

		Per Ton of Lead.			Per Ton of Ore.			
		grs.	oz.	dwt.	grs.	oz.	dwt.	grs.
1. Wt. of ore 400 } ,, lead 312 }	Wt. of Ag	202	21	3	0	16	9	22
2. Wt. of ore 400 } ,, lead 313·8 }	,,	204	21	4	16	16	13	5

§ 125. Table showing amount of silver contained in one ton of ore corresponding to the weight obtained from 400 grains:—

If 400 Grains of Ore yield fine Metal			One Ton of Ore will give			If 400 Grains of Ore yield fine Metal			One Ton of Ore will yield		
gr.	oz.	dwt.	gr.	gr.	oz.	dwt.	gr.	oz.	dwt.	gr.	
·001	0	1	15	·200	16	6	16				
·002	0	3	6	·300	24	10	0				
·003	0	4	21	·400	32	13	8				
·004	0	6	12	·500	40	16	16				
·005	0	8	4	·600	49	0	0				
·006	0	9	19	·700	57	3	8				
·007	0	11	10	·800	65	6	16				
·008	0	13	1	·900	73	10	0				
·009	0	14	16	1·000	81	13	8				
·010	0	16	8	2·000	163	6	16				
·020	1	12	16	3·000	245	0	0				
·030	2	9	0	4·000	326	13	8				
·040	3	5	8	5·000	408	6	16				
·050	4	1	16	6·000	490	0	0				
·060	4	18	0	7·000	571	13	8				
·070	5	14	8	8·000	653	6	16				
·080	6	10	16	9·000	735	0	0				
·090	7	7	0	10·000	816	13	8				
·100	8	3	8								

When the silver obtained in the above processes contains gold, it is separated by dissolving out the silver in nitric acid, and the weight of the gold is deducted before computing the amount of silver present.

### ASSAY OF SILVER ORES, PRODUCTS, AND ALLOYS.

This assay may be conveniently studied under three heads, viz. :—

- I. Treatment of bullion, coin, and plate.
- II. Ores of all kinds containing silver.
- III. Various products and waste matters containing silver, such as litharge, slags, sweep, etc.

#### First Method. BULLION, COIN, AND PLATE.

§ 126. This assay is effected by means of cupellation which has been partly explained under the assay of argentiferous lead. Care must be taken that the sample employed represents the whole mass from which it is obtained, as alloys of silver and copper are liable to undergo liquation, so that different parts of the same ingot will give discordant results.

(a) Professor Roberts-Austen has made a special study of this subject. He cast the alloys in cubical fire-brick moulds, in which they could be cooled either rapidly or slowly. His results are embodied in the following table:—

No.	Name of Alloy.	Parts of Silver in 1000 of Alloy.	Rate of Cooling.	Maximum Variation of Silver in different parts.
I. a	British standard coin	925	rapid	12·8 per 1000
I. b			slow	1·4 "
II. a	Old French standard coin	900	rapid	10·1 "
II. b			slow	1·3 "
III.	Levol's homogeneous alloy	718·93	slow	1·2 "
IV.	AgCu	630·3	slow	21·1 "
V.	AgCu <sub>2</sub>	333·3	slow	12·8 "

In I. *a* and II. *a*, the centres of the cubes were the richest and the corners the poorest.

In I. *b* and II. *b*, the slight variations followed the same law.

In III., the corners were generally richer than the centre.

In IV., Roberts-Austen supposes that gravity has influenced the alloy, the lower parts being richer than the upper.

In V., the variations do not follow any known law.

From the above it will be seen that slow cooling of I. and II. renders them almost homogeneous.

(*b*) Alloys of silver with copper heated in a current of air become oxidised in proportion to the copper present, so that some indication of the amount of each may be obtained from the intensity of the discoloration. Chaudet gives the following table:—

Silver in 1000 parts of Alloy.	Colour of Surface after Heating to Redness.
1000 <i>i.e.</i> , pure silver,	Dull, but white.
950 - - -	Uniform grey-white.
900 - - -	Dull grey-white; black at edges.
880 - - -	Grey, almost black.
860 - - -	" "
840 - - -	Quite black.
820 - - -	"
800 - - -	"

(*c*) The amount of alloy now taken for assay is generally 12 grains, which must be very accurately weighed on a delicate balance. Although the amount taken is of no great consequence, yet the above quantity is convenient, as it saves trouble in computing the results; it

is known as the "assay pound." The silver assay pound is subdivided into aliquot parts, *i.e.*, into 12 ounces, each ounce into 20 pennyweights, and these again into halves, so that each nominal half pennyweight weighs  $\frac{1}{10}$ th part of a troy grain, when the pound = 12 grains. The decimal system in relation to the above weights is also used—the ounce, dwt., and half dwt. are represented by 1 grain, .05 grain, and .025 grain respectively. This is shown by the following table:—

Silver			Assay	Silver			Assay
oz.	dwt.	gr.	Grains.	oz.	dwt.	gr.	Grains.
12	0	0	12	0	10	0	0.500
11	0	0	11	0	5	0	0.250
6	0	0	6	0	3	0	0.150
3	0	0	3	0	2	0	0.100
2	0	0	2	0	1	0	0.050
1	0	0	1	0	0	12	0.025

The assay piece is enclosed in an envelope of sheet lead, the amount of which depends on the quantity of copper present.

(*d*) The following table gives the amount of lead which should be used for various alloys of silver and copper:—

Amount of Silver.	Amount of Copper.	Quantity of Lead for 1 part of Alloy.
1000	0	1
950	50	3
900	100	7
800	200	10
700	300	12
600	400	14
500	500	16-17
400	600	
300	700	
200	800	
100	900	
0	1000	

As metallic lead invariably contains silver, the amount of that metal introduced into the assay must be ascertained and deducted from the quantity of silver obtained.

(e) A number of assays having been prepared, they are placed in the compartments of a wooden tray containing nine to twelve divisions, from which they are transferred to a copper tray in the same order; this tray has forty-five to sixty depressions (Fig. 60), and from thence into previously

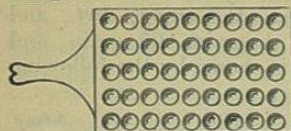


Fig. 60.

heated cupels occupying a corresponding position in the muffle. In a short time the lead melts and becomes covered with greasy-looking drops of litharge which are rapidly absorbed and replaced by others, the oxidation and absorption then continue until the base metals are removed. In from ten to twenty minutes, iridescent films appear, showing that the last traces of lead are being oxidised, which quickly disappear, leaving the bead of silver bright, known as "brightening."

The brightening, when the operation has been successfully performed, should commence with the first row, and finish with the back row. If the brightening occurs irregularly, or commences at the back of the muffle, it shows that the draught has not been properly regulated, and the results cannot be so thoroughly relied on as in the former case. The muffle is now completely closed, and the whole allowed to cool slowly to avoid "spitting," which would render the assay useless when anything like accuracy was desired.

It is advisable to keep the temperature as low as possible to prevent volatilisation of silver and absorption by the cupel. The heat should be so adjusted that only slight fumes are visible over the cupels, and the fused metal should be highly luminous and clearly distinguish-

able from the cupel. Too high a temperature causes fumes to rise to the top of the muffle; with too low a temperature, crystals of litharge will be deposited round the edge of the cupel.

(f) A certain amount of loss of silver always occurs in cupellation, which loss increases with the temperature and the amount of lead used.

To ascertain this loss, a check is used with every nine or twelve assays placed as in Fig. 61. This consists of a known weight of pure silver made up to 12 grains by copper, equal in amount to the copper probably present in the assays, and assayed with same quantity of lead in the same heat. The loss of silver in the check piece is then added to the weight of silver obtained from the assay. In places where large quantities of standard silver assays are made, a specially prepared alloy, known as trial plate, is used for checks.

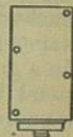


Fig. 61.

(g) The silver beads or "prills" should be round on the top, or only slightly depressed in the centre; should be readily removed from the cupels, and have a bright appearance. A flattened appearance indicates the presence of copper; also when they adhere firmly to the cupel, or throw out projections near the bottom, it shows they are not "fine," and that an insufficient amount of lead has been used.

Before weighing the button of silver, it should be squeezed laterally with a pair of round-nosed pliers, or tapped on the edge with a clean hammer, to detach any adhering bone ash, and brushed with a hard tooth brush. If this fails to remove all foreign matter, soak in dilute hydrochloric acid, and brush till clean.

(h) It is always an advantage to the assayer to know the approximate composition of an alloy before proceeding to

obtain a correct estimate of the silver. The character, and roughly the amount of other metals, may be judged by the indications of the cupel in a preliminary assay. Thus lead produces an orange red colour; manganese a bluish black stain and corrosion of cupel; iron a reddish brown stain at the commencement and dark stain at finish; copper a dark green or brown; cobalt a dark green incrustation and green stain; chromium a dark brick red stain; arsenic a white or pale yellow; antimony a red-brown incrustation, which, if present in any amount, cracks the cupel; nickel a dark green slag and green stain; platinum a greenish stain and crystalline button; tin a grey incrustation; zinc a yellow ring on cupel, the metal burning with a brilliant flame, emitting copious vapours and corroding the cupel.

(i) The presence of platinum, palladium, iridium, and rhodium requires a higher temperature for the cupellation. The surface also of the button is dull after the operation has terminated. Iridium always sinks to the lower part of the button, so that on rolling out the button into a strip a black spot or streak will be observed on the under surface.

*Second Method.* SILVER ORES.

§ 127. (a) The fluxes and re-agents employed are red lead, or litharge, granulated lead, and sheet lead. These should be tested previously to ascertain the amount of silver they contain, if that metal is present in notable quantity. For this purpose the above oxides may be reduced by heating with charcoal at a low red heat, and the buttons of lead obtained, cupelled. Red lead is preferable to litharge, the common varieties of which contain more silver than red lead.

Carbonate of soda and carbonate of potash in powder, charcoal, and anthracite.

Tartar or red argol, which has a higher reducing power than pure cream of tartar.

Nitre in powder.

Fluor-spar, free from lead, which is argentiferous.  
Hoop iron or wrought nails for reducing sulphides.

(b) *Sampling.*—The ore must be roughly crushed, an average sample of  $\frac{1}{2}$  lb. taken and weighed. This portion is then dried at 100° C. and re-weighed, so that the return may be made on the dry or wet sample. The ore is then finely crushed in an iron mortar, and passed through a sieve with 80 meshes to the inch. It sometimes happens that metallic particles remain on the sieve; these are assayed separately, and the proportionate amount of silver added to each aliquot part assayed.

§ 128. *Estimation of the Silver.*

This may be determined by one of two methods, or by both, as a check on each other.

(a) 1st. *By Fusion in a Clay Crucible.*—This method is suitable for ores of silver containing much gangue and only a small proportion of other metals. The following quantities are suitable proportions for an average ore:—

Ore, 200 grains; red lead, 500; charcoal, 25; sodium carbonate, 300; borax, 200.

This mixture, however, would not be suitable in all cases, as carbonate of soda, being a basic substance, must be increased when a large excess of silica or other acid material is present, and the borax increased in case of much lime, oxide of iron, etc. When barytes, gypsum, or phosphate of lime is in the gangue, fluor-spar must be added.

The materials are mixed together, transferred to the crucible, covered with part of the borax, and placed well down in a moderate fire. For the first ten minutes a medium temperature is best, then gradually increase for another eight or ten minutes until the liquid becomes tranquil; then pour into a round mould, allow to cool,

and detach the slag, which, on account of its brittleness, readily separates on hammering from the malleable button of lead. It occasionally happens that the reduced lead is covered with a regulus, or speise, which may contain silver, in which case it would be advisable in assaying another portion to add a strip of iron to decompose the sulphide or arsenide present. But this omission may be remedied by adding iron to the mixture used for cleaning the slag.

The slag is generally glassy, but varies with the nature of the ore, and should be uniform in colour and composition.

(b) *Cleaning the Slag.*—The slag is pounded in a mortar covered with a duster to prevent loss, and mixed with 300 grains red lead, 15 grains charcoal, and 20 grains carbonate of soda. Fuse for about fifteen minutes in the same crucible in which the ore was smelted, and pour into round mould as before. The slag obtained in this case is thrown away, and the lead button after cleaning saved for cupellation.

(c) The two lead buttons are then cupelled, particulars of which have been given in § 124.

§ 129. (a) Sulphides of copper, iron, lead, etc., containing silver, may also be assayed by the crucible method just described; but the sulphur, arsenic, etc., must be oxidised by a preliminary roasting, or a large quantity of red lead must be added in the fusion mixture.

The oxidation is effected by heating the powdered ore at a low temperature at first, in a roasting dish made of fire clay, constantly stirring with an iron rod, as the material, especially in the presence of lead, has a great tendency to fuse and clot together, which prevents oxidation to a great extent. In about fifteen minutes the greater part of the volatile matters will be expelled, and the temperature may then be raised to complete the operation.

(b) The quantity taken for roasting should vary with the richness of the ore, but the following may be taken as an average sample.

Take 200 grains and roast as above, then mix with red lead, 1000 grains; charcoal, 35 grains; borax, 400 grains; and carbonate of soda, 200 grains.

The above mixture is placed in a clay crucible, covered with borax, a strip of iron introduced to decompose any sulphides, and the operation conducted precisely as described in § 128.

(c) If the ore is fused direct without roasting, then take: ore, 200 grains; red lead, 2000 grains; charcoal, 35 grains, and white sand, 150 grains. When there is much sulphur present, the charcoal is unnecessary, and a little nitre may be added with advantage to assist the red lead in its oxidation.

This method of assaying is objectionable because of the large amounts of lead obtained for cupellation; for one part of iron pyrites requires  $8\frac{1}{2}$  times its weight of lead, and grey copper ore 6 to 7 times its weight. The nitre, however, may be judiciously used to assist oxidation. The sand diminishes the action of the bases on the crucible, and may be omitted if an iron crucible be used.

§ 130. As a check on the two preceding methods, digest 200 grains of the ore with strong nitric acid and heat for an hour, adding nitric acid occasionally as it evaporates. Then add water and boil; filter off any insoluble matter; dilute the solution with water, if necessary; boil, and add sufficient hydrochloric acid to precipitate the silver as chloride. If lead is present it will also be partly precipitated, unless the liquid is kept near boiling. Filter off the insoluble silver chloride; well wash with boiling water, which also dissolves out any lead chloride; dry, and heat in a weighed porcelain crucible till the chloride just begins to fuse, then allow to cool and weigh. After deducting the weight of crucible and

filter-ash, the amount of silver is obtained by the following proportion:—

$$143.5 : 108 :: \text{Weight of precipitate} : x.$$

As 200 grains have been taken  $\frac{x}{2}$  = the percentage.

The insoluble residue is mixed with 300 grains red lead, 15 charcoal, 50 borax, and 100 carbonate of soda. Fuse as before in clay crucible, cupel lead, and add the weight of silver button, if any, to the quantity obtained above.

§ 131. In experiments performed on a sample of pyrites to test the three modifications just described, the following results were obtained:—

With prelim. calcination	Direct fusion.	Solution in nitric acid.
* 546 grains Ag.	541 grains Ag.	491 grains Ag.
oz. dwt. grs.	oz. dwt. grs.	oz. dwt. grs.
89 3 14 per ton.	88 7 6 per ton.	80 17 0 per ton.

For the mode of calculating the amount of silver per ton of ore see Lead Assay, § 124 d.

§ 132. 2nd. *By Scorification in a Fireclay Dish.*

(a) This operation is very simple and rapid in its action, but more experience is required to obtain trustworthy results than by fusion in a crucible. It depends on the principle that molten oxide of lead has the power of uniting with infusible silicates such as fireclay, dissolving other bases which may be present, such as oxides of copper, iron, antimony, etc., and forming a fusible slag, leaving unoxidisable metals, such as silver and gold, in combination with the metallic lead present in excess.

(b) All ores and products containing silver may be assayed by this method, but as scorifiers are much

\* In calculating the results, allowance must be made for any silver contained in the red lead employed. See § 139 d.

smaller than crucibles in capacity, a less quantity of ore can be operated on, so that very poor ores are best treated by the crucible plan. It has been explained that oxide of lead in sufficient quantity completely oxidises sulphur, arsenic, etc., as well as base metals, so that this method is well suited for pyritic ores containing silver. Moreover, the lead can be scorified to any convenient size suitable for cupellation. The oxidation of impurities is partly effected by the air and partly by the litharge, which occasionally causes spurting by the effervescence ensuing on this reaction. The lead employed should be in fine grains, which are obtained by pouring lead near its point of solidification into a rectangular box previously blacklead to prevent the metal sticking, and then vigorously shaking to granulate it. Uniformity of grain is desirable, and may be obtained by passing the above through a fine sieve.

(c) The following proportions may be taken, when the ore or product is of medium quality:—

Finely crushed ore passed through No. 80 sieve, 100 grains.

Granulated lead,	1000 grains.
Borax,	10 "

Mix the ore with half the lead; place in the scorifier (Fig. 41); then spread the remaining lead on top, and on this put the borax, whose function is to lessen the action on the vessel and make a more liquid slag. The scorifier and its contents are now placed in a muffle, previously raised to a bright red heat, the door closed until the mass is in full fusion; then the upper half of the door is opened to admit air for oxidation of lead, etc. White vapours now rise from the surface and litharge is rapidly formed. This action is continued until the slag formed just covers the lead beneath; the temperature being now raised the slag becomes very liquid. The size of the

scorifier employed should be such that the contents about half fill it. The slag contains a little silver which is separated by adding 5 to 8 grains of anthracite powder wrapped in tissue paper, and placed quickly on the top of the slag; this causes effervescence and rising of the contents, which, with too small a vessel, will pass over the sides and vitiate the assay. When all action ceases the scorifier and its contents are removed, both metal and slag being poured into the same dry mould. When cold, the slag is detached as usual by hammering on an anvil. The cleaned button of lead is then cupelled and the amount of silver calculated to the ton as before.

(d) The lead obtained should be soft and malleable. If brittle, too little lead has been used, or the scorification has not continued long enough. If the slag is not uniformly liquid when ready to pour, add more borax. In some cases it may be necessary to stir the slag with an iron rod to incorporate any lumps with the slag.

(e) Instead of cleaning the slag with anthracite, it may be cleaned after detaching from the lead, in the manner described under the crucible method, § 128*b*.

§ 133. Gold is occasionally present with the silver obtained by cupellation of the lead. The button should then be flattened and heated with dilute nitric acid for fifteen minutes, the liquid poured off, and the residue boiled with strong acid for a few minutes; the gold is washed, dried, ignited, and weighed. The difference gives the silver.

Full particulars relating to parting gold from silver are given with gold assaying, § 136*e*.

§ 134. *3rd Method. Treatment of Regulus, Litharge, Old Cupels, Slags, Sweep, and other Waste Matters.*

(a) The fluxes employed will of course vary with the

nature of the foreign matter, and the quantity with the amount of matter to be scorified. The following proportions may be taken for jewellers' sweep:—

1 oz. of sweep.  
 $1\frac{1}{2}$  oz. of potash.  
 $1\frac{1}{2}$  oz. of borax.  
 $1\frac{1}{4}$  oz. of salt.  
 $\frac{1}{4}$  oz. of nitre.  
 40 grains anthracite.  
 2 oz. of red lead.

If the substance contains much coal or other carbonaceous matter, omit the anthracite, and substitute  $\frac{1}{2}$  oz. of lime.

If the substance contains lime, then add  $\frac{1}{2}$  oz. of white sand to the above mixture.

In the presence of sulphides, add a piece of hoop iron or a large wrought iron nail.

Another excellent flux is:—

2 oz. potash.  
 1 oz. salt  
 $1\frac{1}{2}$  oz. borax.  
 40 grains nitre.  
 40 grains anthracite.

Well mix the fluxes with the assay sample, leaving the salt as a covering. Commence with a gentle heat, raising the temperature gradually. Towards the close, when the violence of the action has nearly ceased, place a cover on the pot, and finish with a strong heat. About twenty minutes are required. Remove the iron; allow crucible to cool; break open; detach button of lead; and cupel for silver and gold as before. Clean the slag as in previous cases.

(b) *Treatment of Old Cupels for Silver and Gold.*

The crushed bone-ash containing litharge and other oxides is mixed with sufficient charcoal to reduce the

metallic oxides, and as fluxes, the following proportions may be taken:—

Old Cupels, 100 grains; fluor-spar, 150; borax glass, 150; carbonate of soda, 150.

Fuse in a clay crucible, gradually raising the temperature as the operation proceeds, finishing with a strong heat. Pour into mould; allow to cool; tap away the slag, and cupel the button of lead for silver and gold in the usual way.

(c) *Metallurgical Products, Sweep, etc.*

The following system is used at the Royal Mint. 2400 grains are weighed, dried and reweighed; the difference between the two weighings giving the moisture. Calling the second weight found  $W$ , and the weight of moisture  $M$ , then  $W = (2400 - M)$  grains. The dried sample is ground in an iron mortar, passed through a sieve of 60 holes to the linear inch, and the metallic residue cupelled separately. Calling the weight which passes through the sieve  $W_1$ , it is evident that  $\frac{W_1}{4}$  will be the proportion obtained from 600 grains of the initial sample. This weight  $\frac{W_1}{4}$ , is now roasted at a red heat in a muffle to remove all combustible matter. It is again weighed on cooling and its weight  $W_2$ , divided into three equal portions for scorification. Each portion  $\frac{W_2}{3}$  will thus correspond to 200 grains of the original sample.

For each scorification charge are taken 800 grains granulated lead, and 50 to 100 grains of borax. Half the lead is mixed with the charge, and the other half is placed on the top. The scorification is then conducted as described on page 240.

ASSAY OF GOLD ORES, PRODUCTS, AND ALLOYS.

§ 135. As gold is so closely connected with silver, both metals frequently existing in the same material, and as the same process may generally be applied to both, it follows that much of what has been said concerning silver assaying, applies also to gold. However, this metal having a much higher value, substances containing it even in minute quantities may be profitably treated for its extraction, so that in many cases much larger quantities require to be operated upon to obtain the precious metal in a weighable amount, and consequently crucibles of large capacity are required.

§ 136. 1ST. ESTIMATION OF GOLD IN ALLOYS.

(a) In places where a large number of assays have to be conducted, a special set of weights is employed as with silver, the unit quantity being termed the assay pound, which is sub-divided into carats, carat grains, eights, and excess grains. The amount taken as a unit may be 10 grains or half a gramme = 7.716 grains. The relation of the parts are well shown in the following table by Prof. Roberts-Austen:—

				Excess grains.	Decimal equivalent.
			Eighths	1	.1736
		Carat-grains.	1	7.5	1.3021
		Carat.	1	8	10.416
Assay pound	1	4	32	240	41.6
1	24	96	768	5760	1000.0



The excess grains in one assay pound are the same as the number of grains in the troy pounds. Gold is reported to the trade according to the above table in carats or the decimal equivalent; thus pure gold is 24 carat or 1000 fine; standard gold, 22 carat =  $\frac{22 \times 1000}{24} = 916.66$  fine. When an alloy is slightly "worse" than the standard, it is said to be "worse so much." When above the standard, the alloy is called "better so much," the difference being expressed in carat grains, eighths, and excess grains, or in its decimal equivalent. In both cases the excess grains represent gold present in excess of the report.

(b) In America assays of gold ores and bullion are reported in money value per ton. One troy ounce of gold 1000 fine = \$20.67.

Phillips gives the following standard values for different countries:—

Countries.	1000 (24 Ct.)	916.66 (22 Ct.)	900 (21.6 Ct.)
United Kingdom, troy oz.,	£4 4 10	£3 17 10	£3 16 6
United States, "	\$20.67	\$18.95	\$18.6
France, kilogramme,	Fr. 3444.44	Fr. 3157.40	Fr. 3100
Germany, "	Mk. 2790	Mk. 2474.16	Mk. 2511

The principal standards for coinage are:—

	Gold, per 1000 parts.
Austria and Holland (old ducat gold),	986 and 983
United Kingdom, India, Russia (old),	
Portugal, Turkey, and Brazil,	916.66
France, Germany, United States, Italy,	
Spain, Russia (new),	900
Egypt, Mexico, Spanish Philippine Islands,	875
Most other countries	900 fine.

(c) Alloys of gold chiefly contain copper and silver,

but other metals are occasionally present. In the case of silver alone, the alloy is simply adjusted by fusion with silver, so that the silver equals  $2\frac{1}{2}$  times that of the gold, and the latter separated by its insolubility in nitric acid. With copper, cupellation is necessary, and with notable quantities of nickel, antimony, tin, etc., scorification may be required before cupellation.

(d) Method of conducting the process when Silver, Platinum *etc.*, are absent.

Weigh out either 10 grains, or  $\frac{1}{2}$  gramme, and wrap in a quantity of sheet lead, the remaining lead being introduced in the form of a bullet, which may be ascertained by reference to the following table:—

Amount of gold in alloy.	Lead required for cupellation for one part of alloy.
1000	1
900	10
800	16
700	22
600	24
500	26
400	34
300	
200	
100	

The prepared samples are then transferred to trays and thence to the heated cupels in the muffle, as described for silver in § 126*e*, but as gold has a much higher melting point than silver the temperature required to finish must be higher. After brightening, the assay should be cooled in the closed muffle, although it may be removed as soon as it is solid. It is then cleaned and weighed. As most gold contains silver it is better to proceed as follows:—

(e) Silver being present.

The great majority of alloys employed by jewellers and

goldsmiths contain silver as well as copper, in which case the 10-grain sample is wrapped in an envelope of sheet lead, together with  $2\frac{1}{2}$  times as much silver as the gold supposed to be present (which in an unknown alloy is determined by a rough preliminary assay), a lead bullet added as before, and the whole cupelled. Then the cupel is allowed to cool in muffle as before, and the button cleaned from any adhering bone ash by squeezing with pliers or hammering and brushing with a hard tooth brush, or immersing in warm hydrochloric acid; then dried and weighed. It is then flattened on a bright steel anvil with a heavy round-faced hammer whose head is polished bright and smooth. The button is first struck in the centre, then near the edge to elongate it so that it may be more readily gripped by the rolls. It is then annealed and rolled out into an oblong strip about two inches long, again annealed, and coiled into a spiral, the outer surface of which should be that which formed the bottom of the bead. This side of the strip is easily recognised by being less brilliant than the other.

The spiral or "cornet," as it is termed, is then placed in a glass flask having a long neck and capable of holding

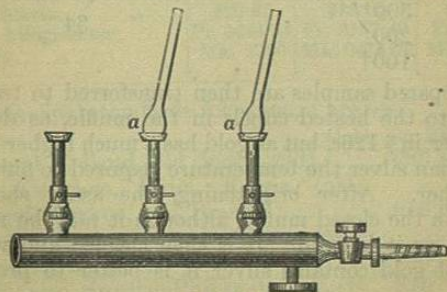


Fig. 62.

about 5 to 6 ounces (Fig. 62). Two ounces of dilute nitric acid, containing two parts water to one of strong

acid, are added, and gently boiled for ten minutes, until all brown fumes have disappeared, then the silver solution is poured off. A small ball of fireclay about the size of a pea is placed in the flask to prevent irregular ebullition, which might break up the cornet and cause loss by spurting. Stronger acid, of the proportion 1 : 1, is then added and boiled for ten minutes. The cornet is boiled in strong acid to finish. Pour off the liquid, wash the gold with pure distilled water, and fill up the flask to the top with same. Now cover the open end with the thumb, invert, and place in a small crucible full of water. This operation of inverting without spilling any liquid may, after a little practice, be easily done, without stopping with the thumb. The gold cornet and any detached fragments fall to the bottom of the crucible in virtue of the high specific gravity of gold; the greater portion of water can now be poured off and the rest evaporated. The dry crucible with the cornet, which is of a dull brown colour, is then placed in a red hot muffle for a short time, when the gold acquires the usual yellow colour with a considerable shrinkage in bulk. The gold is allowed to cool and then weighed.

The amount taken was 10 grains. The weighing after cupellation = silver and gold present, plus the silver added for parting, which was  $2\frac{1}{2}$  times that of the gold. The weighing after parting = gold; and the difference from former weighing = silver.

(f) The accuracy of an assay cannot be relied on without a "check" assay being made at the same time, as silver or copper may be retained, or gold absorbed by the cupel, so that the error may be in excess, or below the true amount. The "check" contains the same amount of pure gold as is supposed present in the alloy, made up to 10 grains (or whatever weight is used in the sample) with pure copper so as to make it completely comparable.

Prof. Roberts-Austen points out "that as absolutely pure gold cannot be obtained, the results of assays indicate an amount of pure gold in excess of that actually present. But the opposite cannot occur, as gold cannot be more than pure. The correction is shown by the following formula:—

Let 1000 represent the weight of alloy taken.

- " p " " " gold obtained.  
 " x " " actual amount of gold in alloy in thousandths.  
 " a " " weight of gold (almost pure) taken as a check which approximately = x.  
 " b " the loss or gain in weight experienced by (a) during the assay, expressed in thousandths.  
 " k " the variation of the 'check gold' from purity.

Then the amount of fine gold in check =  $a(1 - \frac{k}{1000})$  and x the corrected weight of assay will

$$= p - \frac{ak}{1000} \pm b$$

(b) being subtracted or added according as it is loss or gain. If (a) be assumed = x this equation becomes

$$x = \frac{p \pm b}{1 + \frac{k}{1000}}$$

Example. Let p = 917.1 thousandths.

$$a = 920.0$$

$$b = .3$$

$$k = .1$$

Then by first formula,

$$x = 917.1 - \frac{920 \times 0.1}{1000} - 0.3$$

for as (b) is a gain in weight it must be deducted. Hence  $x = 917.1 - 0.092 - 0.3 = 916.708$ , and by second formula

$$x = \frac{917.1 - 0.3}{1 + \frac{0.1}{1000}} = 916.708.$$

These results show that although (a) does not exactly = x, no sensible error is introduced." (Percy's *Gold and Silver*, p. 274.) From the foregoing table it will be seen that gold sometimes retains a little silver by which its weight is increased. This is known as "surcharge," and most commonly occurs when only a small quantity of copper is present, as when the latter is considerable, much lead is required for cupellation, which causes more gold to be absorbed by the cupel. It will also be readily understood that with a medium quantity of copper, the surcharge and absorption may just balance each other.

(g) The following table, showing results of experiments made at the Paris Mint, illustrates this point:—

Copper.	Gold.	Results.	Difference.
100	900	900.25	+ 0.25
200	800	800.5	+ 0.5
300	700	700	0.0
400	600	600	0.0
500	500	499.5	- 0.5
600	400	399.5	
700	300	299.5	
800	200	199.5	
900	100	99.5	

Having weighed the gold obtained from assaying the "check pieces," the excess or deficiency in weight is subtracted or added to the weight of the gold obtained from the assayed sample.

(h) In places where a large number of assays are frequently being made, platinum vessels are often used for parting instead of glass. A shallow platinum dish is

furnished with a rack containing a number of divisions, each holding a small perforated thimble-shaped vessel

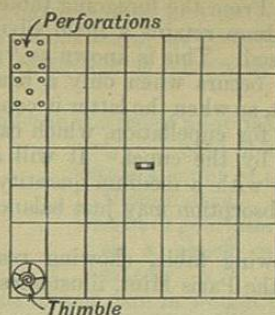


Fig. 63.

made of platinum, which admits of free entrance of the acid contained in the dish. The dish is heated from beneath to boil the acid, and covered at the top with an inverted glass funnel to carry off the fumes. By raising the rack the acid is drained off from all the vessels at once. The apparatus is shown in plan and section in Figs. 63 and 64.

§ 137. *Assay by the Touchstone, etc.*

(a) This is a rough and rapid method of approximately ascertaining the quality of a gold alloy without injury of the article as is the case in dry and wet assaying. An experienced person may determine the correct standard within 1 per cent. of the truth. The method is based on the fact that the richer an alloy is in gold, the more clearly does a streak drawn with it on a black ground exhibit a pure golden yellow colour and the less is it attacked by a test acid.

(b) The touchstone is a hard siliceous stone of a black

colour obtained from Bohemia, Saxony, and Silesia, its surface being prepared and left so that it will just abrade

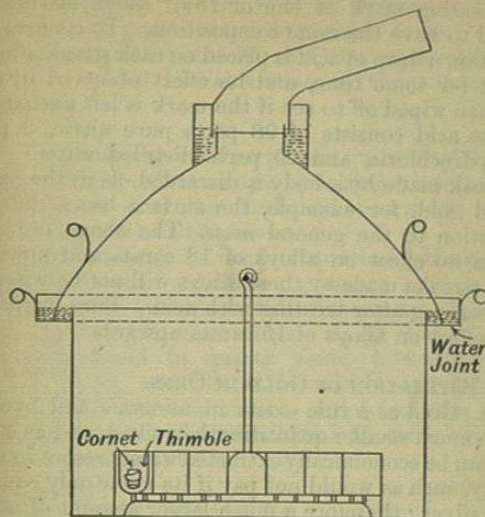


Fig. 64.

the metal from any sharp angle of the alloy when the latter is drawn over the stone.

(c) In order to ascertain the quality of the alloy, its streak is compared with streaks drawn by alloys of known fineness called "touch needles," of which five series are required: 1. *Red series*, consisting of gold and copper, the gold increasing by half carats in successive needles. 2. *White series*, contains gold and silver. 3. *Mixed series*, in which the quantities of silver and copper alloyed with the gold are equal. 4. *Unequal mixed series*, in which the silver is to the copper as 2 : 1. 5th series, in which the silver is to the copper as 1 : 2. Besides these, special

needles are prepared for different kinds of work. The mark left on the stone having been matched with the corresponding mark of one of the "touch needles" is assumed to have the same composition. To confirm this assumption a drop of acid is placed on each streak, allowed to work for some time, and its effect observed in each case; then wiped off to see if the mark is left unchanged. The test acid consists of 98 parts pure nitric, 2 parts pure hydrochloric, and 25 parts distilled water. The first streak made by a body is discarded, as in the case of coloured gold, for example, the surface has a different composition to the general mass. The above test mixture has no effect on alloys of 18 carats and upwards, so that streaks made by these alloys will not be wiped off with linen rag after treating with acid. Pure nitric acid has no effect on alloys of 15 carats upwards.

#### 2nd. ESTIMATION OF GOLD IN ORES.

§ 138. Gold as a rule exists in minerals and bye-products in much smaller quantities than silver, and as stated before can be economically extracted when present in small quantity, such as would not pay if its value only equalled that of silver; therefore a much larger amount of matter must be operated upon to obtain an amount of gold capable of being accurately weighed. Bodies containing gold frequently contain silver also, so that it is usual to examine the button of gold for that metal, which, if present, must be parted from the gold and both metals estimated.

(a) *Sampling.* The greatest care should be taken to obtain a typical sample, which must correctly indicate the average richness of the whole vein. This is an easy matter when the metal is in fine grains uniformly distributed throughout the rock, which is a rare case; but more often it occurs in irregular grains, pockets, nuggets, etc., so that two tons or more should be crushed

and thoroughly mixed in order to select about 20 lbs. for the assay sample. This must be crushed very small and passed through a fine sieve of 80 meshes to the inch. Any flattened grains remaining which cannot be sifted, must be assayed separately, and the proportion of gold and silver added by calculation to the results of the assays.

(b) The assay may be performed in a crucible or in a scorifier as with silver assays, by taking advantage of the solvent power of molten lead, and afterwards cupelling and parting if silver be present.

The matrix may be sand or quartz; pyrites, galena, blende, and other sulphides; granite; oxides of iron, etc. The rocks enclosing gold veins are mostly chloritic, talcose, and argillaceous slates. Gold *in situ* is most frequently found in quartz veins, intersecting metamorphic rocks, and almost invariably associated with pyrites. The most abundant supply of the gold of commerce is obtained from alluvial deposits by washing it from its associated sand.

#### § 139. Assay of Gold Quartz.

Pass 3000 grains of the finely-powdered ore through a sieve with 80 meshes to the inch; collect any "metallics" to assay separately; well mix, and take the weight separately of what has passed the sieve and the metallics that remain.

(a) Weigh out 500 grains ore, 500 grains red lead, 20 to 25 grains charcoal, and 600 grains carbonate of soda. Mix and introduce into a clay pot, which should not be more than half full; cover with a layer of salt, and place well down in a hot fire, a hole having been made previously to receive it, so that the mouth of the crucible is below the lower level of the draught hole; well pack round with red hot coke, the object being to insure as

far as possible a uniform temperature in all parts of the crucible, which will prevent bubbles rising to the top and carrying a portion of the charge over the sides. Raise the temperature gradually as the operation proceeds, by opening damper and closing the back brick on top of furnace, which may be drawn from the wall a little way at first. In about twenty minutes the charge settles down tranquilly in the pot, and should then be poured into a half-round mould; allowed to cool and the button of lead detached, cleaned, and put ready for cupelling.

The button of lead should weigh at least 300 grains or there will be a danger of gold being left in the slag. If less than this amount, it indicates an insufficiency of reducing agent, or the oxidation of foreign matter by the oxide of lead and passage of latter into slag along with these oxides.

(b) *Instead of the foregoing fluxes* take 500 grains of ore, 500 grains red lead, 25 grains charcoal, 400 grains carbonate of soda, and 200 grains borax. Fuse and extract button of lead as before. Weigh the lead obtained.

(c) *Or smelt* 500 grains of ore with 1500 grains red lead and 30 grains charcoal. Treat as before and weigh the lead.

(d) Experiments were made on a given ore with the above mixtures with the following results:—

Weight of lead,	-	380	362	770
"    silver,	-	·020	·019	·011
"    gold,	-	·027	·027	·028
Wt. of Ag in red lead,	-	·012	·012	·036

The following shows the mode of calculation:—

Weight of silver and gold,	-	·059	·058	·074
Weight of silver in red lead,	-	·012	·012	·036
Gold and silver in ore,	-	·047	·046	·038
Weight of gold alone,	-	·027	·027	·028
Weight of silver,	-	·020	·019	·010

(e) The amount of silver and gold per ton was obtained by the following calculation:—

As 500 : ·027 :: 7000 × 2240 : Au per ton.

500 : ·020 :: 7000 × 2240 : Ag per ton.

Table showing results:—

Ore.	Silver.	Gold.	Silver, per ton.			Gold, per ton.		
			oz.	dwts.	grs.	oz.	dwts.	grs.
500	·020	·027	1	3	3	1	15	7
500	·019	·027	1	2	10	1	15	7
500	·010	·028	0	13	2	1	16	4

The above results are given here to show that with a large proportion of oxide of lead a larger amount of gold is obtained, but as silver is lost in cupellation in proportion to the quantity of lead operated upon, the amount of that metal is too low in the 3rd method. Also in the second case the amount of carbonate of soda was too small to flux the acid gangue present; therefore a preliminary assay should be made to ascertain the nature of the foreign matter before making the true assay.

(f) Having obtained the gold in the button of lead the latter is cupelled as before described, the gold weighed and calculated to the ton as in § 139 (e). If the bead is yellow in colour, add  $2\frac{1}{2}$  times its weight of silver; wrap both in lead, and cupel to obtain the

alloy in a single bead; flatten with hammer; roll out thin, and part with nitric acid as described in § 136, to ascertain the amount of silver, if any, as well as the gold.

§ 140. *Assay of Pyritic and other Sulphur Ores.*

(a) Ores containing a large amount of sulphur, arsenic, zinc, etc., should undergo a preliminary roasting to remove the excess of these volatile impurities, or there is danger of the formation of oxysulphides, which, being very fusible, resist reduction and carry silver with them into the slag. The same result may be achieved by adding nitre to the fluxes in smelting, but too much of this re-agent causes effervescence with the consequent liability to boil over. Arsenic and antimony produce arseniates and antimonates which convey silver into the slag. Zinc increases loss of silver by volatilisation.

(b) The roasting is performed in a fire-clay roasting dish placed in a muffle, gradually raised to a bright red heat and occasionally stirred for about half an hour until no further odour of sulphur dioxide is perceived, when the dish is taken out and a current wafted to the nose by means of the hand, in the same way as in roasting copper regulus.

(c) Make two assays, using the following proportions, which must be increased according to the paucity of precious metal present:—

(1) Ore, - - - 1000	(2) Ore, - - - 1000
Carbonate of soda, 1200	Carbonate of soda, 800
Red lead, - - - 500	Borax, - - - 400
Carbon, - - - 30	Red lead, - - - 500
	Carbon, - - - 35

Proceed as in the former case with gold quartz. After introducing the ore mixture into the crucible and covering with salt, force to the bottom a piece of hoop

iron to reduce lead sulphide, etc., which might otherwise retain some of the silver. Even if lead sulphide be absent from the ore, it would be formed by the reaction of lead on iron pyrites.

(d) When the ore contains sufficient pyrites to reduce a suitable quantity of lead for cupellation, the reduction may be effected by fusion with red lead or litharge and carbon, thus:—

(3) Ore, 1000; Red Lead, 3000; Carbon, 75.

These three proportions were applied to a suitable ore, the lead cupelled; the bead alloyed with silver, and parted.

(e) The following results were obtained:—

Weight of lead, - -	430	385	Not weighed.
Silver and gold, - -	·116	·103	·184
Silver in red lead, - -	·024	·024	·072
<hr/>			
Gold and silver in ore, - -	·092	·079	·112
Gold after parting, - -	·066	·058	·078
<hr/>			
Silver in ore, - -	·026	·021	·034

Ore.	Gold.	Silver.	Gold, per ton.			Silver, per ton.		
			oz.	dwt.	grs.	oz.	dwt.	grs.
1000	·066	·026	2	3	3	0	17	0
1000	·058	·021	1	17	21	0	13	17
1000	·078	·034	2	10	3	1	2	5

§ 141. *Assay of Gold Ores by Scorification.*

This method is carried out in the same way as with silver ores, § 132, but larger quantities as a rule require to be operated upon. With poor ores, eight or ten separate scorifications are carried on simultaneously with

the same ore, each scorifier containing 50 to 100 grains of ore and the resulting buttons of lead placed in one scorifier and re-scorified until the lead is reduced to a sufficiently small bulk for cupellation. The quantity of lead required varies with the nature of the gangue. Argentiferous and auriferous lead ores only require five times their weight of lead, while arsenical and pyritic ores require fifteen or sixteen times their weight. The following table may be used as a guide:—

Quartzose, - - - - -	8
Basic (oxides), - - - - -	8
Lead ores, - - - - -	5
Arsenical and antimonial, - - - - -	16
Fahl ore, - - - - -	12 - 16
Pyritic ores, } - - - - -	10 - 15
Blende, } - - - - -	

The borax must be used very sparingly, in just sufficient amount to form a liquid slag, or it may form a crust over the ore and lead, preventing oxidation. It is better to add a few grains at first, and the remainder at intervals as the process advances.

#### ASSAY OF TIN ORES, Etc.

§ 142. The tin of commerce is obtained from its oxide, known as cassiterite or tin-stone, which may generally be recognised by its appearance and high specific gravity which varies from 6.3 to 7.1 in good specimens. When pure, it contains 78.62 per cent. of tin. Tin pyrites is of a yellowish grey colour with strong metallic lustre, having a specific gravity of 4.35, but does not occur in sufficient quantities to be used for extracting the metal on the large scale. It is chiefly found mixed with other sulphides. The following analyses show the composition of good specimens of tin ores:—

Cassiterite.		Tin pyrites.	
Tin, - - - - -	77.50	Tin, - - - - -	27.55
Oxygen, - - - - -	21.50	Copper, - - - - -	29.39
Ferric oxide, - - - - -	0.25	Iron, - - - - -	12.44
Silica, - - - - -	0.75	Sulphur, - - - - -	29.64

Tin ore as taken from the lode contains from 2 to 5 per cent. of cassiterite, which is often associated with one or more of the following bodies:—Quartz felspar, chlorite, schorl, and other stony minerals, mispickel, iron and copper pyrites, iron oxide and wolfram. Cassiterite, having a high specific gravity, is separated from its gangue by a series of washings, which remove the lighter materials. The mispickel and pyrites are converted into oxides by roasting and removed by a subsequent washing. The concentrated product is known as "black tin" (varying from 60 to 72 per cent. of pure tin), and the common impurities are silica, copper and iron oxides, and wolfram. In certain stream tin ores there may be as much as 7 per cent. of titaniferous iron ore.

(a) Before any variety of tin ore is assayed it is necessary to separate the oxide from its associated gangue, which is very skilfully accomplished by an experienced person on a "vanning shovel" (Fig. 23). For this purpose the ore is very finely crushed and sifted so as to obtain all the particles of the same size. The amount of ore taken for the purpose varies with the richness. With poor ores an ounce or 250 grains may be taken; with rich ores half or even a quarter of this amount is used. The weighed portion of ore is placed on a vanning shovel. The vanner stands in front of a tub of water, and allows a little water to run on to the ore. He then raises the shovel a little above the surface of the water, and, holding it nearly horizontal, imparts to it a circular motion until the whole is brought to the centre: by this motion, too, the lighter portions will travel to the top and the