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TITLE If not obvious	Rosebud Sampling Studies
AUTHOR	Bucknam C; Baker E; Clayton R; Dean D; Gribben T; Lane M; Gribben T; Hartman S; McGuire M; McMiller G; Mullin J; Santti S; Wan R; Schutz L; Sigurdson J; Spiller D; Tempel T; Vance R; Voorhees J; Walker P
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Additional Dist_Nos:	
QUAD_NAME	Sulphur 7½'
P_M_C_NAME (mine, claim & company names)	Rosebud Mine; Twin Creeks Plant; Sage Mill; Pinion Mill; American Assay Laboratories; Newmont Metallurgical Service
COMMODITY If not obvious	gold; silver
NOTES	Correspondence; laboratory procedures; waste assays.  31 p.

Keep docs at about 250 pages if no oversized maps attached  
(for every 1 oversized page (>11x17) with text reduce  
the amount of pages by ~25)

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Initials Date

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Initials Date

Rosebud Sampling Studies

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NEWMONT METALLURGICAL SERVICES  
10101 EAST DRY CREEK ROAD  
ENGLEWOOD, COLORADO 80112  
Telephone: (303) 708-4000  
Facsimile: (303) 708-4020

## TRIP REPORT

July 28, 1997  
File 01000

Traveler: C. H. Bucknam/Inverness *CHB*

Destinations: Rosebud Mine, Winnemucca, Nevada  
Twin Creeks Plant, Golconda, Nevada  
American Assay Laboratories, Sparks, Nevada

Dates: June 8-12, 1997

Copy:	E. D. Baker/Inverness	J. Mullin/Elko
	R. Clayton/Rosebud	S. Santti/Carlin
	D. Dean/Twin Creeks	L. Schutz/Lone Tree
	T. Gribben/ Twin Creeks	J. Sigurdson/Twin Creeks
	S. Hartman/Hecla	D. E. Spiller/Inverness
	M. Lane/Carlin	T. Tempel/Carlin
	M. A. McGuire/Inverness	R. Vance/Winnemucca
	G. McMillan/Twin Creeks	J. Voorhees/Twin Creeks
	P. Walker/Twin Creeks	

### Executive Summary

C. H. Bucknam traveled to the Rosebud Mine, Pinion Mill, Sage Mill laboratories, and American Assay Laboratories to review the sampling, sample preparation, and assaying methods being used to process Rosebud ore. Truck sampling procedures at the mine were adequate for moisture determination, but larger sample weights are necessary for proper grade estimation for run of mine ore. Slurry sample presentation at the mill also needs to be improved to reduce the potential for bias. Laboratory processing of slurry samples could also be improved by using sample preparation facilities at the Pinion Mill. Procedures at the commercial laboratory recommended by F. F. Pitard were being followed to properly estimate the grade of the samples received. Verification studies are recommended in order to further optimize the sampling, sample preparation and assay procedures for the joint venture.



## Rosebud Mine—Winnemucca, Nevada

I traveled to the Rosebud Mine, near Winnemucca, on June 9, 1997, to observe the sampling procedures being used prior to ore being shipped to the Twin Creeks plant for processing. I met with Charlie Muerhoff, the Chief Geologist, who provided me with copies of the F. Pitard reports on the subject. Ore was being hauled from the underground operations in 20-ton trucks and dumped into a stockpile which represented one month's production from the mine. A loader was used to mix and stack the ore in the stockpile.

The sampler located his equipment in a pickup truck at the opposite end of the stockpile from the truck scale. Trucks would arrive periodically and tare-weigh. They would then pull up parallel to the stockpile, and the loader would load the first trailer with three nominal 8-ton scoops of ore and the rear trailer with two scoops of ore. On the third and fifth scoop, the loader operator would drive by the sampler so he could collect one shovelful of sample from the scoop. A potential bias was noted in that the loader operator tended to dig closer to the location of the sampler whenever a sample was required.

Each sample was placed in a clear plastic bag and supported in a 5-gallon bucket, which was marked according to the campaign, sample number, and increment number. The bag was sealed with a wire tie, and the weight was measured on a portable balance and recorded on a form for processing at American Assay Laboratories (AAL) in Sparks. The loaded truck was weighed on the scale, and the sampler collected the weight tickets with the samples.

Ten incremental samples were sealed in a 55-gallon drum for collection by AAL at about 4 p.m. for processing in Sparks at night. The sampler complained that the sealing rings for the drums were being returned from the laboratory broken.

The sampling procedure being implemented at the mine appeared to be adequate for the intended purpose of moisture determination. Calculations performed using the heterogeneity data in the Pitard reports, however, indicated that a minimum sample weight of 1.5 tonne should be collected from run-of-mine ore at a nominal particle size of minus 6". This result indicates that there is a significant probability that the sampling is biased for the purposes of grade estimation and that a reference sampling method that is technically correct should be used to verify use of the routine method for grade estimation.

## Twin Creeks Plant—Golconda, Nevada

The Twin Creeks plant site was visited on June 10-11, 1997. A tour of the Pinon Mill and laboratory facilities was conducted, followed by meetings discussing the sampling



issues related to the Rosebud ore campaigns. Preparations were being made for the current ore campaign at the time I needed to travel to Reno to review the laboratory procedures at AAL.

## **Pinon Mill**

Trucks from Rosebud arrive at the plant site and are weighed. A campaign stockpile is then built near the mill-feed grizzly. Nearby, another purge stockpile is built using alternate shovels of ore being fed to the plant before the campaign. The purge stockpile is approximately 6000 tons and takes a couple of days to build. The objective of the purge stockpile is to run material of known grade during the time periods that the Rosebud campaign is being started and ended, so that the grade of the Rosebud contribution to the mill during those time periods can be estimated by difference.

The mill-feed weight belt is also calibrated before and after the ore campaign, and moisture samples are collected from the loader feeding the plant for select campaigns. The ore grade is estimated from the automatic head sampler located after the cyclone overflow. This sampler suffers from geometry problems similar in nature to the Rain Mill, in that a rectangular cutter is passing through a turbulent stream exiting a round pipe. It is recommended that the manufacturer of the sampler be contacted to obtain a recommendation on how the sample presentation may be improved, such as a laminar falling stream from an overflow box and weir.

Problems were also noted in the tortuous path of the piping between the primary and secondary samplers, which made it extremely difficult, if not impossible, to clean out and prevent segregation errors during sampling. It is recommended that the mill consider connecting the samplers with clear plastic hose in the future, as well as cleaning out the samplers on a regular schedule. The secondary sample also traveled through a long hose to a floor below for final collection, which offered an opportunity for segregation and bias.

The feed to the CIL circuit has the potential to be another reference point to collect a head sample. The stream travels through an overflow box and weir and discharges into a splitter box, which divides the stream between two CIL tanks. It may be possible to modify the discharge at this point in order to permit the collection of a reference sample cut.

The tailings sample discharge appeared to be of the proper geometry for proper cutting of a laminar stream; however, the height of the cutter opening should be extended to ensure that the stream is always being sampled under all flow conditions.

## Sage Mill Laboratory

The central laboratory for the Twin Creeks operation is located at the Sage Mill, which requires transportation of the slurries by pickup truck each shift. It was recommended that the JV consider using the laboratory facilities at the Pinon Mill for filtration of the samples prior to transportation of the solids and solutions for assay at the Sage Mill, in order to reduce the potential for losses and contamination.

Slurry samples were processed through a rotary-slurry sample divider, which appeared to be of proper design. The splitter was used to provide the three replicate slurry samples required by the JV agreement. Problems with using the splitter include the height and volume of the feed box and dilution problems associated with keeping the system clean. Modification of the secondary splitter for collection of replicate splits in the field (instead of in the laboratory) is recommended for consideration.

Solutions were assayed by flame atomic absorption spectroscopy (FAAS), solids by gravimetric fire assay for gold, and acid decomposition of a 10 g sample for silver by FAAS. The concentration of hydrochloric acid matrix for FAAS being used may not be adequate to keep the high silver content of Rosebud ores in solution. Newmont Metallurgical Services normally uses 25% v/v HCl as the FAAS matrix for silver. Screen-fire assays were not being performed on Rosebud samples.

In-house-developed standards, not certified reference materials, were being used in the laboratory on a routine basis. A stock of certified reference materials needs to be maintained and used, at least on a daily basis, in conjunction with the in-house standards to be used in each sample batch. Reference materials of similar grade to Rosebud ore need to be used during the campaigns, such as SRM 886 (0.24 oz Au/st) and GBW 07255 (1.4 oz Ag/st).

Establishment and use of quality control charts for each campaign is recommended by the operators as follows:

- 1) Net weight of slurry bucket each shift - Sample Preparation
- 2) Percent solids of slurry each shift - Sample Preparation
- 3) Deviations of replicate split assays - Laboratory
- 4) Performance on gold in-house standards - Laboratory
- 5) Performance on silver in-house standards - Laboratory
- 4) Performance on gold certified reference standards - Laboratory
- 5) Performance on silver certified reference standards - Laboratory

Control charts need to be reported monthly by the Pinion Mill sample preparation, Sage Mill laboratory, and by American Assay Laboratories, to be included in the campaign report.



It is also recommended to build tonnage weighted average composites for each liquid and solid product during each campaign. Analysis of the composites at the end of the campaign will provide another check on the grade estimate. The composites will be used for the proposed interlaboratory study and also can be used for the in-house standards for the next campaign and submitted at some frequency, blind, to the commercial laboratory. This is particularly important for the solutions which do not have an in-house standard or certified reference material at the present time.

### **Review Meeting**

A review meeting was held to discuss the issues involved in proper settlement for the Rosebud ore campaigns. The meeting was attended by C. H. Bucknam, D. Dean, T. Gribben, S. Hartman, G. McMillan, J. Sigurdson, and P. Walker. There had been reasonable agreement between the mine and mill for the first four campaigns, but the fifth campaign resulted in low recovery and grade in the mill. Unfortunately, duplicate moisture sampling at the Pinon Mill had been suspended during the fifth campaign, and the use of the new slurry splitter was also implemented in the laboratory at the same time. A significant amount of sampling review and check assaying has been undertaken in an effort to resolve these problems, including some during my visit.

I came out of the meeting with several assignments, as follows:

- 1) Complete the review of the process from mine to mill and laboratories, using Newmont mine geologists to review the underground sampling.
- 2) Conduct independent sampling studies at NMS on a current sample of Rosebud ore, in order to verify the initial heterogeneity study conducted under the direction of F. F. Pitard and to recommend the optimum sampling protocol for grade estimation.
- 3) Design an interlaboratory quality assurance testing program for Rosebud ores which can be used to interpret check assay results.
- 4) Investigate the possibility of using a commercial laboratory to perform reference analyses on replicate samples taken at the secondary splitter during ore campaigns, using reference analytical methods such as filtration of the entire slurry bucket, neutron activation, and chiddys.

### **American Assay Laboratories—Sparks, Nevada**

AAL in Sparks, Nevada was visited on June 12, 1997. Processing of the Rosebud samples was not observed, since the crew works on graveyard shift; however, a review of the procedures in place was performed. Samples are received in plastic drums from

the site. Locking rings on the drums did not appear to be damaged at the time of the visit, so it is assumed that they are being damaged at the time the drums are returned to the mine site. The AAL staff was not aware that this was a problem and agreed to try to find a solution for it.

Sample weights from the field are not checked, but accepted. Samples are dried during the first day after arrival and prepared the following night. Dried samples are weighed and recorded, and the percentage of moisture is calculated. Ten moisture sample increments are crushed to  $-1/4"$ , combined into one composite sample, mixed in a cement mixer, and transferred to a rotary divider.

A  $1/10^{\text{th}}$  split is collected from the rotary divider and lightly ring-ground in several batches to a nominal 25 mesh, which is then rotary-divided to nominal 1 kg charges. Several of the collection containers are selected at random to fill the feed hopper to approximately 5 kg, in order to obtain the required 500 g charge after rotary division. A screen-fire assay is performed on a randomly selected nominal 500 g portion of the bulk-pulverized product, using a 100 mesh screen to retain a coarse fraction containing coarse metallic particles. The retained fraction is stage ground and screened to a weight of less than 30 g, and duplicate 30 g fire assays are also performed on the minus 100 mesh fraction.

Herman inquarts of 2 mg are added to each fire assay charge and to a blank fire-assay charge, and the dore weights are recorded after cupellation. After parting away the silver, the gold is weighed and recorded. The gold weight is deducted, along with the blank inquart value from the dore weight, to calculate the silver weight. The gold and silver weights from each fire-assay charge are weight-averaged, using the screen fraction weights to calculate the gold and silver assays for the composite sample.

? The mine samples were also being processed by AAL in the same facility, using a slightly different procedure. A modified Keegor pulverizer, rather than batch-ring grinding, is used to produce the minus-25-mesh bulk-pulverized product. The assay portion is ring-ground to nominal 200-mesh, and a single fire assay is performed for mine samples, rather than a screen-fire assay.

### Gold Sampling Models

Gold sampling models for unliberated gold particles and for liberated gold particles were recommended by Francis Pitard<sup>1</sup> at coarse and fine particle sizes, respectively. This approach is based on the premise that the sampling variability at coarse crushing particle sizes is governed by the variability in the coarsest particles of ore in the sample; but when the samples are pulverized, the sampling variability is governed by the liberated gold particles. Newmont Metallurgical Services also endorses this approach, but has developed empirical models for the liberated gold based on gravity



concentration experiments, rather than using the generic formula based on the largest gold particle, which tends to overestimate the sampling variability at fine particle sizes.

A summary of the gold sampling models is shown in Table 1, based on the heterogeneity results used by Pitard for the coarse particle sizes and gravity separation experiments carried out at Hazen Research<sup>2</sup>. Variability is estimated in units of percent of relative standard deviation (%RSD), for the fundamental error in sampling at various particle sizes indicating the required minimum (50%), optimum (15%), and metallurgical (5%) sample weights calculated in grams. Details of the calculation procedures are covered in the appendix of this report.

Sampling constants for the liberated gold model are an order of magnitude larger than for the unliberated gold model, which translates directly into larger theoretical sample weights. For instance, at minus 10-mesh, the unliberated model requires only about 2 kg for 5% variability, and the liberated model requires 42 kg. Some judgment is required in selecting the required sample weight at the cross-over point; but as a general rule of thumb, we do not violate the 50% variability sample weight for the liberated gold at minus 10-mesh (420 g) in any event. Likewise, for field sampling, I would recommend not violating the 50% variability sample weight at 6" for run-of-mine ore (1.5 tonne), if at all possible.

Table 1

## Summary of Gold Sampling Model-Calculated Sample Weights

95% Passing Sieve	Cm	Gy Constant	Minimum 50% RSD Weight, g	Optimum 15% RSD Weight, g	Metallurgical 5%RSD Weight, g
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Heterogeneity Study Results - Unliberated Gold Model

6"	15	112	1516273	16847478	151627298
2"	5	195	97269	1080766	9726896
1"	2.5	275	17195	191054	1719488
0.5"	1.25	389	3040	33774	303965
3/8"	0.95	446	1531	17007	153059
1/4"	0.625	550	537	5970	53734
6 Mesh	0.335	752	113	1256	11302
10 Mesh	0.17	1055	21	230	2073

Gravity Concentration Results - Liberated Gold Model

10 Mesh	0.17	21381	420	4669	42018
28 Mesh	0.06	35989	31	345	3109
48 Mesh	0.03	50897	5	61	550
100 Mesh	0.015	71979	0	11	97
200 Mesh	0.0075	101794	0	2	17

**Conclusions and Recommendations**

Sampling, sample preparation, and assay-sample weight requirements were reviewed with respect to the fundamental error in sampling for Rosebud ores. The use of a 15% RSD is recommended for routine sampling and sample preparation, and 5% RSD is recommended for assay sample weights, if at all possible. Sample weights estimated by using these models should be verified by using independent studies, in order to validate the optimum sample weight for the various steps in the sample processing.

Field sampling of run-of-mine ores is a very difficult task, due to the relatively large particle sizes of the ore (nominal 95% passing 6"), which requires large sample weights according to calculations based on the fundamental error in sampling. This problem presents a challenge to Rosebud ore sampling during routine processing. A reasonable probabilistic approach has been implemented based on the recommendations of F. Pitard, which is a byproduct of the moisture-sampling procedures. This approach could be valid; however, it runs the risk of being biased, due to the omission of the coarse ore particles which are larger than can be collected in



a shovel scoop, as well as potential problems from selection of dig locations during sampling.

Sampling of the ore after milling does not present a problem concerning sample weight requirements based on the fundamental error in sampling. This procedure is the basis of the settlement process for Rosebud ore, but mill sampling and sample preparation procedures need to be carefully controlled on a routine basis to attain the high degree of confidence required in a settlement procedure. Efforts to improve the accountability in this area will result in the greatest pay-back.

If validation of the truck sampling grade is desired, I would base the sampling on collection of at least 100 periodic 1.5 tonne increments, using a small loader or backhoe during the campaign. Each increment could be passed through a 1" grizzly, and the oversize would be field-crushed to a nominal 1". A 1/8 portion of each increment (~200 kg) would be obtained by using an alternate-shovel or cone-and-quarter technique, and the samples would then be transported to a laboratory for drying and processing.

The dried sample would be crushed to at least 95% passing 3/8", rotary divided (10:1), to obtain a nominal 20 kg portion, crushed to minus 10 mesh, rotary divided (10:1) to obtain a nominal 2 kg portion, bulk pulverized to minus 28 mesh, rotary divided (10:1), and two opposite splits combined to obtain a nominal 400 g sample for a screen-fire assay.

The unliberated gold model provides some justification for use of a conventional 30 g fire assay (rather than a screen-fire assay), as long as the sample is ring-ground to minus 200 mesh. At minus 100 mesh, at least 100 g of sample should be used for fire assays, which requires the use of the screen-assay procedures. A variability study should be conducted on a bulk sample pulp at minus 200 mesh for ten replicates each of 15 and 30 g fire assays. These results will be compared to ten replicate screen fire assays from splits taken at minus 28 mesh of 100, 250 and 500 g, to determine the optimum fire assay procedure. This study will require about 1 kg of ore ring-ground to minus 100 mesh and 10 kg of ore bulk-pulverized at minus 28 mesh and will be included in the scope of the sampling study to be conducted at Newmont Metallurgical Services on the 60 kg sample of Rosebud ore.

## APPENDIX GOLD SAMPLING MODEL CALCULATIONS

The fundamental error in gold sampling is calculated in accordance with the theory of Pierre Gy<sup>3</sup>, and the required sample weight necessary for a desired level of precision may be estimated in accordance with Equation 1.

$$M_s = C d^3 / V^2 \quad (\text{Equation 1})$$

where:

- $M_s$  = Minimum Sample Weight, g,
- $C$  = Gy Constant of Heterogeneity for the Particle Size,
- $d$  = 95% Passing Particle Size, cm, and
- $V$  = Relative Standard Deviation, ratio.

### Unliberated Gold Case

In the heterogeneity study carried out by Pitard, 100 fire assays are performed on nominal 30 g portions containing coarse particles of the ore, and the sampling constant is determined empirically according to Equation 2 and estimated for other coarse particle sizes in accordance with Equation 3.

$$C = s^2 / x^2 p D \quad (\text{Equation 2})$$

where:

- $C$  = Gy Constant of Heterogeneity for the Particle Size (425),
- $s$  = Standard Deviation for the Assays (3.55), oz. Au/st,
- $x$  = Mean Gold Assay (1.41), oz. Au/st,
- $p$  = Number of Coarse Ore Particles per Assay (30), and
- $D$  = Specific Gravity of the Ore (2.23), g/cc.

$$C = K / d^{0.5} \quad (\text{Equation 3})$$

where:

- $C$  = Gy Constant of Heterogeneity for the Particle Size,
- $K$  = Particle Size Independent Constant, and
- $d$  = 95% Passing Particle Size, cm.

### Liberated Gold Case

In the case of liberated gold, gravimetric concentration of the gold is used to estimate the amount of gold liberated at the 95% passing particle size tested. In the Hazen work, 23% of the gold was estimated to be liberated at 28 mesh, 44% at 48 mesh, and



37% at 65 mesh, for a composite grading of 6 g Au/t. The effective liberation size of the gold particles, based on the minus 28 mesh test results, was calculated to 31 microns, in accordance with Equation 4. Equation 4 is also used to estimate the liberation factors for the other fine-particle sizes.

$$d_l = l^2 d \quad (\text{Equation 4})$$

where:

$d_l$  = Effective Liberation Size (0.0031), cm,  
 $l$  = Liberation Factor (0.23), ratio, and  
 $d$  = 95% Passing Particle Size, cm.

The Gy constants for each of the fine particle sizes is calculated in the classical manner, assuming that the gold occurs as relatively flat particles at a specific gravity of 19 g/cc, in accordance with Equation 5.

$$C = f g c l \quad (\text{Equation 5})$$

where:

$C$  = Gy Constant of Heterogeneity for the Particle Size,  
 $f$  = Shape Factor (0.2),  
 $g$  = Granulometric Factor (0.25),  
 $c$  = Mineralogical Factor (19/0.000006), and  
 $l$  = Liberation Factor.





Moisture Sample Increment Collection at the Rosebud Mine



Moisture Sample Field Weighing and Logging Station





Truck Weighing Operation at the Rosebud Mine

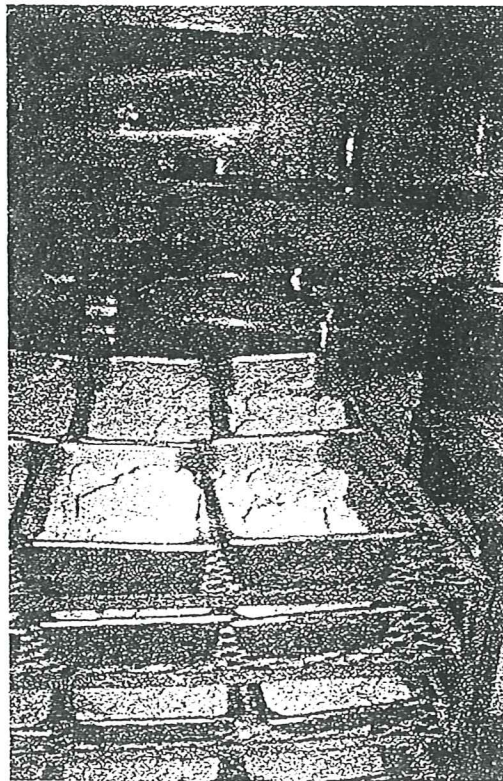


Moisture Sample Preparation for Shipment to Sparks





Moisture Sample Weighing Station AAL

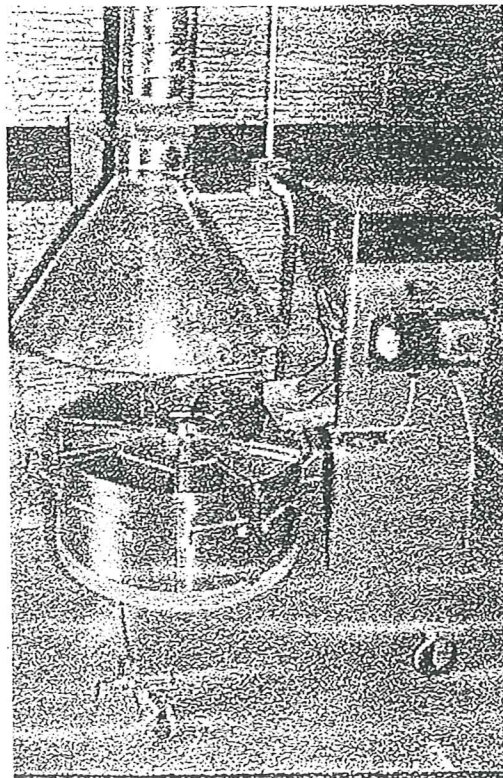


Sample Increment Drying at AAL



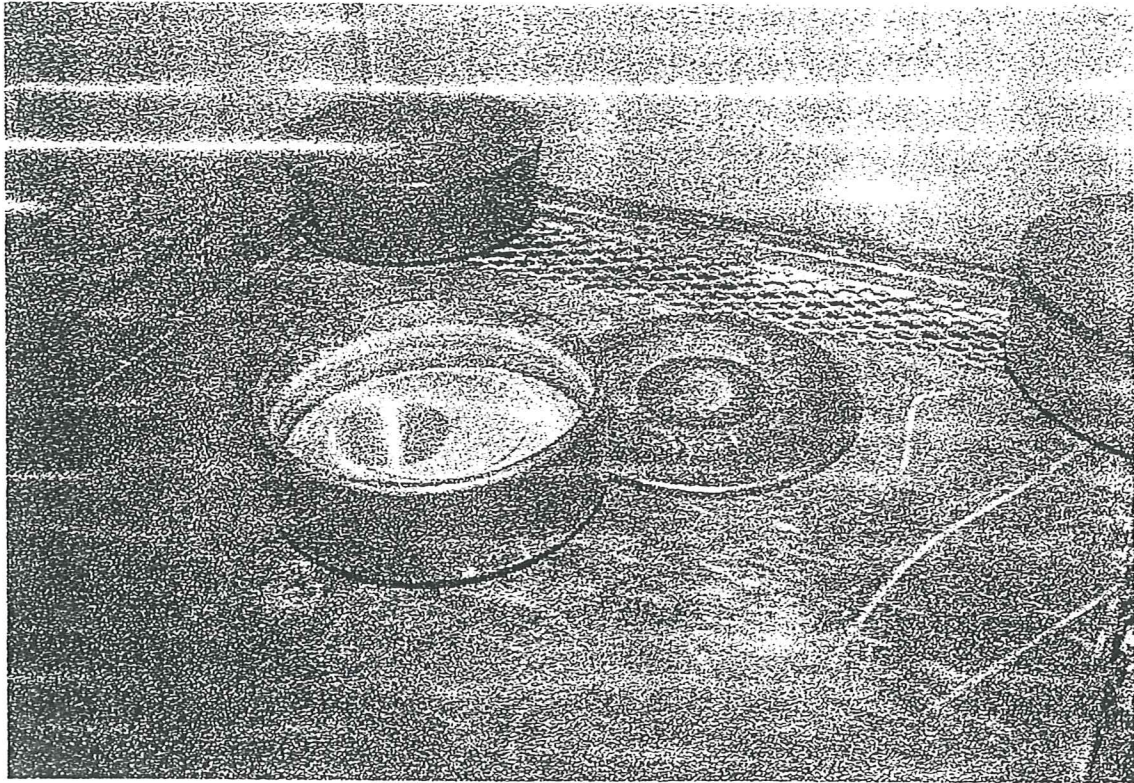


Sample Jaw Crusher at AAL



Rotary Sample Divider at AAL



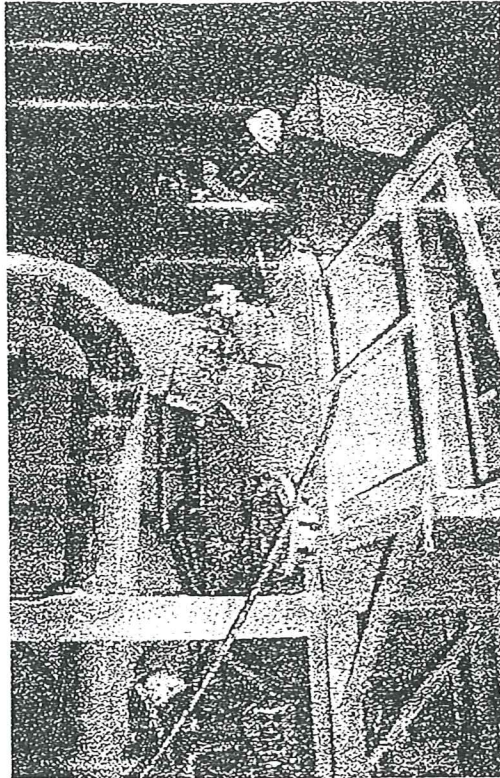


Sample Grinding Mills at AAL



Rosebud Ore and Purge Ore Stockpiles at Twin Creeks



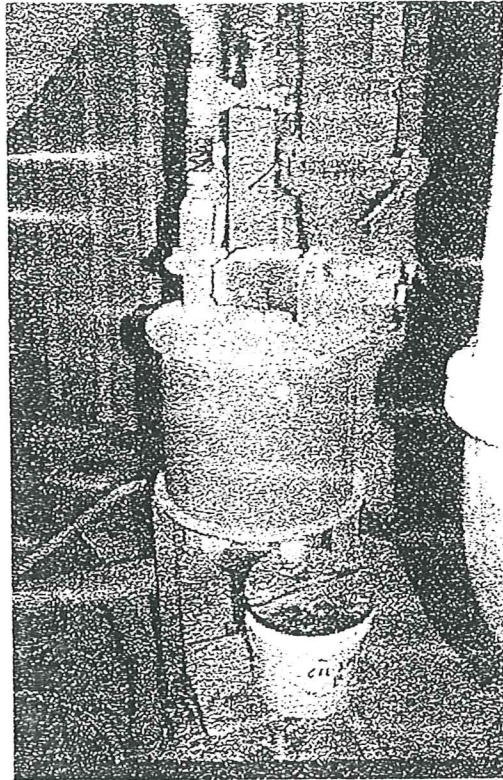


Pinon Mill Head Sampling Box Arrangement



Pinon Mill Leach Feed Slurry Splitter Box



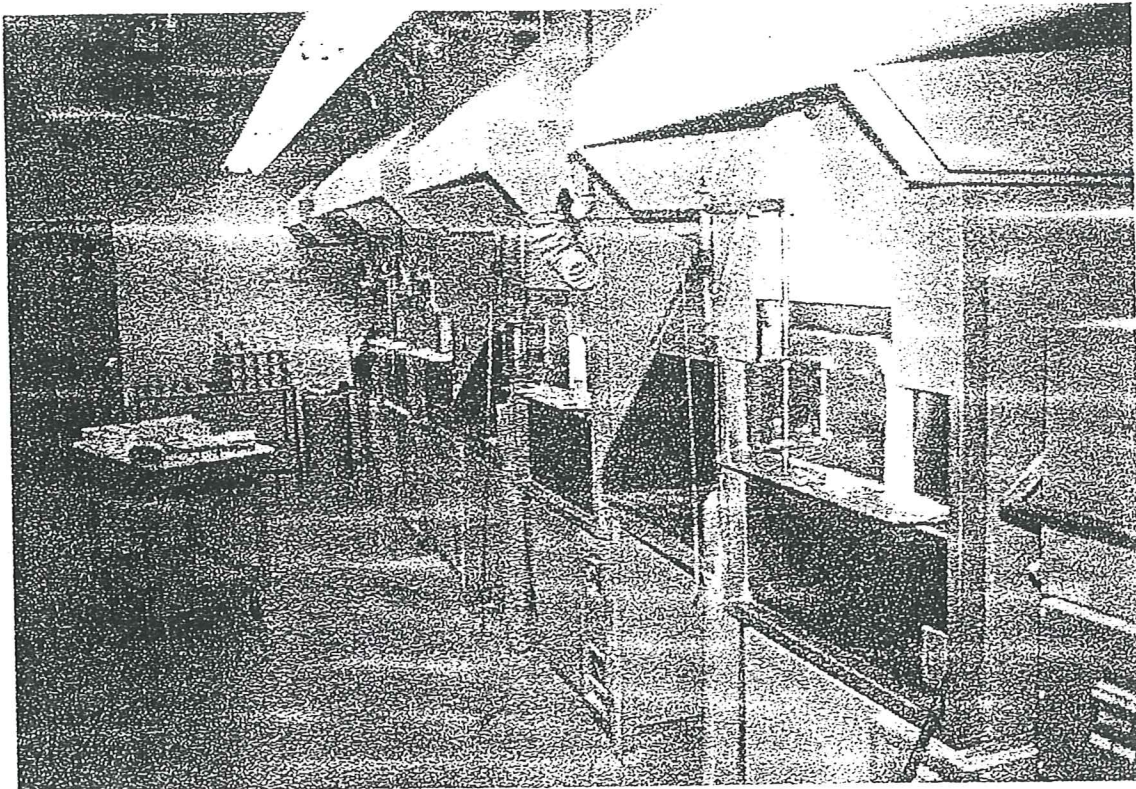


Pinon Mill Tailings Secondary Sampler

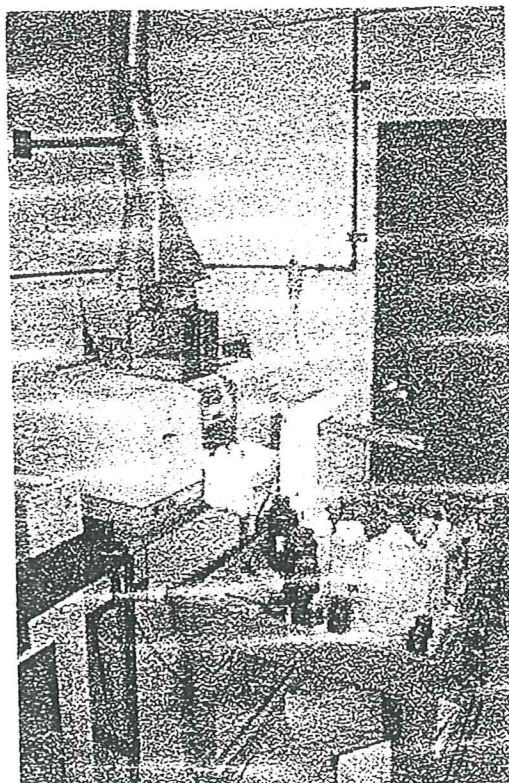


Sage Mill Laboratory Slurry Splitter





Sage Mill Laboratory Fire Assay Room



Sage Mill Laboratory Solution AAS Instrument



## REFERENCES

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- <sup>1</sup> Pitard, F. F., Study of the Heterogeneity of Gold and Silver at the Rosebud Project, Francis Pitard Sampling Consultants report to Hecla Mining Company, August 7, 1996.
  - <sup>2</sup> Dughman, T. L., and Bentzen, E. H., Scoping Studies Beneficiation and Recovery of Gold and Silver, Hazen Research report for Hecla Mining Company, December 16, 1994.
  - <sup>3</sup> Pitard, F. F., Pierre Gy's Sampling Theory and Sampling Practice, CRC Press, 1991.

NEWMONT METALLURGICAL SERVICES  
10101 EAST DRY CREEK ROAD  
ENGLEWOOD, COLORADO 80112  
TELEPHONE: (303) 708-4000  
FACSIMILE: (303) 708-4020

## MEMORANDUM

July 29, 1997  
File 01000/J0927

To: Scott Santti/Carlin

Copy: E. D. Baker/Inverness  
R. Clayton/Hecla-Rosebud  
S. Hartman/Hecla-Winnemucca  
M. A. McGuire/Inverness  
J. Mullin/Elko  
J. Sigurdson/Twin Creeks  
D. E. Spiller/Inverness  
T. Tempel/Carlin  
✓ R. Vance/Winnemucca  
J. Voorhees/Twin Creeks  
P. Walker/Twin Creeks  
R. Y. Wan/Inverness

From: C. H. Bucknam *CHB*

Subject: Rosebud Sampling Studies

Work is progressing at Dawson Metallurgical on processing the bulk ore sample from Rosebud ore under the attached scope of work. Estimated costs at Dawson are \$1,500 and Newmont Metallurgical Services will be able to process the fire assay samples for gold and silver. A supply of standard reference materials was ordered to be used for quality control on the analyses at a cost of about \$2250 for four bottles of each of the following standards: (1) SRM 886 (0.24 oz Au/st), (2) GBW 07277 (1.4 oz Ag/st), and (3) GBW 07256 (3.3 oz Ag/st). A bottle of SRM 886 has already been shipped to J. Herzog at the Twin Creeks plant site to be used for quality control during the Rosebud campaigns.

A Sieving Riffler has been located at a cost of \$4,100 (see attached). It is recommended that this be purchased to perform the screen-fire assay variability studies. The unit will be used without a screen to prepare eight charges at minus 28-mesh for each of the following nominal split weights: 625, 310, and 125 g. The 24 charges will each be processed through the unit with the 100-mesh screen in place, producing one plus 100 fraction and eight minus 100 fractions. The plus 100-mesh fraction will be stage ground, as necessary, until less than 30 grams of oversize is retained. After weighing the coarse and fine fractions, the coarse fraction and two of



the fine fractions will be selected from opposite sides of the turntable and ring ground for processing by the screen-fire assay protocol. The results will be statistically compared with eight 15 g and 30 g portions of the minus 200-mesh ring ground pulps in order to select the optimum sample weight for fire assay of Rosebud ore.

### **Scope of Work for Rosebud Sampling Study**

1. Receive nominal 60 kg bulk sample.
2. Screen at -1" and crush oversize to just pass 1".
3. Determine 95% passing particle size of the crushed ore.
4. Screen and verify that there are approximately 100 X 30 g particles at the nominal 95% passing size or proceed by the rotary division procedure.
5. Weigh and prepare 100 particles for assay by ring grinding to 95% -200 mesh.
6. Crush the fine fraction to 95% passing 10 mesh and split out one nominal 10 kg and one 1 kg charges.
7. Bulk pulverize the 10 kg charge to 95% passing 28 mesh and split out 1 x 5 kg charge, 1 x 2.5 kg charge, 2 x 1 kg charges, and 1 x 500 g charge.
8. Pulverize any excess coarse particle fraction at -28 mesh and prepare a nominal 500 g charge for screen-fire assay.
9. Return the ~30 g assay charges to Newmont Metallurgical Services for 100 gravimetric gold and silver fire assays and two 500 g nominal 28 mesh charges (coarse and fine fractions) for screen-fire assays at 100 mesh for gold and silver.
10. Ship one 1 kg at -10 mesh, two 1 kg charges, one 2.5 kg charge and one 5 kg charge at -28 mesh, and any excess sample material to Newmont Metallurgical Services.



**NEWMONT METALLURGICAL SERVICES**

10101 E. Dry Creek Road  
Englewood, Co 80112  
Telephone (303) 708-4000  
Facsimile (303) 708-4020

C. H. Bucknam  
Coordinator—Analytical Development

Phone: (303) 708-4430  
cbuc4430@corp.newmont.com

**M E M O R A N D U M**

**TO:** S. Santti/Carlin



File 75504-5.1

**FROM:** C. H. Bucknam/Inverness

**DATE:** February 18, 1998

**COPY:** K. Allen/Hecla Rosebud  
R. Clayton/Hecla Rosebud  
D. Dean/Twin Creeks  
T. Gribben/Twin Creeks  
S. Hartman/Hecla Winnemucca  
M. A. McGuire/Inverness  
J. Sigurdson/Twin Creeks  
D. E. Spiller/Inverness  
T. Tempel/Twin Creeks  
R. Vance/Winnemucca  
P. Walker/Twin Creeks

**SUBJECT: Marcasite Waste Sample – Assay Results**

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I requested a sample of marcasite waste from the Rosebud deposit from K. Allen, to be used in a fire assay variability study<sup>1</sup>. It was also used to investigate how the marcasite reacted to hydrochloric acid digestion in a reference acid-base accounting waste characterization test. Results from these studies are reported below.



## FIRE ASSAY VARIABILITY STUDY

The fire assay variability study was conducted to determine how well the sampling model for Rosebud<sup>2</sup> would predict the variability of assays on relatively small samples, due to the fundamental error in sampling. The screen-fire assay variability study on ore showed that the variability in gold assays was relatively constant when the assay charge weight was reduced from 700 to 100 g in the range of 6-7% relative standard deviation (RSD) at about 0.6 g Au/t (0.018 oz Au/st) for 10 g Au/t ore (0.29 oz Au/st). It was recommended to follow up the screen-fire assay study with a study of the variability of conventional fire assays on finely ground pulps, which are being routinely used for ore control.

A nominal 400 g assay pulp was produced from ring grinding a minus ten mesh split of the marcasite waste sample. The technician that prepared the sample reported the formation of "cookies" in the grinding mill from the marcasite caking up. The finely ground split was divided into eight nominal 50 g fractions on the sieving riffler<sup>3</sup>. One 50 g fraction was further divided to prepare eight nominal 6 g fire assay splits. Two fractions were combined and divided to prepare eight nominal 12 g fire assay splits and the remaining five fractions were combined and divided to prepare eight nominal 31 g fire assay splits.

The assayer consumed the smaller splits in total and weighed out an assay ton (29.2 g) for the larger splits. The assay results are summarized in Table 1, the results for the standard reference samples are shown in Table 2 and the assay results are attached. Assay results tended to increase with sample weight, which indicates that a minimum sample weight of 30 g is necessary for direct fire assay of Rosebud waste. Gold assay variability was relatively constant at 0.005-0.006 oz Au/ton, regardless of sample weight, which resulted in the higher than predicted % RSD in the range of 35-52%, due to the relatively low grade of the sample.

Table 1. Summary of Fire Assay Variability for Marcasite Waste

Statistic	Gold, oz Au/st			Silver, oz Ag/st		
	6.9	13.9	29.2	6.9	13.9	29.2
Sample Weight, g						
Mean	0.010	0.011	0.017	0.761	0.995	1.08
Standard Deviation	0.005	0.006	0.006	0.074	0.107	0.025
%RSD	52	51	35	10	11	2.3

Table 2. Summary of Standard Reference Materials Assays

Description	Weight AUFA		AGFA		g Ag/t	Difference %
	g	oz Au/st	g Au/t	oz Ag/st		
CONTROL GBW 07255	15.02	0.006	0.21	1.126	38.60	
Certified					46.9	-21.5
CONTROL GBW 07256	5.46	0.053	1.82	2.477	84.92	
Certified					112	-31.9
CONTROL SRM-886	29.28	0.24	8.23	0.011	0.38	
Certified			8.25			-0.3



Silver assays were biased low, which is attributed to the uncorrected fire assay procedure used and some probable contribution from low sample weight. Silver precision was very good on nominal 30 g samples at only 2% RSD, which is attributed to the relatively high silver grade of about 1 oz Ag/st.

### WASTE CHARACTERIZATION RESULTS

The Newmont standard carbon-sulfur analyses were performed on the sample to calculate net carbonate value (NCV), as well as to reference acid-base accounting tests for comparison. One of the main objectives of the testing was to see whether or not like pyrrhotite, marcasite would produce low results for the pyritic sulfur method, due to solubility in hydrochloric acid. Results are shown in Table 3.

The marcasite waste sample was classified as acidic with an NCV of minus 4.82 % CO<sub>2</sub>. There was no significant carbonate content in the sample with a total carbon of only 0.05 % C and acetic acid soluble calcium of only 0.08 % Ca. The acid neutralization potential (ANP) titration was negative at minus 0.13 % CO<sub>2</sub>, further verified with the acid concentration present (ACP) titration at minus 0.28 % CO<sub>2</sub>, indicating the presence of acidic salts in the sample.

The acid generation potential (AGP) of minus 4.82 was determined by the difference between total and residual sulfur after pyrolysis. The AGP estimate from sulfate sulfur, determined by combustion-infrared analysis after sodium carbonate digestion of the sulfate sulfur, was in good agreement at minus 4.9 % CO<sub>2</sub>. The AGP estimate by pyritic sulfur, using the difference between hydrochloric acid and nitric acid residual sulfur, was even more negative at minus 6.2 % CO<sub>2</sub>, attributed to a high hydrochloric acid residual sulfur value. The gravimetric sulfate sulfur AGP estimate was much lower at minus 1.1% CO<sub>2</sub>, which was probably due to inaccuracy of the gravimetric method at low sulfur content.

Table 3. Waste Characterization Testing Results for Marcasite Waste.

Determination	Value
Net Carbonate Value Calculation	
Carbon	
Total, % C	0.05
Residual, Pyrolysis, % C	0.00
Residual, Hydrochloric Acid, % C	0.00
Acid Neutralization Potential, % CO <sub>2</sub>	0.00
Sulfur	
Total, %S	3.92
Residual, Pyrolysis, % S	0.40
Acid Generation Potential, % CO <sub>2</sub>	-4.82
Net Carbonate Value, % CO <sub>2</sub>	-4.82
NCV Classification	Acidic
Acid-Base Accounting Reference Methods	
Acetic Acid Soluble Calcium, % Ca	0.08
Acid Neutralization Potential, % CO <sub>2</sub>	0.09
ANP Titration, % CO <sub>2</sub>	-0.13
ACP Titration, % CO <sub>2</sub>	-0.28
Pyritic Sulfur	
Residual, Hydrochloric Acid, % S	4.58
Residual, Nitric Acid, % S	0.06
Acid Generation Potential, %CO <sub>2</sub>	-6.19
Sulfate Sulfur	
Residual, Sodium Carbonate, % S	3.58
Acid Generation Potential, %CO <sub>2</sub>	-4.90
Gravimetric Sulfate Sulfur	
Total, % S	4.23
Residual, Sodium Carbonate, % S	0.82
Acid Generation Potential, %CO <sub>2</sub>	-1.12

### CONCLUSIONS AND RECOMMENDATIONS

Results from the testing of the marcasite waste sample indicate that standard (30 g) fire assay and carbon-sulfur analyses are suitable for routine gold grade and waste characterization purposes. The variability in the gold analyses was relatively constant with a standard deviation about 0.005-0.006 oz Au/st, regardless of the sample weight.

Use of fire assay sample weights below 30 g increase the risk of a low assay bias. It is recommended that the Rosebud bulk ore sample be submitted to the same procedure as the waste sample for comparison of the fire assay variability.

Silver results from non-corrected routine fire assays tend to be biased low and the use of a smaller fire assay sample weight appears to increase the bias. The effect of sample weight on silver assays should be studied further to determine what the optimum sample weight should be for accurate silver assays. It is recommended that the sample and standards used in this study be submitted for acid digestion and silver analyses by the normal NMS procedure for comparison with the Twin Creeks procedure.



## REFERENCES

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<sup>1</sup> Bucknam, C. H., "Sample Preparation for the Rosebud Fire Assay Variability Study," NMS memorandum to R. Y. Wan, October 20, 1997.

<sup>2</sup> Bucknam, C. H., Odekirk, J. R, and Seidel, J., Sampling Optimization Study for the Rosebud Bulk Sample, NMS report, November 6, 1997.

<sup>3</sup> Bucknam, C. H., "Rosebud Sampling Studies," NMS memorandum to S. Santti, July 29, 1997.

NEWMONT METALLURGICAL SERVICES

ASSAY RESULTS

February 17, 1998

DESCRIPTION	METHOD	ASSAY	RESULT	UNIT	QC
MARCASITE WASTE 30G #1	AU-AG	AGFA	1.09765	OPT	N/A
	AU-AG		37.6333	PPM	N/A
	AU-AG	AUFA	0.02492	OPT	N/A
	AU-AG		0.85452	PPM	N/A
MARCASITE WASTE 30G #2	AU-AG	AGFA	1.06861	OPT	N/A
	AU-AG		36.6379	PPM	N/A
	AU-AG	AUFA	0.01097	OPT	N/A
	AU-AG		0.37629	PPM	N/A
MARCASITE WASTE 30G #3	AU-AG	AGFA	1.09141	OPT	N/A
	AU-AG		37.4196	PPM	N/A
	AU-AG	AUFA	0.01695	OPT	N/A
	AU-AG		0.58147	PPM	N/A
MARCASITE WASTE 30G #4	AU-AG	AGFA	1.06415	OPT	N/A
	AU-AG		36.4848	PPM	N/A
	AU-AG	AUFA	0.01595	OPT	N/A
	AU-AG		0.54710	PPM	N/A
MARCASITE WASTE 30G #5	AU-AG	AGFA	1.03130	OPT	N/A
	AU-AG		35.3585	PPM	N/A
	AU-AG	AUFA	0.01098	OPT	N/A
	AU-AG		0.37651	PPM	N/A
MARCASITE WASTE 30G #6	AU-AG	AGFA	1.10826	OPT	N/A
	AU-AG		37.9971	PPM	N/A
	AU-AG	AUFA	0.02294	OPT	N/A
	AU-AG		0.78662	PPM	N/A
MARCASITE WASTE 30G #7	AU-AG	AGFA	1.09219	OPT	N/A
	AU-AG		37.4461	PPM	N/A
	AU-AG	AUFA	0.02094	OPT	N/A
	AU-AG		0.71814	PPM	N/A
MARCASITE WASTE 30G #8	AU-AG	AGFA	1.09797	OPT	N/A
	AU-AG		37.6443	PPM	N/A
	AU-AG	AUFA	0.00996	OPT	N/A
	AU-AG		0.34160	PPM	N/A
CONTROL SRM-886	AU-AG	AGFA	0.01095	OPT	N/A
	AU-AG		0.37574	PPM	N/A
	AU-AG	AUFA	0.24011	OPT	N/A
	AU-AG		8.23228	PPM	N/A
MARCASITE WASTE 10G #1	AU-AG	AGFA	1.19203	OPT	N/A
	AU-AG		40.8694	PPM	N/A
	AU-AG	AUFA	0.01231	OPT	N/A
	AU-AG		0.42205	PPM	N/A
MARCASITE WASTE 10G #2	AU-AG	AGFA	1.08041	OPT	N/A
	AU-AG		37.0423	PPM	N/A
	AU-AG	AUFA	0.01891	OPT	N/A
	AU-AG		0.64860	PPM	N/A
MARCASITE WASTE 10G #3	AU-AG	AGFA	1.01925	OPT	N/A
	AU-AG		34.9454	PPM	N/A
	AU-AG	AUFA	0.01935	OPT	N/A
	AU-AG		0.66352	PPM	N/A
MARCASITE WASTE 10G #4	AU-AG	AGFA	0.87072	OPT	N/A
	AU-AG		29.8529	PPM	N/A



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DESCRIPTION	METHOD	ASSAY	RESULT	UNIT	QC
MARCASITE WASTE 10G #4	AU-AG	AUFA	0.00640	OPT	N/A
	AU-AG		0.21950	PPM	N/A
MARCASITE WASTE 10G #5	AU-AG	AGFA	1.03717	OPT	N/A
	AU-AG		35.5597	PPM	N/A
	AU-AG	AUFA	0.00641	OPT	N/A
	AU-AG		0.21995	PPM	N/A
MARCASITE WASTE 10G #6	AU-AG	AGFA	0.92580	OPT	N/A
	AU-AG		31.7414	PPM	N/A
	AU-AG	AUFA	0.00625	OPT	N/A
	AU-AG		0.21446	PPM	N/A
MARCASITE WASTE 10G #7	AU-AG	AGFA	0.92661	OPT	N/A
	AU-AG		31.7693	PPM	N/A
	AU-AG	AUFA	0.00814	OPT	N/A
	AU-AG		0.27929	PPM	N/A
MARCASITE WASTE 10G #8	AU-AG	AGFA	0.91146	OPT	N/A
	AU-AG		31.25	PPM	N/A
	AU-AG	AUFA	0.01040	OPT	N/A
	AU-AG		0.35673	PPM	N/A
CONTROL GBW 07255	AU-AG	AGFA	1.12613	OPT	N/A
	AU-AG		38.6100	PPM	OUT
	AU-AG	AUFA	0.00582	OPT	N/A
	AU-AG		0.19970	PPM	N/A
MARCASITE WASTE 5 G #1	AU-AG	AGFA	0.65124	OPT	N/A
	AU-AG		22.3280	PPM	N/A
	AU-AG	AUFA	0.00434	OPT	N/A
	AU-AG		0.14885	PPM	N/A
MARCASITE WASTE 5 G #2	AU-AG	AGFA	0.70878	OPT	N/A
	AU-AG		24.3009	PPM	N/A
	AU-AG	AUFA	0.00853	OPT	N/A
	AU-AG		0.29278	PPM	N/A
MARCASITE WASTE 5 G #3	AU-AG	AGFA	0.73181	OPT	N/A
	AU-AG		25.0906	PPM	N/A
	AU-AG	AUFA	0.01692	OPT	N/A
	AU-AG		0.58013	PPM	N/A
MARCASITE WASTE 5 G #4	AU-AG	AGFA	0.72385	OPT	N/A
	AU-AG		24.8175	PPM	N/A
	AU-AG	AUFA	0.01703	OPT	N/A
	AU-AG		0.58394	PPM	N/A
MARCASITE WASTE 5 G #5	AU-AG	AGFA	0.81394	OPT	N/A
	AU-AG		27.9063	PPM	N/A
	AU-AG	AUFA	0.00818	OPT	N/A
	AU-AG		0.28046	PPM	N/A
MARCASITE WASTE 5 G #6	AU-AG	AGFA	0.86353	OPT	N/A
	AU-AG		29.6066	PPM	N/A
	AU-AG	AUFA	0.00822	OPT	N/A
	AU-AG		0.28196	PPM	N/A
MARCASITE WASTE 5 G #7	AU-AG	AGFA	0.84916	OPT	N/A
	AU-AG		29.1139	PPM	N/A
	AU-AG	AUFA	0.01230	OPT	N/A
	AU-AG		0.42194	PPM	N/A

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ASSAY RESULTS

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DESCRIPTION	METHOD	ASSAY	RESULT	UNIT	QC
MARCASITE WASTE 5 G #8	AU-AG	AGFA	0.74127	OPT	N/A
	AU-AG		25.4150	PPM	N/A
	AU-AG	AUFA	0.00428	OPT	N/A
	AU-AG		0.14690	PPM	N/A
CONTROL GBW 07256	AU-AG	AGFA	2.47729	OPT	N/A
	AU-AG		84.9350	PPM	OUT
	AU-AG	AUFA	0.05339	OPT	N/A
	AU-AG		1.83049	PPM	N/A



## NEWMONT METALLURGICAL SERVICES

## ASSAY RESULTS

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DESCRIPTION	METHOD	ASSAY	RESULT	UNIT	QC
ROSEBUD MARCASITE WASTE	ACPL	ACPL	-0.2815	%	N/A
	ANP		-0.1334	%	N/A
	C/S ROAST CAP F	0		%	N/A
	C/S ROAST SAP F	0.39908		%	N/A
	C/S TOTAL CTOT	0.04653		%	N/A
	C/S TOTAL	0.03591		%	N/A
	C/S TOTAL STOT	3.92258		%	N/A
	C/S TOTAL	3.74900		%	N/A
	CAAS	CAAS	0.08323	%	N/A
	CAAS		832.332	PPM	N/A
	CAI	CAI F	0	%	N/A
	SCIS	SCIS	3.57713	%	N/A
	SHCL	SHCL	5.10817	%	N/A
	SHCL		4.58252	%	N/A
	SHNO3	SHNO3	.05935	%	N/A
	SHNO3		593.5	PPM	N/A
	SSGM	SO4GR	0.82000	%	N/A
	SSGM		8200.06	PPM	N/A
	STGM	STGM	4.23206	%	N/A
	STGM		42320.6	PPM	N/A
CONTROL SRM-886	ANP		19.0509	%	N/A
	C/S ROAST CAP F	4.78933		%	N/A
	C/S ROAST SAP F	0.61851		%	N/A
	C/S TOTAL CTOT	5.55555		%	N/A
	C/S TOTAL STOT	1.47849		%	N/A
	Certified		1.466		
	CAAS	CAAS	12.3752	%	N/A
	CAAS		123752.	PPM	N/A
	CAI	CAI F	0.57126	%	N/A
	SCIS	SCIS	0.92862	%	N/A
	SHCL	SHCL	1.23371	%	N/A
	SHNO3	SHNO3	.08510	%	N/A
	SHNO3		851	PPM	N/A