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Mass spectrometry

Optimized analysis of semi-volatile organic compounds (SVOC) in environmental samples in compliance with U.S. EPA Method 8270 utilizing GC-MS

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Introduction

Semi-volatile organic compounds (SVOCs) are ubiquitous in our environment and raise concerns regarding health for humans and wildlife. They are released in various everyday processes, including manufacturing and agriculture. Thus, the analysis of SVOCs in extracts of many matrices, such as solid waste, soil, air, and water, is commonly performed by environmental testing laboratories. Regulations are in place worldwide to monitor the presence of SVOCs in environmental samples to protect the environment and human exposure. The United States Environmental Protection Agency (EPA) released the first SVOC method by gas chromatography/mass spectrometry (Method 8270) at the end of 1980.[1] EPA Method 8270D and derivative methods are now used in environmental labs globally to monitor SVOCs.

Analytical testing laboratories that follow EPA 8270 methodology for the analysis of SVOCs must comply with several requirements before starting the analysis. These include the ability for the MS instrument to pass decafluorotriphenyl phosphine (DFTPP) tuning criteria, relative retention time (RRT) of target analytes criteria, linearity of calibration curve criteria, and suggested minimum response factors. This application note shows how the Thermo Scientific™ ISQ™ 7610 single quadrupole GC-MS system can meet EPA Method 8270D requirements.

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One of the unique features of the ISQ 7610 single quadrupole MS systems is NeverVent™ technology, which allows laboratories to minimize instrument downtime with fast removal of the ion source for maintenance and to change the analytical column without venting the system. The ISQ 7610 MS also introduces a new detector, the XLXR™ detector system, with a wider linear dynamic range and longer lifetime compared to previous generation detectors.

Experimental

For these experiments, an ISQ 7610 single quadrupole MS system was equipped with the Thermo Scientific™ ExtractaBrite™ ion source and coupled to a Thermo Scientific™ TRACE™ 1610 GC. The Thermo Scientific™ iConnect™ Split-Splitless (SSL) Injector module (P/N 19070010) was used as the GC inlet. Thermo Scientific™ Chromeleon™ Chromatography Data System (CDS) software was used to acquire, process, and report data. The instrument method parameters are shown in Table 1.

Tuning

The default ISQ 7610 "EI Full Tune" is optimized for maximum sensitivity, allowing the user to achieve low ppb levels of analyte detection. The concentration of SVOC for most analyses using the EPA Method 8270 is in the ppm range; hence, the default EI Full Tune was modified using the "Advanced AutoTune" option to lower the overall sensitivity of the MS system. The tuning emission current was reduced to 10 µA and the detector sensitivity was adjusted to deliver 5 million counts for m/z 219 for perfluorotributylamine (PFTBA). Adjusting the detector sensitivity changes the gain on the detector (and hence the electron multiplier voltage) to achieve the required sensitivity. An added benefit of this AutoTune procedure is that it extends the life of the electron multiplier because the gain, in most cases, is lower than the default EI Full Tune option.

EPA Method 8270 uses a dynamic tuning procedure for checking the MS tuning performance. This is done by using decafluorotriphenyl phosphine (DFTPP) and checking various m/z ions and their relative intensities. Figure 1 shows the total ion chromatogram (TIC) of the GC-MS tuning mixture (Restek Cat. No. 31615) containing DFTPP, and Figure 2 shows the mass spectrum for the DFTPP. Chromeleon CDS software has a dedicated reporting package for environmental laboratories and automatically reports tune evaluation performance with a Pass/Fail indicator. Table 2 shows the DFTPP ion abundance spectrum check criteria according to EPA Method 8270D and passing evaluation result for the injection.

The other components of the GC-MS tuning mix include pentachlorophenol, benzidine, and 4,4'-DDT, which are primarily used to check for inlet inertness and column performance.

Peak tailing for pentachlorophenol and benzidine is an indication of basic and acidic active sites in the inlet and/or column, possibly requiring inlet maintenance including trimming the front end of the column. EPA Method 8270D requires that the tailing factor for these two compounds be not more than 2. The injection of the GC-MS tuning mix gave tailing factors of 1.22 and 1.18 for pentachlorophenol and benzidine, respectively. 4,4'-DDT is used to assess inlet performance with the criteria that the breakdown products DDD and DDE be no more than 20%. No degradation of DDT was observed, thus indicating pristine inlet conditions.

Table 1. Autosampler, GC, and MS instrument method parameters

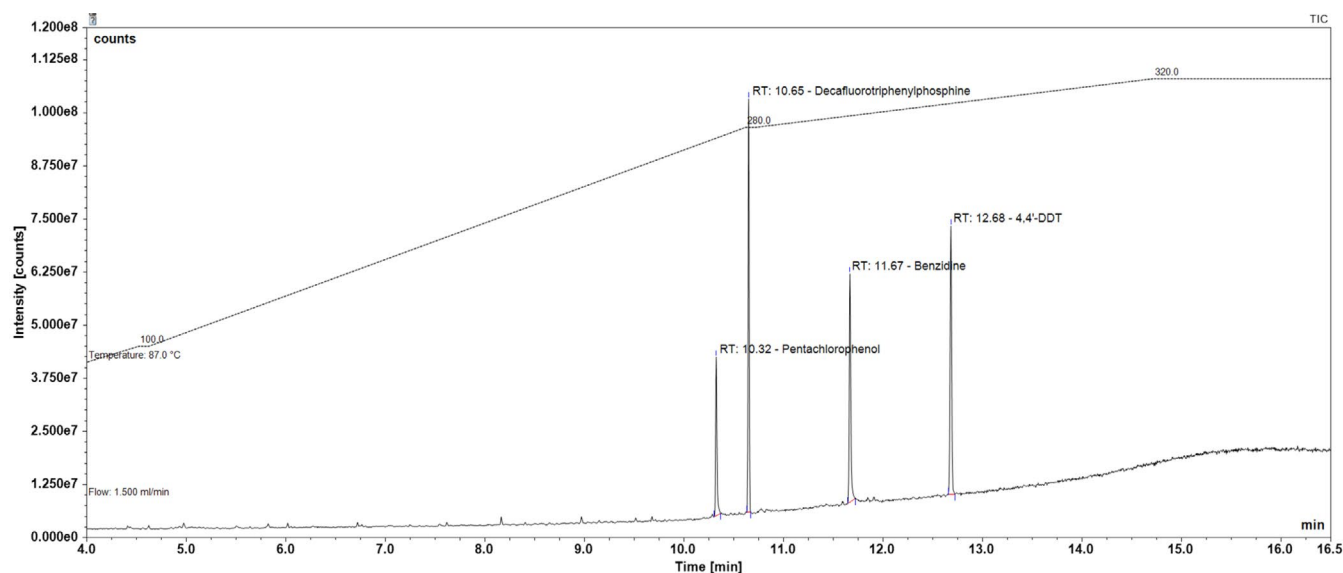| Parameter	Value
AS 1610 Autosampler	
Syringe	10 µL, 25 gauge, 50 mm length, cone tip (P/N 36500525)
Injection volume	1 µL
Pre-injection solvent and cycle	Acetone, 2 cycles
Sample rinses	3
Post-injection solvent and cycles	Dichloromethane, 2 cycles
Injector draw speed	Slow
Fill Strokes	3
Air volume	1 µL
Pre-injection delay time	0.0 min
Post-injection delay time	3.0 min
TRACE 1610 GC system	
Column	Thermo Scientific™ TraceGOLD™ TG-5ms, 30 m × 0.25 mm × 0.5 µm (P/N 26098-2230)
Liner	Splitless liner single taper with wool, 4 mm i.d., 78.5 mm length (P/N 453A1925-UI)
SSL mode	Split
Inlet temperature	300 °C
Split flow	15 mL/min (split ratio = 10)
Septum purge flow	Constant flow of 5.0 mL/min
Carrier flow	Constant He flow of 1.5 mL/min
Oven program	37 °C (2.0 min) 25 °C/min to 100 °C (0.1 min) 30 °C/min to 280 °C (0.1 min) 10 °C/min to 320 °C (5.0 min) Total run time = 19.9 min
ISQ 7610 MS system	
MS transfer line temperature	300 °C
Ion source temperature	320 °C
Source type	ExtractaBrite with EI ion volume
Ionization mode, Electron energy	EI, 70 eV
Emission current	10 µA
Scan range	35–500 Da
Dwell time	0.1 s
Detector gain	Last saved AutoTune

Figure 1. TIC of the GC-MS tuning mixture
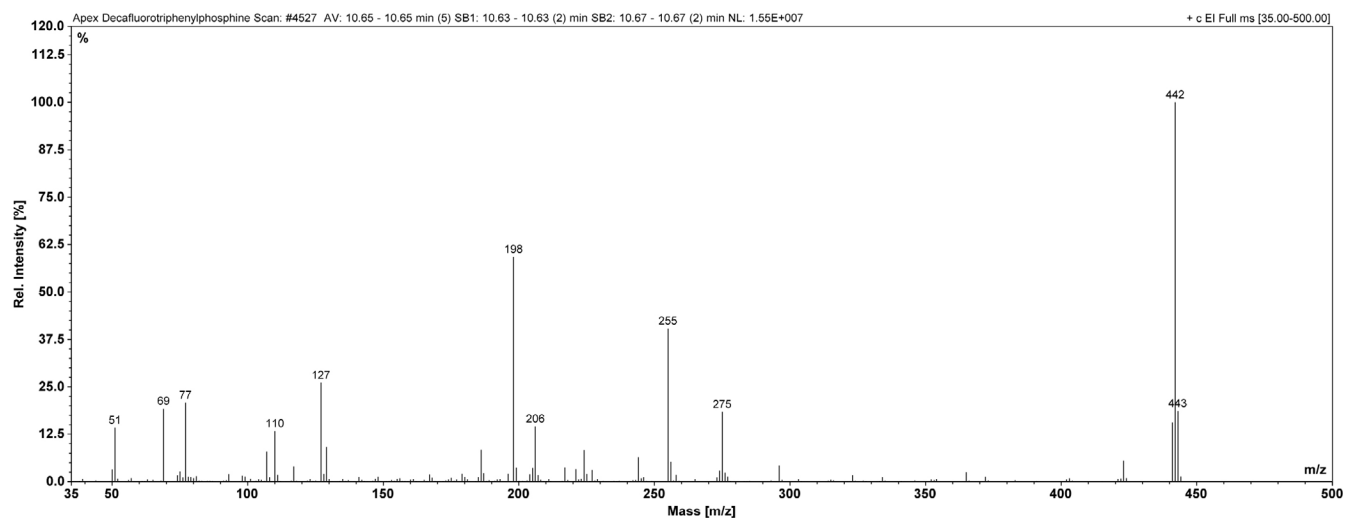
Figure 2. Mass spectrum of the DFTPP peak

Table 2. DFTPP ion abundance spectrum check criteria results according to EPA Method 8270D

Evaluated mass, m/z	Ion abundance criteria	Measured % relative abundance	Criteria evaluation result, Pass/Fail
51	10–80% of base peak	14.2	Pass
68	<2% of m/z 69	1.7	Pass
70	<2% of m/z 69	0.4	Pass
127	10–80% of base peak	26.1	Pass
197	<2% of m/z 198	0.0	Pass
198	Base peak, or >50% of m/z 442	59.3	Pass
199	5–9% of m/z 198	6.2	Pass
275	10–60% of base peak	18.4	Pass
365	>1% of m/z 198	4.2	Pass
441	Present but <24% of m/z 442	15.6	Pass
442	Base Peak, or >50% of m/z 198	Base peak	Pass
443	15–42% of m/z 442	18.6	Pass

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. Target compound concentration ranged from 1 to 400 ppm with internal and surrogate standards spiked at a constant concentration level of 4 ppm over 11 calibration levels. A wider target concentration range, compared to that presented in a previous application note (AN10522), was investigated, and this was made possible due to the extended dynamic range of the new XLRR detector system in the ISQ 7610 MS. A chromatogram for the 40 ppm calibration standard is shown in Figure 3 with the investigated calibration levels outlined in Table 3.

Table 4 provides retention time and quantification ion information for all analytes, internal standards, and surrogates standards. Identities of all compounds were confirmed by comparison with the NIST library match with a total of 76 target analytes, 6 surrogates, and 6 internal standards. The associated internal standard for each analyte is provided in the table and is in accordance with EPA 8270D methodology.

Table 3. Analyte, internal standard, and surrogate concentration levels used for this study

Level	Target analyte concentration ppm	Internal standard concentration, ppm	Surrogate concentration, ppm
Cal 1	1	4	4
Cal 2	2	4	4
Cal 3	5	4	4
Cal 4	10	4	4
Cal 5	20	4	4
Cal 6	40	4	4
Cal 7	50	4	4
Cal 8	75	4	4
Cal 9	100	4	4
Cal 10	200	4	4
Cal 11	400	4	4
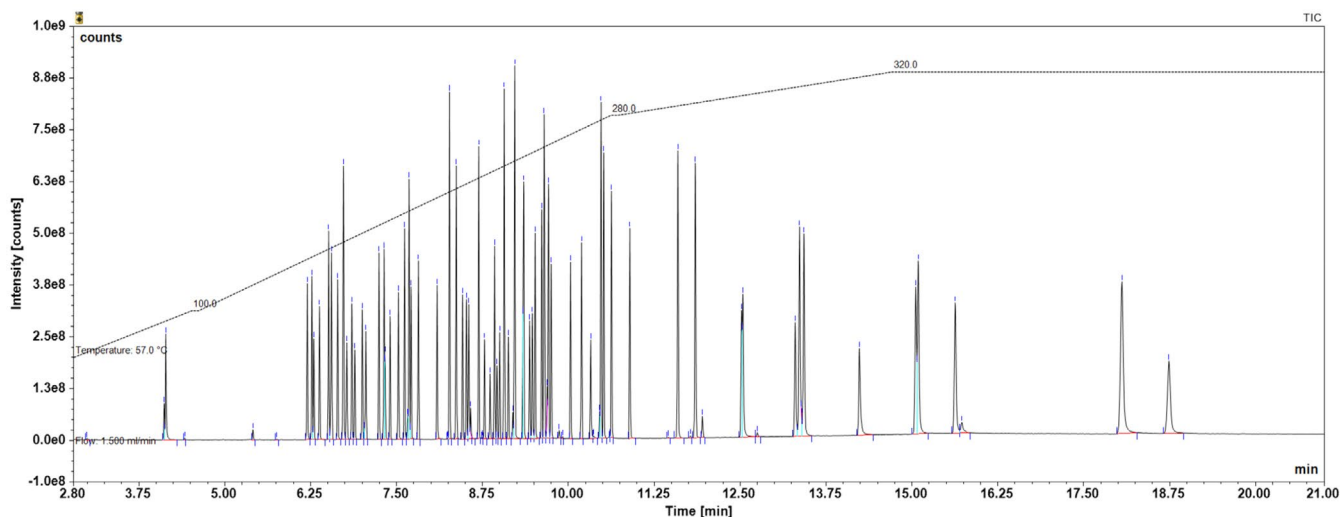
Figure 3. TIC of the 40 ppm semivolatile mix spiked with internal standards and surrogates

Table 4 (part 1). Compound, retention time, internal standard used, and quantitative ion information

Compound name	Retention time, min	Internal standard used	Quantitation ion, m/z
N-Nitrosodimethylamine	4.12	1,4-Dichlorobenzene-d$_4$ (ISTD)	74
Pyridine	4.15	1,4-Dichlorobenzene-d$_4$ (ISTD)	79
2-Fluorophenol, (Surr)	5.41	1,4-Dichlorobenzene-d$_4$ (ISTD)	112
Phenol-d$_6$ (Surr)	6.19	1,4-Dichlorobenzene-d$_4$ (ISTD)	99
Phenol	6.20	1,4-Dichlorobenzene-d$_4$ (ISTD)	94
Aniline	6.27	1,4-Dichlorobenzene-d$_4$ (ISTD)	93
Bis(2-chloroethyl) ether	6.29	1,4-Dichlorobenzene-d$_4$ (ISTD)	93
2-Chlorophenol	6.38	1,4-Dichlorobenzene-d$_4$ (ISTD)	128

Table 4 (part 2). Compound, retention time, internal standard used, and quantitative ion information

Compound name	Retention time, min	Internal standard used	Quantitation ion, m/z
1,3-Dichlorobenzene	6.51	1,4-Dichlorobenzene-d$_4$ (ISTD)	146
1,4-Dichlorobenzene-d$_4$ (ISTD)	6.54	n.a.	152
1,4-Dichlorobenzene	6.55	1,4-Dichlorobenzene-d$_4$ (ISTD)	146
Benzyl alcohol	6.64	1,4-Dichlorobenzene-d$_4$ (ISTD)	108
1,2-Dichlorobenzene	6.72	1,4-Dichlorobenzene-d$_4$ (ISTD)	146
Bis(2-chloroisopropyl) ether	6.77	1,4-Dichlorobenzene-d$_4$ (ISTD)	121
2-Methylphenol	6.85	1,4-Dichlorobenzene-d$_4$ (ISTD)	107
3-Methylphenol/4-Methylphenol	6.85	1,4-Dichlorobenzene-d$_4$ (ISTD)	107
N-Nitroso-di-n-propylamine	6.89	1,4-Dichlorobenzene-d$_4$ (ISTD)	70
Hexachloroethane	7.00	1,4-Dichlorobenzene-d$_4$ (ISTD)	117
Nitrobenzene-d$_5$ (Surr)	7.03	Naphthalene-d$_8$ (ISTD)	82
Nitrobenzene	7.05	Naphthalene-d$_8$ (ISTD)	77
Isophorone	7.24	Naphthalene-d$_8$ (ISTD)	82
2,4-Dimethylphenol	7.32	Naphthalene-d$_8$ (ISTD)	107
2-Nitrophenol	7.33	Naphthalene-d$_8$ (ISTD)	139
Bis(2-chloroethoxy) methane	7.40	Naphthalene-d$_8$ (ISTD)	93
2,4-Dichlorophenol	7.52	Naphthalene-d$_8$ (ISTD)	162
1,2,4-Trichlorobenzene	7.61	Naphthalene-d$_8$ (ISTD)	180
Naphthalene-d$_8$ (ISTD)	7.66	n.a.	136
Naphthalene	7.68	Naphthalene-d$_8$ (ISTD)	128
4-Chloroaniline	7.71	Naphthalene-d$_8$ (ISTD)	127
Hexachlorobutadiene	7.81	Naphthalene-d$_8$ (ISTD)	225
4-Chloro-3-methylphenol	8.09	Naphthalene-d$_8$ (ISTD)	107
2-Methylnaphthalene	8.27	Naphthalene-d$_8$ (ISTD)	142
1-Methylnaphthalene	8.37	Naphthalene-d$_8$ (ISTD)	142
Hexachlorocyclopentadiene	8.46	Acenaphthene-d$_{10}$ (ISTD)	237
2,4,6-Trichlorophenol	8.51	Acenaphthene-d$_{10}$ (ISTD)	196
2,4,5-Trichlorophenol	8.55	Acenaphthene-d$_{10}$ (ISTD)	196
2-Fluorobiphenyl (Surr)	8.57	Acenaphthene-d$_{10}$ (ISTD)	172
2-Chloronaphthalene	8.69	Acenaphthene-d$_{10}$ (ISTD)	162
2-Nitroaniline	8.78	Acenaphthene-d$_{10}$ (ISTD)	65
1,4-Dinitrobenzene	8.86	Acenaphthene-d$_{10}$ (ISTD)	168
Dimethyl phthalate	8.92	Acenaphthene-d$_{10}$ (ISTD)	163
1,3-Dinitrobenzene	8.96	Acenaphthene-d$_{10}$ (ISTD)	168
2,6-Dinitrotoluene	9.00	Acenaphthene-d$_{10}$ (ISTD)	165
Acenaphthylene	9.06	Acenaphthene-d$_{10}$ (ISTD)	152
1,2-Dinitrobenzene	9.07	Acenaphthene-d$_{10}$ (ISTD)	168
3-Nitroaniline	9.12	Acenaphthene-d$_{10}$ (ISTD)	138
Acenaphthene-d$_{10}$ (ISTD)	9.19	n.a.	164
2,4-Dinitrophenol	9.21	Acenaphthene-d$_{10}$ (ISTD)	184
4-Nitrophenol	9.21	Acenaphthene-d$_{10}$ (ISTD)	139
Acenaphthene	9.22	Acenaphthene-d$_{10}$ (ISTD)	153

Table 4 (part 3). Compound, retention time, internal standard used, and quantitative ion information

Compound name	Retention time, min	Internal standard used	Quantitation ion, *m/z*
2,4-Dinitrotoluene	9.33	Acenaphthene-d_{10} (ISTD)	165
Dibenzofuran	9.35	Acenaphthene-d_{10} (ISTD)	168
2,3,5,6-Tetrachlorophenol	9.43	Acenaphthene-d_{10} (ISTD)	232
2,3,4,6-Tetrachlorophenol	9.47	Acenaphthene-d_{10} (ISTD)	232
Diethylphthalate	9.51	Acenaphthene-d_{10} (ISTD)	149
4-Chlorophenyl phenyl ether	9.61	Acenaphthene-d_{10} (ISTD)	204
Fluorene	9.65	Acenaphthene-d_{10} (ISTD)	166
4-Nitroaniline	9.65	Acenaphthene-d_{10} (ISTD)	138
2-Methyl-4,6-dinitrophenol	9.69	Phenanthrene-d_{10} (ISTD)	198
Diphenylamine	9.71	Phenanthrene-d_{10} (ISTD)	169
Azobenzene	9.75	Phenanthrene-d_{10} (ISTD)	182
2,4,6-Tribromophenol (Surr)	9.86	Acenaphthene-d_{10} (ISTD)	332
4-Bromophenyl phenyl ether	10.03	Phenanthrene-d_{10} (ISTD)	248
Hexachlorobenzene	10.19	Phenanthrene-d_{10} (ISTD)	284
Pentachlorophenol	10.32	Phenanthrene-d_{10} (ISTD)	266
Phenanthrene-d_{10} (ISTD)	10.45	n.a.	188
Phenanthrene	10.47	Phenanthrene-d_{10} (ISTD)	178
Anthracene	10.51	Phenanthrene-d_{10} (ISTD)	178
Carbazole	10.63	Phenanthrene-d_{10} (ISTD)	167
Dibutyl phthalate	10.89	Phenanthrene-d_{10} (ISTD)	149
Fluoranthene	11.59	Phenanthrene-d_{10} (ISTD)	202
Pyrene	11.85	Chrysene-d_{12} (ISTD)	202
p-Terphenyl-d14 (Surr)	11.95	Chrysene-d_{12} (ISTD)	244
Butyl benzyl phthalate	12.52	Chrysene-d_{12} (ISTD)	149
Bis(2-ethylhexyl) adipate	12.54	Chrysene-d12 (ISTD)	129
Bis(2-ethylhexyl) phthalate	13.30	Perylene-d12 (ISTD)	149
Benzo[*a*]anthracene	13.36	Chrysene-d_{12} (ISTD)	228
Chrysene-d_{12} (ISTD)	13.39	n.a.	240
Chrysene	13.43	Chrysene-d_{12} (ISTD)	228
Dioctyl phthalate	14.23	Perylene-d_{12} (ISTD)	149
Benzo[*b*]fluoranthene	15.05	Perylene-d_{12} (ISTD)	252
Benzo[*k*]fluoranthene	15.09	Perylene-d_{12} (ISTD)	252
Benzo[*a*]pyrene	15.63	Perylene-d_{12} (ISTD)	252
Perylene-d_{12} (ISTD)	15.73	n.a.	264
Indeno[1,2,3-*cd*]pyrene	18.05	Perylene-d_{12} (ISTD)	276
Dibenzo[*a,h*]anthracene	18.05	Perylene-d_{12} (ISTD)	278
Benzo[*g,h,i*]perylene	18.73	Perylene-d_{12} (ISTD)	276

Results and discussion

Relative retention time results

EPA Method 8270D calls for calculating the relative retention time (RRT) for each target analyte in each calibration standard. Relative retention time is defined by the formula below.

$$RRT = \frac{\text{Retention time of the analyte}}{\text{Retention time of the internal standard}}$$

The RRT was calculated for each of the target analytes in all calibration levels, and the results for the difference between the maximum RRT and minimum RRT for each analyte are given in Table 5. EPA Method 8270D specifies that this difference be no more than 0.06 RRT units. The results obtained in these experiments show the maximum difference obtained over all analytes investigated is 0.0069 RRT units; easily passing this criterion.

Table 5. Maximum relative retention time difference for all the target analytes

Analyte	Relative retention time	Analyte	Relative retention time
N-Nitrosodimethylamine	0.0035	1,2-Dinitrobenzene	0.0021
Pyridine	0.0069	3-Nitroaniline	0.0021
Phenol	0.0027	4-Nitrophenol	0.0021
Aniline	0.0014	Acenaphthene	0.0004
Bis(2-chloroethyl) ether	0.0014	2,4-Dinitrophenol	0.0013
2-Chlorophenol	0.0013	2,4-Dinitrotoluene	0.0013
1,3-Dichlorobenzene	0.0005	Dibenzofuran	0.0004
1,4-Dichlorobenzene	0.0005	2,3,5,6-Tetrachlorophenol	0.0004
Benzyl alcohol	0.0024	2,3,4,6-Tetrachlorophenol	0.0002
1,2-Dichlorobenzene	0.0005	Diethylphthalate	0.0011
Bis(2-chloroisopropyl) ether	0.0008	4-Chlorophenyl phenyl ether	0.0002
2-Methylphenol	0.0026	Fluorene	0.0005
3-Methylphenol/4-Methylphenol	0.0026	4-Nitroaniline	0.0026
N-Nitroso-di-n-propylamine	0.0037	2-Methyl-4,6-dinitrophenol	0.0020
Hexachloroethane	0.0005	Diphenylamine	0.0014
Nitrobenzene	0.0012	Azobenzene	0.0007
Isophorone	0.0025	4-Bromophenyl phenyl ether	0.0004
2,4-Dimethylphenol	0.0014	Hexachlorobenzene	0.0005
2-Nitrophenol	0.0005	Pentachlorophenol	0.0003
Bis(2-chloroethoxy) methane	0.0009	Phenanthrene	0.0007
2,4-Dichlorophenol	0.0011	Anthracene	0.0007
1,2,4-Trichlorobenzene	0.0002	Carbazole	0.0007
Naphthalene	0.0005	Dibutyl phthalate	0.0002
4-Chloroaniline	0.0007	Fluoranthene	0.0008
Hexachlorobutadiene	0.0005	Pyrene	0.0007
4-Chloro-3-methylphenol	0.0003	Butyl benzyl phthalate	0.0003
2-Methylnaphthalene	0.0003	Bis(2-ethylhexyl) adipate	0.0004
1-Methylnaphthalene	0.0003	Bis(2-ethylhexyl) phthalate	0.0004
Hexachlorocyclopentadiene	0.0005	Benzo[a]anthracene	0.0006
2,4,6-Trichlorophenol	0.0004	Chrysene	0.0010
2,4,5-Trichlorophenol	0.0006	Dioctyl phthalate	0.0003
2-Chloronaphthalene	0.0004	Benzo[b]fluoranthene	0.0013
2-Nitroaniline	0.0008	Benzo[k]fluoranthene	0.0018
1,4-Dinitrobenzene	0.0012	Benzo[a]pyrene	0.0018
Dimethyl phthalate	0.0017	Indeno[1,2,3-cd]pyrene	0.0052
1,3-Dinitrobenzene	0.0017	Dibenzo[a,h]anthracene	0.0045
2,6-Dinitrotoluene	0.0011	Benzo[g,h,i]perylene	0.0046
Acenaphthylene	0.0004		

Calibration results

EPA Method 8270D requires that average response factors be used for calibration of the target analytes and the %RSDs for those response factors be less than 20% for use of the results in quantitative analysis. The method allows for alternative curve fits as long as no more than 10% of the compounds have average response factors that do not exceed the 20% RSD criteria. For compounds that need the alternative curve fit, a correlation coefficient (r^2) of 0.99 must be obtainable. Table 6

shows the results for our calibration sequence where only 4 out of 76 analytes had average response factors greater than 20% with their corresponding r^2 values passing the criteria of 0.99 or greater. In addition to the average response factor criteria, the EPA recommends minimum response factors for certain compounds within the method. Table 7 shows the minimum response factors obtained by the method for all compounds and that they meet or exceed the requirements of the EPA.

Table 6 (part 1). Average relative response factor %RSD and r² values for linear regression fit

Compound name	Average relative response factor %RSD	r² of linear fit	Calibration fit	Compound name	Average relative response factor %RSD	r² of linear fit	Calibration fit
N-Nitrosodimethylamine	4.32		AvgCalFact	1-Methylnaphthalene	10.14		AvgCalFact
Pyridine	11.49		AvgCalFact	Hexachlorocyclopentadiene	16.43		AvgCalFact
2-Fluorophenol, (Surr)	5.03		AvgCalFact	2,4,6-Trichlorophenol	9.95		AvgCalFact
Phenol-d$_6$ (Surr)	5.57		AvgCalFact	2,4,5-Trichlorophenol	12.34		AvgCalFact
Phenol	10.26		AvgCalFact	2-Fluorobiphenyl (Surr)	5.16		AvgCalFact
Aniline	8.60		AvgCalFact	2-Chloronaphthalene	10.23		AvgCalFact
Bis(2-chloroethyl) ether	6.63		AvgCalFact	2-Nitroaniline	9.99		AvgCalFact
2-Chlorophenol	3.85		AvgCalFact	1,4-Dinitrobenzene	15.06		AvgCalFact
1,3-Dichlorobenzene	6.63		AvgCalFact	Dimethyl phthalate	8.00		AvgCalFact
1,4-Dichlorobenzene-d$_4$ (ISTD)	6.97		AvgCalFact	1,3-Dinitrobenzene	14.53		AvgCalFact
1,4-Dichlorobenzene	7.12		AvgCalFact	2,6-Dinitrotoluene	10.54		AvgCalFact
Benzyl alcohol	5.15		AvgCalFact	Acenaphthylene	13.23		AvgCalFact
1,2-Dichlorobenzene	6.33		AvgCalFact	1,2-Dinitrobenzene	15.24		AvgCalFact
Bis(2-chloroisopropyl) ether	4.70		AvgCalFact	3-Nitroaniline	11.73		AvgCalFact
2-Methylphenol	5.43		AvgCalFact	Acenaphthene-d$_{10}$ (ISTD)	5.95		AvgCalFact
3-Methylphenol/4-Methylphenol	5.39		AvgCalFact	2,4-Dinitrophenol	-	0.999	Lin
N-Nitroso-di-n-propylamine	5.53		AvgCalFact	4-Nitrophenol	12.28		AvgCalFact
Hexachloroethane	4.12		AvgCalFact	Acenaphthene	-	0.995	Lin
Nitrobenzene-d$_5$ (Surr)	6.85		AvgCalFact	2,4-Dinitrotoluene	13.75		AvgCalFact
Nitrobenzene	5.26		AvgCalFact	Dibenzofuran	12.24		AvgCalFact
Isophorone	9.99		AvgCalFact	2,3,5,6-Tetrachlorophenol	17.64		AvgCalFact
2,4-Dimethylphenol	6.05		AvgCalFact	2,3,4,6-Tetrachlorophenol	16.35		AvgCalFact
2-Nitrophenol	13.40		AvgCalFact	Diethylphthalate	7.68		AvgCalFact
Bis(2-chloroethoxy) methane	5.23		AvgCalFact	4-Chlorophenyl phenyl ether	7.21		AvgCalFact
2,4-Dichlorophenol	7.67		AvgCalFact	Fluorene	11.95		AvgCalFact
1,2,4-Trichlorobenzene	5.59		AvgCalFact	4-Nitroaniline	16.67		AvgCalFact
Naphthalene-d$_8$ (ISTD)	7.14		AvgCalFact	2-Methyl-4,6-dinitrophenol	-	0.998	Lin
Naphthalene	14.55		AvgCalFact	Diphenylamine	7.07		AvgCalFact
4-Chloroaniline	7.55		AvgCalFact	Azobenzene	6.08		AvgCalFact
Hexachlorobutadiene	4.78		AvgCalFact	2,4,6-Tribromophenol (Surr)	8.92		AvgCalFact
4-Chloro-3-methylphenol	8.86		AvgCalFact	4-Bromophenyl phenyl ether	5.14		AvgCalFact
2-Methylnaphthalene	12.66		AvgCalFact	Hexachlorobenzene	3.74		AvgCalFact

Table 6 (part 2). Average relative response factor %RSD and r² values for linear regression fit

Compound name	Average relative response factor %RSD	r² of linear fit	Calibration fit	Compound name	Average relative response factor %RSD	r² of linear fit	Calibration fit
Pentachlorophenol	-	0.997	Lin	Benzo[a]anthracene	4.92		AvgCalFact
Phenanthrene-d$_{10}$ (ISTD)	5.79		AvgCalFact	Chrysene-d$_{12}$ (ISTD)	11.69		AvgCalFact
Phenanthrene	12.18		AvgCalFact	Chrysene	6.18		AvgCalFact
Anthracene	11.72		AvgCalFact	Dioctyl phthalate	15.54		AvgCalFact
Carbazole	9.29		AvgCalFact	Benzo[b]fluoranthene	12.45		AvgCalFact
Dibutyl phthalate	10.68		AvgCalFact	Benzo[k]fluoranthene	13.40		AvgCalFact
Fluoranthene	10.16		AvgCalFact	Benzo[a]pyrene	16.85		AvgCalFact
Pyrene	11.76		AvgCalFact	Perylene-d$_{12}$ (ISTD)	12.08		AvgCalFact
p-Terphenyl-d$_{14}$ (Surr)	6.13		AvgCalFact	Indeno[1,2,3-cd]pyrene	19.02		AvgCalFact
Butyl benzyl phthalate	9.68		AvgCalFact	Dibenzo[a,h]anthracene	18.61		AvgCalFact
Bis(2-ethylhexyl) adipate	10.48		AvgCalFact	Benzo[g,h,i]perylene	13.68		AvgCalFact
Bis(2-ethylhexyl) phthalate	14.11		AvgCalFact				

Table 7 (part 1). Minimum response factors for compounds along with EPA Method 8270D recommended/suggested minimum response factors

Compound name	EPA 8270D recommended minimum response factor	Minimum response factor	Compound name	EPA 8270D recommended minimum response factor	Minimum response factor
N-Nitrosodimethylamine	n.a	0.62	2-Nitrophenol	0.10	0.11
Pyridine	n.a	0.92	Bis(2-chloroethoxy) methane	0.30	0.32
2-Fluorophenol, (Surr)	n.a	1.02	2,4-Dichlorophenol	0.20	0.22
Phenol-d$_6$ (Surr)	n.a	1.23	1,2,4-Trichlorobenzene	n.a	0.31
Phenol	0.80	1.26	Naphthalene-d$_8$ (ISTD)	n.a	n.a
Aniline	n.a	1.56	Naphthalene	0.70	0.75
Bis(2-chloroethyl) ether	0.70	1.01	4-Chloroaniline	0.01	0.36
2-Chlorophenol	n.a	1.17	Hexachlorobutadiene	0.01	0.18
1,3-Dichlorobenzene	n.a	1.38	4-Chloro-3-methylphenol	0.20	0.21
1,4-Dichlorobenzene-d$_4$ (ISTD)	n.a	n.a	2-Methylnaphthalene	0.40	0.56
1,4-Dichlorobenzene	n.a	1.37	1-Methylnaphthalene	n.a	0.52
Benzyl alcohol	n.a	0.68	Hexachlorocyclopentadiene	0.05	0.26
1,2-Dichlorobenzene	n.a	1.31	2,4,6-Trichlorophenol	0.20	0.28
Bis(2-chloroisopropyl) ether	n.a	0.38	2,4,5-Trichlorophenol	0.20	0.29
2-Methylphenol	0.70	1.08	2-Fluorobiphenyl (Surr)	n.a	1.34
3-Methylphenol/4-Methylphenol	0.60	1.08	2-Chloronaphthalene	0.80	0.99
N-Nitroso-di-n-propylamine	0.50	0.67	2-Nitroaniline	0.01	0.19
Hexachloroethane	0.30	0.44	1,4-Dinitrobenzene	n.a	0.12
Nitrobenzene-d$_5$ (Surr)	n.a	0.25	Dimethyl phthalate	0.01	1.02
Nitrobenzene	0.20	0.25	1,3-Dinitrobenzene	n.a	0.14
Isophorone	0.40	0.51	2,6-Dinitrotoluene	0.20	0.20
2,4-Dimethylphenol	0.20	0.29	Acenaphthylene	0.90	1.35

Table 7 (part 2). Minimum response factors for compounds along with EPA Method 8270D recommended/suggested minimum response factors

Compound name	EPA 8270D recommended minimum response factor	Minimum response factor	Compound name	EPA 8270D recommended minimum response factor	Minimum response factor
1,2-Dinitrobenzene	n.a	0.09	Phenanthrene	0.70	0.79
3-Nitroaniline	0.01	0.23	Anthracene	0.70	0.79
Acenaphthene-d$_{10}$ (ISTD)	n.a	n.a	Carbazole	0.01	0.73
4-Nitrophenol	0.01	0.12	Dibutyl phthalate	0.01	0.82
Acenaphthene	0.90	0.92	Fluoranthene	0.60	0.90
2,4-Dinitrophenol	0.01	0.02	Pyrene	0.60	1.00
2,4-Dinitrotoluene	0.20	0.24	p-Terphenyl-d$_{14}$ (Surr)	n.a	0.99
Dibenzofuran	0.80	1.27	Butyl benzyl phthalate	0.01	0.36
2,3,5,6-Tetrachlorophenol	n.a	0.19	Bis(2-ethylhexyl) adipate	0.00	0.32
2,3,4,6-Tetrachlorophenol	0.01	0.21	Bis(2-ethylhexyl) phthalate	0.01	0.50
Diethylphthalate	0.01	0.99	Benzo[a]anthracene	0.80	1.03
4-Chlorophenyl phenyl ether	0.40	0.62	Chrysene-d$_{12}$ (ISTD)	n.a	n.a
Fluorene	0.90	1.01	Chrysene	0.70	0.97
4-Nitroaniline	0.01	0.21	Dioctyl phthalate	0.01	0.72
2-Methyl-4,6-dinitrophenol	0.01	0.03	Benzo[b]fluoranthene	0.70	0.85
Diphenylamine	n.a	0.54	Benzo[k]fluoranthene	0.70	1.07
Azobenzene	n.a	0.20	Benzo[a]pyrene	0.70	0.70
2,4,6-Tribromophenol (Surr)	n.a	0.17	Perylene-d$_{12}$ (ISTD)	n.a	n.a
4-Bromophenyl phenyl ether	0.10	0.23	Indeno[1,2,3-cd] pyrene	0.50	0.79
Hexachlorobenzene	0.10	0.28	Dibenzo[a,h]anthracene	0.40	0.67
Pentachlorophenol	0.05	0.08	Benzo[g,h,i]perylene	0.50	0.76
Phenanthrene-d$_{10}$ (ISTD)	n.a	n.a			

NeverVent technology

Specifically designed to simplify the routine maintenance procedures and to maximize the GC-MS instrument uptime, NeverVent technology includes the proprietary Vacuum Probe Interlock (VPI) and the V-lock solution available on the ISQ 7610 single quadrupole GC-MS system (Figure 4). This allows ion source cleaning, changing of the ionization mode (electron impact vs. chemical ionization), or column replacement to be performed quickly without breaking the MS vacuum. The system is then ready to run samples within minutes, not hours or days. Additionally, the V-lock technology keeps the MS fully isolated from the GC system under vacuum, 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, including replacing the septum and liner or trimming the analytical column, without the use of any additional post column or auxiliary gas flow into the MS.

Figure 4. VPI being removed from the ISQ 7610 MS

Conclusion

The Thermo Scientific ISQ 7610 single quadrupole GC-MS system with the ExtractaBrite ion source and the innovative NeverVent technology is the perfect solution to perform EPA Method 8270D:

- The extended dynamic range of the XLXR detector system used on the ISQ 7610 GC-MS system enables coverage of 1 to 400 ppm in a single injection.

- Seventy-six compounds were reported, and each fulfilled the EPA Method 8270D requirements in terms of minimum relative retention time, minimum response factors, and linearity.

- Chromeleon CDS software, with the Environmental Reporting package, offers unparalleled flexibility, scalability, and compliance. It meets EPA Method 8270D 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.

- NeverVent Technology allows analytical labs to maintain their sample throughput by almost eliminating instrument downtime.

Reference

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/esam/epa-method-8270e-sw-846-semivolatile-organic-compounds-gas-chromatographymass-spectrometry-gc, Accessed March 6, 2022.

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