Synthesis, Molecular Structure from the X-ray Diffraction Data of the Powder (1E,1'E)-1,1'-(1,4-Phenylene)Bis(N-(Adamantan-1-yl)methanimine)

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Abstract
The title compound was synthesized by condensation between two equivalents adamantan-1-ylamine and one equivalent of benzene-1,4-dicarbaldehyde in n-BuOH and produced a good yield 87% of new bis Schiff base. The compound skeleton was affirmed by FTIR, 1H NMR, LC-MS, and X-ray powder diffraction. The structure was solved by a parallel tempering process and refined by using Rietveld refinement. Two adamantan-1-ylimino groups are connected in the anti-positions to the planar central 1,4-dimethylbenzene group. All rings of the adamantyl group possess normal chair conformation.

Keywords: Powder X-ray diffraction, Adamantan-1-ylamine, Bis Schiff base.

Introduction
The molecular design of adamantane and its derivatives are actual importance for researchers in molecular technology. Adamantan-1-ylamine is an alicyclic tricyclic amine which has an unusual skeleton adamantyl fragment consisting of three fused cyclohexane rings in a chair conformation [1]. It is the smallest repeating fragment of the diamond lattice [2]. The synthesis of molecules containing two or more pharmaceutical fragments in their structure allows for the development of new types of biological properties and for the extension of known curative effects. Adamantan-1-amine and its derivatives have been a good source for a wide scope of pharmaceutical drugs. In particular, they have...
been effective in the development of drugs for Parkinson’s disease [3], treatment and protection against influenza virus (A) [4], anti-microbial drugs [5], and anti-inflammatory drugs [6]. Furthermore, compounds that contain azomethines are reactive intermediates for organic synthesis in different fields and have biological [7, 8], anti-viral [9], anti-bacterial [10,11], and anti-fungal [12] effects, as well as inhibiting anti-microbial agents [13]. A search in the CCDC Database survey gave few results containing adamantan-1-ylaminomethylbenzene fragment or 1,4-di(aminomethyl)benzene fragment [14-20]. Structures similar to the title compound are absent in the database. This work presents new applications of bis imine (1E,1’E)-1,1’-(1,4-phenylene)bis(N-(adamantan-1-yl)methanimine) as a predece-ssor for the synthesis of new compounds. The structure of the desired compound was confirmed by the use of the X-ray powder diffraction technique.

Materials and Methods

The materials were purchased from Merck (Germany) and Romil Co. (UK). The melting point was measured by the Stuart SMP-10 apparatus. FTIR, 1H NMR and LC-MS spectra were recorded on Bruker Tensor 27, Bruker UltraShield (300 MHz, CDCl3), and Thermo Scientific Exactive Plus Orbitrap LC-MS spectrometer, respectively. X-ray powder diffraction pattern was measured on the PANalytical Empyrean diffractometer.

Synthesis of the title compound

To a hot solution of adamantan-1-ylamine (4 g, 0.026 mol) in 15 ml n-BuOH a solution of benzene-1,4-dicarbaldehyde (1.74 g, 0.013 mol) in 10 ml n-BuOH was added. The mixture was refluxed with stirring for 30 min and was then left for 3 h. The crude product was filtered and evaporated. The product was collected and re-crystallized from EtOH or i-PrOH.

The properties and spectral data of the title compound:

White needles, yield 4.6 g (87%), m.p 243–244°C. FTIR, ν, cm⁻¹: 1633 (C=N), 3013 (C—H aromatic), 2904, 2846 (C—H aliphatic). 1H NMR (CDCl3, δ, 300 MHz, p.p.m.): (adamantyl group 1.66 (s, 12H, 6CH2), 1.85 (m, 6H, 6CH), 2.23 (broad s, 12H, 6CH2)), 7.92–8.26 (m, 4H, Ar-ring), 8.32 (s, 2H, 2 CH=N). LC-MS, m/z (%): 400.89 [M]+ (100).

X-ray powder structure of the title compound: The powder as very small needles was obtained by allowing the saturated EtOH solution of the compound to stand for four days. The details of crystallographic data are shown in Tables 1-4.

Results and Discussion

The route of the synthesis of the title compound is shown in scheme 1, which includes the synthesis of new bis Schiff base (1E,1’E)-1,1’-(1,4-phenylene)bis(N-(adamantan-1-yl)methanimine) by condensation between two equivalents adamantan-1-ylamine and one equivalent of benzene-1,4-dicarbaldehyde in the presence of n-butanol as a solvent, and produced a good yield (87%). The skeleton of the compound was affirmed by FTIR, 1H NMR, LC-MS, and powder X-ray diffraction.

![Scheme 1](image-url)  
*Scheme 1- For the synthesis of the title structure*
FTIR spectrum displayed evanescence in the stretching vibration bands of groups (-NH₂) and (C=O) for amine and benzene-1,4-dicarbaldehyde respectively with appeared stretching vibration band of the azomethine group (C=N) at 1633 cm⁻¹. The stretching vibration band for (C-H aromatic) appeared at 3013 cm⁻¹, and stretching vibration bands for (C-H aliphatic) appeared at 2846 and 2904 cm⁻¹ (Figure-1). The ¹H NMR spectrum for (2CH=N) protons displayed the presence of a down-field singlet at δ 8.32 ppm and protons of phenyl group displayed two singlets at δ 7.92 and 8.26 ppm. The adamantyl group exhibited signals as a singlet, multiplet, and broad singlet at δ 1.66 (12H, 6CH₂C-N), 1.85 (6H, 6CH) and 2.23 (12H, 6CH₂) ppm, respectively (Figure-2). Moreover, LC—mass spectrum was identical to its molecular weight, m/z (%): 400.89 [M]+ (100) (Figure-3). The formation of the bis imine derivative title compound was occurred according to a similar suggested mechanism in literature [12] but in the absent catalyst. The two pairs of electrons nitrogen of two molecules amine attacks to the dicarbonyl group by nucleophilic addition produced dihemi-aminal and then the left two water molecules and gave the target compound. Scheme-2.

Ad=adamantyl group

Scheme 2-The suggested mechanism of bis imine for target compound
Figure 1-FTIR spectrum of the title compound

Figure 2-$^1$H NMR spectrum of the title compound
Crystallographic Study

The molecular structure of the title compound was displayed in Figure-4. Its crystalline data were as follows: C_{28}H_{36}N_{2}, M.W= 400.59 g.mol^{-1}, system with space group: Monoclinic, C2/c, a=28.249 (10) Å, b= 6.450 (18) Å, c= 12.509 (4) Å, β= 91.88 (4), V=2278 (6) Å³, Z= 4, Rad. type Cu Ka, λ = 1.5418 Å. Specimen shape, size: Flat sheet (15×1) mm.

The X-ray powder diffraction pattern in (Figure-6) was measured by computer programs in the θ range 5-70° on PANalytical Empyrean diffractometer [21]. The structure was solved by parallel tempering technique using FOX [22], and refined by Rietveld refinement procedure was made by MRIA [23] with constrained isotropic thermal parameters for all non-hydrogen atoms. Molecular graphics was made by Mercury [24]. Hydrogen atoms were calculated and not refined. The bond length of (N6-C12) for (N=C) is normal 1.32 (2) Å. In the crystal structure of the target compound, was observed two adamantant-1-ylimino groups are arranged in the anti-positions to the planar central 1, 4-dimethylbenzene group (Figure-4). The molecule is located on the inversion center. All rings of adamantyl moiety possess normal chair conformation. The details of the crystal data are provided in Tables-(1-4).
**Figure 4**-Data block: Showing the molecular structure, atoms numbering and displacement ellipsoids are drawn at 50% probability level of the title compound.

**Figure 5**-Powder diffraction pattern of the title compound.
Table 1-Crystal data and refinement details of the title compound

| Crystal data          |   |
|-----------------------|---|
| M.F                   | C_{28}H_{36}N_{2} |
| M.W                   | 400.59          |
| Crystal system and    | Monoclinic, C2/c |
| group of space        |               |
| Temp.                 | 298 K          |
| a,b,c (Å)             | 28.249 (10), 6.450 (18), 12.509 (4) |
| β (°)                 | 91.88 (4)      |
| V (Å$^3$)             | 2278 (6)       |
| Z                     | 4              |

Type of radiation: Cu Kα, $\lambda=1.5418$ Å

Shape and size of specimen: Flat sheet, 15×1 mm

Collection data

Diffractometer: EMPYREAN (PANanalytical, 2011)

Mounting of specimen: Thin layer over the non-diffracting silicon plate

Mode collection data: Reflection

Scan process: Persistent

$2\theta$ values: $2\theta_{\text{min}} = 5.00°$, $2\theta_{\text{max}} = 70.00°$, $2\theta_{\text{exp}} = 0.01°$

Refinement

R-factors and quality of fit: $R_p=0.026$, $R_{wp}=0.033$, $R_{\text{exp}}=0.013$, $R_{\text{Bragg}}=0.109°$, $\chi^2=7.618$

Parameters number: 104

Restraints number: 48

Treatment of H-atoms: Parameters of H-atoms not refined

Code of symmetry: (i) $-x$, $-y+1$, $-z+1$

CCDC reference, deposition number: 1971146

Table 2-Geometric parameters (Bonds lengths) (Å,$^\circ$) of the title compound

| Bond | d (Å,$^\circ$) | Bond | d (Å,$^\circ$) |
|------|--------------|------|--------------|
| C1-C2| 1.534 (19)   | C1-H1| 0.9800       |
| C1-C9| 1.57 (3)     | C2-H2A| 0.9700      |
| C1-C10| 1.54 (3)    | C2-H2B| 0.9700      |
| C2-C3| 1.55 (2)     | C3-H3 | 0.9800       |
| C3-C4| 1.535 (19)   | C4-H4A| 0.9700      |
| C3-C11| 1.54 (2)    | C4-H4B| 0.9700      |
| C4-C5| 1.528 (16)   | C7-H7A| 0.9700      |
| C5-N6| 1.48 (2)     | C7-H7B| 0.9700      |
| C5-C7| 1.54 (2)     | C8-H8 | 0.9800       |
| C5-C10| 1.55 (2)    | C9-H9A| 0.9700      |
| N6-C12| 1.32 (2)    | C9-H9B| 0.9700      |
| C7-C8| 1.541 (19)   | C10-H10A| 0.9700   |
| C8-C9| 1.555 (19)   | C10-H10B| 0.9700   |
| C8-C11| 1.555 (17)  | C11-H11A| 0.9700   |
| C12-C13| 1.501 (18) | C11-H11B| 0.9700   |
| C13-C14| 1.41 (3)   | C12-H12| 0.9300      |
| C13-C15| 1.39 (2)   | C14-H14| 0.9300      |
| C14-C15i| 1.4213 (2) | C15-H15| 0.9300      |
| C15-C14i| 1.4213 (2) |        |             |
Table 3- Geometric parameters (Bonds angles $\omega$, °) of the title compound

| Angle     | $\omega$, ° | Angle     | $\omega$, ° |
|-----------|-------------|-----------|-------------|
| C2-C1-C9  | 106.5 (7)   | C11-C3-H3 | 110.5       |
| C2-C1-C10 | 108.1 (5)   | C2-C3-H3  | 110.5       |
| C9-C1-C10 | 104.9 (4)   | C5-C4-H4A | 109.7       |
| C1-C2-C3  | 112.5 (3)   | C3-C4-H4A | 109.7       |
| C2-C3-C4  | 107.1 (3)   | C5-C4-H4B | 109.7       |
| C2-C3-C11 | 109.7 (5)   | C3-C4-H4B | 109.7       |
| C4-C3-C11 | 108.6 (3)   | H4A-C4-H4B| 108.2       |
| C3-C4-C5  | 110.0 (5)   | C5-C7-H7A | 109.3       |
| C4-C5-N6  | 111.0 (9)   | C8-C7-H7A | 109.3       |
| C4-C5-C7  | 111.9 (6)   | C5-C7-H7B | 109.3       |
| C4-C5-C10 | 107.4 (4)   | C8-C7-H7B | 109.3       |
| N6-C5-C7  | 108.4 (8)   | H7A-C7-H7B| 108.0       |
| N6-C5-C10 | 108.0 (6)   | C7-C8-H8  | 112.9       |
| C7-C5-C10 | 109.9 (2)   | C9-C8-H8  | 112.9       |
| C5-N6-C12 | 119.4 (2)   | C11-C8-H8 | 112.9       |
| C5-C7-C8  | 111.6 (8)   | C8-C9-H9A | 108.0       |
| C7-C8-C9  | 106.8 (4)   | C1-C9-H9A | 108.0       |
| C7-C8-C11 | 106.0 (3)   | C8-C9-H9B | 108.0       |
| C9-C8-C11 | 104.9 (5)   | C1-C9-H9B | 108.0       |
| C1-C9-C8  | 116.9 (4)   | H9A-C9-H9B| 107.3       |
| C1-C10-C5 | 111.9 (5)   | C1-C10-H10A| 109.2      |
| C3-C11-C8 | 114.5 (6)   | C5-C10-H10A| 109.2      |
| N6-C12-C13| 126.3 (4)   | C1-C10-H10B| 109.2      |
| C12-C13-C14| 118.3 (5) | C5-C10-H10B| 109.2      |
| C12-C13-C15| 121.0 (5) | H10A-C10-H10B| 107.9     |
| C14-C13-C15| 120.6 (8) | C3-C11-H11A| 108.6      |
| C13-C14-C15i| 119.6 (7)| C8-C11-H11A| 108.6      |
| C13-C15-C14i| 119.8 (7) | C3-C11-H11B| 108.6      |
| C2-C1-H1  | 112.3       | C8-C11-H11B| 108.6      |
| C10-C1-H1 | 112.3       | H11A-C11-H11B| 107.6     |
| C9-C1-H1  | 112.3       | N6-C12-H12 | 116.8       |
| C1-C2-H2A | 109.1       | C13-C12-H12| 116.8       |
| C3-C2-H2A | 109.1       | C13-C14-H14| 120.2       |
| C1-C2-H2B | 109.1       | C15i-C14-H14| 120.2     |
| C3-C2-H2B | 109.1       | C13-C15-H15 | 120.1      |
| H2A-C2-H2B| 107.8       | C14i-C15-H15| 120.1      |
| C4-C3-H3  | 110.5       |             |             |
Table 4—Parameters of displacement (Å²) for fractional atomic coordinates and isotropic or equivalent isotropic of the title compound

| atoms | x    | y    | z    | Uiso*/Ueq |
|-------|------|------|------|-----------|
| C1    | 0.1425 (5) | 0.969 (3) | 0.0600 (13) | 0.05066* |
| C2    | 0.1416 (5) | 0.766 (2) | −0.0039 (8)  | 0.05066* |
| C3    | 0.1552 (5) | 0.575 (3) | 0.0662 (13)  | 0.05066* |
| C4    | 0.1191 (4) | 0.5625 (16) | 0.1551 (11)  | 0.05066* |
| C5    | 0.1226 (4) | 0.7559 (19) | 0.2255 (10)  | 0.05066* |
| N6    | 0.0886 (4) | 0.745 (2)  | 0.3131 (8)   | 0.05066* |
| C7    | 0.1726 (4) | 0.785 (2)  | 0.2750 (9)   | 0.05066* |
| C8    | 0.2099 (6) | 0.806 (2)  | 0.1884 (13)  | 0.05066* |
| C9    | 0.1935 (4) | 0.9865 (18) | 0.1138 (13)  | 0.05066* |
| C10   | 0.1091 (4) | 0.9450 (17) | 0.1539 (10)  | 0.05066* |
| C11   | 0.2049 (4) | 0.6075 (19) | 0.1181 (13)  | 0.05066* |
| C12   | 0.0661 (3) | 0.571 (2)  | 0.3308 (8)   | 0.05066* |
| C13   | 0.0311 (4) | 0.5325 (19) | 0.4162 (9)   | 0.05066* |
| C14   | 0.0207 (5) | 0.6963 (12) | 0.4860 (10)  | 0.05066* |
| C15   | 0.0110 (4) | 0.3373 (14) | 0.4298 (8)   | 0.05066* |
| H1    | 0.1345     | 1.0894   | 0.0152       | 0.061*    |
| H2A   | 0.1635     | 0.7769   | −0.0618      | 0.061*    |
| H2B   | 0.1101     | 0.7455   | −0.0353      | 0.061*    |
| H3    | 0.1544     | 0.4481   | 0.0231       | 0.061*    |
| H4A   | 0.0874     | 0.5513   | 0.1236       | 0.061*    |
| H4B   | 0.1252     | 0.4400   | 0.1984       | 0.061*    |
| H7A   | 0.1805     | 0.6671   | 0.3203       | 0.061*    |
| H7B   | 0.1732     | 0.9083   | 0.3194       | 0.061*    |
| H8    | 0.2421     | 0.8256   | 0.2184       | 0.061*    |
| H9A   | 0.2161     | 0.9992   | 0.0574       | 0.061*    |
| H9B   | 0.1950     | 1.1139   | 0.1549       | 0.061*    |
| H10A  | 0.0769     | 0.9282   | 0.1261       | 0.061*    |
| H10B  | 0.1103     | 1.0700   | 0.1970       | 0.061*    |
| H11A  | 0.2277     | 0.6146   | 0.0620       | 0.061*    |
| H11B  | 0.2128     | 0.4877   | 0.1621       | 0.061*    |
| H12   | 0.0725     | 0.4599   | 0.2859       | 0.061*    |
| H14   | 0.0345     | 0.8258   | 0.4771       | 0.061*    |
| H15   | 0.0183     | 0.2293   | 0.3839       | 0.061*    |

**Conclusion**

The research included a synthesis of new bis Schiff base which contains adamantyl moiety by condensation between two equivalents adamantan-1-ylamine and one equivalent of benzen-1,4-dicarbaldehyde in n-BuOH gave (1E,1′E)-1,1′-(1,4-phenylene)bis(N-(adamantan-1-yl)methanimine) with good yield. The skeleton of the compound was affirmed by FTIR, 1H NMR, LC-MS spectroscopy, and X-ray powder diffraction techniques. The structure was solved by a parallel tempering process and refined by Rietveld refinement. The positions of two adamantanyl groups are connected in the anti-conformation to the planar central 1,4-dimethylbenzene group. The rings of the adamantyl fragment showed that have a normal chair conformation.

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