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5-Methyl-*N'*-[5-methyl-1-(4-methylphenyl)-1*H*-1,2,3-triazole-4-carbonyl]-1-(4-methylphenyl)-1*H*-1,2,3-triazole-4-carbohydrazide, C₂₂H₂₂N₈O₂

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Abstract

C₂₂H₂₂N₈O₂, monoclinic, *P*₂/*c* (no. 14), *a* = 15.5175(5) Å, *b* = 7.9715(3) Å, *c* = 17.3941(5) Å, β = 90.005(3)°, *V* = 2151.61(12) Å³, *Z* = 4, *R*_{gt}(*F*) = 0.0592, *wR*_{ref}(*F*²) = 0.1780, *T* = 293(2) K.

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Tables 1 and 2 contain details on crystal structure and measurement conditions and a list of the atoms including atomic coordinates and displacement parameters.

Table 1: Data collection and handling.

Crystal:	Colourless needle
Size:	0.38 × 0.20 × 0.10 mm
Wavelength:	Mo Kα radiation (0.71073 Å)
μ:	0.09 mm ^{−1}
Diffractometer, scan mode:	SuperNova, Atlas, ω
θ _{max} , completeness:	29.9°, >99%
<i>N</i> (<i>hkl</i>) _{measured} , <i>N</i> (<i>hkl</i>) _{unique} , <i>R</i> _{int} :	19956, 5492, 0.027
Criterion for <i>I</i> _{obs} , <i>N</i> (<i>hkl</i>) _{gt} :	<i>I</i> _{obs} > 2 σ(<i>I</i> _{obs}), 3531
<i>N</i> (<i>param</i>) _{refined} :	293
Programs:	CrysAlis ^{PRO} [1], SHELX [2, 3], WinGX/ORTEP [4]

Source of material

The title compound was synthesized as previously reported [5] from reaction of ethyl 2-cyano-3-ethoxyacrylate and 5-methyl-1-(4-methylphenyl)-1*H*-1,2,3-triazole-4-carbohydrazide in boiling ethanol for 2 h. The solid obtained was collected by filtration, washed with ethanol, dried and recrystallized from dimethylformamide to give colourless crystals in 73% yield (Mp. 296–297 °C; lit. Mp. 296–297 °C [5]).

Experimental details

All H atoms were placed in calculated positions and refined using a riding model. For the methyl groups, C–H bonds were fixed at 0.96 Å and *U*_{iso}(H) set to 1.5*U*_{eq}(C) with free rotation around the C–C bond (HFIX 137 in SHELX [3]). For the rest of the hydrogens, *U*_{iso}(H) was set to 1.2*U*_{eq}(C, N) with aromatic C–H and N–H distances of 0.93 and 0.86 Å respectively.

Comment

Compounds containing the triazole moiety show various biological activities [6–9]. Therefore, various efficient

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

Atom	x	y	z	<i>U</i> _{iso} ^a / <i>U</i> _{eq}
C1	0.96790(12)	0.0586(2)	0.79433(10)	0.0437(4)
C2	0.89047(14)	−0.0239(3)	0.80137(12)	0.0547(5)
H2	0.866434	−0.079612	0.759562	0.066*
C3	0.84903(15)	−0.0227(3)	0.87167(13)	0.0602(6)
H3	0.796371	−0.077359	0.876566	0.072*
C4	0.88377(14)	0.0576(3)	0.93492(11)	0.0518(5)
C5	0.96172(14)	0.1376(3)	0.92605(11)	0.0529(5)
H5	0.986290	0.191577	0.968075	0.063*
C6	1.00460(14)	0.1398(3)	0.85619(11)	0.0505(5)
H6	1.057095	0.194996	0.851124	0.061*
C7	0.83736(18)	0.0535(4)	1.01108(13)	0.0710(7)
H7A	0.834672	−0.059948	1.029431	0.106*
H7B	0.779980	0.096337	1.004684	0.106*
H7C	0.867871	0.121508	1.047607	0.106*
C8	1.09266(12)	0.0225(2)	0.70254(10)	0.0442(4)
C9	1.09452(13)	0.0445(3)	0.62392(11)	0.0469(4)
C10	1.15761(14)	−0.0406(3)	0.75754(12)	0.0553(5)
H10A	1.177426	0.050192	0.789216	0.083*
H10B	1.205401	−0.087267	0.729791	0.083*
H10C	1.132189	−0.125651	0.789347	0.083*
C11	1.16846(14)	0.0198(3)	0.57203(11)	0.0514(5)
C12	1.23475(13)	0.0625(3)	0.38549(11)	0.0517(5)
C13	1.29432(13)	−0.0268(3)	0.33374(12)	0.0527(5)
C14	1.33624(13)	0.0293(3)	0.26919(12)	0.0520(5)
C15	1.34021(17)	0.1947(3)	0.23121(14)	0.0688(7)
H15A	1.396466	0.242328	0.238353	0.103*
H15B	1.297701	0.267686	0.253327	0.103*
H15C	1.329043	0.181677	0.177255	0.103*
C16	1.43024(14)	−0.1322(3)	0.17649(13)	0.0599(6)
C17	1.50621(15)	−0.0466(4)	0.17030(15)	0.0695(7)
H17	1.522426	0.029961	0.207951	0.083*
C18	1.55879(16)	−0.0753(4)	0.10730(16)	0.0755(7)
H18	1.610103	−0.015771	0.102806	0.091*
C19	1.53726(18)	−0.1886(4)	0.05159(15)	0.0756(7)
C20	1.4600(2)	−0.2720(4)	0.05912(17)	0.0926(10)
H20	1.443611	−0.348057	0.021283	0.111*
C21	1.40587(18)	−0.2460(4)	0.12147(17)	0.0831(8)
H21	1.354226	−0.304452	0.125872	0.100*
C22	1.5959(2)	−0.2185(5)	−0.01578(19)	0.1049(11)
H22A	1.609256	−0.113447	−0.039952	0.157*
H22B	1.567777	−0.290794	−0.052111	0.157*
H22C	1.648162	−0.270555	0.001692	0.157*
N1	1.01030(10)	0.0598(2)	0.72083(8)	0.0457(4)
N2	0.96332(11)	0.1015(2)	0.65742(9)	0.0553(5)
N3	1.01520(12)	0.0900(2)	0.59885(9)	0.0554(5)
N4	1.14820(12)	0.0166(3)	0.49665(10)	0.0616(5)
H4	1.097628	0.045055	0.481164	0.074*
N5	1.21055(12)	−0.0333(3)	0.44502(10)	0.0617(5)
H5A	1.234788	−0.129357	0.451240	0.074*
N6	1.31080(14)	−0.1927(3)	0.34385(12)	0.0720(6)
N7	1.36070(15)	−0.2444(3)	0.28864(14)	0.0797(7)
N8	1.37675(12)	−0.1092(3)	0.24288(11)	0.0604(5)
O1	1.24163(11)	0.0029(3)	0.59494(9)	0.0775(5)
O2	1.20913(12)	0.2028(2)	0.37390(9)	0.0722(5)

synthetic processes have been developed for the production of 1,2,3-triazoles [10–12]. Recently, the crystal structures for related compounds have been published [13, 14].

The asymmetric unit consists of one molecule of the title compound. The molecule consists of two tolyl rings [A (C1–C7) and D (C16–C22)] and two triazolyl rings [B (N1–N3, C8–C10) and C (N6–N8, C13–C15)]. The twist angles between pairs of rings are 50.6(1)°, 59.2(1)° and 61.0(1)° for A/B, B/C and C/D respectively. The torsion angle (C11–N4–N5–C12) for the formyl dihydrazide group is −124.5(2)°.

The molecules are aligned roughly parallel to [−101] in the crystal. Interactions of π - π type occur between identical tolyl groups (D) of neighbouring molecules with a centroid-to-centroid distance of 4.8 Å. Tolyl (A) and triazolyl (B) rings also interact with a centroid-to-centroid distance of 4.0 Å. Weak C–H···X interactions occur with O and N atoms acting as acceptors and C···O and C···N distances in the range 3.14 Å to 3.46 Å.

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