advantage of leaving the iron, lime, and alumina insoluble at the start and only a small quantity of alkali is introduced into the analysis.

As for the mineral constituents they are better determined on a separate portion. Their accurate determination is made all the more difficult by the presence of an excessive amount of alkali metals as in Peckham's method.

Our practice has been to heat the asphalt at a low temperature in a platinum dish, slowly driving off the volatile constituents in such a way that the asphalt does not burn. At the end the temperature may be raised and the remaining organic matter burned without danger of loss.

The ash is extracted with hydrochloric acid and any insoluble residue fused with a small amount of the mixed carbonates. The solution of the fusion in hydrochloric acid is added to the other and the analysis proceeded with as usual. In this way the quantity of alkaline salts is reduced to a minimum.

Time is actually lost by determining the metals and sulphur in one portion as the determination of the latter must wait until the metals have been separated.

In all but two of the asphalt analyses of Dr. Day, as quoted by Messrs. Peckham, the decimal point is misplaced, making the per cent. sulphur far less than it should be.

LABORATORY OF RICKETTS AND BANKS, NEW YORK.

THE DETERMINATION OF NICKEL IN NICKEL ORES.

BY A. C. LANGMUIR.

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GEORGE WILLIAM SARGENT'S paper on the "Determination of Nickel in Nickel-Steel" in the October number of this Journal, prompts me to publish a somewhat similar method for the determination of nickel in its ores, which has been in use in this laboratory for several months with excellent results. It is particularly advantageous in all cases in which a relatively small quantity of nickel is to be separated from a large amount of iron and surpasses all other methods such as the basic acetate, basic carbonate, or ammonia separations.

One gram of the ore is covered in a No. 2 Griffin's beaker with 15 cc. concentrated nitric acid. One or two cc. of liquid bromine is added and the contents are gently heated until the bromine is expelled. The solution is then boiled down almost to dryness. Bromine in combination with nitric acid effects so thorough an oxidation that no sulphur bead can form. This reaction may also be applied in the analysis of copper mattes, where it is often necessary to filter off the sulphur bead which usually forms after the treatment with nitric acid and to roast it to recover the small amount of undecomposed sulphide which it invariably contains. The addition of bromine renders this extra operation entirely unnecessary.

After expelling the excess of nitric acid, about 50 cc. of concentrated hydrochloric acid are to be added and the solution slowly boiled down to 5 cc. The nitric acid is expelled and any residue carrying nickel and insoluble in nitric acid is brought into solution—at least in the ores handled by us.

Dilute to 250 cc. with hot water and pass hydrogen sulphide to remove copper, etc. Filter and boil the filtrate to expel hydrogen sulphide. Oxidize the ferrous iron by adding nitric acid to the boiling solution, drop by drop, carefully avoiding any excess. Cool slightly and add ammonia until distinctly alkaline. Filter and wash twice with hot water, catching the filtrate which will contain the bulk of the nickel in a large casserole and boil it down while the remaining operations are being performed.

Dissolve the ferric hydrate which carries a considerable amount of nickel in warm dilute hydrochloric acid. Wash into a casserole and boil down rapidly until pasty. Add about 10 cc. of hydrochloric acid (1.10 sp. gr.), warm slightly, rinse into a 250 cc. separating funnel with acid of the same strength, keeping the volume down to 50 cc., if possible, and cool thoroughly. Add now 40 cc. of C. P. ether and agitate vigorously at least five minutes. Invert the funnel occasionally and open the stop-cock cautiously to lower the pressure. The ferric chloride gradually dissolves in the ether leaving the nickel, aluminum, and any cobalt or manganese in the aqueous solution as chlorides.

The first solution obtained by boiling down the nitric acid residue with 50 cc. of hydrochloric acid cannot be profitably treated by the ether separation. On account of the sulphuric acid formed by the oxidation of the sulphur, the removal of the iron by ether is far from complete. The sulphuric acid must be separated from the iron by an ammonia separation so that a solution containing only chlorides may be obtained. There is almost always some slight loss of solution in the treatment with ether in the separatory funnel, but any error from this source becomes negligible if the bulk of the nickel is first separated by ammonia.

After agitating, the funnel is allowed to stand a few minutes and the water solution is drawn off into a second funnel. The ether solution is washed twice with a few cc. of hydrochloric acid (sp. gr. 1.10), the washings being caught in the second funnel. Mr. Sargent contents himself with one ether treatment but we have always found it advisable to remove the iron still remaining with the nickel by a second agitation with 40 cc. of ether. The second operation is carried out in the same way as the first.

The aqueous solution and washings are run into a beaker, the dissolved ether is expelled by boiling, bromine water is added until the solution is strongly colored, and the iron, aluminum and any manganese present are separated by adding ammonia to alkalinity and boiling. If the ether treatment has been properly carried out, a mere trace of iron remains with the nickel and the precipitate may be caught on a 9 cm. paper. The precipitate obtained as above is dissolved in a little hydrochloric acid, bromine is added, and the precipitation with ammonia is repeated. The combined filtrates and washings are added to the casserole containing the now concentrated filtrate from the first ferric hydrate precipitate. The contents of the casserole which now contain all of the nickel in the ore are concentrated to 100 cc. or until ammonium chloride begins to separate. Fifty cc. of concentrated nitric acid are added and the solution is slowly boiled down almost to dryness. In this way the ammonium chloride is entirely destroyed. Ammonium nitrate is unchanged; hence the necessity for avoiding an excess in the oxidation of the ferrous iron. We are now able to concentrate the solution to a volume of a few cc. without the separation of any salts. Ten cc. of concentrated sulphuric acid are added and the casserole heated until dense fumes appear and the nitric acid is entirely expelled. After cooling, dilute with 100 cc. cold water, boil, rinse into the high beaker used for electrolytic determinations, neutralize with ammonia, and add 25 cc. in excess. Dilute to 200 to 250 cc., insert the spiral and the weighed platinum cylinder, and electrolyze with a strong current,—we have used 1.2 amperes. The precipitation of the nickel is generally complete within three hours.

The separation of iron and nickel by ether has not as yet been used in the analysis of nickel ores, or mattes. It is unequaled for cleanliness, rapidity, and accuracy, and deserves the first place among the many methods proposed for the separation of iron and nickel.

The ammonia separation has proved particularly untrustworthy in our hands. Three and even four separations fail to remove the nickel, even if the acid solution containing the iron be poured into water containing an excess of ammonia.

The ready means afforded for destroying almost unlimited quantities of ammonium chloride by boiling down with an excess of nitric acid is not new, having been suggested long ago by Lawrence Smith in connection with his well-known method for alkali determinations. Yet it has been strangely neglected and we believe has never been used in the analysis of nickel ores, where it is particularly useful as it enables us to avoid the troublesome and inaccurate separation of the nickel as sulphide.

The solutions to be tested for nickel, ordinarily obtained in analysis, are choked with ammonium chloride, which prevents the concentration of the solution and necessitates the preliminary separation of the nickel from a dilute solution of sulphide. The difficulties in the way of precipitating and washing nickel sulphide without loss in the filtrate or washings are too well-known to need repetition.

In some analyses made by the author before his adoption of the ether method it was necessary to test for traces of nickel in several samples of steel. At least 5 grams had to be taken and the ammonium chloride accumulating in the filtrates from the ammonia separations of the iron was so large in amount that concentration to a bulk in which ammonium sulphide would reveal nickel was out of the question. But by boiling down slowly, with copious additions of nitric acid, it was possible to destroy all the ammonium chloride and to finally test for nickel in a volume of some 15 cc. under ideal conditions.

The ether separation, as based on the solubility of ferric chloride in ether under the above conditions is, in all probability, not restricted to the determination of nickel or aluminum, but may possibly be applied in the separation of small amounts of zinc from iron. The separation of uranium and iron by ammonium carbonate and sulphide is far from satisfactory if the uranium be small in amount. Possibly in this case as well, the ether treatment may result in a decided improvement.

LABORATORY OF RICKETTS AND BANKS,
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LABORATORY METHOD FOR THE CONTINUOUS AND UNIFORM GENERATION OF ACETYLENE, AND FOR ITS PURIFICATION.

BY J. A. MATHEWS.

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FOR most purposes in which it is desired to employ a stream of acetylene, no purification is necessary, provided the gas has been properly generated from the usual quality of commercial carbide now being produced. Analyses from various sources show that the impurities in acetylene from this source rarely amount to 1 per cent. and a good part of this is likely to be nitrogen. The combined amounts of hydrogen sulphide, hydrogen phosphide, and ammonia seldom exceed 0.2 per cent. In order that the amount of impurities may be as small as possible it is necessary that the gas be produced in the cold. This cannot be done when water is allowed to drip slowly upon the carbide, and furthermore this method causes the gas to be given off spasmodically and the lime residue often bubbles or froths to such an extent that solid matter in considerable amounts may be carried over with the gas. In obviating all these difficulties the following method has proved successful.

The fragments of calcium carbide are placed in a wide-mouthed bottle. They may, with advantage, be suspended in a basket of coarse wire netting, which will keep the carbide away from the