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## 4-Methyl-*N*-(5-methyl-3-oxo-8-oxabicyclo[3.2.1.]oct-6-en-1-ylmethyl)-benzenesulfonamide

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Zinc powder (10.6 g, 160 mmol) was given to an ice-cold solution of methanol (40 mL) saturated with ammonium chloride. With vigorous magnetic stirring in an ice-bath, tetrachloroketone 1 [1] (4.59 g, 10 mmol) was added in small portions (exothermic reaction, the mixture should not be allowed to boil!). Then the ice-bath was removed, and the mixture was allowed to come up to room temperature with continuous stirring. Finally, the mixture was refluxed. A TLC showed that after 1 h the starting material 1 had disappeared. Unreacted zinc powder and inorganic salts were removed by suction and washed with methanol (50 mL). To the combined filtrates, 100 mL of a 0.6 molar solution of EDTA disodium salt (110 g in 500 mL water with 10 g NaOH) was added. The mixture was stirred for 1 h, and extracted with tert-butylmethyl ether (9 ´ 20 mL). The combined extracts were washed with saturated brine (50 mL) and dried overnight with magnesium sulfate. After filtration the solvent was evaporated. The remaining white solid was recrystallized from a mixture of ethanol and tert-butylmethyl ether (2:1 v/v, 30 mL). The slight pink-coloured crystals were filtrated and finally washed with a small amount of n-pentane to yield 2.74 g (85%) product.

Melting Point: 126-127 °C.

TLC (silica, hexane/tert-butylmethyl ether (1:1 v/v): A lemon-coloured spot emerged after spraying the sheet with vanillin/sulfuric acid reagent followed by heating with a hot-air gun;  $R_f = 0.07$ . The starting material (1) showed a pale yellow spot at  $R_f = 0.27$ .

<sup>1</sup>H-NMR (250 MHz, CDCl<sub>3</sub>):  $\delta$ = 1.43 (s, 3 H, 5-CH<sub>3</sub>); 2.43 (s, 3 H, C $H_3$ C<sub>6</sub>H<sub>4</sub>SO<sub>2</sub>); two AB sub-spectra with  $\delta_A$ = 2.43,  $\delta_B$ = 2.36,  $J_{AB}$ = 16.1 Hz =  $J_{gem}$ , and  $\delta_A$ = 2.45,  $\delta_B$ = 2.26,  $J_{AB}$ = 16.5 Hz=  $J_{gem}$  (4 H, H-2 and H-4); ABX sub-spectrum (8 + 4 lines) with  $\delta_A$ = 3.30,  $\delta_B$ = 3.14,  $\delta_X$ = 5.13,  $J_{AB}$ = (-)13.0 Hz,  $J_{AX}$ = 7.8 Hz,  $J_{BX}$ = 4.6 Hz (3 H, diastereotopic C $H_2$ -NH); AB sub-spectrum with  $\delta_A$ = 6.00,  $\delta_B$ = 5.92,  $J_{AB}$ = 5.8 Hz (2 H, H-6 and H-7); AA'BB' sub-spectrum (4 lines) with  $\delta_A$ = 7.75,  $\delta_B$ = 7.32,  $J_{AB}$ = 8.2 Hz (4 H, H-2/6 and H-3/5 from CH<sub>3</sub>C<sub>6</sub>H<sub>4</sub>SO<sub>2</sub>).

 $^{13}$ C-NMR (62.9 MHz, CDCl<sub>3</sub>): d = 21.5 (*C*H<sub>3</sub>C<sub>6</sub>H<sub>4</sub>SO<sub>2</sub>); 22.9 (5-CH<sub>3</sub>); 46.9 (C-2); 47.2 (C-4); 51.1 (CH<sub>2</sub>-N); 84.3 (C-5); 85.5 (C-1); 127.05 (Tos-C-2/6); 129.8 (Tos-C-3/5); 133.2 (C-6); 136.65 (Tos-C-4); 137.9 (C-7); 143.6 (Tos-C-1); 205.2 (C-3).

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IR (CDCl<sub>3</sub> film, cm<sup>-1</sup>): 3380, 3280 (N-H); 2980, 2960, 2930, 2900, 2880 (CH); 1712 (C=O); 1600 (C=C); 1495, 1450, 1410, 1382, 1335, 1175.

Elemental Analysis: Calculated for  $C_{16}H_{19}NO_{4}S$  (321.4): C, 59.79%; H, 5.96%; N, 4.36%; S, 9.98%. Found: C, 59.70%; H, 5.99%; N, 4.19%; S, 9.95%.

## References

1. Meining, H; Föhlisch, B. Molbank 2005, M410.

Sample Availability: Available from MDPI.

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