

original oil. On mixing the castor oil with magnesia, the free fatty acids present in the oil are neutralised, forming minute quantities of magnesia soap, which acts as a kind of catalytic agent, emulsifying the balance of the oil and distributing it very thoroughly. On drying the paste formed by mixing castor oil, magnesia, and water, the oil globules do not unite again, but remain in the state of the original fine subdivision. On examining the powder under the microscope with a magnification of 450, very few fat globules can be observed, almost the entire amount of oil having been absorbed so firmly that no oil separates out when the powder is mixed with hot or cold water and allowed to stand.

Mr. D. T. ATKINS (of Messrs. Eimer and Amend), described the working of the new Emerson bomb calorimeter, giving figures showing its efficiency as compared with the standard Mahler bomb calorimeter. He also explained the working of the Barnes telephone pyrometer, both with the galvanometer and telephone receiver, indicating its advantages over some similar pyrometers. The Wanner pyrometer was also shown.

A further exhibit consisted of a collection of fused silica ware, including dishes, muffles, tubing, and other laboratory appliances, and also a variety of fused alumina ware, such as muffles, crucibles, combustion bulbs, tiles, tubing, &c., which are recommended for metallurgical and other high temperature experiments.

Other exhibits were a Williams modified Westphal specific gravity balance for liquids and solids, and a new graphite rotating cathode for the rapid electrolytic determination of metals.

Sydney Section.

Meeting held at Sydney on Wednesday, May 12, 1909.

PROFESSOR SCHOFIELD IN THE CHAIR.

A RAPID AND ACCURATE METHOD OF ESTIMATING IRON IN IRON ORES.

BY J. S. MACLAURIN, D.S.C., AND W. DONOVAN, M.S.C.

Anyone who has had experience in the estimation of iron in magnetites and hematites must have frequently found difficulty in getting the iron into solution within a reasonable time. Such difficulties led one of us to subject the ore to a preliminary heating in hydrogen or coal gas. The iron could then be readily dissolved in hydrochloric acid, reduced by stannous chloride, and estimated volumetrically. In these estimations, however, potassium permanganate could not be used if really accurate results were required, owing to the disturbing effect of hydrochloric acid, and consequently it was necessary to employ the bichromate method, which has the serious defect of requiring an external indicator. These defects have been entirely removed by modifying the process, which is now carried out in the following manner.

A weighed quantity of finely ground ore is introduced into a hard glass bulb tube by means of a small copper scoop (made of thick copper foil bent into the desired shape and soldered to a stout piece of copper wire). Corks, carrying narrow glass tubes, are then fitted into the ends of the tube. Should the ore contain organic matter, air is drawn through the tube by means of a filter pump or aspirator, and the bulb heated to redness for a few minutes, being gently rotated backwards and forwards during the heating. It is then allowed to cool, the air current stopped, and a stream of hydrogen or coal gas substituted. This is lighted at the exit end of the small glass tube. Should hydrogen be used, the tube may be prevented from closing up by the use of a platinum tip, made by rolling a small strip of platinum foil round the end of the tube. After heating for from 10 to 30 minutes, according to the nature of the ore, the

tube is allowed to cool, the stream of hydrogen or coal gas shut off, and the reduced ore shaken into a flask containing one or two grams of sodium bicarbonate and a little water. Any ore remaining in the tube is washed into the flask with dilute sulphuric acid. By this means sufficient carbon dioxide is evolved to fill the flask, and so prevent oxidation of the iron. In order to still further guard against oxidation, the flask is closed by a rubber cork carrying a short exit tube and an inlet tube which dips beneath the surface of the solution in the flask, through which a steady stream of carbon dioxide is allowed to flow from a Kipp apparatus. The flask is then heated, and the solution boiled for from 5 to 20 minutes, according to the ease with which the iron dissolves. This operation requires little or no attention, and can be carried out while another sample is being heated in the bulb tube. When all the iron is dissolved, the solution is allowed to cool in the air, or more rapidly by closing the exit tube with the finger and allowing water to flow over the outside of the flask, the carbon dioxide apparatus being still, of course, attached. It only remains to shut off the carbon dioxide, remove the cork from the flask, wash the tubes with a little water, and titrate the solution with potassium permanganate.

This process gives very constant results, duplicate determinations agreeing excellently. Thus, in estimating the iron in 55 samples of limonite, the greatest difference in the duplicate determinations, which were made quite independently of one another, was 0.22 per cent. of iron, and the average difference only 0.09 per cent. In many of these determinations, after titrating with permanganate, the solution was reduced with sulphuretted hydrogen, the excess of this gas being removed by passing carbon dioxide through the boiling solution (Hillebrand's method). The iron so reduced was then estimated by titration with permanganate, and in every case the result so found agreed closely with that previously found by our method. This agreement shows that the whole of the iron dissolved out of the reduced ore goes into solution as a ferrous salt, and is therefore estimated in the subsequent titration. Moreover, the residues contained no iron. This was proved by fusing with sodium and potassium carbonates, and testing for iron.

We had thus conclusive proof of the accuracy of our results, but for the sake of comparison with other methods, the following determinations on five limonites were made. The results given under A are by our method. Those under B were determined by dissolving in hydrochloric acid and stannous chloride, adding mercuric chloride to remove the excess of stannous chloride, phosphoric acid and manganese sulphate to improve the conditions for titration, and finally titrating with permanganate. In column C of the table, the results were obtained by dissolving in hydrochloric acid, reducing with lead and copper sulphate, and titrating with potassium bichromate.

The results are percentages of iron.

A.	B.	Difference from A.	C.	Difference from A.
44.03	43.13	-0.90	43.42	-0.61
49.21	49.06	-0.15	49.29	+0.08
38.57	38.09	+0.42	38.66	+0.09
26.73	26.40	-0.27	26.78	+0.05
35.52	35.07	+0.45	36.02	+0.50
Mean diff. from A. -0.09			Mean diff. from A. +0.02	

The titration in the case of B is not nearly so satisfactory as in A, owing to the presence of a large amount of precipitate, which somewhat masks the ending. Duplicates by this method differ to a much greater extent than do those by the A method. In making determinations by method C the solution of the ore is very slow—sometimes taking hours to accomplish, and, owing to the necessity for an external indicator, the titration is not nearly so simple and satisfactory as in the A method.

Our method is thus shown to be well adapted for the estimation of iron in limonites. It may also be used with advantage for the much less soluble magnetites, ilmenites, hematites, &c. In this case the ore requires fine grinding

and half an hour's reduction in hydrogen; coal gas cannot be used, as it does not give complete reduction. If the hydrogen be made from commercial zinc, it should be purified from antimony and arsenic, which interfere with the subsequent titration. The purification is most simply effected by passing through a hard glass tube wrapped in thin sheet asbestos, and heated to redness by a long flat burner. The tube should be from 15—18 ins. long, and about $\frac{1}{2}$ in. in diameter, reduced to $\frac{1}{4}$ in. or less for 4 or 5 ins., to allow of the ready deposition of antimony and arsenic. It is interposed between the source of hydrogen and the hard glass bulb tube in which reduction of the ore takes place. This treatment removes all but minute traces of arsenic and antimony, and these may be got rid of by passing the hydrogen through one or two wash bottles containing glass wool saturated with chromic acid.

Using this method we got the results given in column A of the following table. Those shown under B were obtained from the same solution in the following manner. After titrating with permanganate (for A determinations) the solutions were reduced by hydrogen sulphide (Hillebrand's method), the excess removed by carbon dioxide, and again titrated with permanganate. The results so obtained are shown in column B. As a further check on these figures, the ore was fused with sodium and potassium carbonates, dissolved in sulphuric acid, reduced by means of sulphuretted hydrogen, &c., as before, and titrated with potassium permanganate, the percentages of iron so found being shown in column C. Or it was fused as before, dissolved in hydrochloric acid reduced by stannous chloride, and titrated with bichromate, the results being shown in column D.

	A.	B.	C.	D.
Magnetite, Taranaki, N.Z.	59.62 57.75	59.00 —	60.02 57.72	59.76 —
" Quebec, Canada	64.34	—	—	64.12
" France..	67.17	67.24	—	67.34
Ilmenite, Norway ..	43.48	—	43.53	—
Micaceous iron ore	57.39	—	—	57.50
Ilmenite, Ohio, U.S.A....	61.18	61.24	—	—

These figures are sufficient to show that our method gives very satisfactory results for such refractory ores as magnetites and ilmenites. For these ores it has the further merit of making the estimation of titanium quite a simple matter. This is done by adding hydrogen peroxide to the solution after the permanganate titration, and estimating the percentage of titanium by comparison with a standard solution of that substance. By this means, 9.12 per cent. of titanium dioxide was found in the Taranaki magnetite, as against 9.27 per cent. obtained by fusion. Moreover, it was found that none of the insoluble residues contained more than minute traces of titanium.

To recapitulate very briefly, the process consists of the following steps:—(1). Roasting the finely ground ore. (2.) Reduction of the roasted ore by heating in coal-gas or hydrogen. (3.) Solution of the reduced ore in dilute sulphuric acid in an atmosphere of carbon dioxide. (4.) Titration of the resulting solution by potassium permanganate. The advantages of the process over those commonly employed are the removal of any organic matter which might interfere with the subsequent titration, the ease and completeness with which the ores are dissolved, the accuracy of the titrations, and the possibility of estimating titanium in the same solution.

NOTE.—Since writing this paper our attention has been drawn to the fact that a somewhat similar method is described in Crookes's Select Methods in Chemical Analysis, 3rd Edition. That method we find involves the use of a porcelain tube and platinum boats. It requires from four to six hours for four determinations, and it admittedly fails to extract the whole of the iron when dealing with magnetites, this being due no doubt to incomplete reduction.

In our method, on the other hand, much simpler apparatus is used, complete reduction is easily and quickly obtained in the rotating bulb tube, the whole of the iron goes into solution, and the determination is made accurate and reliable by purifying the hydrogen. This was found to be a very important step in the process, because when using unpurified hydrogen, results differing from the true value by several per cent. were sometimes obtained.

Journal and Patent Literature.

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I.—PLANT, APPARATUS, AND MACHINERY.

PATENTS.

Treating materials with air or other gases or vapours for drying, bleaching and other purposes. Tomlinson-Haas, Ltd., Manchester. From F. Haas, Ges.m.b.H., Lennep, Germany. Eng. Pat. 14,014, July 2, 1908.

THE claim is for improvements in the apparatus described in Eng. Pat. 15,108 of 1903 and consists in providing valves by means of which any one or more of a series of drying chambers can be cut out of the drying system, and atmospheric air, which may be first moistened if desired, can then be drawn through the chambers so cut out of the series. Further, a portion of the air drawn through the apparatus may be circulated through certain of the chambers to admit of its being used continuously.

—W. H. C.

Packing pieces for chemical absorption, condensing and like towers. E. P. Peyton, Birmingham. Eng. Pat. 18,831, Sept. 8, 1908:

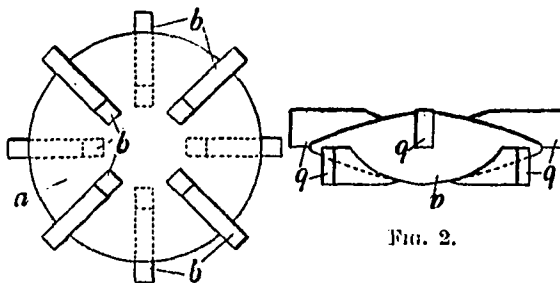


FIG. 1.

FIG. 2.

THE packing pieces have the form shown in plan in Fig. 1, and in cross-section in Fig. 2. They consist of a circular or polygonal central portion, *a*, having curved surfaces provided both above and below with projecting radial ribs or bearers, *b*.—W. H. C.