Advancing Semi-Volatile Organic Compound Detection with GC-MS/MS

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1. Introduction

Traditional solid-phase extraction methods for semi-volatile organic compounds (SVOCs), such as EPA 625.1 and 8270E, often require sample volumes up to 1 liter. However, growing environmental and safety concerns surrounding methylene chloride ($MeCl_2$) have driven interest in micro-extraction techniques that use much smaller sample volumes. These methods not only reduce solvent use but also align with green chemistry principles. While GC-MS remains the gold standard for SVOC analysis, coupling micro-extraction techniques with GC-MS/MS (triple quadrupole) offers enhanced method performance. This study demonstrates that GC-MS/MS provides accurate and compliant quantification of SVOCs, meeting the evolving demands of environmental laboratories for sustainable testing workflows.

2. Methods

The Restek SVOC Megamix 150 Kit and SVOC Internal Standard Mix were used to prepare two sets of calibration standards: (1) a traditional range from 0.2 ppm to 80 ppm, and (2) a lower range from 1 ppb to 1000 ppb. The lower range is designed to evaluate the extraction efficiency of the micro-extraction method using minimal volumes of $MeCl_2$. Calibration curves for both ranges were acquired using the GCMS-TQ8050 NX. Specific method parameters are detailed in Table 1.

Table. 1 Analytical conditions

GC-MS:	GCMS-TQ8050 NX
GC	
Column:	SH-I-SVOC MS (30m×0.25 mm, 0.25µm)
Insert:	Topaz liner splitless single taper
Inlet Temp.:	275 °C
Injection Volume:	1 µL
Injection:	Split 5
Carrier Gas:	Helium
Control Mode:	Constant linear velocity
MS	
IF Temp.:	320 °C
Ion Source:	230 °C
Ionization Mode:	EI
Mode:	Scan/MRM

3. Results

- Calibration Range and Sensitivity - GC-MS/MS was successfully calibrated from 200 ppb to 80,000 ppb (traditional range), confirming that the triple quadrupole system is well-suited for SVOC analysis using conventional sample preparation methods with a 1000x concentration factor. Using the lower calibration range (1 ppb to 1000 ppb) for micro-extraction-based workflows, all compounds met the EPA 625.1/8270E initial calibration (ICAL) requirements. Although some polar compounds (e.g., nitroaromatics and phenols) exhibited poorer chromatography at low concentrations, the method still achieved sufficient performance. For most analytes, the lowest calibration point was 1 or 5 ppb—up to 200x lower than those in the traditional range.
- Improved Detection limits - Compared to MDLs obtained using GC-MS and EPA Method 625.1, GC-MS/MS achieved significantly lower detection limits—up to 500x lower in some cases. This increased sensitivity enables the use of reduced solvent volumes, particularly methylene chloride, supporting safer and more environmentally responsible workflows. Detailed performance data, including calibration ranges (ppm and ppb), %RSE, and IDLs for each compound, are provided in Table 2.
- Operational Improvements GC-MS/MS vs GC-MS - Unlike single quadrupole systems that require DFTPP tuning every 12 hours under EPA Methods 625.1/8270, GC-MS/MS eliminates this need—improving uptime and reducing maintenance. Additionally, the ability to combine MRM quantitation with scan-based spectral qualification in a single run streamlines data review. Figure 1 shows the MRM and scan spectra for 2,3,4,5-tetrachlorophenol. Incorporating spectral pattern matching alongside ion ratio confirmation helps reduce false positives and improves compound identification confidence.

Figure. 1 MRM and Scan spectrum combination in a single run

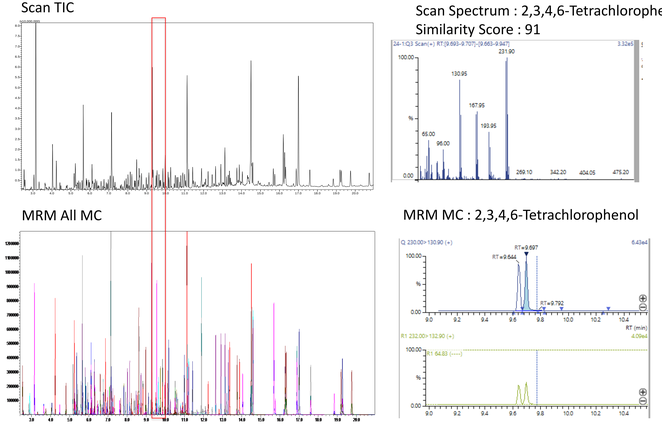

Table. 2 Individual calibration and repeatability results

		Traditional Range, Concentrations - ppm			Lower Range, Concentrations - ppb			
ID	Compound	Lowest	High	RSE	Lowest	High	RSE	IDL (pg)
1	1,4-Dioxane-d8				ISTD			
2	1,4-Dioxane	5.00	80.00	0.91	5	1000	8.99	0.90
3	N-Nitrosodimethylamine	0.20	80.00	1.75	1	500	10.38	1.68
4	Pyridine	1.00	80.00	1.11	50	1000	15.44	14.02
5	Ethyl methacrylate	5.00	80.00	1.42	1	1000	10.88	2.22
6	2-Picolin	5.00	80.00	2.06	1	1000	4.43	1.29
7	N-Nitrosomethylethylamine	5.00	80.00	2.06	5	1000	13.55	0.90
8	Methyl methanesulfonate	5.00	80.00	1.72	1	1000	16.91	0.57
9	2-Fluorophenol	0.20	80.00	2.99	1	500	5.69	0.37
10	N-Nitrosodiethylamine	5.00	80.00	1.50	5	1000	14.56	2.30
11	Ethyl methanesulfonate	5.00	80.00	1.61	5	1000	9.85	1.17
12	Benzaldehyde	5.00	80.00	1.06	5	1000	7.33	1.18
13	Phenol	0.20	80.00	3.33	1	1000	14.90	0.52
14	Phenol-d6(d5) Surr	0.20	80.00	2.92	1	1000	17.41	0.31
15	Aniline	0.20	80.00	3.60	5	500	8.59	2.41
16	Pentachloroethane	5.00	80.00	1.77	1	1000	10.09	1.83
17	Bis(2-chloroethyl) ether	0.20	80.00	0.79	1	1000	14.07	0.22
18	2-Chlorophenol	0.20	80.00	3.67	1	1000	15.42	0.64
19	1,3-Dichlorobenzene	0.20	80.00	1.76	1	1000	16.55	0.98
20	1,4-Dichlorobenzene	0.20	80.00	1.50	1	1000	11.68	0.70
21	1,4-Dichlorobenzene-d4				ISTD			
22	Benzyl Alcohol	0.20	80.00	6.70	5	500	16.44	0.76
23	1,2-Dichlorobenzene	0.20	80.00	1.65	1	1000	14.20	0.94
24	2-Methylphenol	0.20	80.00	4.81	10	500	17.79	2.07
25	2,2'-oxybis(1-chloropropane)	0.20	80.00	1.43	1	1000	13.64	1.09
26	Acetophenone	5.00	80.00	1.76	5	1000	8.73	1.02
27	N-Nitrosomorpholine	5.00	80.00	1.95	1	1000	15.24	2.87
28	3 and 4-Methylphenol	0.20	80.00	4.43	1	1000	19.36	0.69
29	N-Nitroso-di-N-propylamine	0.20	80.00	4.94	1	1000	15.17	2.07
30	2-Toluidine	5.00	80.00	2.22	1	1000	18.49	1.10
31	N-Nitrosopyrrolidine	5.00	80.00	2.22	5	1000	8.89	2.32
32	Hexachloroethane	0.20	80.00	2.11	5	1000	17.23	2.32
33	Nitrobenzene	0.20	80.00	7.22	5	1000	17.71	0.76
34	Nitrobenzene-d5	0.20	80.00	5.40	5	1000	14.90	0.73
35	N-Nitrosopiperidine	5.00	80.00	1.97	5	1000	15.89	2.18
36	Isophorone	0.20	80.00	5.13	1	1000	14.85	0.36
37	2-Nitrophenol	0.20	80.00	8.86	5	500	11.62	2.62
38	2,4-Dimethylphenol	0.20	80.00	4.75	10	500	18.86	1.53
39	Benzoic Acid	5.00	80.00	6.07	100	2000	7.07	43.90
40	Bis(2-chloroethoxy)methane	0.20	80.00	5.52	1	1000	13.25	0.73
41	alpha,alpha-Dimethyl phenethylamine	5.00	80.00	7.18	50	1000	5.22	75.50
42	2,4-Dichlorophenol	0.20	80.00	4.12	5	500	8.12	2.63
43	1,2,4-Trichlorobenzene	0.20	80.00	1.55	1	1000	12.44	0.66
44	Naphthalene	0.20	80.00	1.16	1	1000	10.99	0.26
45	Naphthalene-d8				ISTD			
46	2,6-Dichlorophenol	5.00	80.00	1.13	1	1000	11.67	1.34
47	4-Chloroaniline	0.20	80.00	3.42	1	500	8.39	0.97
48	Hexachlorobutadiene	0.20	80.00	1.42	1	1000	11.35	0.73
49	Caprolactam	5.00	80.00	0.65	5	1000	9.24	4.66
50	N-Nitrosodi-n-butylamine	5.00	80.00	3.47	5	1000	16.85	2.10
51	4-Chloro-3-methylphenol	0.20	80.00	1.85	1	500	5.23	0.85
52	IsoSafrole-1	5.00	80.00	2.18	1	1000	8.80	1.27
53	2-Methylnaphthalene	0.20	80.00	1.17	1	500	11.64	0.59
54	1-Methylnaphthalene	0.20	80.00	8.12	1	1000	11.75	0.54
55	1,2,4,5-Tetrachlorobenzene	5.00	80.00	1.80	1	1000	6.45	1.38
56	Hexachlorocyclopentadiene	0.20	80.00	4.64	5	500	8.06	2.45
57	2,4,6-Trichlorophenol	0.20	80.00	7.69	1	1000	9.21	0.92
58	2,4,5-Trichlorophenol	0.20	80.00	6.03	1	500	8.80	1.00
59	2-Fluorobiphenyl	0.20	80.00	1.54	1	1000	18.14	1.18
60	Isosafrole-2	5.00	80.00	1.87	1	1000	9.12	1.37
61	Safrole	5.00	80.00	1.37	1	1000	19.26	4.13
62	1,1'-Biphenyl	5.00	80.00	2.47	1	1000	7.35	2.24
63	2-Chloronaphthalene	0.20	80.00	2.59	1	1000	11.56	0.32
64	1-Chloronaphthalene	5.00	80.00	3.76	1	1000	8.00	0.76
65	Phenyl ether	1.00	80.00	6.62	1	1000	7.50	1.04
66	2-Nitroaniline	0.20	80.00	8.04	5	500	9.53	2.90
67	1,4-Naphthoquinone	5.00	80.00	0.98	5	1000	153.60	1.25
68	1,4-Dinitrobenzene	0.20	80.00	8.97	10	500	19.39	11.15
69	Dimethylphthalate	0.20	80.00	3.99	1	1000	14.55	0.66
70	1,3-Dinitrobenzene	0.20	80.00	12.63	5	500	10.72	7.06
71	2,6-Dinitrotoluene	1.00	80.00	10.07	5	500	7.37	2.91
72	1,2-Dinitrobenzene	0.20	80.00	6.64	20	1000	17.00	43.23
73	Acenaphthylene	0.20	80.00	1.70	1	1000	19.65	1.44
74	3-Nitroaniline	1.00	80.00	9.45	1	500	7.37	2.34
75	Acenaphthene-d10				ISTD			
76	Acenaphthene	0.20	80.00	5.61	1	500	12.06	1.71
77	2,4-Dinitrophenol	1.00	80.00	8.10	100	1000	18.59	32.07
78	4-Nitrophenol	1.00	80.00	8.11	10	500	13.58	15.52
79	2,4-Dinitrotoluene	0.20	80.00	7.66	1	1000	12.40	3.00
80	Dibenzofuran	0.20	80.00	6.86	1	1000	13.64	0.41
81	2-Naphthylamine	5.00	80.00	4.14	5	1000	12.01	2.18
82	2,3,5,6-Tetrachlorophenol	0.20	80.00	7.47	10	500	7.32	3.46
83	2,3,4,6-Tetrachlorophenol	0.20	80.00	16.03	5	500	9.87	2.69
84	1-Naphthylamine	5.00	80.00	4.18	1	1000	12.60	0.92
85	Diethylphthalate	0.20	80.00	3.77	5	1000	13.06	0.42
86	4-Chlorophenyl phenyl ether	0.20	80.00	1.59	1	1000	13.28	0.98
87	Fluorene	0.20	80.00	2.29	1	1000	13.81	0.60
88	4-Nitroaniline	1.00	80.00	9.74	5	500	7.80	2.44
89	N-Nitro-o-toluidine(5-Nitro-o-toluidine)	5.00	80.00	4.95	1	1000	19.40	1.30
90	4,6-Dinitro-2-methylphenol	1.00	80.00	13.93	20	500	9.54	14.97
91	N-Nitrosodiphenylamine	0.20	80.00	2.08	5	1000	14.96	0.49
92	Diphenylhydrazine	0.20	80.00	6.85	1	500	9.91	0.32
93	2,4,6-Tribromophenol	0.20	80.00	12.67	10	1000	18.36	4.12
94	1,3,5-Trinitrobenzene	5.00	80.00	3.99	10	1000	17.36	6.23
95	Diallate-1	5.00	80.00	1.55	1	1000	10.85	2.99
96	Phenacetin	5.00	80.00	1.06	5	1000	12.54	2.05
97	4-Bromophenyl phenyl ether	0.20	80.00	0.85	1	1000	14.05	1.15
98	Diallate-2	5.00	80.00	2.08	1	1000	15.85	2.54
99	Hexachlorobenzene	0.20	80.00	2.07	1	1000	17.12	1.32
100	Atrazine	5.00	80.00	2.54	5	1000	12.68	2.16
101	4-Aminobiphenyl	5.00	80.00	3.19	1	1000	12.53	1.31
102	Pentachlorophenol	0.20	80.00	7.56	5	500	9.94	1.89
103	Pronamide	5.00	80.00	1.58	5	1000	10.31	1.80
104	Pentachloronitrobenzene	5.00	80.00	1.89	5	1000	15.10	3.46
105	Phenanthrene	0.20	80.00	0.79	1	1000	12.26	0.75
106	Phenanthrene-d10				ISTD			
107	Anthracene	0.20	80.00	1.64	1	1000	16.47	0.49
108	Carbazole	0.20	80.00	3.80	1	1000	13.97	0.51
109	Di-n-butylphthalate	0.20	80.00	9.61	10	1000	19.48	0.25
110	4-nitroquinoline-1-oxide	5.00	80.00	3.16	5	1000	7.68	6.01
111	Isodrin	5.00	80.00	2.48	1	1000	8.77	2.34
112	Fluoranthene	0.20	80.00	3.84	1	1000	10.30	0.15
113	Benzidine	0.20	80.00	7.97	20	500	6.95	22.47
114	Pyrene	0.20	80.00	4.65	1	1000	10.23	0.39
115	Aramite-1	5.00	80.00	0.48	5	1000	7.19	1.88
116	o-Terphenyl-D14	0.20	80.00	2.06	1	1000	10.81	0.55
117	Aramite-2	5.00	80.00	0.75	5	1000	10.00	1.39
118	p-Dimethylaminoazobenzene	5.00	80.00	4.50	5	1000	11.04	0.58
119	Chlorobenzilate	5.00	80.00	2.94	5	1000	8.84	0.76
120	3,3'-Dimethylbenzidine	5.00	80.00	3.75	5	1000	10.03	2.39
121	Butylbenzylphthalate	0.20	80.00	9.30	5	1000	15.57	0.95
122	Bis(2-ethylhexyl)adipate	0.20	80.00	10.55	1	1000	19.64	0.66
123	2-Acetylaminofluorene	5.00	80.00	5.03	5	1000	17.79	1.63
124	3'3-Dichlorobenzidine	0.20	80.00	7.54	1	1000	19.91	0.53
125	Benz[a]anthracene	0.20	80.00	8.25	1	1000	12.97	0.24
126	Bis(2-ethylhexyl)phthalate	0.20	80.00	10.49	5	1000	15.81	0.55
127	Chrysene	0.20	80.00	8.32	1	1000	8.91	0.27
128	Chrysene-D12				ISTD			
129	Di-n-octylphthalate	0.20	80.00	9.76	1	1000	17.32	0.29
130	Benzo[b]fluoranthene	0.20	80.00	4.35	1	1000	10.37	0.22
131	7-12Dimethylbenz[a]anthracene	5.00	80.00	1.12	1	1000	11.24	0.84
132	Benzo[k]fluoranthene	0.20	80.00	3.75	1	1000	11.71	0.34
133	Benzo[a]pyrene	0.20	80.00	6.24	1	1000	9.08	0.30
134	Perylene-D12				ISTD			
135	3-Methylcholanthrene	5.00	80.00	0.74	1	1000	15.23	1.33
136	Dibenz[a,j]acridine	0.20	80.00	7.65	1	1000	15.54	0.76
137	Indeno[1,2,3-cd]pyrene	0.20	80.00	3.33	1	1000	13.08	0.45
138	Dibenz[a,h]anthracene	0.20	80.00	2.13	1	1000	11.78	0.39
139	Benzo[g,h,i]perylene	0.20	80.00	2.61	1	1000	9.56	0.52

4. Conclusions

The GC-MS/MS method demonstrated excellent sensitivity and calibration performance for SVOC analysis, with most compounds reliably quantified down to 5 ppb—more than 40x lower than traditional GC-MS-based methods. All target analytes met the QA/QC criteria of EPA Methods 625.1 and 8270E, including polar compounds that exhibited poorer chromatography at low concentrations. Beyond analytical performance, GC-MS/MS offers key operational benefits, including elimination of routine DFTPP tuning, faster data processing through combined MRM and scan acquisition, and significantly reduced solvent use. These advantages support safer, more efficient, and environmentally sustainable laboratory workflows.