

Analysis of Phenols in Waste Water Using EPA Method 604 with Automated Solid Phase Extraction (FMS TurboTrace®)

Introduction

Phenols are a diverse class of aromatic compounds, used extensively throughout the chemical manufacturing, medical, and pharmaceutical industries. Many of them exhibit well-established human toxicity and are highly water-soluble and thus found at significant concentrations in industrial and municipal wastewater supplies.

The EPA has developed certain methods for their extraction and analysis. The extraction method outlines the use of solid phase extraction for waste water matrix samples employing both cartridges and disks. Consistent with other EPA 600 series methods, EPA 604 incorporates a rigid set of QC and acceptance criteria requiring precise and reproducible analytical practices. The potential for error and the variability associated with manual extractions makes the benefits of automating these processes apparent. Hence, FMS developed a simple automated system which is fast and yields high quality data.

Instrumentation

- FMS TurboTrace® System
- FMS SuperVap®
- Vacuum pump
- Agilent 7890A GC-FID

Consumables

- FMS, Inc. 0.5 g SDVB cartridge
- FMS sodium sulfate cartridge
- Ultra-pure DI water
- Fisher 6 N Hydrochloric Acid
- Fisher Pesticide Grade Methanol
- Fisher Pesticide Grade Dichloromethane
- Restek 604 spiking standards

Procedure

- 6 samples (1L water each) are prepared and acidified with mL HCl till pH ~ 2
- Spike with various 604 standards
- Put sample bottles in place and fill rinse bottles with 13 mL dichloromethane
- Cartridges are installed in each of the six positions.
- Vacuum is turned on
- Cartridges are conditioned with 3 x 3 mL dichloromethane (drain), 3 x 3mL methanol (keep wet), 6 x 3 mL 0.05N HCl (keep wet)
- Samples are loaded across cartridges under vacuum, at a rate of 20 mL/min
- Cartridges are dried under vacuum for 15-30 min (no nitrogen)
- Sample bottles are automatically rinsed from the rinse bottles with 13 mL dichloromethane
- Dichloromethane from sample bottles is loaded across the cartridges (10 mL, then 3 mL) and the eluent is collected.
- The extracts are exchanged into 10 mL isopropanol for analysis into Direct to GC Vial Collection Vessels
- Extracts are dried over sodium sulfate or in line cartridges can be used downstream from SDVB cartridges

FMS SuperVap®

- Pre-heat temp: 40 °C
- Pre-heat time: 15 minutes
- Heat in Sensor mode at 40 °C under nitrogen (7-10 psi)
- Direct to GC Vial Vessel Reduce to 1 mL
- Samples are now ready for analysis



Table 1 with recoveries for various 604 compounds

Compound	%Recovery	Stdev
2-chlorophenol	83.6	1.14
2-nitrophenol	84.9	1.87
2,4-dimethylphenol	95.5	3.42
2,4-dichlorophenol	92.6	3.81
4-chloro-3-methylphenol	102.3	1.55
2,4,6-trichlorophenol	97.2	3.83
2,4-dinitrophenol	85.5	1.98
4-nitrophenol	100.3	2.47
2-methyl-4,6-dinitrophenol	80.2	3.91
Pentachlorophenol	93.7	2.35
Phenol	86.2	1.79

Conclusions

Reviewing the sample data shows high recoveries for a dozen phenols, demonstrating excellent efficiency for these compounds. Samples can be taken from collection bottle to GC vial in one quick, consistent, reproducible process that will save laboratories both time and money.



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FMS TurboTrace® System and SuperVap®