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SOME OBSERVATIONS ON E. C. KENDALL'S METHOD OF ESTIMATING IODINE IN THYROID PREPARATIONS.

S. B. KUZIRIAN.

In presenting this short note on Kendall's method of estimating iodine in thyroid preparations, it is perhaps desirable to give a short review of the method. For the details of manipulation of the method the writer recommends a careful study of the original paper.¹⁻²

The procedure is essentially to oxidize the organic matter by fusion with sodium hydroxide with the aid of small quantities of potassium nitrate in a nickel crucible. The fused mass is dissolved in hot water, one c.c. of a ten per cent solution of sodium bisulphite and a few drops of methyl orange are added. It is acidified with 85 per cent sirupy phosphoric acid. The hydroiodic acid produced is oxidized to iodic acid by addition of a few drops of liquid bromine. The whole mass is placed on a hot plate and with the aid of talc powder is boiled to remove excess of bromine. Ten to fifteen drops of a solution of sodium salicylate are added to take up any bromine that might have escaped expulsion, the mixture is then cooled, after which five c.c. of a ten per cent solution of potassium iodide and three to five c.c. of sirupy phosphoric acid are added and the iodine liberated is titrated with 0.02 N. sodium thio-sulphate.

While Kendall's modified method was used by the author on this paper for analysis of a large number of feeds and samples of ewes' milk for their iodine content, some difficulties were met with on account of interference of ash and added bromine. Forbes, Beegle, and others, seem to have encountered the same difficulties, as shown in their bulletin.³

Satisfactory results were obtained, however, when the following details were carefully observed:

1. In destroying organic matter, it is advantageous to use more sodium hydroxide and less potassium nitrate to obviate the formation of large quantities of nitrite, which tends to liberate iodine.

¹E. C. Kendall, Jour. Biol. Chem., 19, pp. 251-254, 1914.

²E. C. Kendall, Jour. Amer. Chem. Soc., 34, 894, 1912.

³Forbes, Beegle, and others, Bul. 299, Ohio Agr. Exp. Sta., Wooster, O.

2. Organic substances with high ash content often contain iron or some other interfering element, which ought to be eliminated before reliable results can be obtained. According to the author's experiences, filtration at this point did not altogether eliminate the interfering action of ash. When, however, the sodium hydroxide fusion melt was taken up with hot water, a few grams of sodium bicarbonate (C.P.) and some talc added, brought to boiling and kept boiling for a few minutes, set aside for two hours and filtered, the filtrate was free from interfering basic elements.

3. In acidifying the above filtrate with 85 per cent sirupy phosphoric acid, the author's experience was well in accord with Forbes, Beegle, and others, who found that one or two drops of phosphoric acid in excess was insufficient to assure expulsion of all bromine. An addition of even one and a half to two cubic centimeters in excess would boil off all of the bromine without causing any loss of iodine. This was shown by taking an aliquot of accurately standardized alcoholic solution of iodoform and estimating iodine according to Kendall's method. No loss of iodine occurred when two cubic centimeters of acid was added in excess.

4. In boiling off the bromine it was made a practice to dilute the filtrate to over 400 cubic centimeters and boil down to 250 cubic centimeters, thus assuring a complete expulsion of bromine.

Under the conditions specified in this short note the author found the method applicable for tankage, roughage and other organic substances with a high percentage of ash.

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*Bulletin 299, Ohio Agr. Exp. Sta., p. 422.