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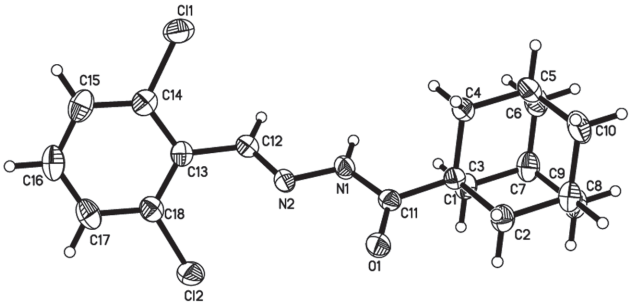
Crystal structure of *N'*-[(1*E*)-(2,6-dichlorophenyl)-methylidene]adamantane-1-carbohydrazide, $C_{18}H_{20}Cl_2N_2O$



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Abstract

$C_{18}H_{20}Cl_2N_2O$, orthorhombic, *Pbca* (No. 61), $a = 8.1023(2)$ Å, $b = 18.7063(4)$ Å, $c = 22.5509(6)$ Å, $V = 3417.91(14)$ Å^3, $Z = 8$, $R_{gt}(F) = 0.0496$, $wR_{ref}(F^2) = 0.1535$, $T = 293(2)$.

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The asymmetric unit of the crystal structure is shown in the figure. Tables 1 and 2 contain details of the measurement method and a list of the atoms including atomic coordinates and displacement parameters.

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Table 1: Data collection and handling.

Crystal:	Colorless prism
	Size 0.26 × 0.03 × 0.02 mm
Wavelength:	Cu K_α radiation (1.5418)
μ:	34.6 cm^{-1}
Diffractometer, scan mode:	Xcalibur, Onyx, Nova, ω
$2\theta_{max}$, completeness:	139°, >99 %
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	14651, 3181, 0.070
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2771
$N(param)_{refined}$:	155
Programs:	CrysAlis^{PRO} [24], SHELXT [25], SHELX [26]

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2).

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
Cl1	0.05709(9)	0.57373(4)	0.43640(3)	0.0529(2)
Cl2	0.45333(9)	0.43699(4)	0.27823(3)	0.0511(2)
O1	0.3971(2)	0.65850(10)	0.18889(8)	0.0411(4)
N1	0.1666(2)	0.61834(11)	0.23417(9)	0.0354(5)
H1	0.0606	0.6167	0.2349	0.043*
N2	0.2591(2)	0.57924(10)	0.27392(8)	0.0322(4)
C1	0.1386(3)	0.70750(12)	0.15542(10)	0.0310(5)
C13	0.2619(3)	0.50570(11)	0.36015(10)	0.0326(5)
C3	0.0248(3)	0.66168(14)	0.11591(11)	0.0375(5)
H3A	0.0909	0.6304	0.0911	0.045*
H3B	−0.0459	0.6322	0.1405	0.045*
C11	0.2475(3)	0.65977(12)	0.19355(9)	0.0304(5)
C12	0.1762(3)	0.54631(12)	0.31357(10)	0.0331(5)
H12	0.0615	0.5480	0.3130	0.040*
C2	0.2492(3)	0.75315(13)	0.11537(11)	0.0387(6)
H2A	0.3158	0.7223	0.0904	0.046*
H2B	0.3229	0.7818	0.1395	0.046*
C4	0.0332(3)	0.75710(14)	0.19440(11)	0.0417(6)
H4A	0.1043	0.7861	0.2193	0.050*
H4B	−0.0375	0.7288	0.2200	0.050*
C17	0.4679(4)	0.42118(15)	0.39580(14)	0.0475(7)
H17	0.5535	0.3895	0.3877	0.057*
C8	0.0304(4)	0.75697(14)	0.03766(12)	0.0453(6)
H8A	0.0963	0.7266	0.0121	0.054*
H8B	−0.0368	0.7877	0.0128	0.054*
C14	0.2153(3)	0.51428(12)	0.41956(11)	0.0381(5)

Table 2 (continued)

Atom	x	y	z	U_{iso}*/U_{eq}
C9	0.1429(3)	0.80229(13)	0.07645(12)	0.0425(6)
H9	0.2148	0.8314	0.0512	0.051*
C10	0.0391(4)	0.85104(15)	0.11556(13)	0.0515(7)
H10A	0.1106	0.8803	0.1401	0.062*
H10B	−0.0271	0.8825	0.0910	0.062*
C7	−0.0810(3)	0.71083(15)	0.07687(12)	0.0451(6)
H7	−0.1536	0.6818	0.0518	0.054*
C16	0.4185(4)	0.43223(15)	0.45313(14)	0.0505(7)
H16	0.4713	0.4082	0.4838	0.061*
C5	−0.0729(4)	0.80563(16)	0.15465(13)	0.0518(7)
H5	−0.1404	0.8370	0.1796	0.062*
C18	0.3903(3)	0.45717(12)	0.34994(12)	0.0374(5)
C15	0.2910(4)	0.47877(15)	0.46575(12)	0.0482(7)
H15	0.2568	0.4860	0.5047	0.058*
C6	−0.1852(4)	0.75930(17)	0.11648(14)	0.0549(8)
H6A	−0.2551	0.7895	0.0921	0.066*
H6B	−0.2557	0.7304	0.1417	0.066*

Source of material

2,6-Dichlorobenzaldehyde (1.75 g, 0.01 mol) was added to a solution of adamantane-1-carbohydrazide (1.94 g, 0.01 mol), in ethanol (15 mL), and the mixture was heated under reflux for 1 h. On cooling, the precipitated crystalline product was filtered, washed with cold ethanol, dried and crystallized from ethanol to yield 3.06 g (87%) of the title compound as colourless prismatic crystals. M.p. 529–531 K [23]. **^{1}H NMR** (DMSO-d_6, 500.13 MHz): δ 1.65–1.69 (m, 6H, adamantane-H), 1.86–1.88 (m, 6H, adamantane-H), 2.0 (s, 3H, adamantane-H), 5.35 (s, 1H, NH), 7.15–7.25 (m, 3H, Ar-H), 7.95 (s, 1H, CH = N). **^{13}C NMR** (DMSO-d_6, 125.76 MHz): δ 26.40, 33.51, 35.01, 39.99 (adamantane-C), 126.87, 130.05, 133.53, 134,95 (Ar-C), 143.69 (CH = N), 175.90 (C = O).

Experimental details

Carbon-bound H atoms were placed in calculated positions and were included in the refinement in the riding model approximation. The U_{iso} values of the nitrogen-bound H atom was set to $1.2U_{eq}$(N).

Discussion

The broad and potent activity of adamantane-based derivatives has established as one of the biologically important scaffolds. Adamantane derivatives were reported to possess a wide spectrum of biological properties including antiviral [1–8], anticancer [9, 10], antibacterial [11–14], antifungal [15], antimalarial [16] and antidiabetic [17] activities. In addition, a number of hydrazide-hydrazone derivatives have been claimed to possess interesting biological activities [18–22]. In the present study, we report the X crystal structure of the title adamantane-1-carohydrazide-hydrazone as intermediate for the synthesis of surpassing potential chemotherapeutic agents [23].

The asymmetric unit cell of the title compound contains one molecule. All bond lengths and angles of the title molecule are in the expected ranges. The molecules are weakly connected in the crystal structure *via* four intermolecular hydrogen bonds, N1—H1···O1^i, N1—H1···N2^i, C4—H4B···O1^i and C12—H12···O1^i. The H···A distances are 2.31, 2.55, 2.50 and 2.46 Å, respectively and the angles are 125, 165, 154 and 125°, respectively. Symmetry codes: (i) $x-1/2$, y, $-z+1/2$.

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