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U.S. Geological Survey Standard Reference Sample Project—Performance Evaluation of Analytical Laboratories

INTRODUCTION

Since 1962, the U.S. Geological Survey (USGS) has operated the Standard Reference Sample Project (SRSP) to evaluate the performance of USGS, cooperator, and contractor analytical laboratories that analyze chemical constituents of environmental samples. The laboratories are evaluated by using performance-evaluation samples, which are called Standard Reference Samples (SRSs). SRSs are submitted to laboratories semi-annually for round-robin laboratory performance comparison purposes. In an interlaboratory comparison study, which was done during the winter of 1997, 146 laboratories were evaluated for their analytical performance on nine SRSs for inorganic and nutrient constituents. As part of the SRSP, a surplus of homogeneous, stable SRSs is maintained for distribution to USGS offices and participating laboratories for use in continuing quality-assurance and quality-control activities.

Statistical evaluation of the laboratory SRSs results provides information to compare the analytical performance of the laboratories and to determine possible analytical deficiencies and problems. SRS results also provide information on the bias and variability of different analytical results and methods used in the SRS analyses.

Although the SRSP is not a certification program, participation is required for all laboratories that provide water-quality data for the USGS. The participating laboratories are identified only by a confidential code number. Laboratory personnel select the SRSs to analyze and must include the analyses that are routinely performed for USGS investigations. Analytical methods of choice can be used by these laboratories to analyze the SRS constituents.

TYPES AND PREPARATION OF THE STANDARD REFERENCE SAMPLES

USGS personnel prepare the following types of SRSs from different natural-matrix water sources throughout the United States:

Trace SRS: surface water with trace and major inorganic constituents.

Major SRS: surface water with major inorganic constituents.

Nutrient SRS: surface and de-ionized water with nutrient constituents.

Low ionic strength SRS: snowmelt with major inorganic constituents.

Mercury SRS: surface water with mercury.

Acid mine water SRS: acid mine water with trace inorganic constituents.

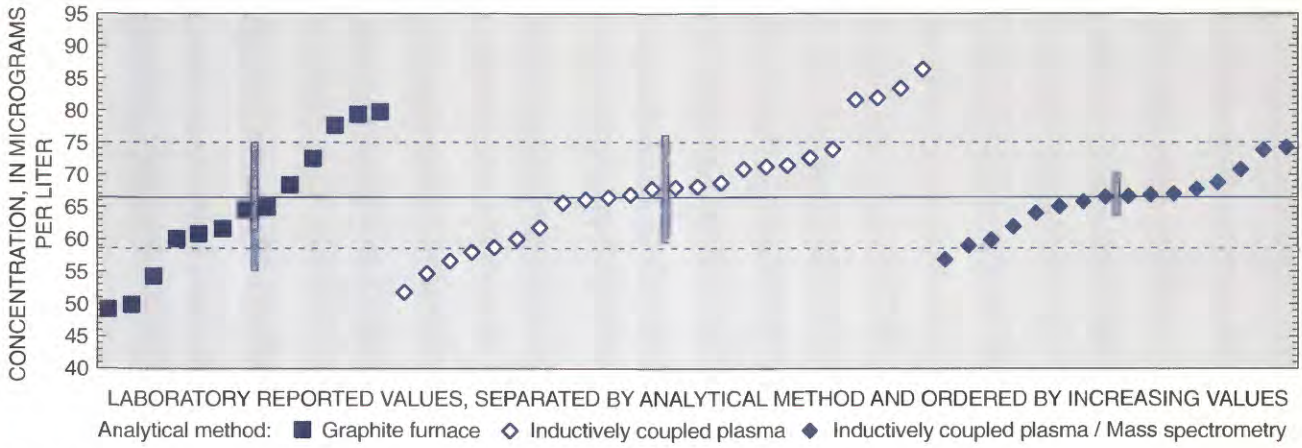
Ground water SRS: ground water with major and trace inorganic constituents.

The SRSs are prepared by pumping the water through a series of 0.45-, 0.2-, and 0.1-micrometer filters and ultraviolet sterilizer into a polyethylene drum. When appropriate, the water is either acidified with nitric or sulfuric acid or chlorinated to 5 parts per million free chloride or both. For some types of SRSs, selected constituents are spiked in the water to desired concentrations with reagent-grade chemicals. The water is then circulated for at least 24 hours. During SRS bottling, the water is pumped through an ultraviolet sterilizer and a 0.1-micrometer filter. The bottles and caps used for SRSs are acid leached, de-ionized water rinsed, and autoclave sterilized.

ANALYSIS AND REPORTING OF THE SRS RESULTS

The results of the SRSP inter-laboratory performance evaluations are compiled and entered into a computer data base and evaluated by using non-parametric statistics. Data for each SRS constituent are tabulated in descending order, and the median (midpoint of all the values) is considered the most probable value (MPV). The midpoint of the values greater than the median is called the upper hinge (Hu); the midpoint of the values less than the median is called the lower hinge (Hl). (The Hu and Hl are similar to quartiles but are not mathematically equivalent.) The range

of data between Hu and Hl, which includes the middle 50-percent of the data, is called the hinge spread (Hspr) and is used to calculate the sample-data deviation, the F-pseudosigma. The F-pseudosigma is equivalent to the sample standard deviation when the sample data have a normal statistical distribution. Non-parametric statistics are best used when the sample data include a few outlier values (Hoaglin and others, 1983). A USGS Open-File Report is prepared for each SRSP interlaboratory evaluation and includes graphs and tables that summarize laboratory performance for each SRS constituent and a table that summarizes the overall laboratory performance (Farrar, 1998). For each SRS constituent, the following type of graphical plot is prepared:



Graphical plot of selected aluminum data for a trace standard reference sample.

The upper and lower boundaries in the above graph at 40 and 95 micrograms per liter are at +3 and -3 F-pseudosigmas from the MPV. The MPV (solid horizontal line) and +/- 1 F-pseudosigma deviations (dashed lines) are determined from the constituent-pooled data. The vertical bar, for each ordered analytical method, extends +/- 1 F-pseudosigma from the MPV for that method.

LABORATORY PERFORMANCE RATINGS

To facilitate the comparison of analytical performances in the SRSP interlaboratory evaluations, a laboratory performance rating is determined from the absolute value of F-pseudosigma, which is the deviation of the laboratory's reported value from the pooled-data MPV. The following rating criteria are used:

Performance rating	Number of F-pseudosigmas
4 (Excellent)	0.00 to 0.5
3 (Good)	0.51 to 1.0
2 (Satisfactory)	1.01 to 1.5
1 (Marginal)	1.51 to 2.0
0 (Unsatisfactory)	Greater than 2.0

These ratings are based on the performance of laboratories in analyzing specific SRS constituents and are used to compare the analytical performance of the participating laboratories on different natural-matrix sample constituents. USGS users of the laboratories can evaluate how these laboratory performances meet their specific project requirements for the type and quality of analytical data needed.

REFERENCES CITED

Farrar, J.W., 1998, Results of the U.S. Geological Survey's analytical evaluation program for standard reference samples: T-151 (trace constituents), M-144 (major constituents), N-55 (nutrient constituents), N-56 (nutrient constituents), P-29 (low ionic strength constituents), GWT-2 (ground-water trace constituents), GWM-2 (ground-water major constituents), AMW-4 (acid mine water constituents), and Hg-25 (mercury) distributed in September 1997: U.S. Geological Survey Open-File Report 98-52, 191 p.

Hoaglin, D.C., Mosteller, F., and Tukey, J.W., 1983, Understanding robust and exploratory data analysis: New York, N.Y., John Wiley & Sons, Inc., p. 38-41.

For additional information, contact:
Standard Reference Sample Project
U.S. Geological Survey
Branch of Quality Systems
P.O. Box 25046, DFC, MS 401
Denver, CO 80225-0046
(303) 236-1870, ext. 316 or 313

Prepared by H. Keith Long, Richard L. Daddow, and Jerry W. Farrar

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