decreased, the amount of material boiling much higher than the ether is increased.

2,3-Dihydroxypropyl *n*-Dodecyl Ether.—A mixture of 3.6 g. (0.015 mole) of the 2,3-epoxypropyl *n*-dodecyl ether and 5.4 g. (0.3 mole of water) of 5% sulfuric acid was heated in the sealed tube at 160° for eight hours. The crude product was rinsed from the tube with 20–30 cc. of ether, neutralized with solid potassium carbonate and the ether layer separated. The aqueous layer was extracted with two 10-cc. portions of ether and the combined ether solutions were distilled on the steam-bath and finally under 15 mm. pressure to remove the last traces of ether and water. The residue weighed 3.0 g., 78% of the theoretical yield; $n^{20}\text{D}$ 1.4534. This compound is a wax-like solid melting about 20°, insoluble in water, soluble in methanol, ethanol, ether, acetone, carbon tetrachloride and benzene.

Preliminary hydrolysis experiments with excess water at 100° for twelve hours, 1% hydrochloric acid at 100° for eight hours, and 1% hydrochloric acid at 130° for eight hours all gave incomplete hydrolysis. The change in refractive index in going from the glycidol ether to the glycerol ether, 1.4434 to 1.4534 at 20°, was used to follow the course of the hydrolysis.

Anal. Calcd. for $C_{15}H_{32}O_3$: C, 69.12; H, 12.38. Found: C, 69.46; H, 12.39.

Samples of the ether were quantitatively oxidized at 50° with lead tetraacetate in glacial acetic acid solution according to the procedure of Criegee.[4] The following results are expressed as percentage of ether in the sample: six hours reaction time, 38.7%; twelve hours, 70.2%; twenty-one hours, 91.2%. By extrapolation of these data it appears that twenty-eight to thirty hours would be required for complete oxidation.

Reaction Tube.—Figure 1 shows a simple, convenient pressure tube made from steel tubing $1^3/_{16}''$ o. d., $^{15}/_{16}''$ i. d., and 15″ in length, closed at one end by a welded steel disk. The open end is machined flat so that a copper–asbestos gasket held in place by a metal plug (2) and threaded cap (3) will make a gas-tight seal.[7] A 22 mm. o. d. glass tube closed at one end is used as a glass liner.

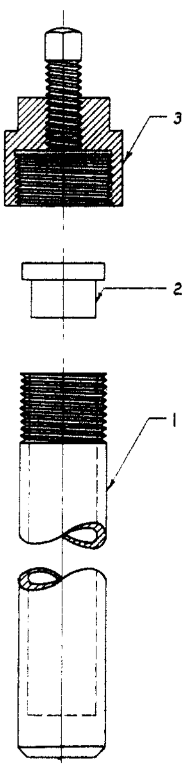

Fig. 1.—Metal bomb tube.

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[7] More complete descriptions of this type of closure are given in "A High Pressure Gas-Compression System," Dilley and Edwards, Circular 61, U. S. Dept. of Agriculture, Jan., 1929; and in J. H. Perry, "Chemical Engineers' Handbook," 2nd ed., McGraw-Hill Book Co., New York, 1941, p. 2181.

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Furfuryl Furoate by Condensation from Furfural

BY ERIK R. NIELSEN

Furfuryl furoate has been prepared by the condensation of furfural in the presence of sodium furfurylate. Previous attempts to carry out this condensation have been unsuccessful and have been cited to show a breakdown in the analogy between benzaldehyde and furfural.[1] The author has modified the method described for benzyl benzoate by Kamm and Kamm[2] to the extent that the condensing agent is prepared and the reaction carried out in the presence of refluxing benzene, whereby the danger of forming the ether rather than the ester is eliminated.

Experimental.—Eighteen grams of metallic sodium was added portionwise to a mixture of 250 g. of distilled furfuryl alcohol and 750 cc. of benzene, contained in a 3-liter flask (reflux condenser). After the condensing agent had been prepared, 1350 g. of distilled furfural was added through the reflux condenser at a rate so as to maintain refluxing by the heat developed by the reaction. The reaction mixture was washed with salt brine and distilled. The ester was recovered as a fraction boiling 121° (1.5 mm.) (77.8%). The solidified ester melted at 18.5° (uncor.) and did not convert to the allotropic form melting at 27.5° which has been reported to be the stable form by Zanetti.[1] The liquid ester showed a specific gravity $d^{25}{}_{25}$ of 1.2384 and a refractive index $n^{20}\text{D}$ of 1.5280. An analyses of the ester showed

	Acidity as furoic acid, %	Saponification equivalent	Carbon,[3] %	Hydrogen,[3] %
Calcd.		192	62.48	4.19
Found	0.23	195	62.25	4.32

The ester was characterized further by saponification to yield furfuryl alcohol and furoic acid.

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[1] H. Gilman and Clarence C. Vernon, THIS JOURNAL, **46**, 2578 (1924); J. E. Zanetti, *ibid.*, **47**, 1452–1453 (1925).

[2] O. Kamm and W. F. Kamm, "Organic Syntheses," Coll. Vol. I, pp. 99–101.

[3] These analyses are by Dr. T. S. Ma, University of Chicago.

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The Preparation of Potassium Triphenylmethide and its Use in Certain Condensations[1]

BY ROBERT LEVINE, ERWIN BAUMGARTEN AND CHARLES R. HAUSER

Sodium triphenylmethide is a useful reagent for the acylation[2] and alkylation[3] of esters and for the carbonation and carbethoxylation of esters[4] and ketones.[5] The sodium triphenylmethide has been prepared from triphenylchloromethane and sodium amalgam. While this method is quite satisfactory, there would be an advantage in preparing sodium triphenylmethide from triphenylmethane which would be regenerated when the reagent is used to effect condensations.

Sodium triphenylmethide is readily prepared from triphenylmethane and sodium amide in liquid ammonia[6] but the reagent is destroyed[7]

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[1] Paper XXVII on "Condensations"; paper XXVI, THIS JOURNAL, **66**, 1220 (1944).

[2] See especially Hauser and Hudson, "Organic Reactions," Roger Adams, Editor-in-Chief, John Wiley and Sons, Inc., New York, N. Y., 1942, Chapter IX.

[3] Hudson and Hauser, (a) THIS JOURNAL, **62**, 2457 (1940); (b) **63**, 3156 (1941).

[4] Baumgarten and Hauser, *ibid.*, **66**, 1037 (1944).

[5] Baumgarten, Levine and Hauser, *ibid.*, **66**, 862 (1944).

[6] Kraus and Kahler, *ibid.*, **55**, 3538 (1933).

[7] Kraus and Rosen, *ibid.*, **47**, 2739 (1925).