SHIMADZU

ePREP

MP 081

Optimizing SVOC Analysis using GC-MS/MS with Automated Pretreatment and High-Speed Analytical Methods

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1. Introduction

- Recent U.S. regulations restricting dichloromethane ($MeCl_2$) have necessitated a shift from traditional liquid-liquid extraction to solvent-minimized micro-extraction.
- Solvent-minimized micro-extraction results in a more than 20 times reduction in analyte concentration, demanding significantly higher sensitivity from analytical systems.
- Shimadzu GCMS-TQ8050 RX low noise detector features noise reduction technology contributing to high sensitivity and Smart MRM secures adequate dwell time in simultaneous multicomponent analysis.
- Environmental labs face increasing pressure to optimize throughput and cost-per-sample.
- Automated sample prep using ePrep in combination with using a 20-meter column to reduce analysis time , minimize labor costs and protect health of environmental lab employees.
- This study presents an optimized workflow that integrates automated micro-extraction with high-sensitivity GC-MS/MS for the simultaneous analysis of multi-component environmental samples, balancing regulatory compliance with operational efficiency.

2. Methods

Figure 1. ePrep Workstation and Shimadzu GCMS-TQ8050 RX

- Tap water samples were collected and placed into high recovery vials.
- Sample preparation was conducted using method 3511 on the ePrep ONE Sample Preparation workstation for automated syringe-based micro-extraction to minimize solvent consumption and manual labor.
- Restek SVOC Megamix 150 kit and SVOC Internal Standard Mix were used for low range calibration standards from 1 ppb to 1000 ppb for demonstrating the extraction efficiency of the micro-extraction method using a minimal volume of MeCl2, which were also prepared using the ePrep system. Workflow are shown in Figure 1.
- Calibration curves and sample data were acquired by GCMS-TQ8050 RX. For Specific method conditions are listed in Table 1.

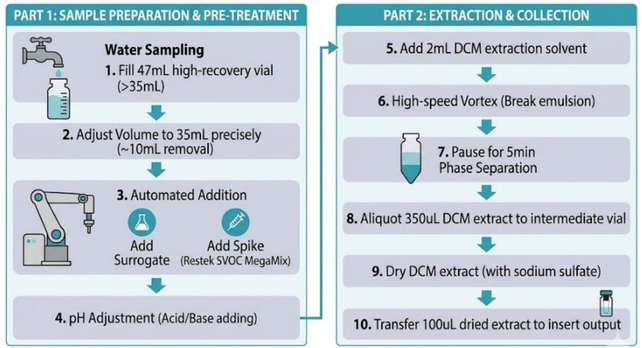

Figure. 2 Automated ePrep workflow

Table. 1 Analytical conditions

GC-MS:	GCMS-TQ8050 RX
GC	
Column:	RMX-5SilMS (20m×0.18 mm, 0.18µm)
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.:	300 °C
Ion Source:	230 °C
Ionization Mode:	EI
Mode:	MRM

3. Results

- Table 2 shows reporting limits in the sample and RSE of calibration curve for each compounds. All compounds pass 8270E requirement for ICAL.
- Figure 3 shows High-speed analysis achieves a 13-minute GC runtime while maintaining sufficient separation of PAH isomers

Table. 2 Initial calibration curve result

ID	Compound Name	RL (ug/L)	RSE	ID	Compound Name	RL (ug/L)	RSE	ID	Compound Name	RL (ug/L)	RSE	ID	Compound Name	RL (ug/L)	RSE
1	1,4-Dioxane	0.057	6.95	33	2-Nitrophenol	0.057	13.31	65	3-Nitroaniline	0.057	8.16	97	Anthracene	0.057	5.51
2	N-Nitrosodimethylamine	0.057	3.54	34	2,4-Dimethylphenol	0.057	6.94	66	Acenaphthene	0.057	6.26	98	Carbazole	0.057	2.38
3	Pyridine	0.286	10.92	35	Bis(2-chloroethoxy)methane	0.057	7.50	67	2,4-Dinitrophenol	0.571	10.14	99	Di-n-butylphthalate	0.057	5.69
4	Ethyl methacrylate	0.286	7.64	36	2,4-Dichlorophenol	0.057	16.58	68	4-Nitrophenol	0.571	6.35	100	4-nitroquinoline-1-oxide	0.571	6.30
5	2-Picolin	0.057	15.59	37	1,2,4-Trichlorobenzene	0.057	7.14	69	2,4-Dinitrotoluene	0.057	8.68	101	Methapyrilene	0.286	8.46
6	N-Nitrosomethylethylamine	0.286	7.47	38	Naphthalene	0.057	3.98	70	Pentachlorobenzene	0.057	4.60	102	Isodrin	0.057	10.04
7	Methyl methanesulfonate	0.286	5.43	39	2,6-Dichlorophenol	0.057	3.62	71	Dibenzofuran	0.057	8.76	103	Fluoranthene	0.057	3.59
8	N-Nitrosodiethylamine	0.057	9.65	40	4-Chloroaniline	0.057	12.48	72	2-Naphthylamine	0.286	11.06	104	Benzidine	0.286	4.06
9	Ethyl methanesulfonate	0.057	6.20	41	Hexachloropropene	0.057	10.44	73	2,3,5,6-Tetrachlorophenol	0.286	12.61	105	Pyrene	0.057	3.68
10	Benzaldehyde	0.057	14.75	42	Hexachlorobutadiene	0.057	6.86	74	2,3,4,6-Tetrachlorophenol	0.286	17.04	106	Aramite-1	0.057	6.76
11	Phenol	0.286	6.25	43	4-Chloro-3-methylphenol	0.057	12.61	75	1-Naphthylamine	0.286	9.79	107	Aramite-2	0.057	10.54
12	Aniline	0.286	13.83	44	IsoSafrole-1	0.057	6.90	76	Diethylphthalate	0.286	5.96	108	p-Dimethylaminoazobenzene	0.057	16.22
13	Pentachloroethane	0.057	12.37	45	2-Methylnaphthalene	0.057	3.15	77	4-Chlorophenyl phenyl ether	0.057	12.33	109	Chlorobenzilate	0.057	7.90
14	Bis(2-chloroethyl) ether	0.057	5.04	46	1-Methylnaphthalene	0.057	5.86	78	Fluorene	0.057	4.32	110	3,3'-Dimethylbenzidine	0.286	6.89
15	2-Chlorophenol	0.057	12.23	47	1,2,4,5-Tetrachlorobenzene	0.057	8.72	79	4-Nitroaniline	0.571	19.65	111	Butylbenzylphthalate	0.057	10.54
16	n-Decane	0.286	11.65	48	Hexachlorocyclopentadie	0.057	13.97	80	N-Nitro-o-toluidine	0.286	18.85	112	Kepone	0.057	12.66
17	1,3-Dichlorobenzene	0.057	3.52	49	2,4,6-Trichlorophenol	0.057	10.71	81	4,6-Dinitro-2-methylphenol	0.571	3.86	113	Bis(2-ethylhexyl)adipate	0.057	1.71
18	1,4-Dichlorobenzene	0.057	7.02	50	2,4,5-Trichlorophenol	0.057	6.84	82	N-Nitrosodiphenylamine	0.057	5.58	114	2-Acetylaminofluorene	0.057	4.74
19	Benzyl Alcohol	0.286	6.57	51	Isosafrole-2	0.057	8.79	83	Diphenylamine	0.057	5.58	115	3,3-Dichlorobenzidine	0.057	7.87
20	1,2-Dichlorobenzene	0.057	4.56	52	Safrole	0.286	10.67	84	Diphenylhydrazine	0.286	12.65	116	Benz[a]anthracene	0.057	4.71
21	2-Methylphenol	0.057	7.93	53	1,1'-Biphenyl	0.057	3.89	85	Diallate-1	0.057	15.43	117	Bis(2-ethylhexyl)phthalate	0.057	15.61
22	2,2'-oxybis(1-chloropropane)	0.286	10.59	54	2-Chloronaphthalene	0.057	5.33	86	Phenacetin	0.286	10.43	118	Chrysene	0.057	3.26
23	Acetophenone	0.057	15.16	55	1-Chloronaphthalene	0.057	4.51	87	4-Bromophenyl phenyl ether	0.057	7.48	119	Di-n-octylphthalate	0.057	14.11
24	N-Nitrosomorpholine	0.057	13.71	56	Phenyl ether	0.057	9.94	88	Diallate-2	0.057	16.16	120	Benzo[b]fluoranthene	0.057	6.04
25	3 and 4-Methylphenol	0.057	7.43	57	2-Nitroaniline	0.286	16.07	89	Hexachlorobenzene	0.057	7.43	121	7-12Dimethylbenz(a)anthracen	0.057	7.24
26	N-Nitrosodi-N-propylamine	0.057	17.60	58	1,4-Naphthoquinone	0.057	17.56	90	Atrazine	0.057	19.14	122	Benzo[k]fluoranthene	0.057	6.01
27	2-Toluidine	0.057	6.79	59	1,4-Dinitrobenzene	0.286	9.12	91	4-Aminobiphenyl	0.057	15.30	123	Benzo[a]pyrene	0.057	6.23
28	N-Nitrosopyrrolidine	0.057	15.99	60	Dimethylphthalate	0.057	6.23	92	Pentachlorophenol	0.286	9.63	124	3-Methylcholanthrene	0.057	6.42
29	Hexachloroethane	0.057	11.67	61	1,3-Dinitrobenzene	0.286	7.08	93	Octadecane	0.286	7.09	125	Dibenz[a,j]acridine	0.057	5.65
30	Nitrobenzene	0.057	14.29	62	2,6-Dinitrotoluene	0.286	7.82	94	Pronamide	0.057	18.16	126	Indeno[1,2,3-cd]pyrene	0.057	5.82
31	N-Nitrosopiperidine	0.286	12.81	63	1,2-Dinitrobenzene	0.571		95	Pentachloronitrobenzene	0.057	15.91	127	Dibenz[a,h]anthracene	0.057	6.92
32	Isophorone	0.057	6.37	64	Acenapthylene	0.057	10.68	96	Phenanthrene	0.057	6.56	128	Benzo[g,h,i]perylene	0.057	8.96

- Figures 4 & 5 show Robust surrogate recoveries demonstrated during spike recovery test.
- Figure 6 shows Excellent extraction efficiency for SVOC components; a 10-ppb spike of the MegaMix and Appendix compounds yielded 70–130% recovery for most target analytes.
- Although direct extraction yielded around 20% recovery for phenols, implementing an isotope dilution approach successfully restored recoveries to approximately 100%.

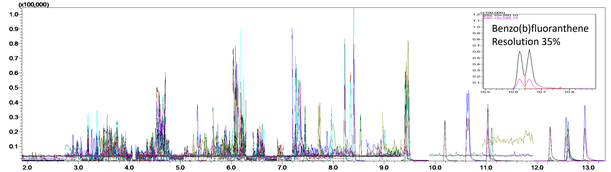

Figure 3. 10 ppb MRM total ion chromatogram

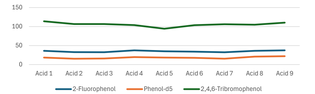

Figure 4. Acid Surrogate Recovery

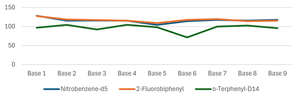

Figure 5. Base Surrogate Recovery

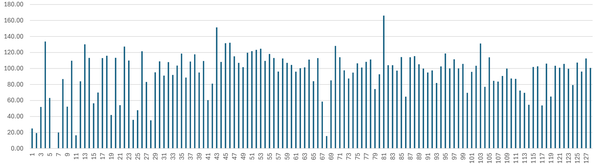

Figure 6. 10 ppb spike recovery test result into Tap water

4. Conclusions

This study successfully demonstrated that the integration of automated sample preparation for EPA Method 3511, a high-speed GC method, and a high-sensitivity triple quadrupole (TQ) mass spectrometer maximizes laboratory productivity while minimizing labor costs per sample. Furthermore, by implementing an isotope dilution approach for the analysis of phenols, we achieved significantly higher recoveries and enhanced analytical reliability. Overall, this combined workflow offers a highly efficient and robust solution for high-throughput routine testing without compromising data quality.