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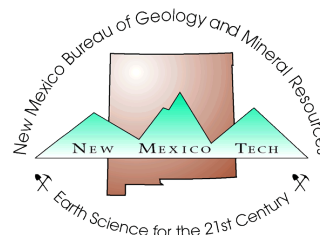
Lynn A. Brandvold

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Reliability of gold and silver analyses by commercial laboratories in the Southwest

by Lynn A. Brandvold, Chemist, New Mexico Bureau of Mines & Mineral Resources, Socorro, NM

Introduction

The rapid rise in the price of gold and silver has led to an increased interest in prospecting for these metals. Because of the high price of gold, an ore that contains 0.05 to 0.1 oz of gold per ton may be profitably mined if large tonnages are available.

When the price of gold was relatively low, determining if a rock contained enough gold to be profitably mined was easy: if the gold could be seen with a magnifying glass, the ore was probably worth mining. Now with the high price of gold, ore containing highly disseminated gold, invisible even under a microscope, may be profitably mined. Consequently, prospectors and small-scale miners must rely on commercial laboratories to determine ore values.

With the exception of a few firms that use a direct atomic-absorption technique, most commercial laboratories in the southwest determine gold and silver values by fire assay; a method for determining metals by using furnace heat and dry reagents, used for gold and silver analysis for thousands of years. A routine commercial fire assay is not run in duplicate and costs between $5 and $10 depending upon the laboratory. If a prospector is selling ore to a smelter, his assay must agree with the smelter's assay within certain limits termed 'splitting limits' (in the case of one well-known smelter, to within 0.02 oz/ton of gold and within 0.5 oz/ton of silver). If assay comparisons differ by more than these limits a control sample must be submitted to an umpire assay unless one wishes to accept the smelter assay. The losing party must stand the cost of the umpire assay. A commercial umpire fire assay, run in quadruplicate, costs two to three times the routine assay price. Because most prospectors must rely on commercial laboratories, the question arises: how reliable *are* routine gold and silver assays? To seek an answer to this question and also to provide an in-house reference sample, the New Mexico Bureau of Mines and Mineral Resources undertook this study in 1977-78.

Methods

A 50-lb sample was donated to the project from a small gold-silver mine near Winston in south-central New Mexico. The sample was approximately 90 percent quartz with small amouts of chlorite, argentite, and fine-grained pyrite.

The ore was crushed and ground so that it would pass an 80-mesh screen. Standard techniques of ore mixing and splitting were used to insure a homogeneous sample. The sample was finally divided into 32 splits. Each split was placed in a separate container.

As a check for homogeneity, three containers were chosen at random and rolled overnight to counteract any settling. A 14.5833-g sample (½-assay-ton) was taken from each container and the samples fire-assayed in the Bureau's analytical laboratory.

To assure each laboratory an adequate working sample, 75 g was sent to each. Samples were prepared in the following manner: Three sample containers were chosen at random. The bottles were emptied onto a plastic sheet and the contents

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Announcements

Items in this column are the responsibility of contributors. Times, dates, and locations are published as received.

NINTH INTERNATIONAL CONGRESS OF CARBONIFEROUS STRATIGRAPHY AND GEOLOGY,
May 10-June 2, 1979,
Washington, D.C. and Urbana, Illinois

Includes: Plenary session, subcommission meetings of International Commission on Stratigraphy, working groups of International Commission on Coal Petrology, technical sessions and symposia, and 24 field trips in eastern and western United States. Field Trip No. 12, Carboniferous of southern New Mexico, May 27-June 2, is being led by J. L. Wilson and F. E. Kottlowski.

Inquiries should be addressed to:
Ellis L. Yochelson, Secretary-General
IX-ICC, 1979
Museum of Natural History
Washington, D.C. 20560

Announcement and call for papers

THE FOUR CORNERS GEOLOGICAL SOCIETY will conduct their 1979 field trip, "Field Symposium on the Permian System of the Colorado Plateau" September 27-30, 1979. The areas to be covered include Canyonlands, Monument Valley, Grand Canyon, and the Mogollon Rim. A guidebook will be prepared for the occasion. If you or your colleagues or students have been researching the Permian rocks of the Colorado Plateau and wish to contribute to the guidebook, please contact D.L. Baars, Fort Lewis College, Durango, CO 81301, phone 303-247-7767. Tentative titles were to be submitted by January 1; manuscript deadline is June 1, 1979.

Announcement and call for papers

THE FORT WORTH GEOLOGICAL SOCIETY plans to host the Southwest Section Regional AAPG Convention in May, 1979. They need your assistance to help provide papers for this convention. The theme will be the three R's of re-evaluation, reworking, and revival. The convention will be held at the Sheraton Hotel in Fort Worth, May 9-11, 1979. Write to George H. Weems, Co-Chairman, Technical Program, Bass Enterprises Production Co., Fort Worth National Bank Building, Fort Worth, TX 76102.

Symposium

The Energy Minerals Division of the AMERICAN ASSOCIATION OF PROFESSIONAL GEOLOGISTS AND THE NEW MEXICO BUREAU OF MINES AND MINERAL RESOURCES will be co-sponsoring a symposium on the Grants Uranium Region to be held at the Albuquerque Convention Center May 13-16, 1979.

Papers dealing with the uranium geology and mining technology of the N.W. quadrant of New Mexico will be featured. For further information, please contact: Dr. Christopher Rautman, Shell Development Company, Bellair Research Center, P.O. Box 481, Houston, TX 77001.

Santa Fe Geological Society

Luncheon Meetings resumed in January 1979 at the Forge Restaurant, Santa Fe.
February 16, 1979—Dr. Doug Brookins, University of New Mexico, Professor of Geology, *Geochemical Study of Grants Mineral Belt*
March 16, 1979—Ben Bader, Sandia Laboratories, *In-Situ Coal Gasification,—Hanna Project, Hanna, WY*
April 20, 1979—(?) Andy Livingston, Chief Geologist, Bokum Resources, *Depositional Model For U_3O_8, Marcus, NM*

mixed by rolling back and forth. The material was coned and quartered; opposite quarters were mixed and then combined. Samples of 75 g were then weighed into envelopes until all the material had been used. This produced 24 samples, all of which were sent to commercial laboratories. Samples were mailed to 10 laboratories under the name of the New Mexico Bureau of Mines and Mineral Resources. After all the results had been returned, two additional splits were mailed to each of four laboratories under different names and from different towns. This was done to compare reproducibility. A random split (not one of the aforementioned 24 samples) was sent to the chemical laboratory of a New Mexico mining company whose chief chemist had offered to determine gold values by fire assay and silver values by both fire assay and atomic absorption.

Results

The in-house homogeneity check produced values listed in table 1. The average for silver is 16.42 oz/ton with a mean deviation of 0.11. The average for gold is 0.23 oz/ton with a mean deviation of 0.02. These results indicate that the sample is homogeneous. Table 2 lists the results from the commercial laboratories that used the fire-assaying technique. Two of the original ten laboratories determined gold and silver by atomic absorption; these results will be considered later.

TABLE 1—RESULTS OF IN-HOUSE FIRE ASSAY TEST FOR HOMOGENEITY

Sample	Gold (oz/ton)	Deviation from average	Silver (oz/ton)	Deviation from average
1	0.21	0.02	16.25	0.17
2	0.26	0.03	16.58	0.16
3	0.22	0.01	16.44	0.02

Average = 0.23 Average = 16.42
Average mean deviation (gold) = 0.02
Average mean deviation (silver) = 0.11

TABLE 2—FIRE-ASSAY RESULTS FROM COMMERCIAL LABORATORIES

Laboratory	Gold (oz/ton)	Silver (oz/ton)
1	0.36	16.84
2	0.70	10.87
3	0.25	18.10
4	0.27	16.10
5	0.22	16.18
6	0.31	16.30
7	0.26	16.40
8	0.30	21.30
Mean =	0.33	16.51
Standard deviation =	0.15	2.87

Standard deviation of the silver assay represents $14.12 per ton assuming the

price of silver is $4.92 per troy oz—a difference in dollars between the highest assay and the lowest assay of $52.11 per ton. The standard deviation of the gold assay represents $31.05 per ton assuming the price of gold is $207 per troy oz—a difference in dollars between the highest and lowest gold assay of $99.36 per ton. Thus the margin for error in determining actual values on an ore shipment could result in substantial losses for a small operator. Better to rely on more than one assay.

An argument could be made that this difference in values was caused by nonhomogeneity between samples. However the in-house homogeneity check on three random samples resulted in an average deviation of 0.02 oz/ton for gold and 0.11 oz/ton for silver.

Furthermore, if the sample were nonhomogeneous, values obtained by atomic absorption would show much larger deviations because the sample size is smaller—15 or 30 g for fire assay (½ or full-assay-ton), whereas for atomic absorption the sample size is 1-5 g. Table 3 lists the atomic absorption results from the two commercial laboratories, the mining company laboratory, and four assays run by various students in the Bureau laboratory. The standard deviations for both gold and silver are much less than those of the commercial fire-assay determinations. Therefore nonhomogeneity of the samples was not an apparent factor in the difference between the determinations from the commercial laboratories.

TABLE 3—ATOMIC ABSORPTION RESULTS

Laboratory	Gold (oz/ton)	Silver (oz/ton)
9[1]	0.21	18.50
10[1]	0.25	19.00
11[2]	—	17.51
12[3]	0.28	17.60
13[3]	0.22	16.37
14[3]	0.22	16.62
15[3]	0.24	16.62
	mean = 0.24	mean = 17.46

Standard deviation (gold) = 0.026
Standard deviation (silver) = 1.01

[1]Commercial labs
[2]Mining company laboratory
[3]Bureau laboratory

The mean silver values obtained by fire assay from the in-house analyses are both lower than the mean silver value obtained by atomic absorption. This is understandable, considering the recognized silver losses during fire assay. When the mining company laboratory fire assayed the sample, a second sample of a known amount of silver was assayed simultaneously and

silver loss determined. The corrected silver value was 17.46 oz/ton—which agrees with the atomic absorption mean in table 3. Although commercial laboratories are certainly aware that silver is lost during fire assay, the low reported silver values indicate that the loss is probably not compensated in the results. A check with three of the eight laboratories confirmed that assaying losses are disregarded in the reported value. The reason given is that smelters do not accept corrected assays as a basis of payment for ores and concentrates, since they lose the same percent silver in smelting the ore.

The mean gold value obtained from atomic absorption (0.24 oz/ton) is much lower than that obtained by the commercial fire assayers (0.33 oz/ton). However, one of the fire assay values for gold (laboratory No. 8, table 2) is unusually high, more than 2 standard deviations from the mean. If this value is discarded, the resultant mean is 0.28 oz/ton. The in-house fire assay mean for gold was 0.23 oz/ton.

Many factors could account for the difference in fire assay values between laboratories: different analysts, different equipment, slightly different methods, and different chemicals. These variables can be eliminated if samples are sent to the same laboratory. Two more samples were sent to each of four laboratories to eliminate these variables and to check the reproducibilities of some of the commercial laboratories. Laboratory No. 2 was excluded because their gold and silver values were more than 2 standard deviations from the mean. This was done to avoid a bias in the reproducibility results. Results are shown in tables 4 and 5. Standard deviations were not calculated because there were only three values in each set. None of the laboratories would have met smelter limits on all three assays for gold or silver, but laboratories 3, 4, and 7 would have met smelter limits on two out of three of the gold assays and laboratories 3, 4, and 6 would have met smelter limits on two out of three of the silver assays. Eliminating the variables between laboratories and analysts improved the analyses, but not appreciably.

TABLE 4—TRIPLICATE FIRE-ASSAY VALUES FOR GOLD

Laboratory	Gold (oz/ton)	Set Mean	Set Range
3	.25 .23 .31	.26	.08
4	.27 .25 .32	.28	.07
6	.31 .21 .44	.32	.23
7	.26 .21 .48	.32	.27
	Overall mean (gold) = .30		

TABLE 5—TRIPLICATE FIRE-ASSAY VALUES FOR SILVER

Laboratory	Silver (oz/ton)			Set Mean	Set Range
3	18.10	16.80	17.90	17.60	1.3
4	16.10	16.90	17.10	16.70	1.0
6	16.30	14.45	15.95	15.57	1.85
7	16.40	15.60	12.55	14.85	3.85
	Overall mean (silver) = 16.18				

Table 6 lists all the fire assay results for silver and gold. A "reasonable" mean was calculated in the following manner: the means and standard deviations for gold and silver analyses were calculated in the usual manner. Those values which differed by more than 1 standard deviation from the means were rejected and new means were calculated. The silver mean represents values uncorrected for losses.

TABLE 6—FIRE-ASSAY RESULTS FOR GOLD AND SILVER

Gold (oz/ton)	Silver (oz/ton)
.36	16.84
.70*	10.87
.25	18.10
.27	16.10
.22	16.18
.31	16.30
.26	16.40
.30	21.30*
.22	15.60
.23	16.80
.25	16.90
.21	14.45
.21	15.60
.22	16.43
.31	17.90
.32	17.10
.44*	15.95
.48*	12.55*
.28	17.11
.24	16.46
.22	16.43
.22	15.60
.24	16.42

Mean	= .29	Mean	= 16.25
Standard deviation	= .12	Standard deviation	= 2.08
Reasonable mean	= .26	Reasonable mean	= 16.45
Standard deviation	= .043	Standard deviation	= 0.82

*indicates values which were rejected for calculation of reasonable means.

Using these reasonable means, some further comparisons can be made with the commercial laboratory fire-assay data in table 2. If the reasonable means are assumed to be the correct values, and the smelter assays agree, then the difference between the commercial assays and the reasonable means can be compared to the splitting limits as shown in table 7.

Three laboratories (Nos. 3, 4, and 7) meet smelter splitting limits on gold, and five laboratories (Nos. 1, 4, 5, 6, and 7) meet smelter splitting limits on silver. Only two laboratories, Nos. 4 and 7, meet smelter splitting limits on both gold and

TABLE 7—DIFFERENCE BETWEEN REASONABLE MEAN VALUES AND COMMERCIAL LABORATORY VALUES

Laboratory	Gold (oz/ton)	Silver (oz/ton)
1	+ .1	+ .39
2	+ .44	− 5.58
3	− .01	+ 1.65
4	+ .01	− .35
5	− .04	− .27
6	+ .05	− .15
7	0.00	− .05
8	+ .04	+ 4.85

Smelter splitting limits: Gold = .02 oz/ton
                        Silver = .5 oz/ton

silver. Looking at it another way (table 2 vs. table 6), four out of eight laboratories are further than 1 standard deviation from the mean on gold or silver or both: two laboratories are further than 2 standard deviations from the mean on gold or silver, or both. One of these two is further than 2 standard deviations from the mean on both gold and silver.

Atomic absorption values have all been previously shown. Because there were so few values none was discarded—and the means are considered reasonable means. For comparison they are shown in table 8 with the fire-assay reasonable means.

TABLE 8—FIRE ASSAY AND ATOMIC ABSORPTION REASONABLE MEANS

Fire assay		Atomic absorption	
Gold (oz/ton)	Silver (oz/ton)	Gold (oz/ton)	Silver (oz/ton)
0.26	16.45	0.24	17.46
Standard deviation		Standard deviation	
0.043	0.82	0.026	1.01
n = 20	n = 20	n = 6	n = 7
n = number of assays			

Conclusions

Results of gold and silver fire assays by commercial laboratories differ markedly, despite the homogeneity of ore samples involved. This variation is evident not only between individual laboratories, but within a single laboratory repeating the same test on different days. Atomic absorption assay results from different laboratories also vary, but not to the degree seen in fire-assay comparisons.

Insufficient analyses were obtained by atomic absorption methods to draw firm conclusions. Further evaluation is needed of atomic-absorption methods, particularly for gold. On the basis of the limited data provided by this study, gold assays by atomic absorption appear to be more reliable than fire assay on this particular ore sample.

The mean silver value is higher for atomic absorption than for fire assay. Thus if an operator is selling to a smelter, he should have his ores assayed by fire assay, not atomic absorption (unless an appropriate correction factor is applied).

An operator should not ship ore to the smelter on the basis of a single commercial assay unless he has either a wide profit margin or positive previous experience with the laboratory.

Commercial laboratories in general need to be more concerned with quality control. ∎

A.P.G.S. Annual Meeting

THE ASSOCIATION OF PROFESSIONAL GEOLOGICAL SCIENTISTS held its 15th annual meeting, November 30 through December 2, 1978, at the Sheraton Old Town Inn in Albuquerque, New Mexico. The theme of the conference was *Government Regulations— Bane or Blessing?* Featured speakers were J. Allen Overton, Jr., President of the American Mining Congress, Charles W. Margolf, Vice-President of Western Coal Operations for W. R. Grace & Co., Gen. Richard Bulgin (Ret.), Executive Director, Associated Nuclear Consultants of America, Ltd., Robert D. Gunn, President, American Association of Petroleum Geologists, and H. Peter Metzger, Administrator of Environmental Affairs, Public Service Company of Colorado.

All agreed that blessings were very difficult to identify. According to Richard W. Everett, Vice-President of Chase Manhattan Bank, a study by a group of banks indicates that the federal tax burden of regulation now totals $103.1 billion, and that regulation is stifling exploration and development and is aggravating the already excessive balance-of-payments deficity. Criticism was not so much of the regulations themselves as of the manner in which the regulations are being administered and applied. A principal complaint voiced was that bureaucrats often lack experience and understanding of the industry they are regulating. Federal requirements are being applied countrywide without regard to extreme variations in local conditions. One paradox cited was the rules regarding sulfur emissions from coal-burning plants, where emissions controls must reduce the sulfur in the stack gases to a small percentage of the feed. In the case of western coals (which are very low in sulfur) the resulting sulfur fraction being specified is economically and metallurgically impossible to achieve. Ironically, eastern coals, which meet the standards, still dump more sulfur into the atmosphere than the western coals would if burned without controls.

Other concerns voiced were the emotional opposition to nuclear power applications, and the difficulties of financing exploration and development—particularly in uranium and coal. The ultimate irony is to force miners back into high-risk, high-cost underground mining at a time when increased coal production has been declared a national goal.

Harrison H. (Jack) Schmitt, U.S. Senator from New Mexico, spoke at the banquet. He described his and other Congressmen's efforts to reassert congressional authority and control over the regulatory process. Congress has tended to delegate too much authority to the executive agencies. His bill, which will be introduced in Congress, will provide for congressional monitoring via hearings and actual recall of regulations for reconsideration should difficulties develop.

A transcript of the proceedings will appear in *Professional Geological Scientist,* the official publication of the A.P.G.S. For information, write to P.O. Box 957, Golden, CO, 80401.—*Clay T. Smith, Publicity Chairman* ☞