LIV. A FURTHER NOTE ON THE DETERMINATION OF IODINE IN BLOOD.

By HARRY ARTHUR ASHWELL AITKEN.

From the Medical School, University of Otago, New Zealand.

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In a recent paper [Aitken, 1930] a method of estimating iodine in blood was described, the essential feature of which was that the iodine was concentrated into a very small volume of solution before being titrated. The titration was performed with a 0.1 cc. pipette, and gave satisfactory results provided the blood was not abnormally low in iodine. However, it is evident that in the case of a blood containing say only 40 γ of iodine per kg. the error involved in the use of a 0.1 cc. pipette and $N/1000$ thiosulphate will be at least 5 %. In order to eliminate such an error the author has devised a convenient and accurate micro-burette in which the standard solution floats on a thread of mercury, the position of the latter being adjusted by means of a fine screw projecting into a mercury reservoir.

The burette, shown in Fig. 1, was made from the stem of a discarded 200° thermometer which had a capillary of internal diameter about 0.18 mm. One end of this was drawn out to a tip *H* and bent over at *G* as shown. The other end of the capillary was opened out slightly and sealed into the steel chamber *A* by means of cement. *B* represents a "stuffing-box" (also of steel) containing vaselined packing which is used to ensure that the chamber *A* is mercury-tight. The "stuffing-box" is screwed on to the chamber *A* which is threaded at the lower end for this purpose, the pitch of the screw being about 0.8 mm. The chamber *A* contains mercury as shown by the black shading. The lower end of the "stuffing-box" (but not of the chamber *A*) carries an internal thread to accommodate the fine steel screw *D*, which is turned by means of the milled head *E* and serves to control the level of the mercury in the thermometer capillary. The pitch of this screw is 0.33 mm. and its diameter 1.4 mm., so that one complete turn of the milled head *E* expels about 0.0005 cc. of mercury from the chamber, corresponding to about 10 divisions on the stem. As regards the other dimensions of the various parts, they may

Fig. 1.

be gathered from the figure if it is remembered that in the actual apparatus the 10° graduations are 2 cm. apart.

The micro-burette, after being filled with mercury (which was done by filling the upper part of the "stuffing-box" with mercury and then attaching it to the chamber), was very carefully calibrated by drawing in threads of mercury at *H*, measuring their lengths in scale divisions and then weighing the threads. A table of corrections for variations in the diameter of the capillary was also drawn up, though these corrections were really negligible. The capacity of the burette showed that *N*/100 thiosulphate would be the most convenient standard to use, in which case 20 divisions on the scale corresponded to 0.21 γ of iodine in the sample.

The burette is filled with standard solution by immersing the tip *H* in a small vessel of the solution and rotating the milled head *E* forward until a small globule of mercury forms at the orifice; the head is then reversed and the standard solution drawn in to any convenient level. The tip is rinsed with distilled water, dried lightly with filter-paper and immersed in the solution in the titration-tube, and the titration is carried out by turning the milled head. Very delicate control is thus obtained as it is possible to raise the mercury level slowly through fractions of a scale division. In practice both the burette and the titration vessel are clamped to a burette stand, the whole being shaken bodily to ensure mixing during titration. The smallness of error arising in the burette can be gathered from the fact that it is easy to read to a quarter of a scale division on the stem and, using *N*/100 thiosulphate, this corresponds to 0.00262 γ of iodine in the sample.

Apart, however, from inaccuracies in measurement, some investigators have found that a large error arises in estimations based on Fellenberg's method on account of the incompleteness of the alcoholic extraction of the potassium iodide. Thus Andrew [1930] finds it necessary to multiply his experimental results by 3/2, while Adolph and Shen-Chao Chen [1930] use the factor 100/85. Andrew [1930, p. 273] finds that there is little or no loss involved in the first alcoholic extraction, while there is a considerable loss in the second. This discrepancy, though perhaps partly due to losses by volatilisation, is probably mainly to be accounted for by differences in the conditions under which the extractions were performed. The present author has had no difficulty in obtaining a quantitative recovery of iodide, provided there is sufficient carbonate present to provide a paste which can be properly treated. In recent work he has found it convenient when performing the second alcoholic extraction to imitate exactly the conditions of the first alcoholic extraction. Small nickel crucibles (about 25 cc. capacity) are used instead of platinum dishes. To the first alcoholic extract in these crucibles is added about 0.4 cc. of the potassium carbonate solution and the residue after evaporation is incinerated, the small crucible being placed in a slightly larger one to avoid the risk of overheating. The residue is carefully moistened with carbonate solution so as to form a paste when rubbed with two small glass rods

flattened at the ends, and successive small amounts of alcohol are employed for the extraction. Thus the only difference between the extractions is one of scale, and it is possible to perform them with equal completeness.

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