Synthesis of Ethoxyethynyl Alcohols

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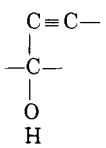
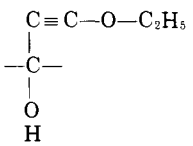
THE STRUCTURE



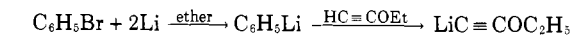
has appeared often in medicinal chemistry. However, the structure,



has not received attention, possibly because of the difficulty of preparing the starting material, ethoxyacetylene. The author has developed a commercial process for ethoxyacetylene and the compound is now available (*1*).

This paper describes the preparation of a number of tertiary alcohols and one secondary alcohol by the reactions of Grignard and lithium salts of ethoxyacetylene with ketones and an aldehyde.

Lithium ethoxyacetylide was prepared by the following sequence of reactions:



The Grignard reagent of ethoxyacetylene can not be used with aldehydes (*2*). The lithium salt generally gives good results.

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LITERATURE CITED

(1) Pfister Chemical Works, Ridgefield, N.J., unpublished work.
(2) Postma, J.C.W., Arens, J.F., *Rec. Trav. Chim.* **75**, 1385 (1956).

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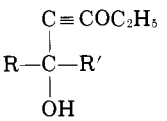
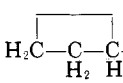
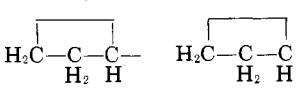
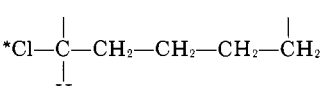


R	R′	Yield %	B.P., ° C. (Mm.)	Formula	Analysis: Carbon Calcd.	Carbon Found	Hydrogen Calcd.	Hydrogen Found	Chlorine Calcd.	Chlorine Found
C_2H_5—	—CH_3	62	57-9/4	$C_8H_{14}O_2$	67.57	67.33	9.92	10.13		
	—CH_3	55	49-50/0.2	$C_9H_{14}O_2$	70.10	70.15	9.15	9.30		
		41	75/0.4	$C_{11}H_{16}O_2$	73.30	73.42	8.95	9.22		
—$C_6H_4OCH_3$	—H	69	...	$C_{12}H_{14}O_3$	69.88	70.03	6.84	7.03		
$ClCH_2$—	—CH_3	67	50/2	$C_7H_{11}O_2Cl$	51.70	51.80	6.82	7.05	21.80	21.71
$ClCH_2CH_2$—	—CH_3	33	70/0.25	$C_8H_{13}O_2Cl$	54.39	54.68	7.42	7.52	20.07	19.94
		50	97.5/0.7	$C_{10}H_{15}O_2Cl$	59.19	59.26	7.56	7.46	17.49	17.60

*chloro cyclohexyl radical