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

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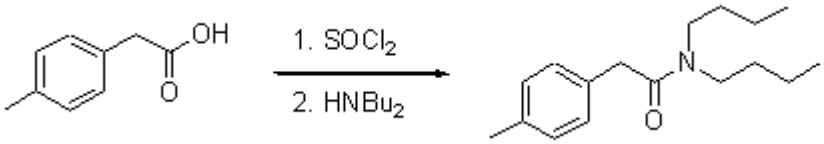

Thionyl chloride (2.18 ml, 3.56 g, 30 mmol, 1.5 eq) was added dropwise to a vigorously stirred solution of p-tolylacetic acid (3 g, 20 mmol, 1 eq) and heated to 100°C for 3 hours. After cooling to room temperature, the volatiles were removed under reduced pressure [1]. The yellow residue was dissolved 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 brime. 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_2CH_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_2CO); 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, $[M\text{-}CONBu_2]^+$).

Reference

1. Tietze, L. F.; Eicher, C. in: *Reaktionen und Synthesen;* Thieme: Stuttgart, **1981**, p.92.

Sample Availability: Samples are available from MDPI.