

Rapid Identification *of*

MINERAL GRAINS IN COMPOSITE SAMPLES

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Abstract

It is stressed that rapid and simple aids to the identification of all the grains of a given mineral species in a composite sample which do not involve preliminary embedding and polishing can often facilitate "close" mill control.

Suitable chemical methods are reviewed and particular prominence is given to differential staining techniques.

Finally, a compilation of those rapid chemical methods of identifying grains of certain specific minerals which are known to the writer, but which have not been described in the body of the paper, is included so that they are readily available to those most likely to find them of use.

Introduction

For "close" mill-control the ideal analytical methods are those which can be employed in the mill itself—preferably by personnel who are not highly trained physicists, chemists or mineralogists—and which will enable data of a sufficient degree of accuracy to be obtained in a comparatively short space of time. Undoubtedly, before long, rapid physical methods will be generally employed in many mills to provide urgent analytical data, and the development, at Boliden, of a tape method of spectrochemical analysis for this specific purpose (Danielsson, 1957) is an important indication of this trend. This trend is further emphasized by the fact that the Japanese have constructed a robust polarigraph, primarily for "on-the-spot" determinations of copper, zinc, etc., in mill products (Mino, 1955), and the recent announcement that the Canadians are manufacturing a berylometer, by means of which beryllium can be quickly determined in all manner of material. The use of the Geiger counter to facilitate the treatment of radioactive ores is an example of the employment of a physical method of analysis well known to all.

Nevertheless, particularly in the operation of pilot plant and other small mills, those simple aids to "close" control which do not require the use of expensive apparatus will continue to prove of value.

Many simple physical and chemical methods exist for separating the various components of a composite sample and for enabling the grains of certain specific minerals to be made so distinctive that quantitative evaluation becomes comparatively quick and easy. Still, these aids are not used

by any means as generally as might be expected, possibly this is because much of the relevant information is widely scattered in the literature and not, therefore, readily available to those to whom it could be of the utmost use.

In this paper the writer has confined himself to reviewing one aspect of the subject, namely those rapid chemical methods by means of which all the grains of a given mineral in a composite sample can be quickly recognized without resorting to preliminary embedding and polishing.

Methods

For convenience, the rapid chemical aids to mineral-grain identification are discussed under the following headings: Insufflating and allied techniques; Staining mineral grains; Grain identification by gas attack, and Identification by fusion tests.

Insufflating Techniques

Williams and Nakhla (1950-51, pp. 282-284) have designed a small metal spraying apparatus, operated by compressed air, by means of which a true sample of fine mineral particles can be caused to adhere to reagent-impregnated gelatin-coated paper. The impregnating reagents used are such that they will impart a distinctive colour to all the grains of a given species. The technique is of somewhat restricted use because it can only be employed to identify those species which can be stained at room temperature by reagents of moderate concentration. Furthermore, the only additional treatment which can be safely applied to the paper after the sample has been blown on to it is to subject it to a gas (Obviously it can neither be sprayed with liquids nor immersed in a bath of reagent as such treatments might cause some of the particles to become detached.)

The apparatus, being robust, could be used in a mill and as a true sample attaches itself to the paper, with experience, the degree of variation in the quantity of a given substance present in a series of samples could be determined simply by inspection, with or without the added assistance of a microscope.

The following related technique is useful for comparing the amounts of certain species in a series of samples of grains within, say, the 40 to 80 mesh range. The method depends on taking prints, on spot-reaction paper, of a series of samples each of the same volume, and composed of loose grains.

and then comparing the prints. It has proved particularly useful to the writer for comparing the amounts of fluorite present in various mill-products consisting essentially of fluorite, quartz, calcite, galena and barite, and the following description of the method will serve to indicate the manner in which the technique may be used as an aid to the solution of similar problems. The test is based on the fact that calcium fluoride reacts with a hydrochloric acid solution of red-violet zirconium-alizarin reagent with the formation of colourless ZrF_6^{--} and Ca^{++} ions and yellow alizarin (Feigl, F., 1947, pp. 202-204). Thus, if a grain of fluorite rests for a short space of time on a piece of spot-reaction paper impregnated with the above reagent and heated with steam, on its removal the spot on which it rested is pale-yellow, whilst the surrounding paper remains pink.

In practice, a circle of 1.5-ins. dia. is drawn on a piece of spot-reaction paper and successive drops of reagent are placed in the centre until the whole of the circle, as a result of diffusion, is pink. A measured volume of grains is scattered as evenly as possible over the coloured zone and the paper is then placed on the top of a small beaker containing some gently-boiling water. After a minute the paper is removed and tapped to remove the grains. The size and number of pale yellow to colourless spots on the pink background gives some idea of the quantity of fluorite in the sample, and by comparing prints made from different samples, all but small variations in fluorite content are immediately recognisable.

Under the conditions of the test fluor-apatite and cryolite react in the same manner as fluorite. If the products have been subjected to flotation it is necessary to know what reagents have been used, as sodium fluoride is sometimes used to depress barite. Tests indicate that barite grains which have been depressed by the addition of 1 lb. of sodium fluoride per ton of ore all react positively to the above test. The reagent is prepared by dissolving 0.05 g. of zirconium nitrate in 50 ml. of water and 10 ml. of concentrated hydrochloric acid, and mixing it with a solution of 0.05 g. of sodium alizarin sulphonate in 50 ml. of water.

Straining Mineral Grains

If grains of a given mineral species are stained so that their colour is markedly different from those of associated grains of other species, an estimate of the quantity of the stained mineral present in the sample can be made rapidly by inspection, or a more accurate assessment can be obtained by the slower process of counting grains under the microscope. Staining is not only used to differentiate between species which appear essentially the same, but also to enhance the differences in appearance between species which can, without treatment, be differentiated by *careful* inspection.

It is worth mentioning that differentiation between the grains of similar transparent minerals can often be facilitated by immersing them in a liquid whose refractive index is such that the grains of one species are almost invisible.

Grains of a given species of transparent mineral may, on occasion, be made readily recognizable in a composite sample by subjecting them to the relatively neglected dispersion staining. The technique depends on the fact that "if particles of a transparent solid are placed in a liquid having the same refractive index as the solid for one wave length of light but a different refractive index for other

wave lengths, the colour for which the refractive index of the liquid and solid match will be transmitted without deviation, but other wave lengths will be deviated. When a microscope is adjusted for dark-field illumination, only the deviated colours reach the eye." (Crossman, 1948.) By immersing a mixture containing corundum, quartz, mica and glass in methylene iodide ($n_{D^{25}} 1.74$ at 25 deg. C.) and employing the above method of observation, the corundum grains appear yellow whilst the other components are white. Under similar circumstances if quartz is immersed in nitrobenzene it appears a brilliant blue. (McCrone, 1955, p. 134.)

That ultraviolet light is of considerable value in identifying untreated grains of certain minerals (e.g., scheelite, zircon, monazite, etc.) is well known, but the fact that visible light from high-energy incandescent lamps often causes minerals to emit infra-red luminescence which may, by being of diagnostic value, have some application in the mill, has not yet been generally appreciated. (See Barnes, D. F. Infra-red luminescence of minerals. U.S. Geological Survey Bulletin 1052-C.)

Usually the alteration of the particular grains is such that they can be readily distinguished in ordinary light, but the development of a superficial deposit which fluoresces characteristically under ultra-violet light may also be employed. The staining of grains of a given species may be effected by heat-treatment alone or followed by immersion in certain reagents, or by direct treatment with one or more reagents. The change, as a result of the treatment, may be due to conversion of much or all of the mineral to opaque oxide; to superficial reduction to metal, etc.; to superficial oxidation, with the resultant development of a transparent film displaying interference colours; to the development of a superficial compound composed of some of the components of the mineral and of the staining reagent, and to dye adsorption. In addition to the above staining methods, leaching may, on occasion, facilitate differentiation between grains of two similar species. Thus if a mixture composed of reddish-brown grains of Nigerian thorite and zircon are boiled with hydrochloric acid the thorite is converted to a fragile, white, silica shell which is readily distinguishable from the glassy, white, bleached zircon. (See Macleod, W.N. and Jones, M. P., 1955, pp. 168-169.)

Opaque Coating

The alteration of grains of a given species in such a manner that their colour is markedly different from grains of associated minerals under ordinary light and at room temperature is an ideal procedure because the grains can be identified without using a dark-room or any special physical apparatus other than a binocular microscope. The time required to stain fragments by the various methods discussed below is, of course, variable, but never great.

Staining by Calcination

It is a well-known fact that when certain minerals are heated in air they suffer an alteration in colour which is persistent at room-temperature. This change is either due to certain imperfectly understood reorganizations of the components or to oxidation. It is common knowledge that by heat-treatment, which results in reorganization within the crystal, brown Brazilian topaz becomes pink, pink Malayan zircon becomes colourless, and pale green beryl assumes a colour similar to that of an aquamarine. Clearly these particular facts are unlikely to be of widespread value to the

mineral-dresser, although it is the practice of Chinese tin-dressers in Malaya to roast certain concentrates in order to differentiate visually between fine zircon and cassiterite. However, another alteration of this type which may be used to advantage in the mill concerns cassiterite. All varieties of cassiterite yet examined by the writer change colour permanently on being heated for about 30 minutes at temperatures fairly near 1,000 deg. C. and, as is well known, this treatment also results in magnetic cassiterite becoming non-magnetic. Dark varieties of cassiterite become vermilion to orange and their transparency increases: even opaque white cassiterite which is slightly veined by knife-edge red veins (of hematite?) becomes pale pink.

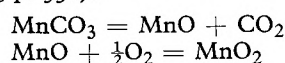
Several Nigerian mining companies have considered heating mill-products consisting essentially of columbite and magnetic cassiterite in order to destroy the magnetic properties of the latter and thus enable them to separate the two species by magnetic means. If this method is ever employed, the colour of the heat-treated cassiterite will greatly facilitate estimations of it in the columbite fraction as the colour of the latter is unaffected by the heat-treatment. The colour change is of added value because the tinning test yields negative tests when applied to thoroughly heat-treated Nigerian cassiterite.

Value of Heat Treatment

It is worth noting that the physical properties of the species are so modified by heat-treatment that their certain subsequent separation from associated minerals is considerably facilitated. Thus, for example, if "normal" spodumene (S.G., 3.13-3.20) is heated for 30 minutes at about 1,050 deg. C. it is converted to the β variety (S.G., 2.4) which will float in bromoform (S.G. c. 2.8). Clearly, then, this fact may be made the basis of a method of separation and evaluation of spodumene in composite samples which do not contain readily fusible components. It may also be noted that if β spodumene is boiled for 5 minutes with a 1.2 per cent aqueous solution of Methylene Blue, and subsequently washed well and examined wet, it appears dark blue whilst the minerals with which it is normally associated are pale blue or colourless.

The development of a characteristic oxide layer by the action of heat may be made use of to differentiate between particles of rhodochrosite and rhodonite in a mixture of these pink minerals. By subjecting the mixture to a low-temperature roast, rhodonite is unaltered, but rhodochrosite is changed super-

ficially to black dioxide in accordance with the following equations (Gaudin, 1935, p. 556):



This is the only useful example of this type known to the writer but it may well be that others exist.

In conclusion it must be pointed out that by employing heat-treatment to enable one species to be identified, the diagnostic features of associated species might be destroyed. Thus, heat-treatment of Franklin Furnace (New Jersey) products, in order to confirm the presence of rhodochrosite, would convert any yellow willemite present—which is readily detected because of its strong yellowish-green fluorescence under ultra-violet light—into a dull-grey, non-fluorescent substance.

Staining by reduction

Grains of cassiterite which have not been strongly heated can almost invariably be superficially reduced, by treatment with zinc and dilute hydrochloric acid, to gray, metallic tin which greatly facilitates their recognition. Before conducting the test, sulphides, etc., should be decomposed by boiling with aqua regia, and it is often necessary to subject roasted samples and samples from the zone of oxidation to preliminary acid clearing in order to expose the whole of the cassiterite to the reducing agents. However, even "clean" cassiterite, which has not been subjected to heat-treatment, does not invariably react positively to the zinc/hydrochloric acid test: this was noted by Beringer (1931, p. 220), and in recent years cassiterite with this unusual property has been found in Nigeria. Furthermore, as stated earlier, thoroughly heat-treated Nigerian magnetic cassiterite will not "tin" when subjected to the above test, and preliminary treatment with a number of chemicals has failed to restore this reducing property. However, certain Cornish cassiterites, when subjected to the same heat-treatment as that given to the Nigerian variety will "tin", though slowly: it thus appears that the ease with which heat-treated cassiterite can be tinned is not only a function (within limits) of the duration of the roast and the temperature, but also of the physical and/or chemical characteristics of the cassiterite tested.

Up to this point only reduction to metal has been considered, but less complete reductions may be employed to develop a characteristic coating on some minerals, notably certain tungstates. Thus, in order to facilitate the recognition under the microscope of scheelite in a mixture composed, for example, of grains of scheelite, quartz and feldspar, the tungstate fragments may be superficially reduced to a blue tungsten

compound. This reduction is effected by warming the sample for a few minutes in a strong solution of stannous chloride in hydrochloric acid. After this treatment the sample is washed slightly with water and examined wet. The blue stain disappears in an hour or so, but this does not constitute a serious drawback as the grains can be quickly restained, if necessary. This treatment also causes tungstic ochre, and all tungstates, except those of the ferberite-hubnerite series, to be stained blue. On occasion certain secondary molybdenum-bearing species may also stain blue or greenish blue.

Staining by precipitation

A variety of methods may be employed for developing a precipitate of diagnostic value on mineral grains. The coating most resorted to are the products of simple chemical reactions, either directly between a reagent and the mineral grains or between a reagent and a suitably reactive coating previously developed on the grains. It is reasonable to believe that coatings of diagnostic value may be developed for a wide variety of minerals by these means.

Although the development of a satisfactory stain for a given species depends to some extent, on trial and error, method of doing this can often be found reasonably rapidly if the following factors are borne in mind:—

- (i) The staining solution must be such that the mineral is only slightly soluble in it. In other words, the mineral must be so attacked that there is a slow emission of ions, which at the moment of liberation from the grain react with a component of the staining solution forming a precipitate at the surface which adheres to the surface.
- (ii) To ensure that the precipitate adheres to the surface, the rate of dissolution of the mineral must be slower than the rate of precipitation of the coloured coating.

These two points indicate that attention must be paid to the pH of the staining solution, the concentration of the attacking reagents, the temperature of the solution during the reaction and possibly the degree of agitation.

- (iii) Ideally, staining of the mineral should be effected without the evolution of gas, as the latter tends to break and displace the film at the surfaces of grains.

Because many sulphides can only be attacked by acids, which result in the liberation of appreciable quantities of hydrogen sulphide, it is necessary either to convert their superficial portions to sulphate, oxide or carbonate—which can often be stained by employing reactions not involving the evolution of gas—or to resort to the development of a

a manner described later—of transparent coatings displaying diagnostic interference colours. Thus, the most convenient way of staining galena grains, in order to distinguish readily between them and grains of somewhat similar lead-free minerals, is to proceed as follows: Roast the sample on the lid of an iron crucible until the grains become dull. (This converts the superficial portions of the galena to lead sulphate.) Transfer the sample to a watch-glass and cover it with 0.2 per cent. Sodium rhodizonate solution. Swirl the grains for a few seconds and then add sufficient buffer solution to discharge the colour of the solution. On examination under the microscope the galena grains appear reddish-purple. The buffer solution is prepared by dissolving 1.5 g. of tartaric acid and 1.9 g. of sodium bitartrate in a 100 ml. of water. (For further information concerning the chemistry of this test see Feigl, F. and Suter, H. A., 1942, p. 840 and Feigl, F. and Braile, N., 1943, p. 52.)

On the other hand, witherite grains can be stained pink by warming them for 3 or 4 minutes at about 60 deg. C. with a saturated solution of potassium permanganate made faintly acid with H_2SO_4 , then decanting and warming the grains with a strong solution of oxalic acid in order to destroy any permanganate which may be attached to certain other species present such as quartz, fluorite and barite.

This method of staining witherite—which was developed by the writer—depends on the fact that when barium sulphate is precipitated in the presence of potassium permanganate, the latter is strongly held by the sulphate and resists destruction by oxalic acid. See Feigl, F., 1947, p. 238.)

(iv) The coating must be insoluble.

(v) The reaction employed to develop the coating must be a fairly sensitive one.

(vi) The reagent chosen to produce the coloured coating must be such that under the conditions of the test it is either specific for a given mineral species or radical, or reacts in a distinctive manner with each of several radicals.

Thus, dithizone may be employed to stain both cerussite and smithsonite red. (Described in detail later.)

The writer found that by shaking certain mill-products from North Portugal with 5 per cent potassium ferrocyanide, slightly acidified with HCl, that he could differentiate rapidly between grains of molybdc ochre and those composed of bismuth ochre or tungstic ochre as the former stained brown, whilst the latter two stained blue on account of the readily soluble iron "impurity" in them.

(vii) By employing differences in reactivity towards a given solution,

species containing a common ion may often be differentially stained. Thus, if a sample of grains of witherite and barite are boiled for about 5 minutes with N. potassium chromate, only the witherite grains are stained yellow.

Suitably Reactive Coatings

Because it is not possible to attack certain minerals by common reagents without the evolution of gas—which, as stated above, is objectionable during staining—and because certain minerals are not reactive towards the available staining reagents, it is necessary as a preliminary to the staining of such types to develop a suitably reactive coating on them. Such coatings are most likely to be obtained by oxidation or double decomposition. Boiling with appropriate solutions is often sufficient to so modify the surface of the grain that it can be stained, but on occasion appropriate surface alteration can only be achieved by immersing the sample for a short while in a melt. The following examples illustrate the application of these processes to this problem.

Oxidation — Sulphides

By subjecting sulphides to a short calcination their superficial portions are usually converted to sulphate and/or oxide which often prove to be suitable media for staining. As noted above, it is necessary to calcine galena before staining it with sodium rhodizonate, and bournonite and jamesonite must be subjected to the same preliminary treatment if they are to be stained by the same reagent.

Oxidation — Carbonates and Hydroxides

As a result of roasting, carbonates and hydroxides are usually converted to oxides which may often be more readily stained than the original material. Thus, grains of brucite and/or hydromagnesite may be easily detected when admixed with grains of calcite by subjecting the sample to a short roast and then placing the cooled grains in a solution of silver nitrate. Brucite and hydromagnesite grains become coated with a brown to black film of silver hydroxide, whilst the calcite grains remain uncoloured. (Holmes, A., 1930, p.273.)

The presence of smithsonite in a sample of grains, which also contains quartz, barite and calcite, may be determined by roasting the sample on a square of asbestos paper. By this treatment smithsonite is converted superficially to zinc oxide, which is canary-yellow when hot. This colour is sufficiently marked to enable an estimate of the amount of smithsonite present to be made, but in

order to facilitate the counting of the smithsonite grains under the microscope it is necessary to confer a colour to the mineral which is apparent when the sample is cold. This may be done by damping the roasted sample with cobalt nitrate and then heating it strongly. On cooling, the smithsonite grains are bright green.

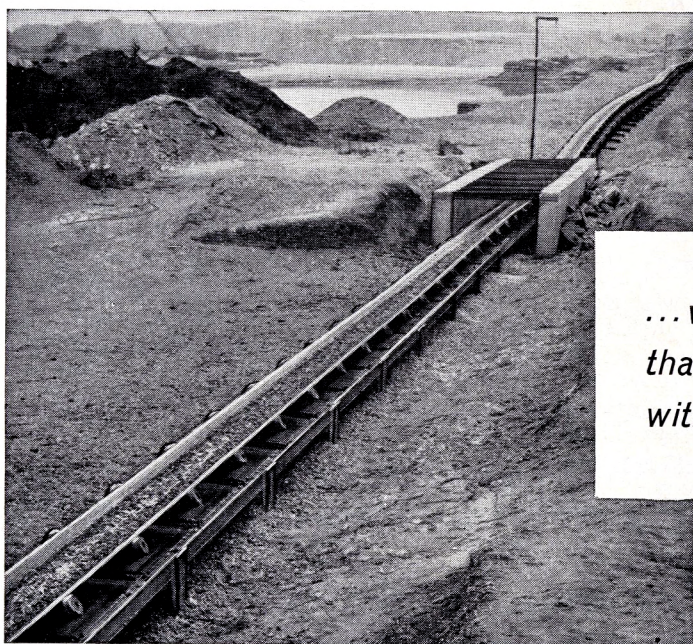
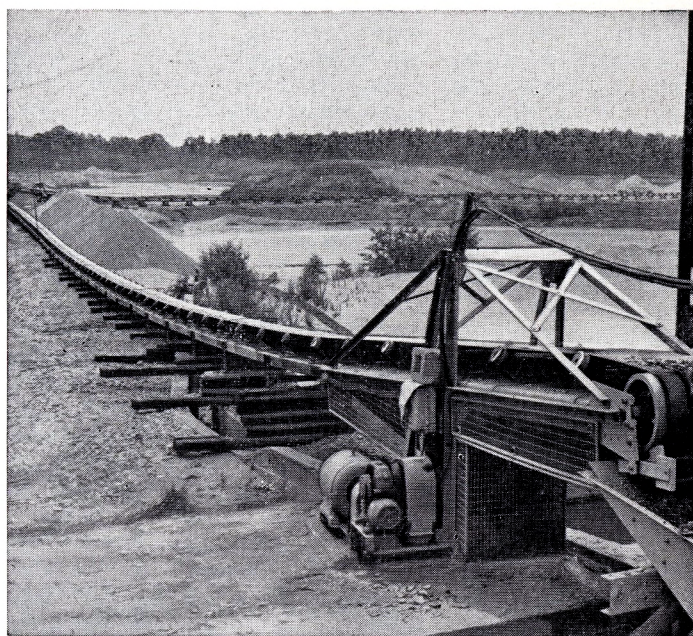
Double Decomposition — Sulphides

By employing double decomposition it is sometimes possible to develop a carbonate coating on grains of a given sulphide which will enable them to be stained easily. Thus, in order to stain nearly white sphalerite grains so that they might be readily recognised in certain mill-products, Paul Raffinot (1953, p. 5) first converts their superficial portions to carbonate and then stains them by employing dithizone in the following manner: The sample is boiled for 10 minutes in a solution of sodium hypochlorite (18 deg. Baume) containing 10 per cent of sodium carbonate. (Presumably the hypochlorite oxidises the sulphide to sulphate which is then converted to carbonate by double decomposition.) After the above treatment the sample is washed repeatedly with water in order to remove all the hypochlorite. The treated sample is then placed in a 0.1 per cent solution of dithizone in 1.0 per cent ammonia and the whole is warmed for 5 minutes at 50 deg. C. As a result of this treatment the sphalerite grains are stained red.

Double Decomposition — "Insolubles"

The following description of a method of staining barite grains, which was devised by Gaudin (1935, p. 555) "to differentiate between quartz and barite in short order so as to control a flotation concentrating operation", contains a typical example of a method of staining an insoluble mineral which depends on the initial development of a suitably reactive coating by double decomposition. "While the machine was running at reduced speed, a small sample of the froth was deslimed crudely, and boiled for a few minutes with an excess of sodium carbonate. This changed the surface of the barite to barium carbonate ($BaSO_4 + CO_3^{--} \rightarrow BaCO_3 \downarrow + SO_4^{--}$). The mineral was then washed and warmed with potassium chromate, which changed the barium carbonate to yellow barium chromate ($BaCO_3 + CrO_4^{--} \rightarrow BaCrO_4 \downarrow + CO_3^{--}$)."

As an example of a means of staining grains which depends first on superficially altering the surface of the minerals by means of attack by a melt, the method advocated by Lancsweert (see Vanden Herrewegen, 1954, pp. 478-480) may be cited for differentiating



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between tantalum/niobium minerals (particularly columbite and tantalite) and ilmenite in certain coarse fractions from the Belgian Congo. In essence the method depends on warming the sample in a porcelain crucible with potassium hydroxide pellets just damped with water. Finally excess potassium hydroxide is decanted off and dilute hydrochloric acid is added to the cold crucible. This removes the excess of potassium hydroxide and causes the columbite/tantalite grains to become white. The colour of grains of ilmenite, silicates, etc., is unchanged.

Jedwab (1957) employed a somewhat similar method to identify *single* grains of beryl: this involves subjecting the fragment to be tested to a brief attack by molten sodium hydroxide, followed by its immersion, for one minute, in a boiling aqueous solution of quinalizarin. This causes beryl to assume an intense blue-mauve hue, whilst quartz is unchanged: feldspars may develop violet patches which, unlike the beryl stain, disappear when the fragments are immersed in water for one hour.

Ampian (1962) developed the following modification of Jedwab's technique which enables all the beryl fragments in a composite sample to be stained simultaneously and in such a way that they can be easily and positively identified: 0.1 g. of the sample is heated with 5 ml. of 0.75 per cent aqueous sodium hydroxide solution, in a 100-ml. nickel beaker, until the water has been expelled and the residual NaOH has just melted. The beaker is then immediately removed from the source of heat and after cooling the melt is leached with c. 20 ml. of distilled water. The grains are collected by filtration through Whatman 41 H filter paper, and after they have been well washed they are transferred, by the aid of 5 ml. water, into a 50-ml. glass beaker. About 40 mg. of quinalizarin are added together with 3 drops of 5.0 per cent sodium hydroxide solution. The mixture in the covered beaker is boiled for 1 minute, then the grains are collected on filter paper, washed and dried. This treatment causes beryl fragments to be stained an intense blue colour, whilst albite, chrysoberyl, microcline, muscovite, orthoclase and phenacite are either unaltered or coated with reddish spots. "The surface alteration of particles subjected to normal mineral-dressing reagents and treatment does not affect the procedure."

Further types of stains depending on precipitation

In all but one of the examples yet cited, staining by precipitation has depended on the development of a coloured compound of a cation of the mineral under examination, but the following will serve to emphasize the fact that

other types exist. Thus, to enhance the difference between Nigerian thorite and zircon obtained—as noted earlier—by bleaching with hydrochloric acid, the following staining test may be employed (Hosking and Patterson, 1956, p. 74): Transfer c. 2 g. of the sample to a 100-ml. beaker and cover with the minimum of concentrated hydrochloric acid. Cover with a watch-glass and boil rapidly for 4 minutes. Decant, and wash the residue 5 or 6 times with water, but leave the solids just covered with water after the last wash. Add c. 0.5 g. of *finely-ground* ammonium molybdate and swirl to effect solution. Add 10 ml. of concentrated nitric acid and boil rapidly for 3 minutes. Decant wash 8 times with water, dry, and examine. This treatment causes thorite grains, unlike grains of other species normally occurring in Nigerian gravity concentrates, to assume a matt, intense lemon-yellow appearance, whilst zircon grains become leached and glassy. Any dark minerals are coated, to varying degrees, with a whitish veneer.

This test depends on reaction between the silicate anion and the staining reagent to produce yellow ammonium silicomolybdate.

The following similar test may be used to stain grains of amblygonite yellow and so enable grains of this species to be detected in a sample which also contains quartz, feldspar, spodumene, petalite, lithium and other micas and apatite (Hosking, 1957, p. 275): Place a few grams of the sample in a small beaker and cover to a depth of about $\frac{1}{2}$ in. with a solution prepared by dissolving 5 g. ammonium molybdate in 100 ml. cold water and pouring it into 35 ml. concentrated nitric acid. Bring the solution nearly to the boil and continue at this temperature for about 5 minutes, occasionally agitating the grains. Finally decant and wash the sample several times with water.

Dye-Adsorption Methods

Minerals of the Clay Group

The identification of minerals of the clay group can be established most satisfactorily by the employment of physical methods—some of which are prolonged—which require the use of costly apparatus operated by skilled personnel. However, for some years considerable researches have been made into the possibility of identifying these minerals by staining techniques, as these are quick, cheap and easily carried out. As a result of these investigations several reasonably reliable methods are now available. [See, for example, Faust, G. T. (1940) and Hambleton, W. W. and Dodd, C. G. (1953).]

It is sometimes desirable to identify clay minerals, as a group, in mill-material, and, therefore, it is pertinent briefly to describe the procedure adopted by Faust (1940, p. 20) for indicating that an exceedingly fine constituent in fluorite ore from Helena (Montana) was clay. The ore was finely crushed and boiled with nitric acid to destroy carbonates and any organic matter. This residue was then treated with perchloric acid to remove the fluorite, and after washing it was immersed in a solution of malachite green in benzene when the fine fraction assumed an emerald-green colour indicating that it was clay.

Other Minerals

Gaudin (1935, pp. 554-555) recommends the use of Malachite Green for staining hydrous oxides, and notes that "the action seems to be specific for minerals with hydroxyl radicals." If the mineral is colourless, Malachite Green stains it blue, but if it is coloured the dye causes it to assume a colour which is intermediate between blue and its natural colour. In particular, Gaudin notes that Malachite Green will stain grains of bauxite and of limonite, and states that similar staining reactions occur when other organic dyes are used.

It is possible to differentiate between grains of calcite and dolomite in certain mixtures by immersing the sample in cold Lemberg's solution. This treatment causes grains of calcite to be stained violet within 5 to 10 minutes, whereas dolomite grains show no colour change for at least 20 minutes, after which they are faintly speckled with blue.

Lemberg's solution is prepared by boiling together 60 parts of water, 4 of Al_2Cl_6 , and 6 of logwood chips for 25 minutes. During the period of boiling the solution is constantly stirred and water is added periodically to replace that lost by evaporation.

The staining of the calcite depends on the fact that it develops a coating of aluminium hydroxide and this adsorbs the logwood dye.

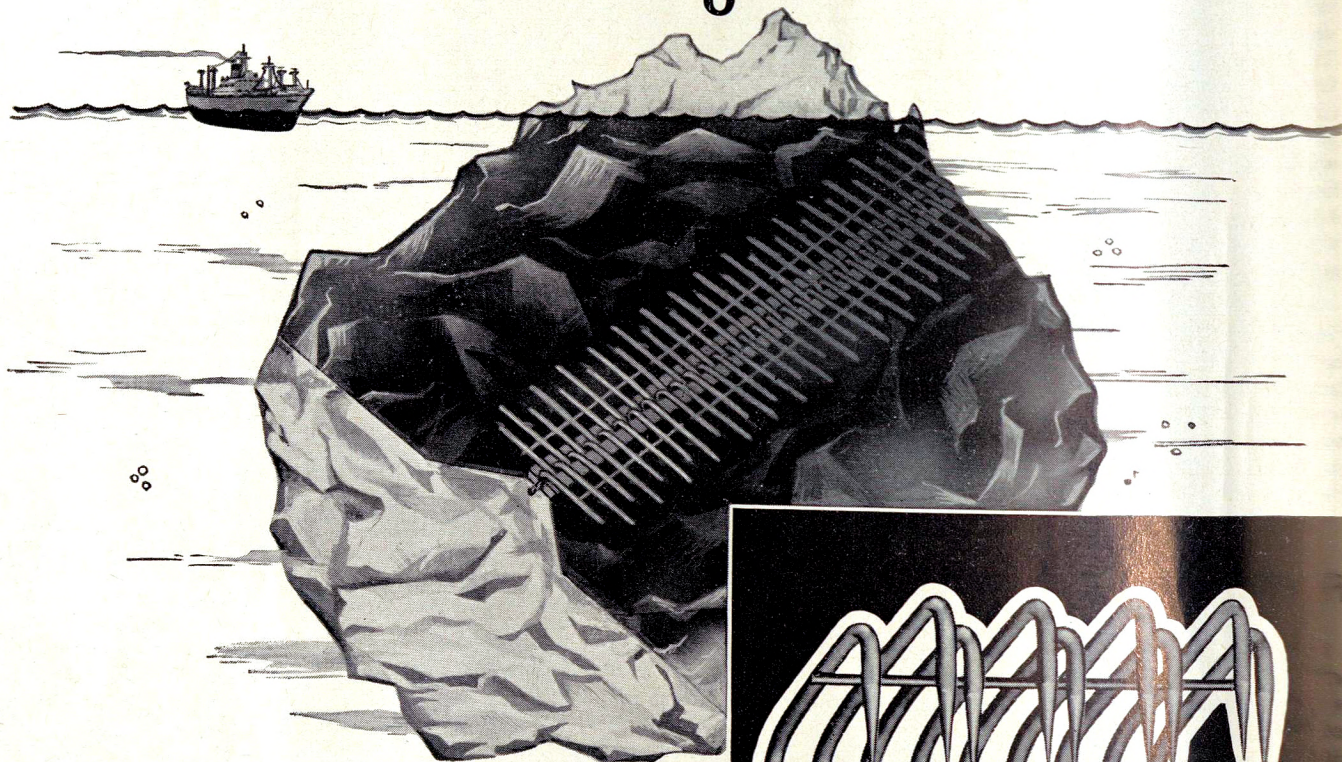
The staining of certain magnesium-bearing minerals by means of Titan Yellow—which is noted later—are further examples of adsorption staining.

Perhaps the staining of β spodumene by methylene blue—which is discussed earlier—is a further example of this general method, but this is uncertain.

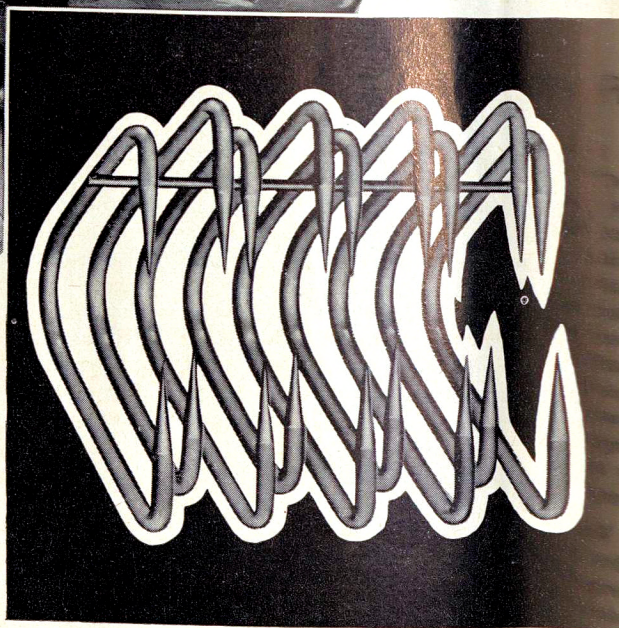
Colour at Elevated Temperatures

It has been noted above that when the superficial portions of fragments of certain zinc species are converted to oxide by roasting they exhibit a strong canary-yellow colour when they are hot

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which enables them to be identified readily. This is the outstanding example of the method of "staining" grains which depends on the use of atmospheric oxygen and the continued application of heat.

Allied to the above method of identification is that which depends on the fact that certain species emit light when subjected to gentle heat. If a sample containing grains of fluorite is heated in a dark-room to about 150 deg. C. the fluorite grains become luminescent, thus enabling an estimate to be made of the amount of this material in the sample. The colour of the light emitted by fluorite varies with the variety: the chlorophane variety emits an emerald-green light, whilst others emit lights which are characterized by purple, blue and reddish tints. (See Dana, E.S., 1932, p. 275.) Grains of fluorite which have been heated and cooled do not become luminescent on reheating.

According to Dana (1932, p. 275) "some varieties of white limestone or marble, after slight heating, emit a yellow light; so also tremolite, danburgite and other species. Doubtless these facts could be used advantageously in work involving the examination of samples containing fragments of these species.

Fluorescent Coatings

As many insoluble, fluorescent compounds are known which are produced by reactions between inorganic cations and organic reagents, it should be possible to develop such compounds on the surfaces of mineral grains, and by so doing facilitate their identification. The writer has shown that this assumption is correct by developing the following procedure for depositing a fluorescent coating on grains of the three most important secondary zinc minerals, namely smithsonite, hydrozincite and hemimorphite:—

Warm the sample for 5 minutes, at about 65 deg. C., with a mixture consisting of equal volumes of a 0.5 per cent solution of 8-hydroxyquinoline in 60 per cent ethanol, and 5N. ammonia. Then decant and wash the grains well in water. This treatment causes the above zinc species to be stained yellow and to fluoresce a brilliant yellow under both long- and short-wave ultraviolet light.

In addition to zinc oxinate, the oxinates of the following metals also fluoresce when treated with ammonia and examined under ultraviolet light:—Ba, Sr, Mg, Ca, Al, Sn(ii), Sn(iv) and Cd. (See Pollard, F. H. and McOmie, J. F. W., 1953, pp. 53-54.) However, minerals containing these elements which are likely to be associated with zinc

Table 1. Reactions between grains and 0.1 N. Silver Nitrate		
Mineral	Nature of coating, etc.	Method of Conducting Test
Arsenopyrite	Grains cohere	Boil for 1 minute
Bornite	Grey coating of silver	Agitate grains in cold solution
Bournonite	No apparent change	Boil for 1 minute
Chalcocite	Grey coating of silver	Agitate grains in cold solution
Chalcopyrite	Blue or purple film	Boil for 1 minute
Cuprite	Grey coating of silver	" " " "
Galena	Grains somewhat dull	" " " "
Niccolite	Grey coating of silver	Agitate grains in cold solution
Pentlandite	Slight orange-yellow film. Patchy	Boil for 1 minute
Pyrite	No apparent change	" " " "
Pyrrhotite	Purplish-brown film	" " " "
Sphalerite	No apparent change	" " " "
Stannite	" " "	" " " "
Tetrahedrite	" " "	" " " "

(i) The silver coating tends to develop somewhat quicker on chalcocite than on bornite. It is possible that the various samples of bornite grains reacted positively because of included chalcocite, but this has not been established.

(ii) When the above staining method is employed as an aid to the estimation of chalcopyrite in a sample which also contains pyrrhotite, the stain on the latter may be removed (to avoid confusion) by washing the sample in 1.0 per cent sodium nitrite. (Raffinot, P., 1953, p. 6.)

deposits will not, in the vast majority of cases, form oxinates by this treatment.

As a further example of mineral grain identification by the development of a characteristic fluorescent coating it can be recorded that after fragments of bauxite have been warmed for a few minutes with equal volumes of the 8-hydroxyquinoline solution noted immediately above, and 5N. acetic acid, that they fluoresce yellow-green under both long- and short-wave ultraviolet light.

Interference Films

It is wellknown that when white light falls upon a surface which is coated with a transparent film that interference colours are seen. The colour which is observed is due primarily to the thickness of the transparent film, but is modified by the body-colour of the film, by differences in the reflectivity of the upper and lower surfaces of the film, and by the colour and other physical characteristics of the substratum. Obviously then, if films are developed on several mineral species by identical treatment, the interference colour displayed by any one is likely to differ from those of the others and so serve as an aid to its identification. Reasoning of this sort led Gaudin (1935, pp. 556-562) to develop some most valuable aids to the recognition of fragments of certain sulphides in mill-products, provided they were first embedded in bakelite and polished. Gaudin stressed the importance of controlling the staining operation rigorously: an obvious necessity in view of the fact that the colour depends largely on the thickness of the coating. He also suggested that transparent oxide films

would probably prove to be the most satisfactory type, from a diagnostic point of view, when examining sulphides, and noted that certain reactions which would result in the development of transparent films on some sulphides would cause opaque coatings to develop on others. Staining methods employing reactions of the latter type were termed "Combination Methods" by him.

Independent investigations by Raffinot (1953, pp. 5-6) and the writer have demonstrated that dilute silver nitrate (one of the staining reagents used successfully by Gaudin) was also capable of staining loose grains of a number of sulphides in a way which made them readily recognisable, provided the temperature of the solution and the immersion times were carefully controlled.

Gas Liberation

Carbonate and sulphide grains may be detected by methods which depend on the liberation of gas. It is well known that one or more of the diluted mineral acids react readily with most mineral carbonates, causing the evolution of carbon dioxide. Thus, if a portion of the sample under test is placed on a glass slide, treated with dilute acid, and observed under the microscope, grains of Carbonate minerals can be identified because of the trains of bubbles emanating from them.

Grains of minerals which contain sulphide ions can be identified rapidly and surely by employing the sodium azide/iodine test. (See Feigl F., 1928, p. 369 and 1947, pp. 227-229 and p.301.)

(To be concluded)

Rapid Identification of

MINERAL GRAINS IN COMPOSITE SAMPLES

K. F. G. HOSKING, M.Sc., Ph.D., A.M.I.M.M.

The author concludes the review of suitable chemical methods for rapidly identifying mineral grains published in the January issue. Differential staining techniques are given particular prominence.

TO examine a sample for sulphides, a portion of it is spread out on a glass slide covered with the reagent, and examined under the microscope. A train of nitrogen bubbles is initiated by every grain which contains sulphide ions.

Use of Nitrogen

Under standard conditions the rate of evolution of the nitrogen bubbles is to some extent governed by the percentage of sulphide sulphur in the grain. It is, for example, quite easy to differentiate between a sample of arsenopyrite and of loellingite because of this fact. Furthermore, because the rare minerals helvite, genthelvite and danalite contain only a small percentage of sulphide sulphur, nitrogen bubbles are liberated very slowly when they are immersed in the azide/iodine reagent: nevertheless, the evolution is sufficiently rapid to permit a distinction to be made between grains of these minerals and garnet grains, which they resemble and with which they are often associated. (A method of identifying these minerals by staining is described later.)

Grains of some sulphides tarnish extremely quickly, and may then be quite unreactive towards the azide/iodine reagent. Pyrite, for example, which might appear quite bright, may not react—presumably because a thin, colourless film has developed on it as a result of oxidation. Therefore, except when testing grains which were taken immediately before from the grinding-circuit of a mill, it is wise to subject the sample to a short treatment with hot concentrated

hydrochloric acid, followed by a wash in distilled water, before applying the azide/iodine test to it.

It must also be noted that some flotation reagents contain sulphide sulphur, and that unless steps are taken to remove all traces of these from a sample by an appropriate acid treatment, misleading results will be obtained on applying the azide/iodine test. (In practice it is often extremely difficult to remove some reagents when they are adsorbed on the surfaces of mineral grains without resorting to very drastic treatment.)

The reagent, which is fairly stable, is prepared by dissolving 3 g. of sodium azide in 100 ml. of 0.1N iodine solution.

Fusion Tests

Grains of certain of the more intractable minerals can be conveniently identified by subjecting them to fusion tests which result in the development of products which are either characteristically coloured, or which fluoresce characteristically under ultraviolet light.

The most noteworthy method of this type is that developed by MacKay, and others (see MacKay, R. A., 1950-51, pp. 129-131) for identifying columbite grains in Nigerian products. Identification depends on the fact that when Nigerian columbite grains are heated on a thin layer of flux composed of sodium bisulphate, sodium fluoride and fusion mixture, each grain becomes surrounded by a reaction-product halo which fluoresces pale-yellow under ultraviolet light. As the test is well known it need not be discussed at length, but it is worth mentioning, however, that it is also extremely useful for detecting grains of

pitchblende in Cornish cassiterite concentrates. Under the conditions of the test the pitchblende grains become encircled by a halo which fluoresces an intense yellow under ultraviolet light.

Orsino Smith (1953, p. 60) notes several elements which when fused with sodium or lithium fluoride form compounds that fluoresce characteristically under ultraviolet light. Possibly these may be used to form the basis of tests—similar to MacKay's test—for identifying grains of certain tungsten, titanium, etc., minerals, although the writer has had no success, as yet, in this sphere.

However, grains of wolframite, and other manganese-rich minerals, may be detected as follows: Place a few pellets of sodium hydroxide on a square of asbestos paper (of side 1.5 inches) and near to one edge. Holding the paper with a tongs, heat it over a large burner in such a way that as the hydroxide melts it runs over the paper, forming first a thin veneer and then sinking into it. At the moment when that portion of the paper over which the melt has spread assumes a matt appearance, sprinkle the sample over it and continue heating for about 30 seconds. (If the grains are coarse they should be gently pre-heated, otherwise they may decrepitate during the test and be lost from the sample.) Manganese-rich grains thus treated become partially coated with green sodium manganate and are surrounded by blue-green haloes.

As an alternative the test may be carried out on a thin veneer of sodium hydroxide on a silica crucible lid.

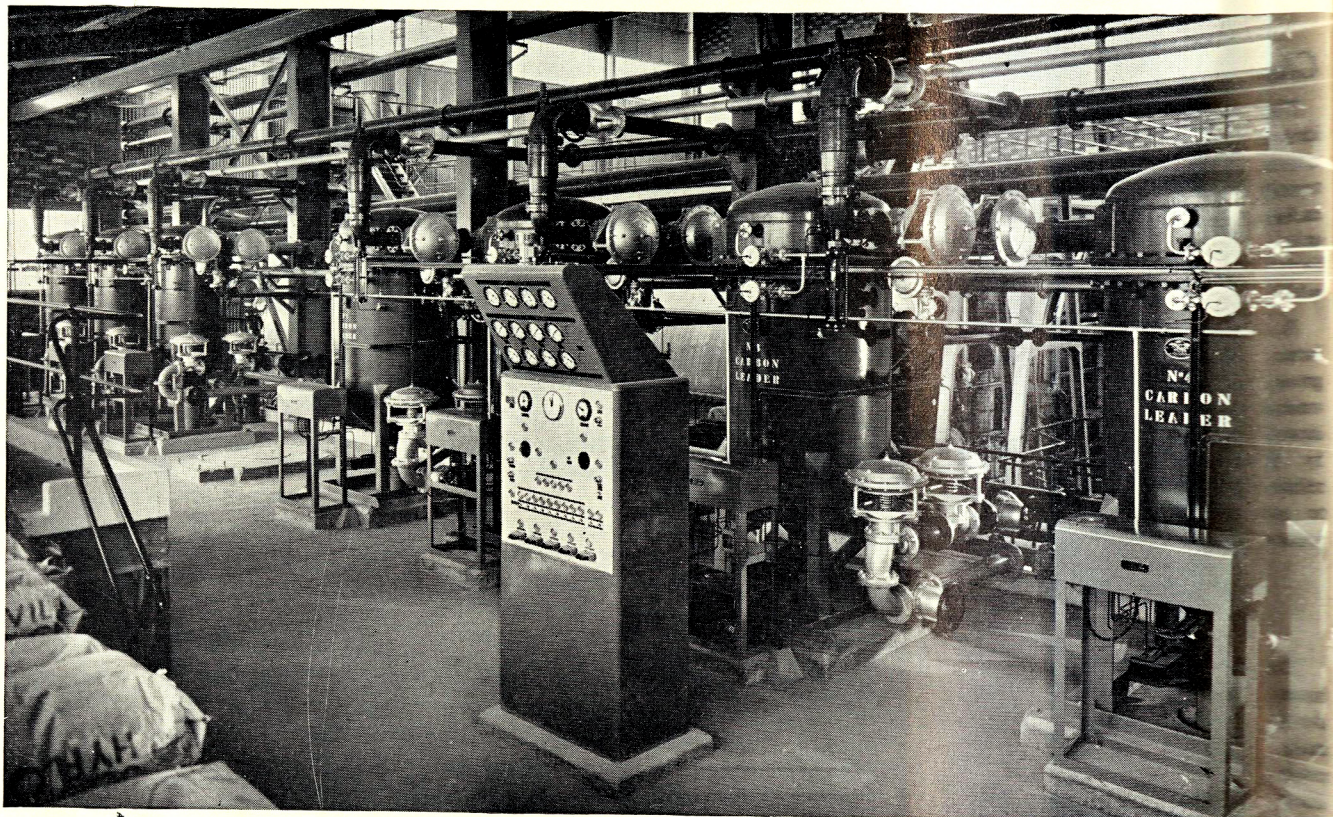
A more convenient way of detecting grains of wolframite in many types of sample is to boil a few grams of the



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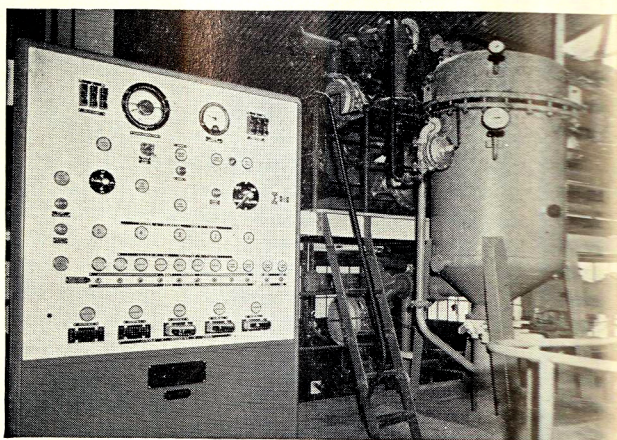
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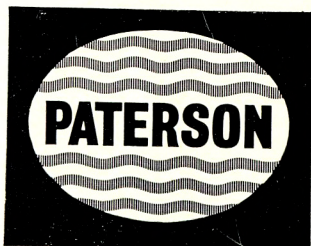
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material for c. 20 minutes with a solution of aqua regia of the following composition: HCl, 25 ml.; HNO₃, 15 ml.; H₂O, 60 ml. This treatment causes grains of wolframite (and scheelite) to assume a yellow colour (Vanden Herrewegen, 1954, p. 477).

Chemical Aids

The following chemical aids to the identification of grains of specific minerals in "loose" composite samples, together with those embodied in the earlier parts of this paper, include not only the vast majority occurring in the literature, but also a number of new ones which have been developed by the writer.

Published methods for differentiating between members of the clay-minerals group have, however, been largely omitted. Those aids which have been described by others have been repeated by the writer in order to check their reliability and attempts have been made to extend their fields of application whenever possible.

The Alizarin S Method

The method of staining grains of bauxite, which is described below, was developed by the writer in order to make rapid comparisons between various products of mill-tests which were conducted in order to examine the possibility of improving the grade of certain bauxitic material.

The samples contained grains of bauxite, kaolinite, felspar, quartz and ilmenite, and a large percentage of the components were coated with a film of limonite.

Boil 10 g. of the sample for a few minutes in HCl in order to dissolve the limonite. Cool; dilute well with water; swirl; allow the solids to settle and decant. Repeat the washing until all the soluble iron salts are removed. Cover the insoluble residue with N. NaOH and after 2 minutes add 0.1 per cent alizarin S solution until an intense violet colour develops. Tumble the insolubles in this solution for 1 minute, and after a further 4 minutes add 5N. acetic acid until the violet colour of the liquid is discharged. Decant and wash the grains well with water. The bauxite fragments are stained purplish-pink.

The method is based on a test for aluminium which was devised by F. W. Atack (1915, p. 936).

Methods of staining grains of bauxite by employing Malachite Green and 8-hydroxyquinoline have been described earlier.

Grains of felspar, but not accompanying grains of quartz, may be stained by the following method which was developed by A. L. Engel (1938, pp. 69-70) in order that the grades of products produced during experimental work on the separation of felspar from quartz by flotation could be rapidly estimated by visual means.

Place a few grams of the material to be examined in a beaker and cover them with equal volumes of HCl and warm distilled water. Digest for 5 min., then wash the sample until it is free of acid and dry on the sand-bath. Cover the dried material with a solution of Safranin O and warm gently on a hot plate for at least 15 minutes. When the staining appears to be complete—that is, when certain components are obviously stained yellow or pink—decant and wash the grains thoroughly with distilled water. (The grains must be tumbled during the washing process in order to remove any traces of the stain on grains other than felspar.) Decant the wash water, then dry the product on a sand-bath and examine it. The felspar grains will either be distinctly yellow or pink. The Safranin O solution is prepared by dissolving 2 g. of Safranin O in 30 ml. of ethyl alcohol and adding 70 ml. of distilled water.

Engel found the method satisfactory for the problem in hand, but inferred that it might not be applicable to all felspar-quartz products. The present writer has applied it to several Cornish samples and has found it completely satisfactory. Feigl (1947, pp. 470-471) describes the test briefly but states, erroneously that the quartz—but not the felspar—is stained.

Quartz, Potash Minerals and Plagioclase Felspars

The following method was devised by Graham (1955, pp. 37-38) primarily to differentiate rapidly between grains of certain silicon-containing species in soil samples, but on occasion it could obviously be used to advantage in the mill.

Having acid-cleaned a known quantity of the sample (in order to remove soluble iron compounds) mount it on a microscope slide in such a manner that the grains are only partially embedded in the cement. Subject the sample to the fumes of hydrofluoric acid for a few minutes and then immerse the slide in a saturated solution of sodium cobaltinitrite.

After 5 minutes remove the slide, wash it with distilled water and immerse it for 5 minutes in 0.1 per cent aqueous Malachite Green. Finally rinse in distilled water and dry. This treatment

causes microcline, orthoclase, and potash mica grains to be stained greenish-yellow, whilst albite becomes greenish-blue. The lime-rich plagioclases are more deeply etched than albite and are stained a paler hue. Quartz remains clear and unetched.

Chrysotile and Other Varieties of Asbestos

Morton and Baker (1941, pp. 515-523) devised the following test for differentiating between small fragments of chrysotile and other varieties of asbestos in order to assist the Canadian asbestos industry.

Place a few drops of a 1 per cent solution of iodine in glycerin on a microscope slide and disperse a little of the sample to be tested in the liquid. Within a short space of time chrysotile—but not other varieties of asbestos—is stained brown. Brucite and serpentine are also stained brown by the same treatment.

Witherite and Similar Species

The chromate and permanganate-sulphuric acid methods of staining grains of witherite in order to differentiate between them and grains of fluorite, quartz, barite, etc. have already been described.

A further method, which is given below, is applicable in the absence of lead minerals, and is based upon the fact that a red salt is precipitated when barium ions react with sodium rhodizonate in a weakly acid solution. (See Feigl, F., 1947, pp. 165-167.)

Place a few grams of the sample in a beaker and cover them with a freshly-prepared 0.2 per cent solution of sodium rhodizonate. Warm at about 60 deg. C. for a few minutes and decant the solution. Add 1:20 HCl; tumble the grains rapidly; decant and wash the residue with water. As a result of this treatment witherite grains are stained pink, whereas grains of quartz, barite, fluorite, calcite, celestite, and strontianite are unstained.

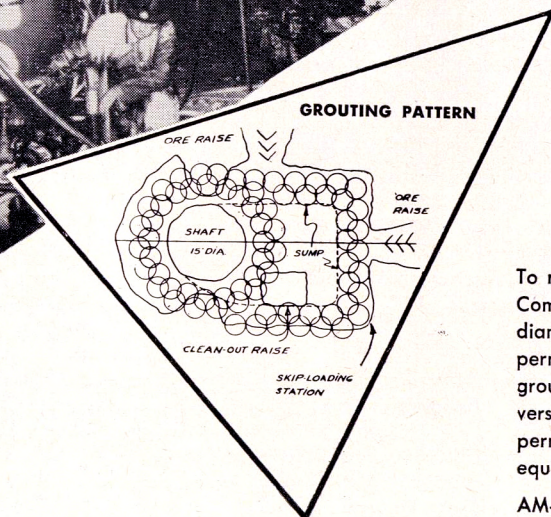
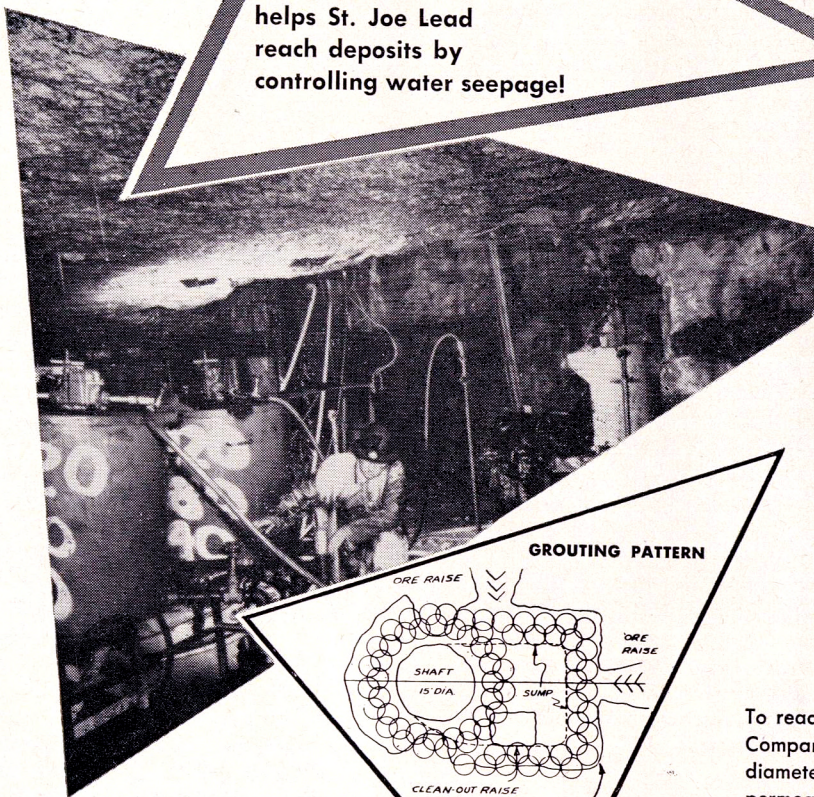
In order not to destroy the pink film on the witherite it is essential that the sample should only remain in the 1:20 HCl for a very short space of time.

Calcite

Theoretically it should be easy to differentiate between grains of calcite and quartz because of differences in their refractive indices and because of the characteristic cleavage of calcite. Yet, as Gaudin remarks (1935, p. 554), "both of these criteria may fail: a coarser quartz grain and a finer calcite grain may give similar interference colours, especially in suitable orientations; a locked particle consisting of calcite and quartz would be very embarrassing". Gaudin also points out that calcite can be

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stained in a variety of ways to suit the requirements of almost any problem and notes that when it is boiled with ferric chloride it assumes a yellow to brown coat of ferric hydroxide; with nickel nitrate, a green coat of nickel hydroxide; with silver nitrate, a brown to black coat of silver hydroxide. Quartz is unaffected by these reagents.

The present writer has found that calcite grains in a quartz-calcite mixture can be strongly stained by employing the following method:—

Boil the sample with 10 per cent copper sulphate for 5 minutes then decant and wash the grains thoroughly with water. Finally, tumble the grains in a 1.0 per cent alcoholic solution of rubenic acid then decant and wash the residue slightly with water. The calcite grains are thus stained dark green to black, whilst the quartz grains are unaffected.

Calcite and Dolomite

Holmes (1930, pp. 265-267) recommends the following methods for differentiating between grains of calcite and dolomite:—

Aluminium Chloride/Logwood Solution

This method has already been described.

Silver Chromate Method—Immerse the sample for 3 to 4 minutes in 10 per cent silver nitrate solution heated to about 65 deg. C., then decant and wash the grains well with distilled water in order to remove all free silver salts. Finally, cover the grains with potassium chromate and after a minute decant and wash the residue. Calcite grains thus treated are reddish-brown, whilst dolomite and quartz grains are unaffected. Aragonite is stained less deeply than calcite.

Copper Nitrate Method—Boil the sample for a few minutes with very dilute copper nitrate solution. This treatment causes calcite grains to be stained green, whilst dolomite and quartz grains are unaffected.

Magnesite Grains

Titan Yellow Method—The following method of staining grains of magnesite—which is based upon a test for magnesium due to I. M. Kolthoff (1927, p. 254)—was developed by the writer in order to differentiate between grains of magnesite

and dolomite and to facilitate the recognition of magnesite grains in a mixture which also contains such minerals as quartz, calcite, fluorite, dolomite and the sulphates and carbonates of the alkaline earths.

Place a few grams of the sample in a beaker and add 0.1 per cent Titan Yellow until the grains are covered with a layer of solution about 1 cm. deep. Heat until the solution just boils, then remove the beaker from the source of heat and add 5N. sodium hydroxide until the solution is vermilion. Tumble the grains gently for about 30 sec., then decant and wash the residue. This treatment causes magnesite grains to be stained pink, whilst grains of the other species noted above are unaffected.

Diphenylcarbazine Method—In order to differentiate between grains of magnesite and dolomite, Feigl (1947, pp. 442-443) recommends the following method.

Cover a little of the test-material with a hot alcoholic alkaline solution of diphenylcarbazine. After 5 minutes decant, and wash the grains repeatedly in hot water until no more colouring matter goes into solution. This treatment causes the magnesite grains to become red-violet, whereas the dolomite grains are unaffected.

Dolomite grains may also be stained by the above procedure provided they are first ignited.

Danalite-Helvite-Genthelvitite Group

The formulae of the end-members of the danalite-helvite-genthelvitite group are as follows:—

Danalite, $[\text{Fe}_8 \text{Be}_6 \text{Si}_6 \text{O}_{24} \text{S}_2]$.
Helvite, $[\text{Mn}_8 \text{Be}_6 \text{Si}_6 \text{O}_{24} \text{S}_2]$.
Genthelvitite, $[\text{Zn}_8 \text{Be}_6 \text{Si}_6 \text{O}_{24} \text{S}_2]$.

At Iron Mountain, New Mexico, deposits containing danalite, helvite and garnets were subjected to examination because it was thought that their beryllium content might be sufficiently high to justify mining them. (See Glass, J., Jahns, R. H. and Stevens, R. E., 1944, pp. 163-191.) During the examination it was found difficult to judge a sample of ore as to its beryllium content because of the close resemblance between small grains of the beryllium species and the accompanying garnets. In order to overcome this difficulty, Gruner developed the following methods of staining the beryllium species which depend on the development of yellow coatings of sulphide of arsenic and red coatings of antimony sulphide as the result of reactions between the appropriate cations and the sulphide ions of the species under discussion.

Place a very small representative sample of the powdered or crushed rock

in a 50 ml. beaker and add sufficient dilute sulphuric acid to cover it. Add a pinch of arsenious oxide and boil the solution for 1 or 2 minutes. Decant and wash the sample twice with water. Examine the contents in the beaker—still covered by a little water—under the microscope. Grains of any of the beryllium species noted appear brilliant canary-yellow.

Even when the gangue is essentially yellow garnet the stained fragments are easily distinguished. However, to simplify recognition under such conditions a little *metallic* antimony should be substituted for arsenious oxide in the above test: this will cause grains of the beryllium mineral species to be stained a brilliant red.

It is claimed that by employing either of the above procedures that a single grain of helvite or danalite can be detected in the midst of thousands of gangue minerals. The writer has confirmed that this claim is correct.

The application of the sodium azide/iodine test to the identification of grains of danalite and related beryllium species when they are associated with garnets has been noted earlier in this paper.

Arsenopyrite

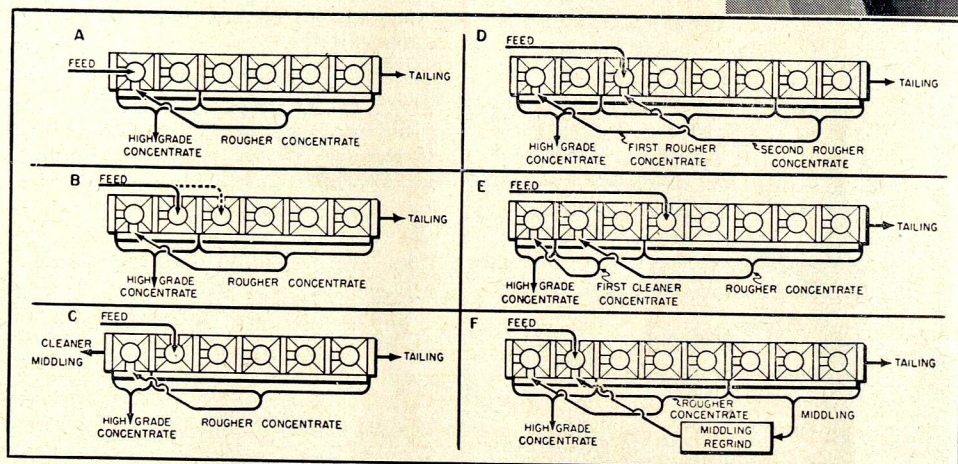
It is often difficult to distinguish between small grains of pyrite and arsenopyrite when a mixture of these is examined under the microscope. Raffinot (1953, p. 6) suggests using the following differential staining method in order to overcome this difficulty:—

Place a few grams of the sample in a small beaker and cover them with a 3 per cent solution of potassium ferrocyanide in 5 per cent hydrochloric acid. Bring the solution just to the boil, then decant and wash the grains with water. Arsenopyrite grains are stained a vivid blue by this treatment, whereas pyrite grains are unaffected.

The writer has shown that chalcopyrite and stannite grains are also unaffected by the above treatment, whereas loellingite grains are stained blue and, therefore, cannot be distinguished from arsenopyrite grains in mixtures by this method. The loellingite which was tested was obtained from the Castle-an-Dinas mine (Cornwall), and although it stained in the same manner as arsenopyrite, it was found that the test could be used to differentiate between *pure* samples of each of the two species for the following reason: Whenever arsenopyrite is warmed with ferrocyanide-HCl reagent, *strong* blue "streamers" ascend through the solution from the

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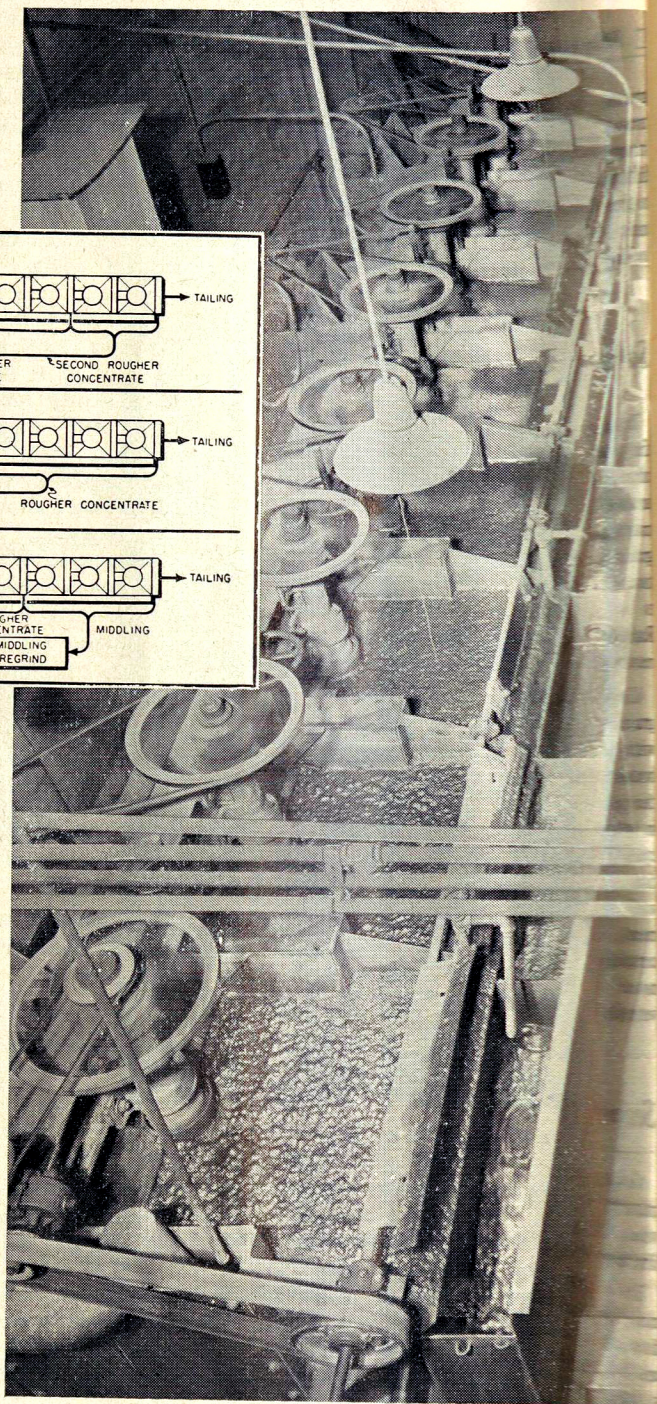


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Mineral Grains

grains, but when loellingite is similarly treated the "streamers" are much less strongly developed or non-existent. This difference in behaviour is obviously due to the fact that arsenopyrite is generally more soluble in the reagent than is loellingite.

Sphalerite

It has been noted earlier that grains of the paler varieties of sphalerite are often difficult to recognise when they are associated with grains of pale gangue minerals. A method of staining sphalerite grains in order to facilitate their recognition has also been noted, but in addition to it the following method due to Raffinot (1953, p. 5) may well be worth recording :

Convert the surfaces of the sphalerite grains to zinc carbonate by treatment with sodium hypochlorite-sodium carbonate solution, as described elsewhere in this paper. Wash the treated grains well in water and place them in cold ferricyanide-oxalic acid-diethylaniline solution. After a few minutes decant and wash the grains in water. By this treatment, sphalerite grains—and grains of certain secondary zinc minerals which are noted below—are stained orange to vermillion.

Secondary Zinc Minerals

Grains of many of the secondary zinc minerals are often difficult to recognise when they are associated with grains of similarly coloured gangue minerals.

To simplify the study of products made during experimental work on the flotation of smithsonite, hydrozincite, and hemimorphite, Raffinot (1953, pp.3-5) has developed Methods (i) and (ii) below for staining these three zinc species. Methods (iii) and (iv)—which are due to the writer—are satisfactory alternatives to the methods that have been proposed by Raffinot.

Dithizone Method—Grains of the three zinc species already noted are stained carmine by treatment with dithizone in ammoniacal solution. Secondary lead minerals, however, react similarly, and if they are present the following procedure should be adopted :

Immerse the sample in 10 per cent sodium sulphide solution for 5 minutes, then decant and wash the grains well with water. (This treatment converts the superficial portions of the grains of secondary lead minerals to black, lead

sulphide which will not react with dithizone under conditions of the test.) Warm the sample for 5 minutes at 50 deg. C. with a 1:1 mixture of a 0.2 per cent solution of dithizone in carbon tetrachloride and 2.0 per cent ammonium hydroxide. Finally decant the solution and then wash the sample with water and examine it under the microscope.

Ferricyanide/Oxalic Acid/Diethylaniline Method

This method of staining certain secondary zinc minerals depends on the following facts which were first investigated by E. Eegriwe (1928, p. 228):

Alkali ferricyanide oxidises diethylaniline to a coloured compound and at the same time ferrocyanide ions are formed. This oxidation proceeds slowly and incompletely unless the ferrocyanide ions are removed as they are formed.

If zinc ions are present ferrocyanide ions are removed because of the formation of sparingly-soluble, white zinc ferrocyanide. The coloured oxidation product is adsorbed on the white zinc ferrocyanide. Obviously, appreciable quantities of cations which form coloured, insoluble ferrocyanides will interfere.

In order to stain grains of the three zinc species under review, Raffinot places a portion of the sample in a cold solution composed of equal volumes of 3 per cent potassium ferricyanide and 3 per cent oxalic acid containing 0.5 per cent diethylaniline. After 3 minutes the solution is decanted and the grains are washed with water. This treatment causes grains of smithsonite, hydrozincite and hemimorphite to be stained reddish-orange.

The present writer has established that grains of the following species are stained orange to red by precisely the same reaction: Descloizite, hopeite, para-hopeite, tarbuttite, and willemite. The following species which sometimes occur in zinc deposits do not react: Barite, celestite, witherite, strontianite, fluorite, quartz, cerussite and anglesite.

Ammonium Mercuric Thiocyanate-Copper Sulphate Method—Zinc and copper ions together react with mercuric thiocyanate ions forming a purple insoluble compound. This reaction may be used as the basis of a method for staining grains of smithsonite, hydrozincite and hemimorphite, and probably grains of other secondary zinc minerals.

Place a few grams of the sample to be examined in a small beaker and add about 1 ml. of 0.1 per cent copper sulphate, 6 ml. of ammonium mercuric thiocyanate and 3 or 4 drops of 5N. HCl. Heat the solution to 65 deg. C. and after 5 minutes remove the beaker

from the source of heat. Tumble the grains gently in the warm solution for a further 30 seconds, then decant and wash the sample lightly with water.

This treatment causes grains of the three zinc species noted, to be stained pinkish-purple. The ammonium mercuric thiocyanate is prepared by dissolving 8 g. of mercuric chloride and 9 g. of ammonium thiocyanate in a 100 ml. of water.

Acridine Hydrochloride Method—An alcoholic solution of acridine hydrochloride reacts with zinc ions in the presence of potassium thiocyanate forming a yellow-green complex. Iron gives a red and cobalt a green product under the same conditions. [The most complete account of reactions involving the use of acridine hydrochloride are due to Welcher (1947, vol. 3, pp. 118-120).] Under certain conditions this reaction may be employed to stain grains of smithsonite, hydrozincite and hemimorphite, and the most satisfactory procedure is as follows:

Place a few grams of the sample in a small beaker and add about 5 ml. of 10 per cent potassium thiocyanate, 4 ml. of a 1 per cent solution of acridine hydrochloride in alcohol and 2 ml. of 2 per cent HCl. Heat the solution to about 70 deg. C. and maintain this temperature for 4 minutes.

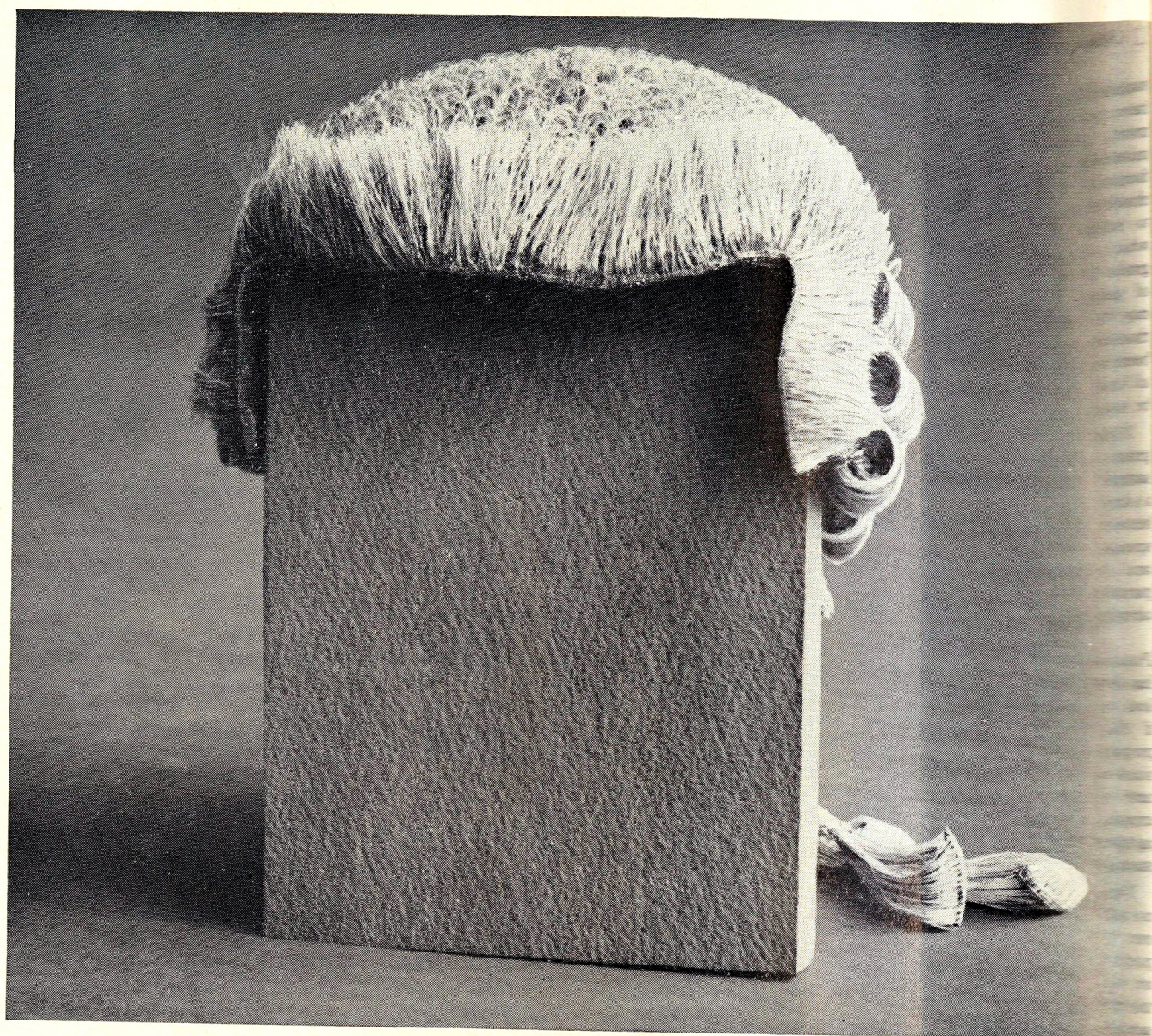
At the end of this stage it is necessary to remove the beaker from the source of heat, decant and wash the grains with water. Grains of the three zinc species noted are stained greenish-yellow by this treatment.

Lead Minerals

Sodium Rhodizonate Method — Feigl (1947, p. 429) notes that lead can be detected in minerals—in the absence of barium species—by spotting a little of the finely-powdered sample first with a drop of buffer solution and then with a drop of sodium rhodizonate solution.

If lead is present this treatment causes the mineral particles to become red. The same writer also states that certain lead species—for example, galena and stolzite—react more positively to the above test if they are first calcined.

The present writer has examined a wide range of secondary lead minerals and has found that the majority of them stain beautifully if they are immersed in a freshly-prepared 0.2 per cent solution of sodium rhodizonate and then, after 30 seconds, sufficient buffer solution is added to discharge the colour of the solution. However, stolzite, raspite, wulfenite and some varieties of pyromorphite and mimetite are only adequately



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stained by the method described if they have previously been calcined. Furthermore, in order to stain grains of lead minerals with a metallic lustre satisfactorily by employing the sodium rhodizonate reaction it is necessary first to subject them to a short calcination which should be stopped as soon as the grains become dull. The fact that grains of galena, jamesonite and bournonite may be stained by employing the sodium rhodizonate method after calcination has been noted earlier and it is probable that all lead species with a metallic lustre can be stained by similar treatment.

This method thus not only enables grains of lead minerals in a composite sample to be detected, but also serves as a simple means of differentiating rapidly between grains of pairs of such similar species as bournonite and tetrahedrite, and jamesonite and stibnite. The preparation of the buffer solution has already been noted.

Potassium Iodide Method—The writer has found that grains of most secondary lead minerals are stained brilliant yellow if they are immersed in a freshly-prepared, fairly strong solution of potassium iodide and 1 : 7 nitric acid is added until the volume of the solution is increased by about a quarter. Little or no agitation of the grains is necessary, and staining is effected almost immediately.

The yellow lead iodide coating, whilst not adhering as firmly as the rhodizonate one, is nevertheless adequate for most purposes. Stolzite, raspite and wulfenite are not stained to any marked extent by this procedure, and the staining of certain samples of pyromorphite and mimetite is greatly enhanced if, on adding the potassium iodide, the whole is warmed somewhat before the addition of the nitric acid.

This method of staining has been applied directly to concentrates on a vanning shovel in order to make an estimate of the amount of cerussite present.

Locating Sulphide and Secondary Lead Mineral Grains—Having applied sodium azide/iodine reagent to a sample in order to locate sulphide grains, the further addition of a little 1 : 7 nitric acid causes cerussite (and doubtless certain other secondary lead minerals) to stain yellow. The stained grains are best seen after the treated sample is washed lightly with water in order to remove the staining reagents.

Raffinot's Method of Staining Cerussite (1953, p. 3)—Raffinot notes that grains of cerussite and of anglesite may be stained black by treating them with a solution of sodium sulphide, but remarks that this is

not always a satisfactory aid to identification as it is not always easy to distinguish between such stained grains and grains of galena. As briefly noted earlier in this section, he recommends staining cerussite by warming the sample for 5 minutes at 70 deg. C. with a solution containing 0.1 per cent of dithizone and 2.0 per cent of sodium cyanide. This treatment causes cerussite grains to become red. Anglesite is not stained effectively by this method as it is too soluble. The addition of the sodium cyanide prevents any secondary zinc minerals which may be present from being stained.

Cerussite and Anglesite

Head and Crawford (Rep. Div. U.S. Bur. Min. 2932) recommend the following methods of staining cerussite and/or anglesite grains in order to distinguish them easily in crushed samples:—

(a) To produce a bright yellow coating of lead chromate of equal intensity on both cerussite and anglesite grains, treat the sample with a cold saturated solution of potassium dichromate for 15 minutes.

(b) To stain anglesite yellow, but not cerussite, treat the sample for one minute with a cold aqueous solution containing 2 per cent potassium dichromate and 0.5 per cent sodium hydroxide.

(c) To stain cerussite yellow, but not anglesite, immerse the sample in a cold 1 per cent solution of CrO_3 for one minute. (If this solution is applied for a greater length of time than that stated, or if a stronger solution than the one recommended is used, some or all of any galena present will be decomposed. If the test is conducted as suggested above, only an orange tarnish develops on the galena.)

The originators of the above tests suggest that the staining of a given sample is best carried out on a watch glass and that the grains should be tumbled during the whole period in which they are immersed in the solution.

Copper Minerals

Chalcocite—Grains of chalcocite—unlike those of similar minerals—are readily stained blue if they are immersed for 2 minutes in a cold 20 per cent solution of ferric chloride. A slight amount of warming tends to increase the intensity of colour.

Chrysocolla and Malachite—Grains of certain varieties of chrysocolla are not

unlike grains of malachite, but whereas the specific gravity of the former mineral varies from 2 to 2.2, that of the latter varies from 3.9 to 4. Therefore, if these are the only green species in the sample, and if they do not occur in composite grains, they may be readily separated by employing bromoform. However, if a considerable quantity of composite grains occur this method is of no value. Under these conditions estimations are greatly simplified if one of the two species under discussion is stained a colour other than green. Often it is possible to differentiate between grains of chrysocolla and of malachite by the following staining method:

Immerse a few grams of the sample in warm 5N. acetic acid and after 30 seconds add sufficient warm 10 per cent sodium xanthate solution to treble the volume. Tumble the grains for a minute, then decant and wash the residue with water. Malachite grains are stained yellow by this treatment, whilst grains of chrysocolla are *usually* found to be unaffected.

The reactivity of different varieties of chrysocolla to these reagents is variable, and therefore it is wise to conduct appropriate preliminary tests on the chrysocolla of a given mine before the above method is applied to the examination of the mill products. A pale blue chrysocolla which was examined by the writer was so reactive that when fragments of it were tumbled for about 2 minutes in cold 10 per cent sodium xanthate solution they assumed a brilliant yellow coating. When malachite grains were similarly treated they were unaffected.

Stibnite

It is not easy to differentiate readily between grains of stibnite and jamesonite by inspection under the microscope, but it is desirable to do so as they often occur in the same orebody. This differentiation may be simplified, either by utilizing the sodium rhodizonate reaction to stain the jamesonite (as described earlier), or by staining the stibnite yellow by placing a few grams of the sample in 40 per cent potassium hydroxide for 5 minutes. This treatment also causes any kermesite grains which may be present to be stained yellow. For examination under the microscope, the solution should be decanted and the grains washed lightly in water. This method of staining stibnite (and kermesite) is based on an etch reaction described by Short (1940, p. 114).

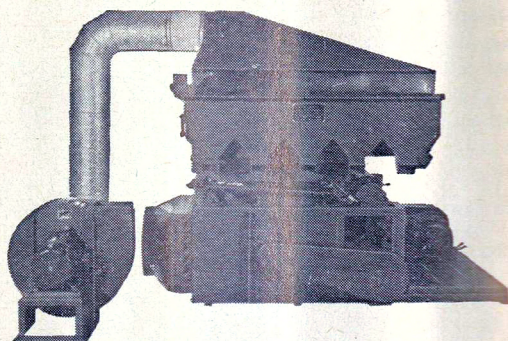
Calaverite $2(\text{Au Te}_2)$ —Grains of tellurides of the precious metals are not

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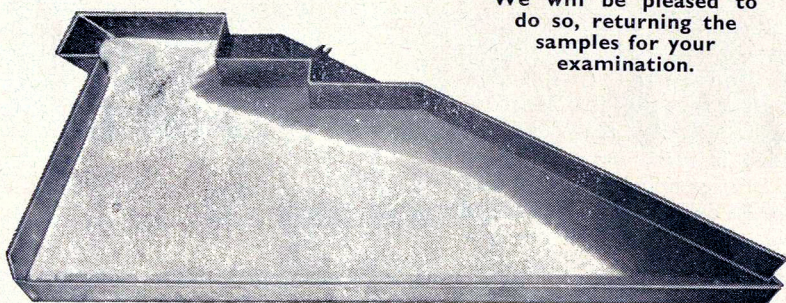
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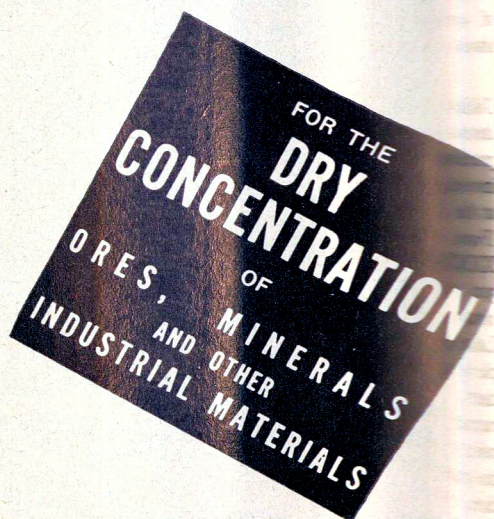


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SCHEELITE
WOLFRAMITE
ILMENITE
BAUXITE
MICA

GALENA
URANIUM
VANADIUM
FELDSPAR
LIME
PYRITE
BARITE

COLUMBITE
MAGNETITE
HEMATITE
LIMONITE
FLUORSPAR
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readily identified under the microscope when they are associated with grains of the common sulphide minerals. The following method of staining grains of calaverite—which could probably be used for staining other tellurides—assists in their recognition:

Immerse a few grams of the sample in 1:1 nitric acid. Bring the acid just to the boiling point, then decant and wash the grains with water. (This treatment causes calaverite to assume a pinchbeck-brown tarnish, whilst pyrrhotite and chalcopryrite display iridescent tarnishes. Gold is unaltered, and pyrite shows little or no tarnish.) Cover the grains with stannous chloride-HCl reagent and warm gently for about 3 minutes, then decant and wash the grains lightly. This treatment causes the calaverite grains to become dull black and tends to remove the tarnish developed on grains of certain species noted above as a result of the preliminary acid treatment.

The stannous chloride-HCl reagent should be prepared immediately before use by boiling 4 or 5 g. of tin with 15 ml. of concentrated hydrochloric acid for about 5 minutes.

Tellurides, etc.—Haultain and Johnston devised the following method to identify precious metal tellurides (see "Milling investigations into the ore as occurring at the Lake Shore Mine". By the Staff. *Trans. Canad. Inst. Min. Metall.*, 1936, XXXIX, 434.) in extremely fine (minus-300 mesh) super-panner concentrates obtained during experimental work on Lake Shore material: A little of the sample is sprinkled on a Pyrex slide which is then heated in the flame of a bunsen burner until the glass is red and on the point of bending. At this temperature the particles adhere to the slide which is then turned over and the grains are heated, for a further few minutes, directly in the flame. This treatment causes each grain of precious metal telluride to be converted to a globule of gold or silver surrounded by a coloured halo which is easily recognised when the cooled slide is examined under the microscope.

Haultain and Johnston (1933) also observed that this treatment facilitated the identification of certain other species. Thus, pyrite becomes a characteristic red and chalcopryrite a characteristic grey, pentlandite sprouts hair-like filaments and exudes globules.

Columbite—Flinter (1955) states that the identification of grains of Malayan columbite is facilitated by subjecting the sample to a preliminary treatment with boiling hydrochloric acid. If the treatment is continued for about 15 minutes hematite and magnetite dissolve

whilst ilmenite either assumes a grey or white coating, or becomes dull. Wolframite grains are partly coated with a canary-yellow product. When the sample is boiled for an hour with the acid, iron-rich magnetic varieties of rutile (derived from ilmenite) react in the same way as ilmenite. Normal rutile, cassiterite, tourmaline, allanite, betafite and columbite are unaltered by this treatment.

Conclusion

In writing this paper it has been regarded as axiomatic that if each grain of a given species in a composite sample of loose grains can be easily recognized "close" mill-control is greatly facilitated. The writer, however, feels certain

that many of the aids to grain identification are not widely known nor has their potential been generally appreciated. This paper has been written primarily with a view to demonstrating the value of these simple aids. It is also hoped that this paper will stimulate research into the development of further simple chemical—and other—tests which will assist the mineral-dresser and thus tend to increase the efficiency of the mill.

Acknowledgements

Of necessity, the preparation of this paper has involved the abundant use of the findings of other workers, but in particular the author wishes to pay tribute to the works of Gaudin and Raffinot from which he has culled a wealth of information.

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Rapid Identification *of*

MINERAL GRAINS IN COMPOSITE SAMPLES

K. F. G. HOSKING, M.Sc., Ph.D., A.M.I.M.M.

Abstract

It is stressed that rapid and simple aids to the identification of all the grains of a given mineral species in a composite sample which do not involve preliminary embedding and polishing can often facilitate "close" mill control.

Suitable chemical methods are reviewed and particular prominence is given to differential staining techniques.

Finally, a compilation of those rapid chemical methods of identifying grains of certain specific minerals which are known to the writer, but which have not been described in the body of the paper, is included so that they are readily available to those most likely to find them of use.

Introduction

For "close" mill-control the ideal analytical methods are those which can be employed in the mill itself—preferably by personnel who are not highly trained physicists, chemists or mineralogists—and which will enable data of a sufficient degree of accuracy to be obtained in a comparatively short space of time. Undoubtedly, before long, rapid physical methods will be generally employed in many mills to provide urgent analytical data, and the development, at Boliden, of a tape method of spectrochemical analysis for this specific purpose (Danielsson, 1957) is an important indication of this trend. This trend is further emphasized by the fact that the Japanese have constructed a robust polarigraph, primarily for "on-the-spot" determinations of copper, zinc, etc., in mill products (Mino, 1955), and the recent announcement that the Canadians are manufacturing a berylometer, by means of which beryllium can be quickly determined in all manner of material. The use of the Geiger counter to facilitate the treatment of radioactive ores is an example of the employment of a physical method of analysis well known to all.

Nevertheless, particularly in the operation of pilot plant and other small mills, those simple aids to "close" control which do not require the use of expensive apparatus will continue to prove of value.

Many simple physical and chemical methods exist for separating the various components of a composite sample and for enabling the grains of certain specific minerals to be made so distinctive that quantitative evaluation becomes comparatively quick and easy. Still, these aids are not used

by any means as generally as might be expected, possibly this is because much of the relevant information is widely scattered in the literature and not, therefore, readily available to those to whom it could be of the utmost use.

In this paper the writer has confined himself to reviewing one aspect of the subject, namely those rapid chemical methods by means of which all the grains of a given mineral in a composite sample can be quickly recognized without resorting to preliminary embedding and polishing.

Methods

For convenience, the rapid chemical aids to mineral-grain identification are discussed under the following headings: Insufflating and allied techniques; Staining mineral grains; Grain identification by gas attack, and Identification by fusion tests.

Insufflating Techniques

Williams and Nakhla (1950-51, pp. 282-284) have designed a small metal spraying apparatus, operated by compressed air, by means of which a true sample of fine mineral particles can be caused to adhere to reagent-impregnated gelatin-coated paper. The impregnating reagents used are such that they will impart a distinctive colour to all the grains of a given species. The technique is of somewhat restricted use because it can only be employed to identify those species which can be stained at room temperature by reagents of moderate concentration. Furthermore, the only additional treatment which can be safely applied to the paper after the sample has been blown on to it is to subject it to a gas (Obviously it can neither be sprayed with liquids nor immersed in a bath of reagent as such treatments might cause some of the particles to become detached.)

The apparatus, being robust, could be used in a mill and as a true sample attaches itself to the paper, with experience, the degree of variation in the quantity of a given substance present in a series of samples could be determined simply by inspection, with or without the added assistance of a microscope.

The following related technique is useful for comparing the amounts of certain species in a series of samples of grains within, say, the 40 to 80 mesh range. The method depends on taking prints, on spot-reaction paper, of a series of samples each of the same volume, and composed of loose grains.

and then comparing the prints. It has proved particularly useful to the writer for comparing the amounts of fluorite present in various mill-products consisting essentially of fluorite, quartz, calcite, galena and barite, and the following description of the method will serve to indicate the manner in which the technique may be used as an aid to the solution of similar problems. The test is based on the fact that calcium fluoride reacts with a hydrochloric acid solution of red-violet zirconium-alizarin reagent with the formation of colourless ZrF_6^- and Ca^{++} ions and yellow alizarin (Feigl, F., 1947, pp. 202-204). Thus, if a grain of fluorite rests for a short space of time on a piece of spot-reaction paper impregnated with the above reagent and heated with steam, on its removal the spot on which it rested is pale-yellow, whilst the surrounding paper remains pink.

In practice, a circle of 1.5-ins. dia. is drawn on a piece of spot-reaction paper and successive drops of reagent are placed in the centre until the whole of the circle, as a result of diffusion, is pink. A measured volume of grains is scattered as evenly as possible over the coloured zone and the paper is then placed on the top of a small beaker containing some gently-boiling water. After a minute the paper is removed and tapped to remove the grains. The size and number of pale yellow to colourless spots on the pink background gives some idea of the quantity of fluorite in the sample, and by comparing prints made from different samples, all but small variations in fluorite content are immediately recognisable.

Under the conditions of the test fluor-apatite and cryolite react in the same manner as fluorite. If the products have been subjected to flotation it is necessary to know what reagents have been used, as sodium fluoride is sometimes used to depress barite. Tests indicate that barite grains which have been depressed by the addition of 1 lb. of sodium fluoride per ton of ore all react positively to the above test. The reagent is prepared by dissolving 0.05 g. of zirconium nitrate in 50 ml. of water and 10 ml. of concentrated hydrochloric acid, and mixing it with a solution of 0.05 g. of sodium alizarin sulphonate in 50 ml. of water.

Straining Mineral Grains

If grains of a given mineral species are stained so that their colour is markedly different from those of associated grains of other species, an estimate of the quantity of the stained mineral present in the sample can be made rapidly by inspection, or a more accurate assessment can be obtained by the slower process of counting grains under the microscope. Staining is not only used to differentiate between species which appear essentially the same, but also to enhance the differences in appearance between species which can, without treatment, be differentiated by *careful* inspection.

It is worth mentioning that differentiation between the grains of similar transparent minerals can often be facilitated by immersing them in a liquid whose refractive index is such that the grains of one species are almost invisible.

Grains of a given species of transparent mineral may, on occasion, be made readily recognizable in a composite sample by subjecting them to the relatively neglected dispersion staining. The technique depends on the fact that "if particles of a transparent solid are placed in a liquid having the same refractive index as the solid for one wave length of light but a different refractive index for other

wave lengths, the colour for which the refractive index of the liquid and solid match will be transmitted without deviation, but other wave lengths will be deviated. When a microscope is adjusted for dark-field illumination, only the deviated colours reach the eye." (Crossman, 1948.) By immersing a mixture containing corundum, quartz, mica and glass in methylene iodide ($n_{D^{25}} 1.74$ at 25 deg. C.) and employing the above method of observation, the corundum grains appear yellow whilst the other components are white. Under similar circumstances if quartz is immersed in nitrobenzene it appears a brilliant blue. (McCrone, 1955, p. 134.)

That ultraviolet light is of considerable value in identifying untreated grains of certain minerals (e.g., scheelite, zircon, monazite, etc.) is well known, but the fact that visible light from high-energy incandescent lamps often causes minerals to emit infra-red luminescence which may, by being of diagnostic value, have some application in the mill, has not yet been generally appreciated. (See Barnes, D. F. Infra-red luminescence of minerals. U.S. Geological Survey Bulletin 1052-C.)

Usually the alteration of the particular grains is such that they can be readily distinguished in ordinary light, but the development of a superficial deposit which fluoresces characteristically under ultra-violet light may also be employed. The staining of grains of a given species may be effected by heat-treatment alone or followed by immersion in certain reagents, or by direct treatment with one or more reagents. The change, as a result of the treatment, may be due to conversion of much or all of the mineral to opaque oxide; to superficial reduction to metal, etc.; to superficial oxidation, with the resultant development of a transparent film displaying interference colours; to the development of a superficial compound composed of some of the components of the mineral and of the staining reagent, and to dye adsorption. In addition to the above staining methods, leaching may, on occasion, facilitate differentiation between grains of two similar species. Thus if a mixture composed of reddish-brown grains of Nigerian thorite and zircon are boiled with hydrochloric acid the thorite is converted to a fragile, white, silica shell which is readily distinguishable from the glassy, white, bleached zircon. (See Macleod, W.N. and Jones, M. P., 1955, pp. 168-169.)

Opaque Coating

The alteration of grains of a given species in such a manner that their colour is markedly different from grains of associated minerals under ordinary light and at room temperature is an ideal procedure because the grains can be identified without using a dark-room or any special physical apparatus other than a binocular microscope. The time required to stain fragments by the various methods discussed below is, of course, variable, but never great.

Staining by Calcination

It is a well-known fact that when certain minerals are heated in air they suffer an alteration in colour which is persistent at room-temperature. This change is either due to certain imperfectly understood reorganizations of the components or to oxidation. It is common knowledge that by heat-treatment, which results in reorganization within the crystal, brown Brazilian topaz becomes pink, pink Malayan zircon becomes colourless, and pale green beryl assumes a colour similar to that of an aquamarine. Clearly these particular facts are unlikely to be of widespread value to the

mineral-dresser, although it is the practice of Chinese tin-dressers in Malaya to roast certain concentrates in order to differentiate visually between fine zircon and cassiterite. However, another alteration of this type which may be used to advantage in the mill concerns cassiterite. All varieties of cassiterite yet examined by the writer change colour permanently on being heated for about 30 minutes at temperatures fairly near 1,000 deg. C. and, as is well known, this treatment also results in magnetic cassiterite becoming non-magnetic. Dark varieties of cassiterite become vermilion to orange and their transparency increases: even opaque white cassiterite which is slightly veined by knife-edge red veins (of hematite?) becomes pale pink.

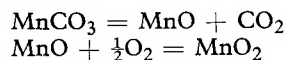
Several Nigerian mining companies have considered heating mill-products consisting essentially of columbite and magnetic cassiterite in order to destroy the magnetic properties of the latter and thus enable them to separate the two species by magnetic means. If this method is ever employed, the colour of the heat-treated cassiterite will greatly facilitate estimations of it in the columbite fraction as the colour of the latter is unaffected by the heat-treatment. The colour change is of added value because the tinning test yields negative tests when applied to thoroughly heat-treated Nigerian cassiterite.

Value of Heat Treatment

It is worth noting that the physical properties of the species are so modified by heat-treatment that their certain subsequent separation from associated minerals is considerably facilitated. Thus, for example, if "normal" spodumene (S.G., 3.13-3.20) is heated for 30 minutes at about 1,050 deg. C. it is converted to the β variety (S.G., 2.4) which will float in bromoform (S.G. c. 2.8). Clearly, then, this fact may be made the basis of a method of separation and evaluation of spodumene in composite samples which do not contain readily fusible components. It may also be noted that if β spodumene is boiled for 5 minutes with a 1.2 per cent aqueous solution of Methylene Blue, and subsequently washed well and examined wet, it appears dark blue whilst the minerals with which it is normally associated are pale blue or colourless.

The development of a characteristic oxide layer by the action of heat may be made use of to differentiate between particles of rhodochrosite and rhodonite in a mixture of these pink minerals. By subjecting the mixture to a low-temperature roast, rhodonite is unaltered, but rhodochrosite is changed super-

ficially to black dioxide in accordance with the following equations (Gaudin, 1935, p. 556):



This is the only useful example of this type known to the writer but it may well be that others exist.

In conclusion it must be pointed out that by employing heat-treatment to enable one species to be identified, the diagnostic features of associated species might be destroyed. Thus, heat-treatment of Franklin Furnace (New Jersey) products, in order to confirm the presence of rhodochrosite, would convert any yellow willemite present—which is readily detected because of its strong yellowish-green fluorescence under ultra-violet light—into a dull-grey, non-fluorescent substance.

Staining by reduction

Grains of cassiterite which have not been strongly heated can almost invariably be superficially reduced, by treatment with zinc and dilute hydrochloric acid, to gray, metallic tin which greatly facilitates their recognition. Before conducting the test, sulphides, etc., should be decomposed by boiling with aqua regia, and it is often necessary to subject roasted samples and samples from the zone of oxidation to preliminary acid clearing in order to expose the whole of the cassiterite to the reducing agents. However, even "clean" cassiterite, which has not been subjected to heat-treatment, does not invariably react positively to the zinc/hydrochloric acid test: this was noted by Beringer (1931, p. 220), and in recent years cassiterite with this unusual property has been found in Nigeria. Furthermore, as stated earlier, thoroughly heat-treated Nigerian magnetic cassiterite will not "tin" when subjected to the above test, and preliminary treatment with a number of chemicals has failed to restore this reducing property. However, certain Cornish cassiterites, when subjected to the same heat-treatment as that given to the Nigerian variety will "tin", though slowly: it thus appears that the ease with which heat-treated cassiterite can be tinned is not only a function (within limits) of the duration of the roast and the temperature, but also of the physical and/or chemical characteristics of the cassiterite tested.

Up to this point only reduction to metal has been considered, but less complete reductions may be employed to develop a characteristic coating on some minerals, notably certain tungstates. Thus, in order to facilitate the recognition under the microscope of scheelite in a mixture composed, for example, of grains of scheelite, quartz and feldspar, the tungstate fragments may be superficially reduced to a blue tungsten

compound. This reduction is effected by warming the sample for a few minutes in a strong solution of stannous chloride in hydrochloric acid. After this treatment the sample is washed slightly with water and examined wet. The blue stain disappears in an hour or so, but this does not constitute a serious drawback as the grains can be quickly restained, if necessary. This treatment also causes tungstic ochre, and all tungstates, except those of the ferberite-hubnerite series, to be stained blue. On occasion certain secondary molybdenum-bearing species may also stain blue or greenish blue.

Staining by precipitation

A variety of methods may be employed for developing a precipitate of diagnostic value on mineral grains. The coating most resorted to are the products of simple chemical reactions, either directly between a reagent and the mineral grains or between a reagent and a suitably reactive coating previously developed on the grains. It is reasonable to believe that coatings of diagnostic value may be developed for a wide variety of minerals by these means.

Although the development of a satisfactory stain for a given species depends to some extent, on trial and error, method of doing this can often be found reasonably rapidly if the following factors are borne in mind:—

- (i) The staining solution must be such that the mineral is only slightly soluble in it. In other words, the mineral must be so attacked that there is a slow emission of ions, which at the moment of liberation from the grain react with a component of the staining solution forming a precipitate at the surface which adheres to the surface.
- (ii) To ensure that the precipitate adheres to the surface, the rate of dissolution of the mineral must be slower than the rate of precipitation of the coloured coating.

These two points indicate that attention must be paid to the pH of the staining solution, the concentration of the attacking reagents, the temperature of the solution during the reaction and possibly the degree of agitation.

- (iii) Ideally, staining of the mineral should be effected without the evolution of gas, as the latter tends to break and displace the film at the surfaces of grains.

Because many sulphides can only be attacked by acids, which result in the liberation of appreciable quantities of hydrogen sulphide, it is necessary either to convert their superficial portions to sulphate, oxide or carbonate—which can often be stained by employing reactions not involving the evolution of gas—or to resort to the development of a

a manner described later—of transparent coatings displaying diagnostic interference colours. Thus, the most convenient way of staining galena grains, in order to distinguish readily between them and grains of somewhat similar lead-free minerals, is to proceed as follows: Roast the sample on the lid of an iron crucible until the grains become dull. (This converts the superficial portions of the galena to lead sulphate.) Transfer the sample to a watch-glass and cover it with 0.2 per cent. Sodium rhodizonate solution. Swirl the grains for a few seconds and then add sufficient buffer solution to discharge the colour of the solution. On examination under the microscope the galena grains appear reddish-purple. The buffer solution is prepared by dissolving 1.5 g. of tartaric acid and 1.9 g. of sodium bitartrate in a 100 ml. of water. (For further information concerning the chemistry of this test see Feigl, F. and Suter, H. A., 1942, p. 840 and Feigl, F. and Braile, N., 1943, p. 52.)

On the other hand, witherite grains can be stained pink by warming them for 3 or 4 minutes at about 60 deg. C. with a saturated solution of potassium permanganate made faintly acid with H_2SO_4 , then decanting and warming the grains with a strong solution of oxalic acid in order to destroy any permanganate which may be attached to certain other species present such as quartz, fluorite and barite.

This method of staining witherite—which was developed by the writer—depends on the fact that when barium sulphate is precipitated in the presence of potassium permanganate, the latter is strongly held by the sulphate and resists destruction by oxalic acid. See Feigl, F., 1947, p. 238.)

(iv) The coating must be insoluble.

(v) The reaction employed to develop the coating must be a fairly sensitive one.

(vi) The reagent chosen to produce the coloured coating must be such that under the conditions of the test it is either specific for a given mineral species or radical, or reacts in a distinctive manner with each of several radicals.

Thus, dithizone may be employed to stain both cerussite and smithsonite red. (Described in detail later.)

The writer found that by shaking certain mill-products from North Portugal with 5 per cent potassium ferrocyanide, slightly acidified with HCl, that he could differentiate rapidly between grains of molybdc ochre and those composed of bismuth ochre or tungstic ochre as the former stained brown, whilst the latter two stained blue on account of the readily soluble iron "impurity" in them.

(vii) By employing differences in reactivity towards a given solution,

species containing a common ion may often be differentially stained. Thus, if a sample of grains of witherite and barite are boiled for about 5 minutes with N. potassium chromate, only the witherite grains are stained yellow.

Suitably Reactive Coatings

Because it is not possible to attack certain minerals by common reagents without the evolution of gas—which, as stated above, is objectionable during staining—and because certain minerals are not reactive towards the available staining reagents, it is necessary as a preliminary to the staining of such types to develop a suitably reactive coating on them. Such coatings are most likely to be obtained by oxidation or double decomposition. Boiling with appropriate solutions is often sufficient to so modify the surface of the grain that it can be stained, but on occasion appropriate surface alteration can only be achieved by immersing the sample for a short while in a melt. The following examples illustrate the application of these processes to this problem.

Oxidation — Sulphides

By subjecting sulphides to a short calcination their superficial portions are usually converted to sulphate and/or oxide which often prove to be suitable media for staining. As noted above, it is necessary to calcine galena before staining it with sodium rhodizonate, and bournonite and jamesonite must be subjected to the same preliminary treatment if they are to be stained by the same reagent.

Oxidation — Carbonates and Hydroxides

As a result of roasting, carbonates and hydroxides are usually converted to oxides which may often be more readily stained than the original material. Thus, grains of brucite and/or hydromagnesite may be easily detected when admixed with grains of calcite by subjecting the sample to a short roast and then placing the cooled grains in a solution of silver nitrate. Brucite and hydromagnesite grains become coated with a brown to black film of silver hydroxide, whilst the calcite grains remain uncoloured. (Holmes, A., 1930, p.273.)

The presence of smithsonite in a sample of grains, which also contains quartz, barite and calcite, may be determined by roasting the sample on a square of asbestos paper. By this treatment smithsonite is converted superficially to zinc oxide, which is canary-yellow when hot. This colour is sufficiently marked to enable an estimate of the amount of smithsonite present to be made, but in

order to facilitate the counting of the smithsonite grains under the microscope it is necessary to confer a colour to the mineral which is apparent when the sample is cold. This may be done by damping the roasted sample with cobalt nitrate and then heating it strongly. On cooling, the smithsonite grains are bright green.

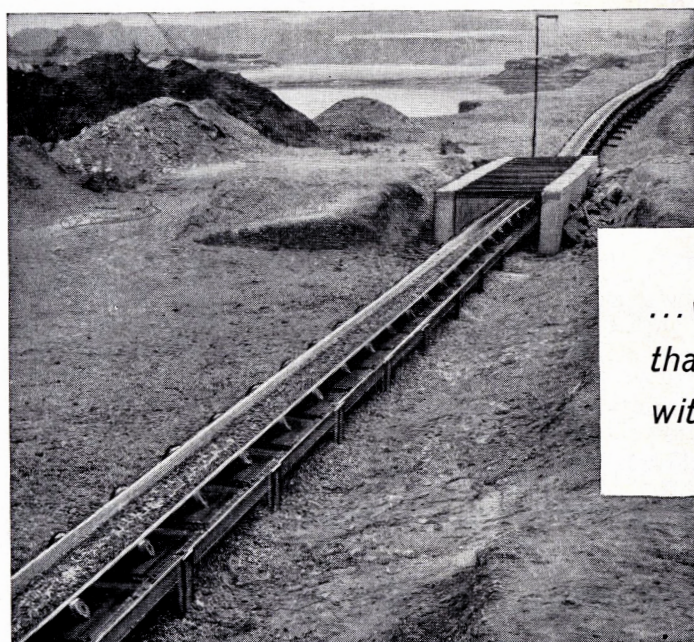
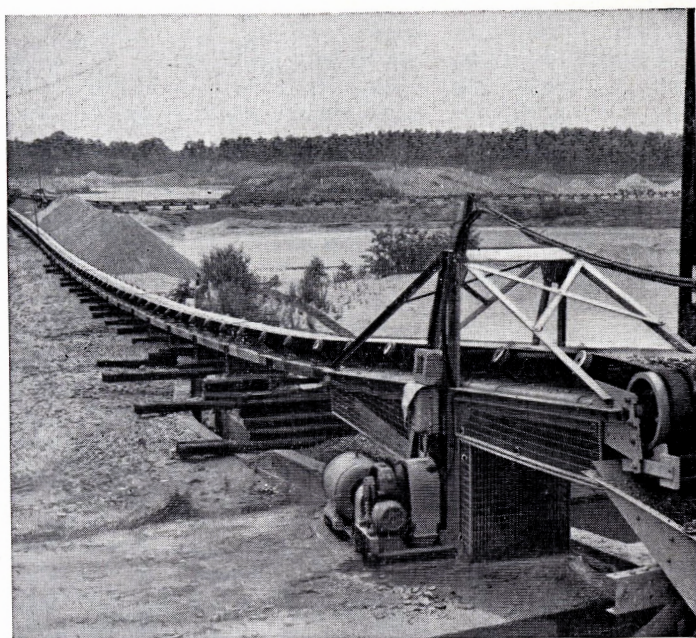
Double Decomposition — Sulphides

By employing double decomposition it is sometimes possible to develop a carbonate coating on grains of a given sulphide which will enable them to be stained easily. Thus, in order to stain nearly white sphalerite grains so that they might be readily recognised in certain mill-products, Paul Raffinot (1953, p. 5) first converts their superficial portions to carbonate and then stains them by employing dithizone in the following manner: The sample is boiled for 10 minutes in a solution of sodium hypochlorite (18 deg. Baume) containing 10 per cent of sodium carbonate. (Presumably the hypochlorite oxidises the sulphide to sulphate which is then converted to carbonate by double decomposition.) After the above treatment the sample is washed repeatedly with water in order to remove all the hypochlorite. The treated sample is then placed in a 0.1 per cent solution of dithizone in 1.0 per cent ammonia and the whole is warmed for 5 minutes at 50 deg. C. As a result of this treatment the sphalerite grains are stained red.

Double Decomposition — "Insolubles"

The following description of a method of staining barite grains, which was devised by Gaudin (1935, p. 555) "to differentiate between quartz and barite in short order so as to control a flotation concentrating operation", contains a typical example of a method of staining an insoluble mineral which depends on the initial development of a suitably reactive coating by double decomposition. "While the machine was running at reduced speed, a small sample of the froth was deslimed crudely, and boiled for a few minutes with an excess of sodium carbonate. This changed the surface of the barite to barium carbonate ($BaSO_4 + CO_3^{--} \rightarrow BaCO_3 \downarrow + SO_4^{--}$). The mineral was then washed and warmed with potassium chromate, which changed the barium carbonate to yellow barium chromate ($BaCO_3 + CrO_4^{--} \rightarrow BaCrO_4 \downarrow + CO_3^{--}$)."

As an example of a means of staining grains which depends first on superficially altering the surface of the minerals by means of attack by a melt, the method advocated by Lancsweert (see Vanden Herrewegen, 1954, pp. 478-480) may be cited for differentiating



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between tantalum/niobium minerals (particularly columbite and tantalite) and ilmenite in certain coarse fractions from the Belgian Congo. In essence the method depends on warming the sample in a porcelain crucible with potassium hydroxide pellets just damped with water. Finally excess potassium hydroxide is decanted off and dilute hydrochloric acid is added to the cold crucible. This removes the excess of potassium hydroxide and causes the columbite/tantalite grains to become white. The colour of grains of ilmenite, silicates, etc., is unchanged.

Jedwab (1957) employed a somewhat similar method to identify *single* grains of beryl: this involves subjecting the fragment to be tested to a brief attack by molten sodium hydroxide, followed by its immersion, for one minute, in a boiling aqueous solution of quinalizarin. This causes beryl to assume an intense blue-mauve hue, whilst quartz is unchanged: feldspars may develop violet patches which, unlike the beryl stain, disappear when the fragments are immersed in water for one hour.

Ampian (1962) developed the following modification of Jedwab's technique which enables all the beryl fragments in a composite sample to be stained simultaneously and in such a way that they can be easily and positively identified: 0.1 g. of the sample is heated with 5 ml. of 0.75 per cent aqueous sodium hydroxide solution, in a 100-ml. nickel beaker, until the water has been expelled and the residual NaOH has just melted. The beaker is then immediately removed from the source of heat and after cooling the melt is leached with c. 20 ml. of distilled water. The grains are collected by filtration through Whatman 41 H filter paper, and after they have been well washed they are transferred, by the aid of 5 ml. water, into a 50-ml. glass beaker. About 40 mg. of quinalizarin are added together with 3 drops of 5.0 per cent sodium hydroxide solution. The mixture in the covered beaker is boiled for 1 minute, then the grains are collected on filter paper, washed and dried. This treatment causes beryl fragments to be stained an intense blue colour, whilst albite, chrysoberyl, microcline, muscovite, orthoclase and phenacite are either unaltered or coated with reddish spots. "The surface alteration of particles subjected to normal mineral-dressing reagents and treatment does not affect the procedure."

Further types of stains depending on precipitation

In all but one of the examples yet cited, staining by precipitation has depended on the development of a coloured compound of a cation of the mineral under examination, but the following will serve to emphasize the fact that

other types exist. Thus, to enhance the difference between Nigerian thorite and zircon obtained—as noted earlier—by bleaching with hydrochloric acid, the following staining test may be employed (Hosking and Patterson, 1956, p. 74): Transfer c. 2 g. of the sample to a 100-ml. beaker and cover with the minimum of concentrated hydrochloric acid. Cover with a watch-glass and boil rapidly for 4 minutes. Decant, and wash the residue 5 or 6 times with water, but leave the solids just covered with water after the last wash. Add c. 0.5 g. of *finely-ground* ammonium molybdate and swirl to effect solution. Add 10 ml. of concentrated nitric acid and boil rapidly for 3 minutes. Decant wash 8 times with water, dry, and examine. This treatment causes thorite grains, unlike grains of other species normally occurring in Nigerian gravity concentrates, to assume a matt, intense lemon-yellow appearance, whilst zircon grains become leached and glassy. Any dark minerals are coated, to varying degrees, with a whitish veneer.

This test depends on reaction between the silicate anion and the staining reagent to produce yellow ammonium silicomolybdate.

The following similar test may be used to stain grains of amblygonite yellow and so enable grains of this species to be detected in a sample which also contains quartz, feldspar, spodumene, petalite, lithium and other micas and apatite (Hosking, 1957, p. 275): Place a few grams of the sample in a small beaker and cover to a depth of about $\frac{1}{2}$ in. with a solution prepared by dissolving 5 g. ammonium molybdate in 100 ml. cold water and pouring it into 35 ml. concentrated nitric acid. Bring the solution nearly to the boil and continue at this temperature for about 5 minutes, occasionally agitating the grains. Finally decant and wash the sample several times with water.

Dye-Adsorption Methods

Minerals of the Clay Group

The identification of minerals of the clay group can be established most satisfactorily by the employment of physical methods—some of which are prolonged—which require the use of costly apparatus operated by skilled personnel. However, for some years considerable researches have been made into the possibility of identifying these minerals by staining techniques, as these are quick, cheap and easily carried out. As a result of these investigations several reasonably reliable methods are now available. [See, for example, Faust, G. T. (1940) and Hambleton, W. W. and Dodd, C. G. (1953).]

It is sometimes desirable to identify clay minerals, as a group, in mill-material, and, therefore, it is pertinent briefly to describe the procedure adopted by Faust (1940, p. 20) for indicating that an exceedingly fine constituent in fluorite ore from Helena (Montana) was clay. The ore was finely crushed and boiled with nitric acid to destroy carbonates and any organic matter. This residue was then treated with perchloric acid to remove the fluorite, and after washing it was immersed in a solution of malachite green in benzene when the fine fraction assumed an emerald-green colour indicating that it was clay.

Other Minerals

Gaudin (1935, pp. 554-555) recommends the use of Malachite Green for staining hydrous oxides, and notes that "the action seems to be specific for minerals with hydroxyl radicals." If the mineral is colourless, Malachite Green stains it blue, but if it is coloured the dye causes it to assume a colour which is intermediate between blue and its natural colour. In particular, Gaudin notes that Malachite Green will stain grains of bauxite and of limonite, and states that similar staining reactions occur when other organic dyes are used.

It is possible to differentiate between grains of calcite and dolomite in certain mixtures by immersing the sample in cold Lemberg's solution. This treatment causes grains of calcite to be stained violet within 5 to 10 minutes, whereas dolomite grains show no colour change for at least 20 minutes, after which they are faintly speckled with blue.

Lemberg's solution is prepared by boiling together 60 parts of water, 4 of Al_2Cl_6 , and 6 of logwood chips for 25 minutes. During the period of boiling the solution is constantly stirred and water is added periodically to replace that lost by evaporation.

The staining of the calcite depends on the fact that it develops a coating of aluminium hydroxide and this adsorbs the logwood dye.

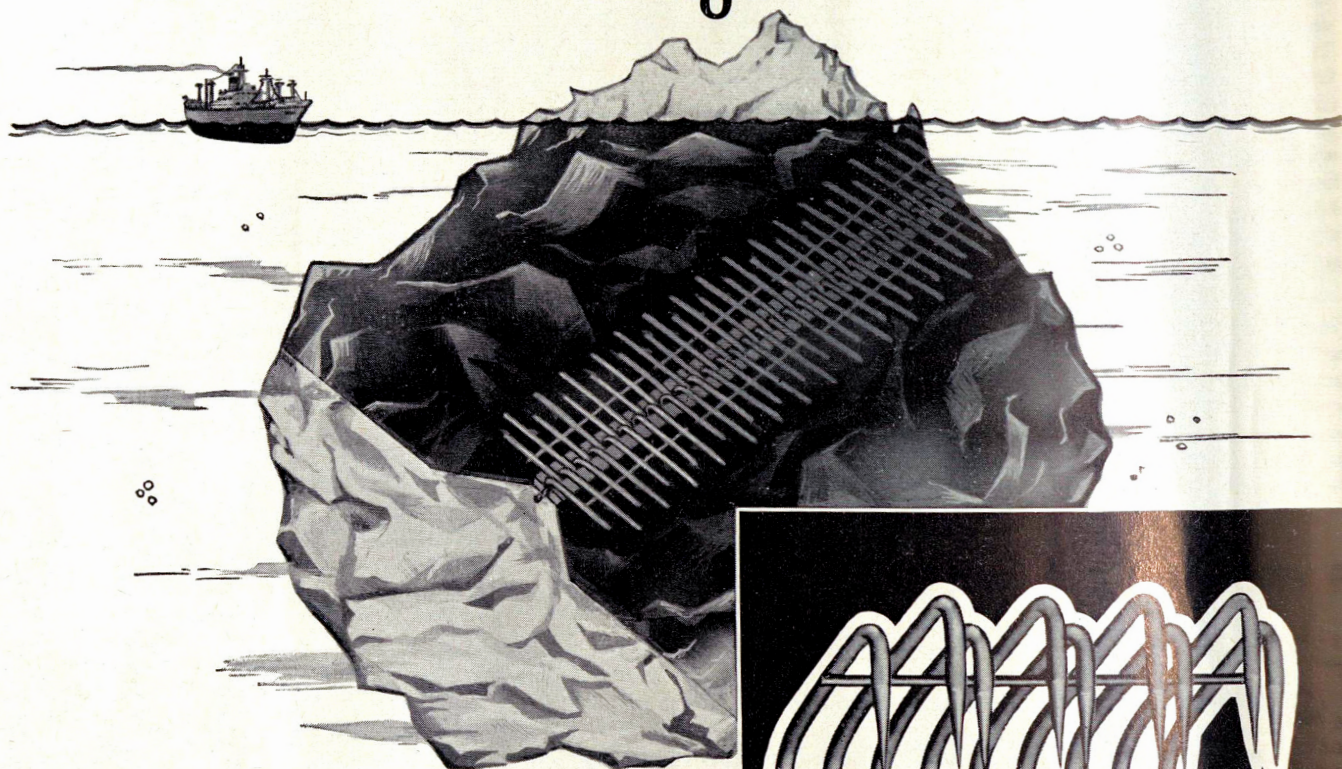
The staining of certain magnesium-bearing minerals by means of Titan Yellow—which is noted later—are further examples of adsorption staining.

Perhaps the staining of β spodumene by methylene blue—which is discussed earlier—is a further example of this general method, but this is uncertain.

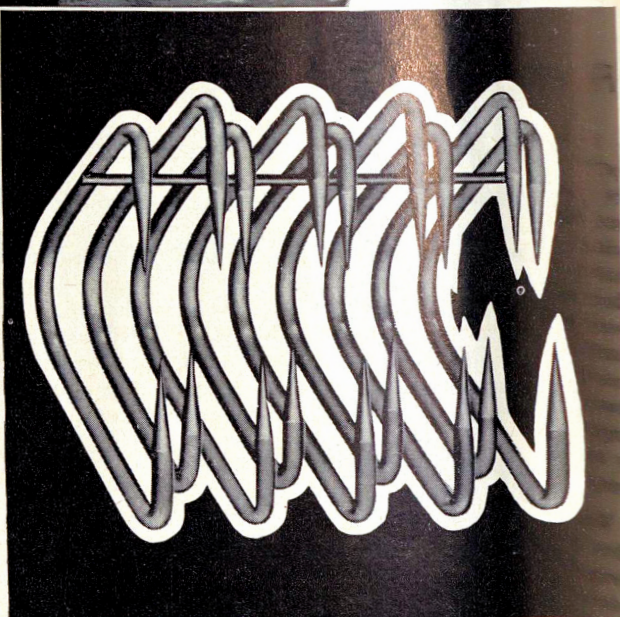
Colour at Elevated Temperatures

It has been noted above that when the superficial portions of fragments of certain zinc species are converted to oxide by roasting they exhibit a strong canary-yellow colour when they are hot

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which enables them to be identified readily. This is the outstanding example of the method of "staining" grains which depends on the use of atmospheric oxygen and the continued application of heat.

Allied to the above method of identification is that which depends on the fact that certain species emit light when subjected to gentle heat. If a sample containing grains of fluorite is heated in a dark-room to about 150 deg. C. the fluorite grains become luminescent, thus enabling an estimate to be made of the amount of this material in the sample. The colour of the light emitted by fluorite varies with the variety: the chlorophane variety emits an emerald-green light, whilst others emit lights which are characterized by purple, blue and reddish tints. (See Dana, E.S., 1932, p. 275.) Grains of fluorite which have been heated and cooled do not become luminescent on reheating.

According to Dana (1932, p. 275) "some varieties of white limestone or marble, after slight heating, emit a yellow light; so also tremolite, danburgite and other species. Doubtless these facts could be used advantageously in work involving the examination of samples containing fragments of these species.

Fluorescent Coatings

As many insoluble, fluorescent compounds are known which are produced by reactions between inorganic cations and organic reagents, it should be possible to develop such compounds on the surfaces of mineral grains, and by so doing facilitate their identification. The writer has shown that this assumption is correct by developing the following procedure for depositing a fluorescent coating on grains of the three most important secondary zinc minerals, namely smithsonite, hydrozincite and hemimorphite:—

Warm the sample for 5 minutes, at about 65 deg. C., with a mixture consisting of equal volumes of a 0.5 per cent solution of 8-hydroxyquinoline in 60 per cent ethanol, and 5N. ammonia. Then decant and wash the grains well in water. This treatment causes the above zinc species to be stained yellow and to fluoresce a brilliant yellow under both long- and short-wave ultraviolet light.

In addition to zinc oxinate, the oxinates of the following metals also fluoresce when treated with ammonia and examined under ultraviolet light:—Ba, Sr, Mg, Ca, Al, Sn(ii), Sn(iv) and Cd. (See Pollard, F. H. and McOmie, J. F. W., 1953, pp. 53-54.) However, minerals containing these elements which are likely to be associated with zinc

Table 1. Reactions between grains and 0.1 N. Silver Nitrate		
Mineral	Nature of coating, etc.	Method of Conducting Test
Arsenopyrite	Grains cohere	Boil for 1 minute
Bornite	Grey coating of silver	Agitate grains in cold solution
Bournonite	No apparent change	Boil for 1 minute
Chalcocite	Grey coating of silver	Agitate grains in cold solution
Chalcopyrite	Blue or purple film	Boil for 1 minute
Cuprite	Grey coating of silver	" " " "
Galena	Grains somewhat dull	" " " "
Niccolite	Grey coating of silver	Agitate grains in cold solution
Pentlandite	Slight orange-yellow film. Patchy	Boil for 1 minute
Pyrite	No apparent change	" " " "
Pyrrhotite	Purplish-brown film	" " " "
Sphalerite	No apparent change	" " " "
Stannite	" " "	" " " "
Tetrahedrite	" " "	" " " "

(i) The silver coating tends to develop somewhat quicker on chalcocite than on bornite. It is possible that the various samples of bornite grains reacted positively because of included chalcocite, but this has not been established.

(ii) When the above staining method is employed as an aid to the estimation of chalcopyrite in a sample which also contains pyrrhotite, the stain on the latter may be removed (to avoid confusion) by washing the sample in 1.0 per cent sodium nitrite. (Raffinot, P., 1953, p. 6.)

deposits will not, in the vast majority of cases, form oxinates by this treatment.

As a further example of mineral grain identification by the development of a characteristic fluorescent coating it can be recorded that after fragments of bauxite have been warmed for a few minutes with equal volumes of the 8-hydroxyquinoline solution noted immediately above, and 5N. acetic acid, that they fluoresce yellow-green under both long- and short-wave ultraviolet light.

Interference Films

It is wellknown that when white light falls upon a surface which is coated with a transparent film that interference colours are seen. The colour which is observed is due primarily to the thickness of the transparent film, but is modified by the body-colour of the film, by differences in the reflectivity of the upper and lower surfaces of the film, and by the colour and other physical characteristics of the substratum. Obviously then, if films are developed on several mineral species by identical treatment, the interference colour displayed by any one is likely to differ from those of the others and so serve as an aid to its identification. Reasoning of this sort led Gaudin (1935, pp. 556-562) to develop some most valuable aids to the recognition of fragments of certain sulphides in mill-products, provided they were first embedded in bakelite and polished. Gaudin stressed the importance of controlling the staining operation rigorously: an obvious necessity in view of the fact that the colour depends largely on the thickness of the coating. He also suggested that transparent oxide films

would probably prove to be the most satisfactory type, from a diagnostic point of view, when examining sulphides, and noted that certain reactions which would result in the development of transparent films on some sulphides would cause opaque coatings to develop on others. Staining methods employing reactions of the latter type were termed "Combination Methods" by him.

Independent investigations by Raffinot (1953, pp. 5-6) and the writer have demonstrated that dilute silver nitrate (one of the staining reagents used successfully by Gaudin) was also capable of staining loose grains of a number of sulphides in a way which made them readily recognisable, provided the temperature of the solution and the immersion times were carefully controlled.

Gas Liberation

Carbonate and sulphide grains may be detected by methods which depend on the liberation of gas. It is well known that one or more of the diluted mineral acids react readily with most mineral carbonates, causing the evolution of carbon dioxide. Thus, if a portion of the sample under test is placed on a glass slide, treated with dilute acid, and observed under the microscope, grains of Carbonate minerals can be identified because of the trains of bubbles emanating from them.

Grains of minerals which contain sulphide ions can be identified rapidly and surely by employing the sodium azide/iodine test. (See Feigl F., 1928, p. 369 and 1947, pp. 227-229 and p.301.)

(To be concluded)

Rapid Identification of

MINERAL GRAINS IN COMPOSITE SAMPLES

K. F. G. HOSKING, M.Sc., Ph.D., A.M.I.M.M.

The author concludes the review of suitable chemical methods for rapidly identifying mineral grains published in the January issue. Differential staining techniques are given particular prominence.

TO examine a sample for sulphides, a portion of it is spread out on a glass slide covered with the reagent, and examined under the microscope. A train of nitrogen bubbles is initiated by every grain which contains sulphide ions.

Use of Nitrogen

Under standard conditions the rate of evolution of the nitrogen bubbles is to some extent governed by the percentage of sulphide sulphur in the grain. It is, for example, quite easy to differentiate between a sample of arsenopyrite and of loellingite because of this fact. Furthermore, because the rare minerals helvite, genthelvite and danalite contain only a small percentage of sulphide sulphur, nitrogen bubbles are liberated very slowly when they are immersed in the azide/iodine reagent: nevertheless, the evolution is sufficiently rapid to permit a distinction to be made between grains of these minerals and garnet grains, which they resemble and with which they are often associated. (A method of identifying these minerals by staining is described later.)

Grains of some sulphides tarnish extremely quickly, and may then be quite unreactive towards the azide/iodine reagent. Pyrite, for example, which might appear quite bright, may not react—presumably because a thin, colourless film has developed on it as a result of oxidation. Therefore, except when testing grains which were taken immediately before from the grinding-circuit of a mill, it is wise to subject the sample to a short treatment with hot concentrated

hydrochloric acid, followed by a wash in distilled water, before applying the azide/iodine test to it.

It must also be noted that some flotation reagents contain sulphide sulphur, and that unless steps are taken to remove all traces of these from a sample by an appropriate acid treatment, misleading results will be obtained on applying the azide/iodine test. (In practice it is often extremely difficult to remove some reagents when they are adsorbed on the surfaces of mineral grains without resorting to very drastic treatment.)

The reagent, which is fairly stable, is prepared by dissolving 3 g. of sodium azide in 100 ml. of 0.1N iodine solution.

Fusion Tests

Grains of certain of the more intractable minerals can be conveniently identified by subjecting them to fusion tests which result in the development of products which are either characteristically coloured, or which fluoresce characteristically under ultraviolet light.

The most noteworthy method of this type is that developed by MacKay, and others (see MacKay, R. A., 1950-51, pp. 129-131) for identifying columbite grains in Nigerian products. Identification depends on the fact that when Nigerian columbite grains are heated on a thin layer of flux composed of sodium bisulphate, sodium fluoride and fusion mixture, each grain becomes surrounded by a reaction-product halo which fluoresces pale-yellow under ultraviolet light. As the test is well known it need not be discussed at length, but it is worth mentioning, however, that it is also extremely useful for detecting grains of

pitchblende in Cornish cassiterite concentrates. Under the conditions of the test the pitchblende grains become encircled by a halo which fluoresces an intense yellow under ultraviolet light.

Orsino Smith (1953, p. 60) notes several elements which when fused with sodium or lithium fluoride form compounds that fluoresce characteristically under ultraviolet light. Possibly these may be used to form the basis of tests—similar to MacKay's test—for identifying grains of certain tungsten, titanium, etc., minerals, although the writer has had no success, as yet, in this sphere.

However, grains of wolframite, and other manganese-rich minerals, may be detected as follows: Place a few pellets of sodium hydroxide on a square of asbestos paper (of side 1.5 inches) and near to one edge. Holding the paper with a tongs, heat it over a large burner in such a way that as the hydroxide melts it runs over the paper, forming first a thin veneer and then sinking into it. At the moment when that portion of the paper over which the melt has spread assumes a matt appearance, sprinkle the sample over it and continue heating for about 30 seconds. (If the grains are coarse they should be gently pre-heated, otherwise they may decrepitate during the test and be lost from the sample.) Manganese-rich grains thus treated become partially coated with green sodium manganate and are surrounded by blue-green haloes.

As an alternative the test may be carried out on a thin veneer of sodium hydroxide on a silica crucible lid.

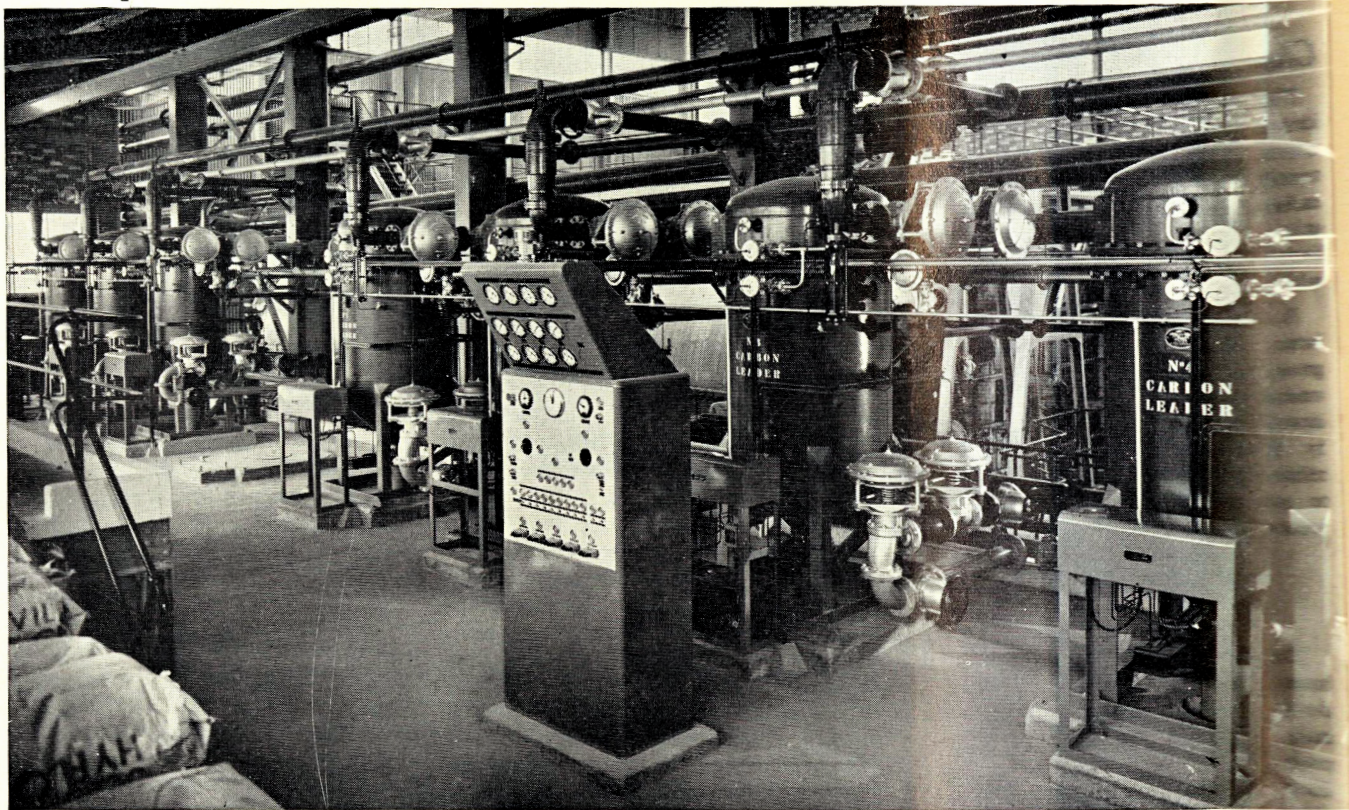
A more convenient way of detecting grains of wolframite in many types of sample is to boil a few grams of the



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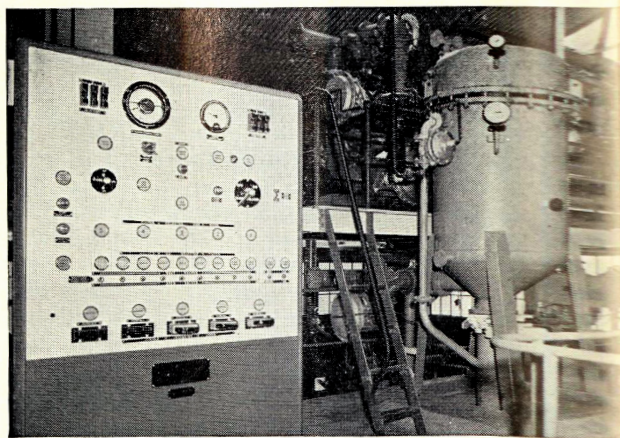
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material for c. 20 minutes with a solution of aqua regia of the following composition: HCl, 25 ml.; HNO₃, 15 ml.; H₂O, 60 ml. This treatment causes grains of wolframite (and scheelite) to assume a yellow colour (Vanden Herrewegen, 1954, p. 477).

Chemical Aids

The following chemical aids to the identification of grains of specific minerals in "loose" composite samples, together with those embodied in the earlier parts of this paper, include not only the vast majority occurring in the literature, but also a number of new ones which have been developed by the writer.

Published methods for differentiating between members of the clay-minerals group have, however, been largely omitted. Those aids which have been described by others have been repeated by the writer in order to check their reliability and attempts have been made to extend their fields of application whenever possible.

The Alizarin S Method

The method of staining grains of bauxite, which is described below, was developed by the writer in order to make rapid comparisons between various products of mill-tests which were conducted in order to examine the possibility of improving the grade of certain bauxitic material.

The samples contained grains of bauxite, kaolinite, felspar, quartz and ilmenite, and a large percentage of the components were coated with a film of limonite.

Boil 10 g. of the sample for a few minutes in HCl in order to dissolve the limonite. Cool; dilute well with water; swirl; allow the solids to settle and decant. Repeat the washing until all the soluble iron salts are removed. Cover the insoluble residue with N. NaOH and after 2 minutes add 0.1 per cent alizarin S solution until an intense violet colour develops. Tumble the insolubles in this solution for 1 minute, and after a further 4 minutes add 5N. acetic acid until the violet colour of the liquid is discharged. Decant and wash the grains well with water. The bauxite fragments are stained purplish-pink.

The method is based on a test for aluminium which was devised by F. W. Atack (1915, p. 936).

Methods of staining grains of bauxite by employing Malachite Green and 8-hydroxyquinoline have been described earlier.

Grains of felspar, but not accompanying grains of quartz, may be stained by the following method which was developed by A. L. Engel (1938, pp. 69-70) in order that the grades of products produced during experimental work on the separation of felspar from quartz by flotation could be rapidly estimated by visual means.

Place a few grams of the material to be examined in a beaker and cover them with equal volumes of HCl and warm distilled water. Digest for 5 min., then wash the sample until it is free of acid and dry on the sand-bath. Cover the dried material with a solution of Safranin O and warm gently on a hot plate for at least 15 minutes. When the staining appears to be complete—that is, when certain components are obviously stained yellow or pink—decant and wash the grains thoroughly with distilled water. (The grains must be tumbled during the washing process in order to remove any traces of the stain on grains other than felspar.) Decant the wash water, then dry the product on a sand-bath and examine it. The felspar grains will either be distinctly yellow or pink. The Safranin O solution is prepared by dissolving 2 g. of Safranin O in 30 ml. of ethyl alcohol and adding 70 ml. of distilled water.

Engel found the method satisfactory for the problem in hand, but inferred that it might not be applicable to all felspar-quartz products. The present writer has applied it to several Cornish samples and has found it completely satisfactory. Feigl (1947, pp. 470-471) describes the test briefly but states, erroneously that the quartz—but not the felspar—is stained.

Quartz, Potash Minerals and Plagioclase Felspars

The following method was devised by Graham (1955, pp. 37-38) primarily to differentiate rapidly between grains of certain silicon-containing species in soil samples, but on occasion it could obviously be used to advantage in the mill.

Having acid-cleaned a known quantity of the sample (in order to remove soluble iron compounds) mount it on a microscope slide in such a manner that the grains are only partially embedded in the cement. Subject the sample to the fumes of hydrofluoric acid for a few minutes and then immerse the slide in a saturated solution of sodium cobaltinitrite.

After 5 minutes remove the slide, wash it with distilled water and immerse it for 5 minutes in 0.1 per cent aqueous Malachite Green. Finally rinse in distilled water and dry. This treatment

causes microcline, orthoclase, and potash mica grains to be stained greenish-yellow, whilst albite becomes greenish-blue. The lime-rich plagioclases are more deeply etched than albite and are stained a paler hue. Quartz remains clear and unetched.

Chrysotile and Other Varieties of Asbestos

Morton and Baker (1941, pp. 515-523) devised the following test for differentiating between small fragments of chrysotile and other varieties of asbestos in order to assist the Canadian asbestos industry.

Place a few drops of a 1 per cent solution of iodine in glycerin on a microscope slide and disperse a little of the sample to be tested in the liquid. Within a short space of time chrysotile—but not other varieties of asbestos—is stained brown. Brucite and serpentine are also stained brown by the same treatment.

Witherite and Similar Species

The chromate and permanganate-sulphuric acid methods of staining grains of witherite in order to differentiate between them and grains of fluorite, quartz, barite, etc. have already been described.

A further method, which is given below, is applicable in the absence of lead minerals, and is based upon the fact that a red salt is precipitated when barium ions react with sodium rhodizonate in a weakly acid solution. (See Feigl, F., 1947, pp. 165-167.)

Place a few grams of the sample in a beaker and cover them with a freshly-prepared 0.2 per cent solution of sodium rhodizonate. Warm at about 60 deg. C. for a few minutes and decant the solution. Add 1:20 HCl; tumble the grains rapidly; decant and wash the residue with water. As a result of this treatment witherite grains are stained pink, whereas grains of quartz, barite, fluorite, calcite, celestite, and strontianite are unstained.

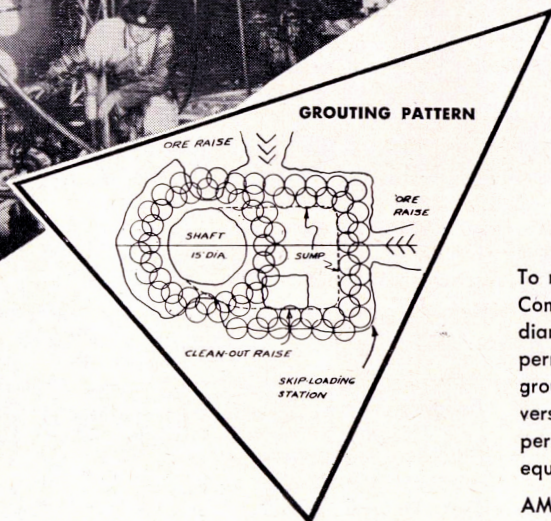
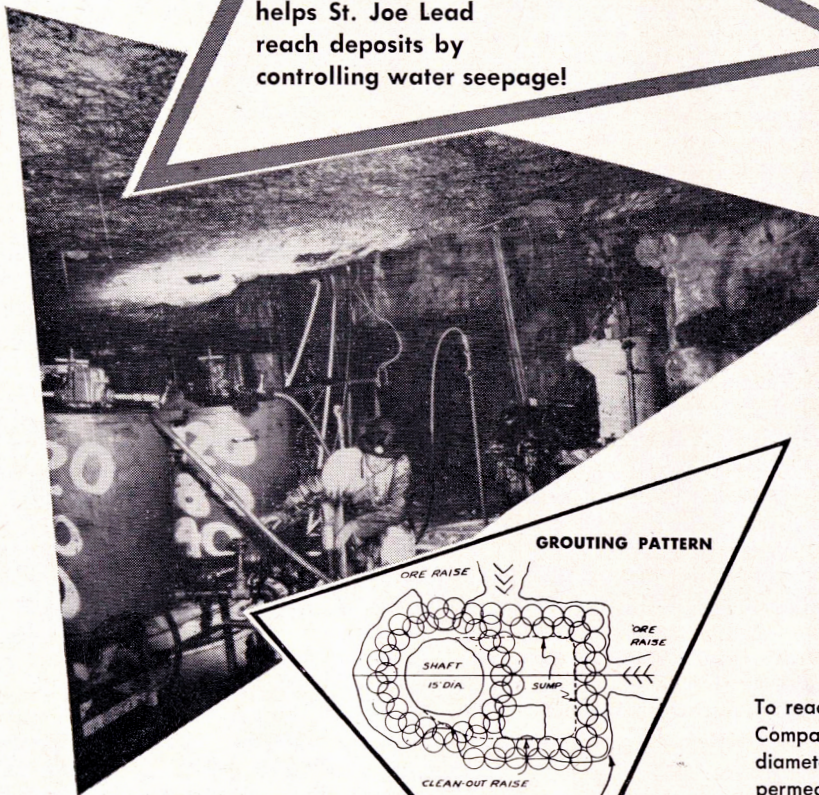
In order not to destroy the pink film on the witherite it is essential that the sample should only remain in the 1:20 HCl for a very short space of time.

Calcite

Theoretically it should be easy to differentiate between grains of calcite and quartz because of differences in their refractive indices and because of the characteristic cleavage of calcite. Yet, as Gaudin remarks (1935, p. 554), "both of these criteria may fail: a coarser quartz grain and a finer calcite grain may give similar interference colours, especially in suitable orientations; a locked particle consisting of calcite and quartz would be very embarrassing". Gaudin also points out that calcite can be

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stained in a variety of ways to suit the requirements of almost any problem and notes that when it is boiled with ferric chloride it assumes a yellow to brown coat of ferric hydroxide; with nickel nitrate, a green coat of nickel hydroxide; with silver nitrate, a brown to black coat of silver hydroxide. Quartz is unaffected by these reagents.

The present writer has found that calcite grains in a quartz-calcite mixture can be strongly stained by employing the following method:—

Boil the sample with 10 per cent copper sulphate for 5 minutes then decant and wash the grains thoroughly with water. Finally, tumble the grains in a 1.0 per cent alcoholic solution of rubenic acid then decant and wash the residue slightly with water. The calcite grains are thus stained dark green to black, whilst the quartz grains are unaffected.

Calcite and Dolomite

Holmes (1930, pp. 265-267) recommends the following methods for differentiating between grains of calcite and dolomite:—

Aluminium Chloride/Logwood Solution

This method has already been described.

Silver Chromate Method—Immerse the sample for 3 to 4 minutes in 10 per cent silver nitrate solution heated to about 65 deg. C., then decant and wash the grains well with distilled water in order to remove all free silver salts. Finally, cover the grains with potassium chromate and after a minute decant and wash the residue. Calcite grains thus treated are reddish-brown, whilst dolomite and quartz grains are unaffected. Aragonite is stained less deeply than calcite.

Copper Nitrate Method—Boil the sample for a few minutes with very dilute copper nitrate solution. This treatment causes calcite grains to be stained green, whilst dolomite and quartz grains are unaffected.

Magnesite Grains

Titan Yellow Method—The following method of staining grains of magnesite—which is based upon a test for magnesium due to I. M. Kolthoff (1927, p. 254)—was developed by the writer in order to differentiate between grains of magnesite

and dolomite and to facilitate the recognition of magnesite grains in a mixture which also contains such minerals as quartz, calcite, fluorite, dolomite and the sulphates and carbonates of the alkaline earths.

Place a few grams of the sample in a beaker and add 0.1 per cent Titan Yellow until the grains are covered with a layer of solution about 1 cm. deep. Heat until the solution just boils, then remove the beaker from the source of heat and add 5N. sodium hydroxide until the solution is vermilion. Tumble the grains gently for about 30 sec., then decant and wash the residue. This treatment causes magnesite grains to be stained pink, whilst grains of the other species noted above are unaffected.

Diphenylcarbazine Method—In order to differentiate between grains of magnesite and dolomite, Feigl (1947, pp. 442-443) recommends the following method.

Cover a little of the test-material with a hot alcoholic alkaline solution of diphenylcarbazine. After 5 minutes decant, and wash the grains repeatedly in hot water until no more colouring matter goes into solution. This treatment causes the magnesite grains to become red-violet, whereas the dolomite grains are unaffected.

Dolomite grains may also be stained by the above procedure provided they are first ignited.

Danalite-Helvite-Genthelvite Group

The formulae of the end-members of the danalite-helvite-genthelvite group are as follows:—

Danalite, $[\text{Fe}_8 \text{Be}_6 \text{Si}_6 \text{O}_{24} \text{S}_2]$.
Helvite, $[\text{Mn}_8 \text{Be}_6 \text{Si}_6 \text{O}_{24} \text{S}_2]$.
Genthelvite, $[\text{Zn}_8 \text{Be}_6 \text{Si}_6 \text{O}_{24} \text{S}_2]$.

At Iron Mountain, New Mexico, deposits containing danalite, helvite and garnets were subjected to examination because it was thought that their beryllium content might be sufficiently high to justify mining them. (See Glass, J., Jahns, R. H. and Stevens, R. E., 1944, pp. 163-191.) During the examination it was found difficult to judge a sample of ore as to its beryllium content because of the close resemblance between small grains of the beryllium species and the accompanying garnets. In order to overcome this difficulty, Gruner developed the following methods of staining the beryllium species which depend on the development of yellow coatings of sulphide of arsenic and red coatings of antimony sulphide as the result of reactions between the appropriate cations and the sulphide ions of the species under discussion.

Place a very small representative sample of the powdered or crushed rock

in a 50 ml. beaker and add sufficient dilute sulphuric acid to cover it. Add a pinch of arsenious oxide and boil the solution for 1 or 2 minutes. Decant and wash the sample twice with water. Examine the contents in the beaker—still covered by a little water—under the microscope. Grains of any of the beryllium species noted appear brilliant canary-yellow.

Even when the gangue is essentially yellow garnet the stained fragments are easily distinguished. However, to simplify recognition under such conditions a little *metallic* antimony should be substituted for arsenious oxide in the above test: this will cause grains of the beryllium mineral species to be stained a brilliant red.

It is claimed that by employing either of the above procedures that a single grain of helvite or danalite can be detected in the midst of thousands of gangue minerals. The writer has confirmed that this claim is correct.

The application of the sodium azide/iodine test to the identification of grains of danalite and related beryllium species when they are associated with garnets has been noted earlier in this paper.

Arsenopyrite

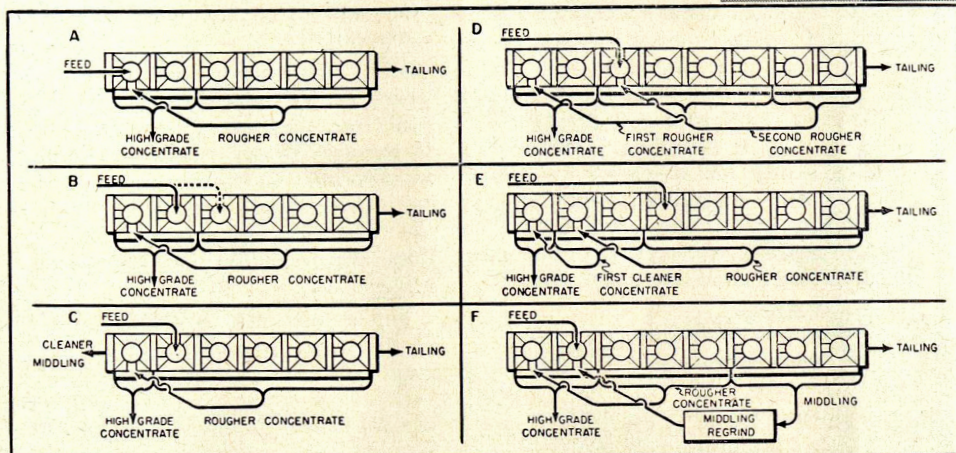
It is often difficult to distinguish between small grains of pyrite and arsenopyrite when a mixture of these is examined under the microscope. Raffinot (1953, p. 6) suggests using the following differential staining method in order to overcome this difficulty:—

Place a few grams of the sample in a small beaker and cover them with a 3 per cent solution of potassium ferrocyanide in 5 per cent hydrochloric acid. Bring the solution just to the boil, then decant and wash the grains with water. Arsenopyrite grains are stained a vivid blue by this treatment, whereas pyrite grains are unaffected.

The writer has shown that chalcopyrite and stannite grains are also unaffected by the above treatment, whereas loellingite grains are stained blue and, therefore, cannot be distinguished from arsenopyrite grains in mixtures by this method. The loellingite which was tested was obtained from the Castle-an-Dinas mine (Cornwall), and although it stained in the same manner as arsenopyrite, it was found that the test could be used to differentiate between *pure* samples of each of the two species for the following reason: Whenever arsenopyrite is warmed with ferrocyanide-HCl reagent, *strong* blue "streamers" ascend through the solution from the

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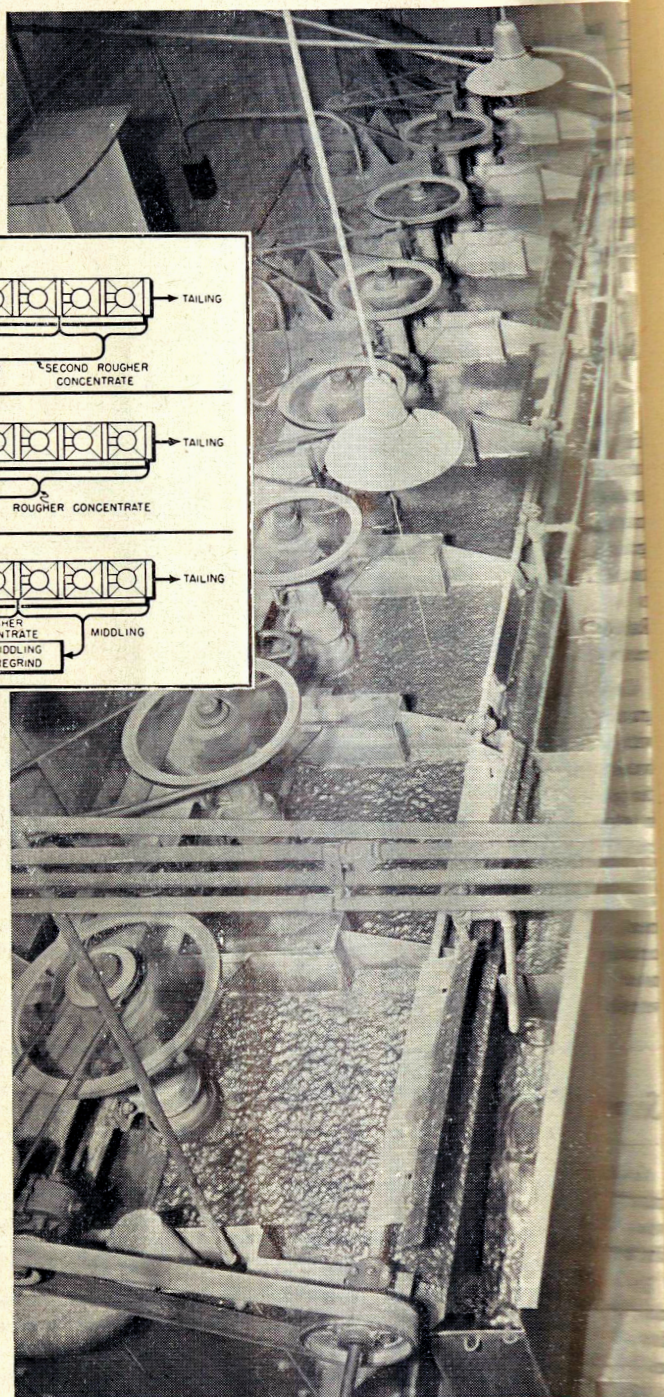


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grains, but when loellingite is similarly treated the "streamers" are much less strongly developed or non-existent. This difference in behaviour is obviously due to the fact that arsenopyrite is generally more soluble in the reagent than is loellingite.

Sphalerite

It has been noted earlier that grains of the paler varieties of sphalerite are often difficult to recognise when they are associated with grains of pale gangue minerals. A method of staining sphalerite grains in order to facilitate their recognition has also been noted, but in addition to it the following method due to Raffinot (1953, p. 5) may well be worth recording :

Convert the surfaces of the sphalerite grains to zinc carbonate by treatment with sodium hypochlorite-sodium carbonate solution, as described elsewhere in this paper. Wash the treated grains well in water and place them in cold ferricyanide-oxalic acid-diethylaniline solution. After a few minutes decant and wash the grains in water. By this treatment, sphalerite grains—and grains of certain secondary zinc minerals which are noted below—are stained orange to vermillion.

Secondary Zinc Minerals

Grains of many of the secondary zinc minerals are often difficult to recognise when they are associated with grains of similarly coloured gangue minerals.

To simplify the study of products made during experimental work on the flotation of smithsonite, hydrozincite, and hemimorphite, Raffinot (1953, pp.3-5) has developed Methods (i) and (ii) below for staining these three zinc species. Methods (iii) and (iv)—which are due to the writer—are satisfactory alternatives to the methods that have been proposed by Raffinot.

Dithizone Method—Grains of the three zinc species already noted are stained carmine by treatment with dithizone in ammoniacal solution. Secondary lead minerals, however, react similarly, and if they are present the following procedure should be adopted :

Immerse the sample in 10 per cent sodium sulphide solution for 5 minutes, then decant and wash the grains well with water. (This treatment converts the superficial portions of the grains of secondary lead minerals to black, lead

sulphide which will not react with dithizone under conditions of the test.) Warm the sample for 5 minutes at 50 deg. C. with a 1:1 mixture of a 0.2 per cent solution of dithizone in carbon tetrachloride and 2.0 per cent ammonium hydroxide. Finally decant the solution and then wash the sample with water and examine it under the microscope.

Ferricyanide/Oxalic Acid/Diethylaniline Method

This method of staining certain secondary zinc minerals depends on the following facts which were first investigated by E. Eegriwe (1928, p. 228):

Alkali ferricyanide oxidises diethylaniline to a coloured compound and at the same time ferrocyanide ions are formed. This oxidation proceeds slowly and incompletely unless the ferrocyanide ions are removed as they are formed.

If zinc ions are present ferrocyanide ions are removed because of the formation of sparingly-soluble, white zinc ferrocyanide. The coloured oxidation product is adsorbed on the white zinc ferrocyanide. Obviously, appreciable quantities of cations which form coloured, insoluble ferrocyanides will interfere.

In order to stain grains of the three zinc species under review, Raffinot places a portion of the sample in a cold solution composed of equal volumes of 3 per cent potassium ferricyanide and 3 per cent oxalic acid containing 0.5 per cent diethylaniline. After 3 minutes the solution is decanted and the grains are washed with water. This treatment causes grains of smithsonite, hydrozincite and hemimorphite to be stained reddish-orange.

The present writer has established that grains of the following species are stained orange to red by precisely the same reaction: Descloizite, hopeite, parahopeite, tarbuttite, and willemite. The following species which sometimes occur in zinc deposits do not react: Barite, celestite, witherite, strontianite, fluorite, quartz, cerussite and anglesite.

Ammonium Mercuric Thiocyanate-Copper Sulphate Method—Zinc and copper ions together react with mercuric thiocyanate ions forming a purple insoluble compound. This reaction may be used as the basis of a method for staining grains of smithsonite, hydrozincite and hemimorphite, and probably grains of other secondary zinc minerals.

Place a few grams of the sample to be examined in a small beaker and add about 1 ml. of 0.1 per cent copper sulphate, 6 ml. of ammonium mercuric thiocyanate and 3 or 4 drops of 5N. HCl. Heat the solution to 65 deg. C. and after 5 minutes remove the beaker

from the source of heat. Tumble the grains gently in the warm solution for a further 30 seconds, then decant and wash the sample lightly with water.

This treatment causes grains of the three zinc species noted, to be stained pinkish-purple. The ammonium mercuric thiocyanate is prepared by dissolving 8 g. of mercuric chloride and 9 g. of ammonium thiocyanate in a 100 ml. of water.

Acridine Hydrochloride Method—An alcoholic solution of acridine hydrochloride reacts with zinc ions in the presence of potassium thiocyanate forming a yellow-green complex. Iron gives a red and cobalt a green product under the same conditions. [The most complete account of reactions involving the use of acridine hydrochloride are due to Welcher (1947, vol. 3, pp. 118-120).] Under certain conditions this reaction may be employed to stain grains of smithsonite, hydrozincite and hemimorphite, and the most satisfactory procedure is as follows:

Place a few grams of the sample in a small beaker and add about 5 ml. of 10 per cent potassium thiocyanate, 4 ml. of a 1 per cent solution of acridine hydrochloride in alcohol and 2 ml. of 2 per cent HCl. Heat the solution to about 70 deg. C. and maintain this temperature for 4 minutes.

At the end of this stage it is necessary to remove the beaker from the source of heat, decant and wash the grains with water. Grains of the three zinc species noted are stained greenish-yellow by this treatment.

Lead Minerals

Sodium Rhodizonate Method — Feigl (1947, p. 429) notes that lead can be detected in minerals—in the absence of barium species—by spotting a little of the finely-powdered sample first with a drop of buffer solution and then with a drop of sodium rhodizonate solution.

If lead is present this treatment causes the mineral particles to become red. The same writer also states that certain lead species—for example, galena and stolzite—react more positively to the above test if they are first calcined.

The present writer has examined a wide range of secondary lead minerals and has found that the majority of them stain beautifully if they are immersed in a freshly-prepared 0.2 per cent solution of sodium rhodizonate and then, after 30 seconds, sufficient buffer solution is added to discharge the colour of the solution. However, stolzite, raspite, wulfenite and some varieties of pyromorphite and mimetite are only adequately



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stained by the method described if they have previously been calcined. Furthermore, in order to stain grains of lead minerals with a metallic lustre satisfactorily by employing the sodium rhodizonate reaction it is necessary first to subject them to a short calcination which should be stopped as soon as the grains become dull. The fact that grains of galena, jamesonite and bournonite may be stained by employing the sodium rhodizonate method after calcination has been noted earlier and it is probable that all lead species with a metallic lustre can be stained by similar treatment.

This method thus not only enables grains of lead minerals in a composite sample to be detected, but also serves as a simple means of differentiating rapidly between grains of pairs of such similar species as bournonite and tetrahedrite, and jamesonite and stibnite. The preparation of the buffer solution has already been noted.

Potassium Iodide Method—The writer has found that grains of most secondary lead minerals are stained brilliant yellow if they are immersed in a freshly-prepared, fairly strong solution of potassium iodide and 1 : 7 nitric acid is added until the volume of the solution is increased by about a quarter. Little or no agitation of the grains is necessary, and staining is effected almost immediately.

The yellow lead iodide coating, whilst not adhering as firmly as the rhodizonate one, is nevertheless adequate for most purposes. Stolzite, raspite and wulfenite are not stained to any marked extent by this procedure, and the staining of certain samples of pyromorphite and mimetite is greatly enhanced if, on adding the potassium iodide, the whole is warmed somewhat before the addition of the nitric acid.

This method of staining has been applied directly to concentrates on a vanning shovel in order to make an estimate of the amount of cerussite present.

Locating Sulphide and Secondary Lead Mineral Grains—Having applied sodium azide/iodine reagent to a sample in order to locate sulphide grains, the further addition of a little 1 : 7 nitric acid causes cerussite (and doubtless certain other secondary lead minerals) to stain yellow. The stained grains are best seen after the treated sample is washed lightly with water in order to remove the staining reagents.

Raffinot's Method of Staining Cerussite (1953, p. 3)—Raffinot notes that grains of cerussite and of anglesite may be stained black by treating them with a solution of sodium sulphide, but remarks that this is

not always a satisfactory aid to identification as it is not always easy to distinguish between such stained grains and grains of galena. As briefly noted earlier in this section, he recommends staining cerussite by warming the sample for 5 minutes at 70 deg. C. with a solution containing 0.1 per cent of dithizone and 2.0 per cent of sodium cyanide. This treatment causes cerussite grains to become red. Anglesite is not stained effectively by this method as it is too soluble. The addition of the sodium cyanide prevents any secondary zinc minerals which may be present from being stained.

Cerussite and Anglesite

Head and Crawford (Rep. Div. U.S. Bur. Min. 2932) recommend the following methods of staining cerussite and/or anglesite grains in order to distinguish them easily in crushed samples:—

(a) To produce a bright yellow coating of lead chromate of equal intensity on both cerussite and anglesite grains, treat the sample with a cold saturated solution of potassium dichromate for 15 minutes.

(b) To stain anglesite yellow, but not cerussite, treat the sample for one minute with a cold aqueous solution containing 2 per cent potassium dichromate and 0.5 per cent sodium hydroxide.

(c) To stain cerussite yellow, but not anglesite, immerse the sample in a cold 1 per cent solution of CrO_3 for one minute. (If this solution is applied for a greater length of time than that stated, or if a stronger solution than the one recommended is used, some or all of any galena present will be decomposed. If the test is conducted as suggested above, only an orange tarnish develops on the galena.)

The originators of the above tests suggest that the staining of a given sample is best carried out on a watch glass and that the grains should be tumbled during the whole period in which they are immersed in the solution.

Copper Minerals

Chalcocite—Grains of chalcocite—unlike those of similar minerals—are readily stained blue if they are immersed for 2 minutes in a cold 20 per cent solution of ferric chloride. A slight amount of warming tends to increase the intensity of colour.

Chrysocolla and Malachite—Grains of certain varieties of chrysocolla are not

unlike grains of malachite, but whereas the specific gravity of the former mineral varies from 2 to 2.2, that of the latter varies from 3.9 to 4. Therefore, if these are the only green species in the sample, and if they do not occur in composite grains, they may be readily separated by employing bromoform. However, if a considerable quantity of composite grains occur this method is of no value. Under these conditions estimations are greatly simplified if one of the two species under discussion is stained a colour other than green. Often it is possible to differentiate between grains of chrysocolla and of malachite by the following staining method:

Immerse a few grams of the sample in warm 5N. acetic acid and after 30 seconds add sufficient warm 10 per cent sodium xanthate solution to treble the volume. Tumble the grains for a minute, then decant and wash the residue with water. Malachite grains are stained yellow by this treatment, whilst grains of chrysocolla are usually found to be unaffected.

The reactivity of different varieties of chrysocolla to these reagents is variable, and therefore it is wise to conduct appropriate preliminary tests on the chrysocolla of a given mine before the above method is applied to the examination of the mill products. A pale blue chrysocolla which was examined by the writer was so reactive that when fragments of it were tumbled for about 2 minutes in cold 10 per cent sodium xanthate solution they assumed a brilliant yellow coating. When malachite grains were similarly treated they were unaffected.

Stibnite

It is not easy to differentiate readily between grains of stibnite and jamesonite by inspection under the microscope, but it is desirable to do so as they often occur in the same orebody. This differentiation may be simplified, either by utilizing the sodium rhodizonate reaction to stain the jamesonite (as described earlier), or by staining the stibnite yellow by placing a few grams of the sample in 40 per cent potassium hydroxide for 5 minutes. This treatment also causes any kermesite grains which may be present to be stained yellow. For examination under the microscope, the solution should be decanted and the grains washed lightly in water. This method of staining stibnite (and kermesite) is based on an etch reaction described by Short (1940, p. 114).

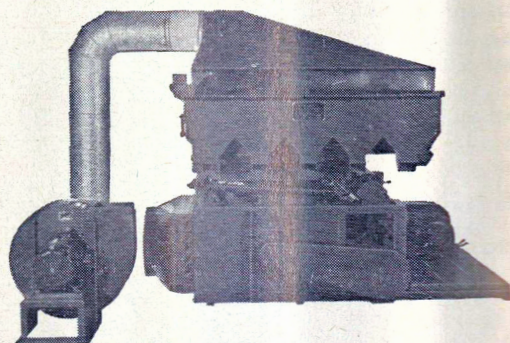
Calaverite $2(\text{Au Te}_2)$ —Grains of tellurides of the precious metals are not

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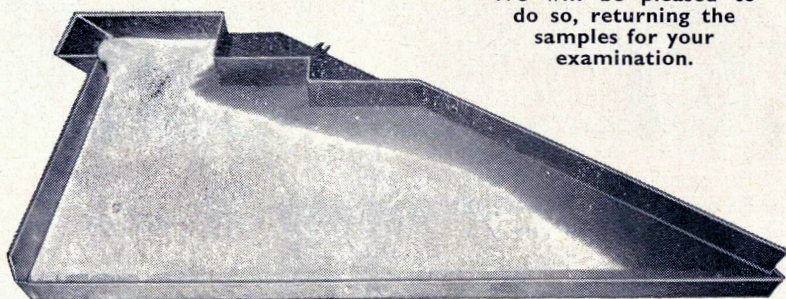
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- Due to the greater sensitivity to slight differences in density of the particles, Dry Concentration will successfully treat ores which are not amenable to wet concentration.
- Ore may be efficiently separated at the economical releasing point of the minerals.



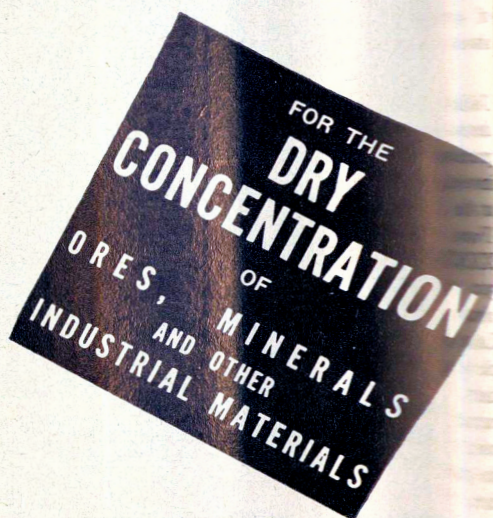
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ILMENITE
BAUXITE
MICA

GALENA
URANIUM
VANADIUM
FELDSPAR
LIME
PYRITE
BARITE

COLUMBITE
MAGNETITE
HEMATITE
LIMONITE
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□ Mineral Grains

readily identified under the microscope when they are associated with grains of the common sulphide minerals. The following method of staining grains of calaverite—which could probably be used for staining other tellurides—assists in their recognition:

Immerse a few grams of the sample in 1:1 nitric acid. Bring the acid just to the boiling point, then decant and wash the grains with water. (This treatment causes calaverite to assume a pinchbeck-brown tarnish, whilst pyrrhotite and chalcopyrite display iridescent tarnishes. Gold is unaltered, and pyrite shows little or no tarnish.) Cover the grains with stannous chloride-HCl reagent and warm gently for about 3 minutes, then decant and wash the grains lightly. This treatment causes the calaverite grains to become dull black and tends to remove the tarnish developed on grains of certain species noted above as a result of the preliminary acid treatment.

The stannous chloride-HCl reagent should be prepared immediately before use by boiling 4 or 5 g. of tin with 15 ml. of concentrated hydrochloric acid for about 5 minutes.

Tellurides, etc.—Haultain and Johnston devised the following method to identify precious metal tellurides (see "Milling investigations into the ore as occurring at the Lake Shore Mine". By the Staff. *Trans. Canad. Inst. Min. Metall.*, 1936, XXXIX, 434.) in extremely fine (minus-300 mesh) super-panner concentrates obtained during experimental work on Lake Shore material: A little of the sample is sprinkled on a Pyrex slide which is then heated in the flame of a bunsen burner until the glass is red and on the point of bending. At this temperature the particles adhere to the slide which is then turned over and the grains are heated, for a further few minutes, directly in the flame. This treatment causes each grain of precious metal telluride to be converted to a globule of gold or silver surrounded by a coloured halo which is easily recognised when the cooled slide is examined under the microscope.

Haultain and Johnston (1933) also observed that this treatment facilitated the identification of certain other species. Thus, pyrite becomes a characteristic red and chalcopyrite a characteristic grey, pentlandite sprouts hair-like filaments and exudes globules.

Columbite—Flinter (1955) states that the identification of grains of Malayan columbite is facilitated by subjecting the sample to a preliminary treatment with boiling hydrochloric acid. If the treatment is continued for about 15 minutes hematite and magnetite dissolve

whilst ilmenite either assumes a grey or white coating, or becomes dull. Wolframite grains are partly coated with a canary-yellow product. When the sample is boiled for an hour with the acid, iron-rich magnetic varieties of rutile (derived from ilmenite) react in the same way as ilmenite. Normal rutile, cassiterite, tourmaline, allanite, betafite and columbite are unaltered by this treatment.

Conclusion

In writing this paper it has been regarded as axiomatic that if each grain of a given species in a composite sample of loose grains can be easily recognized "close" mill-control is greatly facilitated. The writer, however, feels certain

that many of the aids to grain identification are not widely known nor has their potential been generally appreciated. This paper has been written primarily with a view to demonstrating the value of these simple aids. It is also hoped that this paper will stimulate research into the development of further simple chemical—and other—tests which will assist the mineral-dresser and thus tend to increase the efficiency of the mill.

Acknowledgements

Of necessity, the preparation of this paper has involved the abundant use of the findings of other workers, but in particular the author wishes to pay tribute to the works of Gaudin and Raffinot from which he has culled a wealth of information.

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