TABLE I (*continued*).

Boiling point.	Temperature of bath.	Pressure.	Weight.
	F.		
78.5- 79°	100-105°	15	50
79- 85°	105-110°	15	28
85-108°	125-145°	15	8
108-112°	140	15	5
110-114°	140-145°	15	38
112-120°	145-150°	15	8

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DICHLOROAMINE T. AND CHLORINATED EUCALYPTOL 1.2.

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Since the introduction of Dichloroamine T. (Toluene-*p*-sulfondichloroamide) for the treatment of infected wounds by Dakin, Lee, Sweet, Hendricks and LeConte,[1] the production of this substance in pure form and on a large scale, as well as that of suitable solvents for its application, have become problems of importance. As originally used for this purpose, Dichloroamine T. was dissolved in a prepared eucalyptol (chlorinated) and used in this condition or further diluted with Prepared Paraffin Oil (chlorinated).

It was early recognized by the authors that the products obtained by the action of chlorine on eucalyptol may vary considerably according to the conditions of chlorination. By continued chlorination alone several preparations can be obtained, particularly chlorinated eucalyptol, specific gravity 1.2, which are at present the subject of extended study by surgeons.

The present paper is intended to describe briefly an improved method for the large scale production of Dichloroamine T., the preparation of Chlorinated Eucalyptol 1.2, as well as other results of the chlorination of eucalyptol.

Method for Large Production of Dichloroamine T.—Toluene-*p*-sulfonamide is dissolved in ten parts of 1 : 10 caustic soda (39° Bé.) and diluted with twenty parts of water. The solution is carefully filtered to remove ferric hydroxide. Chlorine from a cylinder is then passed into the solution until a voluminous white precipitate of toluene-*p*-sulfondichloroamide is formed. This, collected on a filter, throughly washed twice with 5-8 parts of water and finally with enough 10% alcohol to make a thin paste. The dilute alcohol washing should be done very quickly and the substance separated with the aid of a vacuum filter. It is then dried at a temperature not exceeding 55°, preferably in a vacuum dryer.

[1] *J. Am. Med. Assoc.*, **69**, 27 (1917).

The method has the advantage that it is very rapid and avoids the use of chloroform as a solvent. It was developed in order to produce a Dichloroamine T. which would be comparatively stable. The product has a negligible ash and a good chlorine content, but no free chlorine on standing.

Calc.: C = 29.54%. Found: C = 29.42%, 29.37%.

The very considerable work done in this laboratory on the preparation and distribution of Dichloroamine T. for surgical purposes has suggested the following specifications as most suitable for this material when used as a disinfectant:

Physical properties:	White powder or crystals with slight yellow tinge. M. 78–84°.
Chemical properties:	Soluble in cold chloroform with slight to no turbidity. (Any turbidity must be removable by shaking with anhydrous calcium chloride.) Soluble in Prepared Eucalyptol, Dakin, and Chlorinated Eucalyptol 1.2. Ash, not over 0.2%. Chlorine content, 29.0% to 29.54%: calc.; 29.54% C.

The solubility in chloroform or similar organic solvent is important in that it shows presence of inorganic impurities, calcium salts, etc. The material should not have a strong odor of chlorine, showing instability. The melting point may vary within the limits indicated, owing to moisture. Sharp drying will decompose the substance.

The eucalyptol, as first proposed,[1] was chlorinated with potassium chlorate and hydrochloric acid according to the following method:

Five hundred cc. eucalyptol (U. S. P.) are treated with 15 g. potassium chlorate and 50 cc. concentrated hydrochloric acid. After twelve hours the oil is well washed with water and sodium carbonate solution. Dry sodium carbonate is added to the oil and the mixture is allowed to stand twenty-four hours. It is then filtered and dried with a little calcium chloride.

This method has the disadvantage that no definite amount of chlorine enters the eucalyptol. The chlorination depends on the rate of adding chlorate and hydrochloric acid as well as the light in which the process takes place. Samples may have 1% chlorine present or scarcely any, depending on whether chlorination took place in sunlight or on a dark day. The specific gravity of this oil may vary from 0.930 to 0.935.

It was found that by passing chlorine gas from a cylinder into eucalyptol the reaction proceeded much further than by the chlorate method described above. When a specific gravity of 1.20 was reached a comparatively heavy oil, much less volatile than eucalyptol, was obtained.

The results of the use of this oil will be published elsewhere; briefly, it

[1] Dakin and Dunham, *Brit. Med. J.*, June, 1917.

was found that it is almost as good a solvent for Dichloroamine T.; that it can be used in full strength on the skin and in open wounds and that the use of Paraffin Oil either as a diluent or as a means of preventing sticking of dressings is unnecessary.

The following is a sufficiently detailed statement of the method by which this oil is obtained:

Preparation of Chlorinated Eucalyptol 1.2.—Through a glass tube reaching to the bottom of a five-gallon bottle containing about 10 kilos of eucalyptol, is passed chlorine from a cylinder. The oil used should have a b. p. of 176–177° and a specific gravity of 0.925. The process should be carried on in good daylight.

During the chlorination the temperature rises and should be kept below 80° by regulating the chlorine stream. Hydrochloric acid is given off and may be absorbed in alkali.

When a specific gravity of 1.19 is reached the oil is chlorinates sufficiently and the process in interrupted. The oil is then washed with about four liters of water, then shaken thoroughly with 250 g. dry sodium carbonate and allowed to settle. After carefully decanting from the carbonate, about 500 g. fused calcium chloride are added and the whole again shaken. On standing, preferably overnight, the oil is filtered, bottled and is then ready for use. It is a white or slightly amber-colored oil, specific gravity 1.2, with a chlorine content of about 31%. Such an oil will readily dissolve 20% of Dichloroamine T., which solution may keep in an amber bottle without decomposition for a month.

Further Products of Chlorination.—By the further chlorination of the 1.2 oil, products may be obtained having a specific gravity of 1.4 and higher. This may be carried out by chlorinating directly at 100°, or with a solvent such as chloroform at its boiling point. The oil of specific gravity 1.4 is amber colored and of the consistency of molasses. The Dichloroamine is still soluble, although to a less extent. The future may find a use for this type of oil, since the increased viscosity presents an advantage in certain cases.

Summary.

In this paper is presented a method for the large production of stabile toluene-*p*-sulfon-dichloroamide (Dichloroamine T.); a method for the preparation of Chlorinated Eucalyptol specific gravity 1.2 and further chlorination products.

The constitution and properties of these oil are the subject of investigation in the laboratory. In conclusion the authors wish to thank Dr. Paul A. Lewis for his coöperation.

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