

used, and second, the complete extraction of this chlorinated lime by the amount of water prescribed.

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(To be continued.)

## CHEMISTRY AND PREPARATION OF DECOLORIZED TINCTURE OF IODINE.

BY SIMON MENDELSON.\*

A half-century ago, the colorless tincture was highly recommended for its external application on account of its discutient propensities, and the supposed repulsive tendencies further ascribed to the tincture placed it in position as an indicated remedy by internal administration in general inflammatory affections.<sup>1</sup>

The preparation is practically devoid of the therapeutic virtues ordinarily attributed to elementary iodine, nevertheless constitutes a galenical of staple demand in pharmaceutical practice.

The method of preparation, as prescribed in the N. F. IV,<sup>2</sup> represents a totally inadequate procedure for production of the commodity upon a more or less extensive scale.

Numerous conditions and difficulties, encountered in the course of digestion in particular, contribute to the uncertainty of quantity in the final yield of finished product. The application of heat to facilitate solution and accelerate decolorization, presents a degree of danger due to the liability of precipitation of varying amounts of nitrogen iodide occasioned by the contact of ammonia and iodine. Nitrogen iodide is characterized by a violently explosive tendency, and when purified and dried is susceptible to detonation by the mere contact of a feather.

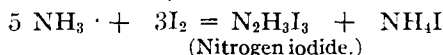
The precipitate, which is subsequently dissipated on completion of decoloriza-

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<sup>1</sup> *Am. J. M. Sc.*, 9, 398, 1865.

<sup>2</sup> "N. F. IV," p. 228.

tion,<sup>3</sup> has the constitution  $H_3N:Nl_3$  and represents a definite compound, formed through the following combination:



The nitrogen iodide is probably the result of decomposition of ammonium hypo-iodate:  $3NH_4OI = N_2H_3I_3 + NH_3 + 3H_2O$ .

The hypo-iodate is formed when iodine is dissolved in an excess of ammonia:



Production of the nitrogen halide compound is accompanied by the simultaneous formation of hydriodic acid, which in turn combines with some of the residual ammonia to form ammonium iodide.

#### REVISED PROCESS.

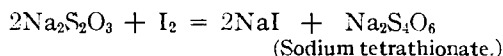
The following method was devised for large-scale production without the application of heat to affect digestion of the ingredients. Prepare a solution of

Sodium thiosulphate	83.0 Gm.
Ammonium hydroxide 28%	65.0 cc.

adding sufficient water to adjust the total volume to 100.0 cc. Solution may be more readily accomplished, if desired, by first dissolving the thiosulphate in a little water, the ammonia added and the required volume completed with water.

The mixture is transferred to a wide-mouthed jar of about 2 liters' capacity and Tincture of Iodine U. S. P. added in small portions with thorough agitation until the end-point is nearly attained. Discontinue the addition of tincture when the mixture retains a trace of color. This can then be readily eliminated by the addition of a few more fragments of thiosulphate with vigorous stirring or shaking.

A volume of approximately 1500.0 cc. of the U. S. P. Tincture is generally required, with a final yield of 1600.0 cc. of finished product. Allow the mixture to stand for several days in order to insure maximum precipitation of the crystalline sodium tetrathionate which is formed as shown in the equation.



The decolorized tincture is filtered through paper after which it is ready for dispensing. The preparation contains in addition to the reaction products noted above, varying amounts of triethylammonium iodide  $N(C_2H_5)_3HI$ , traces of sodium iodide, and sulphate, ethyl iodide, iodoform ( $CHI_3$ ), etc.

The alcoholic contents of the N. F. formula and the one here given are practically identical.

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<sup>3</sup> *Am. J. Pharm.*, 1869, 7.