heat by chemical action, will generate sufficient heat to drive off the excess of ammonia, and this is the main point to observe in securing its neutrality.

This reagent is made up as follows in this laboratory: 740 grams of commercial citric acid are carefully weighed out and placed in a four-liter graduate containing 1900 cc. of ten per cent. ammonium hydroxide. With a suitable glass rod the citric acid is thoroughly and vigorously stirred until the citric acid has all dissolved. Distilled water is now added until the meniscus reads 4000 cc. The solution is again stirred and carefully transferred to a large porcelain evaporating dish. The solution is allowed to stand over night and in the morning large oval crystals are noticeable on the sides of the four-liter graduate, and invariably the solution when tested for neutrality will be found strictly neutral. If the solution is not vigorously stirred sufficient heat will not be evolved to drive off the excess of ammonia, and when tested will be found to be slightly alkaline, but by resorting to vigorous stirring, a strictly neutral reaction will be obtained. The solution, after being transferred to the reagent bottle, is brought to the required temperature, 20° C., and distilled water added until the specific gravity is 1.09. On testing the neutrality of this solution, both with coralline and cochineal as indicators, it will be found unnecessary to alter its neutrality in the least degree, the solution being strictly neutral.

THE ASSAY OF TELLURIDE ORES.

By Charles H. Fulton.

Received June 8, 1898.

THE growing importance of telluride ores and the fact of their relative difficulty of assay in contrast with other ores, has led to this work. The object of the work was to determine where the difficulty and losses lay, and if possible to remedy these defects of the assay by proper methods and precautions.

Ore No. 1 is a telluride ore, having a gangue, mainly of quartz. A very small amount of pyrite is also present.

Ore No. 2 is a very rich telluride from Cripple Creek, Colorado, containing considerable pyrite. The gangue is siliceous.

Both ores were finely crushed, No. 1 crushed to 100-mesh, and No. 2 to 150-mesh, to insure an even sample. The crucible assay was tried on these ores with various modifications of the charge as regards the amount of litharge present. All assays on ore No. 1 were made on one assay ton of ore. The assays on ore No. 2 were made on one-fifth assay ton.

The charges for ore No. 1 were as follows, except that the litharge increased from one assay ton to six assay tons in the series :

1 assay ton ore.
1 assay ton litharge.
½ assay ton silica.
1 assay ton soda.
10 grams borax glass.
2 grams argol.
Salt cover.

The amount of silver in the ore is very small, and was disregarded in the assays. Twenty milligrams of silver foil were added to each crucible assay to give a larger button in cupellation and to avoid subsequent inquartation. Four iron nails were also added to each assay to desulphurize and give a malleable button.

The lead buttons ranged in weight from twenty to forty-eight grams, the increase in weight being due to the increasing litharge. All buttons were fairly malleable and were cupelled directly, even the larger buttons, without previous rescorification. The cupellation was conducted at a low heat, with feathers of litharge.

The assays show a regular increase of gold as the litharge increases, with the exception of B and B_0 and ranged from 5.60 milligrams to 5.78 milligrams. A further increase of litharge showed no better result. (See Table I.)

The slags from these crucible assays were ground up and mixed with one assay ton of litharge and two grams of argol, and fused. The resulting lead button was cupelled with the addition of ten milligrams of silver to give a button which could be parted.

The results of correction of the crucible assay show that con-

TABLE I.—SHOWING EFFECT OF INCREASING LITHARGE. ORE NO. I.

Number of assay.	Amount of litharge	Amount of argol.	Character of slag.	Character of lead button.	Weight of lead button.	Amount of gold.	Gold recovered from crucible slag	Gold recovered from cupel.	Total amount of gold recovered.	Percentage recovered from crucible slag.	Percentage recovered from cupel.	Total percentage recovered.	Amount of gold in corrected assay.
	Assay tons.	Grams.			Grams.	Milli-grams.	Milli-gram.	Milli-gram.	Milli-gram.	Per cent.	Per cent.	Per cent.	Milli-grams.
A	1	2	black glassy	malleable	20	5.60	0.03	0.09	0.12	0.52	0.55	2.07	5.72
B	2	2	" "	brittle	37	5.52	0.16	0.02	0.18	2.76	0.18	2.94	5.70
B_0	2	2	" "	"	47	5.69	0.03	0.01	0.04	0.52	0.09	0.61	5.64
C	4	2	green glassy	"	32	5.67	0.05	0.01	0.06	0.86	0.09	0.95	5.73
C_0	4	2	" "	"	30	5.65	0.08	0.02	0.10	1.38	0.18	1.56	5.75
D	6	2	" "	malleable	48	5.72	0.06	0.01	0.07	1.03	0.09	1.12	5.79
D_0	6	2	" "	"	45	5.78	0.06	0.01	0.07	1.03	0.09	1.12	5.85
				Averages,		5.65	0.068	0.013	0.09	1.16	0.12	1.48	5.74

These assays were run with iron nails to desulphurize.

siderable gold is retained in the slag. The assays which contain the least amount of litharge retain the most gold. The average correction was 0.068 milligram, which is equivalent to 1.16 per cent. The highest corrected assay was taken as 100. This loss of gold in the crucible slag is much greater than that in ordinary ore assays.

The cupels were also ground up and mixed with one assay ton of litharge, one and a half assay tons of borax glass, one assay ton of soda, and two grams of argol. Silver was added to the fusion.

The amount of gold recovered from the cupel is small, only 0.013 milligram, equivalent to 0.12 per cent.

It is also to be noted that the corrected assays increase with the greater quantity of litharge, as well as the uncorrected assays, which shows that part of the loss is due to volatilization.

Table II shows the effect of increasing the litharge on a rich telluride. The charges for this ore were as follows, except as before, for the varying litharge: One-fifth assay ton ore, two assay tons litharge, one assay ton soda, one gram argol. The ore had considerable reducing power, and one gram of argol was found to be sufficient to give the right-sized button.

The crucible slags were remelted and the resulting lead buttons cupelled. The actual loss of gold in slags for this ore was higher than for No. 1, although the percentage loss decreases. The loss in the cupel being small, it was neglected in this case. The silver contents of this ore were determined, but no correction for the loss of silver in slags and cupels was made.

When the button resulting from the fusion of a telluride ore was brittle, it was usually deemed necessary to rescorify it before cupellation. The button resulting from telluride fusions is usually brittle and scorification is almost always resorted to. A set of assays were run on ores No. 1 and No. 2 to determine whether this is of advantage. These sets were run, as the previous ones, with increasing litharge, but no nails were added to desulphurize. All the buttons were rescorified, some with the addition of test-lead.

Tables III and IV show the results of these sets. The gold is considerably lower when the buttons are rescorified, and also it is evident that the loss takes place during the scorification.

TABLE II.—SHOWING EFFECT OF INCREASING LITHARGE ON A RICH TELLURIDE. ORE NO. 2.

Number of assay.	Amount of litharge.	Amount of ore.	Amount of argol.	Character of slag.	Character of lead button.	Weight of lead button.	Amount of gold.	Gold recovered from crucible slag.	Percentage recovered from crucible slag.	Amount of gold in corrected assay.
	Assay tons.	Assay ton.	Gram.			Grams.	Milligrams.	Milligram.	Per cent.	Milligrams.
1	2	$\frac{1}{5}$	1	green glassy	fairly malleable	20	89.10	0.22	1.21	90.20
2	4	$\frac{1}{5}$	1	" "	" "	26	90.50	0.08	0.31	90.90
3	4	$\frac{1}{5}$	1	" "	" "	48	90.26	0.10	0.55	90.36
4	6	$\frac{1}{5}$	1	" "	malleable	28	90.30	0.09	0.49	90.75
					Averages,		90.04	0.09	0.45	90.55

TABLE III.—SHOWING EFFECT OF RESCORIFICATION OF LEAD BUTTON. ORE NO. 1.

Number of assay.	Amount of litharge.	Weight of lead button.	Weight after rescorification.	Amount of gold.	Gold recovered from crucible slag.	Gold recovered from scorification slag.	Gold recovered from cupel.	Total amount of gold recovered.	Percentage recovered from crucible slag.	Percentage recovered from scorification slag.	Percentage recovered from cupel.	Total percentage recovered.	Amount of gold in corrected assay.
	Assay tons.	Grams.	Grams.	Milligrams.	Milligram.	Milligram.	Milligram.	Milligram.	Per cent.	Per cent.	Per cent.	Per cent.	Milligrams.
E	1	17	8	5.32	0.10	0.16	trace	0.26	1.72	2.76	trace	4.84	5.58
F	2	22	14	5.62	0.06	0.04	0.01	0.11	1.03	0.70	0.18	1.91	5.73
G	4	30	18	5.64	0.06	0.04	0.01	0.11	1.03	0.70	0.18	1.91	5.75
H	6	40	14	5.50	0.08	0.05	trace	0.11	1.38	0.52	trace	1.90	5.61
H_0	6	39	14	5.54	0.09	0.04	trace	0.13	1.55	0.70	trace	2.20	5.67
I	8	75	24	5.58	0.07	....	....	....	1.20	....	....	....	5.65
		Averages,		5.54	0.08	0.038	....	0.11	1.31	0.65	....	1.98	5.77

TABLE IV.—SHOWING EFFECT OF RESCORIFICATION OF LEAD BUTTON, RICH TELLURIDE. ORE NO. 2.

Number of assay.	Amount of litharge.	Amount of ore.	Amount of argol.	Character of lead button.	Weight of lead button.	Weight after rescorification.	Amount of gold.	Gold recovered from crux slag.	Corrected assay.
	Assay tons.	Assay ton.			Grams.	Grams.	Milligrams.	Milligram.	Milligrams.
10	4	$\frac{1}{5}$	none	malleable	68	29	89.30	....	....
11	4	$\frac{1}{5}$	"	"	54	26	89.60	....	....
12	6	$\frac{1}{5}$	"	"	76	24	88.50	0.20	88.70
						Averages,	89.13		

These assays were run with niter and iron nails to desulphurize.

This loss is partly in the scorification slag and partly by volatilization. The crucible slags, cupels, and scorification slags were remelted and the buttons cupelled. The loss in the crucible slag is a little higher than in the first set (Table I).

The average amount of gold recovered from the scorification slag is 0.038 milligram, equivalent to 0.65 per cent. The amount recovered from the cupel is very small and less than in Table I.

The assays run on the rich telluride, with rescorification, point to the same conclusion (Table IV). This set of assays was run with niter and iron nails to increase the oxidizing action and to desulphurize. This loss of gold during rescorification is much greater in tellurides than in ordinary ores. With the latter it usually amounts to no more than a trace. The average result, by direct cupellation, is always higher than that obtained when the buttons are first rescorified.

A charge with potassium cyanide was run on ore No. 1. Ore, one assay ton; potassium cyanide, two assay tons; litharge, one assay ton; salt cover and silver. This gave gold 5.40, which is much lower than the results of assays run by the ordinary charges.

The following are the average results obtained from the four sets of assays:

ORE NO. 1.

Average uncorrected assay, when cupelled directly, 5.65 milligrams.

Average corrected assay, when cupelled directly, 5.74 milligrams.

Average uncorrected assay, when button is rescorified, 5.54 milligrams.

Average corrected assay, when button is rescorified, 5.67 milligrams.

Average loss of gold in crucible slag, 0.075 milligram.

Average loss of gold in per cent., 1.23 per cent.

Average loss in cupel, when cupelled directly, 0.013 milligram.

Average loss in cupel, in per cent., 0.12 per cent.

Average loss in scorification slag, 0.038 milligram.

Average loss in per cent., 0.65 per cent.
Total gold recovered when cupelled directly, 0.09 milligram.
Total gold recovered in per cent., 1.48 per cent.
Total gold recovered when rescorified, 0.11 milligram.
Total gold recovered in per cent., 1.98 per cent.

ORE NO. 2.

Average uncorrected assay, cupelled directly, 90.04 milligrams.

Average corrected assay, cupelled directly, 90.55 milligrams.

Average uncorrected assay, when rescorified, 89.13 milligrams.

Average loss of gold in crucible slag, 0.09 milligram.

Average loss of gold in per cent., 0.45 per cent.

A set of assays was also run in which the heat of the fire and time in fire was varied, to determine whether these factors had any influence on the result obtained. The fires were varied from a red to a white heat and the time from thirty minutes to one hour. The difference in results was not appreciable, and a good, moderately hot fire, with from forty to fifty minutes in the furnace, gave the best results. Cupellation, however, should be carried on at a low heat.

SCORIFICATION ASSAYS.

Both ores, Nos. 1 and 2, were run by scorification with very variable results, but uniformly lower than the crucible assay.

Ore No. 1 was assayed by the following charge :

Ore, one-fifth assay ton.
Test-lead, seventy grams.
Borax glass, one-half gram.
Litharge cover, twenty grams.

This gave gold = 4.30 milligrams. (Crucible assay ran 5.62 milligrams.) In the next charge the test-lead was increased to eighty grams. Otherwise charge was the same.

This gave gold = 4.80 ounces.

Then to further increase the ratio of ore to test-lead these charges were run in duplicate.

No. 1.	No. 2.
Ore, one-tenth assay ton.	Ore, one-tenth assay ton.
Test-lead, fifty grams.	Test-lead, sixty grams.
Borax glass, one-half gram.	Borax glass, one-half gram.
Litharge cover, twenty grams.	Litharge cover, twenty grams.

No. 1 gave gold, 4.20 and 4.00 milligrams, respectively.

No. 2 gave gold, 5.00 and 5.40 milligrams, respectively.

The heat was kept low throughout.

More assays were run by various charges, but invariably gave low results. A great drawback to the method is the large amount of test-lead which must be used, giving a button too large to be cupelled easily. The litharge cover seems of advantage, but also tends to increase the size of the lead button by protecting the lead from scorification.

Ore No. 2 was run by the following charge:

Ore, one-tenth assay ton.
Test-lead, fifty grams.
Litharge cover, twenty grams.
Borax glass, one-half gram.

This gave gold, 74.80 ounces (crucible assay, 90.20 ounces).

The test-lead was increased to sixty grams, giving 78.70 milligrams gold.

The scorification slags were ground up and assayed:

No. 1 gave 0.42 milligram = five and six-tenths per cent. of gold.

No. 1 gave 0.38 milligram gold.

This makes corrected result per assay ton on No. 1 = 79.0 milligrams.

This makes corrected result per assay ton on No. 2 = 81.8 milligrams.

This shows the corrected assay by scorification still away below the ordinary crucible assay. The loss of gold in scorification slag is very great, but evidently a still greater loss takes place by volatilization.

These results readily show that the crucible assay is much the better for telluride ores.

CONCLUSIONS.

It is very evident that a large excess of litharge is necessary to obtain good results in assaying a telluride ore. The amount of litharge will vary with the richness of the ore, the proper proportion of litharge to ore being maintained in a rich telluride by decreasing the quantity of ore. A definite ratio of litharge to gold present in telluride ores might be established.

The loss of gold in the crucible slag is many times more than that in assaying ordinary ores, and with both low and high grade ores justifies the making of corrected assays where accuracy is required. The loss of gold by absorption in the cupel is not of much account.

Scorification of any kind is bad for a telluride ore, whether it be the direct scorification of the ore or the rescorification of brittle or large buttons.

The loss in cupelling a large or brittle button directly is much less than if it were first rescorified.

The amount of gold lost in the scorification slag when scorifying the ore directly is very great, amounting to five and six-tenths per cent.

The highest uncorrected result also gives the highest corrected result, showing that loss takes place by volatilization. The loss of gold in a rich telluride is greater than in a low grade ore, but the percentage loss decreases with the richness of the ore.

In general, the crucible assay is best by far for telluride ores. The fire should be moderately hot, the length of time from forty to fifty minutes. The assay should be so conducted as to give a button of good size, from twenty to twenty-eight grams, which can be cupelled directly. The cupellation should be done at a low heat with crystals of litharge forming on the cupel. When the assay is to be accurate, the crucible slag should be remelted and the first result corrected. The cupel correction can be disregarded.

Mr. F. C. Smith publishes an article in the *Trans. Am. Inst. Min. Eng.*, **26**, 485, on "The Occurrence and Behavior of Tellurium in Gold Ores, etc.," which is partly devoted to the assay of telluride ores. His experiments prove that the crucible assay is the better for telluride ores, and that the loss of gold in the assay is very great if the charge is not properly made up as regards the amount of litharge present. He does not locate the loss or make corrected assays.

In the discussion of Mr. Smith's paper, Mr. Wm. P. Jenney states that he believes an oxidizing charge with a small quantity of ore would be beneficial. The excess of litharge, of course, has an oxidizing action, but to try this point further the assays in Table IV were made by the following charge :

Ore, one-fifth assay ton.
Litharge, four assay ton.
Soda, one and a half assay tons.
Silica, one assay ton.
Six loops of wire.
Twenty grams of niter.

The wire acted as the reducing agent with the sulphur. The buttons were good and malleable, but on cupellation gave no better results than charges with litharge alone.

ON THE LINDO-GLADDING METHOD OF DETERMINING POTASH.

BY A. L. WINTON[1] AND H. J. WHEELER.[2]

Received June 15, 1898.

IN view of certain criticisms of the Lindo-Gladding method of potash determination which have appeared of late, notably those by Vogel and Haefcke,[3] we were appointed a committee by the Association of Official Agricultural Chemists of the United States to prepare an article setting forth certain work bearing upon the points involved.

At the regular meeting of the Association in Washington in 1890, Frear and Sweetser[4] called attention to certain comparative determinations made by them, which indicated that the use of sodium chloride was unnecessary. The mean difference in

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[1] Chemist Conn. Agricultural Experiment Station and Reporter on methods of potash determination for the Association of Official Agricultural Chemists for the years 1896 and 1897.

[2] Chemist R. I. Agricultural Experiment Station and Reporter on methods of potash determination for the Association of Official Agricultural Chemists for the years 1894 and 1895.

[3] Die landw. Versuch-Stationen 47 (1896), pp. 112-117.

[4] U. S. Dept. of Agr., Div. of Chem., Bull. 28 (1890), p. 73.