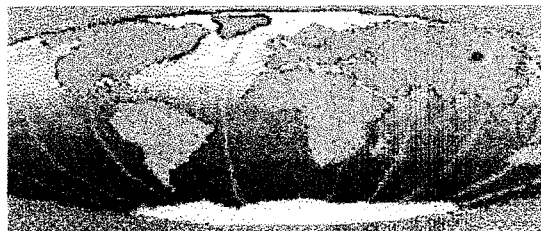


ISGC'01

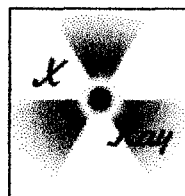
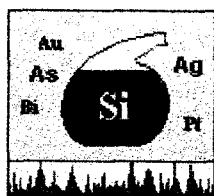


IRKUTSK, JULY 23-27, 2001

RUSSIAN ACADEMY OF SCIENCES
Scientific council of analytical chemistry
SIBERIAN BRANCH
Institute of Geochemistry
Institute of Earth's Crust
Institute of Inorganic Chemistry
United Institute of Geology, Geophysics and Mineralogy

**THE SECOND
INTERNATIONAL
WORKSHOP SIBERIAN
GEOANALYTICAL
SEMINAR
«I N T E R S I B G E O C H E M '01»
July 24-26, 2001, Irkutsk, Russia**

ABSTRACTS



Irkutsk 2001

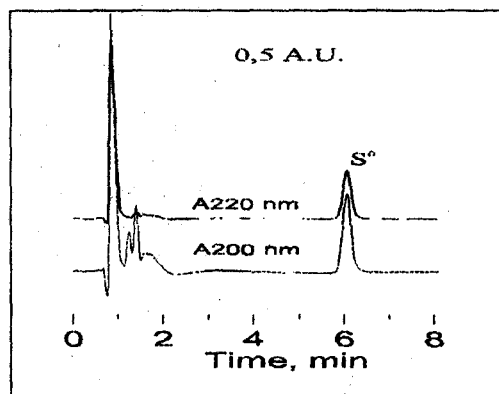
Application HPLC for Analysis Elemental Sulfur in Sediments

A.G.Gorshkov, I.N.Azarova, G.I.Baram

Limnological Institute SB RAS,
Correspondence, P.O. Box 4199, Иркутск-33,
664033, Russia
E-mail: agg@lin.irk.ru

Thanks to its unique properties is a very sensitive indicator of physical and chemical conditions of formation and diagenesis of bottom sediments. Since sulfur can exist both in extremely oxidized and in extremely recovered state it occurs in the sediments as sulfates SO_4^{2-} or sulfites S^{2-} as well as elemental sulfur S^0 and can be within organic compounds. A classical method of analysis that allows determination of all the main forms of sulfur compounds is rather time-consuming and has some bottlenecks. Thus, due to possible surplus of S^0 content due to sample acidification at the first stage when determining the sulfides, S^0 should be analysed using a separate sample by its extraction from the sediment with acetone. Further quantitative analysis suggests the use of gravimetric, titrimetric and photometry methods after S^0 transfer into the forms convenient for analysis, that is SO_4^{2-} , SCN^- , $\text{S}_2\text{O}_3^{2-}$ или S^{2-} .

Determination of S^0 in the solutions of organic solvents can be done using chromatography methods whereas HPLC method alone makes it possible to register sulfur cycles with different number of atoms in the "native" form. Our experience of studying the bottom sediments of Baikal Lake revealed that HPLC can be easily adapted to S^0 determination. It should be stressed that application of HPLC allows the sample to be prepared using a more simple pattern and analyze the crude acetone extracts using a direct input into the chromatograph column.



Crude extract of the core sample (the layer 168-173 cm, Southern Lake Baikal), VER 99-2 St 6, GC, N 52°05'20''; E 105°50'26''). The peak elemental sulfur, $t_R=6.0\pm 0.2$ min, $S_{220}/S_{200} 0.593\pm 0.011$. Content of $\text{S}^0 1.0\pm 0.4$ $\mu\text{g/g}$.

HPLC was performed with the liquid chromatograph "Milichrom A-02", EcoNova, Ltd. Novosibirsk, Russia [1]. Column 2x75 mm with Nucleosil 100-5 C_{18} PAH (Macherey-Nagel, Germany) was used. Isocratic elution was carried out with mixture acetonitrile:water 75:25. UV detection was performed at 200 and 220 nm, simultaneously; flow rate was 0.2 ml/min; the column temperature was kept as 45 °C.

The sample volume was 10 μl .

Conditions of isocratic elution make it possible to separate the sulfur peak from the peaks of associate components of the extract with high resolution and two-wavelength detection allows its identification with a high degree of certainty. Chromatography on the micro column raises the sensitivity by the order of magnitude. Within the range of detection of 200-220 nm the limit of sulfur detection makes 5 ng/peak under the ratio of signal/noise 3:1. Qualitative analysis is characterized by high reproducibility of the results. Relative standard deviation in the quantitative determination of elemental sulfur in the homogenized material of bottom sediments accounts for $S_r = 5.6\%$.

Data of quantitative determination of S^0 evidence a considerable non-homogeneity of its distribution in the cores of bottom sediments of Baikal Lake, i.e., from 4 to 1200 $\mu\text{g/g}$ (dry sediment) both in terms of macro non-homogeneity and micro non-homogeneity of an individual core layer. Therefore, sampling of a part of a non-homogenized layer only results in wide confidence interval of the analysis results. With view of that two sampling methods have been proposed. The first one consists in core division at a step of 1 cm and less and sulfur extraction from the whole sampled layer. The second one suggests thorough averaging of the studied core section before sampling.