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Application Note 159

Quantitative Analysis of Semivolatile Organic Compounds on Meridian MDN-5S

Due to the sensitivity of a mass spectrometric detector, it is imperative to use a column that will not bleed excessively when analyzing semivolatiles. The MDN™-5S offers both low bleed at high temperature and inertness.

Key Words:
● semivolatile ● GC/MS ● bleed

Analytes termed “semivolatiles” include acidic, basic and neutral compounds such as polynuclear aromatics and phthalates. Specific lists of these compounds have been identified as environmental pollutants and are routinely monitored for in soil, water, and air. A common method for the analysis of semivolatiles requires the use of a mass spectrometric detector (MSD) for positive identification and quantification. The analysis of semivolatiles requires that the column be brought to a final temperature in the range of 300°C. Due to the sensitivity of an MSD, it is imperative to use a column that will not bleed excessively at these temperatures and contaminate the ion source of the mass spectrometer.

The MDN-5S offers both low bleed at high temperature and inertness. These attributes are required to analyze semivolatile compounds. We used a 30m x 0.32mm ID, 0.50µm MDN-5S to generate a calibration curve typical for this type of analysis. Using a split injection allowed the calibration range to be run from 10 to 180ppm, and the constant flow option in the GCs EPC kept the analysis time to under 25 minutes. The TIC of an 80ppm standard is presented in Figure A. Very little column bleed was observed, even at 300 °C.

Figure B illustrates the resolution which was achieved between the difficult to separate pairs benzo(b)fluoranthene/benzo(k)fluoranthene and indeno(1,2,3-cd)pyrene/dibenzo(a,h)anthracene. The benzo (b) & (k) isomers have the same quantitation ion, which can lead to miss-identifications by many GC/MS data systems if they are not adequately resolved.

The results of the multipoint calibration are presented in Table 1. The retention times of each compound are listed along with the ions used to determine their relative response factors. Most compounds had RSD values of <15%, indicating that the column showed good linearity in this range. The inertness of the column provides adequate response for very active compounds such as 2,4-dinitrophenol, pentachlorophenol and benzidine despite the very low on-column amounts (2ng for the lowest standard and 36ng for the highest).

Figure A. Semivolatile Standard on a 30m x .32mm ID, 0.50µm Meridian™ MDN-5S Column

Sample:	Semivolatile standard, 80ppm in methylene chloride
Column:	**MDN-5S, 30m x 0.32mm ID, 0.50µm**
Cat. No.:	**24393**
Injection:	2µl, split 10:1
Flow:	2.5 ml/min, He (constant)
Oven:	50°C (1 min) to 120°C at 15°C/min, to 280°C at 20°C/min, to 300°C at 2 °C/min
Inj. Temp.:	280°C
Interface Temp.:	290°C
Scan Range:	HP 5973MSD, 45-450 amu,

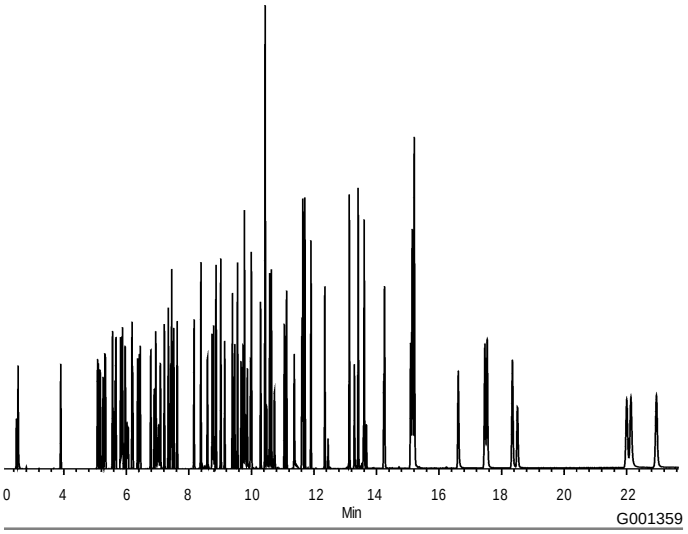

Figure B. Key Semivolatile Resolutions on the 30m x 0.32mm ID, 0.50µm Meridian MDN-5S Column

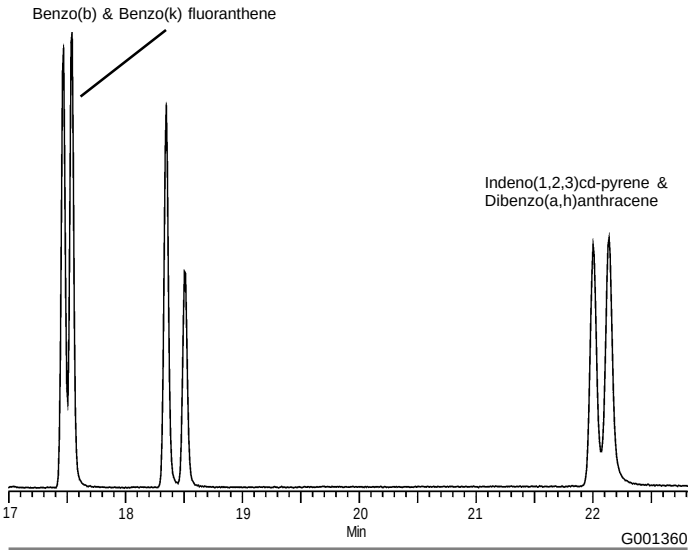

Table 1. Results of Multipoint Calibration on the MDN-5S

Compound	RT	Quant. Ion	Avg. RRF	%RSD	Compound	RT	Quant Ion	Avg. RRF	%RSD
N-nitrosodimethylamine	2.48	74	0.848	4.0	3-Nitroaniline	9.68	138	0.357	4.8
Pyridine	2.54	79	1.54	4.2	Acenaphthene-d10	9.74	164		
2-Fluorophenol	3.90	112	1.247	1.8	Acenaphthene	9.78	153	1.202	6.6
Phenol	5.10	94	1.809	3.6	2,4-Dinitrophenol	9.81	184	0.152	19.8
Phenol-d6	5.09	99	1.76	3.1	4-Nitrophenol	9.87	109	0.248	14.8
Aniline	5.16	66	0.869	9.2	2,4-Dinitrotoluene	9.98	165	0.427	8.1
Bis(2-chloroethylether)	5.26	95	0.436	3.4	Dibenzofuran	10.00	168	1.788	7.4
2-Chlorophenol-d4	5.31	132	1.358	2.2	Diethyl phthalate	10.29	149	1.396	5.6
2-Chlorophenol	5.33	128	1.426	2.9	Fluorene	10.44	166	1.29	8.2
1,3-Dichlorobenzene	5.56	146	1.429	5.2	4-Chlorophenyl phenyl ether	10.44	204	0.740	8.1
1,4-Dichlorobenzene-d4	5.64	152			4-Nitroaniline	10.45	138	0.381	4.7
1,4-Dichlorobenzene	5.67	146	1.466	4.9	2-Methyl-4,6-dinitrophenol	10.49	198	0.101	37.3
Benzyl Alcohol	5.81	108	0.943	3.8	N-nitrosodiphenylamine	10.58	169	0.506	6.8
1,2-Dichlorobenzene-d4	5.86	152	0.919	6.1	Azobenzene	10.64	77	0.752	8.3
1,2-Dichlorobenzene	5.88	146	1.388	4.6	2,4,6-Tribromophenol	10.74	330	0.218	9.7
2-Methylphenol	5.96	108	1.285	4.1	4-Bromophenyl phenyl ether	11.06	248	0.215	3.6
Bis(2-chloroisopropyl)ether	6.02	45	1.214	4.1	Hexachlorobenzene	11.12	284	0.242	4.2
4-Methylphenol	6.18	108	1.401	3.9	Pentachlorophenol	11.37	266	0.129	26.9
N-nitroso-di-n-propylamine	6.21	70	1.082	4.2	Phenanthrene-d10	11.62	188		
Nitrobenzene-d5	6.42	82	0.428	4.2	Phenanthrene	11.65	178	1.028	7.8
Nitrobenzene	6.45	77	0.436	6.0	Anthracene	11.71	178	1.045	6.4
Isophorone	6.78	82	0.721	5.2	Carbazole	11.91	167	1.022	6.4
2-Nitrophenol	6.89	139	0.187	9.4	Di-n-butyl phthalate	12.35	149	1.095	7.3
2,4-Dimethylphenol	6.94	107	0.381	4.2	Fluoranthene	13.13	202	1.232	5.2
Benzoic Acid	7.03	105	0.262	22.6	Pyrene	13.41	202	1.191	6.9
bis(2-Chloroethoxy)methane	7.09	93	0.429	5.4	Benzidine	13.29	184	0.419	12.4
2,4-Dichlorophenol	7.21	162	0.297	4.9	Aramite #1	13.59	185	0.056	21.0
1,2,4-Trichlorobenzene	7.34	180	0.341	6.6	Terphenyl-d14	13.61	244	0.902	5.2
Naphthalene-d8	7.43	136			Aramite #2	13.68	185	0.075	19.7
Naphthalene	7.45	128	1.057	7.5	3,3'-Dimethylbenzidine	14.24	212	0.376	14.3
4-Chloroaniline	7.52	127	0.451	5.9	Butylbenzyl phthalate	14.26	149	0.477	6.0
Hexachlorobutadiene	7.63	225	0.208	4.6	3,3'-Dichlorobenzidine	15.10	252	0.388	11.8
4-Chloro-3-methylphenol	8.17	107	0.348	5.2	Benzo(a)anthracene	15.14	228	1.107	4.2
2-Methylnaphthalene	8.39	142	0.688	5.9	Chrysene-d12	15.15	240		
Hexachlorocyclopentadiene	8.60	237	0.377	10.9	Chrysene	15.21	228	1.028	6.1
2,4,6-Trichlorophenol	8.75	196	0.399	8.2	Bis(2-ethylhexyl)phthalate	15.21	149	0.603	9.4
2,4,5-Trichlorophenol	8.79	196	0.442	2.5	Di-n-octyl phthalate	16.62	149	1.089	22.6
2-Fluorobiphenyl	8.87	172	1.398	7.0	Benzo(b)fluoranthene	17.47	252	1.217	8.1
2-Chloronaphthalene	9.02	162	1.170	7.0	Benzo(k)fluoranthene	17.54	252	1.260	3.5
2-Nitroaniline	9.15	65	0.423	5.0	Benzo(a)pyrene	18.35	252	1.124	7.8
Dimethyl phthalate	9.39	163	1.347	5.6	Perylene-d12	18.50	265		
2,6-Dinitrotoluene	9.47	165	0.305	7.5	Indeno(1,2,3)cd-pyrene	22.01	276	1.010	12.0
Acenaphthylene	9.56	152	1.898	6.5	Dibenzo(a,h)anthracene	22.14	278	1.059	6.4
					Benzo(g,h,I)perylene	22.95	276	1.174	3.3

internal standards

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Meridian – Sigma-Aldrich Co.

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Fused silica columns manufactured under HP US Pat. No. 4,293,415.

Ordering Information:

Description	Cat. No.
Meridian MDN-5S Capillary Column	
30m x 0.32mm ID, 0.50µm film	**24393**

Contact our Technical Service Department (phone 800-359-3041 or 814-359-3041, FAX 800-359-3044 or 814-359-5468) for expert answers to your questions.

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