

QUANTITATIVE DETERMINATION OF IRON IN BASHAM'S MIXTURE.*

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Liquor Ferri et Ammonii Acetatis is frequently submitted to be analyzed for its iron content, and there being no assay method for the preparation in the United States Pharmacopœia nor in other standard reference books consulted, I conducted the following experiments:

(A) Ten cc. of Basham's Mixture were evaporated to dryness and ignited in a platinum dish. Considerable unburnt carbon remaining the ash was moistened with concentrated HNO_3 , and again ignited. This treatment with nitric acid was repeated several times until it was thought that all the carbon had been burned off. Five cc. of concentrated HCl were then added to the ash and the dish and contents placed on a steam-bath to dissolve the iron oxide. There still being unburnt carbon the acid-soluble material was filtered through an ashless filter paper which after thorough washing was returned to the platinum dish and ignited. The carbon now being consumed there were added to the dish 2 cc. concentrated HCl and the material heated on the steam-bath until dissolved when it was added to the main solution in a bottle with a glass stopper. Two grams of KI were added, the glass-stoppered bottle and contents placed in water at a temperature of 40°C . for 30 minutes, the solution cooled and titrated with $N/10 \text{ Na}_2\text{S}_2\text{O}_3$ solution employing starch as indicator. The analysis indicated the presence of 0.1561 Gm. iron per 100 cc.

(B) Ten cc. of Basham's Mixture were diluted with about 100 cc. distilled water, ammonium chloride and an excess of NH_4OH added and H_2S passed to saturation. About 10 cc. of ether were added, the material thoroughly stirred while heating on a steam-bath and filtered through a 9 cm. quantitative filter paper (the addition of ether and thorough stirring make the precipitate settle quickly and cause a very rapid filtration), and washed with H_2S water. The precipitate was dissolved in HCl , catching the solution in a 250-cc. Pyrex beaker; the paper was washed thoroughly and the solution boiled to get rid of H_2S , when it was oxidized with HNO_3 , the iron precipitated with redistilled NH_4OH ; the material was filtered, washed, dried, ignited and weighed as Fe_2O_3 . The weight of $\text{Fe}_2\text{O}_3 \times 0.70 \times 10 =$ weight of iron as metallic iron per 100 cc. The analysis indicated the presence of 0.1650 Gm. iron per 100 cc.

To the crucible containing the iron oxide, 5 cc. concentrated HCl were added and the material heated on a steam-bath until solution was complete after which it was transferred to a bottle with a glass stopper, KI added and the analysis completed as under (A). The analysis indicated the presence of 0.1641 Gm. iron per 100 cc.

(C) To 10 cc. of Basham's Mixture in a weighed platinum dish there were added 5 cc. concentrated H_2SO_4 , and after evaporating the excess of H_2O on a hot plate, the dish and contents were heated to a dull red heat until SO_2 fumes ceased to be evolved, cooled and weighed. The weight of $\text{Fe}_2\text{O}_3 \times 0.70 \times 10 =$ weight of iron per 100 cc. The analysis indicated the presence of 0.1645 Gm. iron per 100 cc. To the dish containing the iron oxide 5 cc. of concentrated HCl were added and the analysis completed as under the iodometric method in (B). The analysis indicated the presence of 0.1641 Gm. of iron per 100 cc.

(D) Ten cc. of Basham's Mixture were heated in a 500-cc. Kjeldahl flask until excess of moisture was expelled, 20 cc. of a mixture of equal quantities of HNO_3 and H_2SO_4 added and the material heated until all organic matter was destroyed, additional quantities of HNO_3 being added from time to time. When the reaction was complete the material was transferred to a beaker, heated to boiling, made alkaline with NH_4OH to precipitate $\text{Fe}(\text{OH})_3$, filtered, washed and dissolved in 5 cc. concentrated HCl , the solution and washings being received in a bottle with a glass stopper, KI added and the analysis completed as under (A). The analysis indicated the presence of 0.1779 Gm. iron per 100 cc.

(E) Ten cc. of Basham's Mixture in a 500-cc. Kjeldahl flask were heated until excess of moisture was expelled, after which 20 cc. concentrated H_2SO_4 and 10 grams K_2SO_4 were added, the material heated until all organic matter was destroyed, transferred to a beaker heated to boil-

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ing, the iron precipitated with NH_4OH , the precipitate washed and dissolved in 5 cc. concentrated HCl , the filtrate and washings being collected in a bottle with a glass stopper, KI added and the analysis completed as under (A). The analysis indicated the presence of 0.1733 Gm. iron per 100 cc.

(F) Following the method I published¹ 10 cc. of Basham's Mixture were diluted with HO_2 *q. s.* 500 cc. Five cc. of this solution were evaporated and ignited in a platinum dish, 5 cc. HCl (1-1) added, the material boiled an instant, poured into a 100-cc. Nessler tube, water added *q. s.* 100 cc., 3 drops of KMnO_4 solution (5-1000) added to oxidize the iron, and after a few minutes 10 cc. of KSCN solution (20-1000) were added, the color produced being immediately compared with the iron standards. The analysis indicated the presence of 0.160 Gm. iron per 100 cc.

(G) Twenty-five cc. of Basham's Mixture were added to a bottle with a glass stopper, 2 grams of KI and 5 cc. concentrated HCl were added and the material placed in H_2O at 40°C . for 30 minutes when it was cooled and titrated with $N/10$ $\text{Na}_2\text{S}_2\text{O}_3$ solution employing starch as indicator. The analysis indicated the presence of 0.1550 Gm. iron per 100 cc.

The results by all methods were as follows:

- A—0.1561 Gm. iron per 100 cc. of Basham's Mixture.
- B—0.1641 Gm. iron per 100 cc. of Basham's Mixture.
- C—0.1641 Gm. iron per 100 cc. of Basham's Mixture.
- D—0.1779 Gm. iron per 100 cc. of Basham's Mixture.
- E—0.1733 Gm. iron per 100 cc. of Basham's Mixture.
- F—0.1600 Gm. iron per 100 cc. of Basham's Mixture.
- G—0.1550 Gm. iron per 100 cc. of Basham's Mixture.

SUMMARY.

1. Due to the large amount of sugar and glycerin which Basham's Mixture contains it is practically impossible to ignite the material without leaving a large amount of carbon which holds iron in such a manner that boiling with HCl does not remove it.

Treatment of the ash by repeatedly moistening it with HNO_3 evaporation and ignition greatly aid but still fail to completely destroy the carbon. If carbon is not entirely consumed the results are low due to the fact that iron is held in such a manner that HCl fails to remove it.

2. Methods D and E give concordant results but are higher than the results of other methods. Notwithstanding the fact that the HNO_3 , H_2SO_4 and K_2SO_4 employed in making the analyses were "analyzed chemicals" we tested them and found that they did not contribute to the high results.

Methods A and G yielded practically the same results.

Method F yielded results between those obtained in A, G, B and C; B and C yielded identical results.

3. Method G placing 25 cc. of the sample in a bottle with a glass stopper, adding 5 cc. HCl , 2 grams KI and keeping the material at 40°C . for half an hour, cooling and titrating with $N/10$ $\text{Na}_2\text{S}_2\text{O}_3$, employing starch as indicator, is not only accurate but easily applied and is suggested for inclusion in the U. S. P. X as a method of assay for Iron in "Liquor Ferri et Ammonii Acetatis."

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¹ "Colorimetric Estimation of Iron in Pharmaceutical Preparations," *JOUR. A. PH. A.*, 5, 517, 1916.