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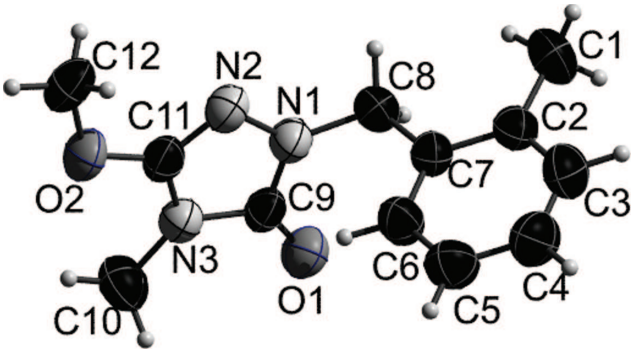
Crystal structure of 5-methoxy-4-methyl-2-(2-methylbenzyl)-2,4-dihydro-3H-1,2,4-triazol-3-one, $C_{12}H_{15}N_3O_2$



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Abstract

$C_{12}H_{15}N_3O_2$, monoclinic, $P2_1/c$, $a = 10.480(3)$ Å, $b = 14.608(4)$ Å, $c = 8.3398(17)$ Å, $\beta = 106.753(18)°$, $V = 1222.6(5)$ Å^3, $Z = 4$, $R_{gt}(F) = 0.0700$, $wR_{ref}(F^2) = 0.1896$, $T = 296(2)$ K.

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The crystal structure is shown in the Figure, Tables 1–3 contain details of the measurement method and a list of the atoms including atomic coordinates and displacement parameters.

Source of material

Reagents and solvents used were of commercially available quality. The title compound was synthesized according references [1]. To a stirred solution of 3-methoxy-4-methyl-1H-1,2,4-triazol-5(4H)-one (0.25 g, 1 mmol) and K_2CO_3 (0.14 g, 1.1 mmol) in DMF (5 mL), a mixture of 1-(chloromethyl)-2-fluorobenzene (1.1 mmol) was added dropwise. The resulting mixture was stirred at room temperature overnight. The mixture was poured into ice water. The precipitate formed was filtered off and recrystallized from petroleum ether/acetone to give the title compound with the yield of ~88%.

Table 1: Data collection and handling.

Crystal:	Colourless, block, size 0.1×0.2×0.3 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	0.89 cm^{-1}
Diffractometer, scan mode:	Bruker APEX-II area-detector diffractometer, φ and ω scans
$2\theta_{max}$:	50°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	8490, 210
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 988
$N(param)_{refined}$:	155
Programs:	SHELX [2, 3]

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

Atom	Site	x	y	z	U_{iso}
H(12A)	4e	0.8811	0.1462	0.3369	0.109
H(12B)	4e	0.8648	0.0548	0.2345	0.109
H(12C)	4e	0.7732	0.1388	0.1625	0.109
H(8A)	4e	0.7507	−0.0417	−0.3435	0.070
H(8B)	4e	0.8489	−0.0209	−0.4492	0.070
H(6A)	4e	0.7417	0.1858	−0.3813	0.076
H(10A)	4e	1.1547	0.2234	0.0821	0.115
H(10B)	4e	1.1470	0.2524	−0.1015	0.115
H(10C)	4e	1.2266	0.1645	−0.0230	0.115
H(5A)	4e	0.5905	0.2778	−0.5672	0.091
H(4A)	4e	0.4392	0.2126	−0.7981	0.094
H(1A)	4e	0.6749	−0.1018	−0.5915	0.117
H(1B)	4e	0.6230	−0.0815	−0.7840	0.117
H(1C)	4e	0.5215	−0.1016	−0.6823	0.117
H(3A)	4e	0.4438	0.0575	−0.8418	0.089

Experimental details

The H atoms bonded to C and N atoms were positioned geometrically and refined using a riding model [aliphatic C–H = 0.97(2) Å and N–H = 0.86 Å, $U_{iso}(H) = 1.2U_{eq}(C)$]. The

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Table 3: Atomic displacement parameters (Å^2).

Atom	Site	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
O(2)	4*e*	0.9576(3)	0.1525(2)	0.1445(3)	0.085(2)	0.081(2)	0.046(1)	−0.008(2)	0.026(1)	−0.007(1)
O(1)	4*e*	1.0479(3)	0.1083(2)	−0.3569(3)	0.075(2)	0.073(2)	0.056(2)	−0.001(2)	0.026(2)	−0.003(1)
N(3)	4*e*	1.0295(3)	0.1463(2)	−0.0912(3)	0.059(2)	0.060(2)	0.048(2)	−0.005(2)	0.016(2)	−0.000(1)
N(2)	4*e*	0.8463(3)	0.0677(2)	−0.0962(4)	0.068(2)	0.065(2)	0.047(2)	−0.000(2)	0.019(2)	0.004(2)
N(1)	4*e*	0.8820(3)	0.0548(2)	−0.2449(3)	0.058(2)	0.065(2)	0.044(2)	−0.007(2)	0.016(1)	−0.000(1)
C(11)	4*e*	0.9404(4)	0.1214(3)	−0.0109(4)	0.064(3)	0.056(2)	0.043(2)	0.005(2)	0.012(2)	0.003(2)
C(7)	4*e*	0.6898(4)	0.0665(2)	−0.5026(4)	0.057(2)	0.056(2)	0.046(2)	−0.005(2)	0.014(2)	−0.004(2)
C(9)	4*e*	0.9924(4)	0.1037(3)	−0.2470(4)	0.063(3)	0.053(2)	0.040(2)	0.004(2)	0.017(2)	0.002(2)
C(2)	4*e*	0.5995(4)	0.0273(3)	−0.6415(4)	0.059(3)	0.060(3)	0.054(2)	−0.011(2)	0.018(2)	−0.003(2)
C(12)	4*e*	0.8610(5)	0.1203(3)	0.2266(5)	0.091(3)	0.082(3)	0.051(2)	0.013(3)	0.028(2)	0.011(2)
C(8)	4*e*	0.7950(4)	0.0071(3)	−0.3855(4)	0.064(3)	0.053(2)	0.055(2)	−0.005(2)	0.014(2)	−0.004(2)
C(6)	4*e*	0.6830(4)	0.1597(3)	−0.4759(5)	0.069(3)	0.052(2)	0.064(2)	0.001(2)	0.007(2)	−0.004(2)
C(10)	4*e*	1.1497(4)	0.2013(3)	−0.0280(5)	0.065(3)	0.086(3)	0.076(3)	−0.016(3)	0.016(2)	−0.014(2)
C(5)	4*e*	0.5916(5)	0.2150(3)	−0.5858(5)	0.081(3)	0.059(3)	0.082(3)	0.001(2)	0.015(3)	0.006(2)
C(4)	4*e*	0.5018(5)	0.1762(3)	−0.7234(5)	0.071(3)	0.081(3)	0.072(3)	0.005(3)	0.002(2)	0.012(2)
C(1)	4*e*	0.6053(5)	−0.0736(3)	−0.6781(5)	0.084(3)	0.069(3)	0.076(3)	−0.013(2)	0.016(2)	−0.024(2)
C(3)	4*e*	0.5055(4)	0.0834(3)	−0.7497(5)	0.064(3)	0.084(3)	0.065(2)	−0.010(2)	0.004(2)	−0.004(2)

H atoms bonded to O atoms were located in a difference Fourier maps and refined with O–H distance restraints of 0.85(2) and $U_{iso}(H) = 1.5U_{eq}(O)$.

Discussion

In recent years, heterocycles have received significant attention due to their biological importance [4–6], especially 1,2,4-triazole ring. The 1,2,4-triazole derivatives are often exhibited diversity biological activities including herbicidal activity [6, 7], antifungal activity [8] and so on. In view of these facts and in continuation of our interest in the agriculture, we attempted to synthesize a series of 1,2,4-oxadiazole derivatives. Generally, the average bond lengths and bond angles of phenyl ring and triazole ring [9, 10] are normal. The C(8)–N(1), C(11)–N(2), C(9)–N(1) and C(11)–N(3) bonds are significantly shorter than a normal single C–N bond which indicates significant electron delocalization in the triazole system. The bond angle of N(1)–C(8)–C(7) is 114.8(3)°.

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