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1894

On the fire assay of gold, silver, and lead

Claude D. Grove

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Thesis
on the fire assay
Gold - Silver and Lead,
for the degree of
Engineer of Mines,

by
C. J. Grover

For June 14-95

14258

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Outfit for Assay Office

Two furnaces are necessary.

(a) A Wind furnace.

(b) A Muffle furnace.

(a) The Wind furnace should be about 18" square in longitudinal cross section and about 18" deep to the grate bars. It should be built of fire brick and covered with an iron cover lined beneath with fire clay. The cover should be hinged at upper side and raised and lowered by means of a weight connected by a chain passing over a pulley above.

Near the bottom of the furnace should be placed grate bars to support the fuel and should be sufficiently far apart to assure the proper draught. The flue from the Wind furnace may lead direct to the chimney from the muffle furnace.

(b) The muffle furnace should be built of brick and lined with fire brick. The grate bars should be properly arranged about 1" below the bottom of the muffle.

The furnace should be fixed from the rear to prevent the heat from the fire door annoying the workers. Beneath the fire grate should be an ash pit and this should be occasionally filled with water to cool the lining of the furnace and also from gases whose combustion helps heat the furnace.

The flues run directly around the muffle on both sides to the chimney which should be about 30' high.

The door to the fire box may be easily handled by the aid of a counterpoise supported from a pulley above.

Stimmins coal may be used as fuel. The muffle is supported by projecting bricks in the rear and shelf in front, & that the most convenient size for muffle is:

- Length 17 1/2"
- Width 12 1/2"
- Height 6"

All inside measurement.

In front of the furnace and on a level with the bottom, the large muffle should be an iron plate. This gives a convenient place for boards of crucibles, & on first the preparatory to charging besides gives a place by which the muffle door may be slid open or shut to any required degree.

The muffle door should be made of -

- (a) asbestos, sheet asbestos,
- (b) an iron door.

The asbestos is cut sufficiently large to cover the front of the muffle.

The iron door should have a knob on the back, so that while lying on the iron plate and leaning back on the iron knob it may be slid backward and forward to adjust the asbestos plate to any required degree.

There should be crucible, & cupel and cupel trays and boards. The cupel boards should be covered on one side with sheet asbestos so as not to be burnt by placing hot cupels on them.

There should also be moulds, Hammer and
Cupril Cupel board and tables, Chisels for
cutting bullion, refined lead etc. also cupel
mould in the furnace room.

A good cupel mould may be made as follows:-
The brass cupel mould is fastened on to an
iron rod 2' long, this wire is vertically through
a hole in the top of the table, this hole is
provided with an iron ferrule just fitting
the mould.

Over the floor a horizontal rod is connected
at one end to this vertical rod and is fastened
by a pin to the leg of the table, which acts
as a fulcrum. The extreme end is formed like
a pedal and power is exerted there by the
foot. The vertical rods drop down, and the
cavity thus formed in the mould filled
with bone ash prepared for making cupels.
Then the pedal is pressed with the foot
and the bone ash kept in place by the
aid of a flat plate of iron fixed with
the top of the table and fastened at one end.

while the other end slides under a roof.
This cover is then removed and the rod
pressed a little higher so the cupel may
be removed. This device while simple is
very expeditious for making cupels.

It is best to have the furnace room
separated from the fuel and balance rooms
by partitions.

The fuel room may consist of three
bins for soft coal, coke and Charcoal, with
a passage between them to the back of the
furnace where the opening door is.

The tools for this room are a ~~rod~~ shovel
cross bar and hose.

The balance room contains bins for crucibles
scrapings and fluxes, also shelves for samples
and tables for scale.

Three balances are necessary, one a cheap
Green scale for the weighing of large amounts
of flux previous to mixing. This flux is charged
by measure. The second balance is for one end
should be sensitive to .01 gm.

The third balance should be very sensitive for weighing gold and silver buttons, it should be of the best make and should weigh to .00001 gm. This room should be well lighted and free from draught that might interfere with the weighing.

Sampling

In sampling the main lot by shovel fulls in proportional alternations.

If lots are small say one or two car loads. $\frac{1}{10}$ is to be crushed down to the size of walnuts this sample then to be quartered down if small lots to 1000, # if two or three car lots to 200, # and if large to 400, # then put through rolls and crushed to size of cherries. Then quartered down to three wheelbarrow loads, then crushed until it will pass through $\frac{1}{4}$ mesh screen. Then quartered down until $\frac{1}{3}$ sack is left if a small lot and $1\frac{1}{2}$ sacks if large lot.

Or if ore is rich, this remainder to be put through the grinder entirely and the ground sample to be quartered down until some 5 to 10

is left. This remainder to be ground still finer on the bucking plate untill all of it will pass through a 50 mesh sieve, then from this sample, # is to be taken by quartering and rubbed down untill all will pass through a 50 mesh sieve.

Assay of lead.

A charge of 10 gms (one measure) of flux is put in a 10 dm crucible, the 10 gms are added and all intricately mixed. Then a second measure of flux is poured on top and if one contains sulphur, then 12 d nails are stuck in charge.

The crucible should be over hot for about 30 min. I find a quick hot fusion of lead renders a higher and more accurate result than a slower and colder fusion.

For running slag when a very small amount of lead exists a larger charge of ore should be used generally about 20 gms, to this add a weighed silver button of about 500 mg. to collect the lead. weigh the resulting button and deduct

The weight of the silver. The button may be assayed at any convenient time and the silver saved.

7. Assay for lead

- Potassium Carbonate 14 parts of wt
- Sodium Carbonate 10 " " "
- Cry. Sulphuric Brox 10 " " "
- What flux will fit in case
- Gold 10 parts of wt

The assay may be in the form of Ore Matte, Specie, Bullion or Doré.

If ore is matte pour in copper run by crucible as a layer charge may be used and thus a greater quantity of gold is obtained to work upon. If Specie or Matte moderately rich in copper run by scorification. Bullion if pure may be directly assayed, if containing numerous quantities of foreign metals other than lead, scorchify the cupel. If ore dissolve a weighed quantity and weigh gold.

Crucible Assay for Gold

Use 20 lb crucibles.

The ore may be in the form of

Sulphide, Chloride, Telluride, Bromide, Carbonate,
Silicate, Arsenide & Selenide, also Slags and
Dull & Hard Acetons and Fine Grout.

Plus for Crucible Assay.

Equal weights of Sodium Carbonate and
litharge are intimately mixed and a measure
of the mixture taken that will contain 30 grs of
the litharge. To this is added 1/2 A.T. of an and an
amounting careful of ground vitrified borax.

If or is Sulphide add Potassium Cyanide,
I find this acts much more uniformly on
or than nitre and diminishes the danger of
the silver being carried off by the evolution of
Sulphur gases, as the Sulphur goes into the
slag, perhaps mostly as an alkaline Sulpho
cyanide.

Potassium Cyanide may take the place of nitre in
all cases in one assay except in the presence of copper
then if charge is in reducing atmosphere a
direct oxidizer must be employed.

To this charge add 4 or 5 - 20 d nails.

If or is Silicate Carbonate, Chloride & Ground

results by trying to cupel this mixture.

Crucible assays give good checks for silver but the results should not be taken as conclusive, for they will not check as close or be as high in silver as scorification assays. I have run matte for gold and silver successfully by crucible containing as high as 18% Cu.

Scorification Assay

Use 2 1/2 in scorifier.

Crossing in copper & arsenic, spines, Matles and Matte Bullion should be run by scorification.

Cham Bullion may be directly cupelled.

If ore contains much volatile constituents it should be started at a low heat so that all these volatile matters may be slowly expelled and the heat gradually raised to the required temperature.

Too rapid expulsion of these volatile substances is always accompanied by loss of silver.

If ore contains volatile matter in small amounts it may be started hot and thus some time saved.

The time of running a scorification varies,

sometimes running as high as 80 min.
 It is a good idea to band up the front of the
 muffle with rope. This increases the heat in
 the front and by regulating the muffle door
 the heat may be kept uniform throughout the
 muffle and thus all the work finished
 at once.

Those charges when scorified until the
 button sinks are found, hammered and weighed.
 Charge for scorification assays:
 Ore 11 A.T.

That lead 42 gms.

A little fused borax or soda increases the fluidity
 of the slag.

In case ore is strong in Copper, Nickel,
 Arsenic & Antimony, or if ore is Blende or rich
 Telluride, as much as 80 gms. test lead is needed,
 but the appearance of the resulting lead button
 will indicate any foreign metals if present,
 and it is of so rare occurrence that such
 strong ores are rare that 42 gms. may be taken
 as general charge for test lead, and in those

few rare occurrences. The buttons may be reconfirmed

After the buttons are obtained from Cupellation they are weighed to determine the amount of silver present. These assays should check to within 1% silver per ton between the lightest and heaviest buttons. Generally four assays are run on each ore.

The Gold should check to .02% per ton on the assay balance should weigh to .02 mg.

After the silver buttons are weighed they are dissolved in Parting fluid with the following

- Nitric Acid 1 Part by measure
- Water 3 parts by measure

This liquid is decanted into the silver residue jar and the remainder subjected to

- Nitric Acid 2 parts
- Water 1 part

The liquid is decanted into the silver residue jar and distilled water found on the gold.

The Parting flask is inverted over an uncuped cup until gold settles in cup. Then the water is poured off and the gold dried and weighed.

The object in using dilute acid first is to obtain the gold in bright heavy particles, If strong acid is used at first, the gold will be in fine shreds of fine & will be more difficult to handle.

In case the button from cupellation contains much gold and little silver, the silver will not all be dissolved by the acid, In this case it will be necessary to fuse some pure silver with the button by blow pipe or some simple means, when all the silver will be readily soluble.

In case the ore is known to contain much gold and little silver, it is well to add to the charge a pure silver button and run this charge for gold alone.

Silver.

All silver assays should be made by decupellation, as lighter and more accurate results may be obtained by this method than by crucible, except when ore is very poor in silver.

Obviously if assay is to be made on pure button the charges may be directly cupelled but if further

Contains gross or marked amount of ~~Co~~ or containing
it should be identified, In the latter case it
may be necessary to add some test lead.

The identification is one as described under
411.

Slag

Run by Crucible

Use 1 A.T. on, 20 gms lead flux, 40 gm Pb,
also little borax. Cupel button obtained & weigh silver.

Refined lead

Use 2 A.T. Identify to required size and cupel

Mattes Strong in Copper

These should be assayed by identification and
checked by some simple wet method.

Wet Method

Dissolve in with Nitric acid, filter if insoluble
residue, To filtrate add little Sulphuric acid
and lead acetate to form a copious precipitate
collect precipitate of silver, then add Sodium
bromide to precipitate silver as Silver Bromide
filter again and add precipitate to insol.
residue and run by crucible or identification

This leaves the copper in the filtrate while the silver and gold are all in the precipitate and insoluble residue.

Copper Scrap

Copper filings or scraps to be assayed for silver should be melted in a graphite crucible in a wind furnace, charcoal should be added to fuse the copper reduced. If the scraps are very dirty and an assay is wanted of the scraps alone, the fused mass should be remelted and the dross removed by skimming, remelted and granulated by pouring in a thin stream while molten from a few feet above into a basin of cool water below. If the scraps are clean they may be granulated the first fusion.

In case an assay is wanted of the entire mass, weigh before melting and after granulating and calculate the results from this data.

Granulated copper gives a finer sample for silver than when cast in moulds.

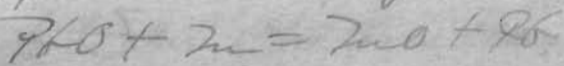
For samples from the granulated copper use wet method.

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Bullion is usually obtained in small "pigs"
and "bonnets". These should be melted quickly
in graphite crucible with Charcoal and
moulded. From the moulded cake take four
samples, one from each side by chipping
through the cake with a cold chisel.

The sample should be chipped directly from
the cake, as the top and bottom of the cake
may vary widely in richness in gold and silver.
For example, if the bullion contains copper or
zinc, the gold will readily alloy with it as will
also some of the silver and as the alloy is
lighter than lead it will settle to the top of
the cake before the mass is solidified, and
thus a sample from the top may give much
higher results, especially in gold than one
directly under it at the bottom of the cake.
5-41. is taken from each of these samples and
directly cupelled if pure, but if bullion contains
marked quantities of foreign matter it will
be necessary to first assay.

Metallic Zinc

Blue powder, strong zinc ore or even metallic zinc may be assayed for gold and silver by crucible by adding sufficient litharge to oxidize the zinc.



The resulting lead button will have to be scorified to reduce size.

Manganese Ores

Strong manganese ores may be run by either crucible or scorification, but often the resulting button should be scorified before cupellation.

Iron Ores

Occasionally, strong iron ores have to be assayed for gold and silver. In this case add sufficient silica to form an acid flux.

Metallic Tin

It is difficult to assay metallic tin for gold and silver & this the best method is to add sufficient silica to oxidize the tin in crucible and scorify the resulting button.

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Fine Silver

To determine the fineness of silver requires some care. The samples are obtained by running some of the silver from the crucible into water to granulate it. Samples are thus taken from each brick of silver cast. From these samples six or eight assays are taken which are rubbed down on a smooth whetstone until they each weigh exactly 500 mg. The same number of samples of pure silver are taken and each button is rolled in lead foil made by running refined lead through rolls until a piece 1/8" in long and about 3/4" long weighs 3 grains.

This lead should only give a trace of silver when 2 H.T. is cupelled.

These buttons are all cupelled in a hot muffle together, the lead foil removed. The crucibles, antimony, lead or other impurities.

These crucibles must be gradually moved toward the front of the muffle after the button has "brightened" if cooled too quickly the buttons will crack and shooting is generally accompanied

by loss of silver

Spitting may be due to two causes

1. Silver at a high heat absorbs oxygen from the air, and if cooled too rapidly, the oxygen in its effort to escape may cause the button to spit.

2. If a silver button is cooled too rapidly the surface will solidify and contract on the molten interior, this force of contraction may be sufficient to cause the crust to break and the button to spit.

After the cupellation is completed the buttons are removed and weighed, the loss of weight by oxidation of the pure silver is added to the weight of the silver being assayed this weight multiplied by 2 gives the fineness of the silver.

Cupellation

There is a diversity of opinions among assayers as to the best method of cupellation.

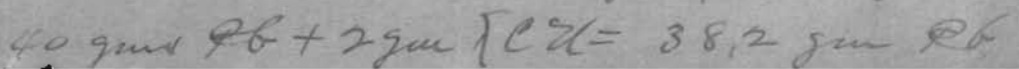
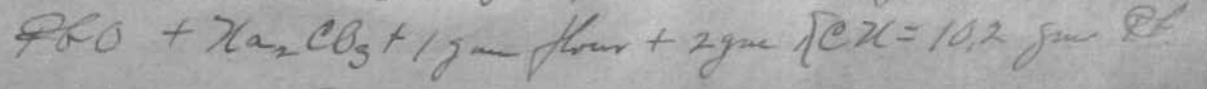
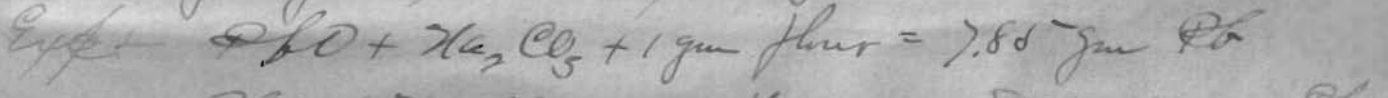
My experience for silver is that much higher and more accurate results may be obtained by cupelling the buttons as "cold" as possible and

Then gradually increase the heat on the "wind up".
 The heat may be kept down in the muffle
 by surrounding the crucible with empty crucibles
 & scraps etc. also by regulating the furnace doors.
 I will mention that I recommend cold cupellation
 on buttons when their weights are between 18 & 40
 gms. I have had no occasion to cupel buttons
 without these eyes.

Special Experiments

1. In running an arsenic ore, flux dust etc by
 crucible, there is generally a dross formed
 that sticks to the top of the lead button.
 This may be thrown away for analysis
 shows it contains none of the silver or gold.
 They having probably alloyed with the lead.
 This dross should be discarded because
 it interferes with the subsequent cupellation.

2. KCN reduces PbO in the presence of an
 reducing agent, and oxidizes it.



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3. Nails are reducing to oxide and phosphate
as well as Sulphide and Silicate ones.

4. Nitric varies in the amount of its action
as an oxidizer in the same compounds.

5. Impure litharge ($PbO_2 + PbO$) oxidizes silver
in the absence of an reducing agent but has
little or no effect in a crucible assay when
reducing agents are present.

Exp: Run 1 gm pure silver in each, and cupelled hot
all at same heat and time.

1. Pure PbO gave	98.22	gms
2. Impure PbO (Red litharge) gave	97.46	
3. " " with flour	97.4	
4. " " " " "	97.22	
5. Pure Ag cupelled with pure Pb	83.4	

Proof submitted for the degree of E. M.

by - C. G. Grover, 24 June 1894.