

tation to secure a precipitate free from copper. Even with this additional manipulation (and consequent loss of time) the results obtained are incorrect. The amount of the standard KCN required to decolorise the solution is affected by varying temperature, varying amount of free and combined ammonia, and volume of solution. The presence of other metals also affects the process. Silver, zinc and nickel, if present, react with KCN like copper, while manganese retains copper in the precipitate to a greater extent than iron, besides obscuring the end reaction.

Having had occasion recently to make a large number of determinations of very low-grade materials, where it was necessary to know the copper contents accurately to within one or two hundredths per cent., I have employed the cyanide-assay with complete success notwithstanding the difficulties above cited. Careful attention to details and the introduction of a system of comparative standardization have resulted in a method which leaves little to be desired as regards speed of working, combined with a very fair degree of accuracy. The principles involved are: (1) The greatest possible uniformity in every stage of the assay; (2) the adoption for each particular class of material of a value for the KCN solution obtained by standardizing in the presence of that material.

#### DETAILS OF WORKING.

The details of an assay or set of assays of material containing from 0.1 to 0.5 per cent. of copper are as follows: 10 grammes of the ore are weighed into a No. 4 porcelain casserole and digested on the hot plate until complete solution is effected. The acids used, degree of concentration, time of heating, etc., will depend on the nature of the ore; and each operator will determine these features for himself, bearing in mind, however, that in this, as well as in all subsequent operations, what is once adopted must be strictly adhered to in every assay of the same class. When solution has been effected, the sides of the dish are rinsed down with a stream of hot water and the proper amount of dilute ammonia is added, the contents of the casserole being well stirred before and after the addition of the ammonia. The assay is then returned to the hot plate, heated moderately for five minutes, and then filtered hot through a 7-in. filter into a narrow beaker of 250 c. c. capacity. Nearly the entire contents of the dish can be poured out at once onto the filter, and the balance is sluiced out with a stream of cold water. The mass on the filter is washed twice with cold water, the jet being forced with the full power of the lungs, so as to stir up and wash the precipitate thoroughly with the minimum quantity of water. The liquid filters rapidly, and the clear blue filtrate, after two washings, should now be almost cold (not over 70 degrees F.); and if the washing has been properly performed the bulk should not be over 180 c. c. If it be less than this, more water should be added. It is convenient to have a strip of white cardboard bearing the 180 c. c. mark for the size of beaker used. By standing this strip alongside of the beaker the volume of the solution can easily be measured.

The solution, if cool and of the proper volume, is now ready for assay. Place the beaker under the burette on a square of white filter paper. Run in the KCN solution rapidly, stirring vigorously meanwhile. As the color fades, proceed more slowly and cautiously, adding the standard solution in drops, instead of a continuous stream, as at first. If the ore solution contains manganese, a precipitate will begin to form as the colour fades from blue to violet. This precipitate, which appears in the form of a dirty bluish-green or

brown discoloration, obscures the end-reaction, and must be removed by filtration before the finishing point is reached.

Keep the attention fixed on the side of the beaker, near the bottom, look down transversely through the cloudy solution. As long as a decided tint of violet can be seen on the mirror-like surface of the glass, the cautious addition of KCN can be continued, drop by drop. Stop before this colour becomes too faint, and filter rapidly through a thin filter-paper. Washing is unnecessary, as the solution is now too close to the finishing point for the trace of copper absorbed by the filter to affect the result appreciably. The filtered solution, which should be very light blue or pale violet in tint, can now be easily brought to a delicate, hardly perceptible rose pink, which should be the finishing point for weak solutions. For solutions containing much copper, it is necessary to finish with a more decided pink, since it takes a little longer for the last color to fade from a solution which was rich in copper at the start. Where no manganese is present, re-filtering will usually be unnecessary, the solution remaining clear to the finishing point. A considerable excess of manganese may cause trouble in the assay, especially in the presence of ammoniac chloride, as the precipitated hydroxide then forms very slowly, and keeps making its appearance as a milky discoloration in successive filtrates. This can be very largely prevented, however, by the presence of sufficient iron, as the iron precipitate seems to carry with it all but a trace of the manganese.

A little longer heating after addition of the ammonia also aids in securing a filtrate free from disturbing quantities of manganese.

#### STANDARDISING THE POTASSIUM CYANIDE SOLUTION.

From what has already been pointed out, it will be clear that the amount of KCN necessary to decolorize the solution does not give the true amount of copper present in the ore, when the latter contains other bases which interfere with the assay. When the acid solution is precipitated with excess of ammonia, Ag, Ni, and Zn, if present in the ore, go into the solution, and react like copper with the KCN; while Fe, Mn and Al are precipitated as hydroxides. This precipitate invariably contains a small proportion of the copper which, in the case of iron, is probably mechanically retained; but where manganese is present there is a further retention, possibly as a manganate.

Arsenic may also form in insoluble arsenate; and Cr also interferes.

In looking for a way to obviate these difficulties, I began by assuming that the amount of copper retained by these causes combined, varied directly as the ratio of the total copper to the disturbing elements presents. Experiment proved this assumption to be a true one, at least to a very close approximation; and, this fact once established, the course of procedure became clear.

In standardizing the KCN solution for any particular ore or class of ores of the same composition, two portions of 10 grammes each are weighed out for separate assays. To one of them is added a weighed amount of pure copper, and the two are run through together, being treated in all respects like regular assays. The difference in the number of c.c. of KCN used in the two assays represents an amount equivalent to the weighed excess of copper taken, and from this the true value of the ore is readily ascertained. Operating on the same weight of identical gangues, this figure (with frequent checking) is used. For a different gangue, or a different weight of the same gangue, another test must be made; and whenever an accurate determination of a new ore is required, the assay is run in duplicate as described.