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LETTERS TO THE EDITOR

SYNTHESIS OF 2-HYDROXY-3,3,5-TRICHLOROTETRAHYDROPYRAN

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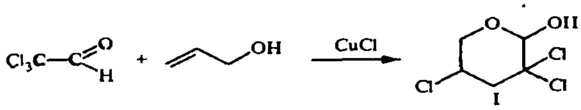
2-Hydroxy-3,3,5-trichlorotetrahydropyran (I), rather than the expected addition product 2,2,4-trichloro-5-hydroxypentanal, was formed as two pairs of diastereomers from the reaction of chloral with allyl alcohol in the presence of CuCl. The structure of heterocycle I was confirmed by ^1H NMR spectroscopy and by reverse synthesis: addition of chloral to allyl acetate followed by hydrolysis of the product and intramolecular cyclization of the allyl hemiacetal of chloral in the presence of CuCl:



The ^1H NMR spectrum showed the absence of the open chain tautomer.

CCl_3CHO (10 cm^3, 0.1 mol), $CH_2{=}CHCH_2OH$ (6.3 cm^3), 0.1 mol, acetonitrile (30 cm^3) and CuCl (1 g, 0.01 mol) were sealed in an ampule. The ampule was heated at 130°C for 10 h. The cooled ampule was opened, the solvent evaporated, and the residue dissolved in ether. The precipitate was filtered off and the filtrate evaporated and distilled in vacuum to give a yellow viscous liquid (6.52 g, 32%), b.p. 120°C (0.12 mmHg).

Found, %: C 29.05, H 3.62. Calc. for $C_5H_7Cl_3O_2$,%: C 29.23, H 3.44. ^1H NMR spectrum: 4.10, 4.15 (2H, m, CH_2O), 3.52, 3.91 (1H, m, CHCl), 3.13, 3.18 (2H, m, CH_2), 4.71, 5.15 (2H, d, CHOHO), 4.85 ppm (1H, s, OH). ^{13}C NMR spectrum: 62.56, 70.68 (CH_2O), 50.05, 50.30 (CHCl), 46.73, 52.05 (CH_2), 94.91, 98.45 (CHOHO), 87.19, 88.29 ppm (CCl_2).

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