



IN REPLY REFER TO:

United States Department of the Interior

U.S. GEOLOGICAL SURVEY

Box 25046 M.S. 407

Denver Federal Center

Denver, Colorado 80225

NATIONAL WATER QUALITY LABORATORY TECHNICAL MEMORANDUM 1994.11

May 4, 1994

To: Assistant Chief Hydrologist for PC&TS
Regional Hydrologists
Chief, Office of Water Quality
Assistant Chief, Office of Water Quality
Deputy ACH for PC&TS for NAWQA
Area Hydrologists
District Chiefs
Regional Water-Quality Specialists
Assistant Regional Hydrologists for NAWQA
District Water-Quality Specialists
Chiefs, NAWQA Study-Units
Chief, Ocala Project Office
Chief, Yucca Mountain HIP
QA Manager, Yucca Mountain Project
NWQL Radiological Advisory Committee
Chief, Branch of Quality Assurance
Employees, National Water Quality Laboratory

From: Peter F. Rogerson, Chief
National Water Quality Laboratory
Branch of Analytical Services

Subject: New lab codes for sulfur isotope (δ -34 S) analysis

Author: Ann H. Mullin, Radchem Unit, Quality Mgmt. Program AHMULLIN (303) 467-8235

Revision: None

SCOPE

Since October 1, 1992, the Reston Stable Isotope Laboratory (RSIL) has been analyzing sulfur isotope (δ -34 S) samples for the National Water Quality Laboratory (NWQL). Recently the NWQL received a request from the RSIL to change the price structure for these samples.

Following is the text of the memorandum received from the RSIL explaining the request:

"Processing samples submitted to NWQL in 1993 for sulfur isotope (δ -34 S) analysis involved a variety of extraction techniques and a wide range in the number of preparation hours required per sample. The majority of samples, submitted as liters of water with appropriately high sulfate concentration, required relatively short processing time. On the other hand, a minority of the samples submitted with a variety of complicating factors including unusually low sulfate concentration (<20 mg/L), high dissolved organic material, H₂S still in solution, and those samples submitted as unprocessed rocks required an additional amount of time and effort to prepare.

"As a result, a new basic price list will be in effect for all future samples submitted to NWQL according to the sample type and analysis requested. The basic types are (1) δ -34 S analysis of dissolved sulfate for water samples with >20 mg/L sulfate, (2) δ -34 S analysis of sulfate collected on ion-exchange resin from water with <20 mg/L sulfate, (3) δ -34 S analysis of dissolved sulfide collected in the field as Ag₂S[1], (4) δ -34 S analysis of sulfate in a solid (rock, sediment, or mineral) sample[2,3] and (5) δ -34 S analysis of sulfide in a solid (rock, sediment, or mineral) sample[2]. If a particular sample requires processing in addition to that included in these basic techniques[2,3,4], additional charges will be assessed.

"There are several things that researchers can do to minimize charges for these samples and assure the best possible analyses. (1) Report the sulfate concentration of the sample on the ASR form. It is very important for us to know whether your sample has 10 or 1,000 mg/L sulfate. (2) If a sample contains dissolved sulfide, strip[1] the sample in the field, even if the sulfate concentration is high enough that you are not worried about contamination of the sulfate with oxidized sulfide. If a sample is submitted with sulfide in solution, it will be stripped in the lab and a charge assessed. We cannot process samples that are emitting toxic H₂S gas. (3) Samples with an off color or odor will be pretreated to remove the organic material before the sulfate is precipitated as BaSO₄. Although a charge is assessed for this additional service, failure to pretreat such samples results in contaminated BaSO₄ precipitate which cannot be analyzed. If you know the DOC content of sample, this can be included on the ASR form. (4) If submitting solid (rock, sediment, or mineral) samples for δ -34 S analysis, the sample should be crushed to pass a 200-mesh (74-micron) sieve. Call Rebecca Carmody in Reston at (703) 648-6338 if you have questions about submitting a particular type of sample."

([1,2,3,4] Please see references at end of document)

RSIL is instituting the following changes which will become effective June 1, 1994. NWQL will change prices as directed by RSIL.

Lab codes 535, 298, and 1138, currently in use for sulfur isotope analysis, will become invalid. They will be replaced by the following:

1. Lab Code 1951.- Sulfur isotopic analysis of dissolved sulfate for sulfate concentrations >20 mg/L. Amount of material required - sufficient sample for 30 mg sulfate.

2. Lab Code 1949.- Sulfur isotopic analysis of low sulfate concentration (<20 mg/L) sample collected on ion-exchange resin. Amount of material required - ion exchange resin containing >30 mg sulfate. (The ion exchange resin can be obtained by contacting the Radchem Unit at the NWQL.)
3. Lab Code 1948.- Sulfur isotopic analysis of dissolved sulfide in water; sample submitted as Ag₂S. Amount of material required - 30 mg Ag₂S
4. Lab Code 1950.- Sulfur isotopic analysis of sulfate in rock or mineral; sample submitted crushed to finer than 200 mesh. Amount of material required - 30 mg as sulfate.
5. Lab Code 1947.- Sulfur isotopic analysis of sulfide in rock or mineral (total sulfide, monosulfide, or disulfide; sample submitted crushed to finer than 200 mesh). Amount of material required - requires rock sample containing >4 mg of S as sulfide in order to make 30 mg Ag₂S.
6. Schedule 921 consisting of Lab Codes:
 - 1952.- Sulfur isotopic analysis of sulfate
 - 1953.- Sulfur isotopic analysis of disulfide
 - 1954.- Sulfur isotopic analysis of monosulfide.The three isotopic compositions will be reported on one sample (sample submitted crushed to finer than 200 mesh). Amount of material required - sufficient sample for 30 mg sulfate; monosulfide and disulfide concentrations to provide 30 mg as Ag₂S, each.

The NWQL understands the difficulties that these add-on charges will cause and regrets the need to impose them. However, all of these additional charges can be avoided with proper collection and preparation of samples in the field.

All of these changes will be implemented June 1, 1994.

Effect on Data: None

Supersedes: None

Key Words: Isotopic, monosulfide, disulfide

Distribution: See above plus the continua USGS.labnews, .water.quality, and .radchem

References:

[1]Carmody, R.W.; Busenberg, E.; Plummer, L.N.; and Coplen, T. (1994) Field procedures for collection of dissolved sulfide and sulfate, unpublished manuscript, 29 pp.

[2]Bates, A. (1993) Procedures for Sulfur Analyses, unpublished manuscript, 4 pp.

[3]Busenberg, E. (1987) Procedure for converting CaSO₄ to BaSO₄, unpublished, 1p.

[4]Carmody, R.W. (1994) Laboratory techniques for analysis of sulfide and sulfate from ground and surface waters, unpublished manuscript, 8 pp.

If you would like copies of any of these references, please call Rebecca Carmody.