Fast Analysis of EPA Method 8270 Acid Extractable Compounds Using Gas Chromatography/Tandem Mass Spectrometry (GC/MS/MS)

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Introduction

EPA Method 8270[1] is commonly used for the analysis of a wide variety of semivolatile organic compounds (SVOCs) in environmental matrices. Due to the large number of compounds the analytical run can be as long as 45 minutes. Using a GC tandem mass spectrometer[2] (GC/MS/MS or GC-Triple Quad) with electron ionization (EI) and dynamic multiple reaction monitoring (dMRM) a 10-minute analysis time was developed to increase efficiency in the laboratory. The analyte list has six internal standards, eight surrogates and 72 target compounds including polycyclic aromatic hydrocarbons[3] (PAHs) and other acid, base, neutral compounds. Results of the fast 8270 method using GC/MS/MS were compared to traditional GC/MS on real extracts from environmental samples prepared by liquid/liquid extraction[4] with dichloromethane (DCM) at pH ≤ 2.

Equipment

Instrument: Agilent 7890/7010A Triple Quadrupole GC/MS
Software: MassHunter B.09
Liner: Agilent 5190-2295 Ultra Inert, wool
Column: Agilent DB5MS UI 20 m x 0.18 mm x 0.18 µm
Sample Preparation: Corning AOS™ liquid-liquid extractors

Reagents and Standards

Calibration standards: Supelco
Alternate source: Restek
Internal standards: Phenova
DCM: Foxpure D454-4
Reagent Water: Milli-Q

GC/MS/MS Operating Conditions

GC Conditions	MS/MS Conditions
Inlet temperature: 280°C	Ion source: EI
Oven temperature: 40°C	Ionization voltage: 70 eV
Ramp: 35°C/min to 320°C	Interface temperature: 280°C
Hold for 2 min	Ion source temperature: 325°C
Injection volume: 1 µL	
Split ratio: 100:1	dMRMs: 176
Carrier gas: Helium	(Examples below)
Flow: 1.0 mL/min	

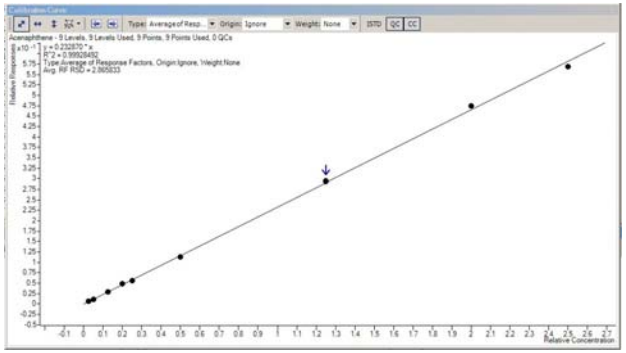
Triple Quadrupole Acquisition Method - MS Parameters Report

Compound Table:

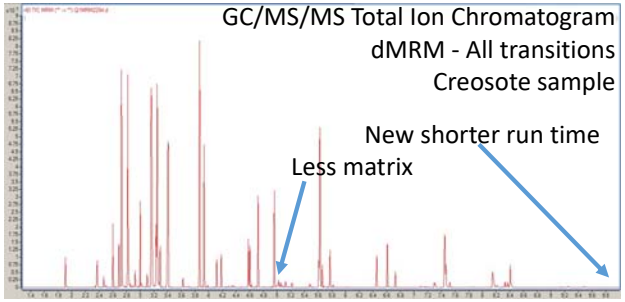
Compound Name	ISTD ?	Precursor ion	MS1 res	Product ion	MS2 res	RT	Left Delta RT	Right Delta RT	Dwell (ms)	CE (V)
2-Chlorophenol-d4		132	Wide	95.9	Wide	2.40	0.50	0.50	19.7	10
2-Chlorophenol-d4		132	Wide	67.8	Wide	2.40	0.50	0.50	19.7	10
Bis(2-chloroethyl)ether		95.1	Wide	65	Wide	2.43	0.10	0.10	10	5
Bis(2-chloroethyl)ether		93.1	Wide	63	Wide	2.43	0.10	0.10	10	0
2-Chlorophenol		128	Wide	64	Wide	2.46	0.10	0.15	9.2	15
2-Chlorophenol		128	Wide	63	Wide	2.46	0.10	0.15	9.2	30
1,3-Dichlorobenzene		146	Wide	111	Wide	2.55	0.20	0.42	8.2	15
1,3-Dichlorobenzene		146	Wide	75	Wide	2.55	0.20	0.42	8.2	30
1,4-Dichlorobenzene		146	Wide	111	Wide	2.60	0.42	0.42	10.3	15
1,4-Dichlorobenzene		146	Wide	75	Wide	2.60	0.42	0.42	10.3	30
1,4-Dichlorobenzene-d4	X	150	Wide	115	Wide	2.60	0.10	0.10	9.2	15
1,4-Dichlorobenzene-d4	X	150	Wide	78	Wide	2.60	0.10	0.10	9.2	30

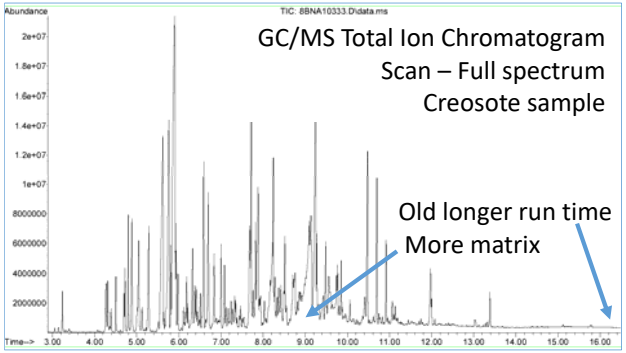
Calibration

Calibration points: 9
Calibration range: 1-100 ug/ml in DCM
Curve fits: Average of Response factors, %RSD≤20
or Quadratic, $r^2 \geq 0.99$



GC/MS/MS versus GC/MS - Creosote sample





Initial Demonstration of Proficiency

Initial Demonstration of Proficiency, examples. Full list available upon request.

Compound, Spiked 50 ug/L	Quant dMRM	RT	IDP-1	IDP-2	IDP-3	IDP-4	AVG	STDEV	%RSD	AVG %REC
Phenol	94.0 -> 66.1	2.4	53	55	55	54	54	1	2	109
1,2-Dichlorobenzene-d4	152.0 -> 78.0	2.7	41	38	41	35	39	3	8	78
2-Methylphenol	107.0 -> 77.0	2.7	53	55	56	54	55	1	2	109
Naphthalene	128.1 -> 102.1	3.4	48	48	48	48	48	0	1	96
Hexachlorocyclopentadiene	237.0 -> 119.0	4.0	47	48	49	48	48	1	2	95
2-Nitroaniline	138.0 -> 65.0	4.3	54	53	55	53	54	1	2	107
Acenaphthene	153.1 -> 77.0	4.6	49	49	50	49	49	0	0	99
4-Nitrophenol	138.9 -> 109.0	4.7	54	55	56	56	55	1	2	111
2,4-Dinitrotoluene	165.0 -> 63.0	4.7	54	54	55	54	54	0	1	108
Pentachlorophenol	265.9 -> 167.0	5.5	51	52	53	53	52	1	2	105
Phenanthrene	178.1 -> 152.1	5.6	51	51	51	51	51	0	0	102
Benzo (b) fluoranthene	252.1 -> 250.1	8.2	54	55	56	57	56	1	2	111
Benzo (a) pyrene	252.1 -> 250.1	8.4	57	57	57	57	57	0	1	114
Benzo (g,h,i) perylene	276.1 -> 274.1	9.5	56	57	57	58	57	1	1	114

Conclusions

The Fast 8270 method using GC/MS/MS has several advantages:

- Shortened run time increases efficiency in the laboratory.
- Tandem mass spectrometry reduces matrix interferences.
- Dynamic MRM eliminates time segments.

Concerns include reproducibility over time and lower limit of quantitation (LLOQ) due to the small amount of extract on column.

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References

[1]U.S. EPA. Method 8270E Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS). *SW-846 VI* (2018)
[2] Yost & Enke. *J. Am. Chem. Soc.*, 100(7), (1978) 2274-2275
[3]Anderson et al. *J Chrom A*, 1419 (2015) 89-98
[4] U.S. EPA. Method 3520C Continuous Liquid-Liquid Extraction, Rev. 3; *SW-846*, (1996)

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