

The Preparation of 2,2,2-Trideuterioacetaldehyde

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WE recently required large quantities of 2,2,2-trideuterioacetaldehyde, in a state of high isotopic purity. Previous studies of the exchange of the methyl protons of acetaldehyde have shown that in alkaline media, although exchange occurs, the aldol condensation is a competing process. Hence these conditions are not feasible for preparative use.^{1,2} We have examined the reaction in the presence of pyridine by n.m.r. spectroscopy and have found that on heating, in a sealed tube, a solution of acetaldehyde (4.0 M) in deuterium oxide with a little pyridine (0.4 M) (12 hr.), 83% exchange of the

methyl protons occurs. The aldehyde was readily recovered by distillation under nitrogen (75%). Two more such exchanges in the same manner gave 2,2,2-trideuterioacetaldehyde (40% overall), b.p. 24° (729 mm.), 99% deuterium in the methyl group by n.m.r. Mass-spectral analysis showed 96% trideuterio-, 2% dideuterio-, and 2% monodeuterio-species were present.† This then represents a simple and economic preparation of a valuable deuteriated compound.

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† At 9.5 ev., standardized against acetaldehyde.

¹ K. F. Bonhoeffer and W. D. Walters, *Z. phys. Chem.*, **1938**, **181**, A, 441.

² R. P. Bell and M. J. Smith, *J. Chem. Soc.*, **1958**, 1691.