LETTER TO THE EDITOR

The Synthesis of 4-hydroxy-6(2':6':6'-trimethylcyclohex-1'-enyl)-4-methyl-hex-5-en-1-yne

SIR,—By condensation of β-ionone (I) with propargyl bromide (II) employing a Reformatsky type of reaction, 4-hydroxy-6(2':6':6':6'-trimethylcyclohex-1'-enyl)-4-methyl-hex-5-en-1-yne (III) has been obtained in good vield. The

Me Me

$$CH = CH - CO$$
 $RCH_3 - C = CH$

Me

 Me
 M

product formed a yellow oil (Found: C, 83·2; H, 10·4; $C_{16}H_{24}O$ requires C, 82·7; H, 10·4 per cent.) which showed selective light absorption at $\lambda_{\rm max} = 232 {\rm m}\mu$, $E_{1}^{1\,{\rm per cent.}} = 255$, and gave a violet-blue colour with the Carr-Price reagent. On catalytic microhydrogenation it absorbed 2·7 molar equivalents of hydrogen, the 1':2'-cyclohexenyl double bond evidently resisting hydrogenation because of its tertiary character. Zerewitinoff determinations revealed the presence of two active hydrogen atoms.

The structure assigned to (III) has been confirmed by reaction of its Grignard reagent with p-methoxybenzaldehyde when 1:5-dihydroxy-7(2':6':6'-trimethylcyclohex-1'-enyl)-1(4"-methoxyphenyl)-5-methyl-hept-6-en-2-yne (IV) Found: C, 77.8; H, 9.7; OMe, 7.6. $C_{24}H_{32}O_3$ requires C, 78.2; H, 8.8; OMe, 8.4 per cent. was obtained. Reaction with p-dimethylaminobenzaldehyde gave the corresponding 4"-dimethylamino-analogue (V) (Found: C, 78.4; H, 9.4; N, 3.7; $C_{25}H_{35}O_2N$ requires C, 78.7; H, 9.2; N, 3.7 per cent.), characterised by selective light absorption at $\lambda_{max} = 260 \text{ m}_{\mu}$, $E_{1}^{1} \frac{\text{per cent.}}{\text{cm}} = 470$.

Experiments on the conversion of (III) into vitamin A alcohol are in progress.

The authors thank Dr. R. E. Stuckey and Mr. P. S. Stross for the absorption data, and the Directors of The British Drug Houses, Ltd., for permission to publish these results.

Research Department, The British Drug Houses, Ltd., London, N.1. 10th October, 1949. B. N. FEITELSON

V. Petrow

O. STEPHENSON.