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Synthesis of (4R,5R)-2-Ethoxy-4,5-bis[(1-methoxy-1-methyl)ethyl]-1,3-dioxolane

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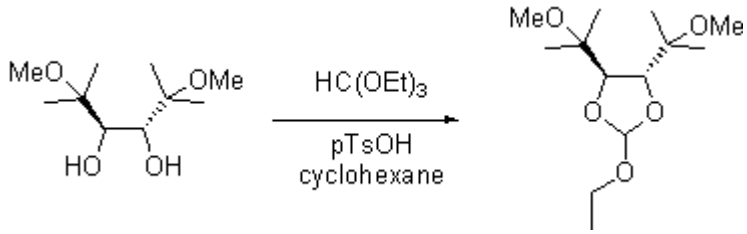

The title compound was prepared from 5 g (89 mmol, 1 eq) of (3R,4R)-2,5-dimethoxy-2,5-dimethyl-3,4-hexanediol [1], 4.4 ml (3.95 g, 27 mmol, 1.1 eq) of triethyl orthoformate, 0.05 g (0.25 mmol, 0.01 eq) of p-toluenesulfonic acid monohydrate in 35 ml of cyclohexane under inert atmosphere. The reaction mixture was stirred for 90 minutes at room temperature followed by distillation for 24 hours at 100°C to remove the formed ethanol. The mixture was allowed to cool to ambient temperature. The crude product was purified by Kugelrohr distillation over 0.5 g of K_2CO_3 at 170°C under reduced pressure (10 Torr) to give 5.64 g (89%) of the product as a colorless liquid.

^{1}H NMR ($CDCl_3$, 200 MHz): 5.89 (*s*, 1 H, CH); 4.07 (*AB*, *J* = 3.2, 1 H, C*H*CH); 4.01 (*AB*, *J* = 3.2, 1 H, CHC*H*); 3.66 (*q*, *J* = 7.0, 2 H, CH_2); 3.23 (*s*, 3 H, OCH_3); 3.22 (*s*, 3 H, OCH_3); 1.13-1.24 (*m*, 15 H, 5xCH_3).

^{13}C NMR ($CDCl_3$, 50 MHz): 117.6 (*D*, *J* = 99, CH); 83.4 (*D*, *J* = 147, *C*HCH); 81.7 (*D*, *J* = 150, CH*C*H); 76.3 (*S*, C); 75.7 (*S*, C); 61.1 (*T*, *J* = 142, CH_2); 49.5 (*Q*, *J* = 141, OCH_3); 49.4 (*Q*, *J* = 141, OCH_3); 22.0 (*Q*, C*C*H_3); 21.0 (*Q*, *J* = 126, C*C*H_3); 20.3 (*Q*, C*C*H_3); 19.3 (*Q*, C*C*H_3); 15.2 (*Q*, *J* = 126, CH_2*C*H_3)

EI-MS: 261 (15, $[M-H]^+$); 247 (5, $[M-Me]^+$); 217 (20, $[M-OEt]^+$); 189 (70, $[M-MeOCMe_2]^+$); 73 (100, $[CMe_2OMe]^+$)

References and Notes

1. The diol was prepared using described procedure : Mash, E. A.; Hemperly, S. B.; Nelson, K. A.; Heidt, P. C.; Vandeusen, S. *J. Org. Chem.* **1990**, *55*, 2045.

Sample Availability: Samples are not available.