

# **THERMAL DESORPTION GAS CHROMATOGRAPHIC METHOD FOR TRACE LEVEL MEASUREMENTS OF HYDROGEN SULFIDE AND OTHER REDUCED SULFUR GASES**

DEVAI, I., DELAUNE, R.D. and GAMBRELL, R.P.

Wetland Biogeochemistry Institute, Louisiana State University  
Baton Rouge, LA 70803-7511, U.S.A.

Methods for measuring natural (emission from both terrestrial and marine ecosystems) and industrial sources of hydrogen sulfide, carbonyl sulfide, carbon disulfide and methylated sulfur compounds such as methanethiol and dimethyl sulfide are reported.

Due to the generally low concentration of these volatile reduced sulfur compounds in ambient air, especially in rural and remote areas (like reservoirs, tailwaters, etc.) a preconcentration step is required before gas chromatographic analysis for determining natural and anthropogenic emissions. In the past reduced sulfur gas emissions measurements have relied mainly on cryogenic sample collection or metal foil adsorption. In the last decade methods reported in the literature widely used adsorption on solid adsorbents followed by thermal desorption and gas chromatography for the collection of volatile organic compounds in air.

Reported are: (1) trapping efficiencies of selected solid adsorbent materials for collecting field emission samples of trace levels of reduced sulfur gases and (2) thermal desorption gas chromatographic method for the separation and analysis of hydrogen sulfide, carbonyl sulfide, methanethiol, dimethyl sulfide and carbon disulfide.

The trapping efficiency of fourteen solid adsorbent tubes for sampling trace levels of these volatile reduced sulfur gases was quantified. Results demonstrated that silica gel and molecular sieves (in conjunction with a calcium chloride drying tube) were the best solid adsorbent materials for trapping reduced sulfur gases if the sweep gas contains moisture (i.e., normal field sampling conditions).

During the laboratory analysis the desorption apparatus was connected directly to the gas chromatograph equipped with a 1.4 m x 1.8" OD Teflon (FEP) column packed with 40/60 Carboxpack B HT 100 and flame photometric detector. The oven temperature was programmed from -20 °C to 75 °C at 30 °C/min rate (using a liquid carbon dioxide subambient accessory) with initial 2 min delay and 3.4 min hold at the end. Adequate separation, resolution, calibration and stable retention times of analyzed volatile reduced sulfur compounds were obtained using this optimized thermal desorption and gas chromatographic method. These methods were successfully used to measure natural and anthropogenic emissions of reduced sulfur gases and in laboratory experiments where the efficiency of various oxidants for controlling emission of hydrogen sulfide from sewage sludge was demonstrated.