Crystal structure of 4-{[1-(4-dimethylaminophenyl)methylidene]amino}-1,5-dimethyl-2-phenyl-1,2-dihydropyrazol-3-one, $C_{20}H_{22}N_4O$

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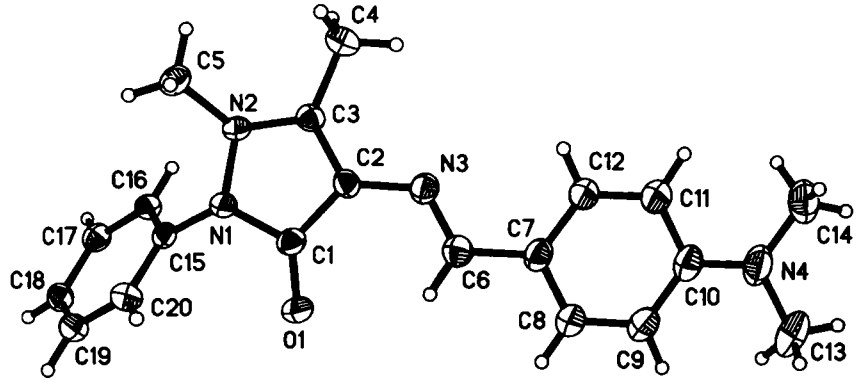

Abstract

$C_{20}H_{22}N_4O$, monoclinic, $C12/c1$ (no. 15),
$a = 17.875(4)$ Å, $b = 6.878(2)$ Å, $c = 29.747(7)$ Å,
$\beta = 101.010(4)°$, $V = 3589.7$ Å^3, $Z = 8$,
$R_{gt}(F) = 0.070$, $wR_{ref}(F^2) = 0.172$, $T = 298$ K.

Source of material

4-Dimethylaminobenzylidene (0.1 mmol, 14.9 mg) and 4-aminoantipyrine (0.1 mmol, 20.3 mg) were dissolved in methanol (10 ml). The mixture was stirred for 30 min at room temperature to give a clear yellow solution. After allowing the resulting solution to stand in air for 7 d, yellow block-shaped crystals were formed at the bottom of the vessel by slow evaporation of the solvent. The crystals were isolated by filtration, washed with methanol and dried in a vacuum desiccator using anhydrous $CaCl_2$ (yield 87 %). Elemental analysis: found – C, 71.6 %; H, 6.7 %; N, 16.9 %; calc. for $C_{20}H_{22}N_4O$ – C, 71.8 %; H, 6.6 %; N, 16.8 %.

Discussion

Antipyrine (2,3-dimethyl-1-phenylpyrazol-5-one) and its derivatives exhibit a wide range of biological activities and applications [1]. As an extension of our work [2,3], a new antipyrine derivative is reported here. The crystal structure of the title compound is built up by only the $C_{20}H_{22}N_4O$ molecules, within which all the bond lengths are in normal ranges and comparable to the corresponding values in other diarylaldimine structures [4]. The dihedral angle between the pyrazoline ring and the C15 – C20 phenyl ring is 55.1(3)°. O1 atom deviates from the pyrazoline best plane by 0.108(4) Å, whereas C4 and C5 deviate from it, on the other side, by 0.102(4) Å and 0.715(4) Å, respectively. Because of conjugation through the imino double bond C6=N3, the pyrazoline ring and the C7 – C12 phenyl ring are approximately coplanar (the deviation from the combined mean plane is 0.118(4) Å); the dihedral angle between the two rings is 17.9(3)°. As expected, the molecule adopts a *trans* configuration about the C6=N3 bond.

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Table 1. Data collection and handling.

Crystal:	yellow block, size 0.13 × 0.29 × 0.45 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	0.79 cm^{-1}
Diffractometer, scan mode:	Siemens SMART CCD, ω
$2\theta_{max}$:	50.06°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	7218, 3169
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 1911
$N(param)_{refined}$:	230
Program:	SHELXTL [4]

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

Atom	Site	x	y	z	U_{iso}
H(4A)	8*f*	0.4690	–0.3832	0.3424	0.094
H(4B)	8*f*	0.4189	–0.2901	0.2987	0.094
H(4C)	8*f*	0.3974	–0.2585	0.3468	0.094
H(5A)	8*f*	0.6260	–0.2741	0.3399	0.088
H(5B)	8*f*	0.6346	–0.1908	0.2921	0.088
H(5C)	8*f*	0.5699	–0.3408	0.2954	0.088
H(6)	8*f*	0.4391	0.3518	0.4044	0.067
H(8)	8*f*	0.3502	0.5743	0.4325	0.079
H(9)	8*f*	0.2366	0.6395	0.4550	0.087
H(11)	8*f*	0.1748	0.0800	0.4307	0.077
H(12)	8*f*	0.2882	0.0220	0.4094	0.071
H(13A)	8*f*	0.1404	0.6024	0.5077	0.148
H(13B)	8*f*	0.0557	0.5979	0.4813	0.148
H(13C)	8*f*	0.1188	0.6960	0.4589	0.148
H(14A)	8*f*	0.0606	0.1803	0.4296	0.137
H(14B)	8*f*	0.0190	0.3072	0.4610	0.137
H(14C)	8*f*	0.0846	0.1655	0.4830	0.137
H(16)	8*f*	0.5673	0.1284	0.2440	0.054
H(17)	8*f*	0.6385	0.3049	0.2010	0.058
H(18)	8*f*	0.7418	0.4856	0.2370	0.061
H(19)	8*f*	0.7751	0.4830	0.3154	0.065
H(20)	8*f*	0.7062	0.3027	0.3587	0.060

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}
O(1)	8*f*	0.5550(1)	0.3614(3)	0.37538(8)	0.071(2)	0.042(2)	0.073(2)	−0.010(1)	0.029(1)	−0.015(1)
N(1)	8*f*	0.5846(1)	0.0998(4)	0.33309(9)	0.049(2)	0.035(2)	0.054(2)	−0.006(1)	0.020(1)	−0.004(1)
N(2)	8*f*	0.5471(2)	−0.0727(3)	0.31590(9)	0.050(2)	0.029(2)	0.060(2)	−0.002(1)	0.018(1)	−0.003(1)
N(3)	8*f*	0.4141(2)	0.0933(4)	0.38221(9)	0.055(2)	0.048(2)	0.056(2)	0.002(1)	0.021(2)	0.001(2)
N(4)	8*f*	0.1223(2)	0.4057(6)	0.4574(1)	0.066(2)	0.105(3)	0.084(2)	0.008(2)	0.034(2)	−0.015(2)
C(1)	8*f*	0.5402(2)	0.1959(5)	0.3598(1)	0.051(2)	0.036(2)	0.048(2)	0.003(2)	0.012(2)	−0.000(2)
C(2)	8*f*	0.4781(2)	0.0654(5)	0.3625(1)	0.042(2)	0.043(2)	0.047(2)	−0.003(2)	0.014(2)	0.004(2)
C(3)	8*f*	0.4872(2)	−0.0940(4)	0.3375(1)	0.045(2)	0.034(2)	0.049(2)	0.000(2)	0.009(2)	0.008(2)
C(4)	8*f*	0.4388(2)	−0.2723(5)	0.3307(1)	0.064(2)	0.049(2)	0.077(3)	−0.014(2)	0.017(2)	−0.002(2)
C(5)	8*f*	0.5989(2)	−0.2336(5)	0.3104(1)	0.064(2)	0.040(2)	0.077(2)	0.010(2)	0.026(2)	0.000(2)
C(6)	8*f*	0.4020(2)	0.2552(5)	0.4010(1)	0.058(2)	0.059(2)	0.052(2)	−0.004(2)	0.016(2)	−0.002(2)
C(7)	8*f*	0.3309(2)	0.2907(6)	0.4173(1)	0.057(2)	0.065(2)	0.045(2)	0.003(2)	0.018(2)	−0.007(2)
C(8)	8*f*	0.3146(2)	0.4752(6)	0.4319(1)	0.069(3)	0.065(3)	0.071(3)	−0.004(2)	0.031(2)	−0.011(2)
C(9)	8*f*	0.2462(2)	0.5144(6)	0.4457(1)	0.084(3)	0.067(3)	0.074(3)	0.002(2)	0.033(2)	−0.013(2)
C(10)	8*f*	0.1913(2)	0.3684(6)	0.4457(1)	0.063(3)	0.081(3)	0.049(2)	0.007(2)	0.017(2)	−0.004(2)
C(11)	8*f*	0.2095(2)	0.1811(6)	0.4313(1)	0.065(3)	0.069(3)	0.062(2)	−0.003(2)	0.019(2)	−0.009(2)
C(12)	8*f*	0.2775(2)	0.1473(6)	0.4182(1)	0.054(2)	0.064(2)	0.063(2)	−0.004(2)	0.017(2)	−0.013(2)
C(13)	8*f*	0.1081(3)	0.5914(7)	0.4781(2)	0.097(4)	0.120(4)	0.087(3)	0.037(3)	0.038(3)	−0.013(3)
C(14)	8*f*	0.0669(2)	0.2517(7)	0.4578(1)	0.069(3)	0.123(4)	0.089(3)	−0.006(3)	0.034(3)	−0.006(3)
C(15)	8*f*	0.6292(2)	0.1997(4)	0.3056(1)	0.037(2)	0.033(2)	0.049(2)	0.005(1)	0.014(2)	0.000(2)
C(16)	8*f*	0.6092(2)	0.1996(4)	0.2585(1)	0.041(2)	0.037(2)	0.056(2)	0.001(2)	0.009(2)	−0.003(2)
C(17)	8*f*	0.6517(2)	0.3057(5)	0.2328(1)	0.054(2)	0.043(2)	0.048(2)	0.007(2)	0.012(2)	0.004(2)
C(18)	8*f*	0.7136(2)	0.4128(5)	0.2542(1)	0.051(2)	0.040(2)	0.066(2)	−0.003(2)	0.025(2)	0.004(2)
C(19)	8*f*	0.7333(2)	0.4111(5)	0.3009(1)	0.045(2)	0.047(2)	0.072(2)	−0.005(2)	0.012(2)	−0.004(2)
C(20)	8*f*	0.6919(2)	0.3042(5)	0.3270(1)	0.052(2)	0.047(2)	0.050(2)	−0.005(2)	0.005(2)	−0.002(2)

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