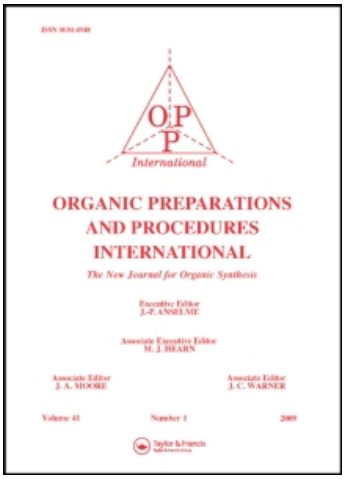

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PREPARATION OF MONOALLYL ETHYLENE GLYCOLS

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all at once to 500 g of ice. The aqueous solution was extracted with two 300 mL portions of ether. The combined ethereal extracts were washed with brine, saturated aqueous sodium bicarbonate solution, brine and were dried ($MgSO_4$). Removal of the solvent afforded 1.6 g of an orange oil which was chromatographed on 140 g of Alcoa neutral alumina eluting with 5:1 pentane-diethyl ether to give 0.87 g (61%) of 3,3,4,4-tetramethylcyclopentanone (1)[1] as a white solid, mp. 125-127°; R_f 0.47 (in 5:1 pentane-diethyl ether).

NMR: δ 1.05 (s, 12H), 2.21 (s, 4H); C-13-NMR: δ 24.02, 40.32, 52.61, 190.73 (very small, C=0); IR (mull) 1745 cm^{-1}; mass spectrum, Calcd. for $C_9H_{16}O$ m/e 140.1201, found m/e 140.1198.

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PREPARATION OF MONOALLYL ETHYLENE GLYCOLS

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Difficulties with the procedure of Riemschneider and Kotzsch [Monatsh. Chem., 90, 787 (1959)] for the synthesis of monoallyl ethylene glycols were immediately apparent. Preparation of the sodium salt of the glycol was accomplished by addition of sodium to excess glycol under toluene. Removal of the toluene required repeated triturations with ether.

This allowed for introduction of water vapor and diminished the yields. Purification by distillation through a Widmer column worked well enough for 2-allyloxyethanol; the higher homologs were not isolated as efficiently. The modified procedure outlined below avoided such difficulties.

EXPERIMENTAL SECTION

Sodium (23 g, 1 mol) was cut into small chunks under toluene. Pieces were added to 10 mol of the glycol under nitrogen. If necessary the solution was warmed to 50° to initiate the reaction. Sodium pieces were added at a rate to keep the pot temperature between 50° and 100°. After cooling below 70°, allyl bromide (86.5 ml, 1 mol) was added and stirred for 48 hrs. The solution was diluted to twice its volume with water and continuously extracted for 7 days with 50% 2,2,4-trimethylpentane in benzene. After evacuation of the solvents, the residue was purified by short-path distillation (see TABLE).

TABLE. Yields for Monoallyl Ethylene Glycol Oligomers

Compound[a]	% Yield (lit.[b])	bp° (mmHg)	Lit.[b]
$CH_2=CHCH_2OCH_2CH_2OH$	87 (99)	159	159
$CH_2=CHCH_2(OCH_2CH_2)_2OH$	78 (88)	73 (.20)	106 (10)
$CH_2=CHCH_2(OCH_2CH_2)_3OH$	84 (63)	101 (.25)	92 (.4)

a. All spectral analyses confirmed structural assignments.

b. See text.

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