

***Determination of volatile organic compounds (VOCs) in environmental water samples using Purge & Trap coupled to Gas chromatography-Mass spectrometry***

The scope of this experimental project/application is the transfer of knowledge regarding to the analysis of purgeable volatile organic compounds (e.g. halocarbons) in environmental water samples using the advanced technique of purge & trap coupled to GC-MS. Through this experimental course the students will have the opportunity to learn some aspects about the instrumentation, method validation and analysis of real samples as well. This project will include four distinguishable experimental sections:

- 1) *Sample preparation*: Due to the nature of the specific analysis automated purge & trap is the predominant sample pretreatment technique. A detailed explanation will be given on the theoretical and practical aspects on this technique including the demonstration of the operating software.

A typical experimental pathway includes the following general steps:

- Purging of 5 mL sample using He at a flow rate of 40 mL/min for 11 min
- Desorption of the trapped analytes at temperature of 250 °C with a flow rate of 300 mL/min



- 2) GC-MS: in this step courses will be provided both on the theoretical and the operational aspects of GC-MS including software handling. The split ratio on the inlet will be optimized in the range of 1/100 to 1/20 in order to obtain the maximum separation efficiency and sensitivity. The oven temperature will be linearly increased from 140 to 160 °C at a rate of 3 °C/min and from 160 °C to 220 °C at a rate of 20 °C/min and hold at this temperature for 3 min. The carrier flow rate will be 1.5 mL/min while the transfer line temperature will be set at 240 °C. All separations will be carried out on GC-GasPro (30 m x 0.320 µm) capillary column.

Looking to the MS parameters the temperature of ion source and the quadrupoles will be adjusted to 230 °C and 150 °C respectively. The quantification of the analytes will be performed in SIM mode. The retention time and the SIM segments are shown in the following table.

Analyte	Retention time (min)	SIM segment start time (min)	Quantification ion	Qualification ion Q1	Qualification ion Q2
chloroform	4,46	4	83	85	
1,1,1,-trichloroethane	6,54	5	97	99	61
Bromodichloro methane	6,62		83	85	129
1,2-dichloroethane	7,53		62	98	
1,4-difluorobenzene (ISTD)	7,85	7,6	114	88	63
Dibromochloro methane	8,592	8,2	129	127	
Bromoform	10,253	9,5	173	175	252
1,1,1,2-tetrachloro ethane	10,894	10,5	131	133	119



- 3) Method validation: method validation parameters including linearity, precision and accuracy, LOD and LOQ will be investigated and discussed.  
Calibration curves will be constructed analyzing a series of standard solutions in the range of 1 to 20 ppb. Statistical evaluation of the calibration curve will be conducted while a set of experiments will be devoted on the investigation of the intra-day reproducibility and to calculate the LOD and LOQ values.
- 4) Application: the final step involves the analysis of real and/or spiked samples while a significant part of this lecture will be focused on the data interpretation and evaluation.

