



LABMAIL #12

August 2011

## TRACE DETERMINATION OF VOCs IN SOLID MATRICES USING GC-MS AFTER 'WATER EXTRACTION'

ALS Czech Republic has validated a new method for the determination of volatile organic compounds in various solid samples (soil, sediment, solid waste) by headspace analysis followed by gas chromatography coupled to mass spectrometry detection. The sample preparation is based on the method US EPA 5021. It uses the 'water extraction', instead of traditional methanol as a solvent.



The benefit of this approach is low reporting limits down to 0.1 µg/kg dry weight (higher limits apply to Chloromethane, Dichloromethane, tert-Butyl alcohol, Toluene and Tetrachloroethene). Also such sample preparation better mimics conditions which occur in nature compared to methanol-based extraction. About 70 volatile compounds (listed in Table I) are determined by this method. ALS experienced staff, high-tech equipment and comprehensive quality control procedures ensure that accurate and defensible analytical data are produced.

LIMS code: S-VOCGMS06

### Method scope and application

The method is applicable to various solid sample matrices such soil, sediment, waste. Due to high sensitivity, the method is convenient for trace content analyses of VOCs. The benefit is extremely low limit of quantification and extraction which mimics the natural processes.

### Sample container and reporting limits (LORs)

It is recommended to use a tightly closed glass container with about 50 grams of sample. The container should be completely filled with no headspace and kept chilled.

The method is capable of LORs down to 0.1 µg/kg dry weight. Higher limits are applicable to Chloromethane, Dichloromethane, tert-Butyl alcohol, Toluene and Tetrachloroethene (1 - 10 µg/kg DW).

### What is water extraction?

The method is based on applying a salted water solution with adjusted pH for extraction, so called 'matrix-modifier'. A low pH of the solution eliminates biodegradation of target compounds and minimizes dehydrohalogenation reactions. The pH of this solution may vary depending on type of determined analytes. The extraction by water corresponds very well with processes in nature and brings the possibility to determine compounds naturally released from the environment.

Table I: List of analytes

1.1.1.2-Tetrachloroethane	1.3.5-Trimethylbenzene	Dibromochloromethane	n-Propylbenzene
1.1.1-Trichloroethane	1.3-Dichlorobenzene	Dibromomethane	ortho-Xylene
1.1.2.2-Tetrachloroethane	1.3-Dichloropropane	Dichlorodifluoromethane	p-Isopropyltoluene
1.1.2-Trichloroethane	1.4-Dichlorobenzene	Dichloromethane	sec-Butylbenzene
1.1-Dichloroethane	2.2-Dichloropropane	Ethyl tert-Butyl Ether (ETBE)	Styrene
1.1-Dichloroethene	2-Chlorotoluene	Ethylbenzene	tert-Butyl alcohol (TBA)
1.1-Dichloropropylene	4-bromofluorobenzene	Hexachlorobutadiene	tert-Amyl Methyl Ether (TAME)
1.2.3-Trichlorobenzene	4-Chlorotoluene	Chlorobenzene	tert-Butylbenzene
1.2.3-Trichloropropane	Benzene	Chloroethane	Tetrachloroethene
1.2.4-Trichlorobenzene	Bromobenzene	Chloroform	Tetrachloromethane
1.2.4-Trimethylbenzene	Bromodichloromethane	Chloromethane	Toluene
1.2-Dibromo-3-chloropropane	Bromoform	Isopropylbenzene	trans-1.2-Dichloroethene
1.2-Dibromoethane (EDB)	Bromochloromethane	meta- & para-Xylene	trans-1.3-Dichloropropene
1.2-Dichlorobenzene	Bromomethane	Methyl tert-Butyl Ether (MTBE)	Trichloroethene
1.2-Dichloroethane	cis-1.2-Dichloroethene	Naphthalene	Trichlorofluoromethane
1.2-Dichloropropane	cis-1.3-Dichloropropylene	n-Butylbenzene	Vinyl chloride
1.3.5-Trichlorobenzene			

### Interference

The sample matrix itself can cause interference. Samples with high content of oily material and organic sludge waste inhibit the partitioning of the volatile analytes into the headspace. Therefore, this method is not appropriate for samples with organic carbon content more than 1% (may yield low recoveries).

Also, samples can be contaminated by diffusion of volatile organics during transport and storage. To avoid false positive results, transport and sample storage areas must be free of target analytes. Samples should be analyzed within 14 days of collection.

Routine TAT is 7-10 days after sample reception.

### References:

- (1) US EPA 5021a, Revision 1, June 2003
- (2) Flores, P., Bellar, T., „Determination of Volatile Organic Compounds in Soils using Equilibrium Headspace Analysis and Capillary Column Gas Chromatography/Mass Spectrometry, „U.S. Environmental Protection Agency, Office of Research and Development, Environmental Monitoring Systems Laboratory, Cincinnati, OH, December, 1992.
- (3) Ioffe, B.V., Vitenberg, A.G., „Headspace Analysis and Related Methods in Gas Chromatography,“ John Wiley and Sons, 1984.

ALS Czech Republic, s.r.o.  
Na Harfě 336/9, 190 00 Praha 9, Czech Republic  
tel.: +420 226 226 228  
email: [customer.support@alsglobal.com](mailto:customer.support@alsglobal.com)

**RIGHT SOLUTIONS RIGHT PARTNER**

[www.alsglobal.eu](http://www.alsglobal.eu)