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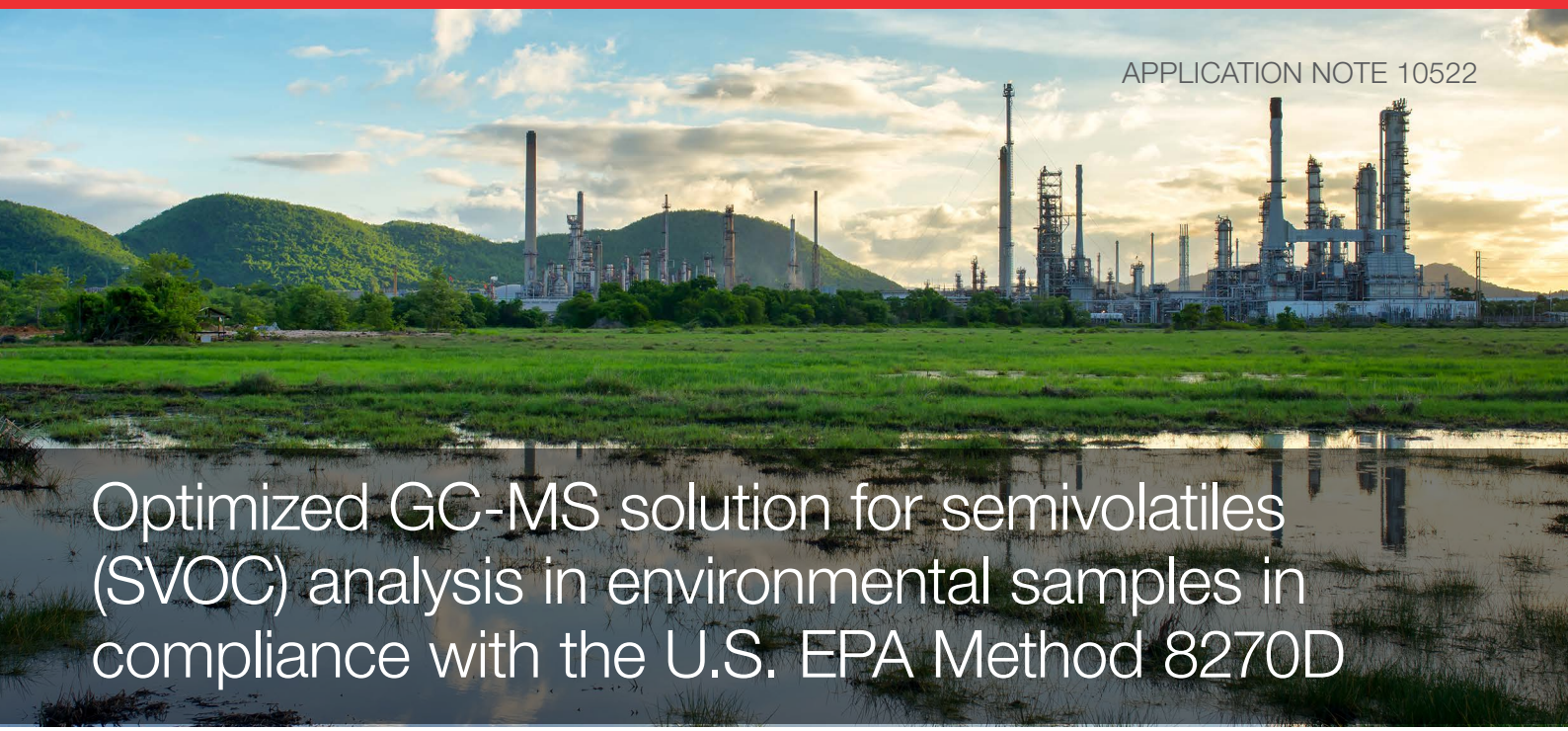

Optimized GC-MS solution for semivolatiles (SVOC) analysis in environmental samples in compliance with the U.S. EPA Method 8270D

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Introduction

The United States Environmental Protection Agency (U.S. EPA) released the first Semivolatile Organic Compounds (SVOC) method by Gas Chromatography/Mass Spectrometry (Method 8270) at the end of 1980. It is a common method used in almost all environmental laboratories looking to analyze semivolatile organic compounds in extracts prepared from many types of solid waste matrices, soils, air sampling media, and water.[1] Since then, single quadrupole mass spectrometers have become much more sensitive and the source fragmentation has changed. Many original assumptions[2] about the origin and nature of the ion species have proven to be wrong or require correction, while the new generations of the mass spectrometers have proven to provide more response in the high-mass region,[3] resulting in adjustment of the tuning criteria to be met.[4] To adjust to these changes, the EPA has changed the ion abundance criteria for the passing of DFTPP ion ratio criteria in EPA Method 8270D.

This application note shows how the Thermo Scientific™ ISQ™ 7000 single quadrupole GC-MS system can meet Method 8270D requirements with the extended dynamic range detection system. The working method range was shown to be 0.2–200 ppm using the same column.

ThermoFisher SCIENTIFIC

Particular attention has been posed on maximizing the uptime of the instrument, as required by high-throughput laboratories. The innovative Thermo Scientific™ NeverVent™ technology available on the ISQ 7000 GC-MS system is a unique solution for speeding up the routine maintenance operations, saving the time typically required to vent the MS system and re-establish the vacuum conditions.

The new Thermo Scientific™ Instant Connect Helium Saver Injector was also assessed in this application note to show that significant financial costs savings can be realized throughout the lifetime of a GC-MS instrument without compromising the instrument's performance.

Experimental

The method was tested on five ISQ 7000 GC-MS systems equipped with the Thermo Scientific™ ExtractaBrite™ ion source to assess method transferability and instrument-to-instrument variability. Both ranges (0.2–50 ppm and 2–200 ppm) were validated using the Instant Connect Helium Saver Injector (P/N 19070013) and the Thermo Scientific™ Instant Connect Split-Splitless (SSL) Injector module (P/N 19070010). The column used was a Thermo Scientific™ TraceGOLD™ TG-5MS GC Column with 5 m guard, 30 m × 0.25 mm × 0.25 µm (P/N 26098-1425). A Thermo Scientific™ Injection Port Deactivated Liner 4 mm ID × 105 mm (P/N 453A1925) was selected for the Split-Splitless injection port. The ISQ 7000 GC-MS system operated in full-scan mode and the Thermo Scientific™ Chromeleon™ Chromatography Data System (CDS) software was used to acquire, process, and report data. The operating parameters for the Thermo Scientific™ TRACE™ 1310 GC system are reported in Table 1a (splitless method, range 0.2–50 ppm) and Table 1b (split method, range 2–200 ppm). The ISQ 7000 single quadrupole MS operating conditions are detailed in Tables 2a and 2b.

Table 1a. TRACE 1310 GC system parameters for splitless method.

Injection Volume (µL)	1.0
Liner	Deactivated Splitless Liner
Inlet Temp (°C)	270
Inlet Module and Mode	SSL in Surge Splitless at 345 kPa for 0.6 min
Splitess Time (min)	0.6
Split Flow (mL/min)	50
Oven Temperature Program	
Initial Temperature 1 (°C)	35
Hold Time (min)	2.25
Rate (°C/min)	25
Temperature 2 (°C)	100
Hold Time (min)	0.1
Rate (°C/min)	30
Temperature 3 (°C)	280
Hold Time (min)	0.1
Rate (°C/min)	10
Temperature 4 (°C)	320
Hold Time (min)	5.00

Table 1b. TRACE 1310 GC system parameters for split method.

Injection Volume (µL)	1.0
Liner	Deactivated Splitless Liner
Inlet Temp (°C)	310
Inlet Module and Mode	SSL in Split Mode
Split Ratio	10:1
Split Flow (mL/min)	15
Carrier Gas (mL/min)	He, 1.5
Oven Temperature Program	
Initial Temperature 1 (°C)	35
Hold Time (min)	2.25
Rate (°C/min)	25
Temperature 2 (°C)	100
Hold Time (min)	0.1
Rate (°C/min)	30
Temperature 3 (°C)	280
Hold Time (min)	0.1
Rate (°C/min)	10
Temperature 4 (°C)	320
Hold Time (min)	5.00

Table 2a. ISQ 7000 Single Quadrupole MS parameters for splitless method.

Transfer Line Temp (°C)	300
Ion Source	ExtractaBrite
Ion Source Temp (°C)	300
Ionization Mode	EI
Electron Energy (eV)	70
Acquisition Mode	Full-scan
Scan Range (*m/z*)	35–500
Emission Current (mA)	10
Dwell Time	0.1

Table 2b. ISQ 7000 Single Quadrupole MS parameters for split method.

Transfer Line Temp (°C)	310
Ion Source	ExtractaBrite
Ion Source Temp (°C)	300
Ionization Mode	EI
Electron Energy (eV)	70
Acquisition Mode	Full-scan
Scan Range (*m/z*)	35–500
Emission Current (mA)	15
Dwell Time	0.1

Tuning for DFTPP

The ISQ 7000 MS system was tuned with a built-in EPA 8270D specifically designed tune (DFTPP Tune). This assures fulfillment of all method requirements in terms of ion abundance criteria. A tune verification DFTPP solution was injected to verify that the ISQ 7000 GC-MS system met the tuning requirements shown in Figure 1. Chromeleon CDS software has a dedicated reporting package for environmental laboratories, and automatically reports tune evaluation performance with a Pass/Fail indicator (Table 3).

Standard and sample preparation

Standards (Restek 8270 MegaMix Cat. No. 31850, AccuStandard Internal Standard Cat. No. Z-014J, AccuStandard Surrogate Cat No. M-8270-SS) were prepared in methylene chloride, and the internal standards were spiked at a concentration of 5 ppm for both the splitless and split methods. Spiking the range of 0.2 to 200 ppm with the same concentration of internal standards eliminated the necessity of preparing two different sets of calibration standards. Table 4 contains the calibration levels of both methods.

A volume of 1 µL of the calibration standards was injected for all methods. Figure 2 shows the chromatogram of the 5 ppm calibration standard acquired in splitless mode.

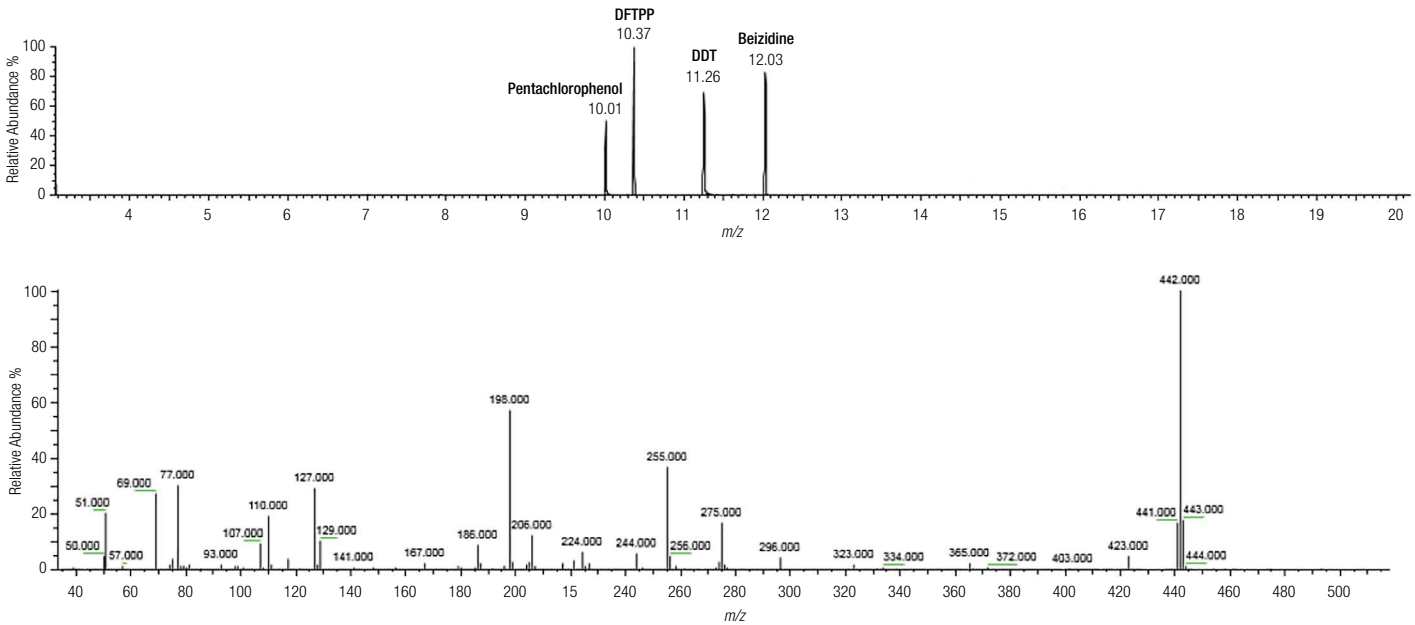

Figure 1. Acquired DFTTP mass spectrum using the ISQ 7000 single quadrupole GC-MS system operated in full-scan at 70 eV ionization energy.

Table 3. DFTPP spectrum check for ion abundance criteria.

Eval Mass (*m/z*)	Ion Abundance Criteria	Measured % Relative Abundance	Criteria Pass/ Fail
51	Greater than or equal to 10% AND less than or equal to 80% of Base Peak	20.7	Pass
68	Less than 2% of *m/z* 69	0.7	Pass
70	Less than 2% of *m/z* 69	0.5	Pass
127	Greater than or equal to 10% AND less than or equal to 80% of Base Peak	29.4	Pass
197	Less than 2% of *m/z* 198	0.1	Pass
198	Greater than 50% AND less than or equal to 100% of Base Peak	57.5	Pass
199	Greater than or equal to 5% AND less than or equal to 9% of *m/z* 198	5.9	Pass
275	Greater than or equal to 10% AND less than or equal to 60% of Base Peak	17.2	Pass
365	Greater than 1% of *m/z* 198	4.6	Pass
441	Greater than 0% AND less than 24% of *m/z* 442	17.4	Pass
442	Greater than 50% AND less than or equal to 100% of Base Peak	100.0	Pass
443	Greater than or equal to 15% AND less than or equal to 24% of *m/z* 442	18.1	Pass

Table 4. Calibration standards used for testing the splitless and split methods.

Calibration Standard	Splitless Conc. (ppm)	Split Conc. (ppm)
Cal 1	0.2	2.0
Cal 2	0.5	5.0
Cal 3	1.0	10.0
Cal 4	2.0	20.0
Cal 5	5.0	35.0
Cal 6	10.0	50.0
Cal 7	20.0	100.0
Cal 8	35.0	200.0
Cal 9	50.0	–

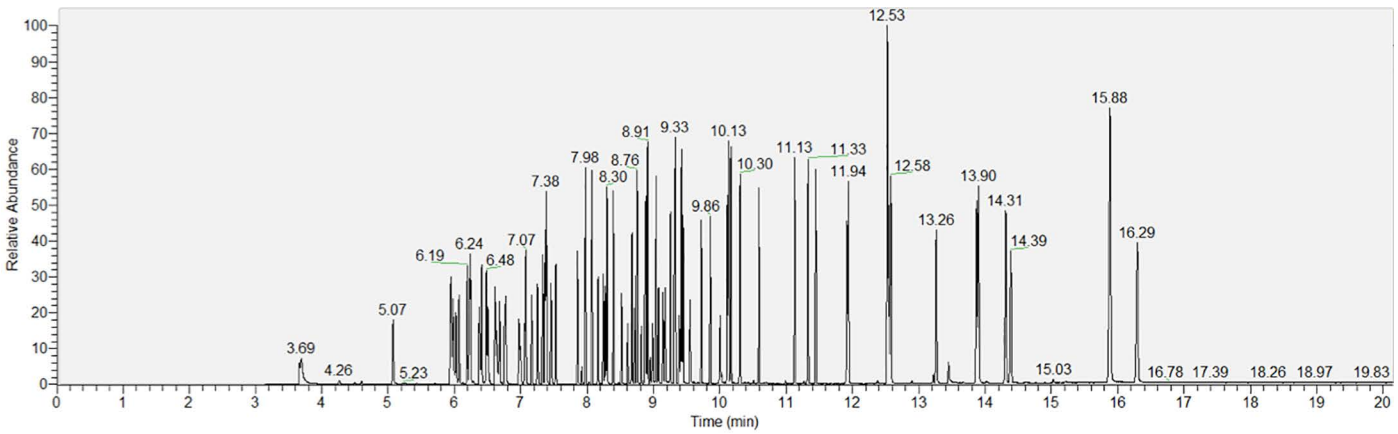

Figure 2. Total ion current (TIC) chromatogram of the 5 ppm EPA 8270 semivolatile calibration standard injected in splitless mode.

Results and discussion

Splitless method 0.2–50 ppm calibration

The average relative response factors of the 76 targeted compounds and six surrogates were calculated by analyzing the nine calibration standards from 0.2 ppm to 50 ppm in methylene chloride. Six compounds had Response Factors %RSD >20% and required an alternative curve fit. The %RSDs of those compounds calibrated using average response factors and r^2 values for the six alternative fit compounds are shown in Table 5.

Split method 2–200 ppm calibration

The average response factors of the 76 targeted compounds and six surrogates were calculated by analyzing eight calibration standards with concentrations ranging from 2 ppm to 200 ppm prepared in methylene chloride. Seven compounds had Response Factors %RSD >20% and required an alternate curve fit. The %RSDs of those compounds calibrated using average response factors and r^2 values for the seven alternative fit compounds are shown in Table 6.

Instant Connect Helium Saver module

Method 8270D was also tested with the Instant Connect Helium Saver module (P/N 19070013). Depending on the experimental conditions, the Helium Saver module allows up to 14 years of GC and GC-MS operation from a single helium cylinder. The inlet is supplied with two different gases: nitrogen is used for the septum purge and split flows with only helium supplying the analytical column. Because of this innovative and patented solution, helium consumption is dramatically reduced.

After time for equilibration, the GC-MS tuning mixture was injected and passed the criteria for EPA Method 8270D. Standards for a calibration curve (0.2–50 ppm and 2–200 ppm) were injected, and the data processed. Table 7a shows the results for splitless method and Table 7b reports split method. In both configurations (SSL and Helium Saver) and for both methods (split and splitless), less than 10% of compounds required an alternative curve fit. All the others had RSD% less than 20% with linear fit.

Minimum response factors

EPA Method 8270D requires a minimum relative response factor (RRF) for any point of the calibration curve for several compounds in the targeted list. Table 8 presents those minimum relative response factor requirements and the minimum RRF across all curves performed on the ISQ 7000 single quadrupole GC-MS system.

Retention times

The four methods: splitless, splitless with Helium Saver, split, and split with Helium Saver, were developed over a period of three weeks. Table 9 demonstrates the stability of the retention times over this period of time. During this time, the liner and septa were changed and the analytical column trimmed. Still, the retention times are reproducible using different methods and different inlet modules. Table 9 shows a comparison of the retention times obtained using different methods and inlet modules.

NeverVent technology

Specifically designed to simplify the routine maintenance procedures and to maximize the GC-MS instrument uptime, the proprietary Vacuum Probe Interlock (VPI) and the V-lock solution available on the ISQ 7000 single quadrupole GC-MS system allow ion source cleaning or column replacement to be performed quickly without breaking the MS vacuum, saving up to 98% of the time typically required to perform those operations. Thanks to the VPI, the ion source can be fully removed—including all of the lenses and the repeller—through the front vacuum interlock, without venting the system. This allows cleaning the source, swapping it, or changing ionization type, and being ready to run samples within minutes, not hours or days. Additionally, the V-lock technology allows the MS under vacuum to be fully isolated from the GC system, permitting not only a quick replacement of the analytical column when necessary, but also quick and safe performance of regular maintenance at the injector side, like replacing the septum or the liner or trimming the analytical column, without the use of any additional post-column or auxiliary gas flow into the MS.

Table 5. Response factors %RSDs as well as coefficient of determination values (r^2) determined from the calibration curve acquired over a concentration range of 0.2–50 ppm (splitless injections).

Compound	%RSD	r^2	Compound	%RSD	r^2
N-Nitrosodimethylamine	11.53	—	Acenaphthylene	8.24	—
Pyridine	10.23	—	1,2-Dinitrobenzene	14.85	—
2-fluorophenol (surrogate)	5.57	—	3-Nitroaniline	8.09	—
Phenol-d6 (surrogate)	4.99	—	**Acenaphthene-d10**	5.78	—
Aniline	6.39	—	Acenaphthene	7.57	—
Phenol	7.30	—	2,4-dinitrophenol	—	0.9867
Bis (2-chloroethyl) ether	7.95	—	Phenol, 4-nitro-	18.15	—
Phenol, 2-chloro-	6.19	—	Dibenzofuran	6.78	—
Benzene, 1,3-dichloro-	6.29	—	2,4-dinitrotoluene	12.32	—
1,4-Dichlorobenzene-d4	4.90	—	Phenol, 2,3,5,6-tetrachloro-	—	0.9957
Benzene, 1,4-dichloro-	7.57	—	Phenol, 2,3,4,6-tetrachloro-	—	0.9965
Benzyl alcohol	7.33	—	Diethyl Phthalate	5.60	—
Benzene, 1,2-dichloro-	7.43	—	4-chlorophenylphenylether	6.50	—
Phenol, 2-methyl-	6.27	—	Fluorene	7.31	—
Bis (2-chloroisopropyl) ether	6.31	—	4-nitroaniline	7.88	—
Phenol, 3&4-methyl-	6.52	—	4,6-Dinitro-2-methylphenol	—	0.9945
N-Nitroso-di-n-propylamine	6.63	—	Diphenylamine	9.61	—
Ethane, hexachloro-	5.80	—	Azobenzene	7.06	—
Nitrobenzene-D5 (surrogate)	5.90	—	2,4,6-tribromophenol (surrogate)	—	0.9963
Benzene, nitro-	3.20	—	4-bromophenylphenylether	4.30	—
Isophorone	3.90	—	Hexachlorobenzene	8.18	—
Phenol, 2-nitro-	13.14	—	Phenol, pentachloro-	—	0.9960
Phenol, 2,4-dimethyl-	4.52	—	Phenanthrene	10.88	—
Bis (2-chloroethoxy) methane	5.17	—	**Phenanthrene-d10-**	3.54	—
Phenol, 2,4-dichloro-	4.76	—	Anthracene	11.38	—
Benzene, 1,2,4-trichloro-	6.17	—	Carbazole	9.69	—
Naphthalene	8.26	—	Di-n-butyl phthalate	8.10	—
Naphthalene-d8	5.02	—	Fluoranthene	10.94	—
p-Chloroaniline	4.95	—	Pyrene	10.68	—
1,3-Butadiene, 1,1,2,3,4,4-hexachloro-	5.36	—	p-Terphenyl-d14 (surrogate)	6.76	—
Phenol, 4-chloro-3-methyl-	4.14	—	Benzyl butyl phthalate	8.69	—
Naphthalene, 2-methyl	7.54	—	Bis (2-ethylhexyl) adipate	6.08	—
Naphthalene, 1-methyl-	7.00	—	Benz[a]anthracene	9.68	—
Hexachlorocyclopentadiene	9.80	—	Chrysene	9.38	—
Phenol, 2,4,5-trichloro-	8.21	—	**Chrysene-d12**	4.02	—
Phenol, 2,4,6-trichloro-	5.90	—	Bis (2-ethylhexyl) phthalate	7.42	—
2-fluorobiphenyl (surrogate)	4.99	—	Di-n-octylphthalate	6.30	—
Naphthalene, 2-chloro-	7.24	—	Benzo[b]fluoranthene	6.70	—
2-Nitroaniline	10.43	—	Benzo[k]fluoranthene	8.48	—
1,4-Dinitrobenzene	16.05	—	Benzo[a]pyrene	6.11	—
Dimethyl phthalate	5.66	—	**Perylene-d12**	5.73	—
Benzene, 1,3-dinitro-	13.75	—	Indeno[1,2,3-cd]pyrene	6.36	—
2,6-dinitrotoluene	6.11	—	Dibenzo[a,h]anthracene	6.39	—
			Benzo[g,h,i]perylene	7.75	—

Boldface indicates Internal Standards

Table 6. Response factors %RSDs as well as coefficient of determination values (r^2) determined from the calibration curve acquired over a concentration range of 0.2–200 ppm (10:1 split injections).

Compound	%RSD	r^2	Compound	%RSD	r^2
N-Nitrosodimethylamine	6.31	—	Acenaphthylene	6.59	—
Pyridine	10.80	—	1,2-Dinitrobenzene	15.11	—
2-fluorophenol (surrogate)	4.30	—	3-Nitroaniline	14.42	—
Phenol-d6 (surrogate)	4.19	—	**Acenaphthene-d10**	**7.23**	—
Aniline	4.89	—	Acenaphthene	7.98	—
Phenol	5.48	—	2,4-dinitrophenol	—	0.9984
Bis (2-chloroethyl) ether	4.45	—	Phenol, 4-nitro-	—	0.9982
Phenol, 2-chloro-	4.94	—	Dibenzofuran	8.91	—
Benzene, 1,3-dichloro-	5.03	—	2,4-dinitrotoluene	18.65	—
1,4-Dichlorobenzene-d4	**6.01**	—	Phenol, 2,3,5,6-tetrachloro-	17.58	—
Benzene, 1,4-dichloro-	5.09	—	Phenol, 2,3,4,6-tetrachloro-	12.33	—
Benzyl alcohol	9.21	—	Diethyl Phthalate	7.83	—
Benzene, 1,2-dichloro-	4.76	—	4-chlorophenylphenylether	7.93	—
Phenol, 2-methyl-	6.77	—	Fluorene	9.13	—
Bis (2-chloroisopropyl) ether	4.85	—	4-nitroaniline	13.30	—
Phenol, 3&4-methyl-	5.92	—	4,6-Dinitro-2-methylphenol	-	0.9983
N-Nitroso-di-n-propylamine	6.23	—	Diphenylamine	8.13	—
Ethane, hexachloro-	4.85	—	Azobenzene	9.24	—
Nitrobenzene-D5 (surrogate)	10.59	—	2,4,6-tribromophenol (surrogate)	13.23	—
Benzene, nitro-	10.24	—	4-bromophenylphenylether	6.37	—
Isophorone	5.18	—	Hexachlorobenzene	5.72	—
Phenol, 2-nitro-	19.20	—	Phenol, pentachloro-	—	0.9981
Phenol, 2,4-dimethyl-	4.92	—	Phenanthrene	6.32	—
Bis (2-chloroethoxy) methane	8.67	—	**Phenanthrene-d10-**	**6.95**	—
Phenol, 2,4-dichloro-	5.68	—	Anthracene	7.23	—
Benzene, 1,2,4-trichloro-	5.74	—	Carbazole	11.25	—
Naphthalene	5.74	—	Di-n-butyl phthalate	6.69	—
Naphthalene-d8	**6.53**	—	Fluoranthene	7.64	—
p-Chloroaniline	6.02	—	Pyrene	6.93	—
1,3-Butadiene, 1,1,2,3,4,4-hexachloro-	5.54	—	p-Terphenyl-d14 (surrogate)	6.38	—
Phenol, 4-chloro-3-methyl-	8.26	—	Benzyl butyl phthalate	6.97	—
Naphthalene, 2-methyl	6.97	—	Bis(2-ethylhexyl)adipate	6.16	—
Naphthalene, 1-methyl-	7.35	—	Benz[a]anthracene	7.43	—
Hexachlorocyclopentadiene	—	0.9991	Chrysene	6.17	—
Phenol, 2,4,5-trichloro-	10.39	—	**Chrysene-d12**	**10.49**	—
Phenol, 2,4,6-trichloro-	7.92	—	Bis (2-ethylhexyl) phthalate	4.95	—
2-fluorobiphenyl (surrogate)	6.45	—	Di-n-octylphthalate	8.70	—
Naphthalene, 2-chloro-	8.16	—	Benzo[b]fluoranthene	7.06	—
2-Nitroaniline	17.03	—	Benzo[k]fluoranthene	6.26	—
1,4-Dinitrobenzene	—	0.9980	benzo[a]pyrene	6.81	—
Dimethyl phthalate	8.30	—	**Perylene-d12**	**14.99**	—
Benzene, 1,3-dinitro-	—	0.9976	Indeno[1,2,3-cd]pyrene	6.15	—
2,6-dinitrotoluene	11.55	—	Dibenzo[a,h]anthracene	6.91	—
			Benzo[g,h,i]perylene	7.06	—

Boldface indicates Internal Standards

Table 7a. Response factors %RSDs for the 76 targeted compounds and internal standards, as well as r^2, for alternative fit calibrations using the Instant Connect Helium Saver module in splitless mode.

Compound	%RSD	r^2	Compound	%RSD	r^2
N-Nitrosodimethylamine	6.62	—	Acenaphthylene	7.34	—
Pyridine	10.56	—	1,2-Dinitrobenzene	16.57	—
2-fluorophenol (surrogate)	6.37	—	3-Nitroaniline	19.06	—
Phenol-d6 (surrogate)	4.82	—	**Acenaphthene-d10**	**3.99**	—
Aniline	13.52	—	Acenaphthene	4.68	—
Phenol	5.41	—	2,4-dinitrophenol	—	0.9938
Bis(2-chloroethyl) ether	17.24	—	Phenol, 4-nitro-	—	0.9950
Phenol, 2-chloro-	6.34	—	Dibenzofuran	6.21	—
Benzene, 1,3-dichloro-	5.80	—	2,4-dinitrotoluene	—	0.9942
1,4-Dichlorobenzene-d4	**2.53**	—	Phenol, 2,3,5,6-tetrachloro-	—	0.9962
Benzene, 1,4-dichloro-	5.17	—	Phenol, 2,3,4,6-tetrachloro-	14.62	—
Benzyl alcohol	18.38	—	Diethyl Phthalate	5.69	—
Benzene, 1,2-dichloro-	5.36	—	4-chlorophenylphenylether	5.32	—
Phenol, 2-methyl-	6.17	—	Fluorene	9.43	—
Bis(2-chloroisopropyl)ether	4.53	—	4-nitroaniline	19.69	—
Phenol, 3&4-methyl-	7.17	—	4,6-Dinitro-2-methylphenol	—	0.9893
N-Nitroso-di-n-propylamine	7.58	—	Diphenylamine	6.12	—
Ethane, hexachloro-	6.39	—	Azobenzene	6.01	—
Nitrobenzene-D5 (surrogate)	8.67	—	2,4,6-tribromophenol (surrogate)	16.16	—
Benzene, nitro-	8.86	—	4-bromophenylphenylether	8.54	—
Isophorone	5.52	—	Hexachlorobenzene	5.49	—
Phenol, 2-nitro-	17.07	—	Phenol, pentachloro-	—	0.9971
Phenol, 2,4-dimethyl-	8.44	—	Phenanthrene	7.12	—
Bis(2-chloroethoxy)methane	8.87	—	**Phenanthrene-d10-**	**2.95**	—
Phenol, 2,4-dichloro-	8.56	—	Anthracene	12.18	—
Benzene, 1,2,4-trichloro-	5.36	—	Carbazole	6.86	—
Naphthalene	5.91	—	Di-n-butyl phthalate	6.59	—
Naphthalene-d8	**2.41**	—	Fluoranthene	8.46	—
p-Chloroaniline	5.82	—	Pyrene	7.82	—
1,3-Butadiene, 1,1,2,3,4,4-hexachloro-	4.82	—	p-Terphenyl-d14 (surrogate)	7.49	—
Phenol, 4-chloro-3-methyl-	8.96	—	Benzyl butyl phthalate	5.81	—
Naphthalene, 2-methyl	5.95	—	Bis(2-ethylhexyl)adipate	9.11	—
Naphthalene, 1-methyl-	6.54	—	Benz[a]anthracene	5.79	—
Hexachlorocyclopentadiene	—	0.9959	Chrysene	6.90	—
Phenol, 2,4,5-trichloro-	13.52	—	**Chrysene-d12**	**4.59**	—
Phenol, 2,4,6-trichloro-	9.81	—	Bis(2-ethylhexyl)phthalate	7.06	—
2-fluorobiphenyl,(surrogate)	6.00	—	Di-n-octylphthalate	7.84	—
Naphthalene, 2-chloro-	5.66	—	Benzo[b]fluoranthene	8.98	—
2-Nitroaniline	17.31	—	Benzo[k]fluoranthene	11.28	—
1,4-Dinitrobenzene	—	0.9962	Benzo[a]pyrene	7.47	—
Dimethyl phthalate	5.88	—	**Perylene-d12**	**5.38**	—
Benzene, 1,3-dinitro-	17.90	—	Indeno[1,2,3-cd]pyrene	8.02	—
2,6-dinitrotoluene	11.80	—	Dibenzo[a,h]anthracene	5.99	—
			Benzo[g,h,i]perylene	7.43	—

Boldface indicates Internal Standards

Table 7b. Response factors %RSDs for the 76 targeted compounds and internal standards, as well as r^2, for alternative fit calibrations using the Instant Connect Helium Saver module in split mode.

Compound	%RSD	r^2	Compound	%RSD	r^2
N-Nitrosodimethylamine	6.62	—	Acenaphthylene	7.25	—
Pyridine	13.09	—	1,2-Dinitrobenzene	17.76	—
2-fluorophenol (surrogate)	6.02	—	3-Nitroaniline	18.05	—
Phenol-d6 (surrogate)	5.71	—	**Acenaphthene-d10**	**4.15**	—
Aniline	6.13	—	Acenaphthene	7.36	—
Phenol	6.52	—	2,4-dinitrophenol	—	0.9965
Bis(2-chloroethyl) ether	5.69	—	Phenol, 4-nitro-	—	0.9978
Phenol, 2-chloro-	7.17	—	Dibenzofuran	6.90	—
Benzene, 1,3-dichloro-	7.28	—	2,4-dinitrotoluene	18.32	—
1,4-Dichlorobenzene-d4	**3.26**	—	Phenol, 2,3,5,6-tetrachloro-	—	0.9957
Benzene, 1,4-dichloro-	8.13	—	Phenol, 2,3,4,6-tetrachloro-	17.05	—
Benzyl alcohol	14.15	—	Diethyl Phthalate	6.09	—
Benzene, 1,2-dichloro-	6.95	—	4-chlorophenylphenylether	8.11	—
Phenol, 2-methyl-	6.68	—	Fluorene	8.51	—
Bis(2-chloroisopropyl)ether	6.28	—	4-nitroaniline	19.17	—
Phenol, 3&4-methyl-	6.42	—	4,6-Dinitro-2-methylphenol	—	0.9987
N-Nitroso-di-n-propylamine	7.31	—	Diphenylamine	7.24	—
Ethane, hexachloro-	9.32	—	Azobenzene	7.28	—
Nitrobenzene-D5 (surrogate)	10.02	—	2,4,6-tribromophenol (surrogate)	14.93	—
Benzene, nitro-	11.59	—	4-bromophenylphenylether	7.06	—
Isophorone	6.70	—	Hexachlorobenzene	7.82	—
Phenol, 2-nitro-	14.78	—	Phenol, pentachloro-	—	0.9991
Phenol, 2,4-dimethyl-	5.90	—	Phenanthrene	8.55	—
Bis(2-chloroethoxy)methane	5.64	—	**Phenanthrene-d10-**	**3.85**	—
Phenol, 2,4-dichloro-	5.96	—	Anthracene	6.87	—
Benzene, 1,2,4-trichloro-	6.67	—	Carbazole	8.99	—
Naphthalene	4.81	—	Di-n-butyl phthalate	7.05	—
Naphthalene-d8	**3.84**	—	Fluoranthene	7.25	—
p-Chloroaniline	5.55	—	Pyrene	6.05	—
1,3-Butadiene, 1,1,2,3,4,4-hexachloro-	7.15	—	p-Terphenyl-d14 (surrogate)	6.25	—
Phenol, 4-chloro-3-methyl-	7.32	—	Benzyl butyl phthalate	5.92	—
Naphthalene, 2-methyl	5.92	—	Bis(2-ethylhexyl)adipate	6.32	—
Naphthalene, 1-methyl-	6.15	—	Benz[a]anthracene	7.37	—
Hexachlorocyclopentadiene	—	0.9985	Chrysene	6.90	—
Phenol, 2,4,5-trichloro-	12.06	—	**Chrysene-d12**	**4.81**	—
Phenol, 2,4,6-trichloro-	12.35	—	bis(2-ethylhexyl)phthalate	6.27	—
2-fluorobiphenyl (surrogate)	7.30	—	di-n-octylphthalate	6.56	—
Naphthalene, 2-chloro-	7.68	—	Benzo[b]fluoranthene	6.55	—
2-Nitroaniline	17.72	—	Benzo[k]fluoranthene	9.18	—
1,4-Dinitrobenzene	19.53	—	benzo[a]pyrene	7.40	—
Dimethyl phthalate	7.46	—	**Perylene-d12**	**8.17**	—
Benzene, 1,3-dinitro-	18.89	—	Indeno[1,2,3-cd]pyrene	8.23	—
2,6-dinitrotoluene	13.59	—	dibenzo[a,h]anthracene	7.15	—
			Benzo[g,h,i]perylene	6.50	—

Boldface indicates Internal Standards

Table 8 (Part 1). EPA Method 8270D minimum relative response factors and those produced by the ISQ 7000 single quadrupole system.

Compound	EPA 8270D Minimum Response	Thermo Minimum		Thermo Minimum	
		Splitless	Splitless Helium Saver	Split (10:1)	Split Helium Saver
Phenol	0.8	1.990	2.895	2.603	2.767
Bis(2-chloroethyl) ether	0.7	1.499	2.225	1.929	2.134
Phenol, 2-chloro-	0.8	1.516	1.884	1.882	1.869
Phenol, 2-methyl-	0.7	1.412	1.802	1.719	1.771
Phenol, 3&4-methyl-	0.6	1.495	1.933	1.767	1.897
N-Nitroso-di-n-propylamine	0.5	1.110	1.886	1.254	1.579
Ethane, hexachloro-	0.3	0.530	0.439	0.716	0.690
Benzene, nitro-	0.2	0.316	0.469	0.404	0.471
Isophorone	0.4	0.708	0.989	0.869	0.995
Phenol, 2-nitro-	0.1	0.160	0.170	0.152	0.157
Phenol, 2,4-dimethyl-	0.2	0.389	0.453	0.430	0.465
Bis(2-chloroethoxy)methane	0.3	0.432	0.589	0.530	0.586
Phenol, 2,4-dichloro-	0.2	0.282	0.269	0.313	0.288
Naphthalene	0.7	1.085	1.247	1.176	1.260
p-Chloroaniline	0.01	0.464	0.493	0.497	0.546
1,3-Butadiene, 1,1,2,3,4,4-hexachloro-	0.01	0.112	0.118	0.175	0.116
Phenol, 4-chloro-3-methyl-	0.2	0.342	0.394	0.382	0.418
Naphthalene, 2-methyl	0.4	0.785	0.730	0.726	0.724
Hexachlorocyclopentadiene	0.05	0.236	0.128	0.213	0.044
Phenol, 2,4,6-trichloro-	0.2	0.345	0.322	0.372	0.298
Phenol, 2,4,5-trichloro-	0.2	0.324	0.286	0.368	0.300
Naphthalene, 2-chloro-	0.8	1.232	1.388	1.314	1.349
2-Nitroaniline	0.01	0.335	0.406	0.339	0.455
Dimethyl phthalate	0.01	1.361	1.511	1.442	1.482
2,6-dinitrotoluene	0.2	0.229	0.259	0.258	0.242
Acenaphthylene	0.9	1.899	2.216	2.063	2.165
3-Nitroaniline	0.01	0.298	0.336	0.428	0.541
2,4-dinitrophenol	0.01	0.055	0.042	0.045	0.025
Acenaphthene	0.9	1.312	1.574	1.383	1.417

Table 8 (Part 2). EPA Method 8270D minimum relative response factors and those produced by the ISQ 7000 single quadrupole system.

Compound	EPA 8270D Minimum Response	Thermo Minimum		Thermo Minimum	
		Splitless	Splitless Helium Saver	Split (10:1)	Split Helium Saver
2,4-dinitrotoluene	0.2	0.304	0.327	0.316	0.330
Dibenzofuran	0.8	1.840	1.907	1.811	1.863
Phenol, 4-nitro-	0.01	0.167	0.042	0.124	0.055
Diethyl Phthalate	0.01	1.335	1.676	1.508	1.518
4-chlorophenylphenylether	0.4	0.740	0.609	0.692	0.621
4-nitroaniline	0.01	0.306	0.360	0.315	0.296
Fluorene	0.9	1.434	1.647	1.471	1.470
4,6-Dinitro-2-methylphenol	0.01	0.079	0.057	0.063	0.047
Diphenylamine	0.01	0.683	0.897	0.750	0.799
4-bromophenylphenylether	0.1	0.477	0.332	0.241	0.206
Hexachlorobenzene	0.1	0.324	0.256	0.283	0.267
Phenol, pentachloro-	0.05	0.131	0.077	0.064	0.049
Phenanthrene	0.7	1.125	1.335	1.289	1.275
Anthracene	0.7	1.270	1.138	1.272	1.347
Carbazole	0.01	1.070	1.407	1.006	1.156
Di-n-butyl phthalate	0.01	1.314	1.856	1.517	1.626
Fluoranthene	0.6	1.263	1.123	1.268	1.234
Pyrene	0.6	1.072	1.326	1.296	1.487
Benzyl butyl phthalate	0.01	0.496	0.906	0.677	0.847
Bis(2-ethylhexyl)phthalate	0.01	0.741	1.225	0.941	1.144
Chrysene	0.7	1.025	1.110	1.164	1.102
Benz[a]anthracene	0.8	1.068	1.228	1.171	1.124
Di-n-octylphthalate	0.01	1.465	2.673	2.084	2.413
Benzo[b]fluoranthene	0.7	1.364	1.417	1.592	1.432
Benzo[k]fluoranthene	0.7	1.292	1.185	1.586	1.396
Benzo[a]pyrene	0.7	1.353	1.420	1.500	1.414
Indeno[1,2,3-cd]pyrene	0.5	1.600	1.794	1.727	1.866
Dibenzo[a,h]anthracene	0.4	1.393	1.645	1.472	1.617
Benzo[g,h,i]perylene	0.5	1.302	1.560	1.406	1.636

Table 9 (Part 1). Retention times (RT) for the four methods.

Compound	Splitless RT (min)	Split (10:1) RT (min)	Split (10:1) Helium Saver RT (min)	Splitless Helium Saver RT (min)
Pyridine	3.66	3.71	3.66	3.29
N-Nitrosodimethylamine	3.71	3.74	3.68	3.33
2-fluorophenol (surrogate)	5.08	5.07	5.04	4.98
Phenol-d6 (surrogate)	5.96	5.93	5.91	5.92
Phenol	5.97	5.94	5.93	5.92
Aniline	5.98	5.95	5.94	5.92
Bis(2-chloroethyl) ether	6.04	6.00	5.98	5.97
Phenol, 2-chloro-	6.08	6.05	6.03	6.02
Benzene, 1,3-dichloro-	6.20	6.17	6.15	6.14
1,4-Dichlorobenzene-d4	6.23	6.20	6.18	6.17
Benzene, 1,4-dichloro-	6.25	6.21	6.20	6.19
Benzyl alcohol	6.39	6.36	6.34	6.34
Benzene, 1,2-dichloro-	6.42	6.38	6.37	6.36
Phenol, 2-methyl-	6.49	6.46	6.45	6.46
Bis(2-chloroisopropyl)ether	6.51	6.48	6.47	6.46
Phenol, 3&4-methyl-	6.63	6.60	6.59	6.59
N-Nitroso-di-n-propylamine	6.67	6.62	6.60	6.61
Ethane, hexachloro-	6.68	6.65	6.64	6.63
Nitrobenzene-D5 (surrogate)	6.77	6.73	6.72	6.72
Benzene, nitro-	6.79	6.75	6.74	6.74
Isophorone	7.00	6.96	6.94	6.95
Phenol, 2-nitro-	7.06	7.03	7.02	7.02
Phenol, 2,4-dimethyl-	7.09	7.06	7.05	7.06
Bis(2-chloroethoxy)methane	7.18	7.14	7.13	7.13
Phenol, 2,4-dichloro-	7.27	7.23	7.22	7.23
Benzene, 1,2,4-trichloro-	7.33	7.30	7.29	7.29
Naphthalene-d8	7.37	7.34	7.33	7.33
Naphthalene	7.39	7.36	7.35	7.35
p-Chloroaniline	7.46	7.43	7.42	7.42
1,3-Butadiene, 1,1,2,3,4,4-hexachloro-	7.53	7.50	7.49	7.49
Phenol, 4-chloro-3-methyl-	7.87	7.84	7.83	7.84
Naphthalene, 2-methyl	7.99	7.95	7.94	7.95
Naphthalene, 1-methyl-	8.08	8.04	8.03	8.04
Hexachlorocyclopentadiene	8.17	8.13	8.12	8.13
Phenol, 2,4,6-trichloro-	8.25	8.21	8.21	8.22
Phenol, 2,4,5-trichloro-	8.28	8.25	8.24	8.25
2-fluorobiphenyl (surrogate)	8.31	8.27	8.26	8.27
Naphthalene, 2-chloro-	8.41	8.37	8.36	8.37
2-Nitroaniline	8.53	8.49	8.49	8.50
1,4-Dinitrobenzene	8.63	8.59	8.58	8.60
Dimethyl phthalate	8.70	8.66	8.64	8.66
Benzene, 1,3-dinitro-	8.74	8.69	8.68	8.70
2,6-dinitrotoluene	8.77	8.72	8.71	8.73
Acenaphthylene	8.77	8.73	8.72	8.73

Table 9 (Part 2). Retention times (RT) for the four methods.

Compound	Splitless RT (min)	Split (10:1) RT (min)	Split (10:1) Helium Saver RT (min)	Splitless Helium Saver RT (min)
1,2-Dinitrobenzene	8.84	8.80	8.78	8.80
Acenaphthene-d10	8.89	8.85	8.84	8.85
3-Nitroaniline	8.90	8.85	8.84	8.86
Acenaphthene	8.92	8.88	8.87	8.89
2,4-dinitrophenol	8.98	8.93	8.92	8.94
Phenol, 4-nitro-	9.02	8.98	8.97	8.99
Dibenzofuran	9.05	9.01	9.00	9.02
2,4-dinitrotoluene	9.10	9.06	9.04	9.06
Phenol, 2,3,5,6-tetrachloro-	9.15	9.11	9.10	9.12
Phenol, 2,3,4,6-tetrachloro-	9.19	9.15	9.14	9.15
Diethyl Phthalate	9.28	9.23	9.22	9.23
4-chlorophenylphenylether	9.33	9.28	9.28	9.29
Fluorene	9.34	9.30	9.29	9.31
4-nitroaniline	9.43	9.38	9.36	9.38
Diphenylamine	9.45	9.40	9.38	9.40
4,6-Dinitro-2-methylphenol	9.45	9.40	9.39	9.41
Azobenzene	9.46	9.42	9.41	9.42
2,4,6-tribromophenol (surrogate)	9.57	9.52	9.51	9.53
4-bromophenylphenylether	9.73	9.69	9.68	9.69
Hexachlorobenzene	9.87	9.82	9.82	9.83
Phenol, pentachloro-	10.02	9.97	9.97	9.98
Phenanthrene-D10-	10.12	10.08	10.07	10.08
Phenanthrene	10.15	10.10	10.09	10.10
Anthracene	10.19	10.14	10.13	10.14
Carbazole	10.32	10.27	10.27	10.28
Di-n-butyl phthalate	10.60	10.55	10.55	10.56
Fluoranthene	11.15	11.10	11.09	11.10
Pyrene	11.35	11.29	11.29	11.30
p-Terphenyl-d14 (surrogate)	11.46	11.40	11.40	11.41
Benzyl butyl phthalate	11.93	11.87	11.87	11.88
Bis(2-ethylhexyl)adipate	11.95	11.89	11.89	11.90
Bis(2-ethylhexyl)phthalate	12.54	12.48	12.47	12.49
Benz[a]anthracene	12.55	12.48	12.48	12.50
Chrysene-d12	12.57	12.50	12.49	12.52
Chrysene	12.61	12.54	12.53	12.55
Di-n-octylphthalate	13.28	13.21	13.20	13.22
Benzo[b]fluoranthene	13.91	13.83	13.82	13.85
Benzo[k]fluoranthene	13.91	13.83	13.85	13.88
Benzo[a]pyrene	14.35	14.26	14.25	14.29
Perylene-d12	14.40	14.32	14.31	14.34
Indeno[1,2,3-cd]pyrene	15.96	15.83	15.81	15.88
Dibenzo[a,h]anthracene	15.96	15.84	15.83	15.88
Benzo[g,h,i]perylene	16.36	16.24	16.23	16.29

Conclusion

The Thermo Scientific ISQ 7000 single quadrupole GC-MS system with the ExtractaBrite ion source and the innovative NeverVent technology is the perfect solution to perform the EPA 8270D Method.

Thanks to the extended dynamic range detection system, the ISQ 7000 GC-MS system allows you to cover a 0.2–200 ppm range with the same column and liner. Seventy-six compounds were reported, and each fulfilled the EPA 8270D requirements in terms of minimum response factors and linearity.

Chromeleon CDS software, with the Environmental Reporting package, offers unparallel flexibility, scalability, and compliance. It provides compliance with EPA 8270D Method requirements offering a full complement of standard reports including DFTPP Tune Check report, Breakdown report, Internal Standard Summary report, Tentatively Identified Compounds report, various quality control reports for check standards, laboratory control samples, matrix spikes, surrogate recoveries, and more.

The Thermo Scientific Instant Connect Helium Saver Module is a unique tool that can be used to reduce the cost per analysis, without compromising the analytical results. The Helium Saver Module makes laboratories more efficient and environmentally friendly, saving 90% of helium during each run.

The ExtractaBrite ion source design, as integrated in the ISQ 7000 GC-MS system, keeps your system cleaner, longer. With heat throughout the ion optics and the patented RF lens, the ISQ 7000 GC-MS system has been proven to be capable to analyze more dirty samples per day, with maximum uptime. Even better, when the instrument finally requires cleaning, the column needs to be replaced or trimmed, or maintenance is required at the injector side, the NeverVent technology offers the user the possibility to operate without venting the MS system, in a very fast and simple way. Why break your workflow when you can have unstoppable productivity?

References

1. 1. U.S. EPA. July 2014. Method 8270D (SW-846): Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS), Revision 5. https://www.epa.gov/sites/production/files/2015-12/documents/8270d.pdf, Accessed February 11th, 2018.
2. 2. James W. Eichelberger, Lawrence E. Harris, and William L. Budde, Reference Compound to Calibrate Ion Abundance Measurements in Gas Chromatography-Mass Spectrometry Systems, *Anal. Chem.,* **1975,** *47* (7), 995–1000.
3. 3. Joseph R. Donnelly, Ion Abundance Criteria for Gas Chromatographic/Mass Spectrometric Environmental Analysis, *J. Assoc. Off. Anal. Chem.,* **1988,** *71* (2), 434–439.
4. 4. Yves Tondeur, Warren J. Niederhutl, Joseph E. Campana, Ronald K. Mitchum, G. Wayne Sovocool, Joseph R. Donnelly, Ion Chemistry of a Gas Chromatographic/Mass Spectrometric Ion Abundance Calibrant, *Biomedical and Environmental Mass Spectrometry Journal of Mass Spectrometry,* **1988,** *15* (8), 429–439.

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