

Synthesis and spectral studies of 3,5-bis(4-chlorophenyl)-1-propyl-1,3,5-triazinane

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ABSTRACT

A hexahydrotriazine derivative was synthesized via a condensation reaction of 4-chloroaniline, propylamine and formalin. The structure of the synthesized compound was confirmed and characterized by using various spectral techniques like FT-IR, ¹H NMR, ¹³C NMR and UV spectroscopy.

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1. Introduction

Hexahydrotriazines belong to a wide range of six-membered ring compounds having three nitrogen atoms in 1, 3 and 5 positions [1]. Over the years, *N*-substituted 1,3,5-hexahydrotriazines have received considerable attention [2]. One of particular interest is the use of these type of products in industrial chemistry [3], as an example *N,N',N''*-trisubstituted 1,3,5-triazinanes can be used as reactants for the preparation of *N*-heterocyclic carbenes, which are a substantial class of ligands in homogenous catalysis [4].

In the previous research work, the single crystal structure of the title compound was characterized by X-ray single crystal diffractometer [5]. In this work, the structure of the synthesized title compound was confirmed and characterized by using various spectral techniques like FT-IR, ¹H NMR, ¹³C NMR and UV spectroscopy.

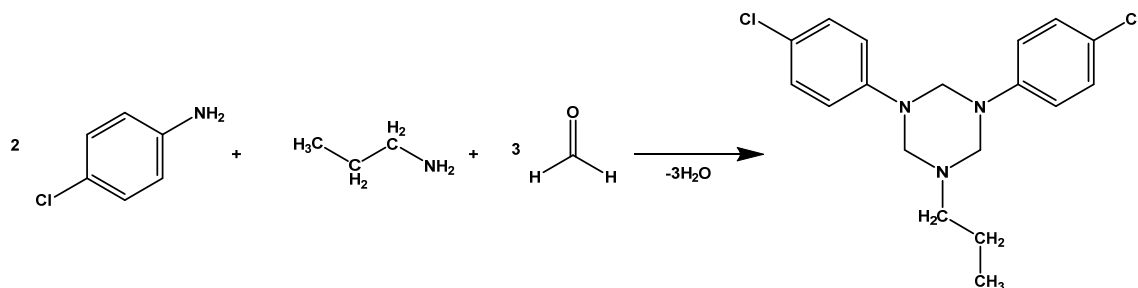
2. Experimental

2.1. Instrumentation

Melting point was determined on a capillary melting point apparatus. The purity of the title compound was screened by an analytical thin layer chromatography (TLC) conducted on percolated TLC plates (Silica gel 60F₂₅₄, Merck) visualized under UV light and using CH₂Cl₂: Petroleum ether (4:1, v:v) as an eluent. ¹H and ¹³C NMR spectra were recorded on Bruker 400 MHz spectrometer at room temperature using TMS as internal reference and chemical shifts are expressed as δppm. The infrared spectra was recorded in KBr pellet on Shimadzu FT-IR 8201 PC (4000-400 cm⁻¹) spectrophotometer. The UV spectra were recorded on a SPECORD 200 plus spectrophotometer (190-1100 nm).

2.2. Synthesis

An excess of formalin was added dropwise to a solution of 1:2 stoichiometric ratio of *n*-propylamine and 4-chloroaniline in 25 mL of ethanol. The mixture was stirred for 12 hours at 20 °C. The resulting solution was evaporated on a rotary evaporator to dryness and the white residue was recrystallized from cyclohexane to afford 3,5-bis(4-chlorophenyl)-1-propyl-1,3,5-triazacyclohexane in 89% yield [3-6].



Scheme 1

M.p.: 135-137 °C. FT-IR (KBr, ν , cm^{-1}): 3089-3033 ($\nu_{\text{C-H}}$, Ar-H), 2954-2805 (CH_3 , CH_2), 1276 (C-N), 815 (C-Cl), 752 ($\delta_{\text{C-H}}$, Ar). ^1H NMR (400 MHz, CDCl_3 , δ , ppm): 0.90 (t, 3H, CH_3), 1.50 (m, 2H, CH_2), 2.52 (t, 2H, CH_2), 4.25 (s, 4H, $\text{C}_3\text{H}_7\text{-N-CH}_2\text{-N-Ar}$), 4.71 (s, 2H, Ar-N- $\text{CH}_2\text{-N-Ar}$), 6.91 (d, 4H, Ar), 7.18 (d, 4H, Ar). ^{13}C NMR (100 MHz, CDCl_3 , δ , ppm): 11.81 ($\text{CH}_3\text{-CH}_2$), 20.88 ($\text{CH}_3\text{-CH}_2$), 53.99 ($\text{C}_2\text{H}_5\text{-CH}_2\text{-N}$), 68.63 ($\text{C}_3\text{H}_7\text{-N-CH}_2\text{-N-Ar}$), 71.27 (Ar-N- $\text{CH}_2\text{-N-Ar}$), 118.75 (CH, Ar), 125.64 ($\text{C}_{\text{Ar-Cl}}$), 129.13 (CH, Ar), 147.93 (N- C_{Ar}). UV (λ_{max} , nm): 327-328 ($\pi\text{-}\pi^*$ transitions), 194-197 ($n\text{-}\pi^*$ transitions).

3. Results and discussion

An unsymmetrically substituted 1,3,5-hexahydrotriazine, 3,5-bis(4-chlorophenyl)-1-propyl-1,3,5-triazacyclohexane (**I**), was synthesized via a mixed condensation reaction of 4-chloroaniline and *n*-propylamine with formaldehyde (formalin) [7,8] (Scheme 1). This compound is stable at room temperature and obtained in high yield (89%). Recrystallized from cyclohexane gave transparent colorless plaques. The synthesis mechanism is probably pass through imines, which trimerizes to give the title compound **I** [3].

The characterization of the title compound has been interpreted by FT-IR, ^1H -NMR and ^{13}C -NMR including 2D measurements. The infrared spectrum reveals a strong sharp band at 815 cm^{-1} resulting from the stretching vibration of C-Cl band. Two absorption bands at 1492 and 1590 cm^{-1} are shown by the six-membered aromatic system ($\nu_{\text{C=C}}$), another absorption band at 752 cm^{-1} characteristic of the C-H out of plane vibration of the aromatic system.

The ^1H NMR spectrum of the title compound shows protons of the methyl group resonate as a three proton triplet centered at δ 0.90 ppm. The two protons of CH_2 group adjacent to both CH_2 and CH_3 groups appear as sextet at δ 1.50 ppm ($\text{CH}_3\text{-CH}_2\text{-CH}_2$). A triplet peak centered at δ 2.52 ppm shows the two protons of the CH_2 group attached to the nitrogen atom ($\text{CH}_2\text{-CH}_2\text{-N}$). The protons of the heterocyclic triazine appear at δ 4.25 and 4.71 ppm. The protons of the aromatic system appear as a two doublets at δ 6.91 and 7.18 ppm.

In the ^{13}C NMR spectra of the title compound, the chemical shift values of different carbon atoms of the propyl group appear at δ 11.81, 20.88 and 53.99 ppm. The ^{13}C NMR spectrum display two signals at δ 68.63 and 71.27 ppm corresponding to two carbon atoms of the triazinane cycle. Those of the aryl groups appear at δ 129.13 and 118.75 ppm, and the C-Cl atom appears at δ 125.64 ppm.

The ultra-violet (UV) spectrum of the crystalline form shows two weak signals at 194 and 197 nm characteristic the $n \rightarrow \pi^*$ transition. The other signals appear as intense at 327 and 328 indicating the $\pi \rightarrow \pi^*$ transitions.

4. Conclusion

In this manuscript, we have described the synthesis of a triazacyclohexane derivative by the condensation of 4-chloro

aniline and *n*-propylamine with formalin in the presence of ethanol. The desired product was obtained with good yield and characterized by the usual spectroscopic methods IR, NMR and UV.

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