

large metal "dry box" through which dry air is passed. Operations are conducted through two holes in the side of the box.

The sample of liquid is poured into the crucible and thoroughly mixed with the solid. Suction is applied to remove any non-adhering liquid; the suction line is then broken. Liquid butane is now prepared. A test-tube is immersed in a solid carbon dioxide-acetone mixture and *n*-butane (Ohio Chemical and Manufacturing Company) is led into this through a glass tube. This operation is also conducted in the dry box. A small amount of the liquid butane (b. p. +0.4°) is poured into the crucible to chill it; a larger addition is then made, and the liquid is mixed with the solid to free the latter from the residue of organic liquid. Suction is again applied. The sample is washed twice more, and then the crucible is weighed (after warming to room temperature).

We cannot claim as yet a very high precision for this method. Thus with methanol containing 20% water, the method gave results in error up to 1%, and the same was true of mixtures containing paraldehyde in addition. Nevertheless, we believe that the simplicity and directness of the method make it worth presentation.

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Aqueous Solubility of 2,2,3-Trimethylpentanol-3

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The aqueous solubility of the least soluble octanol—normal octanol—has been determined by Butler¹ who found a mean of 0.0586 weight per cent. at 25°. Previous investigations of some lower alcohols² indicate that the most soluble octanol should be 2,2,3-trimethylpentanol-3 and it seemed worth while to measure its solubility. This compound was made by the action of *t*-butylmagnesium chloride on methyl ethyl ketone with the usual Grignard procedure, purified and the solubility measured by the same method used with the other alcohols. Results are given in the table. Although this tertiary octanol is of low solubility, it is more than ten times as soluble as the normal octanol.

(1) Butler, Thomson and MacLennan, *J. Chem. Soc.*, 680 (1933).

(2) (Pentanol) Ginnings and Baum, *THIS JOURNAL*, **59**, 1111 (1937); (hexanol) Ginnings and Webb, *ibid.*, **60**, 1388 (1938); (heptanol) Ginnings and Hauser, *ibid.*, **60**, 2581 (1938).

d_{25}^{25} of pure alc. and b. p. °C. (760 mm.)	Temp., °C.	Wt. % alc.		d of liquid phase	
				Alc. rich	Water rich
0.8420	20	0.75	98.02	0.8536	0.9975
153-154	25	.69	97.99	.8503	.9964
	30	.64	97.98	.8455	.9951

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1,4-Diiodobutane from Tetrahydrofuran

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The formation of tetrahydrofuran by the action of silver oxide and water with tetramethylene iodide has been reported by Hamonet.¹ The cleavage of the tetrahydrofuran ring to form tetramethylene iodide needed in a study of the action of metals on 1,4-dihalides, however, has not been described. Since other methods of preparing the iodide described by Hamonet and by Marvel and Tanenbaum,² had been tried or eliminated because of the difficulty of obtaining starting materials or satisfactory yields, the reaction between red phosphorus, iodine and tetrahydrofuran was carried out. Tetrahydrofuran (25 g.) was added dropwise to a mixture of 5.8 g. of red phosphorus and 94.5 g. of powdered iodine (added cautiously) placed in a 100-ml. three-necked flask provided with an efficient mechanical stirrer and a reflux condenser. The mixture was refluxed for an hour on a steam-bath and then poured into water. The lower black layer was drawn off and washed with 5% sodium hydroxide until colorless. Sufficient ether was then added until the extract floated on the solution of the base. The ether solution was washed several times with water, dried over calcium chloride and quickly transferred to a 125-ml. vacuum distillation flask fitted with a 10" (25-cm.) vacuum-jacketed side column packed with 1/4" (6-mm.) Fenske glass spiral rings, condensers, etc. Any oxygen present in the fractionating apparatus or in the solution was displaced by a current of nitrogen. After removing the ether, the 1,4-diiodobutane was distilled at a pressure of 1 mm. using a slow current of nitrogen to prevent bumping. A 51% yield (5.5 g.) of a slightly yellowish product boiling at 105-110° was obtained.

Reduction of furan (59 g.) by hydrogen (100 lb. (7 atm.)) with a Raney nickel catalyst (8 g.) at 80° gave a 91% yield of tetrahydrofuran, b. p.

(1) Hamonet, *Ann. chim.*, [9] **10**, 28 (1913).

(2) Hamonet, *Compt. rend.*, **132**, 346 (1901); Marvel and Tanenbaum, *THIS JOURNAL*, **44**, 2650 (1922).