

N*-Furan-2-ylmethyl-4-methyl-benzenesulfonamide (*N*-Furfuryl-*p*-toluenesulfonamide)*Holger Meining and Baldur Föhlisch***Institut für Organische Chemie der Universität Stuttgart, Pfaffenwaldring 55, D-70569 Stuttgart, Germany
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A solution of furfurylamine[1] (9.77 g, 100 mmol) in *tert*-butylmethyl ether[2] (MTBE, 50 mL) was made in a 250-mL four-necked flask, fitted with two dropping funnels and an immersion thermometer, and cooled in an ice/salt bath to -15 °C. With vigorous stirring, solutions of *p*-toluenesulfonyl chloride (20.97 g, 110 mmol) in MTBE (80 mL) and 20% aqueous sodium hydroxide (22 mL, 110 mmol) were added simultaneously and cautiously. The temperature of the reaction mixture was not allowed to exceed -5 °C. When the addition was completed (60–90 min), the cooling bath was removed, and the mixture stirred for 2–3 hours. The MTBE layer was separated, and the aqueous layer acidified to pH 3–4 and extracted with MTBE (4 × 40 mL). The combined organic solutions were washed with saturated brine (2 × 40 mL) and dried overnight with magnesium sulfate. After filtration, the solvent was removed in a rotary evaporator. The remaining white solid was recrystallized from 50% aqueous ethanol (150 mL) to yield 21.28 g (85%) of colourless platelets with m.p. 114–115°C (Ref.[3]: 88%, m.p. 111–112°C).

¹H NMR (250 MHz, CDCl₃): d = 2.41 (s, 3 H, CH₃C₆H₄SO₂), 4.15 (d, *J* = 6.0 Hz, 2 H, CH₂-N), 5.04 (t, *J* = 6.0 Hz, 1 H, NH); ABX sub-spectrum with *d*_A = 6.20, *d*_B = 6.08, *d*_X = 7.22, *J*_{AB} = 3.2 Hz, *J*_{AX} = 1.8 Hz, *J*_{BX} = 0.8 Hz (3 H, H-4, H-3, H-5); AA'BB' sub-spectrum with *d*_A = 7.71, *d*_B = 7.26, *J*_{AB} = 8.2 Hz, (4 H, H-2/6 of CH₃C₆H₄SO₂, H_{AA'}, and H-3/5 of CH₃C₆H₄SO₂, H_{BB'}).

¹³C NMR/DEPT (62.9 MHz, CDCl₃): d = 21.5 (+, CH₃C₆H₄SO₂), 40.1 (-, CH₂-N), 108.2 (+, C-3), 110.4 (+, C-4), 127.1 (+, Tos-C-2/6), 129.65 (+, Tos-C-3/5), 136.85 (C_q, Tos-C-4), 142.5 (+, C-5), 143.5 (C_q, Tos-C-1), 149.6 (C_q, C-2).

IR (CDCl₃): 3385 (N-H), 2930, 2870 (C-H), 1600 (C=C), 1495, 1410, 1335, 1165 cm⁻¹(SO₂).

Anal. Calcd. for C₁₂H₁₃NO₃S (251.3): C, 57.35; H, 5.21; N, 5.57; S, 12.76. Found: C, 57.49; H, 5.30; N, 5.52; S, 12.53.

References and Notes

1. Commercially available; we used a product from Fluka, Switzerland.

2. *tert*-Butylmethyl ether was recommended as an inexpensive solvent when this experiment was carried out (June 1991, Diploma thesis of H.M.). Nowadays, it is considered environmentally problematic. We also used 1,2-dichloroethane instead of the ether.
3. Chooney, N.; Kuhnert, N.; Sammes, P. G.; Smith, G.; Ward, R. W. *J. Chem. Soc., Perkin Trans 1*, **2002**, 1999–2005. Dry pyridine was used as base and solvent. However, spectra and combustion analysis are not given in this paper.

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