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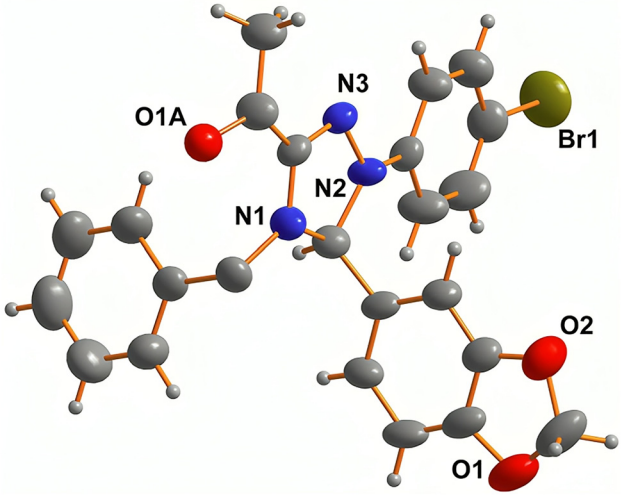
Crystal structure of 1-(5-(benzo[*d*][1,3]dioxol-5-yl)-4-benzyl-1-(4-bromophenyl)-4,5-dihydro-1*H*-1,2,4-triazol-3-yl)ethan-1-one, $C_{24}H_{20}BrN_3O_3$



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Abstract

$C_{24}H_{20}BrN_3O_3$, triclinic, $P\bar{1}$ (no. 2), a = 10.830(2) Å, b = 11.030(2) Å, c = 11.680(2) Å, α = 97.20(3)°, β = 116.70(3)°, γ = 112.50(3)°, V = 1,074.3(5) Å^3, Z = 2, $R_{gt}(F)$ = 0.0809, $wR_{ref}(F^2)$ = 0.1895, T = 298(2) K.

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The molecular structure is shown in the figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

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Table 1: Data collection and handling.

Crystal:	Colorless block
Size:	0.18 × 0.18 × 0.15 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	1.94 mm^{-1}
Diffractometer, scan mode:	STOE IPDS II, ω
θ_{max}, completeness:	25.7°, 99 %
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	9,356, 4,041, 0.072
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2,368
$N(param)_{refined}$:	281
Programs:	X-Area,[1] X-RED,[2] SHELX,[3] WinGX/ORTEP-3,[4] Mercury[5]

1 Source of materials

The title compound was synthesized by 1,3-dipolar cycloaddition reaction (1,3–DPCA) of N-(3,4–methylenedioxybenzylidene) benzylamine and N-(4-bromophenyl)-2-oxopropanehydrazonoyl chloride during our research in the construction of chiral 1,2,4-triazoline derivatives. The reaction mixture was refluxed in ethanol with an excess amount of triethylamine according to the procedure reported earlier.[6] Colorless blocks for the compound, suitable for the X-ray structure analysis, were obtained after slow evaporation of ethanolic solution of the crude compound.

2 Experimental details

All the non-hydrogen atoms were refined as anisotropic except oxygen atom O1 which splits into two positions as O1A and O1B. The hydrogen atoms were generated geometrically with U_{iso}(H) = 1.2.

3 Comment

1,2,4–Triazolines stand as an intriguing yet underexplored category of heterocycles, holding considerable promise for

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
C1	0.7147 (6)	0.4279 (6)	0.6198 (6)	0.0402 (13)
C2	0.5452 (7)	0.3057 (6)	0.6832 (6)	0.0446 (13)
H2	0.594763	0.309714	0.778455	0.053*
C3	0.3783 (7)	0.1801 (6)	0.5945 (6)	0.0426 (13)
C4	0.3347 (7)	0.0712 (6)	0.6399 (7)	0.0485 (14)
H4	0.405515	0.078891	0.727407	0.058*
C5	0.1854 (8)	−0.0510 (7)	0.5564 (8)	0.0575 (16)
H5	0.155767	−0.125535	0.585803	0.069*
C6	0.0865 (8)	−0.0549 (6)	0.4309 (8)	0.0550 (16)
C7	−0.1127 (9)	−0.1163 (8)	0.2164 (9)	0.082 (2)
H7A	−0.122230	−0.176076	0.141066	0.098*
H7B	−0.215049	−0.122365	0.185388	0.098*
C8	0.1284 (7)	0.0550 (7)	0.3872 (7)	0.0501 (14)
C9	0.2737 (7)	0.1751 (6)	0.4653 (6)	0.0478 (14)
H9	0.301277	0.248735	0.434320	0.057*
C10	0.5082 (6)	0.5129 (6)	0.7379 (6)	0.0411 (13)
C11	0.4187 (8)	0.4459 (7)	0.7892 (7)	0.0641 (18)
H11	0.395977	0.354746	0.785918	0.077*
C12	0.3630 (9)	0.5132 (8)	0.8453 (8)	0.070 (2)
H12	0.300873	0.466707	0.877196	0.084*
C13	0.4001 (8)	0.6482 (7)	0.8532 (6)	0.0553 (16)
C14	0.4883 (8)	0.7154 (7)	0.8040 (7)	0.0624 (17)
H14	0.511585	0.806901	0.808485	0.075*
C15	0.5435 (8)	0.6489 (7)	0.7474 (7)	0.0559 (16)
H15	0.605104	0.696358	0.715397	0.067*
C16	0.8261 (8)	0.4646 (7)	0.5740 (7)	0.0559 (16)
C17	0.9037 (8)	0.6083 (7)	0.5764 (8)	0.0649 (18)
H17A	0.896132	0.670508	0.634891	0.097*
H17B	1.013703	0.639024	0.610765	0.097*
H17C	0.851969	0.608948	0.484947	0.097*
C18	0.6856 (7)	0.1896 (6)	0.6341 (6)	0.0486 (14)
C19	0.7971 (7)	0.2025 (6)	0.7786 (6)	0.0445 (13)
C20	0.9383 (8)	0.3262 (7)	0.8729 (7)	0.0625 (17)
H20	0.965055	0.405140	0.848550	0.075*
C21	1.0391 (9)	0.3336 (9)	1.0016 (8)	0.076 (2)
H21	1.133157	0.417343	1.063705	0.091*
C22	1.0024 (10)	0.2195 (10)	1.0390 (8)	0.078 (2)
H22	1.071798	0.224624	1.125893	0.094*
C23	0.8643 (10)	0.0979 (9)	0.9491 (8)	0.079 (2)
H23	0.838071	0.020161	0.975159	0.095*
C24	0.7624 (8)	0.0890 (7)	0.8194 (7)	0.0614 (17)
H24	0.668569	0.004721	0.758500	0.074*
N1	0.6436 (5)	0.3003 (5)	0.6297 (5)	0.0465 (12)
N2	0.5582 (5)	0.4420 (5)	0.6781 (5)	0.0432 (11)
N3	0.6683 (5)	0.5144 (5)	0.6464 (5)	0.0400 (11)
O1	−0.0654 (6)	−0.1612 (5)	0.3314 (6)	0.0773 (15)
O2	0.0054 (6)	0.0257 (5)	0.2589 (5)	0.0739 (14)
O1A[a]	0.8895 (18)	0.3870 (11)	0.5745 (17)	0.054 (4)*
O1B[b]	0.8221 (15)	0.3648 (10)	0.5027 (15)	0.056 (4)*
Br1	0.32084 (11)	0.73907 (10)	0.92824 (9)	0.0899 (4)

[a]Occupancy: 0.46(2), [b]Occupancy: 0.54(2).

diverse biological activities.[7] Their adaptability in organic synthesis and pivotal role as intermediates in the synthesis of various compounds, spanning pharmaceuticals to agrochemicals,[8] have attracted medicinal chemists over the last decade. In our endeavour to synthesize enantio-pure derivatives of 1,2,4-triazolines using carbohydrate moiety as a chiral scaffold,[6] we synthesized the target triazoline via 1,3–DPCA from achiral precursors to investigate the stereo selectivity output of this reaction. The crystallographic analysis provided evidence supporting the proposed structure and confirmed the absolute configuration of the newly formed chiral centre (C2). The unit cell contained two molecules of the compound in a racemic mixture, each exhibiting a distinct absolute configuration at C2. The observed length of the C(1)=N(3) bond is 1.300(7) Å and confirms its double-bond character by falling within the typical range reported in the literature (1.27–1.32 Å) for triazolines and triazoles.[9–11] As expected, the adjacent single C–N bond C(2)–N(2) equals 1.467(7) Å, and is long due to its σ-bonding nature. Bromine is the heaviest atom in the molecule. Its covalent bond C(13)–Br(1) is the longest, 1.903(6) Å, and is comparable to related compounds.[9,11]

Interestingly, the oxygen atom, O1, of the acetyl unit splits into two positions as O1A and O1B. The C16=O bond length is in average 1.27 Å, confirming its bonding character.[12,13] The bromophenyl and 1,2,4-triazoline define the equatorial plane of the compound. In fact, the dioxybenzoyl unit is about 130.7° above this plane. The arm of the benzyl unit is hanging below the plane as the N1–C18–19 is twisted about 113.7°.

Author contribution: All the authors have accepted responsibility for the entire content of this submitted manuscript and approved submission.

Conflict of interest: The authors declare no conflicts of interest regarding this article.

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