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Short Note

1,3,5-Tris-(2,3-dihydro-1*H*-1,5-benzodiazepin-4-yl)-1,2,3,4,5,6-hexahydro-*s*-triazine

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Abstract: A new compound, 1,3,5-tris-(2,3-dihydro-1*H*-1,5-benzodiazepin-4-yl)-1,2,3,4,5,6-hexahydro-*s*-triazine was obtained by the reaction of *ortho*-phenylenediamine **1** with 1,3,5-triacryl-1,2,3,4,5,6-hexahydro-1,3,5-triazine **2**. Spectroscopic (IR, ^{1}H-NMR, ^{13}C-NMR and MS) data are supplied to support the proposed structure for the title compound.

Keywords: 1,5-benzodiazepines; 1,3,5-triazines; *ortho*-phenylenediamine

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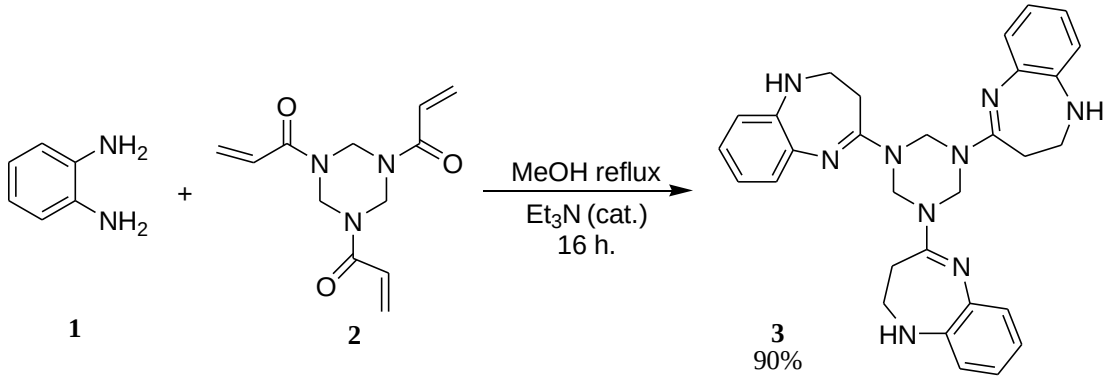
A method for the preparation of 1,5-benzodiazepine derivatives corresponds to the condensation reaction between aromatic *o*-diamines with 1,3-dielectrophilic compounds such as acrylic acid, acrylic esters and acrylamides. This approach is quite versatile, which makes it appropriate for synthetic purposes [1–6]. Benzodiazepine moieties are well-known due to their wide range of pharmacological activities [7,8]. Previous outcomes reported by us have shown that other systems based on the diazepine core, introduced special properties against different cancer types; turning them into promising pharmacophores [9,10].

On the other hand, the (1,3,5-triacryl)-*s*-triazine fragments have been used as cross-linking agents for improving the fixation of dyes on polyamide fibers [11].

*Synthesis of 1,3,5-tris-(2,3-dihydro-1*H*-1,5-benzodiazepin-4-yl)-1,2,3,4,5,6-hexahydro*-s-*triazine* (**3**)

A mixture of *ortho*-phenylenediamine **1** (345 mg, 3.2 mmol) and 1,3,5-triacryl-1,2,3,4,5,6-hexahydro-1,3,5-triazine **2** (248 mg, 1 mmol) in methanol (10 mL) with triethylamine (1 mL) was refluxed for 16 h (see Scheme 1). The precipitate formed was filtered off and then purified by column chromatography on silica gel, using CH_2Cl_2:MeOH (20:1) as eluent.

Scheme 1. Synthesis of 1,3,5-tris-(2,3-dihydro-1*H*-1,5-benzodiazepin-4-yl)-1,2,3,4,5,6-hexahydro-*s*-triazine **3**.



The 1,3,5-tris-(2,3-dihydro-1*H*-1,5-benzodiazepin-4-yl)-1,2,3,4,5,6-hexahydro-*s*-triazine **3** was obtained as a pale-yellow solid (yield: 467 mg, 90%), this compound showed high solubility in solvents such as $CHCl_3$, CH_2Cl_2, DMF and DMSO. Studies on the generality of this procedure to obtain new analogues and on some biological properties of compound **3** are in progress.

Melting point: >350 ℃.

IR (KBr, cm^{-1}): 3385–3338 (NH), 1647 (C=N), 1508 (C=C).

^{1}H-NMR ($CDCl_3$, 400 MHz): δ = 6.82-6.78 (m, 3H, Ar-H); 6.69-6.66 (m, 9H, Ar-H); 5.23 (s, 6H, N-CH_2-N); 3.45 (t, *J* = 6.0 Hz, 6H, CH_2); 3.44 (bs, 3H, NH); 2.86 (bs, 6H, CH_2) ppm.

^{13}C-NMR ($CDCl_3$, 100 MHz): δ = 171.4, 137.2, 135.4, 120.8, 119.6, 116.9, 112.9, 56.1, 39.7, 32.3 ppm.

EI-MS (m/z, %): 519.2 (M^+, 100%), 374.5 (21), 230.1 (16), 145.3 (49),107.3 (35).

Elemental Analysis: Calculated for $C_{30}H_{33}N_9$: C, 69.34%, H, 6.40%, N, 24.26%. Found: C, 69.30%, H, 6.37%, N, 24.24%.

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