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5-Phenyl-3-[1-(phenylsulphonyl)-hexyl]-1,2,4-triazine

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As part of ongoing research programme we synthesised the title compound as valuable intermediate for intermolecular cycloaddition reactions, leading to biologically active heterocycles.

In a round-bottom flask (25 mL), the suspension of NaH (27 mg, 1.1 mmol; washed with Et₂O from 60% oil suspension) in DMF (3 ml), cooled to 0°C, was stirred for 15 min under argon. Then, the 5-phenyl-3-phenylsulphonylmethyl-1,2,4-triazine (1) [1] (312 mg, 1.0 mmol) and 1-iodopentane (200 mg, 1.0 mmol) in DMF (3 ml) was added *via* syringe at 0°C during a period *ca* 5 min. The reaction mixture was stirred at 0°C for *ca* 2 - 4 h until completion (TLC monitoring; CHCl₃). The mixture was poured onto water with ice, the precipitate was filtered and the crude product was purified by column chromatography using CHCl₃/n-hexane mixture (100:1) as eluent. Yield of pure 5-phenyl-

3-[1-(phenylsulphonyl)-hexyl]-1,2,4-triazine (3) - 343 mg (90%). An analytical sample was recrystallized from ethanol/water mixture to give yellow solid.

M.p. 165-166°C (EtOH-H₂O).

IR (KBr, cm⁻¹): 2965 & 2880 (CH₂), 1315 & 1160 (SO₂).

¹H NMR (CDCl₃, 200 MHz): 0.75-0.93, 1.10-1.40, and 2.30-2.62 (3 x m, 11 H, C₅H₁₁), 4.85 (dd, $J_I = 10.8$ Hz, $J_2 = 4.4$ Hz, 1 H, CH(SO₂Ph)), 7.40-7.74 (m, 7 H, H-Ar), 8.07 (apparent d, J = 7.9 Hz, 2 H, H-Ar), 9.58 (s, 1 H, H-triazine).

MS (EI), m/z (% rel. int.): 381 (<1, M^{+•}), 317 (20), 261 (6), 260 (27), 241 (6), 240 (34), 184 (7), 102 (100), 77 (20), 51 (6).

LSIMS(+), *m/z* (% rel. int.): 382 (100, M+H), 240 (14).

HR-LSIMS Calcd for C₂₁H₂₄N₃SO₂ (M+H): 382.1589; Found: 382.1579.

Reference

1. Rykowski, A.; Makosza, M. Liebigs Ann. Chem. 1988, 627.

Sample Availability: Available from the author and from MDPI.

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