

# SCIENTIFIC PROCEEDINGS.

## Minnesota Branch.

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### The Determination of Iodine in Natural Waters.

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A large dishpan is filled with the water, 1 gram of sodium hydroxide is added and evaporated over a stove or gas burner. As the water evaporates, more is added until 100 litres have been added and evaporated to about 1 litre. After the addition of 2 drops of 60 per cent NaOH solution, it is filtered into a porcelain evaporating dish, evaporated to dryness, and heated to burn the organic matter, either over a free flame or in a furnace.\* The temperature should not rise enough to make the dish luminous. It is cooled, 25 cc. of absolute alcohol added, and the residue rubbed with a pestle. The alcohol is decanted through a filter and 10 cc. more added. The process is repeated until 100 cc. have been added. The filtrate is evaporated in a porcelain dish after the addition of 1 drop of 60 per cent NaOH solution, and enough water to keep it in solution. When the volume has reached a convenient size, it is transferred to a platinum or nickel dish or crucible, and evaporated to dryness. The residue is dissolved in 2 cc. of distilled water, transferred to a small beaker, and after the addition of two drops of 0.1 N sulfuric acid, it is neutralized with phosphoric acid, using an indicator paper made by drying an alcoholic solution of brom-phenol-blue or methyl orange on ash-free filter paper. The solution is boiled to remove

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\* Danger of overheating may be avoided by burning in a combustion tube with one end bent down into an NaOH solution to absorb iodine that may escape. The arrangement is shown in *J. Biol. Chem.*, 1924, lx, 293, except that a side-neck test tube and suction allow the cold end of the combustion tube to be left open.

the  $\text{CO}_2$  and  $\text{SO}_2$ , and cooled. The volume is made up to 10 cc. in transferring to a 12 cc. separatory funnel having a 10 cc. graduation mark. One cc. of purified carbon tetrachloride, and 1 or 2 mg. of sodium nitrite are added, and the separatory shaken 200 times.† Eighty-five ninety-fifths of the iodine passes into the carbon tetrachloride, and colors it pink or violet, but it is cloudy with water droplets. It is run into a 1 cc. glass stoppered bottle, centrifuged to remove the water and a colorimetric match made. Ten mg. of iodine are dissolved in 100 cc. of purified carbon tetrachloride, and into 10 cc. glass stoppered bottles are placed 1 to 10 cc., respectively, of this solution of iodine, and the volume made up to 10 cc. in each with purified carbon tetrachloride. Then 1 cc. glass stoppered bottles of the same size and shape as that used to centrifuge the unknown are filled with these different dilutions of the iodine solution. They contain 0.001 to 0.1 mg. of iodine per cc. The unknown is compared with these known solutions colorimetrically in order to determine the quantity of iodine extracted, but if there is more than 0.1 mg., the unknown solution may be diluted quantitatively to bring it within the range of the known solutions. Since only 85/95 of the total iodine were extracted in the separatory funnel, it would be necessary to multiply the result by 1.115 in order to obtain the total iodine of the sample, but this is usually superfluous with the above bottles. If a more accurate determination is desired, a micro-colorimeter made by the Bausch and Lomb Optical Company should be used. In this case only one standard (containing 0.1 mg. of iodine per cc.) is used. About 2 per cent of the iodine is lost by filtering the sample of water (that is being evaporated) at the time it reaches the volume of 1 litre, but a great deal of time is saved and the procedure is justified. Although the colorimetric determination may be made within 1 per cent by the use of the micro-colorimeter, taking

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† Technical carbon tetrachloride may be sufficiently purified by coloring it with bromide or chlorine, allowing to stand in a glass bottle in sunlight for a week, washing out the chlorine or bromide with NaOH solution, water and then with distilled water, syphoning off the water, filtering through paper flooded with  $\text{CCl}_4$  to remove water droplets, and finally through a layer of plaster of Paris and distilling, rejecting the cloudy first portion of the distillate. It may be tested by making up a standard solution of iodine, letting it set for a week and comparing it with a fresh solution as mentioned above. If the color fades, the carbon tetrachloride still contains a reducing substance. This may sometimes be removed by shaking it with a dilute solution of nitrous acid made by adding a little sodium nitrate to very dilute hydrochloric acid or adding a few drops of nitrosyl sulphuric acid to a large volume of distilled water. Nitrosyl sulphuric acid may be made by adding starch to nitric acid in a retort and distilling over the mixture into concentrated sulphuric acid.

the average of 5 or more readings, the absorption coefficient is not known so accurately. It is difficult to reach an absolute equilibrium with a separatory funnel. The method of repeated extractions in a separatory funnel may be used, each extraction estimated separately and the total added together. If it is desired to titrate the iodine rather than to use the colorimetric method, run 10 cc. of distilled water into a separatory funnel, add one drop of 0.1 normal sulfurous acid and the carbon tetrachloride containing the iodine, and shake 200 times or until all of the iodine is reduced to iodide and passes into the water, run off the carbon tetrachloride and transfer the water to a beaker, make volume 100 cc., acidify with 1 cc. of 50 per cent phosphoric acid, oxidize with chlorine or bromine water. Boil off the bromine or chlorine, transferring to a dry beaker to prevent bumping, and reducing the volume to 50 cc., add 2 cc. of KI and some starch solution and titrate with 0.001 N thiosulphate with a micro biuret, and divide the result by 6. This is a complete report.

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### The Prenatal Growth of the Human Pancreas.

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The ponderal growth of the pancreas, with respect to body-weight, has been studied from 319 observations on human fetuses, varying from 26 to 4380 gm. in total body-weight. When the weight of the fetal pancreas is plotted against the weight of the body as a whole, it follows the course of a straight line which may be approximated by the expression:

$$PW = 0.0010335 BW + 0.19 \quad (1)$$

where "PW" is the pancreas weight in grams and "BW" is the total weight of the body in grams. This formula was computed from the means of 500 gm. ranges of body-weight, from 0 to 4500 gm. inclusive, by the method of means (weighting by the square root of the number of cases in each interval). The calculated values thus obtained show a mean, weighted, absolute, deviation of 0.123 gm. and a mean, weighted, relative deviation of 6.07 per cent from the observed averages. A similar relationship to total body-weight is characteristic of the weights of many of the other organs and parts of the body.