

SOLVING ODOR EPISODIES BY HRGC/HRMS CLOSE LOOP STRIPPING ANALYSIS (CLSA) IN NATURAL WATER.

Gomez-Lopez, Mariano, Vazquez, Maria-Jose.

LABAQUA-SANTIAGO. Rúa Secundino López 1. 15702-Santiago de Compostela (Spain).

Introduction: The episodes of odour and flavour in natural and drinking waters are one of the main concerns between drinking water supplier companies and besides, it are the main source of complaint between the drinking water users. In general, the episodes are due to the presence of organic compounds with low molecular weight, low boiling point, low solubility in water and usually apolars that are called volatiles. Without considering (Considering apart) the inputs of organic contaminants with natural origin (i.e. Geosmine), we are in the presence of water quality alterations directly made by the man. They can have the origin in direct spills (meaningful or not) or due to different causes such as incorrect use of materials, bad use/maintenance of installations or presence of sediments in (the case of) drinking water tank.

The present work is the compilation of six examples where the combination of the extraction technique of CLSA with the analysis by CG/MS allowed the identification of compounds that caused the episodes of odour and flavour in natural and drinking waters.

Experimental section: Closed Loop Striping Analysis (CLSA): Analyses were carried out according to the method previously described (1) in a commercial CLSA apparatus (Brechtbüler, Switzerland). 1 L of water samples was spiked with 1-chloroalkanes: C₅, C₆, C₁₀, C₁₂, C₁₆ and C₁₈ (Fluka Switzerland) to give a final concentration of 800 ng/L for each compound. We used 5 mg activated carbon filters to trap organic compound stripped from the water during 1 h. Temperatures of 45°C and 55°C were used for water-bath and carbon filter, respectively. The filters were spiked with C₈ and C₁₄ 1-chloroalkanes (Fluka Switzerland) at 800 ng/L for each compound (final concentration). Filters were then extracted with Carbon disulfide (Merck, Germany) to obtain a final volume of 40 µL.

HRGC-HRMS analysis (Instrumental Conditions): HRGC/HRMS analyses were carried out on a TRACE-MS/TRACE-GC 2000 (Thermo Quest, USA). 1 µL of CLSA extracts was injected "cold on column" (5 s) into a 50 m x 320 µm i.d. x 0.25 µm film CP-Sil 19CB fused silica column (Chrompack, The Netherlands). The GC temperature program was 30°C (5 min) to 280°C (10 min) at a rate of 3°C/min. Helium (42 cm/s) was the carrier gas. Mass spectrometer was operated in IE mode (70 eV) scanning from 35-450 Da with 1 s decade. Transfer line and ion source temperatures were 200°C and 250°C, respectively.

Odor episodies:

1) Hydrocarbon spill in drinking/natural water: as a result of an accident of a tank it was necessary suspend the production and provision of the potable water. The sample analysis by CLSA/HRGC/MS showed a linear hydrocarbon bell (figure 1) characteristic of the fuel-oil.

- 2) Episode of bad ODOR (scent) in the drink water of one builds. The analysis by CLSA/HRGC/MS allowed assigning the episode to a mixture OF BRANCHED (graft) alcohol of 10 and 11 carbon atoms, with great industrial use. The episode was consequence of migrations produced by (the) bad use of pipes. PVC pipes usually used in sewage system instead of pipes apt for potable water conduction were installed.
- 3) Episode of bad flavour in packaged mineral water in tetra brick: The analysis by CLSA/HRGC/MS showed the presence of the 1,4-dichlorobencene as the agent who originated the episode due to a deficiency in the manufacture of the package. The 1,4-dichlorobencene is used as solvent in the manufacture of tetra brick for the union of its different layers.
- 4) Episode of bad water odour in a well used for supplying one urbanization, by a strong odour to garlic. The analysis by CLSA/HRGC/MS showed the presence of a high number of compound with sulphides: di-i-propil-disulfide. The origin of these compounds was assigned to a breakage of a propane gas deposit of the urbanization. The compounds with sulphur are used as additives of gases of domestic use to produce odour and to detect them
- 5) Episode of bad odour in well water (well lower than 10 m of depth) man used. The analysis by CLSA/HRGC/MS showed the presence of bencene, toluene, xylene and alkyl-bencenes, characteristic compounds of gasoline. The well contamination was due to the breaking of a gasoline deposit, which produce the superficial contamination of the aquifer.
- 6) Episode of complaint by bad odour in drinking water of a land. The analysis by CLSA/HRGC/MS showed the presence of toluene, xylenes, alkyl-bencenes and aldehydes. The episode research assigned these compounds from migrations of rubber used between different sections of pipes that are not suitable for such purpose

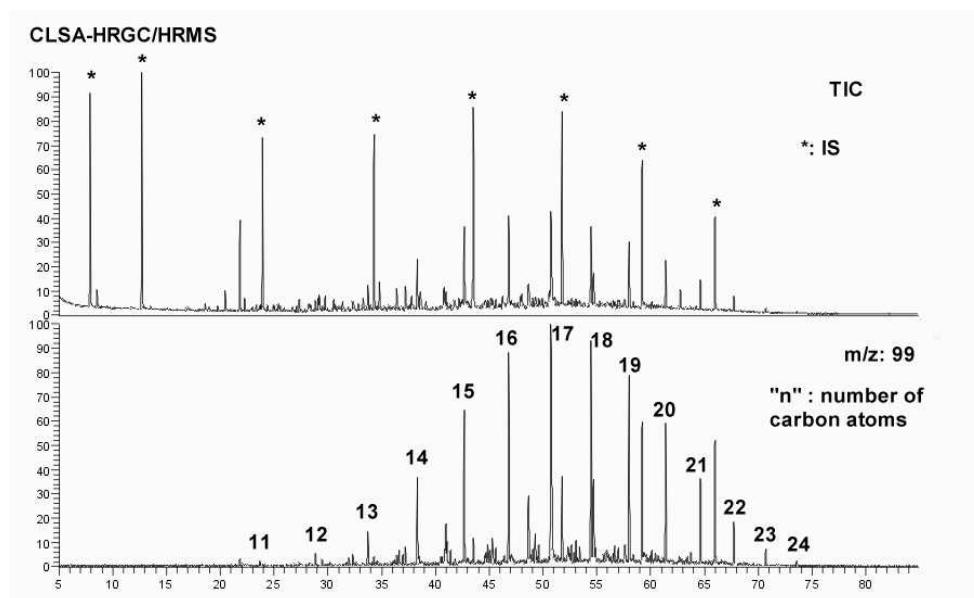


Figure 1

1.- Romero J., Manero I. and Laso J. (2005). Instrumental method accreditation for Geosmin and MIB by CLSA/HRGC/FID. Accreditation and Quality Assurance (submitted)