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The lack of iodine in the food of the inhabitants of the endemic goitre regions of this continent has resulted in the addition to various foods of inorganic iodine calculated to supply this deficiency. The addition of inorganic iodine, whether because of its inferiority, or because of the tendency to overdosage, is generally conceded to be not as successful in the treatment of endemic goitre as the natural iodine containing foods. The iodine content of various foods has been the object of extensive research, and marine foods especially have received their share of attention because of their relatively high iodine content. The general lack of agreement in the results of analysis of the same forms for iodine throws considerable doubts on the accuracy of the methods now in vogue, and it seems necessary to revise at least all values obtained before 1924.

All analytical procedures used in estimations of traces of iodine in organic material consist of the same main features. The method developed by the author differs only in details from those employed by earlier investigators. It consists of three parts.-

(1) Complete ashing of the organic material in an atmosphere of oxygen in a closed system.

(2) The trapping of all gases of combustion in a collection train, consisting of alkali washers and a Cottrell precipitator.

(3) The recovery of the iodine from the ash, and the collection train, and its quantitative determination.

To be able to judge the relative merits of the various methods of ashing it is necessary to have at one's command a dependable method for the recovery and quantitative determination of the iodine from the ash. In the literature a great number of methods for this purpose have been described but none of them are without fault. They may be briefly classified as follows:

(1) Methods using an organic solvent (alcohol) to extract the iodine from the ash.
  (a) Ultimate determination colorimetric.
  (b) Ultimate determination done by titration.

(2) Methods using water to extract the iodine from the alkaline ash.
  (a) Ultimate determination colorimetric.
  (b) Ultimate determination done by titration.

As regards the use of alcohol for the extraction of the iodine from the ash some investigators have found a serious error arising in estimations based on Fellenberg's method on account of the incompleteness of the alcoholic extraction of the potassium iodide. Thus Andrew (1) and Adolph and Shen-chaochen (2) found it necessary to multiply their experimental results by a constant which was not the same in both cases, while Aitken (3) finds the use of a constant unnecessary.

The extraction of the iodine from the alkaline ash by means of water has the disadvantage of dissolving a number of other compounds, as well as the iodine, which if left in the solution till the end of the procedure may cause considerable error.(4).

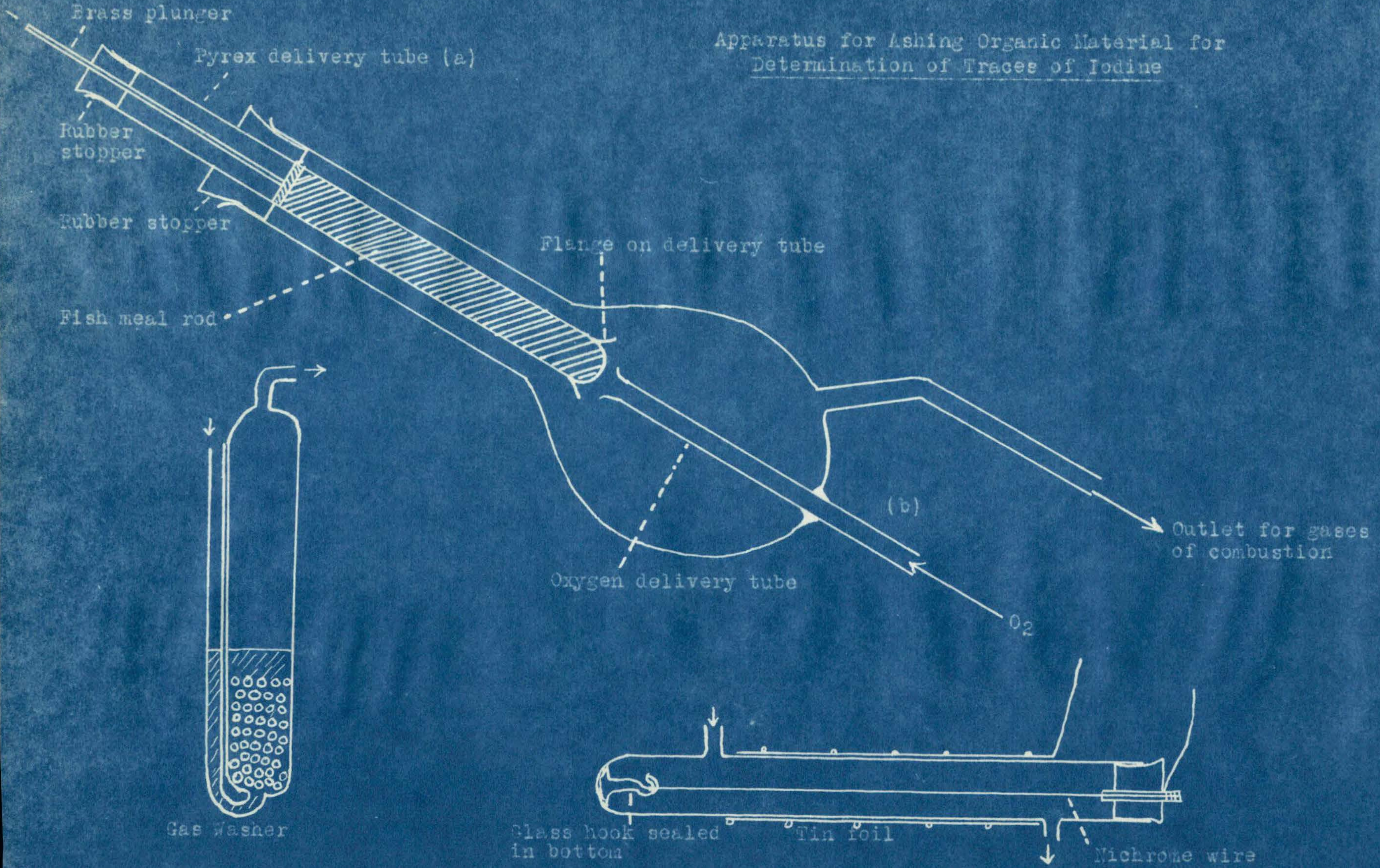
In comparing the relative merits of the colorimetric and titration methods of estimating iodine it must be remembered that although the titration method is capable of much greater accuracy, due to the quantitative multiplication of the titratable quantity of iodine when potassium iodide is added to a solution of potassium iodate, any error (5) that may arise from the presence of oxidizing compounds in the solution is multiplied in proportion to the number of equivalent there are of these oxidizing agents.

In the method of estimation devised by the author an attempt was made to combine the good points of some of these methods while as many as possible of the sources of error were eliminated.

The complete ashing of the muscle proved a very difficult task. The method followed in preparing the muscle for ashing was as follows. The fillets cut from the fresh fish were ground up in a meat chopper, and mixed with a solution of ½% of their weight of Na_2CO_3 and then dried in an oven at 70°C. The dry mass was then ground in a mill to a fine powder, which on analysis gave a water content of 5-7% water. The apparatus most commonly used for ashing of organic material for iodine analysis were not found to be applicable to this muscle meal, although in case of commercial meal they gave satisfactory ashing. This undoubtedly was due to a greater amount of salts in the commercial meal which acted as a filler and facilitated the ashing. The meal when ashed in an apparatus which was a slight modification of that of G. M. Karns (5) formed a tarry mass that "froze" the apparatus and extinguished the flame. The meal in order to ash properly apparently needed a very high concentration of oxygen. An apparatus was therefore built with the object in view to have a maximum concentration of oxygen at the place of ashing. The drawing which follows is self explanatory. The muscle meal was stuffed into a tissue paper cylinder which had been made to fit the pyrex tube (a), the tube was then put in place in the combustion flask and the meal cylinder pushed in by means of the brass plunger until its end was level with the flanged out end of the tube (a). The oxygen which was obtained from a cylinder was then led in through the tube (b) which directed the stream of gas on to the face of the meal cylinder. When all the air had been displaced from the combustion flask the tube (c) was pulled out and a match applied to the end of the meal cylinder, and when the meal began to burn in the oxygen atmosphere the tube was pushed in place again. The flow of oxygen was regulated so as to give optimum burning (1-1½ litres per minute). As the meal cylinder gradually burned at the end it was pushed further in by means of the plunger.

In the beginning the same difficulties were experienced as with the apparatus of G. M. Karns, although to a much less extent, but they were overcome by adding to the meal 20% of its own weight of water just before it was put into the cylinder and by never having more than 10-15 grams of meal in each cylinder. It was found that the water had to be added just before the beginning of each run or otherwise it rather retarded than aided the ashing. In this manner a smokeless burning was obtained, and the snow white ash which resulted deposited in beautiful patterns on the walls of the combustion flask. However it was felt that by having a platinum collar on the end of the tube (a) the time of ashing, which was 1-1½ hours for 25 grams of meal, might be cut down. Attached to the outlet for the gases of combustion was a collection train consisting of a washer of the type shown on the diagram containing 5 cc. of 10% sodium hydroxide solution and a Cottrell precipitator, as shown also in the diagram, through which was passed a current with a potential difference of 10,000 volts, and then 3 more washers of the same type as the first one.

Apparatus for Ashing Organic Material for Determination of Traces of Iodine



On completion of a run the apparatus was washed out quantitatively and the washings and the alkaline solution from the washers evaporated on a water bath. When the salts started to come out of solution the evaporation was stopped, the solution filtered and neutralized. It was then ready for analysis:

25 cc. of the liquid containing the iodine was placed in a 100 cc. separatory funnel and 2 drops of concentrated HCl and 20 cc. of purified carbon tetrachloride added. 3 drops of 0.1N arsenious acid were added, and the funnel was shaken vigorously and then allowed to stand for a few minutes. A few drops of nitrosyl sulphuric acid were added to oxidize the excess arsenious acid and the iodide to iodine, and the separatory funnel shaken until all the iodine has passed into the carbon tetrachloride layer. The separatory funnel was allowed to stand for a few minutes, and the carbon tetrachloride then allowed to run into another separatory funnel of the same size as the first which contained chlorine water made up to a volume of 15 cc. with distilled water. The remaining liquid was treated with two successive portions of carbon tetrachloride of 15 cc. each and the carbon tetrachloride allowed to run into the second separatory funnel. The second separatory funnel was shaken with the chlorine water until all the iodine in the carbon tetrachloride was oxidized to iodate and dissolved in the aqueous solution. Most of the carbon tetrachloride was run off but the remainder was washed into a beaker with the aqueous solution. Hydrochloric acid 1:1 was added to the solution in the beaker until a green colour due to the chlorine was observed and then a few more drops.

The solution was boiled down to a small volume on a water bath during which process the chlorine and carbon tetrachloride were driven off. A small crystal of Kl was added, then a few drops of hydrochloric acid, and the solution titrated against sodium thiosulphate.

In order to test this procedure, two solutions N/10 and N/1000 of potassium iodate were made up. 5 cc. lots of each of them were taken and run through the procedure. Two 5 cc. lots of each solution were kept for control and titrated against standard sodium thiosulphate along with the others. The results obtained are shown below.

Normality of K1O3	Titre for controls	Titre for samples
N/10	4.590 cc.	4.571 cc. 4.574 cc.
N/1000	4.54 cc.	4.52 cc. 4.57 cc.

5 cc. lots of potassium iodate solution N/100 were now added to small sample of muscle meal (5 grams) and the meal ashed in the apparatus designed by the author, and the ash carried through the procedure as described. In some cases the washings and ash from the whole apparatus were analyzed in one lot, in others each individual part of the apparatus was analyzed separately. The following were the results obtained:

Parts of Apparatus and control	Titre $Na_2S_2O_3$	Apparatus and Control	Titre $Na_2S_2O_3$
Control	3.125 cc.	Control	4.578 cc.
Combustion flask	.206-	the whole apparatus	2.598-
Cottrell precipitator	.046-		
1st. washer	.452 cc.		
2nd. washer	.080-		
3rd. washer	.024-		
4th. washer	.023-		

It is quite obvious from these results that some of the iodine is being lost. It has been proven that the method of recovery of the iodine from the ashes is sound, and to all appearances the ashing of the muscle meal was as thorough as could be wished for. The only possible source of error must, therefore, be in the collection train. This view is also upheld by the comparatively great amount of iodine found in the last washer (2% of the total). All earlier investigations have made use of much more efficient collection trains, but they mostly have been dealing with a much faster flow of gases than the author had to contend with.

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