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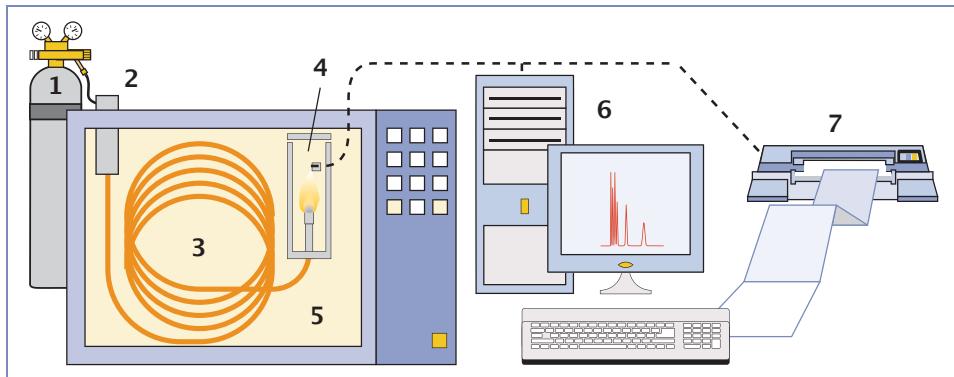
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## The GC system



1 – 5 compose the chromatograph:

1. **gas supplies:** carrier gas and e.g. burner gases for a flame ionisation detector (FID)
2. **sample injector:**  
with **direct injection** the sample is introduced into the column without contact with other parts from glass or metal (on-column injection);  
with **indirect injection** the sample is introduced into an evaporator, and the vapour is then transferred into the column either completely or partially (split technique); both techniques allow low temperatures, high temperatures or temperature programming
3. **capillary column:** the heart of the GC system
4. **detector:** indicates a substance by generation of an electrical signal (response). Some detectors are specific for certain classes of substances or for certain elements (P, N, etc.).
5. **oven** with temperature control

6 and/or 7 serve for evaluation of the separation

6. **data station** for digital evaluation of chromatograms
7. **recorder** for plotting or printing chromatograms

### The separation process

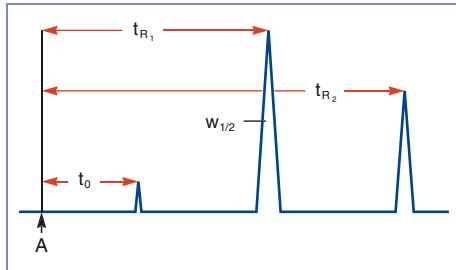
Separation is achieved by repeated distribution of each sample component between two phases: in GC, the **mobile phase** is always a gas (mostly  $N_2$ ,  $H_2$ , He). The **stationary phase** is a mostly viscous gum-like liquid coated to the inner wall of a capillary column (WCOT = Wall Coated Open Tubular). Transport of the analytes is achieved exclusively in the gas phase, separation is accomplished in the stationary phase. The quality of a separation (resolution) depends on how long the components to be separated stay in the stationary phase and on how often they interact with this phase. The type of interaction between component and phase (selectivity) is determined by the functional groups. The polarity of the phase is a function of stationary phase substituents.

# Basic principles of capillary GC



## The chromatogram

A chromatogram consists of a base line and a number of peaks. Peak areas allow quantitative determinations:



A: starting point of a chromatogram = time of injection of a dissolved solute  
A component can be identified by its **retention time** (qualitative determination):

$$t_{R_i} = t_0 + t_{R_i}'$$

**t<sub>0</sub>**: dead time = void time = residence time of a solute in the mobile phase (time required by a component to migrate through the chromatographic system without any interaction with the stationary phase)

**t<sub>Ri</sub>**: retention time = interval between peak i and the point of injection

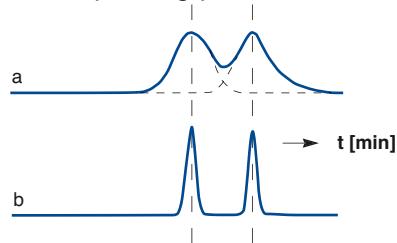
**t<sub>Ri'</sub>**: net retention time = difference between total retention time and dead time t<sub>0</sub>. It indicates how long a substance stays in the stationary phase.

### Other terms characterising a separation:

**k'**: capacity factor: a measure for the position of a sample peak in the chromatogram. The capacity factor is specific for a given compound and constant under constant conditions.

$$k'_i = \frac{t_{R_i} - t_0}{t_0}$$

The relative retention does not provide any information on the quality of a separation, since for equal values of  $\alpha$  two very broad peaks may overlap, (as shown in trace a), or may be completely resolved (as in trace b), if they are correspondingly narrow.



**R**: resolution: a measure for the quality of a separation, taking the peak width at half height ( $w_{1/2}$ ) into account according to

$$R = \frac{t_{R_2} - t_{R_1}}{(w_{1/2})_2 + (w_{1/2})_1}$$

**N<sub>th</sub>**: number of theoretical plates: characterises the quality of a column (should be determined for  $k' > 5$ ). The height equivalent to a theoretical plate (h, HETP) is calculated by dividing the length L of the column by the number of theoretical plates N<sub>th</sub>. The smaller this value the better works the column.

$$N_{th} = 5.54 \cdot \left( \frac{t_{R_i}}{w_{1/2}} \right)^2$$

$$h = HETP = \frac{L}{N_{th}}$$

## MN phases for GC

Phase	Composition	Relative polarity <sup>1</sup>	max. Temperature <sup>2</sup>
OPTIMA® 1	100 % dimethylpolysiloxane		340/360 °C
OPTIMA® 1 MS			
OPTIMA® 1 MS Accent			
OPTIMA® 5	5 % phenyl – 95 % dimethylpolysiloxane		340/360 °C
OPTIMA® 5 MS	5 % diphenyl - 95 % dimethylpolysiloxane		340/360 °C
OPTIMA® 5 MS Accent	silarylene phase equivalent to 5 % diphenyl – 95 % dimethylpolysiloxane		340/360 °C
OPTIMA® δ-3	phase with autoselectivity		340/360 °C
OPTIMA® XLB	phase with optimised silarylene content		340/360 °C
OPTIMA® δ-6	phase with autoselectivity		340/360 °C
OPTIMA® 1301	6 % cyanopropylphenyl – 94 % dimethylpolysiloxane		300/320 °C
OPTIMA® 624	6 % cyanopropylphenyl – 94 % dimethylpolysiloxane		280/300 °C
OPTIMA® 624 LB	as above, low bleed phase		280/300 °C

<sup>1</sup> = nonpolar, = polar properties

<sup>2</sup> first temperature for isothermal operation, second value for short isotherms in a temperature programme (please note, that for columns with 0.53 mm ID and for columns with thicker films temperature limits are generally lower)

## Summary



	Structure	USP	Phases which provide a similar selectivity based on chemical and physical properties	
	$\left[ \begin{array}{c} \text{CH}_3 \\   \\ \text{O} - \text{Si} - \text{C}(\text{CH}_3)_2 \\   \\ \text{CH}_3 \end{array} \right]_n$	G1	PERMABOND® SE-30, OV-1, DB-1, SE-30, HP-1, SPB-1, CP-Sil 5 CB, Rtx®-1, 007-1, BP1, MDN-1, AT™-1, ZB-1, OV-101	
		G2		
		G38	Ultra-1, DB-1MS, HP-1MS, Rtx®-1MS, Equity™-1, AT™-1MS, VF-1MS, CP-Sil 5 CB MS	
	$\left[ \begin{array}{c} \text{CH}_3 \\   \\ \text{O} - \text{Si} - \text{C}(\text{CH}_3)_2 \\   \\ \text{C}_6\text{H}_5 \end{array} \right]_m \left[ \begin{array}{c} \text{CH}_3 \\   \\ \text{O} - \text{Si} - \text{C}(\text{CH}_3)_2 \\   \\ \text{CH}_3 \end{array} \right]_n$	G27	PERMABOND® SE-52, SE-54, SE-52, DB-5, HP-5, SPB-5, CP-Sil 8, Rtx®-5, 007-5, BP5, MDN-5, AT™-5, ZB-5	
		G36		
	$\left[ \begin{array}{c} \text{C}_6\text{H}_5 \\   \\ \text{O} - \text{Si} - \text{C}(\text{CH}_3)_2 \\   \\ \text{C}_6\text{H}_5 \end{array} \right]_m \left[ \begin{array}{c} \text{CH}_3 \\   \\ \text{O} - \text{Si} - \text{C}(\text{CH}_3)_2 \\   \\ \text{CH}_3 \end{array} \right]_n$	G27		
		G36	DB-5MS, HP-5MS, Ultra-2, Equity™-5, CP-Sil 8CB low bleed/MS, Rtx®-5SIL-MS, Rtx®-5MS, 007-5MS, BPX5, MDN-5S, AT™-5MS, VF-5MS	
	$\left[ \begin{array}{c} \text{C}_6\text{H}_5 \\   \\ \text{O} - \text{Si} - \text{C}(\text{CH}_3)_2 \\   \\ \text{C}_6\text{H}_5 \end{array} \right]_m \left[ \begin{array}{c} \text{CH}_3 & \text{H}_3\text{C} \\   &   \\ \text{O} - \text{Si} - & \text{C}(\text{CH}_3)_2 \\   &   \\ \text{CH}_3 & \text{H}_3\text{C} \end{array} \right]_n \left[ \begin{array}{c} \text{H}_3\text{C} \\   \\ \text{O} - \text{Si} \\   \\ \text{H}_3\text{C} \end{array} \right]_o$	G27		
		G36		
see description on page 10		G49	no similar phases	
$\left[ \begin{array}{c} \text{C}_6\text{H}_5 \\   \\ \text{O} - \text{Si} - \text{C}(\text{CH}_3)_2 \\   \\ \text{C}_6\text{H}_5 \end{array} \right]_m \left[ \begin{array}{c} \text{CH}_3 & \text{H}_3\text{C} \\   &   \\ \text{O} - \text{Si} - & \text{C}(\text{CH}_3)_2 \\   &   \\ \text{CH}_3 & \text{H}_3\text{C} \end{array} \right]_n \left[ \begin{array}{c} \text{H}_3\text{C} \\   \\ \text{O} - \text{Si} \\   \\ \text{H}_3\text{C} \end{array} \right]_o$	-	DB-XLB, Rtx®-XLB, MDN-12, VF-XMS		
see description on page 10		-	no similar phases	
$\left[ \begin{array}{c} \text{C}_6\text{H}_5 \\   \\ \text{O} - \text{Si} - \text{C}(\text{CH}_3)_2 \\   \\ \text{NC} - (\text{CH}_2)_3 \end{array} \right]_m \left[ \begin{array}{c} \text{CH}_3 \\   \\ \text{O} - \text{Si} - \text{C}(\text{CH}_3)_2 \\   \\ \text{CH}_3 \end{array} \right]_n$	G43	HP-1301, DB-1301, SPB-1301, Rtx®-1301, CP-1301, 007-1301		
		G43	HP-624, HP-VOC, DB-624, DB-VRX, SPB-624, CP-624, Rtx®-624, Rtx®-Volatiles, 007-624, BP624, VOCOL	
For special phases and phases tested for specific applications see table on page 21				

## MN phases for GC

Phase	Composition	Relative polarity <sup>1</sup>	max. Temperature <sup>2</sup>
OPTIMA® 1701	14 % cyanopropylphenyl – 86 % dimethylpolysiloxane		300/320 °C
OPTIMA® 35 MS	silarylene phase with selectivity similar to a 35 % diphenyl – 65 % dimethylpolysiloxane phase		360/370 °C
OPTIMA® 17	phenylmethylpolysiloxane, 50 % phenyl		320/340 °C
OPTIMA® 210	trifluoropropylmethylpolysiloxane (50 % trifluoropropyl)		260/280 °C
OPTIMA® 225	50 % cyanopropylmethyl – 50 % phenylmethylpolysiloxane		260/280 °C
OPTIMA® 240	33 % cyanopropylmethyl – 67 % dimethylpolysiloxane		260/280 °C
OPTIMA® WAX	polyethylene glycol 20 000 daltons		250/260 °C
OPTIMA® FFAP	polyethylene glycol 2-nitroterephthalate		250/260 °C

<sup>1</sup>  = nonpolar,  = polar properties

<sup>2</sup> first temperature for isothermal operation, second value for short isotherms in a temperature programme (please note, that for columns with 0.53 mm ID and for columns with thicker films temperature limits are generally lower)

## Summary



	<b>Structure</b>	<b>USP</b>	Phases which provide a similar selectivity based on chemical and physical properties
	$\left[ \begin{array}{c} \text{C}_6\text{H}_5 \\   \\ \text{O} - \text{Si} - \text{C}_6\text{H}_5 \\   \\ \text{NC} - (\text{CH}_2)_3 \end{array} \right]_m \quad \left[ \begin{array}{c} \text{CH}_3 \\   \\ \text{O} - \text{Si} - \text{CH}_3 \\   \\ \text{CH}_3 \end{array} \right]_n$	G46	OV-1701, DB-1701, CP-Sil 19 CB, HP-1701, Rtx®-1701, SPB-1701, 007-1701, BP10, ZB-1701
	$\left[ \begin{array}{c} \text{C}_6\text{H}_5 \\   \\ \text{O} - \text{Si} - \text{C}_6\text{H}_5 \\   \\ \text{C}_6\text{H}_5 \end{array} \right]_m \quad \left[ \begin{array}{c} \text{CH}_3 & \text{H}_3\text{C} \\   &   \\ \text{O} - \text{Si} - \text{C}_6\text{H}_4 - \text{Si} - \text{CH}_3 &   \\   &   \\ \text{CH}_3 & \text{H}_3\text{C} \end{array} \right]_n \quad \left[ \begin{array}{c} \text{H}_3\text{C} \\   \\ \text{O} - \text{Si} - \text{H}_3\text{C} \end{array} \right]_o$	G42	DB-35 MS, HP-35, SPB-35, Rtx-35, 007-35, BPX-35, MDN-35, AT-35 MS, ZB-35, OV-11, VF-35 MS
	$\left[ \begin{array}{c} \text{CH}_3 \\   \\ \text{O} - \text{Si} - \text{C}_6\text{H}_5 \\   \\ \text{C}_6\text{H}_5 \end{array} \right]_m$	G3	OV-17, DB-17, HP-50+, HP-17, SPB-50, SP-2250, Rtx®-50, CP-Sil 24 CB, 007-17, ZB-50
	$\left[ \begin{array}{c} \text{CH}_3 \\   \\ \text{O} - \text{Si} - \text{CH}_3 \\   \\ \text{F}_3\text{C} - (\text{CH}_2)_2 \end{array} \right]_n$	G6	OV-210, DB-210, Rtx®-200, 007-210
	$\left[ \begin{array}{c} \text{CH}_3 & \text{CH}_3 \\   &   \\ \text{O} - \text{Si} - \text{O} - \text{Si} - \text{C}_6\text{H}_5 \\   &   \\ \text{NC} - (\text{CH}_2)_3 & \text{C}_6\text{H}_5 \end{array} \right]_n$	G7 G19	DB-225, HP-225, OV-225, Rtx®-225, CP-Sil 43, 007-225, BP225
	$\left[ \begin{array}{c} \text{CH}_3 \\   \\ \text{O} - \text{Si} - \text{CH}_3 \\   \\ \text{NC} - (\text{CH}_2)_3 \end{array} \right]_m \quad \left[ \begin{array}{c} \text{CH}_3 \\   \\ \text{O} - \text{Si} - \text{CH}_3 \\   \\ \text{CH}_3 \end{array} \right]_n$	–	no similar phases
	$\text{H} \left[ \begin{array}{c} \text{H} & \text{H} \\   &   \\ \text{O} - \text{C} - \text{C} - \text{OH} \\   &   \\ \text{H} & \text{H} \end{array} \right]_n$	G16	PERMABOND® CW 20 M, DB-Wax, Supelcowax™, HP-Wax, HP-INNOWax, Rtx®-Wax, CP-Wax 52 CB, Stabilwax, 007-CW, BP20, AT™-Wax, ZB-Wax
	$\left[ \begin{array}{c} \text{O} & \text{O} \\    &    \\ \text{C} - \text{C}_6\text{H}_4 - \text{C} - (\text{OCH}_2\text{CH}_2)_m - \text{O} \\    \\ \text{O}_3\text{N} \end{array} \right]_n$	G25 G35	PERMABOND® FFAP, DB-FFAP, HP-FFAP, CP-SIL 58 CB, 007-FFAP, CP-FFAP CB, Nukol
	For special phases and phases tested for specific applications see table on page 21		

# Phase selection in GC

Selecting the proper stationary phase is the most important decision when selecting a capillary column for a given separation task. In this chapter you will find some guidelines and concepts that simplify the process. There are four major column parameters (primary selection features) and four hardware-and-problem adapted secondary selection features:

## Primary selection features:

- ◎ phase polarity
- ◎ film thickness
- ◎ inner diameter
- ◎ column length

## Secondary selection features:

- ◎ temperature stability
- ◎ deactivation
- ◎ bleeding
- ◎ special selectivities

## Phase polarity

Phase selectivity is determined by the physicochemical interactions of the solute molecules with the stationary phase.

### ◎ General rule: only an adequate polarity of the GC phase leads to satisfying separation and peak shape

**Use similar polarities for phase and target compounds** (e. g. nonpolar molecules require nonpolar polysiloxane phases in the column).

MN offers more than 40 different phases for gas chromatography, from very nonpolar to polar columns.

◎ **Nonpolar** stationary phases, starting with 100% dimethylpolysiloxane, separate by volatility (i. e. boiling point) only.

Typical nonpolar phases:  
OPTIMA® 1 or OPTIMA® 5

Typical analytes:  
linear hydrocarbons (*n*-alkanes).

For structures and relative polarities of MN OPTIMA® phases please refer to the table on the preceding pages.

◎ **Mid-polar** phases offer additional interactions, which may improve a separation. With increasing polarity, e. g. by introducing phenyl and/or cyanopropyl groups, the separation is increasingly influenced by differences in dipole moment and by charge transfer.

Typical mid-polar phase:  
OPTIMA® 1701

Typical analytes:  
molecules which can be polarised (e. g. aromatic compounds).

◎ For **polar** components featuring medium to strong hydrogen bonding capacities, polyethylene glycol phases (WAX) are the best choice for a separation.

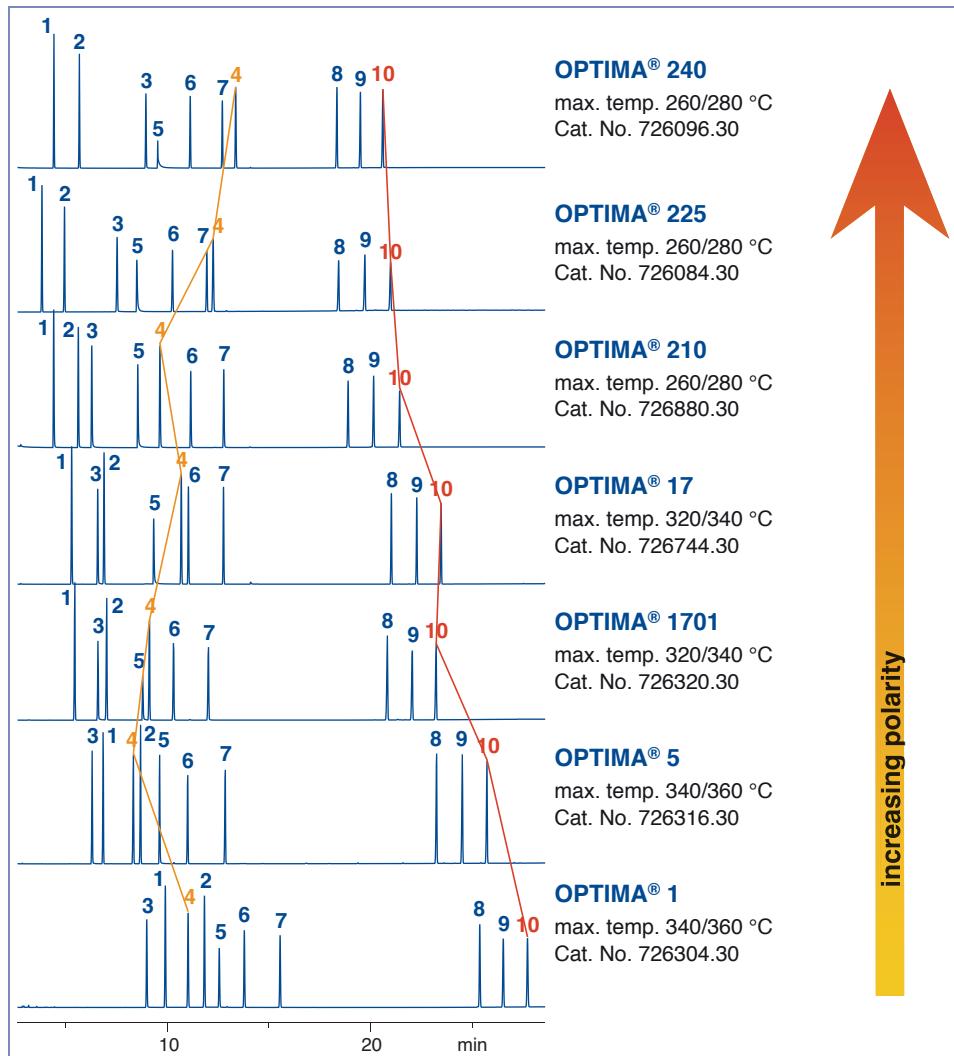
Typical polar phase:  
OPTIMA® WAX

Typical analytes:  
alcohols and carboxylic acids.

# Phase polarity



## Comparison of separation properties of selected OPTIMA® phases



All columns: 0.5 µm film, 30 m x 0.32 mm ID  
 Sample: MN-OPTIMA® test mixture (REF 722316)  
 Injection: 1.0 µl, split 1: 50  
 Carrier gas: 80 kPa N<sub>2</sub>  
 Temperature: 80 °C → T<sub>max</sub> (isothermal), 8 °C/min  
 Detector: FID, 260 – 300 °C

**Peaks:**

1. Undecane	6. Methyl decanoate
2. Dodecane	7. Methyl undecanoate
3. Octanol	8. Henicosane
4. Dimethylaniline	9. Docosane
5. Decylamine	10. Tricosane

## Phase selection in GC

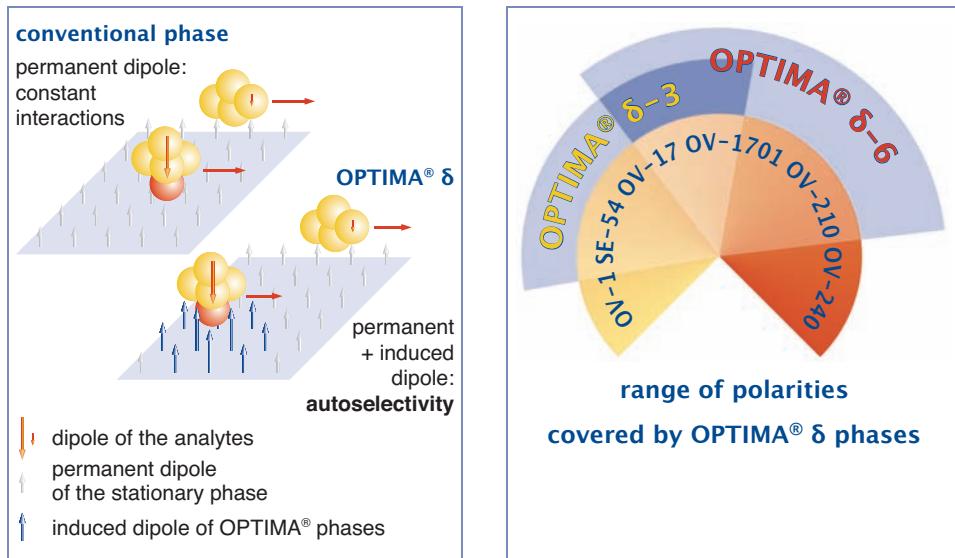
### Autoselectivity · OPTIMA® δ phases

The polarity of all standard phases is defined by their composition.

In conventional mid-polar phases an increased polarity is achieved by an increase of the phenyl content in the dimethylpolysiloxane or by adding e. g. cyanopropyl or trifluoromethyl groups to the dimethylpolysiloxane often resulting in an increased tendency for bleeding.

**OPTIMA® δ phases were developed to adjust themselves to the polarity of the target compounds: with non-polar compounds the phase reacts nonpolar, for polar compounds the phase increases its polarity.**

The OPTIMA® δ phases consist of cross-linked polysiloxane block polymers with defined composition, exclusively produced for MN. Polar molecules are able to induce a dipole moment in the stationary phase, so that the analytes show stronger interactions. We call this phenomenon autoselectivity, because the stationary phase adjusts itself to the polarity of the analytes. Thus OPTIMA® δ phases cover broader ranges of polarity. Due to their structure, OPTIMA® δ phases show very high temperature limits of 340/360 °C and low bleed levels, making them well suited for MSD or PND.



Isomeric phenols are difficult to analyse on standard phases due to coelutions. The autoselective OPTIMA® δ-3 readily separates all of 22 isomeric phenols as shown in application 250060 on page 61.

## Primary selection features



### Polarity · Most frequently used phases

Selectivity has to be optimized for the critical pair of components or the main component.

**You should always select the least polar column which solves your separation task.**

About 70% of all GC separations can be performed on non- to mid-polar columns.

In fact, 5-type phases (5 % phenyl – 95 % methylpolysiloxane) are the most commonly used GC phases in the world, because they generally feature high temperature stability, low column bleeding and good deactivation.

The second most common phases are WAX and/or FFAP. They represent the other end of the polarity range and are well suited for all compounds with strong hydrogen bonding capacity (e. g. compounds with OH, COOH or  $\text{NH}_x$  functionalities).

Mid-polar phases like 1301, 35, 17, 1701, 210 and 225 feature alternative polarities for special separations or for confirming analytical results.

### Film thickness

Film thickness of MN capillary columns reaches from 0.1 to 5.0  $\mu\text{m}$ . Standard film thickness is 0.25  $\mu\text{m}$ . Thin films (0.1 – 0.2  $\mu\text{m}$ ) are very well suited for high-boiling or temperature labile compounds, fast separations, or very closely eluting substances.

Increasing film thickness will increase the capacity, the retention time for low-boiling compounds, and improve inertness. This is especially useful for samples with widely differing concentrations, or for the separation of volatile polar substances.

Better coverage of the column wall by a thicker film and a reduction of the column surface by reducing column length is favourable for extremely active substrates, which in many cases cause noticeable tailing, if they come in contact with uncoated spots of the column wall.

Thick films also mean more phase in the column, and consequently higher bleeding. This results in lower maximum operating temperatures for thick film columns. In addition, thick film columns may have a lower efficiency. Variation of the film thickness is often better than increasing the column length as may be seen in applications 200030 and 200041 page 196.

# Phase selection in GC

## Inner diameter

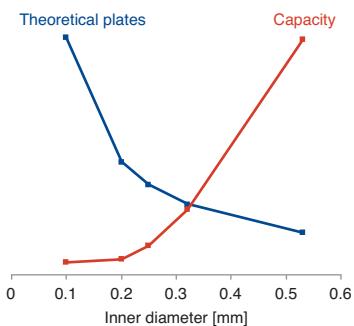
The lower the ID of a GC column, the higher is the theoretically possible number of plates per meter.

- ⌚ **0.1 – 0.2 mm ID**  
for high resolution and short retention times with low carrier gas flows (Fast GC)
- ⌚ **0.25 mm ID**  
all-purpose columns for analyses of complex mixtures
- ⌚ **0.32 mm ID**  
for routine analyses with short retention times, but increased capacity
- ⌚ **0.53 mm ID**  
for rapid separations with inert surface and highest capacity

## Plate number as a function of ID

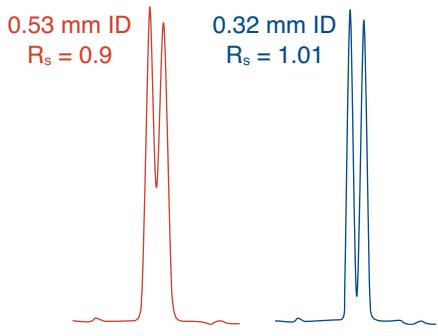
Column ID [mm]	Theoretical plates/m
0.10	12500
0.20	5940
0.25	4750
0.32	3710
0.53	2240

## Plate number and capacity as function of inner diameter



## Influence of inner diameter on resolution

Column: 30 m, identical film thickness



Decreasing the inner diameter and thus increasing resolution is useful for high speed/Fast GC procedures, but some side effects have to be considered:

- ⌚ retention time increases as well (at identical head pressure)
- ⌚ the back pressure of a small ID column increases due to restriction
- ⌚ the capacity of columns decreases (the loadability decreases, see table on previous page)
- ⌚ the hardware requirements for injection systems (high pressure, high speed of injection) and detectors (highest speed of detection) increase

Because of this, GC procedures can not always take profit from the increase in plate numbers!

## Primary column parameters



### Sample capacity (loadability) of a 30 m column in ng

Film thickness [µm]	Inner diameter [mm]			
	0.2	0.25	0.32	0.53
0.10	20 – 35	25 – 50	35 – 75	50 – 100
0.25	35 – 75	50 – 100	75 – 125	100 – 250
0.50	75 – 150	100 – 200	125 – 250	250 – 500
1.00	150 – 250	200 – 300	250 – 500	500 – 1000
3.00		400 – 600	500 – 800	1000 – 2000
5.00		1000 – 1500	1200 – 2000	2000 – 3000

### Phase ratio of capillary columns

Column length (L) and retention times (for identical flow and pressure parameters) show a linear relation; resolution is proportional to the square root of L. If other column parameters (ID, film thickness) have to be adapted – for example in case of a new GC with different dimension requirements – a comparable separation can only be achieved with columns with similar phase ratio!

In general a higher phase ratio implies less retention power and shorter retention times and vice versa.

#### Calculation of the phase ratio:

$$\beta = \frac{r}{2 d_f}$$

with r = column radius and  $d_f$  = film thickness

### Phase ratio as a function of inner diameter and film thickness

↓ ID \ $d_f \rightarrow$	0.1	0.2	0.25	0.35	0.5	1	1.5	2	3	5
0.1	250	125.0	100	71.43	50	25.0	16.67	12.50	8.3	5.0
0.2	500	250.0	200	142.86	100	50.0	33.33	25.00	16.7	10.0
0.25	625	312.5	250	178.57	125	62.5	41.67	31.25	20.8	12.5
0.32	800	400.0	320	228.57	160	80.0	53.33	40.00	26.7	16.0
0.53	1325	662.5	530	378.57	265	132.5	88.33	66.25	44.2	26.5

# Phase selection in GC

## Column length

Column length is directly proportional to the separation efficiency (number of plates N).

Longer columns show increased resolution, however, R is proportional to the square root of N, i. e. a doubled column length only increases the resolution by a factor of 1.4, while cost and analysis time are proportional to the length.

Routine separations are most frequently performed on 25 or 30 m columns, while complex mixtures may require 50 or 60 m columns. 10 m columns with 0.10 mm ID are used for Fast GC.

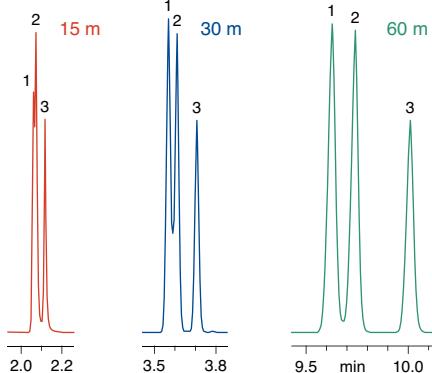
**As a rule of thumb:**  
**use about 1 m column length per component in your mixture.**

### Influence of column length on resolution and retention time

Columns: OPTIMA® 1, 0.25 mm ID, 0.25 µm film

Carrier gas: 1 ml/min He, isothermal

Peaks: FAMEs  
1. C18:2, 2. C18:1, 3. C18



## Fast GC

Characteristics of Fast GC are decreased column diameters, high heating rates and decreased column length for faster GC separations with high resolution efficiency.

- ⌚ small inner diameters combined with fast temperature programs can reduce the analysis time by up to 80%
- ⌚ high heating rates place special demands on stationary phases: OPTIMA® columns meet exactly this requirement, as they show very low bleeding and provide long lifetimes, even when continuously subjected to high heating rates
- ⌚ small inner diameters result in high column inlet pressures and a lower volume flow of the mobile phase, which as a consequence require very fast injection of very small samples against a high pressure
- ⌚ the amount of sample, which can be injected, is limited by the inner diameter and the thin film
- ⌚ high sensitivity detectors with small volume and extremely short response time, as well as a very rapid data acquisition and processing are required

For comparison of a separation on a 50 m standard capillary with a separation on a 10 m fast GC column see application 211260 on page 151.

# Secondary column parameters

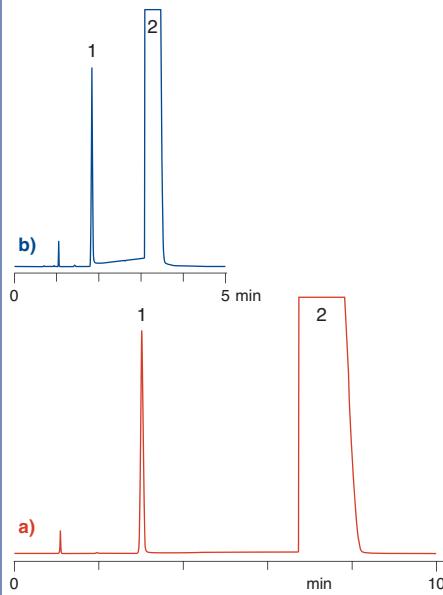


## Temperature stability

High temperature stability of a column is beneficial for high-boiling solutes with very low vapour pressures, which normally have very long retention times and rather broad peak shapes. Since OPTIMA® columns can be operated at increased temperatures, they elute high-boiling compounds faster and with better peak shapes.

### Improved peak shape at higher temperatures

Injection: 0.6 µl, split 1:100  
Carrier gas: 0.4 bar He  
a) Temperature: **100 °C** isothermal  
width of peak 2: 1.4 min  
b) Temperature: **130 °C** isothermal  
width of peak 2: 0.5 min



### Maximum operating temperatures for OPTIMA® phases

OPTIMA® phase	max. Temp. [°C]
<b>1</b>	340/360
<b>1 MS</b>	340/360
<b>1 MS Accent</b>	340/360
<b>5</b>	340/360
<b>5 MS</b>	340/360
<b>5 MS Accent</b>	340/360
<b>8-3</b>	340/360
<b>XLB</b>	340/360
<b>8-6</b>	340/360
<b>1301</b>	300/320
<b>624 / 624 LB</b>	280/300
<b>1701</b>	300/320
<b>35 MS</b>	360/370
<b>17</b>	320/340
<b>210</b>	260/280
<b>225</b>	260/280
<b>240</b>	260/280
<b>WAX</b>	250/260
<b>FFAP</b>	250/260

\* first temperature for isothermal operation, second value for short isotherms in a temperature programme (please note, that for columns with 0.53 mm ID and for columns with thicker films temperature limits are generally lower)



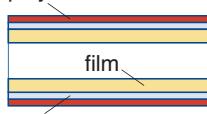
# Phase selection in GC

## Deactivation

The quality of deactivation in GC columns is influenced by

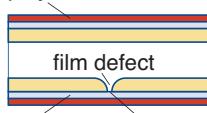
- ⌚ the deactivation of the glass tube itself
- ⌚ the binding process of the polysiloxane or polyethylene glycol to the glass wall
- ⌚ the composition and thickness of the film
- ⌚ the constancy / quality of the coverage of the column wall

polyimide



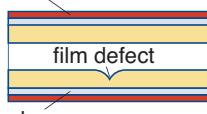
**optimal column**  
with even film and  
perfectly covered  
walls

polyimide



**column with film defect** causing an active area, where analytes can interact with the glass wall, resulting in tailing

polyimide



**thicker films** help to avoid active areas, even when the film is not perfectly smooth resulting in less or no tailing

The possibility of film defects increases with increasing column length and decreasing film thickness. Thus especially for polar compounds thin film columns may cause problems like tailing or deformed peaks due to uncontrolled interactions with active spots on the column wall.

## Quality control of OPTIMA® columns

The deactivation process of OPTIMA® columns is optimized for excellent chromatographic performance, improved peak shape, efficiency and sensitivity.

Performance of each GC column from MN is tested by stringent quality control procedures including

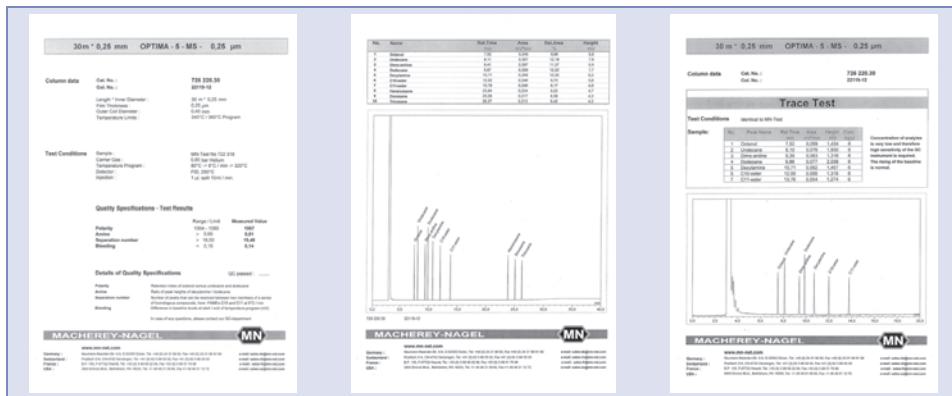
- ⌚ **column efficiency** by measuring the separation number (number of resolved peaks between two members of a series of homologue compounds, in this case FAMEs C11 and C12) in a temperature programme (8 °C/min)
- ⌚ **polarity** by measuring the retention index of octanol versus decane and undecane
- ⌚ **bleeding** as difference in baseline values at start and end of the temperature programme
- ⌚ **inertness** by measuring the peak height ratio for decylamine/undecane (for non- to mid-polar phases)

All MN columns are supplied with test chromatograms of standard test mixtures stating the actual performance of the column (MS columns additionally include the chromatogram of a trace test diluted 1:100). Additionally, we include the corresponding test mixture.

# Secondary column parameters



## Typical test chromatograms of an OPTIMA® column

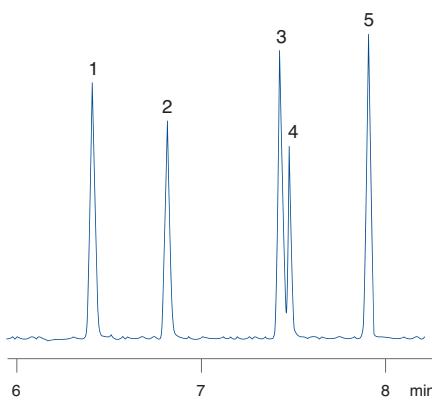


## Examples of OPTIMA® column performance and deactivation

**Column:** OPTIMA® 35 MS,  
30 m x 0.25 mm ID,  
0.25 µm film  
**Injection:** 1 µl, split 1:20, **1 ng/peak**  
**Carrier gas:** 1.0 ml/min He  
**Temperature:** 80 °C  $\xrightarrow{10\text{ °C/min}}$  320 °C (10 min)  
**Detector:** MSD

### Peaks:

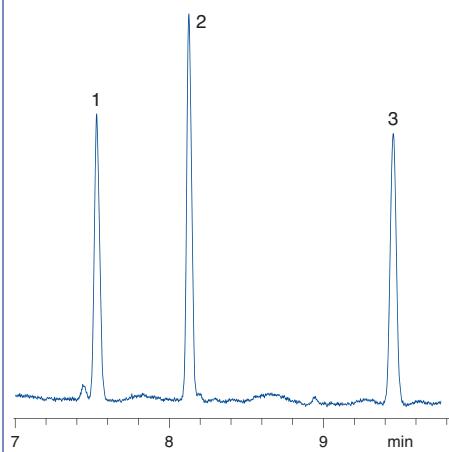
1. *o*-Anisidine
2. 4-Chloroaniline
3. *p*-Cresidine
4. 2,4,5-Trimethylaniline
5. 4-Chloro-*o*-toluidine



**Column:** OPTIMA® 5 MS Accent,  
30 m x 0.25 mm ID,  
0.25 µm film  
**Injection:** 1 µl, split 1:50, **0.2 ng/peak**  
**Carrier gas:** 80 kPa He  
**Temperature:** 80 °C  $\xrightarrow{8\text{ °C/min}}$  360 °C  
**Detector:** MSD

### Peaks:

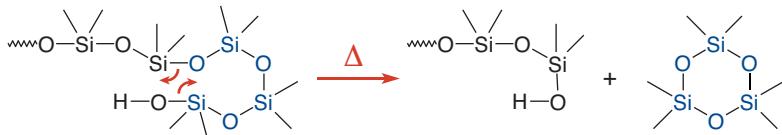
1. Octanol
2. Undecane
3. Dimethylamine



## Phase selection in GC

### Column bleeding

Degradation of a methylpolysiloxane chain forming hexamethylcyclotrisiloxane



Column bleeding is a thermal degradation of the siloxane polymer caused by splitting of siloxane bonds with lower bond energies (see formula above).

Bleeding is accelerated near the upper temperature limit of the stationary phase (normally above 180 °C). In the presence of oxygen or water bleeding even starts at much lower temperatures. This is why the purity of the carrier gas is very important, especially when working at high temperatures.

Column bleed increases as column length increases, because longer columns contain more of the stationary phase. However, don't let this fact discourage you to use a longer column if necessary.

Column bleed also increases as film thickness increases. Since thicker films are more retentive, later eluting peaks may shift into a region of much higher column bleed when increasing film thickness as is shown in the chromatogram below.

#### Column bleeding at elevated temperature

Column: OPTIMA® 5 Amine, 30 x 0.32 mm ID, 1.5 µm film, REF 726356.30,  
max. temperature 300/320 °C

Injection: 1 µl, split 1:35

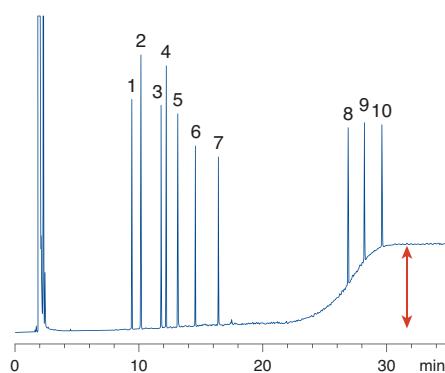
Carrier gas: 1.5 bar He

Temperature: 80 °C  $\xrightarrow{8\text{ °C/min}}$  320 °C (10 min)

Detector: FID

#### Peaks:

1. Octanol
2. Undecane
3. Dimethylaniline
4. Dodecane
5. Decylamine
6. Methyl decanoate
7. Methyl undecanoate
8. Henicosane
9. Docosane
10. Tricosane



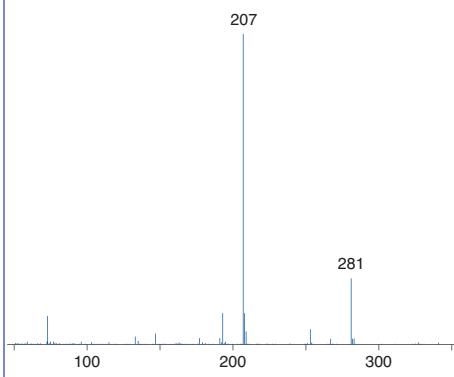
## Secondary column parameters



As is shown in the mass spectrum below, the major “bleed” ion from a 5-type phase is  $m/z = 207$  resulting from hexamethylcyclotrisiloxane ( $D_3$ ), the second important ion  $m/z = 281$  results from the cyclic degradation product with 4 units ( $D_4$ ).

### Mass spectrum of a 5-type phase

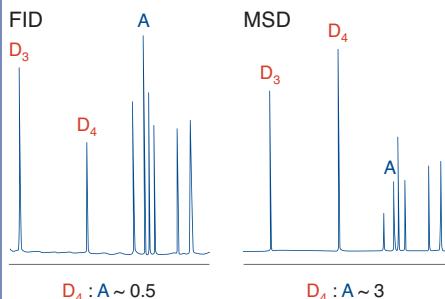
320 °C, TIC, 50 – 500 amu



### Influence of the type of detector

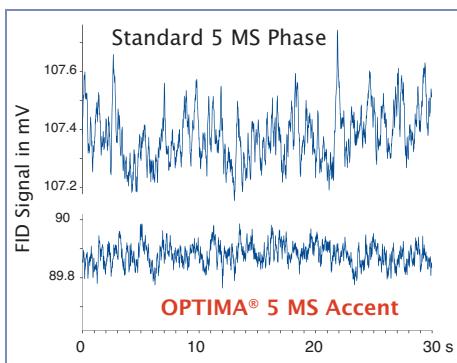
The impact of bleeding on the signal to noise ratio of a chromatogram is much more pronounced for ion trap MS detectors than for FID detectors as is shown in the graphs below. With FID detection, the ratio of  $D_4$  to the analyte peak A is about 0.5; with MS detection it is about 3! This is why columns with low bleed characteristics are especially important for use with MS detection.

### Chromatogram of the same sample analysed with FID and MSD



### Benefits of low bleed columns in GC

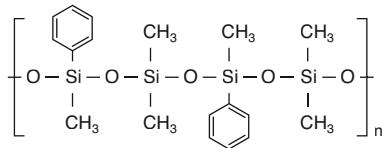
- Low bleed columns allow
- ⌚ higher operating temperatures resulting in shorter run times
  - ⌚ less pollution of the detection system
  - ⌚ improved detectability of solutes in GC/MS analyses
  - ⌚ increased detectability due to an improved signal to noise ratio as is shown in the traces at right



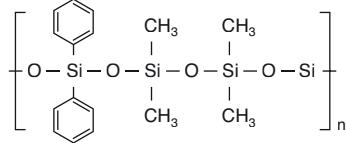
## Phase selection in GC

### Low-bleed GC/MS columns

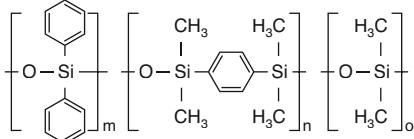
**Standard columns** with 5-type phases contain phenyl and methyl substituted siloxanes in the ratio 5:95 as shown in the structure below



The first generation of low-bleed **GC/MS columns** contains diphenyl siloxane groups which improve bleed characteristics due to steric hindrance of the formation of D<sub>3</sub> and D<sub>4</sub>.



**Silyl ene-type columns** (see figure below) are stabilised by insertion of arylene groups in the siloxane chain, offering lowest bleeding and high inertness even at higher temperatures.

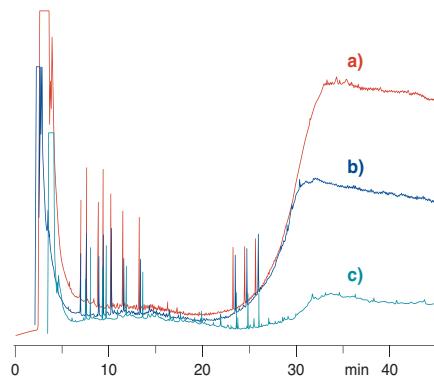


### Comparison of bleeding characteristics of 5-type columns

Columns: 30 m x 0.25 mm ID,  
0.25 µm film  
Injection: 1 µl, split 1:50  
Carrier gas: 80 kPa He  
Temperature: 80 °C → 360 °C  
8 °C/min  
Detector: FID

#### Chromatograms:

- a) conventional 5-type phase
- b) conventional 5-MS phase
- c) OPTIMA® 5 MS Accent



## Special selectivities



### Achiral columns for special separations

Certain analytical separations can be performed more easily with chromatographic columns, which have been especially developed or tested for the respective task. The following table summarises our programme of GC speciality capillaries. Page numbers refer to the cited applications.

Phase composition	Recommended application	Typical application	Page
<b>OPTIMA® 5 Amine</b>			
5% phenyl - 95% methylpolysiloxane especially deactivated	polyfunctional amines such as ethanolamines, amino-functionalised diols	250050 210280	138 140
<b>FS-CW 20 M-AM</b>			
polyethylene glycol, basic, non-immobilised	amines	201520 201530	139 139
<b>PERMABOND® P-100</b>			
dimethylpolysiloxane	petrochemical products	200071	199
<b>PERMABOND® SE-54 HKW</b>			
1% vinyl - 5% phenyl - 94% methylpolysiloxane	volatile halogenated hydrocarbons	212480 200150	40 46
<b>OPTIMA® 1-TG</b>			
dimethylpolysiloxane	triglycerides according to carbon number	200050	197
<b>OPTIMA® 17-TG</b>			
phenyl-methyl-polysiloxane (50% phenyl)	triglycerides according to degree of unsaturation	201800	176
<b>PERMABOND® Silane</b>			
	monomeric silanes and chlorosilanes	200090	148
<b>PERMABOND® CW 20 M-DEG</b>			
polyethylene glycol, tested for diethylene glycol	diethylene glycol in wine	201500	160

## Phase selection in GC

### Chiral columns for enantiomer separation

For chiral columns it is not possible to make a general prediction, which phase could solve a given separation task. Even for compounds with small structural differences the enantio-differentiation can be quite different. For numerous chiral separations – arranged by increasing molecular size – refer to pages 201 – 275. Page numbers in the table below refer to the cited typical applications.

Phase	Composition	max. Temp. [° C]	Typical application	Page
<b>LIPODEX® cyclodextrin phases</b>				
LIPODEX® is patented under EP 0407 412 and US Re. 36.092				
LIPODEX® A	hexakis-(2,3,6-tri-O-pentyl)- $\alpha$ -cyclodextrin	200/220	202851	212
LIPODEX® B	hexakis-(2,6-di-O-pentyl-3-O-acetyl)- $\alpha$ -cyclodextrin	200/220	202861	205
LIPODEX® C	heptakis-(2,3,6-tri-O-pentyl)- $\beta$ -cyclodextrin	200/220	201880	210
LIPODEX® D	heptakis-(2,6-di-O-pentyl-3-O-acetyl)- $\beta$ -cyclodextrin	200/220	202871	227
LIPODEX® E	octakis-(2,6-di-O-pentyl-3-O-butyryl)- $\gamma$ -cyclodextrin	200/220	212761	202
LIPODEX® G	octakis-(2,3-di-O-pentyl-6-O-methyl)- $\gamma$ -cyclodextrin	220/240	250410	259
<b>HYDRODEX cyclodextrin phases</b> , diluted with optimized polysiloxanes				
HYDRODEX $\beta$ -PM	heptakis-(2,3,6-tri-O-methyl)- $\beta$ -cyclodextrin	230/250	202030	272
HYDRODEX $\beta$ -3P	heptakis-(2,6-di-O-methyl-3-O-pentyl)- $\beta$ -cyclodextrin	230/250	201920	266
HYDRODEX $\beta$ -6TBDM	heptakis-(2,3-di-O-methyl-6-O-t-butyldimethyl-silyl)- $\beta$ -cyclodextrin	230/250	250170	274
HYDRODEX $\beta$ -TBDAc	heptakis-(2,3-di-O-acetyl-6-O-t-butyldimethyl-silyl)- $\beta$ -cyclodextrin	220/240	212430	265
HYDRODEX $\gamma$ -TBDAc	octakis-(2,3-di-O-acetyl-6-O-t-butyldimethyl-silyl)- $\gamma$ -cyclodextrin	220/240	212980	262



# The mobile phase in GC



Sometimes it may be difficult to apply the data of a given GC application in reality, because every manufacturer of GC equipment has its own standards and specifications (depending on the country). The physical parameters are fixed, but an adaption has to be made.

Sometimes an older application uses different or out-dated parameters which are no longer in use. The tables and information in this chapter were compiled to separate the "jungle" in order to give practicable professional hints.

## Selection of the optimum carrier gas

For optimum column performance, hydrogen carrier gas offers some strong advantages over helium or nitrogen. Hydrogen yields higher plate numbers (= better resolution) at rapid linear velocities and achieves higher velocities at lower pressures. At the same time, the presence of extra hydrogen is advantageous for flame ionization and other detectors that use hydrogen fuel gas. Nevertheless, in practical work H<sub>2</sub> is not used so often, because it is the gas with the highest price and the highest danger potential!

Helium is not so dangerous, cheaper, with a similar separation efficiency and for this reason the most recommended gas for modern routine analysis. For sure nitrogen has the lowest price, but many separation problems can be avoided or solved directly by switching from N<sub>2</sub> to He or H<sub>2</sub>. The following tables show, that H<sub>2</sub> has the highest gas velocity and the lowest viscosity of all commonly used gases for GC (H<sub>2</sub>, He, N<sub>2</sub>).

We recommend: if a high resolution (e.g. for chiral separations) or a short analysis time is required, use hydrogen!

## Physical parameters of GC gases

Gas	Viscosity *		Linear velocity at 25 °C					
	300 K	500 K	g/mol	m/s	km/h	Factor N <sub>2</sub>	Faktor He	Faktor H <sub>2</sub>
H <sub>2</sub>	9.0	12.7	2.016	1920	6920	3.8	1.4	1
He	20.0	28.4	4.003	1370	4930	2.7	1	0.71
N <sub>2</sub>	17.9		28.014	510	1840	1	0.37	0.26
O <sub>2</sub>			32	480	1720			

\* from Handbook of Chemistry and Physics, 72<sup>nd</sup> Edition

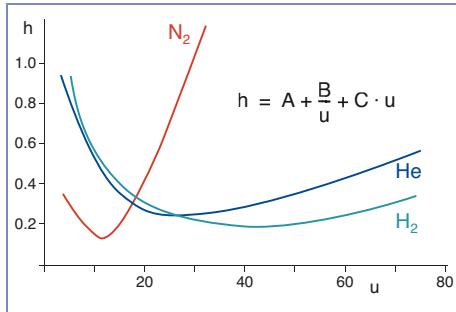
## Optimized working conditions for GC

The table below shows the optimum working conditions for standard columns with common lengths and inner diameters. If you adjust your GC parameters to these values, you can be sure to work in the optimum chromatographic region according to the van Deemter curves. If your parameters are outside these values, you will lose separation efficiency and the results may no longer be ideal (poor separation, analysis time too long, dead time too short).

The short dead times mirror the high speed of H<sub>2</sub> compared to the other gases. If the same inlet pressures are applied for hydrogen as for helium, peaks are eluted in less time, because the linear velocity of H<sub>2</sub> is faster than that of helium. In general, when switching from helium to hydrogen retention times will be roughly cut in half if the inlet pressure is unchanged.

### Plate height and gas velocity

The Van Deemter equation shows how the plate height h depends on the flow velocity u for 3 different GC gases:



- A Eddy diffusion; for WCOT capillary columns  $A = 0$
  - B molecular axial diffusion; B is a function of the diffusion coefficient of the component in the respective carrier gas
  - C resistance to mass transfer
- In practice often higher velocities than  $u_{opt}$  are chosen, if separation efficiency is sufficient, since higher carrier velocities mean shorter retention times.

Gas	Column length [m]		Average velocity [cm/s]	Outlet flow [ml/min]	Head pressure			Dead time [min]
	ID [mm]	[mm]			[kPa]	[psi]	[bar]	
N <sub>2</sub>	10	0.10	8 – 16	0.04 – 0.1	55 – 112	8 – 16	0.5 – 1.1	2.1 – 1.0
	25	0.20	8 – 16	0.14 – 0.33	34 – 69	5 – 10	0.3 – 0.69	5.2 – 2.6
	30	0.25	8 – 16	0.21 – 0.48	26 – 53	3.8 – 7.6	0.26 – 0.53	6.3 – 3.1
	30	0.32	8 – 16	0.33 – 0.72	16 – 32	2.3 – 4.6	0.16 – 0.32	6.3 – 3.1
	30	0.53	8 – 16	0.88 – 1.79	5.8 – 11.7	0.8 – 1.7	0.06 – 0.11	6.3 – 3.1
He	10	0.10	20 – 40	0.14 – 0.45	156 – 330	22 – 48	1.6 – 3.3	0.83 – 0.42
	25	0.20	20 – 40	0.46 – 1.29	95 – 200	14 – 29	0.95 – 1.99	2.1 – 1.0
	30	0.25	20 – 40	0.65 – 1.74	72 – 150	10 – 22	0.7 – 1.5	2.5 – 1.25
	30	0.32	20 – 40	0.95 – 2.29	43 – 89	6.3 – 12.9	0.43 – 0.89	2.5 – 1.25
	30	0.53	20 – 40	2.3 – 4.92	16 – 32	2.3 – 4.6	0.16 – 0.32	2.5 – 1.25
H <sub>2</sub>	10	0.10	30 – 55	0.18 – 0.47	113 – 215	16 – 31	1.1 – 2.1	0.55 – 0.3
	25	0.20	30 – 55	0.62 – 1.43	69 – 130	10 – 19	0.69 – 1.3	1.4 – 0.7
	30	0.25	30 – 55	0.9 – 2.0	53 – 99	7.6 – 14	0.5 – 0.9	1.7 – 0.9
	30	0.32	30 – 55	1.35 – 2.8	32 – 59	4.6 – 8.5	0.32 – 0.59	1.7 – 0.9
	30	0.53	30 – 55	3.35 – 6.5	11.5 – 21.4	1.7 – 3.1	0.12 – 0.21	1.7 – 0.9

Oven temperature 100 °C, split 10:1, injector temperature 175 °C

## Conversion tables



## Pressure conversion chart

Multiply value for unit in the left column by the factor given in the table below the desired unit.

$\downarrow$ Unit →	kPa	bar	atm	psi	$\text{kg cm}^{-2}$	Torr
kPa	1	0.0100	0.00987	0.1450	0.0102	7.52
bar	100	1	0.9869	14.5038	1.0197	751.88
atm	101.32	1.0133	1	14.696	1.0332	760
psi	6.8948	0.06895	0.068	1	0.0703	51.713
$\text{kg cm}^{-2}$	98.06	0.9806	0.9678	14.223	1	735.56
Torr	0.1330	0.00133	0.00132	0.0193	0.00136	1

Examples: to convert 250 bar to psi, multiply 250 by 14.5038 = 3626 psi

to convert 3000 psi to kPa, multiply 3000 by 6.8948 = 20684.4 kPa

## Conversion of concentrations and amounts of sample per injection

The table shows common concentrations and injection volumes for GC. The upper half (blue) is the standard region of detectable concentrations (TCD and FID detectors) and the lower half (yellow) is the region of ultra trace analysis (MS, Ion Trap, Quadrupol MS, ECD).

[%]	Concentration			Injection 1 µl		
	[g/g] [g/ml]	[g/kg] [g/l]	[ppm]	[ppb]	splitless [ng]	split 1: 10 [ng]
100	1	1 000	1 000 000	1 000 000 000	1 000 000	100 000
10	0.1	100	100 000	100 000 000	100 000	10 000
1	0.01	10	10 000	10 000 000	10 000	1 000
0.1	0.001	1	1 000	1 000 000	1 000	100
0.01	0.0001	0.1	100	100 000	100	10
0.001	0.00001	0.01	10	10 000	10	1
		[µg/g] [µg/ml]	[mg/kg] [mg/l]			
0.000 1	1	1	1	1 000	1	0.1
0.000 01	0.1	0.1	0.1	100	0.1	0.01
0.000 001	0.01	0.01	0.01	10	0.01	0.001
0.000 000 1	0.001	0.001	0.001	1	0.001	0.000 1
0.000 000 01	0.000 1	0.000 1	0.000 1	0.1	0.000 1	0.000 01
0.000 000 001	0.000 01	0.000 01	0.000 01	0.01	0.000 01	0.000 001
0.000 000 000 1	0.000 001	0.000 001	0.000 001	0.001	0.000 001	0.000 000 1

## Derivatisation

### The purpose of derivatisation

In gas chromatography it is often advantageous to derivatise polar functional groups (mainly active hydrogen atoms) with suitable reagents. Prerequisite for successful derivatisation is quantitative, rapid and reproducible formation of only one derivative. Aim of this reaction is an improved volatility, better thermal stability or a lower limit of detection due to improved peak symmetry.

The halogen atoms introduced by derivatisation (e. g. trifluoroacetates) allow specific detection (ECD) with the advantage of high sensitivity. Elution orders and fragmentation patterns in mass spectroscopy can be influenced by a specific derivatisation. The derivatisation examples in the table are meant as a first orientation and have to be adjusted or optimized for special problems.

### Selection guide for derivatisation of important functional groups in GC

Function	method	derivative	recommended reagents
<b>Alcohols, Phenols</b>	silylation	R'O – TMS	BSA, MSTFA, MSHFBA, TSIM, SILYL-2110, SILYL-21, SILYL-1139
R'OH	acylation	R'O – CO – R	TFAA, HFBA, MBTFA, MBHFBA
	alkylation	R'O – R	TMSH
ster. hindered	silylation	R'O – TMS	TSIM, BSTFA, SILYL-991
<b>Amines</b>	silylation	R' – NR" – TMS	BSA, MSTFA, MSHFBA, SILYL-991
prim., sec.	acylation	R' – NR" – CO – R	TFAA, HFBA, MBTFA, MBHFBA
hydrochlorides	silylation	R' – NR" – TMS	MSTFA
<b>Amides</b>	silylation	not stable	
	acylation	R' – CO – NH – CO – R	TFAA, MBTFA, HFBA, MBHFBA
<b>Amino acids</b>	silylation	$\begin{array}{c} \text{CO} - \text{O} - \text{TMS} \\   \\ \text{R}' - \text{CH} \\   \\ \text{NH} - \text{TMS} \end{array}$	BSA, BSTFA, MSTFA, MSHFBA
	alkyl. (a) + acyl. (b)	$\begin{array}{c} \text{CO} - \text{O} - \text{R} \\   \\ \text{R}' - \text{CH} \\   \\ \text{NH} - \text{CO} - \text{R} \end{array}$	a) MeOH/TMCS, TMSH b) TFAA, HFBA, MBTFA, MBHFBA
<b>Carboxylic acids</b> (fatty acids)	silylation	R' – CO – O – TMS susceptible to hydrolysis	BSA, MSTFA, MSHFBA, TMCS, TSIM, SILYL-2110, SILYL-21, Silyl-1139
salts	alkylation	R' – CO – O – R	DMF-DMA, MeOH/TMCS (1 M), TMSH
	silylation	R' – CO – O – TMS susceptible to hydrolysis	TMCS
<b>Carbo-hydrates</b>	silylation acylation		MSTFA, TSIM, HMDS, SILYL-1139 TFAA, MBTFA
<b>Steroids</b>	silylation acylation		BSA, TSIM TFAA, MBTFA, HFBA, MBHFBA

# Do's and don'ts in GC



## Water on GC columns

Water causes trouble right from the beginning because of its high expansion volume. This overload effect of the injector is called "backflash" and leads to poor separations and bad peak shapes. Reproducibility is not guaranteed for repeated injections. The table shows the different gas volumes of common GC solvents. Here you find the reason, why nonpolar solvents are always preferred in GC. Even very polar analytes should be injected in nonprotic solvents (if possible).

### Volume expansion of solvents

Solvent	Gas volume [ $\mu\text{l}$ ]
n-Hexane	140
Ethyl acetate	185
Acetone	245
Dichloromethane	285
Acetonitrile	350
Methanol	450
Water	1010

Injection 1  $\mu\text{l}$ , injector temperature 250 °C, pressure 20 psi

**Attention: a water sample can contain a (maybe unknown) concentration of salts. This salt load can damage each column immediately.**

If water cannot be avoided, some points have to be considered:

- ⌚ In order to prevent condensation on the column, the start temperature in the oven should be at least 100 °C or higher for a better peak shape.

- ⌚ Water can extinguish the FID flame or decrease the sensitivity of an ECD.
- ⌚ Because nonpolar GC phases cannot be wetted well by water, a compound with good solubility in water can show poor peak shapes or double peaks.

GC phases which can be used with water samples – without risk of a direct damage – are phases without oxidizable groups, e. g. OPTIMA® 1, 5, 35, 17, Wax, FFAP types and OPTIMA® 8-3 and 8-6.

More critical are 1301, 1701, 225 and 240 types, because the cyano groups can be oxidised or destroyed catalytically (by acids, bases or metals). The phase has still its correct film thickness, but it is chromatographically "dead".

All Cyclodextrin columns are directly destroyed by water, so the samples must be dried before injection. We recommend the use of drying cartridges like CHROMAFIX® Dry (REF 731852).

**Rule: the higher film thickness is, the more stable the column is towards water.**

Water itself is not so critical, but the (possible) ingredients can be (salts, catalysts, acids, bases). When columns have been run with aqueous samples, they should be heated at least at 150 °C in order to remove the water film completely from the column. Lower temperatures have no effect resulting in poor reproducibility.

## Do's and don'ts in GC

Observation / Possible causes	Suggested remedy
<b>No peaks, no gas flow</b>	
detector has no power	check detector power supply and cables
no FID flame	check FID; reignite it
syringe defective / clogged	use a different syringe or clean it
temperature too low for the analytes	check temperature programme, oven temperature (external thermometer)
detector / software / computer hardware failure	check integrator, cables; restart computer
no gas flow	check gas tubes, valves, seals; test gas flow; shorten front of GC column, change injection septum
column connection leaks	use new ferrules
broken GC column	if breakage is at the beginning or at the end, remove the short piece; breakage in the middle can be mended with a glass connector; for multiple breakages replace column

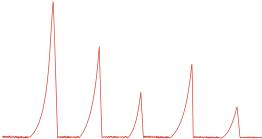
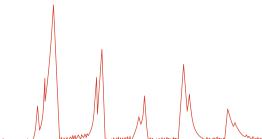
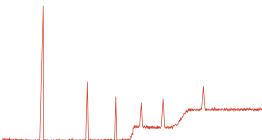
### Tailing



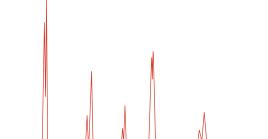
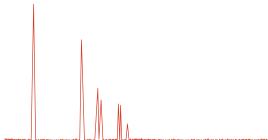
sample vaporises too slowly, not evenly or condenses	increase injector temperature (consider max. temperature limits of the column)
high-boiling analytes	derivatise polar, basic or high boiling compounds
system leaks	check column installation; search for leaks; replace ferrules
analytes coelute	change temperature programme or use column with different selectivity
sample decomposes	check temperature programme, oven temperature (external thermometer); if analytes are not temperature-stable, reduce injector temperature; replace liner by a deactivated one
column absorbs or decomposes analytes	check capillary ends; check intact deactivation using the test mixture; for poor results shorten both column ends by about 10 cm; or replace column; if column test does not show any defects: a) use a column with thicker film b) use phase with better deactivation c) use column with special selectivity
split rate too low	increase split rate
analytes always tending to tail	no chance for symmetric peaks

# GC troubleshooting



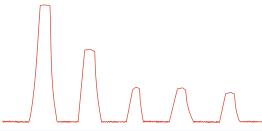
Observation / Possible causes	Suggested remedy
<b>Fronting</b>	
column overload	decrease injection volume; dilute sample
sample vaporises too slowly, not evenly or condenses	increase injector temperature (consider max. temperature limits of the column)
analytes coelute	change temperature programme or use column with different selectivity
sample decomposes	check temperature programme, oven temperature (external thermometer); if analytes are not temperature-stable, reduce injector temperature; replace liner
column absorbs or decomposes analytes	check capillary ends; check intact deactivation using the test mixture; for poor results shorten both column ends by about 10 cm; or replace column; if column test does not show any defects: a) use a column with thicker film b) use phase with better deactivation c) use column with special selectivity
<b>Small peaks on fronting or tailing of bigger peaks</b>	
column not properly installed	check capillary ends; check tight and correct fit in injector and detector
temperature of injection too low	check injector temperature; if analytes are stable, increase temperature
solvent not compatible with GC phase	change solvent
splitter defect	measure flow and adjust splitter
poorly deactivated column, film thickness too low	check capillary ends; check intact deactivation using the test mixture; for poor results shorten both column ends by about 10 cm; or replace column
<b>Plateaus at certain temperatures</b>	
steps in temperature programme too drastic	avoid very short and strong heating periods

## Do's and don'ts in GC

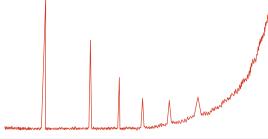
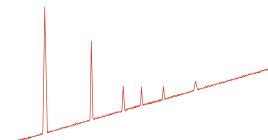
Observation / Possible causes	Suggested remedy
<b>Double peaks, doubled peak tops</b>	
solvent and column not compatible	change solvent or use guard column
solvent mixtures with large differences in boiling point and polarity	use just one solvent
sample decomposes	check temperature programme, oven temperature (external thermometer); if analytes are not temperature-stable, reduce injector temperature; replace liner by a deactivated one
analytes coelute	modify temperature programme or use longer column; possibly change column polarity
detector overload	inject less; control make-up flow
<b>Missing or overlapping peaks, poor separation efficiency</b>	
syringe defective / clogged	use a different syringe or clean it
sample too diluted	increase injection volume; concentrate sample
sample concentration too high	decrease injection volume; dilute sample
column connection leaks, column not properly installed	check column installation; search for leaks; replace ferrules
perforated injection septum	replace septum
injector temperature too low	check temperature programme; increase injector temperature
sample decomposes in the injector	check temperature programme; reduce injector temperature; replace liner; check capillary ends
column oven too hot	check temperature programme, oven temperature (external thermometer); decrease temperature
wrong flow rate	measure flow and correct it if necessary
column absorbs or decomposes analytes	check capillary ends; check intact deactivation using the test mixture; for poor results shorten both column ends by about 10 cm; or replace column; if column test does not show any defects: a) use a column with thicker film b) use phase with better deactivation c) use column with special selectivity

# GC troubleshooting



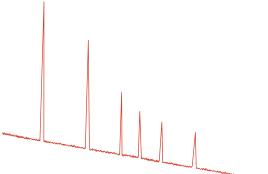
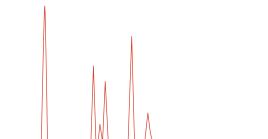
Observation / Possible causes	Suggested remedy
<b>Broad peaks</b>	
poor focussing	decrease start temperature of the programme
flow too high or too low	measure flow, control and adjust it if necessary
split rate too low	increase split rate
column overloaded	decrease injection volume, dilute sample or increase split flow
no solvent focussing effect	decrease oven temperature or use solvent with higher boiling point
<b>Cut tops of peaks, broad peaks</b>	
detector overload	decrease injection volume; dilute sample; increase the split flow
column overload	decrease injection volume; increase split flow
zero point is outside the display	change scale
<b>Strong noise, waves</b>	
leak at column entrance or injection septum	check column installation; search for leaks; replace ferrules
bleeding of septum / injector contaminated	make a run with lower injector temperature; if the baseline improves, replace liner, use low bleed or high temperature septa
septum particles in column entrance	cut 1 turn from column entrance; replace injection septum
column contaminated	cut two turns from column entrance; rinse column with solvent (only chemically bonded phases); otherwise replace column or use guard column
column not properly conditioned	condition column according to manufacturers' instructions (while column is not connected to the detector)
hardware defect	check temperature programme, oven temperature (external thermometer); contact your GC manufacturer
detector contaminated	clean detector
increase of temperature too fast	decrease temperature gradient and end temperature
poor gas quality	use gas grades recommended for GC; for longer supply lines from gas source to GC use gas purification cartridges directly connected to the GC

## Do's and don'ts in GC

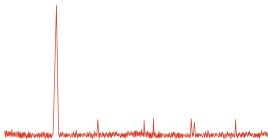
Observation / Possible causes	Suggested remedy
<b>Increasing baseline, at high temperature bleeding or noise</b>	
septum particles in column entrance	cut one turn from column entrance; replace injection septum
column contaminated	cut two turns from column entrance; rinse column with solvent (only chemically bonded phases); otherwise replace column or use guard column
increase of temperature too fast / end temperature too high	decrease temperature gradient and end temperature
column not properly conditioned	condition column according to manufacturers' instructions (while column is not connected to the detector)
detector contaminated	clean detector
poor gas quality	use gas grades recommended for GC; for longer supply lines from gas source to GC use gas purification cartridges directly connected to the GC
film thickness in column too high	use column with thinner film
column stability not sufficient for detector sensitivity	use special low bleed column types
<b>Constantly rising baseline</b>	
leak at column entrance or injection septum	check column installation; search for leaks; replace ferrules
bleeding of septum / injector contaminated	make a run at lower injector temperature; if the baseline improves, replace liner, use low bleed or high temperature septa
septum particles in column entrance	cut one turn from column entrance; replace injection septum
column contaminated	cut two turns from column entrance; rinse column with solvent (only chemically bonded phases); otherwise replace column or use guard column
detector contaminated	clean detector
increase of temperature too fast	decrease temperature gradient and end temperature
poor gas quality	use gas grades recommended for GC; for longer supply lines from gas source to GC use gas purification cartridges directly connected to the GC

# GC troubleshooting



Observation / Possible causes	Suggested remedy
<b>Constantly declining baseline</b>	
gas flow changes with temperature gradient	check gas content in gas cylinder; pressure must be a few bar above the required pressure at max. temperature; otherwise exchange gas cylinder
<b>Short lifetime, poor resolution, lack of separation efficiency</b>	
impurities on the column	cut two turns from column entrance; rinse column with solvent (only chemically bonded phases); otherwise replace column or use guard column
contamination from vials / septa or sample preparation	check SPE and / or autosampler vials; use low bleed or high temperature septa
polymerisation on the column	use guard column (at least 10 m)
separation efficiency decreases for repeated injections, improves after reconditioning	use guard column; reduce injection volume; modify temperature programme; for repeated injections increase end temperature (if possible) and use longer temperature programme
temperature too high / temperature increase too fast	decrease oven temperature and / or temperature gradient (should not be higher than 25 °C/min)
cooling too fast	do not open oven door at high temperatures
temperature too low / condensation	increase injector temperature and/or start temperature
poor deactivation	check capillary ends; check intact deactivation using the test mixture; for poor results shorten both column ends by about 10 cm; or replace column; if column test does not show any defects: a) use a column with thicker film b) use phase with better deactivation c) use column with special selectivity
air in the system	use oxygen absorber or gas grade with less oxygen
water content too high	reduce water content
head-space analysis: permanent air injections	displace oxygen from vials with an inert gas

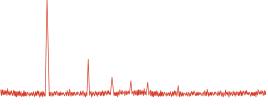
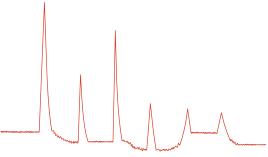
## Do's and don'ts in GC

Observation / Possible causes	Suggested remedy
<b>Regular interfering peaks</b>	
bleeding of silicon septa	replace injection septum, use low bleed or high temperature septa
poor gas quality	use gas grades recommended for GC; for longer supply lines from gas source to GC use gas purification cartridges directly connected to the GC
FID: dust or contaminants in the detector	clean detector; if particles are visible in the column or column ends are not cut precisely (frayed edges), cut two turns from the column entrance
electronic defect, damaged cable or detector	replace cable, contact your GC manufacturer
<b>Irregular interfering peaks, spikes, ghost peaks</b>	
contamination from vials / septa or sample preparation	control SPE and / or autosampler vials; use low bleed or high temperature septa
derivatisation not quantitative	check derivatisation protocol; use more reactive derivatisation reagents
dirty syringe	use a different syringe or clean it
sample decomposes	check temperature programme, oven temperature (external thermometer); if analytes are not temperature-stable, reduce injector temperature; replace liner
column absorbs or decomposes analytes	check capillary ends; check intact deactivation using the test mixture; for poor results shorten both column ends by about 10 cm; or replace column; if column test does not show any defects: a) use a column with thicker film b) use phase with better deactivation c) use column with special selectivity
sample volume too high, double injection	reduce sample volume or add a blank run after a high volume injection
poor gas quality	use gas grades recommended for GC; for longer supply lines from gas source to GC use gas purification cartridges directly connected to the GC

# GC troubleshooting



Observation / Possible causes	Suggested remedy
<b>Decreased or differing retention times</b>	
speed of gas too high	compare flow at column entrance and outlet with preset flow; check and / or clean gas tubes; in case of pressure build-up cut and remove one turn (20 cm) from the column or replace column
oven temperature too high	check temperature programme, oven temperature (external thermometer); decrease temperature
<b>Increased or differing retention times/ low gas flow</b>	
speed of gas too low	increase flow
column connection leaks, column not properly installed	check column installation; search for leaks; replace ferrules.
oven temperature too low	check temperature programme, oven temperature (external thermometer); if the analytes are stable, increase temperature
strong decrease of gas pressure	replace septum; for an instrument with pressure / temperature control, flow must be higher than 15 psi above the demand at max. temperature of the programme
tubes / capillaries / column constricted or blocked	compare flow at column entrance and outlet with preset flow; check and / or clean gas tubes; in case of pressure build-up cut and remove one turn (20 cm) from the column or replace column
<b>Negative peaks, negative signals</b>	
polarity of integrator is inverted	invert polarity at the instrument
sample injected into wrong column	inject sample in proper column
column overload	decrease injection volume; dilute sample
pressure fluctuations	check gas tubes, valves, seals; test gas flow; change injection septum; contact hardware manufacturer
detector contaminated	clean detector

Observation / Possible causes	Suggested remedy
<b>Peaks too small, poor quantification, concentrations not reproducible</b>	
dirty syringe	use a different syringe or clean it
concentration of sample too low	increase injection volume; concentrate sample
split too high	reduce split
sensitivity of detector too low	inject standard in order to test detector sensitivity
column connection leaks, column not properly installed	check column installation; search for leaks; replace ferrules
injector temperature too low	check temperature programme, increase injector temperature
dirty ECD	clean ECD
FID, TCD gas flow too low	correct flow according to manufacturers' instructions
sample decomposes	check capillary ends; check intact deactivation using the test mixture; for poor results shorten both column ends by about 10 cm; or replace column; if column test does not show any defects: a) use a column with thicker film b) use phase with better deactivation c) use column with special selectivity
<b>Baseline increases or decreases before or after a peak</b>	
injection volume too high	decrease injection volume; dilute sample; clean injection system
column bleeding due to poor conditioning	condition column according to manufacturers' instructions (while column is not connected to the detector)
pressure fluctuations	check gas tubes, valves, seals; test gas flow; change injection septum; contact hardware manufacturer
baseline not properly adjusted	readjust baseline; calibrate integrator
injector temperature too low	check injector temperature; if the analytes are stable, increase temperature
injection septum perforated	replace septum
wrong TCD gas flow	adjust flow according to manufacturers' instructions

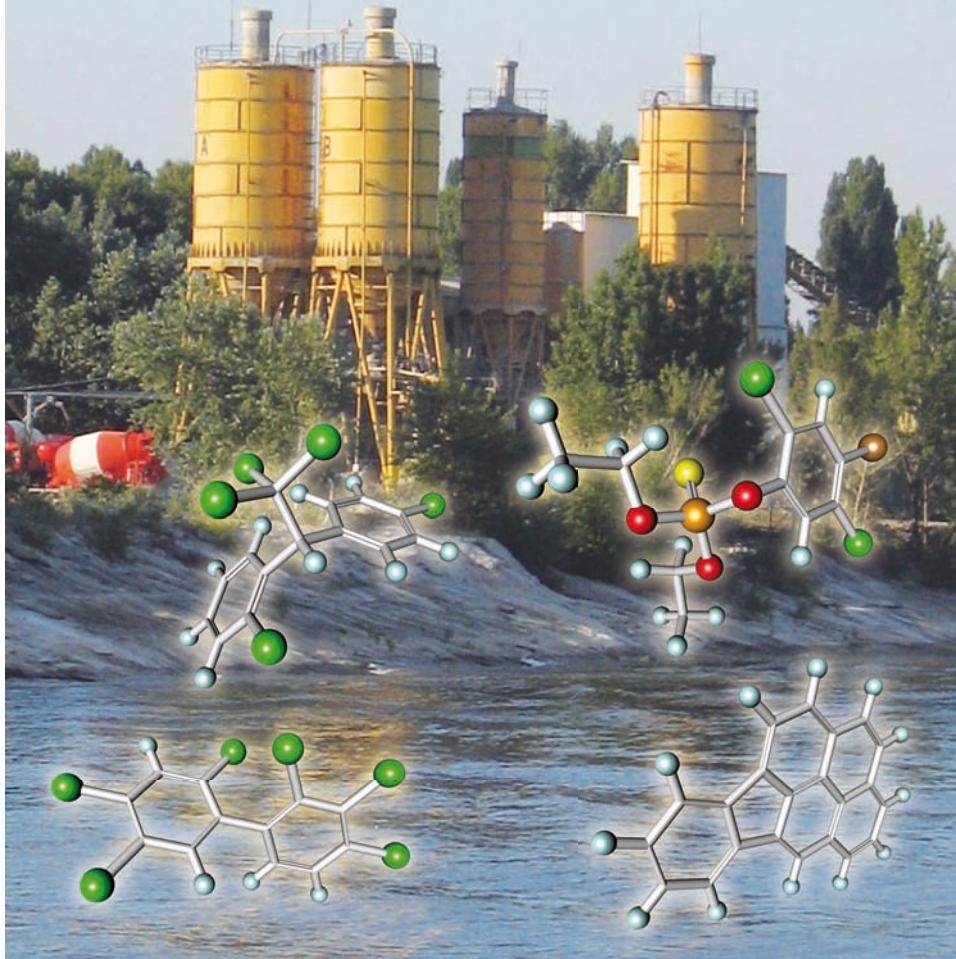


# Environmental Pollutants

hydrocarbons · PAH

PCB · phenols

pesticides



# Environmental pollutants

## Separation of PAH, PCB, pesticides and phthalates (EPA 525) MN Appl. No. 212810

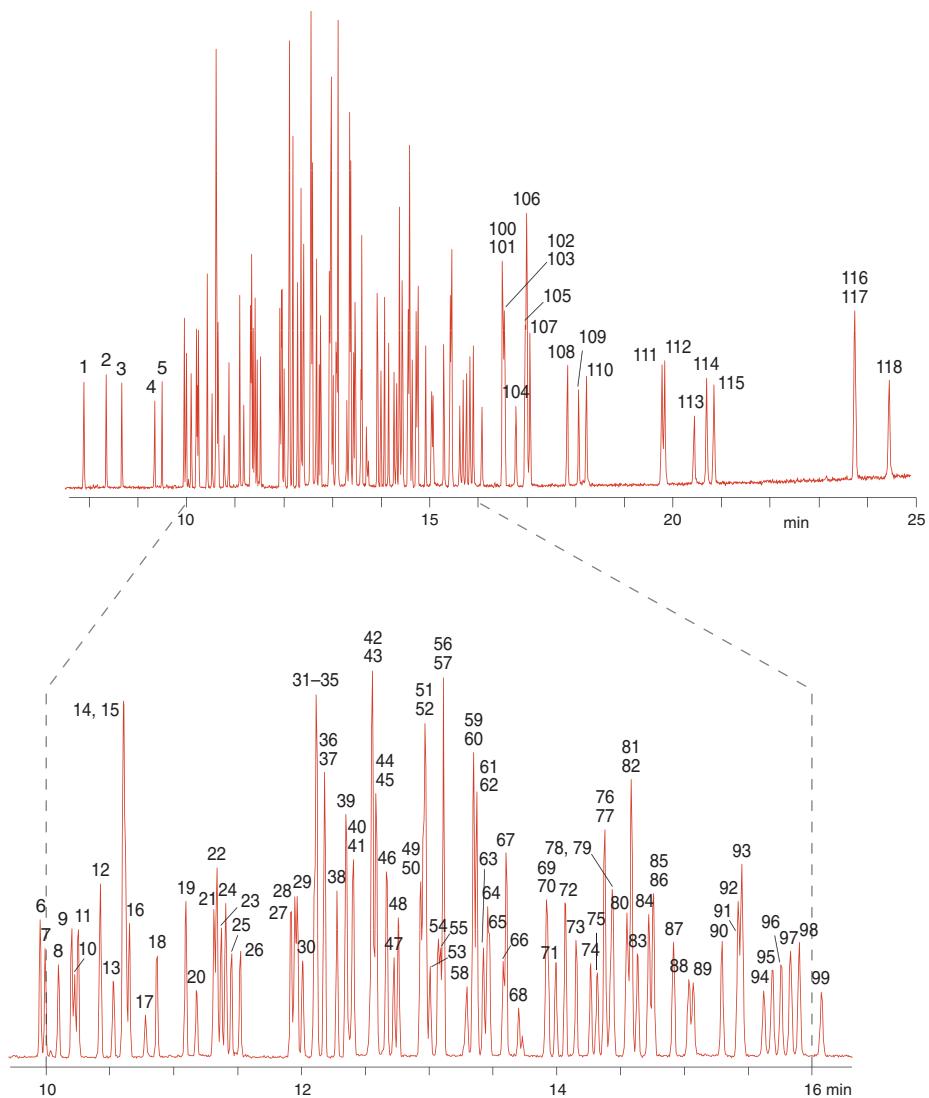
Column: OPTIMA® XLB, 30 m x 0.25 mm ID, 0.25 µm film, REF 725850.30,  
max. temperature 340/360 °C  
 Sample: standard according to EPA method 525, 5 ng per component  
 Injection: 1 µl, 300 °C, pressure pulsed (0.4 min 30 psi), splitless for 0.4 min  
 Carrier: 1.0 ml/min He  
 Temperature: 35 °C (2 min)  $\xrightarrow{20\text{ °C/min}}$  260 °C  $\xrightarrow{6\text{ °C/min}}$  330 °C (5 min)  
 Detector: MSD, 280 °C, scan range 45 – 550 amu

**Peaks:**

1. Isoporphone	40. β-BHC	79. <i>trans</i> -Nonachlor
2. 2-Nitro-m-xylene	41. Disulfoton	80. Pyrene-d <sub>10</sub>
3. Dichlorvos	42. Terbacil	81. Pyrene
4. Hexachlorocyclopentadiene	43. Phenanthrene-d <sub>10</sub>	82. 4,4'-DDE
5. EPTC = S-ethyl N,N-dipropyl-thiocarbamate	44. Parathion-methyl	83. PCB-154
6. Butylate	45. Phenanthrene	84. <i>p</i> -Terphenyl-d <sub>14</sub>
7. Mevinphos	46. Anthracene	85. Dieldrin
8. Vernolate	47. γ-BHC (lindane)	86. Carboxin
9. Pebulate	48. PCB-29	87. Chlorobenzilate
10. Etridiazole (terrazole)	49. Alachlor	88. Tricyclazole
11. Dimethyl phthalate	50. Prometryn	89. Endrin
12. Acenaphthene	51. Ametryn	90. 4,4'-DDD
13. 2,6-Dinitrotoluene	52. Simetryn	91. Bis(2-ethylhexyl) adipate
14. Acenaphthene-d <sub>10</sub>	53. δ-BHC	92. Butyl benzyl phthalate
15. PCB-1	54. Heptachlor	93. Endosulfan II
16. Chloroneb	55. Chlorothalonil	94. Endrin aldehyde
17. Tebuthiuron	56. Di- <i>n</i> -butyl phthalate	95. Norflurazon
18. Molinate	57. Terbutryl	96. 4,4'-DDT
19. Diethyl phthalate	58. Bromacil	97. Triphenyl phosphate
20. 2,4-Dinitrotoluene	59. Chlorpyrifos	98. Hexazinone
21. Propachlor	60. Metolachlor	99. Endosulfan sulphate
22. Fluorene	61. Chlorthal-methyl	100. Bis(2-ethylhexyl) phthalate
23. Ethoprop	62. PCB-47	101. Methoxychlor
24. Cycloate	63. Aldrin	102. PCB-201
25. Trifluralin	64. Triadimefon	103. PCB-171
26. Chlorpropham	65. Cyanazine (Bladex)	104. Endrin ketone
27. PCB-5	66. MGK-264 (N-octyl bicycloheptene dicarboximide)	105. Benz[a]anthracene
28. Atraton	67. Diphenamid	106. Chrysene-d <sub>12</sub>
29. Prometon	68. Merphos	107. Chrysene
30. α-BHC	69. PCB-98	108. Fenarimol
31. Hexachlorobenzene	70. Heptachlor epoxide B	109. <i>cis</i> -Permethrin
32. Propazine	71. Heptachlor epoxide A	110. <i>trans</i> -Permethrin
33. Simazin	72. Butachlor	111. Benzo[b]fluoranthene
34. Atrazine	73. Stirofos (tetrachlorvinphos)	112. Benzo[k]fluoranthene
35. Metribuzin	74. Fenamiphos	113. Fluridone (Sonar <sup>®</sup> )
36. Diazinon	75. α-Chlordane	114. Benzo[a]pyrene
37. Terbufos	76. Napropamide	115. Perylene-d <sub>12</sub>
38. Pronamide (propyzamide)	77. γ-Chlordane	116. Dibenz[ah]anthracene
39. Pentachlorophenol	78. Endosulfan I	117. Indeno[1,2,3-cd]pyrene
		118. Benzo[ghi]perylene

For formulas of selected compounds see structure index from page 291.

## Complex mixture



# Environmental pollutants

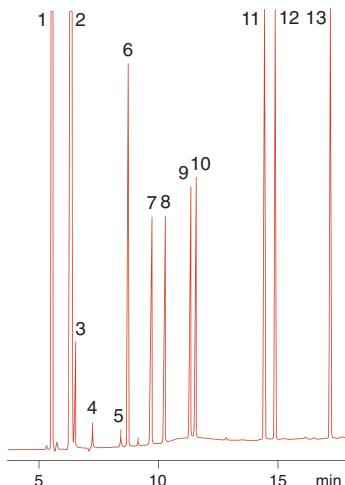
## Analysis of volatile halogenated hydrocarbons

MN Appl. No. 210560

Column: OPTIMA® 5,  
60 m x 0.32 mm ID,  
1.0 µm film, REF 726325.60,  
max. temperature 340/360 °C  
Injection: 0.1 µl  
Carrier gas: 56 ml/min N<sub>2</sub> 10 °C/min  
Temperature: 50 °C (5 min) → 200 °C  
Detector: ECD 300 °C

### Peaks:

1. Trichlorofluoromethane (F11)
2. 1,1,2-Trichlorotrifluoroethane (F113)
3. Dichloromethane
4. *trans*-1,2-Dichloroethene
5. *cis*-1,2-Dichloroethene
6. Trichloromethane
7. 1,1,1-Trichloroethane + 1,2-dichloroethane
8. Tetrachloromethane
9. Trichloroethene
10. Bromodichloromethane
11. Dibromochloromethane
12. Tetrachloroethene
13. Tribromomethane



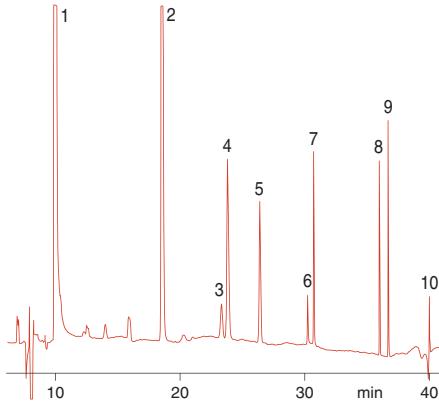
## Analysis of a haloform test mixture

MN Appl. No. 212480

Column: PERMABOND® SE-54-HKW,  
50 m x 0.32 mm ID,  
REF 723945.50,  
max. temperature 300/320 °C  
Injection: 1 µl, split ~1:30  
Carrier gas: 0.9 bar He 10 °C/min  
Temperature: 35 °C (25 min) → 160 °C  
(5 min)  
Detector: ECD 300 °C

### Peaks [ng/ml]:

1. Dichloromethane [795]
2. Trichloromethane [75]
3. 1,1,1-Trichloroethane [67]
4. 1,2-Dichloroethane [100]
5. Tetrachloromethane [15.9]
6. Trichloroethene [14.6]
7. Bromodichloromethane [20]
8. Dibromochloromethane [122]
9. Tetrachloroethene [81]
10. Tribromomethane [28.9]



# Volatile hydrocarbons



## Separation of *cis*-1,2-dichloroethene and bromochloromethane from excess trichloromethane (1:1:1000)

MN Appl. No. 212570

Column: OPTIMA® 624 LB,  
30 m x 0.32 mm ID,  
1.8 µm film, REF 726786.30  
max. temperature 280/300 °C

Injection: 1 µl, 280 °C, split 300 ml/min

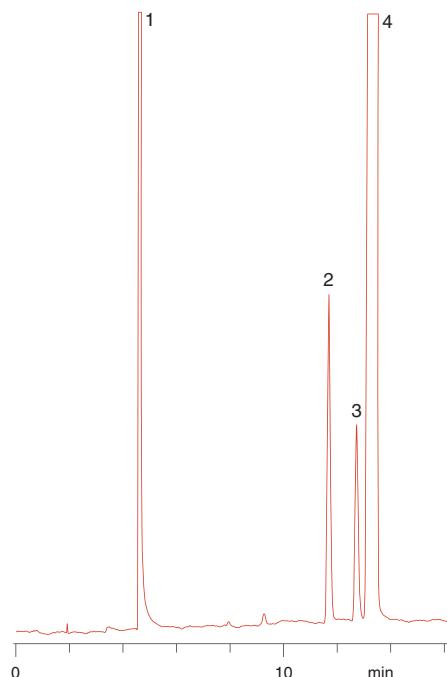
Carrier gas: 55 kPa He (1.7 ml/min)

Temperature: isothermal, 40 °C

Detector: FID 280 °C

**Peaks:**

1. Ethanol (stabilizer)
2. *cis*-1,2-Dichloroethene
3. Bromochloromethane
4. Trichloromethane



Please note that for such a long isothermal separation little differences in film thickness may result in longer or shorter retention times. In this case it may be necessary to adjust pressure or flow rate.

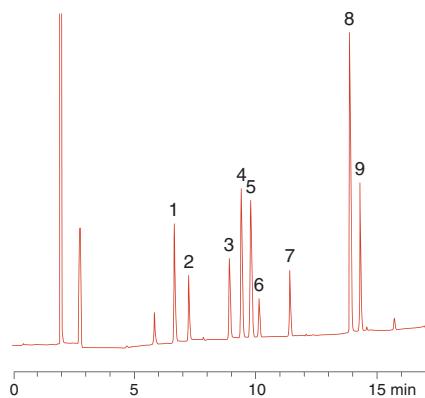
## Environmental pollutants

### Analysis of volatile hydrocarbons with headspace GC MN Appl. No. 210330

Column: OPTIMA® 624, 50 m x 0.32 mm ID, 1.8 µm film, REF 726787.50,  
max. temperature 280/300 °C  
Injection: HS Cryofocussion, splitless, 150 °C  
Carrier gas: 2.11 ml/min N<sub>2</sub>  
Temperature: 36 °C (4 min)  $\xrightarrow{10 \text{ °C/min}}$  200 °C (2 min)  
Detector: ECD 260 °C

**Peaks:**

1. Dichloromethane
2. *trans*-1,2-Dichloroethene
3. *cis*-1,2-Dichloroethene
4. Trichloromethane
5. 1,1,1-Trichloroethane
6. Tetrachloromethane
7. Trichloroethene
8. 1,1,2-Trichloroethane
9. Tetrachloroethene



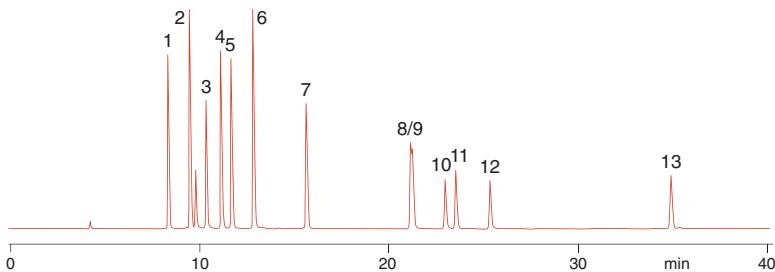
Courtesy of R. Mueller, G&P Torsten Plaar Umweltanalytik, Oldenburg, Germany

### Analysis of chlorinated hydrocarbons MN Appl. No. 210080

Column: OPTIMA® 624, 30 m x 0.25 mm ID, 1.4 µm film, REF 726785.30,  
max. temperature 280/300 °C  
Sample: 6.25 µl/l  
Temperature: 35 °C (4 min)  $\xrightarrow{4 \text{ °C/min}}$  180 °C (8 min)  
Detector: FID

**Peaks:**

- |                             |                                      |                                                 |
|-----------------------------|--------------------------------------|-------------------------------------------------|
| 1. Isoallyl chloride        | 6. 1,5-Hexadiene                     | 11. 2-Chloromethyloxirane<br>(Epichlorohydrine) |
| 2. 1-Chloro-2-methylpropane | 7. 2,2-Dichloropropane               | 12. ( <i>E</i> )-1,3-Dichloropropene            |
| 3. 1-Chloropropene          | 8. 1,1-Dichloropropane               | 13. 1,2,3-Trichloropropane                      |
| 4. 3-Chloropropene          | 9. 2,3-Dichloropropene               |                                                 |
| 5. 1-Chloropropane          | 10. ( <i>Z</i> )-1,3-Dichloropropene |                                                 |



Courtesy of Mrs. Kehl, DOW, Stade, Germany

# Volatile hydrocarbons

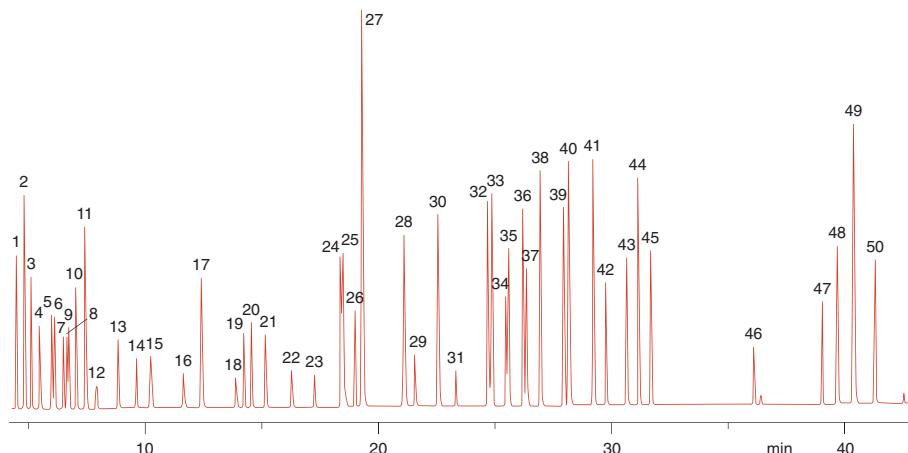


## Analysis of volatile organic compounds (EPA 502/524) MN Appl. No. 250461

Column: OPTIMA® δ-6, 50 m x 0.20 mm ID, 0.2 µm film, REF 726465.50,  
max. temperature 340/360 °C  
Sample: EPA 502 Volatile Organics Calibration Mix, 20 µg/ml per component in methanol  
Injection: 1 µl, split 20 ml/min  
Carrier gas: 1.5 bar He  
Temperature: 40 °C (5 min)  $\xrightarrow{2.5\text{ °C/min}}$  70 °C  $\xrightarrow{3\text{ °C/min}}$  100 °C  $\xrightarrow{4\text{ °C/min}}$  160 °C (10 min)  
Detector: MSD

### Peaks:

- |                                                |                                              |                                 |
|------------------------------------------------|----------------------------------------------|---------------------------------|
| 1. 1,1-Dichloroethene                          | 17. Toluene                                  | 34. 1,2,3-Trichloropropane      |
| 2. Dichloromethane                             | 18. <i>trans</i> -1,3-Dichloropropene        | 35. 2-Chlorotoluene             |
| 3. <i>trans</i> -1,2-Dichloroethene            | 19. 1,1,2-Trichloroethane                    | 36. 1,3,5-Trimethylbenzene      |
| 4. 1,1-Dichloroethane                          | 20. Tetrachloroethene                        | 37. 4-Chlorotoluene             |
| 5. 2,2-Dichloropropane                         | 21. 1,3-Dichloropropane                      | 38. <i>t</i> -Butylbenzene      |
| 6. <i>cis</i> -1,2-Dichloroethene              | 22. Dibromochloromethane                     | 39. 1,2,4-Trimethylbenzene      |
| 7. Trichloromethane                            | 23. 1,2-Dibromoethane                        | 40. sec-Butylbenzene            |
| 8. Bromochloromethane                          | 24. Chlorobenzene                            | 41. <i>p</i> -Isopropyltoluene  |
| 9. 1,1,1-Trichloroethane                       | 25. Ethylbenzene                             | 42. 1,3-Dichlorobenzene         |
| 10. 1,1-Dichloroprop-1-ene + tetrachloroethane | 26. 1,1,2-Tetrachloroethane                  | 43. 1,4-Dichlorobenzene         |
| 11. Benzene                                    | 27. <i>m</i> -Xylene + <i>p</i> -xylene      | 44. <i>n</i> -Butylbenzene      |
| 12. 1,2-Dichloroethane                         | 28. <i>o</i> -Xylene                         | 45. 1,2-Dichlorobenzene         |
| 13. Trichloroethene                            | 29. Styrene                                  | 46. 1,2-Dibromo-3-chloropropane |
| 14. 1,2-Dichloropropane                        | 30. Isopropylbenzene                         | 47. Hexachlorobutadiene         |
| 15. Dibromomethane + bromodichloromethane      | 31. Tribromomethane                          | 48. 1,2,4-Trichlorobenzene      |
| 16. <i>cis</i> -1,3-Dichloropropene            | 32. <i>n</i> -Propylbenzene                  | 49. Naphthalene                 |
|                                                | 33. Bromobenzene + 1,1,2,2-tetrachloroethane | 50. 1,2,3-Trichlorobenzene      |



# Environmental pollutants

## Analysis of volatile organic compounds (EPA 502/524)

*MN Appl. No. 211280*

Column: OPTIMA® 624, 50 m x 0.25 mm ID, 1.4 µm film, REF 726785.50, max. temperature 280/300 °C

Sample: EPA 502 Volatile Organics Calibration Mix, Supelco 502 111, 2000 µg/ml per component in methanol

Injection: 1 µl, split 1:50

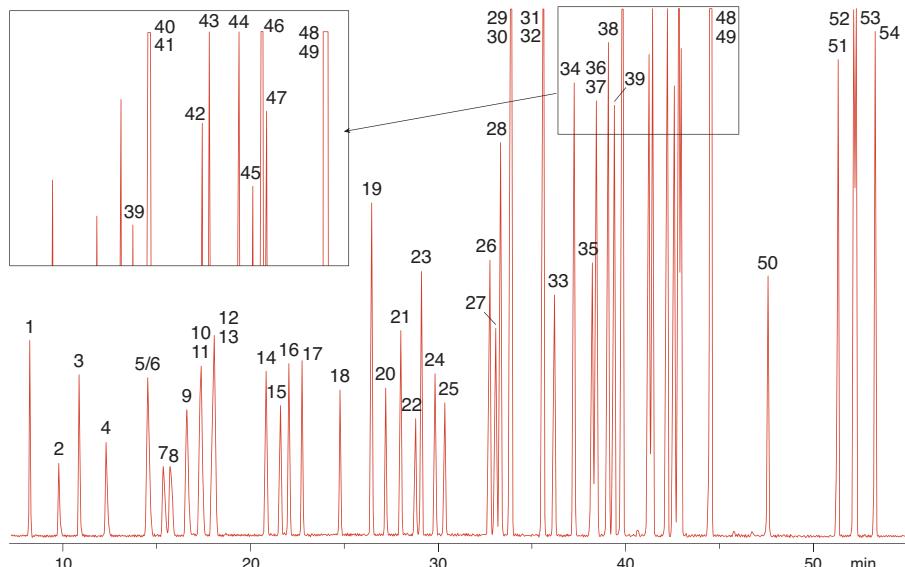
Carrier gas: 1.5 bar He      2.5 °C/min      3 °C/min      4 °C/min

Temperature: 40 °C (5 min) → 70 °C → 100 °C → 220 °C (5 min)

Detector: MSD

**Peaks:**

- |                                     |                                       |                                 |
|-------------------------------------|---------------------------------------|---------------------------------|
| 1. 1,1-Dichloroethene               | 19. Toluene                           | 37. Bromobenzene                |
| 2. Dichloromethane                  | 20. <i>trans</i> -1,3-Dichloropropene | 38. <i>n</i> -Propylbenzene     |
| 3. <i>trans</i> -1,2-Dichloroethene | 21. 1,1,2-Trichloroethane             | 39. 2-Chlorotoluene             |
| 4. 1,1-Dichloroethane               | 22. 1,3-Dichloropropane               | 40. 1,3,5-Trimethylbenzene      |
| 5. 2,2-Dichloropropane              | 23. Tetrachloroethene                 | 41. 4-Chlorotoluene             |
| 6. <i>cis</i> -1,2-Dichloroethene   | 24. Dibromochloromethane              | 42. <i>t</i> -Butylbenzene      |
| 7. Bromochloromethane               | 25. 1,2-Dibromoethane                 | 43. 1,2,4-Trimethylbenzene      |
| 8. Trichloromethane                 | 26. Chlorobenzene                     | 44. <i>sec</i> -Butylbenzene    |
| 9. 1,1,1-Trichloroethane            | 27. 1,1,1,2-Tetrachloroethane         | 45. 1,3-Dichlorobenzene         |
| 10. 1,1-Dichloropropene             | 28. Ethylbenzene                      | 46. <i>p</i> -Isopropyltoluene  |
| 11. Tetrachloromethane              | 29. <i>m</i> -Xylene                  | 47. 1,4-Dichlorobenzene         |
| 12. 1,2-Dichloroethane              | 30. <i>p</i> -Xylene                  | 48. <i>n</i> -Butylbenzene      |
| 13. Benzene                         | 31. <i>o</i> -Xylene                  | 49. 1,2-Dichlorobenzene         |
| 14. Trichloroethene                 | 32. Styrene                           | 50. 1,2-Dibromo-3-chloropropane |
| 15. 1,2-Dichloropropane             | 33. Tribromomethane                   | 51. 1,2,4-Trichlorobenzene      |
| 16. Dibromomethane                  | 34. Isopropylbenzene                  | 52. Hexachlorobutadiene         |
| 17. Bromodichloromethane            | 35. 1,1,2,2-Tetrachloroethane         | 53. Naphthalene                 |
| 18. <i>cis</i> -1,3-Dichloropropene | 36. 1,2,3-Trichloroproppane           | 54. 1,2,3-Trichlorobenzene      |



# Volatile hydrocarbons

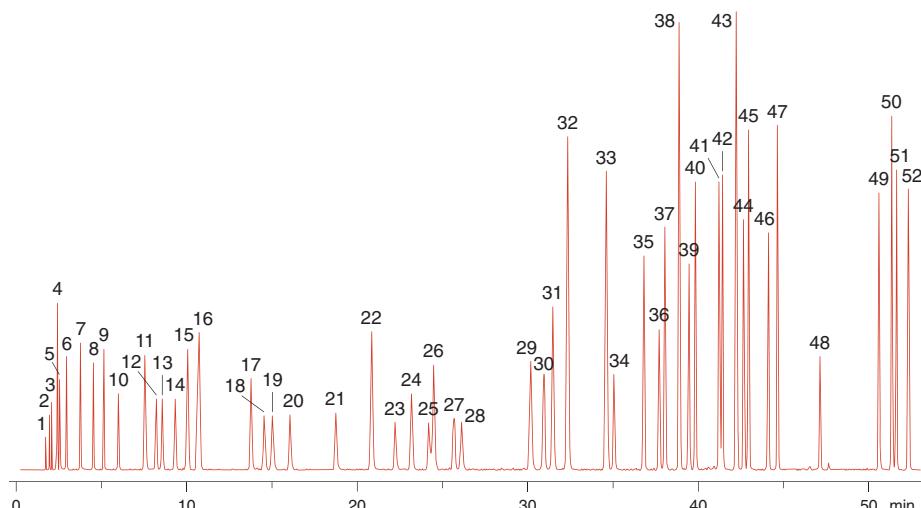


## Analysis of volatile organic compounds (EPA 624) MN Appl. No. 250200

Column: OPTIMA® 624, 25 m x 0.20 mm ID, 1.1 µm film, REF 726784.25,  
max. temperature 280/300 °C  
Sample: EPA 624 Volatile Organics, 20 µg/ml per component in methanol  
Injection: 1 µl, split 1:50  
Carrier gas: 20 cm/s He  
Temperature: 40 °C (8 min)  $\xrightarrow{1.5\text{ °C/min}}$  70 °C  $\xrightarrow{4\text{ °C/min}}$  180 °C  
Detector: MSD, EI

### Peaks:

1. Dichlorofluoromethane
2. Chloromethane
3. Vinyl chloride
4. Bromomethane
5. Chloroethane
6. Trichlorofluoromethane
7. 1,1-Dichloroethene
8. Dichloromethane
9. *trans*-1,2-Dichloroethene
10. 1,1-Dichloroethane
11. *cis*-1,2-Dichloroethene
12. Bromochloromethane + 2,2-dichloropropane
13. Trichloromethane
14. 1,1,1-Trichloroethane
15. Tetrachloromethane + 1,1-dichloropropene
16. Benzene + 1,2-dichloroethane
17. Trichloroethene
18. 1,2-Dichloropropane
19. Dibromomethane
20. Bromodichloromethane
21. *cis*-1,3-Dichloropropene
22. Toluene
23. *trans*-1,3-Dichloropropene
24. 1,1,2-Trichloroethane
25. 2-Chloro-1-propene
26. Tetrachloroethene
27. Dibromochloromethane
28. 1,2-Dibromoethane
29. Chlorobenzene
30. 1,1,1,2-Tetrachloroethane
31. Ethylbenzene
32. *m*-Xylene + *p*-xylylene
33. Styrene + *o*-xylylene
34. Tribromomethane
35. Isopropylbenzene
36. Bromobenzene
37. 1,2,3-Trichloropropane + 1,1,2,2-tetrachloroethane
38. Benzyl chloride
39. 2-Chlorotoluene + 3-chlorotoluene
40. 1,3,5-Trimethylbenzene
41. *t*-Butylbenzene
42. 1,2,4-Trimethylbenzene
43. *sec*-Butylbenzene + 1,3-dichlorobenzene
44. 1,4-Dichlorobenzene
45. *o*-Isopropyltoluene
46. 1,2-Dichlorobenzene
47. *n*-Butylbenzene
48. 1,2-Dibromo-3-chloropropane
49. 1,2,4-Trichlorobenzene
50. Naphthalene
51. Hexachloro-1,3-butadiene
52. 1,2,3-Trichlorobenzene



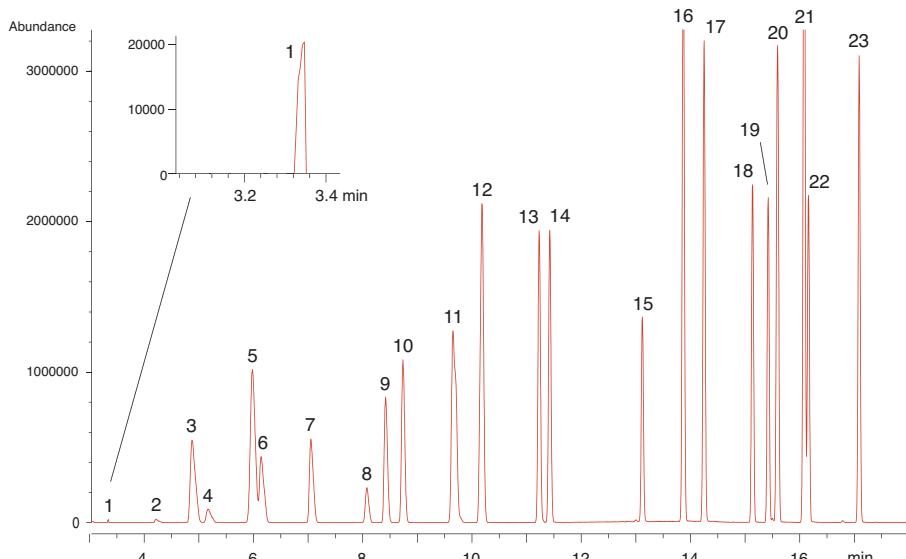
# Environmental pollutants

## Analysis of volatile halogenated hydrocarbons and BTX MN Appl. No. 200150

Column: PERMABOND® SE-54-HKW, 50 m x 0.32 mm ID, REF 723945.50,  
max. temperature 300/320 °C  
Injection: 1 µl, 0.2% in methanol, split 15 ml/min  
Carrier gas: 1.5 ml/min He (constant flow)  
Temperature: 40 °C (5 min)  $\xrightarrow{10 \text{ °C/min}}$  160 °C  
Detector: MSD

**Peaks:**

- |                                                |                                   |
|------------------------------------------------|-----------------------------------|
| 1. Vinyl chloride                              | 13. Trichloroethene               |
| 2. Ethanol                                     | 14. Dichlorobromomethane          |
| 3. Trichlorofluoromethane (F 11)               | 15. Toluene                       |
| 4. Pentane                                     | 16. Chlorodibromomethane          |
| 5. 1,1,2-Trichlorotrifluoroethane (F 113)      | 17. Tetrachloroethene             |
| 6. Dichloromethane                             | 18. Chlorobenzene                 |
| 7. <i>trans</i> -1,2-Dichloroethene            | 19. Ethylbenzene                  |
| 8. Hexane                                      | 20. <i>m</i> - + <i>p</i> -Xylene |
| 9. <i>cis</i> -1,2-Dichloroethene              | 21. Tribromomethane               |
| 10. Trichloromethane                           | 22. <i>o</i> -Xylene              |
| 11. 1,1,1-Trichloroethane + 1,2-dichloroethane | 23. Bromobenzene                  |
| 12. Tetrachloromethane + benzene               |                                   |



# Chlorinated hydrocarbons



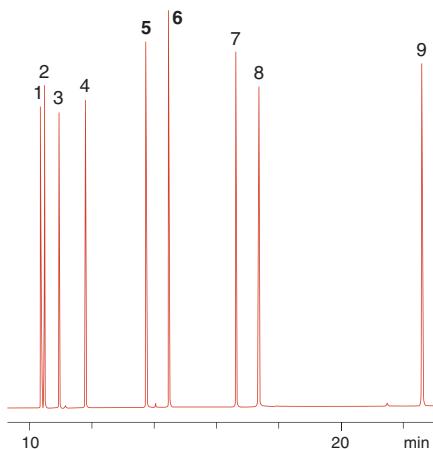
## Analysis of chlorinated hydrocarbons (EPA 612)

### Peaks:

- |                        |                           |                              |
|------------------------|---------------------------|------------------------------|
| 1. 1,3-Dichlorobenzene | 4. Hexachloroethane       | 7. Hexachlorocyclopentadiene |
| 2. 1,4-Dichlorobenzene | 5. 1,2,4-Trichlorobenzene | 8. 2-Chloronaphthalene       |
| 3. 1,2-Dichlorobenzene | 6. Hexachlorobutadiene    | 9. Hexachlorobenzene         |

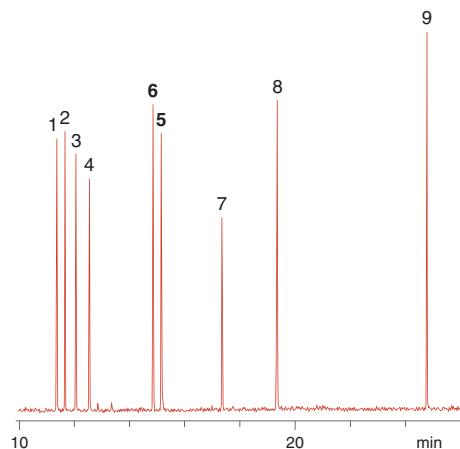
### MN Appl. No. 211290

Column: OPTIMA® 5,  
50 m x 0.20 mm ID,  
0.20  $\mu\text{m}$  film, REF 726857.50,  
max. temperature 340/360 °C  
Sample: chlorinated hydrocarbon  
standard EPA 612,  
2000  $\mu\text{g}/\text{ml}$  per component in  
isooctane  
Injection: 1  $\mu\text{l}$ , split 1:20  
Carrier gas: 2.5 bar He  
Temperature: 50 °C  $\xrightarrow{8 \text{ °C/min}}$  260 °C (10 min)  
Detector: MSD



### MN Appl. No. 250440

Column: OPTIMA® δ-6,  
50 m x 0.20 mm ID,  
0.20  $\mu\text{m}$  film, REF 726465.50,  
max. temperature 340/360 °C  
Sample: chlorinated hydrocarbon  
standard EPA 612,  
200  $\mu\text{g}/\text{ml}$  per component in  
isooctane  
Injection: 1  $\mu\text{l}$ , split 1:20  
Carrier gas: 2.0 bar He  
Temperature: 50 °C  $\xrightarrow{8 \text{ °C/min}}$  260 °C (10 min)  
Detector: MSD



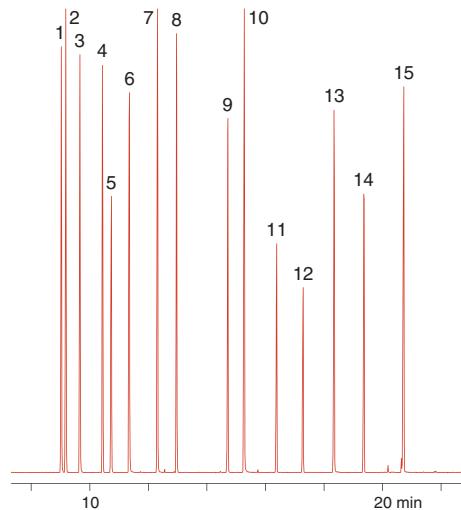
## Environmental pollutants

### Analysis of a mixture of neutral and basic organic compounds MN Appl. No. 200320

Column: OPTIMA® 5,  
25 m x 0.20 mm ID,  
0.2  $\mu$ m film, REF 726857.25,  
max. temperature 340/360 °C  
Sample: 200  $\mu$ g/ml each in 2-propanol  
Injection: 1.0  $\mu$ l; split 1:150  
Carrier gas: 25 cm/s He  
Temperature: 50 °C (5 min)  $\xrightarrow{10\text{ °C/min}}$  220 °C  
 $\xrightarrow{20\text{ °C/min}}$  330 °C  
Detector: MSD

**Peaks:**

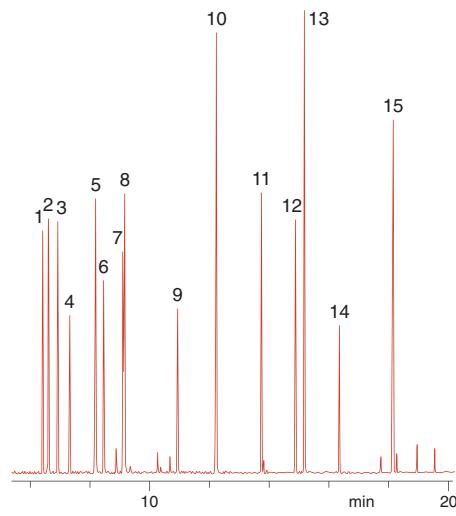
1. 1,3-Dichlorobenzene
2. 1,4-Dichlorobenzene
3. 1,2-Dichlorobenzene
4. Hexachloroethane
5. Nitrobenzene
6. Isophorone
7. 1,2,4-Trichlorobenzene
8. Hexachlorobutadiene
9. Hexachlorocyclopentadiene
10. 1-Chloronaphthalene
11. 2-Methyl-1,3-dinitrobenzene
12. 1-Methyl-2,4-dinitrobenzene
13. Azobenzene
14. Hexachlorobenzene
15. Carbazole



Column: OPTIMA® δ-3,  
25 m x 0.20 mm ID,  
0.20  $\mu$ m film, REF 726400.25,  
max. temperature 340/360 °C  
Injection: 1  $\mu$ l, split 1:150  
Carrier gas: 25 cm/s He  
Temperature: 50 °C (5 min)  $\xrightarrow{10\text{ °C/min}}$  220 °C  
 $\xrightarrow{20\text{ °C/min}}$  330 °C  
Detector: MSD

**Peaks:**

1. 1,4-Dichlorobenzene
2. 1,3-Dichlorobenzene
3. 1,2-Dichlorobenzene
4. Hexachloroethane
5. Nitrobenzene
6. Isophorone
8. 1,2,4-Trichlorobenzene
7. Hexachlorobutadiene
9. Hexachlorocyclopentadiene
10. 1-Chloronaphthalene
11. 2-Methyl-1,3-dinitrobenzene
12. 1-Methyl-2,4-dinitrobenzene
13. Azobenzene
14. Hexachlorobenzene
15. Carbazole



# Halogenated hydrocarbons



## Analysis of chlorotoluene isomers

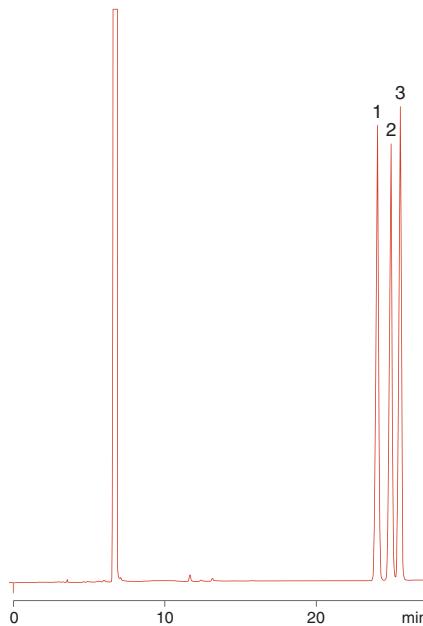
Column: OPTIMA® 624, 50 m x 0.32 mm ID, 1.8  $\mu\text{m}$  film, REF 726787.50,  
max. temperature 280/300 °C  
Carrier gas: 150 kPa H<sub>2</sub> (7.7 ml/min)  
Temperature: 70 °C  
Detector: FID 250 °C

### MN Appl. No. 200340

Injection: 1  $\mu\text{l}$  (1 % each in toluene)  
split 220 ml/min

**Peaks:**

1. *o*-Chlorotoluene
2. *m*-Chlorotoluene
3. *p*-Chlorotoluene

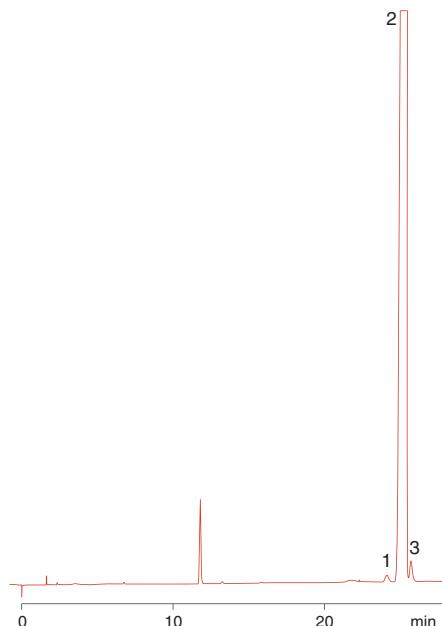


### MN Appl. No. 200350

Injection: 1  $\mu\text{l}$  (undiluted sample)  
split 220 ml/min

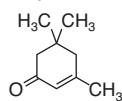
**Peaks:**

1. *o*-Chlorotoluene [0.16 %]
2. *m*-Chlorotoluene [98.31 %]
3. *p*-Chlorotoluene [0.39 %]

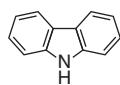


## Structures of miscellaneous organics

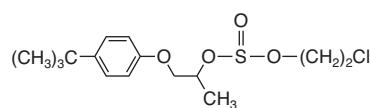
Isophorone



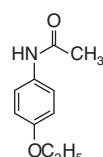
Carbazole



Aramite



Phenacetin



# Environmental pollutants

## Analysis of semivolatile organics (EPA 8270) MN Appl. No. 212790

Column: OPTIMA® 5 MS Accent, 30 m x 0.25 mm ID, 0.25 µm film, REF 725820.30,  
max. temperature 340/360 °C

Sample: 16 µg/ml in CH<sub>2</sub>Cl<sub>2</sub>

Injection: 1 µl splitless (hold 0.4 min), 300 °C

Carrier gas: 1.0 ml/min He

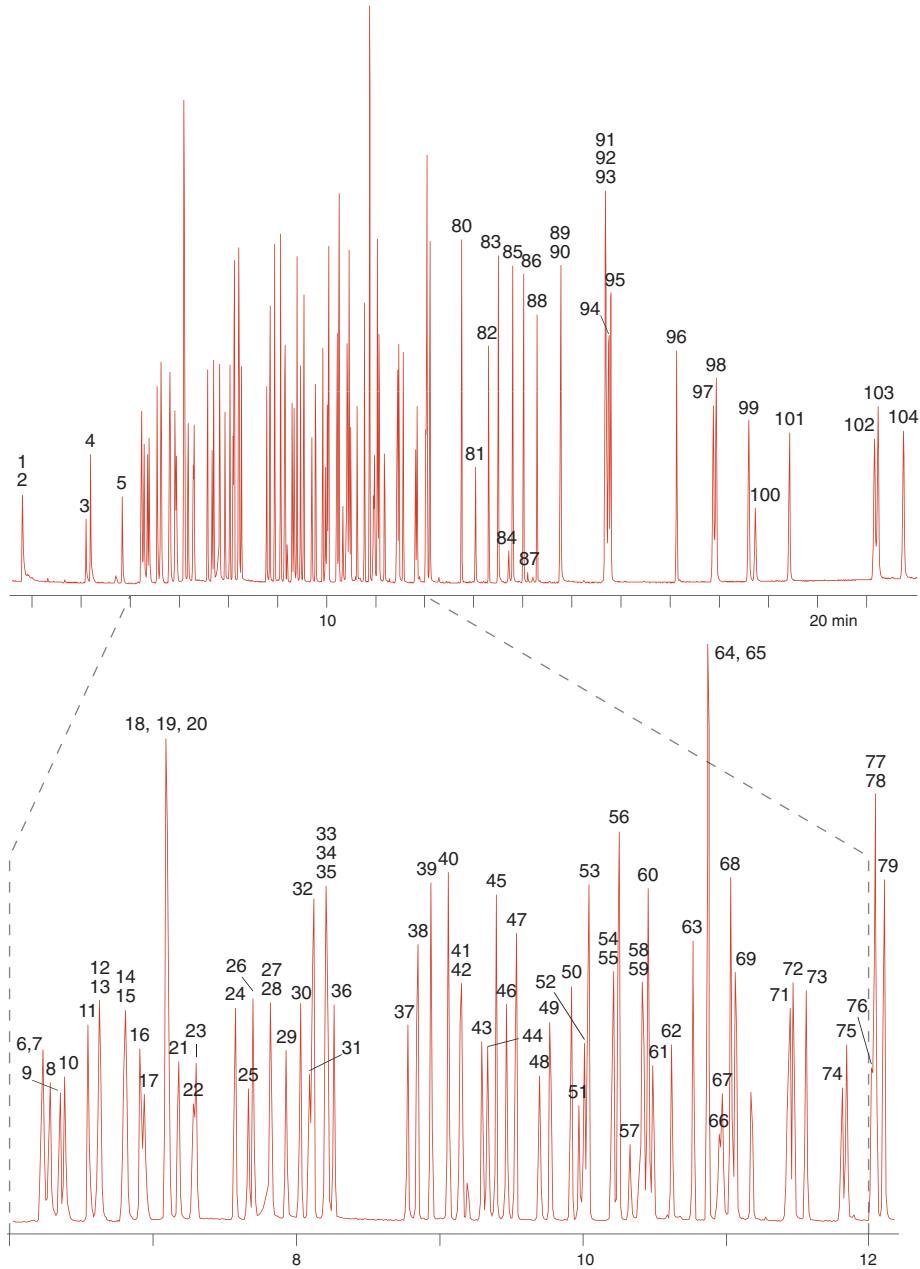
Temperature: 35 °C (2 min)  $\xrightarrow{20\text{ °C/min}}$  260 °C  $\xrightarrow{6\text{ °C/min}}$  330 °C (1 min)

Detection: MSD 280 °C

### Peaks:

- |                                        |                                  |                                  |
|----------------------------------------|----------------------------------|----------------------------------|
| 1. N-Nitrosodimethylamine              | 35. Hexachloropropene            | 70. 2,4,6-Tribromophenol         |
| 2. Pyridine                            | 36. Hexachlorobutadiene          | 71. Phenacetin                   |
| 3. Methyl methanesulfonate             | 37. 4-Chloro-3-methylphenol      | 72. 4-Bromophenyl phenyl ether   |
| 4. 2-Fluorophenol                      | 38. Isosafrole                   | 73. Hexachlorobenzene            |
| 5. Ethyl methanesulfonate              | 39. 2-Methylnaphthalene          | 74. Pentachlorophenol            |
| 6. Phenol-d <sub>6</sub>               | 40. 1-Methylnaphthalene          | 75. Pentachloronitrobenzene      |
| 7. Phenol                              | 41. Hexachlorocyclopentadiene    | 76. Phenanthrene-d <sub>10</sub> |
| 8. Aniline                             | 42. 1,2,4,5-Tetrachlorobenzene   | 77. Dinoseb                      |
| 9. Bis(2-chloroethyl) ether            | 43. 2,4,6-Trichlorophenol        | 78. Phenanthrene                 |
| 10. 2-Chlorophenol                     | 44. 2,4,5-Trichlorophenol        | 79. Anthracene                   |
| 11. 1,3-Dichlorobenzene                | 45. 2-Fluorobiphenyl             | 80. Di-n-butyl phthalate         |
| 12. 1,4-Dichlorobenzene-d <sub>4</sub> | 46. Safrole                      | 81. 4-Nitro-quinoline 1-oxide    |
| 13. 1,4-Dichlorobenzene                | 47. 2-Chloronaphthalene          | 82. Isodrin                      |
| 14. 1,2-Dichlorobenzene                | 48. 2-Nitroaniline               | 83. Fluoranthene                 |
| 15. Benzyl alcohol                     | 49. 1,4-Naphthoquinone           | 84. Benzdione                    |
| 16. 2-Methylphenol                     | 50. Dimethyl phthalate           | 85. Pyrene                       |
| 17. Bis(2-chloroisopropyl) ether       | 51. 1,3-Dinitrobenzene           | 86. p-Terphenyl-d <sub>14</sub>  |
| 18. Acetophenone                       | 52. 2,6-Dinitrotoluene           | 87. Aramide                      |
| 19a. 4-Methylphenol                    | 53. Acenaphthylene               | 88. Chlorobenzilate              |
| 19b. 3-Methylphenol                    | 54. Acenaphthene-d <sub>10</sub> | 89. Kepone                       |
| 20. N-Nitroso-di-n-propylamine         | 55. 3-Nitroaniline               | 90. Butyl benzyl phthalate       |
| 21. Hexachloroethane                   | 56. Acenaphthene                 | 91. Benz[a]anthracene            |
| 22. Nitrobenzene-d <sub>5</sub>        | 57. 2,4-Dinitrophenol            | 92. 3,3'-Dichlorobenzidine       |
| 23. Nitrobenzene                       | 58. Pentachlorobenzene           | 93. Chrysene-d <sub>12</sub>     |
| 24. Isophorone                         | 59. 4-Nitrophenol                | 94. Chrysene                     |
| 25. 2-Nitrophenol                      | 60. Dibenzofuran                 | 95. Bis(2-ethylhexyl) phthalate  |
| 26. 2,4-Dimethylphenol                 | 61. 2,4-Dinitrotoluene           | 96. Di-n-octyl phthalate         |
| 27. Bis(2-chloroethoxy)methane         | 62. 2,3,4,6-Tetrachlorophenol    | 97. Benzo[b]fluoranthene         |
| 28. Benzoic acid                       | 63. Diethyl phthalate            | 98. Benzo[k]fluoranthene         |
| 29. 2,4-Dichlorophenol                 | 64. Fluorene                     | 99. Benzo[a]pyrene               |
| 30. 1,2,4-Trichlorobenzene             | 65. 4-Chlorophenyl phenyl ether  | 100. Perylene-d <sub>12</sub>    |
| 31. Naphthalene-d <sub>8</sub>         | 66. 4-Nitroaniline               | 101. 3-Methylcholanthrene        |
| 32. Naphthalene                        | 67. 2-Methyl-4,6-dinitrophenol   | 102. Indeno[1,2,3-cd]pyrene      |
| 33. 2,6-Dichlorophenol                 | 68. Diphenylamine                | 103. Dibenz[ah]anthracene        |
| 34. 4-Chloroaniline                    | 69. Azobenzene                   | 104. Benzo[ghi]perylene          |

## Semivolatile organics



# Environmental pollutants

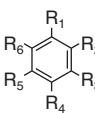
## Analysis of semivolatile organics (EPA 625) MN Appl. No. 212830

Column: OPTIMA® 5 MS Accent, 30 m x 0.25 mm ID, 0.25 µm film, REF 725820.30,  
max. temperature 340/360 °C  
 Injection: 1.0 µl, 10 ppm (20 ppm int. std.), 20 psi 0.3 min, pulsed splitless (hold 0.3 min),  
300 °C  
 Carrier gas: 1.0 ml/min He  
 Temperature: 35 °C (1 min)  $\xrightarrow{18\text{ °C/min}}$  270 °C  $\xrightarrow{5\text{ °C/min}}$  305 °C  $\xrightarrow{30\text{ °C/min}}$  330 °C (1 min)  
 Detector: MSD 280 °C

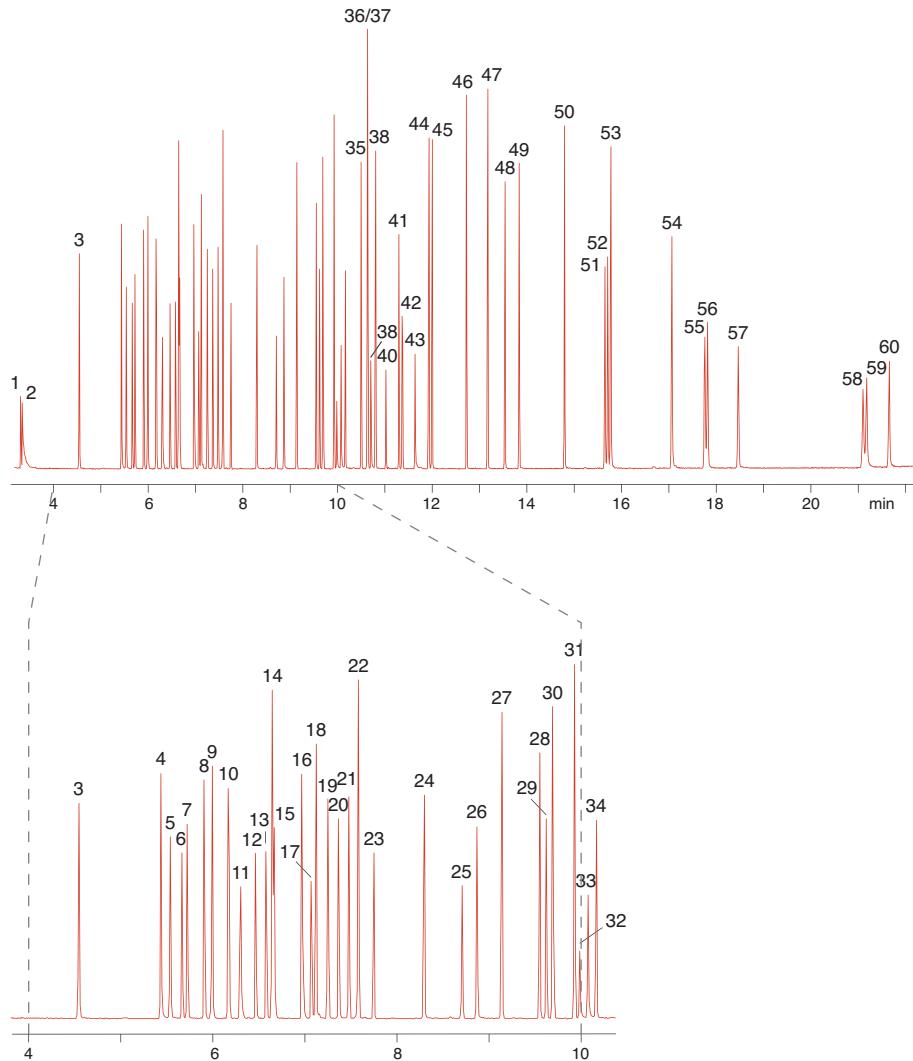
**Peaks:**

- |                                             |                                    |                                      |
|---------------------------------------------|------------------------------------|--------------------------------------|
| 1. N-Nitrosodimethylamine                   | 21. 1,2,4-Trichlorobenzene         | 41. 4-Bromophenyl phenyl ether       |
| 2. Pyridine-d <sub>5</sub>                  | 22. Naphthalene                    | 42. Hexachlorobenzene                |
| 3. 2-Fluorophenol (surrogate std.)          | 23. Hexachlorobutadiene            | 43. Pentachlorophenol                |
| 4. Pentafluorophenol (int. std.)            | 24. 4-Chloro-3-methylphenol        | 44. Phenanthrene                     |
| 5. Phenol                                   | 25. Hexachlorocyclopentadiene      | 45. Anthracene                       |
| 6. Bis(2-chloroethyl) ether                 | 26. 2,4,6-Trichlorophenol          | 46. Di-n-butyl phthalate             |
| 7. 2-Chlorophenol                           | 27. 2-Chloronaphthalene            | 47. 4,4'-Dibromobiphenyl (int. std.) |
| 8. 1,3-Dichlorobenzene                      | 28. Dimethyl phthalate             | 48. Fluoranthene                     |
| 9. 1,4-Dichlorobenzene                      | 29. 2,6-Dinitrotoluene             | 49. Pyrene                           |
| 10. 1,2-Dichlorobenzene                     | 30. Acenaphthylene                 | 50. Butyl benzyl phthalate           |
| 11. Bis(2-chloroisopropyl) ether            | 31. Acenaphthene                   | 51. Benz[a]anthracene                |
| 12. N-Nitroso-di-n-propylamine              | 32. 2,4-Dinitrophenol              | 52. Chrysene                         |
| 13. Hexachloroethane                        | 33. 4-Nitrophenol                  | 53. Bis(2-ethylhexyl) phthalate      |
| 14. Nitrobenzene-d <sub>5</sub> (int. std.) | 34. 2,4-Dinitrotoluene             | 54. Di-n-octyl phthalate             |
| 15. Nitrobenzene                            | 35. Diethyl phthalate              | 55. Benzo[b]fluoranthene             |
| 16. Isophorone                              | 36. Fluorene                       | 56. Benzo[k]fluoranthene             |
| 17. 2-Nitrophenol                           | 37. 4-Chlorophenyl phenyl ether    | 57. Benzo[a]pyrene                   |
| 18. 2,4-Dimethylphenol                      | 38. 4,6-Dinitro-2-methylphenol     | 58. Indeno[1,2,3-cd]pyrene           |
| 19. Bis(2-chloroethoxy)methane              | 39. Diphenylamine                  | 59. Dibenz[ah]anthracene             |
| 20. 2,4-Dichlorophenol                      | 40. 4,4'-Dibromooctafluorobiphenyl | 60. Benzo[ghi]perylene               |

### Substituted benzene common names

Structure	Compound	R <sub>1</sub>	R <sub>2</sub>	R <sub>3</sub>	R <sub>4</sub>	R <sub>5</sub>	R <sub>6</sub>
	HCB = Hexachlorobenzene	Cl	Cl	Cl	Cl	Cl	Cl
	Chloroneb	Cl	OCH <sub>3</sub>	H	Cl	OCH <sub>3</sub>	H
	Chlorthal	Cl	COOH	Cl	Cl	COOH	Cl
	Dichlobenil	Cl	CN	Cl	H	H	H
	Chlorothalonil	Cl	CN	Cl	CN	Cl	Cl
	Tecnazene	Cl	Cl	NO <sub>2</sub>	Cl	Cl	H
	Quintozene	Cl	Cl	NO <sub>2</sub>	Cl	Cl	Cl
	Dicloran	Cl	NH <sub>2</sub>	Cl	H	NO <sub>2</sub>	H
	Dinoseb	OH	sec-C <sub>4</sub> H <sub>9</sub>	H	NO <sub>2</sub>	H	NO <sub>2</sub>

## Semivolatile organics



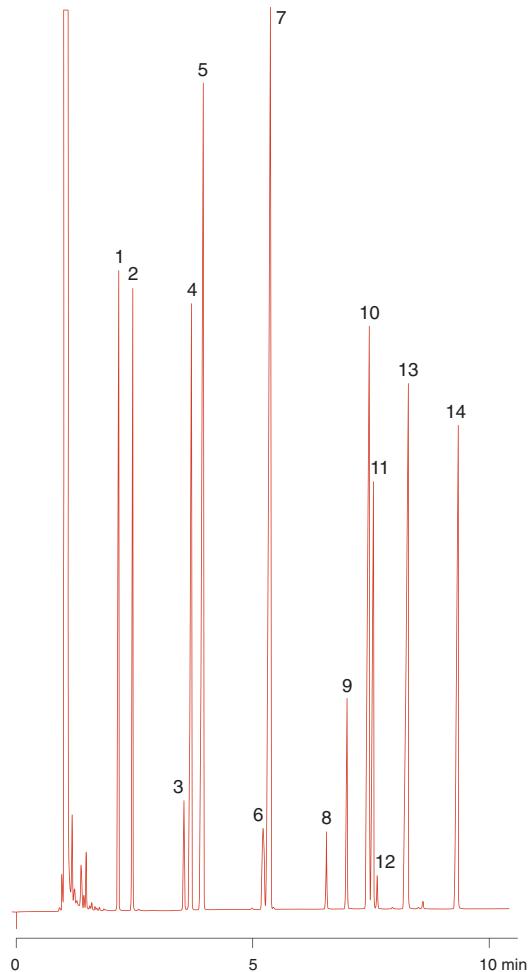
# Environmental pollutants

## Analysis of nitro- and chloronitroaromatics MN Appl. No. 200300

Column: OPTIMA® 1301, 25 m x 0.32 mm ID, 0.25 µm film, REF 726777.25,  
max. temperature 300/320 °C  
Injection: 0.5 µl, split 55 ml/min  
Carrier gas: 60 kPa H<sub>2</sub> (3.3 ml/min)  
Temperature: 120 °C (3 min)  $\xrightarrow{15\text{ °C/min}}$  230 °C  
Detector: FID 250 °C

**Peaks:**

1. Nitrobenzene
2. Isophorone
3. 3-Nitrochlorobenzene
4. 2-Nitrochlorobenzene
5. 4-Nitrochlorobenzene
6. 2,5- and 2,6-Dichloronitrobenzene
7. 2,4-Dichloronitrobenzene
8. 2,6-Dinitrotoluene
9. 2,5-Dinitrotoluene
10. 2,4-Dinitrotoluene
11. 2,3-Dinitrotoluene
12. 3,5-Dinitrotoluene
13. 3,4-Dinitrotoluene
14. 2,4,6-Trinitrotoluene



# Aromatic hydrocarbons



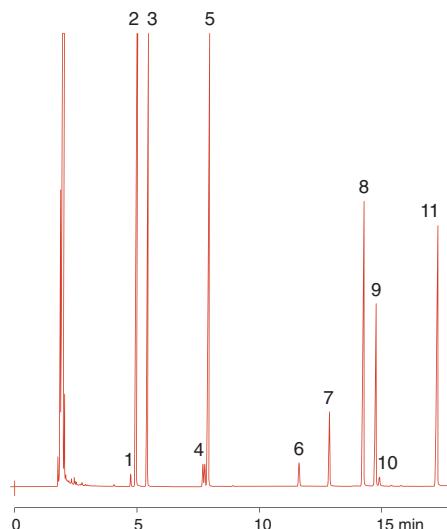
## Analysis of nitro- and chloronitro-aromatics

MN Appl. No. 200290

Column: OPTIMA® 1701,  
50 m x 0.32 mm ID,  
0.25  $\mu\text{m}$  film, REF 726318.50,  
max. temperature 300/320 °C  
Injection: 1  $\mu\text{l}$ , split 125 ml/min  
Carrier gas: 100 kPa H<sub>2</sub> 4 °C/min  
Temperature: 140 °C (6 min)  $\xrightarrow{10\text{ °C/min}}$  190 °C  
Detector: FID 250 °C

### Peaks:

1. 3-Nitrochlorobenzene
2. 4-Nitrochlorobenzene
3. 2-Nitrochlorobenzene
4. 2,5- and 2,6-Dichloronitrobenzene
5. 2,4-Dichloronitrobenzene
6. 2,6-Dinitrotoluene
7. 2,5-Dinitrotoluene
8. 2,4-Dinitrotoluene
9. 2,3-Dinitrotoluene
10. 3,5-Dinitrotoluene
11. 3,4-Dinitrotoluene



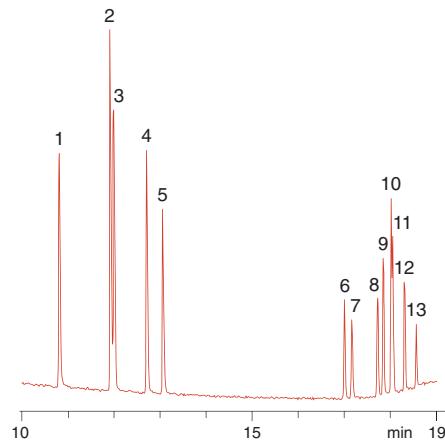
## Analysis of nitroaromatic compounds (explosives)

MN Appl. No. 210350

Column: OPTIMA® δ-3,  
30 m x 0.25 mm ID,  
0.25  $\mu\text{m}$  film, REF 726420.30,  
max. temperature 340/360 °C  
Injection: 1.0  $\mu\text{l}$ , on column, 250 °C  
Carrier gas: 15 psi He  
Temperature: 40 °C (1 min)  $\xrightarrow{10\text{ °C/min}}$  250 °C  
Detector: MSD

### Peaks:

1. Nitrobenzene
2. 2-Nitrotoluene-D<sub>7</sub>
3. 2-Nitrotoluene
4. 3-Nitrotoluene
5. 4-Nitrotoluene
6. 2,6-Dinitrotoluene
7. 1,3-Dinitrotoluene
8. 2,5-Dinitrotoluene
9. 2,3-Dinitrotoluene
10. 2,4-Dinitrotoluene-D<sub>3</sub>
11. 2,4-Dinitrotoluene
12. 3,5-Dinitrotoluene
13. 3,4-Dinitrotoluene



Courtesy of Mr. Steinbach, Inst. of Organic Chemistry, Univ. of Marburg, Germany

# Environmental pollutants

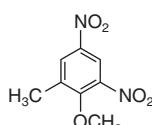
## Analysis of nitromusk compounds in domestic dust MN Appl. No. 211851

Column: OPTIMA® δ-3, 30 m x 0.25 mm ID, 0.25 µm film, REF 726420.30,  
max. temperature 340/360 °C  
Sample: 100 µl/l standard with 2-methyl-4,6-dinitroanisole in *n*-hexane  
Injection: 260 °C, split 10 ml/min  
Carrier gas: H<sub>2</sub>  
Temperature: 150 °C (5 min)  $\xrightarrow{7 \text{ °C/min}}$  290 °C (5 min)  
Detector: ECD 320 °C

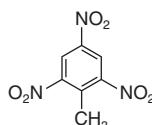
### Peaks for applications 211851

– 211853:

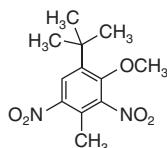
1. 2-Methyl-4,6-dinitroanisole



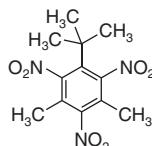
2. 2,4,6-Trinitrotoluene (TNT)



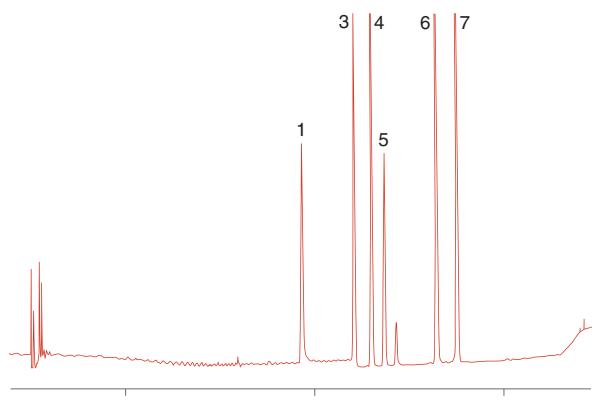
3. Musk ambrette (MA)



4. Musk xylene (MX)

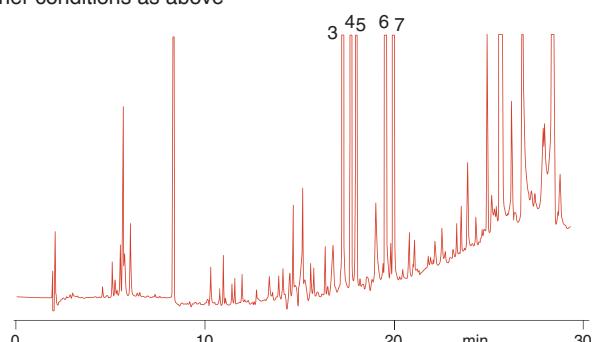


(continued next page)



### MN Appl. No. 211852

Column: OPTIMA® δ-6, 30 m x 0.25 mm ID, 0.25 µm film, REF 726470.30, max. temperature 340/360 °C  
Sample: pooled dust sample extracted by steam distillation with *t*-butyl methyl ether, spiked with 50 µg/l standard and 2-methyl-4,6-dinitroanisole  
Other conditions as above



# Aromatic hydrocarbons



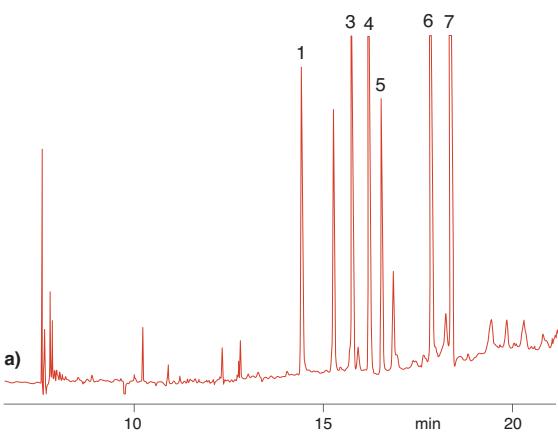
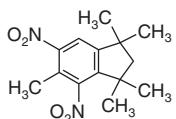
## Analysis of nitromusk compounds in domestic dust (cont.) MN Appl. No. 211853

Samples:  
a) hexane extract of a single dust sample, spiked with 100 µg/l standard and 50 µg/l 2-methyl-4,6-dinitroanisole  
b) hexane extract of a pooled dust sample, spiked with 50 µg/l standard, 2-methyl-4,6-dinitroanisole and 2,4,6-trinitrotoluene (TNT)  
Other conditions as in application 211851

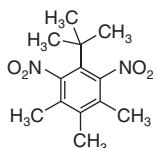
### Peaks for applications 211851

- 211853 (cont.):

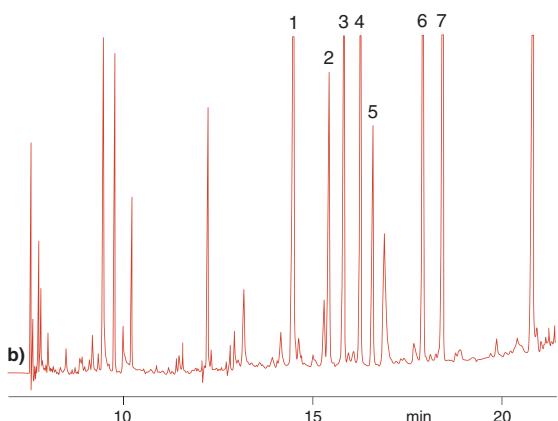
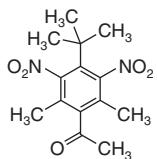
5. Musk moskene (MM)



6. Musk tibetene (MT)



7. Musk ketone (MK)



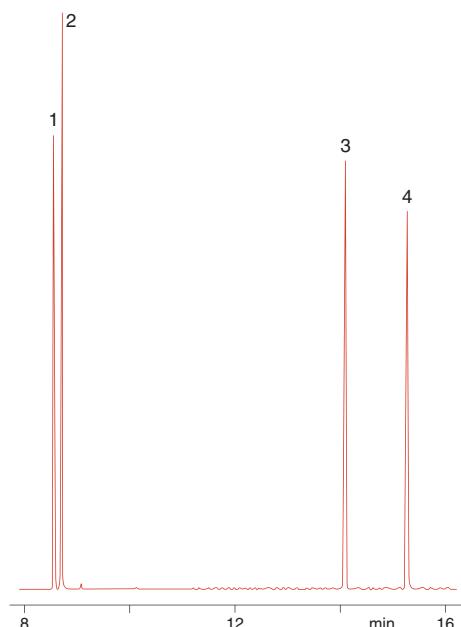
Courtesy of Sven Heegmann, AK Prof. Butte, Diplomarbeit, University of Oldenburg, Germany

## Environmental pollutants

### Analysis of nitroaromatics (EPA 609) MN Appl. No. 250450

Column: OPTIMA® δ-6,  
50 m x 0.20 mm ID, 0.20 µm  
film, REF 726465.50,  
max. temperature 340/360 °C  
Sample: EPA 609 Nitroaromatics and  
Isophorone Mix, 2000 µg/ml  
per substance in hexane  
Injection: 1.0 µl, split 1:50  
Carrier gas: 2.0 bar He  
Temperature: 100 °C (1 min)  $\xrightarrow{10 \text{ °C/min}}$  300 °C  
(5 min)  
Detector: MSD

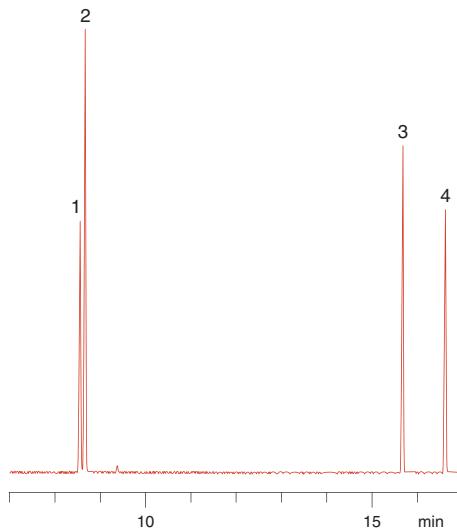
**Peaks:**  
1. Nitrobenzene  
2. Isophorone  
3. 2,6-Dinitrotoluene  
4. 2,4-Dinitrotoluene



### Analysis of nitroaromatics (EPA 609) MN Appl. No. 213200

Column: OPTIMA® 35 MS,  
30 m x 0.25 mm ID,  
0.25 µm film, REF 726154.30,  
max. temperature 360/370 °C  
Injection: 1.0 µl, split 1:50  
Carrier gas: 1 ml/min He  
Temperature: 60 °C  $\xrightarrow{8 \text{ °C/min}}$  250 °C  
Detector: MSD

**Peaks:**  
1. Nitrobenzene  
2. Isophorone  
3. 2,6-Dinitrotoluene  
4. 2,4-Dinitrotoluene



# Aromatic hydrocarbons

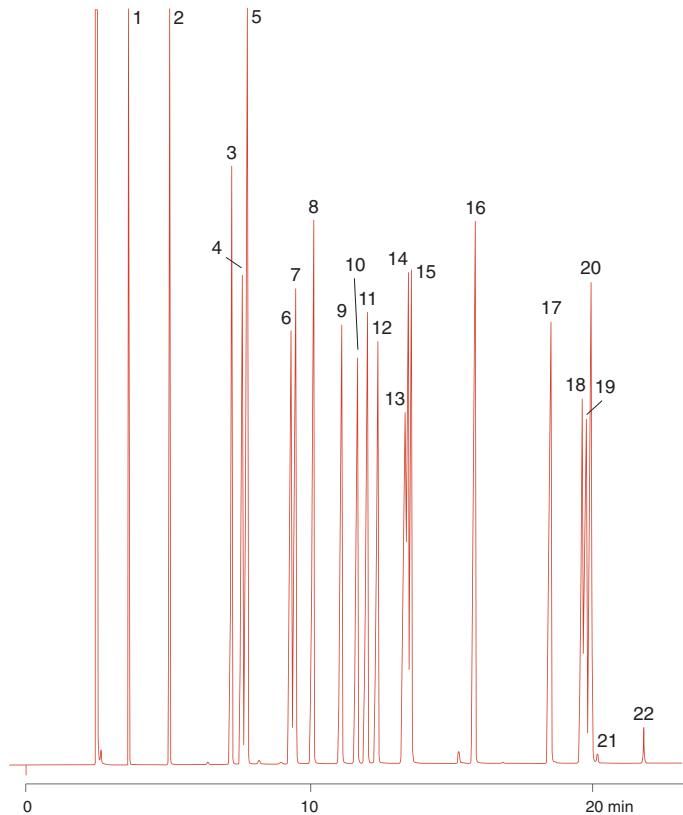


## Analysis of aromatic hydrocarbons MN Appl. No. 200370

Column: OPTIMA® 210, 50 m x 0.25 mm ID, 0.5 µm film, REF 726874.50,  
max. temperature 260/280 °C  
Injection: 0.5 µl, split 64 ml/min  
Carrier gas: 120 kPa H<sub>2</sub> (1.5 ml/min)  
Temperature: 60 °C (10 min)  $\xrightarrow{5\text{ °C/min}}$  120 °C  
Detector: FID 250 °C

**Peaks:**

1. Benzene
2. Toluene
3. Ethylbenzene
4. *p*-Xylene
5. Chlorobenzene  
+ *m*-xylene
6. *o*-Xylene
7. *i*-Propylbenzene
8. Styrene
9. *n*-Propylbenzene
10. Bromobenzene
11. 1,3,5-Trimethylbenzene
12. *o*-Chlorotoluene
13. *m*-Chlorotoluene
14. *p*-Chlorotoluene
15. *p*-Isopropyltoluene
16. *n*-Butylbenzene
17. 2-Methylphenol
18. 2,6-Dichlorotoluene
19. 3-Methylphenol
20. 2,4-Dichlorotoluene  
+ 3-methylphenol
21. 2,5-Dichlorotoluene
22. 2,3-Dichlorotoluene



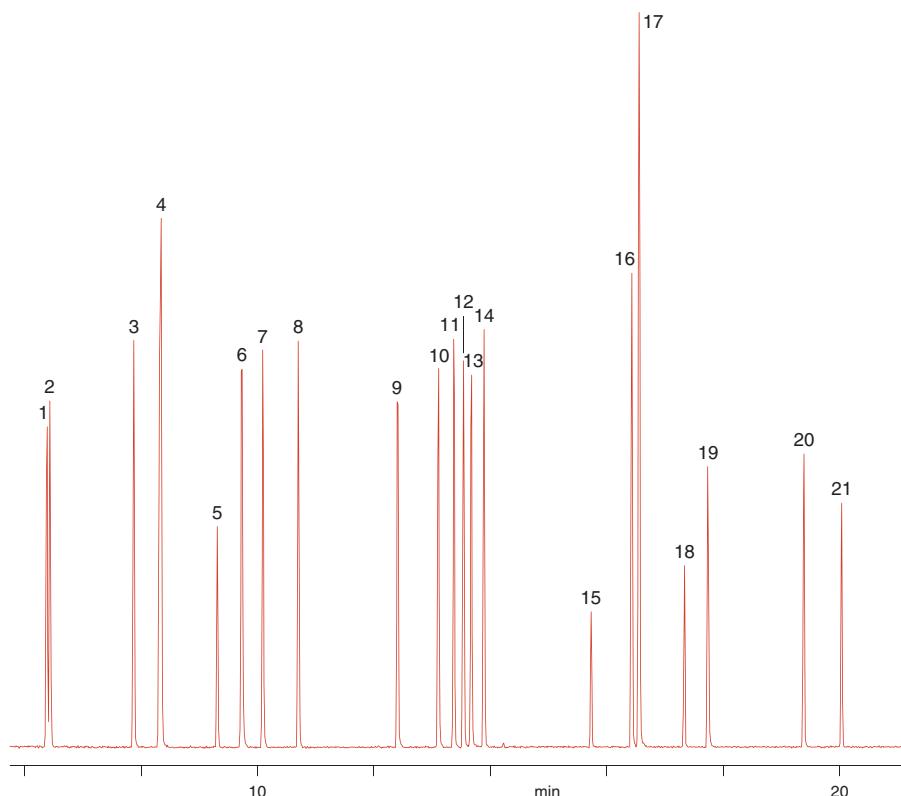
## Environmental pollutants

### Analysis of phenols (EPA 8040 A) MN Appl. No. 200410

Column: OPTIMA® 5, 25 m x 0.20 mm ID, 0.2 µm film, REF 726857.25,  
max. temperature 340/360 °C  
Sample: phenols (EPA 8040 A), 50 µg/ml in 2-propanol  
Injection: 1.0 µl, split 1:150  
Carrier gas: 25 cm/s He  
Temperature: 60 °C (3 min)  $\xrightarrow{8\text{ °C/min}}$  330 °C  
Detector: MSD

**Peaks:**

- |                       |                            |                                   |
|-----------------------|----------------------------|-----------------------------------|
| 1. Phenol             | 8. 2,6-Dichlorophenol      | 15. 2,4-Dinitrophenol             |
| 2. 2-Chlorophenol     | 9. 4-Chloro-3-methylphenol | 16. 2,3,4,6-Tetrachlorophenol     |
| 3. 2-Methylphenol     | 10. 2,3,5-Trichlorophenol  | 17. 2,3,5,6-Tetrachlorophenol     |
| 4. 4-Methylphenol     | 11. 2,4,6-Trichlorophenol  | 18. 2-Methyl-4,6-dinitrophenol    |
| 5. 2-Nitrophenol      | 12. 2,4,5-Trichlorophenol  | 19. 3,4,5-Trichlorophenol         |
| 6. 2,4-Dimethylphenol | 13. 2,3,4-Trichlorophenol  | 20. Pentachlorophenol             |
| 7. 2,4-Dichlorophenol | 14. 2,3,6-Trichlorophenol  | 21. 2-Isopropyl-4,6-dinitrophenol |



# Phenols



## Analysis of isomeric phenols (EPA 8040 A) MN Appl. No. 250060

Column: OPTIMA® δ-3, 60 m x 0.25 mm ID, 0.25 µm film, REF 726420.60,  
max. temperature 340/360 °C

Injection: 1.0 µl, split 1:80

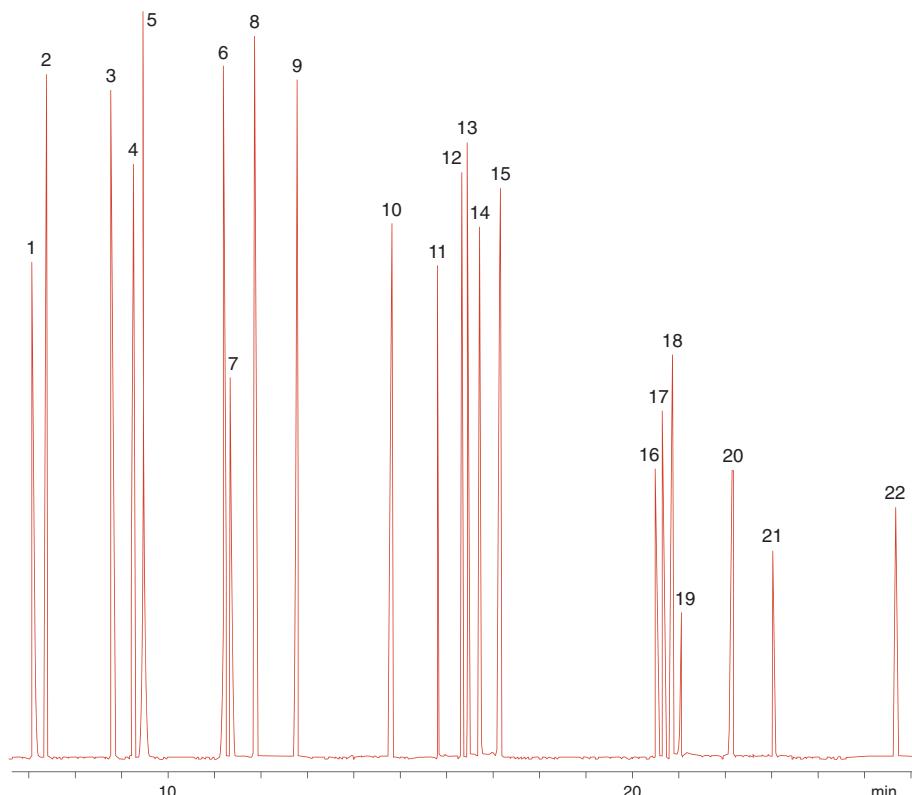
Carrier gas: 1.3 bar He

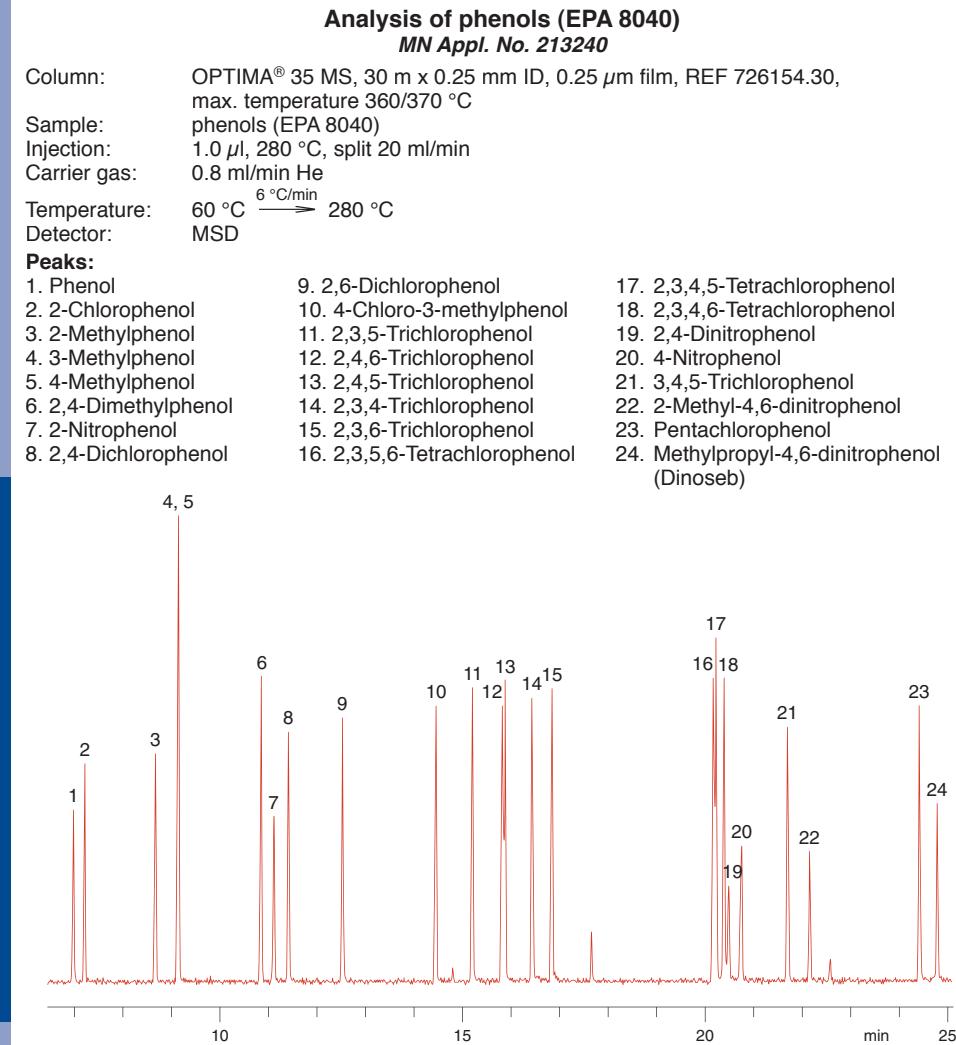
Temperature: 60 °C (3 min)  $\xrightarrow{6\text{ °C/min}}$  320 °C

Detector: MSD

### Peaks:

- |                       |                             |                                   |
|-----------------------|-----------------------------|-----------------------------------|
| 1. Phenol             | 9. 2,6-Dichlorophenol       | 16. 2,3,5,6-Tetrachlorophenol     |
| 2. 2-Chlorophenol     | 10. 4-Chloro-3-methylphenol | 17. 2,3,4,5-Tetrachlorophenol     |
| 3. 2-Methylphenol     | 11. 2,3,5-Trichlorophenol   | 18. 2,3,4,6-Tetrachlorophenol     |
| 4. 4-Methylphenol     | 12. 2,4,6-Trichlorophenol   | 19. 2,4-Dinitrophenol             |
| 5. 3-Methylphenol     | 13. 2,4,5-Trichlorophenol   | 20. 3,4,5-Trichlorophenol         |
| 6. 2,4-Dimethylphenol | 14. 2,3,4-Trichlorophenol   | 21. 2-Methyl-4,6-dinitrophenol    |
| 7. 2-Nitrophenol      | 15. 2,3,6-Trichlorophenol   | 22. 2-Isopropyl-4,6-dinitrophenol |
| 8. 2,4-Dichlorophenol |                             |                                   |





# Phenols



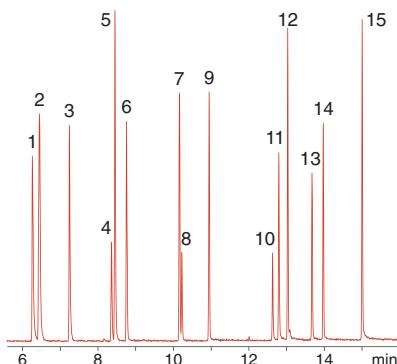
## Analysis of phenols (EPA 528)

MN Appl. No. 212840

Column: OPTIMA® 5 MS Accent, 30 m x 0.25 mm ID, 0.25 µm film, REF 725820.30,  
max. temperature 340/360 °C  
Sample: 1 µl, 5 ppm, 5 ng/compound  
Injection pulsed splitless (hold 0.5 min),  
pulsed pressure 50 psi (hold 0.5 min)  
Carrier gas: 1.3 ml/min He  
Temperature: 40 °C (1 min)  $\xrightarrow{12 \text{ °C/min}}$  200 °C  
 $\xrightarrow{30 \text{ °C/min}}$  300 °C (1 min)  
Detector: MSD 280 °C

### Peaks:

1. Phenol
2. 2-Chlorophenol-3,4,5,6-d<sub>4</sub> + 2-chlorophenol
3. 2-Methylphenol
4. 2-Nitrophenol
5. 2,4-Dimethylphenol-3,5,6-d<sub>3</sub> + 2,4-dimethylphenol
6. 2,4-Dichlorophenol
7. 4-Chloro-3-methylphenol
8. 1,2-Dimethyl-3-nitrobenzene (int. std. 1)
9. 2,4,6-Trichlorophenol
10. 2,4-Dinitrophenol
11. 4-Nitrophenol
12. 2,3,4,5-Tetrachlorophenol (int. std. 2)
13. 2-Methyl-4,6-dinitrophenol
14. 2,4,6-Tribromophenol (surrogate std.)
15. Pentachlorophenol



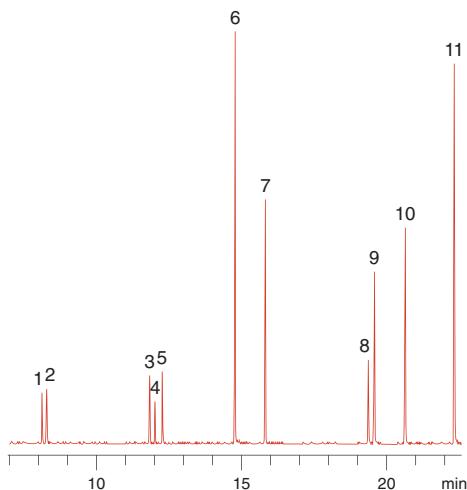
## Analysis of phenols (EPA 604)

MN Appl. No. 213170

Column: OPTIMA® 35 MS, 30 m x 0.25 mm ID, 0.25 µm film, REF 726154.30, max. temperature 360/370 °C  
Injection: 1.0 µl, split 1:20  
Carrier gas: 1.5 ml/min He  
Temperature: 60 °C (4 min)  $\xrightarrow{5 \text{ °C/min}}$  230 °C  
Detector: MSD

### Peaks:

1. Phenol
2. 2-Chlorophenol
3. 2,4-Dimethylphenol
4. 2-Nitrophenol
5. 2,4-Dichlorophenol
6. 4-Chloro-3-methylphenol
7. 2,4,6-Trichlorophenol
8. 2,4-Dinitrophenol
9. 4-Nitrophenol
10. 2-Methyl-4,6-dinitrophenol
11. Pentachlorophenol



# Environmental pollutants

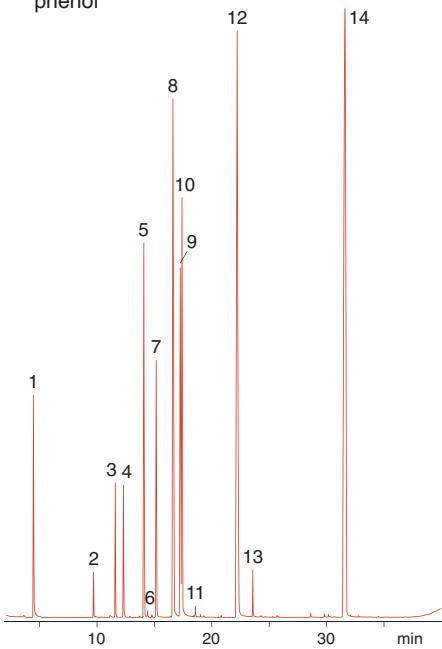
## Analysis of various phenols

*MN Appl. No. 210110*

Column: OPTIMA® 5 MS,  
30 m x 0.25 mm ID, 0.25 µm film,  
REF 726220.30,  
max. temperature 340/360 °C  
Sample: 5 ppm of each compound except  
*N-i*-propylaniline (9.4 ppm)  
Method: SPME  
Temperature: 40 °C (2 min)  $\xrightarrow[20\text{ °C/min}]{6\text{ °C/min}}$  240 °C  
 $\xrightarrow{320\text{ °C}}$   
Detector: MSD

### Peaks:

- |                                          |                              |
|------------------------------------------|------------------------------|
| 1. Toluene-D <sub>8</sub>                | 9. 3-Bromophenol             |
| 2. Phenol                                | 10. 4-Chloro-3-methyl-phenol |
| 3. 2-Methylphenol<br>( <i>o</i> -Cresol) | 11. 2,4-Dibromophenol        |
| 4. Nitrobenzene-D <sub>5</sub>           | 12. 2-Hydroxybiphenyl        |
| 5. <i>N-i</i> -Propylaniline             | 13. 2-Cyclohexylphenol       |
| 6. 2,4-Dichlorophenol                    | 14. Hexafluorobis-phenol A   |
| 7. 4-Chlorophenol                        |                              |
| 8. 4-Bromo-2-chloro-phenol               |                              |



Courtesy of Riedel-de-Haën, Seelze, Germany

## PAH structures

Naphthalene



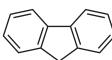
Acenaphthylene



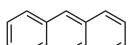
Acenaphthene



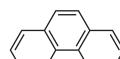
Fluorene



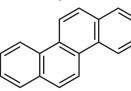
Anthracene



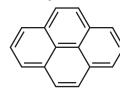
Phenanthrene



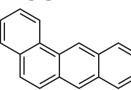
Chrysene



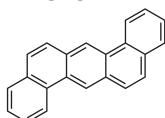
Pyrene



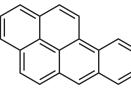
Benz[a]anthracene



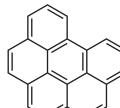
Dibenz[ah]anthracene



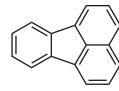
Benzo[a]pyrene



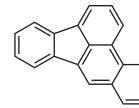
Benzo[ghi]perylene



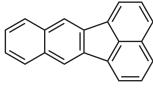
Fluoranthene



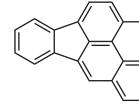
Benzo[b]fluoranthene



Benzo[k]fluoranthene



Indeno[1,2,3-cd]pyrene





### Analysis of 6 PAH according to German drinking water specifications MN Appl. No. 212540

Column: OPTIMA® δ-6, 50 m x 0.2 mm, 0.2 µm film, REF 726465.50,

max. temperature 340/360 °C

Sample: test mixture acc. to German drinking water specifications (REF 722331),  
20 µg/ml in toluene

Injection: 1 µl, 300 °C, splitless

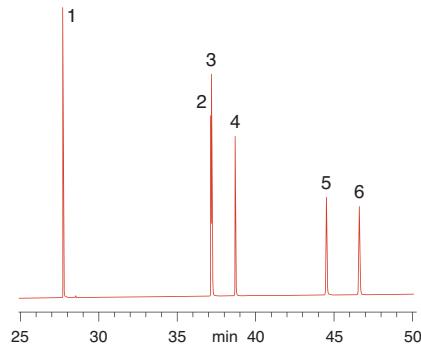
Carrier gas: 0.7 ml/min He

Temperature: 100 °C (1 min)  $\xrightarrow{7\text{ °C/min}}$  340 °C  
(15 min)

Detector: MSD 280 °C

**Peaks:**

1. Fluoranthene
2. Benzo[b]fluoranthene
3. Benzo[k]fluoranthene
4. Benzo[a]pyrene
5. Indeno[1,2,3-cd]pyrene
6. Benzo[ghi]perylene



### Analysis of 16 PAH (EPA 610)

MN Appl. No. 212800

Column: OPTIMA® 5 MS Accent, 30 m x 0.25 mm ID, 0.25 µm film, REF 725820.30,  
max. temperature 340/360 °C

Injection: 0.1 µl (20 µg/ml in toluene), 300 °C, splitless (hold 1 min)

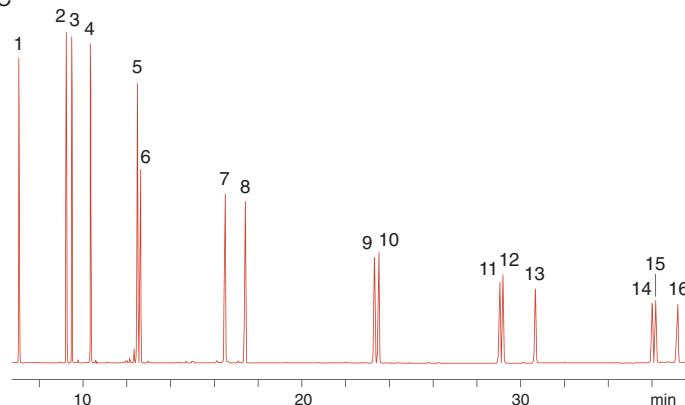
Carrier gas: 40 cm/s H<sub>2</sub>

Temperature: 40 °C (1 min)  $\xrightarrow{20\text{ °C/min}}$  200 °C  $\xrightarrow{4\text{ °C/min}}$  310 °C (5 min)

Detector: FID 310 °C

**Peaks:**

1. Naphthalene
2. Acenaphthylene
3. Acenaphthene
4. Fluorene
5. Phenanthrene
6. Anthracene
7. Fluoranthene
8. Pyrene
9. Benz[a]anthracene
10. Chrysene
11. Benzo[b]fluoranthene
12. Benzo[k]fluoranthene
13. Benzo[a]pyrene
14. Indeno[1,2,3-cd]pyrene
15. Dibenz[ah]anthracene
16. Benzo[ghi]perylene



## Environmental pollutants

### Analysis of 16 PAH (EPA 610) MN Appl. No. 213190

Column: OPTIMA® 35 MS, 30 m x 0.25 mm ID, 0.25 µm film, REF 726154.30,  
max temperature 360/370 °C

Injection: 1 µl, split 1:10

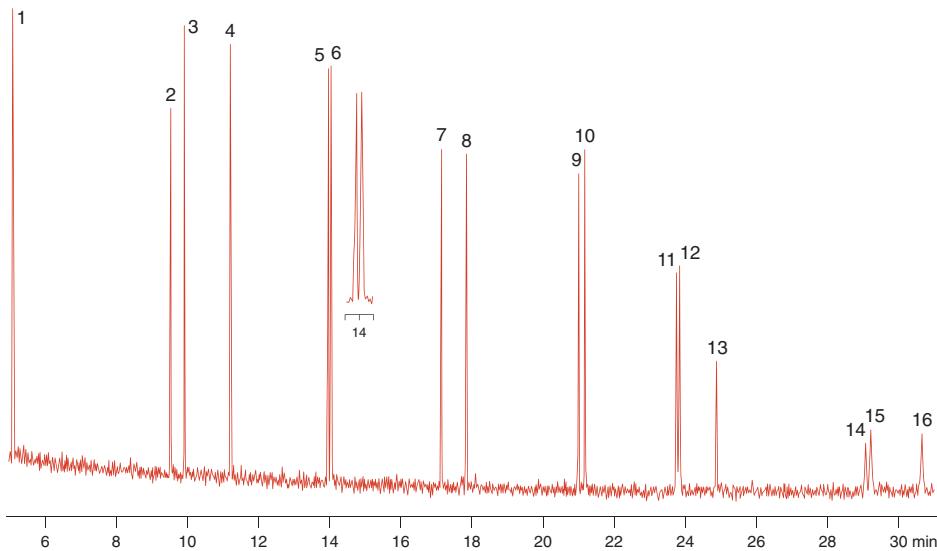
Carrier gas: 0.6 bar H<sub>2</sub>      6 °C/min

Temperature: 100 °C (3 min)      300 °C (10 min)

Detector: MSD

**Peaks:**

- |                   |                 |                          |                            |
|-------------------|-----------------|--------------------------|----------------------------|
| 1. Naphthalene    | 5. Phenanthrene | 9. Benz[a]anthracene     | 13. Benzo[a]pyrene         |
| 2. Acenaphthylene | 6. Anthracene   | 10. Chrysene             | 14. Indeno[1,2,3-cd]pyrene |
| 3. Acenaphthene   | 7. Fluoranthene | 11. Benzo[b]fluoranthene | 15. Dibenz[ah]anthracene   |
| 4. Fluorene       | 8. Pyrene       | 12. Benzo[k]fluoranthene | 16. Benzo[ghi]perylene     |





## Rapid separation of PCB and PAH MN Appl. No. 212920/212930

Column: OPTIMA® XLB, 30 m x 0.25 mm ID, 0.25 µm film, REF 725850.30

max. temperature 340/360 °C

Injection: 1 µl, standard 0.005 ng/µl; 250 °C, pulsed, splitless, pulse 1.38 bar in 1 min

Purge flow: He with purge flow 60 ml/min

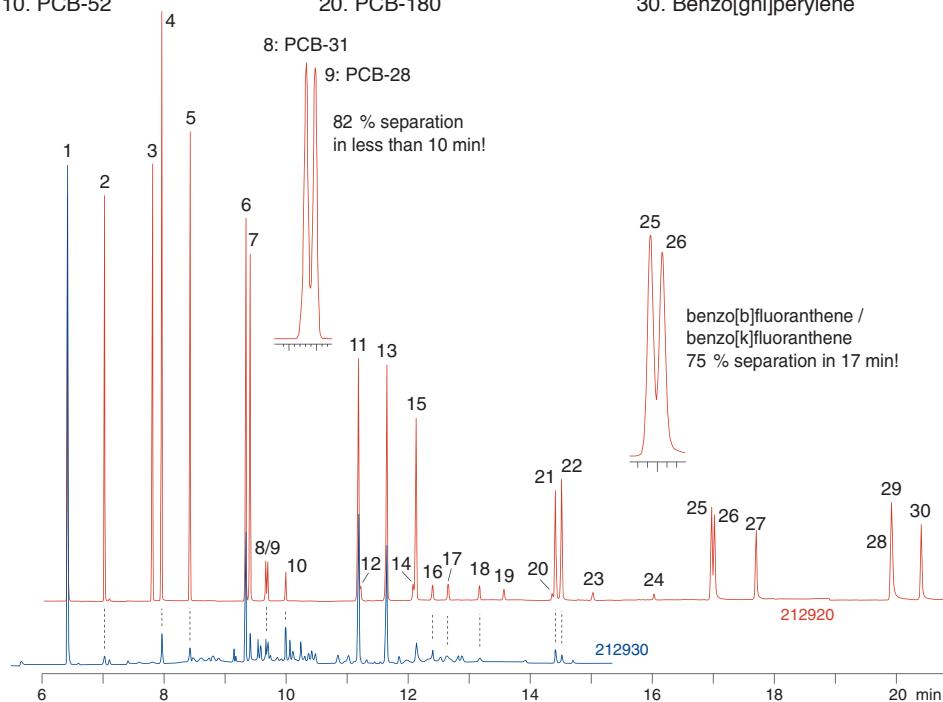
Temperature: 40 °C (2 min)  $\xrightarrow{30 \text{ °C/min}}$  240 °C (2 min)  $\xrightarrow{10 \text{ °C/min}}$  340 °C (5 min)

Detection: MS source 230 °C, interface 280 °C, quadrupol 150 °C

**a) application 212920: standard; b) application 212930 PCB and PAH from slag**

### Peaks:

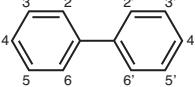
- |                        |                          |                            |
|------------------------|--------------------------|----------------------------|
| 1. Naphthalene         | 11. Fluoranthene         | 21. Benz[a]anthracene      |
| 2. 2-Methylnaphthalene | 12. PCB-101              | 22. Chrysene               |
| 3. Acenaphthylene      | 13. Pyrene               | 23. PCB-169                |
| 4. Acenaphthene        | 14. PCB-77               | 24. PCB-194                |
| 5. Fluorene            | 15. 2-Methylfluoranthene | 25. Benzo[b]fluoranthene   |
| 6. Phenanthrene        | 16. PCB-118              | 26. Benzo[k]fluoranthene   |
| 7. Anthracene          | 17. PCB-153              | 27. Benzo[a]pyrene         |
| 8. PCB-31              | 18. PCB-138              | 28. Dibenz[ah]anthracene   |
| 9. PCB-28              | 19. PCB-126              | 29. Indeno[1,2,3-cd]pyrene |
| 10. PCB-52             | 20. PCB-180              | 30. Benzo[ghi]perylene     |



Courtesy of Centre d'Analyses de Recherche, Lab. d'Hydrologie, Illkirch, France

# Environmental pollutants

## PCB nomenclature

BZ	
	
1	2-Chlorobiphenyl
5	2,3-Dichlorobiphenyl
18	2,2',5-Trichlorobiphenyl
20	2,3,3'-Trichlorobiphenyl
28	2,4,4'-Trichlorobiphenyl
29	2,4,5-Trichlorobiphenyl
31	2,4',5-Trichlorobiphenyl
44	2,2',3,5'-Tetrachlorobiphenyl
47	2,2',4,4'-Tetrachlorobiphenyl
49	2,2',4,5'-Tetrachlorobiphenyl
52	2,2',5,5'-Tetrachlorobiphenyl
61	2,3,4,5-Tetrachlorobiphenyl
66	2,3',4,4'-Tetrachlorobiphenyl
70	2,3',4,5-Tetrachlorobiphenyl
98	2,2',3',4,6-Pentachlorobiphenyl
99	2,2',4,4',5-Pentachlorobiphenyl
101	2,2',4,5,5'-Pentachlorobiphenyl
103	2,2',4,5',6-Pentachlorobiphenyl
105	2,3,3',4,4'-Pentachlorobiphenyl
114	2,3,4,4',5-Pentachlorobiphenyl
118	2,3',4,4',5-Pentachlorobiphenyl
128	2,2',3,3',4,4'-Hexachlorobiphenyl
138	2,2',3,4,4',5-Hexachlorobiphenyl
149	2,2',3,4',5',6-Hexachlorobiphenyl
153	2,2',4,4',5,5'-Hexachlorobiphenyl
154	2,2',4,4',5,6'-Hexachlorobiphenyl
156	2,3,3',4,4',5-Hexachlorobiphenyl
170	2,2',3,3',4,4',5-Heptachlorobiphenyl
171	2,2',3,3',4,4',6-Heptachlorobiphenyl
180	2,2',3,4,4',5,5'-Heptachlorobiphenyl
183	2,2',3,4,4',5',6-Heptachlorobiphenyl
189	2,3,3',4,4',5,5'-Heptachlorobiphenyl
194	2,2',3,3',4,4',5,5'-Octachlorobiphenyl
201	2,2',3,3',4,5',6,6'-Octachlorobiphenyl
209	Decachlorobiphenyl

## Analysis of PCB (Aroclor 1248)

MN Appl. No. 210510

Column: OPTIMA® δ-3,  
50 m x 0.20 mm ID, 0.20 µm  
film, REF 726400.50,  
max. temperature 340/360 °C

Injection: 1.0 µl, split 1:25

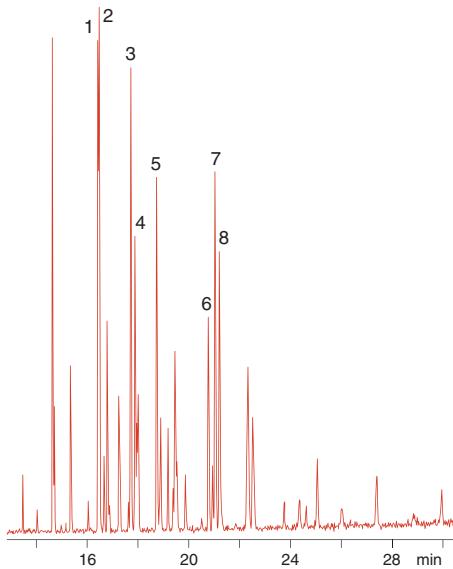
Carrier gas: 25 cm/s He

Temperature: 100 °C  $\xrightarrow{12\text{ °C/min}}$  220 °C  
 $\xrightarrow{1\text{ °C/min}}$  260 °C

Detector: MSD

### Peaks:

1. PCB-31
2. PCB-28
3. PCB-52
4. PCB-49
5. PCB-44
6. PCB-61
7. PCB-70
8. PCB-66



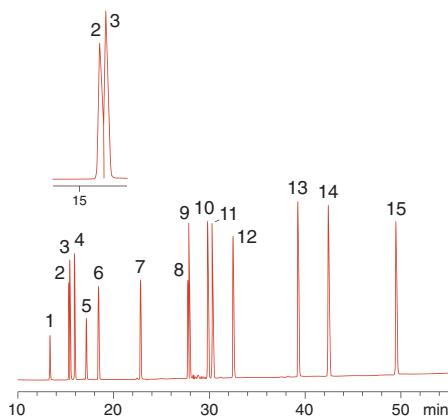


**Analysis of PCB (W22 congener mix)**  
MN Appl. No. 211180

Column: OPTIMA® 5 MS,  
50 m x 0.20 mm ID,  
0.2  $\mu$ m film, REF 726210.50,  
max. temperature: 340/360 °C  
Injection: 1  $\mu$ l PCB W22 congener mix,  
10  $\mu$ l/ml, split 80 ml/min  
Carrier gas: 0.5 bar He  
Temperature: 220 °C  $\xrightarrow{1.5\text{ °C/min}}$  300 °C (15 min)  
Detector: ECD 300 °C

**Peaks:**

- |            |             |
|------------|-------------|
| 1. PCB-18  | 9. PCB-118  |
| 2. PCB-31  | 10. PCB-153 |
| 3. PCB-28  | 11. PCB-105 |
| 4. PCB-20  | 12. PCB-138 |
| 5. PCB-52  | 13. PCB-180 |
| 6. PCB-44  | 14. PCB-170 |
| 7. PCB-101 | 15. PCB-194 |
| 8. PCB-149 |             |

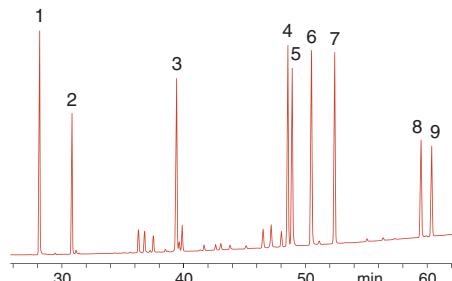


**Analysis of PCB**  
MN Appl. No. 210770

Column: OPTIMA®  $\delta$ -3,  
60 m x 0.25 mm ID, 0.25  $\mu$ m  
film, REF 726420.60,  
max. temperature 340/360 °C  
Injection: 1  $\mu$ l, splitless, 36 pg/45 pg,  
depending on component  
Carrier gas: 2.6 ml/min He  
Temperature: 60 °C (1 min)  $\xrightarrow{1.5\text{ °C/min}}$  200 °C  
 $\xrightarrow{20\text{ °C/min}}$  290 °C (3 min)  
Detector: ECD 300 °C,  
25 ml/min Ar/CH<sub>4</sub> 5%

**Peaks:**

- |            |
|------------|
| 1. PCB-28  |
| 2. PCB-52  |
| 3. PCB-101 |
| 4. PCB-114 |
| 5. PCB-153 |
| 6. PCB-105 |
| 7. PCB-138 |
| 8. PCB-156 |
| 9. PCB-180 |

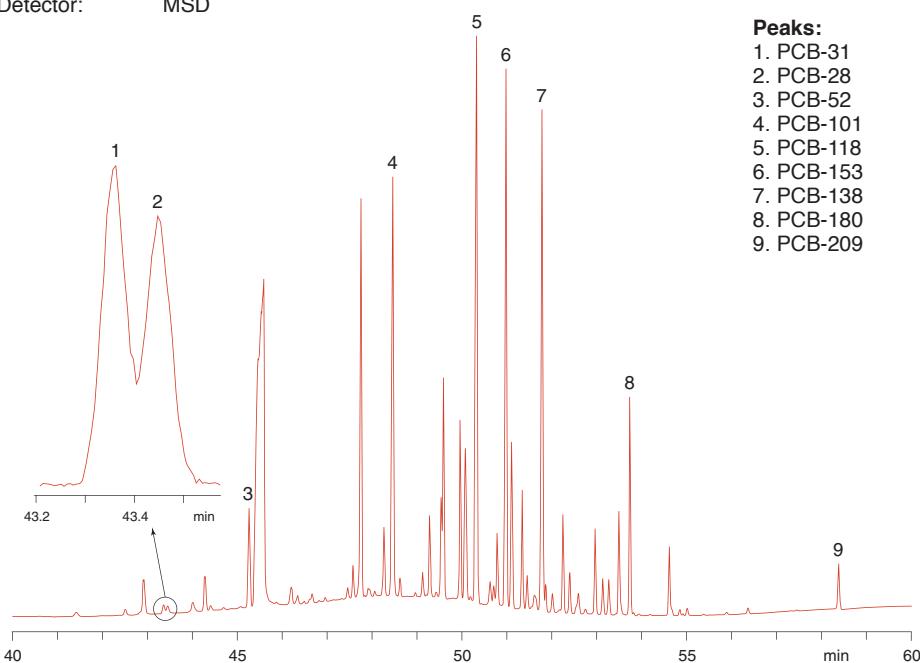


Courtesy of Mr. Bauer, Mr. Drichelt, Zentr. Inst.  
d. San.dienstes BW, Kiel, Germany

## Environmental pollutants

### Analysis of PCB from window putty MN Appl. No. 212071

Column: OPTIMA® 5 MS, 50 m x 0.20 mm ID, 0.2 µm film, REF 726210.50,  
max. temperature 340/360 °C  
Injector temp.: 200 °C  
Carrier gas: 32 psi He  
Temperature: 120 °C (1 min)  $\xrightarrow{10\text{ °C/min}}$  200 °C  $\xrightarrow{15\text{ °C/min}}$  290 °C (5 min)  $\xrightarrow{20\text{ °C/min}}$  320 °C  
Detector: MSD



Courtesy of N. Bertram, LTA Labor f. Toxikol. u. Analytik, Königswinter, Germany

# PCDD • PCDF

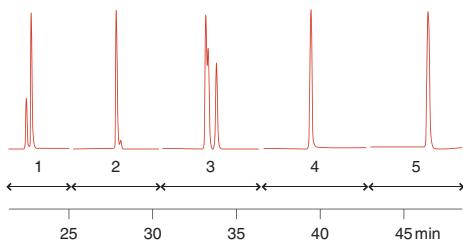


## Analysis of polychlorinated dibenzodioxins (PCDDs)

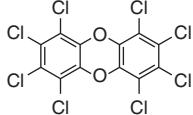
MN Appl. No. 210180

Column: OPTIMA® δ-3,  
60 m x 0.32 mm ID,  
0.25 µm film, REF 726440.60,  
max. temperature 340/360 °C  
Sample: <sup>13</sup>C-labelled standards  
Injection: 1.0 µl, splitless  
Carrier gas: He  
Temperature:  $200\text{ }^{\circ}\text{C} \xrightarrow[2\text{ }^{\circ}\text{C/min}]{4\text{ }^{\circ}\text{C/min}} 280\text{ }^{\circ}\text{C}$   
 $\xrightarrow{} 320\text{ }^{\circ}\text{C (10 min)}$   
Detector: MSD

	Peaks:	m/z
1.	Tetrachlorodibenzodioxins	333.9
2.	Pentachlorodibenzodioxins	367.9
3.	Hexachlorodibenzodioxins	401.9
4.	Heptachlorodibenzodioxins	435.8
5.	Octachlorodibenzodioxin	471.8



Courtesy of Dr. Klostermann, Mr. Ludwig, SGS Intercontrol, Wismar, Germany



Octachlorodibenzodioxin

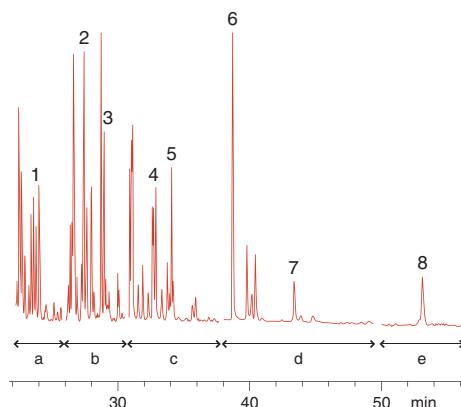
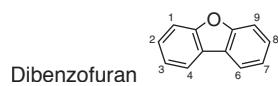
## Analysis of polychlorinated dibenzofurans (PCDFs) in dust

MN Appl. No. 250520

Column: OPTIMA® δ-3,  
60 m x 0.25 mm ID,  
0.25 µm film, REF 726440.60  
max. temperature 340/360 °C  
Injection: 2.0 µl, 6 – 160 pg/µl depending on component  
Carrier gas: 1.4 ml/min He  
Temperature:  $120\text{ }^{\circ}\text{C (1 min)} \xrightarrow[3\text{ }^{\circ}\text{C/min}]{25\text{ }^{\circ}\text{C/min}} 220\text{ }^{\circ}\text{C}$   
 $\xrightarrow{} 300\text{ }^{\circ}\text{C (25 min)}$   
Detector: MSD

Peaks:
1. 2,3,7,8-Tetrachlorodibenzofuran
2. 1,2,3,7,8-Pentachlorodibenzofuran
3. 2,3,4,7,8-Pentachlorodibenzofuran
4. 1,2,3,4,7,8-Hexachlorodibenzofuran + 1,2,3,6,7,8-hexachlorodibenzofuran
5. 2,3,4,6,7,8-Hexachlorodibenzofuran
6. 1,2,3,4,6,7,8-Heptachlorodibenzofuran
7. 1,2,3,4,7,8,9-Heptachlorodibenzofuran
8. Octachlorodibenzofuran

- m/z:  
a) 307  
b) 341  
c) 375  
d) 409  
e) 443



Courtesy of A. Dockwilla, Tredi, Strasbourg, France

# Environmental pollutants

## Analysis of halogenated hydrocarbons, organochlorine pesticides and PCB MN Appl. No. 200530

Column: OPTIMA® 1301, 25 m x 0.32 mm ID, 0.25 µm film, REF 726777.25,  
max. temperature 300/320 °C

Injection: 0.8 µl on column

Carrier gas: 50 kPa H<sub>2</sub>

Temperature: 80 °C  $\xrightarrow{30\text{ °C/min}}$  200 °C  $\xrightarrow{4\text{ °C/min}}$  270 °C

Detector: ECD 310 °C

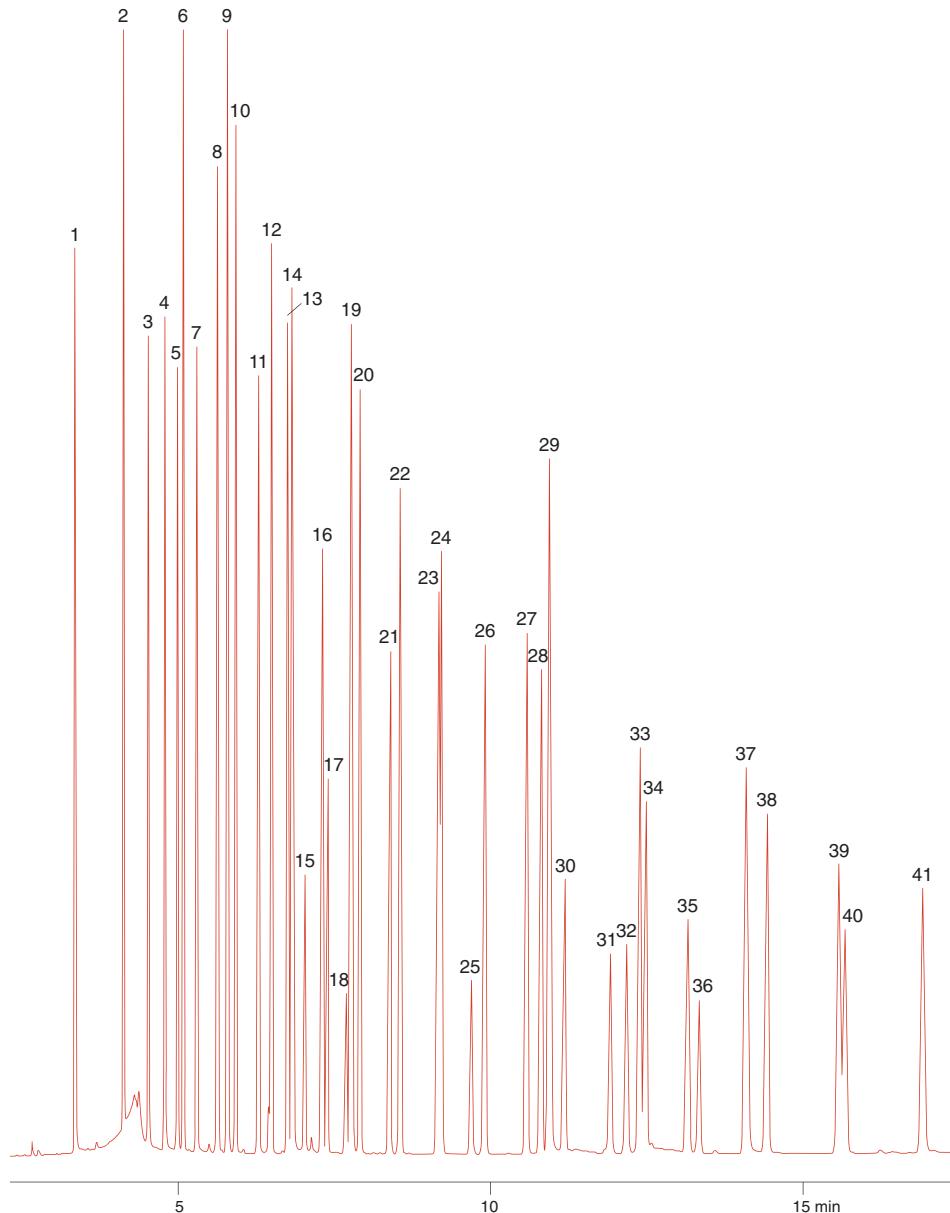
**Peaks:** [pg]

- |                                             |                                            |                                |
|---------------------------------------------|--------------------------------------------|--------------------------------|
| 1. Hexachlorocyclopentadiene [160]          | 14. 2,3,4,5-Tetrachloroaniline [160]       | 27. <i>p,p'</i> -DDE [80]      |
| 2. Etridiazole [160]                        | 15. PCB-28 [80]                            | 28. Dieldrin [80]              |
| 3. <i>α</i> -2,4-Trichloroacetophenone [80] | 16. Heptachlor epoxide [80]                | 29. Chlorfenson [160]          |
| 4. 2,4,5,6-Tetrachloroxylene [80]           | 17. $\beta$ -BHC [80]                      | 30. <i>o,p'</i> -DDD [80]      |
| 5. Tecnazene [80]                           | 18. PCB-52 [80]                            | 31. <i>o,p'</i> -DDT [80]      |
| 6. 2,3,5,6-Tetrachloroaniline [240]         | 19. Methyl pentachlorophenyl sulphide [80] | 32. PCB-153 [80]               |
| 7. Trifluralin [320]                        | 20. $\delta$ -BHC [80]                     | 33. Endosulfan $\beta$ [80]    |
| 8. HCB [80]                                 | 21. Telodrin [80]                          | 34. <i>p,p'</i> -DDD [80]      |
| 9. Pentachloroanisole [80]                  | 22. Octachlorostyrene [80]                 | 35. PCB-138 [80]               |
| 10. <i>α</i> -BHC [80]                      | 23. <i>cis</i> -Heptachlor epoxide [80]    | 36. <i>p,p'</i> -DDT [80]      |
| 11. Quintozene [80]                         | 24. Alodan (int. std.) [80]                | 37. Chlorbenside sulphone [80] |
| 12. Lindane [80]                            | 25. PCB-101 [80]                           | 38. Endosulfan sulphate [80]   |
| 13. Bromocyclen [80]                        | 26. Endosulfan $\alpha$ [80]               | 39. PCB-180 [80]               |
|                                             |                                            | 40. Mirex [80]                 |
|                                             |                                            | 41. Tetradifon [80]            |

### Pesticide structures: bridged diphenyl derivatives

Structure	Compound	X	R <sub>1</sub>	R <sub>2</sub>	R <sub>3</sub>	R <sub>4</sub>	R <sub>5</sub>
	Bifenoxy	-O-		Cl	Cl	H	NO <sub>2</sub>
	Fluorodifen	-O-	CF <sub>3</sub>	NO <sub>2</sub>	H	NO <sub>2</sub>	H
	Tetrasul	-S-	Cl	H	Cl	Cl	Cl
	Tetradifon	-SO <sub>2</sub> -	Cl	H	Cl	Cl	Cl
	Chlorbenside sulphone	-CH <sub>2</sub> -SO <sub>2</sub> -	Cl	H	H	Cl	H
	Chlorfenson	-O-SO <sub>2</sub> -	Cl	H	H	Cl	H
	<i>o,p'</i> -DDD	>CH-CHCl <sub>2</sub>		H	Cl	H	Cl
	<i>p,p'</i> -DDD	>CH-CHCl <sub>2</sub>	Cl	H	H	Cl	H
	<i>o,p'</i> -DDE	>C=CCl <sub>2</sub>		H	Cl	H	Cl
	<i>p,p'</i> -DDE	>C=CCl <sub>2</sub>	Cl	H	H	Cl	H
	<i>o,p'</i> -DDT	>CH-CCl <sub>3</sub>		H	Cl	H	Cl
	<i>p,p'</i> -DDT	>CH-CCl <sub>3</sub>	Cl	H	H	Cl	H
	<i>p,p'</i> -Methoxychlor	>CH-CCl <sub>3</sub>		OCH <sub>3</sub>	H	H	OCH <sub>3</sub>
	Chlorobenzilate	>(OH)-CO-O-C <sub>2</sub> H <sub>5</sub>	Cl	H	H	Cl	H
	Bromopropylate	>(OH)-CO-O-i-C <sub>3</sub> H <sub>7</sub>	Br	H	H	Br	H
	Fenarimol	>(OH)-CO-O-CH <sub>2</sub> -CH=CH <sub>2</sub>	Cl	H	Cl	H	H
	Nuarimol	>(OH)-CO-O-CH <sub>2</sub> -CH=CH <sub>2</sub>	F	H	Cl	H	H

## Halogenated hydrocarbons • Pesticides



Courtesy of Mr. Lembacher, Hipp Nährmittel, Pfaffenhofen, Germany

# Environmental pollutants

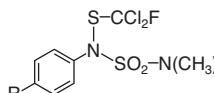
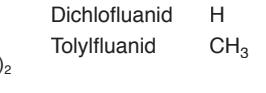
## Analysis of pesticides and PCB MN Appl. No. 210690

Column: OPTIMA® δ-6, 50 m x 0.20 mm ID, 0.20 µm film, REF 726465.50,  
max. temperature 340/360 °C  
 Injection: 0.03 – 0.5 µg/ml depending on component, splitless, purge time 0.8 min, 270 °C  
 Carrier gas: 2.3 ml/min He  
 Temperature: 130 °C (1 min)  $\xrightarrow{25\text{ °C/min}}$  180 °C (5 min)  $\xrightarrow{2\text{ °C/min}}$  260 °C  $\xrightarrow{6\text{ °C/min}}$  310 °C (14 min)  
 Detector: ECD 300 °C

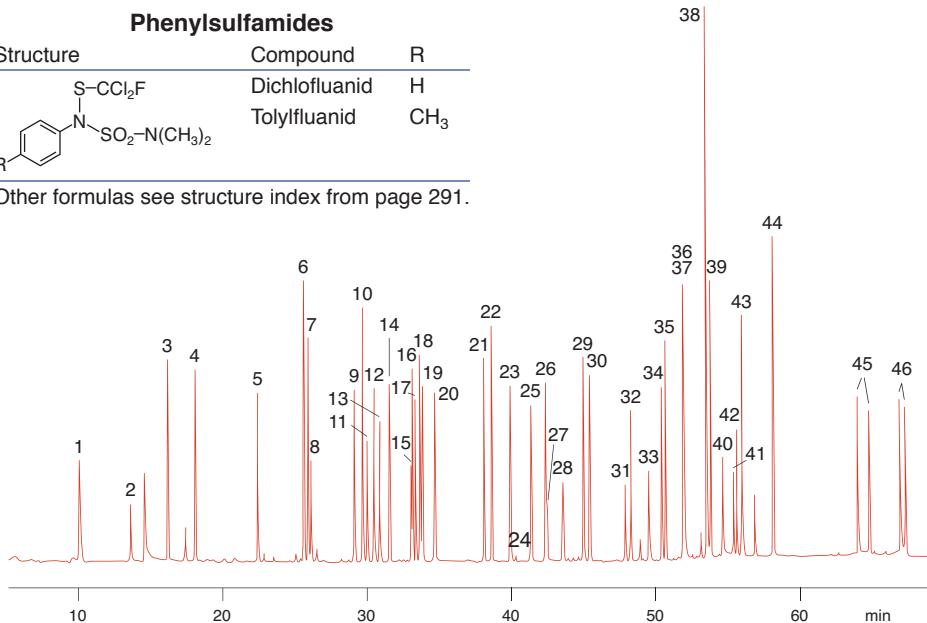
**Peaks:**

- |                         |                       |                  |                               |
|-------------------------|-----------------------|------------------|-------------------------------|
| 1. Dichlobenil          | 13. S 421             | 25. PCB-101      | 37. PCB-138                   |
| 2. Chloroneb            | 14. δ-BHC             | 26. α-Endosulfan | 38. Nuarimol                  |
| 3. Trifluralin          | 15. PCB-52            | 27. Captan       | 39. Endosulfan sulphate       |
| 4. Tecnazene            | 16. Aldrin            | 28. Folpet       | 40. Bromopropylate            |
| 5. α-BHC                | 17. Chlorothalonil    | 29. Dieldrin     | 41. Captafol                  |
| 6. Dichloran            | 18. Chloryrifos-ethyl | 30. o,p'-DDD     | 42. Methoxychlor              |
| 7. γ-BHC                | 19. Dichlofluanid     | 31. Endrin       | 43. PCB-180                   |
| 8. Quintozene           | 20. Triadimefon       | 32. o,p'-DDT     | 44. Tetradifon                |
| 9. β-BHC                | 21. Fluorochloridone  | 33. PCB-153      | 45. Flucythrinate (2 isomers) |
| 10. Vinclozolin         | 22. Tolyfluanid       | 34. Tetrasul     | 46. Flualinate (2 isomers)    |
| 11. PCB-28 + heptachlor | 23. Procymidone       | 35. β-Endosulfan |                               |
| 12. p-Chloroaniline     | 24. Anilazine         | 36. p,p'-DDT     |                               |

**Phenylsulfamides**

Structure	Compound	R
	Dichlofluanid	H
	Tolyfluanid	CH <sub>3</sub>

Other formulas see structure index from page 291.



Courtesy of Mr. Grabher, CLUA, Sigmaringen, Germany

# Chlorinated hydrocarbons • Pesticides



Analysis of chlorinated hydrocarbons, organochlorine pesticides and PCB  
acc. to German Health Administration (BGA 1988)

MN Appl. No. 200700

Column: OPTIMA® 5, 60 m x 0.25 mm ID, 0.25 µm film, REF 726056.60,  
max. temperature 340/360 °C

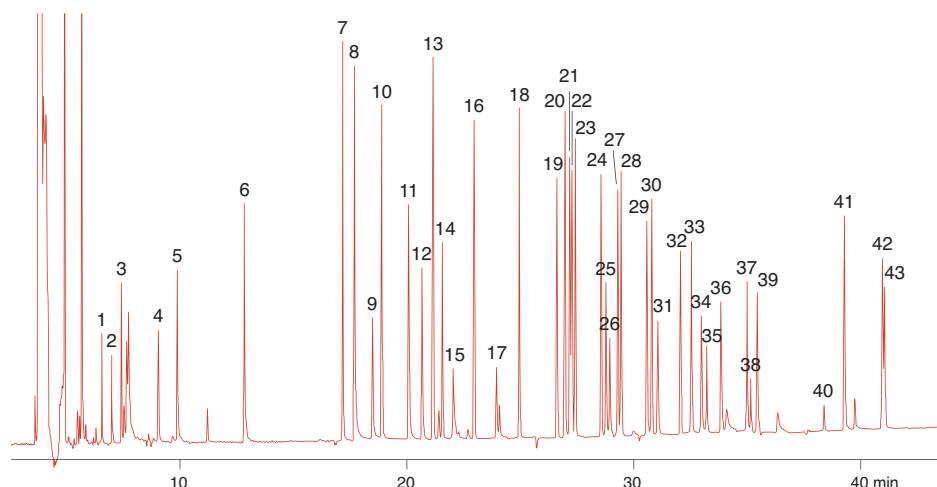
Split: 64 ml/min

Carrier gas: 160 kPa H<sub>2</sub> (2.3 ml/min)

Temperature: 60 °C → 150 °C (1 min) → 300 °C (5 min)  
Detector: ECD 320 °C

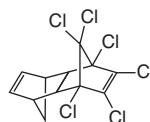
**Peaks:**

- |                               |                            |                              |
|-------------------------------|----------------------------|------------------------------|
| 1. 1,2,3-Trichlorobenzene     | 13. Bromocyclen            | 23. <i>trans</i> -Heptachlor |
| 2. 1,2,4-Trichlorobenzene     | 14. Musk xylene            | 34. <i>p,p'</i> -DDD         |
| 3. 1,3,5-Trichlorobenzene     | 15. PCB-28                 | 35. <i>o,p'</i> -DDT         |
| 4. 1,2,4,5-Tetrachlorobenzene | 16. Heptachlor             | 24. <i>trans</i> -Chlordane  |
| 5. 1,2,3,4-Tetrachlorobenzene | 17. PCB-52                 | 36. PCB-153                  |
| 6. Pentachlorobenzene         | 18. Aldrin                 | 25. <i>o,p'</i> -DDE         |
| 7. α-BHC                      | 19. Isodrin (int. std.)    | 26. PCB-101                  |
| 8. HCB                        | 20. Octachlorostyrene      | 27. Endosulfan α             |
| 9. β-BHC                      | 21. <i>cis</i> -Heptachlor | 28. <i>cis</i> -Chlordane    |
| 10. γ-BHC (lindane)           | epoxide                    | 29. <i>p,p'</i> -DDE         |
| 11. δ-BHC                     | 22. Oxychlordane           | 30. Dieldrin                 |
| 12. ε-BHC                     |                            | 31. <i>o,p'</i> -DDD         |
|                               |                            | 32. Endrin                   |
|                               |                            | 33. Endosulfan β             |
|                               |                            | 34. <i>p,p'</i> -DDT         |
|                               |                            | 35. PCB-138                  |
|                               |                            | 36. PCB-180                  |
|                               |                            | 37. Endosulfan sulphate      |
|                               |                            | 38. PCB-170                  |
|                               |                            | 39. Mirex                    |

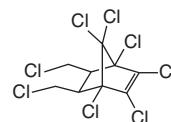


## Organochlorine pesticide structures

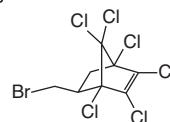
Aldrin



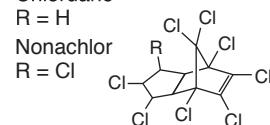
Alodan



Bromocyclen



Chlordane



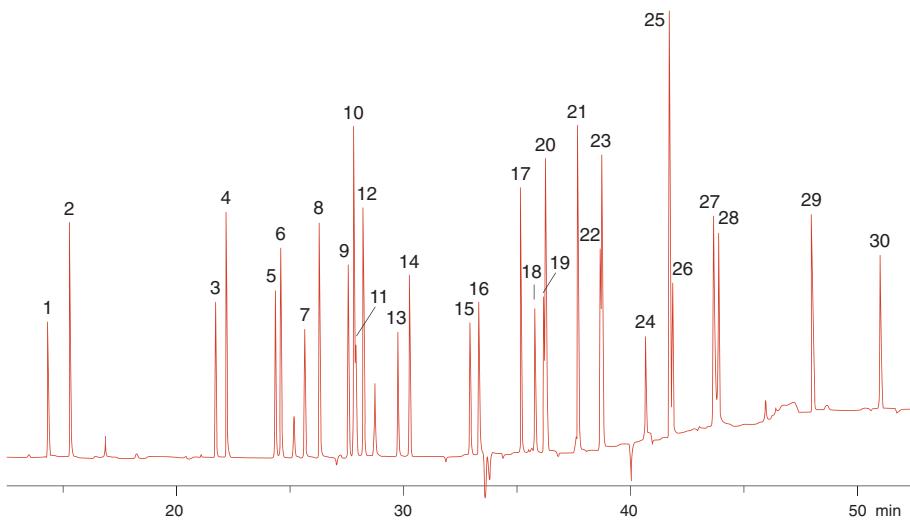
# Environmental pollutants

## Analysis of organochlorine pesticides and PCB MN Appl. No. 210230

Column: OPTIMA® δ-3, 50 m x 0.20 mm ID, 0.20 µm film, REF 726400.50,  
max. temperature 340/360 °C  
Injection: 0.01 – 0.15 µg/ml depending on component, splitless, purge time 0.5 min  
Carrier gas: 1.37 ml/min He  
Temperature: 130 °C (1 min)  $\xrightarrow{25\text{ °C/min}}$  180 °C (5 min)  $\xrightarrow{3\text{ °C/min}}$  260 °C (2 min)  $\xrightarrow{4\text{ °C/min}}$  300 °C  
(20 min)  
Detector: ECD

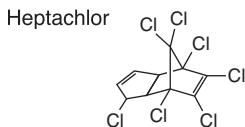
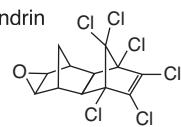
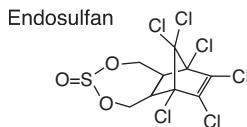
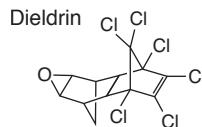
**Peaks:**

- |                       |                             |                             |                      |
|-----------------------|-----------------------------|-----------------------------|----------------------|
| 1. Chloroneb          | 9. PCB-28                   | 17. <i>o,p'</i> -DDE        | 25. <i>p,p'</i> -DDD |
| 2. Pentachlorobenzene | 10. <i>p</i> -Chloroaniline | 18. PCB-101                 | 26. PCB-153          |
| 3. α-BHC              | 11. Heptachlor              | 19. <i>trans</i> -Nonachlor | 27. <i>p,p'</i> -DDT |
| 4. HCB                | 12. δ-BHC                   | 20. <i>cis</i> -Chlordane   | 28. PCB-138          |
| 5. γ-BHC              | 13. PCB-52                  | 21. <i>p,p'</i> -DDE        | 29. PCB-180          |
| 6. Quintozene         | 14. Aldrin                  | 22. Dieldrin                | 30. Mirex            |
| 7. Bromocyclen        | 15. Isodrin                 | 23. <i>o,p'</i> -DDD        |                      |
| 8. β-BHC              | 16. Oxychlordane            | 24. Endrin                  |                      |



Courtesy of Mr. Grabher, CLUA, Sigmaringen, Germany

### Organochlorine pesticide structures



# Organochlorine Pesticides • PCB

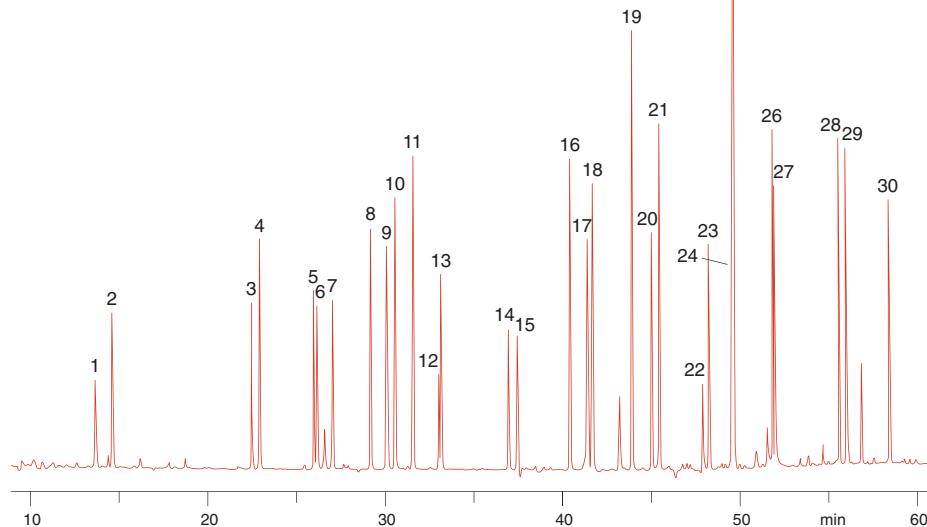


## Analysis of organochlorine pesticides and PCB MN Appl. No. 210700

Column: OPTIMA® δ-6, 50 m x 0.20 mm ID, 0.20 µm film, REF 726465.50,  
max. temperature 340/360 °C  
Injection: 0.03 – 0.5 µg/ml depending on component, splitless, purge time 0.8 min, 270 °C  
Carrier gas: 2.3 ml/min He  
Temperature: 130 °C (1 min)  $\xrightarrow{25\text{ °C/min}}$  180 °C (5 min)  $\xrightarrow{2\text{ °C/min}}$  260 °C  $\xrightarrow{6\text{ °C/min}}$  310 °C (14 min)  
Detector: ECD 300 °C

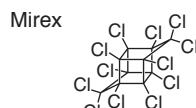
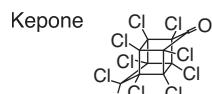
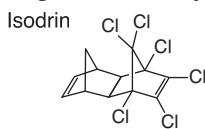
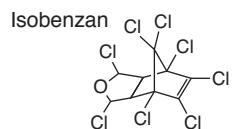
### Peaks:

- |                             |                           |                      |
|-----------------------------|---------------------------|----------------------|
| 1. Chloroneb                | 11. δ-BHC                 | 21. <i>o,p'</i> -DDD |
| 2. Pentachlorobenzene       | 12. PCB-52                | 22. Endrin           |
| 3. α-BHC                    | 13. Aldrin                | 23. <i>o,p'</i> -DDT |
| 4. HCB                      | 14. Isodrin               | 24. PCB-153          |
| 5. γ-BHC                    | 15. Oxychlordane          | 25. <i>p,p'</i> -DDD |
| 6. Quintozene               | 16. <i>o,p'</i> -DDE      | 26. <i>p,p'</i> -DDT |
| 7. Bromocyclen              | 17. PCB-101               | 27. PCB-138          |
| 8. β-BHC                    | 18. <i>cis</i> -Chlordane | 28. Methoxychlor     |
| 9. PCB-28 + heptachlor      | 19. <i>p,p'</i> -DDE      | 29. PCB-180          |
| 10. <i>p</i> -Chloroaniline | 20. Dieldrin              | 30. Mirex            |



Courtesy of Mr. Grabher, CLUA, Sigmaringen, Germany

### Organochlorine pesticide structures



## Environmental pollutants

### Analysis of halogenated hydrocarbons, organochlorine pesticides and PCB MN Appl. No. 250220

Column: OPTIMA® 1701, 50 m x 0.20 mm ID, 0.20 µm film, REF 726841.50,  
max. temperature 300/320 °C

Injection: accu-pesticide test mixture, 20 µg/ml, split 1:10

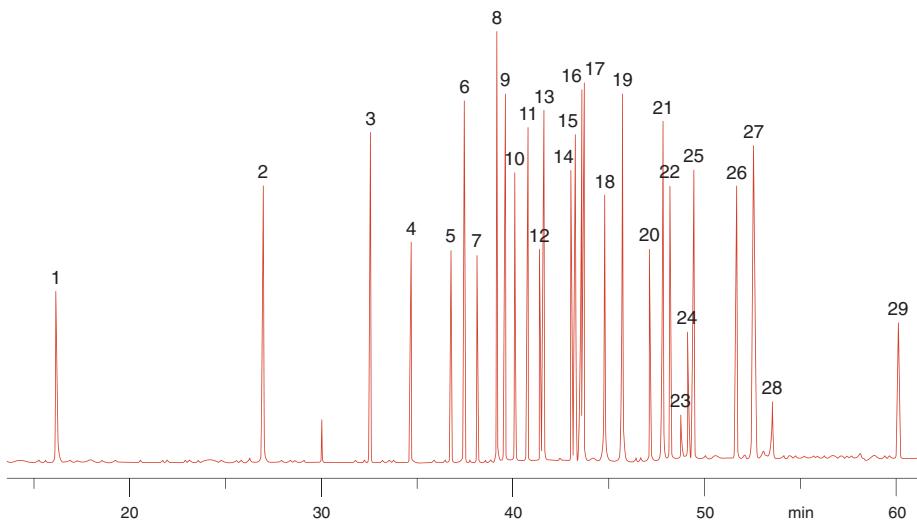
Carrier gas: 1.1 bar He 4 °C/min

Temperature: 100 °C (3 min) → 250 °C, then isothermal

Detector: MSD EI

**Peaks:**

- |                        |                          |                            |
|------------------------|--------------------------|----------------------------|
| 1. Hexachlorobutadiene | 11. Isobenzan            | 21. <i>o,p'</i> -DDD       |
| 2. Pentachlorobenzene  | 12. δ-BHC                | 22. PCB-118                |
| 3. Hexachlorobenzene   | 13. Isodrin              | 23. Endrin                 |
| 4. α-BHC               | 14. Heptachlor epoxide A | 24. <i>p,p'</i> -DDD       |
| 5. β-BHC               | 15. Heptachlor epoxide B | 25. PCB-138                |
| 6. PCB-28              | 16. PCB-101              | 26. <i>o,p'</i> -DDT       |
| 7. Heptachlor          | 17. <i>o,p'</i> -DDE     | 27. PCB-153 + endosulfan 2 |
| 8. PCB-52              | 18. Endosulfan 1         | 28. <i>p,p'</i> -DDT       |
| 9. Aldrin              | 19. <i>p,p'</i> -DDE     | 29. PCB-180                |
| 10. γ-BHC              | 20. Dieldrin             |                            |



# Organochlorine pesticides • PCB

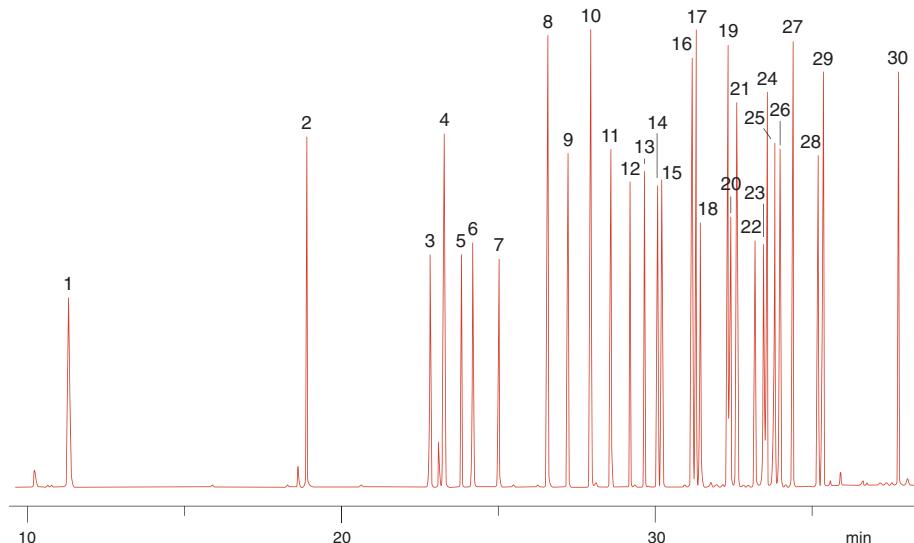


## Analysis of halogenated hydrocarbons, organochlorine pesticides and PCB MN Appl. No. 250350

Column: OPTIMA® 5 MS, 25 m x 0.20 mm ID, 0.35 µm film, REF 726215.25,  
max. temperature 340/360 °C  
Sample: accu-pesticide test mixture, 10 ng/peak  
Injection: 1 µl, 1 min splitless  
Carrier gas: 30 cm/s He    5 °C/min  
Temperature: 75 °C (1 min) → 280 °C  
Detector: MSD

**Peaks:**

- |                        |                          |                      |
|------------------------|--------------------------|----------------------|
| 1. Hexachlorobutadiene | 11. Aldrin               | 21. <i>o,p'</i> -DDD |
| 2. Pentachlorobenzene  | 12. Isobenzan            | 22. Endrin           |
| 3. α-BHC               | 13. Isodrin              | 23. Endosulfan 2     |
| 4. Hexachlorobenzene   | 14. Heptachlor epoxide A | 24. PCB-118          |
| 5. β-BHC               | 15. Heptachlor epoxide B | 25. <i>p,p'</i> -DDD |
| 6. γ-BHC               | 16. <i>o,p'</i> -DDE     | 26. <i>o,p'</i> -DDT |
| 7. δ-BHC               | 17. PCB-101              | 27. PCB-153          |
| 8. PCB-28              | 18. Endosulfan 1         | 28. <i>p,p'</i> -DDT |
| 9. Heptachlor          | 19. <i>p,p'</i> -DDE     | 29. PCB-138          |
| 10. PCB-52             | 20. Dieldrin             | 30. PCB-180          |



# Environmental pollutants

## Analysis of organochlorine pesticides and PCB MN Appl. No. 210500

Column: OPTIMA® δ-3, 60 m x 0.25 mm ID, 0.25 µm film, REF 726420.60,  
max. temperature 340/360 °C

Injection: 1 µl, 3 – 18 pg/µl depending on component, 270 °C

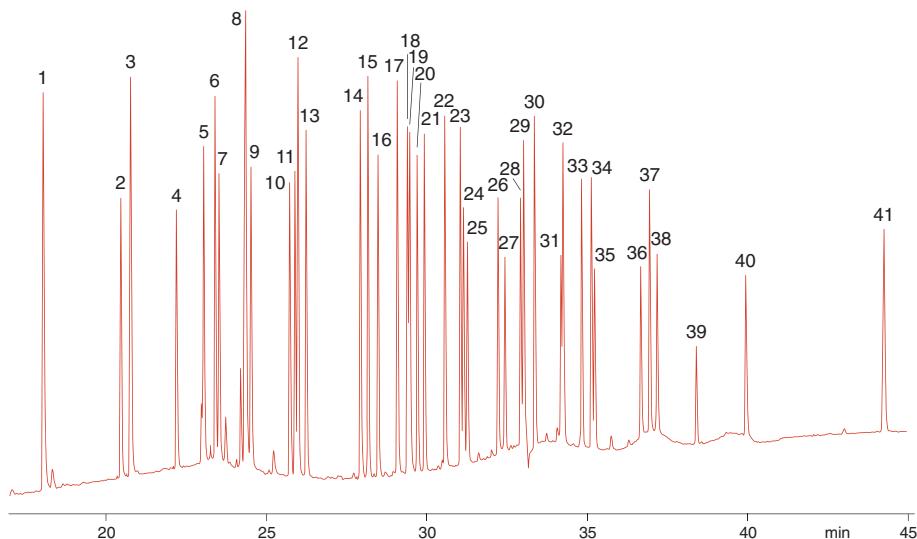
Carrier gas: 1.37 ml/min H<sub>2</sub> (1.76 bar)

Temperature: 120 °C (1 min)  $\xrightarrow{5\text{ °C/min}}$  300 °C (45 min)

Detector: ECD 300 °C

**Peaks:**

- |                                |                                      |                         |
|--------------------------------|--------------------------------------|-------------------------|
| 1. TCNB = 1,2,4,5-Tetrachloro- | 14. Oxychlordane                     | 28. <i>p,p'</i> -DDD    |
| 3-nitrobenzene = Tecnazene     | 15. <i>cis</i> -Heptachlor epoxide   | 29. PCB-153             |
| 2. $\alpha$ -BHC               | 16. <i>trans</i> -Heptachlor epoxide | 30. $\beta$ -Endosulfan |
| 3. HCB                         | 17. <i>o,p'</i> -DDE                 | 31. <i>p,p'</i> -DDT    |
| 4. Lindane                     | 18. <i>trans</i> -Chlordane          | 32. PCB-138             |
| 5. Bromocyclen                 | 19. PCB-101                          | 33. PCB-183             |
| 6. Musk xylene                 | 20. <i>cis</i> -Chlordane            | 34. Endosulfan sulphate |
| 7. $\beta$ -BHC                | 21. $\alpha$ -Endosulfan             | 35. Parlar 50           |
| 8. PCB-28/31                   | 22. <i>p,p'</i> -DDE                 | 36. PCB-156             |
| 9. Heptachlor                  | 23. Dieldrin                         | 37. PCB-180             |
| 10. PCB-52                     | 24. <i>o,p'</i> -DDD                 | 38. Ketoendrin          |
| 11. PCB-49                     | 25. Parlar 26                        | 39. Parlar 62           |
| 12. Aldrin                     | 26. Endrin                           | 40. PCB-189             |
| 13. Musk ketone                | 27. <i>o,p'</i> -DDT                 | 41. Deca-PCB = PCB-209  |



Courtesy of Mr. Münch, Staatl. Med. Lebensm. u. Vet. UA, Gießen, Germany

# Organochlorine pesticides • PCB



## Analysis of organochlorine pesticides and PCB MN Appl. No. 200610

Column: OPTIMA® 17, 50 m x 0.25 mm ID, 0.25 µm film, REF 726022.50,

max. temperature 320/340 °C

Injection: 0.5 µl (100 pg each), splitless, 280 °C

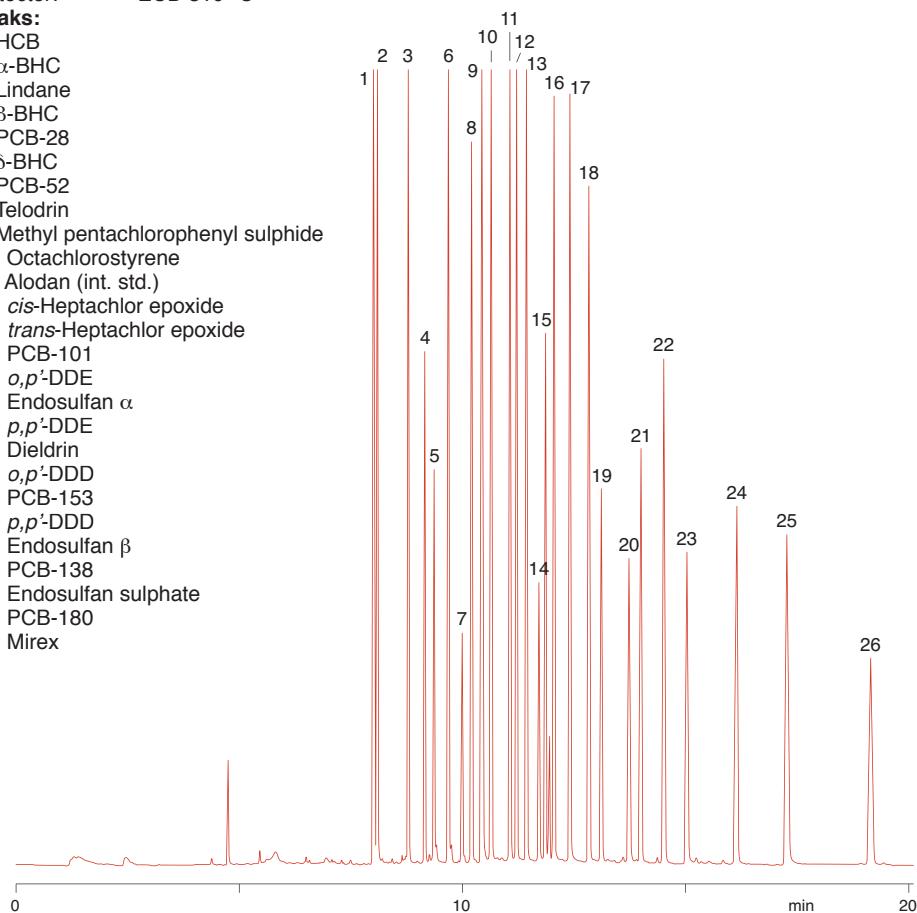
Carrier gas: 250 kPa H<sub>2</sub>

Temperature: 80 °C  $\xrightarrow{30\text{ °C/min}}$  240 °C  $\xrightarrow{6\text{ °C/min}}$  280 °C

Detector: ECD 310 °C

**Peaks:**

1. HCB
2.  $\alpha$ -BHC
3. Lindane
4.  $\beta$ -BHC
5. PCB-28
6.  $\delta$ -BHC
7. PCB-52
8. Telodrin
9. Methyl pentachlorophenyl sulphide
10. Octachlorostyrene
11. Alodan (int. std.)
12. *cis*-Heptachlor epoxide
13. *trans*-Heptachlor epoxide
14. PCB-101
15. *o,p'*-DDE
16. Endosulfan  $\alpha$
17. *p,p'*-DDE
18. Dieldrin
19. *o,p'*-DDD
20. PCB-153
21. *p,p'*-DDD
22. Endosulfan  $\beta$
23. PCB-138
24. Endosulfan sulphate
25. PCB-180
26. Mirex



Courtesy of Mr. Lembacher, Hipp Nährmittel, Pfaffenhofen, Germany

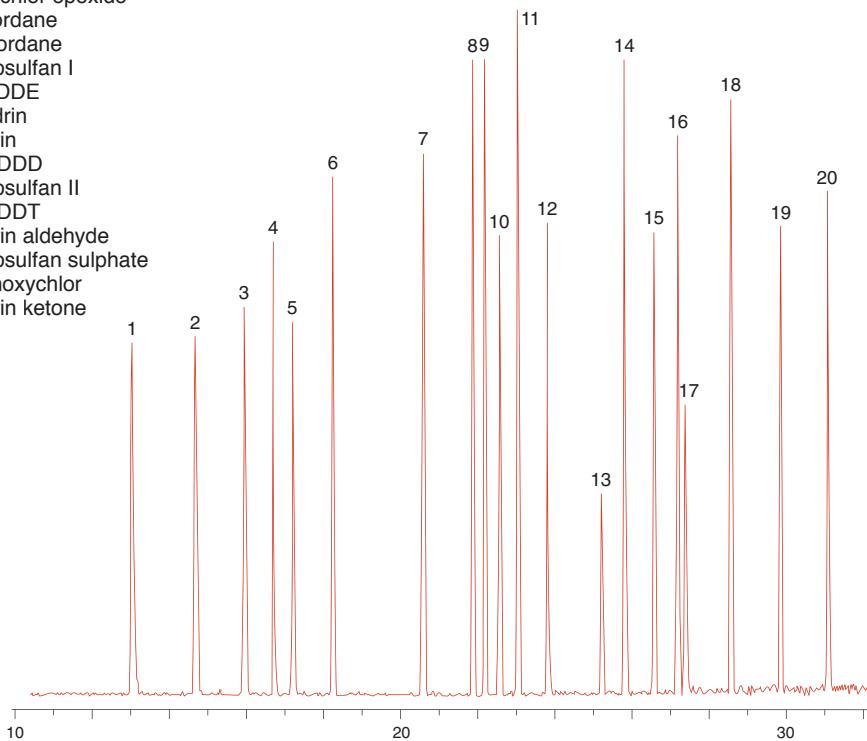
## Environmental pollutants

### Separation of organochlorine pesticides (EPA 8081) MN Appl. No. 250430

Column: OPTIMA® δ-6, 50 m x 0.20 mm ID, 0.2 µm film, REF 726465.50,  
max. temperature 340/360 °C  
Sample: EPA 8081 organochlorine pesticide calibration mix (Restek), 200 µg/ml each in  
toluene – hexane (1:1, v/v)  
Injection: 1 µl, split 1:30  
Carrier gas: 2.0 bar He  
Temperature: 180 °C  $\xrightarrow{4\text{ °C/min}}$  300 °C (10 min)  
Detector: MSD

**Peaks:**

1. α-BHC
2. γ-BHC (lindane)
3. β-BHC
4. Heptachlor
5. δ-BHC
6. Aldrin
7. Heptachlor epoxide
8. γ-Chlordane
9. α-Chlordane
10. Endosulfan I
11. 4,4'-DDE
12. Dieldrin
13. Endrin
14. 4,4'-DDD
15. Endosulfan II
16. 4,4'-DDT
17. Endrin aldehyde
18. Endosulfan sulphate
19. Methoxychlor
20. Endrin ketone



# Organochlorine pesticides



## Separation of organochlorine pesticides (EPA 608) MN Appl. No. 213220

Column: OPTIMA® 35 MS, 30 m x 0.25 mm ID, 0.25 µm film, REF 726154.30,

max. temperature 360/370 °C

Sample: EPA 608 organochlorine pesticide mix

Injection: 1 µl, split 20 ml/min

Carrier gas: 0.8 ml/min He

Temperature: 160 °C  $\xrightarrow{6 \text{ °C/min}}$  260 °C (10 min)

Detector: MSD

**Peaks:**

1.  $\alpha$ -BHC

2.  $\gamma$ -BHC (lindane)

3.  $\beta$ -BHC

4. Heptachlor

5.  $\delta$ -BHC

6. Aldrin

7. Heptachlor epoxide

8. Endosulfan I

9. 4,4'-DDE

10. Dieldrin

11. Endrin

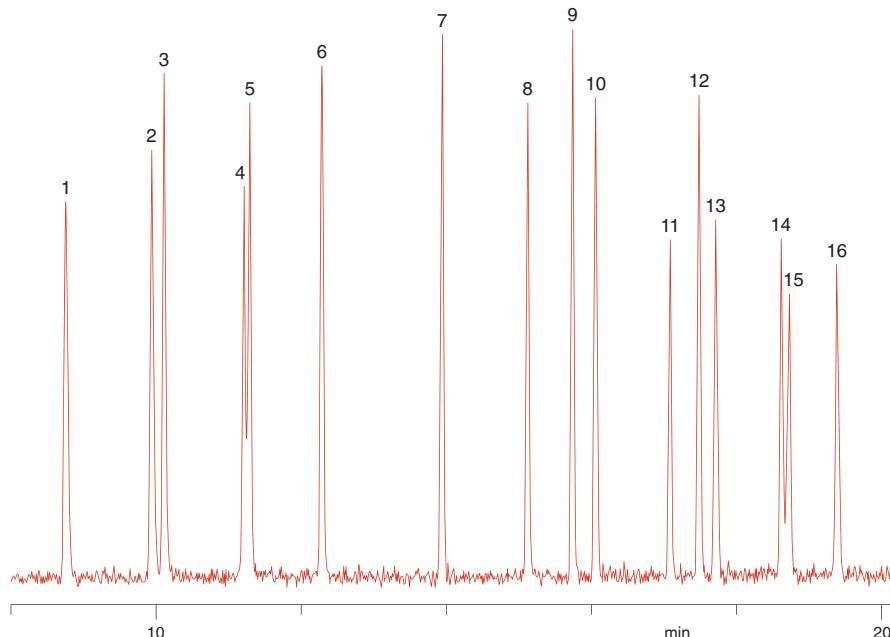
12. 4,4'-DDD

13. Endosulfan II

14. 4,4'-DDT

15. Endrin aldehyde

16. Endosulfan sulphate



# Environmental pollutants

## Analysis of organochlorine pesticides from cod liver oil MN Appl. No. 213270

Column: OPTIMA® δ-3, 60 m x 0.25 mm ID, 0.25 µm film, REF 726420.60, max. temperature 340/360 °C

Sample: a) pesticide standard (150 pg/µl of each compound)  
b) NP-HPLC group separation of NIST reference material SRM 1588 (semivolatile organohalogen compounds in cod liver oil) on 250 x 4 mm NUCLEOSIL® 100-10 NH<sub>2</sub> using 10 ml *n*-hexane (fraction 1) and 32 ml *n*-hexane – dichloromethane (90:10 v/v, fraction 2), flow rate 1 ml/min (see application 250510 for chromatogram of fraction 1)

Injection: 2 µl of fraction 2, on column

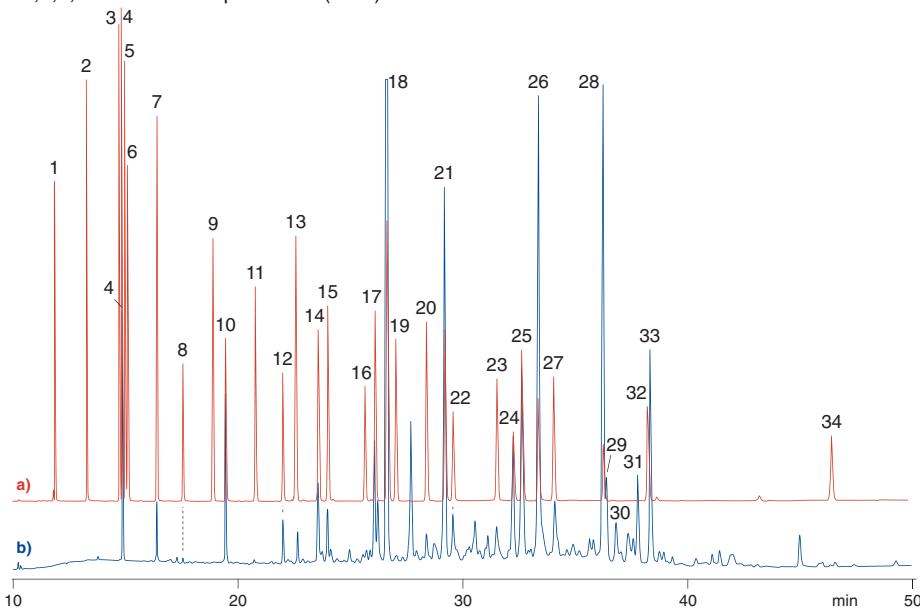
Carrier gas: 180 kPa H<sub>2</sub> 20 °C/min

Temperature: 80 °C (3 min) → 200 °C 1.5 °C/min → 290 °C (5 min)

Detector: ECD 300 °C

**Peaks:**

- |                                          |                                                        |                           |
|------------------------------------------|--------------------------------------------------------|---------------------------|
| 1. Pentachlorobenzene                    | 13. PCB-103                                            | 24. 2,4'-DDT              |
| 2. 2,4,6-Tribromoanisole                 | 14. Oxychlordane                                       | 25. <i>cis</i> -Nonachlor |
| 3. Tetrachloro-1,4-dimethoxybenzene      | 15. <i>cis</i> -Heptachlor epoxide                     | 26. 4,4'-DDD              |
| 4. α-BHC                                 | 16. 2,4'-DDE                                           | 27. Endosulfan 2          |
| 5. Pentachloroanisole                    | 17. <i>trans</i> -Chlordane                            | 28. 4,4'-DDT              |
| 6. HCB                                   | 18. <i>cis</i> -Chlordane<br>+ <i>trans</i> -nonachlor | 29. Parlar 41             |
| 7. γ-BHC                                 | 19. Endosulfan 1                                       | 30. Parlar 40             |
| 8. β-BHC                                 | 20. 4,4'-DDE                                           | 31. Parlar 44             |
| 9. Heptachlor                            | 21. Dieldrin                                           | 32. Endosulfan sulphate   |
| 10. ε-BHC                                | 22. 2,4'-DDD                                           | 33. Parlar 50             |
| 11. Aldrin                               | 23. Endrin                                             | 34. Mirex                 |
| 12. 1,2,3,4-Tetrachloronaphthalene (TCN) |                                                        |                           |



R. Looser, K. Ballschmiter, J. Chromatogr. A 836 (1999) 271 – 284

# Halogenated hydrocarbons

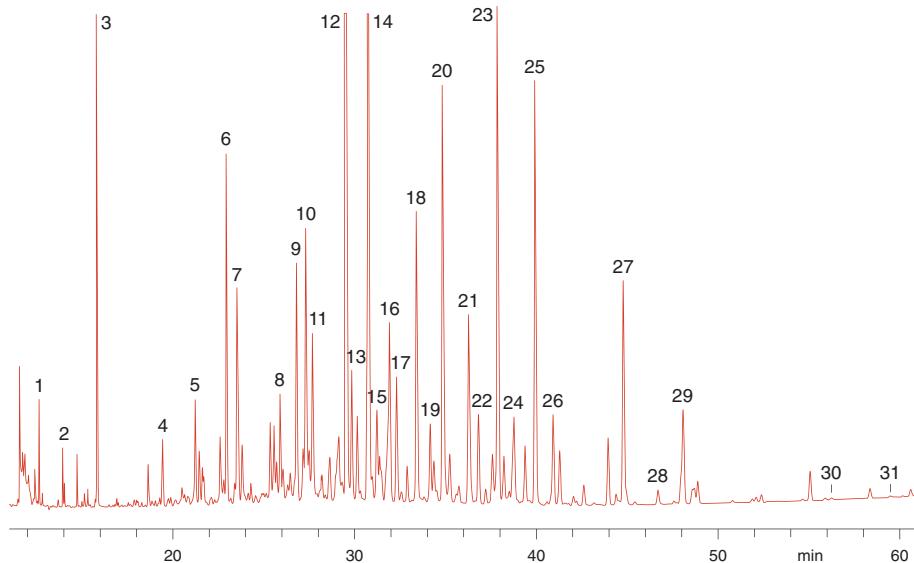


## Analysis of organohalogen compounds incl. PCB from cod liver oil MN Appl. No. 250510

Column: OPTIMA® δ-3, 60 m x 0.25 mm ID, 0.25 µm film, REF 726420.60, max. temperature 340/360 °C  
Sample: NP-HPLC group separation of NIST reference material SRM 1588 (semivolatile organohalogen compounds in cod liver oil) on 250 x 4 mm NUCLEOSIL® 100-10 NH<sub>2</sub> using 10 ml *n*-hexane (fraction 1) and 32 ml *n*-hexane – dichloromethane (90:10 v/v, fraction 2), flow rate 1 ml/min (see application 213270 for chromatogram of fraction 2)  
Injection: 2 µl of fraction 1, on column  
Carrier gas: 180 kPa H<sub>2</sub>      Temperature: 80 °C (3 min)  $\xrightarrow{20 \text{ °C/min}}$  200 °C  $\xrightarrow{1.5 \text{ °C/min}}$  290 °C (5 min)  
Detector: ECD 300 °C

**Peaks:**

- |                                                            |               |               |                                          |
|------------------------------------------------------------|---------------|---------------|------------------------------------------|
| 1. Pentachlorobenzene                                      | 8. PCB-66     | 17. PCB-139   | 26. PCB-128                              |
| 2. 2,4,6-Tribromoanisole                                   | 9. 2,4'-DDE   | 18. PCB-118   | 27. PCB-180                              |
| 3. HCB                                                     | 10. PCB-101   | 19. PCB-146   | 28. 2,2',4,4'-Tetrabromodiphenyl ether   |
| 4. PCB-28                                                  | 11. PCB-99    | 20. PCB-153   | 29. Mirex                                |
| 5. PCB-52                                                  | 12. 4,4'-DDE  | 21. PCB-105   | 30. 2,2',4,4',6-Pentabromodiphenyl ether |
| 6. 1,2,3,4-Tetrachloronaphthalene (TCN, recovery standard) | 13. PCB-87    | 22. PCB-137   | 31. 2,2',4,4',5-Pentabromodiphenyl ether |
| 7. PCB-103 (int. std.)                                     | 14. Parlar 26 | 23. PCB-138   |                                          |
|                                                            | 15. PCB-151   | 24. PCB-187   |                                          |
|                                                            | 16. PCB-82    | 25. Parlar 50 |                                          |



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# Environmental pollutants

## Analysis of a pesticide mixture

*MN Appl. No. 210710*

Column: OPTIMA® δ-6,  
50 m x 0.20 mm ID, 0.20 µm  
film, REF 726465.50,  
max. temperature 340/360 °C

Injection: 0.03 – 0.5 µg/ml, depending  
on component, splitless,  
purge time 0.8 min, 270 °C

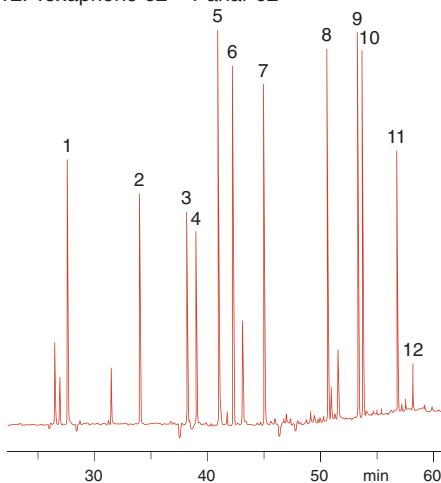
Carrier gas: 2.3 ml/min He

Temperature: 130 °C (1 min)  $\xrightarrow{25\text{ °C/min}}$   
 $\xrightarrow{2\text{ °C/min}}$  180 °C (5 min)  
 $\xrightarrow{6\text{ °C/min}}$  260 °C  
 $\xrightarrow{310\text{ °C (14 min)}}$

Detector: ECD 300 °C

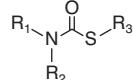
**Peaks:**

1. Musk xylene
2. Musk ketone
3. *cis*-Heptachlor epoxide
4. *trans*-Heptachlor epoxide
5. *trans*-Chlordane
6. α-Endosulfan
7. Toxaphene 26 = Parlar 26
8. β-Endosulfan
9. Toxaphene 50 = Parlar 50
10. Endosulfan sulphate
11. Ketoendrin
12. Toxaphene 62 = Parlar 62



Courtesy of Mr. Grabher, CLUA, Sigmaringen,  
Germany

## Pesticide structures: carbamates



Compound	R <sub>1</sub>	R <sub>2</sub>	R <sub>3</sub>
Butylate	i-C <sub>4</sub> H <sub>9</sub>	i-C <sub>4</sub> H <sub>9</sub>	C <sub>2</sub> H <sub>5</sub>
Cycloate	C <sub>2</sub> H <sub>5</sub>	cyclohexyl	C <sub>2</sub> H <sub>5</sub>
EPTC	n-C <sub>3</sub> H <sub>7</sub>	n-C <sub>3</sub> H <sub>7</sub>	C <sub>2</sub> H <sub>5</sub>
Prothiocarb	H	(CH <sub>2</sub> ) <sub>3</sub> — N(CH <sub>3</sub> ) <sub>2</sub>	C <sub>2</sub> H <sub>5</sub>
Pebulate	C <sub>2</sub> H <sub>5</sub>	n-C <sub>4</sub> H <sub>9</sub>	n-C <sub>3</sub> H <sub>7</sub>
Vernolate	n-C <sub>3</sub> H <sub>7</sub>	n-C <sub>3</sub> H <sub>7</sub>	n-C <sub>3</sub> H <sub>7</sub>
Thiobencarb	C <sub>2</sub> H <sub>5</sub>	C <sub>2</sub> H <sub>5</sub>	

Compound	Structure
Carbaryl	
Carbofuran	
Molinate	
Pirimicarb	

# Pesticides

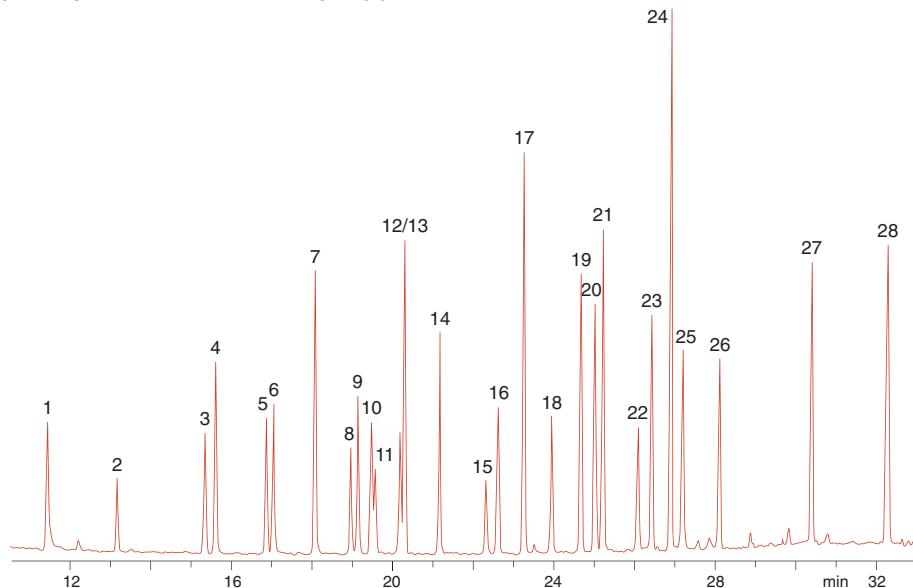


## Analysis of 28 pesticides (Ökotex 100) MN Appl. No. 210270

Column: OPTIMA® δ-3, 30 m x 0.25 mm ID, 0.25 µm film, REF726420.30,  
max. temperature 340/360 °C  
Injection: 250 °C, splitless, purge time 1 min  
Carrier gas: He  
Pressure: 21.4 kPa (1 min)  $\xrightarrow{9 \text{ kPa/min}}$  42.9 kPa  $\xrightarrow{1.1 \text{ kPa/min}}$  72 kPa (20 min)  
Temperature: 60 °C (1 min)  $\xrightarrow{40 \text{ °C/min}}$  150 °C  $\xrightarrow{4.8 \text{ °C/min}}$  280 °C (20 min)  
Detector: MSD 320 °C

**Peaks:**

- |                     |                                      |                         |
|---------------------|--------------------------------------|-------------------------|
| 1. Carbaryl         | 11. Parathion-methyl                 | 21. <i>o,p'</i> -DDD    |
| 2. Trifluralin      | 12. Malathion                        | 22. Endrin              |
| 3. $\alpha$ -BHC    | 13. Aldrin                           | 23. <i>o,p'</i> -DDT    |
| 4. HCB              | 14. Parathion                        | 24. <i>p,p'</i> -DDD    |
| 5. Lindane          | 15. <i>cis</i> -Heptachlor epoxide   | 25. $\beta$ -Endosulfan |
| 6. Quintozene       | 16. <i>trans</i> -Heptachlor epoxide | 26. <i>p,p'</i> -DDT    |
| 7. $\beta$ -BHC     | 17. <i>o,p'</i> -DDE                 | 27. Methoxychlor        |
| 8. Heptachlor       | 18. $\alpha$ -Endosulfan             | 28. Mirex               |
| 9. $\delta$ -BHC    | 19. <i>p,p'</i> -DDE                 |                         |
| 10. $\epsilon$ -BHC | 20. Dieldrin                         |                         |



Courtesy of K. Friedrichs, Chem. Untersuchungsamt, Bielefeld, Germany

# Environmental pollutants

## Analysis of toxaphenes MN Appl. No. 211920

Column: OPTIMA® 17, 50 m x 0.25 mm ID, 0.25 µm film, REF 726022.50,  
max. temperature 320/340 °C

Injection: 1 – 2 µl, 1.0 min splitless, 240 °C

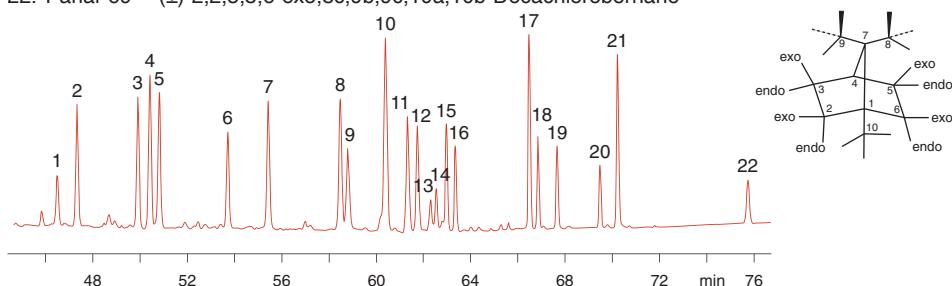
Carrier gas: He

Temperature: 70 °C (2 min)  $\xrightarrow{15\text{ °C/min}}$  180 °C  $\xrightarrow{1.5\text{ °C/min}}$  250 °C (5 min)  $\xrightarrow{10\text{ °C/min}}$  290 °C (15 min)

Detector: MSD

**Peaks:**

1. Parlar 11 ( $\pm$ )-2,2,3-exo-Trichloro,5,5-bis(chloromethyl),6-(E)-chloromethylene-8,9,10-trinorbornane
2. Parlar 12 ( $\pm$ )-5-exo,6-endo-Dichloro,2-endo-chloromethyl,3-(E)-chloromethylene-8,9,10-trinorbornane
3. Parlar 15 ( $\pm$ )-5-exo,6-endo,7-anti-Trichloro,2,2-bis(chloromethyl),3-(E)-chloromethylene-8,9,10-trinorbornane
4. Parlar 21 ( $\pm$ )-2,2,5,5-9c,10a,10b-Heptachlorobornane
5. Parlar 26 ( $\pm$ )-2-endo,3-exo,5-endo,6-exo,8b,8c,10a,10c-Octachlorobornane
6. Parlar 25 ( $\pm$ )-2,2,3,-exo-Trichloro,5-endo-chloromethyl,6-(E)-chloromethylene-5-dichloromethyl,8,9,10-trinorbornane
7. Parlar 31 ( $\pm$ )-2,2,3-exo-Trichloro,6-(E)-chloromethylene,5,5-bis(dichloromethyl),8,9,10-trinorbornane
8. Parlar 32 ( $\pm$ )-2,2,5-endo,6-exo,8c,9b,10a-Heptachlorobornane
9. Parlar 38 ( $\pm$ )-2,2,5,5,9b,9c,10a,10b-Octachlorobornane
10. Parlar 39 ( $\pm$ )-2,2,3-exo,5-endo,6-exo,8c,9b,10a-Octachlorobornane
11. Parlar 41 ( $\pm$ )-2-exo,3-endo,5-exo,8c,9b,9c,10a,10b-Octachlorobornane
12. Parlar 40 ( $\pm$ )-2-endo,3-exo,5-endo,6-exo,8b,9c,10a,10c-Octachlorobornane
13. Parlar 42a ( $\pm$ )-2,2,5-endo,6-exo,8b,8c,9c,10a-Octachlorobornane
14. Parlar 42b ( $\pm$ )-2,2,5-endo,6-exo,8c,9b,9c,10a-Octachlorobornane
15. Parlar 44 ( $\pm$ )-2-exo,5,5,8c,9b,9c,10a,10b-Octachlorobornane
16. Parlar 50 ( $\pm$ )-2-endo,3-exo,5-endo,6-exo,8b,8c,9c,10a,10c-Nonachlorobornane
17. Parlar 51 ( $\pm$ )-2,2,5,5,8c,9b,10a,10b-Octachlorobornane
- + Parlar 56 ( $\pm$ )-2,2,5-endo,6-exo,8b,8c,9c,10a,10b-Nonachlorobornane
18. Parlar 58 ( $\pm$ )-2,2,3-exo,5,5,8c,9b,10a,10b-Nonachlorobornane
19. Parlar 59 ( $\pm$ )-2,2,5,endo,6-exo,8c,9b,9c,10a,10b-Nonachlorobornane
20. Parlar 62 ( $\pm$ )-2,2,5,5,8c,9b,9c,10a,10b-Nonachlorobornane
21. Parlar 63 ( $\pm$ )-2-exo,3-endo,5-exo,6-exo,8b,8c,9c,10a,10c-Nonachlorobornane
22. Parlar 69 ( $\pm$ )-2,2,5,5,6-exo,8c,9b,9c,10a,10b-Decachlorobornane



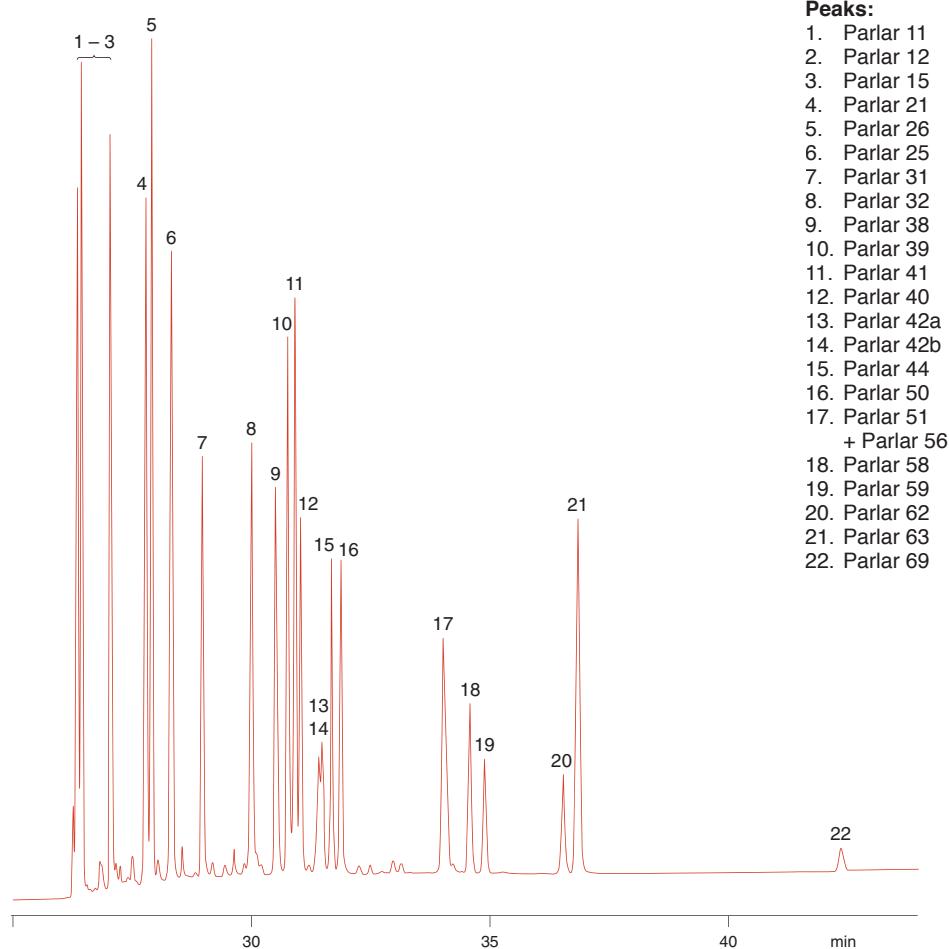
M. Kaltenegger, Dissertation, Universität Wuppertal, Germany and M. Kaltenegger, K.-H. Schwind, K.-H. Ueberschär, H. Hecht, M. Petz, Organohalogen Compounds **35** (1998) 281 – 285

# Toxaphenes



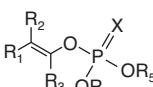
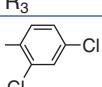
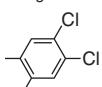
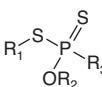
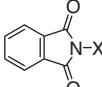
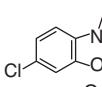
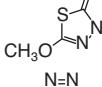
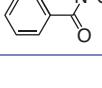
## Analysis of a complex toxaphene mixture MN Appl. No. 250130

Column: OPTIMA® δ-3, 40 m x 0.20 mm ID, 0.35 µm film, custom-made column  
we recommend REF 726400.50, max. temperature 340/360 °C  
Injection: 1 µl, splitless  
Carrier gas: 1.3 ml/min He  
Temperature: 100 °C  $\xrightarrow{7\text{ °C/min}}$  270 °C  
Detector: ECD

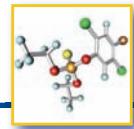


Courtesy of R. Baycan-Keller, M. Oehme, Inst. Org. Anal. Chem., University of Basel, Switzerland

# Environmental pollutants

Structures of organophosphorus pesticides							
Structure	Compound	X	R <sub>1</sub>	R <sub>2</sub>	R <sub>3</sub>	R <sub>4</sub>	R <sub>5</sub>
	Chlorfenvinphos	O	Cl	H		C <sub>2</sub> H <sub>5</sub>	C <sub>2</sub> H <sub>5</sub>
	Crotoxyphos	O	CO-O-CH(C <sub>6</sub> H <sub>5</sub> )-CH <sub>3</sub>	H	CH <sub>3</sub>	CH <sub>3</sub>	CH <sub>3</sub>
	Dichlorphos	O	Cl	Cl	H	CH <sub>3</sub>	CH <sub>3</sub>
	Dicrotophos	O	CO-N(CH <sub>3</sub> ) <sub>2</sub>	H	CH <sub>3</sub>	CH <sub>3</sub>	CH <sub>3</sub>
	Mevinphos	O	CO-O-CH <sub>3</sub>	H	CH <sub>3</sub>	CH <sub>3</sub>	CH <sub>3</sub>
	Monocrotophos	O	CO-NH-CH <sub>3</sub>	H	CH <sub>3</sub>	CH <sub>3</sub>	CH <sub>3</sub>
	Phosphamidon	O	CO-N(C <sub>2</sub> H <sub>5</sub> ) <sub>2</sub>	Cl	CH <sub>3</sub>	CH <sub>3</sub>	CH <sub>3</sub>
	Tetrachlorvinphos	O	Cl	H		CH <sub>3</sub>	CH <sub>3</sub>
	Methacrifos	S	CO-OCH <sub>3</sub>	CH <sub>3</sub>	H	C <sub>2</sub> H <sub>5</sub>	C <sub>2</sub> H <sub>5</sub>
Structure	Compound		R <sub>1</sub>		R <sub>2</sub>	R <sub>3</sub>	
	Chlormephos		CH <sub>2</sub> Cl		C <sub>2</sub> H <sub>5</sub>	OC <sub>2</sub> H <sub>5</sub>	
	Fonofos		C <sub>6</sub> H <sub>5</sub>		C <sub>2</sub> H <sub>5</sub>	C <sub>2</sub> H <sub>5</sub>	
	Malathion		CH <sub>2</sub> -CO-OC <sub>2</sub> H <sub>5</sub>		CH <sub>3</sub>	OCH <sub>3</sub>	
	Dimethoate		CH <sub>2</sub> -CO-NH-CH <sub>3</sub>		CH <sub>3</sub>	OCH <sub>3</sub>	
	Amidithion		CH <sub>2</sub> -CO-NH-(CH <sub>2</sub> ) <sub>2</sub> -OCH <sub>3</sub>		CH <sub>3</sub>	OCH <sub>3</sub>	
	Mecarbam		CH <sub>2</sub> -CO-N(CH <sub>3</sub> )-CO-OC <sub>2</sub> H <sub>5</sub>		C <sub>2</sub> H <sub>5</sub>	OC <sub>2</sub> H <sub>5</sub>	
	Phorate		CH <sub>2</sub> -S-C <sub>2</sub> H <sub>5</sub>		C <sub>2</sub> H <sub>5</sub>	OC <sub>2</sub> H <sub>5</sub>	
	Terbufos		CH <sub>2</sub> -S-C(CH <sub>3</sub> ) <sub>3</sub>		C <sub>2</sub> H <sub>5</sub>	OC <sub>2</sub> H <sub>5</sub>	
	Carbophenothion		CH <sub>2</sub> -S-4-C <sub>6</sub> H <sub>5</sub> Cl		C <sub>2</sub> H <sub>5</sub>	OC <sub>2</sub> H <sub>5</sub>	
	Phenkaptone		CH <sub>2</sub> -S-2,5-C <sub>6</sub> H <sub>4</sub> Cl <sub>2</sub>		C <sub>2</sub> H <sub>5</sub>	OC <sub>2</sub> H <sub>5</sub>	
	Disulfoton		(CH <sub>2</sub> ) <sub>2</sub> -S-C <sub>2</sub> H <sub>5</sub>		C <sub>2</sub> H <sub>5</sub>	OC <sub>2</sub> H <sub>5</sub>	
	Thiometon		(CH <sub>2</sub> ) <sub>2</sub> -S-C <sub>2</sub> H <sub>5</sub>		CH <sub>3</sub>	OCH <sub>3</sub>	
	Phosmet			X = CH <sub>2</sub>	CH <sub>3</sub>	OCH <sub>3</sub>	
	Dialifos			X = CH-CH <sub>2</sub> Cl	C <sub>2</sub> H <sub>5</sub>	OC <sub>2</sub> H <sub>5</sub>	
	Phosalone				C <sub>2</sub> H <sub>5</sub>	OC <sub>2</sub> H <sub>5</sub>	
	Methidathion				CH <sub>3</sub>	OCH <sub>3</sub>	
	Azinphos-methyl				CH <sub>3</sub>	OCH <sub>3</sub>	
	Azinphos-ethyl				C <sub>2</sub> H <sub>5</sub>	OC <sub>2</sub> H <sub>5</sub>	

# Organophosphorus pesticides



**Structures of organophosphorus pesticides**

Structure	Compound	R <sub>1</sub>	R <sub>2</sub>	R <sub>3</sub>	
	Acephate	NH-CO-CH <sub>3</sub>	OCH <sub>3</sub>	SCH <sub>3</sub>	
	Dimefox	N(CH <sub>3</sub> ) <sub>2</sub>	N(CH <sub>3</sub> ) <sub>2</sub>	F	
	Edifenphos	OC <sub>2</sub> H <sub>5</sub>	SC <sub>6</sub> H <sub>5</sub>	SC <sub>6</sub> H <sub>5</sub>	
	Ethoprophos	OC <sub>2</sub> H <sub>5</sub>	S-n-C <sub>3</sub> H <sub>7</sub>	S-n-C <sub>3</sub> H <sub>7</sub>	
	Heptenophos	-O- 	OCH <sub>3</sub>	OCH <sub>3</sub>	
	Naled	OCHBr-CBrCl <sub>2</sub>	OCH <sub>3</sub>	OCH <sub>3</sub>	
	Trichlorfon	CH(OH)-CCl <sub>3</sub>	OCH <sub>3</sub>	OCH <sub>3</sub>	
	Vamidothion	S-(CH <sub>2</sub> ) <sub>2</sub> -S-CH(CH <sub>3</sub> )-CO-NH-CH <sub>3</sub>	OCH <sub>3</sub>	OCH <sub>3</sub>	
	Coumaphos		C <sub>2</sub> H <sub>5</sub>	C <sub>2</sub> H <sub>5</sub>	
	Chlorpyrifos		C <sub>2</sub> H <sub>5</sub>	C <sub>2</sub> H <sub>5</sub>	
	Diazinon	X <sub>1</sub> = CH <sub>3</sub> , X <sub>2</sub> = i-C <sub>3</sub> H <sub>7</sub>	C <sub>2</sub> H <sub>5</sub>	C <sub>2</sub> H <sub>5</sub>	
	Etrimfos	X <sub>1</sub> = OC <sub>2</sub> H <sub>5</sub> , X <sub>2</sub> = C <sub>2</sub> H <sub>5</sub>	CH <sub>3</sub>	CH <sub>3</sub>	
	Pirimiphos-ethyl	X <sub>1</sub> = CH <sub>3</sub> , X <sub>2</sub> = N(C <sub>2</sub> H <sub>5</sub> ) <sub>2</sub>	C <sub>2</sub> H <sub>5</sub>	C <sub>2</sub> H <sub>5</sub>	
	Pirimiphos-methyl	X <sub>1</sub> = CH <sub>3</sub> , X <sub>2</sub> = N(C <sub>2</sub> H <sub>5</sub> ) <sub>2</sub>	CH <sub>3</sub>	CH <sub>3</sub>	
	Pyridaphenthion		C <sub>2</sub> H <sub>5</sub>	C <sub>2</sub> H <sub>5</sub>	
	Quinalphos		C <sub>2</sub> H <sub>5</sub>	C <sub>2</sub> H <sub>5</sub>	
	Thionazin		C <sub>2</sub> H <sub>5</sub>	C <sub>2</sub> H <sub>5</sub>	
	Isazofos	X <sub>1</sub> -N=C=X <sub>2</sub>	X <sub>1</sub> = i-C <sub>3</sub> H <sub>7</sub> , X <sub>2</sub> = Cl	C <sub>2</sub> H <sub>5</sub>	C <sub>2</sub> H <sub>5</sub>
	Triazophos	X <sub>2</sub>	X <sub>1</sub> = C <sub>6</sub> H <sub>7</sub> , X <sub>2</sub> = H	C <sub>2</sub> H <sub>5</sub>	C <sub>2</sub> H <sub>5</sub>

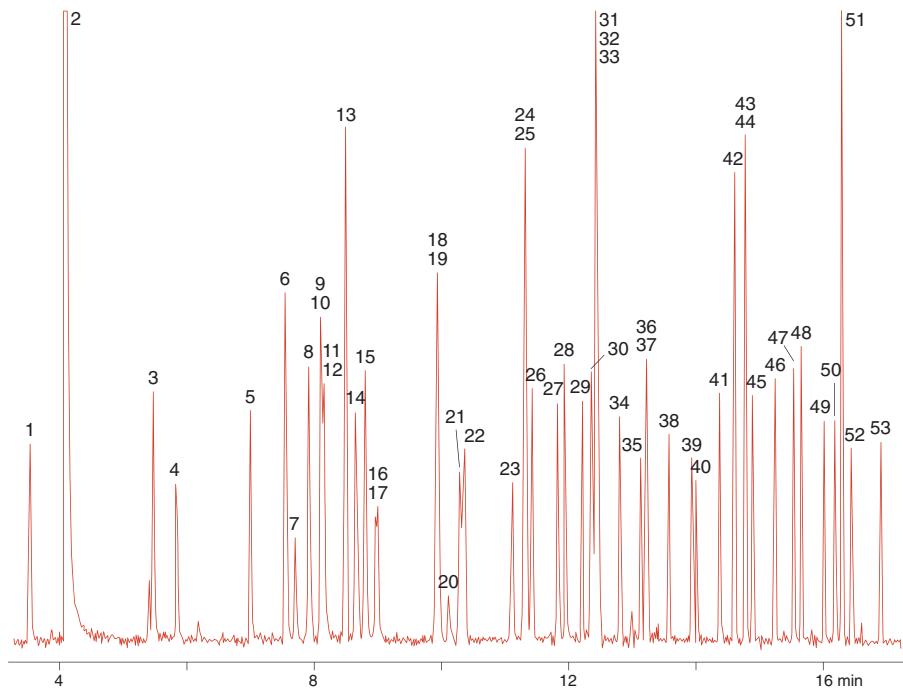
Structure	Compound	X <sub>1</sub>	X <sub>2</sub>	R <sub>1</sub>	R <sub>2</sub>
	Demeton-O	O	S	C <sub>2</sub> H <sub>5</sub>	C <sub>2</sub> H <sub>5</sub>
	Demeton-S	S	O	C <sub>2</sub> H <sub>5</sub>	C <sub>2</sub> H <sub>5</sub>
	Demeton-O-methyl	O	S	C <sub>2</sub> H <sub>5</sub>	CH <sub>3</sub>
	Demeton-S-methyl	S	O	C <sub>2</sub> H <sub>5</sub>	CH <sub>3</sub>
	Demephion-O	O	S	CH <sub>3</sub>	CH <sub>3</sub>
	Demephion-S	S	O	CH <sub>3</sub>	CH <sub>3</sub>
	Sulfotep	O	S	C <sub>2</sub> H <sub>5</sub>	-
	TEPP	O	O	C <sub>2</sub> H <sub>5</sub>	-
	Ethion	S-CH <sub>2</sub> -S-	S	C <sub>2</sub> H <sub>5</sub>	-
	Dioxathion		S	C <sub>2</sub> H <sub>5</sub>	-

## Environmental pollutants

### Analysis of organophosphorus pesticides (EPA 8140 / 8141 / 8141 A) MN Appl. No. 213030

Column: OPTIMA® 1 MS Accent, 30 m x 0.32 mm ID, 0.50 µm film, REF 725807.30  
Sample: 0.2 µg/ml in hexane,  
8140/8141 OP pesticides calibration mix A and 8141 OP pesticides calibration mix  
B; IS triphenyl phosphate and tributyl phosphate  
Injection: splitless (hold 1 min), 250 °C  
Carrier gas: He, 1 ml/min, constant pressure  
Temperature: 100 °C  $\xrightarrow{10 \text{ °C/min}}$  180 °C (2 min)  $\xrightarrow{18 \text{ °C/min}}$  300 °C (3 min)  
Detector: FPD (Flame Photometric Detector), 280 °C

**Peaks:**  
1. Dichlorvos, 2. Hexamethylphosphoramide, 3. Mevinphos, 4. Trichlorfon, 5. TEPP, 6. Thionazin,  
7. Demeton-O, 8. Ethoprop, 9. Tributyl phosphate (IS), 10. Dicrotophos, 11. Monocrotophos,  
12. Naled, 13. Sulfotepp, 14. Phorate, 15. Dimethoate, 16. Demeton-S, 17. Dioxathion, 18. Terbufos,  
19. Fonofos, 20. Phoshamidon isomer, 21. Diazinon, 22. Disulfoton, 23. Phoshamidon,  
24. Dichlofenthion, 25. Parathion-methyl, 26. Chlorpyrifos methyl, 27. Ronnel, 28. Fenitrothion,  
29. Malathion, 30. Fenthion, 31. Chlordane, 32. Parathion-ethyl, 33. Chlorpyrifos, 34. Trichloronate,  
35. Chlorfenvinphos, 36. Merphos, 37. Crotoxyphos, 38. Stirofos, 39. Tokuthion (prothiophos),  
40. Merphos oxidation product, 41. Fensulfothion, 42. Famphur, 43. Ethion, 44. Bolstar, 45. Carbophenothion,  
46. Triphenyl phosphate (IS), 47. Phosmet, 48. EPN, 49. Azinphos-methyl, 50. Lepto-phos,  
51. Tri-o-cresyl phosphate, 52. Azinphos-ethyl, 53. Coumaphos



# Organophosphorus pesticides



## Separation of organophosphorus pesticides (EPA 8140 / 8141) MN Appl. No. 250420

Column: OPTIMA® δ-6, 50 m x 0.20 mm ID, 0.2 µm film, REF 726465.50,  
max. temperature 340/360 °C

Sample: EPA 8140 OP pesticide calibration mix (Restek),  
200 µg/ml each in hexane – acetone (95:5, v/v)

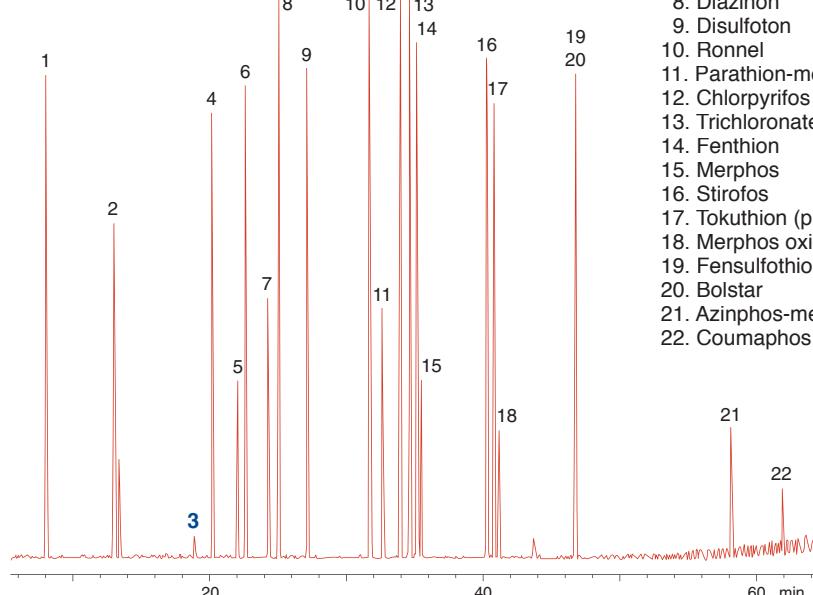
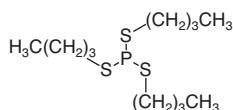
Injection: 1 µl, split 1:30

Carrier gas: 2.0 bar He

Temperature: 150 °C  $\xrightarrow{2.5 \text{ °C/min}}$  300 °C (10 min)

Detector: MSD

Morphos:



### Peaks:

1. Dichlorvos
2. Mevinphos
3. Demeton-S
4. Ethoprop
5. Naled
6. Phorate
7. Demeton-O
8. Diazinon
9. Disulfoton
10. Ronnel
11. Parathion-methyl
12. Chloryrifos
13. Trichloronate
14. Fenthion
15. Morphos
16. Stirofos
17. Tokuthion (prothiophos)
18. Morphos oxidation product
19. Fensulfothion
20. Bolstar
21. Azinphos-methyl
22. Coumaphos

# Environmental pollutants

## Analysis of organophosphorus insecticides MN Appl. No. 200940

Column: OPTIMA® 1301, 25 m x 0.32 mm ID, 0.25 µm film, REF 726777.25,  
max. temperature 300/320 °C

Injection: 0.5 µl on column

Carrier gas: 80 kPa H<sub>2</sub>

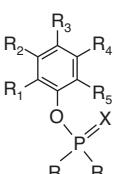
Temperature: 80  $\xrightarrow{30\text{ °C/min}}$  220 °C  $\xrightarrow{4\text{ °C/min}}$  270 °C

Detector: PND

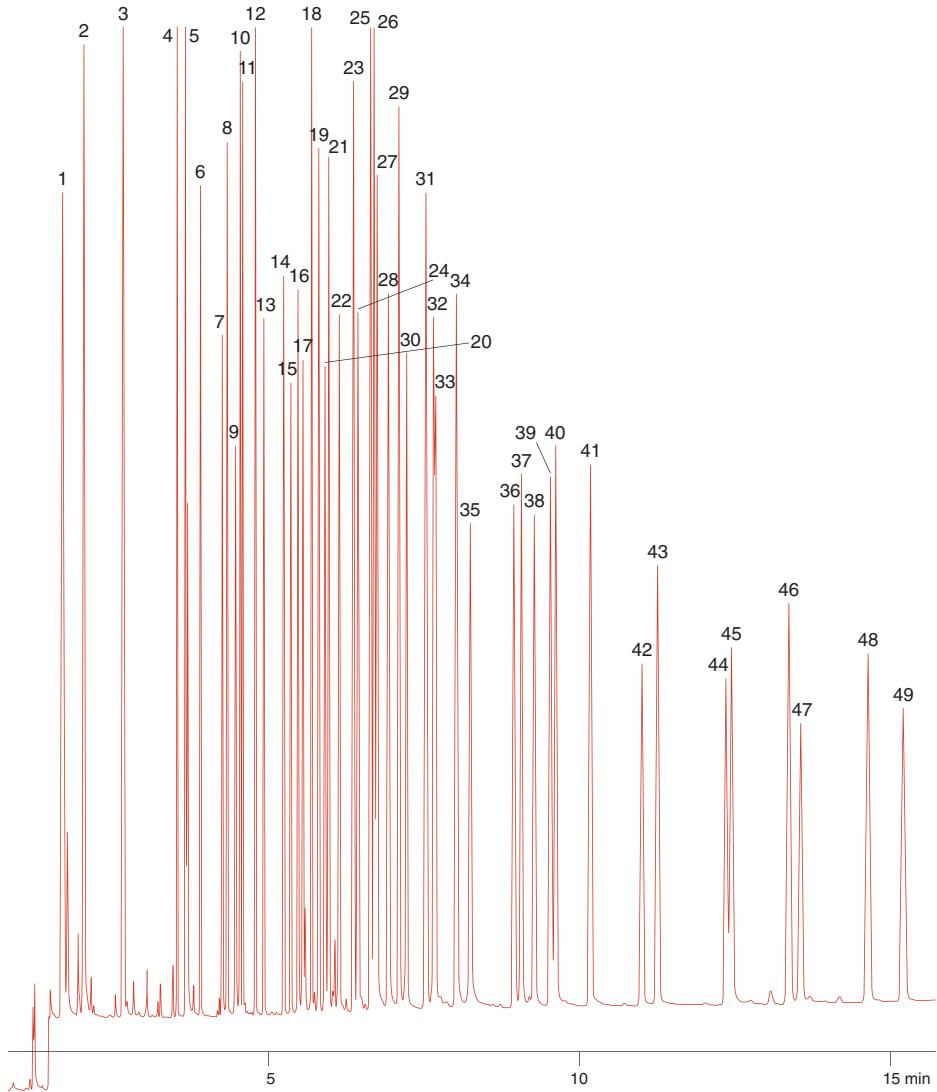
**Peaks: [ng]**

- |                                         |                                                |                                  |
|-----------------------------------------|------------------------------------------------|----------------------------------|
| 1. Dimefox [0.25]                       | 18. Dichlofenthion [0.5]                       | 34. Fenamiphos [0.5]             |
| 2. Triethyl phosphate [0.25]            | 19. Dursban-methyl = Chlorpyrifos-methyl [0.5] | 35. Vamidothion [0.5]            |
| 3. Dichlorvos [0.25]                    | 20. Tolclofos-methyl [0.25]                    | 36. Ethion [0.25]                |
| 4. Chlormephos [0.25]                   | 21. Fenchlorphos [0.5]                         | 37. Sulprofos [0.5]              |
| 5. Mevinphos [0.5]                      | 22. Pirimiphos-methyl [0.5]                    | 38. Carbofenothonion [0.5]       |
| 6. Methacrifos [0.25]                   | 23. Dursban-ethyl = Chlorpyrifos-ethyl [0.5]   | 39. Edifenphos [0.5]             |
| 7. Demephion [0.5]                      | 24. Malathion [0.5]                            | 40. Triazophos [0.5]             |
| 8. Heptenophos [0.5]                    | 25. Pirimiphos-ethyl [1.0]                     | 41. Triphenyl phosphate [0.5]    |
| 9. TEPP [0.25]                          | 26. Parathion-ethyl [0.5]                      | 42. Phenkapton [0.5]             |
| 10. Ethoprophos [0.25]                  | 27. Amidithion [0.5]                           | 43. Pyridaphenthion [0.5]        |
| 11. Tributyl phosphate [0.25]           | 28. Crufomate [0.5]                            | 44. Azinphos-methyl [0.5]        |
| 12. Sulfotep [0.25]                     | 29. Quinalphos [0.5]                           | 45. Phosalone [0.5]              |
| 13. Thiometon [0.25]                    | 30. Bromophos-ethyl [0.5]                      | 46. Azinphos-ethyl [0.5]         |
| 14. Diazinon [0.25]                     | 31. Methidathion [0.5]                         | 47. Dialifos [0.5]               |
| 15. Disulfoton [0.25]                   | 32. Iodofenphos [0.5]                          | 48. Tri-p-cresyl phosphate [0.5] |
| 16. Tris-2-chloroethyl phosphate [0.25] | 33. Prothiophos [0.5]                          | 49. Coumaphos [0.5]              |
| 17. Isazofos [0.25]                     |                                                |                                  |

## Structures of organophosphorus pesticides

Structure	Compound	X	R <sub>1</sub>	R <sub>2</sub>	R <sub>3</sub>	R <sub>4</sub>	R <sub>5</sub>	R <sub>6</sub>	R <sub>7</sub>
	Bromophos-ethyl	S	Cl	H	Br	Cl	H	OC <sub>2</sub> H <sub>5</sub>	OC <sub>2</sub> H <sub>5</sub>
	Chlorthion	S	H	Cl	NO <sub>2</sub>	H	H	OCH <sub>3</sub>	OCH <sub>3</sub>
	Crufomate	O	Cl	H	C(CH <sub>3</sub> ) <sub>3</sub>	H	H	OCH <sub>3</sub>	NH-CH <sub>3</sub>
	Dichlofenthion	S	Cl	H	Cl	H	H	OC <sub>2</sub> H <sub>5</sub>	OC <sub>2</sub> H <sub>5</sub>
	EPN	S	H	H	NO <sub>2</sub>	H	H	OC <sub>2</sub> H <sub>5</sub>	C <sub>6</sub> H <sub>5</sub>
	Famphur	S	H	H	SO <sub>2</sub> -N(CH <sub>3</sub> ) <sub>2</sub>	H	H	OCH <sub>3</sub>	OCH <sub>3</sub>
	Fenamiphos	O	H	CH <sub>3</sub>	SCH <sub>3</sub>	H	H	OC <sub>2</sub> H <sub>5</sub>	NH-i-C <sub>3</sub> H <sub>7</sub>
	Fenchlorphos	S	Cl	H	Cl	Cl	H	OCH <sub>3</sub>	OCH <sub>3</sub>
	Fenitrothion	S	H	CH <sub>3</sub>	NO <sub>2</sub>	H	H	OCH <sub>3</sub>	OCH <sub>3</sub>
	Fensulfothion	S	H	H	SO-CH <sub>3</sub>	H	H	OC <sub>2</sub> H <sub>5</sub>	OC <sub>2</sub> H <sub>5</sub>
	Fenthion	S	H	CH <sub>3</sub>	SCH <sub>3</sub>	H	H	OCH <sub>3</sub>	OCH <sub>3</sub>
	Iodofenphos	S	Cl	H	I	Cl	H	OCH <sub>3</sub>	OCH <sub>3</sub>
	Leptophos	S	Cl	H	Br	Cl	H	OCH <sub>3</sub>	C <sub>6</sub> H <sub>5</sub>
	Parathion-ethyl	S	H	H	NO <sub>2</sub>	H	H	OC <sub>2</sub> H <sub>5</sub>	OC <sub>2</sub> H <sub>5</sub>
	Profenofos	O	Cl	H	Br	H	H	OC <sub>2</sub> H <sub>5</sub>	S-n-C <sub>3</sub> H <sub>7</sub>
	Prothiophos	S	Cl	H	Cl	H	H	OC <sub>2</sub> H <sub>5</sub>	S-n-C <sub>3</sub> H <sub>7</sub>
	Sulprofos	S	H	H	SCH <sub>3</sub>	H	H	OC <sub>2</sub> H <sub>5</sub>	S-n-C <sub>3</sub> H <sub>7</sub>
	Tolclofos-methyl	S	Cl	H	CH <sub>3</sub>	H	Cl	OCH <sub>3</sub>	OCH <sub>3</sub>
	Trichloronat	S	Cl	H	Cl	Cl	H	OC <sub>2</sub> H <sub>5</sub>	C <sub>2</sub> H <sub>5</sub>

## Organophosphorus pesticides



Courtesy of Mr. Lembacher, Hipp Nährmittel, Pfaffenhofen, Germany

# Environmental pollutants

## Analysis of organophosphorus pesticides MN Appl. No. 213180

**Column:** OPTIMA® 35 MS, 30 m x 0.25 mm ID, 0.25 µm film, REF 726154.30,  
max. temperature 360/370 °C

**Injection:** 1.0 µl, 250 °C, split 1:25

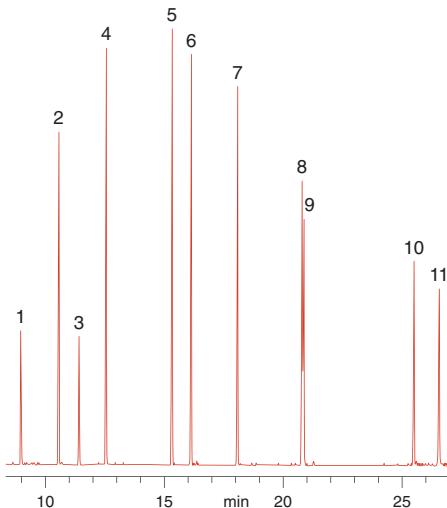
**Carrier gas:** 1.5 ml/min He

**Temperature:** 130 °C  $\xrightarrow{6\text{ °C/min}}$  300 °C

**Detector:** MSD

**Peaks:**

1. Demeton-O
2. Phorate
3. Demeton-S
4. Disulfoton
5. Trichloronate
6. Fenthion
7. Prothiophos
8. Sulprofos
9. Fensulfothion
10. Azinphos-methyl
11. Coumaphos



### Pesticide structures: dinitroaniline herbicides

Structure	Compound	R <sub>1</sub>	R <sub>2</sub>	R <sub>3</sub>	R <sub>4</sub>
	Ethalfluralin	C <sub>2</sub> H <sub>5</sub>	CH <sub>2</sub> -C(CH <sub>3</sub> )=CH <sub>2</sub>	H	CF <sub>3</sub>
	Fluchloralin	C <sub>2</sub> H <sub>4</sub> Cl	n-C <sub>3</sub> H <sub>7</sub>	H	CF <sub>3</sub>
	Isopropalin	n-C <sub>3</sub> H <sub>7</sub>	n-C <sub>3</sub> H <sub>7</sub>	H	CH(CH <sub>3</sub> ) <sub>2</sub>
	Pendimethalin	H	CH(C <sub>2</sub> H <sub>5</sub> ) <sub>2</sub>	CH <sub>3</sub>	CH <sub>3</sub>
	Profluralin	n-C <sub>3</sub> H <sub>7</sub>	cyclopropylmethyl	H	CF <sub>3</sub>
	Trifluralin	n-C <sub>3</sub> H <sub>7</sub>	n-C <sub>3</sub> H <sub>7</sub>	H	CF <sub>3</sub>

### Pesticide structures: triazines

Structure	Compound	R <sub>1</sub>	R <sub>2</sub>	R <sub>3</sub>
	Isomethiozin	C(CH <sub>3</sub> ) <sub>3</sub>	SCH <sub>3</sub>	N=CH-CH(CH <sub>3</sub> ) <sub>2</sub>
	Metamitron	C <sub>6</sub> H <sub>5</sub>	CH <sub>3</sub>	NH <sub>2</sub>
	Metribuzin	C(CH <sub>3</sub> ) <sub>3</sub>	SCH <sub>3</sub>	NH <sub>2</sub>
	Hexazinone	—	—	—

For more triazine structures see page 102.

# Pesticides



## Pesticide structures: triazole and imidazole derivatives, conazoles

Structure	Compound	R <sub>1</sub>	R <sub>2</sub>	R <sub>3</sub>	R <sub>4</sub>
	Cyproconazole	H	OH	1-cyclopropylethyl	H
	Diclobutrazole	CH(OH)-C(CH <sub>3</sub> ) <sub>3</sub>	H	H	Cl
	Hexaconazole	H	OH	n-C <sub>4</sub> H <sub>9</sub>	Cl
	Myclobutanil	H	CN	n-C <sub>4</sub> H <sub>9</sub>	H
	Penconazole	H	H	n-C <sub>3</sub> H <sub>7</sub>	Cl
	Bitertanol	CH(OH)-C(CH <sub>3</sub> ) <sub>3</sub>	C <sub>6</sub> H <sub>5</sub>	—	—
	Triadimefon	CO-C(CH <sub>3</sub> ) <sub>3</sub>	Cl	—	—
	Triadimenol	CH(OH)-C(CH <sub>3</sub> ) <sub>3</sub>	Cl	—	—
Flutriafol	Tebuconazole			Imazalil	
	Propiconazole			Prochloraz	
				Triflumizole	

## Pesticide structures: urea derivatives

Structure	Compound	R <sub>1</sub>	R <sub>2</sub>	R <sub>3</sub>
	Fluometuron		H	CH <sub>3</sub>
	Tebuthiuron		CH <sub>3</sub>	H

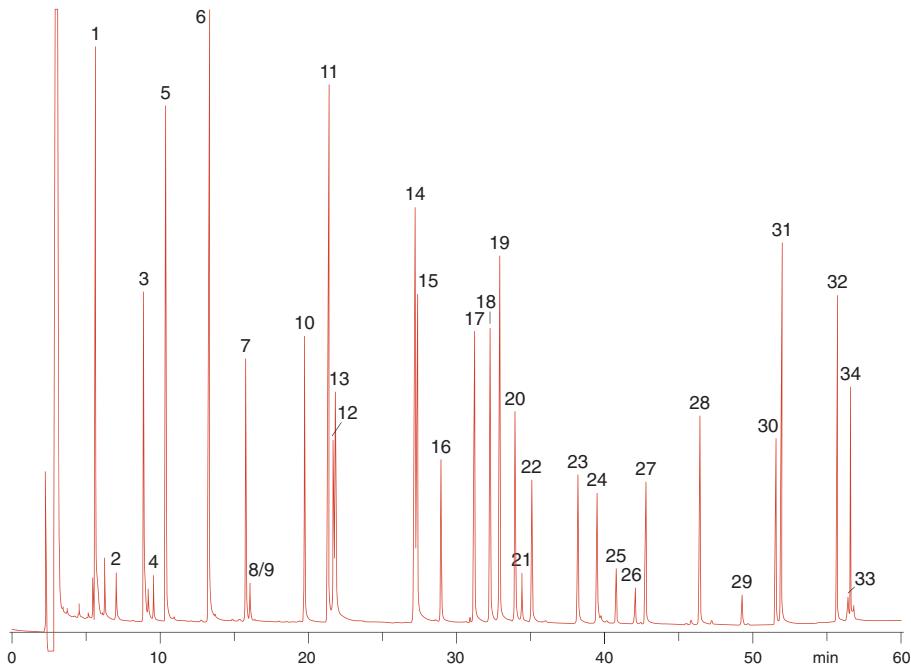
# Environmental pollutants

## Analysis of a pesticide mixture MN Appl. No. 210740

Column: OPTIMA® δ-6, 50 m x 0.20 mm ID, 0.20 µm film, REF 726465.50,  
max. temperature 340/360 °C  
Injection: 0.03 – 0.5 µg/ml depending on component, splitless, purge time 0.8 min, 270 °C  
Carrier gas: 2.3 ml/min He  $\xrightarrow{25\text{ °C}/\text{min}}$  180 °C (5 min)  $\xrightarrow{2\text{ °C}/\text{min}}$  260 °C  $\xrightarrow{6\text{ °C}/\text{min}}$  310 °C (4 min)  
Temperature: 130 °C (1 min)  $\xrightarrow{25\text{ °C}/\text{min}}$  180 °C (5 min)  $\xrightarrow{2\text{ °C}/\text{min}}$  260 °C  $\xrightarrow{6\text{ °C}/\text{min}}$  310 °C (4 min)  
Detector: PND 300 °C

**Peaks:**

- |                                                 |                             |                     |
|-------------------------------------------------|-----------------------------|---------------------|
| 1. Dichlorvos                                   | 11. Dioxathion              | 23. Methidathion    |
| 2. Degradation product of ethion/<br>dioxathion | 12. Dimethoate              | 24. Imazalil        |
| 3. Mevinphos                                    | 13. Etrimfos                | 25. Flusilazole     |
| 4. Propham                                      | 14. Fenchlorphos/pirimiphos | 26. Myclobutanil    |
| 5. Methacrifos                                  | 15. Tolclofos-methyl        | 27. Ethion          |
| 6. Heptenophos                                  | 16. Malathion               | 28. Carbophenothion |
| 7. Sulfotep/<br>degradation product of diazinon | 17. Fenthion                | 29. Tebuconazole    |
| 8. Diphenylamine                                | 18. Bromophos-methyl        | 30. Pyridaphenthion |
| 9. Chlorpropham                                 | 19. Chlorthion              | 31. Phenkapton      |
| 10. Diazinon                                    | 20. Chlorenvinphos          | 32. Azinphos-methyl |
|                                                 | 21. Penconazole             | 33. Bitertanol      |
|                                                 | 22. Quinalphos              | 34. Azinphos-ethyl  |



Courtesy of Mr. Grabher, CLUA, Sigmaringen, Germany

# Pesticides



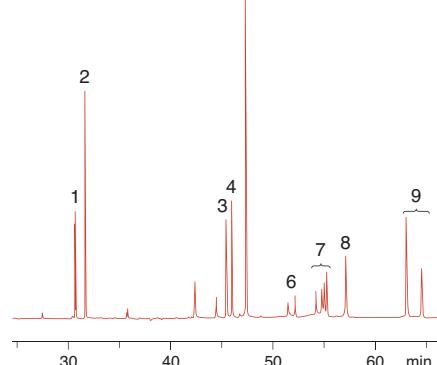
## Analysis of pyrethroid insecticides MN Appl. No. 210720

Column: OPTIMA® δ-6, 50 m x 0.20 mm ID, 0.20 µm film, REF 726465.50,  
max. temperature 340/360 °C  
Injection: 0.03 – 0.5 µg/ml depending on component, splitless, purge time: 0.8 min, 270 °C  
Carrier gas: 2.3 ml/min He  
Temperature: 130 °C (1 min)  $\xrightarrow{25\text{ °C/min}}$  180 °C (5 min)  $\xrightarrow{2\text{ °C/min}}$  260 °C  $\xrightarrow{6\text{ °C/min}}$  310 °C (14 min)  
Detector: ECD 300 °C

### Peaks:

1. Bioallethrin isomers
2. Chlorothion
3. Fenpropathrin + tetramethrin isomer 1
4. Tetramethrin isomer 2
5. Cyhalothrin
6. Permethrin isomers
7. Cyfluthrin isomers
8. Alphamethrin =  $\alpha$ -Cypermethrin
9. Fenvalerate isomers

Courtesy of Mr. Grabher, CLUA, Sigmaringen,  
Germany



## Pesticide structures: pyrethroids

Structure	Compound	R <sub>1</sub>	R <sub>2</sub>	R <sub>3</sub>	R <sub>4</sub>
	Cyfluthrin	CH=CCl <sub>2</sub>	H	CN	F
	Cyhalothrin	CH=CCl-CF <sub>3</sub>	H	CN	H
	$\alpha$ -Cypermethrin	CH=CCl <sub>2</sub>	H	CN	H
	Fenpropathrin	CH <sub>3</sub>	CH <sub>3</sub>	CN	H
	Permethrin	CH=CCl <sub>2</sub>	H	H	H
	Fenvalerate	4-Cl-phenyl	–	–	–
	Flucythrinate	4-CHF <sub>2</sub> O-phenyl	–	–	–
	Fluvalinate	4-CF <sub>3</sub> ,2-Cl-phenyl-NH	–	–	–
	Bioallethrin		–	–	–
	Tetramethrin		–	–	–

# Environmental pollutants

## Analysis of acaricides and fungicides for peel-treatment MN Appl. No. 210210

Column: OPTIMA® δ-3, 30 m x 0.25 mm ID, 0.25 µm film, REF 726420.30,  
max. temperature 340/360 °C

Injection: 1.0 µl, 250 °C, 1 min splitless, then 1:25

Carrier gas:

He

Pressure: 26.5 kPa (1 min)  $\xrightarrow{7 \text{ kPa/min}}$  42.9 kPa  $\xrightarrow{1.1 \text{ kPa/min}}$  71 kPa (20.2 min)

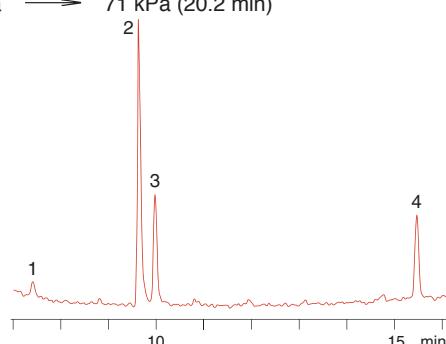
Temperature: 80 °C (1 min)  $\xrightarrow{30 \text{ °C/min}}$  150 °C  
 $\xrightarrow{4.8 \text{ °C/min}}$  280 °C (20 min)

Detector: MSD

**Peaks:**

1. Mecarbam
2. Imazalil
3. Chlorgenson
4. Tetradifon

Courtesy of K. Friedrichs,  
Chem. Untersuchungsamt, Bielefeld, Germany



### Pesticide structures: carboxylic acid amides

Structure	Compound	R <sub>1</sub>	R <sub>2</sub>	R <sub>3</sub>	R <sub>4</sub>
	Acetochlor	CH <sub>2</sub> Cl	CH <sub>2</sub> -OC <sub>2</sub> H <sub>5</sub>	CH <sub>3</sub>	C <sub>2</sub> H <sub>5</sub>
	Alachlor	CH <sub>2</sub> Cl	CH <sub>2</sub> -OCH <sub>3</sub>	C <sub>2</sub> H <sub>5</sub>	C <sub>2</sub> H <sub>5</sub>
	Butachlor	CH <sub>2</sub> Cl	CH <sub>2</sub> -O-n-C <sub>4</sub> H <sub>9</sub>	C <sub>2</sub> H <sub>5</sub>	C <sub>2</sub> H <sub>5</sub>
	Fenfuram	o-methylfuranyl	H	H	H
	Metalaxyl	CH <sub>2</sub> -OCH <sub>3</sub>	CH(CH <sub>3</sub> )-CO-OCH <sub>3</sub>	CH <sub>3</sub>	CH <sub>3</sub>
	Metazachlor	CH <sub>2</sub> Cl	CH <sub>2</sub> -N <sub>3</sub>	CH <sub>3</sub>	CH <sub>3</sub>
	Methfuroxam	trimethylfuranyl	H	H	H
	Metolachlor	CH <sub>2</sub> Cl	CH(CH <sub>3</sub> )-CH <sub>2</sub> -OCH <sub>3</sub>	CH <sub>3</sub>	C <sub>2</sub> H <sub>5</sub>
	Ofurace	CH <sub>2</sub> Cl	γ-butyrolactone	CH <sub>3</sub>	CH <sub>3</sub>
	Propachlor	CH <sub>2</sub> Cl	CH(CH <sub>3</sub> ) <sub>2</sub>	H	H
	Chlorpropham	O-i-C <sub>3</sub> H <sub>7</sub>	H	Cl	H
	Flamprop	C <sub>6</sub> H <sub>5</sub>	CH(CH <sub>3</sub> )-COOH	Cl	F
	Pentanochlor	CH(CH <sub>3</sub> )-i-C <sub>3</sub> H <sub>7</sub>	H	Cl	CH <sub>3</sub>
	Propanil	C <sub>2</sub> H <sub>5</sub>	H	Cl	Cl
	Propham	O-i-C <sub>3</sub> H <sub>7</sub>	H	H	H
	Diethyltoluamide	C <sub>2</sub> H <sub>5</sub>	C <sub>2</sub> H <sub>5</sub>	CH <sub>3</sub>	H
	Propyzamide	C(CH <sub>3</sub> ) <sub>2</sub> -C≡CH	H	Cl	Cl
	Diphenamid	CH <sub>3</sub>	CH <sub>3</sub>	CH(C <sub>6</sub> H <sub>5</sub> ) <sub>2</sub>	
	Isocarbamid	CH <sub>2</sub> -CH(CH <sub>3</sub> ) <sub>2</sub>	H		
	Napropamide	C <sub>2</sub> H <sub>5</sub>	C <sub>2</sub> H <sub>5</sub>	CH(CH <sub>3</sub> )-O-1-naphthyl	

# Pesticides



## Analysis of fungicides MN Appl. No. 200910

Column: OPTIMA® 1, 25 m x 0.32 mm ID, 0.25 µm film, REF 726302.25,  
max. temperature 340/360 °C

Injection: 1 µl on column

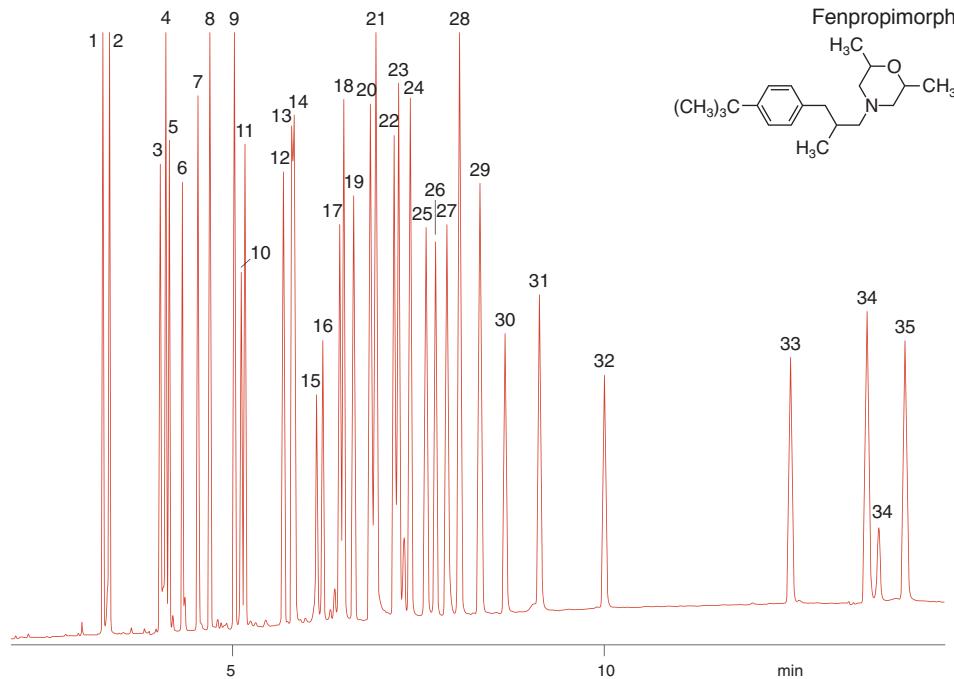
Carrier gas: 50 kPa H<sub>2</sub>

Temperature: 80 °C  $\xrightarrow{40\text{ °C/min}}$  200 °C  $\xrightarrow{6\text{ °C/min}}$  280 °C

Detector: PND 300 °C

### Peaks: [ng]

- |                          |                           |                       |
|--------------------------|---------------------------|-----------------------|
| 1. Etridiazole [2]       | 13. Alachlor [5]          | 25. Flutriafol [2]    |
| 2. Prothiocarb [2.5]     | 14. Metalaxyl [5]         | 26. Flurodififen [5]  |
| 3. Diphenylamine [2]     | 15. Dichlofluanid [2]     | 27. Hexaconazole [2]  |
| 4. Azobenzene [2]        | 16. Thiobencarb [2.5]     | 28. Myclobutanil [2]  |
| 5. Cycloate [2.5]        | 17. Methfuroxam [4]       | 29. Diclobutrazol [2] |
| 6. Ethalfluralin [2]     | 18. Triadimefon [2]       | 30. Thioquinox [2]    |
| 7. Dicloran [2.5]        | 19. Fenpropimorph [5]     | 31. Ofurace [5]       |
| 8. Ethoxyquin [5]        | 20. Thiabendazole [2.5]   | 32. Nuarimol [2]      |
| 9. Benzo[h]quinoline [2] | 21. Penconazole [2]       | 33. Fenarimol [2.5]   |
| 10. Fenfuram [2.5]       | 22. Triadimenol [2.5]     | 34. Bitertanol [2.5]  |
| 11. Fluchloralin [2.5]   | 23. Quinomethionate [2.5] | 35. Prochloraz [2]    |
| 12. Vinclozolin [5]      | 24. Triflumizole [2.5]    |                       |



Courtesy of Mr. Lembacher, Hipp-Nährmittel, Pfaffenhofen, Germany

# Environmental pollutants

## Analysis of herbicides MN Appl. No. 200770

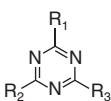
Column: OPTIMA® 1301, 25 m x 0.32 mm ID, 0.25 µm film, REF 726777.25,  
max. temperature 300/320 °C  
Injection: 0.5 µl, splitless, 280 °C  
Carrier gas: 50 kPa H<sub>2</sub>  
Temperature: 80  $\xrightarrow{30\text{ °C/min}}$  200 °C  $\xrightarrow{4\text{ °C/min}}$  270 °C  
Detector: PND 310 °C

**Peaks: [ng]**

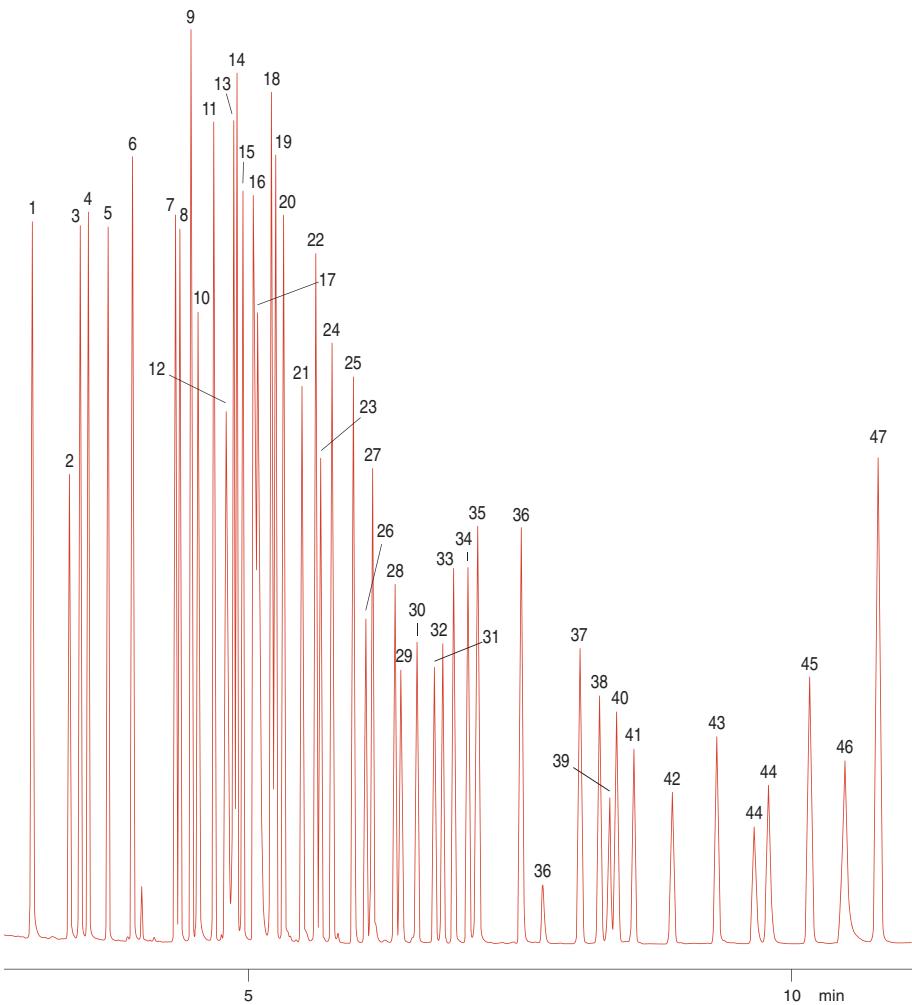
- |                                        |                                  |                            |
|----------------------------------------|----------------------------------|----------------------------|
| 1. EPTC [5]                            | 17. Prometon [2.5]               | 33. Dimethametryn [1.25]   |
| 2. Butylate [5]                        | 18. Trietazine [1.25]            | 34. Metazachlor [2.5]      |
| 3. Vernolate [5]                       | 19. Atrazine [1.25]              | 35. Bromacil [5]           |
| 4. Pebulate [5]                        | 20. Sebutylazine desethyl [1.25] | 36. Triadimenol [5]        |
| 5. Propham [5]                         | 21. Isocarbamid [2.5]            | 37. Oxadiazon [5]          |
| 6. Molinate [5]                        | 22. Fluchloralin [5]             | 38. Methoprotryn [1.25]    |
| 7. Azobenzene [2.5]                    | 23. Sebutylazine [1.25]          | 39. Fluazifop-butyl [5]    |
| 8. Cycloate [5]                        | 24. Desmetryn [1.25]             | 40. Thioquinox [2.5]       |
| 9. Diphenylamine [5]                   | 25. Simetryn [1.25]              | 41. Fluorodifen [5]        |
| 10. Ethalfluralin [5]                  | 26. Metalaxyl [5]                | 42. Flamprop-isopropyl [5] |
| 11. Terbumeton desethyl [5]            | 27. Terbutryn [1.25]             | 43. Metamitron [2.5]       |
| 12. Desethyldeisopropylatrazine [1.25] | 28. Isomethiozin [1.25]          | 44. Propiconazole [5]      |
| 13. Desisopropylatrazine [1.25]        | 29. Metolachlor [5]              | 45. Fluotrimazole [5]      |
| 14. Desethylatrazine [1.25]            | 30. Propanil [5]                 | 46. Lenacil [5]            |
| 15. Terbutylazine desethyl [1.25]      | 31. Pentanochlor [5]             | 47. Hexazinone [5]         |
| 16. Atraton [2.5]                      | 32. Isopropalin [2.5]            |                            |

**Pesticide structures: triazines**

Compound	R <sub>1</sub>	R <sub>2</sub>	R <sub>3</sub>
Atrazine	Cl	NH-C <sub>2</sub> H <sub>5</sub>	NH-CH(CH <sub>3</sub> ) <sub>2</sub>
Cyanazine	Cl	NH-C <sub>2</sub> H <sub>5</sub>	NH-C(CH <sub>3</sub> ) <sub>2</sub> -CN
Propazine	Cl	NH-CH(CH <sub>3</sub> ) <sub>2</sub>	NH-CH(CH <sub>3</sub> ) <sub>2</sub>
Sebutylazine	Cl	NH-C <sub>2</sub> H <sub>5</sub>	NH-(CH <sub>2</sub> ) <sub>3</sub> -CH <sub>3</sub>
Simazin	Cl	NH-C <sub>2</sub> H <sub>5</sub>	NH-C <sub>2</sub> H <sub>5</sub>
Terbutylazine	Cl	NH-C <sub>2</sub> H <sub>5</sub>	NH-C(CH <sub>3</sub> ) <sub>3</sub>
Trietazine	Cl	NH-C <sub>2</sub> H <sub>5</sub>	N(C <sub>2</sub> H <sub>5</sub> ) <sub>2</sub>
Anilanzine	Cl	Cl	NH-(o-Cl-C <sub>6</sub> H <sub>4</sub> )
Atratone	O-CH <sub>3</sub>	NH-C <sub>2</sub> H <sub>5</sub>	NH-CH(CH <sub>3</sub> ) <sub>2</sub>
Prometon	O-CH <sub>3</sub>	NH-CH(CH <sub>3</sub> ) <sub>2</sub>	NH-CH(CH <sub>3</sub> ) <sub>2</sub>
Secbumeton	O-CH <sub>3</sub>	NH-C <sub>2</sub> H <sub>5</sub>	NH-CH(CH <sub>3</sub> )-C <sub>2</sub> H <sub>5</sub>
Terbumeton	O-CH <sub>3</sub>	NH-C <sub>2</sub> H <sub>5</sub>	NH-C(CH <sub>3</sub> ) <sub>3</sub>
Ametryn	S-CH <sub>3</sub>	NH-C <sub>2</sub> H <sub>5</sub>	NH-CH(CH <sub>3</sub> ) <sub>2</sub>
Desmetryn	S-CH <sub>3</sub>	NH-CH <sub>3</sub>	NH-CH(CH <sub>3</sub> ) <sub>2</sub>
Dimethametryn	S-CH <sub>3</sub>	NH-C <sub>2</sub> H <sub>5</sub>	NH-CH(CH <sub>3</sub> )-CH(CH <sub>3</sub> ) <sub>2</sub>
Methoprotryn	S-CH <sub>3</sub>	NH-CH(CH <sub>3</sub> ) <sub>2</sub>	NH-(CH <sub>2</sub> ) <sub>3</sub> -OCH <sub>3</sub>
Prometryn	S-CH <sub>3</sub>	NH-CH(CH <sub>3</sub> ) <sub>2</sub>	NH-CH(CH <sub>3</sub> ) <sub>2</sub>
Simetryn	S-CH <sub>3</sub>	NH-C <sub>2</sub> H <sub>5</sub>	NH-C <sub>2</sub> H <sub>5</sub>
Terbutryn	S-CH <sub>3</sub>	NH-C <sub>2</sub> H <sub>5</sub>	NH-C(CH <sub>3</sub> ) <sub>3</sub>



## Herbicides



Courtesy of Mr. Lembacher, Hipp Nährmittel, Pfaffenhofen, Germany

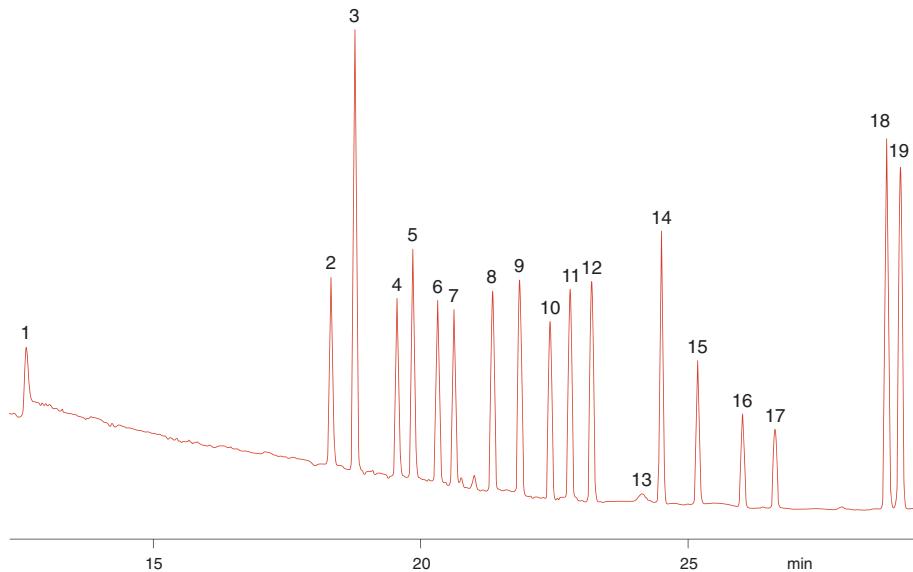
# Environmental pollutants

## Analysis of triazine herbicides MN Appl. No. 200820

Column: PERMABOND® FFAP, 25 m x 0.25 mm ID, 0.1 µm film, REF 723936.25,  
max. temperature 220/240 °C  
Injection: 2.5 µl, 1 min splitless, then split 1:50  
Carrier gas: 250 kPa He  
Temperature: 100 °C  $\xrightarrow{5\text{ °C/min}}$  240 °C  
Detector: PND 250 °C

**Peaks: [ng/ml]**

- |                        |                                |
|------------------------|--------------------------------|
| 1. Azobenzene [4768]   | 11. Simazin [856]              |
| 2. Prometon [716]      | 12. Ametryn [764]              |
| 3. Terbumeton [1646]   | 13. Cyanazine [728]            |
| 4. Atraton [740]       | 14. Simetryn [836]             |
| 5. Propazine [820]     | 15. Desethylatrazine [872]     |
| 6. Terbutylazine [780] | 16. Metribuzin [744]           |
| 7. Secbumeton [748]    | 17. Desisopropylatrazine [784] |
| 8. Atrazine [828]      | 18. Anilazine [3488]           |
| 9. Prometryn [792]     | 19. Methoprotyn [1544]         |
| 10. Terbutryn [784]    |                                |



Courtesy of J. Heering, Diplomarbeit FH Köln, Germany

# Herbicides

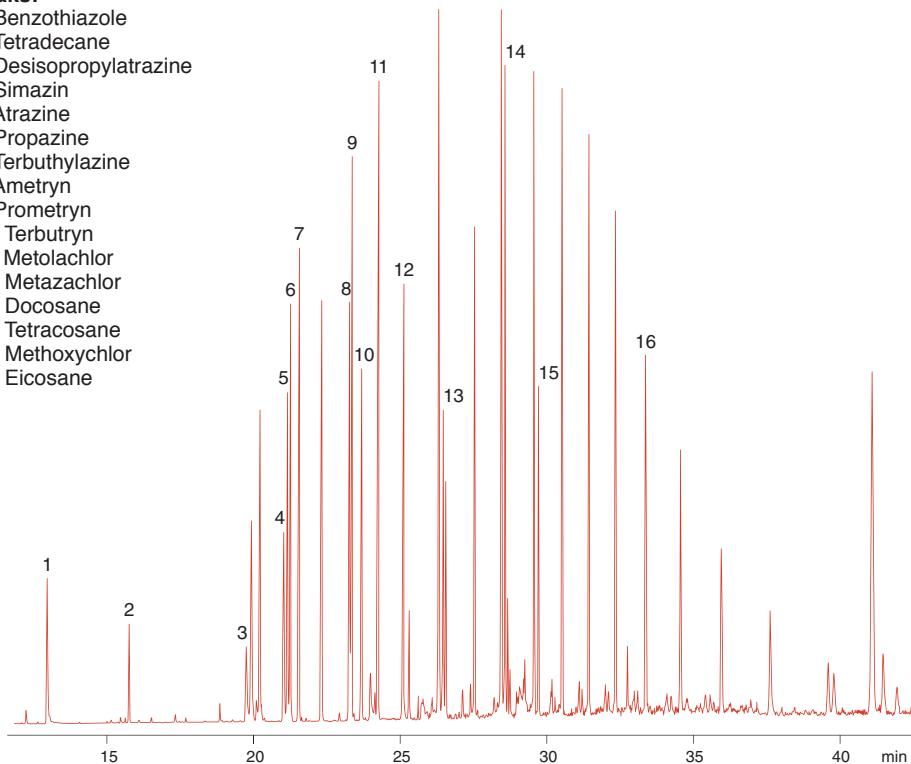


## Analysis of triazine herbicides MN Appl. No. 210410

Column: OPTIMA® 5 Amine, 25 m x 0.20 mm ID, 0.35 µm film, REF 726355.25,  
max. temperature 300/320 °C  
Carriergas: 24 cm/s He    8 °C/min  
Temperature: 70 °C (3 min) ————— 300 °C  
Detector: MSD

**Peaks:**

1. Benzothiazole
2. Tetradecane
3. Desisopropylatrazine
4. Simazin
5. Atrazine
6. Propazine
7. Terbutylazine
8. Ametryn
9. Prometryn
10. Terbutryn
11. Metolachlor
12. Metazachlor
13. Docosane
14. Tetracosane
15. Methoxychlor
16. Eicosane



## Environmental pollutants

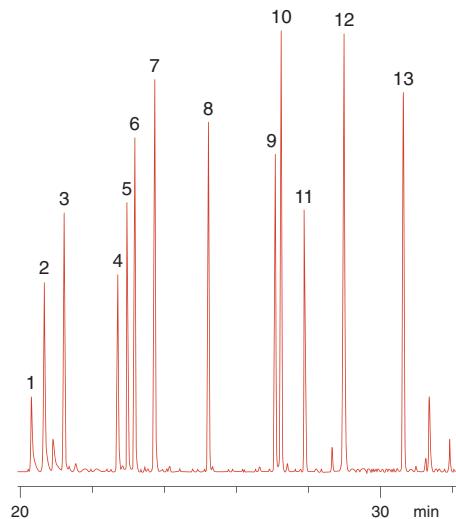
### Analysis of triazines

MN App. No. 250210

Column: OPTIMA® 5 MS,  
50 m x 0.25 mm ID,  
0.25  $\mu$ m film,  
REF 726220.50,  
max. temperature 340/360 °C  
Injection: splitless  
Carrier gas: 25 cm/s He  
Temperature: 120 °C (1 min)  $\xrightarrow{4 \text{ °C/min}}$  280 °C  
Detector: MSD

**Peaks:** [ppm]

1. Desisopropylatrazine [10.0]
2. Desethylatrazine [11.4]
3. Desethylterbutylazine [10.9]
4. Simazin [9.8]
5. Atrazine [10.2]
6. Propazine [10.8]
7. Terbutylazine [12.2]
8. Sebutylazine [12.1]
9. Ametryn [9.7]
10. Prometryn [12.1]
11. Terbutryn [9.3]
12. Metolachlor [14.8]
13. Metazachlor [12.6]



### Analysis of dinitroaniline herbicides

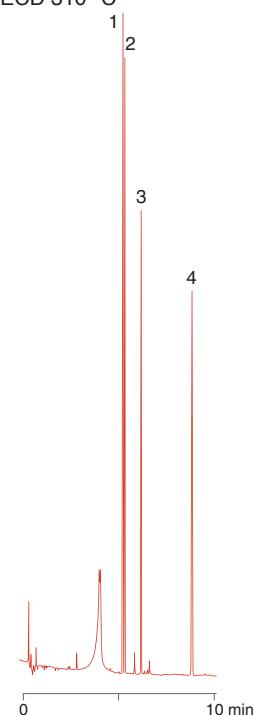
(EPA 627)

MN Appl. No. 200740

Column: OPTIMA® 1301,  
25 m x 0.32 mm ID,  
0.25  $\mu$ m film,  
REF 726777.25,  
max. temperature 300/320 °C  
Injection: 0.5  $\mu$ l on column, 100 pg each  
Carrier gas: 50 kPa H<sub>2</sub>  
Temperature: 80 °C  $\xrightarrow{30 \text{ °C/min}}$  200 °C  $\xrightarrow{4 \text{ °C/min}}$   
270 °C  
Detector: ECD 310 °C

**Peaks:**

1. Trifluralin
2. Ethalfluralin
3. Profluralin
4. Isopropalin



Courtesy of Mr. Lembacher, Hipp Nährmittel, Pfaffenhofen, Germany

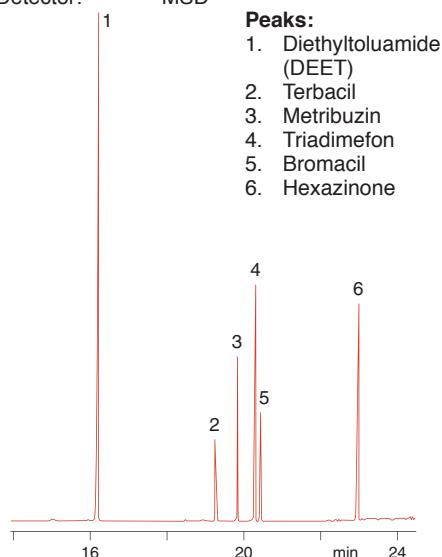
# Pesticides



## Analysis of organo-nitrogen pesticides (EPA 633)

MN Appl. No. 250110

Column: OPTIMA® δ-3,  
50 m x 0.25 mm ID,  
0.25 µm film,  
REF 726420.50,  
max. temperature 340/360 °C  
Injection: 1.0 µl, 3 s splitless  
Carrier gas: 1.3 bar He  
Temperature: 50 °C  $\xrightarrow{10\text{ °C/min}}$  220 °C  $\xrightarrow{20\text{ °C/min}}$   
320 °C  
Detector: MSD



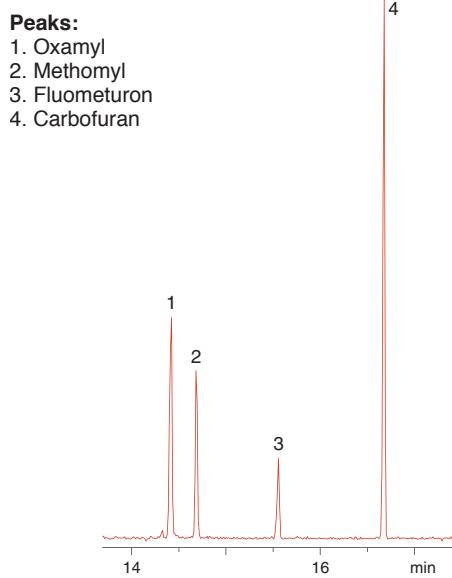
## Pesticide structures: uracil derivatives

Structure	Compound	R <sub>1</sub>	R <sub>2</sub>
	Bromacil	Br	CH(CH <sub>3</sub> )—C <sub>2</sub> H <sub>5</sub>
	Terbacil	Cl	C(CH <sub>3</sub> ) <sub>3</sub>
	Lenacil	—	—

## Analysis of carbamate/urea pesticides (EPA 632)

MN Appl. No. 200860

Column: OPTIMA® 5,  
25 m x 0.20 mm ID,  
0.20 µm film,  
REF 726857.25,  
max. temperature 340/360 °C  
Injection: 1.0 µl carbamate/urea pesticides (EPA 632), 10 µg/ml each in MeOH, 3 s splitless  
Carrier gas: 25 cm/s He  
Temperature: 50 °C (5 min)  $\xrightarrow{20\text{ °C/min}}$  220 °C  
 $\xrightarrow{10\text{ °C/min}}$  330 °C  
Detector: MSD



## Pesticide structures: oxime carbamates

Structure	Compound	R <sub>1</sub>
	Methomyl	CH <sub>3</sub>
	Oxamyl	CO—N(CH <sub>3</sub> ) <sub>2</sub>

Courtesy of Mr. Lembacher, Hipp Nährmittel, Pfaffenhofen, Germany

## Environmental pollutants

### Analysis of phenoxycarboxylic acid herbicides MN Appl. No. 201060

Column: OPTIMA® 1301, 25 m x 0.32 mm ID, 0.25 µm film, REF 726777.25,  
max. temperature 300/320 °C

Injection: 0.5 µl on column

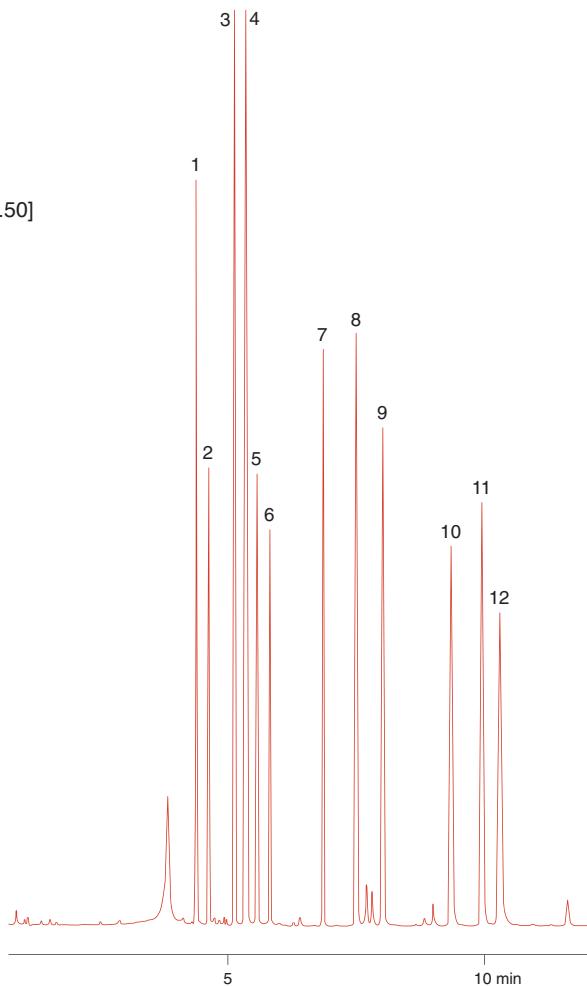
Carrier gas: 70 kPa H<sub>2</sub>

Temperature: 80  $\xrightarrow{30\text{ °C/min}}$  220 °C  $\xrightarrow{4\text{ °C/min}}$  270 °C

Detector: ECD 310 °C

**Peaks:** [ng]

1. Chlорfenprop-methyl [0.25]
2. 2,4-D methyl ester [0.25]
3. 2,4,5-TP methyl ester [0.125]
4. 2,4,5-T methyl ester [0.125]
5. 2,4-D isobutyl ester [0.25]
6. 2,4-D 1-butyl ester [0.25]
7. Alodan (int. std.)[0.05]
8. Dichlorprop 2-ethyl 1-hexyl ester [0.50]
9. 2,4-D isoctyl ester [0.50]
10. 2,4,5-T isoctyl ester [0.125]
11. Dichlorprop methyl ester [0.50]
12. 2,4,5-T octyl ester [0.50]



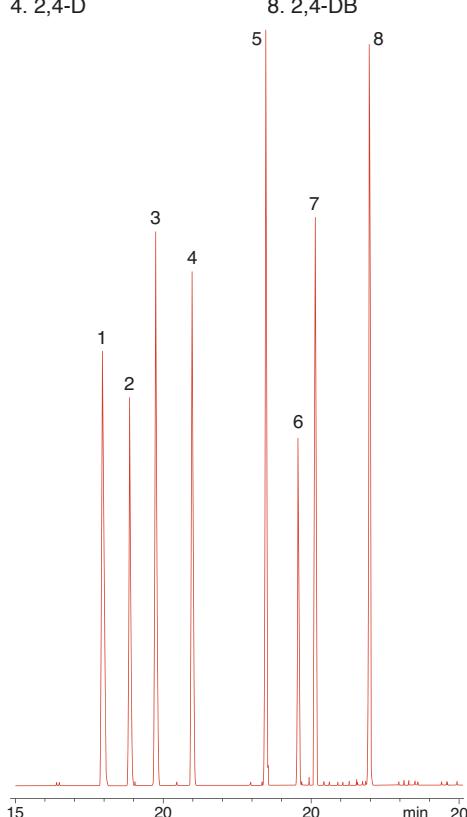
# Phenoxycarboxylic acid pesticides



## Analysis of phenoxycarboxylic acid herbicides (DIN 38407-14)

MN Appl. No. 213280

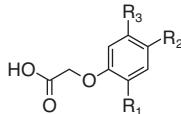
Column:	OPTIMA® 35 MS, 30 m x 0.25 mm ID, 0.25 µm film, REF 726154.30, max. temperature 360/370 °C
Injection:	2 µl, 280 °C, analytes derivatised with TMSH split 10 ml/min
Carrier gas:	1 bar He
Temperature:	145 °C $\xrightarrow{3\text{ °C/min}}$ 220 °C
Detector:	MSD
<b>Peaks:</b> methyl esters of	
1. Mecoprop	5. Fenoprop
2. MCPA	6. MCPB
3. Dichlorprop	7. 2,4,5-T
4. 2,4-D	8. 2,4-DB



## Pesticide structures: phenoxycarboxylic acids

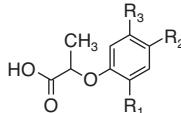
Compound	R <sub>1</sub>	R <sub>2</sub>	R <sub>3</sub>
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### Phenoxyacetic acid derivatives



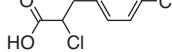
2,4-D	Cl	Cl	H
2,4,5-T	Cl	Cl	Cl
MCPA	CH <sub>3</sub>	Cl	H

### Phenoxypropionic acid derivatives

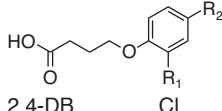


2,4-DP = Dichlorprop	Cl	Cl	H
2,4,5-TP = Fenoprop	Cl	Cl	Cl
MCPP = Mecoprop	CH <sub>3</sub>	Cl	H
Fluazifop	H	-O- N   CF <sub>3</sub>	H

### Chlorfenprop



### Phenoxybutyric acid derivatives



2,4-DB	Cl	Cl	-
MCPB	CH <sub>3</sub>	Cl	-

# Environmental pollutants

## Analysis of a pesticide mixture MN Appl. No. 210680

Column: OPTIMA® δ-3,  
50 m x 0.20 mm ID,  
0.20 µm film,  
REF 726400.50,  
max. temperature 340/360 °C

Injection: 2 µl, 8 ng/µl, splitless,  
KAS 60 °C  $\xrightarrow{12\text{ °C/s}}$  250 °C  
(2 min)

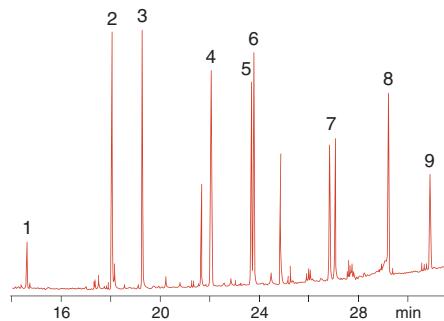
Carrier gas: 50 ml/min He, 35 psi

Temperature: 100 °C (12 min)  $\xrightarrow{15\text{ °C/min}}$   
220 °C (1 min)  $\xrightarrow{5\text{ °C/min}}$  340 °C  
(10 min)

Detector: MSD

**Peaks:**

1. Acephate
2. Pirimicarb
3. Metalaxyl
4. Metazachlor
5. α-Endosulfan
6. Profenofos
7. Propiconazole
8. Methoxychlor
9. Phosalone



Courtesy of Mr. Heinzler, Staatl. Medizinal-, Lebensmittel- und Veterinär-Untersuchungsamt, Kassel, Germany

## Analysis of organochlorine pesticides MN Appl. No. 210840

Column: OPTIMA® δ-6,  
30 m x 0.25 mm ID,  
0.25 µm film, REF 726470.30,  
max. temperature 340/360 °C

Injection: 10 µg/ml in acetone

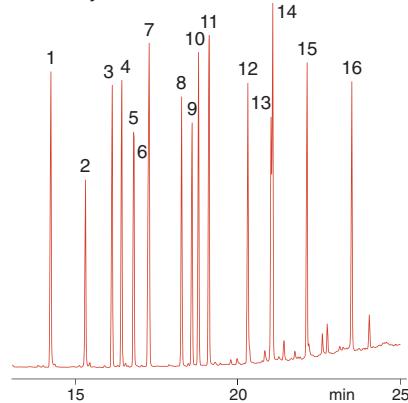
Carrier gas: H<sub>2</sub>

Temperature: 60 °C (2 min)  $\xrightarrow[8\text{ °C/min}]{30\text{ °C/min}}$  150 °C  
 $\xrightarrow{300\text{ °C}}$  300 °C (6 min)

Detector: MSD (ion trap)

**Peaks:**

1. α-BHC
2. Quintozene
3. β-BHC
4. Heptachlor
5. δ-BHC
6. Chlorothalonil
7. Aldrin
8. Isodrin
9. *cis*-Heptachlor epoxide
10. *trans*-Heptachlor epoxide
11. *p,p'*-DDE
12. Dieldrin
13. Endrin
14. *o,p'*-DDT
15. *p,p'*-DDT
16. Methoxychlor



Courtesy of Mrs. Geilen, Berg. Wasser- u. Umweltlabor, Wuppertal Germany

# Pesticides

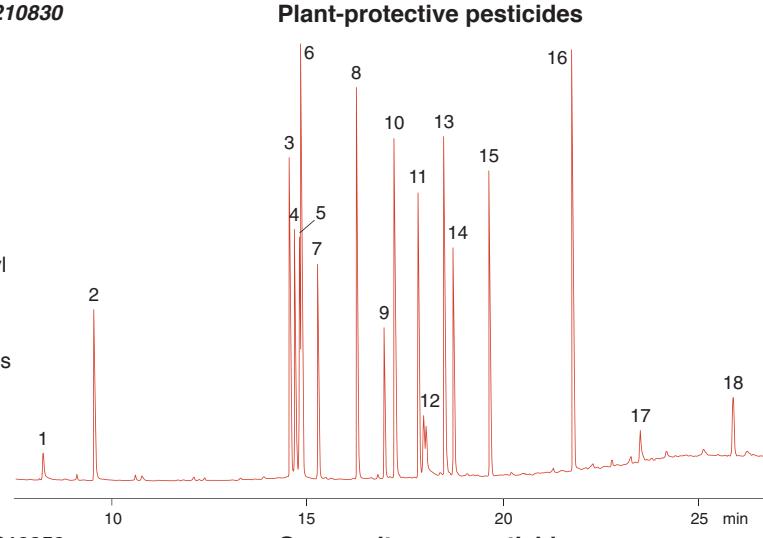


Column and conditions as in application 210840

**MN Appl. No. 210830**

**Peaks:**

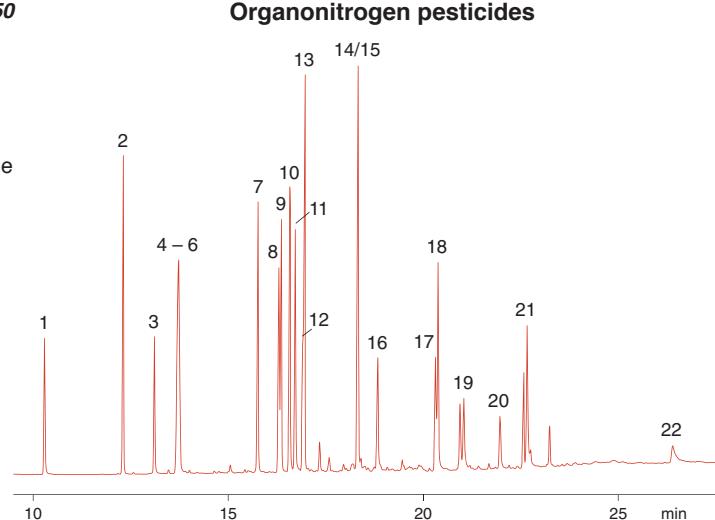
1. Carbofuran
2. Dichlobenil
3. Propazine
4. Atrazine
5. Simazin
6. Terbutylazine
7.  $\gamma$ -BHC
8. Alachlor
9. Parathion-methyl
10. Metolachlor
11. Parathion-ethyl
12. Bromacil
13. Chlорfenvinphos
14. Metazachlor
15.  $\alpha$ -Endosulfan
16.  $\beta$ -Endosulfan
17. Chloridazon
18. Azinphos-ethyl



**MN Appl. No. 210850**

**Peaks:**

1. Propham
2. Trifluralin
3. Chlorpropham
4. Desethylatrazine
5. Desethylterbutylazine
6. Desisopropylatrazine
7. Sebutylazine
8. Vinclozolin
9. Desmetryn
10. Prometryn
11. Ametryn
12. Metribuzin
13. Terbutryn
14. Cyanazine
15. Pendimethalin
16. Triadimenol
17. Flusilazole
18. Methoprotyn
19. Cyproconazole
20. Metamitron
21. Tebuconazole
22. Prochloraz



Courtesy of Mrs. Geilen, Berg. Wasser- u. Umweltlabor, Wuppertal Germany

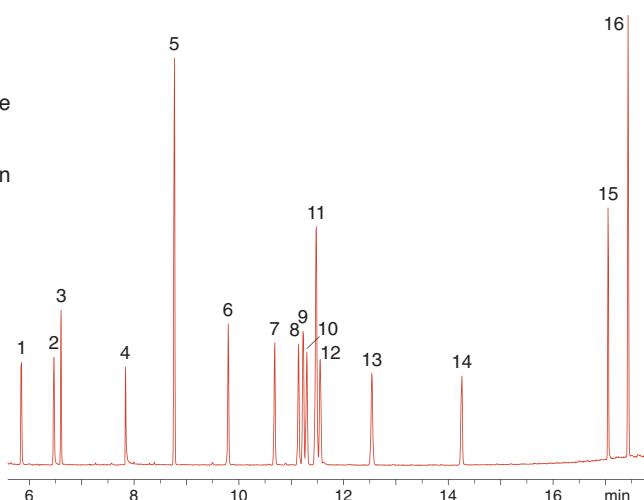
# Environmental pollutants

## Separation of pesticides and aromatic hydrocarbons (EPA 526 mix) MN Appl. No. 212820

Column: OPTIMA® 5 MS Accent, 30 m x 0.25 mm ID, 0.25 µm film, REF 725820.30, max. temperature 340/360 °C  
 Sample: EPA method 526 mix, 10 ppm (20 ppm internal standard)  
 Injection: 1 µl splitless, hold 0.3 min, 300 °C  
 Carrier gas: 0.8 ml/min He  
 Temperature: 50 °C (1 min)  $\xrightarrow{20\text{ °C/min}}$  200 °C (5 min)  $\xrightarrow{30\text{ °C/min}}$  310 °C (3 min)  
 Detector: MSD 280 °C

**Peaks:**

1. Nitrobenzene
2. 2,4-Dichlorophenol
3. 1,3-Dimethyl-2-nitrobenzene
4. 2,4,6-Trichlorophenol
5. Acenaphthene-d<sub>10</sub> (int. st.)
6. Azobenzene (decomposition product of 1,2-diphenyl-hydrazine)
7. Prometon
8. Terbufos
9. Diazinon
10. Fonofos
11. Phenanthrene-d<sub>10</sub> (int. st.)
12. Disulfoton
13. Acetochlor
14. Cyanazine
15. Triphenyl phosphate
16. Chrysene-d<sub>12</sub>



## Pesticide structures: miscellaneous heterocyclic aromatic compounds

Structure	Compound	R <sub>1</sub>	R <sub>2</sub>	R <sub>3</sub>	R <sub>4</sub>	R <sub>5</sub>
	Fluorochloridone		H	CF <sub>3</sub>	H	H
	Iprodione		H	Cl	H	Cl
	Oxadiazon	(CH <sub>3</sub> ) <sub>3</sub> C	Cl	H	Cl	O-CH(CH <sub>3</sub> ) <sub>2</sub>
	Procyomidone		H	Cl	H	Cl
	Vinclozolin		H	Cl	H	Cl

# Pesticides



## On-column analysis of a pesticide mixture MN Appl. No. 210430

Column: OPTIMA® 5, 50 m x 0.25 mm ID, 0.25 µm film, REF 726056.50,  
max. temperature 340/360 °C

Retention gap: Me-Sil deactivated, 1.6 m x 0.25 mm ID, REF 723706.10 (10 m)

Injection: 2 µl on column

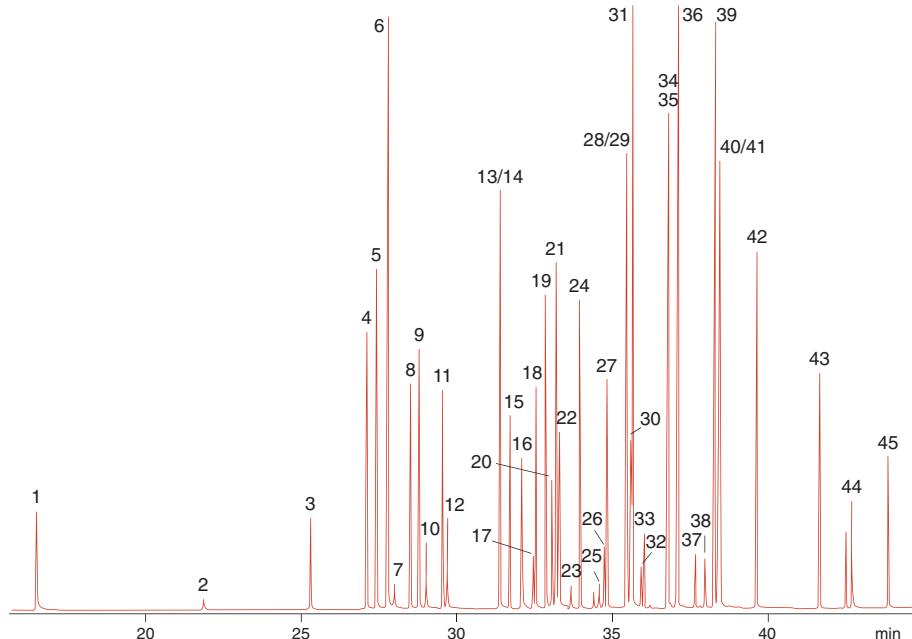
Flow: 0.8 bar → 2.0 bar, 0.025 bar/min

Temperature: 60 °C (1 min)  $\xrightarrow{5 \text{ °C/min}}$  270 °C  $\xrightarrow{20 \text{ °C/min}}$  310 °C (8 min)

Detector: MSD

### Peaks:

- |                            |                                      |                           |
|----------------------------|--------------------------------------|---------------------------|
| 1. Dichlorvos              | 16. Heptachlor                       | 31. <i>o,p'</i> -DDE      |
| 2. Trichlorfon             | 17. Fenitrothion                     | 32. $\alpha$ -Endosulfan  |
| 3. Tecnazene               | 18. Pirimiphos-methyl                | 33. <i>cis</i> -Chlordane |
| 4. Sulfotep                | 19. Malathion                        | 34. <i>p,p'</i> -DDE      |
| 5. $\alpha$ -BHC           | 20. Aldrin                           | 35. Dieldrin              |
| 6. HCB                     | 21. Fenthion                         | 36. <i>o,p'</i> -DDD      |
| 7. Dimethoate              | 22. Chlorpyrifos-ethyl               | 37. Endrin                |
| 8. $\beta$ -BHC            | 23. Chlorthion                       | 38. $\beta$ -Endosulfan   |
| 9. $\gamma$ -BHC           | 24. Bromophos-methyl                 | 39. <i>p,p'</i> -DDD      |
| 10. Quintozene             | 25. <i>trans</i> -Heptachlor epoxide | 40. <i>o,p'</i> -DDT      |
| 11. Diazinon               | 26. <i>cis</i> -Heptachlor epoxide   | 41. Ethion                |
| 12. Disulfoton             | 27. Chlorfenvinphos                  | 42. <i>p,p'</i> -DDT      |
| 13. Chlorpyrifos-methyl    | 28. Methidathion                     | 43. Methoxychlor          |
| 14. Parathion-methyl       | 29. <i>trans</i> -Chlordane          | 44. Azinphos-methyl       |
| 15. Demeton-S-methylsulfon | 30. Bromophos-ethyl                  | 45. Azinphos-ethyl        |



# Environmental pollutants

**Analysis of pesticide mixtures**

Column: OPTIMA® 1301, 60 m x 0.25 mm ID, 0.25 µm film, REF 726771.60,  
max. temperature 300/320 °C

Injection: 3 µl, 0.1 ng/µl, 80 °C (1 min) → 250 °C (1 min), pulsed splitless

Carrier gas: 54 ml/min He

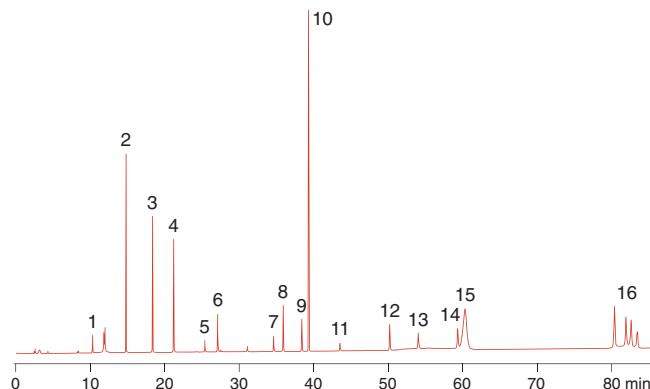
Temperature: 80 °C (2 min)  $\xrightarrow{20\text{ °C/min}}$  190 °C (12 min)  $\xrightarrow{2\text{ °C/min}}$  240 °C (23 min)  $\xrightarrow{10\text{ °C/min}}$  260 °C  
(20 min)

Detector: ECD

**MN Appl. No. 210630**

**Peaks:**

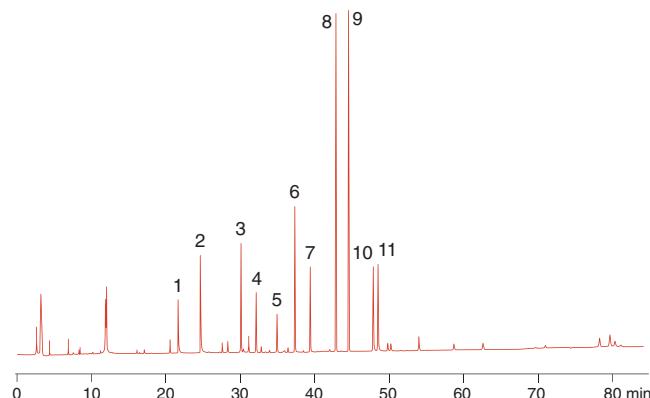
1. Phosphamidon
2. Tecnazene
3. α-BHC
4. Lindane
5. Chlorothalonil
6. Fenchlorphos
7. Metazachlor
8. Bromophos-ethyl
9. Prothiophos
10. Dieldrin  
+ profenofos
11. o,p'-DDT
12. Nuarimol
13. Bromopropylate
14. Bifenox
15. Tetradifon
16. – 19. Cypermethrin,  
4 stereoisomers



**MN Appl. No. 210640**

**Peaks:**

1. Dimethoate
2. Propyzamide
3. Demeton-S-methyl  
+ chlorpyrifos-ethyl
4. Parathion-ethyl
5. Quinalphos  
+ penconazole
6. Procymidone  
+ methidathion
7. Dieldrin  
+ profenofos
8. Myclobutanil
9. PCB-128  
+ β-endosulfan  
+ ethion
10. PCB-153
11. Propiconazole



Courtesy of Mrs. Goda, Lebensmitteluntersuchungsamt, Braunschweig, Germany

# Pesticides



## Analysis of a pesticide mixture

Sample: pesticide standard of Kantonal Laboratory Schaffhausen (Switzerland)  
0.1 mg/ml or 0.01 mg/ml each

Injection: 1.0  $\mu$ l, 3 s splitless

Carrier gas: 25 cm/s He

Temperature: 100 °C (3 min)  $\xrightarrow{8\text{ °C/min}}$  250 °C  $\xrightarrow{10\text{ °C/min}}$  320 °C

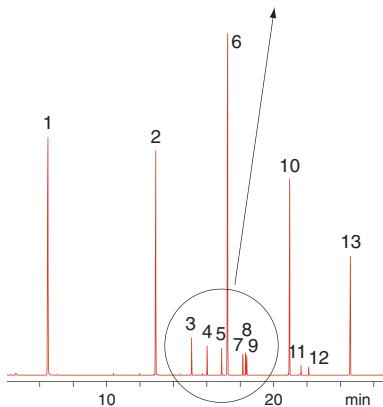
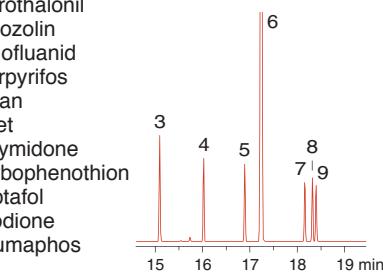
Detector: MSD

### MN Appl. No. 200920

Column: OPTIMA® 5,  
25 m x 0.20 mm ID,  
0.20  $\mu$ m film, REF 726857.25,  
max. temperature 340/360 °C

#### Peaks:

1. Dichlorvos
2. Naled
3. Chlorothalonil
4. Vinclozolin
5. Dichlofluanid
6. Chlorpyrifos
7. Captan
8. Folpet
9. Procymidone
10. Carbophenothion
11. Captafol
12. Iprodione
13. Coumaphos

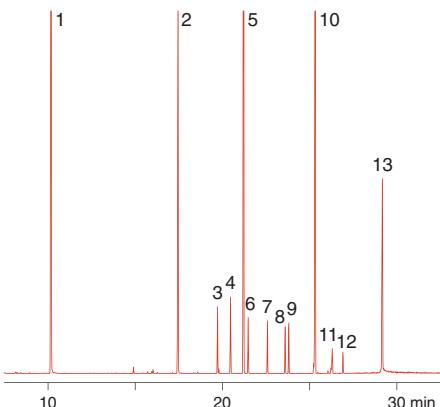


### MN Appl. No. 200930

Column: OPTIMA® 17,  
25 m x 0.20 mm ID,  
0.20  $\mu$ m film, REF 726065.25,  
max. temperature: 320/340 °C

#### Peaks:

1. Dichlorvos
2. Naled
3. Vinclozolin
4. Chlorothalonil
5. Chlorpyrifos
6. Dichlofluanid
7. Procymidone
8. Captan
9. Folpet
10. Carbophenothion
11. Iprodione
12. Captafol
13. Coumaphos



## Pesticide structures: phthalimide derivatives

Structure	Compound	R	Structure	Compound
	Captan	CCl <sub>3</sub>		Folpet
	Captafol	CCl <sub>2</sub> -CCl <sub>2</sub> H		

## Environmental pollutants

Miscellaneous pesticide structures					
Structure	Compound	R <sub>1</sub>	R <sub>2</sub>	Structure	Compound
<chem>*N(*)c1cc(Cl)c(=O)n2c(*)cccc2n1</chem>	Chlоридазон	H	H	<chem>C2H5O-S=C(CCl3)=N</chem>	Етидазол
<chem>*c1nc2sc(S(=O)(=O)R2)nc2n1</chem>	Норфлуразон	CH <sub>3</sub>	CF <sub>3</sub>		
<chem>*c1cc2c(cc1S(=O)(=O)R2)nc3sc2n1</chem>	Квинометионат	CH <sub>3</sub>	O		Флуридон
<chem>*c1cc2c(cc1S(=O)(=O)R2)nc3sc2n1</chem>	Тиохинокс	H	S		
<chem>*c1cc2c(cc1S(=O)(=O)R2)nc3sc2n1</chem>	Карбоксин	—	—	<chem>c1nc2scn2n1</chem>	Тиабендазол
<chem>*c1cc2c(cc1S(=O)(=O)R2)nc3sc2n1</chem>	Этокоин	—	—	<chem>c1nc2scn2n1</chem>	Трициклазол

### Analysis of halogenated acetic acids (EPA 552.2)

*MN Appl. No. 213210*

Column: OPTIMA® 35 MS, 30 m x 0.25 mm ID, 0.25 µm film, REF 726154.30,  
max. temperature 360/370 °C

Injection: 1 µl, split 1:20

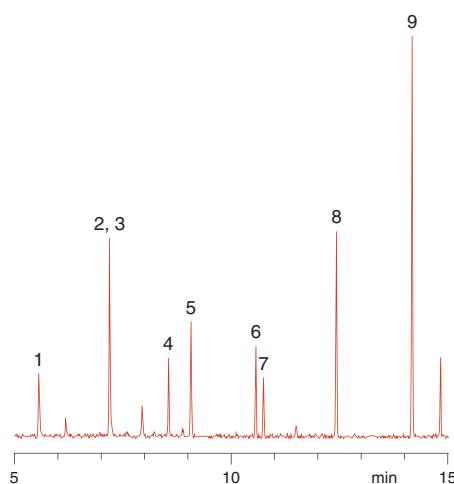
Carrier gas: 1 ml/min He

Temperature: 50 °C (3 min)  $\xrightarrow{10\text{ °C/min}}$  200 °C (10 min)

Detector: MSD

**Peaks:**

1. Члороакетицкый кислоты метил эстэр
2. Диchlороакетицкый кислоты метил эстэр
3. Бромоакетицкый кислоты метил эстэр
4. Триchlороакетицкый кислоты метил эстэр
5. Бромochlороакетицкый кислоты метил эстэр
6. Бромодиchlороакетицкый кислоты метил эстэр
7. Дибромоакетицкый кислоты метил эстэр
8. Дибромochlороакетицкый кислоты метил эстэр
9. Трибромоакетицкый кислоты метил эстэр



# Organic acids



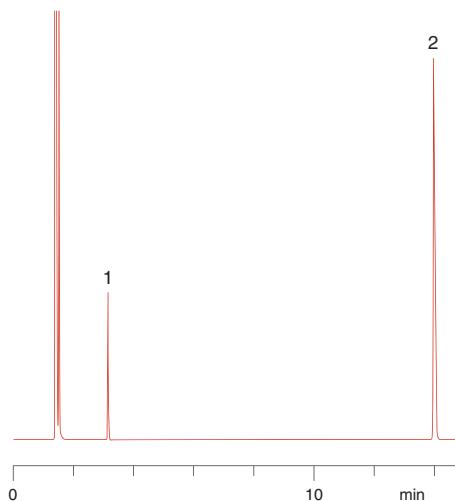
## Analysis of acetic acid and trifluoroacetic acid

MN Appl. No. 213250

Column: OPTIMA® FFAP,  
25 m x 0.25 mm ID,  
0.25 µm film, REF 726116.25,  
max. temperature 250/260 °C  
Injection: 1 µl, split 1:40  
Carrier gas: 0.95 bar He     $\frac{10 \text{ °C/min}}{\text{Temperature: } 40 \text{ °C (5 min)}}$     120 °C  
Detector: FID 250 °C

### Peaks:

1. Trifluoroacetic acid (TFA)
2. Acetic acid



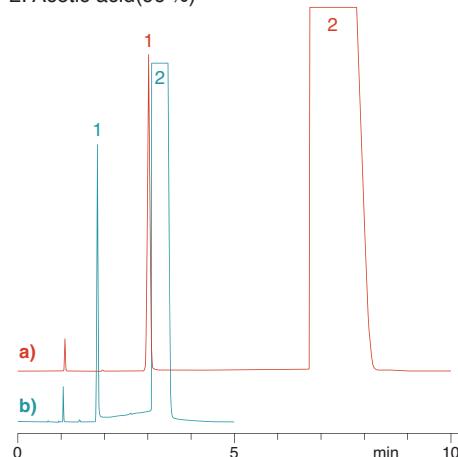
## Analysis of acetic acid and acetic acid anhydride

MN Appl. No. 250620

Column: PERMABOND® FFAP,  
25 m x 0.53 mm ID,  
1.0 µm film, REF 723555.25,  
max. temperature 200/220 °C  
Injection: 0.6 µl, split 1:100  
Carrier gas: 0.4 bar He  
Temperature: a) 100 °C, b) 130 °C  
Detector: FID 200 °C

### Peaks:

1. Acetic acid anhydride (1 %)
2. Acetic acid(99 %)



Although retention times are drastically decreased at higher temperatures, the title compounds are still perfectly separated at 130 °C with an even better peak shape.

## Solvents • Chemicals

# Solvents Chemicals



# Aromatic hydrocarbons



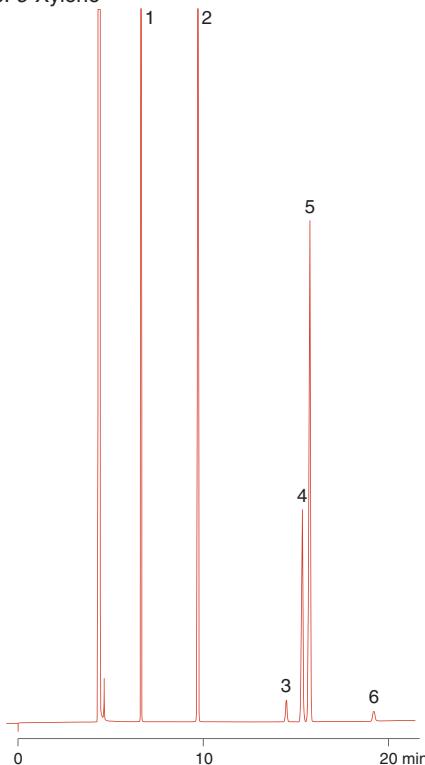
## Analysis of aromatic hydrocarbons (BTX)

MN Appl. No. 200230

Column: OPTIMA® 210,  
50 m x 0.25 mm ID,  
0.5 µm film, REF 726874.50,  
max. temperature 260/280 °C  
Injection: 0.5 µl, split 105 ml/min  
Carrier gas: 130 kPa N<sub>2</sub> (1.1 ml/min)  
Temperature: 50 °C  
Detector: FID 250 °C

**Peaks:**

1. Benzene
2. Toluene
3. Ethylbenzene
4. *p*-Xylene
5. *m*-Xylene
6. *o*-Xylene



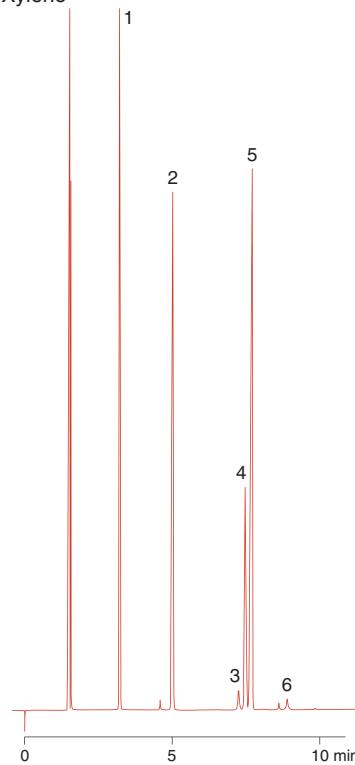
## Analysis of aromatic hydrocarbons (BTX)

MN Appl. No. 200210

Column: PERMABOND® CW 20 M,  
25 m x 0.25 mm ID,  
0.25 µm film, REF 723060.25,  
max. temperature 220/240 °C  
Injection: 0.2 µl, split 1:50  
Carrier gas: 0.8 bar N<sub>2</sub>  
Temperature: 40 °C (3 min) → 80 °C  
Detector: FID 260 °C

**Peaks:**

1. Benzene
2. Toluene
3. Ethylbenzene
4. *p*-Xylene
5. *m*-Xylene
6. *o*-Xylene



## Solvents • Chemicals

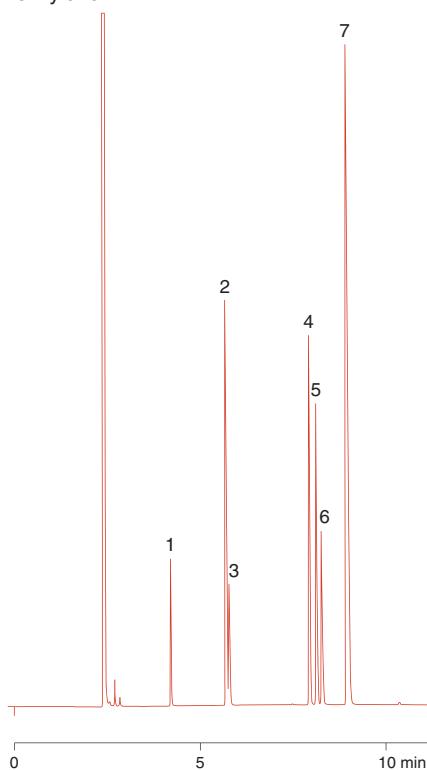
### Analysis of aromatic hydrocarbons (BTX with toluene-d<sub>8</sub>)

MN Appl. No. 200260

Column: FS-HYDRODEX β-PM,  
50 m x 0.25 mm ID,  
REF 723370.50,  
max. temperature 230/250 °C  
Split: 185 ml/min  
Carrier gas: 120 kPa H<sub>2</sub> (2 ml/min)  
Temperature: 60 °C → 100 °C  
Detector: FID 250 °C

#### Peaks in n-hexane:

1. Benzene
2. Toluene-d<sub>8</sub>
3. Toluene
4. p-Xylene
5. m-Xylene
6. Ethylbenzene
7. o-Xylene

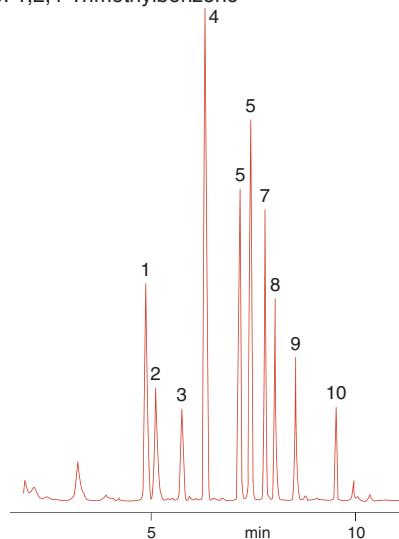


### Analysis of aromatic hydrocarbons MN Appl. No. 210590

Column: OPTIMA® δ-3,  
30 m x 0.25 mm ID,  
0.25 µm film, REF 726420.30,  
max. temperature 340/360 °C  
Injection: 0.5 µl, 250 °C, split 1:10  
Carrier gas: He 12 °C/min  
Temperature: 50 °C → 200 °C  
Detector: MSD

#### Peaks:

1. Ethylbenzene
2. p-Xylene
3. o-Xylene
4. Cumene
5. n-Propylbenzene
6. 4-Ethyltoluene
7. 1,3,5-Trimethylbenzene
8. 2-Ethyltoluene
9. 1,2,3-Trimethylbenzene
10. 1,2,4-Trimethylbenzene



Courtesy of Mr. B. Sievers, Riedel-de Haen AG,  
Seelze, Germany

# Aromatic hydrocarbons

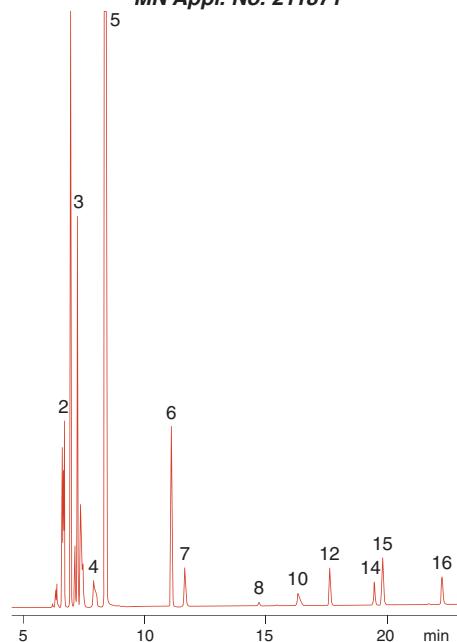


## Analysis of aromatic solvents

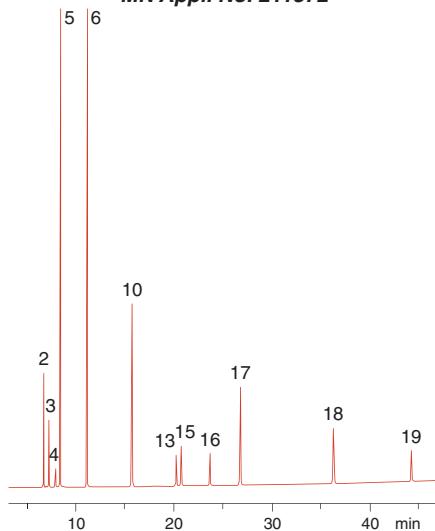
Column: OPTIMA® 210,  
50 m x 0.25 mm ID,  
0.25  $\mu\text{m}$  film, REF 726871.50,  
max. temperature 260/280 °C  
Injection: 0.2  $\mu\text{l}$ , split 89 ml/min  
Carrier gas: 60 kPa N<sub>2</sub>  
Temperature: 50 °C (10 min)  $\xrightarrow{2\text{ °C/min}}$  180 °C  
(20 min)  
Detector: FID 260 °C

MN Appl. No. 211871

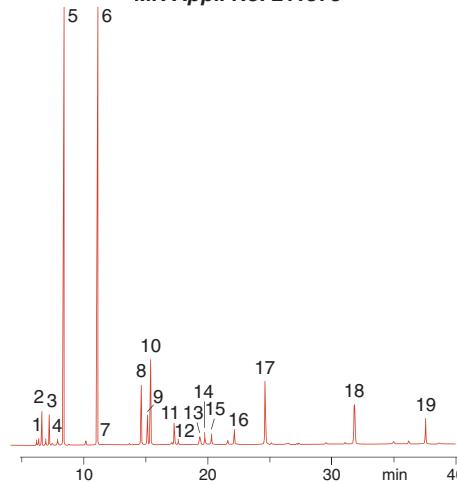
Peaks:	
1. <i>n</i> -Butane	13. <i>n</i> -Propylbenzene
2. <i>n</i> -Hexane	14. <i>p</i> -Ethyltoluene
3. Cyclohexane	15. <i>m</i> -Ethyltoluene
4. Methylcyclohexane	16. 1,2,4-Trimethylbenzene
5. Benzene	17. Indane
6. Toluene	
7. <i>n</i> -Nonane	18. Tetralin
8. Ethylbenzene	
9. <i>p</i> -Xylene	19. Naphthalene
10. <i>m</i> -Xylene	
11. <i>o</i> -Xylene	
12. <i>i</i> -Propylbenzene	



MN Appl. No. 211872



MN Appl. No. 211873



Courtesy of Aral Aromatics GmbH, Gelsenkirchen, Germany

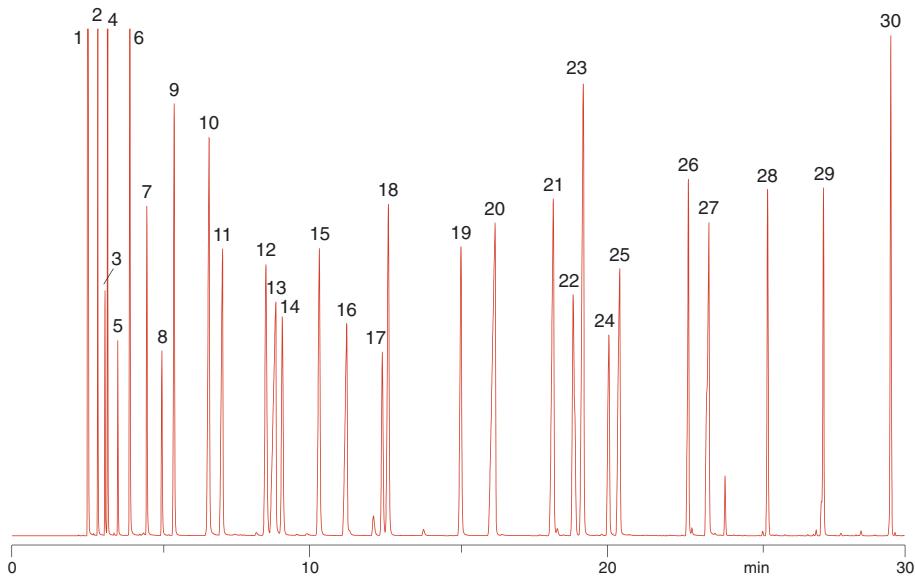
## Solvents • Chemicals

### Analysis of solvents MN Appl. No. 201390

Column: OPTIMA® 1, 60 m x 0.32 mm ID, 1.0 µm film, REF 726323.60,  
max. temperature 340/360 °C  
Sample: solvent mixture, courtesy of J. Lutz, Alcan Rorschach, Switzerland  
Injection: 0.4 µl, split 1:60  
Carrier gas: 120 kPa H<sub>2</sub>  
Temperature: 50 °C (9 min) → 90 °C → 280 °C (2 min)  
Detector: FID 300 °C

**Peaks:**

- |                            |                                    |
|----------------------------|------------------------------------|
| 1. Methanol                | 16. 1-Ethoxy-2-propanol            |
| 2. Ethanol                 | 17. Toluene                        |
| 3. Acetone                 | 18. <i>i</i> -Butyl acetate        |
| 4. Propanol-2              | 19. <i>n</i> -Butyl acetate        |
| 5. Methyl acetate          | 20. 4-Hydroxy-4-methyl-2-pentanone |
| 6. Propanol-1              | 21. 1-Methoxy-2-propyl acetate     |
| 7. Methyl ethyl ketone     | 22. Xylene                         |
| 8. Ethyl acetate           | 23. Cyclohexanone                  |
| 9. Isobutanol              | 24. Ethyl glycol acetate           |
| 10. Butanol-1              | 25. Butyl glycol                   |
| 11. 1-Methoxy-2-propanol   | 26. Heptanol                       |
| 12. Isooctane              | 27. Ethyldiglycol                  |
| 13. Ethylglycol            | 28. Butyldiglycol                  |
| 14. Isoheptane             | 29. Butyl glycol acetate           |
| 15. Methyl isobutyl ketone | 30. Butyldiglycol acetate          |



# Solvents

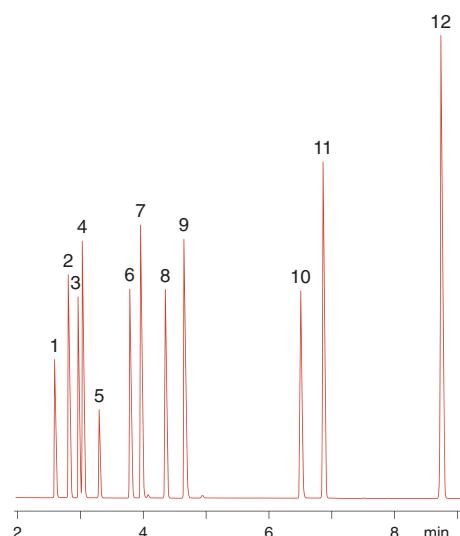


## Analysis of solvents MN Appl. No. 213150

Column: OPTIMA® 1 MS Accent,  
60 m x 0.32 mm ID,  
0.5  $\mu$ m film,  
REF 725807.60,  
max. temperature 340/360 °C  
Injection: 1  $\mu$ l, split 1:150  
Carrier gas: 1 bar He  
Temperature: 40 °C (5 min)  $\xrightarrow{10\text{ °C/min}}$  60 °C  
(2 min)  
Detector: FID 280 °C

**Peaks:**

1. Methanol
2. Ethanol
3. Acetone
4. Propanol-2
5. Dichloromethane
6. *t*-Butyl ethyl ether
7. Methyl ethyl ketone
8. Hexane
9. Tetrahydrofuran (THF) 
10. Dioxane 
11. Heptane
12. Toluene

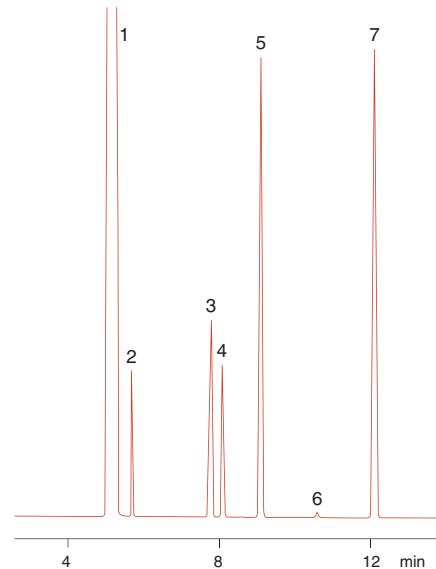


## Analysis of volatile solvents MN Appl. No. 210300

Column: OPTIMA®  $\delta$ -3,  
60 m x 0.32 mm ID,  
1  $\mu$ m film, REF 726442.60,  
max. temperature 340/360 °C  
Injection: 1  $\mu$ l, split 1:75  
Carrier gas: 1.2 bar He  
Temperature: 34 °C (1 min)  $\xrightarrow{4\text{ °C/min}}$   
50 °C  $\xrightarrow{6\text{ °C/min}}$  100 °C  $\xrightarrow{8\text{ °C/min}}$   
200 °C (5 min)  
Detector: FID

**Peaks:**

1. Methanol
2. Methyl formate
3. Methyl acetate
4. Acetonitrile
5. *n*-Hexane
6. Ethyl acetate
7. Cyclohexane



# Solvents • Chemicals

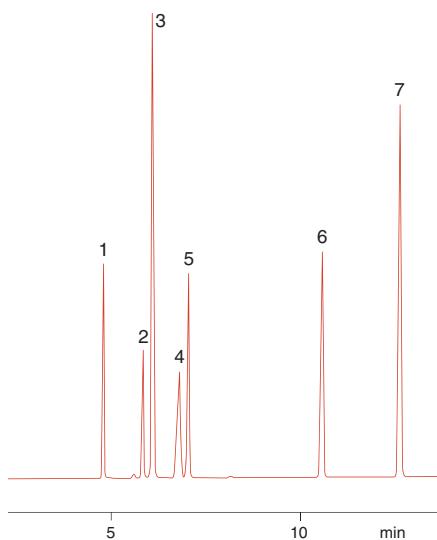
## Analysis of solvents

MN Appl. No. 210260

Column: OPTIMA® δ-3,  
60 m x 0.32 mm ID,  
1 µm film, REF 726442.60,  
max. temperature 340/360 °C  
  
Injection: 0.5 µl, split 1:100  
Carrier gas: 1.2 bar He       $6\text{ °C/min}$   
Temperature: 60 °C (1 min)  $\xrightarrow{6\text{ °C/min}}$  240 °C  
(5 min)  
Detector: FID

**Peaks:**

1. Methanol
2. Ethanol
3. Pentane
4. Propanol-2
5. Acetone
6. Ethyl acetate
7. Benzene



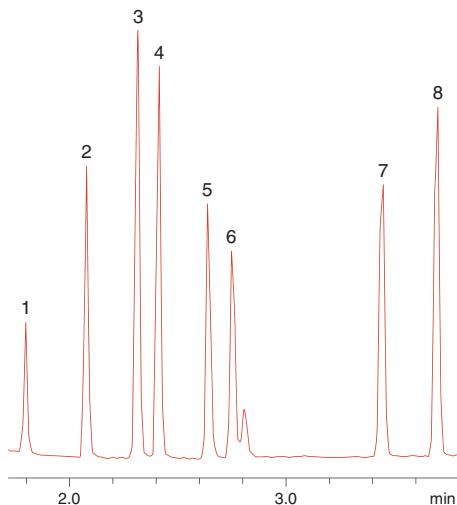
## Analysis of solvents by headspace GC

MN Appl. No. 212960

Column: Optima® WAX,  
30 m x 0.25 mm ID,  
0.25 µm film, REF 726600.30,  
max. temperature 250/260 °C  
  
Sample: headspace: heat 1 ml water +  
20 µl of each solvent to 80 °C  
in a headspace vial for 15 min  
  
Injection: 240 °C, split 3.64:1  
Carrier gas: 7.35 psi He, 1 ml/min  
Temperature: 45 °C (1 min)  $^{10\text{ °C/min}}$  180 °C  
(2 min)  $^{30\text{ °C/min}}$   $\xrightarrow{240\text{ °C}}$  240 °C (3 min)  
Detector: MSD

**Peaks:**

1. Cyclohexane
2. Acetone
3. Tetrahydrofuran
4. Ethyl acetate
5. Dichloromethane
6. Propanol-2
7. Trichloromethane
8. Toluene



Courtesy of V. Cirimele, Laboratoire ChemTox,  
Illkirch Graffenstaden, France

# Solvents

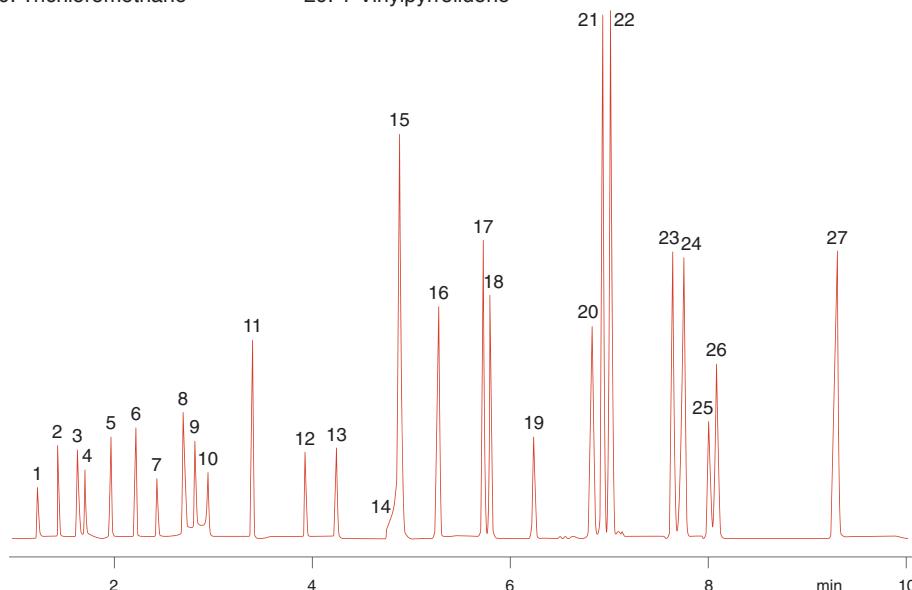


## Rapid analysis of a complex solvent mixture MN Appl. No. 250500

Column: OPTIMA® δ-3, 30 m x 0.25 mm ID, 0.25 µm film, REF 726420.30,  
max. temperature 340/360 °C  
Injection: 1.0 µl, split 1:80  
Carrier gas: 100 kPa He  
Temperature: 35 °C (1.8 min)  $\xrightarrow{25\text{ °C/min}}$  50 °C  $\xrightarrow{70\text{ °C/min}}$  200 °C (15 min)  
Detector: FID

**Peaks:**

- |                      |                        |                            |
|----------------------|------------------------|----------------------------|
| 1. Methanol          | 11. Benzene            | 21. Menthofuran + menthone |
| 2. Ethanol           | 12. Trichloroethene    | 22. Isomenthone + pulegone |
| 3. Propanol-2        | 13. 1,4-Dioxane        | 23. Methyl acetate         |
| 4. Acetone           | 14. Propylene glycol   | 24. Carvone                |
| 5. Dichloromethane   | 15. Toluene + pentenol | 25. Anethole               |
| 6. <i>n</i> -Hexane  | 16. Heptanol-1         | 26. Anisaldehyde           |
| 7. Vinyl acetate     | 17. Limonene           | 27. Dodecanol-1            |
| 8. Butanol-2         | 18. Cineole            |                            |
| 9. Ethyl acetate     | 19. Linalool           |                            |
| 10. Trichloromethane | 20. 1-Vinylpyrrolidone |                            |



Courtesy of M. Bürkler, Solco Basel AG, Birsfelden, Switzerland

## Solvents • Chemicals

### Analysis of solvents and semi-volatiles MN Appl. No. 212520

Column: OPTIMA® 624 LB, 30 m x 0.32 mm ID, 1.8  $\mu$ m film, REF 726786.30  
max. temperature 280/300 °C, retention gap Phe-Sil 0.5 m x 0.53 mm,  
REF 723711.10

Injection: 1  $\mu$ l, cold on-column, 10 ppm per substance in acetone

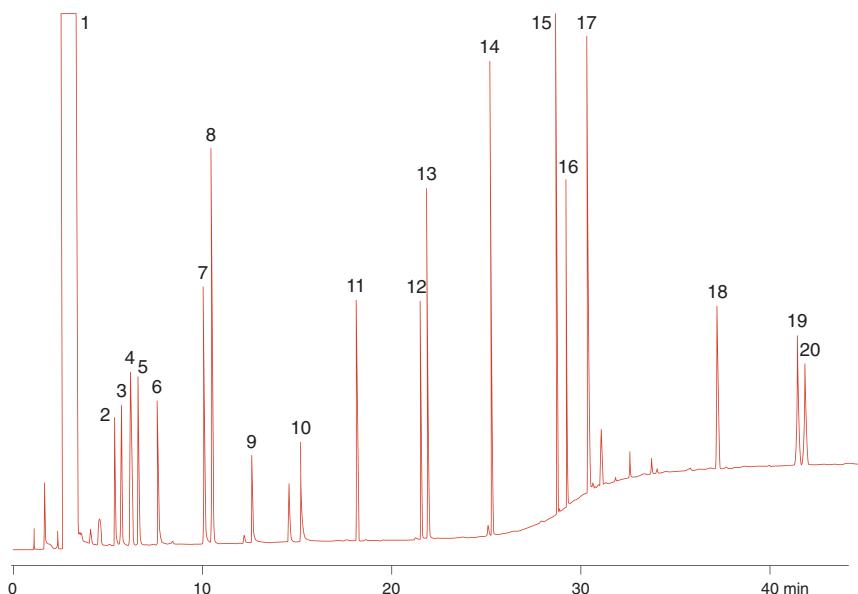
Carrier gas: 1.1 bar He

Temperature: 45 °C (3 min)  $\xrightarrow{6\text{ °C/min}}$  150 °C  $\xrightarrow{18\text{ °C/min}}$  300 °C (20 min)

Detection: FID 280 °C

**Peaks:**

- |                       |                                       |
|-----------------------|---------------------------------------|
| 1. Acetone            | 11. Decane                            |
| 2. Ethyl acetate      | 12. Octanol-1                         |
| 3. Tetrahydrofuran    | 13. Acetophenone                      |
| 4. Cyclohexane        | 14. Butyrophenone                     |
| 5. 2-Methylbutanol-2  | 15. Heptanophenone                    |
| 6. Butanol-1          | 16. Methoxy-5-indole                  |
| 7. Pyridine           | 17. Dibenzylamine                     |
| 8. Toluene            | 18. Methyl eicosanoate                |
| 9. Dimethylformamide  | 19. Methyl <i>cis</i> -13-docosenoate |
| 10. Dimethylsulfoxide | 20. Methyl docosanoate                |



# Solvents

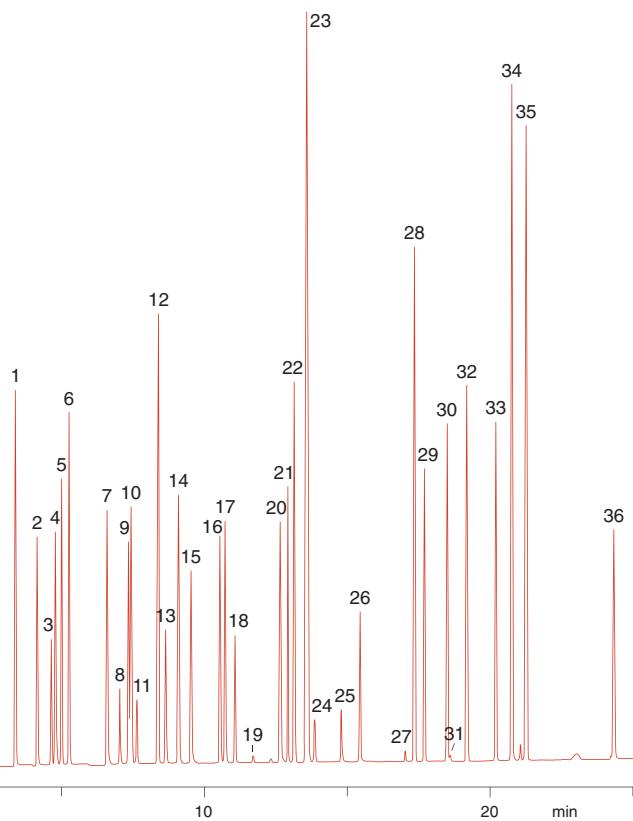


## Analysis of solvents MN Appl. No. 201410

Column: OPTIMA® 1701, 50 m x 0.32 mm ID, 1.0  $\mu\text{m}$  film, REF 726929.50,  
max. temperature 300/320 °C  
Injection: 0.1  $\mu\text{l}$ , split 147 ml/min  
Carrier gas: 150 kPa He (3 ml/min)  
Temperature: 50 °C (5 min)  $\xrightarrow{5\text{ °C/min}}$  160 °C  
Detector: FID 240 °C

**Peaks:**

1. Methanol
2. Ethanol
3. Acetone
4. Propanol-2  
+ methyl acetate
5. Dichloromethane
6. *t*-Butanol
7. Propanol-1
8. Ethyl acetate
9. Butanone (MEK)
10. Trichloroethane
11. Trichloromethane
12. Benzene
13. *i*-Propyl acetate
14. *i*-Butanol
15. Methyl glycol
16. 1-Methoxypropanol-2
17. Butanol-1
18. *n*-Propyl acetate
19. Glycolic acid
20. Ethyl glycol
21. Toluene
22. *i*-Butyl methyl ketone
23. *i*-Butyl acetate
24. Tetrachloroethene
25. Ethoxypropanol
26. *n*-Butyl acetate
27. Ethylbenzene
28. *m*-Xylene + *p*-xylene
29. Methyl glycol acetate
30. 1-Methoxypropyl acetate-2
31. *o*-Xylene
32. 2-Methoxypropyl acetate-1
33. Ethyl glycol acetate
34. Butyl glycol  
+ ethoxypropyl acetate
35. Cyclohexanone
36. Glycolic acid *n*-butyl ester



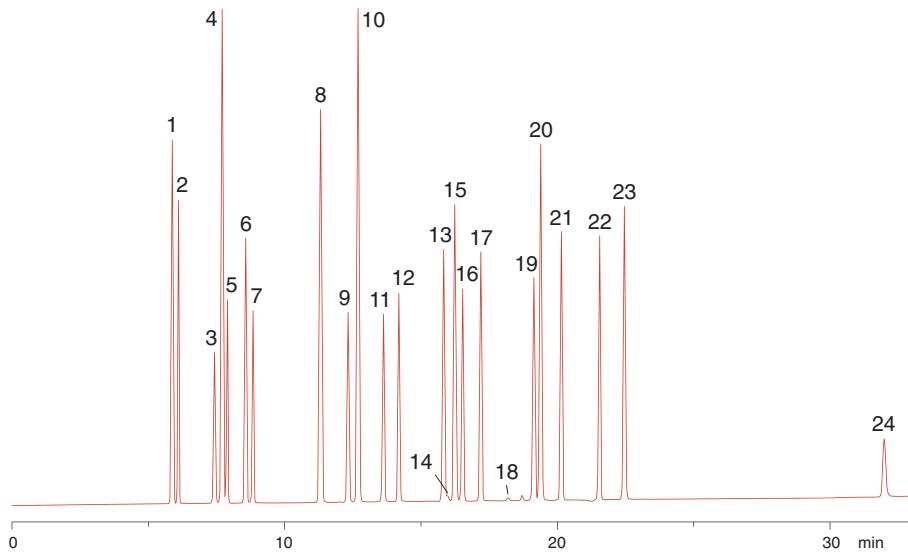
## Solvents • Chemicals

### Analysis of solvents MN Appl. No. 201420

Column: PERMABOND® CW 20 M, 50 m x 0.32 mm ID, 0.5 µm film, REF 723296.50,  
max. temperature 220/240 °C  
Injection: 0.2 µl, split 90 ml/min  
Carrier gas: 1 bar N<sub>2</sub>  
Temperature: 60 °C (5 min)  $\xrightarrow{5\text{ °C/min}}$  210 °C  
Detector: FID 250 °C

**Peaks:**

- |                                 |                                  |
|---------------------------------|----------------------------------|
| 1. Acetone                      | 13. 1-Methoxypropanol-2          |
| 2. Methyl acetate               | 14. Ethylbenzene                 |
| 3. Ethyl acetate                | 15. Butanol-1 + <i>p</i> -xylene |
| 4. Methanol + isopropyl acetate | 16. <i>m</i> -Xylene             |
| 5. Butanone (MEK)               | 17. Ethoxypropanol               |
| 6. Propanol-2                   | 18. <i>o</i> -Xylene             |
| 7. Ethanol                      | 19. 1-Methoxypropyl acetate-2    |
| 8. Isobutyl methyl ketone       | 20. Ethyl glycol                 |
| 9. Propanol-1                   | 21. 2-Methoxypropyl acetate-1    |
| 10. Toluene                     | 22. Ethyl glycol acetate         |
| 11. <i>n</i> -Butyl acetate     | 23. Cyclohexanone                |
| 12. <i>i</i> -Butanol           | 24. Ethylene glycol              |



# Solvents



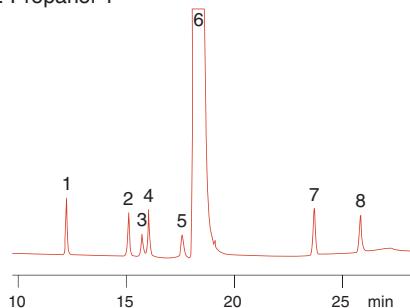
## Analysis of solvent residues in ethanolic tinctures

MN Appl. No. 210880

Column: PERMABOND® CW 20 M,  
60 m x 0.32 mm ID,  
0.5 µm film, REF 723296.60,  
max. temperature 220/240 °C  
Injection: 1 µl, split 50 ml/min  
Carrier gas: 50 kPa N<sub>2</sub>  
Temperature: 60 °C (19 min)  $\xrightarrow{5\text{ °C/min}}$  100 °C  
Detector: FID 250 °C

**Peaks:**

1. Acetone
2. Ethyl acetate
3. Methanol
4. Methyl ethyl ketone
5. Propanol-2
6. Ethanol
7. Isobutyl methyl ketone
8. Propanol-1



Courtesy of Mr. Fischer, Pharmaz. Fabrik Evers & Co. GmbH, Hamburg, Germany

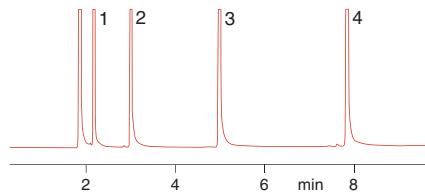
## Analysis of glycols

MN Appl. No. 210950

Column: OPTIMA® δ-3,  
30 m x 0.25 mm ID,  
0.25 µm film, REF 726420.30,  
max. temperature 340/360 °C  
Injection: 1 µl, split 100 ml/min  
Carrier gas: 70 kPa He  
Temperature: 150 °C  $\xrightarrow{10\text{ °C/min}}$  250 °C  
(15 min)  
Detector: FID

**Peaks:**

1. Diethylene glycol
2. Triethylene glycol
3. Tetraethylene glycol
4. Pentaethylene glycol

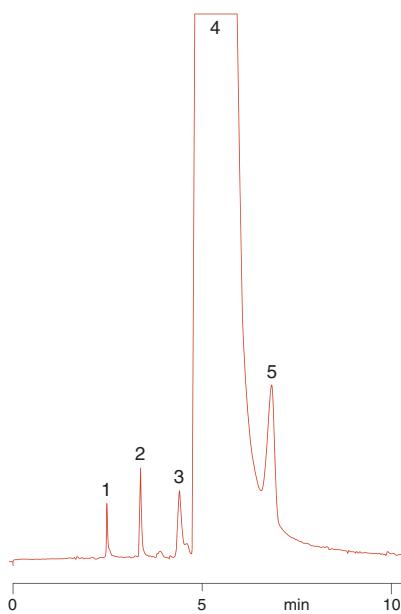


## Solvents • Chemicals

### Analysis of alcohols in dichloromethane *MN Appl. No. 201460*

Column: OPTIMA® 5,  
25 m x 0.32 mm ID,  
5.0  $\mu$ m film,  
REF 726934.25,  
max. temperature 300/320 °C  
Injection: 2  $\mu$ l, split 30 ml/min  
Carrier gas: 1 bar N<sub>2</sub>  
Temperature: 60 °C  
Detector: FID 240 °C

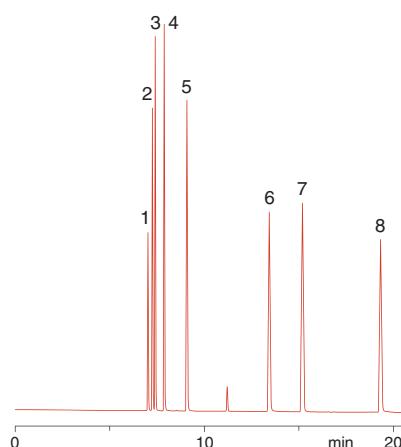
**Peaks:**  
1. Methanol  
2. Ethanol  
3. Propanol-2  
4. Dichloromethane  
5. Propanol-1



### Analysis of alcohols *MN Appl. No. 201430*

Column: OPTIMA® 17,  
50 m x 0.32 mm ID,  
0.5  $\mu$ m film, REF 723744.50,  
max. temperature 320/340 °C  
Injection: 1  $\mu$ l, split 1:50  
Carrier gas: 50 kPa N<sub>2</sub>  
Temperature: 50 °C  $\xrightarrow{4\text{ °C/min}}$  200 °C  
Detector: FID 250 °C

**Peaks:**  
1. Methanol  
2. Ethanol  
3. Propanol-2  
4. Propanol-1  
5. Butanol-1  
6. Hexanol-1  
7. Cyclohexanol  
8. Octanol-1



# Alcohols



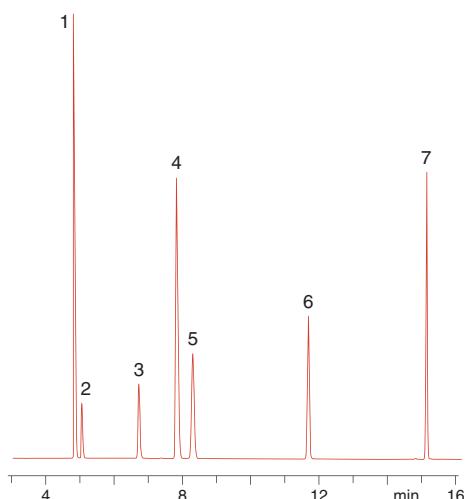
## Headspace analysis of alcohols

MN Appl. No. 212630

Column: OPTIMA® 624,  
60 m x 0.32 mm ID,  
1.80 µm film, REF 726787.60,  
max. temperature 280/300 °C  
Injection: headspace 200 °C, sample  
temperature 60 °C for 20 min,  
needle temperature 70 °C,  
transfer line 80 °C  
Carrier gas: 130 kPa He    20 °C/min  
Temperature: 40 °C (5 min) → 200 °C  
Detector: FID 200 °C

### Peaks:

1. Acetaldehyde
2. Methanol
3. Ethanol
4. Acetone
5. Propanol-2
6. Propanol-1
7. Butanol-1



Courtesy of Mr. Kress, INSTAND e.V., Düsseldorf, Germany

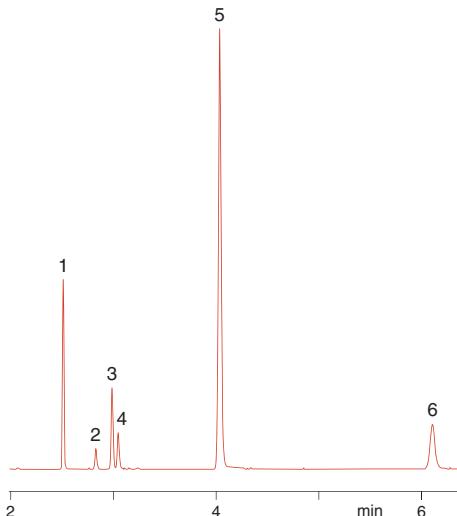
## Headspace analysis of alcohols

MN Appl. No. 212940

Column: OPTIMA® WAX,  
30 m x 0.25 mm ID,  
0.25 µm film, REF 726600.30,  
max. temperature 250/260 °C  
Sample: headspace: heat 1 ml water +  
20 µl of each alcohol to 80 °C  
in a headspace vial for 15 min  
Injection: 250 °C, split 30:1  
Carrier gas: 11.78 psi He, 1 ml/min  
Temperature: 70 °C (10 min)  
Detector: MSD

### Peaks:

1. Acetone
2. Methanol
3. Propanol-2
4. Ethanol
5. Propanol-1
6. Butanol-1



Courtesy of V. Cirimele, Laboratoire ChemTox, Illkirch Graffenstaden, France

## Solvents • Chemicals

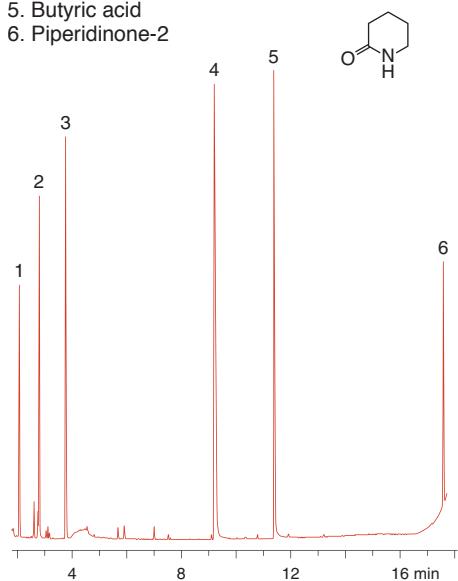
### Headspace analysis of alcohols

MN Appl. No. 212950

Column: OPTIMA® WAX,  
30 m x 0.25 mm ID,  
0.25 µm film, REF 726600.30,  
max. temperature 250/260 °C  
Injection: headspace: heat 1 ml blood  
+ 20 µl int. std. (50 mg/l  
propanol-1) to 80 °C in a  
headspace vial for 15 min;  
240 °C, split 3.64:1  
Carrier gas: 7.35 psi He, 1 ml/min  
Temperature: 45 °C (1 min)  $\xrightarrow{10\text{ °C/min}}$  180 °C  
(2 min)  $\xrightarrow{30\text{ °C/min}}$  240 °C (3 min)  
Detector: MSD

**Peaks:**

1. Acetone
2. Ethanol
3. Propanol-1
4. Acetic acid
5. Butyric acid
6. Piperidinone-2



Courtesy of V. Cirimele, Laboratoire ChemTox,  
Illkirch Graffenstaden, France

# Neutral and basic organics



## Analysis of a mixture of neutral and basic organic compounds

Sample: Base/Neutrals Mix 1, 200 µg/ml each in 2-propanol

Injection: 1.0 µl, split 1:150

Carrier gas: 25 cm/s He

Detector: MSD

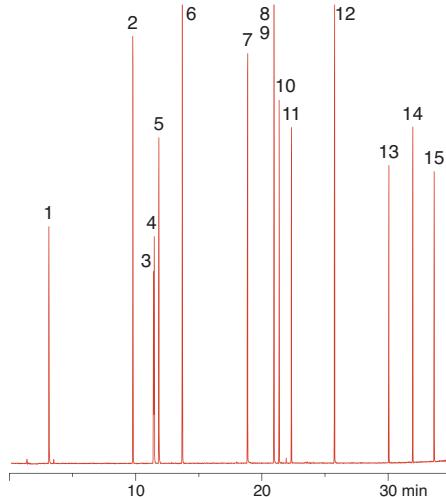
### Peaks:

- |                                 |                                 |
|---------------------------------|---------------------------------|
| 1. N-Nitrosodimethylamine       | 9. Diethyl phthalate            |
| 2. s-Dichloroethyl ether        | 10. N-Nitrosodiphenylamine      |
| 3. Bis(1-chloroisopropyl) ether | 11. 4-Bromophenoxybenzene       |
| 4. Bis(2-chloroisopropyl) ether | 12. Dibutyl phthalate           |
| 5. N-Nitroso-di-n-propylamine   | 13. Benzyl butyl phthalate      |
| 6. Bis(2-chloroethoxy)methane   | 14. Bis(2-ethylhexyl) phthalate |
| 7. Dimethyl phthalate           | 15. Di-n-octyl phthalate        |
| 8. 4-Chlorophenoxybenzene       |                                 |

### MN Appl. No. 201170

Column: OPTIMA® 5,  
25 m x 0.20 mm ID,  
0.20 µm film, REF 726857.25,  
max. temperature: 340/360 °C

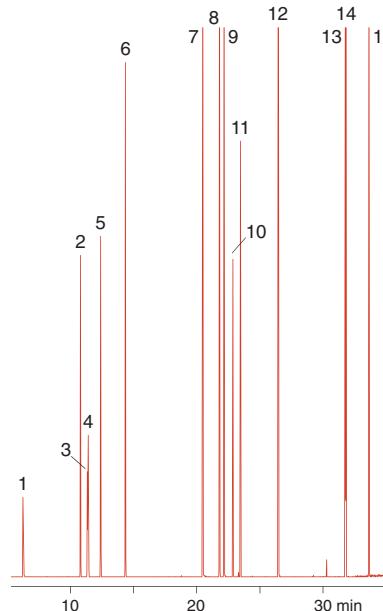
Temperature: 45 °C (5 min)  $\xrightarrow{8\text{ °C/min}}$  330 °C



### MN Appl. No. 201180

Column: OPTIMA® 17,  
25 m x 0.20 mm ID,  
0.20 µm film, REF 726065.25,  
max. temperature 320/340 °C

Temperature: 50 °C (3 min)  $\xrightarrow{8\text{ °C/min}}$  320 °C



## Solvents • Chemicals

### Analysis of organic pollutants from water-packaging materials

MN Appl. No. 212870

Column: OPTIMA® 17,  
15 m x 0.53 mm ID,  
1.0  $\mu$ m film, REF 726747.15  
max. temperature 300/320 °C

Injection: splitless, 270 °C

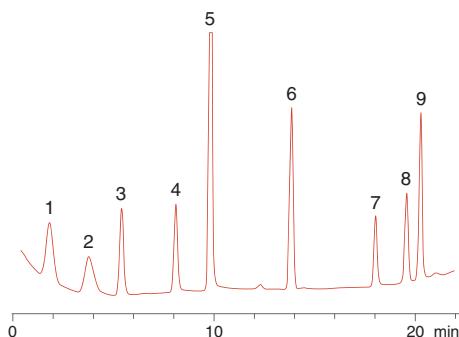
Carrier gas: 10 ml/min N<sub>2</sub> 10 °C/min

Temperature: 50 °C (1 min) → 270 °C  
(10 min)

Detector: FID 300 °C

**Peaks:**

1. Chlorobenzene
2. Isopentyl acetate
3. Styrene
4. Phenol
5. Benzyl alcohol
6. Cyclohexylbenzene
7. Benzophenone
8. Diazinon
9. Dibutyl phthalate



S. Guillot, M. T. Kelly, H. Fenet, M. Larroque,  
J. Chromatogr. A 1101 (2006) 46 – 52

### Analysis of 2,4-dinitrophenyl-hydrzones

MN Appl. No. 201250

Column: OPTIMA® 1,  
10 m x 0.53 mm ID,  
0.50  $\mu$ m film,  
REF 726519.10,  
max. temperature 320/340 °C

Injection: 1  $\mu$ l, split 1:50

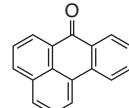
Carrier gas: 0.3 bar H<sub>2</sub>

Temperature: 60 °C → 160 °C 8 °C/min  
300 °C

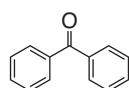
Detector: FID 320 °C

**Peaks:** DNP hydrazones of

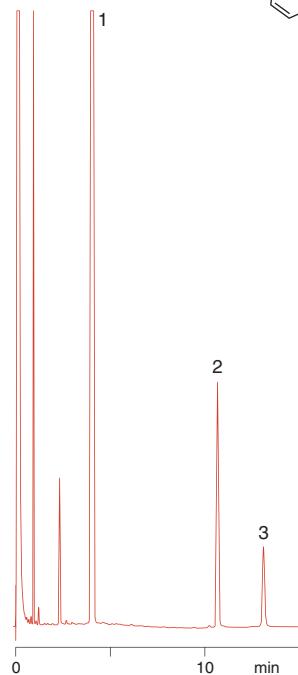
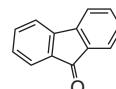
1. Benzanthrone



2. Benzophenone



3. Fluorenone



## Miscellaneous



### Analysis of plasticizers MN Appl. No. 201200

Column: OPTIMA® 5, 50 m x 0.25 mm ID, 0.25 µm film, REF 726056.50,  
max. temperature 340/360 °C

Injection: 1 µl on column, 20 ng each

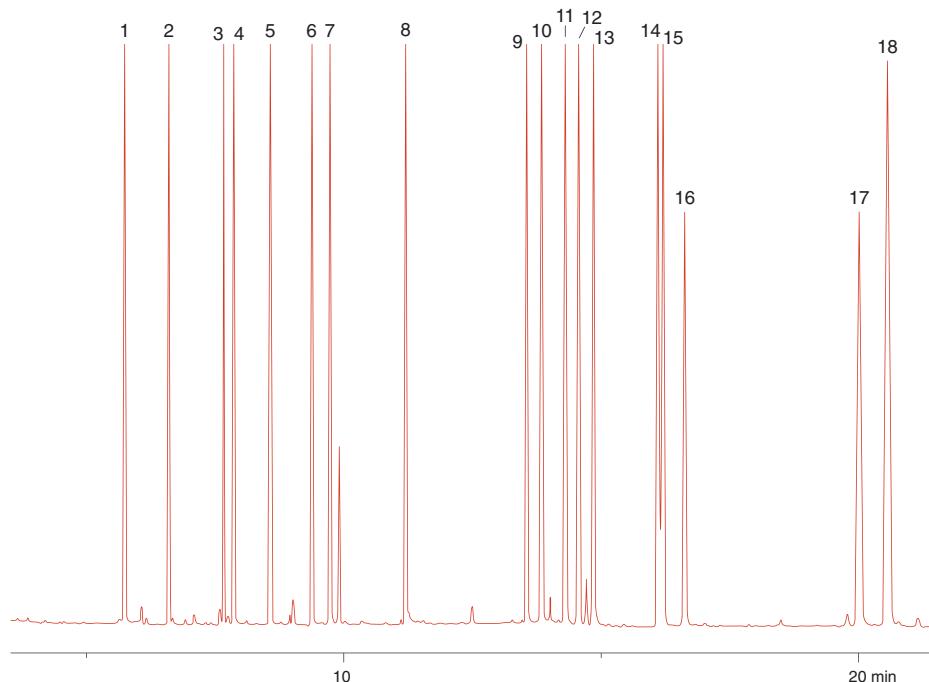
Carrier gas: 140 kPa H<sub>2</sub>

Temperature: 80  $\xrightarrow{40\text{ °C/min}}$  240 °C  $\xrightarrow{6\text{ °C/min}}$  280 °C

Detector: FID

#### Peaks:

- |                            |                                       |
|----------------------------|---------------------------------------|
| 1. Dimethyl phthalate      | 10. Di(2-ethylhexyl) adipate          |
| 2. Diethyl phthalate       | 11. Triphenyl phosphate               |
| 3. Diallyl phthalate       | 12. Diphenyl-(2-ethylhexyl) phosphate |
| 4. Dipropyl phthalate      | 13. Tri(2-ethylhexyl) phosphate       |
| 5. Diisobutyl phthalate    | 14. Dicyclohexyl phthalate            |
| 6. Di-n-butyl phthalate    | 15. Di(2-ethylhexyl) phthalate        |
| 7. Methyl glycol phthalate | 16. Diphenyl phthalate                |
| 8. Dibutyl sebacate        | 17. Di-n-octyl phthalate              |
| 9. Butyl benzyl phthalate  | 18. Tri-p-cresyl phosphate            |



Courtesy of Mr. Lembacher, Hipp Nährmittel, Pfaffenhofen, Germany

## Solvents • Chemicals

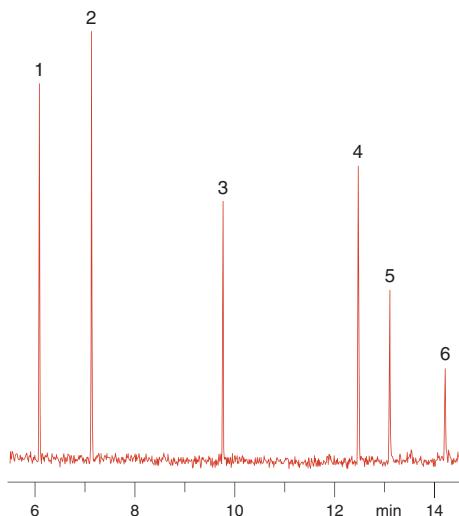
### Analysis of phthalates (EPA 606)

MN Appl. No. 213160

Column: OPTIMA® δ-3,  
30 m x 0.25 mm ID,  
0.25 µm film, REF 726420.30,  
max. temperature 340/360 °C  
Injection: 1 µl, split 1:10  
Carrier gas: 1.4 ml/min H<sub>2</sub>  
Temperature: 100 °C → 320 °C  
Detector: MSD

**Peaks:**

1. Dimethyl phthalate
2. Diethyl phthalate
3. Dibutyl phthalate
4. Butyl benzyl phthalate
5. Di(2-ethylhexyl) phthalate
6. Di-n-octyl phthalate



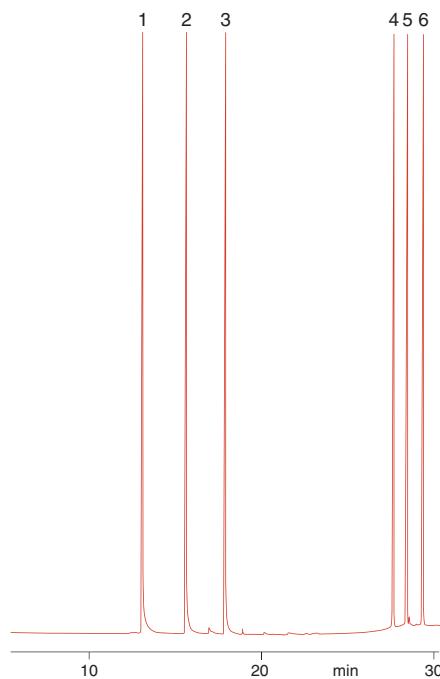
### Analysis of phosphonic acid diethyl esters

MN Appl. No. 211860

Column: OPTIMA® δ-3,  
30 m x 0.25 mm ID,  
0.25 µm film, REF 726420.30,  
max. temperature 340/350 °C  
Injection: 0.5 µl, 250 °C, split 40 ml/min  
Carrier gas: 80 kPa He  
Temperature: 60 °C → 150 °C → 300 °C  
Detector: FID 300 °C

**Peaks:**

1. Diethyl methylphosphonate
2. Diethyl ethylphosphonate
3. Diethyl allylphosphonate
4. Tetraethyl-ethylene-1,2-diphosphonate
5. Tetraethyl-propylene-1,3-diphosphonate
6. Tetraethyl-butylene-1,4-diphosphonate



VeZerf Laborsynthesen GmbH, Idar-Oberstein,  
Germany



## Analysis of halogeno- and phosphororganic compounds MN Appl. No. 210340

Column: OPTIMA® 5, 30 m x 0.25 mm ID, 0.25 µm film, REF 726056.30,  
max. temperature 340/360 °C

Injection: 1.0 µl, splitless

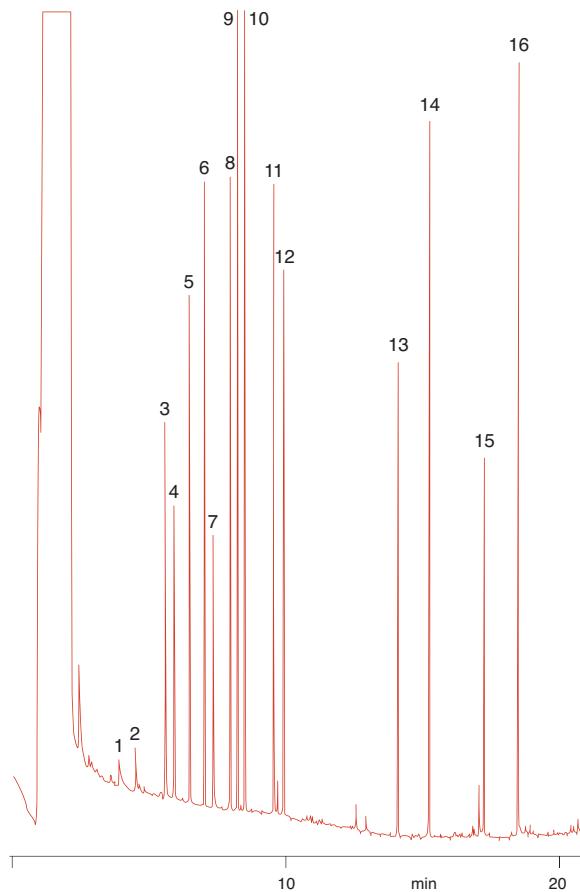
Carrier gas: 1.5 bar H<sub>2</sub>

Temperature: 39 °C (1 min)  $\xrightarrow{10\text{ °C/min}}$  120 °C  $\xrightarrow{12\text{ °C/min}}$  280 °C (10 min)

Detector: MSD

**Peaks:**

1. Dimethyl methylphosphonate
2. Trimethyl phosphate
3. 4-Fluorophenol
4. 1,2-Dichlorobenzene
5. 1-Octanol
6. 2,6-Dimethylphenol
7. Triethyl phosphate
8. 2,6-Dimethylbenzylamine
9. Naphthalene
10. *n*-Dodecane
11. 1-Dodecanol
12. 5-Chloro-2-methylbenzylamine
13. Tributyl phosphate
14. Dibenzothiophene
15. Malathion
16. Methyl octadecanoate



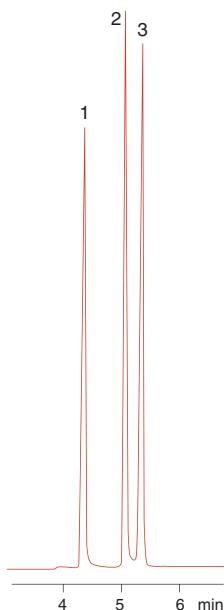
Courtesy of Mr. Kremer, Wehrwissenschaftl. Inst. für Schutztechnologien, Munster, Germany

## Solvents • Chemicals

### Analysis of methylamines

MN Appl. No. 210490

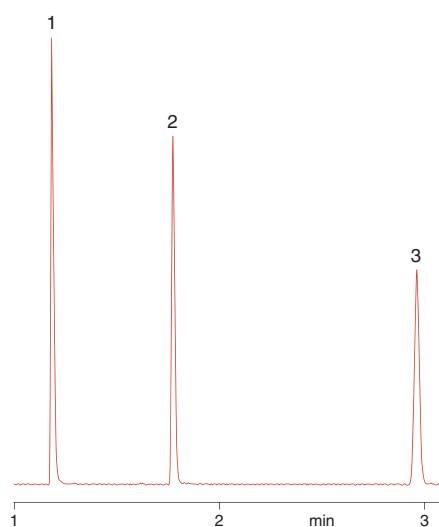
Column: OPTIMA® 5,  
50 m x 0.32 mm ID,  
5.0  $\mu\text{m}$  film, REF 726934.50,  
max. temperature 300/320 °C  
Split: 108 ml/min  
Carrier gas: 2.2 ml/min He  
Temperature: 85 °C  
Detector: FID  
**Peaks:** 20 ng/peak  
1. Methylamine  
2. Dimethylamine  
3. Trimethylamine



### Analysis of ethylamines

MN Appl. No. 250050

Column: OPTIMA® 5 Amine,  
30 m x 0.32 mm ID,  
1.0  $\mu\text{m}$  film, REF 726353.30,  
max. temperature 300/320 °C  
Injection: 1  $\mu\text{l}$ , split 1:70  
Carrier gas: 0.5 bar H<sub>2</sub>  
Temperature: 40 °C  $\xrightarrow{5\text{ °C/min}}$  100 °C  
Detector: FID  
**Peaks:**  
1. Ethylamine  
2. Diethylamine  
3. Triethylamine



# Amines

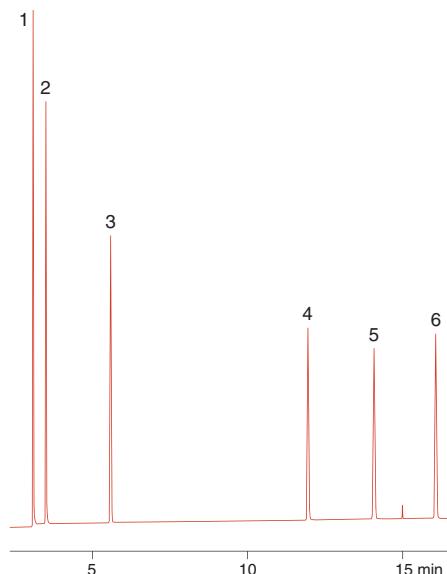


## Analysis of amines MN Appl. No. 201520

Column: FS-CW 20 M-AM,  
25 m x 0.25 mm ID,  
0.25  $\mu\text{m}$  film, REF 733110.25,  
max. temperature 220/240 °C  
Injection: 0.2  $\mu\text{l}$ , split 1:100  
Carrier gas: 0.45 bar N<sub>2</sub> 8 °C/min  
Temperature: 80 °C (5 min)  $\xrightarrow{8\text{ °C/min}}$  180 °C  
Detector: FID 250 °C

**Peaks:**

1. Butylamine
2. Di-*i*-propylamine
3. Dibutylamine
4. Nonylamine
5. Decylamine
6. Undecylamine

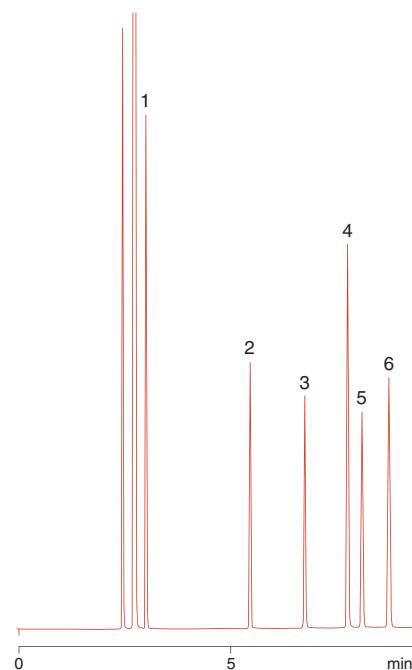


## Analysis of amines MN Appl. No. 201530

Column: FS-CW 20 M-AM,  
25 m x 0.32 mm ID,  
0.25  $\mu\text{m}$  film, REF 733299.25,  
max. temperature 220/240 °C  
Injection: 0.5  $\mu\text{l}$   
Carrier gas: 0.5 bar N<sub>2</sub>  
Temperature: 80 °C  $\xrightarrow{10\text{ °C/min}}$  190 °C  
Detector: FID 240 °C

**Peaks:**

1. Dibutylamine
2. *n*-Nonylamine
3. *n*-Decylamine
4. Benzylamine
5. *n*-Undecylamine
6. Dicyclohexylamine



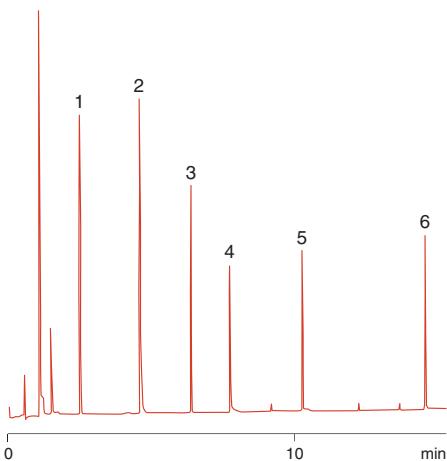
## Solvents • Chemicals

### Separation of the OPTIMA® Amine test mixture (REF 722317) MN Appl. No. 250020

Column: OPTIMA® 5 Amine,  
30 m x 0.32 mm ID,  
1.0  $\mu$ m film, REF 726353.30,  
max. temperature 300/320 °C  
Injection: 1  $\mu$ l, split 1:40  
Carrier gas: 0.6 bar H<sub>2</sub>  
Temperature: 100 °C  $\xrightarrow{10\text{ °C/min}}$  280 °C  
Detector: FID

**Peaks:**

1. Di-*i*-butylamine
2. Diethanolamine
3. 2,6-Dimethylaniline
4. *o*-Propanol-pyridine
5. Dicyclohexylamine
6. Dibenzylamine

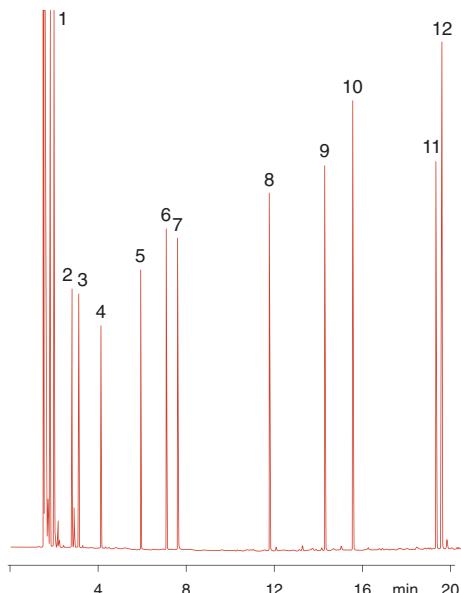


### Analysis of secondary and tertiary amines MN Appl. No. 210280

Column: OPTIMA® 5 Amine,  
30 m x 0.25 mm ID,  
1.0  $\mu$ m film, REF 726358.30,  
max. temperature 300/320 °C  
Injection: 1.0  $\mu$ l, 0.01 % in pentane  
split 1:100  
Carrier gas: 0.55 bar H<sub>2</sub>  
Temperature: 50 °C (3 min)  $\xrightarrow{10\text{ °C/min}}$  280 °C  
(10 min)  
Detector: FID 280 °C

**Peaks:**

1. Diethylamine
2. Di-*i*-propylamine
3. Triethylamine
4. Di-*n*-propylamine
5. Di-*n*-butylamine
6. Tri-*n*-propylamine
7. Di-*i*-butylamine
8. Tri-*n*-butylamine
9. Di-*n*-hexylamine
10. Dicyclohexylamine
11. Dibenzylamine
12. Tri-*n*-hexylamine

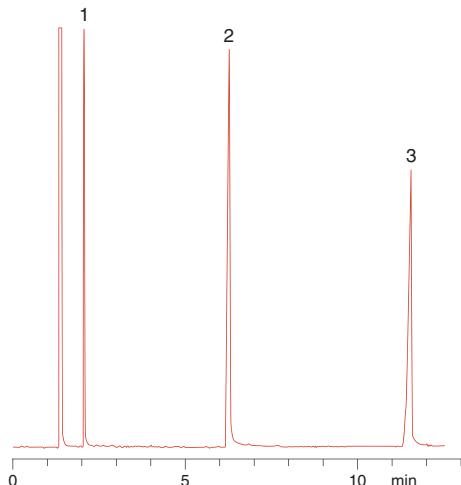


# Amines



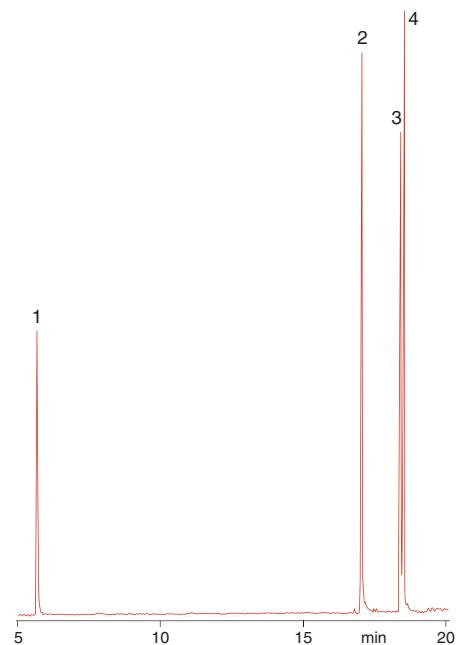
## Analysis of ethanolamines MN Appl. No. 250040

Column: OPTIMA® 5 Amine,  
30 m x 0.32 mm ID,  
1.0  $\mu$ m film, REF 726353.30,  
max. temperature 300/320 °C  
Injection: 1  $\mu$ l, 20 % in methanol  
split 1:30  
Carrier gas: 0.6 bar H<sub>2</sub>  
Temperature: 80 °C  $\xrightarrow{10\text{ °C/min}}$  200 °C  
Detector: FID  
**Peaks:**  
1. Ethanolamine  
2. Diethanolamine  
3. Triethanolamine



## Analysis of pyridine compounds MN Appl. No. 210360

Column: OPTIMA® 5 Amine,  
25 m x 0.20 mm ID,  
0.35  $\mu$ m film, REF 726355.25,  
max. temperature 300/320 °C  
Injection: 310 °C  
Carrier gas: 25 cm/s He  $\xrightarrow{8\text{ °C/min}}$  290 °C  
Temperature: 50 °C (3 min)  $\xrightarrow{8\text{ °C/min}}$  290 °C  
Detector: MSD  
**Peaks:**  
1. Pyridine  
2. *o*-Propanolpyridine  
3. *m*-Propanolpyridine  
4. *p*-Propanolpyridine



## Solvents • Chemicals

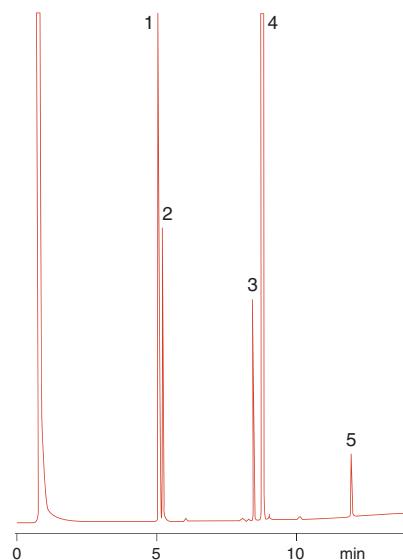
### Analysis of an oxazole derivative of N,N-dimethylformamide

**MN Appl. No. 210520**

Column: OPTIMA® 240,  
25 m x 0.25 mm ID,  
0.25 µm film, REF 726089.25,  
max. temperature 260/280 °C  
Injection: 0.5 µl, split 100 ml/min  
Carrier gas: 0.8 bar He  
Temperature: 50 °C  $\xrightarrow{7\text{ °C/min}}$  200 °C  
Detector: FID 280 °C

**Peaks:**

1. N,N-Dimethylformamide
2. 5-Cyano-4-methyloxazole
3. Methyl decanoate
4. N-Methylpyrrolidone
5. 4-Methyl-5-oxazole-carboxylic acid amide



Courtesy of Hoffmann-La Roche AG, Basel, Switzerland

N-Methylpyrrolidone      Oxazole



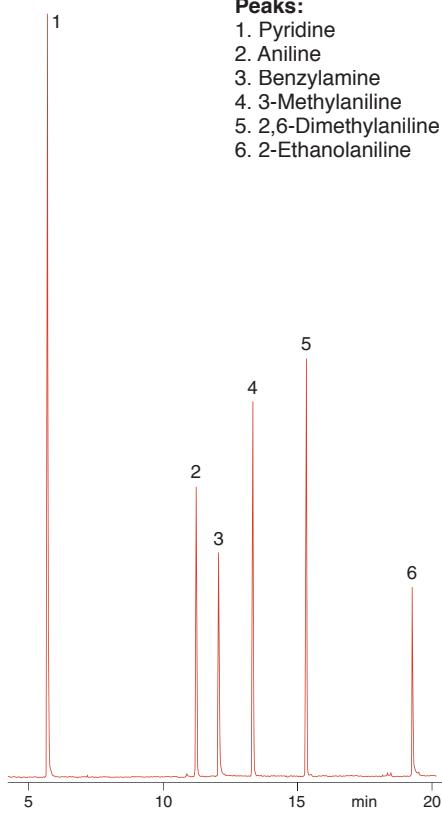
### Analysis of aniline derivatives

**MN Appl. No. 210380**

Column: OPTIMA® 5 Amine,  
25 m x 0.20 mm ID,  
0.35 µm film, REF 726355.25,  
max. temperature 300/320 °C  
Injection: 310 °C  
Carriergas: 25 cm/s He  
Temperature: 50 °C (3 min)  $\xrightarrow{8\text{ °C/min}}$  290 °C  
Detector: MSD

**Peaks:**

1. Pyridine
2. Aniline
3. Benzylamine
4. 3-Methylaniline
5. 2,6-Dimethylaniline
6. 2-Ethanolaniline



# Aromatic amines



## Analysis of substituted anilines MN Appl. No. 201560

Column: FS-CW 20 M-AM, 25 m x 0.25 mm ID, 0.25 µm film, REF 733110.25,  
max. temperature 220/240 °C

Injection: 2 µl, split 1:20

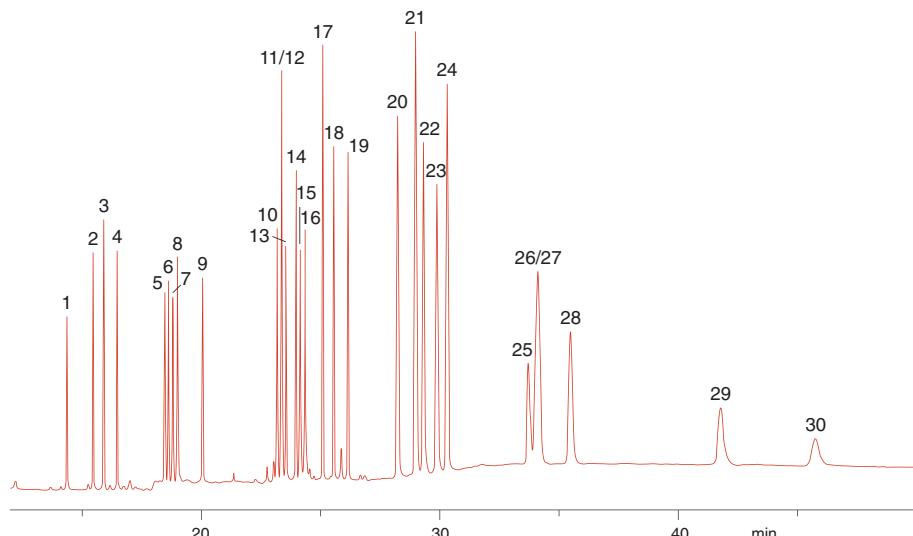
Carrier gas: 80 kPa He

Temperature: 80 °C (5 min)  $\xrightarrow{5\text{ °C/min}}$  210 °C (10 min)

Detector: MSD

### Peaks:

- |                                  |                              |                                 |
|----------------------------------|------------------------------|---------------------------------|
| 1. Aniline                       | 11. 2,4,6-Trichloroaniline   | 21. 3,4-Dichloroaniline         |
| 2. <i>o</i> -Tolidine            | 12. <i>m</i> -Chloroaniline  | 22. 2,4,5-Trichloroaniline      |
| 3. <i>p</i> -Tolidine            | 13. 3-Chloro-2-methylaniline | 23. 1-Naphthylamine (int. std.) |
| 4. <i>m</i> -Tolidine            | 14. 4-Chloro-2-methylaniline | 24. 2,3,4-Trichloroaniline      |
| 5. <i>o</i> -Chloroaniline       | 15. 5-Chloro-2-methylaniline | 25. 5-Chloro-2-nitroaniline     |
| 6. 2-Chloro-6-methylaniline      | 16. <i>m</i> -Phenetidine    | 26. 2-Chloro-5-nitroaniline     |
| 7. 2,6-Dichloroaniline           | 17. 2,4-Dichloroaniline      | 27. 4-Chloro-2-nitroaniline     |
| 8. 4-i-Propylaniline (int. std.) | 18. 2,5-Dichloroaniline      | 28. 3,4,5-Trichloroaniline      |
| 9. 2-Chloro-4-methylaniline      | 19. 2,3-Dichloroaniline      | 29. 2-Chloro-4-nitroaniline     |
| 10. <i>p</i> -Chloroaniline      | 20. 3,5-Dichloroaniline      | 30. 4-Chloro-3-nitroaniline     |



B. Scholz, N. Palauschek, Z. Anal. Chem. 331 (1988) 282 – 289

## Solvents • Chemicals

### Analysis of aromatic amines

MN Appl. No. 213230

Column: OPTIMA® 35 MS, 30 m x 0.25 mm ID, 0.25 µm film, REF 726154.30,  
max. temperature 360/370 °C

Injection: 1 µl, split 1:20

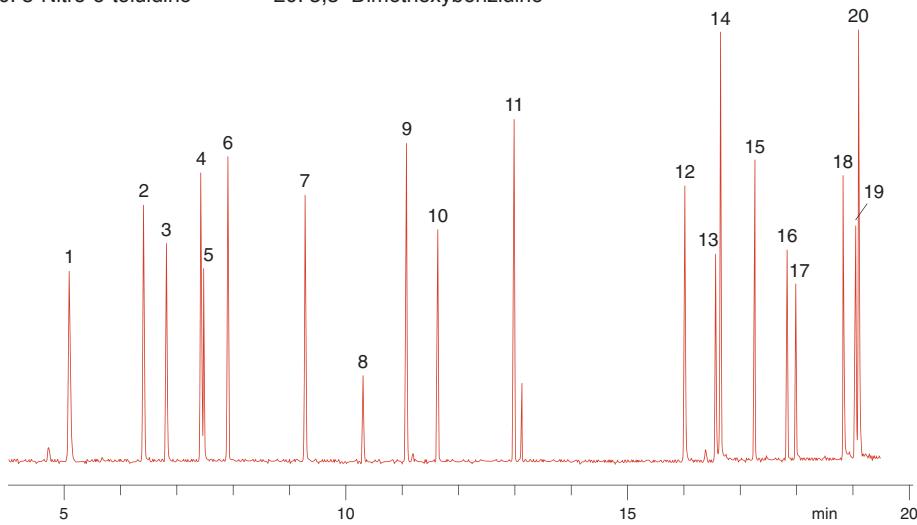
Carrier gas: 1.0 ml/min He

Temperature: 80 °C  $\xrightarrow{10\text{ °C/min}}$  320 °C (10 min)

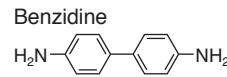
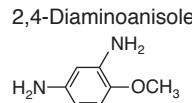
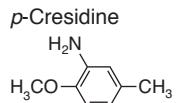
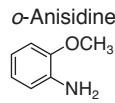
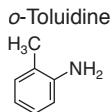
Detector: MSD

**Peaks:**

- |                           |                                                                   |
|---------------------------|-------------------------------------------------------------------|
| 1. o-Toluidine            | 11. 4-Aminobiphenyl                                               |
| 2. o-Anisidine            | 12. 4-Aminoazobenzene                                             |
| 3. 4-Chloroaniline        | 13. 4,4'-Oxydianiline                                             |
| 4. p-Cresidine            | 14. Benzidine + 4,4'-diaminodiphenylmethane                       |
| 5. 2,4,5-Trimethylaniline | 15. o-Aminoazotoluene + 3,3'-dimethyl-4,4'-diaminodiphenylmethane |
| 6. 4-Chloro-o-toluidine   | 16. 3,3'-Dimethylbenzidine                                        |
| 7. 2,4-Diaminotoluene     | 17. 4,4'-Thiodianiline                                            |
| 8. 2,4-Diaminoanisole     | 18. 3,3'-Dichlorobenzidine                                        |
| 9. 2-Naphthylamine        | 19. 4,4'-Methylene-bis-(2-chloroaniline)                          |
| 10. 5-Nitro-o-toluidine   | 20. 3,3'-Dimethoxybenzidine                                       |



### Structures of selected aromatic amines



# Aromatic amines



## Analysis of azo-dyes and aromatic amines by fast GC MN Appl. No. 210820

Column: OPTIMA® δ-3, 10 m x 0.1 mm ID, 0.1 µm film, REF 726410.10,  
max. temperature 340/360 °C

Injection: 1.0 µl, 250 °C

Carrier gas: 84 ml/min, splitless

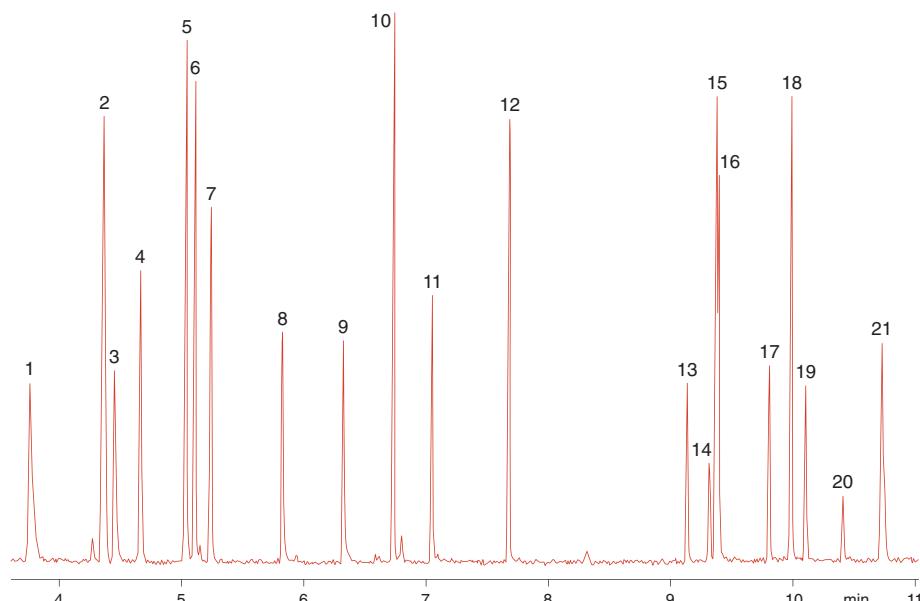
Pressure: 580 kPa (1 min)  $\xrightarrow{42 \text{ kPa/min}}$  966 kPa (5 min)

Temperature: 50 °C (1 min)  $\xrightarrow{25 \text{ °C/min}}$  280 °C (5 min)

Detector: MSD 320 °C

### Peaks:

- |                                  |                                               |
|----------------------------------|-----------------------------------------------|
| 1. <i>o</i> -Toluidine           | 13. 4-Aminoazobenzene                         |
| 2. 2,4- and 2,6-Dimethylaniline  | 14. 4,4'-Oxydianiline                         |
| 3. <i>o</i> -Anisidine           | 15. 4,4'-Diaminodiphenylmethane               |
| 4. 4-Chloroaniline               | 16. Benzidine                                 |
| 5. <i>p</i> -Cresidine           | 17. <i>o</i> -Aminoazotoluene                 |
| 6. 2,4,5-Trimethylaniline        | 18. 3,3'-Dimethyl-4,4'-diaminodiphenylmethane |
| 7. 4-Chloro- <i>o</i> -toluidine | 19. 3,3'-Dimethylbenzidine                    |
| 8. 2,4-Diaminotoluene            | 20. 4,4'-Thiodianiline                        |
| 9. 2,4-Diaminoanisole            | 21. 3,3'-Dimethoxybenzidine                   |
| 10. 2-Naphthylamine              | + 4,4'-methylene-bis-(2-chloroaniline)        |
| 11. 5-Nitro- <i>o</i> -toluidine | + 3,3'-dichlorobenzidine                      |
| 12. 4-Aminobiphenyl              |                                               |



Courtesy of Mrs. K. Friedrichs, Chem. Untersuchungsamt, Bielefeld, Germany

# Solvents • Chemicals

## Analysis of azo-dyes and aromatic amines MN Appl. No. 210170

Column: OPTIMA® 5 Amine, 30 m x 0.25 mm ID, 0.5 µm film, REF 726354.30,  
max. temperature 300/320 °C

Injection: 2.0 µl

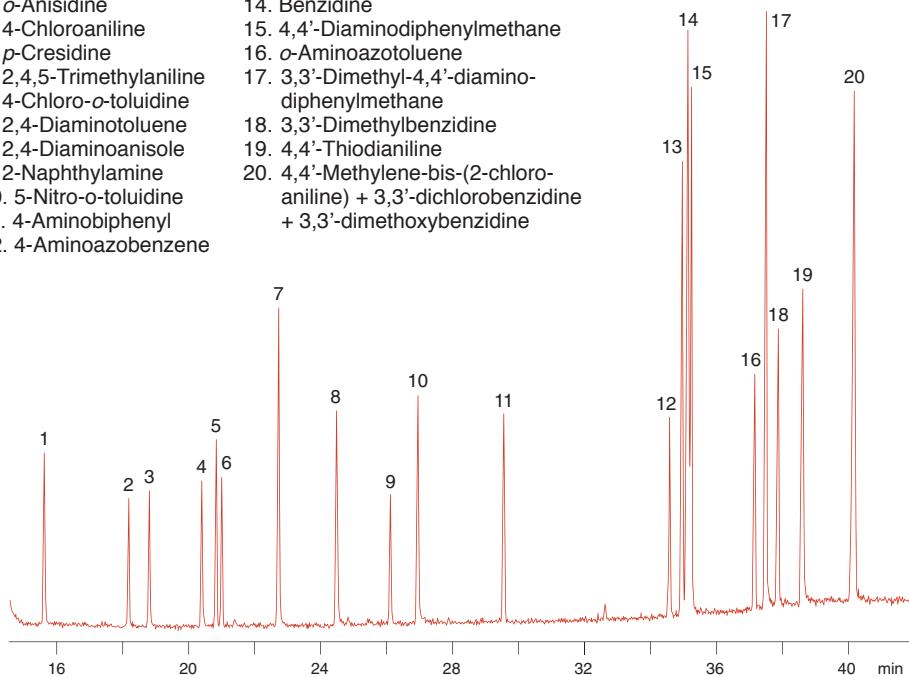
Carrier gas: He 7 °C/min

Temperature: 50 °C → 300 °C

Detector: MSD

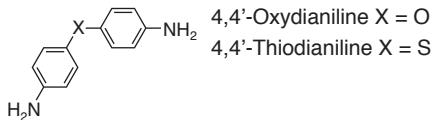
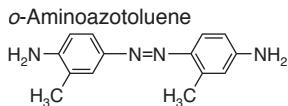
**Peaks:**

- |                           |                                                                                              |
|---------------------------|----------------------------------------------------------------------------------------------|
| 1. o-Toluidine            | 13. 4,4'-Oxydianiline                                                                        |
| 2. o-Anisidine            | 14. Benzidine                                                                                |
| 3. 4-Chloroaniline        | 15. 4,4'-Diaminodiphenylmethane                                                              |
| 4. p-Cresidine            | 16. o-Aminoazotoluene                                                                        |
| 5. 2,4,5-Trimethylaniline | 17. 3,3'-Dimethyl-4,4'-diamino-diphenylmethane                                               |
| 6. 4-Chloro-o-toluidine   | 18. 3,3'-Dimethylbenzidine                                                                   |
| 7. 2,4-Diaminotoluene     | 19. 4,4'-Thiodianiline                                                                       |
| 8. 2,4-Diaminoanisole     | 20. 4,4'-Methylene-bis-(2-chloro-aniline) + 3,3'-dichlorobenzidine + 3,3'-dimethoxybenzidine |
| 9. 2-Naphthylamine        |                                                                                              |
| 10. 5-Nitro-o-toluidine   |                                                                                              |
| 11. 4-Aminobiphenyl       |                                                                                              |
| 12. 4-Aminoazobenzene     |                                                                                              |



Courtesy of Mrs. Friedrichs, Chem. Untersuchungsamt Bielefeld, Germany

### Structures of selected aromatic amines



## Miscellaneous



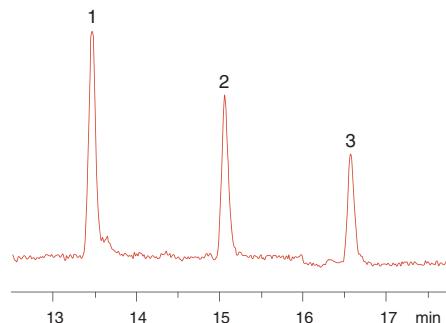
### Analysis of organotin compounds MN Appl. No. 210730

The sample is dissolved in *n*-hexane. After the Grignard reaction with pentylmagnesium bromide the residue is extracted with diethyl ether and dried with Na<sub>2</sub>SO<sub>4</sub>.

Column: OPTIMA® 5 MS,  
12 m x 0.20 mm ID,  
0.35 µm film, REF 726215.12,  
max. temperature 340/360 °C  
Septum purge: 4.5 ml/min  
Injection: 1 µl, 250 °C, splitless  
Carrier gas: 1 ml/min He (20 kPa)  
Temperature: 90 °C → 280 °C (3 min)  
Detector: MSD 280 °C, EI, SIM

**Peaks:**

1. Tetrabutyl tin (internal standard)
2. Tributyl pentyl tin
3. Dibutyl dipentyl tin



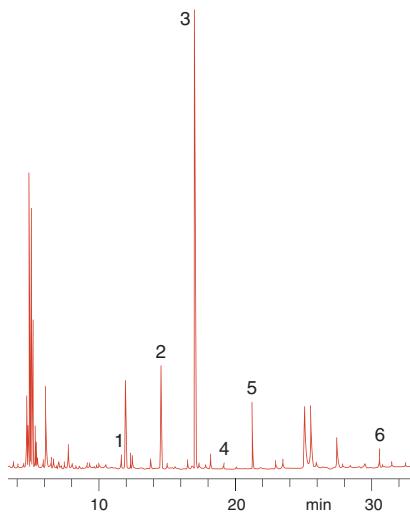
Courtesy of Mr. Wohlfarth, Staatl. Med. Leb-  
ensm., Vet. UA, Wiesbaden, Germany

### Qualitative determination of organotin compounds MN Appl. No. 212140

Column: OPTIMA® 1,  
25 m x 0.2 mm ID,  
0.35 µm film, REF 726837.25,  
max. temperature 340/360 °C  
Injection: 1 µl, split 15 ml/min  
Carrier gas: He  
Temperature: 50 °C → 300 °C  
Detector: MSD, transfer line 300 °C

**Peaks:**

1. BuSnEt<sub>3</sub>
2. Bu<sub>2</sub>SnEt<sub>2</sub>
3. Bu<sub>3</sub>SnEt
4. Bu<sub>4</sub>Sn
5. *i*-OcBu<sub>2</sub>SnEt
6. Oc<sub>3</sub>SnEt



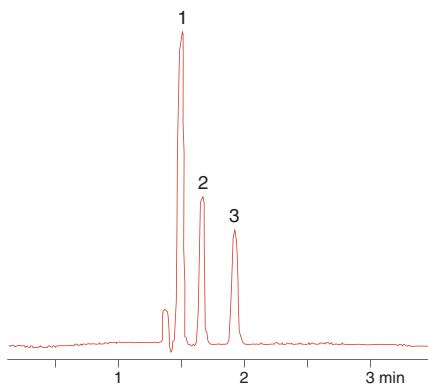
## Solvents • Chemicals

### Analysis of methyl silanes MN Appl. No. 200080

Column: PERMABOND® Silane,  
50 m x 0.32 mm ID,  
REF 723409.50,  
max. temperature 260/280 °C  
Injection: 1 ml gas  
Carrier gas: 60 kPa He  
Temperature: 35 °C  
Detector: MSD

**Peaks:**

1. Methylsilane
2. Dimethylsilane
3. Trimethylsilane



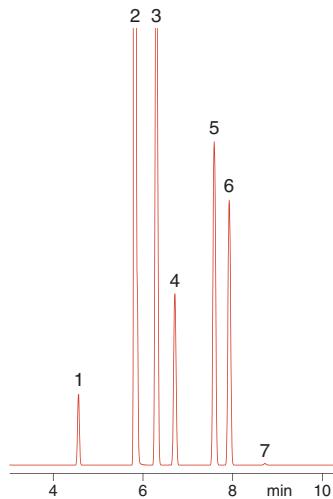
Courtesy of Th. Goldschmidt AG, Essen,  
Germany

### Analysis of chloromethyl silanes MN Appl. No. 200090

Column: PERMABOND® Silane,  
50 m x 0.32 mm ID,  
REF 723409.50,  
max. temperature 260/280 °C  
Injection: 0.5 µl, split 80 ml/min  
Carrier gas: 1 ml/min He (constant flow)  
Temperature: 50 °C (1 min)  $\xrightarrow{5\text{ °C/min}}$  100 °C  
Detector: MSD

**Peaks:**

1. Tetramethylsilane
2. Dichloromethane
3. Tetrachlorosilane
4. Chlorotrimethylsilane
5. Methyltrichlorosilane
6. Dichlorodimethylsilane
7. Hexamethyldisiloxane



# Silanes



## Analysis of silanes MN Appl. No. 200100

Column: PERMABOND® Silane, 50 m x 0.32 mm ID, REF 723409.50,

max. temperature 260/280 °C

Injection: 0.5 µl, split 80 ml/min

Carrier gas: 1.5 ml/min He (constant flow)

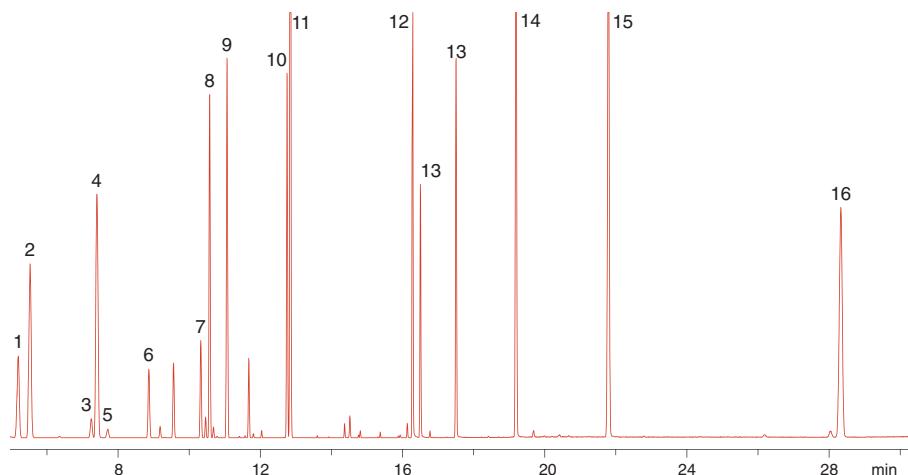
Temperature: 60 °C (6 min)  $\xrightarrow{20 \text{ °C/min}}$  250 °C (15 min)

Detector: MSD

### Peaks:

1. Trimethylmethoxysilane
2. Methylhydrodimethoxysilane
3. Vinyldimethylmethoxysilane
4. Dimethylidimethoxysilane
5. Hexamethylidisiloxane
6. 1,1,3,3-Tetramethylidisiloxane
7. Hexamethyldisilazane (HMDS)
8. Vinyltrimethoxysilane

9. 1,3-Divinyltetramethylidisiloxane
10. 1,3-Divinyltetramethyldisilazane
11. Octamethylcyclotetrasiloxane ( $D_4$ )
12. (Cyanopropyl)methyldimethoxysilane
13. (Phenylpropyl)methyldimethoxysilane
14. Diphenyldimethoxysilane
15. 1,3-Diphenyltetramethylidisilazane
16. 1,3-Dicyanopropyltetramethylidisiloxane



## Solvents • Chemicals

### Analysis of bifunctional alkyl nitrates MN Appl. No. 210290

Column: OPTIMA® 1701, 50 m x 0.32 mm ID, 0.25 µm film, REF 726318.50,  
max. temperature 300/320 °C

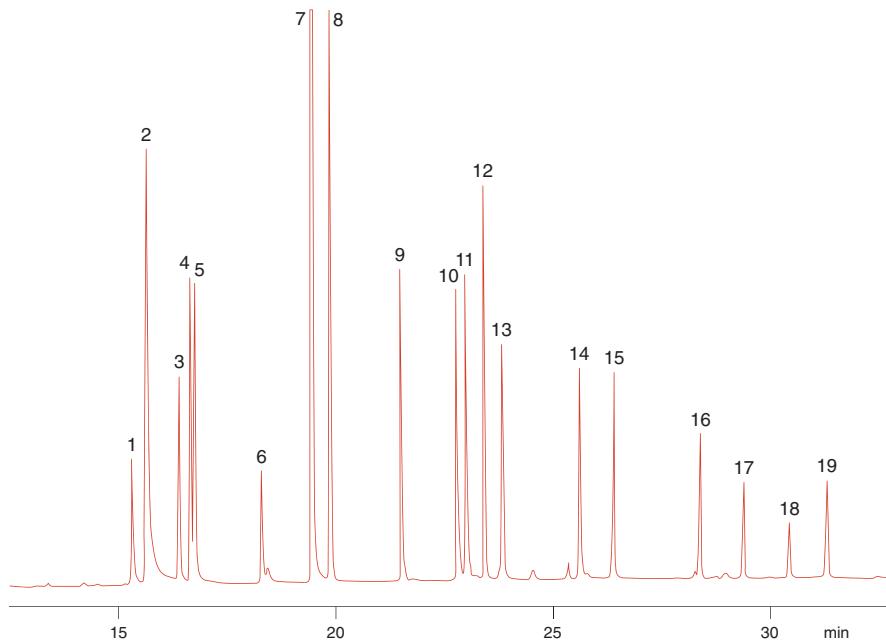
Carrier gas: H<sub>2</sub>

Temperature: 40 °C (3 min)  $\xrightarrow{5\text{ °C/min}}$  200 °C (10 min)

Detector: ECD 260 °C

**Peaks:**

- |                                                                               |                                                                                    |
|-------------------------------------------------------------------------------|------------------------------------------------------------------------------------|
| 1. 1-Nitrooxypropanol-2                                                       | 11. 1,3-Propanediol dinitrate                                                      |
| 2. 2-Nitrooxyethanol                                                          | 12. 1,3-Butanediol dinitrate                                                       |
| 3. RR/SS-3-Nitrooxybutanol-2                                                  | 13. 1,2-Pantanediol dinitrate                                                      |
| 4. 2-Nitrooxypropanol-1                                                       | 14. 1-Hydroxy-2-nitrooxycyclohexane                                                |
| 5. RS/SR-3-Nitrooxybutanol-2                                                  | 15. 1,2-Hexanediol dinitrate                                                       |
| 6. 1-Nitrooxybutanol-2                                                        | 16. trans-1,2-Cyclohexanediol dinitrate                                            |
| 7. 1,2-Ethanediol dinitrate / 1,2-Propanediol dinitrate / 2-Nitrooxybutanol-1 | 17. cis-1,2-Cyclohexanediol dinitrate                                              |
| 8. 2,3-Butanediol dinitrate                                                   | 18. cis/trans-1,3-Cyclohexanediol dinitrate                                        |
| 9. 1,2-Butanediol dinitrate                                                   | 19. trans-1,2-Cycloheptanediol dinitrate / cis/trans-1,3-Cyclohexanediol dinitrate |
| 10. 1-Hydroxy-2-nitrooxycyclopentane                                          |                                                                                    |



J. Kastler, K. Ballschmiter, Fresenius J Anal Chem 360 (1998) 812 – 816

## Miscellaneous



### Comparison of a separation on a 50 m standard capillary with separation on a 10 m fast GC column

MN Appl. No. 211260

#### A) Fast GC column

Column: OPTIMA® 5, 10 m x 0.1 mm ID,  
0.1  $\mu\text{m}$  film, REF 726846.10,  
max. temperature 340/360 °C  
injection 1  $\mu\text{l}$ , split 1:40,  
carrier gas 0.75 bar He

#### B) standard GC column

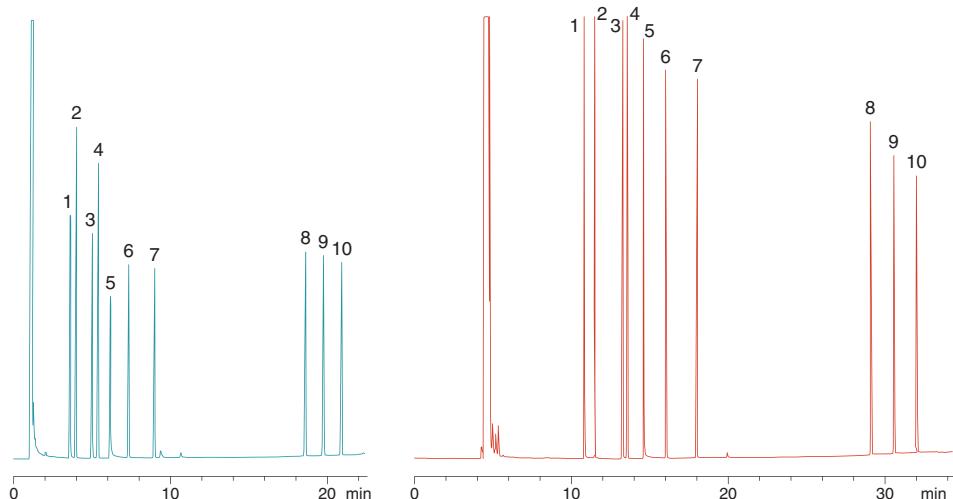
Column: OPTIMA® 5, 50 x 0.25 mm ID,  
0.25  $\mu\text{m}$  film, REF 726056.50,  
max. temperature 340/360 °C  
injection 1  $\mu\text{l}$ , split 1:35,  
carrier gas 1.5 bar He

both separations: temperature: 80 °C  $\xrightarrow{8 \text{ °C/min}}$  320 °C (10 min), detector: FID

While maintaining the temperature programme and halving the pressure a time saving of 30 % results with identical separation efficiency.

#### Peaks:

1. Octanol
2. Undecane
3. Dimethylaniline
4. Dodecane
5. Decylamine
6. Methyl decanoate
7. Methyl undecanoate
8. Henicosane
9. Docosane
10. Tricosane



## Food and cosmetic components

# Food and Cosmetic Components

Fragrances

FAMEs



# Fragrances

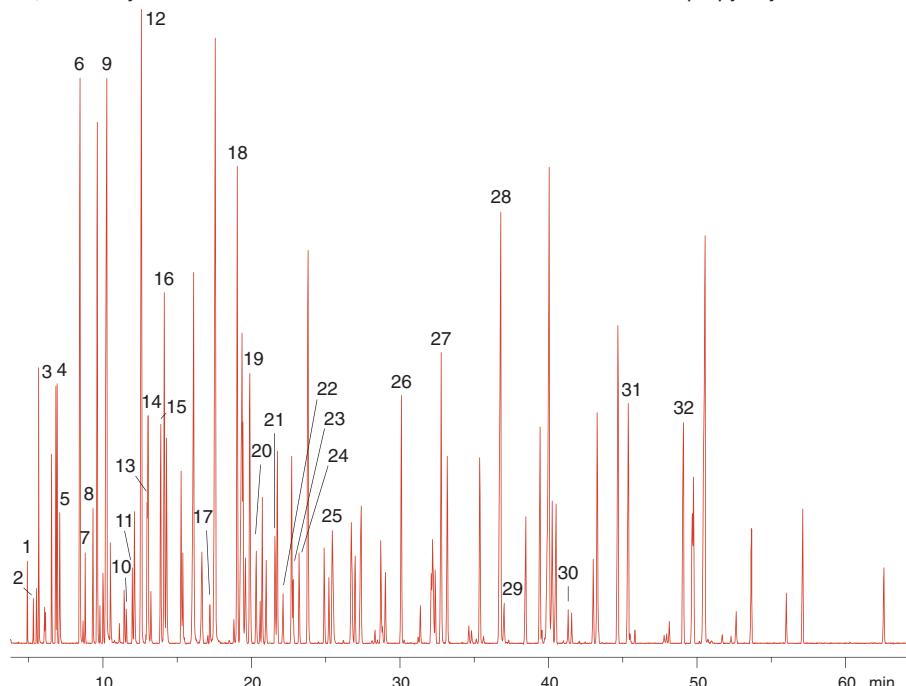


## Analysis of a perfume standard MN Appl. No. 201350

Column: OPTIMA® 5, 25 m x 0.20 mm ID, 0.20 µm film, REF 726857.25,  
max. temperature 340/360 °C  
Injection: 1.0 µl of a standard of perfume ingredients, 1:100 in EtOH, split 1:100  
sample courtesy of Mr. Vogel, Luzi AG, Dietlikon, Switzerland  
Carrier gas: 25 cm/s He      2 °C/min  
Temperature: 70 °C (3 min) → 320 °C  
Detector: MSD

### Peaks:

- |                                   |                                    |                                      |
|-----------------------------------|------------------------------------|--------------------------------------|
| 1. Benzaldehyde                   | 12. Phenylacetic acid methyl ester | 22. 2-Aminobenzoic acid methyl ester |
| 2. β-Pinene                       | 13. Menthol                        | 23. 2-Carene                         |
| 3. Limonene                       | 14. Acetic acid isonyl ester       | 24. Eugenol                          |
| 4. Eucalyptol (Cineole)           | 15. Linalyl propanoate             | 25. Diphenyl ether                   |
| 5. Benzyl alcohol                 | 16. α-Methylbenzyl acetate         | 26. α-Isomethyl ionone               |
| 6. 2,6-Dimethyl-7-octen-2-ol      | 17. 4-Methoxybenzaldehyde          | 27. Lilial                           |
| 7. Hexanoic acid 2-propenyl ester | 18. Bornyl acetate                 | 28. Diethyl phthalate                |
| 8. Benzoic acid methyl ester      | 19. Nonanoic acid ethyl ester      | 29. 2-Naphthyl methyl ketone         |
| 9. Phenylethanol                  | 20. 3-Phenyl-2-propen-1-ol         | 30. Methyl dihydrojasmonate          |
| 10. p-Menth-1(7)-en-9-ol          | 21. Piperonal                      | 31. Benzyl benzoate                  |
| 11. 3,7-Dimethyl-6-octenal        |                                    | 32. Isopropyl myristate              |



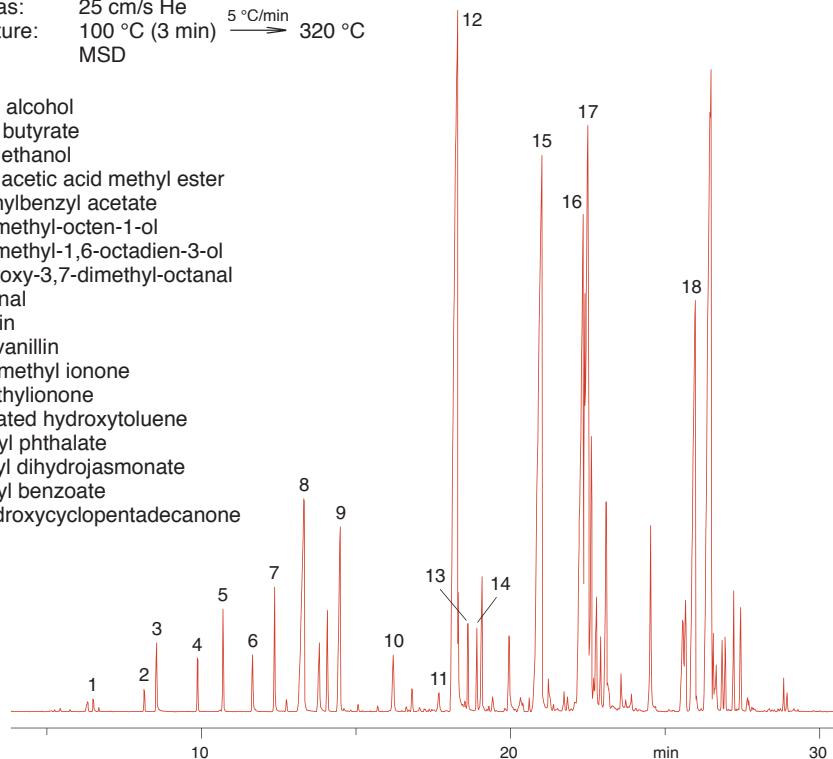
# Food and cosmetic components

## Analysis of commercial perfume MN Appl. No. 201360

Column: OPTIMA® 5, 25 m x 0.20 mm ID, 0.20 µm film, REF 726857.25,  
max. temperature 340/360 °C  
Injection: 1.0 µl perfume sample, 1:100 in EtOH, 3 s splitless  
Carrier gas: 25 cm/s He  
Temperature: 100 °C (3 min)  $\xrightarrow{5\text{ °C/min}}$  320 °C  
Detector: MSD

**Peaks:**

1. Benzyl alcohol
2. Linalyl butyrate
3. Phenylethanol
4. Phenylacetic acid methyl ester
5.  $\alpha$ -Methylbenzyl acetate
6. 3,7-Dimethyl-octen-1-ol
7. 3,7-Dimethyl-1,6-octadien-3-ol
8. 7-Hydroxy-3,7-dimethyl-octanal
9. Piperonal
10. Vanillin
11. Ethylvanillin
12.  $\alpha$ -Isomethyl ionone
13.  $\beta$ -Methylionone
14. Butylated hydroxytoluene
15. Diethyl phthalate
16. Methyl dihydrojasmonate
17. Benzyl benzoate
18. 2-Hydroxycyclopentadecanone



## Structures of selected perfume ingredients

Structure	Compound	R	Structure	Compound
<chem>O=C(O)c1ccc(O)c(R)c1</chem>	Vanillin	CHO	<chem>CC(=O)C/C=C\C1CCC(C)C(C)C1</chem>	$\beta$ -Methylionone
<chem>Oc1ccc(Oc2ccccc2)cc1</chem>	Vinylguaiacol	CH=CH <sub>2</sub>	<chem>CCCCC1OCOC1</chem>	Methyl dihydrojasmonate
<chem>CC=CC=Cc1ccccc1</chem>	Eugenol	CH <sub>2</sub> -CH=CH <sub>2</sub>	<chem>C1CCCC1</chem>	2-Carene
<chem>Oc1ccc(Oc2ccccc2)cc1</chem>	Piperonal	CHO		
<chem>Oc1ccc(Oc2ccccc2)cc1</chem>	Safrole	CH <sub>2</sub> -CH=CH <sub>2</sub>		
<chem>Oc1ccc(Oc2ccccc2)cc1</chem>	Anisaldehyde	CHO		
<chem>Oc1ccc(Oc2ccccc2)cc1</chem>	Anethole	CH=CH-CH <sub>3</sub>		

# Fragrances



## Analysis of a perfume standard MN Appl. No. 250530

Column: OPTIMA® δ-6, 50 m x 0.20 mm ID, 0.20 µm film, REF 726465.50,  
max. temperature 340/360 °C

Injection: 1.0 µl of a standard of perfume ingredients, 1:30 in EtOH, split 1:40  
sample courtesy of Mr. Vogel, Luzi AG, Dietlikon, Switzerland

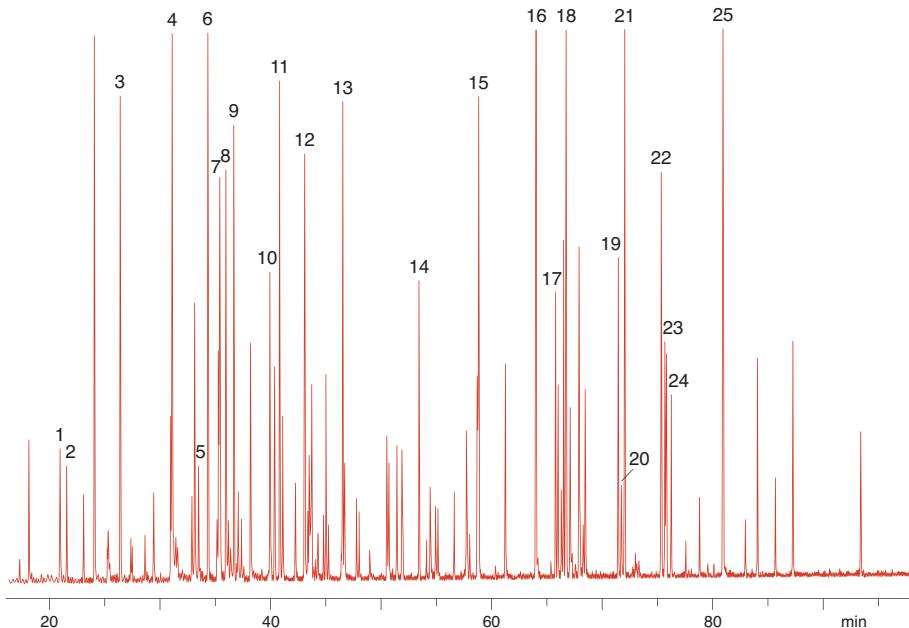
Carrier gas: 1.8 bar He

Temperature: 60 °C  $\xrightarrow{2^{\circ}\text{C}/\text{min}}$  280 °C (5 min)

Detector: MSD

**Peaks:**

- |                                             |                                                                    |
|---------------------------------------------|--------------------------------------------------------------------|
| 1. Limonene                                 | 14. α-Isomethyl ionone                                             |
| 2. Cineole                                  | 15. Lilial                                                         |
| 3. Linalool                                 | 16. Diethyl phthalate                                              |
| 4. 2-Phenylethanol                          | 17. 3,5-Di-t-butylphenol                                           |
| 5. Menthol                                  | 18. Methyl dihydrojasmonate                                        |
| 6. Benzyl acetate                           | 19. Isopropyl myristate                                            |
| 7. Styralyl acetate (1-phenylethyl acetate) | 20. Methyl atratate<br>(methyl 2,4-dihydroxy-3,6-dimethylbenzoate) |
| 8. 3,7-Dimethyl-6-octen-1-ol                | 21. Hexylcinnamic aldehyde                                         |
| 9. Linalyl anthranilate                     | 22. Benzyl benzoate                                                |
| 10. Ethyl nonanoate                         | 23. AETT (acetyl ethyltetramethyltetralin)                         |
| 11. Bornyl acetate                          | 24. Oxacyclohexadecan-2-one                                        |
| 12. Hydroxycitronellal                      | 25. Benzyl salicylate                                              |
| 13. 4-t-Butylcyclohexyl acetate             |                                                                    |



# Food and cosmetic components

## Analysis of essential oils

*MN Appl. No. 201340*

Column: PERMABOND® CW 20 M, 50 m x 0.32 mm ID, 0.5 µm film, REF 723296.50,  
max. temperature 220/240 °C

Injection: 1 µl

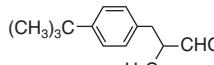
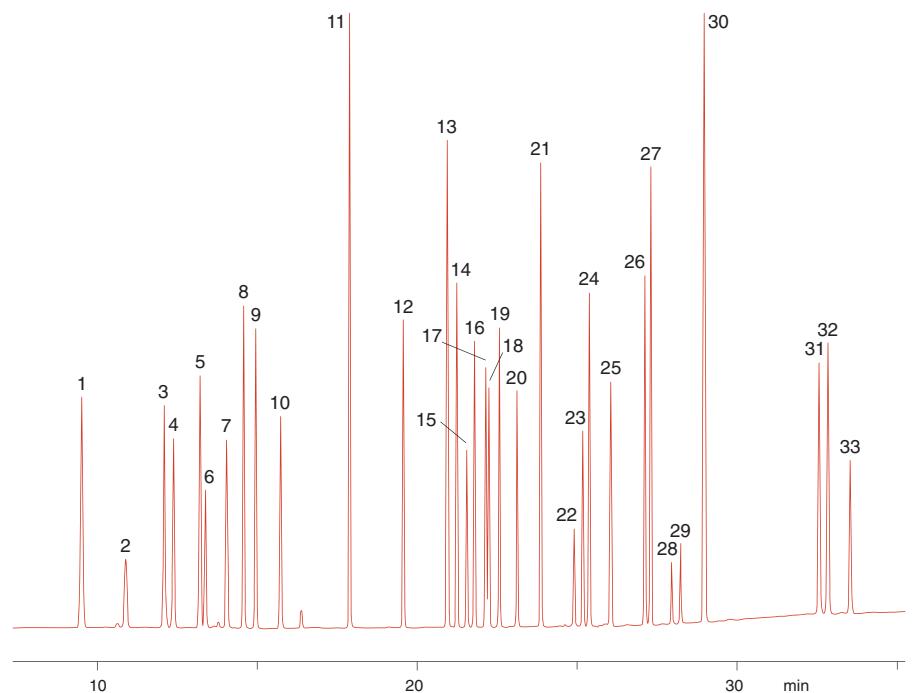
Carrier gas: 1.5 bar He

Temperature: 60 °C (5 min)  $\xrightarrow{8\text{ °C/min}}$  250 °C (10 min)

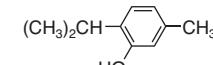
Detector: FID 250 °C

**Peaks:**

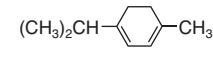
1. α-Pinene	8. Limonene	15. Isomenthone	22. α-Terpineol	28. Benzyl alcohol
2. Camphene	9. Cineole	16. Linalool	23. Borneol	29. Safrole
3. β-Pinene	10. γ-Terpinene	17. Camphor	24. Menthyl valerenate	30. Dodecanol
4. Sabinene	11. Hexanol-1	18. Linalyl acetate	25. Carvone	31. Thymol
5. Carene	12. Fenchone	19. Menthyl acetate	26. Geraniol	32. Eugenol
6. Myrcene	13. L-Menthone	20. Bornyl acetate	27. Anethole	33. α-Bisabolol
7. α-Terpinene	14. Menthofuran	21. Menthol		



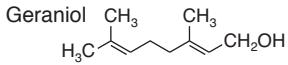
Lilial



Thymol



α-Terpinene



Geraniol

Myrcene

# Essential oils

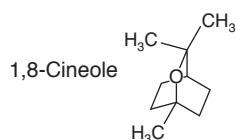
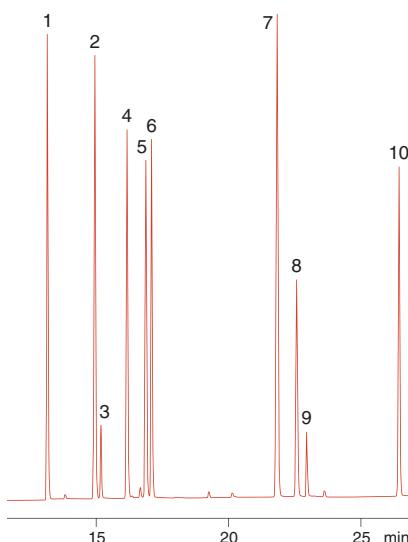


## Analysis of essential oils MN Appl. No. 201330

Column: OPTIMA® 5,  
50 m x 0.25 mm ID,  
0.35 µm film,  
REF 726623.50,  
max. temperature: 340/360 °C  
Injection: 0.5 µl, split 1:50  
Carrier gas: 1 bar N<sub>2</sub>  
Temperature: 95 °C (10 min) → 200 °C  
Detector: FID 280 °C

**Peaks:**

1. α-Pinene
2. β-Pinene
3. Int. std.
4. Δ<sup>3</sup>-Carene
5. Limonene
6. 1,8-Cineole
7. Camphor
8. Menthol
9. Int. std.
10. Bornyl acetate

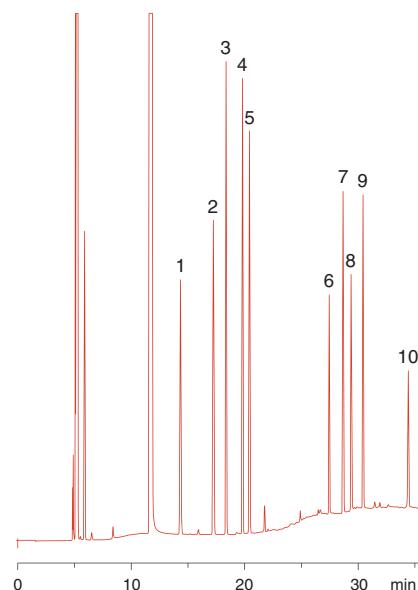


## Analysis of essential oils MN Appl. No. 201370

Column: PERMABOND® FFAP,  
50 m x 0.32 mm ID,  
0.5 µm film,  
REF 723344.50,  
max. temperature 220/240 °C  
Injection: 0.5 µl, split 1:50  
Carrier gas: 0.7 bar N<sub>2</sub>  
Temperature: 60 °C (6 min) → 200 °C  
(20 min)  
Detector: FID 250 °C

**Peaks:**

1. α-Pinene
2. β-Pinene
3. Δ<sup>3</sup>-Carene
4. Limonene
5. Eucalyptol (Cineole)
6. Camphor
7. Linalool
8. Bornyl acetate
9. Menthol
10. Carvone



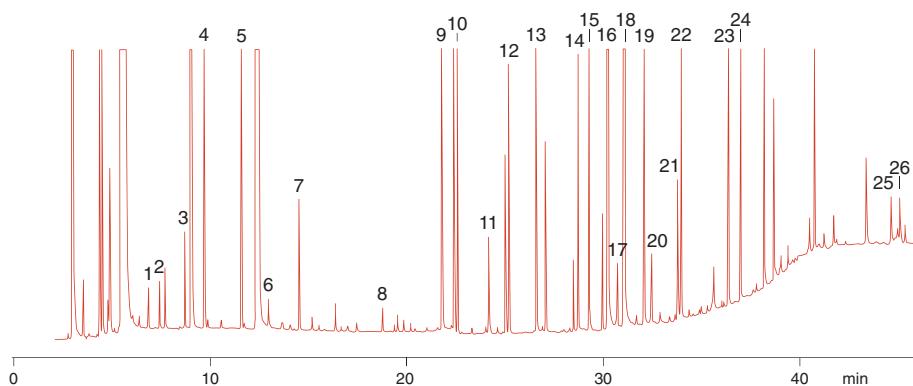
# Food and cosmetic components

## Key odorants in beer MN Appl. No. 211880

Column: PERMABOND® FFAP, 60 m x 0.25 mm ID, 0.25 µm film, REF 723116.60,  
max. temperature 220/240 °C  
Injection: 1 µl, splitless (split open at 1.0 min)  
Carrier gas: H<sub>2</sub> (5.0)  
Temperature: 40 °C (2 min)  $\xrightarrow{5\text{ °C/min}}$  230 °C (40 min)  
Detector: FID

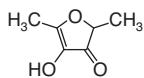
**Peaks:**

- |                           |                                           |                         |
|---------------------------|-------------------------------------------|-------------------------|
| 1. 2-Methylpropyl acetate | 10. 2-Methylpropionic acid                | 18. 2-Phenylethanol     |
| 2. Ethyl butanoate        | 11. Butyric acid                          | 19. (E)-2-Hexenoic acid |
| 3. Methyl pentanoate      | 12. 3-Methylbutyric acid                  | 20. Maltol              |
| 4. 3-Methylbutyl acetate  | 13. 3-Methylthiopropanol                  | 21. Furaneol            |
| 5. Methyl hexanoate       | 14. 2-Phenylethyl acetate                 | 22. Octanoic acid       |
| 6. Ethyl hexanoate        | 15. Hexanoic acid                         | 23. Eugenol             |
| 7. Methyl heptanoate      | 16. Benzyl alcohol                        | 24. 4-Vinylguaiacol     |
| 8. Ethyl octanoate        | 17. 3-Ethyl-2-hydroxy-2-cyclopenten-1-one | 25. Phenylacetic acid   |
| 9. 2-Methylthioethanol    |                                           | 26. Vanillin            |



B. Thum, W. Back, EBC Congress 1999

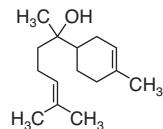
Furaneol



Maltol



Bisabolol



# Alcoholic beverages

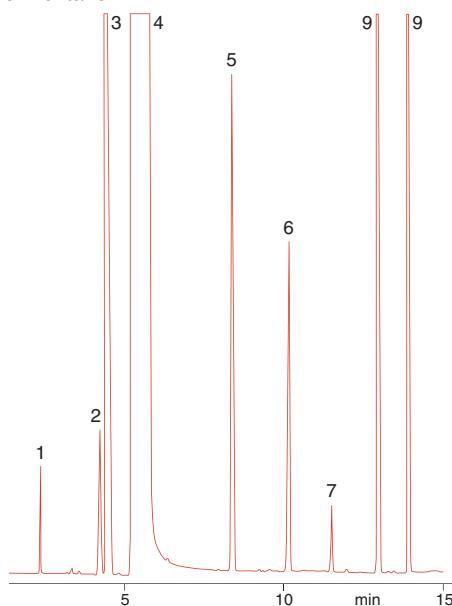


## Analysis of a plum brandy MN Appl. No. 210930

Column: PERMABOND® CW 20 M, 25 m x 0.32 mm ID, 0.5 µm film, REF 723296.25, max. temperature 220/240 °C  
Injection: 1 ml, 150 °C, split 1:20  
Carrier gas: N<sub>2</sub>, 40 Kpa (1 – 2 ml/min)  
Temperature: 50 °C (2 min) → 60 °C  
(5 min) → 150 °C (9 min)  
→ 170 °C (5 min)  
Detector: FID 180 °C

**Peaks:**

1. Acetaldehyde
2. Ethyl acetate
3. Methanol
4. Ethanol
5. Propanol-1
6. 2-Methylpropanol-1
7. Butanol-1
8. Isoamyl alcohol
9. Pentanol-1

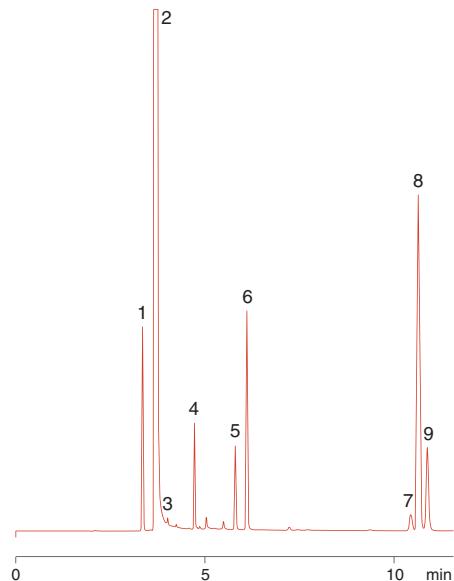


## Analysis of brandy made from wine MN Appl. No. 201480

Column: OPTIMA® 5, 50 m x 0.32 mm ID, 1.0 µm film, REF 726325.50, max. temperature 340/360 °C  
Injection: 0.5 µl, split 30 ml/min  
Carrier gas: 1 bar N<sub>2</sub>  
Temperature: 60 °C (10 min) → 80 °C  
Detector: FID 250 °C

**Peaks:**

1. Acetaldehyde/methanol
2. Ethanol
3. Acetone
4. Propanol-1
5. Ethyl acetate
6. i-Butanol
7. Diethyl acetal
8. 3-Methyl-1-butanol
9. 2-Methyl-1-butanol



Courtesy of Dr. Nilles, Wein- u. Bodenlabor,  
Volkach, Germany

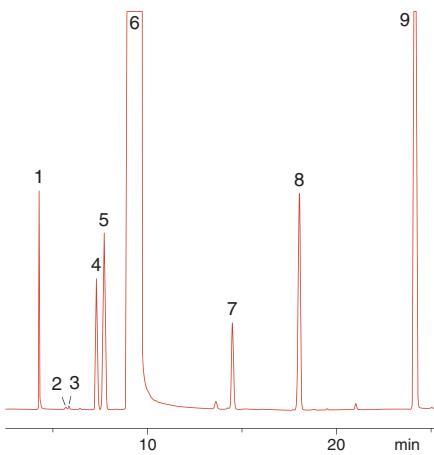
## Food and cosmetic components

### Analysis of brandy made from wine MN Appl. No. 201490

Column: PERMABOND® CW 20 M,  
50 m x 0.32 mm ID,  
0.5 µm film,  
REF 723296.50,  
max. temperature 220/240 °C  
Injection: 1 µl, split 20 ml/min  
Carrier gas: 1 bar N<sub>2</sub>  
Temperature: 60 °C (10 min) → 140 °C  
Detector: FID 250 °C

**Peaks:**

1. Acetaldehyde
2. Acetone
3. Methyl acetate
4. Ethyl acetate
5. Methanol
6. Ethanol
7. Propanol-1
8. *i*-Butanol
9. 2-Methyl-1-butanol/3-methyl-1-butanol

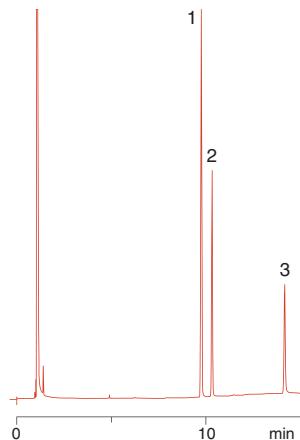


### Analysis of DEG standard (5 g/l ethanol) for wine analysis MN Appl. No. 201500

Column: PERMABOND® CW 20 M-DEG  
25 m x 0.25 mm ID,  
0.25 µm film, REF 723063.25,  
max. temperature 220/240 °C  
Injection: 0.5 µl, split 1:40  
Carrier gas: 1.2 bar N<sub>2</sub>  
Temperature: 80 °C → 200 °C  
Detector: FID 260 °C

**Peaks:** DEG standard

1. 1,4-Butanediol
2. Diethylene glycol (DEG)
3. Glycerol



# Fatty acid methyl esters (FAME)



## Summary of important saturated fatty acids

Code	Common name	Systematic name
C2:0	acetic acid	ethanoic acid
C3:0	propionic acid	propanoic acid
C4:0	butyric acid	butanoic acid
C4:0 iso	isobutyric acid	2-methylpropanoic acid
C5:0	valeric acid	pentanoic acid
C5:0 iso	isovaleric acid	3-methylbutanoic acid
C6:0	caproic acid	hexanoic acid
C6:0 iso	isocaproic acid	4-methylvaleric acid
C7:0	enanthic acid	heptanoic acid
C8:0	caprylic acid	octanoic acid
C9:0	pelargonic acid	nonanoic acid
C10:0	capric acid	decanoic acid
C12:0	lauric acid	dodecanoic acid
C14:0	myristic acid	tetradecanoic acid
C14:0 iso	isomyristic acid	12-methyltridecanoic acid
C15:0		pentadecanoic acid
C15:0 iso	13-methylmyristic acid	13-methyltetradecanoic acid
C15:0 anteiso	12-methylmyristic acid	12-methyltetradecanoic acid
C16:0	palmitic acid	hexadecanoic acid
C16:0 iso	isopalmitic acid	14-methylpentadecanoic acid
C17:0	margaric acid	heptadecanoic acid
C17:0 iso	isomargaric acid	15-methylhexadecanoic acid
	15-methylpalmitic acid	
C17:0 anteiso	anteisomargaric acid	14-methylhexadecanoic acid
	14-methylpalmitic acid	
C18:0	stearic acid	octadecanoic acid
C19:0 cyclo	methylene stearic acid	methylene octadecanoic acid
C20:0	arachidic acid	eicosanoic acid
C22:0	behenic acid	docosanoic acid
C23:0	tricosanoic acid	tricosanoic acid
C24:0	lignoceric acid	tetracosanoic acid
C26:0	cerotic acid	hexacosanoic acid

# Food and cosmetic components

## Summary of important unsaturated fatty acids

Code	Systematic name	$\omega$ reference system	Common name	$\omega$ -3 acid	$\omega$ -6 acid
C3:1	propenoic acid		acrylic acid		
C6:2	2,4-hexadienoic acid	C6:2n2	sorbic acid		
C14:1	<i>cis</i> -9-tetradecenoic acid	C14:1n5c	myristoleic acid		
	<i>trans</i> -9-tetradecenoic acid	C14:1n5t	myristelaidic acid		
C15:1	<i>cis</i> -10-pentadecenoic acid	C15:1n5			
C16:1	<i>cis</i> -9-hexadecenoic acid	C16:1n7c	palmitoleic acid		
	<i>trans</i> -9-hexadecenoic acid	C16:1n7t	palmitelaidic acid		
C17:1	<i>cis</i> -10-heptadecenoic acid	C17:1n7			
C18:1	<i>cis</i> -9-octadecenoic acid	C18:1n9c	oleic acid		
	<i>trans</i> -9-octadecenoic acid	C18:1n9t	elaidic acid		
	<i>cis</i> -11-octadecenoic acid	C18:1n7	vaccenic acid		
C18:2	<i>cis</i> -9,12-octadecadienoic acid	C18:2n6c	linoleic acid	x	
	<i>trans</i> -9,12-octadecadienoic acid	C18:2n6t	linolelaidic acid	x	
C18:3	<i>cis</i> -9,12,15-octadecatrienoic acid	C18:3n3	$\alpha$ -linolenic acid	x	
	<i>cis</i> -6,9,12-octadecatrienoic acid	C18:3n6	$\gamma$ -linolenic acid		x
C18:4	<i>cis</i> -6,9,12,15-octadecatetraenoic acid	C18:4n3	stearidonic acid	x	
C20:1	<i>cis</i> -11-eicosenoic acid	C20:1n9	gondoic acid		
C20:2	<i>cis</i> -11,14-eicosadienoic acid	C20:2n6			x
C20:3	<i>cis</i> -5,8,11-eicosatrienoic acid	C20:3n9	mead acid		
	<i>cis</i> -8,11,14-eicosatrienoic acid	C20:3n6		x	
	<i>cis</i> -11,14,17-eicosatrienoic acid	C20:3n3			x
C20:4	<i>cis</i> -5,8,11,14-eicosatetraenoic acid	C20:4n6	arachidonic acid		x
	<i>cis</i> -8,11,14,17-eicosatetraenoic acid	C20:4n3		x	
C20:5	<i>cis</i> -5,8,11,14,17-eicosapentaenoic acid	C20:5n3			x
C22:1	<i>cis</i> -13-docosenoic acid	C22:1n9	erucic acid		
C22:2	<i>cis</i> -13,16-docosadienoic acid	C22:2n6			x
C22:3	<i>cis</i> -13,16,19-docosatrienoic acid	C22:3n3		x	
C22:4	<i>cis</i> -7,10,13,16-docosatetraenoic acid	C22:4n6	adrenic acid		x
C22:5	<i>cis</i> -7,10,13,16,19-docosapentaenoic a.	C22:5n3	clupanodonic acid	x	
C22:6	<i>cis</i> -4,7,10,13,16,19-docosahexaenoic a.	C22:6n3		x	
C24:1	<i>cis</i> -15-tetracosenoic acid	C24:1n9	nervonic acid		

# Fatty acids



## Determination of butyric acid content in edible fats

MN Appl. No. 210130

In so-called mixed spreads unsaturated vegetable oils and fats replace a certain percentage of milk fat (MF). The proportion of MF in the product has to be labelled to protect the consumer from fraudulent malpractise, since the price of MF is higher than that of other relevant raw materials. In order to check correct labelling of mixed spreads, food inspection authorities need a reliable analytical method to determine the percentage of MF in the spread. The most distinctive feature of MF, i.e. the unique occurrence of butyric acid, is most often determined by chromatography and used as an indicator for calculating the MF content in foodstuffs.

MF (100 mg) is weighed in a 10 ml HS vial, 1 ml sodium methoxide (0.5% metallic sodium in methanol), 1 ml internal standard solution (isobutyric acid, valeric acid) are added, and the vial is sealed with a PTFE-lined crimp cap.

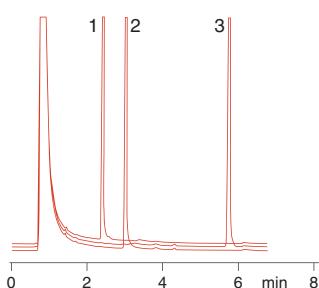
Column: OPTIMA® 5,  
25 m x 0.32 mm ID,  
1.0  $\mu\text{m}$  film, REF 726325.25,  
max. temperature 340/360 °C

Injection: head space, split 1:34, 150 °C  
Carrier gas: 3 ml/min  $\text{H}_2$ , 50 kPa head-  
pressure

Temperature: 50 °C (7 min)  $\xrightarrow{4 \text{ °C/min}}$  60 °C  
Detector: FID 250 °C

### Peaks:

1. C4 iso
2. C4
3. C5



F. Ulberth, Z. Lebensm. Unters. Forsch. A, 206  
(1998) 305 – 307

## Analysis of free carboxylic acids C2 – C7

MN Appl. No. 201570

Column: PERMABOND® FFAP,  
10 m x 0.25 mm ID,  
0.25  $\mu\text{m}$  film, REF 723116.10,  
max. temperature 220/240 °C

Injection: 0.5  $\mu\text{l}$ , split 1:50

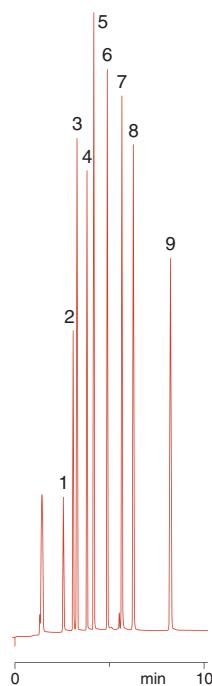
Carrier gas: 0.3 bar  $\text{N}_2$

Temperature: 120 °C  $\xrightarrow{6 \text{ °C/min}}$  150 °C  
(10 min)

Detector: FID 260 °C

### Peaks:

1. C2
2. C3
3. C4 iso
4. C4
5. C5 iso
6. C5
7. C6 iso
8. C6
9. C7

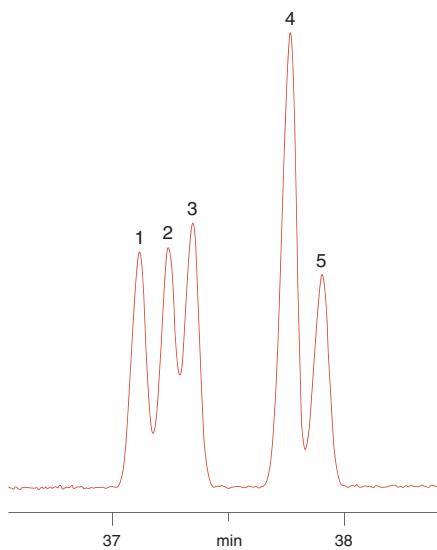


## Food and cosmetic components

### Separation of C18 FAMEs MN Appl. No. 212640

Column: OPTIMA<sup>®</sup> δ-6,  
50 m x 0.20 mm ID,  
0.2 µm film, REF 726465.50,  
max. temperature 340/360 °C  
Injection: 1 µl, 0.1 % FAME in hexane  
split 20 ml/min  
Carrier gas: 2.2 bar He  
Temperature: 120 °C → 180 °C  
2 °C/min → 300 °C (5 min)  
Detector: MSD 280 °C

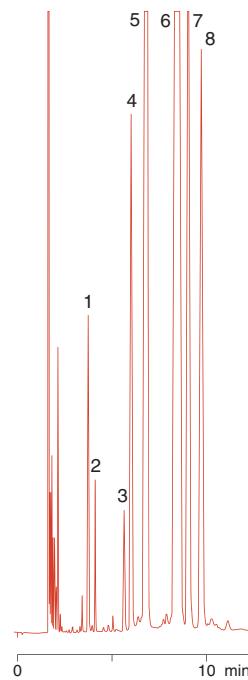
**Peaks:**  
1. C18:1n9c  
2. C18:2n6c  
3. C18:1n9t  
4. C18:0  
5. C18:3n3



### Analysis of FAMEs C10:0 – C18:2 MN Appl. No. 201700

Column: PERMABOND<sup>®</sup> FFAP,  
25 m x 0.25 mm ID,  
0.25 µm film,  
REF 723116.25,  
max. temperature 220/240 °C  
Injection: 0.5 µl, split 1:50  
Carrier gas: 1 bar N<sub>2</sub>  
Temperature: 200 °C  
Detector: FID 260 °C

**Peaks:**  
1. C10:0  
2. C16:0  
3. C18:0  
4. C18:1n9c  
5. C18:2n6c  
6. C18:2 (cis, cis, 9, 11)  
7. C18:2 (cis, trans, 9, 11)  
8. C18:2 (trans, trans, 9, 11)





### Analysis of FAMEs

*MN Appl. No. 250120*

Column: OPTIMA® δ-3, 25 m x 0.20 mm ID, 0.20 µm film, REF 726400.25,  
max. temperature 340/360 °C

Injection: 1 µl, split 1:40

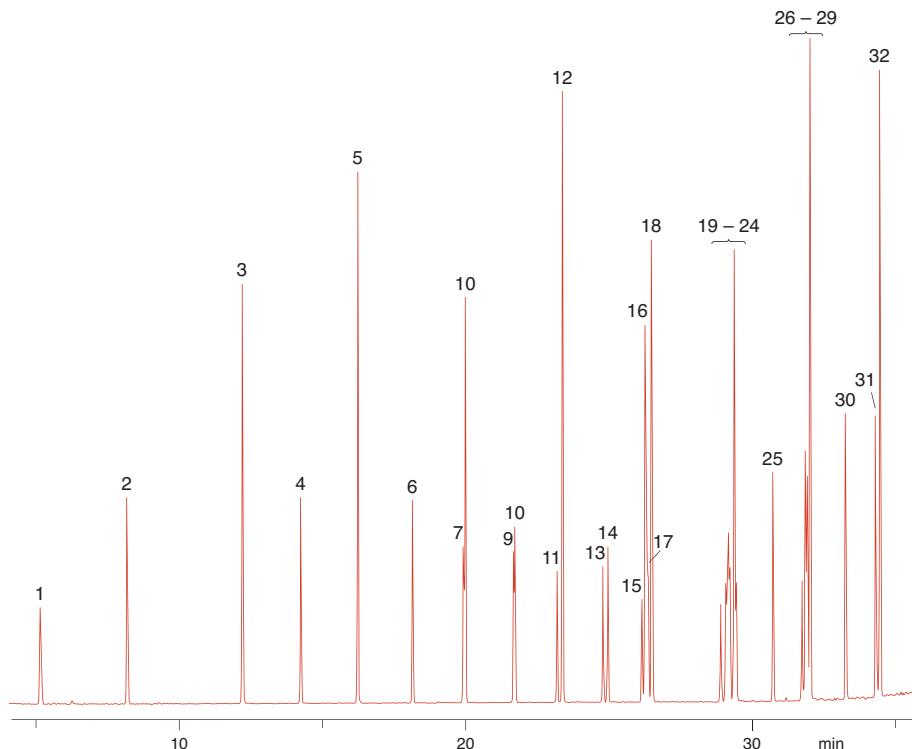
Carrier gas: 1.1 bar He

Temperature: 100 °C (1 min)  $\xrightarrow{6 \text{ °C/min}}$  300 °C (5 min)

Detector: MSD

**Peaks:**

1. C6:0	9. C15:1	17. C18:0	25. C21:0
2. C8:0	10. C15:0	18. C18:2	26. C20:5
3. C10:0	11. C16:1	19. C20:4	27. C22:1
4. C11:0	12. C16:0	20. C20:3	28. C22:2
5. C12:0	13. C17:1	21. C20:1	29. C22:0
6. C13:0	14. C17:0	22. C20:2	30. C23:0
7. C14:1	15. C18:3	23. C20:0	31. C24:1
8. C14:0	16. C18:1	24. C20:3	32. C24:0



## Food and cosmetic components

### Analysis of FAMEs MN Appl. No. 250470

Column: OPTIMA® δ-6, 50 m x 0.20 mm ID, 0.20 µm film, REF 726465.50,  
max. temperature 340/360 °C

Injection: 1 µl, 10 mg FAME mix per ml in CH<sub>2</sub>Cl<sub>2</sub>, split 1:50

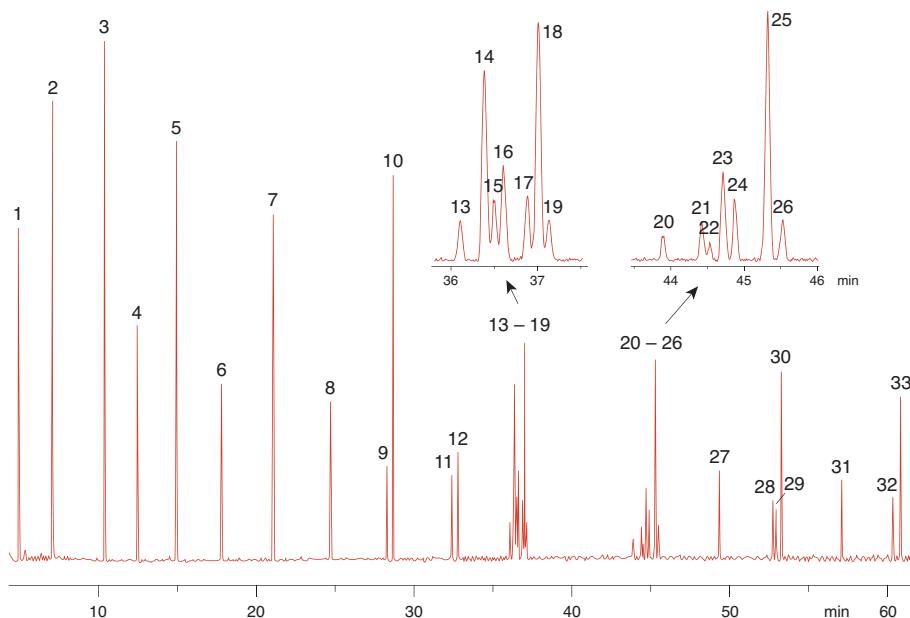
Carrier gas: 2.2 bar He

Temperature: 120 °C → 180 °C → 300 °C (5 min)

Detector: MSD

**Peaks:**

1. C6:0	10. C16:0	18. C18:0	26. C20:3n3
2. C8:0	11. C17:1n7	19. C18:3n3	27. C21:0
3. C10:0	12. C17:0	20. C20:4n6	28. C22:1n9
4. C11:0	13. C18:3n6	21. C20:3n6	29. C22:2n6
5. C12:0	14. C18:1n9c	22. C20:5n3	30. C22:0
6. C13:0	15. C18:2n6c	23. C20:1n9	31. C23:0
7. C14:0 + C14:1n5c	16. C18:1n9t	24. C20:2n6	32. C24:1n9
8. C15:0 + C15:1n5c	17. C18:2n6t	25. C20:0	33. C24:0
9. C16:1n7c			





### Analysis of FAMEs in porcine fat MN Appl. No. 210060

Column: OPTIMA® 225, 25 m x 0.32 mm ID, 0.25 µm film, REF 726352.25,  
max. temperature 260/280 °C

Injection: 1 µl, split 1:40

Carrier gas: 60 kPa H<sub>2</sub>

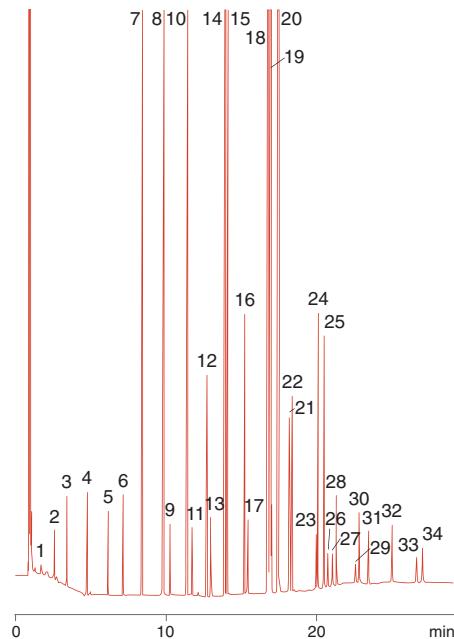
Temperature: 50 °C (2 min)  $\xrightarrow{30\text{ °C/min}}$  125 °C  $\xrightarrow{5\text{ °C/min}}$  160 °C  $\xrightarrow{20\text{ °C/min}}$  180 °C  $\xrightarrow{3\text{ °C/min}}$  200 °C  
 $\xrightarrow{20\text{ °C/min}}$  220 °C (10 min)

Detector: FID 260 °C

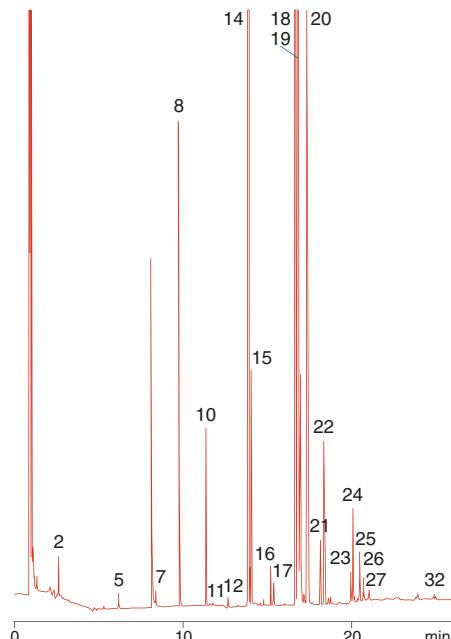
**Peaks:**

1. C4:0	6. C11:0	11. C14:1n5c	16. C17:0	21. C18:3	26. C20:4n6	31. C22:2
2. C5:0	7. C12:0	12. C15:0	17. C17:1	22. C19:0	27. C20:3	32. C22:6
3. C6:0	8. C13:0	13. C15:1	18. C18:0	23. C20:0	28. C20:5	33. C24:0
4. C8:0	9. C13:1	14. C16:0	19. C18:1n9c	24. C20:1	29. C22:0	34. C24:1n9
5. C10:0	10. C14:0	15. C16:1n7c	20. C18:2n6c	25. C20:2	30. C22:1	

FAME standard



FAMEs in porcine fat



Courtesy of Dr. Bantleon, Mr. Leusche, Mr. Hagemann, VFG-Labor, Versmold, Germany

## Food and cosmetic components

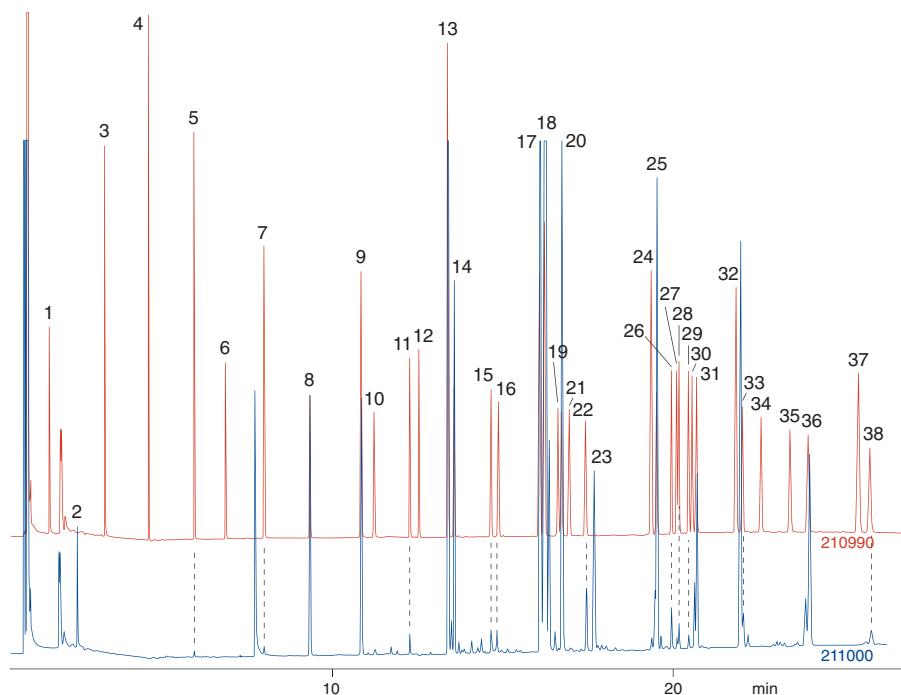
### Analysis of FAMEs from pork and salmon MN Appl. No. 210990 / 211000

Column: OPTIMA® 225, 25 m x 0.32 mm ID, 0.25 µm film, REF 726352.25,  
max. temperature 260/280 °C  
Injection: 1 µl, split 1:40  
Carrier gas: 60 kPa H<sub>2</sub>  
Temperature: 50 °C (2 min)  $\xrightarrow{30\text{ °C/min}}$  125 °C  $\xrightarrow{5\text{ °C/min}}$  160 °C  $\xrightarrow{20\text{ °C/min}}$  180 °C  $\xrightarrow{3\text{ °C/min}}$  200 °C  
 $\xrightarrow{20\text{ °C/min}}$  220 °C (10 min)  
Detector: FID 260 °C

**Application 210990: FAME mixture · Application 211000: FAMEs from pork and salmon**

**Peaks:**

1. C4:0	9. C14:0	17. C18:0	25. C20:1n9	33. C22:1n9
2. C5:0	10. C14:1n5	18. C18:1n9c+t	26. C20:2n6	34. C22:2n6
3. C6:0	11. C15:0	19. C18:2n6t	27. C20:3n6	35. C23:0
4. C8:0	12. C15:1n5	20. C18:2n6c	28. C20:4n6	36. C22:6n3
5. C10:0	13. C16:0	21. C18:3n6	29. C20:3n3	37. C24:0
6. C11:0	14. C16:1n7	22. C18:3n3	30. C21:0	38. C24:1n9
7. C12:0	15. C17:0	23. C19:0	31. C20:5n3	
8. C13:0	16. C17:1n7	24. C20:0	32. C22:0	



Courtesy of Dr. Bantleon, Mr. Leusche, Mr. Hagemann, VFG-Labor, Versmold, Germany

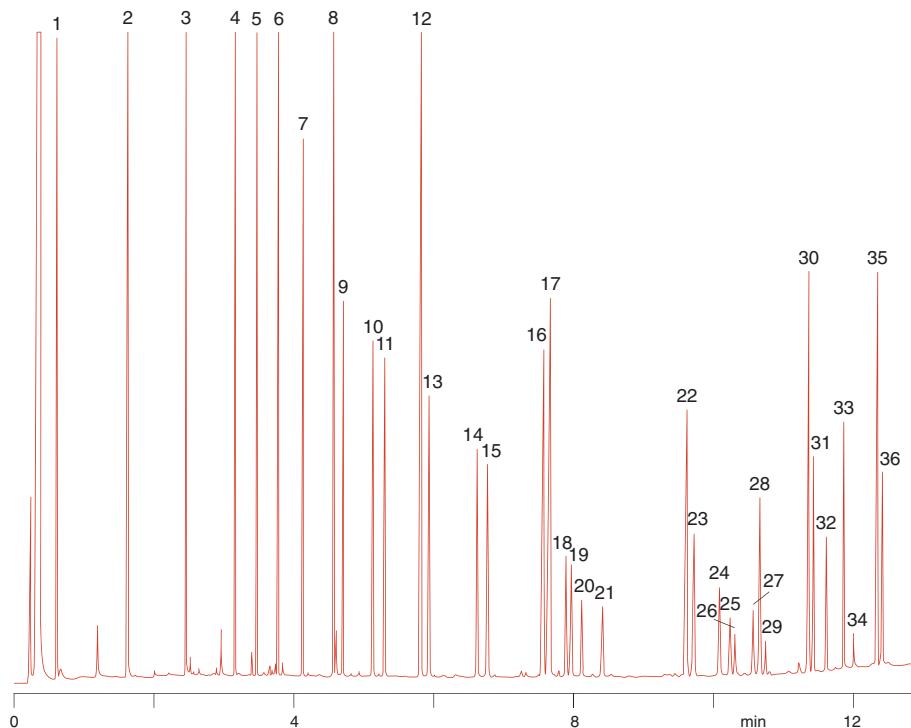


**Fast analysis of a FAME mixture**  
*MN Appl. No. 211820*

Column: OPTIMA® 225, 10 m x 0.1 mm ID, 0.1 µm film, REF 726080.10,  
max. temperature 260/280 °C  
Injection: 1 µl, split 1:250  
Carrier gas: 280 kPa H<sub>2</sub>  
Temperature: 50 °C (1 min) → 160 °C → 200 °C → 240 °C (2 min)  
Detector: FID 260 °C

**Peaks:**

1. C4:0	10. C15:0	19. C18:2n6c	28. C21:0
2. C6:0	11. C15:1n5	20. C18:3n6	29. C20:5n3
3. C8:0	12. C16:0	21. C18:3n3	30. C22:0
4. C10:0	13. C16:1	22. C20:0	31. C22:1n9
5. C11:0	14. C17:0	23. C20:1n9	32. C22:2n6
6. C12:0	15. C17:1n7	24. C20:2n6	33. C23:0
7. C13:0	16. C18:0	25. C20:3n6	34. C22:6n3
8. C14:0	17. C18:1n9c+t	26. C20:4n6	35. C24:0
9. C14:1	18. C18:2n6t	27. C20:3n3	36. C24:1



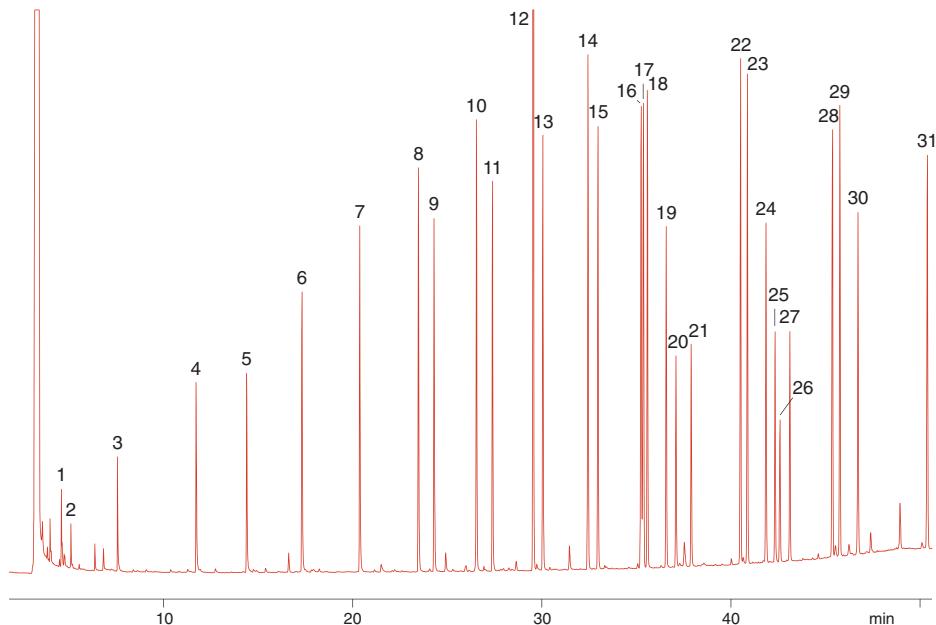
## Food and cosmetic components

### Analysis of FAMEs incl. *cis/trans* C18:1 MN Appl. No. 201620

Column: OPTIMA® 240, 60 m x 0.25 mm ID, 0.25 µm film, REF 726089.60,  
max. temperature 260/280 °C  
Injection: 1.0 µl of a FAME mixture, split 1:25  
Carrier gas: 150 kPa H<sub>2</sub>  
Temperature: 80 °C  $\xrightarrow{20\text{ °C/min}}$  120 °C  $\xrightarrow{3\text{ °C/min}}$  260 °C (10 min)  
Detector: FID 280 °C

**Peaks:**

1. C4:0	9. C14:1	17. C18:1n9t	25. C20:3
2. C5:0	10. C15:0	18. C18:1n9c	26. C20:4
3. C8:0	11. C15:1	19. C18:2	27. C20:3
4. C10:0	12. C16:0	20. C18:3	28. C22:0
5. C11:0	13. C16:1	21. C18:3	29. C22:1
6. C12:0	14. C17:0	22. C20:0	30. C22:3
7. C13:0	15. C17:1	23. C20:1	31. C24:1
8. C14:0	16. C18:0	24. C20:2	





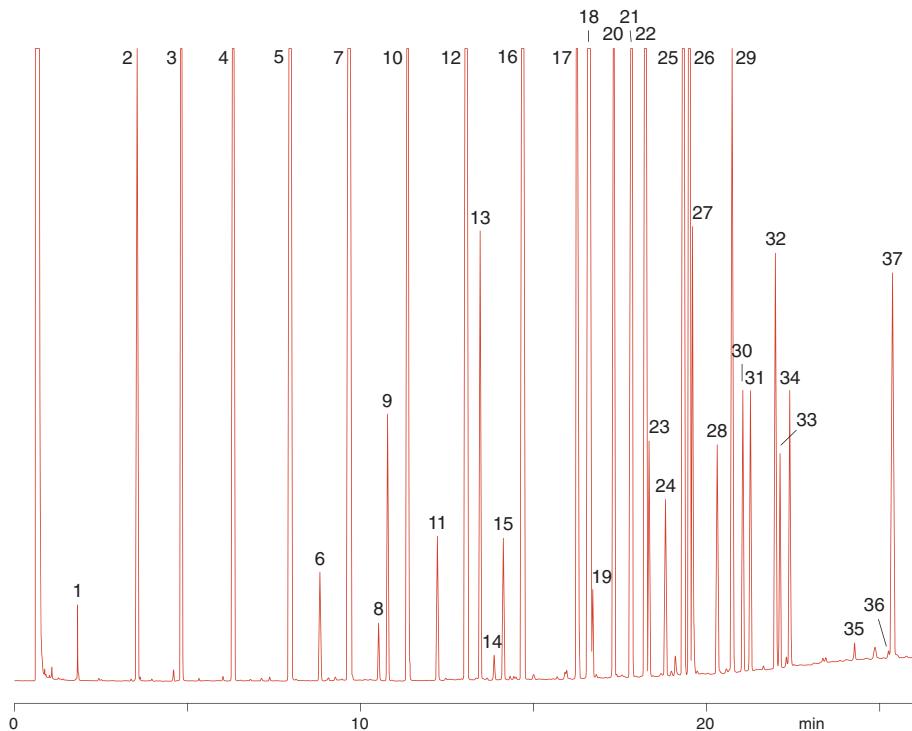
### Analysis of FAMEs

*MN Appl. No. 201660*

Column: PERMABOND® FFAP, 25 m x 0.25 mm ID, 0.1 µm film, REF 723936.25,  
max. temperature 220/240 °C  
Injection: 0.5 µl, split 120 ml/min  
Carrier gas: 0.75 bar H<sub>2</sub>  
Temperature: 100 °C  $\xrightarrow{6\text{ °C/min}}$  240 °C (5 min)  
Detector: FID 250 °C

**Peaks:**

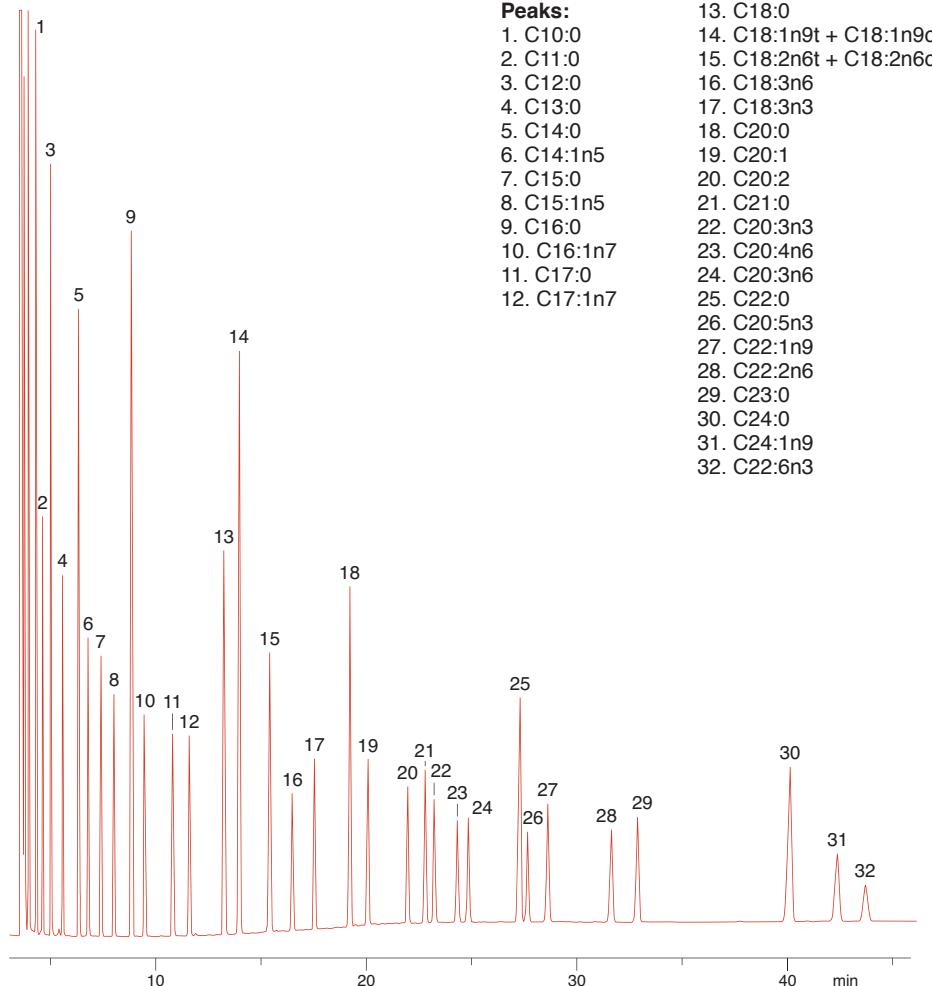
1. C8:0	11. C16:0 iso	21. C19:0 + C18:3n6	31. C20:3n3
2. C10:0	12. C16:0	22. C19:0 cyclo	32. C20:5
3. C11:0	13. C16:1	23. C18:3n3	33. C22:0
4. C12:0	14. C17:0 iso	24. C18:4	34. C22:1
5. C13:0	15. C17:0 anteiso	25. C20:0	35. C24:0
6. C14:0 iso	16. C17:0	26. C20:1	36. C24:1
7. C14:0	17. C18:0	27. C20:1	37. C22:6
8. C15:0 iso	18. C18:1n9c	28. C20:2	
9. C15:0 anteiso	19. C18:1n9t	29. C21:0	
10. C15:0	20. C18:2	30. C20:4	



## Food and cosmetic components

### Analysis of a FAME standard MN Appl. No. 212750

Column: PERMABOND® FFAP, 50 m x 0.25 mm ID, 0.25 µm film, REF 723116.50,  
max. temperature 220/240 °C  
Injection: 1.0 µl, FAME standard, 250 °C  
Carrier gas: 80 kPa H<sub>2</sub>  
Temperature: 200 °C (10 min)  $\xrightarrow{2\text{ °C/min}}$  220 °C  
Detector: FID 250 °C



Courtesy of Dr. E. Most, Institut für Tierernährung und Ernährungsphysiologie,  
Justus-Liebig-Universität, Gießen, Germany

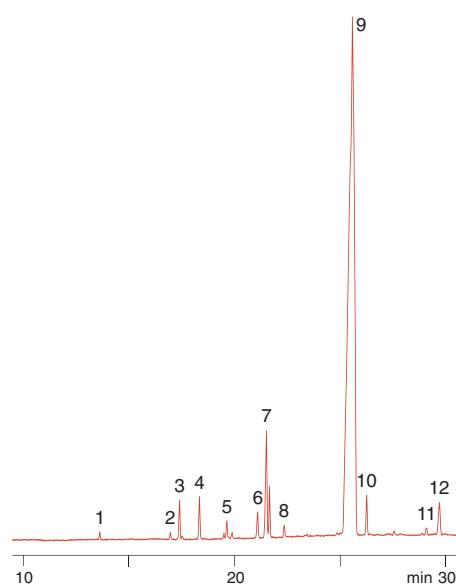


### Analysis of FAMEs in rapeseed oil by fast GC

MN Appl. No. 212010

Column: PERMABOND® FFAP,  
20 m x 0.10 mm ID,  
0.1 $\mu$ m film, REF 723180.20,  
max. temperature 220/240 °C  
Injection: 0.5  $\mu$ l  
Carrier gas: 170 kPa  
Temperature: 60 °C  $\xrightarrow{15\text{ °C/min}}$  120 °C  $\xrightarrow{10\text{ °C/min}}$   
210 °C (5 min)  $\xrightarrow{15\text{ °C/min}}$  240 °C  
(30 min)  
Detector: FID 300 °C

**Peaks:**  
1. C16:0              7. C20:1  
2. C18:0              8. C20:2  
3. C18:1n9c           9. C22:0 + C22:1n9  
4. C18:2              10. C22:2  
5. C18:3              11. C24:0  
6. C20:0              12. C24:1

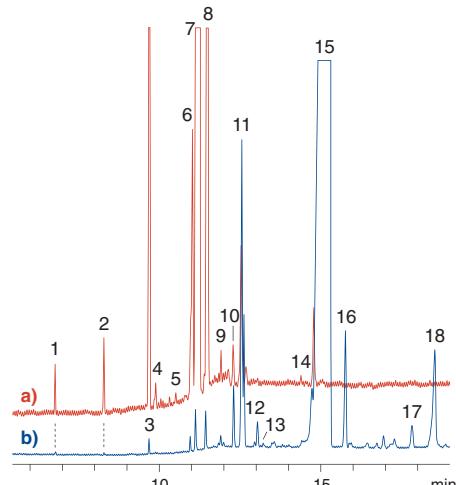


### Analysis of FAMEs in rapeseed oil by fast GC

MN Appl. No. 212150

Column: PERMABOND® FFAP,  
10 m x 0.10 mm ID,  
0.25  $\mu$ m film, REF 723181.10,  
max. temperature 220/240 °C  
Injection: 1.0  $\mu$ l, 250 °C  
Carrier gas: He  
Temperature: 60 °C  $\xrightarrow{15\text{ °C/min}}$  230 °C  
(13.67 min)  
Detector: FID 300 °C

**Peaks:**  
a) fraction rich in oleic acid  
b) fraction of rapeseed oil rich in erucic acid  
1. C12:0              7. C18:1n9c      13. C21:0  
2. C14:0              8. C18:2              14. C22:0  
3. C16:0              9. C18:3              15. C22:1n9  
4. C16:1              10. C20:0              16. C22:2  
5. C17:0              11. C20:1              17. C24  
6. C18:0              12. C20:2              18. C24:1



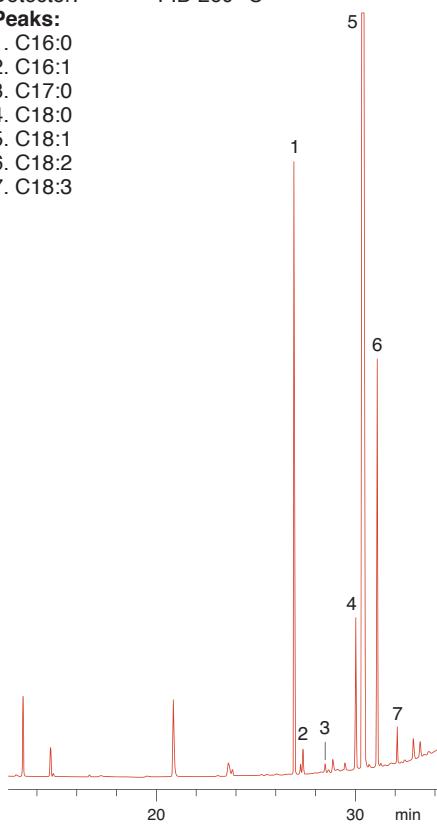
## Food and cosmetic components

### Analysis of FAMEs from olive oil MN Appl. No. 210550

Column: PERMABOND® FFAP,  
25 m x 0.32 mm ID,  
0.25  $\mu$ m film, REF 723341.25,  
max. temperature 220/240 °C  
Injection: 1  $\mu$ l, split 110 ml/min  
Carrier gas: 3.3 ml/min H<sub>2</sub>  
Temperature: 50 °C (4 min)  $\xrightarrow{6\text{ °C/min}}$  240 °C  
Detector: FID 260 °C

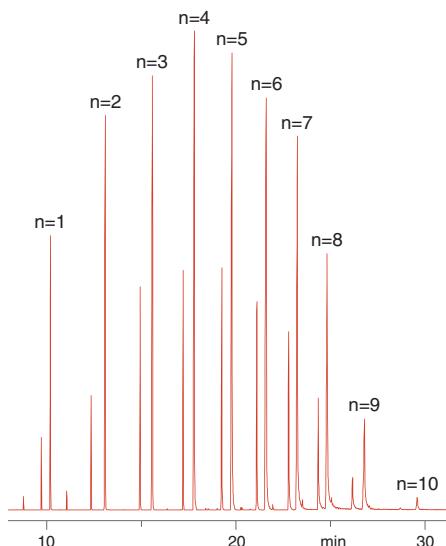
**Peaks:**

1. C16:0
2. C16:1
3. C17:0
4. C18:0
5. C18:1
6. C18:2
7. C18:3



### Analysis of ethoxylated fatty alcohols C12 and C14 (TMS) MN Appl. No. 201760

Column: OPTIMA® 5,  
25 m x 0.20 mm ID,  
0.2  $\mu$ m film, REF 726857.25,  
max. temperature 340/360 °C  
Sample: ethoxylated fatty alcohols  
R – (O – CH<sub>2</sub> – CH<sub>2</sub>)<sub>n</sub> – O – TMS, silylated with BSTFA,  
R = C12 and C14  
Injection: 0.5  $\mu$ l, 300 °C, split 1:150  
Carrier gas: 25 cm/s He  
Temperature: 120 °C (3 min)  $\xrightarrow{10\text{ °C/min}}$  330 °C  
Detector: MSD  
n = degree of ethoxylation



# Lipids

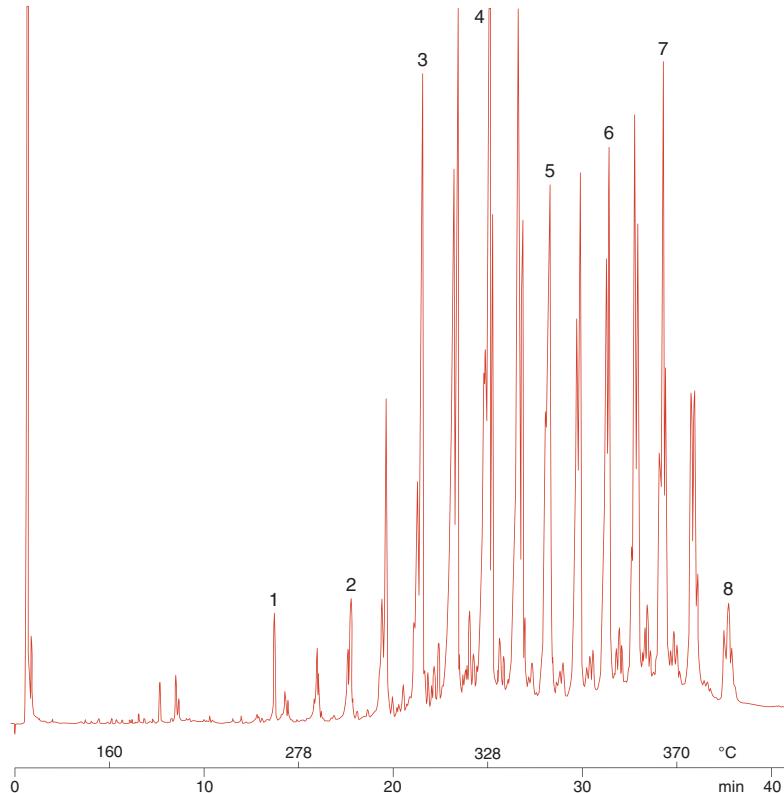


## Analysis of triglycerides from butter MN Appl. No. 201790

Column: OPTIMA® 1-TG, 25 m x 0.32 mm ID, REF 726132.25, max. temperature 370 °C  
Injection: 0.5 µl  
Carrier gas: 80 kPa H<sub>2</sub>  
Temperature: 80 °C (1 min)  $\xrightarrow{20 \text{ °C/min}}$  250 °C  $\xrightarrow{5 \text{ °C/min}}$  370 °C (10 min)  
Detector: FID 380 °C

**Peaks:**

1. Cholesterol
2. T-30
3. T-34
4. T-38
5. T-42
6. T-46
7. T-50
8. T-54



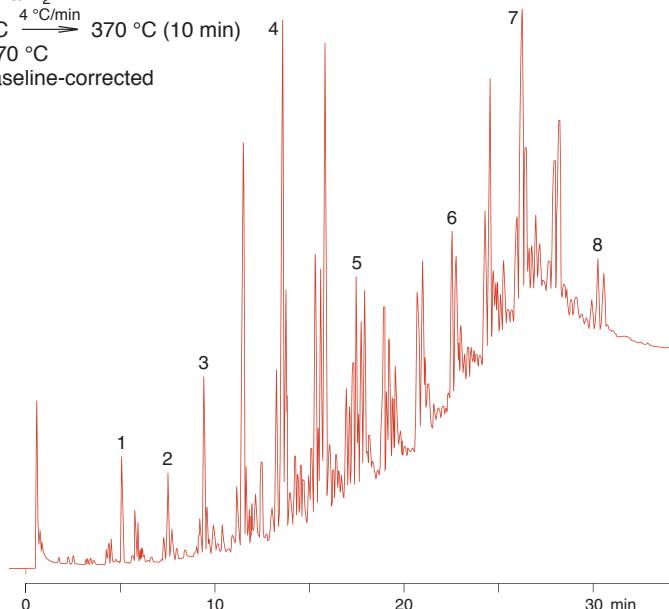
## Food and cosmetic components

### Analysis of triglycerides from butter MN Appl. No. 201800

Column: OPTIMA® 17-TG, 25 m x 0.25 mm ID, REF 726130.25, max. temperature 370 °C  
Injection: 1 µl  
Carrier gas: 100 kPa H<sub>2</sub>  
Temperature: 260 °C  $\xrightarrow{4\text{ °C/min}}$  370 °C (10 min)  
Detector: FID 370 °C  
not baseline-corrected

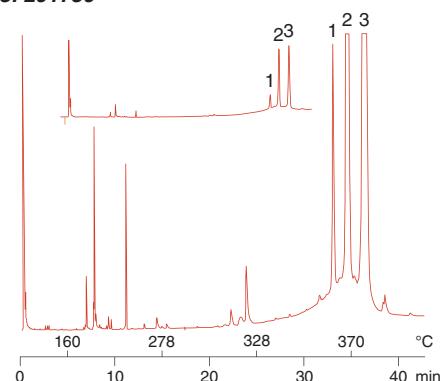
**Peaks:**

1. T-26
2. Cholesterol
3. T-30
4. T-36
5. T-40
6. T-46
7. T-50
8. T-54



### Analysis of triglycerides from olive oil MN Appl. No. 201780

Column: OPTIMA® 1-TG,  
25 m x 0.32 mm ID,  
REF 726132.25,  
max. temperature 370 °C  
Injection: 0.5 µl  
Carrier gas: 80 kPa H<sub>2</sub>  
Temperature: 80 °C (1 min)  $\xrightarrow{20\text{ °C/min}}$  250 °C  
 $\xrightarrow{5\text{ °C/min}}$  370 °C (10 min)  
Detector: FID 380 °C

**Peaks:**  
1. T-50  
2. T-52  
3. T-54

## Miscellaneous

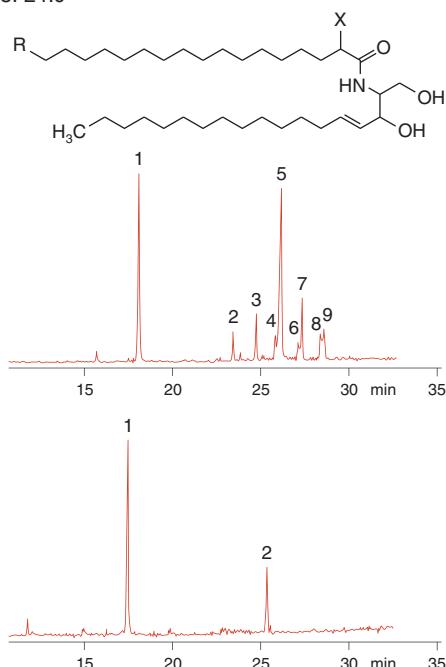


### Ceramide analysis by GC/MS MN Appl. No. 211980

Column: OPTIMA® 5,  
30 m x 0.32 mm ID,  
0.1  $\mu$ m film, REF 726313.30,  
max. temperature 340/360 °C  
Injection: 0.5  $\mu$ l, 320 °C, splitless  
Carrier gas: He, 0.6 bar He  
Temperature: 260 °C  $\xrightarrow{3\text{ °C/min}}$  360 °C  
(20 min)  
Detector: MSD

**Peaks:**

- |                                                               |                                                           |
|---------------------------------------------------------------|-----------------------------------------------------------|
| a) Ceramide IV<br>(2-hydroxy fatty acid ceramides,<br>X = OH) | b) Ceramide III (non-hydroxy fatty acid ceramides, X = H) |
| 1. 18:0      6. 25:1                                          | 1. 18:0                                                   |
| 2. 22:0      7. 25:0                                          | 2. 24:1                                                   |
| 3. 23:0      8. 26:1                                          |                                                           |
| 4. 24:1      9. 26:0                                          |                                                           |
| 5. 24:0                                                       |                                                           |



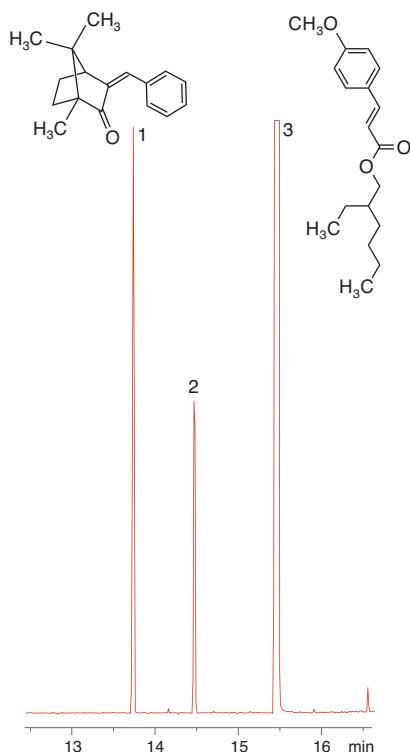
K. Raith, J. Darius, R.H.H. Neubert,  
J. Chromatography A, **876** (2000) 229 – 233

### Analysis of parsols MN Appl. No. 201240

Column: OPTIMA® 5,  
25 m x 0.20 mm ID,  
0.20  $\mu$ m film, REF 726857.25,  
max. temperature 340/360 °C  
Injection: 1.0  $\mu$ l parsol mixture,  
1:1000 in MeOH, split 1:150  
Carrier gas: 25 cm/s He  
Temperature: 120 °C (3 min)  $\xrightarrow{10\text{ °C/min}}$  220 °C  
 $\xrightarrow{20\text{ °C/min}}$  330 °C  
Detector: MSD

**Peaks:**

1. Parsol 5000 = 3-(4'-Methylbenzylidene)camphor
2. Parsol MCX = *p*-Methoxycinnamic acid 2-ethylhexyl ester (*cis* isomer)
3. Parsol MCX (*trans* isomer)



# Food and cosmetic components

## Determination of benzimidazole anthelmintics in meat samples MN Appl. No. 212040

Column: OPTIMA® 1, 10 m x 0.25 mm ID, 0.25 µm film, REF 726050.10,  
max. temperature 340/360 °C

Injection: 1 µl, 270 °C, splitless

Carrier gas: 1 ml/min He

Temperature: 60 °C (0.5 min)  $\xrightarrow{30\text{ °C/min}}$  150 °C  $\xrightarrow{6\text{ °C/min}}$  300 °C

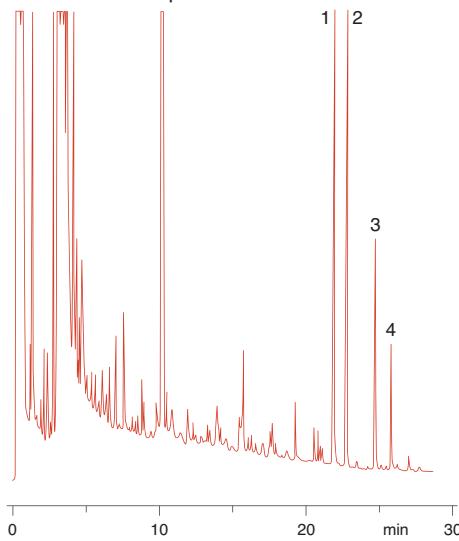
Detector: PND 350 °C

### Chromatogram a)

extract of a kidney sample from a pig treated with fenbendazole, derivatized with pentafluorobenzyl bromide

#### Peaks:

1. and 2. PFB derivatives of fenbendazole
3. and 4. PFB derivatives of the metabolite fenbendazole sulphone

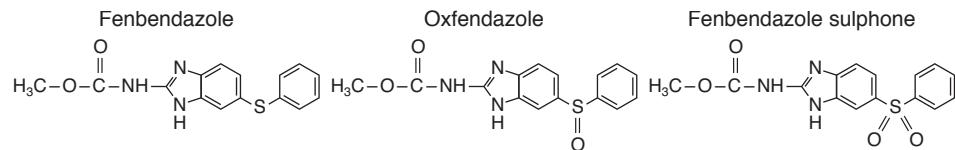
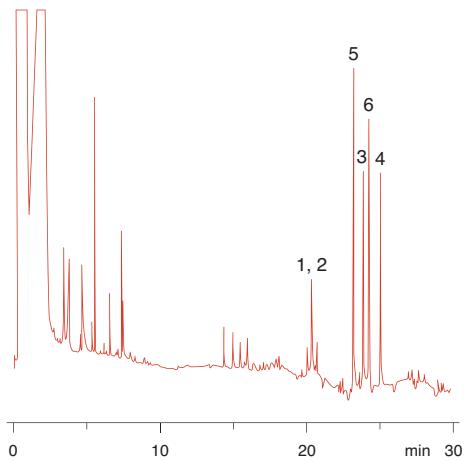


### Chromatogram b)

extract of a blood sample from a pig treated with fenbendazole, derivatized with methyl iodide

#### Peaks:

1. and 2. Me derivatives of fenbendazole
3. and 4. Me derivatives of the metabolite fenbendazole sulphone
5. and 6. Me derivatives of the metabolite oxfendazole



A.M. Marti, A.E. Mooser, H. Koch, J. Chromatography **498** (1990) 145 – 157

## Miscellaneous



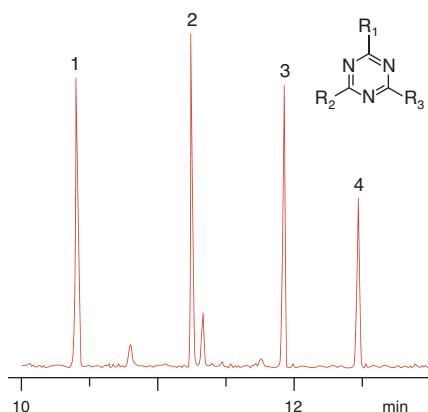
### Determination of melamine, ammeline, ammelide and cyanuric acid in accordance with FDA regulations

*MN Appl. No. 213300*

Column: OPTIMA® 5 MS,  
30 m x 0.25 mm ID,  
0.25  $\mu$ m film, REF 726220.30,  
max. temperature 340/360 °C  
Injection: 1  $\mu$ l, 250 °C, split 10 ml/min  
Carrier gas: 0.5 ml/min He  
Temperature: 75 °C (1 min)  $\xrightarrow{15\text{ °C/min}}$  320 °C  
Detector: MSD

#### Peaks:

1. Cyanuric acid ( $R_1 = R_2 = R_3 = OH$ )
2. Ammelide ( $R_1 = NH_2$ ,  $R_2 = R_3 = OH$ )
3. Ammeline ( $R_1 = R_2 = NH_2$ ,  $R_3 = OH$ )
4. Melamine ( $R_1 = R_2 = R_3 = NH_2$ )



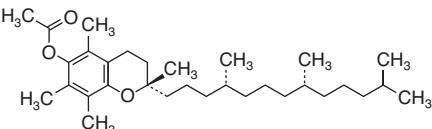
### Analysis of D, L- $\alpha$ -tocopherol acetate (vitamin E acetate)

*MN Appl. No. 210440*

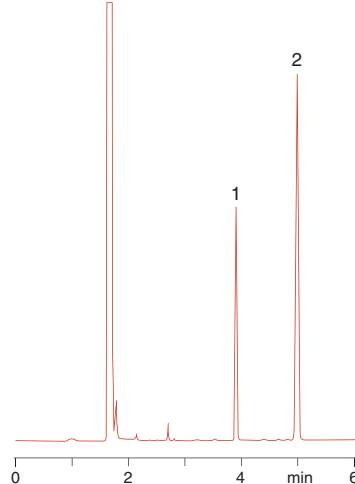
Column: OPTIMA® 1,  
25 m x 0.25 mm ID,  
0.25  $\mu$ m film, REF 726050.25,  
max. temperature 340/360 °C  
Injection: 1  $\mu$ l, 320 °C, split 1:100  
Carrier gas: 10 psi N<sub>2</sub>  
Temperature: 50 °C (3 min)  $\xrightarrow{8\text{ °C/min}}$  290 °C  
Detector: FID 320 °C

#### Peaks:

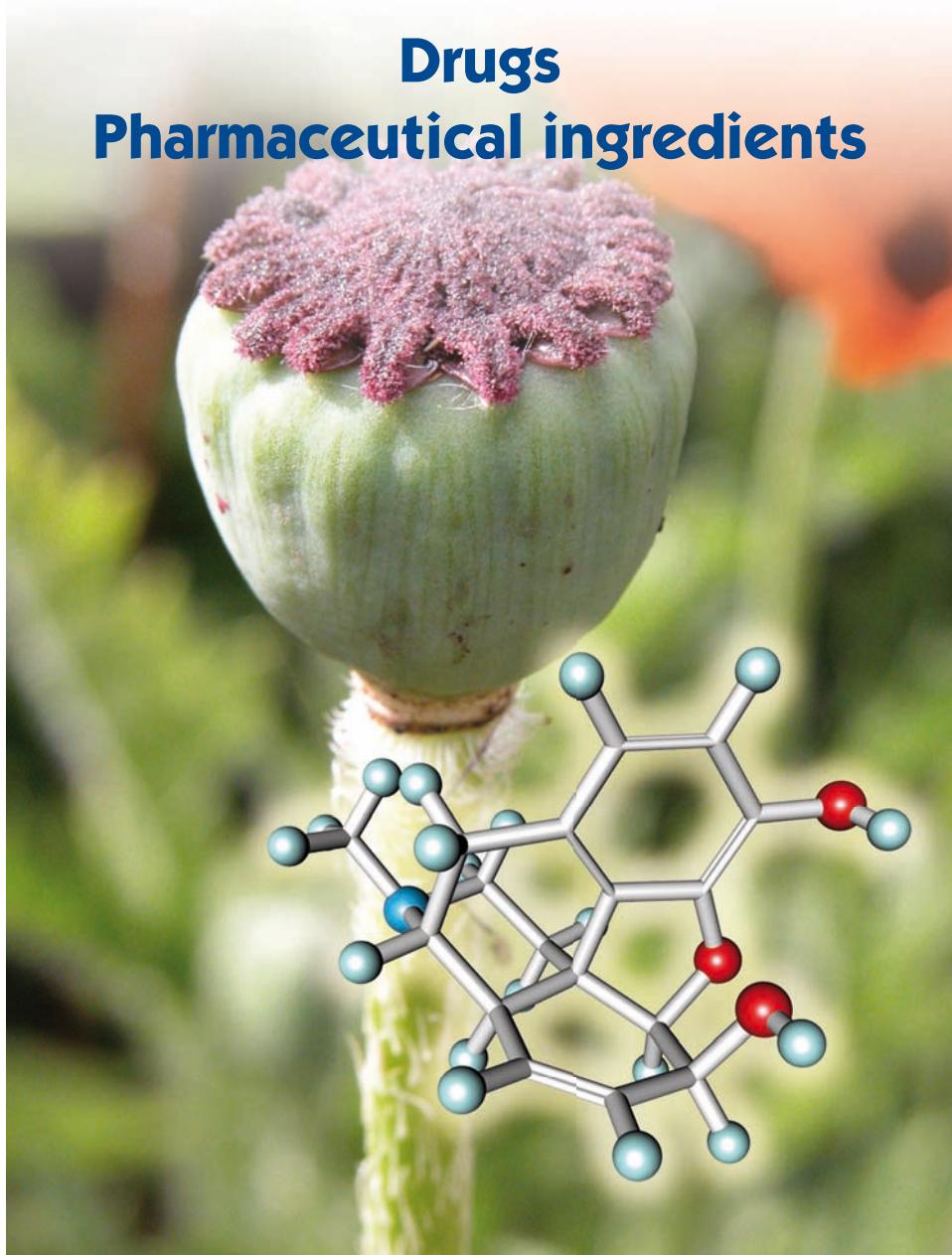
1. D,L- $\alpha$ -Tocopherol acetate [2.5 ng/ml]



2. Tetratriacontane [5.0 ng/ml]



# Drugs Pharmaceutical ingredients



# Drugs of abuse

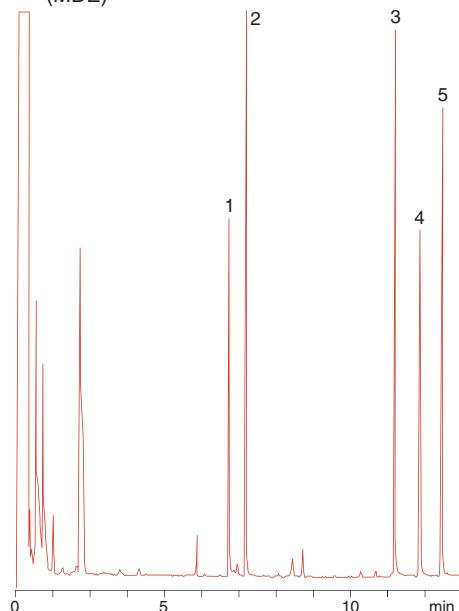


## Analysis of amphetamines MN App. No. 210460

Column: OPTIMA® δ-3,  
30 m x 0.25 mm ID,  
0.25 µm film,  
REF 726420.30,  
max. temperature 340/360 °C  
Injection: 1.0 µl, 20 µg/ml in methanol,  
250 °C, split 10 ml/min  
Carrier gas: 2 ml/min He (5.0)  
Temperature: 60 °C (2 min)  $\xrightarrow{20\text{ °C/min}}$  150 °C  
 $\xrightarrow{6\text{ °C/min}}$  250 °C (5 min)  
Detector: MSD (Ion Trap)

**Peaks:**

1. Amphetamine
2. Methamphetamine
3. 3,4-Methylenedioxymethamphetamine (MDA)
4. 3,4-Methylenedioxymethamphetamine (MDMA)
5. 3,4-Methylenedioxymethylamphetamine (MDE)

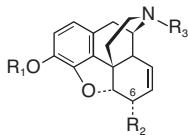


Courtesy of Mr. S. Motsch, Mr. P. Stein, Zollkriminalamt, Köln, Germany

## Structures of selected drugs

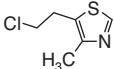
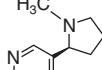
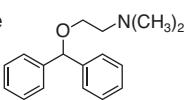
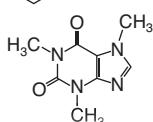
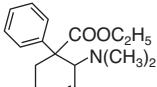
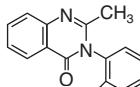
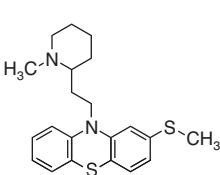
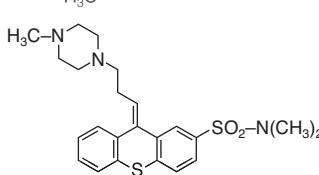
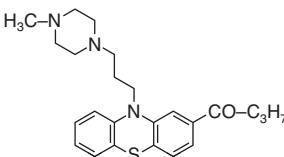
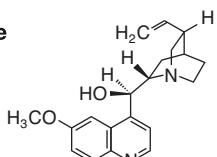
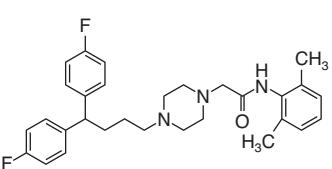
Compound	Structure
Alprazolam	
Midazolam	
Noscapine = Narcotine	
Papaverine	
Meconin	
Methadone	

## Morphine alkaloids



Substance	R <sub>1</sub>	R <sub>2</sub>	R <sub>3</sub>
Morphine	H	OH	CH <sub>3</sub>
Nalorphine	H	OH	CH <sub>2</sub> -CH=CH <sub>2</sub>
Codeine	CH <sub>3</sub>	OH	CH <sub>3</sub>
Heroin	COCH <sub>3</sub>	OCOCH <sub>3</sub>	CH <sub>3</sub>

## Drugs • Pharmaceutical ingredients

Compound	Structure	Compound	Structure
Clomethiazole		Nicotine	
Diphenhydramine		Caffeine	
Tilidine		Methaqualone	
Thioridazine		Thiothixene	
Butaperazine		Quinine	
Lidoflazine			

# Drug screening



## Drug screening analysis MN Appl. No. 201300

Column: OPTIMA® 1, 15 m x 0.53 mm ID, 2.0 µm film, REF 726521.15,  
max. temperature 320/340 °C

Injection: 1 µl in methanol, 0.5 min splitless, 270 °C

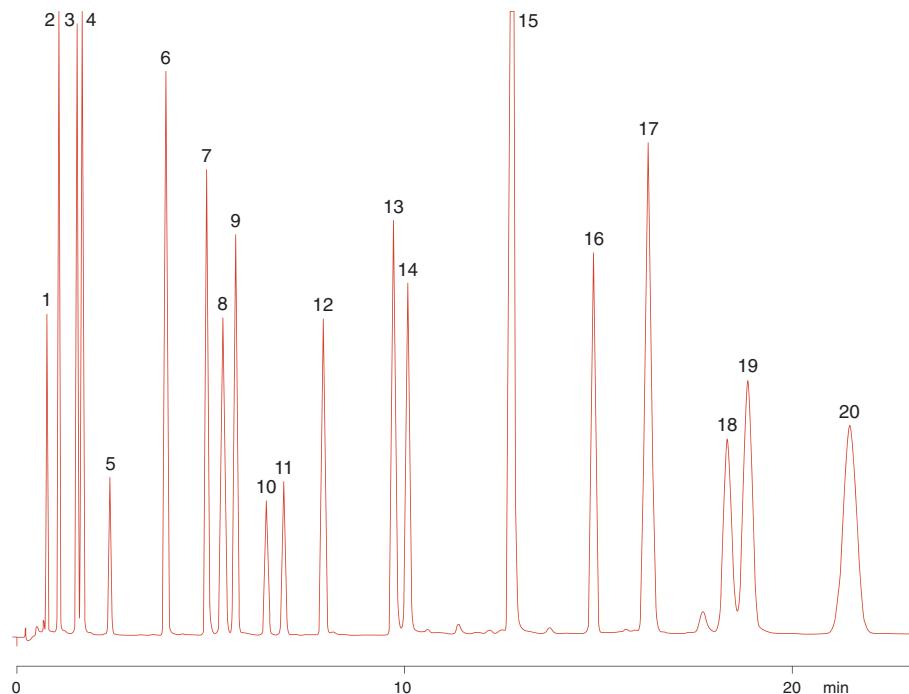
Carrier gas: 10 ml/min H<sub>2</sub> (constant flow)

Temperature: 134 °C  $\xrightarrow{10\text{ °C/min}}$  277 °C

Detector: N-FID 300 °C

### Peaks: [ng]

- |                         |                                                   |
|-------------------------|---------------------------------------------------|
| 1. Amphetamine [50]     | 11. 5-Chloro-2-aminobenzophenone (int. std.) [50] |
| 2. Clomethiazole [50]   | 12. Methaqualone [50]                             |
| 3. Nicotine [20]        | 13. Codeine [100]                                 |
| 4. Ephedrine [100]      | 14. Morphine [100]                                |
| 5. Barbital [100]       | 15. Quinine [200]                                 |
| 6. Phenacetin [100]     | 16. Thioridazine (int. std.) [100]                |
| 7. Caffeine [20]        | 17. Butaperazine [1006}                           |
| 8. Diphenhydramine [50] | 18. Thiothixene } [100]                           |
| 9. Tilidine [100]       | 19. Thiothixene artefact }                        |
| 10. Cyclobarbital (200) | 20. Lidoflazine [100]                             |



Courtesy of Institut für Rechtsmedizin der Heinrich-Heine-Universität, Düsseldorf, Germany

# Drugs • Pharmaceutical ingredients

## Analysis of drugs of abuse MN Appl. No. 211900

**Column:** OPTIMA® 5 MS,  
12 m x 0.2 mm ID,  
0.2 µm film, REF 726210.12,  
max. temperature 340/360 °C

**Injection:** OCI/PTV, split ratio 28

**Carrier gas:** 1.0 ml/min, 57.9 cm/s

**Total flow:** 30.0 ml/min

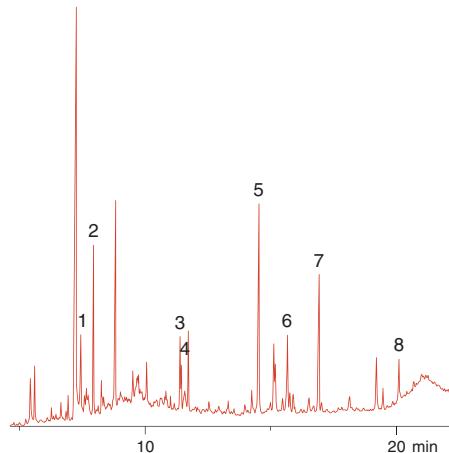
**Pressure:** 58.7 kPa (3 min)  $\xrightarrow{7 \text{ kPa/min}}$   
108 kPa  $\xrightarrow{2.6 \text{ kPa/min}}$  133 kPa  
 $\xrightarrow{8 \text{ kPa/min}}$  150 kPa (4.7 min)

**Temperature:** 70 °C (3 min)  $\xrightarrow{20 \text{ °C/min}}$   
200 °C  $\xrightarrow{7 \text{ °C/min}}$  270 °C  
 $\xrightarrow{20 \text{ °C/min}}$  300 °C (4.7 min)

**Detector:** MSD 280 °C

**Peaks:**

1. Amphetamine
2. Methamphetamine
3. Methadone
4. Methaqualone
5. Codeine
6. Morphine
7. Nalorphine
8. Alprazolam



Courtesy of Mr. Szigan, Laboratorium Lembke-Lempfrid, Köln, Germany

## Analysis of cocaine MN Appl. No. 210020

**Column:** OPTIMA® 1,  
30 m x 0.25 mm ID, 0.25 µm film,  
REF 726050.30,  
max. temperature 340/360 °C

**Sample:** 4 mg of drug mixture dissolved  
in 600 µl CH<sub>2</sub>Cl<sub>2</sub> and derivatised  
with MSTFA (REF 701270.201).

**Injection:** 1 µl

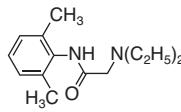
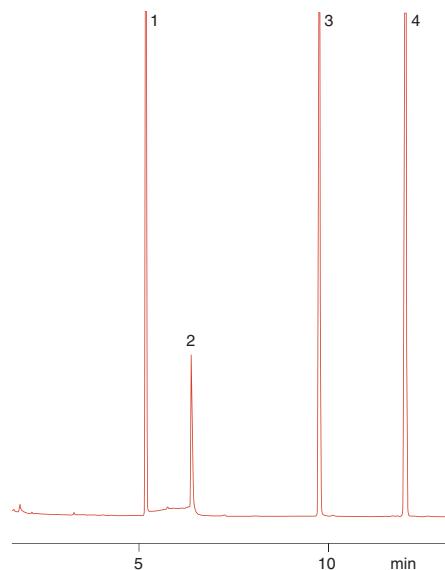
**Carrier gas:** N<sub>2</sub>  $\xrightarrow{7.5 \text{ °C/min}}$  280 °C (4 min)

**Temperature:** 170 °C  $\xrightarrow{20 \text{ °C/min}}$  280 °C (4 min)

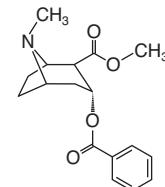
**Detector:** FID 330 °C

**Peaks:**

1. H-Lidocaine, MSTFA derivative
2. N-Lidocaine, MSTFA derivative
3. Cocaine
4. Internal standard



Lidocain



Cocaine

# Drugs of abuse



## Analysis of heroin

MN Appl. No. 210010

Column: OPTIMA® 1,  
30 m x 0.25 mm ID,  
0.25 µm film, REF 726050.30,  
max. temperature 340/360 °C

Sample preparation: 4 mg of a drug mixture are dissolved in 600 µl CH<sub>2</sub>Cl<sub>2</sub> and derivatised with MSTFA (REF 701270.201).

Injection: 1 µl

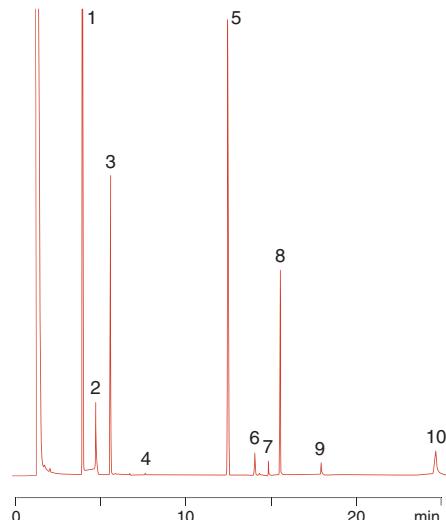
Carrier gas: N<sub>2</sub>

Temperature: 170 °C → 280 °C  
(9.5 min)

Detector: FID 330 °C

### Peaks:

1. Diparacetamol (MSTFA derivative)
2. Monoparacetamol ((MSTFA derivative)
3. Caffeine
4. Glucose (MSTFA derivative)
5. Internal standard
6. Acetylcodeine
7. Acetylmorphine (MSTFA derivative)
8. Diacetylmorphine (heroin)
9. Papaverine
10. Narcotine (MSTFA derivative)



## Analysis of commercial heroin

MN Appl. No. 250360

Column: OPTIMA® 5,  
30 m x 0.20 mm ID,  
0.35 µm film, REF 726860.30,  
max. temperature 340 °C

Injection: 1 µl, 30 s splitless

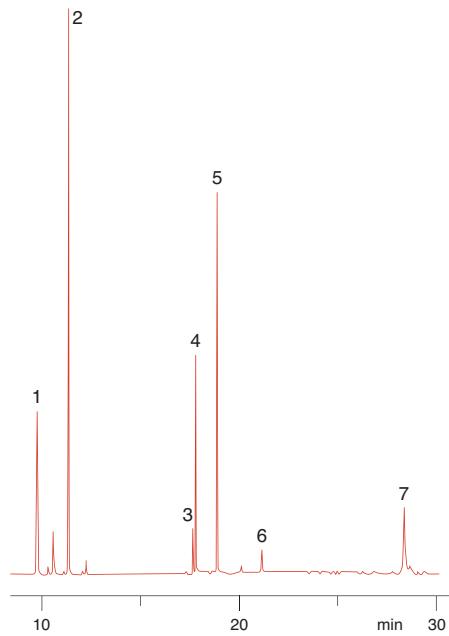
Carrier gas: 30 cm<sup>3</sup>/min He

Temperature: 60 °C (1 min) → 120 °C  
10 °C/min → 280 °C

Detector: MSD

### Peaks:

1. Paracetamol
2. Caffeine
3. Acetylcodeine
4. Monoacetylmorphine
5. Diacetylmorphine (heroin)
6. Papaverine
7. Narcotine



Courtesy of A. Jeger, Gerichtschemisches Institut, Basel, Switzerland

## Drugs • Pharmaceutical ingredients

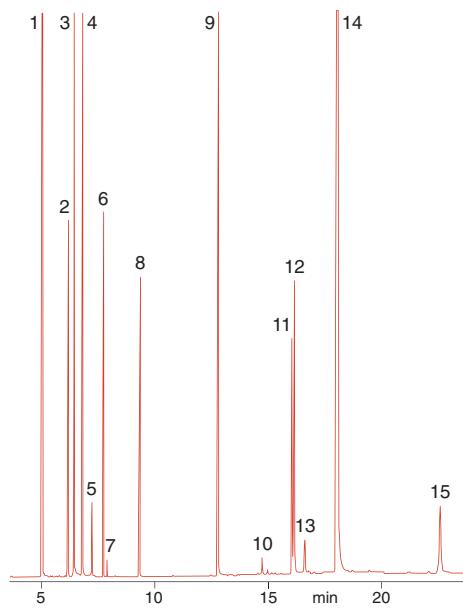
### Analysis of heroin (street quality)

*MN Appl. No. 210470*

Column: OPTIMA® δ-3,  
30 m x 0.25 mm ID,  
0.25 µm film, REF 726420.30,  
max. temperature 340/360 °C  
Sample: 5 mg heroin (street quality) +  
1 mg tetracosane + 1 ml CHCl<sub>3</sub> +  
200 µl pyridine + 150 µl MSTFA  
Injection: 1.0 µl, 250 °C, split 30 ml/min  
Carrier gas: 2 ml/min He (5.0)  
Temperature: 150 °C → 280 °C (25 min)  
Detector: MSD (ion trap)

**Peaks:**

- |                       |                               |
|-----------------------|-------------------------------|
| 1. Paracetamol di-TMS | 10. Morphine di-TMS           |
| 2. Glucose TMS peak 1 | 11. Monoacetyl-morphine TMS   |
| 3. Citric acid 4-TMS  | 12. Acetylcodeine             |
| 4. Glucose TMS peak 2 | 13. Acetylthebaol             |
| 5. Paracetamol TMS    | 14. Diacetylmorphine (heroin) |
| 6. Glucose TMS peak 3 | 15. Papaverine                |
| 7. Meconin            |                               |
| 8. Caffeine           |                               |
| 9. Tetracosane        |                               |



Courtesy of Mr. S. Motsch, Mr. P. Stein,  
Zollkriminalamt, Köln, Germany

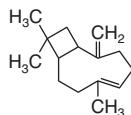
### Analysis of hashish

*MN Appl. No. 212072*

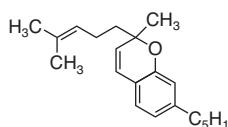
Column: OPTIMA® δ-3,  
60 m x 0.25 mm ID,  
0.25 µm film, REF 726420.60,  
max. temperature 340/360 °C  
Carrier gas: 180 kPa (26 psi) He  
Temperature: 100 °C → 360 °C  
Detector: MSD

**Peaks:**

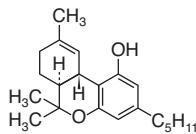
1. Caryophyllene



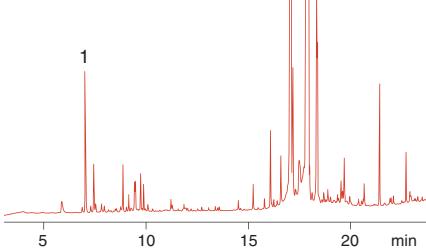
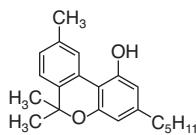
2. Cannabichromene



3. Tetrahydrocannabinol (THC)



4. Cannabinol



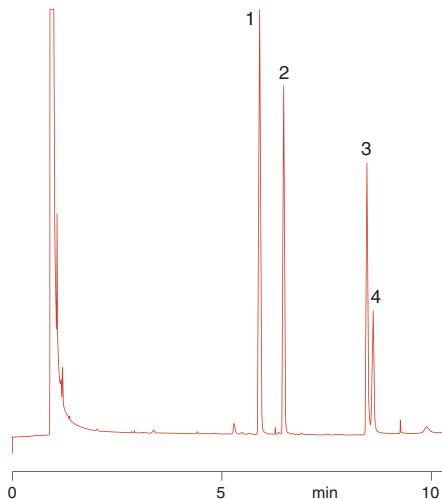
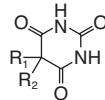
N. Bertram, LTA Labor f. Toxikol. u. Analytik,  
Königswinter, Germany



## Analysis of barbiturates MN Appl. No. 201290

Column: OPTIMA® 5,  
25 m x 0.25 mm ID,  
0.25 µm film, REF 726056.25,  
max. temperature 340/360 °C  
Injection: 1 µl, split 1:10  
Carrier gas: 1.8 bar N<sub>2</sub>  
Temperature: 150 °C → 300 °C  
Detector: FID 280 °C

**Peaks:**  
1. Pentobarbital  
2. Secobarbital  
3. Phenobarbital  
4. Cyclobarbital

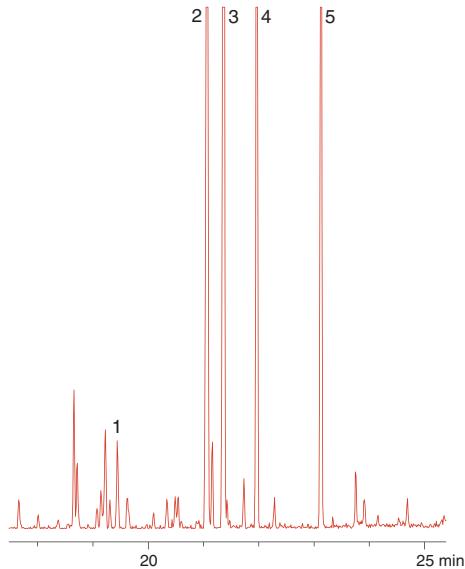


Compound	R <sub>1</sub>	R <sub>2</sub>
Barbital	C <sub>2</sub> H <sub>5</sub>	C <sub>2</sub> H <sub>5</sub>
Pentobarbital	C <sub>2</sub> H <sub>5</sub>	CH(CH <sub>3</sub> )-C <sub>3</sub> H <sub>7</sub>
Secobarbital	CH <sub>2</sub> -CH=CH <sub>2</sub>	CH(CH <sub>3</sub> )-C <sub>3</sub> H <sub>7</sub>
Phenobarbital	C <sub>2</sub> H <sub>5</sub>	C <sub>6</sub> H <sub>5</sub>
Cyclobarbital	C <sub>2</sub> H <sub>5</sub>	

## Analysis of narcotic analgesic drugs in urine MN Appl. No. 201270

Column: OPTIMA® 17,  
25 m x 0.20 mm ID,  
0.20 µm film, REF 726065.25,  
max. temperature 320/340 °C  
Injection: 1.0 µl of a liquid extract of a  
urine sample, 1:10 in MeOH,  
concentration of peaks 2 – 5  
approximately 0.5 µg/ml,  
3 s splitless  
Carrier gas: 25 cm/s He  
Temperature: 130 °C (3 min) → 320 °C  
Detector: MSD

**Peaks:**  
1. Levomethorphan  
2. Dextrorphan  
3. unknown metabolite  
4. Di(2-ethylhexyl) phthalate  
5. unknown metabolite

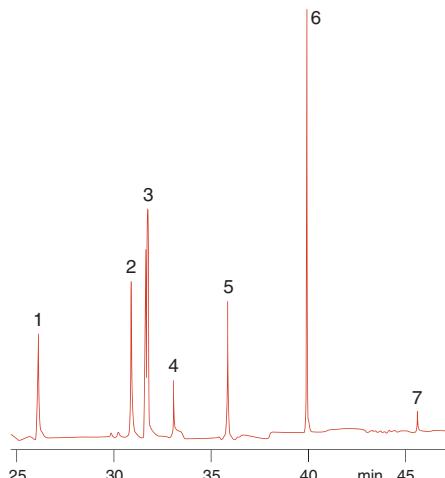


Dextromethorphan and metabolites courtesy of  
Dr. Baumann, Hôpital Prilly, Switzerland

## Drugs • Pharmaceutical ingredients

### Analysis of $\beta$ -agonists from urine MN Appl. No. 210670

Column: OPTIMA®  $\delta$ -3, 30 m x 0.25 mm ID, 0.25  $\mu$ m film, REF 726420.30,  
max. temperature 340/360 °C  
Injection: 1.0  $\mu$ l, 10  $\mu$ g/l from urine, derivatised with HMDS/TMCS 9:1, 280 °C, splitless  
Temperature: 100 °C (1 min)  $\xrightarrow{5\text{ °C/min}}$  300 °C  
Detector: MSD 300 °C  
**Peaks:**  
1. Mabuterol-TMS  
2. Salbutamol-TMS  
3. Clenbuterol-TMS + Clenbuterol-D<sub>6</sub>-TMS  
4. Cimaterol-TMS  
5. Hydroxymethyl-clenbuterol-TMS  
6. Isoxsuprine-TMS  
7. Ractopamine-TMS



Courtesy of Mr. Wohlfarth, Mr. Rosensprung, Staatl. Untersuchungsamt, Wiesbaden, Germany

Structure	Compound	R <sub>1</sub>	R <sub>2</sub>	R <sub>3</sub>	R <sub>4</sub>
<chem>*c1cc(OCC(CN(*)*)C)c(*)cc1</chem>	Mabuterol	CF <sub>3</sub>	NH <sub>2</sub>	Cl	C(CH <sub>3</sub> ) <sub>3</sub>
<chem>*c1cc(Cl)cc(*)cc1</chem>	Clenbuterol	Cl	NH <sub>2</sub>	Cl	C(CH <sub>3</sub> ) <sub>3</sub>
<chem>*c1cc(C#N)cc(*)cc1</chem>	Cimaterol	CN	NH <sub>2</sub>	H	CH(CH <sub>3</sub> ) <sub>2</sub>
<chem>*c1cc(OCC(CN(*)*)C)c(*)cc1</chem>	Salbutamol	CH <sub>2</sub> OH	OH	H	C(CH <sub>3</sub> ) <sub>3</sub>
<chem>Oc1ccc(CC(CN(*)*)C(*)*)cc1</chem>	Ioxsuprine	CH <sub>3</sub>	O-C <sub>6</sub> H <sub>5</sub>	—	—
<chem>Oc1ccc(CC(CN(*)*)C(*)*)cc1</chem>	Ractopamine	H	CH <sub>2</sub> -p-C <sub>6</sub> H <sub>4</sub> -OH	—	—



## Analysis of acidic drugs from waste water MN Appl. No. 212000

Sample preparation: 100  $\mu\text{l}$  sample is derivatised with 30  $\mu\text{l}$  of a 0.2 M methanolic TMSH solution (REF 701520.101).

Column: OPTIMA<sup>®</sup> δ-3, 30 m x 0.25 mm ID, 0.25  $\mu\text{m}$  film, REF 726420.30,

max. temperature 340/360 °C

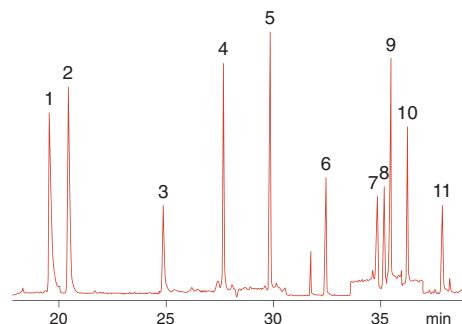
Injection: 2  $\mu\text{l}$  (5  $\mu\text{l}/\text{s}$ ), 275 °C, splitless

Temperature: 70 °C (1 min)  $\xrightarrow{15 \text{ °C/min}}$  280 °C  $\xrightarrow{5 \text{ °C/min}}$  300 °C (10 min)

Detector: MSD

**Peaks:**

1. Clofibric acid
2. Ibuprofen
3. 2,3-Dichlorophenoxyacetic acid (int. std.)
4. Gemfibrozil
5. Fenoprofen
6. Naproxen
7. Ketoprofen
8. Indomethacin
9. Tolfenamic acid
10. Diclofenac
11. Meclofenamic acid



Courtesy of Staatliches Umweltamt Aachen, Germany

Structure	Compound	R <sub>1</sub>	R <sub>2</sub>	R <sub>3</sub>	R <sub>4</sub>
	Clofibric acid	H		Cl	$\text{C}(\text{CH}_3)_2-\text{COOH}$
	Gemfibrozil	$\text{CH}_3$		$\text{CH}_3$	$(\text{CH}_2)_3-\text{C}(\text{CH}_3)_2-\text{COOH}$
	Diclofenac	Cl		H	$\text{CH}_2-\text{COOH}$
	Meclofenamic acid	Cl		$\text{CH}_3$	COOH
	Tolfenamic acid	H		Cl	COOH
	Naproxen	—		—	—

Other formulas see structure index from page 291.

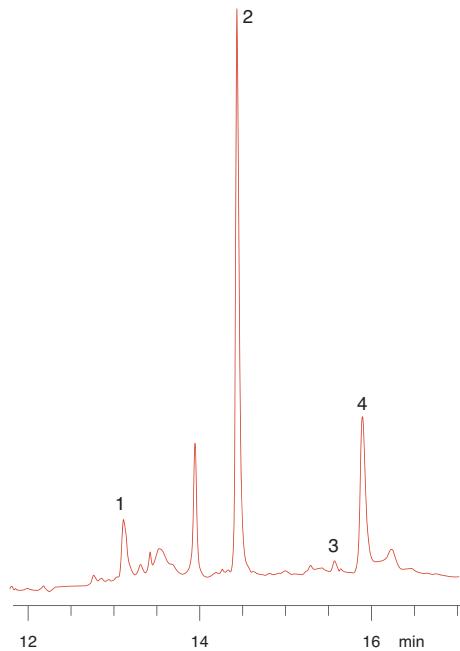
## Drugs • Pharmaceutical ingredients

### Determination of picogram levels of midazolam and metabolites in human plasma

**MN Appl. No. 212580**

Column: OPTIMA® 1,  
25 m x 0.25 mm ID,  
0.25  $\mu$ m film, REF 726050.25,  
max. temperature 340/360 °C  
Injection: 3  $\mu$ l, 300 °C, pulsed splitless  
Carrier gas: 1.1 ml/min He  
(8.6 psi, 43 cm/s)  
Temperature: 85 °C (1 min)  $\xrightarrow[10\text{ °C/min}]{30\text{ °C/min}}$  200 °C  
 $\xrightarrow{310\text{ °C (1 min)}}$   
Detector: MSD

**Peaks:**  
 1. Midazolam  
 2. N-Ethyloxazepam (int. std.)  
 3. 4-Hydroxymidazolam  
 4. 1-Hydroxymidazolam



C. B. Eap et al., J. Chromatography B, **802**  
(2004) 339 – 345

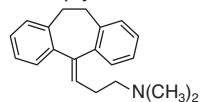
### Analysis of amitriptyline

**MN Appl. No. 210370**

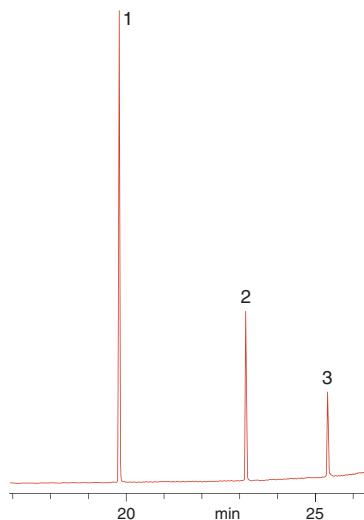
Column: OPTIMA® 5 Amine,  
25 m x 0.20 mm ID,  
0.35  $\mu$ m film, REF 726355.25,  
max. temperature 300/320 °C  
Injection: 310 °C  
Carrier gas: 25 cm/s He  
Temperature: 70 °C (3 min)  $\xrightarrow{10\text{ °C/min}}$  290 °C  
Detector: MSD

**Peaks:**

1. Diphenhydramine
2. Amitriptyline



3. 5-(*p*-Methylphenyl)-5-phenylhydantoin (MPPH)



# Steroids

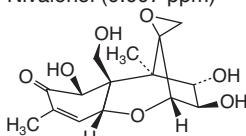


## Analysis of *Fusarium* mycotoxins MN Appl. No. 210660

Column: OPTIMA® 1701,  
25 m x 0.32 mm ID,  
0.35  $\mu$ m film, REF 726824.25,  
max. temperature 300/320 °C  
Injection: 1  $\mu$ L, 250 °C, splitless  
Carrier gas: 30 cm/s N<sub>2</sub>, linear velocity at  
160 °C  
Temperature: 160 °C (3 min)  $\xrightarrow{6\text{ °C/min}}$  240 °C  
 $\xrightarrow{30\text{ °C/min}}$  270 °C (3 min)  
Detector:  $^{63}\text{Ni}$ -ECD 320 °C

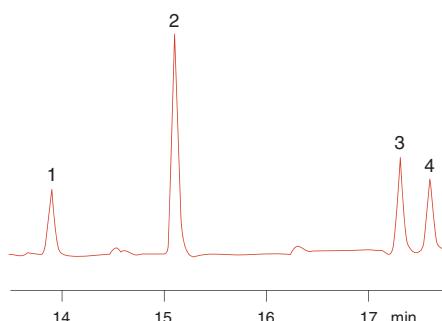
**Peaks:**

1. Nivalenol (0.007 ppm)



2. Deoxynivalenol (0.006 ppm)

3. 15- $\alpha$ -Acetyl-4-deoxynivalenol (0.006 ppm)  
4. 3-Acetyldeoxynivalenol (0.005 ppm)



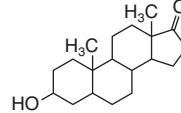
F. Walker, B. Meier, Journal of AOAC Int. Vol. 81, No. 4 (1998) 741 – 748

## Analysis of free steroids MN Appl. No. 201310

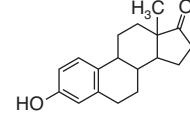
Column: OPTIMA® 17,  
25 m x 0.53 mm ID,  
1.0  $\mu$ m film, REF 726747.25,  
max. temperature 300/320 °C  
Injection: 1  $\mu$ L (0.1 % in CH<sub>2</sub>Cl<sub>2</sub>)  
split 1:30  
Carrier gas: 40 kPa N<sub>2</sub>  
Temperature: 260 °C  
Detector: FID 280 °C

**Peaks:**

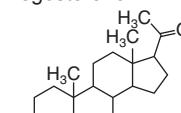
1. Androsterone



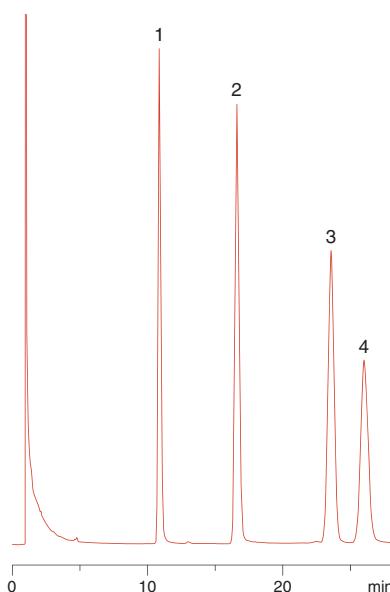
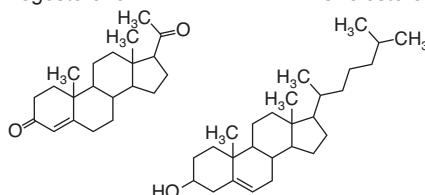
2. Estrone



3. Progesterone



4. Cholesterol



# Drugs • Pharmaceutical ingredients

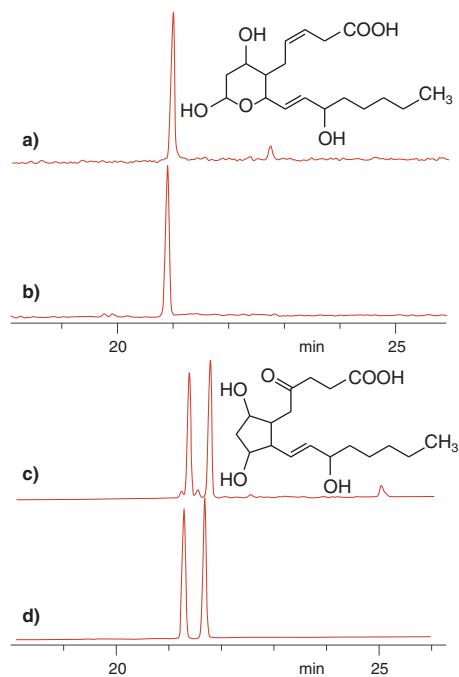
## Quantification of 2,3-dinor-thromboxane B<sub>2</sub> and 2,3-dinor-6-oxo-prostaglandin F<sub>1α</sub> in human urine

**MN Appl. No. 211950**

Column: OPTIMA® 17,  
30 m x 0.25 mm ID,  
0.25 µm film, REF 726022.30,  
max. temperature 320/340 °C  
Injection: 280 °C, splitless  
Carrier gas: 55 kPa He  
Temperature: 70 °C (2 min)  $\xrightarrow[4\text{ °C/min}]{25\text{ °C/min}}$  280 °C  
 $\xrightarrow{} 320\text{ °C}$   
Detector: MSD (quadrupole)

### Chromatograms:

- a) endogenous 2,3-dinorthromboxane B<sub>2</sub>
- b) tetradeuterated 2,3-dinorthromboxane B<sub>2</sub>
- c) endogenous 2,3-dinor-6-oxoprostaglandin F<sub>1α</sub>
- d) tetradeuterated 2,3-dinor-6-oxoprostaglandin F<sub>1α</sub>



D. Tsikas, F.M. Böhme, I. Fuchs, J. Fröhlich, J. Chromatography A, **885** (2000) 351 – 359

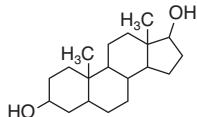
## Analysis of steroids

**MN Appl. No. 250300**

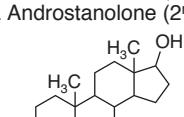
Column: OPTIMA® 5,  
30 m x 0.20 mm ID,  
0.35 µm film, REF 726860.30,  
max. temperature 340/360 °C  
Carrier gas: He  
Temperature: 200 °C (2 min)  $\xrightarrow[3\text{ °C/min}]{3\text{ °C/min}}$  320 °C  
Detector: MSD

### Peaks:

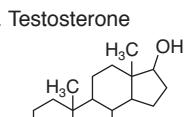
1. Androstanediol



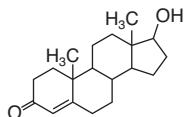
2. Androstanolone (1st isomer)



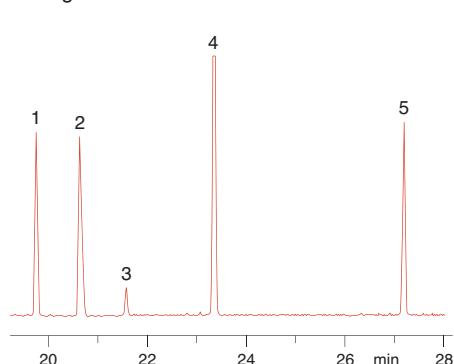
3. Androstanolone (2nd isomer)



4. Testosterone



5. Progesterone





## Separation of endocrinic compounds MN Appl. No. 212620

Column: OPTIMA® δ-6, 30 m x 0.32 mm ID, 0.35 µm film, REF 726481.30,

max. temperature 340/360 °C

Injection: 5.0 µl standards 10 mg/l, nonylphenol (NP) 20 mg/l in methanol, splitless

Carrier gas: 5.0 ml/min H<sub>2</sub>, constant flow

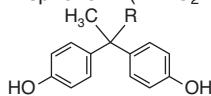
Temperature: 80 °C  $\xrightarrow{12^{\circ}\text{C}/\text{min}}$  305 °C  $\xrightarrow{60^{\circ}\text{C}/\text{min}}$  340 °C

Detector: FID 300 °C

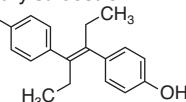
**Peaks:**

1. 4-t-Octylphenol
2. tech. 4-Nonylphenol (NP)
3. Bisphenol A (R = CH<sub>3</sub>)

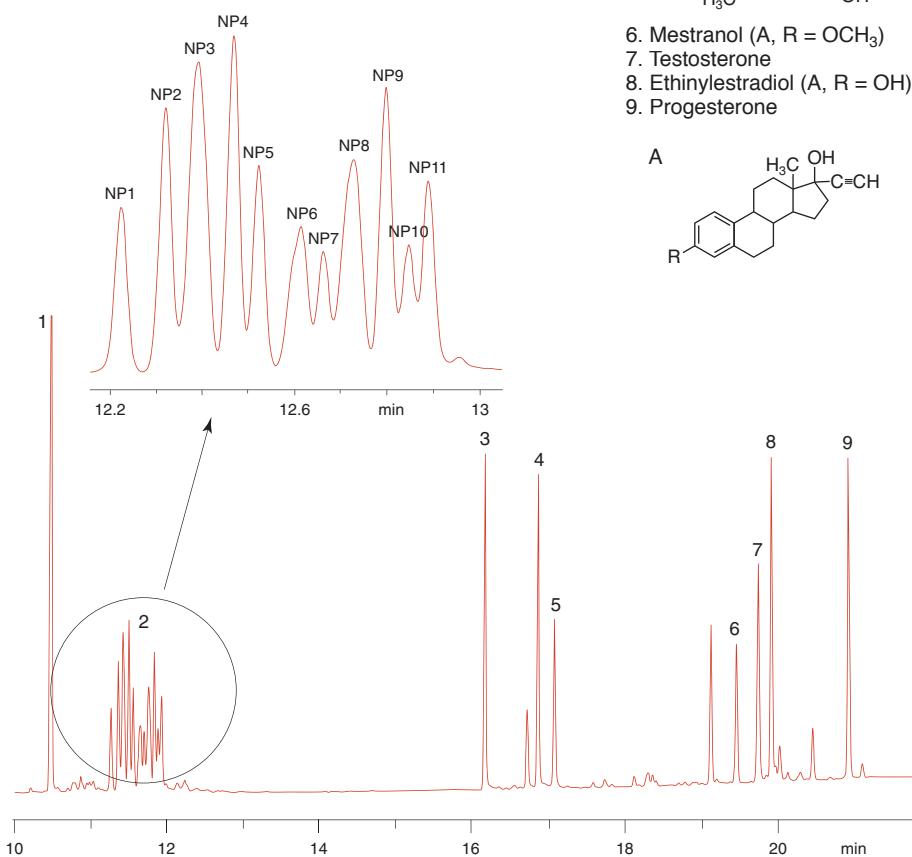
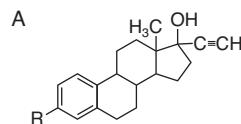
4. Bisphenol B (R = C<sub>2</sub>H<sub>5</sub>)



5. Diethylstilbestrol



6. Mestranol (A, R = OCH<sub>3</sub>)
7. Testosterone
8. Ethinylestradiol (A, R = OH)
9. Progesterone



Courtesy of Mr. Stollenwerk, Inst. f. Chemische Verfahrenstechnik, RWTH Aachen, Germany

# Drugs • Pharmaceutical ingredients

## Separation of dicarboxylic acids for profiling of urinary acidic metabolites

**MN Appl. No. 212590**

Column: OPTIMA® δ-6, 30 m x 0.25 mm ID, 0.25 µm film, REF 726470.30,  
max. temperature 340/360 °C

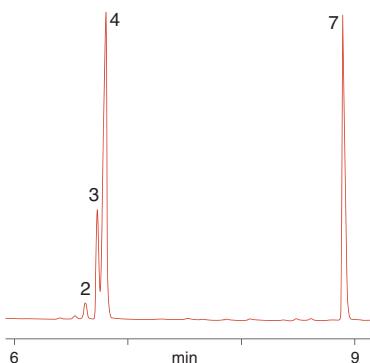
Injection: 2 µl, 220 °C, split 1:10

Carrier gas: 80 kPa H<sub>2</sub>

Temperature: 80 °C → 310 °C

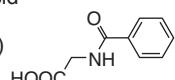
Detector: FID 280 °C

**Sample:** urine of a patient with fumarase enzyme impaired function, derivatised with ethyl chloroformate

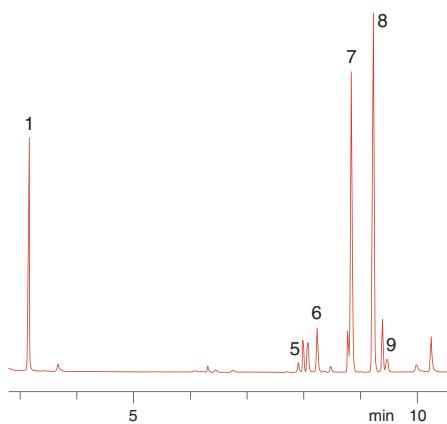


**Peaks:**

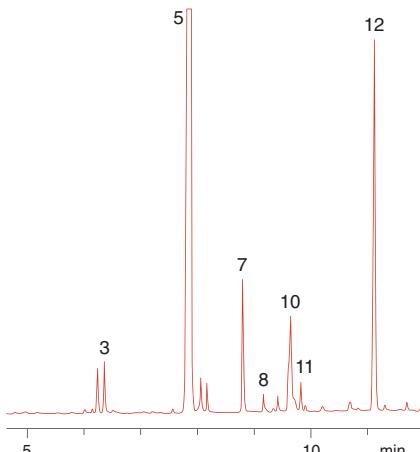
1. 3-Hydroxyisovaleric acid  
(CH<sub>3</sub>)<sub>2</sub>C(OH)-CH<sub>2</sub>-COOH
2. Lactic acid [H<sub>3</sub>C)-CH(OH)-COOH]
3. Succinic acid [HOOC-(CH<sub>2</sub>)<sub>2</sub>-COOH]
4. Fumaric acid [HOOC-CH=CH-COOH]
5. Glutaric acid [HOOC-(CH<sub>2</sub>)<sub>3</sub>-COOH]
6. 3-Methylglutaric acid
7. 2-Phenylbutyric acid (int. std.)
8. 3-Hydroxy-3-methylglutaric acid
9. Adipic acid [HOOC-(CH<sub>2</sub>)<sub>4</sub>-COOH]
10. 3-Hydroxyglutaric acid
11. 2-Oxoglutaric acid
12. Hippuric acid  
(benzoylglycine)



**Sample:** urine of a patient with 3-hydroxy-3-methylglutaric acidurea, derivatised with ethyl chloroformate



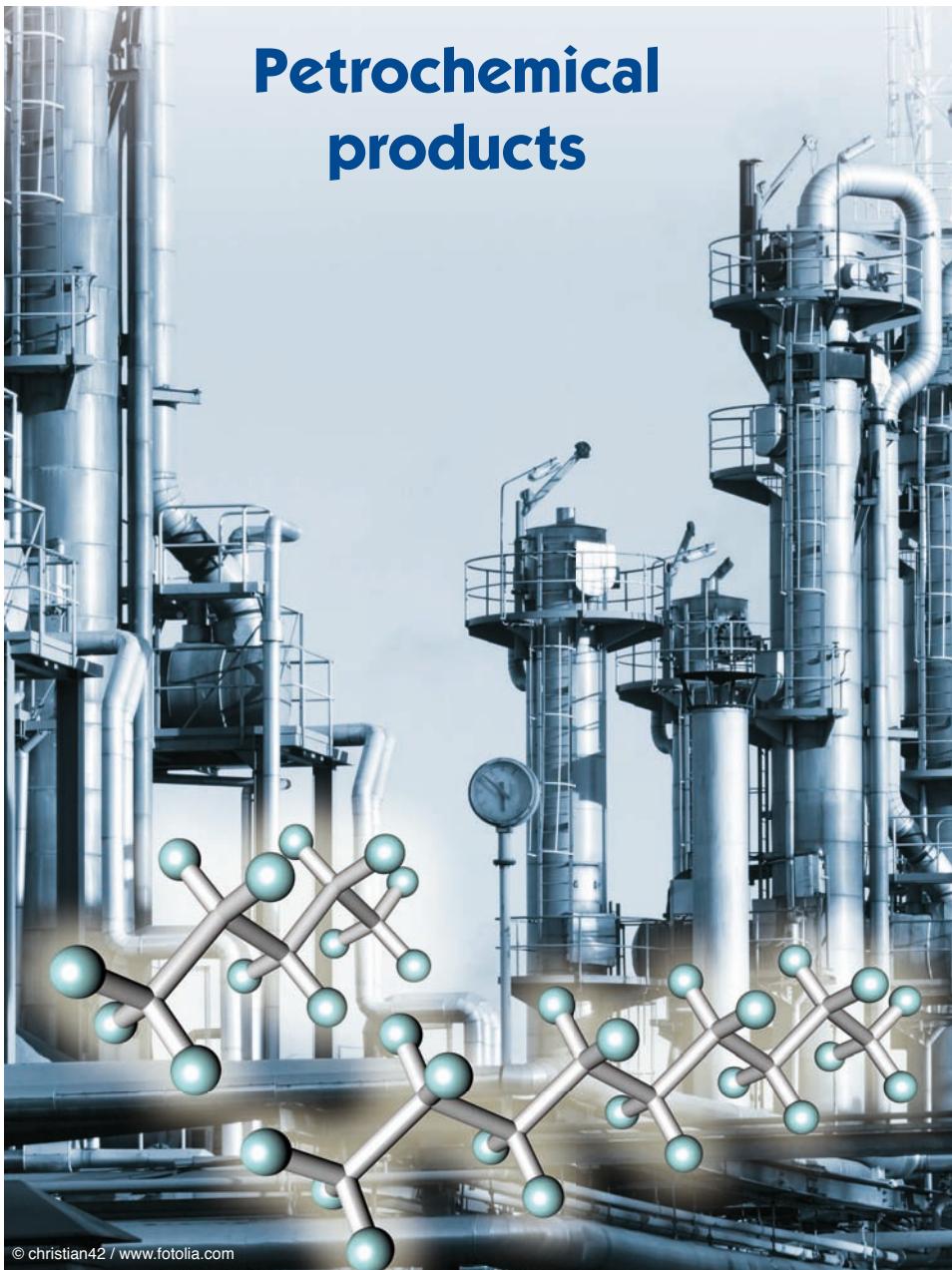
**Sample:** urine of a patient with glutaric acidurea of the I. type, derivatised with ethyl chloroformate



P. Hušek et al., Chromatographia 58 (2003) 623 – 630



## Petrochemical products

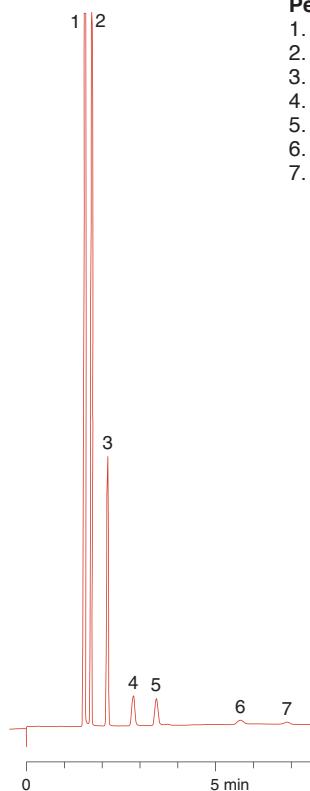


© christian42 / www.fotolia.com

## Petrochemical products

### Analysis of hydrocarbons C<sub>1</sub> – C<sub>5</sub> MN Appl. No. 200030

Column: OPTIMA® 5,  
15 m x 0.32 mm ID,  
5.0  $\mu$ m film, REF 726934.15,  
max. temperature 300/320 °C  
Injection: 100  $\mu$ l, split 60 ml/min  
Carrier gas: 0.08 bar N<sub>2</sub>  
Temperature: 36 °C  
Detector: FID

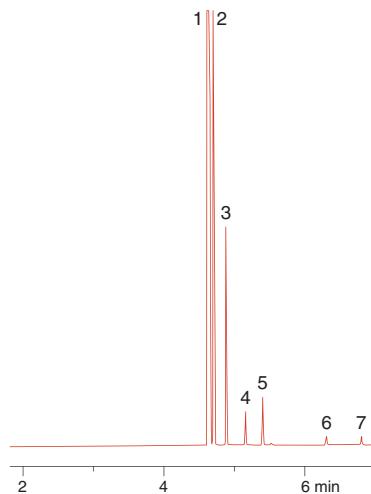


- Peaks:**  
1. Methane  
2. Ethane  
3. Propane  
4. *i*-Butane  
5. Butane  
6. Methylbutane  
7. Pentane

### Analysis of hydrocarbons C<sub>1</sub> – C<sub>5</sub> MN Appl. No. 200041

Column: PERMABOND® P-100,  
100 m x 0.25 mm ID,  
0.5  $\mu$ m film, REF 723890.100,  
max. temperature 300/320 °C  
Injection: 100  $\mu$ l, split 200 ml/min  
Carrier gas: 200 kPa H<sub>2</sub> (2.3 ml/min)  
Temperature: 31 °C  
Detector: FID 250 °C

- Peaks:**  
1. Methane  
2. Ethane  
3. Propane  
4. *i*-Butane  
5. Butane  
6. Methylbutane  
7. Pentane



# Hydrocarbons

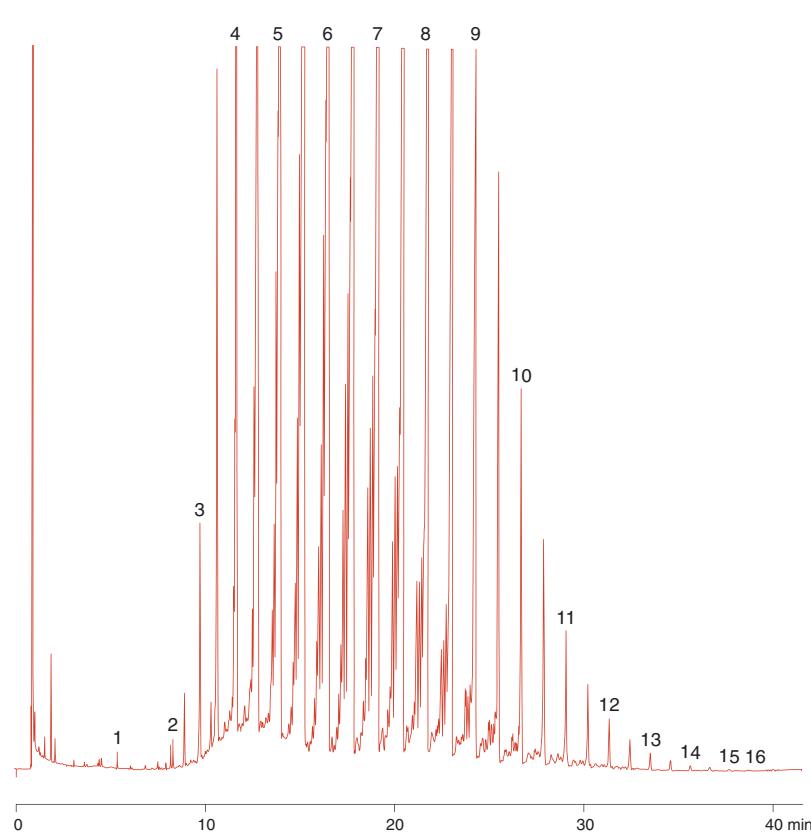


## Analysis of tea warmer candle MN Appl. No. 200050

Column: OPTIMA® 1-TG, 25 m x 0.32 mm ID, REF 726132.25, max. temperature: 370 °C  
Injection: 1.0  $\mu$ l, split 100 ml/min  
Carrier gas: 50 kPa H<sub>2</sub> (3.5 ml/min)  
Temperature: 80 °C (1 min)  $\xrightarrow{20\text{ °C/min}}$  200 °C  $\xrightarrow{5\text{ °C/min}}$  370 °C (5 min)  
Detector: FID 380 °C, baseline corrected

**Peaks:**

- 1. C14
- 2. C18
- 3. C20
- 4. C22
- 5. C24
- 6. C26
- 7. C28
- 8. C30
- 9. C32
- 10. C34
- 11. C36
- 12. C38
- 13. C40
- 14. C42
- 15. C44
- 16. C45



## Petrochemical products

### Analysis of hydrocarbons acc. to ISO DIS 9377-4 (H-53) MN Appl. No. 210600

Sample: standard (1 mg/l) extracted with 50:900 ml *n*-hexane:H<sub>2</sub>O, pH 2 with H<sub>2</sub>SO<sub>4</sub>, (extraction solvent *n*-hexane with *n*-C10 and *n*-C40 standard), then evaporated with N<sub>2</sub> to 1 ml

Column: OPTIMA® 1, 25 m x 0.32 mm ID, 0.25 µm film, REF 726302.25, max. temperature 340/360 °C

Injection: 1.0 µl on column

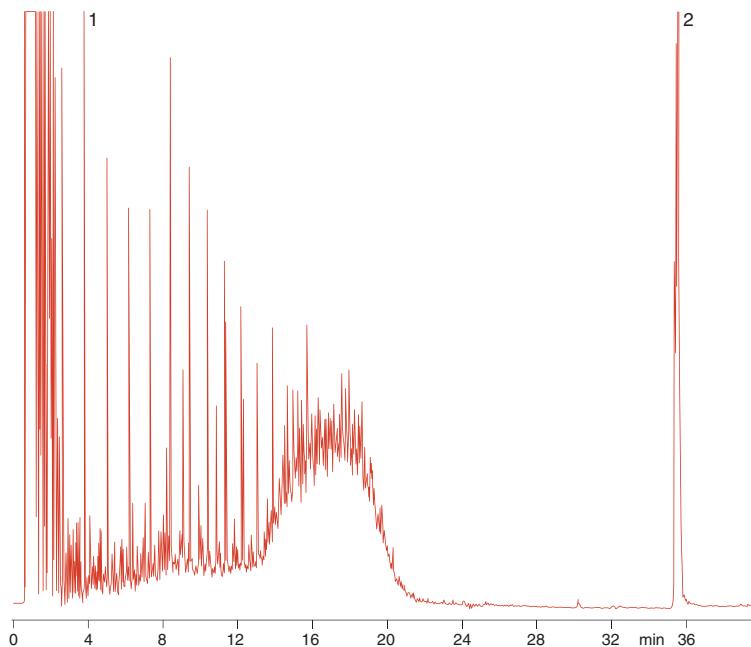
Carrier gas: 3 ml/min H<sub>2</sub> 15 °C/min

Temperature: 60 °C (1 min) → 350 °C (19 min)

Detector: FID (30:300:40 ml/min H<sub>2</sub>:O<sub>2</sub>:He)

**Peaks:**

1. *n*-C<sub>10</sub>
2. *n*-C<sub>40</sub>



For a sample chromatogram of a mixture of diesel and heavy oil see application 210600 at [www.mn-net.com](http://www.mn-net.com)

Courtesy of Mr. W. Elling, INFU, Dortmund, Germany

# Hydrocarbons

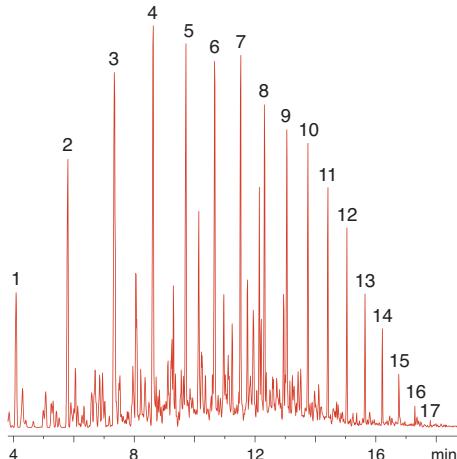


## Analysis of commercial gas oil MN Appl. No. 200060

Column: OPTIMA® 17,  
25 m x 0.20 mm ID,  
0.2 µm film, REF 726065.25,  
max. temperature 320/340 °C  
Injection: 2.0 µl gas oil, diluted 1:100 in  
MeOH, split 1:120  
Carrier gas: 25 cm/s He       $\frac{15 \text{ °C/min}}{\text{80 } \text{°C (4 min)}}$       320 °C  
Temperature: 80 °C (4 min)  $\xrightarrow{15 \text{ °C/min}}$  320 °C  
Detector: MSD

**Peaks:**

- |        |         |
|--------|---------|
| 1. C10 | 10. C19 |
| 2. C11 | 11. C20 |
| 3. C12 | 12. C21 |
| 4. C13 | 13. C22 |
| 5. C14 | 14. C23 |
| 6. C15 | 15. C24 |
| 7. C16 | 16. C25 |
| 8. C17 | 17. C26 |
| 9. C18 |         |



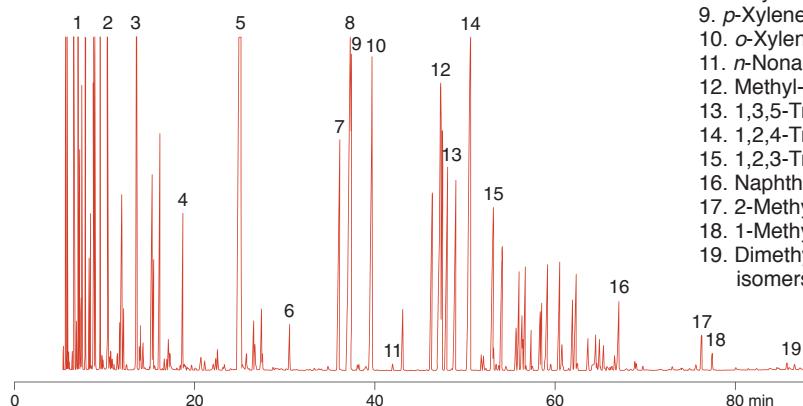
## Analysis of unleaded gasoline

MN Appl. No. 200071

Column: PERMABOND® P-100, 100 m x 0.25 mm ID,  
REF 726890.100, max. temperature 300/320 °C  
Injection: 0.5 µl, split 70 ml/min  
Carrier gas: 200 kPa H<sub>2</sub> (2.3 ml/min)  
 $\frac{2 \text{ °C/min}}{\text{35 } \text{°C (15 min)}}$       200 °C (5 min)  
Temperature:  
Detector: FID 250 °C

**Peaks:**

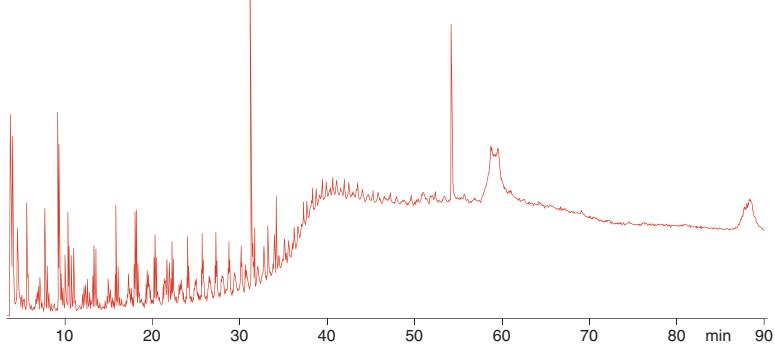
- |                                    |
|------------------------------------|
| 1. n-Pentane                       |
| 2. n-Hexane                        |
| 3. Benzene                         |
| 4. n-Heptane                       |
| 5. Toluene                         |
| 6. n-Octane                        |
| 7. Ethylbenzene                    |
| 8. m-Xylene                        |
| 9. p-Xylene                        |
| 10. o-Xylene                       |
| 11. n-Nonane                       |
| 12. Methyl-3-ethylbenzene          |
| 13. 1,3,5-Trimethylbenzene         |
| 14. 1,2,4-Trimethylbenzene         |
| 15. 1,2,3-Trimethylbenzene         |
| 16. Naphthalene                    |
| 17. 2-Methylnaphthalene            |
| 18. 1-Methylnaphthalene            |
| 19. Dimethylnaphthalene<br>isomers |



## Petrochemical products

### Pyrolysis GC of polyethylene and polypropylene MN Appl. No. 210030

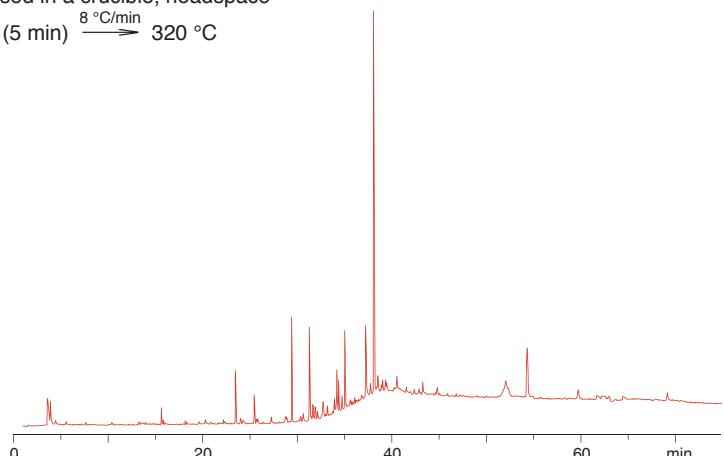
Column: OPTIMA® 5 MS, 60 m x 0.32 mm ID, 1 µm film, REF 726212.60,  
max. temperature 340/360 °C  
Injection: 270 µg solid, headspace  
Temperature: 60 °C (5 min)  $\xrightarrow{8\text{ °C/min}}$  320 °C  
Detector: MSD



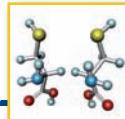
Courtesy of Mrs. Engel, VW, Wolfsburg, Germany

### Pyrolysis GC of chloroprene rubber MN Appl. No. 210040

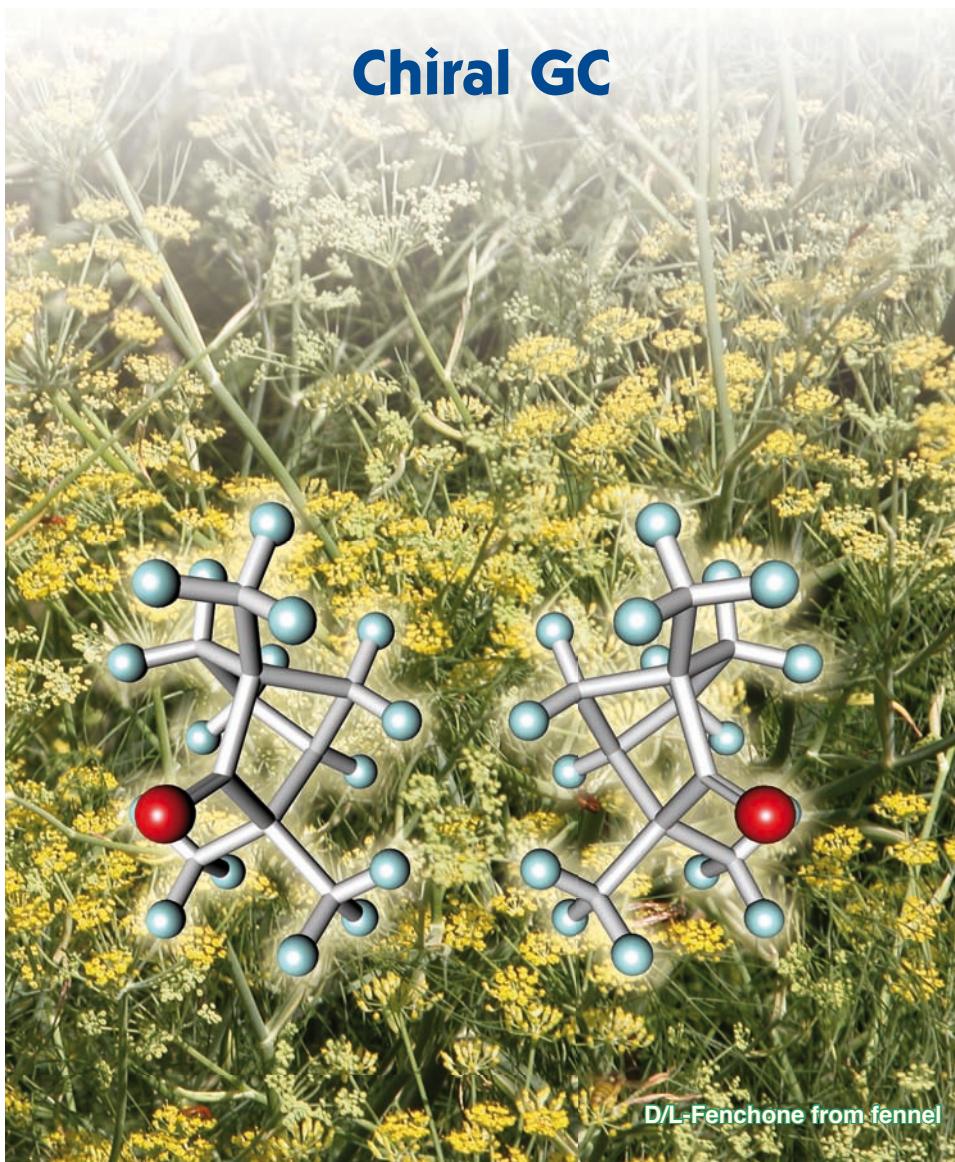
Column: OPTIMA® 5 MS, 60 m x 0.32 mm ID, 1 µm film, REF 726212.60,  
max. temperature 340/360 °C  
Injection: 1 g solid is dissolved in 10 ml of hexane or methanol, 100 µl of this solution are  
pyrolysed in a crucible, headspace  
Temperature: 60 °C (5 min)  $\xrightarrow{8\text{ °C/min}}$  320 °C  
Detector: MSD



Courtesy of Mrs. Engel, VW, Wolfsburg, Germany



# Chiral GC



Please note: enantiomer separations have been arranged by increasing molecular size. Several chromatograms show separations of homologous series; they are listed according to the smallest molecule of the mixture. Related molecules or families of compounds may be found on earlier or later pages depending on size. Specific compounds can be found in the chromatogram index from page 276.

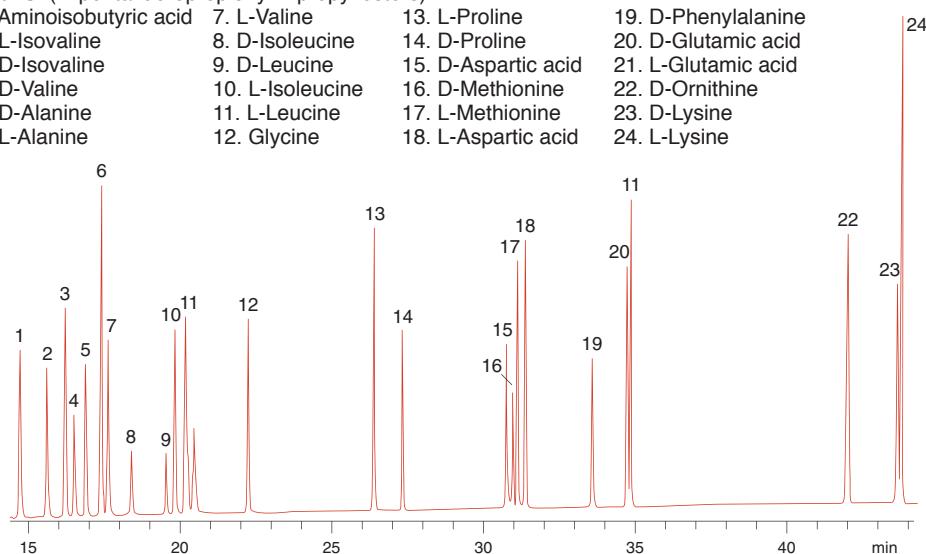
# Chiral separations

## Analysis of amino acid enantiomers MN Appl. No. 212761

Column: FS-LIPODEX® E, 25 m x 0.25 mm ID, REF 723368.25, max. temp. 200/220 °C  
 Injection: 250 °C  
 Carrier gas: 1.2 ml/min He, purge flow 65.9 ml/min  
 Temperature: 60 °C (5 min)  $\xrightarrow{3\text{ °C/min}}$  185 °C (15 min)  
 Detector: MSD 280 °C

**Peaks:** (N-pentafluoropropionyl 2-propyl esters)

- |                         |                  |                     |                     |
|-------------------------|------------------|---------------------|---------------------|
| 1. Aminoisobutyric acid | 7. L-Valine      | 13. L-Proline       | 19. D-Phenylalanine |
| 2. L-Isovaline          | 8. D-Isoleucine  | 14. D-Proline       | 20. D-Glutamic acid |
| 3. D-Isovaline          | 9. D-Leucine     | 15. D-Aspartic acid | 21. L-Glutamic acid |
| 4. D-Valine             | 10. L-Isoleucine | 16. D-Methionine    | 22. D-Ornithine     |
| 5. D-Alanine            | 11. L-Leucine    | 17. L-Methionine    | 23. D-Lysine        |
| 6. L-Alanine            | 12. Glycine      | 18. L-Aspartic acid | 24. L-Lysine        |

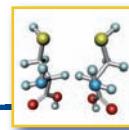


O. Vandenabeele-Trambouze et al., Chromatographia 53 (2001) Suppl., S-332 – S-339

### Summary of important amino acids

Formula	Common name	Systematic name	Structure
C <sub>2</sub> H <sub>5</sub> NO <sub>2</sub>	glycine	aminoethanoic acid	NH <sub>2</sub> -CH <sub>2</sub> -COOH
C <sub>3</sub> H <sub>7</sub> NO <sub>2</sub>	alanine	2-aminoopropanoic acid	CH <sub>3</sub> -CH(NH <sub>2</sub> )-COOH
C <sub>4</sub> H <sub>7</sub> NO <sub>4</sub>	aspartic acid	2-aminobutanedioic acid	HOOC-CH <sub>2</sub> -CH(NH <sub>2</sub> )-COOH
C <sub>4</sub> H <sub>9</sub> NO <sub>2</sub>	aminoisobutyric acid	2-amino-2-methylpropanoic acid	(CH <sub>3</sub> ) <sub>2</sub> C(NH <sub>2</sub> )-COOH
C <sub>5</sub> H <sub>9</sub> NO <sub>2</sub>	proline	pyrrolidine-2-carboxylic acid	
C <sub>5</sub> H <sub>9</sub> NO <sub>4</sub>	glutamic acid	2-aminopentanedioic acid	HOOC-(CH <sub>2</sub> ) <sub>2</sub> -CH(NH <sub>2</sub> )-COOH
C <sub>5</sub> H <sub>11</sub> NO <sub>2</sub>	valine	2-amino-3-methylbutanoic acid	(CH <sub>3</sub> ) <sub>2</sub> CH-CH(NH <sub>2</sub> )-COOH
C <sub>5</sub> H <sub>11</sub> NO <sub>2</sub>	isovaline	2-amino-2-methylbutanoic acid	H <sub>5</sub> C <sub>2</sub> -C(CH <sub>3</sub> )(NH <sub>2</sub> )-COOH
C <sub>5</sub> H <sub>11</sub> NO <sub>2</sub> S	methionine	2-amino-4-(methylsulfanyl)-butanoic acid	CH <sub>3</sub> -S-(CH <sub>2</sub> ) <sub>2</sub> -CH(NH <sub>2</sub> )-COOH
C <sub>5</sub> H <sub>12</sub> N <sub>2</sub> O <sub>2</sub>	ornithine	2,5-diaminopentanoic acid	H <sub>2</sub> N-(CH <sub>2</sub> ) <sub>3</sub> -CH(NH <sub>2</sub> )-COOH
C <sub>6</sub> H <sub>13</sub> NO <sub>2</sub>	leucine	2-amino-4-methylpentanoic acid	(CH <sub>3</sub> ) <sub>2</sub> CH-CH <sub>2</sub> -CH(NH <sub>2</sub> )-COOH
C <sub>6</sub> H <sub>13</sub> NO <sub>2</sub>	isoleucine	2-amino-3-methylpentanoic acid	C <sub>2</sub> H <sub>5</sub> -CH(CH <sub>3</sub> )-CH(NH <sub>2</sub> )-COOH
C <sub>6</sub> H <sub>14</sub> N <sub>2</sub> O <sub>2</sub>	lysine	2,6-diaminohexanoic acid	H <sub>2</sub> N-(CH <sub>2</sub> ) <sub>4</sub> -CH(NH <sub>2</sub> )-COOH
C <sub>9</sub> H <sub>11</sub> NO <sub>2</sub>	phenylalanine	2-amino-3-phenyl-propanoic acid	C <sub>6</sub> H <sub>5</sub> -CH <sub>2</sub> -CH(NH <sub>2</sub> )-COOH

# Compounds C<sub>2</sub> and greater

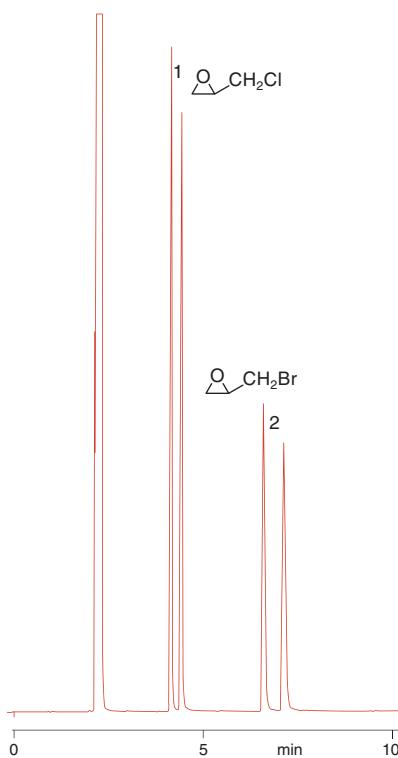


## Enantiomer separation of epichloro- and epibromohydrin MN Appl. No. 202621

Column: FS-LIPODEX® A,  
50 m x 0.25 mm ID,  
REF 723360.50,  
max. temperature 200/220 °C  
Injection: 0.5 µl (1% in CH<sub>2</sub>Cl<sub>2</sub>)  
split 245 ml/min  
Carrier gas: 110 kPa H<sub>2</sub> (2.3 ml/min)  
Temperature: 65 °C  
Detector: FID 240 °C

### Peaks:

1. Epichlorohydrin (C<sub>3</sub>H<sub>5</sub>ClO)
2. Epibromohydrin (C<sub>3</sub>H<sub>5</sub>BrO)



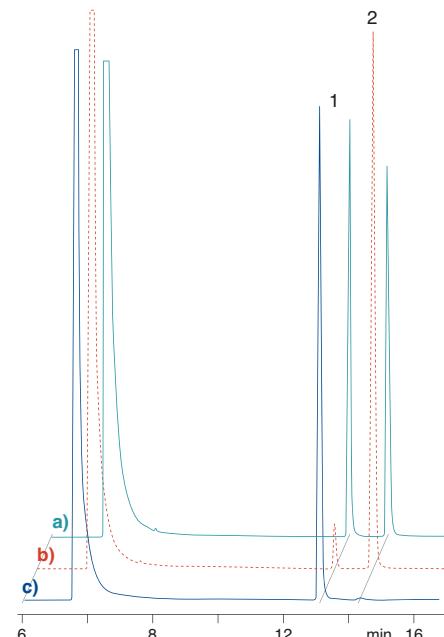
## Enantiomer separation of epichlorohydrin MN Appl. No. 212880

Column: FS-LIPODEX® A,  
50 m x 0.25 mm ID,  
REF 723360.50,  
max. temperature 200/220 °C  
Sample: compounds dissolved in MeOH (1:100)  
Injection: 0.2 µl sample + 0.3 µl air, split 80 ml/min, 230 °C  
Carrier gas: 85 kPa N<sub>2</sub>  
Temperature: 50 °C  
Detector: FID 250 °C

- a) racemate (S/R 50:50)
- b) S/R 2:98
- c) S/R 99:1

### Peaks:

1. S-Epichlorohydrin (C<sub>3</sub>H<sub>5</sub>ClO)
2. R-Epichlorohydrin



Courtesy of B. Mischke, Schulung und Chromatographie, Berlin (Germany)

## Chiral separations

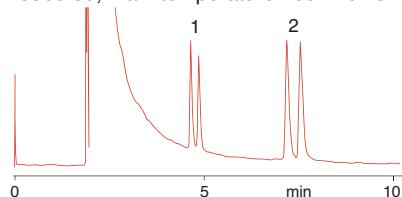
### Enantiomer separation of propane-1,2-diol and butane-1,3-diol (TFA) MN Appl. No. 202881

Column: LIPODEX® D, 25 m x 0.25 mm ID, REF 723366.50, max. temperature 200/220 °C  
 Carrier gas: 0.7 bar H<sub>2</sub>  
 Temperature: 105 °C  
 Detector: FID

**Peaks:**

1. Propane-1,2-diol (C<sub>3</sub>H<sub>8</sub>O<sub>2</sub>)
2. Butane-1,3-diol (C<sub>4</sub>H<sub>10</sub>O<sub>2</sub>)

W.A. König et al., HRC 11 (1988) 506 – 509

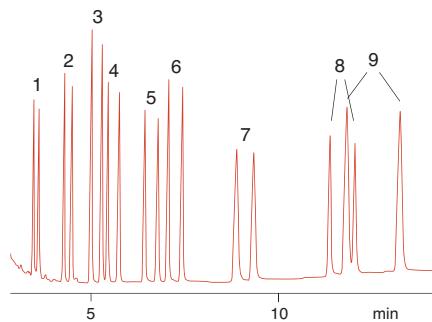


### Enantiomer separation of chiral amines and amino alcohols (N,O-TFA) MN Appl. No. 202421

Column: LIPODEX® D, 50 m x 0.25 mm ID, REF 723366.50, max. temperature 200/220 °C  
 Carrier gas: 1 bar H<sub>2</sub>  
 Temperature: 140 °C → 170 °C  
 Detector: FID

**Peaks:** R enantiomers are eluted first.
 

1. 2-Aminopentane (C<sub>5</sub>H<sub>13</sub>N)
2. 2-Aminohexane (C<sub>6</sub>H<sub>15</sub>N)
3. 2-Amino-5-methylhexane (C<sub>7</sub>H<sub>17</sub>N)
4. 2-Aminoheptane (C<sub>7</sub>H<sub>17</sub>N)
5. 2-Amino-6-methylheptane (C<sub>8</sub>H<sub>19</sub>N)
6. 2-Aminooctane (C<sub>8</sub>H<sub>19</sub>N)
7. Valinol (C<sub>5</sub>H<sub>13</sub>NO)
8. Phenylethylamine (C<sub>8</sub>H<sub>11</sub>N)
9. Alaninol (C<sub>3</sub>H<sub>9</sub>NO)



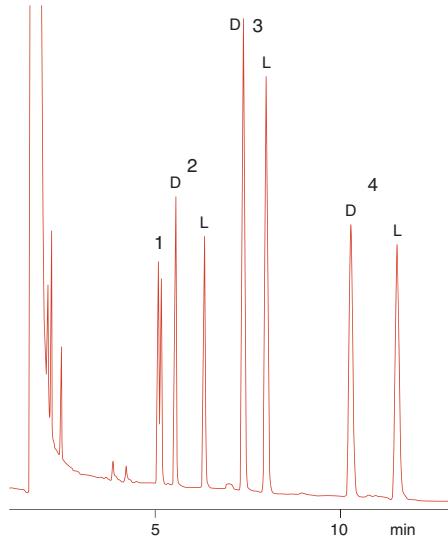
W.A. König et al., HRC 11 (1988) 506 – 509

### Enantiomer separation of amino alcohols (N,O-TFA) MN Appl. No. 202431

Column: LIPODEX® B, 50 m x 0.25 mm ID, REF 723362.50, max. temperature 200/220 °C  
 Carrier gas: H<sub>2</sub>  
 Temperature: 150 °C  
 Detector: FID

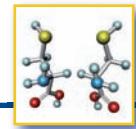
**Peaks:**

1. 1-Aminopropan-2-ol (C<sub>3</sub>H<sub>9</sub>NO)
2. Alaninol = 2-aminopropan-1-ol (C<sub>3</sub>H<sub>9</sub>NO)
3. 2-Aminobutanol (C<sub>4</sub>H<sub>11</sub>NO)
4. Leucinol (C<sub>6</sub>H<sub>15</sub>NO)



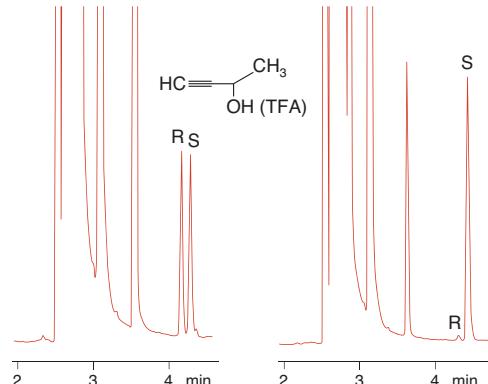
Courtesy of Prof. W.A. König, Hamburg, Germany

## Compounds C<sub>3</sub> and greater



### Enantiomer separation of 1-buten-3-ol (TFA) MN Appl. No. 202751

Column: LIPODEX® C,  
50 m x 0.25 mm ID,  
REF 723364.50,  
max. temperature 200/220 °C  
Carrier gas: 1 bar H<sub>2</sub>  
Temperature: 20 °C  
Detector: FID  
C<sub>4</sub>H<sub>6</sub>O



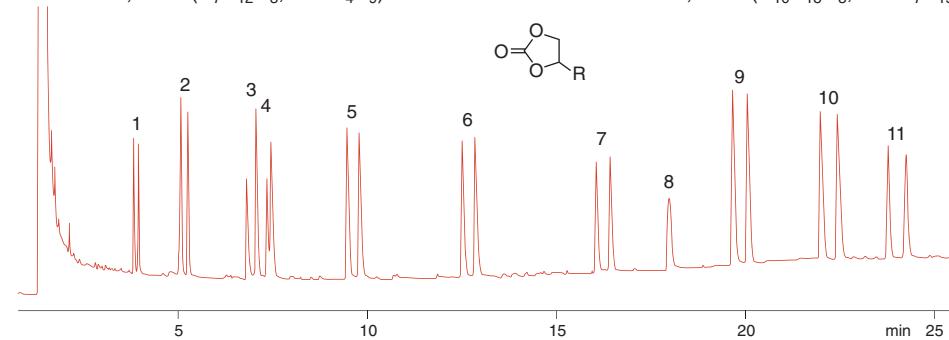
W.A. König et al., HRC 12 (1989) 35 – 39

### Enantiomer analysis of 1,2-diols (cyclic carbonates) MN Appl. No. 202861

Column: LIPODEX® B, 50 m x 0.25 mm ID, REF 723362.50, max. temperature 200/220 °C  
Carrier gas: 1 bar H<sub>2</sub>  
Temperature: 165 °C  $\xrightarrow{2 \text{ °C/min}}$  210 °C  
Detector: FID

#### Peaks:

- 1. 3,3-Dimethylbutane-1,2-diol (C<sub>7</sub>H<sub>12</sub>O<sub>3</sub>, R = t-C<sub>4</sub>H<sub>9</sub>)
- 2. 3-Methylbutane-1,2-diol (C<sub>6</sub>H<sub>10</sub>O<sub>3</sub>, R = CH(CH<sub>3</sub>)<sub>2</sub>)
- 3. Propane-1,2-diol (C<sub>4</sub>H<sub>6</sub>O<sub>3</sub>, R = CH<sub>3</sub>)
- 4. Butane-1,2-diol (C<sub>5</sub>H<sub>8</sub>O<sub>3</sub>, R = C<sub>2</sub>H<sub>5</sub>)
- 5. Pentane-1,2-diol (C<sub>6</sub>H<sub>10</sub>O<sub>3</sub>, R = C<sub>3</sub>H<sub>7</sub>)
- 6. Hexane-1,2-diol (C<sub>7</sub>H<sub>12</sub>O<sub>3</sub>, R = C<sub>4</sub>H<sub>9</sub>)
- 7. Heptane-1,2-diol (C<sub>8</sub>H<sub>14</sub>O<sub>3</sub>, R = C<sub>5</sub>H<sub>11</sub>)
- 8. Phenylglycol (C<sub>9</sub>H<sub>8</sub>O<sub>3</sub>, R = C<sub>6</sub>H<sub>5</sub>)
- 9. Octane-1,2-diol (C<sub>9</sub>H<sub>16</sub>O<sub>3</sub>, R = C<sub>6</sub>H<sub>13</sub>)
- 10. 1-Octene-7,8-diol (C<sub>9</sub>H<sub>14</sub>O<sub>3</sub>, R = (CH<sub>2</sub>)<sub>4</sub>-CH=CH<sub>2</sub>)
- 11. Nonane-1,2-diol (C<sub>10</sub>H<sub>18</sub>O<sub>3</sub>, R = C<sub>7</sub>H<sub>15</sub>)

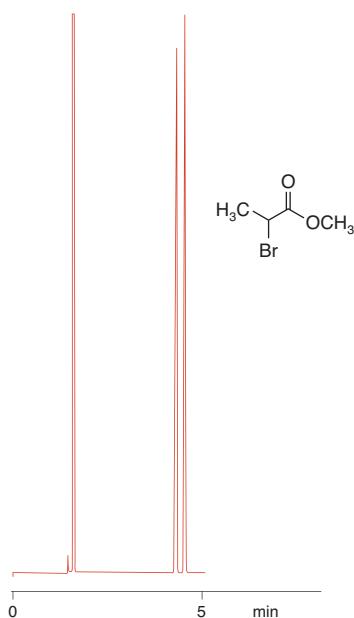


W.A. König et al., HRC 11 (1988) 621 – 625

## Chiral separations

### Enantiomer separation of 2-bromo-propionic acid methyl ester MN Appl. No. 210580

Column: FS-LIPODEX® A, 25 m x 0.25 mm ID, REF 723360.25, max. temperature 200/220 °C  
Injection: 1 µl (1% in CH<sub>2</sub>Cl<sub>2</sub>) split 200 ml/min  
Carrier gas: 11 psi He  
Temperature: 60 °C  
Detector: FID 250 °C  
C<sub>4</sub>H<sub>7</sub>BrO<sub>2</sub>



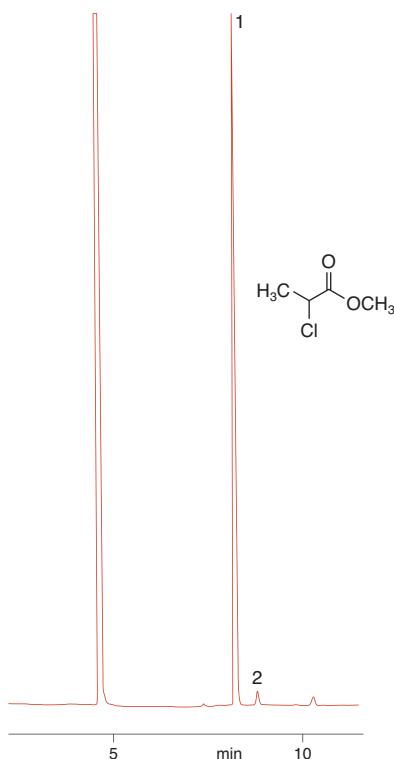
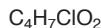
Courtesy of Mr. B. Sievers, Riedel-de Haen AG,  
Seelze, Germany

### Enantiomer separation of 2-chloropropionic acid methyl ester MN Appl. No. 202512

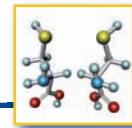
Column: FS-LIPODEX® C, 50 m x 0.25 mm ID, REF 723364.50, max. temperature 200/220 °C  
Split: 200 ml/min  
Carrier gas: 150 kPa He  
Temperature: 70 °C  
Detector: FID 200 °C

#### Peaks:

1. L-2-Chloropropionic acid methyl ester
2. D-2-Chloropropionic acid methyl ester



## Compounds C<sub>4</sub> – C<sub>8</sub>



### Enantiomer separation of 1,2-epoxyalkanes MN Appl. No. 202611

Column: FS-LIPODEX® E,  
25 m x 0.25 mm ID,  
REF 723368.25,  
max. temperature  
200/220 °C

Injection: 0.5 µl  
split 135 ml/min

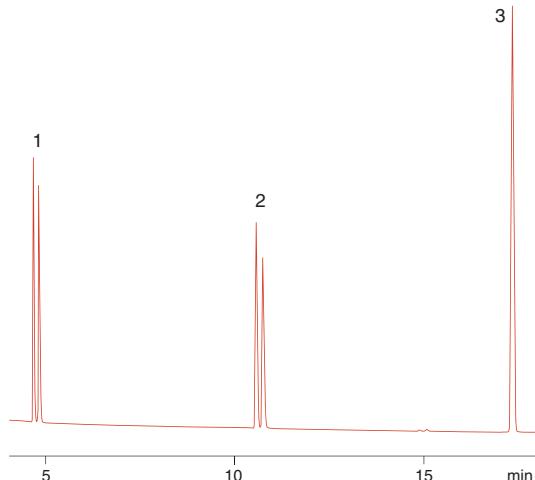
Carrier gas: 50 kPa H<sub>2</sub>  
(1.2 ml/min)

Temperature: 40 °C (3 min)  $\xrightarrow{4\text{ °C/min}}$   
120 °C

Detector: FID 250 °C

**Peaks:**

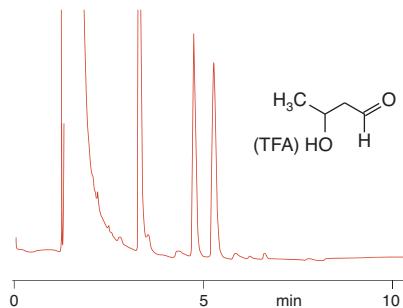
1. 1,2-Epoxybutane (C<sub>4</sub>H<sub>8</sub>O)
2. 1,2-Epoxyhexane (C<sub>6</sub>H<sub>12</sub>O)
3. 1,2-Epoxyoctane (C<sub>8</sub>H<sub>16</sub>O)



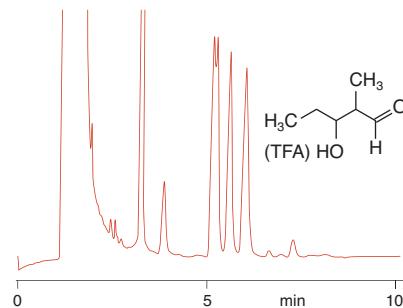
### Enantiomer separation of aldols (TFA) MN Appl. No. 202961

Column: LIPODEX® B, 50 m x 0.25 mm ID, REF 723362.50, max. temperature 200/220 °C  
Carrier gas: 1 bar H<sub>2</sub>  
Temperature: 85 °C  
Detector: FID

3-Hydroxybutyraldehyde (C<sub>4</sub>H<sub>8</sub>O<sub>2</sub>)



2-Methyl-3-hydroxyvaleraldehyde (C<sub>6</sub>H<sub>12</sub>O<sub>2</sub>)



W.A. König et al., HRC 11 (1988) 621 – 625

## Chiral separations

### Enantiomer separation of methyl lactate

MN Appl. No. 202762

Column: FS-LIPODEX® A,  
50 m x 0.25 mm ID,  
REF 723360.50,  
max. temperature 200/220 °C

Injection: 0.1 µl (1 % in CH<sub>2</sub>Cl<sub>2</sub>)  
split 320 ml/min

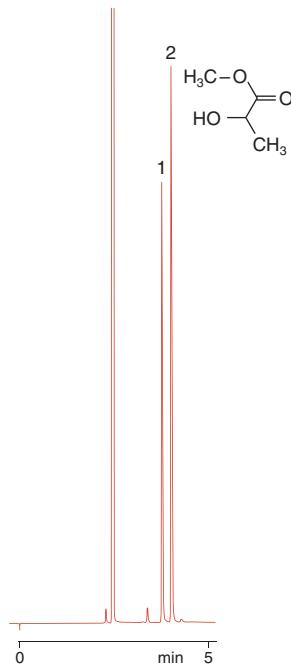
Carrier gas: 120 kPa H<sub>2</sub> (2.2 ml/min)

Temperature: 80 °C

Detector: FID 250 °C

**Peaks:** (C<sub>4</sub>H<sub>8</sub>O<sub>3</sub>)

1. S(-)
2. R(+)



MN Appl. No. 202772

Column: FS-HYDRODEX β-PM,  
50 m x 0.25 mm ID,  
REF 723370.50,  
max. temperature 230/250 °C

Injection: 0.1 µl (1 % in CH<sub>2</sub>Cl<sub>2</sub>)  
split 150 ml/min

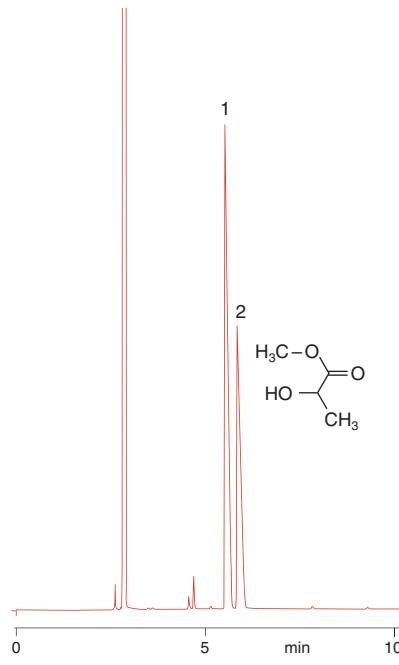
Carrier gas: 120 kPa H<sub>2</sub> (1.7 ml/min)

Temperature: 90 °C

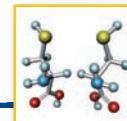
Detector: FID 250 °C

**Peaks:** (C<sub>4</sub>H<sub>8</sub>O<sub>3</sub>)

1. R(+)
2. S(-)



## Compounds C<sub>4</sub> – C<sub>6</sub>



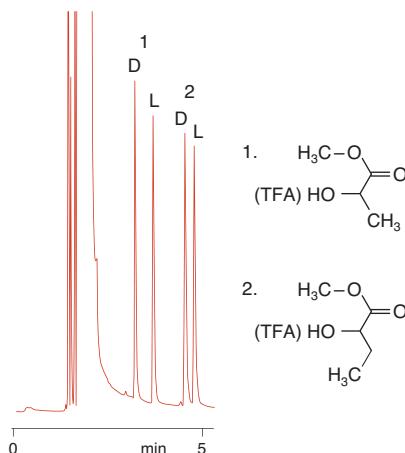
### Enantiomer separation of lactic and 2-hydroxybutyric acid methyl esters (TFA)

MN Appl. No. 202752

Column: LIPODEX® A, 50 m x 0.25 mm ID, REF 723360.50, max. temperature 200/220 °C  
Carrier gas: H<sub>2</sub>  
Temperature: 50 °C  
Detector: FID

**Peaks:**

1. Lactic acid methyl ester (C<sub>4</sub>H<sub>8</sub>O<sub>3</sub>)
2. 2-Hydroxybutyric acid methyl ester (C<sub>5</sub>H<sub>10</sub>O<sub>3</sub>)



Courtesy of Prof. W.A. König, Hamburg, Germany

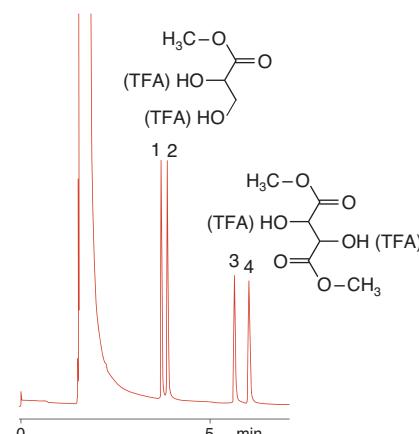
### Enantiomer separation of glyceric and tartaric acid methyl esters (TFA)

MN Appl. No. 202732

Column: LIPODEX® A, 50 m x 0.25 mm ID, REF 723360.50, max. temperature 200/220 °C  
Carrier gas: 1 bar H<sub>2</sub>  
Temperature: 90 °C  
Detector: FID

**Peaks:**

1. D-Glyceric acid methyl ester (C<sub>4</sub>H<sub>8</sub>O<sub>4</sub>)
2. L-Glyceric acid methyl ester
3. L-Tartaric acid dimethyl ester (C<sub>6</sub>H<sub>10</sub>O<sub>6</sub>)
4. D-Tartaric acid dimethyl ester



W.A. König, S. Lutz, G. Wenz, Angew. Chem. Int. Ed. Engl. 27 (1988) 979-980

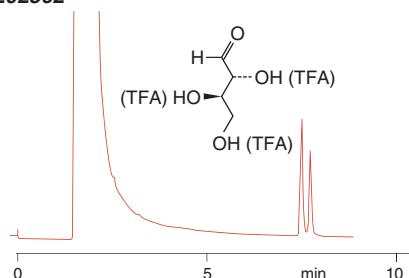
### Enantiomer separation of erythrose (TFA)

MN Appl. No. 202362

Column: LIPODEX® A, 50 m x 0.25 mm ID, REF 723360.50, max. temperature 200/220 °C

Carrier gas: H<sub>2</sub>  
Temperature: 80 °C  
Detector: FID

C<sub>4</sub>H<sub>8</sub>O<sub>4</sub>



Courtesy of Prof. W.A. König, Hamburg, Germany

## Chiral separations

### Enantiomer separation of 3-methyl-1-hexene, 2-bromobutane and 3-methylcyclohexene

MN Appl. No. 201880

Column: LIPODEX® C, 50 m x 0.25 mm ID, REF 723364.50, max. temperature 200/220 °C

Injection: headspace

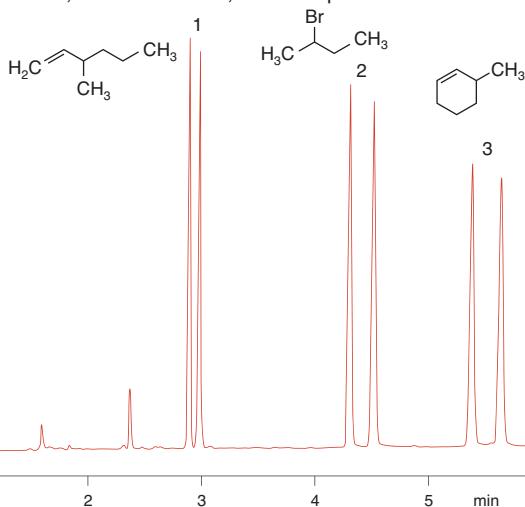
Carrier gas: 1 bar H<sub>2</sub>

Temperature: 30 °C

Detector: FID

**Peaks:**

1. 3-Methyl-1-hexene (C<sub>7</sub>H<sub>14</sub>)
2. 2-Bromobutane (C<sub>4</sub>H<sub>9</sub>Br)
3. 3-Methylcyclohexene (C<sub>7</sub>H<sub>12</sub>)



W.A. König et al., HRC **12**  
(1989) 35 – 39

### Enantiomer separation of chiral alkyl bromides

MN Appl. No. 202250

Column: LIPODEX® C, 50 m x 0.25 mm ID, REF 723364.50, max. temperature 200/220 °C

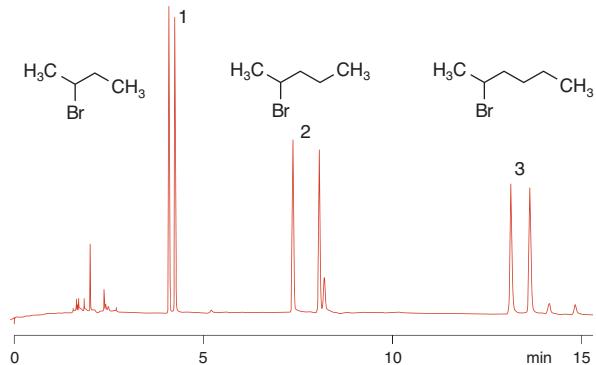
Carrier gas: H<sub>2</sub>

Temperature: 50 °C

Detector: FID

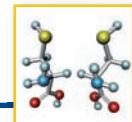
**Peaks:**

1. 2-Bromobutane (C<sub>4</sub>H<sub>9</sub>Br)
2. 2-Bromopentane (C<sub>5</sub>H<sub>11</sub>Br)
3. 2-Bromohexane (C<sub>6</sub>H<sub>13</sub>Br)



Courtesy of Prof. W.A. König,  
Hamburg, Germany

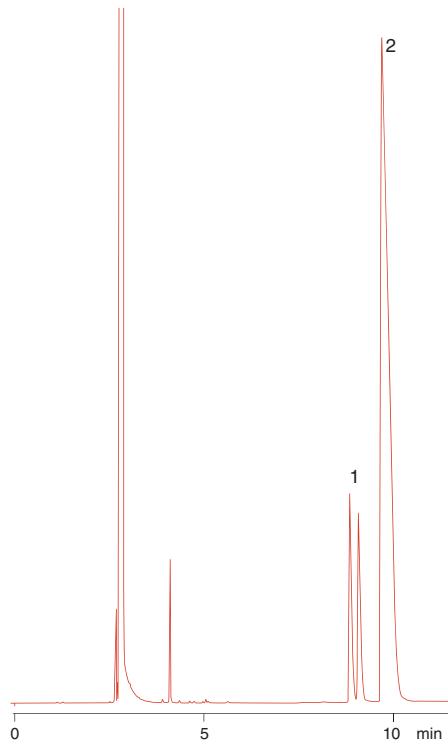
## Compounds C<sub>4</sub> – C<sub>7</sub>



### Enantiomer separation of butane-2,3-diol

MN Appl. No. 202901

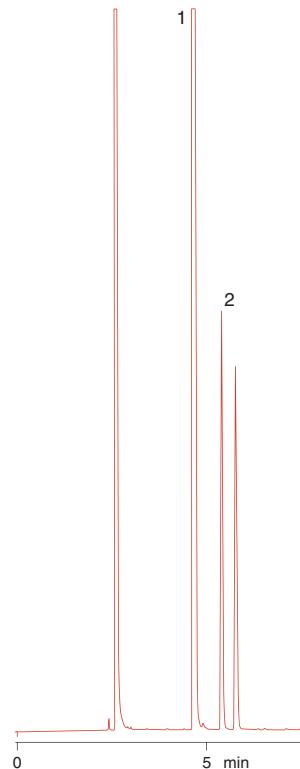
Column: FS-HYDRODEX  $\beta$ -PM,  
50 m x 0.25 mm ID  
REF 723370.50,  
max. temperature 230/250 °C  
Injection: 1  $\mu$ l (1% in CH<sub>2</sub>Cl<sub>2</sub>)  
split 150 ml/min  
Carrier gas: 120 kPa H<sub>2</sub> (1.7 ml/min)  
Temperature: 100 °C  
Detector: FID 250 °C  
**Peaks:** (C<sub>4</sub>H<sub>10</sub>O<sub>2</sub>)  
1. RR/SS  
2. meso



### Enantiomer separation of butane-2,3-diol (TFA)

MN Appl. No. 202891

Column: FS-LIPODEX® E,  
50 m x 0.25 mm ID,  
REF 723368.50,  
max. temperature 200/220 °C  
Injection: 0.5  $\mu$ l (1% in CH<sub>2</sub>Cl<sub>2</sub>)  
split 200 ml/min  
Carrier gas: 120 kPa H<sub>2</sub> (2.1 ml/min)  
Temperature: 80 °C  
Detector: FID 250 °C  
**Peaks:** (C<sub>4</sub>H<sub>10</sub>O<sub>2</sub>)  
1. meso  
2. RR/SS



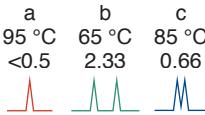
## Chiral separations

### Enantiomer separation of butane-1,3-diol MN Appl. No. 211320

Columns: all 25 m x 0.25 mm ID:  
 a) FS-HYDRODEX  $\beta$ -6TBDM (REF 723381.25),  
 b) FS-LIPODEX® E (REF 723368.25),  
 c) FS-HYDRODEX  $\beta$ -PM (REF 723370.25)

Injection: 1  $\mu$ l, split, 250 °C  
 Carrier gas: 1.1 ml/min He  
 Detector: FID 250 °C

Temperature: a 95 °C    b 65 °C    c 85 °C  
 Resolution: <0.5    2.33    0.66



### partial TFA derivative MN Appl. No. 211330

Column a)  
 Temperature: 75 °C  
 Resolution: 9.98



### TFA derivative MN Appl. No. 211340

Column a)  
 Temperature: 75 °C  
 Resolution: 8.79



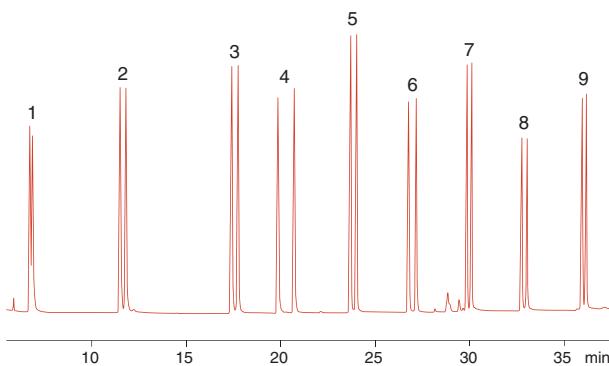
Courtesy of H. Casabianca, CNRS Service Central d'Analyse, Vernaison, France

### Enantiomer analysis of 1,2-diols (TFA) MN Appl. No. 202851

Column: LIPODEX® A,  
 50 m x 0.25 mm ID,  
 REF 723360.50,  
 max. temperature 200/220 °C  
 Carrier gas: H<sub>2</sub>  
 Temperature: 48 °C (5 min)  $\xrightarrow{2\text{ °C/min}}$  120 °C  
 Detector: FID

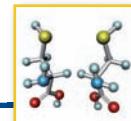
#### Peaks:

1. Butane-1,2-diol (C<sub>4</sub>H<sub>10</sub>O<sub>2</sub>)
2. Pentane-1,2-diol (C<sub>5</sub>H<sub>12</sub>O<sub>2</sub>)
3. Hexane-1,2-diol (C<sub>6</sub>H<sub>14</sub>O<sub>2</sub>)
4. Cyclohexane-*trans*-1,2-diol (C<sub>6</sub>H<sub>12</sub>O<sub>2</sub>)
5. Heptane-1,2-diol (C<sub>7</sub>H<sub>16</sub>O<sub>2</sub>)
6. Cycloheptane-*trans*-1,2-diol (C<sub>7</sub>H<sub>14</sub>O<sub>2</sub>)
7. Octane-1,2-diol (C<sub>8</sub>H<sub>18</sub>O<sub>2</sub>)
8. Phenylglycol (C<sub>8</sub>H<sub>10</sub>O<sub>2</sub>)
9. Nonane-1,2-diol (C<sub>9</sub>H<sub>20</sub>O<sub>2</sub>)



Courtesy of Prof. W.A. König, Hamburg, Germany

## Compounds C<sub>4</sub> and greater



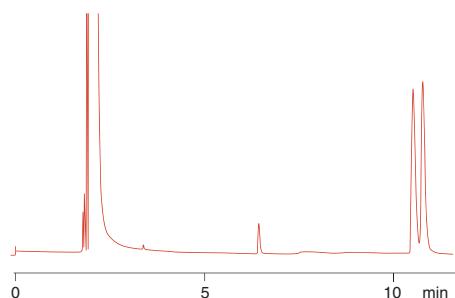
### Enantiomers separation of butane-1,2,4-triol (TFA)

MN Appl. No. 202931

Column: LIPODEX® A,  
50 m x 0.25 mm ID,  
REF 723360.50,  
max. temperature 200/220 °C

Carrier gas: H<sub>2</sub>  
Temperature: 70 °C  
Detector: FID

C<sub>4</sub>H<sub>10</sub>O<sub>3</sub>



### Enantiomer separation of 2-butylamine (TFA)

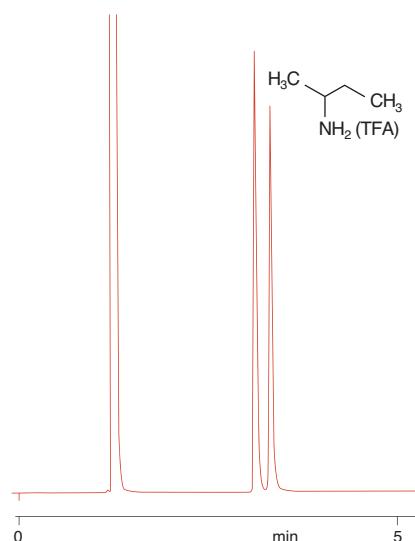
MN Appl. No. 202340

Column: FS-LIPODEX® D,  
25 m x 0.25 mm ID,  
REF 723366.25,  
max. temperature 200/220 °C

Injection: 0.1 µl (1% in CH<sub>2</sub>Cl<sub>2</sub>)  
split 190 ml/min

Carrier gas: 50 kPa H<sub>2</sub> (1.5 ml/min)  
Temperature: 105 °C  
Detector: FID 250 °C

C<sub>4</sub>H<sub>11</sub>N

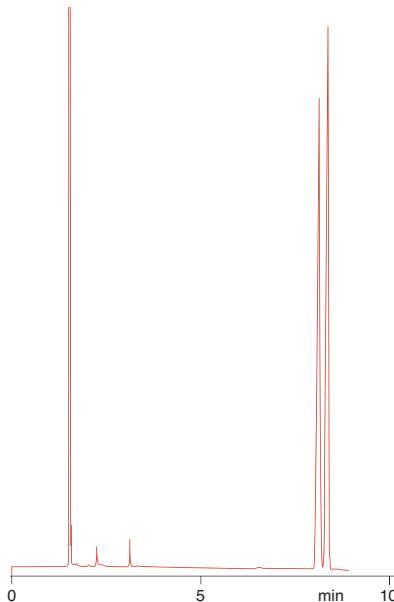


Courtesy of Prof. W.A. König, Hamburg,  
Germany

## Chiral separations

### Enantiomer separation of 2-aminobutanol-1 (N,O-TFA) *MN Appl. No. 210570*

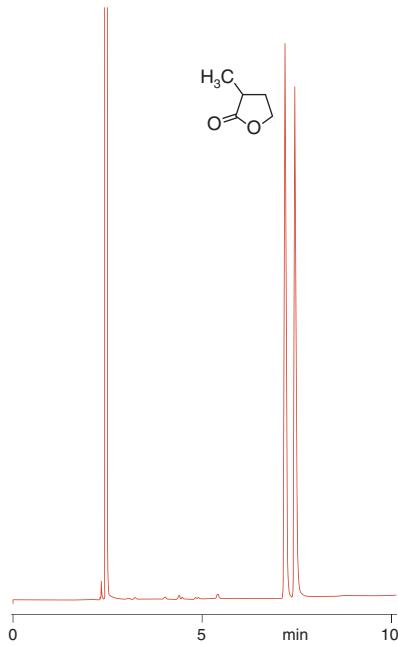
Column: FS-LIPODEX® A,  
25 m x 0.25 mm ID,  
REF 723360.25,  
max. temperature 200/220 °C  
Injection: 1 µl (1% in CH<sub>2</sub>Cl<sub>2</sub>)  
split 200 ml/min  
Carrier gas: 11 psi He  
Temperature: 90 °C  
Detector: FID 250 °C  
C<sub>4</sub>H<sub>11</sub>NO



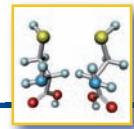
Courtesy of Mr. B. Sievers, Riedel-de Haen AG,  
Seelze, Germany

### Enantiomer separation of α-methylbutyrolactone *MN Appl. No. 202842*

Column: FS-LIPODEX® A,  
50 m x 0.25 mm ID,  
REF 723360.50,  
max. temperature 200/220 °C  
Injection: 0.1 µl (1% in CH<sub>2</sub>Cl<sub>2</sub>)  
split 245 ml/min  
Carrier gas: 110 kPa H<sub>2</sub> (2.3 ml/min)  
Temperature: 90 °C  
Detector: FID 240 °C  
C<sub>5</sub>H<sub>8</sub>O<sub>2</sub>



# Compounds C<sub>4</sub> and greater

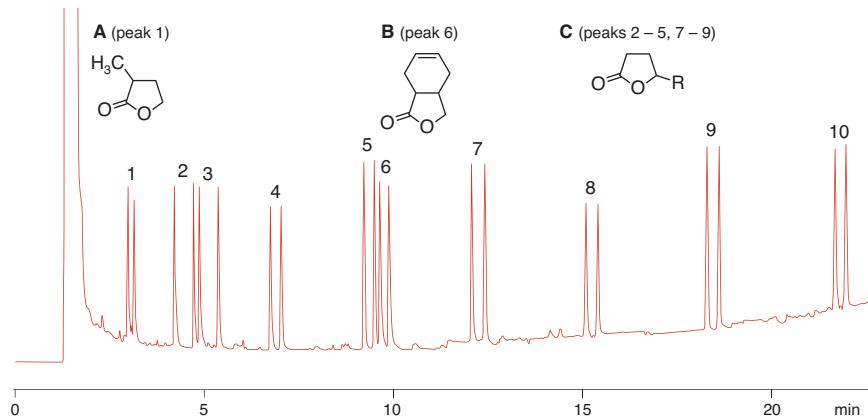


## Enantiomer analysis of $\gamma$ -lactones MN Appl. No. 202982

Column: LIPODEX® B, 50 m x 0.25 mm ID, REF 723362.50,  
max. temperature 200/220 °C  
Carrier gas: H<sub>2</sub>  
Temperature: 135 °C  $\xrightarrow{3\text{ °C/min}}$  200 °C  
Detector: FID

**Peaks:**

1.  $\alpha$ -Methylbutyrolactone (structure A, C<sub>5</sub>H<sub>8</sub>O<sub>2</sub>)
2.  $\gamma$ -Valerolactone (structure C, C<sub>5</sub>H<sub>8</sub>O<sub>2</sub>, R = CH<sub>3</sub>)
3.  $\gamma$ -Hexalactone (structure C, C<sub>6</sub>H<sub>10</sub>O<sub>2</sub>, R = C<sub>2</sub>H<sub>5</sub>)
4.  $\gamma$ -Heptalactone (structure C, C<sub>7</sub>H<sub>12</sub>O<sub>2</sub>, R = C<sub>3</sub>H<sub>7</sub>)
5.  $\gamma$ -Octalactone (structure C, C<sub>8</sub>H<sub>14</sub>O<sub>2</sub>, R = C<sub>4</sub>H<sub>9</sub>)
6. Tetrahydroisobenzofuranone (structure B, C<sub>8</sub>H<sub>10</sub>O<sub>2</sub>)
7.  $\gamma$ -Nonalactone (structure C, C<sub>9</sub>H<sub>16</sub>O<sub>2</sub>, R = C<sub>5</sub>H<sub>11</sub>)
8.  $\gamma$ -Decalactone (structure C, C<sub>10</sub>H<sub>18</sub>O<sub>2</sub>, R = C<sub>6</sub>H<sub>13</sub>)
9.  $\gamma$ -Undecalactone (structure C, C<sub>11</sub>H<sub>20</sub>O<sub>2</sub>, R = C<sub>7</sub>H<sub>15</sub>)
10.  $\gamma$ -Dodecalactone (structure C, C<sub>12</sub>H<sub>22</sub>O<sub>2</sub>, R = C<sub>8</sub>H<sub>17</sub>)



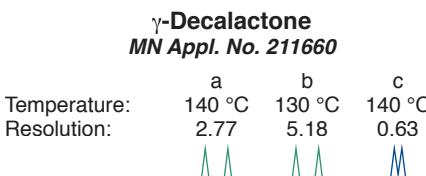
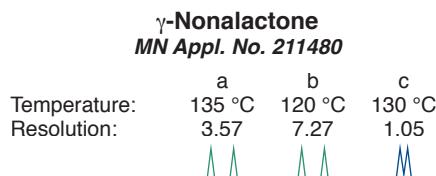
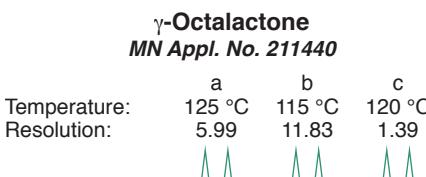
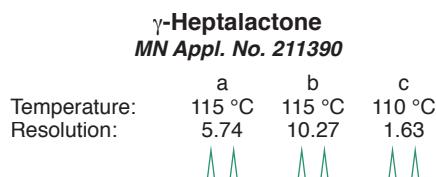
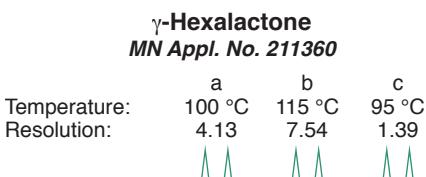
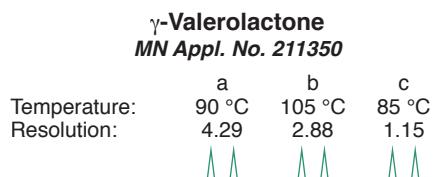
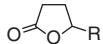
Courtesy of Prof. W.A. König, Hamburg, Germany

# Chiral separations

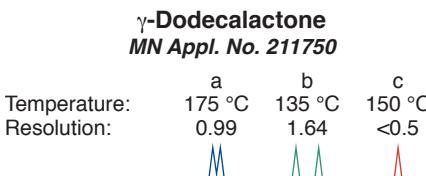
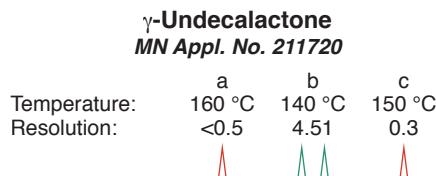
## Enantiomer analysis of $\gamma$ -lactones

Columns: all 25 m x 0.25 mm ID:  
 a) FS-HYDRODEX  $\beta$ -6TBDM (REF 723381.25),  
 b) FS-LIPODEX® E (REF 723368.25),  
 c) FS-HYDRODEX  $\beta$ -PM (REF 723370.25)

Injection: 1  $\mu$ l, split, 250 °C  
 Carrier gas: 1.1 ml/min He  
 Detector: FID 250 °C

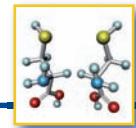


For separation of  $\gamma$ -decalactone also see appl. 212530 at [www.mn-net.com](http://www.mn-net.com)



Courtesy of H. Casabianca, CNRS Service Central d'Analyse, Vernaison, France

# Compounds C<sub>5</sub> and greater



## Enantiomer separation of 1-O-alkylglycerols (cyclic carbonates)

MN Appl. No. 202951

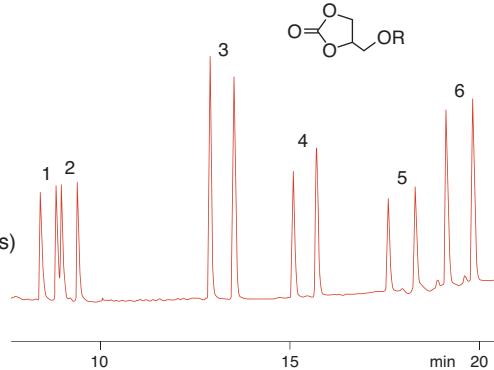
Column: LIPODEX® B,  
50 m x 0.25 mm ID,  
REF 723362.50,  
max. temperature 200/220 °C  
Sample: O-alkylglycerols derivatised  
with phosgene (very toxic)  
Carrier gas: 1 bar H<sub>2</sub>  
Temperature: 160 °C → 200 °C  
Detector: FID

**Peaks:**

(R enantiomers are eluted before S enantiomers)

1. 1-O-Methylglycerol  
(C<sub>5</sub>H<sub>8</sub>O<sub>4</sub>, R = -CH<sub>3</sub>)
2. 1-O-Ethylglycerol  
(C<sub>6</sub>H<sub>10</sub>O<sub>4</sub>, R = -C<sub>2</sub>H<sub>5</sub>)
3. 1-O-Isobutylglycerol  
(C<sub>8</sub>H<sub>14</sub>O<sub>4</sub>, R = -CH<sub>2</sub>-CH(CH<sub>3</sub>)<sub>2</sub>)
4. 1-O-Butylglycerol  
(C<sub>8</sub>H<sub>14</sub>O<sub>4</sub>, R = -(CH<sub>2</sub>)<sub>3</sub>-CH<sub>3</sub>)
5. 1-O-Isopentylglycerol  
(C<sub>9</sub>H<sub>16</sub>O<sub>4</sub>, R = -(CH<sub>2</sub>)<sub>2</sub>-CH(CH<sub>3</sub>)<sub>2</sub>)
6. 1-O-Pentylglycerol  
(C<sub>9</sub>H<sub>16</sub>O<sub>4</sub>, R = -(CH<sub>2</sub>)<sub>4</sub>-CH<sub>3</sub>)

W.A. König et al., HRC 11 (1988) 621 – 625



## Enantiomer separation of cyanohydrins (TFA)

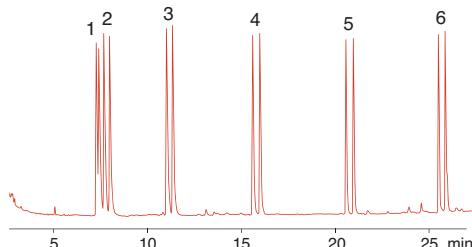
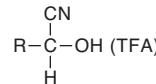
MN Appl. No. 202280

Column: LIPODEX® C,  
50 m x 0.25 mm ID,  
REF 723364.50,  
max. temperature 200/ 220 °C  
Carrier gas: 1 bar H<sub>2</sub>  
Temperature: 50 °C → 180 °C  
Detector: FID

**Peaks:**

1. R = *n*-propyl (C<sub>5</sub>H<sub>9</sub>NO)
2. R = *i*-butyl (C<sub>6</sub>H<sub>11</sub>NO)
3. R = *n*-butyl (C<sub>6</sub>H<sub>11</sub>NO)
4. R = *n*-pentyl (C<sub>7</sub>H<sub>13</sub>NO)
5. R = *n*-hexyl (C<sub>8</sub>H<sub>15</sub>NO)
6. R = *n*-heptyl (C<sub>9</sub>H<sub>17</sub>NO)

R enantiomers are eluted before S enantiomers.



W.A. König et al., HRC 12 (1989) 35 – 39

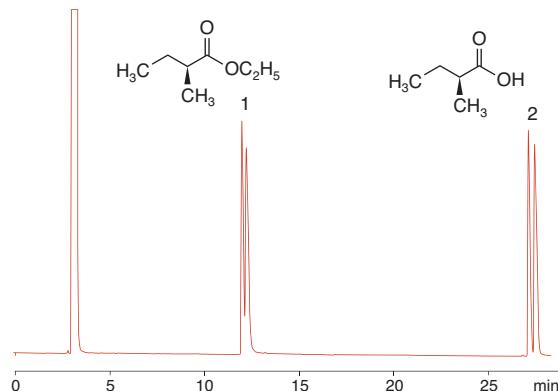
## Chiral separations

### Enantiomer separation of 2-methylbutyric acid and ethyl ester MN Appl. No. 202462

Column: FS-HYDRODEX  $\beta$ -PM,  
 50 m x 0.25 mm ID,  
 REF 723370.50,  
 max. temperature  
 230/250 °C  
 Injection: 1  $\mu$ l (0.5 % in CH<sub>2</sub>Cl<sub>2</sub>)  
 split 1:100  
 Carrier gas: 110 kPa H<sub>2</sub>  
 Temperature: 65 °C (12 min)  
 $\xrightarrow{4\text{ °C/min}}$  130 °C  
 Detector: FID 250 °C

**Peaks:**

1. 2-Methylbutyric acid ethyl ester  
(C<sub>7</sub>H<sub>14</sub>O<sub>2</sub>)
2. 2-Methylbutyric acid  
(C<sub>5</sub>H<sub>10</sub>O<sub>2</sub>)



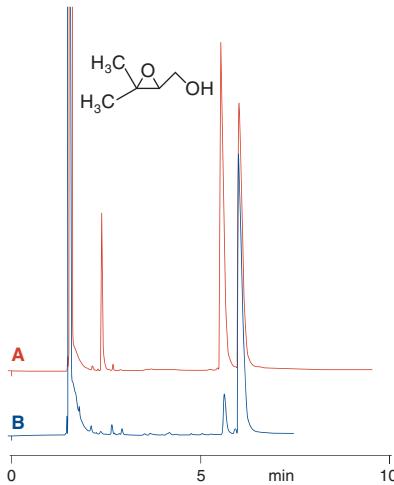
### Enantiomer separation of 2,3-epoxy-3-methylbutanol-1 MN Appl. No. 202681/202691

Column: FS-LIPODEX® E, 25 m x 0.25 mm ID, REF 723368.25,  
 max. temperature 200/220 °C  
 Injection: 0.5  $\mu$ l, split 135 ml/min  
 Carrier gas: 50 kPa H<sub>2</sub> (1.2 ml/min)  
 Temperature: 100 °C  
 Detector: FID 250 °C

- A) racemate (Appl. 202681)**  
**B) ee = 83 % (Appl. 202691)**

C<sub>5</sub>H<sub>10</sub>O<sub>2</sub>

2,3-epoxy-3-methylbutanol-1 can also be separated on HYDRODEX  $\beta$ -PM (applications 202661/202671) or LIPODEX® A (applications 202701/202711). See our application database at [www.mn-net.com](http://www.mn-net.com).



## Compounds C<sub>5</sub> – C<sub>7</sub>



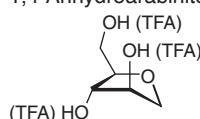
### Enantiomer separation of 1,4- and 1,5-anhydroarabinitol (TFA) MN Appl. No. 203021

Column: LIPODEX® A,  
50 m x 0.25 mm ID,  
REF 723360.50,  
max. temperature 200/220 °C

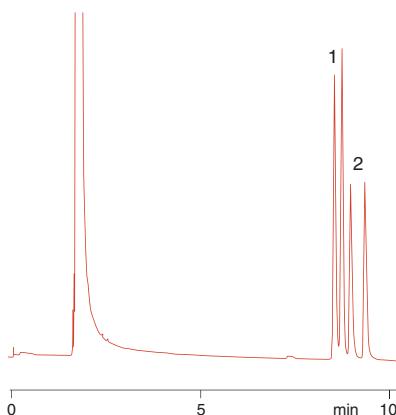
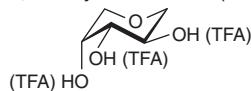
Carrier gas: H<sub>2</sub>  
Temperature: 90 °C  
Detector: FID

**Peaks:**

1. 1,4-Anhydroarabinitol (TFA), C<sub>5</sub>H<sub>10</sub>O<sub>4</sub>



2. 1,5-Anhydroarabinitol (TFA), C<sub>5</sub>H<sub>10</sub>O<sub>4</sub>



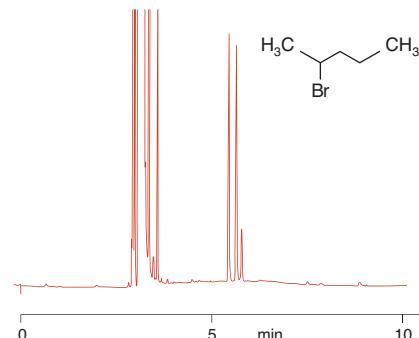
Courtesy of Prof. W.A. König, Hamburg,  
Germany

### Enantiomer separation of 2-bromopentane MN Appl. No. 202240

Column: FS-LIPODEX® C,  
50 m x 0.25 mm ID,  
REF 723364.50,  
max. temperature 200/220 °C

Injection: 1 µl, split 1:100  
Carrier gas: 100 kPa H<sub>2</sub>  
Temperature: 60 °C  
Detector: FID 250 °C

C<sub>5</sub>H<sub>11</sub>Br



## Chiral separations

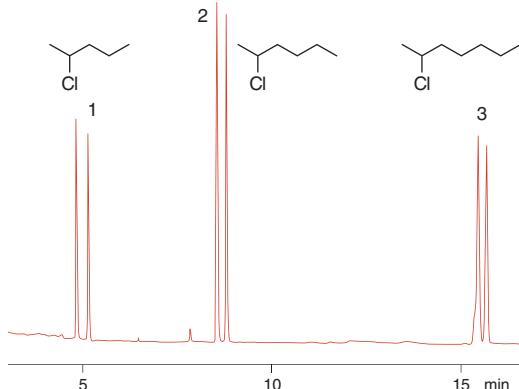
### Enantiomer separation of chiral alkyl chlorides MN Appl. No. 202260

Column: LIPODEX® C,  
50 m x 0.25 mm ID,  
REF 723364.50,  
max. temperature  
200/ 220 °C

Carrier gas: 1 bar H<sub>2</sub>  
Temperature: 60 °C  
Detector: FID

**Peaks:**  
1. 2-Chloropentane (C<sub>5</sub>H<sub>11</sub>Cl)  
2. 2-Chlorohexane (C<sub>6</sub>H<sub>13</sub>Cl)  
3. 2-Chloroheptane (C<sub>7</sub>H<sub>15</sub>Cl)

W.A. König et al., HRC 12 (1989) 35 – 39



### Enantiomer separation of 2-alkanols MN Appl. No. 202451

Column: FS-LIPODEX® E,  
50 m x 0.25 mm ID,  
REF 723368.50,  
max. temperature 200/ 220 °C

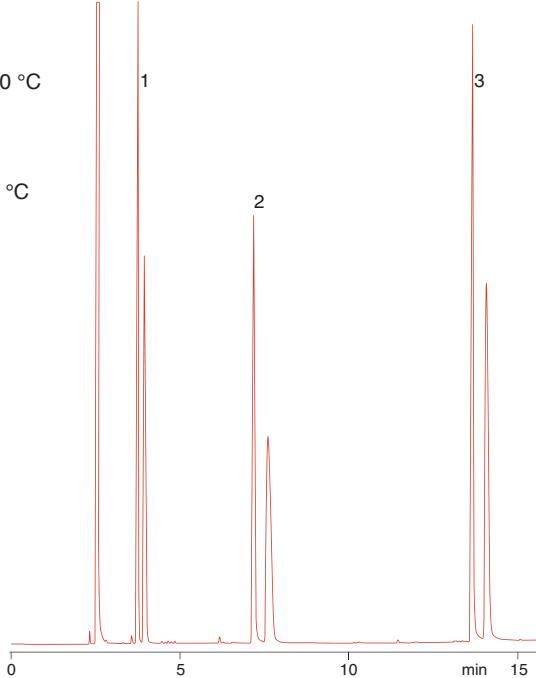
Injection: 0.1 µl (1% in CH<sub>2</sub>Cl<sub>2</sub>)  
split 200 ml/min

Carrier gas: 120 kPa H<sub>2</sub> (2.1 ml/min)

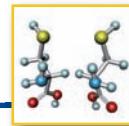
Temperature: 80 °C (4 min)  $\xrightarrow{4\text{ °C/min}}$  130 °C  
Detector: FID 250 °C

**Peaks:**

1. R/S-Pentanol-2 (C<sub>5</sub>H<sub>12</sub>O)  
2. R/S-Heptanol-2 (C<sub>7</sub>H<sub>16</sub>O)  
3. R/S-Nonanol-2 (C<sub>9</sub>H<sub>20</sub>O)



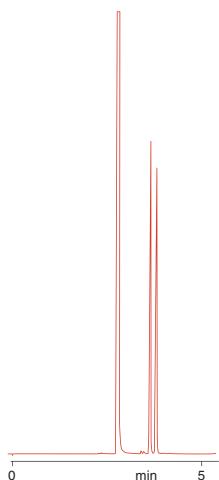
# Compounds C<sub>5</sub> and greater



## Enantiomer separation of pentanol-2 (TFA)

MN Appl. No. 202471

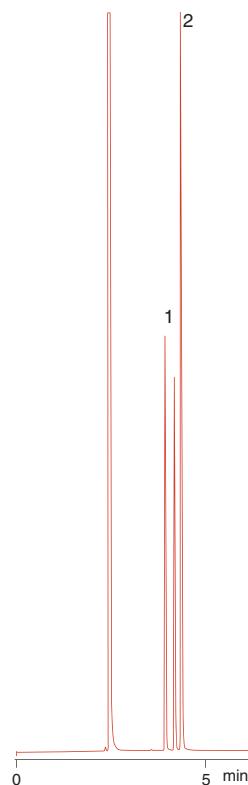
Column: FS-LIPODEX® E,  
50 m x 0.25 mm ID,  
REF 723368.50,  
max. temperature 200/220 °C  
Injection: 0.1 µl (1% in CH<sub>2</sub>Cl<sub>2</sub>)  
split 200 ml/min  
Carrier gas: 120 kPa H<sub>2</sub> (2.1 ml/min)  
Temperature: 60 °C  
Detector: FID 250 °C  
C<sub>5</sub>H<sub>12</sub>O



## Enantiomer separation of pentane-2,4-diol (TFA)

MN Appl. No. 202911

Column: FS-LIPODEX® E,  
50 m x 0.25 mm ID,  
REF 723368.50,  
max. temperature 200/220 °C  
Injection: 0.1 µl (1% in CH<sub>2</sub>Cl<sub>2</sub>)  
split 200 ml/min  
Carrier gas: 120 kPa H<sub>2</sub> (2.1 ml/min)  
Temperature: 100 °C  
Detector: FID 250 °C  
Peaks: (C<sub>5</sub>H<sub>12</sub>O<sub>2</sub>)  
1. RR/SS  
2. meso



## Chiral separations

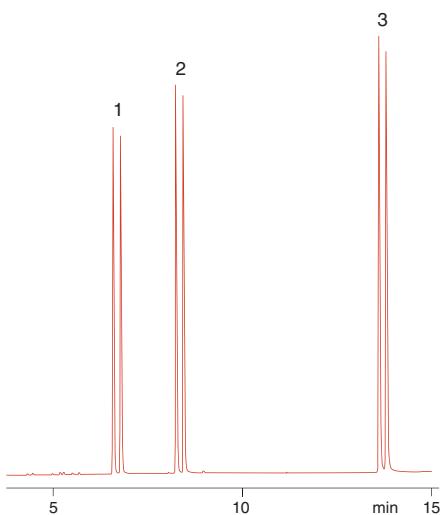
### Enantiomer separation of 1,2-diols (TFA)

MN Appl. No. 202921

Column: FS-LIPODEX® E,  
50 m x 0.25 mm ID,  
REF 723368.50,  
max. temperature 200/220 °C  
Injection: 0.1 µl (1% in CH<sub>2</sub>Cl<sub>2</sub>)  
split 200 ml/min  
Carrier gas: 120 kPa H<sub>2</sub> (2.1 ml/min)  
Temperature: 80 °C → 130 °C  
Detector: FID 250 °C

**Peaks:**

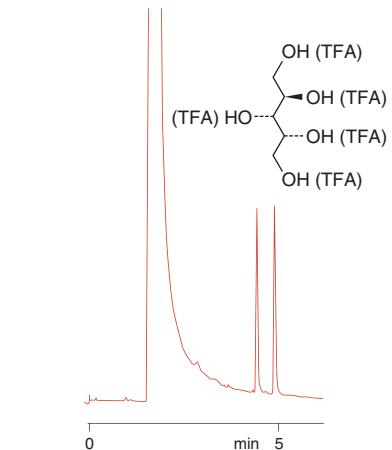
1. Pentane-1,2-diol (TFA), C<sub>5</sub>H<sub>12</sub>O<sub>2</sub>
2. Hexane-1,2-diol (TFA), C<sub>6</sub>H<sub>14</sub>O<sub>2</sub>
3. Octane-1,2-diol (TFA), C<sub>8</sub>H<sub>18</sub>O<sub>2</sub>



### Enantiomer separation of arabinitol (TFA)

MN Appl. No. 203011

Column: LIPODEX® A,  
50 m x 0.25 mm ID,  
REF 723360.50,  
max. temperature 200/220 °C  
Carrier gas: H<sub>2</sub>  
Temperature: 110 °C  
Detector: FID  
C<sub>5</sub>H<sub>12</sub>O<sub>5</sub>



Courtesy of Prof. W.A. König, Hamburg, Germany

## Compounds C<sub>5</sub> – C<sub>8</sub>



### Enantiomer separation of R/S-1-(2-furyl)ethanol and R/S-1-(2-furyl)ethanol acetate

MN Appl. No. 213260

Column: equivalent to FS-HYDRODEX  
 β-6TBDM, 25 m x 0.25 mm  
 ID, REF 723381.25, max.  
 temperature 230/250 °C

Injector: 250 °C

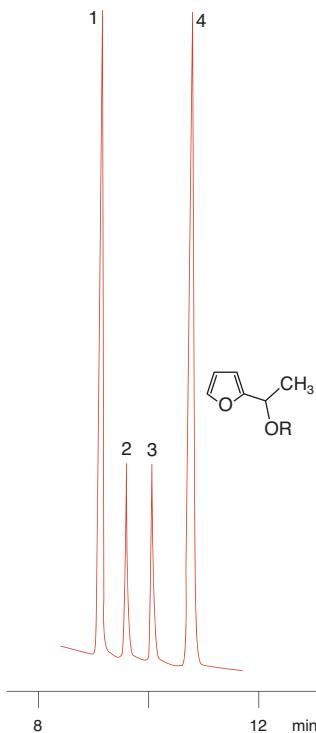
Carrier gas: 60 kPa H<sub>2</sub>

Temperature: 85 °C

Detector: FID 300 °C

**Peaks:**

1. (S)-1-(2-furyl)ethanol acetate (R = CO-CH<sub>3</sub>)
2. (R)-1-(2-furyl)ethanol (R = H)
3. (S)-1-(2-furyl)ethanol (C<sub>6</sub>H<sub>8</sub>O<sub>2</sub>)
4. (R)-1-(2-furyl)ethanol acetate (C<sub>8</sub>H<sub>10</sub>O<sub>3</sub>)



Courtesy of A. Ghanem, V. Schurig, Institute of Organic Chemistry, University of Tübingen, Germany

### Enantiomer separation of lactide

MN Appl. No. 202832

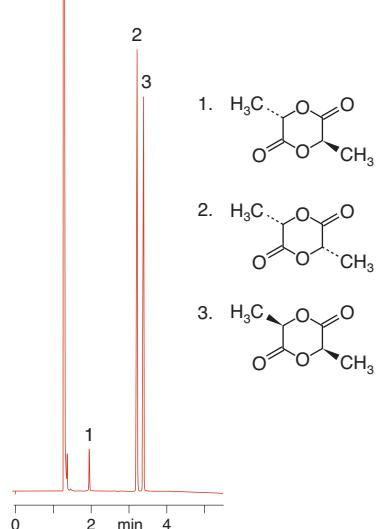
Column: FS-LIPODEX® A,  
 25 m x 0.25 mm ID,  
 REF 723360.25,  
 max. temperature 200/220 °C

Injection:  
 0.5 µl (1 % in CH<sub>2</sub>Cl<sub>2</sub>)  
 split 180 ml/min

Carrier gas:  
 60 kPa H<sub>2</sub> (1.8 ml/min)  
 Temperature: 150 °C  
 Detector: FID 250 °C

**Peaks:** (C<sub>6</sub>H<sub>8</sub>O<sub>4</sub>)

1. meso
2. SS
3. RR



Lactide can also be separated on HYDRODEX  
 β-PM  
 (see application 202822 at [www.mn-net.com](http://www.mn-net.com)).

## Chiral separations

**Analysis of 4 stereoisomers of  
3,4-dimethylbutyrolactone  
*MN Appl. No. 202892***

Column: LIPODEX® B,  
50 m x 0.25 mm ID,  
REF 723362.50,  
max. temperature 200/220 °C

Carrier gas: 1 bar H<sub>2</sub>  
Temperature: 135 °C  
Detector: FID

**Peaks:** (C<sub>6</sub>H<sub>5</sub>-O-)

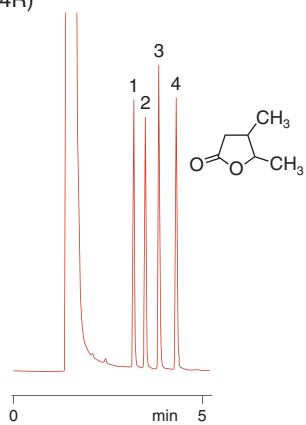
**Peaks:** ( $C_6H_{10}O_2$ )  
1 (3S, 1R)

1. (3S, 4R),  
2. (3R, 4S)

2. (3R, 4S)

3. (3S, 4S)

#### 4. (3R, 4R)



## **Enantiomer analysis of pantolactone MN Appl. No. 202912**

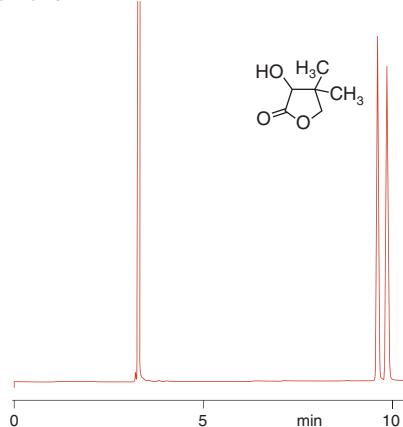
Column: FS-HYDRODEX  $\beta$ -PM,  
50 m x 0.25 mm ID,  
REF 723370.50,  
max. temperature 230/250 °C  
0.5  $\mu$ m film thickness

Injection: 0.5  $\mu$ l, split 1:100

Carrier gas: 100 kPa H<sub>2</sub>

Temperature: 160 °C

Detector: FID 250 °C



Pantolactone can also be separated on LIPODEX® E (applications 202902). See our application database at [www.mn-net.com](http://www.mn-net.com).

W.A. König et al., HRC 11 (1988) 621 – 625

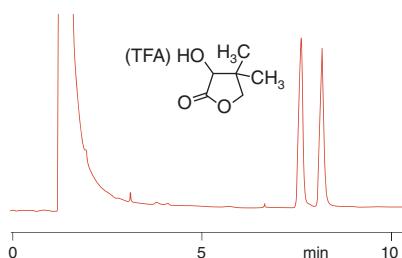
## Enantiomer analysis of pantolactone (TFA) MN Appl. No. 202922

Column: LIPODEX® B,  
50 m x 0.25 mm ID,  
REF 723362.50,  
max. temperature 200/220 °C

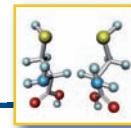
Carrier gas: H<sub>2</sub>  
Temperature: 110 °C  
Detector: FID

C<sub>6</sub>H<sub>10</sub>O<sub>3</sub>

Courtesy of Prof. W.A. König, Hamburg, Germany



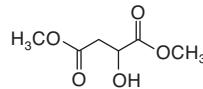
# Compounds C<sub>6</sub>



## Enantiomer separation of dimethyl malate MN Appl. No. 211370

Column: FS-HYDRODEX  $\beta$ -TBDM, 25 m x 0.25 mm ID, REF 723381.25  
Injection: 1  $\mu$ l, split, 250 °C  
Carrier gas: 1.1 ml/min He  
Temperature: 100 °C  
Detector: FID 250 °C

C<sub>6</sub>H<sub>10</sub>O<sub>5</sub>



Resolution: 8.22



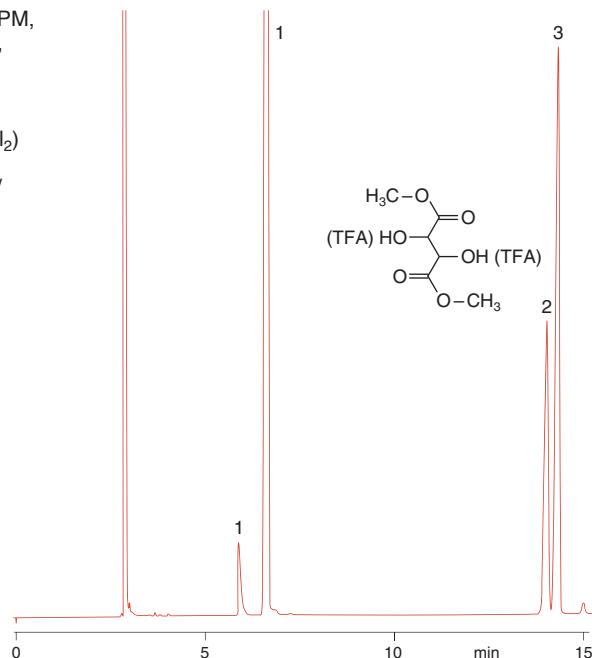
Courtesy of H. Casabianca, CNRS Service Central d'Analyse, Vernaison, France

## Enantiomer separation of tartaric acid dimethyl esters (TFA) MN Appl. No. 202712

Column: FS-HYDRODEX  $\beta$ -PM,  
50 m x 0.25 mm ID,  
REF 723370.50,  
max. temperature  
230/250 °C  
Injection: 0.5  $\mu$ l (1% in CH<sub>2</sub>Cl<sub>2</sub>)  
split 150 ml/min  
Carrier gas: 120 kPa H<sub>2</sub> (1.7 ml/  
min)  
Temperature: 135 °C  
Detector: FID 250 °C

**Peaks:** (C<sub>6</sub>H<sub>10</sub>O<sub>6</sub>)

1. impurities
2. D-( - )
3. L-( + )



Tartaric acid dimethyl ester can also be separated on HYDRODEX  $\beta$ -PM (application 202722) or LIPODEX® A (application 202702).  
See our application database at [www.mn-net.com](http://www.mn-net.com).

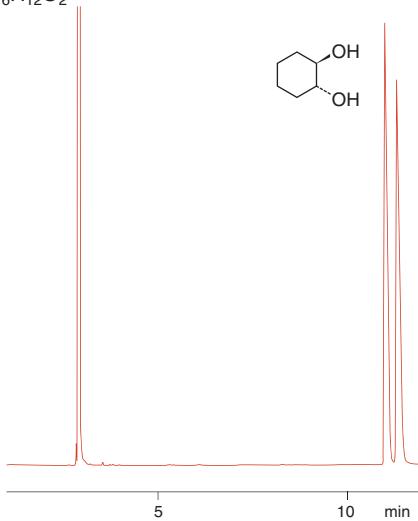
## Chiral separations

### Enantiomer separation of *trans*-1,2-cyclohexanediol

MN Appl. No. 202841

Column: FS-HYDRODEX  $\beta$ -PM,  
50 m x 0.25 mm ID,  
REF 723370.50,  
max. temperature 230/250 °C  
Injection: 0.5  $\mu$ l, split 1:100  
Carrier gas: 100 kPa H<sub>2</sub>  
Temperature: 150 °C  
Detector: FID 250 °C

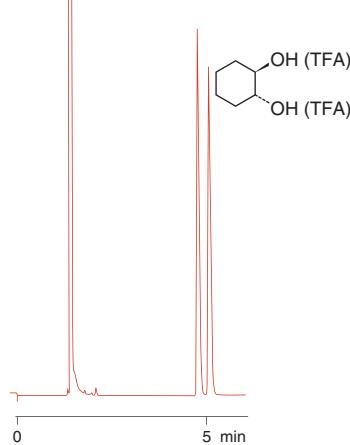
C<sub>6</sub>H<sub>12</sub>O<sub>2</sub>



### Enantiomer separation of *trans*-1,2-cyclohexanediol (TFA)

MN Appl. No. 202831

Column: FS-LIPODEX® E,  
25 m x 0.25 mm ID,  
REF 723368.25,  
max. temperature 200/ 220 °C  
Injection: 0.5  $\mu$ l, split 1:100  
Carrier gas: 60 kPa H<sub>2</sub>  
Temperature: 105 °C  
Detector: FID 250 °C



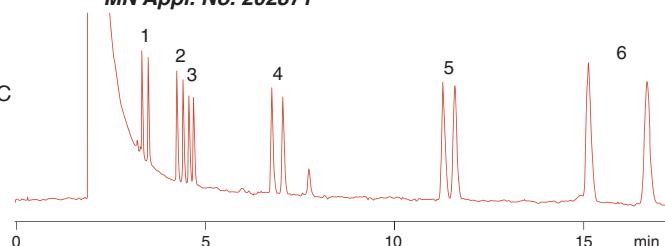
*trans*-1,2-cyclohexanediol can also be separated on LIPODEX® A (see application 202821 at [www.mn-net.com](http://www.mn-net.com)).

## Compounds C<sub>6</sub> and greater



## Enantiomer analysis of *trans*-1,2- and *trans*-1,3-cycloalkanediols (TFA) MN Appl. No. 202871

Column: LIPODEX® D,  
25 m x 0.25 mm ID,  
REF 723366.25,  
max. temperature 200/220 °C  
Carrier gas: 0.7 bar H<sub>2</sub>  
Temperature: 150 °C  
Detector: FID



Peaks:

- Pairs:**

  1. Cyclohexane-*trans*-1,2-diol ( $C_6H_{12}O_2$ )
  2. Cycloheptane-*trans*-1,2-diol ( $C_7H_{14}O_2$ )
  3. Cycloheptane-*trans*-1,3-diol ( $C_7H_{14}O_2$ )
  4. Cyclooctane-*trans*-1,2-diol ( $C_8H_{16}O_2$ )
  5. Cyclodecane-*trans*-1,2-diol ( $C_{10}H_{20}O_2$ )
  6. Cyclodecane-*trans*-1,3-diol ( $C_{10}H_{20}O_2$ )

W.A. König, S. Lutz, G. Wenz, E. von der Leyen, HRC 11 (1988) 506 – 509

**Enantiomer separation of  
2-methylbutyric acid methyl ester  
*MN Appl. No. 211380***

Columns: all 25 m x 0.25 mm ID:  
 a) FS-HYDRODEX  $\beta$ -6TBDM  
 (REF 723381.25),  
 b) FS-LIPODEX<sup>®</sup> E (REF  
 723368.25),  
 c) FS-HYDRODEX  $\beta$ -PM  
 (REF 723370.25)

Injection: 1  $\mu$ l, split, 250 °C

Carrier gas: 1.1 ml/min He

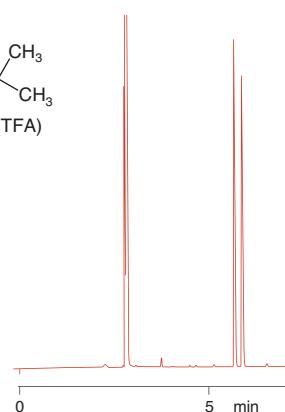
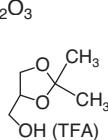
Detector: FID 250 °C

$C_6H_{12}O_2$

Courtesy of H. Casabianca, CNRS Service  
Central d'Analyse, Vernaison, France

## Enantiomer separation of solketal (TFA) MN Appl. No. 202971

Column: FS-HYDRODEX  $\beta$ -PM,  
 50 m x 0.25 mm ID,  
 REF 723370.50,  
 max. temperature 230/250 °C  
 Injection: 0.1  $\mu$ l (1% in  $\text{CH}_2\text{Cl}_2$ )  
 split 150 ml/min  
 Carrier gas: 120 kPa  $\text{H}_2$  (1.7 ml/min)  
 Temperature: 120 °C  
 Detector: FID 250 °C



Solketal can also be separated on LIPODEX® A (see application 202981 at [www.mn-net.com](http://www.mn-net.com)).

## Chiral separations

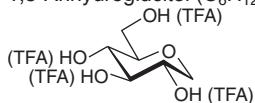
**Enantiomer separation of 1,5-anhydroglucitol, -galactitol und -mannitol (TFA)**  
**MN Appl. No. 203031**

Column: LIPODEX® A, 50 m x 0.25 mm ID, REF 723360.50,  
max. temperature 200/220 °C

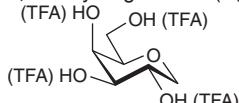
Carrier gas: H<sub>2</sub>  
Temperature: 120 °C  
Detector: FID

**Peaks:**

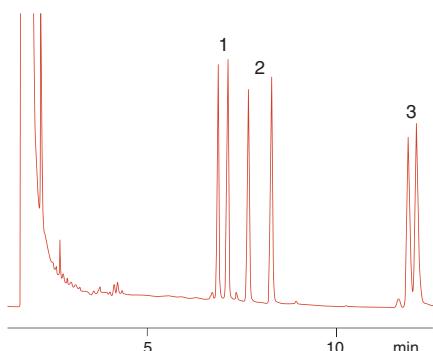
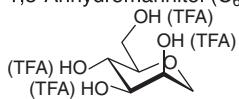
1. 1,5-Anhydroglucitol ( $C_6H_{12}O_5$ )



2. 1,5-Anhydrogalactitol ( $C_6H_{12}O_5$ )



3. 1,5-Anhydromannitol ( $C_6H_{12}O_5$ )



Courtesy of Prof. W.A. König, Hamburg,  
Germany

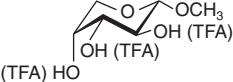
**Enantiomer separation of arabinose ( $\alpha$ - and  $\beta$ -anomers, methylglycosides, TFA)**  
**MN Appl. No. 202372**

Column: LIPODEX® A,  
50 m x 0.25 mm ID,  
REF 723360.50,  
max. temperature 200/220 °C

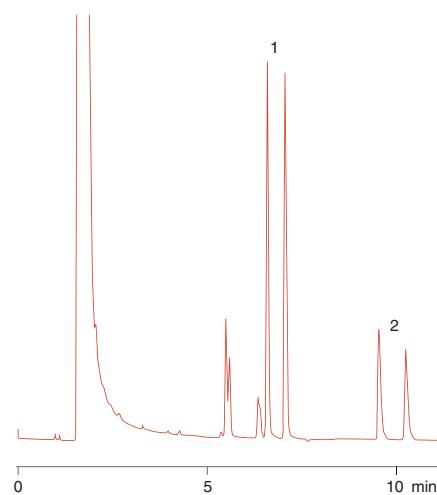
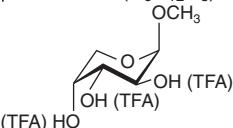
Carrier gas: H<sub>2</sub>  
Temperature: 100 °C  
Detector: FID

**Peaks:**

1.  $\alpha$ -Arabinose ( $C_6H_{12}O_5$ )



2.  $\beta$ -Arabinose ( $C_6H_{12}O_5$ )



Courtesy of Prof. W.A. König, Hamburg,  
Germany

For separation of other sugar anomers on  
LIPODEX® A see applications 202432, 202392,  
202382 at [www.mn-net.com](http://www.mn-net.com).

# Compounds C<sub>6</sub> and greater



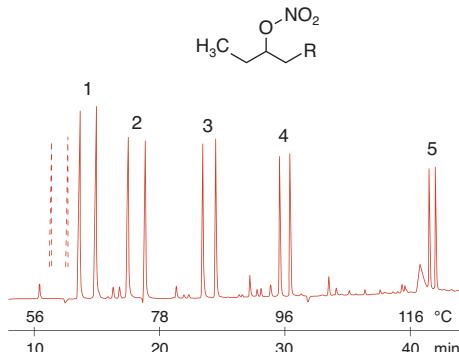
## Separation of chiral alkyl nitrates

MN Appl. No. 212050

Column: FS-LIPODEX® D, 25 m x 0.25 mm ID, REF 723366.25,  
max. temperature 200/220 °C  
Injection: on-column  
Carrier gas: 70 kPa H<sub>2</sub>, 55 cm/s, 100 °C  
Temperature: 40 °C (2 min)  $\xrightarrow{2\text{ °C/min}}$  190 °C  
Detector: ECD 240 °C

### Peaks:

1. *n*-Hexyl-3-nitrate (C<sub>6</sub>H<sub>13</sub>NO<sub>3</sub>, R = C<sub>2</sub>H<sub>5</sub>)
2. *n*-Heptyl-3-nitrate (C<sub>7</sub>H<sub>15</sub>NO<sub>3</sub>, R = C<sub>3</sub>H<sub>7</sub>)
3. *n*-Octyl-3-nitrate (C<sub>8</sub>H<sub>17</sub>NO<sub>3</sub>, R = C<sub>4</sub>H<sub>9</sub>)
4. *n*-Nonyl-3-nitrate (C<sub>9</sub>H<sub>19</sub>NO<sub>3</sub>, R = C<sub>5</sub>H<sub>11</sub>)
5. *n*-Undecyl-3-nitrate (C<sub>11</sub>H<sub>23</sub>NO<sub>3</sub>, R = C<sub>7</sub>H<sub>15</sub>)



dashed lines indicate where peak 1 would be expected to elute

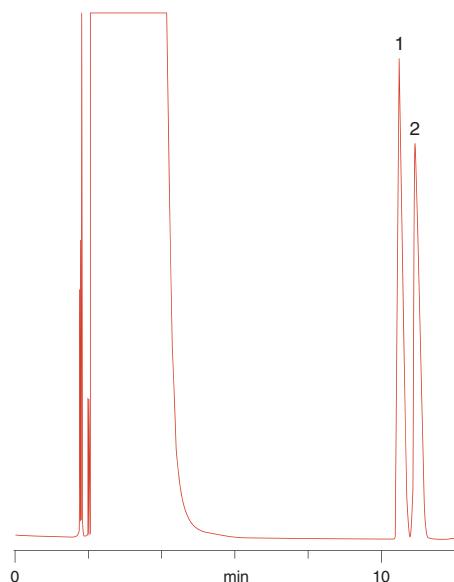
M. Schneider, K. Ballschmiter,  
J. Chromatography A **852** (1999) 525 – 534

## Enantiomer separation of 2-methylpentanol-1

MN Appl. No. 212450

Column: FS-HYDRODEX®  $\beta$ -TBDAC, 50 m x 0.25 mm ID, REF 723384.50, max. temperature 220/240 °C  
Sample: 0.1% in CH<sub>2</sub>Cl<sub>2</sub>  
Carrier gas: 1.5 bar H<sub>2</sub>  
Temperature: 80 °C  
Detector: FID

**Peaks:** (C<sub>6</sub>H<sub>14</sub>O)  
1. (*R*)-2-Methylpentanol-1  
2. (*S*)-2-Methylpentanol-1

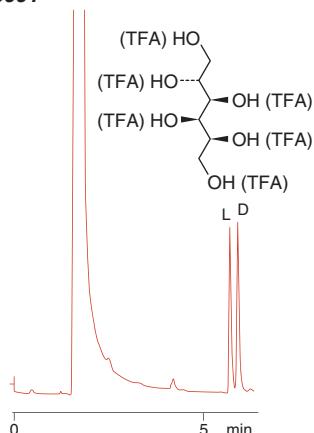


## Chiral separations

### Enantiomer separation of sorbitol (TFA) *MN Appl. No. 203001*

Column: LIPODEX® A,  
50 m x 0.25 mm ID,  
REF 723360.50,  
max. temperature 200/220 °C  
Carrier gas: H<sub>2</sub>  
Temperature: 110 °C  
Detector: FID  
C<sub>6</sub>H<sub>14</sub>O<sub>6</sub>

Courtesy of Prof. W.A. König, Hamburg,  
Germany



### Enantiomer separation of thioacetic acid cyclopent-2-enyl ester

Carrier gas: 100 kPa H<sub>2</sub>  
Temperature: 70 °C (2 min)  $\xrightarrow{10 \text{ °C/min}}$  100 °C (2 min)  $\xrightarrow{10 \text{ °C/min}}$  130 °C (2 min)  
Detector: FID

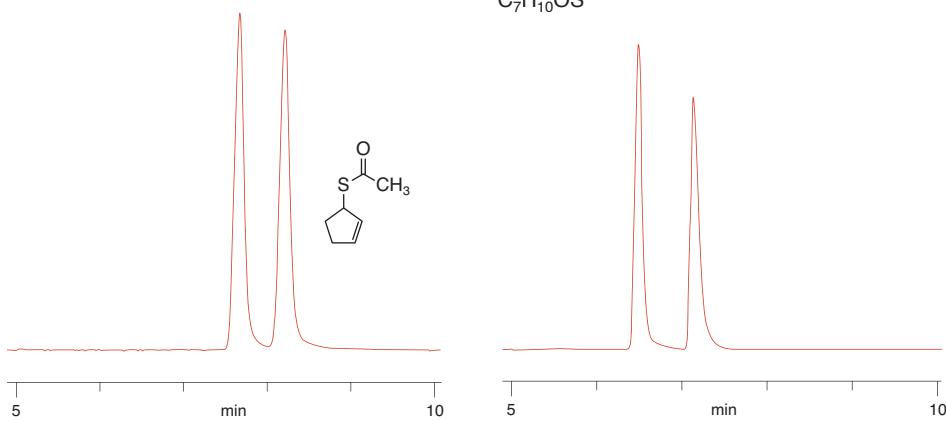
**MN Appl. No. 212270**

Column: FS-LIPODEX® E,  
25 m x 0.25 mm ID,  
REF 723368.25,  
max. temperature 200/220 °C

**MN Appl. No. 212280**

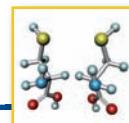
Column: FS-LIPODEX® G,  
25 m x 0.25 mm ID,  
REF 723379.25,  
max. temperature 220/240 °C

C<sub>7</sub>H<sub>10</sub>OS



Courtesy of Mrs. Vermeeren, AK Prof. Gais, Inst. für Org. Chemie, RWTH Aachen, Germany

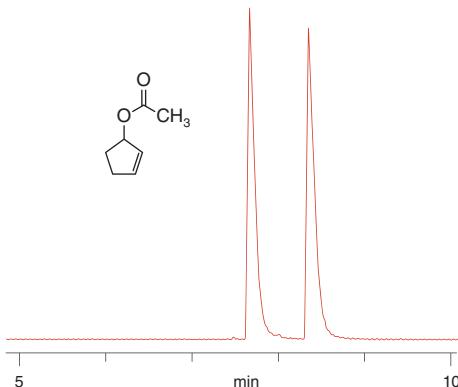
## Compounds C<sub>6</sub> – C<sub>7</sub>



### Enantiomer separation of acetic acid cyclopent-2-enyl ester

MN Appl. No. 212300

Column: FS-LIPODEX® G,  
25 m x 0.25 mm ID,  
REF 723379.25,  
max. temperature 220/240 °C  
Carrier gas: 100 kPa H<sub>2</sub>  
Temperature: 50 °C (5 min) → 80 °C  
(5 min)  
Detector: FID  
C<sub>7</sub>H<sub>10</sub>O<sub>2</sub>

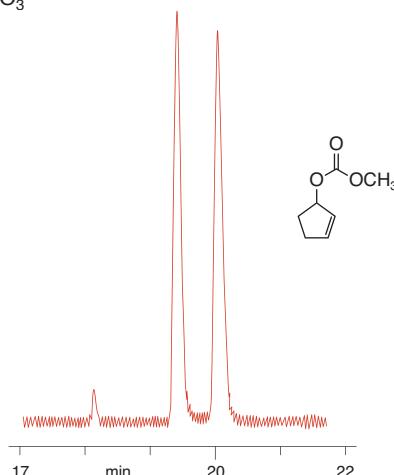


Courtesy of Mrs. Vermeeren, AK Prof. Gais,  
Inst. für Org. Chemie, RWTH Aachen, Germany

### Enantiomer separation of cyclopent-2-enyl methyl carbonate

MN Appl. No. 212330

Column: FS-LIPODEX® E, 25 m x 0.25 mm ID,  
REF 723368.25, max. temperature 200/220 °C  
Carrier gas: 100 kPa H<sub>2</sub>  
Temperature: 50 °C (15 min) →  
10 °C/min (5 min) → 120 °C (5 min)  
Detector: FID  
C<sub>7</sub>H<sub>10</sub>O<sub>3</sub>



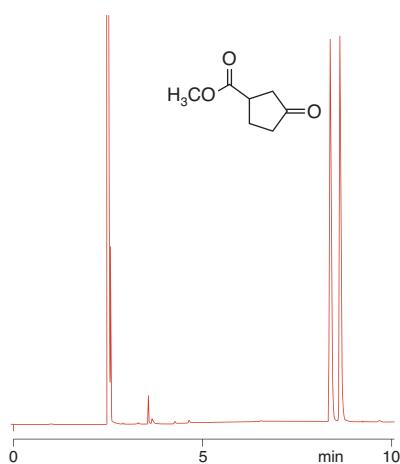
Courtesy of Mrs. Vermeeren, AK Prof. Gais,  
Inst. für Org. Chemie, RWTH Aachen, Germany

## Chiral separations

### Enantiomer separation of cyclopentanonecarboxylic acid methyl ester

MN Appl. No. 202080

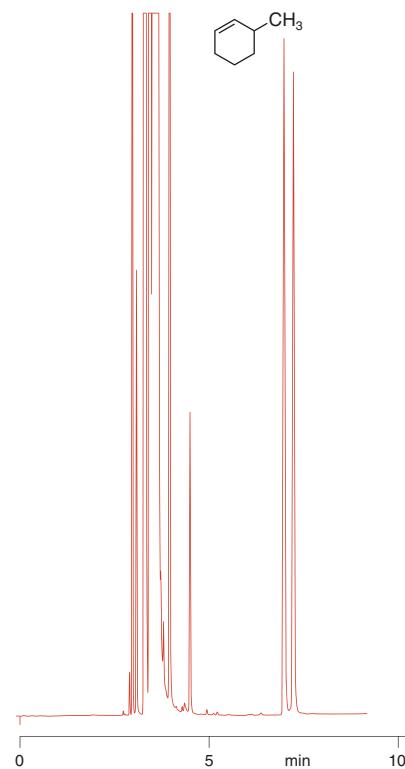
Column: FS-LIPODEX® A,  
50 m x 0.25 mm ID,  
REF 723360.50,  
max. temperature 200/220 °C  
Injection: 0.1 µl (1% in CH<sub>2</sub>Cl<sub>2</sub>)  
split 320 ml/min  
Carrier gas: 120 kPa H<sub>2</sub> (2.2 ml/min)  
Temperature: 120 °C  
Detector: FID 250 °C  
C<sub>7</sub>H<sub>10</sub>O<sub>3</sub>



### Enantiomer separation of 3-methylcyclohexene

MN Appl. No. 201860

Column: FS-LIPODEX® C,  
50 m x 0.25 mm ID,  
REF 723364.50,  
max. temperature 200/220 °C  
Injection: 1.0 µl, split 1:100  
Carrier gas: 100 kPa H<sub>2</sub>  
Temperature: 35 °C  
Detector: FID 250 °C  
C<sub>7</sub>H<sub>12</sub>



3-methylcyclohexene can also be separated on LIPODEX® A (see application 201870 at [www.mn-net.com](http://www.mn-net.com)).



## Compounds C<sub>7</sub> – C<sub>8</sub>

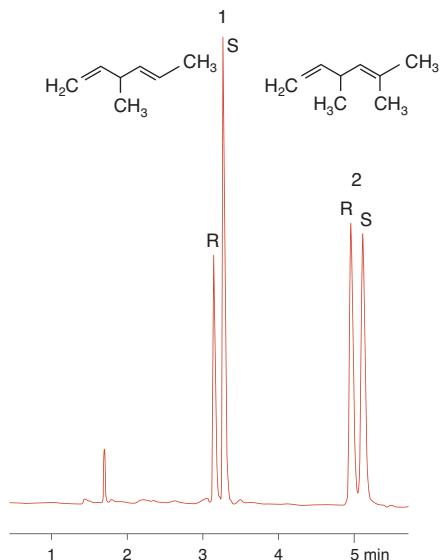
### Enantiomer separation of 3-methyl-1,4(Z)-hexadiene and 3,5-dimethyl-1,4-hexadiene

MN Appl. No. 201890

Column: LIPODEX® C,  
50 m x 0.25 mm ID,  
REF 723364.50,  
max. temperature 200/220 °C  
Injection: headspace  
Carrier gas: 1 bar H<sub>2</sub>  
Temperature: 30 °C  
Detector: FID

**Peaks:**

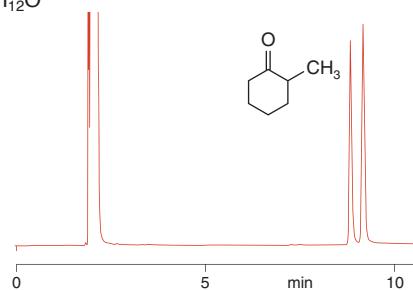
1. 3-Methyl-1,4-hexadiene (C<sub>7</sub>H<sub>12</sub>)
2. 3,5-Dimethyl-1,4-hexadiene (C<sub>8</sub>H<sub>14</sub>)



### Enantiomer separation of 2-methylcyclohexanone

MN Appl. No. 202050

Column: FS-LIPODEX® A, 50 m x 0.25 mm ID, REF 723360.50,  
max. temperature 200/220 °C  
Injection: 1 µl (1 % in CH<sub>2</sub>Cl<sub>2</sub>)  
split 245 ml/min  
Carrier gas: 110 kPa H<sub>2</sub> (2.3 ml/min)  
Temperature: 70 °C  
Detector: FID 240 °C  
C<sub>7</sub>H<sub>12</sub>O



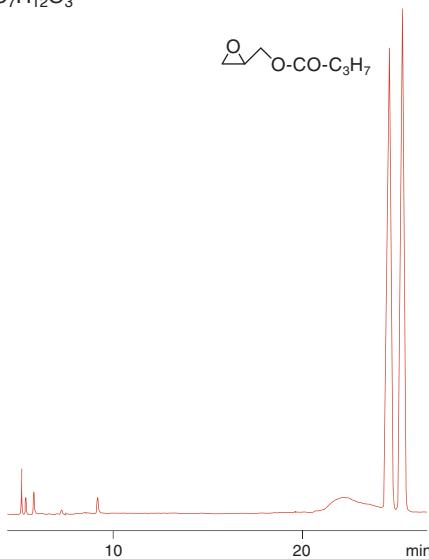
W.A. König et al., HRC 12 (1989) 35 – 39

## Chiral separations

### Enantiomer separation of glycidyl butyrate

MN Appl. No. 202731

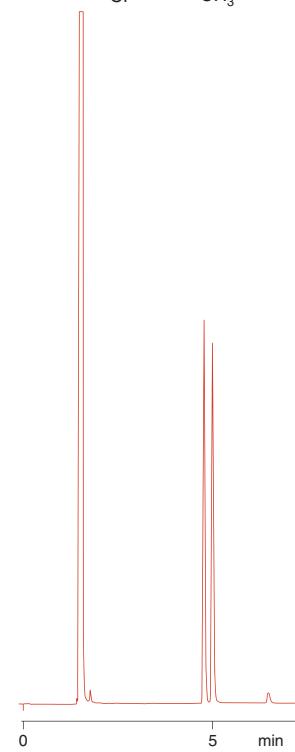
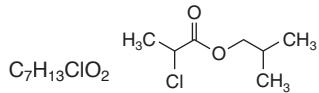
Column: FS-LIPODEX® A,  
25 m x 0.25 mm ID,  
REF 723360.25,  
max. temperature 200/220 °C  
Injection: 1 µl (1% in CH<sub>2</sub>Cl<sub>2</sub>)  
split 180 ml/min  
Carrier gas: 60 kPa H<sub>2</sub> (1.8 ml/min)  
Temperature: 60 °C  
Detector: FID 250 °C  
C<sub>7</sub>H<sub>12</sub>O<sub>3</sub>



### Enantiomer separation of 2-chloropropionic acid isobutyl ester

MN Appl. No. 202532

Column: FS-LIPODEX® E, 25 m x 0.25 mm ID, REF 723368.25,  
max. temperature 200/220 °C  
Injection: 1 µl, 1% in CH<sub>2</sub>Cl<sub>2</sub>, split 125 ml/min  
Carrier gas: 50 kPa H<sub>2</sub> (1.5 ml/min)  
Temperature: 90 °C  
Detector: FID 250 °C

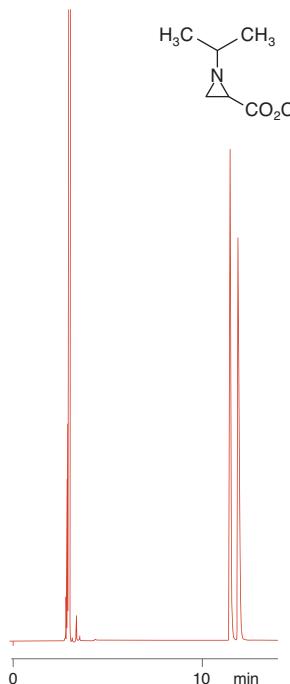


# Compounds C<sub>7</sub>



## Enantiomer separation of 1-*i*-propyl-2-carboxymethyl aziridine *MN Appl. No. 202361*

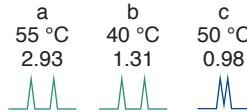
Column: FS-HYDRODEX  $\beta$ -PM,  
50 m x 0.25 mm ID,  
REF 723370.50,  
max. temperature 230/250 °C  
Injection: 0.1  $\mu$ l (1 % in CH<sub>2</sub>Cl<sub>2</sub>)  
split 100 ml/min  
Carrier gas: 120 kPa H<sub>2</sub> (1.7 ml/min)  
Temperature: 110 °C  
Detector: FID 250 °C  
C<sub>7</sub>H<sub>13</sub>NO<sub>2</sub>



## Enantiomers separation of 2-methylbutyric acid ethyl ester *MN Appl. No. 211400*

Columns: all 25 m x 0.25 mm ID:  
a) FS-HYDRODEX  $\beta$ -6TBDM  
(REF 723381.25),  
b) FS-LIPODEX® E  
(REF 723368.25),  
c) FS-HYDRODEX  $\beta$ -PM  
(REF 723370.25)  
Injection: 1  $\mu$ l, split, 250 °C  
Carrier gas: 1.1 ml/min He  
Detector: FID 250 °C  
C<sub>7</sub>H<sub>14</sub>O<sub>2</sub>

Temperature:	a 55 °C	b 40 °C	c 50 °C
Resolution:	2.93	1.31	0.98

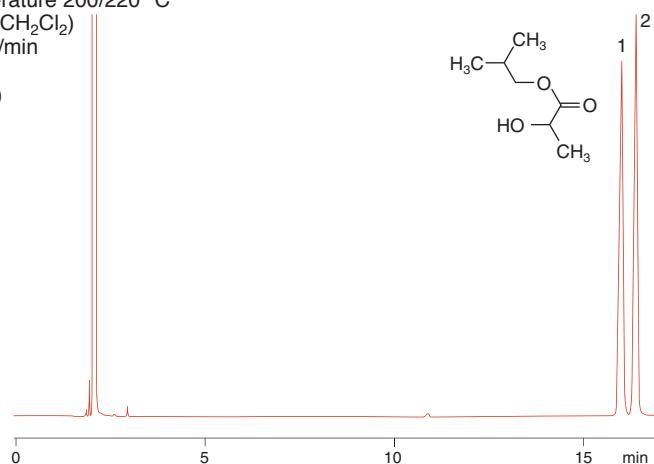


Courtesy of H. Casabianca, CNRS Service Central d'Analyse, Vernaison, France

## Chiral separations

### Enantiomer separation of isobutyl lactate MN Appl. No. 202802

Column: FS-LIPODEX® A, 50 m x 0.25 mm ID, REF 723360.50,  
max. temperature 200/220 °C  
Injection: 1 µl (1% in CH<sub>2</sub>Cl<sub>2</sub>)  
split 220 ml/min  
Carrier gas: 120 kPa H<sub>2</sub>  
(2.5 ml/min)  
Temperature: 60 °C  
Detector: FID 250 °C  
C<sub>7</sub>H<sub>14</sub>O<sub>3</sub>

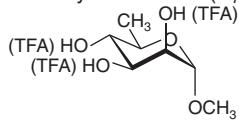


### Enantiomer separation of α-methylrhamnoside and α-methylquinovoside (TFA) MN Appl. No. 203041

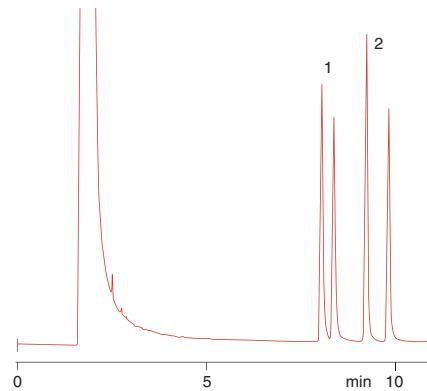
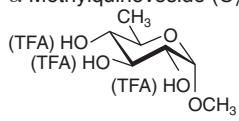
Column: LIPODEX® C, 50 m x 0.25 mm ID, REF 723364.50, max. temperature 200/220 °C  
Carrier gas: 1 bar H<sub>2</sub>  
Temperature: 90 °C  
Detector: FID

**Peaks:**

1. α-Methylrhamnoside (C<sub>7</sub>H<sub>14</sub>O<sub>5</sub>)

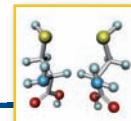


2. α-Methylquinovoside (C<sub>7</sub>H<sub>14</sub>O<sub>5</sub>)



W.A. König et al., HRC **12** (1989) 35 – 39

# Compounds C<sub>7</sub>

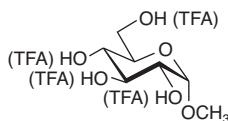


**Enantiomer separation of glucose and mannose ( $\alpha$ - and  $\beta$ -anomers, methylglycosides, TFA)**  
**MN Appl. No. 202412**

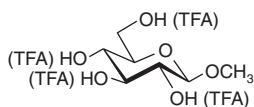
Column: LIPODEX® B, 50 m x 0.25 mm ID, REF 723362.50,  
 max. temperature 200/220 °C

Carrier gas: H<sub>2</sub>  
 Temperature: 155 °C  
 Detector: FID

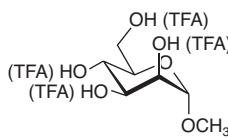
**Peaks:** (C<sub>7</sub>H<sub>14</sub>O<sub>6</sub>)  
 1.  $\alpha$ -Glucose



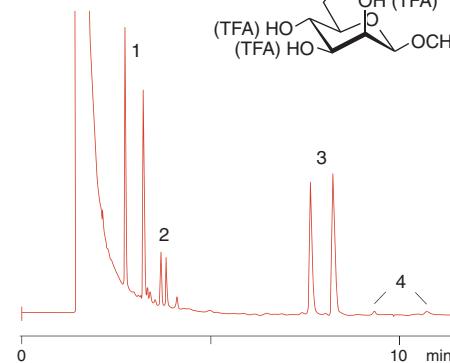
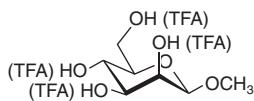
2.  $\beta$ -Glucose



3.  $\alpha$ -Mannose



4.  $\beta$ -Mannose



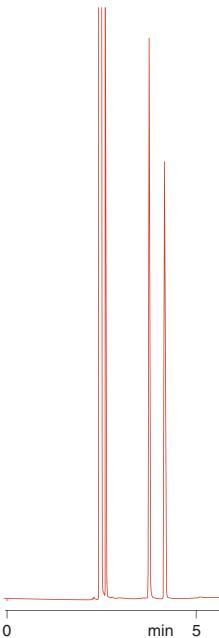
Courtesy of Prof. W.A. König, Hamburg,  
 Germany

See application 202402 at [www.mn-net.com](http://www.mn-net.com) for  
 separation of glucose anomer methylglycosides  
 on LIPODEX® A

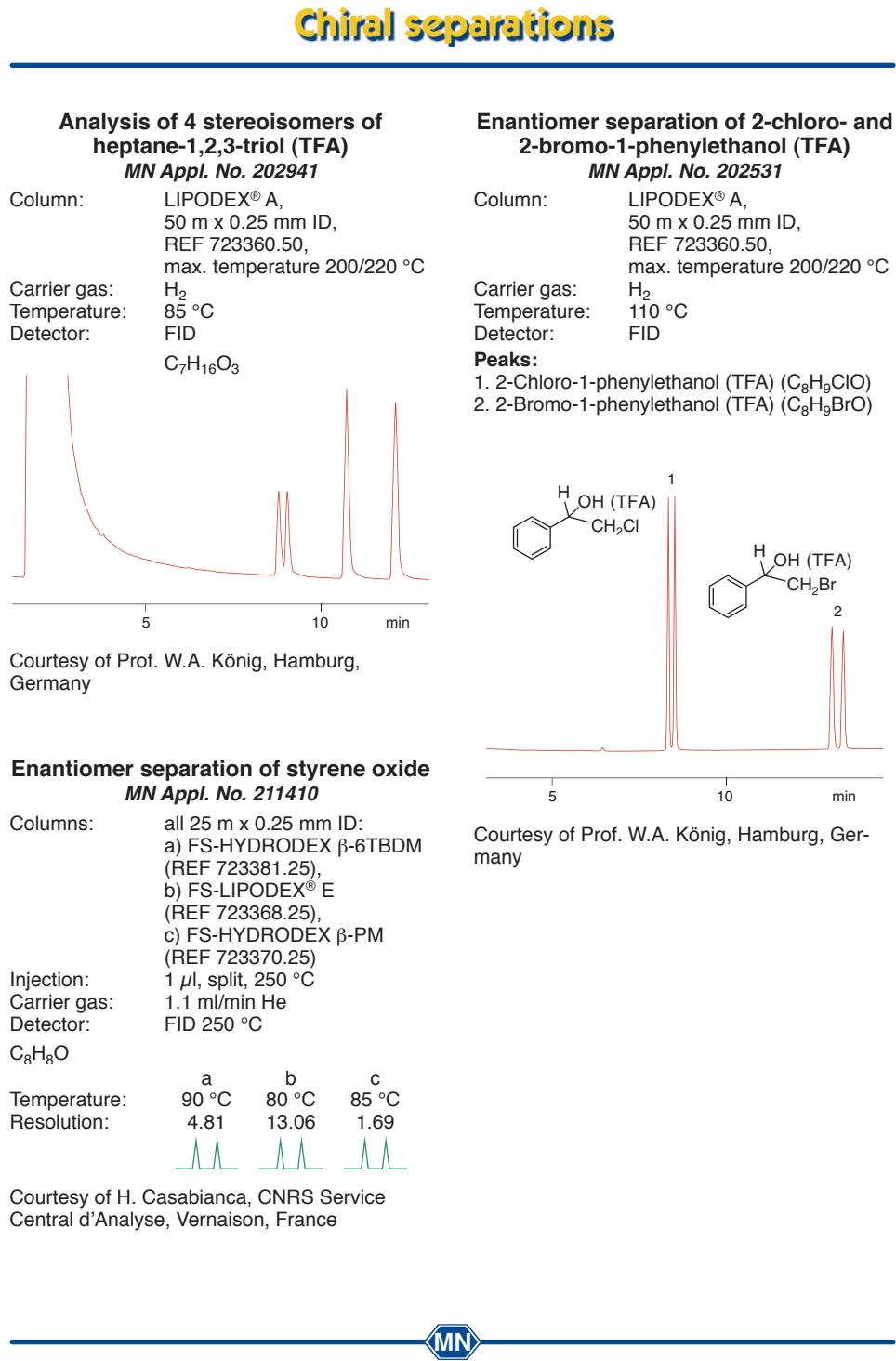
**Enantiomer separation of 2-heptanol (TFA)**  
**MN Appl. No. 202511**

Column: FS-LIPODEX® A,  
 50 m x 0.25 mm ID,  
 REF 723360.50,  
 max. temperature 200/220 °C

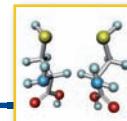
Injection: 0.1  $\mu$ l (1% in CH<sub>2</sub>Cl<sub>2</sub>)  
 split 320 ml/min  
 Carrier gas: 120 kPa H<sub>2</sub> (2.2 ml/min)  
 Temperature: 80 °C  
 Detector: FID 250 °C  
 C<sub>7</sub>H<sub>16</sub>O



2-heptanol (TFA) can also be separated on  
 LIPODEX® E  
 (see application 202501 at [www.mn-net.com](http://www.mn-net.com)).



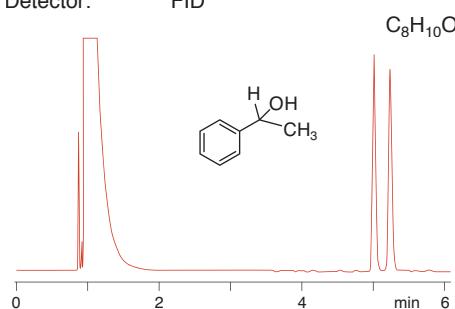
## Compounds C<sub>7</sub> – C<sub>8</sub>



### Enantiomer separation of $\alpha$ -methylbenzyl alcohol = $\alpha$ -phenylethanol

MN Appl. No. 212400

Column: FS-HYDRODEX®  $\beta$ -TBDAC,  
25 m x 0.25 mm ID,  
REF 723384.25,  
max. temperature 220/240 °C  
Carrier gas: 0.6 bar H<sub>2</sub>  
Temperature: 130 °C  
Detector: FID

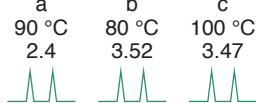


$\alpha$ -Phenylethanol can also be separated on HYDRODEX  $\beta$ -PM (application 202541), HYDRODEX  $\beta$ -3P (application 202571), LIPODEX® A (application 202561). See our application database at [www.mn-net.com](http://www.mn-net.com).

### Enantiomer separation of $\alpha$ -phenylethanol (TFA)

MN Appl. No. 211430

Columns: all 25 m x 0.25 mm ID:  
a) FS-HYDRODEX  $\beta$ -6TBDM  
(REF 723381.25),  
b) FS-LIPODEX® E  
(REF 723368.25),  
c) FS-HYDRODEX  $\beta$ -PM  
(REF 723370.25)  
Injection: 1  $\mu$ l, split, 250 °C  
Carrier gas: 1.1 ml/min He  
Detector: FID 250 °C  
Temperature: 90 °C  
Resolution: 2.4  
90 °C 2.4  
80 °C 3.52  
100 °C 3.47



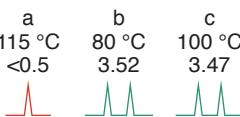
Courtesy of H. Casabianca, CNRS Service Central d'Analyse, Vernaison, France

$\alpha$ -Phenylethanol (TFA) can also be separated on HYDRODEX  $\beta$ -3P (application 202601) or LIPODEX® A (application 202591). See our application database at [www.mn-net.com](http://www.mn-net.com).

MN Appl. No. 211420

Columns: all 25 m x 0.25 mm ID:  
a) FS-HYDRODEX  $\beta$ -6TBDM  
(REF 723381.25),  
b) FS-LIPODEX® E  
(REF 723368.25),  
c) FS-HYDRODEX  $\beta$ -PM  
(REF 723370.25)  
Injection: 1  $\mu$ l, split, 250 °C  
Carrier gas: 1.1 ml/min He  
Detector: FID 250 °C

Temperature: 115 °C    80 °C    100 °C  
Resolution: <0.5    3.52    3.47



Courtesy of H. Casabianca, CNRS Service Central d'Analyse, Vernaison, France

## Chiral separations

### Enantiomer separation of 1-phenylethanol and *o*-, *m*-, *p*-methyl derivatives (TFA)

MN Appl. No. 202521

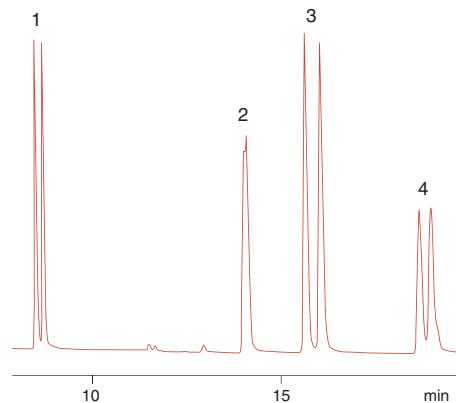
Column: LIPODEX® A, 50 m x 0.25 mm ID, REF 723360.50,  
max. temperature 200/220 °C

Carrier gas: H<sub>2</sub>  
Temperature: 75 °C

Detector: FID

**Peaks:**

1. 1-Phenylethanol (C<sub>8</sub>H<sub>10</sub>O)
2. 1-(2-Methylphenyl)ethanol (C<sub>9</sub>H<sub>12</sub>O)
3. 1-(3-Methylphenyl)ethanol
4. 1-(4-Methylphenyl)ethanol



Courtesy of Prof. W.A. König, Hamburg,  
Germany

### Enantiomer separation of $\alpha$ -phenylethylamine (TFA)

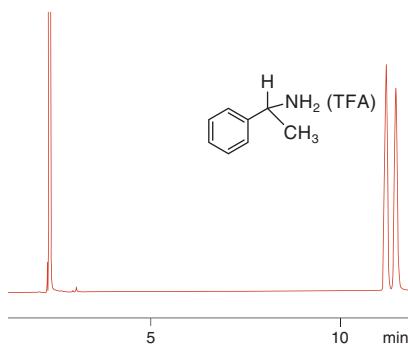
MN Appl. No. 202290

Column: FS-LIPODEX® A,  
50 m x 0.25 mm ID,  
REF 723360.50,  
max. temperature 200/220 °C

Injection: 1 µl (1% in CH<sub>2</sub>Cl<sub>2</sub>)  
split 245 ml/min

Carrier gas: 110 kPa H<sub>2</sub> (2.3 ml/min)  
Temperature: 120 °C

Detector: FID 240 °C  
C<sub>8</sub>H<sub>11</sub>N



## Compounds C<sub>8</sub> – C<sub>9</sub>

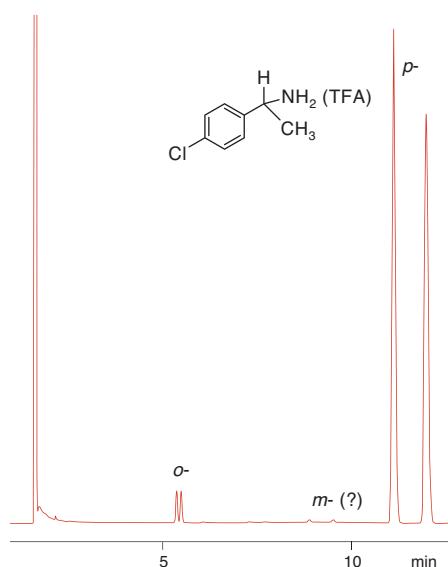


### Enantiomer separation of *p*-chlorophenylethylamine (TFA)

MN Appl. No. 202300

Column: FS-LIPODEX® D,  
25 m x 0.25 mm ID,  
REF 723366.25,  
max. temperature 200/220 °C  
Injection: 0.1 µl (5% in CH<sub>2</sub>Cl<sub>2</sub>)  
split 130 ml/min  
Carrier gas: 50 kPa H<sub>2</sub> (1.1 ml/min)  
Temperature: 170 °C  
Detector: FID 250 °C  
R is eluted before S.

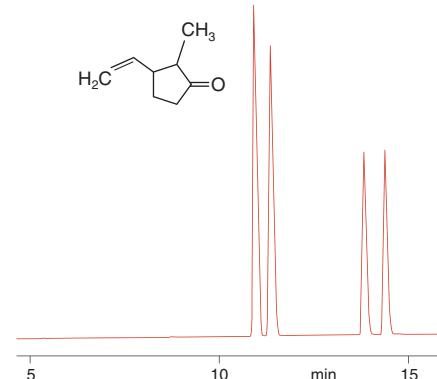
C<sub>8</sub>H<sub>11</sub>CIN



### Enantiomer separation of 2-methyl-3-vinylcyclopentanone

MN Appl. No. 202060

Column: FS-LIPODEX® E,  
25 m x 0.25 mm ID,  
REF 723368.25,  
max. temperature 200/220 °C  
Injection: 0.2 µl, split 80 ml/min  
Carrier gas: 50 kPa H<sub>2</sub> (1.2 ml/min)  
Temperature: 70 °C (3 min)  $\xrightarrow{2\text{ °C/min}}$  110 °C  
Detector: FID 250 °C  
C<sub>8</sub>H<sub>12</sub>O

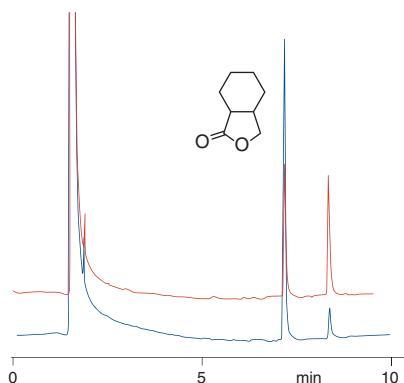


## Chiral separations

### Enantiomer composition of a bicyclic lactone (hexahydroisobenzofuranone)

MN Appl. No. 202962

Column: LIPODEX® D,  
50 m x 0.25 mm ID,  
REF 723366.50,  
max. temperature 200/220 °C  
Carrier gas: H<sub>2</sub>  
Temperature: 170 °C  
Detector: FID  
C<sub>8</sub>H<sub>12</sub>O<sub>2</sub>

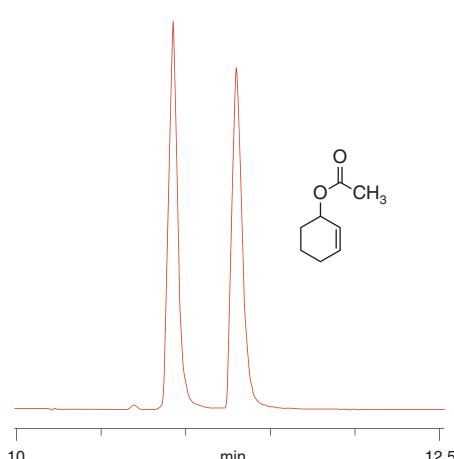


Courtesy of Prof. W.A. König, Hamburg,  
Germany

### Enantiomer separation of acetic acid cyclohex-2-enyl ester

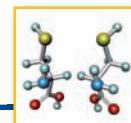
MN Appl. No. 212290

Column: FS-LIPODEX® G,  
25 m x 0.25 mm ID,  
REF723379.25,  
max. temperature 220/240 °C  
Carrier gas: 100 kPa H<sub>2</sub>  
Temperature: 50 °C (5 min) → 80 °C  
(5 min) 10 °C/min  
Detector: FID  
C<sub>8</sub>H<sub>12</sub>O<sub>2</sub>



Courtesy of Mrs. Vermeeren, AK Prof. Gais,  
Inst. für Org. Chemie, RWTH Aachen, Germany

# Compounds C<sub>8</sub>

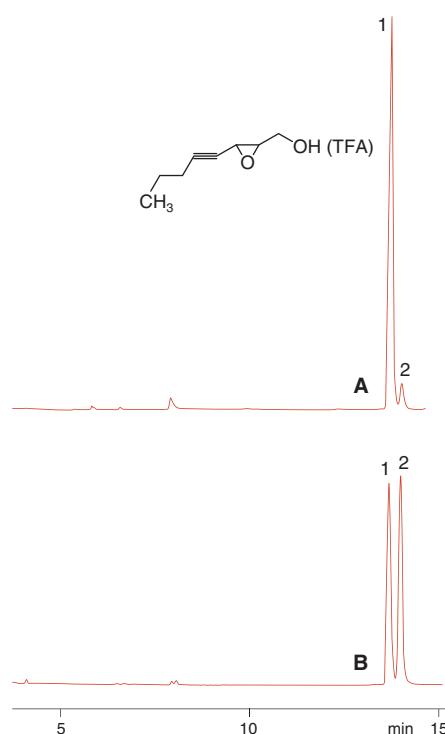


**Enantiomer separation of  
(E)-2,3-epoxy-4-octin-1-ol (TFA)**  
MN Appl. No. 202631

Column: LIPODEX® A,  
50 m x 0.25 mm ID,  
REF 723360.50,  
max. temperature 200/220 °C  
Carrier gas: 1 bar H<sub>2</sub>  
Temperature: 80 °C  
Detector: FID

**Peaks:**

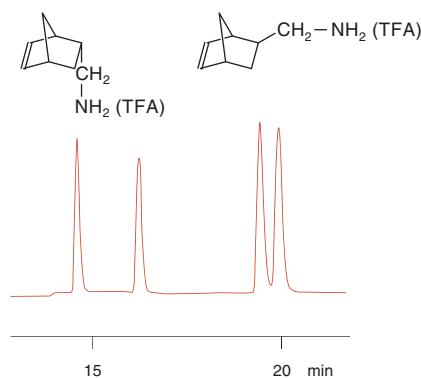
C<sub>8</sub>H<sub>12</sub>O<sub>2</sub>, 1. (2S,3S), 2. (2R,3R)  
Trace A: ee = 89.3 %, trace B = racemate



**Separation of 4 stereoisomers of  
5-aminomethylnorbornene (N-TFA)**  
MN Appl. No. 202350

Column: LIPODEX® D,  
50 m x 0.25 mm ID,  
REF 723366.50,  
max. temperature 200/220 °C  
Temperature: 150 °C  
Carrier gas: 1 bar H<sub>2</sub>  
Detector: FID

C<sub>8</sub>H<sub>13</sub>N



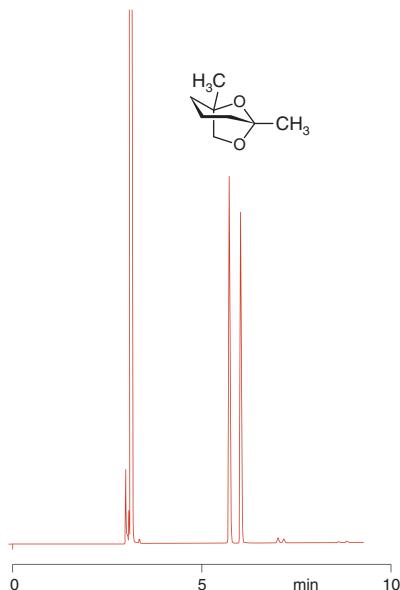
W.A. König, S. Lutz, G. Wenz, E. von der Bey,  
HRC **11** (1988) 506 – 509

W.A. König et al., Angew. Chem. Int. Ed. Engl.  
**28** (1989) 178

## Chiral separations

### Enantiomer separation of frontalin MN Appl. No. 202190

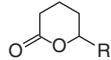
Column: FS-LIPODEX® E,  
50 m x 0.25 mm ID,  
REF 723368.50,  
max. temperature 200/220 °C  
Injection: 0.5 µl, split 180 ml/min  
Carrier gas: 100 kPa H<sub>2</sub> (1.5 ml/min)  
Temperature: 100 °C  
Detector: FID 250 °C  
C<sub>8</sub>H<sub>14</sub>O<sub>2</sub>



### Enantiomer analysis of δ-lactones

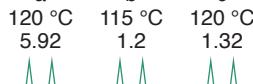
Columns: all 25 m x 0.25 mm ID:  
a) FS-HYDRODEX β-6TBDM  
(REF 723381.25),  
b) FS-LIPODEX® E  
(REF 723368.25),  
c) FS-HYDRODEX β-PM  
(REF 723370.25)

Injection: 1 µl, split, 250 °C  
Carrier gas: 1.1 ml/min He  
Detector: FID 250 °C



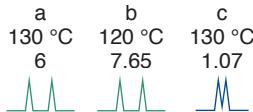
### δ-Octalactone (C<sub>8</sub>H<sub>14</sub>O<sub>2</sub>) MN Appl. No. 211450

Temperature: 120 °C  
Resolution: 5.92  
a  
115 °C  
b  
120 °C  
c



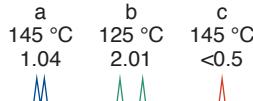
### δ-Nonalactone (C<sub>9</sub>H<sub>16</sub>O<sub>2</sub>) MN Appl. No. 211490

Temperature: 130 °C  
Resolution: 6  
a  
120 °C  
b  
130 °C  
c



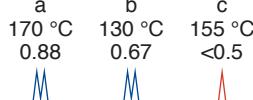
### δ-Decalactone (C<sub>10</sub>H<sub>18</sub>O<sub>2</sub>) MN Appl. No. 211670

Temperature: 145 °C  
Resolution: 1.04  
a  
125 °C  
b  
145 °C  
c



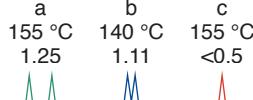
### δ-Undecalactone (C<sub>11</sub>H<sub>20</sub>O<sub>2</sub>) MN Appl. No. 211730

Temperature: 170 °C  
Resolution: 0.88  
a  
130 °C  
b  
155 °C  
c



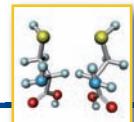
### δ-Dodecalactone MN Appl. No. 211760

Temperature: 155 °C  
Resolution: 1.25  
a  
140 °C  
b  
155 °C  
c



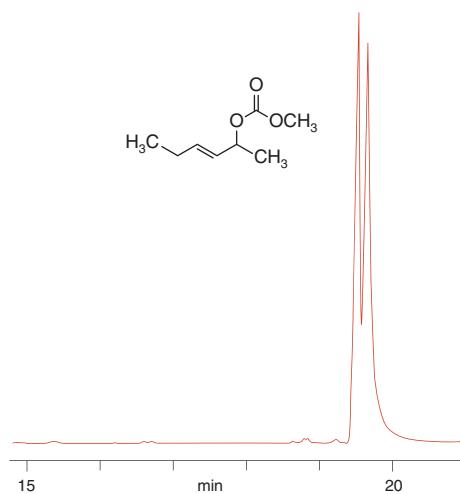
Courtesy of H. Casabianca, CNRS Service  
Central d'Analyse, Vernaison, France

## Compounds C<sub>8</sub> and greater



### Enantiomer separation of 2-hex-3-enyl methyl carbonate MN Appl. No. 212310

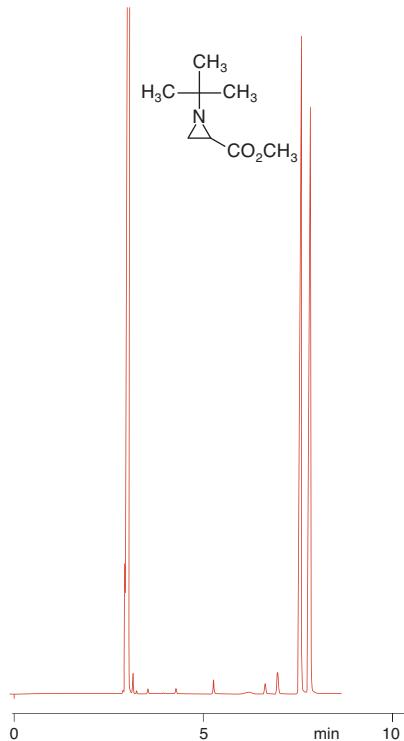
Column: FS-LIPODEX® G,  
25 m x 0.25 mm ID,  
REF723379.25,  
max. temperature 220/240 °C  
Carrier gas: 100 kPa H<sub>2</sub>  
Temperature: 50 °C (20 min)  $\xrightarrow{10\text{ °C/min}}$  80 °C  
(5 min)  $\xrightarrow{10\text{ °C/min}}$  120 °C (5 min)  
Detector: FID  
C<sub>8</sub>H<sub>14</sub>O<sub>3</sub>



Courtesy of Mrs. Vermeeren, AK Prof. Gais,  
Inst. für Org. Chemie, RWTH Aachen, Germany

### Enantiomer separation of 1-t-butyl-2-carboxymethyl aziridine MN Appl. No. 202371

Column: FS-HYDRODEX β-PM,  
50 m x 0.25 mm ID,  
REF 723370.50,  
max. temperature 230/250 °C  
Injection: 0.1 μl (1% in CH<sub>2</sub>Cl<sub>2</sub>)  
split 100 ml/min  
Carrier gas: 120 kPa H<sub>2</sub> (1.7 ml/min)  
Temperature: 130 °C  
Detector: FID 250 °C  
C<sub>8</sub>H<sub>15</sub>NO<sub>2</sub>

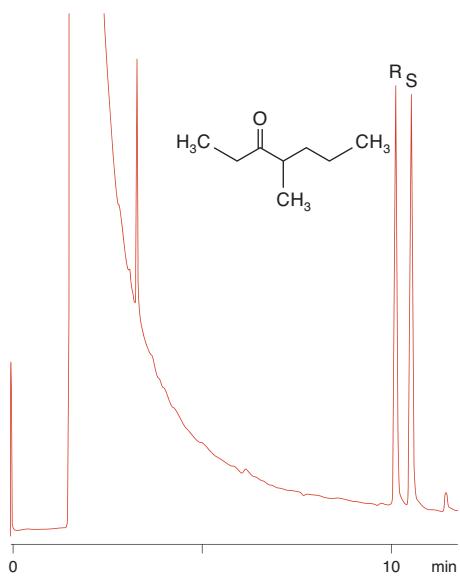


## Chiral separations

### Enantiomer separation of 4-methyl-3-heptanone (ant pheromone)

**MN Appl. No. 201900**

Column: LIPODEX® A,  
50 m x 0.25 mm ID,  
REF 723360.50,  
max. temperature 200/220 °C  
  
Carrier gas:  $\text{H}_2$   
Temperature: 85 °C  
Detector: FID  
  
 $\text{C}_8\text{H}_{16}\text{O}$



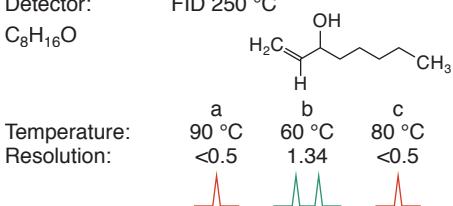
Courtesy of Prof. W.A. König, Hamburg,  
Germany

### Enantiomer separation of 1-octen-3-ol

**MN Appl. No. 211460**

Columns: all 25 m x 0.25 mm ID:  
a) FS-HYDRODEX  $\beta$ -6TBDM  
(REF 723381.25),  
b) FS-LIPODEX® E  
(REF 723368.25),  
c) FS-HYDRODEX  $\beta$ -PM  
(REF 723370.25)  
  
Injection: 1  $\mu\text{l}$ , split, 250 °C  
Carrier gas: 1.1 ml/min He  
Detector: FID 250 °C  
  
 $\text{C}_8\text{H}_{16}\text{O}$

Temperature: 90 °C  
Resolution: <0.5  
60 °C 1.34  
80 °C <0.5



Courtesy of H. Casabianca, CNRS Service  
Central d'Analyse, Vernaison, France

1-octen-3-ol can also be separated on  
LIPODEX® A  
(see application 202761 at [www.mn-net.com](http://www.mn-net.com)).

### Enantiomer separation of 1-octen-3-ol (TFA)

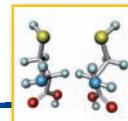
**MN Appl. No. 211470**

Columns: FS-HYDRODEX  $\beta$ -6TBDM,  
25 m x 0.25 mm ID,  
REF 723381.25  
  
Injection: 1  $\mu\text{l}$ , split, 250 °C  
Carrier gas: 1.1 ml/min He  
Temperature: 80 °C  
Detector: FID 250 °C  
Resolution: 1.49



Courtesy of H. Casabianca, CNRS Service  
Central d'Analyse, Vernaison, France

## Compounds C<sub>8</sub> – C<sub>9</sub>



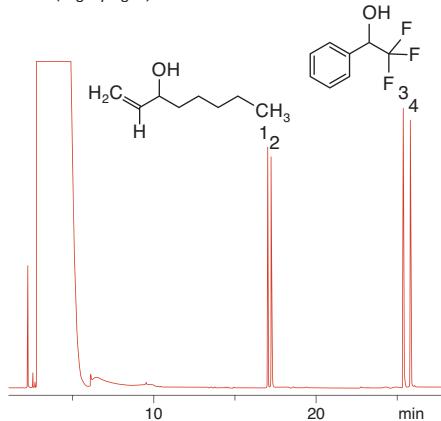
### Enantiomer separation of 1-octen-3-ol and 2,2,2-trifluorophenylethanol

MN Appl. No. 212460

Column: FS-HYDRODEX®  $\beta$ -TBDAC,  
50 m x 0.25 mm ID,  
REF 723384.50,  
max. temperature 220/240 °C  
Sample: 0.1 % in CH<sub>2</sub>Cl<sub>2</sub>  
Carrier gas: 1.2 bar H<sub>2</sub>  
Temperature: 80 °C (5 min)  $\xrightarrow{3\text{ °C/min}}$  140 °C  
(5 min)  
Detector: FID

**Peaks:**

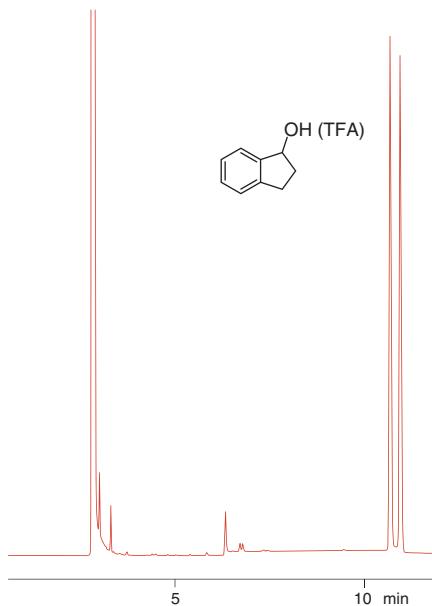
1. (S)-1-Octen-3-ol (C<sub>8</sub>H<sub>16</sub>O)
2. (R)-1-Octen-3-ol
- 3./4. (R)/(S)-2,2,2-Trifluorophenylethanol (C<sub>8</sub>H<sub>7</sub>F<sub>3</sub>O)



### Enantiomer separation of 1-indanol (TFA)

MN Appl. No. 202781

Column: FS-HYDRODEX  $\beta$ -PM,  
50 m x 0.25 mm ID,  
REF 723370.50,  
max. temperature 230/250 °C  
Injection: 1  $\mu$ l (1 % in CH<sub>2</sub>Cl<sub>2</sub>)  
split 150 ml/min  
Carrier gas: 120 kPa H<sub>2</sub> (1.7 ml/min)  
Temperature: 130 °C  
Detector: FID 250 °C  
C<sub>9</sub>H<sub>10</sub>O



1-indanol can also be separated on LIPODEX® A (see application 202771 at [www.mn-net.com](http://www.mn-net.com)).

## Chiral separations

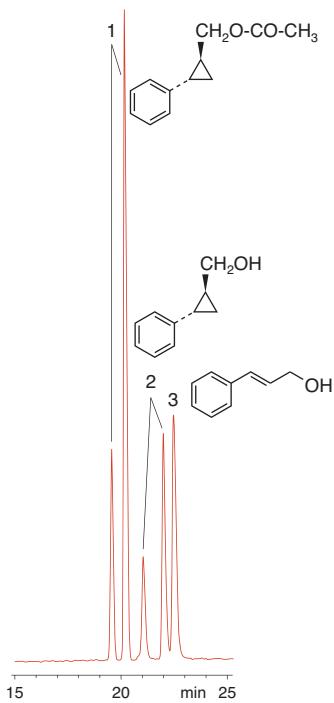
### Enantiomer separation of phenylcyclopropylmethanol, phenylcyclopropylmethyl acetate and cinnamyl alcohol (TFA)

MN Appl. No. 212680

Column: FS-HYDRODEX  $\beta$ -TBDAC,  
25 m x 0.25 mm ID,  
REF 723384.25,  
max. temperature 220/240 °C  
Injection: 1  $\mu$ l (0.1 % in methyl *t*-butyl  
ether), 220 °C, split 15 ml/min  
Carrier gas: 0.6 bar H<sub>2</sub>  
Temperature: 130 °C  
Detector: FID 220 °C

**Peaks:**

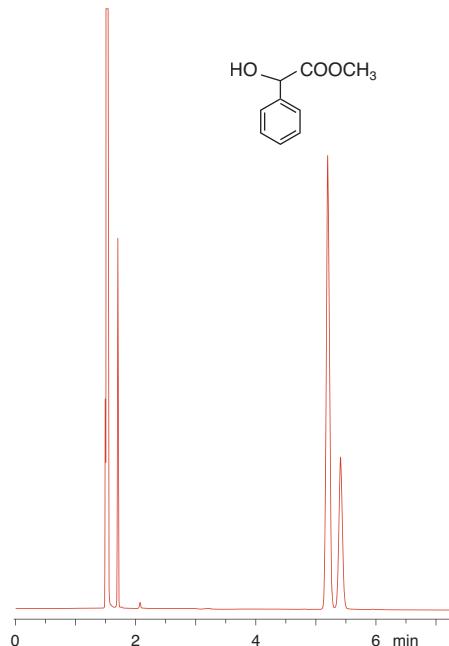
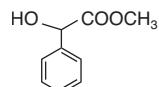
1. ( $\pm$ )-trans-2-Phenylcyclopropylmethyl acetate (C<sub>12</sub>H<sub>14</sub>O<sub>2</sub>)
2. ( $\pm$ )-trans-2-Phenylcyclopropylmethanol (C<sub>10</sub>H<sub>12</sub>O)
3. Cinnamyl alcohol (C<sub>9</sub>H<sub>10</sub>O)



### Enantiomer separation of mandelic acid methyl ester

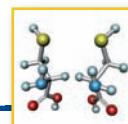
MN Appl. No. 213020

Column: FS-HYDRODEX  $\gamma$ -TBDAC,  
25 m x 0.25 mm ID,  
REF 723387.25,  
max. temperature 220/240 °C  
Injection: 1  $\mu$ l, split 50 ml/min  
Carrier gas: 0.6 bar H<sub>2</sub>  
Temperature: 170 °C  
Detector: FID  
C<sub>9</sub>H<sub>10</sub>O<sub>3</sub>



Mandelic acid methyl ester can also be separated on LIPODEX® A (application 202672) or LIPODEX® C (application 202682). See our application database at [www.mn-net.com](http://www.mn-net.com).

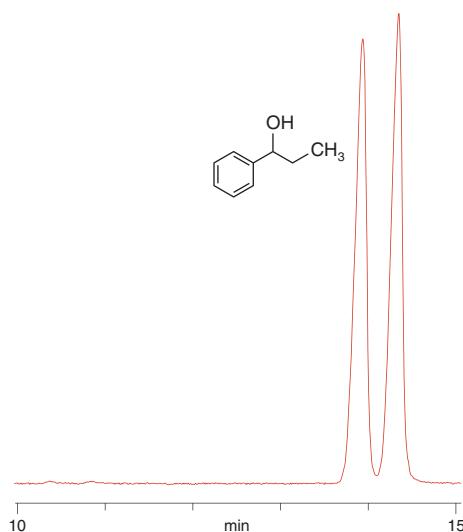
## Compounds C<sub>9</sub> – C<sub>12</sub>



### Enantiomer separation of 1-phenyl-propan-1-ol

MN Appl. No. 212320

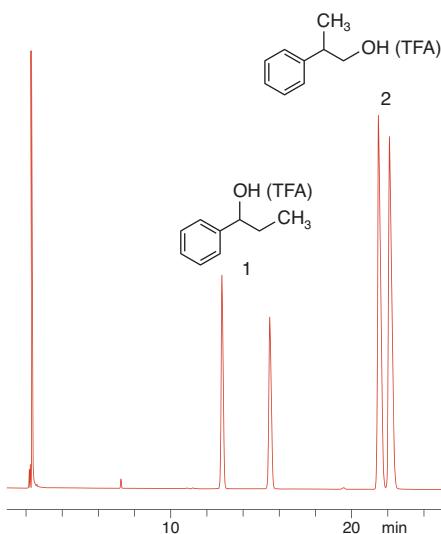
Column: FS-LIPODEX® G,  
25 m x 0.25 mm ID,  
REF723379.25,  
max. temperature 220/240 °C  
Carrier gas: 100 kPa H<sub>2</sub>  
Temperature: 50 °C (5 min) → 80 °C  
(5 min)  
Detector: FID  
C<sub>9</sub>H<sub>12</sub>O



### Enantiomer separation of phenylpropanols (TFA)

MN Appl. No. 212970

Column: FS-HYDRODEX γ-TBDAC,  
50 m x 0.25 mm ID,  
REF723378.50,  
max. temperature 220/240 °C  
Injection: 1 µl, split 50 ml/min  
Carrier gas: 1.2 bar H<sub>2</sub>  
Temperature: 100 °C  
Detector: FID  
**Peaks:** (C<sub>9</sub>H<sub>12</sub>O)  
1. 1-Phenylpropan-1-ol (TFA)  
2. 2-Phenylpropan-1-ol (TFA)



Courtesy of Mrs. Vermeeren, AK Prof. Gais,  
Inst. für Org. Chemie, RWTH Aachen, Germany

## Chiral separations

## Enantiomer separation of amphetamine, ephedrine and norephedrine (N,O-TFA)

MN Appl. No. 202310

Column: LIPODEX® D,  
           50 m x 0.25 mm ID,  
           REF 723366.50,  
           max. temperature 200/220 °C

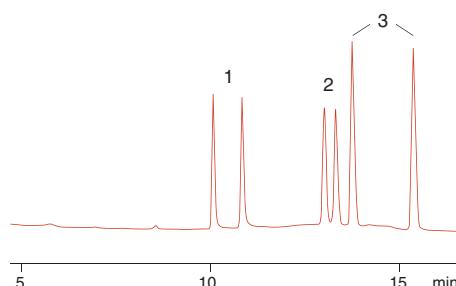
Carrier gas: H<sub>2</sub>

Temperature: 150 °C

Detector: FID

**Peaks:**

1. Amphetamine
2. Ephedrine
3. Norephedrine



Courtesy of Prof. W.A. König, Hamburg,  
Germany

## Enantiomer separation of sympathomimetic amines (N,O-TFA)

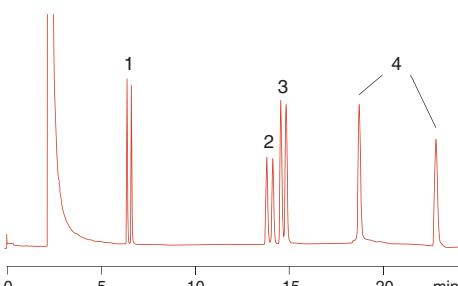
MN Appl. No. 202320

Column: LIPODEX® D,  
50 m x 0.25 mm ID,  
REF 723366.50,  
max. temperature 200/220 °C

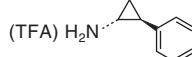
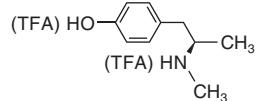
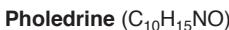
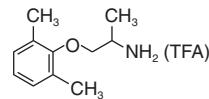
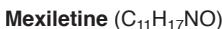
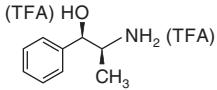
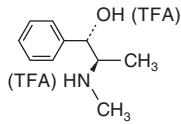
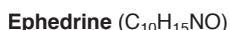
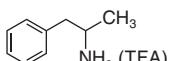
Carrier gas: 1 bar H<sub>2</sub>  
Temperature: 175 °C  
Detector: FID

**Peaks:**

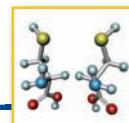
1. Amphetamine
2. Mexiletine
3. Pholedrine
4. Tranylcypromine



W.A. König, S. Lutz, G. Wenz, E. von der Bey,  
HBC 11 (1988) 506 – 509



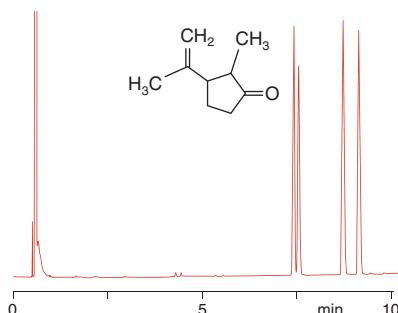
## Compounds C<sub>9</sub> – C<sub>11</sub>



### Enantiomer separation of 3-isopropenyl-2-methylcyclopentanone

MN Appl. No. 202070

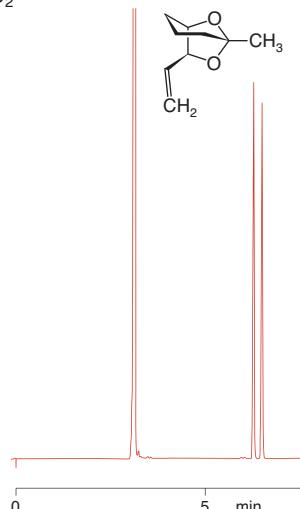
Column: FS-LIPODEX® E,  
25 m x 0.25 mm ID,  
REF 723368.25,  
max. temperature 200/220  
°C  
Injection: 0.2 µl, split 80 ml/min  
Carrier gas: 50 kPa H<sub>2</sub> (1.2 ml/min)  
Temperature: 70 °C (3 min)  $\xrightarrow{2^{\circ}\text{C}/\text{min}}$  110  
°C  
Detector: FID 250 °C  
C<sub>9</sub>H<sub>14</sub>O



### Enantiomer separation of vinyl-endo-brevicomin

MN Appl. No. 202130

Column: FS-LIPODEX® E,  
50 m x 0.25 mm ID,  
REF 723368.50,  
max. temperature 200/220 °C  
Injection: 0.5 µl, split 180 ml/min  
Carrier gas: 100 kPa H<sub>2</sub> (1.5 ml/min)  
Temperature: 120 °C  
Detector: FID 250 °C  
C<sub>9</sub>H<sub>14</sub>O<sub>2</sub>



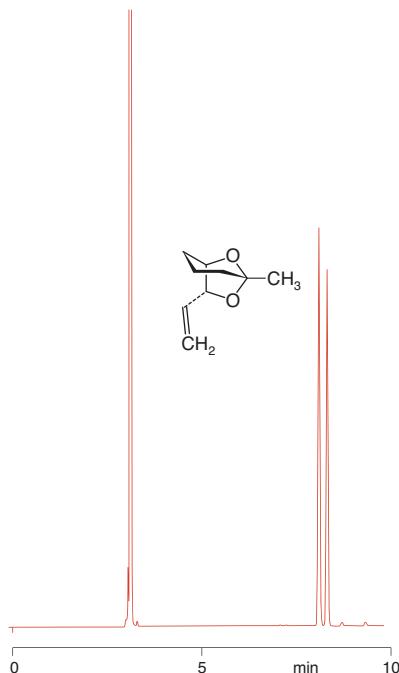
Vinyl-endo-brevicomin can also be separated on HYDRODEX β-PM (see application 202120 at [www.mn-net.com](http://www.mn-net.com)).

## Chiral separations

### Enantiomer separation of vinyl-exo-brevicomin

MN Appl. No. 202150

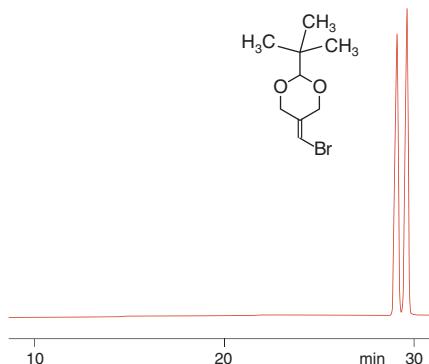
Column: FS-LIPODEX® E,  
50 m x 0.25 mm ID,  
REF 723368.50,  
max. temperature 200/220 °C  
Injection: 0.5 µl, split 180 ml/min  
Carrier gas: 100 kPa H<sub>2</sub> (1.5 ml/min)  
Temperature: 105 °C  
Detector: FID 250 °C  
C<sub>9</sub>H<sub>14</sub>O<sub>2</sub>



### Enantiomer separation of 2-t-butyl-5-bromomethylene 1,3-dioxane

MN Appl. No. 202230

Column: FS-LIPODEX® A,  
50 m x 0.25 mm ID,  
REF 723360.50,  
max. temperature 200/220 °C  
Injection: 0.5 µl (2 % in CH<sub>2</sub>Cl<sub>2</sub>)  
split 92 ml/min  
Carrier gas: 130 kPa H<sub>2</sub> (2.0 ml/min)  
Temperature: 105 °C  
Detector: FID 250 °C  
C<sub>9</sub>H<sub>15</sub>BrO<sub>2</sub>



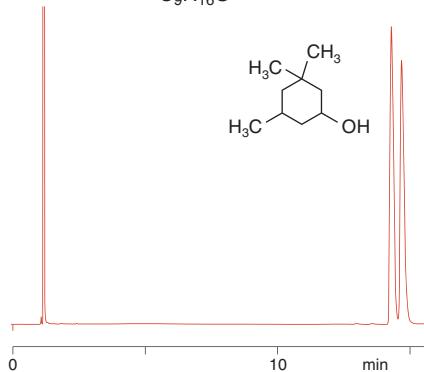
Vinyl-exo-brevicomin can also be separated on HYDRODEX β-PM (see application 202140 at [www.mn-net.com](http://www.mn-net.com)).

## Compounds C<sub>9</sub>



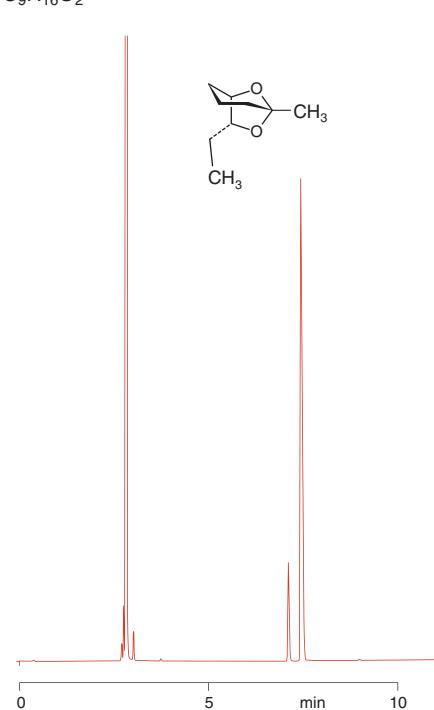
### Enantiomer separation of cis-3,3,5-trimethylcyclohexanol MN Appl. No. 201970

Column: FS-LIPODEX® E,  
25 m x 0.25 mm ID,  
REF 723368.25,  
max. temperature 200/220 °C  
Injection: 0.1 µl (1% in CH<sub>2</sub>Cl<sub>2</sub>)  
split 125 ml/min  
Carrier gas: 60 kPa H<sub>2</sub> (1.6 ml/min)  
Temperature: 80 °C  
Detector: FID 250 °C  
C<sub>9</sub>H<sub>16</sub>O



### Enantiomer separation of (-)-exo-brevicomin MN Appl. No. 202160

Column: FS-HYDRODEX β-PM,  
50 m x 0.25 mm ID,  
REF 723370.50,  
max. temperature 230/250 °C  
Injection: 0.1 µl (1% in CH<sub>2</sub>Cl<sub>2</sub>)  
split 150 ml/min  
Carrier gas: 120 kPa H<sub>2</sub> (1.7 ml/min)  
Temperature: 120 °C  
Detector: FID 250 °C  
C<sub>9</sub>H<sub>16</sub>O<sub>2</sub>



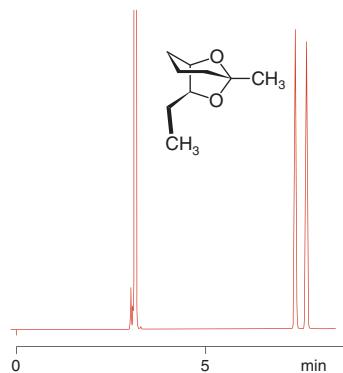
(-)-exo-brevicomin can also be separated on  
LIPODEX® E  
(see application 202170 at [www.mn-net.com](http://www.mn-net.com)).

## Chiral separations

### Enantiomer separation of endo-brevicomin

MN Appl. No. 202180

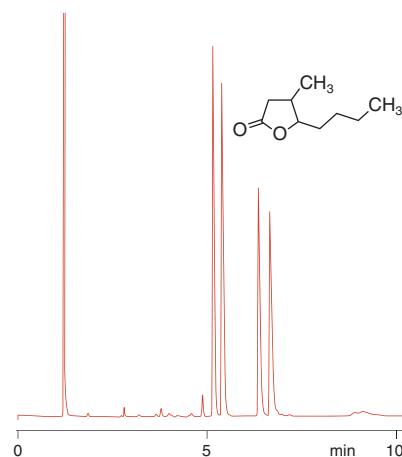
Column: FS-LIPODEX® E,  
50 m x 0.25 mm ID,  
REF 723368.50,  
max. temperature 200/220 °C  
Injection: 0.5 µl, split 180 ml/min  
Carrier gas: 100 kPa H<sub>2</sub> (1.5 ml/min)  
Temperature: 110 °C  
Detector: FID 250 °C  
C<sub>9</sub>H<sub>16</sub>O<sub>2</sub>



### Enantiomer analysis of 4-butyl-3-methylbutyrolactone (Whisky lactone)

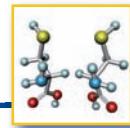
MN Appl. No. 202862

Column: FS-LIPODEX® E,  
25 m x 0.25 mm ID,  
REF 723368.25,  
max. temperature 200/220 °C  
Injection: 0.1 µl (1 % in CH<sub>2</sub>Cl<sub>2</sub>)  
split 95 ml/min  
Carrier gas: 60 kPa H<sub>2</sub> (1.7 ml/min)  
Temperature: 145 °C  
Detector: FID 250 °C  
C<sub>9</sub>H<sub>16</sub>O<sub>2</sub>



Whisky lactone can also be separated on HYDRODEX β-PM (application 202882) or LIPODEX® A (application 202852). See our application database at [www.mn-net.com](http://www.mn-net.com).

## Compounds C<sub>9</sub> – C<sub>11</sub>



### Enantiomer separation of spiroacetals MN Appl. No. 202200

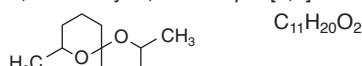
Column: LIPODEX® A, 50 m x 0.25 mm ID, REF 723360.50,  
max. temperature 200/ 220 °C  
Temperature: 80 °C  
Carrier gas: H<sub>2</sub>  
Detector: FID

**Peaks:**

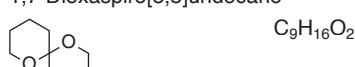
1. 7-Methyl-1,6-dioxaspiro[4,5]decane



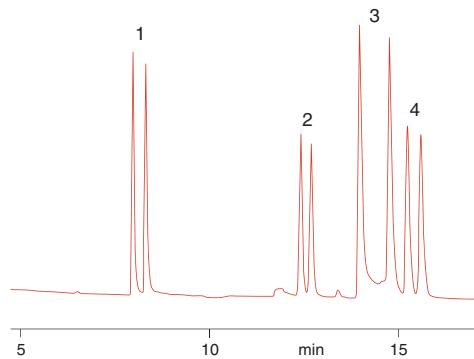
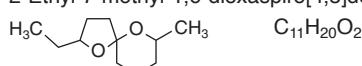
2. 2,8-Dimethyl-1,7-dioxaspiro[5,5]undecane



3. 1,7-Dioxaspiro[5,5]undecane



4. 2-Ethyl-7-methyl-1,6-dioxaspiro[4,5]decane



Courtesy of Prof. W.A. König, Hamburg, Germany

### Enantiomer separation of 1,7-dioxaspiro[5,5]undecane MN Appl. No. 202200

Column: FS-LIPODEX® A,  
50 m x 0.25 mm ID,  
REF 723360.50,  
max. temperature 200/220 °C

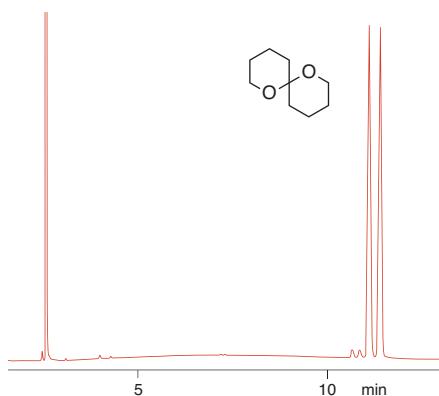
Injection: 0.5 µl (1% in CH<sub>2</sub>Cl<sub>2</sub>)  
split 320 ml/min

Carrier gas: 120 kPa H<sub>2</sub> (2.2 ml/min)

Temperature: 95 °C

Detector: FID 240 °C

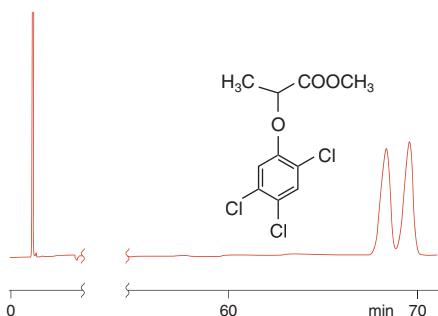
C<sub>9</sub>H<sub>16</sub>O<sub>2</sub>



## Chiral separations

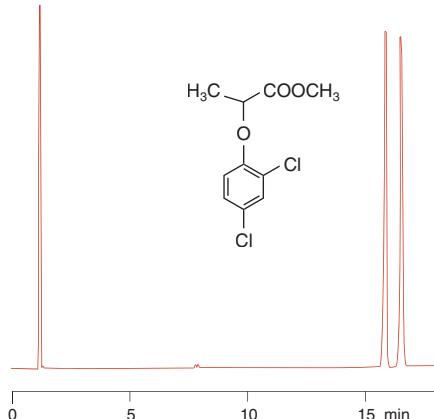
### Enantiomers separation of fenoprop methyl ester MN Appl. No. 202552

Column: FS-HYDRODEX  $\beta$ -3P,  
25 m x 0.25 mm ID,  
REF 723358.25, max. tem-  
perature 230/250 °C  
Injection: 1  $\mu$ l (1% in CH<sub>2</sub>Cl<sub>2</sub>)  
split 130 ml/min  
Carrier gas: 60 kPa H<sub>2</sub> (1.9 ml/min)  
Temperature: 140 °C  
Detector: FID 250 °C  
C<sub>10</sub>H<sub>9</sub>Cl<sub>3</sub>O<sub>3</sub>

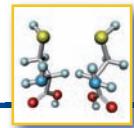


### Enantiomer separation of dichlorprop methyl ester MN Appl. No. 202542

Column: FS-HYDRODEX  $\beta$ -3P,  
25 m x 0.25 mm ID,  
REF 723358.25,  
max. temperature 230/250 °C  
Injection: 0.1  $\mu$ l (1% in CH<sub>2</sub>Cl<sub>2</sub>)  
split 130 ml/min  
Carrier gas: 60 kPa H<sub>2</sub> (1.9 ml/min)  
Temperature: 160 °C  
Detector: FID 250 °C  
C<sub>10</sub>H<sub>10</sub>Cl<sub>2</sub>O<sub>3</sub>

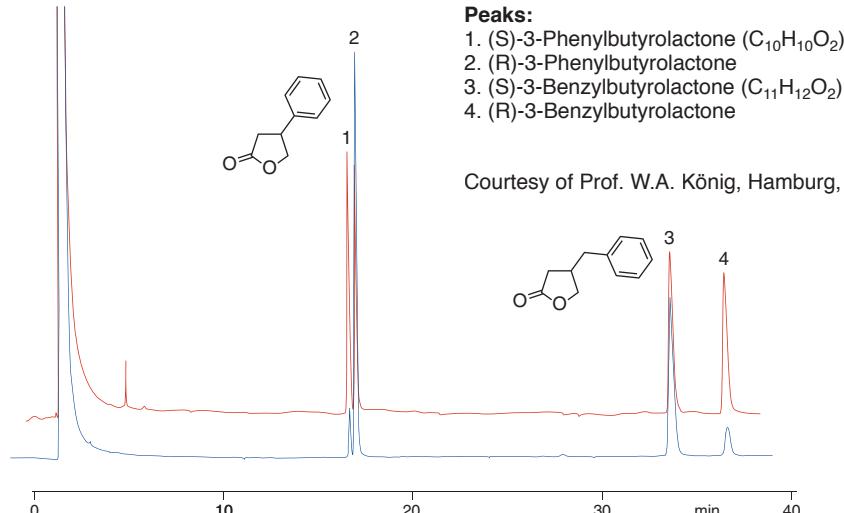


## Compounds C<sub>10</sub> – C<sub>11</sub>



### Enantiomer composition of 3-phenyl- and 3-benzylbutyrolactone MN Appl. No. 202932

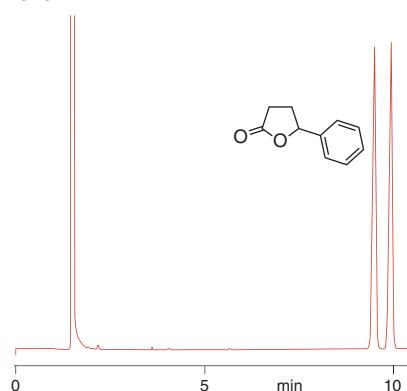
Column: LIPODEX® B, 50 m x 0.25 mm ID, REF 723362.50,  
max. temperature 200/220 °C  
Carrier gas: H<sub>2</sub>  
Temperature: 175 °C  
Detector: FID



### Enantiomer analysis of γ-phenylbutyrolactone MN Appl. No. 202942

Column: FS-LIPODEX® E, 25 m x 0.25 mm ID, REF 723368.25,  
max. temperature 200/220 °C  
Injection: 0.5 µl, split 1:100  
Carrier gas: 60 kPa H<sub>2</sub>  
Temperature: 170 °C  
Detector: FID 250 °C  
C<sub>10</sub>H<sub>10</sub>O<sub>2</sub>

γ-phenylbutyrolactone can also be separated on HYDRODEX β-PM  
(see application 202952 at [www.mn-net.com](http://www.mn-net.com)).



## Chiral separations

**Enantiomer separation of  
2-phenylpropionic acid (hydratropic  
acid) methyl ester**

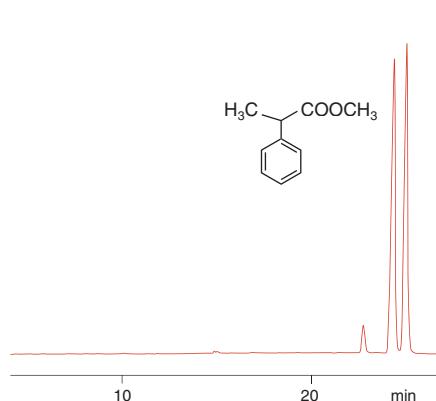
**MN Appl. No. 202572**

Column: FS-LIPODEX® E,  
25 m x 0.25 mm ID,  
REF 723368.25,  
max. temperature  
200/220 °C

Injection: 0.1 µl (0.1% in CH<sub>2</sub>Cl<sub>2</sub>)  
split 100 ml/min

Carrier gas: 60 kPa H<sub>2</sub>  
Temperature: 80 °C  
Detector: FID 250 °C

C<sub>10</sub>H<sub>12</sub>O<sub>2</sub>



**Enantiomer separation of  
desoxyephedrine (TFA)**

**MN Appl. No. 202330**

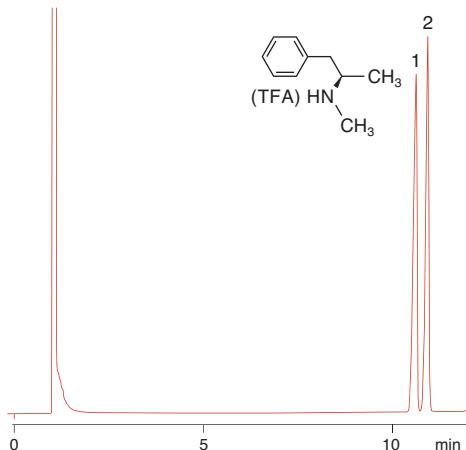
Column: FS-LIPODEX® E,  
25 m x 0.25 mm ID,  
REF 723368.25,  
max. temperature 200/220 °C

Injection: 0.5 µl (1% in CH<sub>2</sub>Cl<sub>2</sub>)  
split 70 ml/min

Carrier gas: 60 kPa H<sub>2</sub> (1.3 ml/min)  
Temperature: 130 °C  
Detector: FID 250 °C

**Peaks:**

1. D-Desoxyephedrine (TFA) C<sub>10</sub>H<sub>15</sub>N
2. L-Desoxyephedrine (TFA)



**Enantiomer separation of carvone**

**MN Appl. No. 211500**

Columns: all 25 m x 0.25 mm ID: a) FS-HYDRODEX β-6TBDM (REF 723381.25),  
b) FS-LIPODEX® E (REF 723368.25),  
c) FS-HYDRODEX β-PM (REF 723370.25)

Injection: 1 µl, split, 250 °C

Carrier gas: 1.1 ml/min He

Detector: FID 250 °C

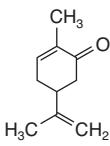
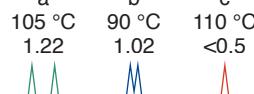
C<sub>10</sub>H<sub>14</sub>O<sub>2</sub>

Temperature: a 105 °C  
Resolution: b 90 °C  
c 110 °C

1.22

1.02

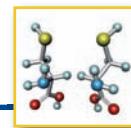
<0.5



Courtesy of H. Casabianca, CNRS Service Central d'Analyse, Vernaison, France

Carvone can also be separated on LIPODEX® A (see applications 202110 and 211940 at [www.mn-net.com](http://www.mn-net.com)).

## Compounds C<sub>10</sub> – C<sub>12</sub>



Chiral constituents of peppermint oil  
MN Appl. No. 250410

Column: FS-LIPODEX® G, 25 m x 0.25 mm ID, max. temp. 220/240 °C, REF 723379.25

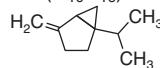
Carrier gas: 50 kPa H<sub>2</sub>

Temperature: 75 °C, isothermal

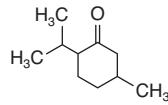
Detector: FID

**Peaks:**

1. (+)-trans-Sabinene hydrate (C<sub>10</sub>H<sub>16</sub>)



2. (+)-Menthone



3. (+)-Isomenthone

4. (-)-Menthone

5. (-)-Isomenthone

(C<sub>10</sub>H<sub>18</sub>O)

6. (+)-Menthofuran

(C<sub>10</sub>H<sub>14</sub>O)

7. (-)-Isopulegol

(C<sub>10</sub>H<sub>18</sub>O)

8. (-)-Menthyl acetate

(C<sub>12</sub>H<sub>22</sub>O<sub>2</sub>)

9. (+)-Pulegone

(C<sub>10</sub>H<sub>16</sub>O)

10. (+)-Neomenthol

11. (-)-Neomenthol

12. (+)-Neoisomenthol

13. (+)-Menthol

14. (-)-Neoisomenthol

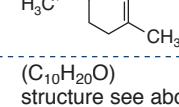
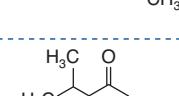
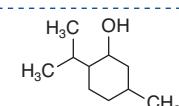
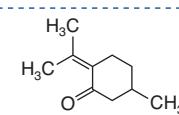
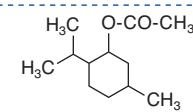
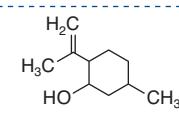
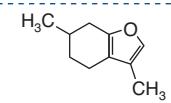
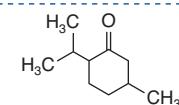
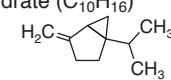
15. (+)-Piperitone

(C<sub>10</sub>H<sub>16</sub>O)

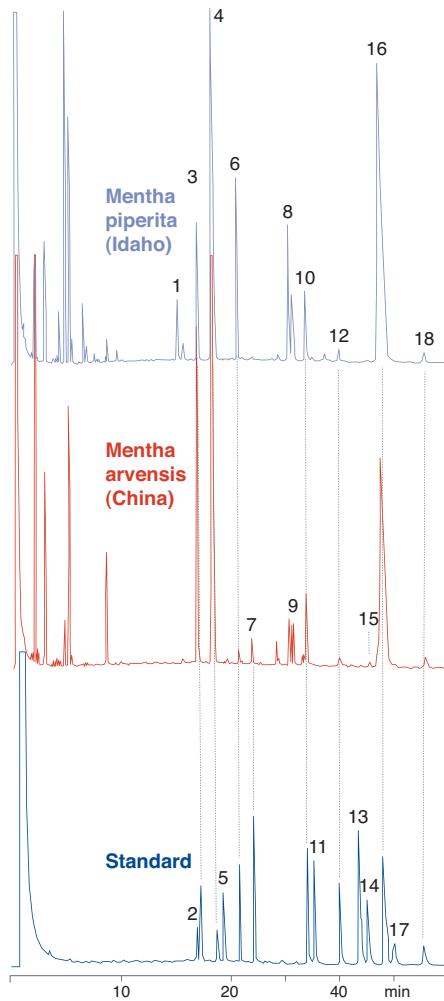
16. (-)-Menthol

17. (+)-Isomenthol

18. (-)-Isomenthol



(C<sub>10</sub>H<sub>20</sub>O) structure see above



W. A. König et al., High Resol. Chromatogr. **20** (1997) 55 – 61

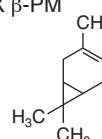
## Chiral separations

### Enantiomer separation of $\Delta^3$ -carene MN Appl. No. 211530

Columns: all 25 m x 0.25 mm ID:  
 a) FS-HYDRODEX  $\beta$ -6TBDM (REF 723381.25),  
 b) FS-LIPODEX® E (REF 723368.25),  
 c) FS-HYDRODEX  $\beta$ -PM (REF 723370.25)

Injection: 1  $\mu$ l, split, 250 °C  
 Carrier gas: 1.1 ml/min He  
 Detector: FID 250 °C  
 $C_{10}H_{16}$

Temperature: a 75 °C      b 50 °C      c 70 °C  
 Resolution: 3.01      8.65      2.41



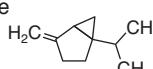
Courtesy of H. Casabianca, CNRS Service Central d'Analyse, Vernaison, France

### Enantiomer separation of sabinene MN Appl. No. 211540

Columns: all 25 m x 0.25 mm ID:  
 a) FS-HYDRODEX  $\beta$ -6TBDM (REF 723381.25),  
 b) FS-LIPODEX® E (REF 723368.25),  
 c) FS-HYDRODEX  $\beta$ -PM (REF 723370.25)

Injection: 1  $\mu$ l, split, 250 °C  
 Carrier gas: 1.1 ml/min He  
 Detector: FID 250 °C  
 $C_{10}H_{16}$

Temperature: a 75 °C      b 50 °C      c gradient  
 Resolution: 5.19      2.1      1.29

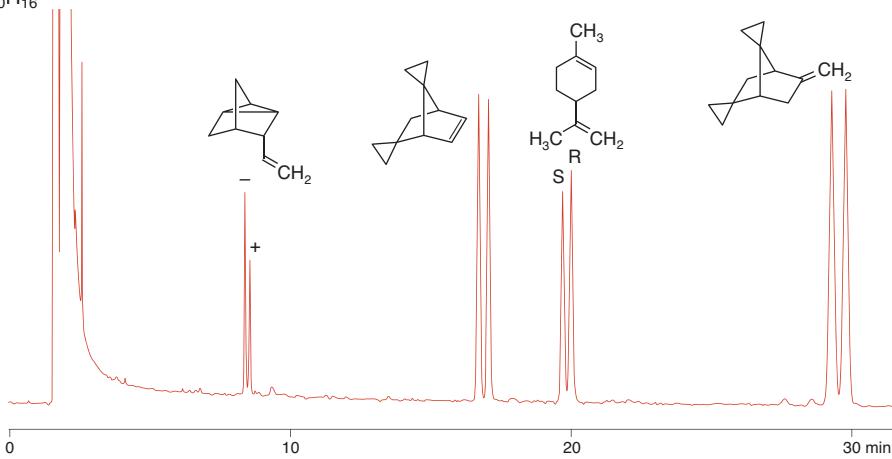


Courtesy of H. Casabianca, CNRS Service Central d'Analyse, Vernaison, France

### Enantiomer separation of cyclic olefins including limonene MN Appl. No. 201830

Column: LIPODEX® C, 50 m x 0.25 mm ID, REF 723364.50,  
 max. temperature 200/220 °C  
 Carrier gas: 1 bar H<sub>2</sub>  
 Temperature: 70 °C  
 Detector: FID

$C_{10}H_{16}$



W.A. König et al., HRC 12 (1989) 35 – 39

# Terpenes C<sub>10</sub>



## Enantiomer separation of limonene

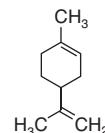
MN Appl. No. 211550

Columns: all 25 m x 0.25 mm ID:  
 a) FS-HYDRODEX β-6TBDM  
 (REF 723381.25),  
 b) FS-LIPODEX® E  
 (REF 723368.25),  
 c) FS-HYDRODEX β-PM  
 (REF 723370.25)

Injection: 1 µl, split, 250 °C  
 Carrier gas: 1.1 ml/min He  
 Detector: FID 250 °C

C<sub>10</sub>H<sub>16</sub>

	a	b	c
Temperature:	80 °C	55 °C	70 °C
Resolution:	6.48	<0.5	0.55



Courtesy of H. Casabianca, CNRS Service  
 Central d'Analyse, Vernaison, France

Limonene can also be separated on  
 HYDRODEX β-3P (see application 201850 at  
[www.mn-net.com](http://www.mn-net.com)).

## Enantiomer separation of α-pinene

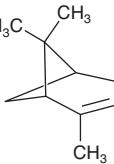
MN Appl. No. 211510

Columns: all 25 m x 0.25 mm ID:  
 a) FS-HYDRODEX β-6TBDM  
 (REF 723381.25),  
 b) FS-LIPODEX® E  
 (REF 723368.25),  
 c) FS-HYDRODEX β-PM  
 (REF 723370.25)

Injection: 1 µl, split, 250 °C  
 Carrier gas: 1.1 ml/min He  
 Detector: FID 250 °C

C<sub>10</sub>H<sub>16</sub>

	a	b	c
Temperature:	68 °C	40 °C	60 °C
Resolution:	1.79	1.66	3.33



Courtesy of H. Casabianca, CNRS Service  
 Central d'Analyse, Vernaison, France

## Enantiomer separation of β-pinene

MN Appl. No. 211520

Columns: all 25 m x 0.25 mm ID:  
 a) FS-HYDRODEX β-6TBDM  
 (REF 723381.25),  
 b) FS-LIPODEX® E  
 (REF 723368.25),  
 c) FS-HYDRODEX β-PM  
 (REF 723370.25)

Injection: 1 µl, split, 250 °C  
 Carrier gas: 1.1 ml/min He  
 Detector: FID 250 °C

C<sub>10</sub>H<sub>16</sub>

	a	b	c
Temperature:	75 °C	50 °C	70 °C
Resolution:	3.23	4.26	1.23



Courtesy of H. Casabianca, CNRS Service  
 Central d'Analyse, Vernaison, France

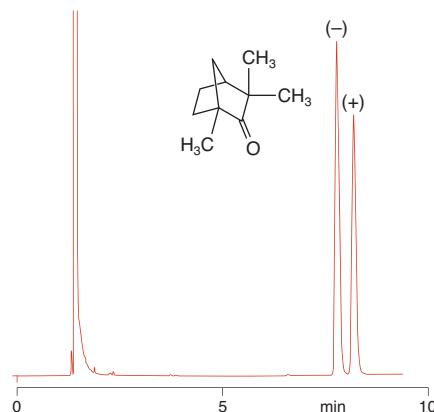
## Enantiomer separation of fenchone

MN Appl. No. 202090

Column: FS-LIPODEX® E,  
 25 m x 0.25 mm ID,  
 REF 723368.25,  
 max. temperature 200/220 °C

Injection: 0.5 µl, split 1:100  
 Carrier gas: 60 kPa H<sub>2</sub>  
 Temperature: 90 °C  
 Detector: FID 250 °C

C<sub>10</sub>H<sub>16</sub>O



## Chiral separations

### Enantiomer separation of camphor MN Appl. No. 211560

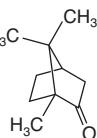
Columns: all 25 m x 0.25 mm ID:  
 a) FS-HYDRODEX  $\beta$ -6TBDM  
 (REF 723381.25),  
 b) FS-LIPODEX® E  
 (REF 723368.25),  
 c) FS-HYDRODEX  $\beta$ -PM  
 (REF 723370.25)

Injection: 1  $\mu$ l, split, 250 °C

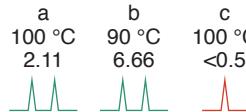
Carrier gas: 1.1 ml/min He

Detector: FID 250 °C

$C_{10}H_{16}O$



Temperature: a 100 °C, b 90 °C, c 100 °C  
 Resolution: 2.11, 6.66, <0.5



Courtesy of H. Casabianca, CNRS Service  
 Central d'Analyse, Vernaison, France

### Separation of essential oils MN Appl. No. 212980 / 212990 / 213000

Column: FS-HYDRODEX  $\gamma$ -TBDAc, 50 m x 0.25 mm ID, max. temp. 220/240 °C, REF 723387.50

Injector: 220 °C

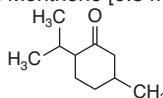
Carrier gas: 1.2 bar  $H_2$

Temperature: 125 °C

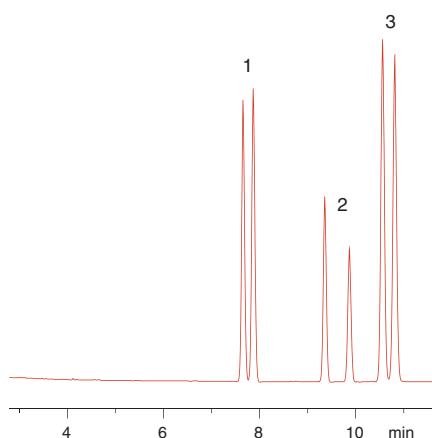
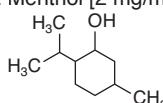
Detector: FID 220 °C

#### Peaks:

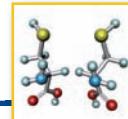
1. Fenchone [1.5 mg/ml] ( $C_{10}H_{16}O$ )
2. Menthone [0.5 mg/ml] ( $C_{10}H_{18}O$ )



3. Menthol [2 mg/ml] ( $C_{10}H_{20}O$ )

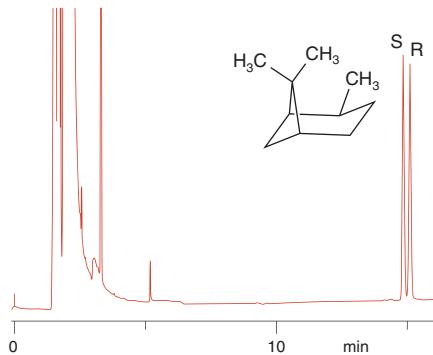


# Terpenes C<sub>10</sub>



## Enantiomer separation of *cis*-pinane MN Appl. No. 201810

Column: LIPODEX® C,  
50 m x 0.25 mm ID,  
REF 723364.50,  
max. temperature 200/220 °C  
  
Carrier gas: H<sub>2</sub>  
Temperature: 70 °C  
Detector: FID  
  
C<sub>10</sub>H<sub>18</sub>



Courtesy of Prof. W.A. König, Hamburg,  
Germany

## Enantiomer separation of terpinen-4-ol MN Appl. No. 211630

Columns: all 25 m x 0.25 mm ID:  
a) FS-HYDRODEX β-6TBDM  
(REF 723381.25),  
b) FS-LIPODEX® E  
(REF 723368.25),  
c) FS-HYDRODEX β-PM  
(REF 723370.25)  
  
Injection: 1 µl, split, 250 °C  
Carrier gas: 1.1 ml/min He  
Detector: FID 250 °C  
  
C<sub>10</sub>H<sub>18</sub>O  
Temperature: a 110 °C, b 75 °C, c 70 °C  
Resolution: 2.1, <0.5, 0.65

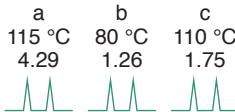
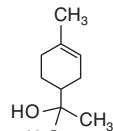
Courtesy of H. Casabianca, CNRS Service  
Central d'Analyse, Vernaison, France

## Enantiomer separation of α-terpineol MN Appl. No. 211640

Columns: all 25 m x 0.25 mm ID:  
a) FS-HYDRODEX β-6TBDM  
(REF 723381.25),  
b) FS-LIPODEX® E  
(REF 723368.25),  
c) FS-HYDRODEX β-PM  
(REF 723370.25)

Injection: 1 µl, split, 250 °C  
Carrier gas: 1.1 ml/min He  
Detector: FID 250 °C  
C<sub>10</sub>H<sub>18</sub>O

Temperature: a 115 °C, b 80 °C, c 110 °C  
Resolution: 4.29, 1.26, 1.75

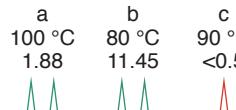
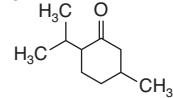


Courtesy of H. Casabianca, CNRS Service  
Central d'Analyse, Vernaison, France

## Enantiomer separation of menthone MN Appl. No. 211600

Columns: all 25 m x 0.25 mm ID:  
a) FS-HYDRODEX β-6TBDM  
(REF 723381.25),  
b) FS-LIPODEX® E  
(REF 723368.25),  
c) FS-HYDRODEX β-PM  
(REF 723370.25)  
  
Injection: 1 µl, split, 250 °C  
Carrier gas: 1.1 ml/min He  
Detector: FID 250 °C  
C<sub>10</sub>H<sub>18</sub>O

Temperature: a 100 °C, b 80 °C, c 90 °C  
Resolution: 1.88, 11.45, <0.5



Courtesy of H. Casabianca, CNRS Service  
Central d'Analyse, Vernaison, France

## Chiral separations

### Enantiomer separation of isomenthone MN Appl. No. 211610

Columns: all 25 m x 0.25 mm ID:  
 a) FS-HYDRODEX  $\beta$ -6TBDM  
 (REF 723381.25),  
 b) FS-LIPODEX® E  
 (REF 723368.25),  
 c) FS-HYDRODEX  $\beta$ -PM  
 (REF 723370.25)

Injection: 1  $\mu$ l, split, 250 °C  
 Carrier gas: 1.1 ml/min He  
 Detector: FID 250 °C

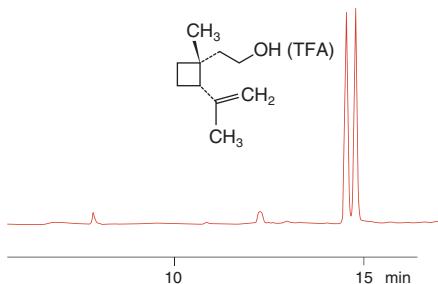
$C_{10}H_{18}O$

	a 100 °C	b 90 °C	c 90 °C
Resolution:	10.01	10.11	2.04

Courtesy of H. Casabianca, CNRS Service Central d'Analyse, Vernaison, France

### Enantiomer separation of grandisol (TFA) MN Appl. No. 202811

Column: LIPODEX® C,  
 50 m x 0.25 mm ID,  
 REF 723364.50,  
 max. temperature 200/220 °C  
 Carrier gas: 1 bar  $H_2$   
 Temperature: 90 °C  
 Detector: FID  
 $C_{10}H_{18}O$



W.A. König et al., HRC 12 (1989) 35 – 39

### Enantiomer separation of borneol MN Appl. No. 211570

Columns: all 25 m x 0.25 mm ID:  
 a) FS-HYDRODEX  $\beta$ -6TBDM  
 (REF 723381.25),  
 b) FS-LIPODEX® E  
 (REF 723368.25),  
 c) FS-HYDRODEX  $\beta$ -PM  
 (REF 723370.25)

Injection: 1  $\mu$ l, split, 250 °C  
 Carrier gas: 1.1 ml/min He  
 Detector: FID 250 °C

$C_{10}H_{18}O$

	a 115 °C	b 80 °C	c 110 °C
Resolution:	4.99	1.77	2.95

Courtesy of H. Casabianca, CNRS Service Central d'Analyse, Vernaison, France

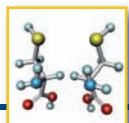
### Enantiomer separation of isoborneol MN Appl. No. 211580

Columns: all 25 m x 0.25 mm ID:  
 a) FS-HYDRODEX  $\beta$ -6TBDM  
 (REF 723381.25),  
 b) FS-LIPODEX® E  
 (REF 723368.25),  
 c) FS-HYDRODEX  $\beta$ -PM  
 (REF 723370.25)

Injection: 1  $\mu$ l, split, 250 °C  
 Carrier gas: 1.1 ml/min He  
 Detector: FID 250 °C

$C_{10}H_{18}O$

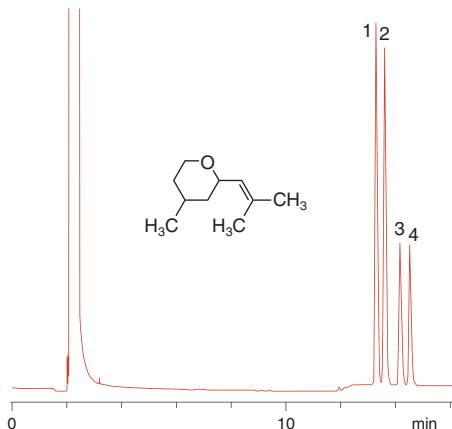
	a 115 °C	b 90 °C	c 110 °C
Resolution:	3.07	1.36	1.25



## Terpenes C<sub>10</sub> – C<sub>12</sub>

### Enantiomer separation of rose oxide MN Appl. No. 212430

Column: FS-HYDRODEX®  $\beta$ -TBDAC,  
50 m x 0.25 mm ID,  
REF 723384.50,  
max. temperature 220/240 °C  
Sample: 0.1 % in hexane  
Carrier gas: 1.4 bar H<sub>2</sub>  
Temperature: 90 °C  
Detector: FID  
**Peaks:** (C<sub>10</sub>H<sub>18</sub>O)  
1. (+)-*cis*-Rose oxide  
2. (-)-*cis*-Rose oxide  
3. (-)-*trans*-Rose oxide  
4. (+)-*trans*-Rose oxide



### MN Appl. No. 211620

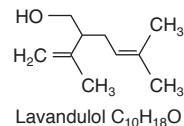
Columns: all 25 m x 0.25 mm ID:  
a) FS-HYDRODEX  $\beta$ -6TBDM  
(REF 723381.25),  
b) FS-LIPODEX® E  
(REF 723368.25)  
Carrier gas: 1.1 ml/min He  
Detector: FID 250 °C  
Temperature: 90 °C      60 °C  
Resolution: 1.41      <0.5  
a      b

Courtesy of H. Casabianca, CNRS Service  
Central d'Analyse, Vernaison, France

### Enantiomer separation of lavandulol MN Appl. No. 211590

Columns: all 25 m x 0.25 mm ID:  
a) FS-HYDRODEX  $\beta$ -6TBDM  
(REF 723381.25),  
b) FS-LIPODEX® E  
(REF 723368.25),  
c) FS-HYDRODEX  $\beta$ -PM  
(REF 723370.25)  
Injection: 1  $\mu$ l, split, 250 °C  
Carrier gas: 1.1 ml/min He  
Detector: FID 250 °C  
Temperature: 110 °C      80 °C      100 °C  
Resolution: 6      0.95      2.82  
a      b      c

Courtesy of H. Casabianca, CNRS Service  
Central d'Analyse, Vernaison, France



### Enantiomer separation of lavandulyl acetate MN Appl. No. 211740

Columns: all 25 m x 0.25 mm ID:  
a) FS-HYDRODEX  $\beta$ -6TBDM  
(REF 723381.25),  
b) FS-LIPODEX® E  
(REF 723368.25),  
c) FS-HYDRODEX  $\beta$ -PM  
(REF 723370.25)  
Injection: 1  $\mu$ l, split, 250 °C  
Carrier gas: 1.1 ml/min He  
Detector: FID 250 °C  
Temperature: 105 °C      85 °C      100 °C  
Resolution: 1.9      <0.5      0.54  
a      b      c

Courtesy of H. Casabianca, CNRS Service  
Central d'Analyse, Vernaison, France

## Enantiomer separation of citronellal and citronellool

MN Appl. No. 212440

Column: FS-HYDRODEX®  $\beta$ -TBDAC,  
50 m x 0.25 mm ID,  
REF 723384.50,  
max. temperature 220/240 °C

Sample: 0.1 % in  $\text{CH}_2\text{Cl}_2$

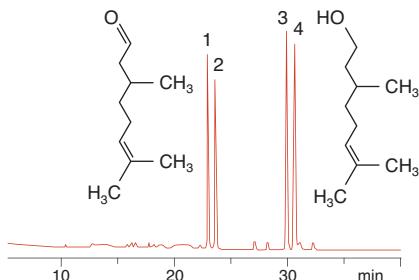
Carrier gas:  $\text{H}_2$ , 1.5 bar

Temperature: 100 °C

Detector: FID

**Peaks:**

1. (R)/(S)-Citronellal ( $\text{C}_{10}\text{H}_{18}\text{O}$ )
2. (S)/(R)-Citronellal
3. (S)-Citronellool ( $\text{C}_{10}\text{H}_{20}\text{O}$ )
4. (R)-Citronellool



Also see application 211680 at  
[www.mn-net.com](http://www.mn-net.com)

## Enantiomer separation of citronellool (TFA)

MN Appl. No. 211690

Columns: all 25 m x 0.25 mm ID:  
a) FS-HYDRODEX  $\beta$ -6TBDM  
(REF 723381.25),  
b) FS-LIPODEX® E  
(REF 723368.25)

Carrier gas: 1.1 ml/min He

Detector: FID 250 °C

Temperature: a 95 °C      b 85 °C  
Resolution: 0.67      1.75

Courtesy of H. Casabianca, CNRS Service  
Central d'Analyse, Vernaison, France

## Enantiomer separation of linalool

MN Appl. No. 201920

Column: FS-HYDRODEX  $\beta$ -3P,  
25 m x 0.25 mm ID,  
REF 723358.25,  
max. temperature 230/250 °C

Injection: 0.1  $\mu\text{l}$  (1 % in  $\text{CH}_2\text{Cl}_2$ )  
split 130 ml/min

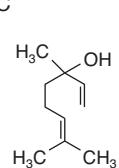
Carrier gas: 60 kPa  $\text{H}_2$  (1.9 ml/min)

Temperature: 120 °C

Detector: FID 250 °C

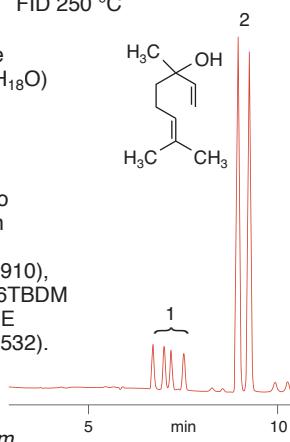
**Peaks:**

1. Linalool oxide
2. Linalool ( $\text{C}_{10}\text{H}_{18}\text{O}$ )



Linalool can also be separated on LIPODEX® C (application 201910), HYDRODEX  $\beta$ -6TBDM and LIPODEX® E (application 210532).

See our application database at  
[www.mn-net.com](http://www.mn-net.com)



## Enantiomer separation of linalool oxide

MN Appl. No. 210533

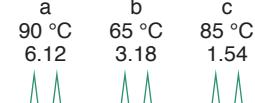
Columns: all 25 m x 0.25 mm ID:  
a) FS-HYDRODEX  $\beta$ -6TBDM  
(REF 723381.25),  
b) FS-LIPODEX® E  
(REF 723368.25),  
c) FS-HYDRODEX  $\beta$ -PM  
(REF 723370.25)

Injection: 1  $\mu\text{l}$ , split, 250 °C

Carrier gas: 1.1 ml/min He

Detector: FID 250 °C

$\text{C}_{10}\text{H}_{18}\text{O}_2$   
Temperature: a 90 °C      b 65 °C      c 85 °C  
Resolution: 6.12      3.18      1.54



Courtesy of H. Casabianca, CNRS Service  
Central d'Analyse, Vernaison, France

## Compounds C<sub>10</sub> – C<sub>12</sub>



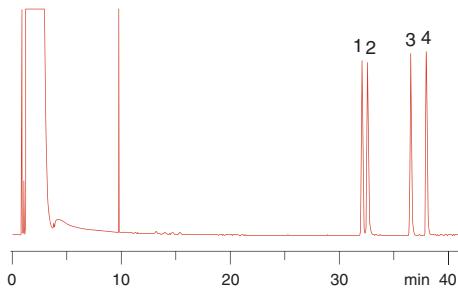
### Enantiomer separation of linalool and linalyl acetate

*MN Appl. No. 212410*

Column: FS-HYDRODEX®  $\beta$ -TBDAC,  
25 m x 0.25 mm ID,  
REF 723384.25,  
max. temperature 220/240 °C  
Sample: 0.1 % in CH<sub>2</sub>Cl<sub>2</sub>  
Carrier gas: 0.8 bar H<sub>2</sub>  
Temperature: 75 °C (20 min)  $\xrightarrow{1\text{ °C/min}}$  110 °C  
(5 min)  
Detector: FID

**Peaks:**

1. (*R*)-Linalyl acetate (C<sub>12</sub>H<sub>20</sub>O<sub>2</sub>)
2. (*S*)-Linalyl acetate
3. (*R*)-Linalool (C<sub>10</sub>H<sub>18</sub>O)
4. (*S*)-Linalool



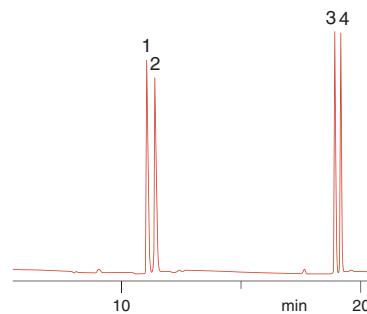
### Enantiomer separation of $\delta$ -decalactone and $\delta$ -dodecalactone

*MN Appl. No. 212420*

Column: FS-HYDRODEX®  $\beta$ -TBDAC,  
25 m x 0.25 mm ID, REF  
723384.25,  
max. temperature 220/240 °C  
Sample: 0.1 % in hexane  
Carrier gas: H<sub>2</sub>, 0.8 bar  
Temperature: 160 °C (12 min)  $\xrightarrow{4\text{ °C/min}}$   
200 °C (10 min)  
Detector: FID

**Peaks:**

1. (*S*)- $\delta$ -Decalactone (C<sub>10</sub>H<sub>18</sub>O<sub>2</sub>)
2. (*R*)- $\delta$ -Decalactone
3. (*S*)- $\delta$ -Dodecalactone (C<sub>12</sub>H<sub>18</sub>O<sub>2</sub>)
4. (*R*)- $\delta$ -Dodecalactone

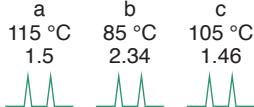


## Chiral separations

### Enantiomer separation of menthol MN Appl. No. 211700

Columns: all 25 m x 0.25 mm ID:  
 a) FS-HYDRODEX  $\beta$ -6TBDM (REF 723381.25),  
 b) FS-LIPODEX® E (REF 723368.25),  
 c) FS-HYDRODEX  $\beta$ -PM (REF 723370.25)  
 Injection: 1  $\mu$ l, split, 250 °C  
 Carrier gas: 1.1 ml/min He  
 Detector: FID 250 °C  
 $C_{10}H_{20}O$

	a 115 °C	b 85 °C	c 105 °C
Resolution:	1.5	2.34	1.46



Courtesy of H. Casabianca, CNRS Service Central d'Analyse, Vernaison, France

Menthol can also be separated on LIPODEX® A (see application 210090 at [www.mn-net.com](http://www.mn-net.com)).

### Enantiomer separation of menthol (TFA) MN Appl. No. 211710

Columns: FS-HYDRODEX  $\beta$ -6TBDM, 25 m x 0.25 mm ID, REF 723381.25  
 Injection: 1  $\mu$ l, split, 250 °C  
 Carrier gas: 1.1 ml/min He  
 Temperature: 85 °C  
 Detector: FID 250 °C  
 Resolution: 2.52



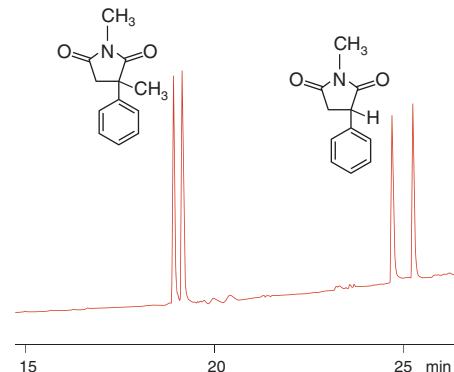
Courtesy of H. Casabianca, CNRS Service Central d'Analyse, Vernaison, France

### Enantiomer separation of antiepileptic drugs mesuximide and phensuximide MN Appl. No. 202381

Column: LIPODEX® B, 50 m x 0.25 mm ID, REF 723362.50, max. temperature 200/220 °C  
 Carrier gas: 1 bar  $H_2$ , 3 °C/min  
 Temperature: 135 °C  $\xrightarrow{3\text{ °C/min}}$  200 °C  
 Detector: FID

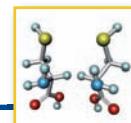
#### Peaks:

1. Mesuximide ( $C_{12}H_{13}NO_2$ )
2. Phensuximide ( $C_{11}H_{11}NO_2$ )



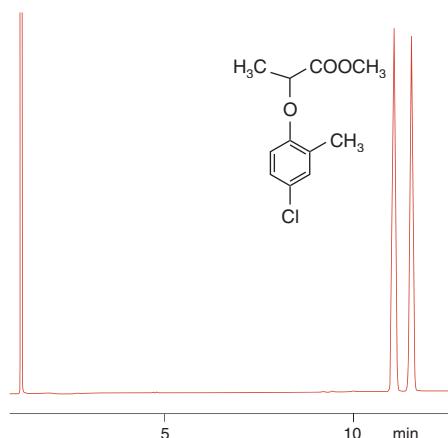
W.A. König et al., HRC 11 (1988) 621 – 625

## Compounds C<sub>10</sub> – C<sub>12</sub>



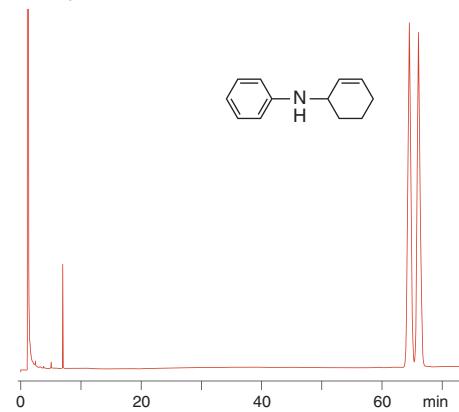
### Enantiomer separation of mecoprop methyl ester MN Appl. No. 202562

Column: FS-HYDRODEX β-3P,  
25 m x 0.25 mm ID,  
REF 723358.25,  
max. temperature 230/250 °C  
Injection: 0.1 µl (1% in CH<sub>2</sub>Cl<sub>2</sub>)  
split 130 ml/min  
Carrier gas: 60 kPa H<sub>2</sub> (1.9 ml/min)  
Temperature: 160 °C  
Detector: FID 250 °C  
C<sub>11</sub>H<sub>13</sub>ClO<sub>3</sub>



### Enantiomer separation of N-phenyl-cyclohex-2-enamine MN Appl. No. 212250

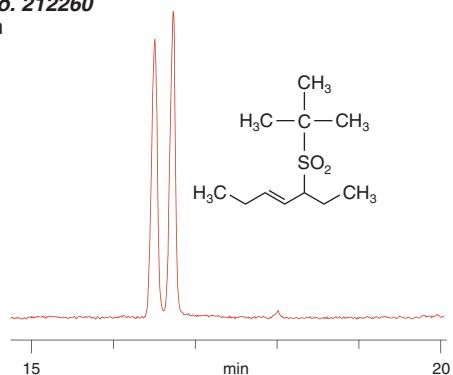
Column: FS-LIPODEX® E,  
25 m x 0.25 mm ID,  
REF 723368.25,  
max. temperature 200/220 °C  
Sample: 10 µl in 3 ml CH<sub>2</sub>Cl<sub>2</sub>  
Carrier gas: 0.6 bar H<sub>2</sub>  
Temperature: 110 °C  
Detector: FID  
C<sub>12</sub>H<sub>15</sub>N



Courtesy of Mrs. Eschmann, Institut für Brennstoffchemie, RWTH Aachen, Germany

### Enantiomer separation of 5-(2-methyl-propan-2-sulphonyl)-hept-3-ene MN Appl. No. 212260

Column: FS-LIPODEX® G, 25 m x 0.25 mm  
ID, REF 723379.25,  
max. temperature 220/240 °C  
Carrier gas: 100 kPa H<sub>2</sub>  
Temperature: 60 °C (15 min)  $\xrightarrow{10\text{ °C/min}}$  100 °C  
(5 min)  
Detector: FID  
C<sub>11</sub>H<sub>22</sub>O<sub>2</sub>S



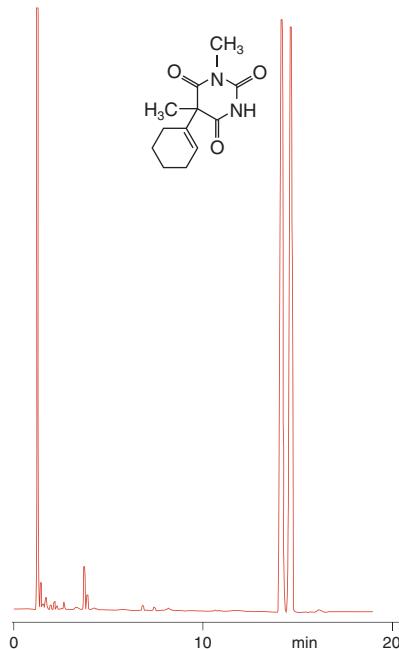
Courtesy of Mrs. Vermeeren, AK Prof. Gais,  
Inst. für Org. Chemie, RWTH Aachen, Germany

## Chiral separations

### Enantiomer separation of hexobarbital (TFA)

MN Appl. No. 202401

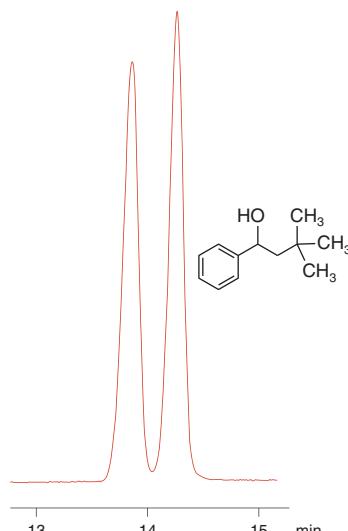
Column: FS-HYDRODEX  $\beta$ -3P,  
25 m x 0.25 mm ID,  
REF 723358.25,  
max. temperature 230/250 °C  
Injection: 0.1  $\mu$ l, split 130 ml/min  
Carrier gas: 60 kPa H<sub>2</sub> (1.9 ml/min)  
Temperature: 210 °C  
Detector: FID 250 °C  
C<sub>12</sub>H<sub>16</sub>N<sub>2</sub>O<sub>3</sub>



### Enantiomer separation of 3,3-dimethyl-1-phenyl-butan-1-ol

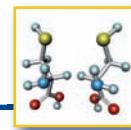
MN Appl. No. 212350

Column: FS-LIPODEX® G,  
25 m x 0.25 mm ID,  
REF 723379.25,  
max. temperature 200/220 °C  
Carrier gas: 100 kPa H<sub>2</sub>  
Temperature: 100 °C (5 min)  $\xrightarrow{10\text{ °C/min}}$  120 °C  
(5 min)  $\xrightarrow{10\text{ °C/min}}$  140 °C (5 min)  
 $\xrightarrow{10\text{ °C/min}}$  170 °C (45 min)  
Detector: FID  
C<sub>12</sub>H<sub>18</sub>O



Courtesy of Mrs. Vermeeren, AK Prof. Gais,  
Inst. für Org. Chemie, RWTH Aachen, Germany

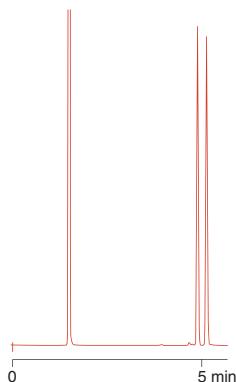
# Compounds C<sub>12</sub> and greater



## Enantiomer separation of menthyl acetate

MN Appl. No. 202010

Column: FS-HYDRODEX  $\beta$ -3P,  
25 m x 0.25 mm ID,  
REF 723358.25,  
max. temperature 230/250 °C  
Injection: 0.1  $\mu$ l (1% in CH<sub>2</sub>Cl<sub>2</sub>)  
split 130 ml/min  
Carrier gas: 60 kPa H<sub>2</sub> (1.9 ml/min)  
Temperature: 150 °C  
Detector: FID 250 °C  
C<sub>12</sub>H<sub>22</sub>O<sub>2</sub>



Menthyl acetate can also be separated on HYDRODEX  $\beta$ -PM (see application 202000 at [www.mn-net.com](http://www.mn-net.com)).

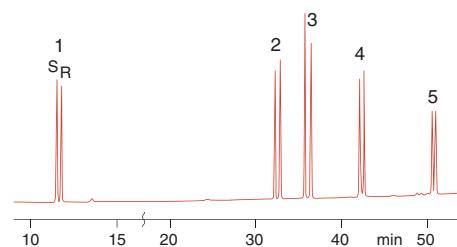
## Separation of isomeric antiinflammatory drugs

MN Appl. No. 210150

Column: FS-HYDRODEX  $\beta$ -6TBDM,  
25 m x 0.25 mm ID,  
max. temperature 250 °C,  
REF 723381.25  
Carrier gas: H<sub>2</sub>  
Temperature: 135 °C  $\xrightarrow{1\text{ °C/min}}$  200 °C  
Detector: FID

### Peaks:

- |                 |                                                 |  |
|-----------------|-------------------------------------------------|--|
| 1. Ibuprofen    | C <sub>13</sub> H <sub>18</sub> O <sub>2</sub>  |  |
| 2. Flurbiprofen | C <sub>15</sub> H <sub>13</sub> FO <sub>2</sub> |  |
| 3. Fenoprofen   | C <sub>15</sub> H <sub>14</sub> O <sub>3</sub>  |  |
| 4. Naproxen     | C <sub>14</sub> H <sub>14</sub> O <sub>3</sub>  |  |
| 5. Ketoprofen   | C <sub>16</sub> H <sub>14</sub> O <sub>3</sub>  |  |

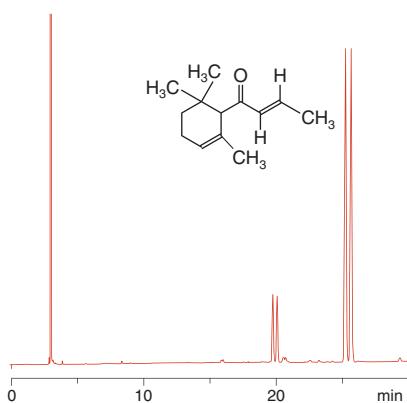


Courtesy of Prof. W.A. König, Hamburg,  
Germany

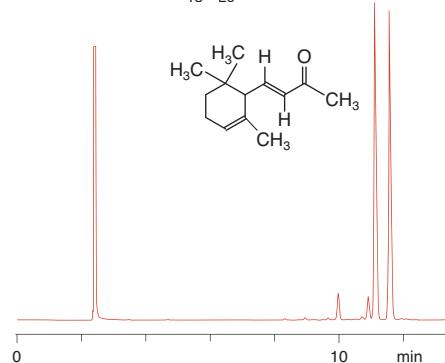
## Chiral separations

**Enantiomer separation of  $\alpha$ -damascone**  
*MN Appl. No. 202030*

Column: FS-HYDRODEX  $\beta$ -PM,  
 50 m x 0.25 mm ID,  
 REF 723370.50,  
 max. temperature 230/250 °C  
 Injection: 0.1  $\mu$ l (1% in  $\text{CH}_2\text{Cl}_2$ )  
 split 150 ml/min  
 Carrier gas: 120 kPa  $\text{H}_2$  (1.7 ml/min)  
 Temperature: 130 °C  
 Detector: FID 250 °C  
 $\text{C}_{13}\text{H}_{20}\text{O}$


**Enantiomer separation of  $\alpha$ -ionone**  
*MN Appl. No. 213010*

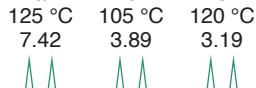
Column: FS-HYDRODEX  $\gamma$ -TBDAC,  
 50 m x 0.25 mm ID,  
 REF 723370.50,  
 max. temperature 230/250 °C  
 Injection: 0.5  $\mu$ l, split 5.0 ml/min  
 Carrier gas: 1.2 bar  $\text{H}_2$   
 Temperature: 155 °C  
 Detector: FID  
 $\text{C}_{13}\text{H}_{20}\text{O}$


*MN Appl. No. 211770*

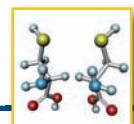
Columns: all 25 m x 0.25 mm ID:  
 a) FS-HYDRODEX  $\beta$ -6TBDM  
 (REF 723381.25),  
 b) FS-LIPODEX® E  
 (REF 723368.25),  
 c) FS-HYDRODEX  $\beta$ -PM  
 (REF 723370.25)

Injection: 1  $\mu$ l, split, 250 °C  
 Carrier gas: 1.1 ml/min He  
 Detector: FID 250 °C

Temperature: 125 °C      105 °C      120 °C  
 Resolution: 7.42      3.89      3.19



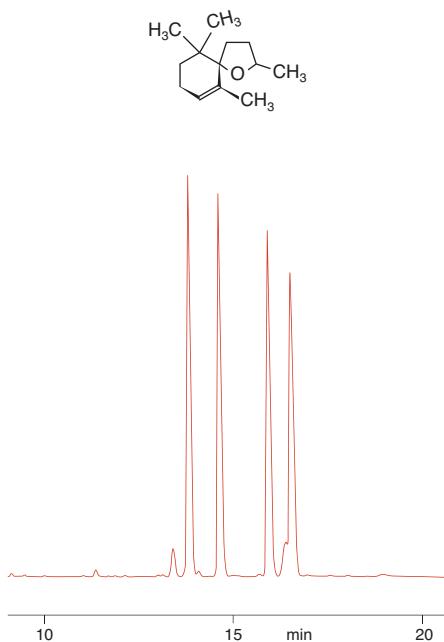
Courtesy of H. Casabianca, CNRS Service  
 Central d'Analyse, Vernaison, France



## Compounds C<sub>13</sub> – C<sub>14</sub>

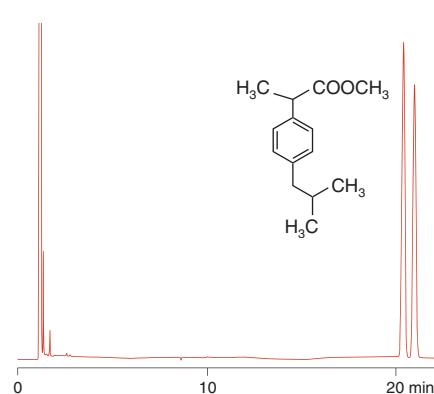
### Enantiomer separation of theaspiran MN Appl. No. 202210

Column: FS-HYDRODEX  $\beta$ -PM,  
50 m x 0.25 mm ID,  
REF 723370.50,  
max. temperature 230/250 °C  
Injection: 0.5  $\mu$ l (1 % in CH<sub>2</sub>Cl<sub>2</sub>)  
split 1:100  
Carrier gas: 200 kPa H<sub>2</sub>  
Temperature: 120 °C  
Detector: FID 250 °C  
C<sub>13</sub>H<sub>22</sub>O



### Enantiomer separation of ibuprofen methyl ester MN Appl. No. 202582

Column: FS-HYDRODEX  $\beta$ -3P,  
25 m x 0.25 mm ID,  
REF 723358.25,  
max. temperature 230/250 °C  
Injection: 0.5  $\mu$ l (1 % in CH<sub>2</sub>Cl<sub>2</sub>)  
split 130 ml/min  
Carrier gas: 60 kPa H<sub>2</sub> (1.9 ml/min)  
Temperature: 140 °C  
Detector: FID 250 °C  
C<sub>14</sub>H<sub>20</sub>O<sub>2</sub>



## Chiral separations

### Separation of isomeric sesquiterpenes MN Appl. No. 250170

Column: FS-HYDRODEX  $\beta$ -6TBDM,  
15 m x 0.25 mm ID,  
max. temperature 230/250 °C

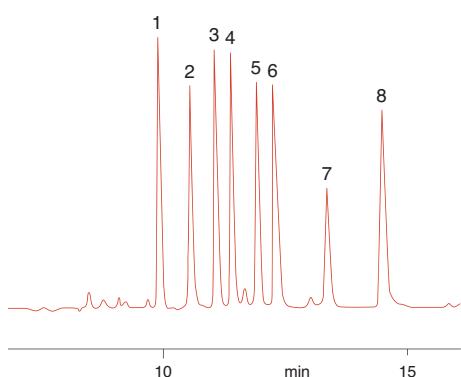
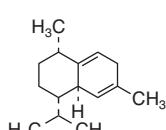
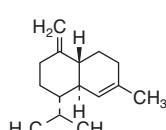
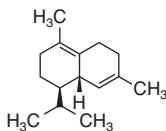
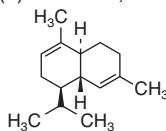
Carrier gas: H<sub>2</sub>

Temperature: 100 °C  $\xrightarrow{2\text{ °C/min}}$  200 °C

Detector: FID

**Peaks:** (C<sub>15</sub>H<sub>24</sub>)

1. (–)- $\gamma$ -Cadinene
2. (–)- $\delta$ -Cadinene
3. (–)- $\alpha$ -Cadinene
4. (+)- $\gamma$ -Cadinene
5. (+)- $\alpha$ -Cadinene
6. (+)- $\delta$ -Cadinene
7. (+)-Cadina-1,4-diene
8. (–)-Cadina-1,4-diene



Courtesy of Prof. W.A. König, Hamburg,  
Germany

### Enantiomer separation of fluoxetine and norfluoxetine MN Appl. No. 212220

Column: FS-HYDRODEX  $\beta$ -6TBDM,  
25 m x 0.25 mm ID,  
REF 723358.25,  
max. temperature 230/250 °C

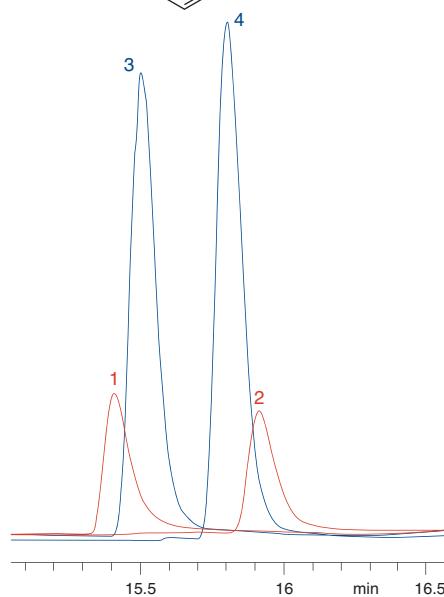
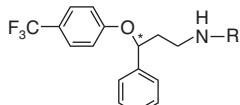
Temperature: 170 °C  $\xrightarrow{1\text{ °C/min}}$  200 °C

Carrier gas: 1.1 ml/min H<sub>2</sub>

Detector: PND 300 °C

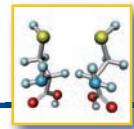
**Peaks:**

1. (S)-Norfluoxetine (R= H)
2. (R)-Norfluoxetine
3. (S)-Fluoxetine (R = CH<sub>3</sub>)
4. (R)-Fluoxetine



S. Ulrich, J. Chromatography B, 783 (2003)  
481 – 490

# Compounds C<sub>15</sub> and greater

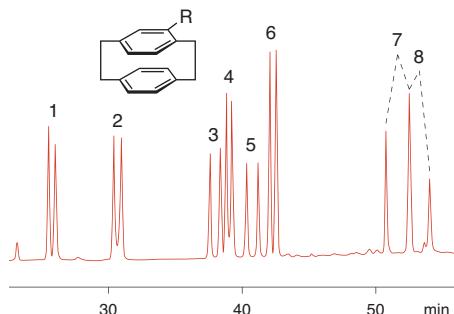


## Separation of isomeric paracyclophanes MN Appl. No. 250190

Column: FS-HYDRODEX β-6TBDM, 25 m x 0.25 mm ID, REF 723381.25  
max. temperature 230/250 °C  
Carrier gas: H<sub>2</sub>  
Temperature: 135 °C  $\xrightarrow{1\text{ °C/min}}$  200 °C  
Detector: FID

**Peaks:**

1. 4-Methyl-[2.2]-paracyclophane (C<sub>17</sub>H<sub>18</sub>, R = -CH<sub>3</sub>)
2. 4-Ethynyl-[2.2]-paracyclophane (C<sub>18</sub>H<sub>16</sub>, R = -C≡CH)
3. 4-Allyloxy-[2.2]-paracyclophane (C<sub>19</sub>H<sub>20</sub>O, R = -O-CH<sub>2</sub>-CH=CH<sub>2</sub>)
4. 4-Acetyl-[2.2]-paracyclophane (C<sub>18</sub>H<sub>18</sub>O, R = -CO-CH<sub>3</sub>)
5. 4-Formyl-[2.2]-paracyclophane (C<sub>17</sub>H<sub>16</sub>O, R = -CHO)
6. 4-Cyano-[2.2]-paracyclophane (C<sub>17</sub>H<sub>15</sub>N, R = -CN)
7. 4-Hydroxymethyl-[2.2]-paracyclophane (C<sub>17</sub>H<sub>18</sub>O, R = -CH<sub>2</sub>OH)
8. 4-Hydroxy-[2.2]-paracyclophane (C<sub>16</sub>H<sub>16</sub>O, R = -OH)



Courtesy of Prof. W.A. König, Hamburg, Germany

(e) = chiral (enantiomer) separation

# Chromatogram index

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**C**

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(e) = chiral (enantiomer) separation		1,7-Dioxaspiro[5,5]undecane (e)	255



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(e) = chiral (enantiomer) separation



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(e) = chiral (enantiomer) separation			
			



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(e) = chiral (enantiomer) separation		(+)- <i>trans</i> -Sabinene hydrate (e)	259



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Alachlor	100	2-Bromopentane	219	Cholesterol	191
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Dimethyl malate	225	Fluchloralin	96	Isobenzan	77
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2,3-Dinorthromboxane	192	Fluorenene	64	Isodrin	77
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Estrone	191	Glycidyl butyrate	234	Linalool	266
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Methomyl	107	Nuarimol	72	Prometryn	102
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2-Methylbutyric acid methyl ester	227	$\gamma$ -Octalactone	215	Propazine	102
2-Methylbutyric acid	218	1-Octen-3-ol	246	Propham	100
$\alpha$ -Methylbutyrolactone	214	Octyl-3-nitrate	229	Propiconazole	97
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4-Methyl-3-heptanone	246	Oxfendazole	178	Pyrene	64
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3-Methyl-1-hexene	210	Pantolactone	224	Quinalphos	91
2-Methyl-3-hydroxy-valeraldehyde	207	Papaverine	181	Quinine	182
$\beta$ -Methylionone	154	Paracyclophanes	275	Quinomethionat	116
Methyl lactate	208	Parathion	94	Quintozene	52
5-(2-Methyl-propan-2-sulphonyl)-hept-3-ene	269	PCB	68	Ractopamine	188
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$\alpha$ -Methylquinovoside	236	Penconazole	97	Sabinene	260
$\alpha$ -Methylrhhamnoside	236	Pendimethalin	96	Safrole	154
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Terbutryn	102	Thiordiazine	182	3,3,5-Trimethylcyclohexanol	253
$\alpha$ -Terpinene	156	Thiothixene	182	2,4,6-Trinitrotoluene	56
$\alpha$ -Terpineol	263	Thymol	156	$\delta$ -Undecalactone	244
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Tetrahydrocannabinol	186	<i>o</i> -Toluidine	144	$\gamma$ -Valerolactone	215
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Tetrahydroisobenzofuranone	215	Toxaphenes	88	Vamidothion	91
Tetralin	121	2,4,5-TP	109	Vanillin	154
Tetramethrin	99	Tranylcypromine	250	Vernolate	86
Tetrasul	72	Triadimefon	97	Vinclozolin	112
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Thioacetic acid cyclopent-2-enyl ester	230	Trichlorfon	91	Whisky lactone	254
		Trichloronat	94		

### Product use restriction

MACHEREY-NAGEL chromatography products are intended, developed, designed and sold FOR RESEARCH AND DEVELOPMENT PURPOSES AND ANALYTICAL QUALITY CONTROL / ROUTINE MEASUREMENTS ONLY, except, however, any other function of the product being EXPRESSLY set forth in original MACHEREY-NAGEL product leaflets.

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201340	156	202350	243	202942	257	210700	77
201350	153	202361	235	202951	217	210710	86
201360	154	202362	209	202961	207	210720	99
201370	157	202371	245	202962	242	210730	147
201390	122	202372	228	202971	227	210740	98
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201420	128	202401	270	203001	230	210820	145
201430	130	202412	237	203011	222	210830	110
201460	130	202421	204	203021	219	210840	110
201480	159	202431	204	203031	228	210850	110
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201520	139	202471	221	210020	184	210950	129
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201570	163	202521	240	210060	167	211180	69

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211600	263	212570	41	250300	192
211610	264	212580	190	250350	79
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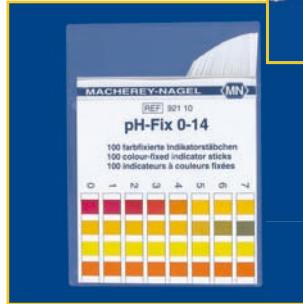
## HPLC • SPE • TLC



## Filtration



## Rapid Tests



## Bioanalysis



## Water Analysis