



## Determination of Volatile Alcohols, Acetates and Ketones in Waters by Headspace GC-MS

### Scope and Range

This method quantitatively determines a range of twenty two volatile alcohols, acetates and ketones by headspace GC-MS analysis.

This method is applicable for the analysis of surface waters and soils and similar environmental samples.

Compound	Low cal std (µg/l)	High cal std (µg/l)	Water sample range (mg/l)	Soil sample range (mg/l)
Methanol	95	18987	0.095-18.987	0.190-37.974
Ethanol	95	18927	0.095-18.987	0.190-37.974
Acetone	95	18927	0.095-18.987	0.190-37.974
iso-Propanol	94	18809	0.094-18.809	0.188-37.618
Methyl Acetate	112	22388	0.112-22.388	0.224-44.776
Tertiary Butanol	93	18576	0.093-18.576	0.186-37.152
Di-isopropyl-Ether	43	8696	0.043-8.696	0.086-17.392
n-Propanol	97	19355	0.097-19.355	0.194-38.710
2-Butanone	97	19355	0.097-19.355	0.194-38.710
Ethyl Acetate	108	21661	0.108-21.661	0.216-43.322
sec-Butanol	97	19417	0.097-19.417	0.194-38.834
Tetra Hydro Furan	107	21352	0.107-21.352	0.214-42.704
Iso-Butanol	96	19231	0.096-19.231	0.192-38.462
iso-Propyl Acetate	52	10453	0.052-10.453	0.104-20.906
n-Butanol	97	19417	0.097-19.417	0.194-38.834
n-Propyl Acetate	50	10033	0.05-10.033	0.100-20.066
MIBK	48	9585	0.048-9.585	0.096-19.170
n-Pentanol	98	19544	0.098-19.544	0.196-39.088
n-Butyl Acetate	53	10601	0.053-10.601	0.106-21.202
2-Furaldehyde	139	27778	0.139-27.778	0.278-55.556
n-Hexanol	98	19544	0.098-19.544	0.196-39.088
n-Heptanol	98	19672	0.098-19.672	0.196-39.344

### Principle

#### Preparation and Extraction

The vials containing the samples ( $10 \pm 0.25$ ml of the water) and 5g of sodium chloride are heated to 80°C and shaken using a CTC/Gerstel MPS2 headspace auto-sampler. Any volatile organic compounds present are partitioned into the headspace in the vial.

#### Analysis

After 40 minutes of shaking, a volume of the headspace is sampled and injected into a Gas Chromatograph (GC) and is analysed by temperature programmed capillary chromatography and Mass Selective Detection (MSD). Identification is performed using the SIM Mode.

Quantification of the components present is carried out by means of the internal standard with relative response factors calibration routine, using deuterated n-Butanol as an internal standard. The relative response of the detector to the various components is taken into consideration in the calculations together with any dilution factors, which may be required.

Analyse the calibration standards, blanks, AQC's, samples and drift checks according to the GC-MS and headspace sampler conditions given in Tables 2a and 2b.



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Table 2a GC/MS Operating Conditions

<b>Inlet</b> Type Gas saver Temperature Split Ratio Split Flow	Split Off 175°C 2:1 4 ml/minute
<b>Column</b> Material Length x ID x Film Thickness Stationary Phase Suitable Supplier  Mode Initial Flow	Fused Silica Capillary Column 30m x 0.32mm x 1.8µm DB624 Agilent  Constant Flow 2 ml/minute
<b>Oven</b> Column oven	40°C Isothermal for 4.0minutes 40-220°C Programmed at 40.0°C/minute
<b>Detector</b> Acquisition Mode Resolution Run time	MSD SIM Low 13.40mins

Table 2b Agilent 7694 Headspace Sampler Conditions

<b>Zone Temperatures</b> Oven Loop Transfer Line Shake	80°C 150°C 150°C High
<b>Event Times</b> GC Cycle Time Vial Eq. Time Pressurization Time Loop Fill Time Loop Eq. Time Inject Time	16.0 minutes 40 minutes 0.2 minutes 0.25 minutes 0.08 minutes 0.50minutes

### **Interferences**

Some compounds of interest, such as methanol, acetone and isopropyl alcohol can be present in the atmosphere of the laboratory. Vials, standard vials and water bottles may become contaminated once they are exposed to the atmosphere, and care should be taken that samples are stored and prepared in areas free from these solvents.

Any interference by such compounds or any external contamination of water, standards or glassware can be detected by analysis of method blanks. Raw GC-MS data from all deionised water blanks must be evaluated for interferences. Any source of interference must be determined and corrective action taken to eliminate the problem where appropriate.