

CHAPTER 7

GOLD ASSAYING

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The purpose of this chapter is to define briefly the more common practices involved in assay department work, and to indicate where further detailed information may be obtained. It provides information concerning what has been general practice in the recent past, and incorporates some information relating to new developments.

The assayer's main task relates to determining the gold value of the ore. Facts supplied by him make it possible for the mine management to select areas in which payable quantities of ore may be mined. With regard to gold extraction his reports on the gold content of all products in the metallurgical department are of vital importance. These are based on samples taken daily in the treatment plant, and present a comprehensive picture of the efficiency of the process. In addition, the assay department is able to undertake special analytical work and also to carry out metallurgical extraction tests.

Assay Practice on the Witwatersrand edited by V. S. Dillon is a book which will frequently be referred to in this chapter. All formerly well-known procedures are given in its pages, and it can be regarded as a *vade-mecum* on gold assaying in South Africa. Although published in 1955 it incorporates all but a few of the latest methods.

The Fire Assay

The fire assay has, for many generations, been used as the method for determining the gold and silver content of ores.

As described by Agricola in Book VII of *De Re Metallica*,

"The method of assaying ore used by mining people, differs from smelting only by the small amount of material used. Inasmuch as, by smelting a small quantity, they learn whether the smelting of a large quantity will compensate them for their expenditure; hence if they are not particular to employ assays, they may, as I have already said, sometimes smelt the metal from the ore with a loss or sometimes without profits; for they can assay the ore

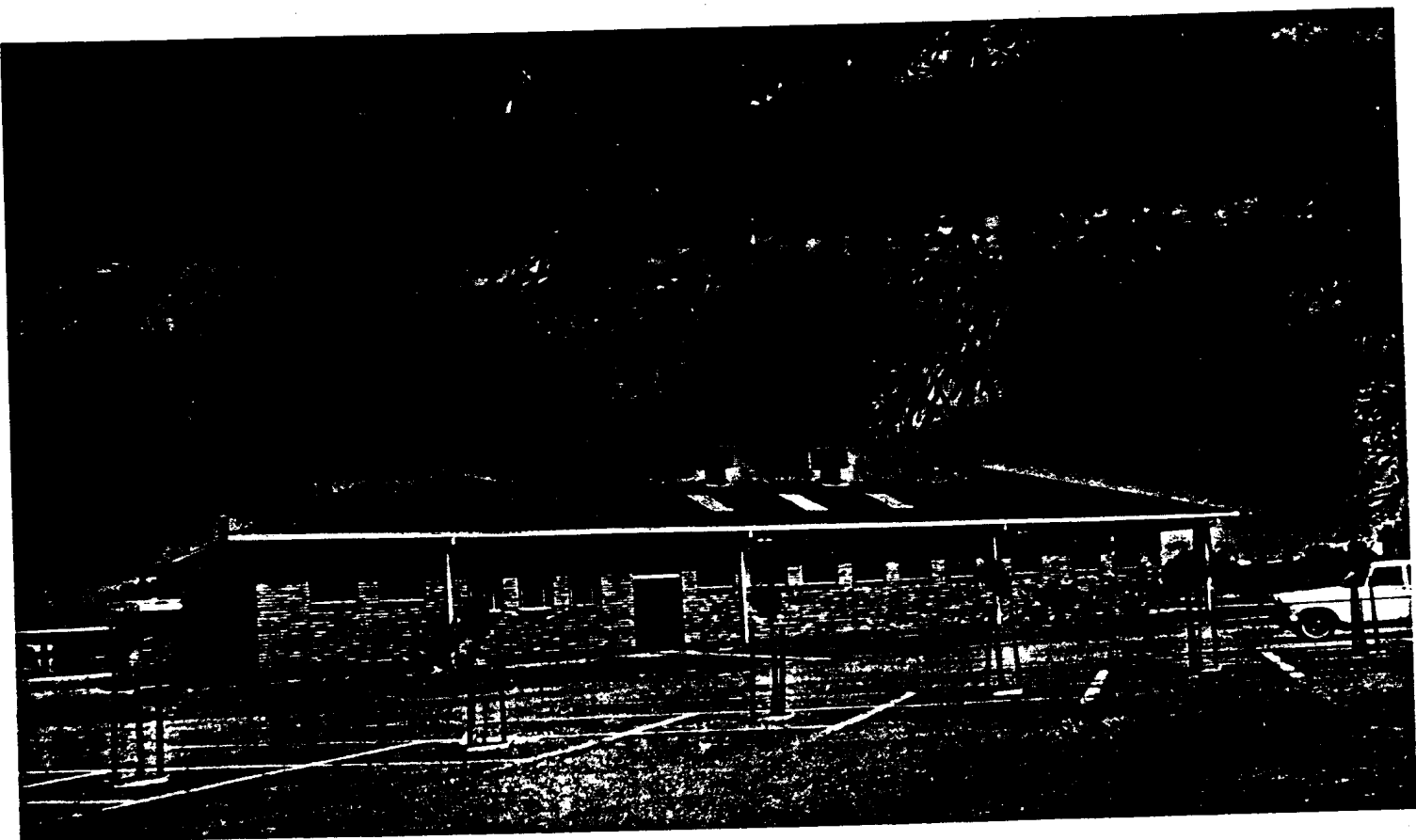


Figure 1. Assay Office exterior

at a very small expense, and smelt it only at a great expense."

Further advice given by him in 1556 included,

"It is necessary that the assayer who is testing ore or metals should be prepared and instructed in all things necessary in assaying, and that he should close the doors of the room in which the assay furnace stands, lest anyone coming at an inopportune moment might disturb his thoughts when they are intent on work. It is also necessary for him to place his balance in a case, so that when he weighs the little buttons of metal the scales may not be agitated by a draught of air, for that is a hindrance to his work."

To follow such well-established procedures with reasonable care presents no difficulties, and provides the information needed with sufficient accuracy. Furthermore, the end point of a fire assay is the physical production of the gold content of the sample, thus affording advantages that the gold pill obtained can be scrutinized, its mass measured and then retained for further reference.

The following diagram shows an elementary flow plan of the fire assay.

MINE SAMPLE ASSAY PROCEDURE

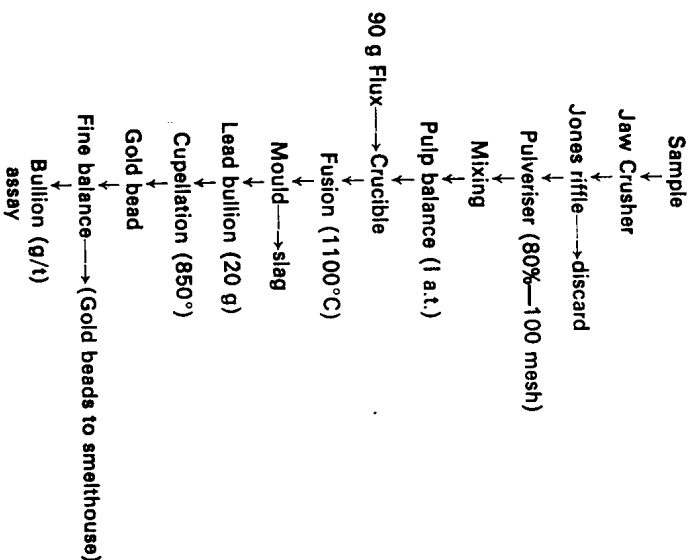


Figure 2. Fire Assay Flow Diagram

A fire assay therefore requires that a suitable amount of pulverized ore be subjected to a small-scale smelting operation to recover the gold content in a form convenient for determining its mass. Since 1869 the quantity used for this purpose was the Chandler's assay ton of 29,166 grams but in order to bring assaying procedure into line with the metrication policy of South Africa the metric assay ton of 25 grams has been adopted. Thus to conduct an assay, one metric assay ton of the pulverized ore is weighed (to an accuracy of about 0,1 g), mixed with three times its weight of flux and fused in a reducing atmosphere at a temperature of about 1 100°C.

The flux referred to is made up in approximately the following proportions:

Litharge	30%
Sodium carbonate	50%
Borax	18%
Maize meal	2%

It will be seen that this flux is strongly basic, a characteristic required for most South African gold ores.

The object of the assay fusion is to concentrate the precious metals (gold and silver) in a button of lead and to retain the gangue in the slag. The flux ingredients provide the necessary means for this to be achieved when the temperature is raised adequately. (N.B. For the theoretical basis and much detail relating to the assay fusion, reference should be made to pages 50 to 55 of *Assay Practice on the Witwatersrand*.)

Fire-clay crucibles are used to hold the mixture of the 25 grams of pulverized ore and 80 to 90 grams of the flux. On these being placed in a furnace and the temperature brought up to a white heat, the material in the crucible melts and the silica of the ore (as well as any dolomite that may be present) combines with the sodium carbonate, borax and litharge to form a molten body, while the carbon of the maize meal combines with the oxygen of the litharge. This reaction causes the fused mass to boil due to the escape of the CO₂ gas and at the same time causes the metallic lead particles (generated from the litharge) to move down through the fusion in a "raining" action to the bottom of the crucible. After about 30 minutes when the boiling action has ceased, the lead, at the high temperature of 1 100°C, will have formed an alloy with the precious metals and will have

reached the bottom of the fusion. Pouring the molten mass into a conical mould permits the recovery of the lead bullion which solidifies on cooling.

The cone-shaped lead buttons so formed are covered in slag and have to be cleaned by hammering into cubes. These are then cupelled in order to remove the lead by oxidation. This is accomplished by placing the lead cubes on magnesium oxide cupels which await them in a muffle furnace heated to 900°C. In the muffle the lead oxidizes in the stream of hot air, and most of this oxide is absorbed into the cupel although a small percentage passes off as fumes. A good draft through the muffle is fundamental to the cupellation procedure to oxidize the metallic lead to litharge. The precious metals do not oxidize and therefore remain on the surface of the cupel in the form of a small globule. After cooling this small bead of gold bullion is accurately mass measured on a balance.

Mine Samples

These consist of portions of the ore chipped from the reef underground at predetermined intervals in each working

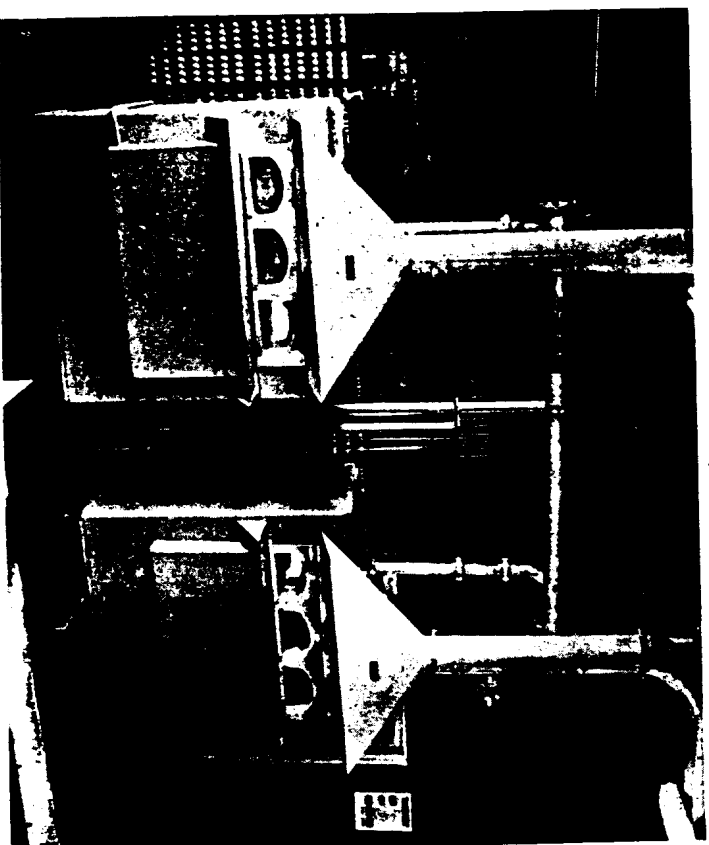


Figure 3. Electric muffle furnaces

area in such a way as to represent a correct cross-section of the gold-bearing body. In the assay department mine samples are put through a jaw crusher set to give a product of about 90% minus 10 millimetres in size. After dividing this material down (by a Jones riffle) to a mass of about 300 grams it is pulverized to about 80% minus 100 mesh (Tyler), a fineness that makes it possible to get a good distribution of the gold particles during mixing and before taking the aliquot for fluxing. Pulverizers are provided with a mechanical device which causes the plates to move apart when the blow-out valve is operated, thus ensuring an adequate removal of residual sample to avoid contamination. In order to remove the dust produced in all crushing operations, dust hoods connected to efficient suction devices are installed and a potential health hazard is eliminated.

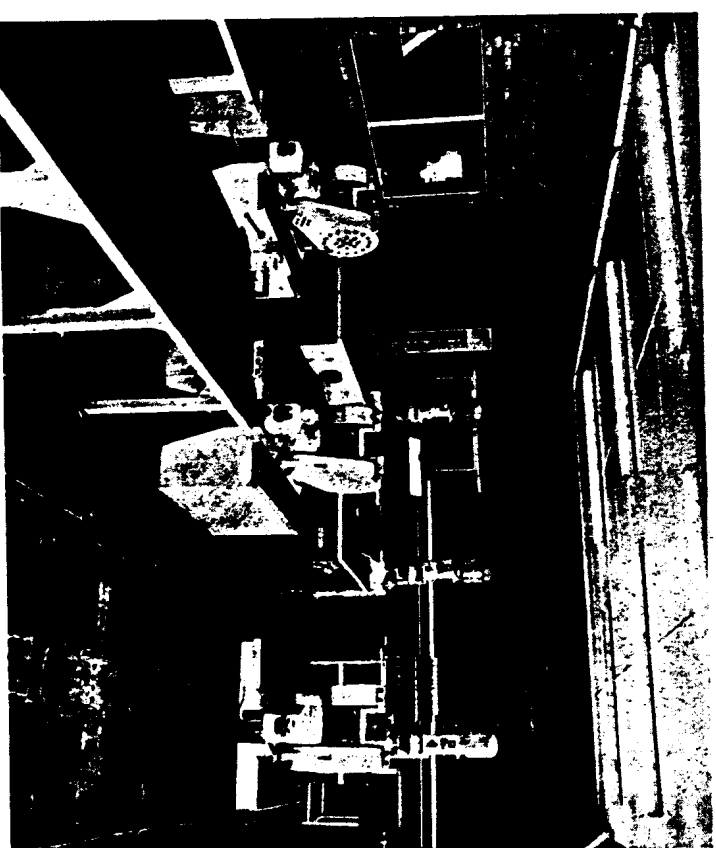


Figure 4. Crushers and pulverizers

Regular checking of mine sample assays is made in order to guard against cross-overs during furnace handling. To achieve this, two samples out of every tray of 36 or 48 samples are set aside in a separate tray to be assayed. Any evidence that these do not check with those in the routine

set would demand an immediate re-assay of the batch concerned.

NOTE. On those mines that produce uranium as well as gold, a portion of the pulverized sample is removed and tested by means of Geiger or scintillation counters to assess the quantity of U_3O_8 present. This is reported as grams of U_3O_8 per kilogram in a column adjacent to the gold figures.

The Metric Assay Ton

This assay ton—"a.t." in contradistinction to the old "A.T."—is a mass of exactly 25 grams. The assayer finds it convenient to use the following quantities when assaying gold, silver or platinum, and using the accompanying factors to convert the milligrams on the balance to grams per ton.

Quantity taken	Mass of bull	Grams per ton
For 1 a.t. of ore (25 g)	a mg	a × 40 i.e. 40a
For 2 a.t. of ore (50 g)	b mg	b × 40 i.e. 20b
		$\frac{2}{2}$
For 4 a.t. of ore (100 g)	c mg	c × 40 i.e. 10c
		$\frac{4}{4}$
For 8 a.t. of ore (200 g)	d mg	d × 40 i.e. 5d
		$\frac{8}{8}$
For 80 a.t. of solution (2 000 g).	s mg	s × 40 i.e. $\frac{s}{2}$
		$\frac{80}{80}$

This system is derived as follows:

One ton = 1 000 000 grams, therefore

$$\frac{x \text{ milligrams per balance}}{1\,000} \times \frac{1\,000\,000}{25} = x \text{ grams per ton.}$$

(See E. A. D. Rubidge, *Journal Institute of Assayers and Analysts*, October 1965.)

The Chandler's assay ton (abbreviated to "A.T.") was based on the ratio:

$$\frac{1 \text{ oz troy} \leftrightarrow 1 \text{ mg}}{1 \text{ ton} \leftrightarrow 1 \text{ A.T.}} \text{ i.e., } \frac{20 \text{ dwt}}{2\,000 \text{ lb}} \leftrightarrow \frac{1 \text{ mg}}{29,166 \text{ g}}$$

N.B. The factor to convert pennyweights per short ton to grams per metric ton is 1,714 3.

Furnaces—Fusion and Cupellation

Several types of furnaces are in use and are classified here by the method of heating.

- 1. Coal-fired by hand.
- 2. Coal-fired by mechanical stoker.
- 3. Oil-fired.
- 4. Electrically-heated, now the predominant type.

The first two types which frequently embraced both crucible hearths and muffles are illustrated in Dillon's book on pages 20 to 29. Oil firing did not prove popular, largely owing to the noise created, but also to the relatively high cost of fuel in comparison with the cheap and readily available Transvaal coal. Therefore separate electric hearths and electric muffles are the accepted installations in modern South African assay offices.

Although more expensive to install, the electrically-heated furnaces have many advantages over the former types. There is more control over temperature, the crucible and muffles are clean at all times and last longer, as does the furnace lining itself. The switch-on is automatic and no night-shift operator is required.

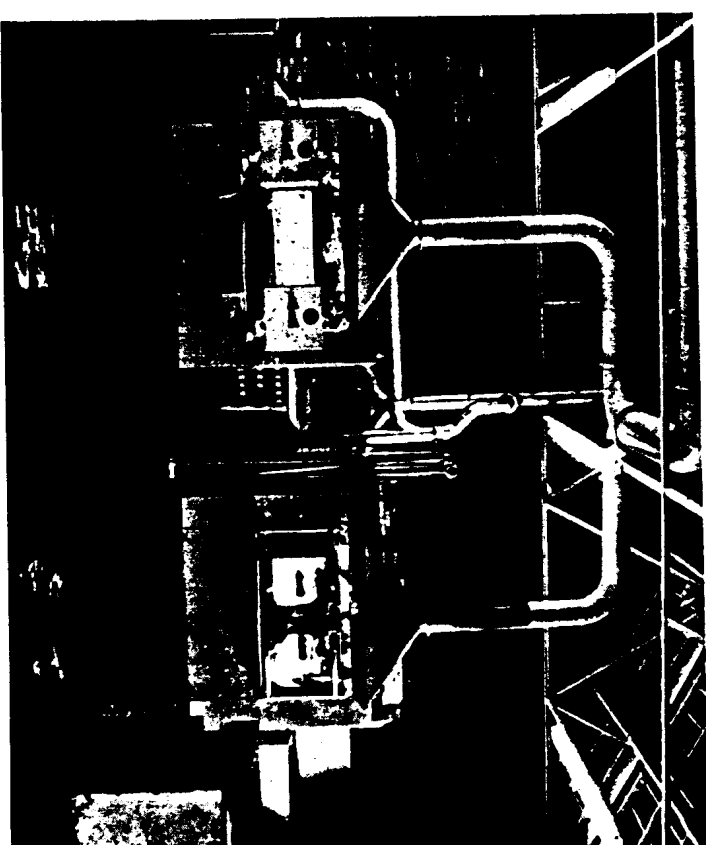


Figure 5. Electrically heated crucible furnaces

The heating elements (composed of silicon carbide) are placed above the hearth of the fusion furnace, and in the case of the muffle furnace, 40 or 50 millimetres above the tops of the muffles. The cupellation furnace usually incorporates four carborundum muffles, but a single muffle can be equipped with internal elements 120 to 140 millimetres above the base on which the cupels are placed. Both the hearth area of the crucible furnace and the cupel-holding muffle have to be provided with draw-off ducts to remove fusion and cupellation fumes, both of which contain poisonous lead oxide.

The crucible furnace, frequently referred to as a "pot furnace", should attain a temperature of about 1 100°C before it is charged with assays. Muffles operate at approximately 900°C.

Because of the large number of mine assays (up to 2 000 daily) that have to be dealt with in the larger offices, assayers often make use of multiple furnace-charging devices, handling either 36 or 48 crucibles in each batch, both for crucible and muffle loading and unloading.

Balance Room

Mass determinations of beads are performed in a balance room where there is no vibration, where the temperature is regulated and there is a minimum of dust.

Gold assaying has attained a high degree of accuracy that has been carefully assessed by means of research. Refinements in measuring instruments have brought about greater precision and dependability and have in the process speeded up this final step in the assay.

For mine sample beads the torsion balance is now greatly used and has completely replaced the old free-swinging balance. The air-damped balance (which was the first type to displace the free-swinging balance) is also extensively used and is preferred by some assayers. The accuracy of both these balances is about 0,005 mg. The torsion balance measures beads of from 0,005 mg to 5,0 mg to a precision of 0,005 mg (or 0,2 g per ton on a 1 a.t. portion). It can, however, be made to measure five to ten times more, without putting any additional strain on the mechanism, by merely cutting away from the aluminium pan a mass equivalent to the additional capacity demanded—this additional mass being replaced by the requisite number of 5 mg riders suspended from the weighing arm. Much accurate work can be done in a considerably shorter time than heretofore.

A finer, i.e., more sensitive, torsion balance is generally used for surface sample pills. It has a sensitivity of 0,001 mg, and has a dial subdivided from 0 to 1 mg with subdivisions of 0,01.

An important aspect of the torsion type of balance is its consistency, i.e., no difficulties arise as a result of temperature changes in the atmosphere—a feature not enjoyed by any other sensitive balance.

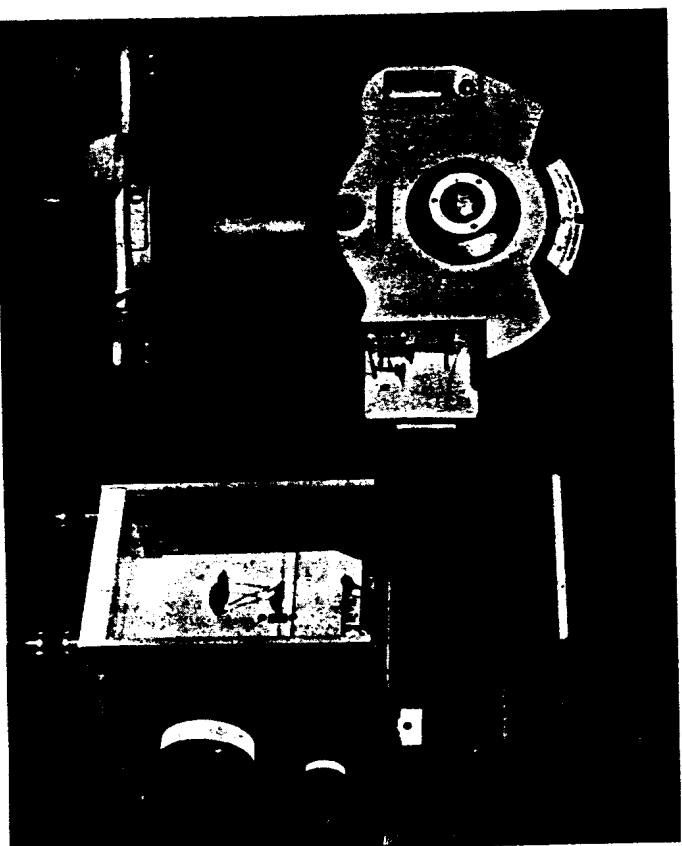


Figure 6. Two types of torsion balances

A new torsion design, working on the same principle, became available in 1970. It is in a small glass case, 21 cm high and 11 cm across, with an upward sliding glass front, having a capacity of from 10 to 20 mg, and has a sensitivity equal to that of the "residue pill" balance (i.e. 0,001 mg), and shows a reading in actual figures.

If the air-damped balance is used it is desirable for the balance room to be air-conditioned. Alternatively it would be necessary for the doors of the balance to be left open for about an hour before mass measuring starts so that the atmosphere inside the balance can adjust to that of the room. This prevents any slight movement of air that might occur due to diffusion of small air masses of different temperature and humidity.



Figure 7. Air damped balance in balance room

Silver in Mine-sample Beads

During the mass measuring operations the assayer at the balance has to make adjustments to the mass obtained before entering the figures on the report sheet. This is done to allow for the silver content of the bullion bead, since the gold in the ore is accompanied by silver to an average of about 10%. By this means the "parting," i.e. silver separation, from a very large number of beads is obviated.

REDUCTION WORK SAMPLES

In contrast to the large number of mine samples assayed each day, those received from the reduction works are few in number. They are, however, of vital importance to the treatment operations, and the accuracy required is such that from 2 a.t. to 8 a.t. aliquots form the basis of each assay, which are also made either in duplicate or triplicate. Consisting of both solids and solutions, the gold content only is required and therefore parting is essential in every case. The main samples of solids comprise slime originals, slime residues and mill feed. In the case of solutions, unprecipitated and precipitated solutions are the essential samples, but filter wash and return dam solutions are generally assayed for control purposes. On those plants where milling in cyanide solution is practised, the gold values of the mill solution and thickener overflow have to be determined.

Both solids and solution samples are normally taken over a period of eight hours and therefore three samples of each type are taken daily. In addition a composite twenty-four hour check sample is frequently taken. Special samples of waste rock, classifier products, pachuca contents, etc, also require assaying as and when investigations are being conducted, but are not part of the daily routine.

Mill Feed

These samples, having a mass of 5 kg to 10 kg, have to be crushed and quartered before 2 a.t. aliquots are assayed in triplicate with 180 grams of flux. The crushing is completed by means of a pulverizer as it is necessary to ensure that the material in the sample should be sufficiently fine to obtain an even distribution of the gold particles, and thus contribute towards agreement of the triplicate assays.

Slime Originals

This is the name generally used to describe the sample of pulp entering the slime plant before gold dissolution. It is an essential guide to mine management regarding the underground operations and thus accuracy must be ensured. The slime is sufficiently fine in particle size to dispense with pulverizing, and therefore after thorough mixing three portions of 2 a.t. each are measured out and fused with 180 grams of flux. The resultant lead button is cupelled and the gold bead parted and its mass determined.

ASSAY PROCEDURE FOR SAMPLES FROM REDUCTION WORKS

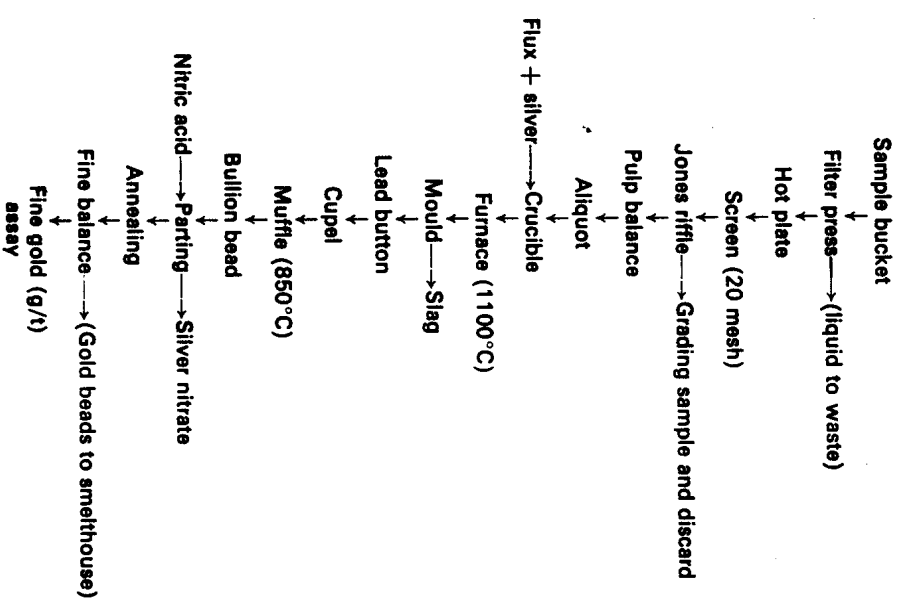


Figure 8

Unwashed and Washed Slime Residues

These samples are taken from the filter discharge and, as the names imply, one sample is a direct residue sample while the second has been subjected to water washes to eliminate the residual cyanide solution which contains a slight gold value. Thus the unwashed residue is always slightly higher in gold content than the washed residue—usually between 0.01 to 0.03 gram per ton. Alternative names for these samples are “total gold loss” and “undissolved gold loss.” Because of the small amount of gold contained in slime residues these samples are assayed in aliquots of 4, 5 or even 8 a.t., but only in duplicate, as variations in individual assays are usually slight. A large quantity of flux is used per aliquot, from 250 to 750 grams, which requires bigger crucibles. With the larger quantities involved, fusion and cupellation times are longer, and special care has to be taken in parting on account of the minute mass of gold involved.

Parting

As already stated, it is important to report the fine gold content of reduction plant samples, and therefore the associated silver in the bead obtained after cupellation has to be removed. This is achieved by dissolving the silver in nitric acid and the procedure is described as “parting”. To obtain satisfactory results in parting it is essential that the soluble metal predominates in the alloy. As the bullion in South African ores normally comprises about 90% gold and 10% silver it is necessary to add silver to the sample in sufficient amount to reverse the gold/silver ratio. This procedure is termed “inquantation” and consists of adding either metallic silver or a silver salt such as silver nitrate to the sample before fusion. The amount added is important—the critical ratio for adequate parting is 2.5 silver to 1 of gold. If however, the ratio increases much above this figure the gold bead breaks down to a fine powder, and this results in inconvenience in measuring its mass and danger of particles of gold being lost. Consequently no more silver is added than will produce a three-to-one ratio, and after parting the porous gold bead is annealed in a muffle furnace to provide a more coherent pill for mass measuring. The nitric acid solution employed is usually 30% strength, and silver dissolution is carried out at approximately 80°C in a fume cupboard.

Inter-mine Checking of Surface Sample Assays

Mining groups usually arrange for their assay offices to exchange a number of samples for checking at monthly intervals in order to have a measure of the performance of the different offices. A list of the comparative figures is then circulated to each mine and the differences studied. If these are considered excessive, corrective action is taken. An internal checking practice has been adopted in some assay offices known as "the two-stream assay". Pulp and residue samples are involved in this procedure where assayer "A" pilots one batch of single fusions right through to the final mass measuring, while assayer "B" attends to the same samples in a separate batch. The results are brought together for reporting. In order to maintain interest, the results of "A" and "B" are graphed. Any trend in difference would call for investigation.

Gold-bearing Solutions

In solution-assaying the gold and silver are separated from the liquid, either by precipitation or by deposition on lead, and six methods of assaying are available:

1. Evaporation on litharge in plastic-lined porcelain basins, followed by fluxing and fusion.
2. Precipitation by copper sulphate, then filtering, fluxing and fusion.
3. Precipitation by cuprous chloride, followed by filtration, fusion and cupellation.
4. Precipitation by a zinc-lead couple, employing lead acetate and zinc dust. After the gold is precipitated, surplus zinc is dissolved with boiling hydrochloric acid and removed by decantation. The residual lead sponge is cupelled, and the resultant gold bead parted, annealed and its mass determined.
5. Deposition on lead foil. Corrugated lead foil is introduced into the solution at boiling point, together with potassium permanganate and hydrochloric acid. The solutions, after 30 minutes boiling, are decanted and the lead foil cupelled.
6. Electrolytic deposition. This is conducted in glass jars with platinum anodes and lead foil cathodes. After the gold has been deposited on the cathodes, these are removed and cupelled, followed by parting and annealing. Unprecipitated solutions have to be very accurately

ly assayed and methods 4, 5 or 6 are to be preferred. For all other solutions, such as filter wash and return dam, method 3 is generally used. For detailed descriptions see Dillon, pages 91 to 98.

Check-gold for Solution Assay

In solutions of high gold content it is advisable to run a check with every batch, using a quantity that will compare with the expected value of the routine sample and following the same procedure as that of the sample assay. Check solution for this purpose is made up by dissolving exactly 100 mg of proof gold in aqua regia and making up to 1 000 ml with water.

To indicate differences in operation between pulp and solution assaying, the frequently-used cuprous chloride method, No. 3, is here given in some detail.

In high-value samples, a 10 a.t. aliquot is run into a 1 000 ml boiling flask, 10 ml of Cu_2Cl_2 added, and the assay well mixed. Potassium ferrocyanide is added to coagulate the white precipitate, and the contents of the flask then filtered. The filter paper with precipitate is opened and partially dried by being placed on a flat suction pad; solution flux is added and the assay put into a crucible for fusion and cupellation.

The solution flux is made up in the following proportions:

Sodium carbonate	36%
Litharge	25%
Silica	25%
Borax	13%
Maize meal	1%

For low-value samples, specially shaped plastic containers are filled at the plant. On receiving these the assayer draws off the excess volume so that 80 a.t. (2 000 ml) remains to be treated with cuprous chloride as described above, but using a larger quantity of Cu_2Cl_2 . These containers are then inverted over the filters to drain automatically, thus saving a considerable amount of manipulation.

Rapid Evaluation of Precipitated Solutions

With the development of the atomic absorption technique for rapid assessing of the gold content of low-value solutions, the "Purple of Cassius" test may fall into disuse in the extraction plants on modern mines. At present,

however, it is a regular means of assaying precipitated solution on site and can be rapidly performed by the plant operator. The adaptation of atomic absorption analysis to precipitated cyanide solutions is under consideration. Although the apparatus is not installed in assay offices, confirmation of the values determined by atomic absorption are nevertheless dependent on fire assays. The assayer is also likely to be called upon to prepare solutions containing standard quantities of gold for use in the atomic absorption apparatus. As each such standard requires to be made up of solution containing impurities similar to the plant solutions the assayer is required to blend solutions of suitable content and confirm their values by accurate solution assays.

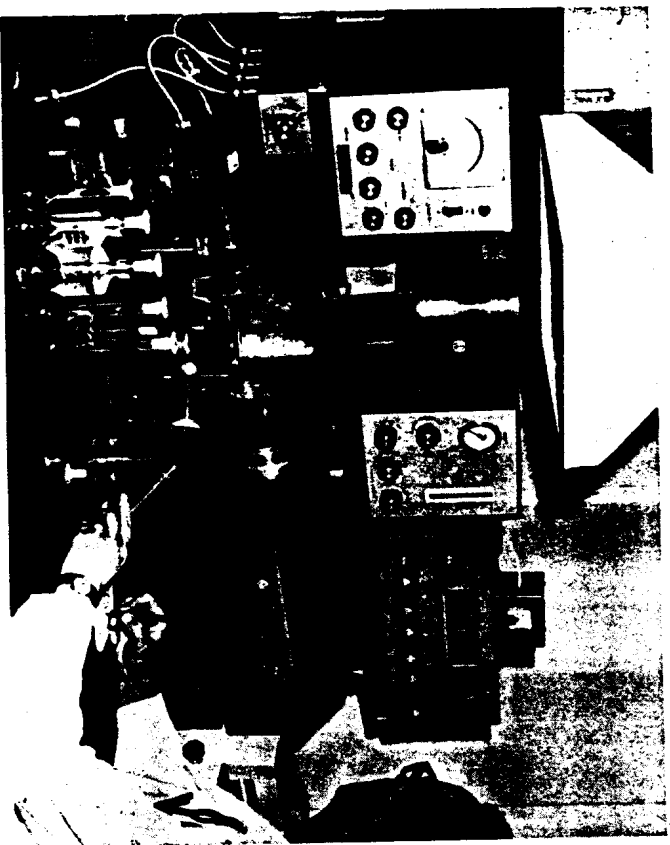


Figure 9. Atomic absorption unit

Assaying Gold Bullion

The bullion assay involves:

1. Preparing aliquots of the gold sample.
 2. Wrapping in lead foil.
 3. Cupelling.
 4. Parting.
 5. Mass-measuring.
- (Gold bar samples are usually assayed together in a batch, and the above steps in brief detail are;

1. Mass-measuring accurately three portions of bullion, each of 0.5 gram, from the respective samples.
2. Placing each portion into a cone of lead foil, to two of which, 25 mg of copper and a 1.25 g disc of pure silver have been added; and one to which no addition has been made. Wrapping each loaded cone and pressing it into button form.
3. Cupelling the batch of compressed buttons at a temperature of about 950°C.
4. Cleaning and rolling the resulting silver-gold buttons into strips, which are then annealed and wound into loose coils, referred to as "cornets". These are put into the parting apparatus which is then inserted into hot dilute nitric acid (1:2) for 10 minutes, and then transferred to hot strong nitric acid (2:1) for 30 minutes. The parted cornets are then washed and annealed.
5. Determining the mass of the cornets and then calculating the pure gold to parts per thousand and ascertaining the silver content by deducting the mass of gold from that of the bullion of the third portion mentioned in 2 above.

The purity of gold bullion is reported in "fineness" as parts per thousand, to the nearest 0.5 part. The metal mass pieces used by the assayer are therefore calibrated from 1 000 half-milligrams down to 10 half-milligrams, the rider on the balance beam being used for assessing the units and the fractions. This mass is chosen for the assay because the 500-milligram mass of bullion is a convenient quantity for handling—the halving of the denominations being merely a device to simplify the "parts per thousand" calculations. Because it is not possible to remove every trace of silver during parting, the assayer is obliged to run one or two check assays with every batch of bullion cupellations. This is done by mass-measuring a portion of proof gold (obtained from the Rand Refinery) to correspond with the estimated fineness of the sample being assayed.

An example of the calculations of one aliquot of parted gold is given below:

Mass of original aliquot	1 006.5 half-milligrams
Mass of pill after parting	920.7 "
Mass gained by check-gold portion	1.2 "

Therefore the assayed fineness is $\frac{920.7}{1 006.5} \times 1 000 = 914.7$

Correcting for the check-gold gain the actual fineness is 914.7 less 1.2 = 913.5.

NOTE: The silver reported is found by difference and could more accurately be defined as "metal (other than gold)" retained after cupellation.

For detailed procedure see pages 109 to 114 of *Assay Practice on the Witwatersrand*.

Assay of Concentrates from Arsenical Ore

The following method is used in assaying concentrate from mines in the Eastern Transvaal when these are submitted for sale to a purchasing company.

As the gold content is usually high (over 170g per ton) it is permissible to use small aliquots, these proving an advantage owing to the high percentage of arsenic present in the ore.

The assay should therefore proceed in the following manner:

Each portion of 6.25 g is treated with 10 g of red lead, 40 g of stock flux (see below), mixed with 35 g of flake caustic soda, and fused in a 3 assay ton fire-clay crucible at a temperature of about 1 100°C. After cupellation the bead is mass-measured, re-inquartered with 2.5 times its mass of silver and parted.

It is essential to assay at least five aliquots from each sample. An ordinary stock flux (with a few grams of caustic soda added) is quite suitable for the run-of-mine ore in all cases except for highly pyritic material. In no instance should metallic iron be added to any fusion of an arsenical sample.

Stock flux required is 30 g of sodium carbonate, 50 g of litharge and 20 g of borax and *no reducing agent in the concentrate assay*.

By-products and Complex Gold Ores

Because certain substances that are sometimes associated with gold cause difficulties in assaying, it is necessary in the case of unknown ores for the assayer to get guidance through qualitative analysis before selecting his fluxes. Typical examples of interfering substances can be found in:

1. *Matte*, where complex metallic sulphides occur and cause trouble in cupellation.
2. Certain metallic oxides, not completely taken up in the slag during smelting but which appear in the lead button.

On cupellation, the lead dispels a scoria on to the perimeter of the cupel in the form of a ring which often holds up some of the gold and silver.

3. Tellurium and copper, which tend to cause some loss of gold during cupellation.

In order to obviate the effects referred to, certain procedures can be adopted:

- (a) *Acid treatment* before fusion is used only when it is known that a large proportion consists of the interfering substance. It is then advisable to use a comparatively small aliquot. Sulphuric acid removes iron and copper when dilute and hot nitric acid is used to attack sulphides.
- (b) *Roasting* of a mass-measured aliquot, spread out over a layer of powdered silica, removes sulphides, producing oxides which can be fluxed. Roasting must be done at a low temperature—preferably not above 600°C so that oxidation takes place without flashing or spitting.

- (c) *Scorification* is resorted to when interfering substances such as metals, arsenic or sulphur, are removed from a lead button before cupellation.

The process is one of oxidation. The scorifier is a fireclay saucer, somewhat smaller and deeper than the roasting dish and is placed in a hot muffle where the metals can be oxidized and thus caused to enter the slag. The slag is provided by adding borax which is joined by the litharge of the oxidizing lead together with the silica of the dish.

- (d) *Fritting* is sometimes resorted to where the interfering element, such as tellurium, is likely to remove gold with it during fusion and cupellation at high temperatures. Fritting is conducted in the crucible in which the assay is to be completed. It is achieved by mixing the aliquot of powdered ore with the chosen flux, containing extra litharge, and placing the crucible in a furnace when the temperature is less than 500°C. The temperature of the furnace is then raised gradually for about 30 minutes to 700°C before being brought up to the usual fusion temperature to complete the fusion. The purpose of this treatment is to allow the oxidation of the metal to take place and for the oxides so formed to become incorporated in the slag.

(e) *Special fluxes* are generally used in the assay of by-products that originate from the metallurgical processes on the mines. Some auxiliary ingredients that go towards making up special fluxes are sodium or potassium nitrate, caustic soda, sulphur, iron, red lead and silica. The nitrates are used as oxidizing agents, as is red lead which also acts as a solvent of metallic oxides in a fusion, while silica contributes to fluidity in the presence of sodium carbonate. Sulphur will convert metallic iron to a fusible ferrous sulphide, while metallic iron can be used to give the reverse action where sulphur is present in excess.

By-products from the gold mines consist of borax slag, pyritic concentrates (or "black-sands"), furnace bricks, graphite and carborundum crucibles, fire-clay liners, furnace ashes, fine iron, sweepings, flue dust, matte, ferric oxide from pyrite-roasting plants and miscellaneous concentrates. These are currently despatched to the Rand Refinery for treatment and sale of the gold and silver content.

A general flux is used for the above work with certain additions to suit the particular material being assayed. This is known as "by-product flux" and consists of:

sodium carbonate	2 parts	by mass
litharge	4	" "
borax	1 part	" "

Details relating to variations in additions are given on pages 124-136 in *Assay Practice on the Witwatersrand*.

Certain additional directions are, however, given here to permit the assay procedure to be more adequately completed.

Borax Slag (silver and gold to be reported) is itself fusible, and so the flux to be used for the assay should be brought into approximate balance by the addition of silica to the by-products flux. The mass-measured pills from this assay must be annealed before parting, first with dilute (1 in 3) nitric acid and then with 3 to 1 nitric acid. (Original mass less gold found = silver.)

Lead Oxide Wash. A "litharge wash" is used in all high-value by-products assays. It consists of 20 g of litharge mixed with 2 g of maize meal and is added by means of a furnace spoon about five minutes before the fusion is due to be poured. This "wash" has the effect of providing a rain of

lead particles that comb through the molten slag to retrieve what little gold might otherwise be retained by slag.

Pots and Liners usually contain a high percentage of carbon, and roasting should be resorted to before fusion. As much borax slag will be present, re-inquartation and parting will be necessary.

Platinum Group Metals. The assay of these metals is not dealt with, but a detailed report on research done on P.G.M. assaying appears in the *Journal of the South African Institute of Assayers and Analysts*, No. 8 of July 1970, by W. R. Inman.

Calcium Sulphate. Because this salt tends to crystallize on the rotary-filter cloths (its solubility being little more than 0.2%), information is sometimes required relating to its concentration in the circulating cyanide solution. In this assay, the protective lime present in the solution has to be determined separately, and the CaSO_4 equivalent of the CaO deducted from the CaSO_4 determined in order to report " CaSO_4 in solution".

Standard Solution for Cyanide Works. The assayer is usually required to prepare the standard solutions that are used by operators on the plant to keep a check on the lime and cyanide strengths. These solutions constitute:

1. Silver nitrate, 13.08 g per litre, where 1 ml = 0.01% KCN when 100 ml of solution is titrated with potassium iodide indicator.
 2. Silver nitrate, 17.35 g per litre, where 1 ml = 0.01% NaCN when 100 ml of solution is titrated.
 3. Oxalic acid, 6.3 g per litre, where 1 ml = 0.01% CaO when 100 ml of solution is titrated.
- Determinations of minor constituents of plant solutions such as ferrocyanide, sulphocyanide, cyanate, etc., are described in *Assay Practice on the Witwatersrand* pages 309-23.

GRADING SAMPLES

Although in many cases the grading of samples of slime, mill pulps and crusher products is conducted in the reduction works, more frequently the assay department attends to grading tests. In either event the procedure is similar.

The most important samples to be sized are the slime originals and the slime residues. Whether they are taken by hand or by automatic samplers, it is customary for these samples to cover an eight hour shift and therefore three of

each type are collected daily. It is also general practice for a confirmatory twenty-four hour sample both of the pulp and residue to be taken as well. Thus a total of eight slime samples have to be graded each day through 48, 100 and 200 mesh Tyler sieves. All samples as received are moist and have to be dried either on hot plates, in ovens or by slow passage under infra-red lamps. Each dried sample is thoroughly mixed on a rubber or plastic sheet and if necessary is reduced in quantity by passing it through a Jones riffle sampler. After final mixing an amount of 100 grams is measured out, preferably on a direct reading balance and is sized through 200 mm diameter, 50 mm deep, testing sieves.

The separation of the screen sizes can be conducted either with water or in the dry state. In the former procedure the 100 gram sample is tipped into a bucket sieve with a 200 mesh Tyler screen forming the base and washed with water to remove the minus 200 mesh portion. The plus 200 mesh material remaining on the screen is removed, dried and put into the top screen of a nest of three testing sieves which are almost invariably 48, 100 and 200 Tyler mesh. However, if desired, other sizes such as 65 mesh or 150 mesh may be interpolated. The first screen, 48 mesh, is fitted with a lid and the last screen, 200 mesh, fits into a bottom pan. The nest is then placed in a mechanical shaker which is automatically timed to operate for seven minutes. After the shaker has stopped, the mass of the contents of each screen is determined and recorded. The sum of the plus 48, plus 100 and plus 200 mesh mass measurements are deducted from 100 to give the percentage of minus 200 mesh.

In the dry method the 100 g sample is placed on the top of the 48 mesh screen and after the lid has been fitted the nest of screens is agitated in the shaker for ten minutes. The contents of each screen are removed and their mass determined. The mass of the contents of the bottom pan is seldom determined to obtain the percentage of -200 mesh, as usually this figure is calculated by difference as in the case of the wet method. The -200 mesh material can be used in determining the plus and minus 325 mesh percentages if these are wanted.

When gold determinations of graded fractions are required, separate nests of screens are used for testing pulp samples and residues. Also the screens are not used for the grading of any other samples to avoid the possibility of gold contamination. The various fractions are retained in separate

containers until sufficient of the smallest quantity (plus 48) is available for assay. Each product is then assayed separately to determine its gold content. From these assays the distribution of gold in both the untreated slime and the cyanide residues is determined as shown in the following two examples. Periodic studies of the values so obtained can be of considerable assistance in assessing the effects of changes in milling parameters.

SLIME PULP

Size	Grading %	Assay g/l	Total gold	Gold distribution %
+ 48 mesh	1	2,1	0,021	0,20
+ 100 "	4	3,3	0,132	1,23
+ 200 "	20	6,2	1,240	11,51
- 200 "	75	12,5	9,375	87,06
Totals	100	—	10,768	100,00

SLIME RESIDUES

Size	Grading %	Assay g/l	Total gold	Gold distribution %
+ 48 mesh	1	1,42	0,014	4,67
+ 100 "	4	0,95	0,038	12,67
+ 200 "	20	0,64	0,128	42,66
- 200 "	75	0,16	0,120	40,00
Totals	100	—	0,300	100,00

From the assay values of the slime pulp and slime residue, the theoretical percentage extraction can be calculated for each size range.

Size	Head Value g/l	Residue g/l	Extraction
+ 48 mesh	2,10	1,42	32,4%
+ 100 "	3,30	0,95	71,2%
+ 200 "	6,20	0,64	89,7%
- 200 "	12,50	0,16	98,7%
Total	10,768	0,300	97,2%

The above exercises on gold distribution and extraction efficiencies are of greater value if performed on monthly aggregates, but can be applied over shorter periods or to a specially collected sample. Owing to the disparity in the proportions of each grading size, some attention has to be paid to preserving only part of the -200 portion of the daily samples, as otherwise too great a bulk would have to be stored and subsequently sampled. Special measures have to be taken to obtain a representative sample of the -200 mesh slime if the wet grading method is adopted.

Samples from the milling circuit are usually taken on a twenty-four hour basis and comprise mill outlets, hydro-cyclone overflows and spigot discharges. They are dried and then graded, usually dry, through a separate series of screens but of the same range, viz. 48, 100 and 200 Tyler mesh, i.e. 0.295 mm, 0.147 mm and 0.074 mm respectively.

Coarse size grading is the term applied when the samples contain material varying from 50 mm to 5 mm. They usually consist of crusher discharge products, screen undersize and the mill feed, the last being the most important. As samples of this type are bulky, around 0.03 m³, special sieves, 0.75 m square and 0.10 m deep, are mounted in a light frame resting on a rocking base. It is customary to handle each dried sample in toto and after determining its mass to tip it into the top screen of a tier of screens, usually 50 mm, 25 mm, 10 mm and 5 mm. The tier is rocked by hand until the sieves appear clear of undersize. A water spray is used to wash the adhering fines through the sieves and to damp the dust that is generated. The mass of the contents of each tared screen is determined after drying, and the combined mass of the various fractions subtracted from the original mass to determine the amount of undersize. The percentage of each size is then computed by reference to the total mass of the sample. With the close control exercised on the sizing of the mill feed by closed circuit screens, coarse size grading tests are rarely conducted as a daily routine, but mill feed samples are often assayed for gold content. Further description of grading practice is given in Assay Practice on the Witwatersrand, pages 100 to 108.

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PART II

CHAPTER 8

THE RAND REFINERY

Historical

For many years after gold was first produced on the Witwatersrand the bullion was sent to London for refining. In due course the idea of establishing a local refinery was mooted but a considerable time elapsed before any action was taken. The first move could be said to have been made in 1909, when the Transvaal Chamber of Mines founded a co-operative smelting works, situated on the Robinson Gold Mining property, to deal with the gold-and-silver-bearing by-products from reduction works. Such material required special treatment and being produced intermittently on individual mines was obviously best handled by a joint concern under the sponsorship of the Chamber. The company, which was known as the Witwatersrand Co-operative Smelting Works, Limited, started operating in April, 1910. The next stage was reached in 1917 when the South African Government proposed establishing a mint in South Africa. After a thorough investigation, this project was accepted and a branch of the Royal Mint was opened in Pretoria in 1922. During the same period the Transvaal Chamber of Mines investigated the possibility of establishing a refinery to treat the gold output of the mines¹. Technical discussions were held with Sir T. K. Rose on the process to be used, and the Miller chlorination process was decided upon. A private company, Rand Refinery, Limited, was registered on 27th November, 1920. The initial capital of £50 000 was raised from shares which were confined to the gold mining companies who were members of the Transvaal Chamber of Mines. An additional £165 000 was borrowed and secured by registered debentures. Following a comprehensive survey, a site in Germiston, Transvaal, was chosen. Here services were readily available, rail facilities were adequate and the position was close to the geographical centre of the Witwatersrand. By December, 1921, the refinery, with a capacity of twelve million Troy ounces (373 250 kilograms) of fine gold per annum, was completed.