APPARENT DETERIORATION OF DONOVAN'S SOLUTION.*

BY JOSEPH ROSIN.

The U. S. P. IX gives a rubric, and assay methods for the constituents of Solution of Arsenous and Mercuric Iodide. The arsenous iodide is assayed by titration with tenth-normal iodine in the presence of sodium bicarbonate. This method is based on the oxidation of trivalent arsenic to pentavalent by the iodine. It is used for the determination of arsenic trioxide and it was also used in the U. S. P. VIII for assaying arsenous iodide.

Having had occasion to examine a few samples of Donovan's Solution, it was noticed that the arsenous iodide was lower than expected, while the contents of mercuric iodide were fairly well within the limits required by the U. S. P. To account for this discrepancy was a simple matter. Blame the chemical. Yet to a less prejudiced mind other possibilities might suggest themselves; the solutions were not properly prepared, or the arsenic became partly oxidized, the oxidation being aided, perhaps, by the mercuric iodide. Accordingly, a solution of arsenous and mercuric iodide was prepared according to the directions of the U. S. P. with the only difference that the solution was made up to a liter instead of 1000 Gm. as directed in the U. S. P. The arsenous iodide used for this solution tested 99.4 percent by titration with iodine. The mercuric iodide complied with U. S. P. requirements and tested 99.6 percent. Immediately after preparation, 20 mils of the solution were titrated for arsenous iodide by the U. S. P. method. It was then set aside and, at intervals, 20 mil portions withdrawn and assayed for arsenous iodide by the same method. The solution was kept in a dark amber-colored bottle in diffused light. At no time did the solution assume a darker color than it was when freshly prepared—pale yellow.

After a few observations were made with this solution designated I, another solution, II, was prepared with the same materials and tested in the same manner as solution I. 20 mils of these solutions should have required 8.72 mils tenth-normal iodine.

I.

Age of solution.	Mils $\frac{N}{10}$ iodine consumed by 20 mils of solution.
Freshly prepared	8.68
1 day	8.62
3 days	8.55
7 days	8.52
20 days	8.42
4 months	7.68
8 months	5.06
1 year 11 days	3.04

II.

Age of solution.	Mils $\frac{N}{10}$ iodine consumed by 20 mils of solution.
Freshly prepared	8.66
1 day	8.62
7 days	8.54
30 days	8.38
55 days	7.90
70 days	7.66

These experiments show that the arsenous iodide in this solution gradually undergoes oxidation and is therefore not shown by the U. S. P. assay. The maximum rate of oxidation appears to be within a day or so of its preparation. Among other conditions, the temperature probably also influences the rate of oxidation.

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Since the efficiency of the solution depends on the amount of arsenic it contains and probably not on its state of oxidation, the deterioration is thus only apparent. Yet if a solution several months old, as it is likely to be in a drug store, is examined by the official method it may cause unjust annoyance.

To determine the total arsenic present in Donovan's Solution, irrespective of its state of oxidation, the well-known Gooch-Browning method for determination of arsenic can be used to advantage. In this method all of the arsenic is first reduced to the arsenous condition by potassium iodide and sulphuric acid (= hydriodic acid) which is then titrated with standard iodine. The details of the method as applied to Solutions of Arsenous and Mercuric Iodide are as follows:

Transfer 25 mils of the solution, accurately weighed, into a 500 mil Erlenmeyer flask, add 4 mils concentrated sulphuric acid and 1 Gm. of potassium iodide, dilute to about 100 mils and gently boil until the volume is reduced to about 40 mils or until the solution is of a pale yellow color. Cool, dilute to about 200 mils with water, add a little starch solution and just discharge the blue color by the addition, drop by drop, of tenth-normal sodium thiosulphate. Add to the decolorized mixture 20 percent sodium hydroxide solution until it is slightly alkaline to litmus paper, then make at once slightly acid with diluted sulphuric acid, cool if necessary, then make again alkaline with sodium bicarbonate and titrate with tenth-normal iodine using starch as indicator.

Tested by this method after the last experiments recorded above, 20 mils of each of the solutions consumed 8.68 mils of tenth-normal iodine.

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INFLUENCE OF VISCOSITY ON THE EMULSIFICATION OF OILS.

BY CHARLES H. STOCKING.

The ease with which the emulsification of an oil may be brought about, and the permanence of the finished product depend upon a number of factors. With the view to establishing, if possible, a "viscosity rule" for manufacturing permanent emulsions from fixed oils the author selected a number of the more commonly used oils, made viscosity tests with an Engler Viscosimeter and then emulsified the oils by the Continental Method, producing emulsions varying in strength from 10 to 60 percent.

The classification of the oils according to their viscosity was determined by comparing the rate of outflow under definite conditions (same initial pressure and same temperature) with the rate of outflow of water from the viscosimeter. The quotient of the time of outflow of 200 mils of oil divided by the time of outflow of 200 mils of water at 20° C. is taken as the measure of the viscosity. This quotient is known as the Engler degree.

The following table shows the results of the tests on the oils selected: