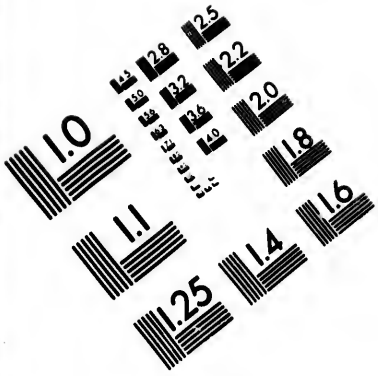

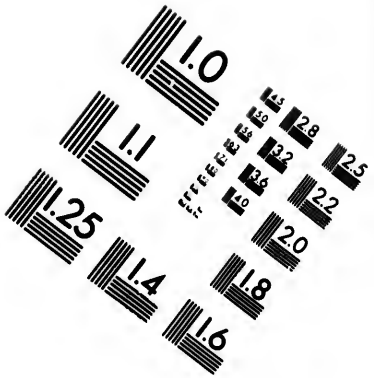

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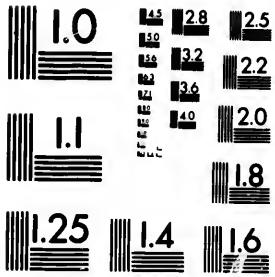

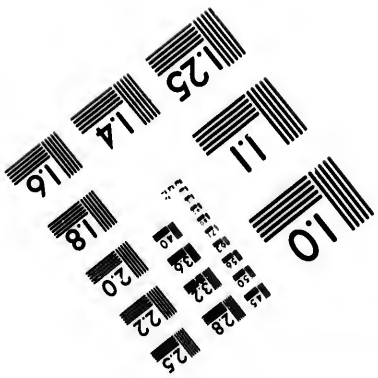

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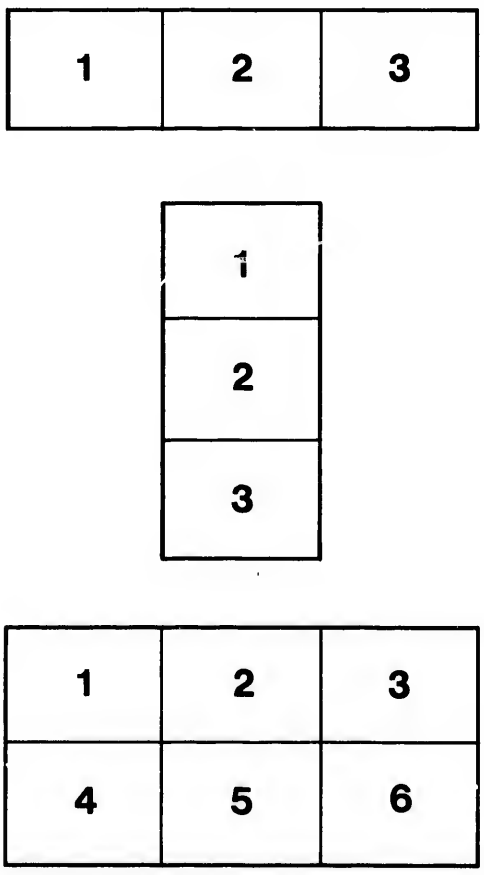

PRACTICAL INSTRUCTIONS

FOR THE DETERMINATION

BY FURNACE ASSAY

OF

GOLD AND SILVER

IN ROCKS AND ORES.

BY

E. J. CHAPMAN,

PH.D., LL.D.

Professor of Mineralogy and Geology in University College, Toronto: and Acting Professor of Mining Geology, Blowpipe Practice, and Assaying, in the Toronto School of Practical Science.

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1881.

CHAPMAN'S

ASSAY NOTES.

INSTRUCTIONS

FOR THE DETERMINATION OF GOLD AND SILVER IN ROCKS AND ORES.

BY THE SAME AUTHOR.

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The following notes contain definite instructions for the examination or assay by furnace operations of all rock-matters and other naturally occurring mineral bodies in which gold or silver is known or suspected to be present. Although given expressly in condensed form, the notes embody various tables and many explanatory directions not given, to the writer's knowledge, in other publications; whilst, at the same time, they are kept free from sundry complicated details belonging solely to the assay of bullion and other artificial alloys. As they are intended for actual use in the laboratory, it has not been thought necessary to add figures of the balances, furnaces, muffles, scorifiers, tongs, cupel-moulds, and other pieces of apparatus employed in these operations. The student will necessarily see these things for himself; and he will find their proper form and manipulation fully described in the text.

School of Practical Science, Toronto,
May 30th, 1881.

CONTENTS.

CHAPTER I.

INTRODUCTORY OBSERVATIONS.

CHAPTER II.

SAMPLING AND PREPARATION OF ASSAY-MATTERS.

CHAPTER III.

THE SCORIFICATION PROCESS.

CHAPTER IV.

THE CRUCIBLE PROCESS.

CHAPTER V.

CUPELLATION.

CHAPTER VI.

ESTIMATION OF GOLD AND SILVER IN CUPEL BUTTONS.

CHAPTER VII.

ASSAY TABLES.

FURNACE ASSAYS

OF

GOLD AND SILVER ORES.

CHAPTER I.

Introductory Observations.

[*Nature of Assays. Enumeration of Processes. Gold and Silver Ores.*]

§ 1. *Assays in general. Rock and Mineral Assays for Gold and Silver* :—Assaying is strictly a branch of quantitative chemical analysis, differing from the latter, properly so-called, by determining only those components of a substance which impart to it a commercial value. In some cases, the assay is performed by furnace operations; in others, by solution and precipitation, &c., as in ordinary chemical analysis; and in others, again, by volumetric methods. The present Notes refer solely to furnace assays of mineral matters containing, or suspected to contain, gold or silver.* These assays, as regards their *modus operandi*, are of two general kinds: *Scorification*

* Mining Assays of this description are quite distinct, it must be understood, from Mint or jeweller's assays, in which bullion and metallic alloys containing gold and silver alone come under examination. The assays to which the present publication refers are not gold and silver assays, properly so-called, but assays of rocks, vinestones, and mineral ores generally, for the detection and determination of any gold or silver that may be present in these matters.

Assays, applied to comparatively rich ores; and *Crucible Assays*, adopted essentially in the examination of poor ores. In both, however, the preparatory stages (including the sampling and preparation of the assay-matter) are alike; and both terminate in the common process of *Cupellation*. In the scorification assay a small portion, only, of the prepared ore is directly operated on; whilst in the crucible assay a much larger portion of the ore can be taken. In all cases, at least a couple of assays should be made simultaneously.* The complete assay comprises the following processes:

(1) Sampling and Preparation of the Assay-matter (Chapter II.)

(2) { Scorification (Chapter III.), or Crucible Treatment (Chapter IV.)

(3) Cupellation (Chapter V.)

(4) Treatment of the Cupel-Button (Chapter VI.)

* Objections are often ignorantly urged against laboratory assays, as being made on portions of matter too small to give trustworthy results regarding the quality of the vein or other deposit to which the assays refer; but if the samples be properly taken (as directed in Chapter II), assays of even a few grammes of matter (forming in most cases, it must be remembered, portions of samples weighing several pounds) may be thoroughly relied on as regards the general character of the deposit—*i. e.*, its comparative richness or poverty—although this may of course vary at different depths. Very frequently an assay of this kind serves to reveal the absolute barrenness of deposits from which large returns are falsely stated to have been obtained; and much subsequent loss and disappointment is thus prevented. In preliminary mining operations, again, small assays are especially useful; and when the ore is not of an exceedingly variable character their results are quite as trustworthy as those derived from the treatment of large quantities of the deposit. This will apply especially to argentiferous lead-ores and auriferous pyrites. Assays, also, are indispensable in checking the results of furnace operations on the large scale; and in the purchase and sale of ores generally.

§ 2. *Gold Ores*:—The metal gold occurs in Nature chiefly under the following conditions: (1) as "free gold" (*i. e.*, in a simple, uncombined state, although commonly alloyed with a little silver) disseminated either in small nuggets or in more or less imperceptible particles through quartz or other solid rock-matter; (2) in the same condition in river-sands and gravels or other alluvial matters forming the "*placer diggings*," &c., of Western explorers, the "*Seifenlager*" of German miners; (3) in probably the metallic state (or as an arsenide or sulphide?) in some examples of mispickel, iron pyrites, copper pyrites, blende and other similar ores, in which it is present in very minute quantity as an "accidental component;" and (4), in some rare tellurides. Assay-samples consist very generally of pieces of quartzose gangue or veinstone, carrying interspersed particles of pyrites, mispickel, zinc-blende, galena, &c., without any visible show of gold.

§ 3. *Silver Ores*:—These are somewhat numerous. The principal comprise: (1) Native silver (usually in small grains or scales, or in leafy or filiform examples) in quartzose or calcareous gangues; (2) Silver Glance (Ag^2S with 87 per cent silver, usually in black, malleable, leafy masses or small crystals); (3) Red silver ores (the dark-red consisting of S 17.7, Sb 22.5, Ag 59.8, the light-red of S 19.4, As 15.2, Ag 65.4: both distinguished by their red streak and blowpipe reactions); (4) Horn-silver ore (chloride of silver with 75.3 per cent metal); and (5) argentiferous varieties of Galena, Grey Copper Ore, and

other sulphides. Assay.samples consist very commonly of galena (more or less pure), or of associated native silver, silver.glance, argentiferous galena, blende, pyrites, &c., in a mixed gangue of quartz and calcite, or calcite and heavy spar. As regards galena, the structure of the mineral is no criterion of the silver-percentage. In some mines the fine-grained varieties are the more highly argentiferous; but, in others, the coarsely-crystalline examples yield the largest amount of silver. Where the amount reaches one per cent., it invariably arises from intermixed native silver or silver-glance. Rich examples of galena shew very frequently in places a peculiar purple tarnish.

CHAPTER II.

Sampling and Preparation of Assay-Matters.

[*Selection of Samples. Preparation of Ore for Assay: Crushing, Sieving, &c.; Apparatus required.*]

§ 1. *Assay Samples:*—It happens very commonly that the assayer has no hand in the selection of the samples sent to him for assay, in which case the sender is, of course, alone responsible for the accuracy of the assay results as applied to the deposit generally from which the samples were obtained. But in some cases, the assayer, especially if competent to report on mineral locations, selects the samples personally, or is able to give directions for their proper selection. Unless the sample be fairly chosen, the assay-results as a rule are not only of

little value, but are often absolutely mischievous in conveying to the public an erroneous impression of the ore. Mineral properties have frequently been grossly exaggerated in value by the assay of unfairly selected samples; whilst in other instances they have been unduly depreciated from the same cause. In the selection of a sample, a number of small pieces should be taken (by the pick or hammer, or by properly placed shots) from various parts of the deposit, and from each of these, smaller fragments may be broken for final reduction to powder in the mortar. A sample consisting of a single piece of ore, however large, is practically of little value. As to the amount to be taken, much must of course be left to the judgment of the collector; but ten or twelve pounds, in quarter or half pound pieces, taken from as many different spots as possible, will in general be sufficient. In deposits of comparatively uniform character less will be required than in other cases. Two or three small pieces of galena, for instance, will be sufficient to show if the lead carry a workable amount of silver; but in sinking on a galena vein, the ore should be tested at different levels, and also at different spots during the extension of the drifts.

§ 2. *Preparation of the Assay-matter; Implements required*:—Before commencing operations in this and other cases, the student should arrange on the work-bench or table, before him, the various implements (apart, of course, from balances, furnaces, anvil, and

other larger pieces of apparatus) required in the operation immediately under hand; and, after use, each piece should be carefully wiped with a soft cloth, and returned to its proper place in the laboratory. In the preparation of the assay-matter, the following articles are required: a strong hammer, with head of about 1 lb. weight; an iron pestle and mortar; a painter's brush; a sieve with metal bottom; two large sheets of paper; a large porcelain capsule or other vessel to hold the powdered ore. These are described below.

The portion of the sample finally selected for assay must be reduced to a fine powder in an iron mortar furnished with heavy pestle; and care must be taken that the whole of this portion of the sample be so treated. If the harder parts, after a few poundings, be discarded, the assay is certain to be either richer or poorer than the proper average. Assistants frequently shirk the labour of pounding, after a certain bulk of pounded matter is obtained. But the easily pounded portions in the case of soft ores in quartz or other hard gangue will necessarily give too rich an average, whilst in other cases, as when the gangue consists of calcite or of more or less decomposed rock-matter, the assay will be too poor to fairly represent the sample. The *whole of the selected portion of the sample* must be reduced to powder in small pieces at a time.

The disintegration of very hard samples may be facilitated by first "astonishing" the larger pieces of rock. This is effected by heating them strongly in a

furnace (or in an ordinary stove or fire, or in a crucible over a large Bunsen burner), and then dropping them into water. After this, *when thoroughly dried*, they are easily broken up.

In pounding, the pestle is brought down by repeated blows upon the ore, grinding being avoided in order to avoid abrasion of the mortar. A convenient size of mortar is one of about eight inches diameter at the top, and eight or ten inches in depth; but it is desirable to have in the laboratory an additional mortar of somewhat larger size, and another of smaller dimensions. Between the separate poundings the ore is emptied out of the mortar on to a sheet of paper, and from this into a a sieve over another sheet of paper. The sieve may have a diameter of about six inches, and depth of four inches, with wooden rim and bottom of fine iron or brass trellis with about thirty meshes to the square inch. It is convenient to keep at hand a cylindrical brush (such as painters use) with which to sweep out the finer portion of the powdered ore, as this often adheres to the sides of the mortar. After passing through the sieve, the powder should be poured into a large porcelain capsule or other suitable receptacle, and a label should be attached at once by a drop of mucilage to the side of the vessel. A precaution of this kind is especially necessary where several assays are being made at one time. It is also advisable to preserve the powdered sample, thus labelled, for a time, in case a repetition of the assay should be necessary.

CHAPTER III.

The Scorification Process.

[*Object of the Process. Apparatus and Reagents. Details of Process. Tables of Assay-Mixtures.*]

§ 1. *Object and general explanation of the Process:*—The process of scorification has for its direct object the combination of all the gold and silver, present in the assay-matter, with pure lead (from which these metals are afterwards separated by cupellation, Chapter V.); whilst iron, arsenic, sulphur, intermixed rock-matter, &c., become "scorified" or pass into slag. The process is performed in small, thick, saucer-shaped vessels of fireclay, known as "scorifiers." These are strongly heated in a muffle or thin fireclay oven according to the method described in detail under § 5, below, and the "workable lead" is then poured into an iron or copper mould, previously warmed to prevent the lead from spitting or throwing off small portions of its mass. The lead button thus obtained, is squared up and cleaned from adherent slag on the anvil, after which it is cupelled as described in Chapter V.

§ 2. *Apparatus:*—The following pieces of apparatus are needed in preparing the assay-mixture and carrying out the process of scorification. (1) An ordinary chemical-balance, with gramme weights ranging from fifty

grammes downwards.† (2) A porcelain capsule or crucible to hold the weighed matter, and a tare for these vessels. The tare may consist of a thin brass box with screwed-on top, holding a few particles of test-lead or some fine shot. (3) A small copper-scoop, or two, for taking up the powdered ore, test-lead, &c., and a small spatula for mixing the assay-matters. (4) A fixed or portable muffle-furnace. For ordinary work, a small portable furnace is amply sufficient. The square fire-clay furnaces, bound with sheet-iron bands, made at the Battersea Works (London, England,) are especially serviceable. The cracks, which commonly occur in them when they are first lighted, are of no consequence. A convenient size is that denoted by the letter C, stamped on the moveable doors and other pieces of the furnace. This size takes a muffle 8 inches long by $4\frac{3}{4}$ inches broad. The small "Luhme furnaces," made of sheet-iron thickly lined with fire-clay, are also very useful; but the fire-clay lining will require to be patched or replaced pretty constantly if the furnace be in active use. Repairs are effected by kneading some good fire-clay with a very small quantity of water into a stiff paste, and pressing this strongly into the damaged places by a trowel, the surface being properly smoothed over afterwards. The patchings must be left to dry thoroughly

† A very expensive balance is not required in these preliminary assaying processes, but for weighing the cupellation buttons and the fine gold separated from the latter, a delicate assay-balance is, of course, essential (see Chapter VI.). For crucible operations it is convenient to have certain special weights, as described under the crucible process in Chapter IV.

for several days before the furnace is again used.* The fuel for these furnaces consists of charcoal chopped by a small light hatchet into blocks of about two inches square. Large pieces do not fall properly, and dust destroys the draught. (5) A fire-clay muffle of suitable dimensions to fit the furnace. Formerly, these muffles had always slits or air-passages at the sides and back, but they are now generally made without ·em, and are consequently stronger. Under both forms, however, they are easily broken. Duplicates should, therefore, be kept at hand.† It is advisabe to spread a little boneash thinly over their floor to prevent the scorifiers from adhering in case some of the contents of the latter should be allowed by mismanagement to overflow, or should escape through the cracking of a faulty scorifier. (6) An assortment of fire-clay scorifiers. Those turned out by the Battersea Works are of excellent quality. The

* For private laboratories, or occasional use, the small gas-furnaces made by Fletcher and others, if properly handled, are also very serviceable.

† Fire-clay muffles, in remote places, cannot always be procured. The writer has thus been compelled at times to use cast iron muffles. In these, a slit at the back can of course be made in the casting; but if holes at the side be required, they must be bored subsequently. Muffles of this kind answer well enough in small furnaces; and, as a rule, they will serve for thirty or forty assays before becoming too much corroded for additional use. In wind-furnaces, however, or where the draught is very strong, they are rapidly melted and destroyed. Wrought iron muffles, made in two pieces, answer better. In these, the floor has a slight flange turned up on each side, and the dome consists of a separate piece, bent into the form of an arch and kept by the flanges in its place. The back is entirely open, but it can of course be closed or partially closed by a piece of fire-brick roughly cut into shape. A large crucible, laid on its side, may sometimes serve as a makeshift muffle where nothing else is at hand. In the small gas-furnaces, plumbago muffles are commonly used.

more generally useful sizes measure two inches, and an inch-and-three-quarters, respectively, across the top. (7) A pair of tongs, about two feet in length, with spring blades flattened and somewhat enlarged at the ends, for feeding the furnace with charcoal. These are commonly known as furnace-tongs or charcoal-tongs. (8) A pair of scorifier-tongs for placing the scorifiers in the muffle, and for removing them from the latter, after scorification, and pouring their contents into the assay-mould described below. These tongs have flat spring blades of about three feet in length. One is the under and the other the upper blade. The under blade is fashioned into a kind of fork or horseshoe-shaped expansion to fit the bottom of the scorifier. The upper blade is of one width throughout. When in use it goes across the flat top of the scorifier, and as the base of the latter rests within the horseshoe of the lower blade, the scorifier is held very securely, both when placed in the muffle and when removed and reversed over the assay-mould. (9) A thick slab of iron or copper, with two (or several) saucer-shaped depressions on one of its flat surfaces, and a metal handle (with wooden "grasp") strongly screwed into one end. This is the assay-mould for the reception of the "workable lead" from the scorifier. The bottom of the saucer-shaped receptacles must be rubbed lightly with chalk or red ochre to prevent the lead from adhering, and the entire mould must be warmed strongly immediately before use. This is best effected by placing it for a few minutes on the ledge before the muffle door, or on

the outside of the furnace at the top. (10) An anvil or block of hard steel from four to six inches square and of about the same thickness, with a tongue or projection several inches in length at the under side for insertion into a block of hard wood. The latter may consist of part of a tree-trunk eight or nine inches or a foot in diameter and about 2½ or 3 feet high. It should be perfectly level at the base so as to stand firmly and securely. A good hammer, with head of about 1 lb. or 1¼ lb. in weight, should accompany the anvil, and be hung up (between a couple of nails supporting the head) on the adjacent wall. One end of the hammer-head must be chisel-shaped, the other end flat and square.

§ 3. *Reagents :*—The reagents required in the scorification-process comprise merely granulated lead (commonly known as "test-lead"), dried borax, and (for occasional use) a little charcoal powder. Every sample of lead purchased for the laboratory should be tested by cupellation before actual use, to ensure the absence of silver. See Chapter V.

§ 4. *Weighing and Dressing of Samples for Scorification :*—The sieved and thoroughly mixed powder (§ 2, Chapter II.) is poured from its receptacle on to a sheet of paper and is made up into a conical heap. This is divided roughly by a spatula into quarters, and from each quarter small portions are taken and placed in the weighing capsule until 5 grammes are thus weighed out for assay. This amount is placed in a scorifier, and is

dressed with its proper amount of granulated lead, varying from about 20 to over 50 grammes, according to the nature of the substance, as shewn in the Tables of Assay mixtures under § 5, below. About one-third, or rather more, of the lead, is mixed in the scorifier with the powdered ore, and the rest is spread over the surface of the mixture,—a small amount of dried borax (usually about a gramme: see the Tables) being finally strewed over the whole. The lead, to save time where many assays are being made, may be measured instead of being weighed, as a gramme more or less is of no moment; and the amount of borax, after a little practice, may be estimated with sufficient accuracy by the eye. A lead-measure may be made by marking off with a file on a stout test-tube the spaces occupied respectively by weighed quantities of lead, as 20, 30, 40, 60, 80 and 100 grammes.

A couple of scorifiers should in all cases be dressed in this manner for each assay; and it is often advisable to operate on an additional quantity of the ore, if the muffle will accommodate a larger number of scorifiers. Ordinary muffles will take four or even six. As a rule, however, if the assay-matter be reduced in the mortar to a very fine powder, and the particles of this be intimately mixed, a couple of scorifications will give the same average as half-a-dozen. The weight of ore taken for assay is, of course, more or less arbitrary; but the amount recommended, five grammes for each scorifier, is about the maximum that can be safely used.

§ 5. *Examples of Assay-mixtures:* — The following mixtures will meet cases of general occurrence. In intermediate cases, the operator must use his own judgment as to the proper amount of lead required. It is always better to use too much than too little. With too small a quantity, the scorification is incomplete, and the lead button does not separate properly from the slag, or is brittle from retention of sulphur, arsenic, &c. (see under the description of the process in § 6). But with too much lead, on the other hand, an inconveniently large button for cupellation is obtained, and the operation occupies more time. As a rule, assay-matters containing nickel, copper, or arsenic, require a large quantity of lead for proper scorification; whilst quartzose and other rock-matters sparingly interspersed with pyrites, or other metallic particles, require comparatively little: practice soon suggests the proper quantity. With siliceous gangues, very little borax is wanted (unless much nickel, arsenic, copper, or iron be present). A gramme is about the average quantity required. With so-called "basic" gangues, on the other hand, that is, when the veinstone or rock-matter is essentially calcareous or barytic, or when the assay-matter consists largely of brown iron ore arising from decomposed pyrites, somewhat more must be used. If too much be taken, however, it is apt to impede the scorification, by covering the metallic bath with a thick coating of fused glass, and so preventing the access of air. Where much, therefore, is needed, it is better to strew a portion, only, over the assay mixture at first, and

to add the remainder towards the close of the operation. It may be folded in a piece of screwed-up tissue-paper, and placed in this manner by the furnace-tongs on the top of the crust or slag. Occasionally, also, it is advisable to add a little powdered charcoal in the same manner.

(1) Quartzose, granitic, gneissoid, or other essentially siliceous rock-matters with sparingly interspersed metallic particles (*i. e.*, specks of pyrites, galena, &c.):

5 grms. ore.
25 to 30 grms. lead.
½ grm. borax.

(2) Quartzose or other siliceous rock-matters with thickly disseminated pyrites:

5 grms. ore.
35 to 40 grms. lead.
¾ to 1 gram. borax.

(3) Iron Pyrites in quartzose gangue:

5 grms. ore.
45 to 50 grms. lead.
¾ to 1 grm. borax.

(4) Copper Pyrites, or intermixed Copper and Iron Pyrites, in quartzose gangue:

5 grms. ore.
60 to 70 grms. lead.
1 grm. borax.

(5) Intermixed Iron Pyrites, Copper Pyrites and Blende in quartzose, or mixed quartzose and calcareous gangue:

5 grms. ore.*
60 to 70 grms. lead.
1½ to 2 grams. borax.

* Take half-weights, or divide into two portions, unless very strong heat can be got up. The greater part of the borax should be added to the crust or slag, at the end of the second stage of the process: see § 6.

(6) Arsenical Pyrites in quartzose or calcareo-quartzose gangue.

5 grms. ore.*
80 grms. of lead.
2 to 3 grms. borax.

(7) Nickel-holding, arsenical, sulphurized ores in quartzose or calcareo-quartzose gangue.

5 grms. ore.*
80 to 100 grms. lead.
5 grms. borax.

(With ores of this character it is often necessary to repeat the scorification).

(8) Arsenical or Antimonial lead or copper ores, or mixture of these, in calcareous or quartzose-calcareous gangue :

5 grms. ore.*
100 grms. lead.
3 to 3½ grms. borax.

(With these ores, also, it is often necessary to repeat the scorification; and, as a rule, the assay mixture should be scorified in two or three separate portions.)

(9) Intermixed Blende and Galena (or Blende, Galena, and Iron Pyrites) in essentially calcareous or barytic gangue.

5 grms. ore.
40 to 50 grms. lead. †
2 to 3 borax.

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* See preceding foot-note.

† If the Blende greatly preponderate, 55 or 60 grammes of lead must be taken, or in some cases as much as 80 grammes.

(10) Pyritous Galena (or mixtures of pyritous galena with Native Silver and Silver Glance.)

5 grms. ore.
35 to 40 grms. lead.
1 grm. borax.

(11) Galena (or intermixed galena, Native Silver and Silver Glance) free or practically free from pyrites and blende.

5 grms. ore.
25 to 30 grms. lead.
$\frac{3}{4}$ to 1 grm. borax.

(12) Native Gold or Native Silver in visible particles in quartz or other rock-matter.

5 grms. ore.
40 to 50 grms. lead.
1 grm. borax.

Care must be taken in cleaning up the workable lead-button on the anvil, as the presence of gold may render it more or less brittle.

(13) Native Gold or Native Silver mixed with copper-pyrites, or other copper ores, in quartzose or other gangue.

5 grms. ore.
55 to 60 grms. lead.
1 $\frac{1}{2}$ grm. borax.

If Native Copper, or much copper ore be present, 80 to 90 grammes of lead must be taken.

(14) Silver Glance or other Rich Silver minerals, or mixtures of Native Silver with argentiferous galena, &c.

5 grms. ore.
35 to 40 grms. lead.
$\frac{1}{2}$ to 1 grm. borax.

§ 6. *Details of the Scorification Process :*—This process includes three stages : the fusion stage ; the oxidation, or scorification stage, proper ; and the *coup-de-grace* or final stage.

(1) *The fusion stage :*—The scorifiers containing the dressed ore (as described under § 4) are taken separately by the forked tongs (§ 2 (7)) and are placed in the glowing muffle.* A few hot coals are heaped about the opening of this, and the door is rapidly closed. Complete fusion of the assay-mixture is thus soon effected, but the muffle door is kept closed for from 20 to 30 minutes. In ordinary cases, a period of 20 minutes is sufficient for this stage of the scorification process.

(2) *The oxidation or slagging stage :*—The muffle door is now opened, and the hot coals are removed from its mouth. A current of air is thus carried over the scorifiers, by which sulphur, arsenic, and other volatile matters, if present, become eliminated ; and intermixed rock-matters, iron, &c., pass into slag or scum. In this manner, the surface of the fused bath becomes gradually covered or partially covered with an infusible crust, when the operation is completed. The time necessary to effect this, varies from about 25 to 35 minutes. If several scorifiers be in the muffle, those nearest the opening may

* The furnace should be lighted and well filled with charcoal about half-an-hour before required for use ; and, during operations, the heat must be maintained by the addition of a few pieces of charcoal from time to time. Beginners often fail in their work (more especially, perhaps, in the cupellation process) by neglecting to keep the furnace properly fed.

have a hot coal placed against them to keep up the heat; but otherwise it is better to let the air have full access by removing all the hot coals when the door is withdrawn at the commencement of this second stage.

(3) *Final or Coup-de-grace stage* :—In this stage, the muffle-door is again closed, and a good heat is kept up for five or six minutes, the object being to cause a complete precipitation or separation of the workable lead from the infusible scum.

§ 7. *Separation and cleaning up of the workable lead* :— The scorification process finished, as described in the preceding section, and the iron or copper mould (previously heated, § 2 [9]) being placed conveniently on a flat stone or on the brick floor in front of the operator, the muffle door is opened, and each scorifier is seized carefully by the forked tongs (§ 2 [8]), and its fluid contents are poured into one of the cavities of the mould. This should be done rapidly, but without undue haste, by a continuous turn of the wrist, until the scorifier is completely reversed over the mould. It is then tapped gently against the edge of the cavity, and laid aside. The lead generally forms a flat cake or disc in the centre of the cavity, surrounded by a vitreous, brittle slag; and it is easily removed, after standing to cool for a few minutes, if the surface of the cavity has been previously rubbed with chalk or ochre as directed under § 2. The lead disc or button is then taken to the anvil and carefully freed from adhering portions of slag under the hammer.

It should not, however, be beaten out too thin, but should be worked square by gentle blows on the edges, and the corners should be slightly rounded in order to prevent abrasion of the cupel in the after process of cupellation (Chapter V.). A so-called "scratch brush," made of iron wire, is sometimes used to clean the surface of the button more thoroughly, but this is rarely necessary. The lead-button should be soft and malleable. If brittle, the scorification will not have been properly conducted or sufficiently prolonged. In this case the button must be re-scorified with the addition of 15 or 20 grammes of test-lead and a little borax.

CHAPTER IV.

The Crucible Process.

[*Object of the Process. Apparatus and Reagents. Manipulation.*]

§ 1. *Object and general explanation of the Crucible process:*—In the case of very poor ores, or in the examination of rock-samples merely suspected to contain traces of metal, the scorification process, described in the preceding Chapter, cannot conveniently be employed, as only a small amount of material is capable of being assayed at a time by that method. Where, for instance, a substance contains merely a few dwts. per ton of gold or silver, the workable lead obtained from five grammes of ore necessarily yields on the cupel a scarcely appreciable amount of metal. Several scorification lead-buttons might of course be obtained, but the large amount of lead to be

got rid of would then become exceedingly inconvenient. In cases of this kind, therefore, a crucible fusion should be resorted to, by which 25 or 50 grammes of the sieved ore—or even more, if thought desirable—may be operated upon at one time. The ore (after previous roasting, if necessary, as described in detail under § 5 of this Chapter) is fused in a fire-clay crucible with its own weight, or rather more, of litharge, and three or four times its weight of a mixture of carb. soda and borax or other suitable flux (see § 3). The litharge becomes reduced to metallic lead. This takes up all the gold and silver (together with copper, &c.) that may be in the ore, and sinks down to the bottom of the pot, where it forms a workable button. When cold, the pot is broken, and the lead button is freed from slag, and squared up on the anvil. It is then cupelled. Both the scorification and crucible processes, therefore, are merely preparatory to cupellation (see Chapter V.).

§ 2. *Apparatus Employed in the Crucible Process:*— (1) A balance with set of gramme weights (see § 2 of the preceding Chapter); also a couple of special weights: one of 29.17 grammes, and the other of 32·67 grammes.* The balance should be capable of carrying 100 grammes.

* These represent, respectively, the short or American ton and the long or British ton, the former consisting of 29166·666, and the latter of 32666·666, Troy ounces. When, therefore, an amount of ore equal to 29·17 is taken for assay, each milligramme of gold or silver obtained on the cupel corresponds to an oz. Troy per ton of 2000 lbs. (avoirdupois) of ore. And, in like manner, when 32 67 grammes of ore are taken for assay, a milligramme of the cupel-metal equals an oz. Troy per ton of 2240 lbs. (See Tables IV. and V., Chapter VII.)

If turning with a milligramme when loaded with the above weight, it will be sufficiently delicate for crucible and scorification weighings, but a proper assay-balance indicating the tenth of a milligramme is necessary for the estimation of the final cupellation-products (see Chapter VI). (2) A porcelain capsule (and tare) to hold the assay-matter whilst being weighed. (3) A copper scoop with flat floor and raised edge, provided with a short wooden handle. This serves for mixing the assay-matter with litharge and flux, and pouring it into the crucible. It may be about 8 inches long and 4 inches wide at the upper end, tapering to about $1\frac{1}{2}$ inch at the point. (4) A light steel spatula with wooden handle, for mixing the assay-matters in the copper scoop. (5) A melting or fusion furnace. The portable furnaces of sheet-iron thickly lined with fire-clay, made by Luhme, of Berlin, answer perfectly; or, in place of these, the fire-clay, iron-bound, melting furnaces of the Battersea Company may be used. As regards size, those with a diameter of 8 or 9 inches and a depth of about $9\frac{1}{2}$ inches will be found most convenient. The domed top with its short chimney is removable. When this is taken off in order to remove the pots, after the fusion of the assay, it must be placed on a brick floor or on the broad sandbath which accompanies the Luhme furnaces. The method of fixing the crucibles and igniting the furnace is described in § 6 of this Chapter. The proper fuel is good charcoal in pieces of about two inches square; but a few larger pieces to put over the pots should also be at hand. (6) A supply of fire-clay

crucibles or "pots." Those of the Battersea Works cannot be surpassed. The round pattern, 4 inches high, 3 inches diameter at top, and $1\frac{3}{4}$ inch at bottom, is most suitable. A good stock of this size should be provided, as each assay (if made, as it should be, in duplicate) entails the sacrifice of two crucibles. It is also desirable to have at hand a few pots of larger and some of smaller size, but very large and very small crucibles are comparatively useless. (7) A few crucible supports. These are small circular blocks of fire-clay, 2 inches in diameter and $1\frac{1}{2}$ inch high. In their absence, a squared piece of of fire-clay brick, or a stout scorifier (reversed), may be used. If the crucibles are placed directly on the furnace bars, they are liable to adhere to these; and the heat is greater an inch or two above the grate than immediately upon it. (8) A pair of crucible tongs, scissor-shaped, about two feet long with the ends of the blades flattened inside and bent slightly downwards. A pair of elastic tongs for feeding the furnace with charcoal (see Chapter IV., § 2 [7]) are also necessary. (9) An iron pan, such as a small cast-iron frying-pan, to hold under the hot crucible when this is removed from the furnace, as described in § 6 of this Chapter. (10) A small supply of fire-clay roasting-dishes, about three inches in diameter. These, of course, require a muffle-furnace, but they are used only in special cases (see § 5). The flat-based dishes are more conveniently handled than those of watch-glass form, but the latter are more durable. The inside is rubbed or smeared with chalk, red ochre, or plumbago,

before use. (11) A piece of stout iron wire ten or twelve inches long (slightly flattened at one extremity, and inserted at the other into a wooden handle) to serve as a stirrer during the roasting process. (12) An anvil, on a solid block of wood, and a good hammer of about a pound in weight at the head, with one end square and the other chisel-shaped. These are described in Chapter IV., § 2 (10). They are used for breaking the crucible and extracting and squaring up the workable lead-button, as as described in § 5 of the present Chapter.

§ 3. *Reagents:*—In the examination of rock-matters for gold and silver by crucible assay, but few reagents are required. These comprise, essentially: (1) Litharge; (2) Fluxing materials; (3) Covering materials.

(1) *Litharge:*—This is the red or yellow lead oxide, Pb O. During the fusion of the assay-matters it becomes reduced to metallic lead, and in this state absorbs the easily reducible metals, and more especially the unoxidizable gold and silver, if any be present in the ore. These are afterwards separated from the reduced lead by cupellation, as described in Chapter 5. Fresh supplies of litharge should always be assayed by reduction and cupellation before use, as ordinary samples frequently contain a small amount of silver. When a laboratory sample is thus found to contain silver, the same amount (say, 25 grammes or other definite quantity) must always be weighed out for assays, and the known amount of silver, of course, deducted from the assay results.

(2) *Fluxing Materials* :—These consist essentially of a mixture of carbonate of soda and calcined borax, to which may be added a small amount of sugar or other carbonaceous substance to promote the reduction of the litharge. Where the gangue or rock-matter is essentially siliceous or "acid," the flux may consist chiefly of carb. soda, but a certain amount of borax should always be added to take up iron oxides, &c., and to assist the fusion. With strongly "basic" gangues, on the other hand, as where the rock-matter consists of calc spar or heavy spar, or when ferruginous matter is present, the amount of borax may equal or exceed that of the soda. A useful flux, employed largely by the writer during the last ten or twelve years, and which has been found both in his own practice and in that of others to yield good results in all general cases, has the composition given below :

CHAPMAN'S CRUCIBLE FLUX.

3 lbs. carb. soda.
2 lbs. dried borax.
¼ lb. cream of tartar.
2 oz. white sugar.

The reagents in these proportions must be intimately intermixed. The flux is conveniently kept in a large wide-mouthed jar with covered top, and a porcelain scoop for its removal should be kept in the same receptacle. The above quantities will dress from 18 to 20 crucibles, when about 25 grammes of ore are taken for assay.

(3) *Covering Materials*:—When the crushed ore, litharge and flux are mixed together and poured into the

crucible it is advisable to place an additional spoonful of flux over the mixture, and to cover this with a layer of common salt, previously ignited to prevent decrepitation. The salt, in fusing, forms a very liquid cover, and thus prevents any reduced globules of lead from adhering to the upper part of the crucible. (See § 5.)

§ 4. *Roasting:*—It has been already stated that in the case of strongly pyritous or arsenical samples the crushed assay-matter must be roasted in order to drive off sulphur, &c., before it is subjected to fusion with litharge. The lead-button would otherwise be liable to break on the anvil, and would not cupel properly. As a rule, however, a substance that requires roasting may be assayed by scorification, a workable lead-button being then obtained by one operation, as described in Chapter III.

When it becomes necessary to resort to roasting, the operation is performed as follows: The quantity of crushed and sieved ore required for assay (see § 5) is weighed out and spread over the bottom of a roasting-dish, previously smeared with red ochre, chalk, or plumbago, to prevent adhesion. The dish is then placed in a moderately hot muffle (see Chapter III.), and its contents are carefully stirred from time to time: the dish being drawn when thoroughly ignited to the front of the muffle, or even on to the ledge before the muffle-opening, so as as to keep it exposed to a full supply of air. Fusion must be carefully prevented by constant stirring. If this be neglected, or if the dish be left too long in the hot

part of the muffle, the surface of the ore may easily cake, and in that case a complete roasting becomes impossible. The operation is thus somewhat tedious, especially in the case of arsenical compounds; but too much care cannot be bestowed upon it, as if it be incompletely performed, a brittle lead-button, which cannot be properly cupelled, is almost certain to result in the subsequent fusion with litharge. When the roasting is completed, the ore no longer scintillates when put back into the hot part of the muffle, and it ceases on continued stirring to give off fumes or perceptible odour. When cold, it presents an earthy aspect, and has usually a dull red or rusty black colour. It is then ready to be transferred to the mixing-scoop and crucible, as described in the next section.

§ 5. *Weighing and Dressing Samples for crucible assay:*— The amount of test-matter taken for crucible assay may vary from about 20 to 50 grammes, but a weight of 25 grammes is about the most convenient amount for ready handling. Table 2 in Chapter VII. shews the value in Troy ounces, &c., (per ton of 2,000 lbs. and 2,240 lbs., respectively), corresponding to each milligramme and tenth of a milligramme obtained after cupellation from a weight of this amount. But when the returns are required to correspond to only one of these tons, a weight of 29.17 grammes may be taken to represent the smaller or American ton, and a weight of 32·67 grammes to represent the British ton. Each milligramme of gold or silver, as obtained by cupellation, will then equal a Troy

ounce. See Tables 4 and 5, Chapter VII. ; also foot note on page 27, above.

The weighed ore (assuming that it does not require roasting, § 4) is transferred from the weighing capsule to the copper mixing-scoop, and a weighed or measured amount of litharge (equal to or somewhat exceeding the weight of test-matter) is placed upon or beside it, together with three or four large spoonfuls of flux (§ 3, above), and the whole is carefully mixed by a steel spatula. The amount of flux need not be weighed out, but as much should be taken as the crucible can conveniently accommodate—a clear space of half-an-inch in depth being left for the salt cover, and another space of about the same depth between this and the top of the crucible. If the crucible be filled to the top, loss will be occasioned by its contents boiling over. The best plan is to mix the ore and litharge with about three or four times their volume of flux in the scoop, to pour this into the crucible, and then to stir in more flux if there be room for it. An additional layer of flux should be added to the top of the mixture, and a layer of salt is finally spread to the depth of about half-an-inch over all—care being taken, as explained above, to leave a clear space between this and the rim of the crucible. A couple of pots should be dressed and filled in this manner and the assay thus made in duplicate, so that one result may serve as a check on the accuracy of the other.

In the examination of galena for silver by crucible assay (when the small amount of silver renders a scorifi-

cation assay unsatisfactory), the ore (25 or 50 grammes) may be mixed with about 3 times its weight of the above flux, and 5 grammes of saltpetre; and two or three large iron-nails may be placed upright in the mixture, the whole being covered with a layer of salt. All the silver will go into the lead button, but there will be a considerable loss of lead. This, however, is no disadvantage, as there is thereby less to cupel.

§ 6. *Furnace Manipulation*:—The dressed crucibles being ready for the furnace, the operator puts on his thick furnace-gloves, and seeing that the spaces between the bars of the grate are free from slag or cinder, he places a couple of crucible supports (see § 2 [7] of this Chapter) upon the grate, fills in loosely the space around these with small blocks of charcoal, and sets each crucible firmly on its support. More charcoal is then carefully added, until the pots are entirely covered. The dome of the furnace is then put on, and three or four pieces of incandescent charcoal (previously ignited in another furnace, or in an ordinary stove or fire-place) are dropped through the door at the top, and the air-way is opened below. In about ten minutes or a quarter of an hour the whole body of the charcoal will be in ignition, and flame will appear at the top of the short chimney. A fresh supply of charcoal must then be added through the door in the dome, and the supply must be kept up, by dropping in five or six additional pieces every eight or ten minutes, for about fifty minutes or an hour after the first appear-

ance of flame at the top of the furnace-chimney. The furnace is then allowed to burn down, and the dome is removed, by its side handles, and set on a large Luhme sandbath or on the brick floor. When the crucibles thus become sufficiently exposed, the operator grasps them, one at a time, on the outside, a little below the top, by a pair of large scissor-tongs (§ 2 [8]) held in his right hand, whilst he holds with his left hand a flat iron vessel of any kind (such as a cast-iron frying-pan) under the lifted crucible. The crucibles are thus safely removed to a brick placed in the laboratory sink, or are carefully set on the brick floor. In putting them down, they may be tapped gently two or three times, but must not be roughly shaken, as in the latter case some of the reduced lead might spurt up among the slag and remain there. It sometimes happens that the support adheres to the bottom of the crucible and is lifted with it; but that is of no consequence. A slight tap of the hammer when the crucible has cooled, easily separates the two. Until the crucible be set down it must be kept in the grasp of the tongs, although supported at the same time by the pan held beneath it; but the latter is only intended to guard against the risk of accident.

§ 7. *Manipulation of the Crucible Button*:—When sufficiently cool to handle, which will not be until the lapse of twenty minutes or half-an-hour, the crucible is laid on its side on the anvil, and is broken by a series of blows given with the sharp end of the hammer. If the

operation be skilfully performed, the pot separates into halves longitudinally, and the button of workable lead becomes exposed. The slag above the latter should be perfectly free from small globules of metal. The broad or square end of the hammer is then used to break up the bottom of the crucible and liberate the button. This, which should be soft and malleable, is hammered free from slag, and squared up on the anvil. It is then ready for cupellation.

CHAPTER V.

Cupellation.

[*Object and General Nature of the Process. Cupelling Apparatus and Materials. Details of Manipulation.*

§ 1. *Object and essential character of Cupellation* :— When metallic lead containing gold or silver is exposed to a high temperature in a current of air, it becomes oxidized; and if the operation be performed on a porous support which is not attacked by lead oxide, the latter is gradually absorbed, carrying with it any copper or other so-called base metal that may be present, and leaving the unoxidizable gold and silver in the form of a fused globule on the surface of the support. In assay practice, the support is made of well burnt and finely ground boneash, and is known as a cupel (see § 3). When gold and silver are present together in the cupel-globule, thus obtained, they are separated by dilute nitric acid (Chapter VI.), the silver being readily dissolved by that reagent, whilst the gold remains untouched. Cupel-

lation is thus resorted to in order to ascertain if these metals (or either of them) be present in the comparatively large button of lead obtained in the scorification or crucible process. As already explained (Chapters III. and IV.) all the gold and silver present in the amount of ore taken for assay will be contained in this button. The lead separates the precious metal from the ore, and cupellation separates it again from the lead.

§ 2. *Cupelling Apparatus and Appliances* :—The following are necessary: (1) A cupelling furnace, with a supply of fire-clay muffles. Where a furnace of this kind is not built up permanently as a fixture in the laboratory, its place is conveniently supplied by one of the square fire-clay furnaces made by the Battersea Company; or by a sheet-iron Luhme furnace, thickly lined with fire-clay. These have been already described in Chapter II., together with their accompanying muffles. (See page 15.) They should stand, if possible, on a brick or stone floor, and be raised sufficiently above the ground to enable the operator to look into the muffle without inconvenient stooping. They require no special flue; but they should be placed, nevertheless, under a large hood communicating with a chimney, or should otherwise stand in an open, well-ventilated part of the laboratory, in order that the carbonic acid and other products of combustion may by rapidly dissipated. (2) Cupel moulds and supply of bone ash. These moulds are sometimes made in two pieces, entirely of brass or gun-metal—

consisting of a thick, slightly conical ring (the mould, proper,) about an inch and-a-half or an inch and three-quarters in diameter and an inch deep; and a pestle or stamper, three or four inches long, with its broad end smoothly convex to form the concave surface of the cupel. A flat rim surrounds this convex portion of the stamper in order to prevent the latter from being driven too deeply into the mould. Another and more common form of cupel-mould is in three pieces, made of hard steel. These consist of the mould proper, a thick, slightly conical ring (about $1\frac{1}{2}$ or $1\frac{3}{4}$ inch deep, $1\frac{1}{2}$ inch inside diameter at the top, and $1\frac{1}{4}$ inch diameter at bottom); a flat disc of steel, $\frac{1}{4}$ inch thick, forming a moveable bottom to the mould; and a pestle or stamper, consisting of a steel head (with convex surface surrounded by a flat border) screwed into a solid box-wood handle, five or six inches long. The method of using these moulds, in the formation of cupels, is described in detail under § 3, below. Moulds which turn out cupels of about an inch, or three-fourths of an inch deep, and an inch and a-half, or inch and a-quarter in diameter, are the most convenient as regards size, but somewhat larger and somewhat smaller moulds should also be kept in a properly appointed laboratory. The bone-ash ought to be quite white, thoroughly well-burnt, and finely ground and sifted. Purchased bone-ash should be tested by dilute hydrochloric acid. If it effervesce strongly, it will contain carbonic acid, and in that case it should be discarded (or subjected to more prolonged ignition), as cupels made

from impure bone-ash, of this kind, are very liable to become cracked or fissured in the muffle. (3) A wooden mallet to drive the pestle or stamper into the cupel-mould. (4) A steel spatula, with wooden handle, for mixing or kneading the moistened bone-ash, filling the moulds, &c. (5) A large porcelain-mortar, or earthen-ware bowl or pan, to hold the bone-ash whilst being moistened and prepared for the moulds, as described under § 3, below. (6) Cupel tongs of light sheet-iron or steel, about three feet in length, either curved or bowed at the free end to fit the cupel, or slightly bent downward at that extremity. The beginner should practice on empty cupels with these tongs, as he is very liable to take hold of the cupel too low, so as to cause it to tilt, or otherwise to grasp it too high and with too strong a pressure, causing it to break or crumble at the edges. (7) Furnace tongs for feeding the furnace with charcoal. These, already described in Chapter III., consist of a pair of short spring-blades slightly enlarged and flattened at the free ends. (8) A wash-bottle for conveniently moistening the bone-ash in preparing it for the cupel-mould. (9) A block of solid wood (or an anvil: see Chapter III.) for the support of the cupel-mould whilst the pestle or stamper is being struck by the mallet. (10) An old silk handkerchief, or soft, clean rag, for wiping the mould and stamper during and after use.

§ 3. *Preparation of Cupels*:—The assayer should always prepare his own cupels. Those purchased, ready-

made, are frequently of doubtful quality, either from being composed of impure bone-ash, or from being mixed with mucilaginous matter in order to render them sufficiently compact to bear packing and transportation. Half-an-hour's practice will enable anyone to make them successfully. The bone-ash is placed in a large porcelain mortar or other convenient receptacle, and is well sprinkled with water from an ordinary wash-bottle, or simply by the hand. It is then thoroughly mixed and kneaded by a light steel spatula, more water being added, if necessary, to make the particles "ball" or just cohere together. Very little water, however, must be used. Beginners generally use too much, simple moistening being all that is required. The proper amount is readily learnt by practice. If the bone-ash be too wet, it sticks to the mould and stamper, and the cupels are ill-formed and otherwise unsatisfactory. "Failures" of this kind should be returned to the porcelain mortar, and ground up with some additional bone-ash. If, on the other hand, the bone-ash be too dry, the cupels crumble or fall to pieces when handled. Some assayers damp their bone-ash, roll it up into balls, and leave these for ten or twelve hours, to dry partially, before being made into cupels.

The properly moistened bone-ash is taken up by the spatula and inserted into the cupel-mould until the latter is entirely filled. The mould is then placed upon the laboratory anvil or other solid support, and the stamper is set upon it, in as vertical a position as possible, and struck smartly three or four times by the mallet. The

latter is then laid aside and the stamper is removed carefully in imparting to it a circular motion by a slight turn of the wrist. This effected, a little bone-ash is dusted over the work-table, and the mould is reversed upon it. The pressure of the thumbs on the moveable bottom of the mould (or where the mould is without this extra piece, on the bottom of the cupel itself) is generally sufficient to effect the removal of the cupel; but sometimes a light tap or two with the wooden handle of the spatula is necessary to loosen it. The mould is then carefully raised, and the cupel is taken up lightly, turned right-side upwards and set to dry on a porous brick. The next day it may be placed to dry more thoroughly on the top of a warm furnace, or on the platform around the short chimney with which many cupelling furnaces are furnished for that purpose, but quick drying is always to be avoided. If a cupel be made and dried hastily so as to be used on the same occasion, it is very liable to crack or fall to pieces in the muffle. A batch of cupels should therefore be made now and then, and be kept for use, after drying, in one of the laboratory drawers or cupboards reserved for that purpose. After each cupel is made, the mould and stamper must be carefully wiped, and they must always be cleaned from adhering particles of bone-ash before being put away; otherwise they will soon lose their polish and become unfit for use, as bone-ash readily attracts moisture from the atmosphere. A cupel is assumed to absorb its own weight of lead, but it is safer to allow a few grammes extra weight in favour

of the cupel. When the lead button is very large and there is danger of the cupel becoming saturated before the close of the operation, another cupel, in reversed position, may be placed under it.

§ 4. *Details of the Cupellation Process:*—The chief points to be observed in cupellation are the following. The cupels must first be placed alone in the glowing muffle to become thoroughly ignited. If a lead button be placed upon a cold or even moderately warm cupel, it will rapidly become covered with a crust of infusible oxide, and the operation will be spoilt. This, in technical language, is known as "freezing."

The cupels are placed, therefore, at first, alone in the hot muffle, and the doors are closed for eight or ten minutes. By that time the cupels will have become sufficiently ignited for the reception of the scorification or crucible buttons. These are seized singly in the assay tongs (those with downward bent points answer best), and the door being opened or removed, each button is placed quickly but carefully in its cupel, a hot coal or two is placed at the mouth of the muffle, and the door is again closed. Where several cupels are under treatment in the same muffle, they are set in two rows along (but not quite touching) the sides; and those near the front of the muffle should have a hot coal laid against their base throughout the operation. When only a couple of cupels form the muffle charge, they should be set towards the centre of the muffle or not too near the door.

After the lapse of another interval of about ten minutes, the muffle door is again opened cautiously, and if, as commonly happens, the lead buttons are then in full fusion, forming a liquid "bath," the door is left open so as to allow a current of air to flow over the cupels. The cupellation goes kindly when a thin vapour rises in delicate coils from the oxidizing metal, and a ring of yellow scales of litharge forms around the inner edge of the cupel. If dark, thick fumes are rapidly evolved, the temperature will be too high; and in that case the draft-opening below the grate must be closed, and the upper door in the dome of the furnace should be opened for a few minutes. If, on the other hand, there is no perceptible lowering of the molten bath, and scarcely any fumes can be seen to arise from it, the temperature will be too low, and there will be danger of freezing. A glowing coal must then be placed immediately against the cupel, and the air-way beneath the grate must be opened to its full extent,—care being taken at the same time to see that the coal has not burnt down too low in the furnace, so as to leave the top of the muffle exposed. It may be advisable, also, to close or partly close the muffle door for a moment; or in extreme cases, to drop from a ladle some fused lead on the cupel, and to close the muffle entirely for a brief space of time. If the furnace, however, is well supplied with fuel, and the cupels are fully ignited before the buttons are placed upon them, there will be little risk of failure—provided always that the amount of lead in the button is suffici-

ent to effect the cupellation of any copper or nickel that may be present in it. These metals require about sixteen times their weight of lead for proper cupellation. when they occur therefore in any form, either singly or together, in samples of ore, and insufficient lead has been used in the scorification (Chapter III.), the fused bath on the cupel is liable to become encrusted (especially towards the end of the operation) with a semi-crystalline coating of infusible oxides, and the process becomes suddenly arrested. It may sometimes be started again by pouring some hot lead into the cupel, or by putting a hot coal over it; but this does not always prove successful, so that, as a rule in cases of this kind, it is better to remove the cupel, break it up carefully when sufficiently cold to be handled, and re-scorify its contents with the addition of 40 or 50 grammes of granulated lead and a little borax.*

Assuming, however, the cupellation to proceed kindly, it is always advisable to let the operation go on by itself —simply adding a little fuel from time to time through the door in the furnace-dome, in order to keep the top of the muffle well covered. Gradually, the fused lead in the cupel will sink lower and lower, and finally pass out of view. As a rule, with buttons of from 15 to 20 grammes weight, the lead will be absorbed or eliminated

* The silver ore of the 3 A Mine, Lake Superior, is associated in this manner with a nickelferous mineral, by which its assay is rendered more or less difficult to beginners. The effect of the nickel must be destroyed by a somewhat prolonged scorification and the use of lead in excess. See Chapter III.

in about 40 or 45 minutes after the opening of the muffle-door.* The muffle is then again closed, and a strong fire is got up for five or six minutes to cause the absorption of the last vestiges of lead. This effected, the door is opened or removed, and the cupels are drawn carefully to the mouth of the muffle, where they are left to cool down slowly for a short time, in order to prevent the cupel-button from "spitting." If, for instance, the button or globule of gold or silver, which remains on the cupel, be comparatively large, it is very apt to crack or throw off excrescences, or, in technical language, to "spit" or "sprout," if rapidly cooled, and from this cause too low a result might be obtained. In the assay of bullion, &c., this has to be carefully guarded against; but in the assay of ordinary ores and rock-matters the globule is usually too minute to need any very rigorous precautions of this kind. Commonly, as it cools, it emits a sudden gleam or flash of light, caused, it is generally thought, by the escape of oxygen absorbed during fusion; but as this gleam or "blick" is exhibited by gold as well as by silver, it is probably due to an incipient crystallization, or to a sudden change, on cooling, in the molecular condition of the metal.

Finally, the cupels are removed from the furnace and set to cool on a flat-brick or on some large scorifiers arranged on a tray for their reception. In large assay-

* It is perhaps needless to observe that a good clock should always occupy a prominent place in the assay-laboratory, together with a black board or slate on which the operator ought to mark down the time at which the various stages of his work commence.

offices, a cast-iron tray, with sunk and numbered compartments, is commonly used for this purpose. When sufficiently cooled, the cupel-button (if there be one as the result of the assay) is easily detached by a pair of light forceps or the point of a pen-knife. If free from lead, it adheres very slightly to the surface of the cupel. When thus detached, it is examined by a pocket-lens,* and is cleaned, if necessary, by being rubbed in the palm of the left hand by the moistened tip of one of the right hand fingers. It is then ready to be weighed in the assay-balance, and to be treated in other respects, as directed in the ensuing Chapter.

In cupellation, silver always suffers a small amount of loss, arising chiefly from absorption by the cupel, and depending more especially, as regards the actual amount, on the quantity of lead employed, the temperature of the muffle, and the condition of the cupel. This has to be taken into consideration into the assay of bullion; but in ordinary mineral assays, in which the cupel-button commonly weighs a few milligrammes, and scarcely ever reaches or exceeds 100 milligrammes in weight, the loss is insufficient to render a correction necessary, and in practice it is always neglected. Cupelled silver, in fact, is never, in the strict sense of the term, *absolutely* pure, and the minute amount of impurity present in it thus

* The little globule or button is taken up and held very conveniently, during examination, by a short piece of tobacco-pipe with some soft wax at one extremity. This is also a useful support for small crystals, &c., as it can be turned round between the thumb and forefinger of the left hand, so as to bring all parts of the object under view.

partly makes up for the cupel-loss. Any legitimate assay-loss, moreover, is certain to be exceeded by the loss which accrues from the metallurgical treatment of the ore on the large scale, and the assay returns, if the cupel-loss be neglected, are thus brought into nearer agreement with the furnace yield. In special cases, nevertheless, it may happen that rich silver ores, yielding 10 to 20 per cent., or even a higher percentage, of metal, may come under assay. In cases of this kind the same class of ore (as the product of a particular mine or mining district) will probably be constantly under examination, and the assayer must draw up for himself a table of corrections based on actual experiment, *i.e.*, by comparing his assay-results with percentages obtained by careful liquid-analysis. It is not possible to give a table of this kind adapted to all cases, or indeed to the work of any individual assayer or assay-furnace.

CHAPTER VI.

Estimation of Gold and Silver in Cupel Buttons.

[*Explanatory Remarks. Apparatus. Weighing. Treatment with Dilute Nitric Acid. Quartation. Blowpipe Treatment.*]

§ 1. *Separation of Gold and Silver. Explanatory Remarks:*—The little button or globule, obtained on the cupel, may consist of gold or silver, only, or of an alloy of these metals. Silver frequently comes out alone, but cupelled gold almost invariably contains a certain amount

of silver. When the latter exceeds the gold in weight, the cupel-button will be quite white or very light in colour. Perfectly pure gold is known as 24 carat gold, the pure metal being always assumed to consist of 24 equal parts or carats. British "standard gold" is of 22 carats: that is to say, every 24 parts contain 2 parts of silver (or silver and copper combined). Twelve carat gold, therefore, consists of half gold and half silver (or silver-copper alloy). In the language of bullion assayers, a gold which differs from "standard gold" is so many carats better or worse. Thus 23 carat gold is one carat better; and jewellers' gold (the best, of 18 carats) is four carats worse.

The cupel button on removal from the cupel is weighed in a delicate assay balance. It is then placed in a small capsule or other convenient vessel (see § 2 below), and is treated with nitric acid. By this treatment, in general cases, the silver becomes rapidly dissolved, whilst the gold separates in the form of a dark brown or black powder. The latter is then carefully washed by decantation, dried, and weighed. Its weight deducted from that of the cupel-button, gives, of course, the weight of the silver If, however, the silver be not somewhat largely in excess, the cupel-button becomes simply blackened in the acid, and very little silver is disolved. In this case, the weighed button must be fused with a piece of pure silver of about twice its weight, together with some pure lead, and the resulting button subjected to cupellation. All the silver becomes then readily taken

out by nitric acid. The lead-fusion and subsequent cupellation, when the globule (as so commonly happens) is of minute size, are most rapidly performed by the blowpipe, as described under § 4 below. But where the gold is suspected to be of high standard, a small piece of pure silver, of known weight, may be added to the original lead-button during cupellation, and its weight afterwards deducted from that of the entire amount of silver, as determined by the loss in nitric acid, or by precipitation from the acid as silver chloride. Finally, the corresponding values per ton of the gold and silver, thus found, are calculated by reference to the Tables given in Chapter VII.

§ 2. *Apparatus* :—(1) Assay Balance.—The principal piece of apparatus required by the assayer at this stage of his work, is a delicate assay-balance. This need not carry a greater weight than 2 or 3 grammes (if used solely for weighing cupel-buttons and the separated gold), but it must indicate readily the tenth of a milligramme. The 10-gramme balances of Oertling, of London, or Becker, of New York (costing about £19 or $95), are sensible to $\frac{1}{100}$ of a milligramme when fully charged, and are especially to be recommended; but very serviceable balances may be obtained at about half this cost. The assay-balance should, of course, be provided with a glass house, and its beam should be divided for the determination of the lesser weights by means of a 1-milligramme rider, made of finely-drawn aluminium wire. It should

also be provided with a pair of light weighing-capsules of thin, gold-plated brass or of horn, a pair of ivory-tipped forceps, and a small camel's-hair brush. The weights should range from a gramme, or two grammes, downwards, and should be accompanied by two or three 10-milligramme and 1-milligramme riders: all, of course, accurately tested. (2) Two or three small capsules of porcelain (an inch and a half to two inches in diameter), with attached handle. These little capsules are especially useful as receptacles for the cupel-buttons when removed from the cupels and taken to the balance-room; and they serve also for the treatment of the button with nitric acid. They can be covered with a watch-glass during the solution of the silver, in order to prevent the escape of nitrous fumes; and they are held very conveniently over the flame of a small Bunsen-burner or spirit-lamp by their short handle. In regular assay offices were bullion is treated, conical beaker glasses, known as "parting flasks," are the orthodox vessels for this operation. The beakers narrow upwards and have a thick glass band a little below their neck, by which they are taken up by a pair of suitable tongs, when heated. In operating on very small buttons, however, the porcelain capsule is a far more convenient vessel, especially as regards the drying and removal of the washed gold. (3) A larger capsule or two, to hold the liquid poured from the smaller capsule during the washing of the separated gold by decantation. (4) A common spirit-lamp or small Bunsen-burner. (5) A blowpipe

and set of blowpipe cupel-moulds, or, in place of the latter, a cylinder of baked clay or pumice (about three inches long and three-fourths of an inch in diameter), with a slight concavity at each extremity for the reception of the boneash forming the cupel.

§ 3. *Reagents :*—The following, at this final stage of the work, are all that are necessary : – (1) Nitric acid, perfectly free from hydrochloric acid. It should be diluted slightly and tested with nitrate of silver before being used. A cloudiness or milky precipitate will be produced if hydrochloric acid be present. (2) A solution of common salt (NaCl), or some hydrochloric acid, for re-obtaining the silver of the cupel-button (if this be desired) from the nitric acid solution. (3) A piece or two of pure lead foil, and some finely-sifted boneash, for re-cupelling the gold buttons when necessary. (4) A small strip of pure silver. This may be obtained from an ordinary silver-coin by separating the copper alloy either by fusion with lead and subsequent cupellation, or by solution of the coin in nitric acid and precipitation of the silver as chloride, the latter, after thorough washing, being fused with some reducing flux in a porcelain crucible, and the reduced silver hammered out for use.

§ 4. *Detailed Treatment of the Cupel-button :*—The small button or globule, after being carefully separated from the cupel, and cleaned from adhering matter, as explained at the close of Chapter V., is taken in a little porcelain capsule to the balance room, and its weight is

very accurately ascertained. The button is then replaced in the capsule, and some pure nitric acid diluted with about an equal bulk of water is dropped upon it, and the capsule is held for a few minutes, or until the acid begins to boil, over the flame of a spirit-lamp or Bunsen-burner. One of three results will then ensue: (*i*) the globule will dissolve wholly; or (*ii*) it will dissolve in part only, the gold separating as a dark-brown or black powder; or (*iii*) it will merely become black on the surface, and in this latter case scarcely any orange-brown fumes will be evolved from the acid, whilst copious fumes of this kind will be given off if the button be dissolved in part or wholly.

If the solution be complete, so that nothing remain undissolved, the button will have consisted entirely of silver. Its corresponding value per ton of ore may therefore at once be calculated or otherwise determined by reference to the proper table in Chapter VII.

If the button be in part dissolved, with separation of brown flakes or powder, the liquid must be carefully decanted from the capsule, and the operation repeated with undiluted acid. The dark powder is then washed two or three times with distilled water, this being decanted cautiously after each washing. As the powder is very heavy, the washing is easily effected without risk of loss. The last remains of the water are drawn off by a fold of filtering or blotting paper, or are expelled by cautiously warming the capsule. The dry powder is then shaken or swept by a camel's-hair pencil into one of

the little weighing capsules of the assay-balance, and its weight, to the tenth of a milligramme, is carefully ascertained. If desired, it can be folded afterwards in a piece of lead foil, and melted and cupelled in one operation by the blow-pipe, and the resulting gold globule can be preserved in a short piece of glass tube closed at each end by a piece of cork. Or, to show its true nature, the dark powder may be compressed strongly by a glass stirring-rod, or rubbed in an agate mortar, when it will quickly assume the yellow colour and metallic lustre of ordinary gold. The weight of the silver is, of course, obtained "by difference," *i.e.*, by deducting the weight of the gold from that of the cupel-button.

When, on the other hand, the cupel-button merely becomes darkened on the surface by treatment with nitric acid (as in all buttons which show a rich gold colour) the process known as "quartation" must be resorted to for the purpose of extracting the silver, so as to obtain the true weight of the gold in the button. It is commonly assumed that unles the silver be to the gold in the proportion of 3 to 1, the gold prevents the acid from thoroughly dissolving it. Complete solution of the silver is effected, however, when the proportions are as 2 (or even $1\frac{3}{4}$) to 1. In the case of very rich buttons, therefore, a small cutting of silver three or four times at least, the size of the button, must be placed with the latter, in contact with a piece of lead foil, on a blowpipe cupel, and the whole fused together and then subjected to cupellation on the same support. The weight of the

added silver need not, of course, be taken. The resulting silver-gold globule will be quite white, and all the silver will be readily extracted from it by treatment with nitric acid, as directed above. The weight of the gold deducted from that of the original cupel-button gives, of course, the weight of the silver present in the assay-matter.

In some exceptional cases the auriferous button left on the cupel may be too large to be subjected to blowpipe treatment. A button of this kind must be wrapped with a cutting of silver in a piece of lead foil, and cupelled in the muffle.

Finally, the amount of gold and silver in the assay-matter being thus obtained, the corresponding amount in the ton of ore must be determined—the portion of ore taken for assay being assumed to represent the ore generally. The determination is, of course, very simple. If x grammes (the assay amount) represent the ton of 2000 lbs. or 2240 lbs. of ore—how many ounces, pennyweights, and grains, Troy, will x milligrammes (the amount of gold or silver obtained in the assay) represent? The Tables given in the ensuing Chapter will show the required result, in ordinary cases, without calculation.

CHAPTER VII.

Assay Tables.

Table I., of the following series, will be found useful in calculating the ton values of returns obtained from

Scorification Assays. Tables II. to V., inclusive, apply to Crucible Assays. Table VI., which also applies to Crucible Assays, is calculated for grain weights. Table VII. shows the ton values corresponding to percentages from 1 to 0·0001. These Tables have been drawn up expressly for the present work. Table IX. does not perhaps belong properly to the series, but may be found useful in certain cases. It is the result of repeated determinations in the writer's laboratory.

The following example will serve to explain the Tables generally:—Five grammes of a given ore are scorified with the proper quantity of lead, &c. (see Chapter III.), and the lead-button, thus obtained, yields a cupel-button which weighs 8 milligrammes. This leaves 2·3 milligrammes of gold after treatment with nitric acid (see Chapter VI.). The amount of silver in the button was consequently equal to 5·7 milligrammes. Hence (see Table I.) the gold is equivalent to 13oz. 8dwts. 8gr. (= 11oz. 13dwts, 8grs. + 1oz. 15dwts. 0grs.) per ton of 2000 lbs. of ore; and to 15oz. 0dwts. 13grs. (= 13oz. 1dwt. 8grs. + 1oz. 19dwts. 5grs.) per ton of 2240 lbs. The silver, in like manner, is shown by the Table to correspond to 33oz. 4dwts. 14grs., and to 37oz. 4dwts. 20grs., per short and long ton, respectively. As already stated, gold and silver ounces are invariably understood to be Troy ounces.

TABLE I.

5 Grammes taken for Assay.

Milligrammes of Gold or Silver Obtained.	Ton of 2000 Lbs.			Ton of 2240 Lbs.		
	Oz.	Dwts.	Grs.	Oz.	Dwts.	Grs.
10	58	6	16	65	6	16
9	52	10	0	58	16	0
8	46	13	8	52	5	8
7	40	16	16	45	14	16
6	35	0	0	38	16	0
5	29	3	8	32	13	8
4	23	6	16	26	2	16
3	17	10	0	19	8	0
2	11	13	8	13	1	8
1	5	16	16	6	10	16
0.9	5	5	0	5	17	14
0.8	4	13	8	5	4	13
0.7	4	1	16	4	11	12
0.6	3	10	0	3	18	10
0.5	2	18	8	3	5	8
0.4	2	6	16	2	12	16
0.3	1	15	0	1	19	5
0.2	1	3	8	1	6	3
0.1		11	16		13	1½

TABLE II.

25 Grammes taken for Assay.

Milligrammes of Gold or Silver Obtained.	Ton of 2000 Lbs.			Ton of 2240 Lbs.		
	Oz.	Dwts.	Grs.	Oz.	Dwts.	Grs.
10	11	13	8	13	1	8
9	10	10	0	11	15	4
8	9	6	16	10	9	2
7	8	3	8	9	2	22
6	7	0	0	7	16	19
5	5	16	16	6	10	16
4	4	13	8	5	4	12
3	3	10	0	3	18	10
2	2	6	16	2	12	6
1	1	3	8	1	6	3
0.9	1	1	0	1	3	12
0.8		18	16	1	0	21
0.7		16	8		18	17
0.6		14	0		15	16
0.5		11	16		13	2
0.4		9	8		10	11
0.3		7	0		7	20
0.2		4	16		5	5½
0.1		2	8		2	14¾

TABLE III.

50 Grammes taken for Assay.

Milligrammes of Gold or Silver Obtained.	Ton of 2000 Lbs.			Ton of 2240 Lbs.		
	Oz.	Dwts.	Grs.	Oz.	Dwts.	Grs.
10	5	16	16	6	10	16
9	5	5	0	5	17	14
8	4	13	8	5	4	13
7	4	1	16	4	11	12
6	3	10	0	3	18	10
5	2	18	8	3	5	8
4	2	6	16	2	12	16
3	1	15	0	1	19	5
2	1	3	8	1	6	3
1		11	16		13	2
0.9		10	2		11	18
0.8		9	8		10	11
0.7		8	4		9	3½
0.6		7	0		7	20
0.5		5	20		6	13
0.4		4	16		5	5½
0.3		3	12		3	22
0.2		2	8		2	14¾
0.1		1	4		1	7½

TABLE IV.

29.17 Grammes taken for Assay.

Assay-Result in Gold or Silver.	Ton of 2000 Lbs.		
	Oz.	Dwts.	Grs.
1 milligramme.	1	0	0
0.9 ———	——	18	0
0.8 ———	——	16	0
0.7 ———	——	14	0
0.6 ———	——	12	0
0.5 ———	——	10	0
0.4 ———	——	8	0
0.3 ———	——	6	0
0.2 ———	——	4	0
0.1 ———	——	2	0
0.05 ———	——	1	0

TABLE V.

32.67 Grammes taken for Assay.

Assay-Result in Gold or Silver.	Ton of 2240 Lbs.		
	Oz.	Dwts.	Grs.
1 milligramme.	1	0	0
0.9 ———	——	18	0
0.8 ———	——	16	0
0.7 ———	——	14	0
0.6 ———	——	12	0
0.5 ———	——	10	0
0.4 ———	——	8	0
0.3 ———	——	6	0
0.2 ———	——	4	0
0.1 ———	——	2	0
0.05 ———	——	1	0

TABLE VI.

1 Oz. Avoirdupois taken for Assay.

Assay-Result in Gold or Silver.	Ton of 2000 Lbs.			Ton of 2240 Lbs.		
	Oz.	Dwts.	Grs.	Oz.	Dwts.	Grs.
1 grain.	66	13	8	74	13	8
0.9	60	0	0	67	4	0
0.8	53	6	16	59	14	16
0.7	46	13	8	52	5	8
0.6	40	0	0	44	16	0
0.5	33	3	16	37	6	16
0.4	26	13	8	29	17	8
0.3	20	0	0	22	8	0
0.2	13	6	16	14	18	6
0.1	6	13	8	7	9	8
0.09	6	0	0	6	14	8
0.08	5	6	16	5	19	10
0.07	4	13	8	5	4	13
0.06	4	0	0	4	9	13
0.05	3	6	16	3	14	15
0.04	2	13	8	2	19	17
0.03	2	0	0	2	4	19½
0.02	1	6	16	1	9	21
0.01		13	8		14	22½

TABLE VII.

A Table of Percentages and Corresponding Values per Ton.

Percentage.	Ton of 2000 Lbs.			Ton of 2240 Lbs.		
	Oz.	Dwts.	Grs.	Oz.	Dwts.	Grs.
1	291	13	8	326	13	8
0.9	262	10	0	294	0	0
0.8	233	6	16	261	6	16
0.7	204	3	8	228	13	8
0.6	175	0	0	196	0	0
0.5	145	16	16	163	6	16
0.4	116	13	8	130	13	8
0.3	87	10	0	98	0	0
0.2	58	6	16	65	6	16
0.1	29	3	8	32	13	8
0.09	26	5	0	29	8	0
0.08	23	6	16	36	2	16
0.07	20	8	8	22	17	8
0.06	17	10	0	19	12	0
0.05	14	11	16	16	6	16
0.04	11	13	8	13	1	8
0.03	8	15	0	9	16	0
0.02	5	16	16	6	10	16
0.01	2	18	8	3	5	8
0.009	2	12	12	2	18	19
0.008	2	6	16	2	12	6
0.007	2	0	20	2	5	17
0.006	1	15	0	1	19	5
0.005	1	9	4	1	12	15
0.004	1	3	8	1	6	3
0.003		17	12		19	4
0.002		11	16		13	2
0.001		5	20		6	13
0.0009		5	6		5	21
0.0008		4	16		5	5
0.0007		4	2		4	14
0.0006		3	12		3	22
0.0005		2	22		3	6
0.0004		2	8		2	15
0.0003		1	18		1	23
0.0002		1	4		1	7
0.0001			14			15

TABLE VIII.

Average Value of Gold per oz., Troy.

Fineness in Carats.	$	£	s.	d.
24	20.65	4	5	0
22	18.93	3	17	11
20	17.21	3	10	10
18	15.49	3	3	9
16	13.77	2	16	8
14	12.05	2	9	7
12	10.32	2	2	6

Note.—The value of silver is subject at present to constant fluctuation. The average Bar or Standard Silver will probably oscillate for some time at a few cents over $1 per oz. Troy. Standard Silver consists of 92·5 % pure silver, and 7·5 % copper alloy. Cupelled or Cake Silver will average about a twelfth more in value.

TABLE IX.

Approximate Sp. Gr. of Gold Alloys.

Fineness in Carats.	Sp. Gr.	Gold, %.	Alloy (Cu-Ag) %.
23	18·56	95·83	4·17
22	17·74	91·67	8·33
21	17·22	87·50	12·50
20	16·60	83·34	16·66
19	16·00	79·17	20·83
18	15·42	75·00	25·00
17	14·92	70·83	29·17
16	14·48	66·67	33·33
15	14·06	62·50	37·50
14	13·65	58·33	41·67
13	13·28	54·17	45·83
12	12·91	50·00	50·00

INDEX.

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