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A New Method for Introducing the 14-Hydroxymethyl Group into the Steroidal Nucleus*

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Standard sample of the 32-oxygenated lanostane compounds which are key intermediates of cholesterol and ergosterol biosynthesis, have been available only through Barton-Kalvoda reaction of the appropriate 7β-hydroxylanostane derivative. As an alternative, we have now developed a convenient method for introducing the 14-hydroxymethyl group into the steroidal nucleus.

The methoxymethyl (MOM) ether of cholest-7-en-3β-ol was oxidized to the 7α,8α-epoxide with m-chloroperbenzoic acid, which was then treated with borontrifluoride etherate in tetrahydrofuran (THF), yielding the 7α-hydroxy-8(14)-ene system in 65% yield. When this allylic alcohol was deprotonated (KH/THF) and alkylated with iodotrimethyltin, the corresponding stanylmethyl ether was formed in 71% yield. Treatment of the ether with excess n-butyllithium in THF at -78℃ to ambient temperature induced a smooth [2,3] -sigmatropic rearrangement to give the 14-hydroxymethyl derivative in 79% yield. Removal of the MOM group with dilute HCl gave 14α-hydroxymethyl-7-cholesten-3β-ol (98%). Similarly, 4,4-dimethyl-7-cholesten-3β-ol was trnsformed into the 32-hydroxy-7-lanosten-3β-ol. Conversion of these 7-ene compounds into the biologically more important 8-ene isomer has already been established. Thus the present procedures pave a convenient way leading to various 32-oxygenated sterols.

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