TO13A Semi-Volatile Organic Compounds

Method TO-13A is used for the analysis of samples collected in either high or low volume PUF/XAD cartridges with a filter. The filter and cartridge are extracted with hexane/ethyl ether in a soxhlet extractor as described in the method. The solvent is concentrated and analyzed by GC/MS in either the full scan or SIM mode. The target list for the method is a select group of polynuclear aromatic hydrocarbons (PAH). The method is also used for a modified 8270 semi-volatile compound list, including phthalates, and phenols. Pesticides can also be run by this method. EAS also does SW-846 Method 8270 on Modified Method 5 XAD cartridges, wipe samples, and product samples.

The EAS modifications to the method include the target list, calibration, and QC criteria. The QC criteria for Method 8270 is different than TO-13A.

Summary of Extraction Method for TO-13A

Samples are extracted using hexane/diethyl ether in a soxhlet extractor as described in the method and are concentrated to a 1 ml volume.

Parameter	EAS Method	TO-13A
Extraction	Soxhlet for 18 hours in 10% diethyl ether in hexane. EAS uses a combination PUF and XAD sampling media, so dichloromethane is not suitable for extraction.	Soxhlet for 18 hours in 10% diethyl ether in hexane. Extraction solvent for XAD extraction solvent is listed as dichloromethane. PUF extraction solvent is listed is hexane.
Sample Medium	Whatman Quartz Filter and PUF Cartridge, High Volume	Whatman Quartz Filter and PUF Cartridge, High Volume
Holding Times	Store at 4C and extract in 7 days. Analyze within 40 days of extraction	Store at 4C and extract in 7 days. Analyze within 40 days of extraction

Table 13.6a
Summary of QC Criteria for TO-13A

Parameter	EAS TO-13A Modified	TO-13A Method
Extraction	18 hours with 10% ethyl ether/hexane mix.	18 hours with 10% ethyl ether/hexane mix.
DFTPP Tune	Every 12 hours	Every 12 hours
Tuning Criteria with DFTPP	TO-13 50 ng DFTPP TO-13A criteria	TO-13 50 ng DFTPP TO-13A criteria
Initial Calibration	5 Points Minimum 1 ug/ml to 50 ug/ml See Table 13.6d	5 Points Minimum 0.1 – 2.5 ug/ml <30% RSD for compounds listed in the method.
Calibration Check Sample (CCS)	After Initial Calibration	After Initial Calibration
Continuing Calibration Verification (CCV)	12 hours – Midpoint on Curve See Table 13.6d	12 hours – Midpoint on Curve Same criteria as initial calibration
Internal Standard (IS)	See Table 13.6b RT +/-0.06 RRT units	1,4-Dichlorobenzene-d4 is not listed as an internal standard in the TO-13A method, but does appear in EPA 8270. Response 50-200%
Surrogate (lab)	All samples prior to extraction See Table 13..6c	Fluorene-d10 Pyrene-d10 20 uL of a 50 ug/mL solution 60-120% recovery
Method Blank	1 per batch of 20 Less than LOQ	With each batch of up to 20 samples. Less than MDL
Laboratory Control Spike	1 per batch of 20 samples See Table 13.6d	With each batch of up to 20 samples 60-120% recovery
Duplicate Lab Control Dup Sample Dup	1 per batch of 20 samples See Table 13.6d	Method does not specify an LCD

Table 13.6b
Internal Standards

Compound	Recovery %
1,4-Dichlorobenzene-d4	50- 200
Naphthalene-d8	50- 200
Acenaphthene-d10	50- 200
Phenanthrene-d10	50- 200
Chrysene-d12	50- 200
Perylene-d12	50- 200

Table 13.6c
Surrogate Standards

Compound	Recovery %
Nitrobenzene-d5	50- 150
2-Fluorobiphenyl	50- 150
p-Terphenyl-d14	50- 150

Table 13.6d
Method TO-13 PAH Compound List

Analyte	Full Scan LOQ ug	SIM LOQ ug	Criteria ICAL CCV %D	Criteria Duplicate %D
naphthalene	1.0	0.1	<30	<30
acenaphthene	1.0	0.1	<30	<30
fluorene	1.0	0.1	<30	<30
phenanthrene	1.0	0.1	<30	<30
anthracene	1.0	0.1	<30	<30
fluoranthene	1.0	0.1	<30	<30
pyrene	1.0	0.1	<30	<30
chrysene	1.0	0.1	<30	<30
benz[a]anthracene	1.0	0.1	<30	<30
benzo(b)fluoranthene	1.0	0.1	<30	<30
benzo(k)fluoranthene	1.0	0.1	<30	<30
benzo(a)pyrene	1.0	0.1	<30	<30
indeno(1,2,3-cd)pyrene	1.0	0.1	<30	<30
dibenz(a,h)anthracene	1.0	0.1	<30	<30
benzo(g,h,i)perylene	1.0	0.1	<30	<30

Table 13.6e
Extracted Laboratory Control Standards

Analyte	LCS %R
1,4-dichlorobenzene	50-150
N-nitrosodi-n-propylamine	50-150
1,2,4-trichlorobenzene	50-150
2,4-dinitrotoluene	50-150
Acenaphthene	50-150
Pyrene	50-150

Table 13.6f
Method TO-13 SVOC Compound List

Analyte	LOQ ug	Criteria		
		ICAL %D	CCV %D	Duplicate %RPD
bis(2-chloroethyl)ether	1.0	<30	<30	<30
1,3-dichlorobenzene	0.5	<30	<30	<30
1,4-dichlorobenzene	1.0	<30	<30	<30
1,2-dichlorobenzene	1.0	<30	<30	<30
bis(2-chloroisopropyl)ether	1.0	<30	<30	<30
Hexachloroethane	1.0	<30	<30	<30
N-nitrosodi-n-propylamine	1.0	<30	<30	<30
Nitrobenzene	1.0	<30	<30	<30
Isophorone	1.0	<30	<30	<30
bis(2-chloroethoxy)methane	1.0	<30	<30	<30
1,2,4-trichlorobenzene	1.0	<30	<30	<30
Naphthalene	1.0	<30	<30	<30
Hexachlorobutadiene	1.0	<30	<30	<30
hexachlorocyclopentadiene	1.0	<30	<30	<30
2-chloronaphthalene	1.0	<30	<30	<30
dimethyl phthalate	1.0	<30	<30	<30
2,6-dinitrotoluene	1.0	<30	<30	<30
Acenaphthene	1.0	<30	<30	<30
2,4-dinitrotoluene	1.0	<30	<30	<30
Fluorene	1.0	<30	<30	<30
diethyl phthalate	1.0	<30	<30	<30
4-chlorophenyl phenyl ether	1.0	<30	<30	<30
Azobenzene	1.0	<30	<30	<30
4-bromophenyl phenyl ether	1.0	<30	<30	<30
Hexachlorobenzene	1.0	<30	<30	<30
Phenanthrene	1.0	<30	<30	<30
Anthracene	1.0	<30	<30	<30
di-n-butyl phthalate	1.0	<30	<30	<30
Fluoranthene	1.0	<30	<30	<30
Pyrene	1.0	<30	<30	<30
butyl benzyl phthalate	1.0	<30	<30	<30
Chrysene	1.0	<30	<30	<30
benz[a]anthracene	1.0	<30	<30	<30
bis(2-ethylhexyl)phthalate	1.0	<30	<30	<30
di-n-octyl phthalate	1.0	<30	<30	<30
benzo(b)fluoranthene	1.0	<30	<30	<30
benzo(k)fluoranthene	1.0	<30	<30	<30
benzo(a)pyrene	1.0	<30	<30	<30
indeno(1,2,3-cd)pyrene	1.0	<30	<30	<30

Analyte	LOQ	Criteria		
		ICAL	CCV	Duplicate
	ug	%D	%D	%RPD
dibenz(a,h)anthracene	1.0	<30	<30	<30
benzo(g,h,i)perylene	1.0	<30	<30	<30