The Agilent 5977B GC/MSD and High Efficiency Source (HES) Lowers Detection for Semivolatile Compounds

Application Note

Authors

Dale Walker, Melissa Churley, and Harry Prest

Abstract

The enhanced signal produced by the High Efficiency Source (HES) of the Agilent 5977B GC/MSD allows for flexibility in analytical approach. To assist setting expectations for strategies in semivolatile organic compound analysis, this application note suggests preliminary instrument detection limits for a range of analytes of interest. The results show that picogram or sub-picogram detection in scan mode is possible for a wide range of compounds.

Agilent Technologies

Introduction

Semivolatile organic compounds (SVOCs) are a broad class of environmentally significant contaminants of global interest. These compounds are found on a variety of target analyte lists in GC/MS methods such as the USEPA 8270 and 525 methods, and comparable methods elsewhere. Although listed as targets and appropriate to selected ion monitoring (SIM) in GC/MS analysis, surveying samples by scanning GC/MS provides advantages such as full scan spectra for compound confirmation, tentatively identifying unexpected unknowns in samples that would escape SIM. In the past, scan sensitivity was borderline or insufficient when compared to SIM or the required detection limits. The High Efficiency Source (HES) of the Agilent 5977B GC/MSD represents a revolution in ion source design with greatly enhanced sensitivity that can be exploited to produce scan detection limits for SVOCs that were formerly only approached by SIM. This application note provides preliminary results for instrument detection limits (IDLs) for a few SVOCs across the classes of compounds typical to this analysis.

Experimental

Many configurations and approaches are used for SVOCs, but common to all is a 5-phase column with a thickness appropriate to the loaded range. Because this was a survey of compounds and a preliminary investigation, an Agilent J&W DB-UI 8270D Ultra Inert GC column (0.25 mm × 30 m, 0.5 µm) was used, as is common in this analysis. This choice would favor higher amounts on-column, although the data suggest that a better approach would be based on a thinner film. Standards were prepared in dichloromethane, and 0.5 µL was injected using a 5-µL syringe in pressure-pulsed splitless mode into a double-taper liner. Replicate injections of a 5 ng/mL standard were used to determine an IDL for each compound. The Agilent 7890B GC with a 5977B HES was operated in scan mode from 50 to 550 u (sampling = 4), and a very low gain factor (0.1) to be pertinent to the desire for a working concentration range. This is essentially a standard configuration, operated in a conservative mode to survey possible IDLs.

Results and Discussion

Estimated IDLs for the scan data were calculated using the external standard method, and were based on eight consecutive injections of 12 total injections. The average of five IDL determinations was reported as the IDL. As can be seen in Table 1, sub-picogram scan detection is common, with a few compounds showing picogram levels due primarily to lowered compound target ion response. Compound chromatography also played a role in some cases (for example, benzo[b]- and [k]fluoranthene, and so forth).

Conclusions

Clearly, compound detection in scan mode is now able to discern amounts previously attained only in SIM mode. This advantage allows several analytical strategies to be explored and applied. The shoot less and get more approach means applying split injections with accelerated run times if high concentration levels wish to be maintained. Shooting less sample would also put less matrix in the liner, column, and so forth, and allow the analyst to get more runs before servicing is required. The prep less and save more approach means processing less sample. This would save time and costs not only in collection and transport, but in solvent use and disposal. These dramatically lowered scan IDLs also suggest that SIM IDLs will be enhanced, and so a combination of both strategies is possible to result in the most time and cost effective analysis possible.

Table 1. Agilent 5977B Scan Mode Instrument Detection Limits for SVOCs

Compound	Scan IDL (pg)	Compound	Scan IDL (pg)
Dimethyl phthalate	0.4	*o*-Cresol	2.5
Diethyl phthalate	1.1	*p*-Cresol	2.6
Di-*n*-butyl phthalate	0.9	2,4-Dimethylphenol	0.5
Butyl benzyl phthalate	5.7	2,4-Dichlorophenol	0.5
Bis(2-ethylhexyl) phthalate	0.5	4-Chloro-3-methylphenol	1.6
Di-*n*-octyl phthalate	2.1	2,4,6-Trichlorophenol	9.1
1,3-Dichlorobenzene	0.3	2,4,5-Trichlorophenol	3.4
1,4-Dichlorobenzene	0.3	Naphthalene	0.2
Benzyl alcohol	3.1	2-Methylnaphthalene	0.4
1,2-Dichlorobenzene	0.3	2-Chloronaphthalene	0.3
1,2,4-Trichlorobenzene	0.3	Acenapthylene	0.4
Azobenzene	0.6	Acenaphthene	0.8
Hexachlorobenzene	2.1	Dibenzofuran	0.3
Bis(2-chloroethyl) ether	1.4	Fluorene	0.4
Bis(2-chloro-1-methylethyl) ether	0.6	Phenanthrene	0.2
Aniline	0.4	Anthracene	0.3
N Nitroso-di-*n*-propylamine	2.2	Fluoranthene	0.8
Nitrobenzene	0.4	Pyrene	0.8
4-Chloroaniline	0.9	Benz[a]anthracene	0.4
2-Nitroaniline	1.3	Chrysene	0.3
2,6-Dinitrotoluene	1	Benzo[b]fluoranthene	0.7
3-Nitroaniline	2.8	Benzo[k]fluoranthene	0.7
2,4-Dinitrotoluene	1.4	Benzo[a]pyrene	0.9
4-Nitroaniline	3.8	Indeno[1,2,3-cd]pyrene	0.7
Diphenylamine	0.6	Dibenz[a,h]anthracene	1.3
Phenol	0.60	Benzo[g,h,i]perylene	0.6
2-Chlorophenol	0.5		

For More Information

These data represent typical results. For more information on our products and services, visit our Web site at www.agilent.com/chem.

www.agilent.com/chem

Printed in the USA
January 21, 2016
5991-6612EN

Agilent Technologies