SCIENCE:

Published by N. D. C. HODGES, 874 Broadway, New York.

Subscriptions to any part of the World, $3.50 a year.

To any contributor, on request in advance, one hundred copies of the issue containing his article will be sent without charge. More copies will be supplied at about cost, also if ordered in advance. Reprints are not supplied, as for obvious reasons we desire to circulate as many copies of *Science* as possible. Authors are, however, at perfect liberty to have their articles reprinted elsewhere. For illustrations, drawings in black and white suitable for photo-engraving should be supplied by the contributor. Rejected manuscripts will be returned to the authors only when the requisite amount of postage accompanies the manuscript. Whatever is intended for insertion must be authenticated by the name and address of the writer; not necessarily for publication, but as a guaranty of good faith. We do not hold ourselves responsible for any view or opinions expressed in the communications of our correspondents.

Attention is called to the "Wants" column. It is invaluable to those who use it in soliciting information or seeking new positions. The name and address of applicants should be given in full, so that answers will go direct to them. The "Exchange" column is likewise open.

IRON AND ALUMINIUM IN BONE BLACK: THEIR QUANTITATIVE DETERMINATION.

BY DR. F. G. WIECHMANN, COLUMBIA COLLEGE, NEW YORK.

The determination of iron and aluminium in bone black has thus far been commonly effected by the so-called ammonia-acetate method, which, until quite recently, has also been the favorite method employed for the determination of the constituents mentioned in mineral phosphates.

As this method, however, is open to serious objections, it was decided to test its accuracy, and to compare the results obtained with those yielded, respectively, by the method of E. Glaser,[1] and by the combination of Glaser's method with that of A. Stutzer,[2] first suggested by R. Jones[3] for the analysis of fertilizers.

For valuable analytical work performed in this connection, the writer's thanks are due his assistant, Mr. E. C. Brainerd.

The schemes of analysis used in this investigation are minutely given in the following directions: —

Method I. Acetate of ammonia process.

(This method is based on the solubility of calcium phosphate in acetic acid, and on the insolubility of the phosphates of iron and aluminium in this medium.)

1. Powder sample. 2. Dry thoroughly. 3. Weigh out 3.0 grammes. 4. Dissolve in distilled water + 25 cubic centimetres HCl (conc.), boiling gently for one hour. 5. Filter. 6. Wash residue on filter, until the wash-water no longer reacts for Cl with $Ag\,NO_3$. 7. Add excess of $Ba\,Cl_2$, boil till $Ba\,SO_4$ is granular. 8. Filter. 9. Wash the $Ba\,SO_4$ on the filter till no more reaction for Cl with $Ag\,NO_3$. 10. To filtrate and wash-waters combined add NH_4OH, until the precipitate formed begins to appear permanent. 11. Then add acetic acid to pronounced acid reaction, and boil. 12. Filter. 13. Wash the precipitate well. 14. Dry, ignite, weigh. 15. Regard the precipitate as $Fe\,PO_4 + Al\,PO_4$, calculate to $Fe_2\,O_3 + Al_2\,O_3$, and so report.

Method II. Glaser's method.

(In this process the calcium is removed from an alcoholic solution by means of sulphuric acid before the precipitation of the iron and aluminium is effected.)

1. Powder sample. 2. Dry thoroughly. 3. Weigh out 5 0 grammes. 4. Dissolve in distilled H_2O + 30 cubic centimetres $H\,Cl$ (conc.) + 10 cubic centimetres $H\,NO_3$ (conc.). 5. Make the solution up to 500 cubic centimetres with distilled water. 6. Filter. 7. Of the filtrate take 100 cubic centimetres (equal to 1.00 gramme), place in a 250 cubic centimetre flask, add 25 cubic centimetres $H_2\,SO_4$ (conc.). Shake frequently, and allow to stand for five minutes. 8. Add absolute ethyl alcohol, cool, fill up to the mark with alcohol, and shake well. 9. As volume contraction will take place, fill up to the mark repeatedly with alcohol, and shake each time. Continue this filling up to the mark until no more contraction takes place. 10. Allow the solution to stand for 12 hours. 11. Filter. 12. Of the filtrate take 100 cubic centimetres (= 0.4 gramme), place in a large platinum dish on a water-bath, and heat until all the alcohol is removed. 13. Wash the remaining solution into a beaker with 50 cubic centimetres of distilled water. 14. Heat to boiling, and then remove the flame. 15. Add NH_4OH very carefully to alkaline reaction. 16. Boil until the ammonia is completely expelled. 17. Filter. 18. Wash the precipitate thoroughly with boiling, distilled water. 19. Dry, incinerate, weigh. 20. Regard the precipitate as $Fe\,PO_4 + Al\,PO_4$, calculate to $Fe_2\,O_3 + Al_2\,O_3$, and so report.

Method III. Combination of the methods of Glaser and Stutzer.

(Stutzer's method consists essentially in precipitating the iron and aluminium, principally as phosphates, in a solution of ammonium acetate; treating this precipitate with a solution of ammonium molybdate, to remove the phosphoric acid as phospho-ammonium molybdate; filtering out this precipitate, and in the resulting filtrate precipitating the iron and aluminium as hydrates, by ammonium hydrate; drying and igniting this precipitate, weighing it as $Fe_2\,O_3 + Al_2\,O_3$, and reporting it as such.)

The following scheme, it is believed, offers all the advantages of both the Glaser and the Stutzer methods.

Proceed exactly as in Method II. up to and inclusive of section No. 18.

Then continue as follows: —

1. Place filter and contents in a beaker which contains 150 cubic centimetres molybdic solution,[4] at a temperature of about 40° C. 2. Keep the mixture at a temperature of about 65° C. for from 12 to 15 hours. 3. Filter out the precipitate. 4. Wash the precipitate thoroughly with $NH_4\,NO_3$ solution (1:10). 5. To filtrate add $NH_4\,OH$ till it is well alkaline. 6. Heat for 2 or 3 hours over a gentle flame, replacing any loss by evaporation by the addition of water and ammonic hydrate. 7. Filter out the precipitate. 8. Dissolve this precipitate from the filter with $H\,Cl$. 9. Precipitate with NH_4OH, and boil out all free ammonia. 10. Filter. 11. Wash precipitate, dry, incinerate, and weigh. 12. Regard the precipitate as $Fe_2\,O_3 + Al_2\,O_3$, and so report.

The mixture on which these three methods were tested consisted of: —

Tri-calcic phosphate	20.00 grammes
Aluminium sulphate	0.10 "
Ferrous sulphate	0.10 "

These amounts of the sulphates of iron and aluminium corresponded to 0.67 per cent of $Fe_2\,O_3 + Al_2\,O_3$, as was ascertained by analysis.

The mixture was dissolved in $H_2\,O + H\,Cl$, and made up to 500 cubic centimetres. 24.75 cubic centimetres of this solution contain 1.00 gramme of the dry substance.

In Method I., used 3.00 grammes; in Method II., 1.00 gramme; in Method III., used 1.00 gramme of the "dry substance" for analysis.

Results of Analysis.

		Method.		
		I.	II.	III.
		Per cent.	Per cent.	Per cent.
$Fe_2\,O_3 + Al_2\,O_3$	Present	0.67	0.67	0.67
	Found	0.56	0.60	0.63

Method I. has evidently yielded the least satisfactory result.

---

[1] Zeitschrift für Angewandte Chemie, 1889, p. 636.

[2] Zeitschrift für Angewandte Chemie, 1890, p. 43.

[3] Chemiker Zeitung, 1890, p. 269.

[4] Dissolve 100 grammes molybdic acid in 40 grammes, or 417 cubic centimetres of ammonium hydrate (sp. gr. 0.96), and pour the solution thus obtained into 1500 grammes, or 1250 cubic centimetres, of nitric acid (sp. gr. 1.20). Keep in a warm place for several days, decant the solution from any sediment, and preserve in glass-stoppered vessel.