hydrate. This crude material contained 96% of the theoretical electropositive chlorine content. When it was distilled at 76–78° (10 mm.) this chlorine content was found to be 99% of theoretical. The distilled product melted at 4–4.5° cor., d^{20}_4 1.544. Since the crude material is sufficiently pure for most purposes, the distillation, which is dangerous, ought to be avoided.

i-**Propyldichloramine** was prepared by a modification of the above procedures. To an ice-cooled concentrated aqueous solution of 95 g. (1 mole) of *i*-propylamine hydrochloride (prepared *in situ*) was added slowly 2.25 moles of aqueous sodium hypochlorite (prepared by adding chlorine to cold 15% aqueous sodium hydroxide, 1.6 moles hypochlorous acid per liter). Simultaneously with this addition over two to three hours was added 6 *N* hydrochloric acid at such a rate that the acidity was maintained between *p*H 5.6 and 6.6. Brom cresol green and chlor phenol red papers can be used as criteria if the solution is allowed to creep into the paper so as to give an indicator zone ahead of the bleached zone. After two hours' subsequent stirring in the cold, the heavy oil was separated, washed once with 50 cc. cold water, twice with 50-cc. portions of 5% sodium thiosulfate, once with cold water, twice with cold 50% sulfuric acid and finally twice with cold water. The crude yield (97 g., 76% of theoretical) contained 94% of the theoretical electropositive chlorine content. Distillation at 41–43° (15 mm.) resulted in 70% recovery of yellow oil (d^{20}_4 1.165; n^{23}_D 1.4572) which contained 99.8% of the calculated electropositive chlorine content.

Anal. Calcd. for $C_3H_7NCl_2$: N, 10.9. Found: N, 10.6 (av.).

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A Modification of Wenker's Method of Preparing Ethyleneimine

BY PHILIP A. LEIGHTON, WILLIAM A. PERKINS AND MELVIN L. RENQUIST

The most convenient method for laboratory preparation of ethyleneimine is that of Wenker.[1] This is a two-step process involving the preparation of β-aminoethylsulfuric acid from monoethanolamine and sulfuric acid followed by treatment of the ester with alkali. An improved technique is suggested for the first step which involves less effort, gives a better quality of the intermediate ester, and leads to higher yields.

Six moles each of ethanolamine (b. p. 169.5–170.1°) and 95% sulfuric acid are separately diluted with half their weight of water and cooled in an ice-bath. The amine is added slowly to the acid with constant stirring in a round-bottom flask also cooled in an ice-bath. The mixture is then boiled under reduced pressure using a water aspirator attached to the flask. Bumping is prevented by the addition of glass beads and the use of a full flame to maintain vigorous boiling.

When the temperature of the liquid reaches 145°, only enough heat is applied to keep the solution boiling, and when a definite turbidity appears, usually between 155 and 160°, heating is stopped unless the temperature begins to fall. Quite suddenly crystallization takes place, causing the temperature to rise sharply to about 185°. After cooling, the cake is softened with 300 cc. of 95% ethanol, removed from the flask and ground with an additional 400 cc. of ethanol followed by filtering and drying.

The yield is 90–95%. The product is white, showing no evidence of charring as was the case when the heating was carried out in an open vessel according to Wenker's directions. Approximately one hour is required to remove the water from the above quantity of material. While Wenker was able to remove only 75% of the theoretical amount of water, in the above method a trap placed between the flask and aspirator collected virtually 100% of the amount expected.

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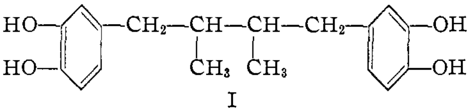
[1] H. Wenker, THIS JOURNAL, **57**, 2328 (1935).

A Synthesis of Nordihydroguaiaretic Acid

BY S. V. LIEBERMAN, GEORGE P. MUELLER AND ERIC T. STILLER

Nordihydroguaiaretic acid or 2,3-*bis*-(3,4-dihydroxybenzyl)-butane [4,4¹-(2,3-dimethyltetramethylene)-dipyrocatechol] (I) is of practical interest as an antioxidant used in preserving edible fats and oils.[1] It is obtained for that purpose by the alkaline extraction of dried plants of the species *Larrea divaricata*.[2]

Schroeter and his co-workers first obtained this compound from the dimethyl ether of guaiaretic acid (II) by hydrogenation and subsequent demethylation.[3] The dimethyl ether of guaiaretic acid has since been synthesized by Haworth, *et al.*[4] These two syntheses constitute a proof of the structure of nordihydroguaiaretic acid.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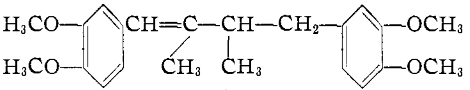
II

The new synthesis described here confirms this structure. The first step involves the coupling of two molecules of 1-piperonyl-1-bromoethane, yielding the corresponding dimethylene ether of nordihydroguaiaretic acid (IV), a compound reported by Orcutt and Bogert as a crystalline solid, m. p. 74°.[5] Apparently a mixture of the diastereoisomers of this compound is produced by the reaction of 1-piperonylethylmagnesium bro-

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[1] U. S. Patent 2,373,192; Higgins and Black, *Oil & Soap*, **21**, 277 (1944).

[2] U. S. Patent 2,382,475.

[3] Schroeter, Lichtenstadt and Irineu, *Ber.*, **51**, 1587 (1918).

[4] Haworth, Mavin and Sheldrick, *J. Chem. Soc.*, 1423 (1934).

[5] Orcutt and Bogert, THIS JOURNAL, **58**, 2057 (1936).