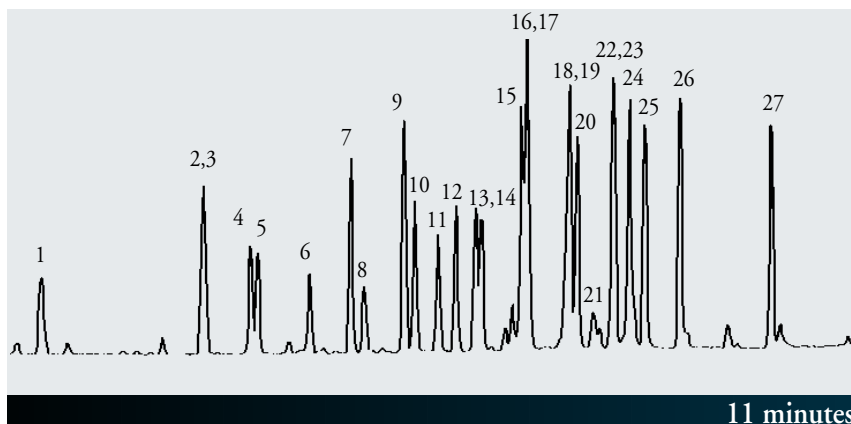


Rapid-MS of Chlorinated Pesticides & PCB's

Fast analysis of traces chlorinated pesticides & PCB's using Rapid-MS and Saturn 2000 ion trap detection



- **Faster analysis:** A factor 3-5 for temperature programmed up to a factor 10 for isothermal runs.
- **Higher sensitivity:** Typically signal-to-noise ratio will increase by at least a factor 3.
- **Compatible** with existing injection techniques and ion trap detection technology.
- **Positive inlet pressure** with short columns.
- **Less contamination** due to low elution temperatures.

- **Easy installation:** Standard fittings and ferrules can be used, no special skills required.
- **High loadability:** Filmthickness from 0.1 –1 microns.
- **More forgiving:** 0.53 mm ID capillaries can take more stress than smaller bore columns.
- **Easy setting of inlet flows/ pressures:** Optimum flowrate for ion trap is software controlled by Star Workstation (one button set-up).

Traces of chlorinated pesticides are difficult to determine. Usually these components are present at low levels, which requires optimum system performance to generate good data. Often they are analyzed together with a range of PCB's. Conventional (30-60 m) columns result in long analysis time and reduced response due to peak broadening. To solve this problem, the retention time in the separation column must be as short as possible. By using the new Rapid-MS with the Saturn 2000 ion trap, a significant reduction in retention time is obtained, which typically is a factor 3, but can be as high as a factor 10. Besides this, by using fast analysis the peaks will be sharper and higher, which allows lower detection limits. In this datasheet the results are presented for the analysis of chlorinated pesticides and PCB's using the Rapid-MS/Saturn 2000 combination. Analysis times, sensitivity, spectral integrity, linearity, repeatability and quantitation are shown. Comparisons are made with a conventional (30 m x 0.25 mm) capillary. The conclusion is that the Rapid-MS/Saturn combination offers a unique combination of advantages to improve the existing MS methods.



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Faster analysis

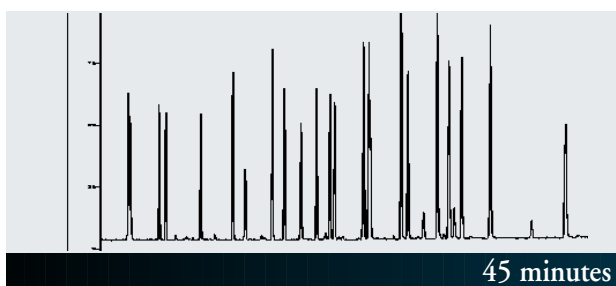
The analysis using Rapid-MS technique can be done in very short time because the optimum carrier gas velocity is about 100 cm per second for helium. This allows components to elute at lower temperatures, which can be beneficial for sensitive components. Below is a comparison of a conventional and the Rapid-MS method. The conventional analysis takes 45 minutes, while the Rapid-MS is completed in less than 11 minutes. Despite of the faster analysis, the peak shape for the chlorinated pesticides as well as the PCB's is symmetrical due to the high inertness of the Rapid-MS system.

Conditions:

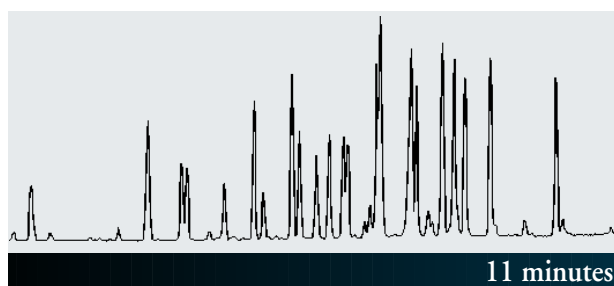
GC: Varian 3800 with Saturn 2000 ion trap
 Column: Rapid-MS, CP-Sil 8 CB-low-bleed/MS 10 m x 0.53 mm, df = 0.25 um, #CP 8132
 Oven: 65° C, 1,5 min → 280° C, 18° C/min
 Carrier gas: Helium, 120kPa 1.2 ml/min, constant flow
 Injection: Splitless : 1.0 ul
 Splitless time : 90 s
 Sample: 1 ng/ul in hexane

Peak Identification		14 Heptachlorepoide
1	Benzene, pentachloro	15 O,P'-DDE
2	Benzene, hexachloro	16 PCB 101
3	Alpha lindane	17 Endosulfan
4	Beta lindane	18 Dieldrin
5	Gamma lindane	19 P,P'-DDE
6	Delta lindane	20 O,P'-DDD
7	PCB 28	21 Endrin
8	Heptachlor	22 PCB 118
9	PCB 52	23 Endosulfan II
10	Aldrin R	24 P,P'-DDT
11	Isobenzan	25 PCB 153
12	Isodrin	26 PCB 138
13	Heptachlor epoxide	27 PCB 180

Conventional



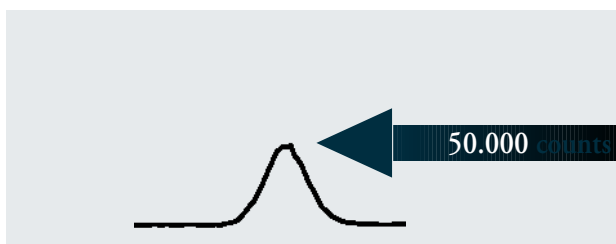
Rapid-MS



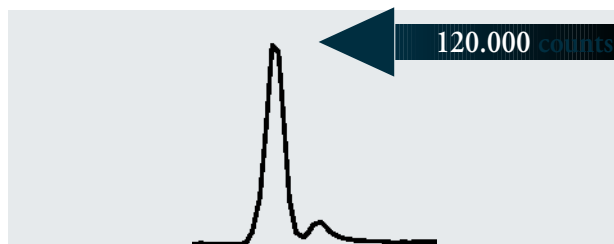
Increased sensitivity & higher signal-to-noise ratio

The signal of an eluting peak depends on its retention time and the peak broadening in the column. Rapid-MS allows peaks to elute up to 10 times faster, which improves the signal by a factor 2-3. An example of typical improvements in signal is shown in the figures below for PCB 180.

Conventional



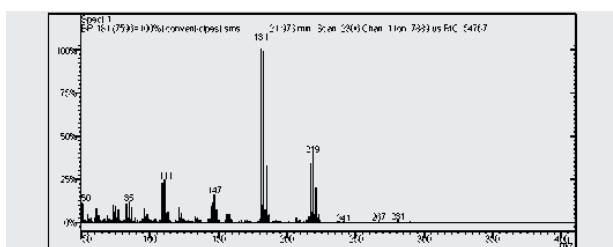
Rapid-MS



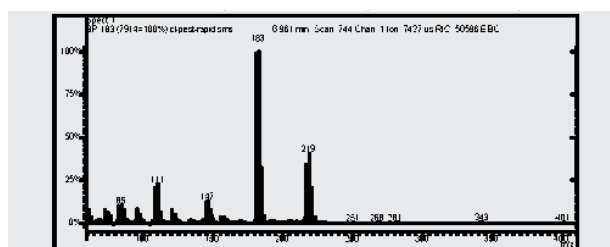
Spectral Integrity

The mass spectra produced with the Rapid-MS columns show direct equivalency with the spectra obtained with the conventional GC-MS. Below see the Rapid-MS spectrum of alpha-lindane on conventional and Rapid MS. Note that the amount of component analyzed here is around 0.5 nanogram.

Conventional

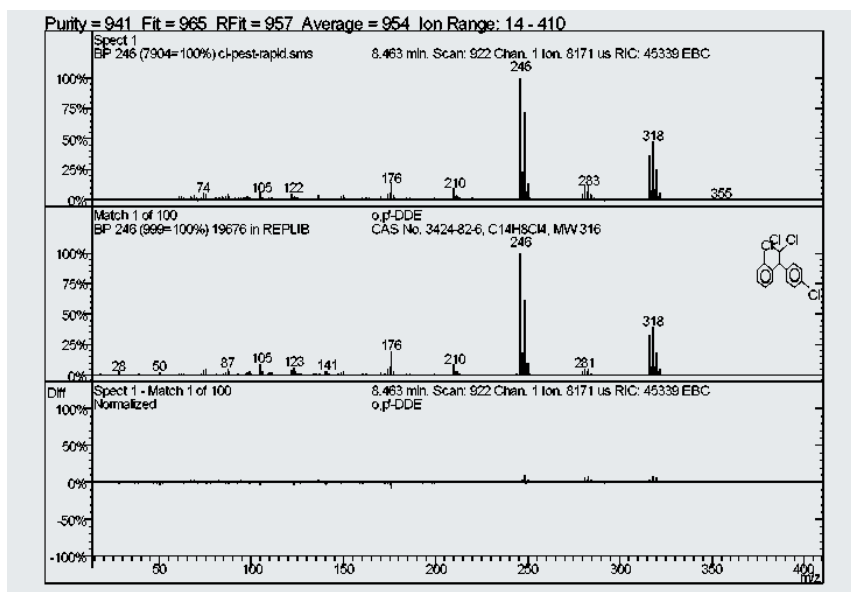


Rapid-MS



Comparison with NIST, Fit factors

The spectra below show the comparison of the isodrin spectrum of the Rapid-MS related to the NIST library. As can be seen, the spectra match quite well. A Reversed Fit(match) factor of 957 was found for o,p'-DDE. The Fit factors of a few other components measured on standard GC/MS and Rapid-MS are listed in the table below.

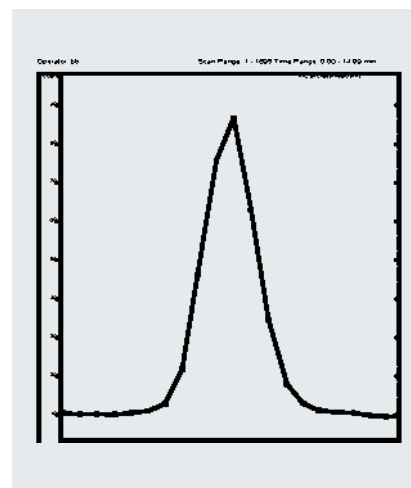


Fit factors

Rapid-MS			Conventional GC/MS		
	Fit	Rfit	Fit	Rfit	
Isodrin	872	880	Isodrin	851	875
O,P-DDE	965	957	O,P-DDE	887	896
Heptachlor	828	841	Heptachlor	843	902

Quantification/data points

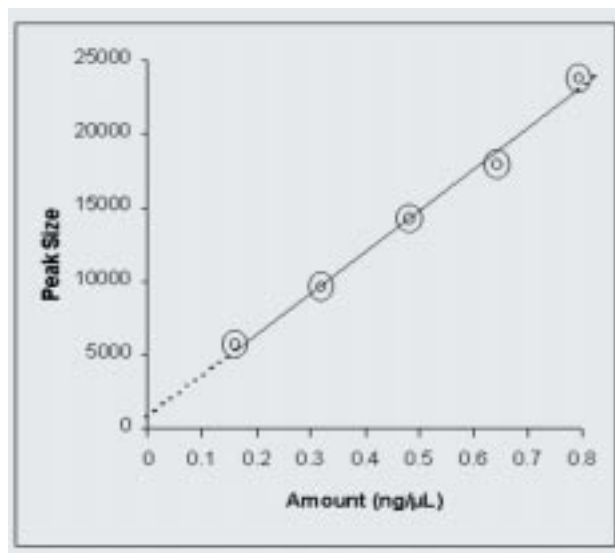
One of the unique characteristics of the Rapid-MS technology is that the eluting peaks from the Rapid-MS column are very fast, but relatively broad. This allows the ion trap system to collect a sufficient number of data points for accurate quantification. This can even be done for fast eluting components. For Chlorinated pesticides, under the conditions mentioned, we have 9 data points per peak where each data point consists of 2 micro-scans. Accuracy is very good: Standard deviation on response factors is within 3%. One could speed up this analysis even more using a faster temperature program.



Sufficient datapoints per peak

Linearity

The linearity for nitrogen pesticides analyzed with Rapid-MS is very good: Five point calibration curves were prepared showing near perfect linearity. The graph for pentachlorobenzene is shown below. Correlation coefficients for pentachlorobenzene is 0.995. Typical correlation coefficients of several components are listed in the table.



Correlation coefficients

Benzene, pentachloro	0.995
Gamma lindane	0.993
PCB 28	0.992
Aldrin R	0.991
Isobenzan	0.994
Heptachlor epoxide	0.995
O,P'-DDE	0.996
Endosulfan	0.993
Dieldrin	0.988
P,P'-DDE	0.996
O,P'-DDD	0.998
Endosulfan II	0.998
P,P'-DDT	0.996



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