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$_2$CO$_3$ 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, CHCH); 4.01 (AB, $J = 3.2$, 1 H, CHCH); 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 ($D$, $J = 147$, CHCH); 81.7 ($D$, $J = 150$, CHCH); 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 ($Q$, CCH$_3$); 21.0 ($Q$, $J = 126$, CCH$_3$); 20.3 ($Q$, CCH$_3$); 19.3 ($Q$, CCH$_3$); 15.2 ($Q$, $J = 126$, CH$_2$CH$_3$)

EI-MS: 261 (15, [M-H]$^+$); 247 (5, [M-Me]$^+$); 217 (20, [M-OEt]$^+$); 189 (70, [M-MeOCMe$_2$]$^+$); 73 (100, [CMe$_2$OMe]$^+$)

References and Notes


Sample Availability: Samples are not available.

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