ART. XX.—*On the detection of Iodine;* by M. CAREY LEA.

WHERE iodine exists in the form of hydriodic acid, or the iodid of a base, two methods are commonly employed to put it into a condition to be detected by the starch test. One of these is by the action of nitric acid, the other by chlorine or bromine water. The latter is the more delicate, but has the disadvantage that if the chlorine or bromine be added in excess, the reaction is missed.

It occurred to me while engaged in testing for iodine, that the facility with which that body is eliminated from its hydrogen and metallic combinations by *chromic acid* would make the latter substance a valuable means of bringing about the starch reaction, and a few experiments completely confirmed this view.

If, for example, we take an extremely dilute solution of iodid of potassium, such that the addition of nitric acid and starch produces no perceptible effect, the further addition of a single drop of very dilute solution of bichromate of potash will instantly bring about the characteristic reaction.

When chlorhydric acid is substituted for nitric, the effect of the bichromate is (as was to be expected) still more marked. The test has then the full delicacy at least of the chlorine test, with this great advantage, that an excess of the reagent does not prevent the reaction.

As to the delicacy of this test, the following observations were made.

With solutions of iodid of potassium up to one hundred thousandth (1 : 100,000) the precipitate was abundant, becoming less blue and more tawny as the dilution increased. Beyond this point the distinctness rapidly fell off. The indications were observable at one-four-hundred-thousandth. With a solution of one-eight-hundred-thousandth it was doubtful whether any effect was evident though still it was thought that a darkening was produced.

The experiment can be made in two ways, according to the result desired.

If it is wished to observe the effect of the chromic acid in increasing the delicacy of the indication, add the acid and starch to the very dilute solution of iodid, and then when the extreme dilution is such that no reaction appears, a drop of solution of bichromate instantly produces it.

But in employing the reagent in the search for iodine, add the starch to the liquid to be tested, stir it up, add a drop of dilute solution of bichromate, enough to communicate a pale yellow color, and finally add a few drops of chlorhydric acid. The test is then the production of the characteristic precipitate,

or in case of great dilution, approaching to a half-millionth, merely a tawny shade given to the solution.

It seems scarcely necessary to say that if a very great excess of acid is used, and too much bichromate, the starch may be made to reduce the bichromate. Even this, however, cannot deceive, for a bluish-green solution is thereby produced, whereas the indications of iodid are in the order of their strength: blue precipitate, tawny precipitate, tawny solution. Unless in the case of very exceptional dilution above spoken of, a well marked blue precipitate is always obtained.

The examination of the delicacy of the reaction with very dilute solutions was made at a temperature of 65° F. or thereabouts. This fact requires to be taken into account, as according to some experiments of Fresenius to be found in the Jahresbericht for 1857, the delicacy of the starch test increases as the temperature falls, so that at 0° C. a fainter trace can be rendered evident than at 12° C., and so on: the difference is asserted to be material. Fresenius's experiments were made with sulphuric acid and hyponitric acid, and the delicacy of the reaction obtained by him at corresponding temperatures seems to fall a little short of the above.