Crystal structure of 1-(1-(2,4-dichlorobenzyl)-5-methyl-1*H*-1,2,3-triazol-4-yl)ethanone, $C_{12}H_{11}Cl_2N_3O$

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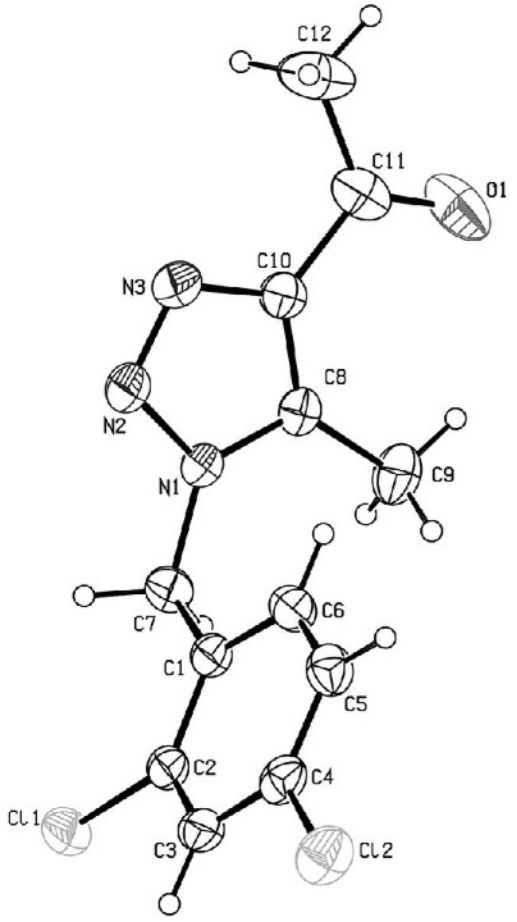

Abstract

$C_{12}H_{11}Cl_2N_3O$, triclinic, $P\bar{1}$ (no. 2), a = 7.0448(14) Å, b = 8.6159(17) Å, c = 11.760(2) Å, α = 98.53(3)°, β = 99.99(3)°, γ = 109.55(3)°, V = 645.9 Å^3, Z = 2, $R_{gt}(F)$ = 0.0425, $wR_{ref}(F^2)$ = 0.1247, T = 290 K.

Source of material

To 11.0 g (54.4 mmol) of 2,4-dichlrobenzyl chloride, 4.60 g (70.68 mmol) of sodium azide in 150 ml of acetonitrile were added [1]. The mixture of reaction was refluxed for 15 hours. Thereafter, the mixture is cooled and 50 mL of water was added and extracted with chloroform (3×40 mL). The organic solution was dried with sodium sulfate and distilled at vacuum. The yellow oily product obtained was dissolved in 70 mL of DMSO and then, 16.19 mL (156.84 mmol) of acetylacetone and 21.7 g (214.8 mmol) of potassium carbonate were added. The mixture was left to react for 48 hours at room temperature [2]. Finally, 60 mL of saturated solution of NaCl was added and the precipitate formed was washed, dried and crystallized in ethyl acetate, obtaining 12.69 g (yield 84%) of yellow crystals. Analysis: Solid crystalline **m.p.** 385 K **IR** (cm^{-1}) ν: 3076 (C_{Ar}-H); 2974 and 2924 (C_{sp^3}-H); 1680 (C=O); 1588 and 1562 (C_{Ar}-C_{Ar}). **^{1}H-NMR** ($CDCl_3$, 500 MHz) δ (ppm): 2,50 (3H, s, H15), 2,70 (3H, s, H18), 5,58 (2H, s, H7), 6,79 (1H, d, J = 8,3 Hz, H6), 7,21 (1H, d, J = 8,4 Hz, H1), 7,46 (1H, s, H3). **^{13}C-NMR** ($CDCl_3$, 125,70 MHz) δ (ppm): 9.17(C15), 27.85(C18), 48.21(C7), 128.12(C1), 129.69(C6), 129.86(C3), 130.59(C5), 133.37(C2), 135.44(C4), 137.28(C14), 144.04(C13), 194.41(C16).

Table 1. Data collection and handling.

Crystal:	white blocks, size 0.07×0.11×0.16 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	4.93 cm^{-1}
Diffractometer, scan mode:	Enraf Nonius CAD4, non-profiled ω
$2\theta_{max}$:	52.01°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	5082, 2545
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2092
$N(param)_{refined}$:	166
Programs:	SHELX, PLATON, XCAD4, OLEX [8–11]

Experimental details

H atoms were located in the difference Fourier map, but refined with fixed individual displacement parameters, using a riding mode with C–H distances of 0.93 Å (for aromatic rings), 0.97 Å (for CH_2), 0.96 Å (for CH_3), with U_{iso}(H) values of 1.2U_{eq}(C) (for CH in aromatic and methylene group), and 1.5U_{eq}(C) (for methyl group).

Discussion

1,2,3-Triazoles are an important class of heterocyclic compounds due to their wide range of applications as pharmaceutical agents [3, 4]. The diversity of chemical structures of the 1,2,3-triazole family and their useful biological activities made these compounds attractive targets in synthetic organic chemistry and many studies have been reported on the synthesis of these compounds [5, 7]. The title compound crystallizes in space group $P\bar{1}$. The bond lengths and angles are in the expected ranges. The dihedral angles between the mean plane of the benzene ring and the triazol ring is 86.64(12)° [for the non-H atoms, r.m.s. deviations = 0.006 and 0.002 Å, respectively]. The crystal packing is stabilized by a weak intermolecular C–H···O^i hydrogen bond, [symmetry code

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(i): –x+1,–y+2,–z+1] which links the molecules into centrosymetric ring with graph-set notation $R_2^2(18)$ [10]. The packing also features π–π stacking interactions between triazole rings [centroid–centroid[ii] distance 3.6770(13) Å, symmetry code (ii) –x+1,–y+1,–z+1] and between benzene rings [centroid–centroid[iii] distance 3.8279(15) Å, symmetry code (iii): –x,–y+1,–z].

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	*x*	*y*	*z*	U_{iso}
H(3)	2*i*	0.1602	0.4135	–0.1406	0.061
H(5)	2*i*	0.3813	0.8825	0.0640	0.065
H(6)	2*i*	0.4367	0.7745	0.2295	0.062
H(7A)	2*i*	0.2304	0.3958	0.2693	0.063
H(7B)	2*i*	0.4274	0.3737	0.2363	0.063
H(9A)	2*i*	0.1661	0.6854	0.3746	0.102
H(9B)	2*i*	0.2263	0.7301	0.5134	0.102
H(9C)	2*i*	0.1486	0.5416	0.4443	0.102
H(12A)	2*i*	0.9090	1.0698	0.7624	0.141
H(12B)	2*i*	0.9770	1.0622	0.6422	0.141
H(12C)	2*i*	0.9575	0.9140	0.7079	0.141

Table 3. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Cl(1)	2*i*	0.17456(8)	0.20684(6)	0.01875(5)	0.0546(3)	0.0450(3)	0.0705(4)	0.0137(2)	0.0084(2)	–0.0027(2)
Cl(2)	2*i*	0.2356(1)	0.74072(9)	–0.17872(5)	0.0732(4)	0.0917(5)	0.0634(4)	0.0379(3)	0.0206(3)	0.0302(3)
O(1)	2*i*	0.5387(4)	0.9439(3)	0.6552(2)	0.122(2)	0.076(1)	0.082(1)	0.027(1)	0.045(1)	–0.007(1)
N(1)	2*i*	0.4945(2)	0.5826(2)	0.3595(1)	0.0377(8)	0.0492(9)	0.0493(8)	0.0179(7)	0.0081(6)	0.0067(7)
N(2)	2*i*	0.7031(3)	0.6251(2)	0.3794(2)	0.0402(9)	0.065(1)	0.072(1)	0.0227(8)	0.0129(8)	0.0020(9)
N(3)	2*i*	0.7860(3)	0.7430(2)	0.4754(2)	0.0440(9)	0.059(1)	0.069(1)	0.0170(8)	0.0036(8)	0.0044(9)
C(1)	2*i*	0.3287(3)	0.5293(2)	0.1471(2)	0.0338(9)	0.0460(9)	0.052(1)	0.0146(7)	0.0109(7)	0.0038(8)
C(2)	2*i*	0.2436(3)	0.4243(2)	0.0342(2)	0.0311(8)	0.0443(9)	0.058(1)	0.0121(7)	0.0111(7)	0.0017(8)
C(3)	2*i*	0.2140(3)	0.4859(3)	–0.0661(2)	0.0363(9)	0.061(1)	0.050(1)	0.0168(9)	0.0099(8)	0.0005(9)
C(4)	2*i*	0.2666(3)	0.6586(3)	–0.0532(2)	0.039(1)	0.067(1)	0.054(1)	0.0221(9)	0.0161(8)	0.0165(9)
C(5)	2*i*	0.3480(3)	0.7670(3)	0.0565(2)	0.053(1)	0.049(1)	0.064(1)	0.0186(9)	0.0189(9)	0.0122(9)
C(6)	2*i*	0.3796(3)	0.7013(2)	0.1554(2)	0.051(1)	0.048(1)	0.051(1)	0.0156(9)	0.0128(8)	0.0025(8)
C(7)	2*i*	0.3635(3)	0.4552(2)	0.2538(2)	0.050(1)	0.045(1)	0.057(1)	0.0141(8)	0.0086(9)	0.0051(8)
C(8)	2*i*	0.4444(3)	0.6754(2)	0.4429(2)	0.045(1)	0.049(1)	0.049(1)	0.0219(8)	0.0158(8)	0.0160(8)
C(9)	2*i*	0.2271(4)	0.6565(3)	0.4439(2)	0.052(1)	0.079(2)	0.085(2)	0.031(1)	0.028(1)	0.024(1)
C(10)	2*i*	0.6324(3)	0.7781(2)	0.5169(2)	0.052(1)	0.046(1)	0.048(1)	0.0179(8)	0.0102(8)	0.0122(8)
C(11)	2*i*	0.6779(4)	0.9095(3)	0.6238(2)	0.088(2)	0.049(1)	0.049(1)	0.017(1)	0.016(1)	0.0131(9)
C(12)	2*i*	0.9003(5)	0.9967(4)	0.6900(2)	0.115(2)	0.062(2)	0.067(2)	0.007(2)	–0.016(2)	0.006(1)

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