factory to work, and quicker. It obviates the no small difficulty of precipitating all the sulphuric acid without adding an excess of barium chloride in the Stassfurt, and avoids the tedious evaporations and filtrations entailed by the alternate. Against all these advantages there has appeared so far only one disadvantage: the necessity of using a greater quantity of platinic chloride.

THE RAPID AND ACCURATE ANALYSIS OF BONE-BLACK.[1]

By W. D. Horne, Ph.D.

SEVERAL years' experience in the analysis of bone-black have led me to the adoption of the following method for attaining accurate results with comparative rapidity.

In new char, bought from the dealer, the determination of moisture is of importance, being frequently limited by contract to three per cent.

In char, in constant use in sugar refineries, the moisture in the revivified sample is of no importance under usual conditions.

The moisture is determined by heating two grams of the unground char for two hours in a hot air-bath at 140° C. It can be weighed between watch-glasses held by a clip to prevent absorption of moisture.

A convenient way of estimating calcium carbonate is with Lunge's nitrometer, using mercury. Two grams of the finely pulverized sample are introduced into a two-ounce Erlenmeyer flask and moistened with three cc. of a saturated solution of mercuric chloride, the tube of ten cc. of hydrochloric acid (sp. gr. = 1.12) introduced and the flask closed with a rubber stopper, which, with a short glass tube and a rubber tube, is hung to the tube of the three-way cock. By tipping the acid out upon the char and properly manipulating the stop-cock the carbon dioxide is liberated, collected, and measured. Any hydrogen sulphide given off is absorbed by the mercuric chloride. Corrections are made for temperature and atmospheric pressure.

For the determination of carbon, iron, calcium sulphate, and insoluble ash, one portion is used. In preparing the sample

[1] Read before the New York Section, November 9, 1894.

for analysis the iron particles must first be removed by a magnet and the char then ground to an almost impalpable powder.

Two grams of this are moistened with water and treated in a four-ounce covered beaker, with twenty cc. hydrochloric acid, and boiled gently until solution is complete, which is within thirty minutes. The beaker is filled up with boiling water and after settling, which takes place almost immediately, decanted upon a weighed platinum Gooch crucible with asbestos plug. The carbon is washed by decantation in this way five or six times, until the filtrate is free from chlorine. The original filtrate and first decanted washing contain practically all of the iron and calcium sulphate. The following wash-waters can usually be rejected. The carbon is washed out of the beaker into the Gooch, and is then washed with eighty per cent. alcohol, ninety-five per cent. alcohol and ether, each two or three times, set in a water-jacketed air-bath, and heated at 100° C. to a constant weight, which will take about three hours. The increased weight is due to carbon and ash. The carbon is burned off over the blast. The loss of weight is then carbon, and the difference is ash.

To the acid solution containing iron and calcium sulphate a drop of methyl orange solution is added and ammonia to nearly neutral reaction. Ammonium acetate is added until the solution turns yellow and then a few drops in excess. The solution is now heated below 70° C. until complete precipitation of iron and aluminum phosphate takes place, which is usually in about twenty minutes. The precipitate, after being washed free from chlorides, is dissolved by dilute sulphuric acid into a six-ounce Erlenmeyer flask, ten cc. of a ten per cent. solution of sodium sulphite added, and boiled to reduce the iron and to expel the sulphur dioxide. To ascertain when this gas ceases to come over, the vapor is led from the flask through a glass tube fitting through the rubber stopper and inclining vertically downward at its outer end. Under this is held a test-tube of dilute potassium permanganate acidified with sulphuric acid. So long as sulphur dioxide, issues it will decolorize the permanganate solution in the tube when the gas is caused to bubble up through the upper half inch of the solution. When the sulphur dioxide

is all out a drop of the solution from the flask is tested with potassium sulphocyanide for ferric iron. When the reduction is complete the solution is cooled and titrated with permanganate. If alumina is to be determined the above-mentioned precipitate of the iron and aluminum phosphates can be treated with 150 cc. of ammonium molybdate solution, to precipitate the phosphoric acid, and filtered. In the filtrate iron and alumina are precipitated as hydroxides by ammonia, filtered off, dissolved in hydrochloric acid, reprecipitated with ammonia, collected, ignited, and weighed. The two may then be separated by any of the well-known methods.

The filtrate from the iron and alumina precipitation contains the calcium sulphate. This solution is acidified with hydrochloric acid, and barium chloride added to precipitate the sulphuric acid. The barium sulphate is treated in the usual way and calculated to calcium sulphate.

Calcium sulphide may be determined by treating five grams of char with twenty cc. of nitric acid and evaporating nearly dry, adding twenty cc. of hydrochloric acid, and evaporating again very low to expel nitric acid, taking up in hydrochloric acid, and in an aliquot portion of the filtered solution precipitating the sulphuric acid in the usual way with barium chloride. From the weight of barium sulphate found, is deducted that found as above, due to calcium sulphate. The remainder is that due to calcium sulphide.

The physical condition of the char is of great importance and should be thoroughly examined into. The grist is estimated by throwing 100 grams upon a sieve of known mesh, shaking gently, and weighing what passes through. This portion may, in turn, be passed through successively finer sieves, and weighed after each. In shaking the sieve should be tapped only very gently—otherwise particles of char will be forced through which, correctly, belong above.

To determine the density of the char it should be carefully filled into a weighed flask of 50 or 100 cc. capacity, and of very gently sloping sides. Admitted into such a flask through a funnel the char fills it completely without leaving vacant recesses at the shoulders. This is weighed, from which is cal-

culated the specific gravity of the char when loose and the pounds per cubic foot. Now the flask is tapped lightly on the table and as the char settles down more is added until no further settling takes place, and the flask is filled to the mark. This gives the weight of char when packed, from which may be calculated, as before, the specific gravity and weight per cubic foot.

As char grows old in use its porosity decreases and its specific gravity increases. It is sometimes desirable to know its porosity. To do this the weighed flask is filled with distilled water, the char introduced, little by little, displacing part of the water from the flask. At the same time the water remaining displaces from the char its contained air, which rises in minute bubbles. The flask is tapped to pack the char, and, when filled to the mark, is heated on the water-bath to expel all air. After cooling, the supernatant water is removed and the flask and contents weighed. The increased weight over that of the flask packed with char is that of the contained water, from which can be calculated the capacity of the char for any liquid of known specific gravity.

The repeated handling which bone-black receives in continued use in sugar refineries, etc., tells on it very seriously, as the friction of the grains against each other and the machinery wears it into dust. This change is slow, but in the long run is very costly, as the fine dust has to be discarded and replaced by new char.

Any method, therefore, which would enable one to judge of the relative durability of samples of char under this continued friction should be worthy of consideration. I have attempted to contrive such a test, and after a good many experiments have found one which promises well. Twenty-five grams of the char to be tested, between sixteen and twenty-four grist, approximately, are thrown on a sieve with circular holes, one-fiftieth of an inch in diameter, the sieve shaken back and forth ten times, tapped three times, and the shaking and tapping repeated twice. This drives the dust through, and it is weighed. Dust and char are then both put into a cylinder of tinned iron, four inches in diameter, and two inches deep; then six glazed porcelain marbles five-sixths of an inch in diameter and weighing together 74.66 grams are added. Now the can is shaken back and forth

with slightly rotary motion 200 times, the marbles removed, and the char sifted as before. The increased weight of dust is calculated to per cent. of the char used. These tests are pretty constant for different portions of the same sample of char.

The dust formed thus from a good new char was in three cases 1.72 per cent., 1.46 per cent., and 1.76 per cent. From another new char 2.68 per cent. and 2.44 per cent. Two grades made by one firm gave 2.16 per cent. and 2.16 per cent. in one case, and 2.86 per cent. and 2.87 per cent. in the other. Char which had been in use ten months and whose softer parts had already been separated by use gave 0.92 per cent. and 0.94 per cent., showing that it was then in a better wearing condition than when it first entered into use.

THE ASPHALT QUESTION.

By S. F. Peckham.

THERE has lately been published some very interesting reading concerning this question. To go back a few months, in October, 1892, Consul Pierce made a report that seemed to leave very little to be said in reference to Trinidad asphaltum from any standpoint. A few months later, Mr. Clifford Richardson published in the *Journal of Analytical and Applied Chemistry*, for Dec. *1892*, and Jan. *1893*, a paper in which he embodied the results of a most elaborate series of technical analyses of Trinidad asphalts. Within the last twelve months there has been published a voluminous report on Trinidad asphalt by Mr. Richardson, in the "Mineral Resources of the United States." In several late numbers of *Paving*, Mr. D. Torrey has shown that a method of analysis of asphalts may be based on successive solutions in alcohol; and he has also discussed in a very suggestive and intelligent manner, the general subject. In the July number of *Paving*, Messrs. Richardson and Bowen pay their respects to the report of Messrs. Leffman and Sadtler, made to the Citizens' Municipal Association and Trades League, of Philadelphia. In the August number of the same journal Messrs. Leffman and Sadtler reply to their critics. In the December number of this JOURNAL Miss Laura Linton publishes a paper, in which she gives the result of a very careful