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The characterization of USGS gold reference standard, DGPM-1

by

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INTRODUCTION

Disseminated gold ore from the Pinson Mine in northern Nevada was collected, prepared, and analyzed as a U.S. Geological Survey reference standard, DGPM-1. The Pinson Mine was chosen as the collection site for the standard because the gold deposit here is a Carlin-type deposit with the gold occurring as sub-micron size particles. The small particle size distribution of gold in this deposit type is ideal for the collection of a reference standard where gold homogeneity in a bulk sample is critical. Analytical results from 16 laboratories are presented for major elements, gold, and the ore-associated elements arsenic, antimony, and tungsten. Total element concentrations of gold, arsenic, antimony and tungsten in DGPM-1 are, respectively: 730 ± 50 ng/g (ppb), 180 ± 12 µg/g (ppm), 14 ± 1 µg/g and 70 ± 9 µg/g.

LOCATION AND DESCRIPTION OF THE SAMPLE

The Pinson Mine is located 40 miles northeast of Winnemucca, Nevada, on the east flank of the Osgood Mountains (Fig. 1). Other gold deposits in the area which are currently being mined include the Gatchell and Twin Creeks deposits. Material for DGPM-1 was collected from one of the five open pits at the Pinson Mine, identified as the Mag pit. Ore from the Mag pit is hosted by carbonate-bearing argillite and shale within the Late Ordovician upper Comus Formation (Foster and Kretschmer, 1991). According to Foster and Kretschmer (1991), the gold predominately occurs in pyrite and iron oxides as particles less than 0.5 microns in size. The Mag deposit is also enriched in the Carlin pathfinder elements arsenic, antimony, and tungsten.

SAMPLE COLLECTION AND PREPARATION

Because the vertical and horizontal distribution of gold in a deposit can be quite variable, a preliminary sampling scheme on an exposed and accessible bench elevation in the Mag pit was designed. Grab samples were collected along the 4960 ft bench of the west wall of the Mag deposit. The samples were crushed, pulverized, and analyzed in the field using a U.S. Geological Survey mobile laboratory. Analytical results from the reconnaissance samples identified a 12 sq ft area at site 26 (Fig. 2) that contained the desired grade of ore material. Approximately 400 kg of DGPM-1 was hand picked from this site.

The coarse bulk sample was crushed, and ground to minus 200 mesh (0.075 mm) using conventional milling techniques. The pulverized material was homogenized in a 10 ft³ cross flow V-blender for 24 hours and bottled in approximately 200 g portions.

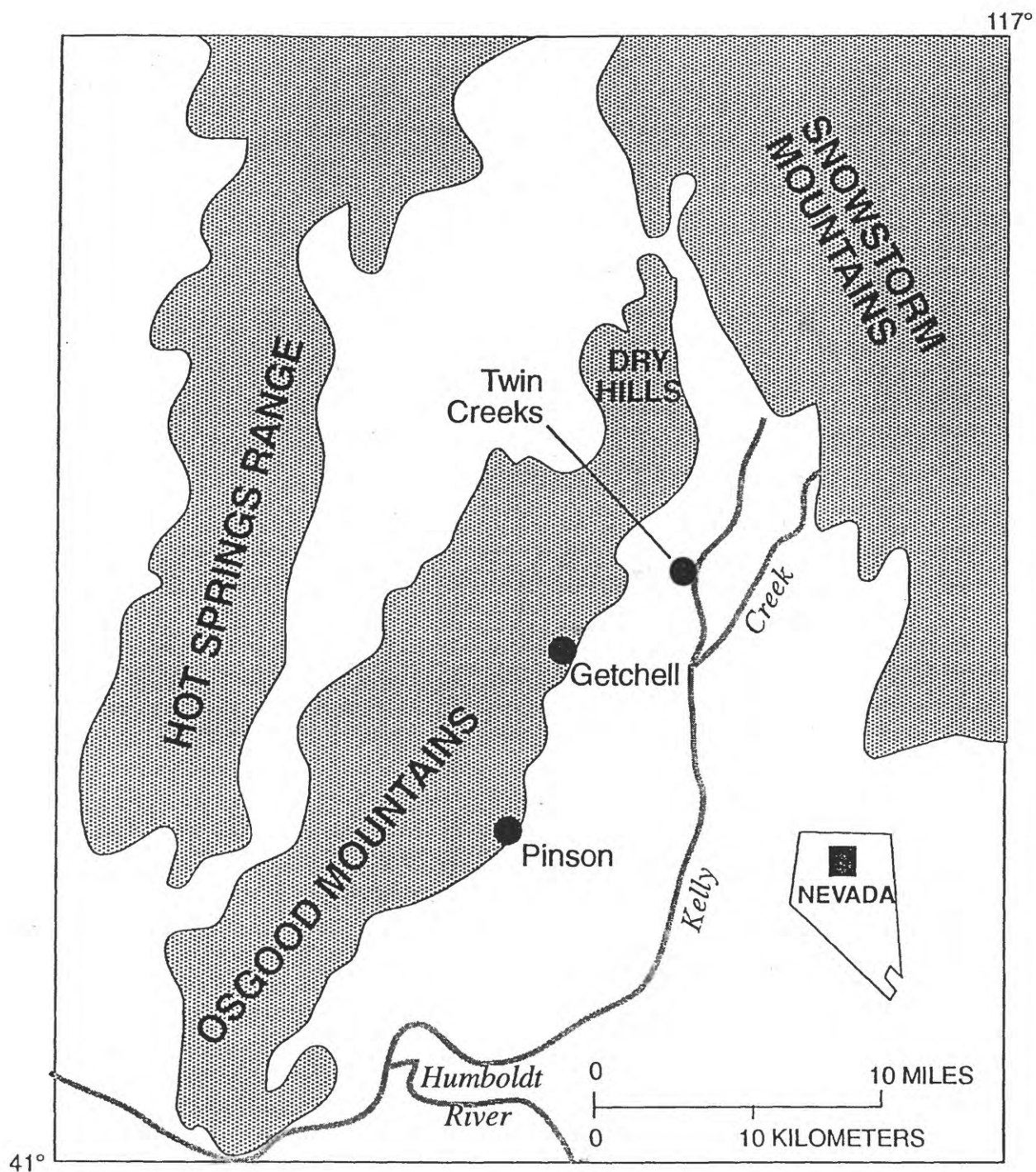


Figure 1. Index map showing the location of the Pinson, Getchell, and Twin Creeks disseminated gold deposits in northern Nevada

The particle size distribution of the pulverized sample is shown on Table 1. Mineralogical analysis by X-ray diffraction show DGPM-1 to consist of 80 percent quartz, 12 percent clay (kaolinite, montmorillonite, and illite), 5 percent orthoclase, and 3 percent muscovite.

Homogeneity of Standard

Based on U.S. Geological Survey neutron activation analysis (NAA) and analysis of variance (ANOVA), bottles of DGPM-1 are considered homogeneous with respect to gold, arsenic, antimony, and tungsten ($p > 0.95$). Data obtained for within- and between-bottle replicates are shown in Table 2. Data from wavelength dispersive X-ray fluorescence analysis (WDXRF) and ANOVA show that the bottles are also homogeneous for the major element oxides of silicon (SiO_2), aluminum (Al_2O_3), iron (Fe_2O_3), magnesium (MgO), potassium (K_2O), and calcium (CaO) ($p > 0.95$). The major oxide of sodium (Na_2O) was not detectable at the determination limit (0.15%) for the WDXRF procedure.

Analytical Results

Analytical results from each laboratory were compiled and the laboratory mean (LM) and standard deviation (SD) for each element calculated. Analytical results from all the laboratories were then combined and a grand mean (GRD-M) and grand standard deviation (GRD-SD) was calculated for each element. Assuming a normal distribution, a 95% confidence interval (CI) was then determined for each element using the GRD-M, GRD-SD, and the total number of observations (n). The LM for each element was then compared to the boundaries of the 95% CI for the GRD-M. If the LM was greater than or equal to the boundaries of the 95% CI, the laboratory results for that element were rejected and the GRD-M and GRD-SD recalculated. This data rejection process was continued until all LM values for an element fell within the 95% CI. For the four elements and seven oxides evaluated only a single iteration process was usually required.

Identification of a GRD-M as a recommended or information value for an element was based on the number of independent techniques used and the number of laboratories reporting information for that element. Recommended values are reported if three or more independent techniques from three or more independent laboratories were used in the final data compilation. Information values are listed if fewer than three independent techniques were used. All total element concentrations listed are based on the analysis of the standard on an as received basis.

Presented in tables 3 and 4 are the arithmetic means, standard deviations and number of observations for each laboratory providing information on that element. When provided

by participating laboratories, information on the method of sample decomposition and final method of analysis is also indicated. Examination of the trace element results in table 3 reveals that gold was quantified by the greatest number of laboratories, using the greatest variety of analytical procedures. Antimony in contrast was analyzed using the fewest analytical procedures. Major element results are derived primarily from WDXRF procedures, and ICP-AES procedures. Only total iron (Fe_2O_3) is determined by a total of three independent analytical procedures. Final recommended and information values for the total element concentrations in DGPM-1 are reported in table 5 along with their standard deviation.

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GLOSSARY

NAA	Neutron activation analysis
AAS	Atomic Absorption spectrometry
WDXRF	Wavelength dispersive X-ray fluorescence
ICP-AES	Inductively Coupled Plasma-Atomic Emission Spectroscopy
ICP/MS	Inductively Coupled Plasma Mass Spectrometry
HG-AAS	Hydride generation Atomic Absorption Spectrometry .
Grav	Gravimetric analysis
acid	Multiacid sample decomposition
acid/xtr	Multiacid sample decomposition followed by solvent extraction
FA	Fire Assay
fusion	Sample decomposition by alkaline fusion
acid/fusion	Multiacid sample decomposition followed by alkaline fusion
ANOVA	Analysis of variance
GRD-M	Grand mean for compiled data
GRD-ST	Grand standard deviation for compiled data

REFERENCES CITED

- Kane, J.S., Arbogast, B., and Leventhal, J., 1990, Characterization of Devonian Ohio shale SDO-1 as a USGS geochemical reference sample: Geostandards Newsletter, v. 14, p. 169-196.
- Foster, J.M. and Kretschmer, E.L., 1991. Geology of the Mag deposit, Pinson Mine, Humboldt County, Nevada, in Raines, G.L., Schafer, R.G., and Wilkinson, W.H., eds., Geology and Ore Deposits of the Great Basin - Symposium Proceedings: Reno, Nevada, Geological Society of Nevada, p. 845-856.

Table 1. Particle size distribution of DGPM-1 reference standard

Mesh size		Weight percent
	+100	0.01
-100	+150	0.01
-150	+200	0.18
-200	+230	0.59
-230	+325	4.73
	-325	94.48

Table 2. Neutron activation analyses for gold, arsenic, antimony, and tungsten in DGPM-1 as a test for homogeneity. Samples 236A-O are sample splits taken from the same bottle

<u>BOTTLE</u>	<u>Au (ng/g)</u>	<u>As (μg/g)</u>	<u>Sb (μg/g)</u>	<u>W (μg/g)</u>
618	768	162	13.6	71
879	732	163	13.5	72
685	755	165	13.7	74
473	731	164	13.4	71
440	777	165	13.5	73
283	777	165	13.7	72
1851	739	164	13.5	72
1702	744	163	13.4	70
1695	771	165	13.7	72
1950	737	166	13.7	71
2129	728	165	13.6	71
236A	790	163	13.5	72
236B	764	164	13.6	69
236C	745	164	13.4	72
236D	779	166	13.5	71
236E	762	164	13.6	73
236F	786	163	13.5	74
236G	763	165	13.5	70
236H	773	162	13.4	70
236I	716	160	13.3	66
236J	745	161	13.3	69
236K	755	164	13.4	71
236L	726	163	13.3	71
236M	724	164	13.5	72
236N	737	165	13.5	71
236O	733	164	13.4	68
11	764	168	13.6	73
1969	760	166	13.4	71
2039	746	165	13.5	70

Table 3. Analytical results for gold, arsenic, antimony and tungsten in DGPM-1.

Au (ng/g)				
<u>Technique</u>	<u>Decomp. proced.</u>	<u>Lab mean</u>	<u>std. dev.</u>	<u># obs</u>
NAA	-	740	10	5
NAA	-	760	23	39
NAA	-	771	37	9
NAA	-	686	13	9
AAS	FA	699	14	38
AAS	FA	740	25	9
AAS	FA	880	-	1
Grav	FA	764	21	3
NAA	-	699	-	1
ICP/MS	acid	817	58	9
ICP-AES	acid/xtr	680	20	9

As (µg/g)				
HGAAS	acid	178	9	38
HGAAS	"	184	11	9
HGAAS	"	183	5	9
HGAAS	"	186	5	9
ICP-AES	acid/xtr	160	0.5	9
ICP-AES	acid	196	4	9
NAA	-	202	3	6
NAA	-	166	3	38
NAA	-	185	3	9
NAA	-	178	2	9

Sb (µg/g)				
HGAAS	acid	13	1	9
NAA	-	14	0.1	38
NAA	-	14	0.2	5
NAA	-	14	0.3	9
NAA	-	14	0.4	9

W (µg/g)				
NAA	-	83	1	6
NAA	-	73	5	38
NAA	-	88	1	9
NAA	-	77	1	9
AAS	acid	62	5	38
ICP-AES	acid	76	3	9
ICP-AES	fusion	57	4	9
ICP-AES	acid/fusion	88	6	3

Table 4. Analytical results for major element oxides in DGPM-1.

Al_2O_3 (%)				
<u>Technique</u>	<u>Decomp. proced.</u>	<u>Lab mean</u>	<u>std. dev.</u>	<u># obs</u>
WDXRF	fusion	9.45	0.03	31
WDXRF	fusion	9.79	0.06	9
WDXRF	fusion	9.62	0.04	9
WDXRF	fusion	9.66	0.05	9
ICP-AES	acid	9.37	0.17	9
ICP-AES	acid	9.35	0.05	9
ICP-AES	fusion	9.62	0.05	9
ICP-AES	fusion	9.2	-	1
CaO (%)				
WDXRF	fusion	0.19	0.002	31
WDXRF	fusion	0.24	0.004	9
WDXRF	fusion	0.29	0.006	9
WDXRF	fusion	0.19	0.005	9
ICP-AES	fusion	0.20	0.009	9
ICP-AES	acid	0.22	0.014	3
WDXRF	fusion	0.16	<0.001	9
Fe_2O_3 (%)				
WDXRF	fusion	1.94	0.01	31
WDXRF	fusion	1.93	0.01	9
WDXRF	fusion	2.03	0.04	9
WDXRF	fusion	1.82	0.05	9
ICP-AES	acid	2.01	0.02	3
ICP-AES	fusion	1.89	-	1
ICP-AES	acid	2.01	0.05	9
NAA	-	1.89	0.06	9
SiO_2 (%)				
WDXRF	fusion	79.7	0.14	31
WDXRF	fusion	80.4	0.16	9
WDXRF	fusion	80.0	0.20	9
WDXRF	fusion	80.3	0.23	9
WDXRF	fusion	78.8	0.20	9
WDXRF	fusion	79.2	0.31	9
K_2O (%)				
WDXRF	fusion	2.76	0.01	31
WDXRF	fusion	2.73	0.05	9
WDXRF	fusion	2.72	0.02	9
ICP-AES	fusion	2.81	0.23	9
ICP-AES	acid	2.60	0.02	9

Table 4 (cont.)

MgO (%)				
<u>Technique</u>	<u>Decomp. proced.</u>	<u>Lab mean</u>	<u>std. dev.</u>	<u># obs</u>
WDXRF	fusion	0.57	0.01	31
WDXRF	fusion	0.50	0.00	9
WDXRF	fusion	0.58	0.006	9
WDXRF	fusion	0.62	0.009	9
ICP-AES	acid	0.55	0.02	3
ICP-AES	fusion	0.53	0.011	9

Table 5. Summary of constituent concentrations for U.S. Geological Survey gold reference standard, DGPM-1

Recommended element concentrations

<u>Element</u>		GRD-M	GRD-SD
Au	(ng/g)	730	50
As	(μ g/g)	180	12
W	(μ g/g)	70	9
<u>Oxide</u>		GRD-M	GRD-SD
Fe ₂ O ₃	(%)	1.96	0.05

Information values

<u>Element</u>		GRD-M	GRD-SD
Sb	(μ g/g)	14	1
<u>Oxide</u>		GRD-M	GRD-SD
Al ₂ O ₃	(%)	9.54	0.15
CaO	(%)	0.20	0.04
K ₂ O	(%)	2.74	0.09
MgO	(%)	0.58	0.03
SiO ₂	(%)	79.7	0.53
TiO ₂	(%)	0.56	0.02