supply him with the particulars of any instances of failure of the act or inconsistencies in appraisement which may have come personally to their notice.

Respectfully substituted,
CHARLES E. MUNROE,
Chairman.

EXPERIMENTS ON THE ESTIMATION OF GRAPHITE IN PIG METAL.

BY FRANK L. CROBAUGH.

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THE determination of graphite in pig iron is often required. At furnace laboratories there is rarely time and equipment necessary to do this work by combustion. Appended are some details of experiments made in the search for a simple, yet accurate, method. In all cases counterpoised two 9 cm. filters were folded together and the filtration performed upon them. After partial unfolding and drying at a temperature not exceeding 100° C., (higher temperature chars paper after treatment) the excess of weight of the inner paper will be the weight of the graphite or graphite plus impurities. If now the paper containing graphite plus impurities (chiefly silica) be burned and the residual weight subtracted from the excess of weight of the inner paper, the difference may be graphite. Five grams of the same drillings were taken for each operation.

The metal contained 1.68 per cent. silicon. The washing in every instance was first with water and hydrochloric acid 1.1 sp. gr. until all iron was removed. The last washings were invariably with water. When there was intermediate treatment on the filter, it will be described with the experiment. In duplicate experiments, A and B, 125 cc. nitric acid, 1.135 sp. gr. was used as solvent and continued at gentle heat for one hour.

	A	B
Weight graphite plus silica	0.2105	0.2500
Weight of residual silica	0.0255	0.0658
	5)0.1850	5)0.1842
	3.70	3.68

The larger amount of silica in B is due to its standing at a gentle heat while A was being filtered. This strength nitric acid dissolves the most silicon according to Dr. Drown, but our experience has been that it separates on slight evaporation or on standing.

In duplicate experiments C and D, 60 cc. hydrochloric acid 1.1 sp. gr. was used as a solvent.

After complete solution 100 cc. of water was added to each and allowed to stand over night.

	C	D
Weight graphite plus silica ·····	0.3500	0.3489
Weight of residual silica········	0.1732	0.1721
	5)0.1768	5)0.1768
	3.53	3.53

The larger amount of silica separated rendered the burning of the graphite more difficult than in A and B. The amount of silica separated represents nearly all the silicon of the metal.

In experiment E, the solvent was 125 cc. nitric acid sp. gr. 1.135 added first, then immediately 12 cc. of Baker and Adamson's 40 per cent. hydrofluoric acid. Solution was perfect within five minutes at a gentle heat, after which the solution was boiled vigorously for five minutes longer, being constantly kept covered to prevent evaporation.

	E
Weight graphite plus silica·················	0.1768
Weight of residual silica·····················	0.0000
	5)0.1768
	3.53

It was thought that hydrofluoric acid would either cause more of the silicon to go into solution and remain there or volatilize it from the boiling acid.

Some glaze was taken from the beaker but no severe corrosion occurred in several treatments in the same beaker.

Intermediate washing with a mixture, one-third strong ammonia and two-thirds water, was made with the hope of dissolving any silica that might still cling to the graphite.

On burning the filter containing the graphite nothing was left in the crucible but some minute dark spots, (likely traces of elements seldom estimated in pig iron) too slight to effect the determination in question. In experiments A, B, C, and D, I feared that the silica weighed with the graphite might be hydrated and thus give too high results in graphite.

Experiment E provided means for eliminating this objection by wholly freeing from silica. Thus far no attention has been paid to the extraction of combined carbon from the graphite, except as far as the solvents used would do this work,, either by dissolving and retaining it, or evolving it. When the graphite of experiment E was washed with ammonia, after all iron was removed, brownish coloration appeared in the funnel tube during the first two washings, which suggested combined carbon in some form dissolved in ammonia. The following experiments were all started exactly the same as was E. The washing will be described along with results given.

	F	G
Weight graphite plus silica ·····	0.1818	0.1800
Weight of residual silica ········	0.0000	0.0000
	5)0.1818	5)0.1800
	3.63	3.60

Both F and G were washed with alcohol, ether, and ammonia in order. Each of the three washings brought the brownish coloration as noted in E.

	H	I
Weight graphite plus silica ·····	0.1820	0.1855
Weight of residual silica ········	0.0000	0.0025
	5)0.1820	5)0.1830
	3.65	3.66

H was washed with ammonia only. Copious brown color came. I was washed with alcohol and ether only. Brown color came.

	J	K	L
Weight graphite plus silica ······	0.1878	0.1830	0.1790
Weight of residual silica ·········	0.0000	0.0000	0.0000
	5)0.1878	5)0.1830	5)0.1790
	3.75	3.66	3.58

J was also washed with alcohol and ether only. Brown color came. K was washed with alcohol, ether, and ammonia, in order named as were F and G. It was observed that more color came with ammonia than with alcohol and ether. The alcohol-ether and ammonia filtrates of K were preserved and will receive attention later. L was washed with ammonia, alcohol, and ether in order. Scarce a trace of color came with alcohol and ether.

In none of these experiments except I was any residue left in the crucible except the dark spots previously alluded to, (soluble in boiling hydrochloric acid sp. gr. 1.20) and in that case, residual silica, showing that ammonia may be necessary to dissolve the last traces of silica, but not always necessary as proven by J.

From a comparison of results and character of washings, it is evident that ammonia is certainly as efficacious a wash as alcohol and ether for removing combined carbon or its compounds, and that where ammonia is used, alcohol and ether are unnecessary. The variation of the hydrofluoric acid results is not so great as to be inadmissible from a commercial stand-point.

This variation may be due to errors of manipulation, or to small graphitic crystals or nuggets crumbled rather than drilled from the pig and larger than the remainder of drillings thus rendering the sample of drillings not homogeneous.

These interlaced spongy particles of graphite, sometimes three mm. on a side are often noticed after solution of pig in 1.135 sp. gr. nitric acid. It may also be due to small amounts of fine graphite penetrating the inner paper and being retained by the outer paper or penetrating it also.

This phenomenon is sometimes noticed especially where retained by the second paper thus giving a dark color. Finally the chemical or mechanical action of the filtration may have disturbed the equality in weight of the two papers. Why then setting aside these sources of error and the dark spots on the crucible are we not weighing pure graphite, if the analysis be conducted as typified in experiment E? This modus operandi is shorter and free from the perplexities of the caustic potash method of Karsten. The alcohol-ether filtrate from K was evaporated to gentle dryness and formed a black mirror-like residue on the beaker, completely soluble in the ammonia wash when warmed gently. The residue from the ammonia filtrate presented the same appearance but was not soluble in alcohol and ether, but readily taken up by ammonia.

Both filtrates on evaporation threw out the dissolved matter in brownish flocks. We hope to investigate this point more thoroughly and also the action of ammonia on the total carbon of pig iron as obtained by use of the double chlorides. Such work may aid in obtaining a better knowledge of the forms of combination of carbon with iron and the forms in which it is separated under different conditions.

Attention will also be paid to the character of spots on the crucible, but these would not be common to all pig iron and might exist to a greater extent in some than in others. I may note in conclusion that all filtrations were performed by aid of the pump, and that more closely concordant results might have been obtained had the filtrations been performed upon asbestos. It would also be interesting to determine whether the silicon of the metal under this treatment is volatilized or largely in solution.

LABORATORY OF STEWART IRON CO., L'T'D.,
SHARON, PA., DECEMBER, 1893.

THE DETECTION OF STRYCHNINE IN AN EXHUMED HUMAN BODY.[1]

BY W. A. NOYES.

PROFESSOR T. G. Wormley in his book on the Micro-Chemistry of Poisons states that strychnine has never been found in an *exhumed* human body after a longer period of burial than forty-three days. His authority is the Ann. d'Hyg., April, *1881*, 359. I have not been able to find in any chemical journal a contradiction of this statement, though there is a statement in the old edition of *Watt's Chemical Dictionary* which seems to be inconsistent with it. Under these circumstances the following account of a recent toxical case may be of some interest.

On April 26th of this year Drs. W. L. Athon and O. Mitchell, of Marshall, Ill., brought to me for examination the stomach, liver and a portion of the intestines of a child who died on June

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[1] Read at the Baltimore Meeting, December 28, 1893.