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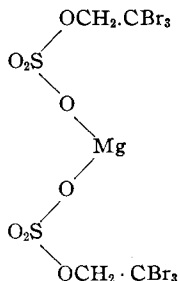
## MAGNESIUM SALT OF TRIBROM METHYL SULPHURIC ACID.\*

BY E. MONESS AND W. G. CHRISTIANSEN.

As part of a research on rectal anesthetics we became interested in determining whether or not the anesthetic properties of magnesium sulphate and polyhalogen aliphatic compounds could be combined in a single, water-soluble compound. Polyhalogen compounds such as ethyl chloride, methylene chloride and chloroform are inhalation anesthetics and tribrom ethanol is a rectal anesthetic. It seemed

\* Section on Practical Pharmacy and Dispensing, Madison meeting, 1933.

possible that the magnesium salt of tribromomethyl sulphuric acid (I) might be a useful, water-soluble, rectal anesthetic.



The acid ester was prepared by reacting tribromomethanol with fuming sulphuric acid in the cold. It was then converted into the magnesium salt. This salt was water-soluble but its activity as a rectal anesthetic was about one-eighth of that of tribromomethanol, *i. e.*, approximately eight times the quantity of this salt, as compared with tribromoethanol, were required to produce rectal analgesia.

The tests were made using 5%, 10%, 15% and 20% aqueous solutions of the salts and injecting a constant volume (25 cc./Kg.) into rats. Results were entirely negative with the 5% and 10% solutions. The 15% solution (dose 3.75 Gm./Kg.) produced a weakened ataxic condition which persisted up to the time the animal was sacrificed four hours later. The rat to which the 20% solution was administered failed to respond to tail pinching 55 minutes after dosage. This analgesic condition, with marked and continually increasing weakness, persisted from then until the animal died 35 minutes later. This behavior indicates that the compound cannot be advantageously used as a rectal anesthetic.

#### EXPERIMENTAL.

*Preparation of the Magnesium Salt of the Acid Sulphate of Tribromomethanol (CBr<sub>3</sub>.CH<sub>2</sub>.OSO<sub>3</sub>)<sub>2</sub>.Mg.*—The method is an adaptation of the one disclosed in German Patent 7278, December 31, 1893, for the preparation of sodium amyl sulphate.

4.2 Gm. of tribromomethanol was placed in a small test-tube and to it was added 1.5 Gm. of concentrated sulphuric acid. The viscous solution was cooled in an ice-salt bath and 2 Gm. of 60% fuming sulphuric acid was added from a small burette—one drop at a time, stirring with a thermometer, and not permitting the temperature to rise above 0° C.—about 25 minutes were required. After about half of the fuming sulphuric acid had been added, the reaction mixture became semi-solid. It was allowed to come to room temperature and poured into crushed ice and water. The excess sulphuric acid was precipitated with a solution of a slight excess of Ba(OH)<sub>2</sub> and the barium sulphate was filtered off. The excess barium hydroxide in the filtrate was then precipitated with a solution of magnesium sulphate, the precipitate filtered off and the filtrate evaporated to dryness *in vacuo* at a temperature of 50° C. The residue was now extracted twice with absolute alcohol, and the extract evaporated to a small volume by the addition of a large volume of ether. The compound was precipitated from this solution as a white crystalline salt, which was washed with ether by decantation and dried *in vacuo*. Yield—2.2 Gm. of a water-soluble and alcohol-soluble salt.

Analysis.	Found.	Calculated for (C <sub>2</sub> H <sub>3</sub> Br <sub>3</sub> SO <sub>4</sub> ) <sub>2</sub> .Mg.
Br	58.4%	64.2%
Mg	4.72%	3.25%
S	9.06%	8.55%

The analysis shows that our compound was contaminated with a small amount of  $MgSO_4$ , but this was held not to interfere with the tests for rectal anesthesia.

The compound, when heated for a long time at  $100^\circ C$ . decomposes with the liberation of  $HBr$ , and the formation of water and alcohol-insoluble substance.

The biological tests on compounds reported herein were made in the biological Research Laboratories of E. R. Squibb and Sons and we gratefully acknowledge their assistance.

RESEARCH DEPARTMENT OF THE CHEMICAL  
AND PHARMACEUTICAL LABORATORIES,  
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### A METHOD FOR THE PREPARATION OF PARENTERAL DEXTROSE SOLUTIONS.

BY HARVEY A. K. WHITNEY.\*

As a first premise it may be stated that a solution of dextrose suitable for parenteral administration may be made from a simple solution of chemically pure dextrose in chemically pure water. This statement is made with due consideration for the arguments for and against such factors as distilling apparatus, water, dextrose, hydrogen-ion concentration, buffers, preservatives and glass-ware. These modifying factors must be given consideration and together with the following elements, introduced at the time of administration, such as infusion apparatus, temperature of solution, velocity of injection and individual idiosyncrasy serve to complicate the apparently simple procedure issued as the original premise.

*Distilling apparatus* should be of a design that incorporates the following desirable and necessary features. Preferably the entrance of the raw water to the boiling chamber should be through condensers, permitting a preheating of the raw water, and venting the consequent discharge of many volatile substances. The insertion of a spray-trap or baffle-plates is also requisite to prevent the mechanical carry-over of boiling water or wet steam into the distillate. And lastly, the final delivery of the pure dry steam into vented condensers that will permit, if necessary, the delivery of the distillate into a closed system. It is pertinent to add that the whole interior of the distilling system should be lined with block tin.

*Water* obtained from the apparatus described, and under the circumstances necessary for the proper operation of the still, will provide a suitable solvent that may be used for the preparation of Aqua Destillata Sterilisata, U. S. P. Water distilled within the working hours of the morning or afternoon should be collected and disposed in a sterilized container of insoluble glass. The containers should be stoppered and sterilized under steam pressure giving a temperature of  $115^\circ C$ . for thirty minutes. If an autoclave is not available, close the mouth of flask containing freshly distilled water with a plug of purified absorbent cotton wrapped in gauze, and boil contents actively for one hour. Until ready for use, protect the mouth of flask, and plug from infection through dust, by wrapping top of flask tightly with paper. Sterilized distilled water so prepared should be used within

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