Identification of Mineral Grains

A review of simple and rapid physical aids to identification

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Abstract

It is stressed that there is a need in the spheres of assaying, prospecting, and mineral-dressing for simple and rapid tests which will enable the appropriate mineralogical, chemical, and physical data to be obtained when examining composite samples of loose mineral grains.

Special attention is paid to problems confronting the mill-superintendent and a survey is made of rapid and simple *physical* methods which are suitable for the examination of mill products.

Introduction

In assaying, prospecting, and mineral-dressing it is often desirable, or necessary, to identify members of a given species occurring in a composite sample of loose grains and to make semi-quantitative estimations of some of the species or radicals present. It is in mineral-dressing, perhaps, that examinations of this nature assume dominant importance.

When mineral-dressing research is being conducted, either with a view to developing an efficient flow-sheet, or to determine the efficiency of a new mineral beneficiation process, the amount of time taken for the examination of products and the complexity of the tests are not usually important. Nevertheless, even during such work rapid and simple aids to identification, etc., can usually be employed to advantage. However, during the operation of a pilot-mill, and even more so during the working of a full-scale mill, extremely rapid tests are the only real aids to " close " control. It is not normally feasible, for example, to use a method in which the sample must first be embedded in bakelite and polished. Ideally also, the tests should require a minimum of apparatus and be such that they can be conducted within the mill by personnel with little or no analytical training.

It is impossible to maintain the highest degree of efficiency obtainable in any mill unless certain characteristics—physical, chemical, and mineralogical—of some, or all, of the material being treated can be readily ascertained within a short space of time. In other words, it is essential to employ such tests as will rapidly give information concerning the number of species present in a given sample ; the percentage of a given element, or species, in a sample ; the physical characteristics of fragments of a given species, etc.

Despite the obvious importance of such tests a perusal of relevant literature, discussions with mill-superintendents from large and small mines, and personal experience of several mills indicate that the development of suitable tests has been sadly neglected. Even when useful tests have been described, they are by no means as widely used as might be expected ; this is partly due to the fact that some are only to be found in journals not readily accessible to many mineral-dressers.

For the above reasons the present article is devoted to a review of physical methods which are primarily of value to the mill-superintendent (although some of those described are also useful to the assayer, prospector and the research worker in mineral-dressing). It must, however, be stressed that there are numerous simple, rapid and extremely valuable chemical aids which can often be used to advantage in conjunction with physical tests, but these require separate treatment.

Rapid Physical Methods

Rapid physical methods suitable for the routine examination of mill feeds and products are most useful when they involve the use of comparatively simple apparatus. Such methods have been classified as follows :

(1) Optical examination, under " white " or ultraviolet light, of samples of loose grains

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immersed in air or in liquids.

(2) Specific gravity methods.

(3) Magnetic methods.

(4) Vincent's electro-chemical magnetic method.

(5) Physical methods for the examination of radioactive substances.

(1) *Optical examination.*

If all the grains of a given species in a sample can be readily recognized because of their appearance (broadly a function of colour, degree of transparency, lustre and shape), the approximate percentage of that species can be determined by inspection, or, more accurately, by counting under the microscope.

It is important to realize that the ease with which a grain of a given mineral is identified under the microscope is often dependent on its size. Thus, whilst little difficulty is usually experienced in differentiating between large grains of pyrite and chalcopyrite by inspection alone, it is necessary to resort to differential staining, or other chemical techniques, in order to distinguish between fine grains of these species with any degree of certainty.

With experience remarkably small differences in the appearance of grains of different species occurring in *minus* 100-mesh fractions are often sufficient for each grain to be identified by *simple* optical methods. Thus, some of the experienced mineral-dressers of Nigeria are often able to make reasonably good estimates of the quantities of ilmenite and columbite present in a sample merely by examining some of the material under a microscope. Differentiation largely depends on the fact that columbite normally displays much higher reflectivity than ilmenite. However, when the columbite crystals are pitted or water-worn, the difference in reflectivity is slight, and then their estimates—which on several occasions have been checked by the writer against chemical analyses—are often very inaccurate. Even when made by men of experience, optical estimates of mixtures composed of grains of ilmenite and unworn and unpitted columbite, are sometimes much in error. Because of this Mackay (1950–51) [1] recommends heating a sample scattered over a thin layer of potassium bisulphate—sodium fluoride fusion mixture flux and then examining the cooled product under ultra-violet light. Each grain of columbite can then be identified because it is surrounded by a pale-yellow halo.

Further errors may arise when making rapid identifications by the simpler optical methods if the grains of a given species occur in a variety of colours or crystal habits. Thus, because of the relative development of the first order prism and second order pyramid, certain Nigerian zircon crystals resemble dodecahedra and so could, from a purely morphological point of view, be mistaken for garnets.

If a given species fluoresces under either short- or long-wave ultraviolet light use may be made of this property to establish its identity and to obtain semi-quantitative information concerning it. However, an estimate of the percentage of a fluorescent mineral present in a sample may be very inaccurate if it is based solely upon the appearance of the irradiated grains to the naked eye. For example, an extremely thin film of scheelite on wolframite grains—so thin that in daylight the latter appear quite free from contamination—will cause them to fluoresce quite strongly under short-wave ultraviolet light. Inaccuracies may also arise as a result of investigators assuming that all the grains of given species in a sample fluoresce, when in point of fact both fluorescent and non-fluorescent varieties are present. Thus, in many Nigerian alluvial or eluvial deposits both fluorescent and non-fluorescent zircons occur. Furthermore, when examining fine grains under ultraviolet light it is virtually impossible to determine the degree of liberation of fluorescent from non-fluorescent species, and examination of the untreated grains under ordinary light may not materially help in the solution of this problem if the appearances of the fluorescent and other species present are similar. This problem often arises when examining products containing scheelite together with such minerals as quartz and felspar, and therefore a method of imparting a characteristic colour to scheelite grains is clearly desirable. (Scheelite grains can, in fact, be stained blue by warming them with a solution of stannous chloride in HCl.)

Sometimes differentiation between grains of similar transparent species may be assisted by immersing the sample in a liquid of refractive index approximately equal to the mean refractive index of one of the species so that its grains almost disappear. Thus, the

[1] A brief list of references is given at the end of this article.

difference in appearance between grains of quartz and topaz may be emphasized by immersing them in ethylene dibromide, when the quartz grains become ghost-like whilst the topaz grains appear dark-edged. Although this technique is so well-known it is rarely employed in the mill.

Finally, the examination of very fine material under the microscope is facilitated by dispersing the components in a drop of detergent.

(2) *Specific Gravity.*

Because mill operators are familiar with gravity methods of concentration, rapid tests based on differences in the specific gravities of components of their products have been utilized by them more frequently than any other methods. Many of these tests possess the important advantage of requiring little apparatus.

Panning and vanning are extremely useful means of isolating heavy minerals from light and thereby enabling estimations to be made of the percentages of the former. However, to effect satisfactory separations considerable experience in the use of the pan or vanning shovel is necessary (although this difficulty may be by-passed by using a mechanical concentrator, such as the Super-Panner), but conclusions concerning the mineralogical character of a sample based almost entirely on panning or vanning may be quite inaccurate ; fine zircon and axinite have both been mistaken for cassiterite.

If the mineralogical characteristics of a given assemblage are known, rapid semi-quantitative data may be obtained by employing methods which depend for their success on differences in the specific gravities of the components. Thus, Hamilton (1953, pp. 263–5), states that, as there is a close relationship between the chromic oxide content of South African chromite ore and its specific gravity, the grade may be readily estimated. He also states that the discrepancy between the grades deduced from specific gravity and derived from chemical analyses has seldom been greater than 0·5% and never more than 1·0%.

In Nigeria, in order to estimate the columbite present in certain cassiterite-free mill products, specific gravity determinations of samples are made by a displacement method. Fifty or a hundred grams. of sample are placed in a burette which has been previously filled to a known level with petrol, and the new level is noted. Reference to a table then enables the percentage of columbite present to be determined, but with varying degrees of accuracy. The method depends on the assumption that the specific gravity of the columbite is 5·3 and that the average specific gravity of all the other components with which it is admixed is 4·5. Such an assumption is obviously dangerous, yet the results obtained by this method rarely differ seriously from those derived from chemical analyses.

It is well-known that a heavier mineral fraction can be separated from a lighter one by agitating the mixture in a liquid of intermediate specific gravity. Stable suspensions of metals in liquids, ranging in specific gravity from 3·5 to 7·5, have recently been marketed by the R. P. Cargille Company of America, so that the possibilities of this method of separation have been vastly increased. Heavy liquids and suspensions of metals in liquids could be used for numerous simple mill tests, yet the former have been largely neglected in the mill and the writer knows of no case in which suspensions of metals in liquids are used for control purposes.

The simplest and quickest way of separating high specific gravity grains from low is to mix a little of the sample with the appropriate liquid on a microscope slide and then lower another slide until it just touches the surface of the liquid. On raising the upper slide the floating grains are removed with some of the liquid. If the liquid has a refractive index approximating to one of the components of the sample this component may be readily recognized by optical examination of the appropriate fraction.

Occasionally melts can be usefully employed to separate some of the denser minerals. Thus ilmenite will float in molten lead chloride whilst columbite will sink (Flinter, 1955).

(3) *Magnetic Methods.*

The employment of permanent and electromagnets sometimes enables considerable qualitative and quantitative data to be determined fairly rapidly. Tests based on magnetic methods of separation are often used in the mill, probably because mill-men are familiar with their large-scale application. It is, however, important that fractions made by magnetic separations should be checked by optical and/or other methods in order to ascertain their composition and, in particular, to confirm that liberation of the

various species present is, for all practical purposes, complete, before the fractions are used to obtain quantitative data.

The following method, which is used by the writer to determine the approximate percentages of magnetic and non-magnetic cassiterite in certain closely-sized Malayan samples, illustrates not only the application of magnetic separation to routine mill-tests, but also the importance of combining simple physical and chemical techniques in order to obtain results quickly :—

A weighed sample is split into three fractions by means of an electro-magnet which has a variable resistance included in its circuit. Essentially, the strongly magnetic fraction consists of ilmenite, magnetic rutile, and cassiterite, whilst the weakly magnetic fraction is largely composed of monazite. The non-magnetic fraction contains cassiterite, zircon, quartz, tourmaline, and topaz. The fractions containing cassiterite are weighed and the cassiterite in each is " tinned " on a tray of sheet zinc. After the completion of the tinning the solution in the tray is diluted and the then buoyant cassiterite is persuaded by agitation to migrate to one corner—an operation which is usually not difficult to carry out. The corner of the tray is bent down and the cassiterite washed off. The cassiterite-free fraction is then removed, washed, dried, and weighed, and the cassiterite originally present is determined by difference. Hence the percentages of magnetic and non-magnetic cassiterite present in the original sample can be calculated.

The fact that the magnetic properties of some species are permanently altered by heat or by prolonged treatment with acid is sometimes of value in facilitating separations. Thus, magnetic cassiterite and the magnetic varieties of zircon occurring in Nigeria become non-magnetic when heated to about 1,000° C. for 10 to 15 minutes. If monazite or magnetic zircon is subjected to a prolonged leach with warm concentrated HCl both become non-magnetic (Jones, M.P., 1953). The possibility of altering the magnetic properties of other species by simple chemical or physical means is well worth while investigating.

By far the most satisfactory way of separating grains of species which differ but little in their magnetic properties is to use an " Isodynamic " separator. This type of apparatus is, however, expensive, and although easy to operate it takes a considerable time to effect precise separations. It is most profitably employed in research centres.

(4) *Vincent's Electro-Chemical Magnetic Method.*

Comparatively recently an interesting electro-chemical magnetic method of separating certain *minus* 100-mesh grains has been described by Vincent (1951, p. 1074). The apparatus consists of a small iron cylindrical container about 7 cm. high and of 7 cm. to 10 cm. internal diameter. The interior of the vertical walls is varnished. About 10 g. of the grains under examination are placed in the container which is then partially filled with a mixed solution of ferrous chloride and calcium chloride. An iron disc-shaped anode is inserted into the liquid and a current of density 0·05A. per square cm. is passed through the system for 3 or 4 minutes. The sample is then removed, washed and dried, and subjected to a magnet which removes the conducting grains because they have been thinly coated with iron. The iron is removed with dilute HCl, and the grains are washed, dried, and weighed.

It is claimed that this method yields quantitative results. Clearly the technique is an ingenious one, but considerable work remains to be done before its full possibilities can be assessed. For example, it would be interesting to determine whether " tinned " cassiterite grains could be similarly coated with iron and thus removed from associated non-conducting minerals.

Examples of conducting and non-conducting minerals (as far as this method is concerned) are listed below :—

Conductors.	*Non-conductors.*	
Pyrite	Quartz	Zircon
Galena	Silicates	Hematite
Copper sulphides	Barite	Ilmenite
Native metals	Wolframite	Cinnabar
Arsenopyrite	Apatite	Sphalerite
Graphite	Rutile	Manganese ores

(5) *Radioactive Substances.*

Of the numerous purely physical methods designed for the examination of radioactive substances only those employing a Geiger counter are of general use in mills. By using an appropriate type, not only may radioactive material be readily detected in a sample, but the approximate amount present may also be simply and rapidly determined. A counter does not, however, assist in the

identification of individual grains of radioactive mineral in a composite sample.

Some of the secondary uranium species are readily recognized, either because of their characteristic colour, or because they fluoresce characteristically under ultraviolet light. However, primary species are non-fluorescent, but some of the chemically simpler ones, including pitchblende, may be identified in a grain sample by utilizing Mackay's columbite test which has been already described. Under the conditions of the test such grains are surrounded by a brilliant yellow halo.

Conclusion

The great merit of the physical tests discussed above is their simplicity and yet it is probably this characteristic which has caused some to underrate their value. If this paper prompts mineral dressers to make fuller use of simple physical tests in order to effect close mill control it will have achieved the purpose for which it has been written.

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Witwatersrand Gold and Uranium

Notes on the analytical appraisal of value distribution

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Part I

Much thought has been given to the origin of gold in the conglomerate beds of the Upper Witwatersrand quartzites and uranium has now entered this field of interest. The weight of opinion on the origin of these deposits seems to the author to favour the placer theory rather than the solution theories. It is worth recalling that in the placer theory (1) [1] gold is supposed to have been deposited from suspension with the conglomerates and may subsequently have undergone redistribution by " solution ", whereas, according to the hydrothermal (2), as well as the infiltration and precipitation theories, it was introduced in solution. It is noteworthy that some evidence for the placer theory has recently come from the lead-isotope age determination of Witwatersrand uraninites by Louw (3), which is supported by the X-ray diffraction analyses of Wasserstein (4).

Payable gold is recovered on the Witwatersrand from reefs varying in thickness from a fraction of an inch to several feet. The quantities involved are often minute—say, one part in 200,000. Uranium may be a by-product, although in some mines it has now relegated gold into second place.

Values fluctuate wildly; their distribution is almost invariably positively skew, or log-normal (5). More often than not skewness is marked, but occasionally it may be mild. Relating the gold output of a mine to the area from which it is drawn yields a skew relationship that is characteristic of deposition in the area (5). That this should be so seems to be confirmed by Fig. 1, which illustrates the skewness of the general trend of values across pay-shoots.

The character of these skew curves would indicate that the formation of the deposit follows an exponential law. This view appears to be substantiated by Fig. 2, which yields an exponential relationship between the average development grade and payability of numerous reefs in mines fringing some

[1] Figures in parentheses refer to the list of references given at the end of this article.