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A new purge-and-trap headspace technique to analyse volatile organohalogens from fluid inclusions of rocks and minerals

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Recently, advances were made in the detection of low boiling point volatile organohalogens (VOCs) at trace gas concentrations of air samples employing sophisticated and complex experimental setups (Sive et al. 2005, Miller et al. 2008) while conventional fluid inclusion gas analysis via GC/MS (gas chromatography coupled with mass spectrometry) do not include halogenated VOCs in their analytical routine (e.g. Samson et al. 2003).
At the same time Svensen et al. (2009) have just confirmed the release of chlorinated and brominated VOC from halites after heat treatment using GC/MS into which they injected compounds previously trapped on adsorption tubes. Already in 1998, Harnish and Eisenhauer reported the presence of CF4 and SF6 released from natural fluorite and granite samples after milling but appear to have received little resonance in the scientific community.
In this work we present the development of a new method for the analysis of halogenated VOCs from fluid inclusions. The mineral or rock sample is milled in an air-tight tempered steel container that fits into a regular planetary mill. Starting at a particle size of around 2-3 mm a final mean particle fineness of 1000 to 750 nm for quartz and fluorites, respectively, is achieved. The grinding container is equipped with two septa that can be pierced by the two needles through which the sample is connected to the GC/MS system and through which the gases are purged similar to a standard purge-and-trap system. The gases are trapped at liquid nitrogen temperatures before entering directly onto the column of the GC/MS system. Compounds that were released during grinding are separated and detected by an ion trap mass spectrometer. To prevent contamination with fine particles a 0.5 μm sintered steel filter element is interconnected after the sample needle.
Optimizations and calibrations were conducted using diluted pure gases.
First results show that this modified GC/MS purge-and-trap method appears to be an effective, simple and relatively low cost alternative for VOC analysis that opens up new territories in GC/MS analysis of fluid inclusions.