Crystal structure of 4-(3,4-dimethylphenyl)-3,5-bis(2-pyridyl)-1,2,4-triazole, $C_{20}H_{17}N_5$

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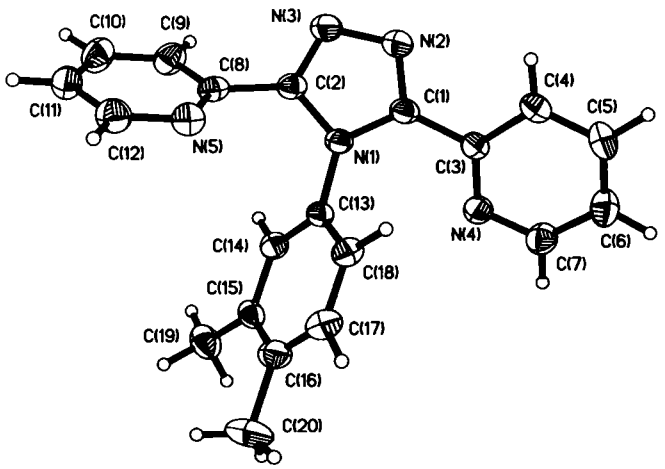

Abstract

$C_{20}H_{17}N_5$, triclinic, $P\bar{1}$ (no. 2), $a = 10.006(3)$ Å, $b = 10.318(3)$ Å, $c = 10.371(3)$ Å, $\alpha = 82.630(5)°$, $\beta = 62.925(5)°$, $\gamma = 63.291(5)°$, $V = 847.7$ Å^3, $Z = 2$, $R_{gt}(F) = 0.043$, $wR_{ref}(F^2) = 0.102$, $T = 298$ K.

Source of material

All reagents and solvents were used as purchased without further purification. C, H and N elemental analyses were performed on a Perkin-Elmer elemental analyzer.

The compound was prepared by the similar procedure as described in [1]. 3,4-Dimethylphenylamine and 2-pyridinecarboxaldehyde were used instead of 4-methoxyphenylamine and 4-pyridinecarboxaldehyde, respectively (yield 45 %). Elemental analysis: found – C, 73.00 %; H, 5.40 %; N, 21.25 %; calc. for $C_{20}H_{17}N_5$ – C, 73.37 %; H, 5.23 %; N, 21.39 %.

Experimental details

The low ratio of N_{gt}/N_{param} is caused by the poor quality of the crystals indicated by the appearance of micro-blebs.

Discussion

All bond lengths and bond angles are within normal ranges. All four rings are well-defined planes. The dihedral angles between the triazole ring and the pyridine planes are 36.5(2)° (with N4/C3/C4/C5/C6/C7 ring) and 50.1(3)° (with N5/C8/C9/C10/C11/C12 ring), respectively. The dihedral angle between the triazole ring and the substituted phenyl is 57.0(2)°. During refinement the torsions of the methyl groups were set as parameters, which lead to angles of 175.6° and 171.7° for ∠H19B–C19–C15–C14 and ∠H20A–C20–C16–C15, respectively. There is no obvious weak interaction, such as hydrogen bonds, in the molecular packing in the crystal.

Table 1. Data collection and handling.

Crystal:	colorless rod-like, size 0.20 × 0.26 × 0.32 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	0.80 cm^{-1}
Diffractometer, scan mode:	Bruker SMART CCD, φ/ω
$2\theta_{max}$:	50.06°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	4482, 2952
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 1528
$N(param)_{refined}$:	228
Programs:	SHELXTL [2], SHELXL-97 [3]

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

Atom	Site	x	y	z	U_{iso}
H(4)	2a	0.4745	0.0589	0.1393	0.068
H(5)	2a	0.4608	–0.0060	0.3663	0.080
H(6)	2a	0.2601	0.1599	0.5673	0.082
H(7)	2a	0.0767	0.3798	0.5392	0.077
H(9)	2a	0.1471	0.4888	–0.3005	0.074
H(10)	2a	0.1828	0.6400	–0.4861	0.088
H(11)	2a	0.3496	0.7533	–0.5207	0.082
H(12)	2a	0.4599	0.7226	–0.3630	0.079
H(14)	2a	0.0008	0.6814	0.0625	0.056
H(17)	2a	0.3307	0.7228	0.2352	0.073
H(18)	2a	0.4033	0.4988	0.1373	0.062
H(19A)	2a	–0.2109	0.9890	0.2646	0.111
H(19B)	2a	–0.1806	0.9269	0.1184	0.111
H(19C)	2a	–0.1048	1.0317	0.1159	0.111
H(20A)	2a	0.1580	0.9688	0.3109	0.153
H(20B)	2a	–0.0293	1.0255	0.3433	0.153
H(20C)	2a	0.0988	1.0470	0.1935	0.153

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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}
N(1)	2*a*	0.2574(2)	0.4342(2)	0.0183(2)	0.045(1)	0.039(1)	0.046(1)	−0.0124(9)	−0.0221(9)	−0.0021(9)
N(2)	2*a*	0.3250(2)	0.2050(2)	−0.0288(2)	0.076(1)	0.048(1)	0.061(1)	−0.020(1)	−0.035(1)	−0.001(1)
N(3)	2*a*	0.3243(2)	0.2764(2)	−0.1508(2)	0.069(1)	0.050(1)	0.059(1)	−0.018(1)	−0.033(1)	−0.004(1)
N(4)	2*a*	0.1624(2)	0.3565(2)	0.3288(2)	0.050(1)	0.051(1)	0.051(1)	−0.012(1)	−0.022(1)	−0.001(1)
N(5)	2*a*	0.3796(2)	0.5877(2)	−0.2440(2)	0.051(1)	0.062(1)	0.062(1)	−0.025(1)	−0.027(1)	0.005(1)
C(1)	2*a*	0.2859(3)	0.3003(2)	0.0704(2)	0.045(1)	0.040(1)	0.052(1)	−0.012(1)	−0.021(1)	−0.004(1)
C(2)	2*a*	0.2844(2)	0.4128(2)	−0.1205(2)	0.043(1)	0.047(1)	0.048(1)	−0.015(1)	−0.023(1)	−0.003(1)
C(3)	2*a*	0.2803(3)	0.2604(2)	0.2135(2)	0.047(1)	0.041(1)	0.052(1)	−0.018(1)	−0.023(1)	0.004(1)
C(4)	2*a*	0.3944(3)	0.1234(2)	0.2222(2)	0.053(2)	0.043(1)	0.064(2)	−0.014(1)	−0.025(1)	0.001(1)
C(5)	2*a*	0.3862(3)	0.0852(3)	0.3566(3)	0.062(2)	0.054(2)	0.080(2)	−0.021(1)	−0.038(2)	0.022(1)
C(6)	2*a*	0.2672(3)	0.1827(3)	0.4754(3)	0.064(2)	0.075(2)	0.061(2)	−0.029(2)	−0.030(1)	0.024(1)
C(7)	2*a*	0.1586(3)	0.3148(3)	0.4570(2)	0.057(2)	0.070(2)	0.051(2)	−0.022(1)	−0.020(1)	0.002(1)
C(8)	2*a*	0.2879(3)	0.5192(2)	−0.2285(2)	0.043(1)	0.049(1)	0.046(1)	−0.014(1)	−0.019(1)	−0.007(1)
C(9)	2*a*	0.2119(3)	0.5367(3)	−0.3158(2)	0.066(2)	0.077(2)	0.054(1)	−0.036(1)	−0.033(1)	0.008(1)
C(10)	2*a*	0.2333(3)	0.6261(3)	−0.4258(3)	0.078(2)	0.087(2)	0.064(2)	−0.033(2)	−0.042(2)	0.009(2)
C(11)	2*a*	0.3301(3)	0.6943(3)	−0.4453(3)	0.073(2)	0.060(2)	0.056(2)	−0.021(2)	−0.025(1)	0.009(1)
C(12)	2*a*	0.3973(3)	0.6733(2)	−0.3511(3)	0.066(2)	0.066(2)	0.066(2)	−0.034(1)	−0.025(1)	0.005(1)
C(13)	2*a*	0.2092(3)	0.5703(2)	0.0882(2)	0.043(1)	0.038(1)	0.039(1)	−0.015(1)	−0.015(1)	−0.001(1)
C(14)	2*a*	0.0678(3)	0.6905(2)	0.0966(2)	0.042(1)	0.047(1)	0.046(1)	−0.019(1)	−0.016(1)	0.003(1)
C(15)	2*a*	0.0233(3)	0.8268(2)	0.1562(2)	0.045(1)	0.037(1)	0.045(1)	−0.011(1)	−0.009(1)	0.004(1)
C(16)	2*a*	0.1267(3)	0.8369(3)	0.2060(2)	0.068(2)	0.056(2)	0.054(1)	−0.033(1)	−0.020(1)	−0.001(1)
C(17)	2*a*	0.2644(3)	0.7150(3)	0.1992(2)	0.058(2)	0.074(2)	0.054(2)	−0.032(1)	−0.022(1)	−0.004(1)
C(18)	2*a*	0.3088(3)	0.5805(2)	0.1410(2)	0.048(1)	0.057(2)	0.046(1)	−0.020(1)	−0.019(1)	−0.003(1)
C(19)	2*a*	−0.1322(3)	0.9551(2)	0.1645(3)	0.064(2)	0.054(2)	0.077(2)	−0.017(1)	−0.020(1)	0.009(1)
C(20)	2*a*	0.0847(4)	0.9830(3)	0.2693(3)	0.124(3)	0.072(2)	0.114(2)	−0.048(2)	−0.044(2)	−0.021(2)

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