

Synthesis and characterization of 1,3-bis(2-fluorophenyl)-5-pentyl-1,3,5-triazinane

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ARTICLE INFORMATION



DOI: 10.5155/eurjchem.8.1.18-19.1510

Received: 14 November 2016

Received in revised form: 14 January 2017

Accepted: 14 January 2017

Published online: 31 March 2017

Printed: 31 March 2017

KEYWORDS

Triazinane
 Pentylamine
 Formaldehyde
 2-Fluoroaniline
 IR spectroscopy
 Heterocyclic nitrogen compound

ABSTRACT

A new triazinane derivative was obtained by condensation reaction of pentylamine, 2-fluoroaniline and formaldehyde (formalin) in basic solution to yield 1,3-bis(2-fluorophenyl)-5-pentyl-1,3,5-triazinane. The structure of the synthesized compound was characterized by spectroscopic methods: FT-IR, UV-Vis, ¹³C, ¹⁹F and ¹H NMR.

Cite this: *Eur. J. Chem.* 2017, 8(1), 18-19

1. Introduction

Triazacyclohexanes, as heterocyclic nitrogen compounds, have been proved to play crucial roles in several fields. They represent an important class in coordination chemistry [1]. They are used a framework for conformational studies in structural chemistry [2,3].

Theoretical studies using density functional theory (DFT) at B3LYP/6-31++G (d,p) were carried out on five various *sym*-1,3,5-triaryl-1,3,5-triazacyclohexanes utilized as a corrosion inhibitors mild steel in acidic medium [4] and in organometallic as ligands in preparation of various complexes [5] which can be used as catalyst in the trimerisation and polymerization reactions [6].

The triazacyclohexane was synthesized via condensation reaction between primary amines (arylamine and alkylamine) and formaldehyde (formalin) in basic solution (in aqueous solution of potassium hydroxide) [7,8].

In this study, triazacyclohexane was synthesized from condensation reaction with primary amines and formaldehyde (formalin) in basic solution [8,9].

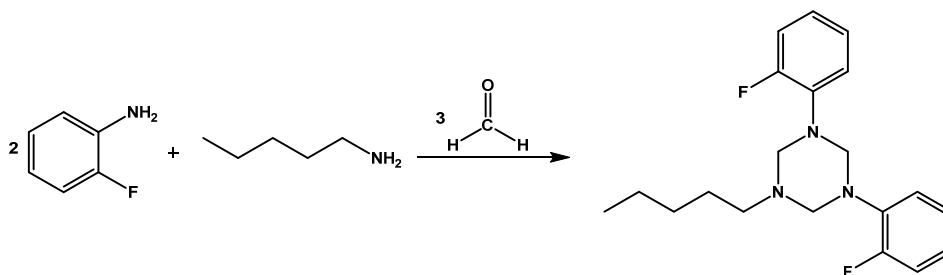
2. Experimental

2.1. Instrumentation

Melting point was determined on a capillary melting point apparatus. Analytical thin layer chromatography (TLC) was conducted on percolated TLC plates (silicagel 60F₂₅₄, Merck) visualized under UV light and stained with dichloromethane as an eluent. ¹H (300 MHz), ¹³C (75 MHz) and ¹⁹F NMR (376 MHz) spectra were recorded on a Bruker spectrometer in CDCl₃ (internal standard and TMS, δ = 0.0 ppm) at room temperature. IR spectra were recorded in KBr pellet on Shimadzu FT-IR 8201 PC (4000-400 cm⁻¹).

2.2. Synthesis

A mixture of 2-fluoroaniline and pentylamine (2:1, v:v) ratio were stirred overnight at ambient temperature with water (10 cm³), potassium hydroxide (1.18 g, 30 mmol) and an excess of formalin (6 mL). The resulting oily phase was extracted with CH₂Cl₂, dried with MgSO₄ and evaporated to dryness [3]. The product was solidified on standing after 2 days [7]. Recrystallization from dichloromethane affords the triazacyclohexane derivative as needles in a high yield.



Scheme 1

1,3-Bis(2-fluorophenyl)-5-pentyl-1,3,5-triazinane: Color: Colorless. Yield: 90%. M.p.: 123-125 °C. FT-IR (KBr, ν , cm^{-1}): 3087 (Ar-H), 2958-2871 (CH_3 , CH_2), 1598, 1496 (C=C), 1461 (CH_2), 1376 (CH_3), 1201 (C-F), 754 (Ar-H). ^1H NMR (300 MHz, CDCl_3 , δ , ppm): 0.88 (t, 3H, CH_3), 1.51 (m, 6H, CH_2), 2.58 (m, 2H, CH_2), 4.28 (s, 4H, $\text{C}_5\text{H}_{11}\text{-N-CH}_2\text{-N-Ar}$), 4.73 (s, 2H, Ar-N- $\text{CH}_2\text{-N-Ar}$), 6.86-7.28 (m, 8H, Ar-H). ^{13}C NMR (75 MHz, CDCl_3 , δ , ppm): 11.90 ($\text{CH}_3\text{-CH}_2$), 20.62 ($\text{CH}_3\text{-CH}_2$), 54.07 ($\text{C}_4\text{H}_9\text{-CH}_2\text{-N}$), 69.58 ($\text{C}_5\text{H}_{11}\text{-N-CH}_2\text{-N-Ar}$), 71.23 (Ar-N- $\text{CH}_2\text{-N-Ar}$), 115.30 (C, Ar-H), 120.07 (C, Ar-F), 122.65 (C, Ar-H). ^{19}F NMR (376.46 MHz, CDCl_3 , δ , ppm): -125.042, -135.617 (C-F). UV/Vis (CHCl_3 , λ_{max} , nm): 238(4).

3. Results and discussion

The condensation of 2-fluoroaniline and pentylamine with formalin afforded an unsymmetrically substituted triazacyclohexane (Scheme 1) [5,6]. This product is stable at room temperature and obtained in high yield 90%. Recrystallization of the obtained solid product from dichloromethane gave transparent needles. The mechanism of this condensation involves the attack of nucleophilic species with formaldehyde to form an imine which trimerizes to give 1,3,5-triazacyclohexane [6,7,9-11].

The characterization of title compound has been explained by FT-IR, UV-Vis, ^1H , ^{13}C and ^{19}F NMR. The infrared spectrum reveals a weak stretching band at 3087 cm^{-1} of C-H aryl system, four strong bands at $2958\text{-}2871\text{ cm}^{-1}$ due to stretching vibration of asymmetric and symmetric stretching of the C-H band (CH_2 and CH_3), at 1598 and 1496 cm^{-1} strong bands characterize the stretching band (C=C) of the aromatic system, a strong band at 1461 cm^{-1} due to in the plane symmetric deformation of the C-H band of the methylene group CH_2 , a medium strong band at 1376 cm^{-1} due to symmetric deformation of three C-H methyl group CH_3 , a strong sharp band at 1201 cm^{-1} resulting from the stretching vibration of C-F, in the deformation zone, a strong band at 754 cm^{-1} due to out of the plane deformation C-H aryl system.

The ^1H NMR spectrum shows protons of the methyl group resonate as triplet at δ 0.88 ppm, the six protons of the three CH_2 group adjacent to both $\text{-CH}_2\text{-N-}$ and CH_3 groups appear as multiplet at δ 1.51 ppm. The multiplet centered at δ 2.58 ppm shows the resonance of the protons of CH_2 of the alkyl attached to the nitrogen. The two various pics of methylene group at δ 4.28 ppm ($\text{CH}_2\text{-N}(\text{C}_5\text{H}_{11})\text{-CH}_2$) and at δ 4.73 ppm (Ar-N- $\text{CH}_2\text{-N-Ar}$) indicate the formation of triazinane derivative. Finally the protons of the aromatic system appear between δ 6.86 and 7.28 ppm.

The ^{13}C NMR spectra shows the carbons atoms of pentyl group appear at δ 11.90, 20.62 and 54.07 ppm, the carbon atoms of triazinane cycle appears at δ 69.58 and 71.23 ppm, the carbons of aryl ring which has a fluorine atoms appear at δ 120.07 ppm, the other carbon atoms of the aryl group appears at δ 115.30 and 122.65 ppm.

4. Conclusion

In conclusion, we have synthesized a new triazinane derivative; the synthesized compound was identified by spectral data. This compound can be a useful ligand for the preparation of organometallic complexes.

Acknowledgements

We gratefully acknowledge Professor Mustapha Bouhenguel Director of the Laboratory of Applied Chemistry and Materials Technology of the University of Oum El Bouaghi for his support of this work. We gratefully acknowledge Mr. Paul Mosset Doctor at the University of Rennes 1, France for providing spectroscopic analysis and we would like to thank Professor Randolph Kohn at University of Bath, England for his help.

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