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Synthesis and characterization of 1,3-*bis*(4-bromophenyl)-5-propyl-1,3,5-triazinane

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ABSTRACT

Condensation of *n*-propyl amine and 4-bromoaniline with formaline in basic solution gives 1,3-*bis*(4-bromophenyl)-5-(propyl)-1,3,5-triazinane. Structure of the synthesized compound was characterized by FT-IR and ^{1}H- and ^{13}C-NMR spectroscopy.

1. Introduction

The formation of 1,3,5-triazacyclohexanes from primary amines and formaldehyde has been known for over a century [1]. The different triazines were synthesized in the various laboratories according to the procedure described elsewhere [2]. The 1,3,5-triazacyclohexane are the subject of several structural studies considering their use in the industrial chemistry. They can be used as ligands for the preparation of new complexes that can be served as catalyst in the polymerization and trimerization of olefines [3]. However, interest in 1,3,5-triazacyclohexane as ligand seems to growing rapidly [4-8].

2. Experimental

2.1. Instrumentation

Purity of the title compound was checked by thin layer chromatography (TLC) using CH_2Cl_2:*n*-hexane (1:1) as an eluent. IR spectra were recorded in KBr pellet on Shimadzu FT-IR 8201 PC (4000-400 cm^{-1}). ^{1}H- and ^{13}C-NMR spectrum of the title compound were recorded on a Bruker Advanced DPX-250 (^{1}H-NMR 250 MHz and ^{13}C-NMR 62.9 MHz) spectrophotometer in $CDCl_3$ using TMS as an internal standard.

2.2. Synthesis

The title compound was obtained by mixing a 2:1:4:4 stoichiometric ratio of *n*-propyl amine, 4-bromo aniline, formaline and potassium hydroxide (in water 15 mL) in ethanol (25 mL) at 293 K for 48h. The resulting solution was extracted with CH_2Cl_2, dried with $MgSO_4$, evaporated on a rotary evaporator to dryness. The oil residue was crystallized from cyclohexane [5-8]. 1,3-*Bis*(4-bromophenyl)-5-(propyl)-1,3,5-triazinane. Yield: 95%. M.p.: 115-117 °C. FT-IR (KBr, ν, cm^{-1}): 2925 (C-H), 1583, 1498 (C=C), 1276 (C-N), 516 (C-Br), 758 (C-H, Ar). ^{1}H NMR (250 MHz, $CDCl_3$, δ, ppm): 0.95 (t, 3H, CH_3), 1.50 (m, 2H, CH_2), 2.51 (t, 2H, CH_2), 4.40 (s, 4H, C_2H_5-N-CH_2-N-Ar), 4.70 (s, 2H, Ar-N-CH_2-N-Ar), 6.80-7.00 (m, 8H, Ar). ^{13}C NMR (62.89 MHz, $CDCl_3$, δ, ppm): 11.87 (*C*H_3-CH_2), 20.92 (CH_3-*C*H_2), 53.98 (C_2H_5-*C*H_2-N), 68.26 (C_3H_7-N-*C*H_2-N-Ar), 71.12 (Ar-N-*C*H_2-N-Ar), 112.97 (C-Br) 119.10, 132.14 (*C*H=C-), 148.38 (N-C=).

3. Results and discussion

An unsymmetrically substituted triazinane, 1,3-*bis*(4-bromophenyl)-5-(propyl)-1,3,5-triazinane, was prepared from the condensation reaction of *n*-propyl amine and 4-bromoaniline with formaldehyde [6] (Scheme 1). This compound is stable at room temperature and high yield (95%) with a transparent color.

The mechanism of interaction is the production of Schiff base, which polymerize to give 1,3-*bis*(4-bromophenyl)-5-(propyl)-1,3,5-triazinane (Scheme 2).

Structure of the title compound has been elucidated by FT-IR, ^{1}H-NMR and ^{13}C-NMR including 2D, J-mod, HSQC measurements. The infrared spectrum shows a strong stretching vibration at 516 cm^{-1} for characteristic C-Br bond. Two absorption bands at 1583 and 1498 cm^{-1} are shows by the six-membered aromatic system $\nu_{C=C}$ an absorption bond at 758 cm^{-1} characteristic of the C-H out of plane vibration of the aromatic system.

The ^{1}H NMR spectrum shows protons of the methyl group resonate as a three proton triplet centered at 0.95 ppm, A triplet arises be cause the methyl group has two equivalent protons on an adjacent carbon atom. The two protons of CH_2 group adjacent to both CH_2 and CH_3 groups appear as sexlet at 1.50 ppm (CH_3-CH_2-CH_2).

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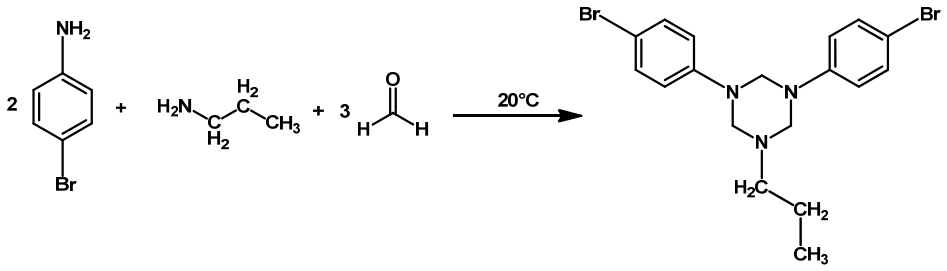
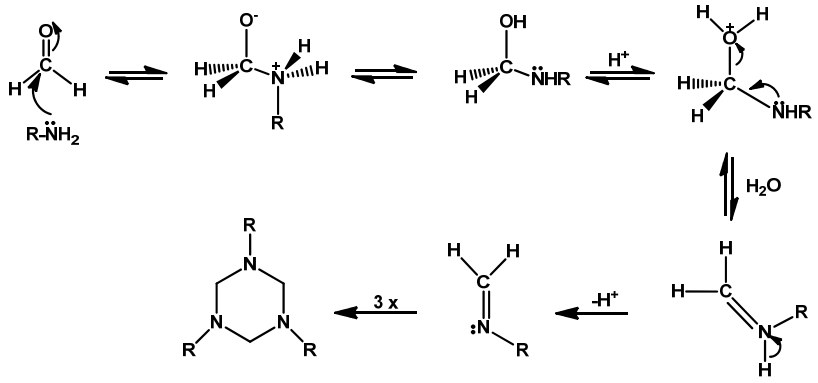


Scheme 1



Scheme 2

The two protons of the CH_2 group attached to the nitrogen atom (CH_2-C*H*$_2$-N) shows a triplet centered at 2.51 ppm. The protons of the heterocyclic triazinane appear as two singlet at 4.40 ppm (C_3H_7-N-C*H*$_2$-N-Ar) and 4.70 (Ar-N-C*H*$_2$-N-Ar), the protons of the aromatic system appear as multiple signal at 6.80-7.00 ppm.

The carbon atoms of the propyl group appear at 11.87, 20.92, 53.98 ppm, the carbon atoms of the triazinane group appears at 68.26 and 71.12 ppm, the carbon atoms of the aryl group appears at 119.10, 132.14 and 148.38 ppm, the carbon ring aryl which has a bromine atom appears at 112.97 ppm.

4. Conclusion

We have synthesized and characterized a new unsymmetrical 1,3,5-triazinane derivative. The synthesis was achieved by condensation of *n*-propyl amine and 4-bromo aniline with formalin. The synthesized compound is very stable in air and can be a useful ligand for the preparation of new metal complexes that can be served as catalyst in the polymerization and trimerization.

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