Molecules 2000, 5, M161

N,N-Dibutyl-2-p-tolyl-acetamide

Olivier Comte, Sylvie Genillard and Céline Nkubana

IPSC - BCH, Université de Lausanne, 1015 Lausanne, Switzerland. E-mail: olivier.comte@etu.unil.ch

Received: 4 April 2000 / Accepted: 6 April 2000 / Published: 28 April 2000

Thionyl chloride (2.18 ml, 3.56 g, 30 mmol, 1.5 eq) was added dropwise to a vigorously stirred solution of p-tolylacetic acid (3 g, 20 mmol, 1 eq) and heated to 100°C for 3 hours. After cooling to room temperature, the volatiles were removed under reduced pressure [1]. The yellow residue was dissolved in 20 ml of dry ether and dibutylamine (11.1 ml, 8.28 g, 64 mmol, 3.2 eq.) was added dropwise at 0°C. After the addition was complete, the solution was allowed to warm to room temperature and stirred for 3 hours. The crude solution was washed with 2 x 25 ml of saturated NaHCO$_3$ solution and 2 x 25 ml of brine. The organic phase was dried over MgSO$_4$ and concentrated under reduced pressure. The crude product was purified by horizontal distillation (Kugelrohr) at 150°C (p=0.05 mbar) to yield 2.7 g (52 %) of the title product as a colorless oil.

IR (FT): 2958; 2872; 1642 (C=O); 1515; 1456; 1377.

$^1$H NMR (CDCl$_3$, 400 MHz): 7.13 (AB, $J = 8.0$, 2 H, H$_{arom}$); 7.11 (AB, $J = 8.0$, 2 H, H$_{arom}$); 3.64 (s, 2 H, CH$_2$); 3.31 (m, 2 H, NCH$_2$); 3.19 (m, 2 H, NCH$_2$); 2.31 (s, 3 H, CH$_3$); 1.24-1.56 (m, 8 H, 2xCH$_2$CH$_2$); 0.92 (t, $J = 7.3$, 6 H, 2xCH$_3$).

$^{13}$C NMR CDCl$_3$, 100 MHz): 170.2 (S, C=O); 135.6 (S, C$_{arom}$); 132.4 (S, C$_{arom}$); 128.9 (D, $J = 157$, CH$_{arom}$); 128.3 (D, $J = 157$, CH$_{arom}$); 47.7 (T, $J = 134$, NCH$_2$); 45.3 (T, $J = 134$, NCH$_2$); 40.4 (T, $J = 128$, CH$_2$CO); 30.9 (T, CH$_2$); 29.5 (T, CH$_2$); 20.7 (Q, CH$_3$); 20.0 (T, CH$_2$); 19.8 (T, CH$_2$); 13.6 (Q, $J = 126$, CH$_3$).

Mass (CI, NH$_3$): 262 (100, [M+H]$^+$); 156 (15, [CONBu$_2$]$^+$); 105 (8, [M–CONBu$_2$]$^+$).

Reference


Sample Availability: Samples are available from MDPI.

©1999 MDPI. All rights reserved. Molecules website www.mdpi.org/molecules/