Crystal structure of 1,3,5-tris(4-methoxyphenyl)-1*H*-1,2,4-triazole-2-oxide, $C_{23}H_{21}N_3O_4$

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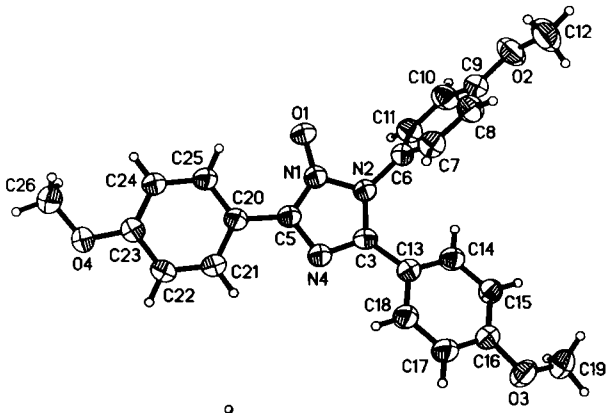

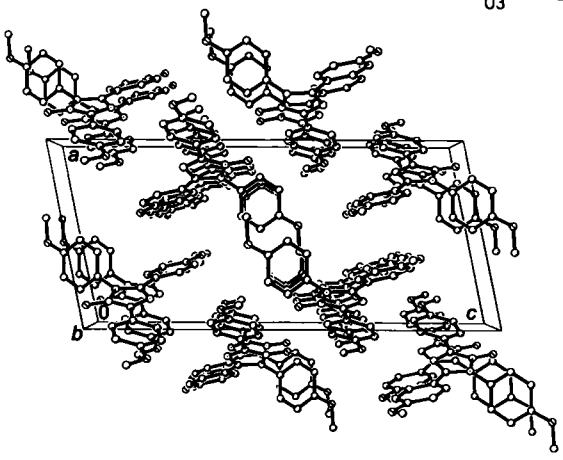

Abstract

$C_{23}H_{21}N_3O_4$, monoclinic, $P12_1/c1$ (no. 14), $a = 10.7855(7)$ Å, $b = 8.3439(6)$ Å, $c = 22.960(2)$ Å, $\beta = 101.620(6)°$, $V = 2023.9$ Å^3, $Z = 4$, $R_{gt}(F) = 0.056$, $wR_{ref}(F^2) = 0.185$, $T = 293$ K.

Source of material

The title compound [1-4] was obtained as a by-product of an attempted 1,3-dipolar cycloaddition, when the nitrile oxide precursor, 4-methoxybenz-hydroximoyl chloride [5], and a rather unreactive dipolarophile [5-8] (a bi-cyclic isoxazolo-cyclopentenone) were heated under reflux in toluene for 16 h. Purification by column chromatography and crystallization from ethyl acetate and petroleum ether furnished the title compound in the form of colorless crystals (m.p. 483-484 K) [9].

Discussion

The anisyl moieties are showing a step-by-step deviation from the coplanar orientation in relation to the triazole core (figure, top) The anisyl group attached at the 1-position shows the strongest out-of-plane orientation, indicated by the torsion angle C3–N2–C6–C7 of –56.5°. This behavior is forced by the steric influence of the neighboring oxygen atom at the 2-position and another anisyl moiety at the 5-position. The anisyl group at the 3-position is only slightly influenced by the oxide at C2. Consequently, we found a nearly coplanar orientation indicated by the torsion angle N4–C5–C20–C21 of –6.4°. The torsion angle N4–C3–C13–C18 of –20.6° of the anisyl moiety at C5 characterizes a medium out-of-plane orientation. In the packing we observe hydrophobic layers in the *a,b* plane stacking along the *c* axis. The cell plot also shows hydrophobic channels along the *b* axis built up by a "face-to-face" and a "side-by-side" orientation of the anisyl groups (figure, bottom).

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Table 1. Data collection and handling.

Crystal:	colorless plate, size 0.05 × 0.20 × 0.40 mm
Wavelength:	Cu K_α radiation (1.54178 Å)
μ:	7.54 cm^{-1}
Diffractometer, scan mode:	Siemens P4, ω
$2\theta_{max}$:	129.96°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	3537, 3350
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 2200
$N(param)_{refined}$:	272
Programs:	SHELXS-97 [10], SHELXL-97 [11]

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

Atom	Site	x	y	z	U_{iso}
H(7)	4*e*	0.4507	–0.0566	0.0990	0.059
H(8)	4*e*	0.5564	–0.1946	0.0362	0.063
H(10)	4*e*	0.2214	–0.3096	–0.0673	0.065
H(11)	4*e*	0.1173	–0.1783	–0.0030	0.057
H(12A)	4*e*	0.6101	–0.4178	–0.0843	0.127
H(12B)	4*e*	0.6201	–0.4041	–0.0154	0.127
H(12C)	4*e*	0.6182	–0.2487	–0.0540	0.127
H(14)	4*e*	0.2637	–0.3454	0.1289	0.056
H(15)	4*e*	0.3312	–0.5570	0.1921	0.054
H(17)	4*e*	0.3589	–0.2614	0.3319	0.068
H(18)	4*e*	0.2916	–0.0515	0.2688	0.063
H(19A)	4*e*	0.4096	–0.7803	0.3232	0.091
H(19B)	4*e*	0.4347	–0.7239	0.2614	0.091
H(19C)	4*e*	0.2956	–0.7366	0.2717	0.091
H(21)	4*e*	0.0383	0.3015	0.2165	0.060
H(22)	4*e*	–0.0569	0.5402	0.2289	0.064
H(24)	4*e*	–0.0435	0.6650	0.0613	0.055
H(25)	4*e*	0.0557	0.4256	0.0495	0.054
H(26A)	4*e*	–0.1768	0.9788	0.1300	0.099
H(26B)	4*e*	–0.1969	0.8460	0.0807	0.099
H(26C)	4*e*	–0.0620	0.9195	0.1034	0.099

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)	4*e*	0.1507(2)	0.1224(3)	0.07843(9)	0.047(1)	0.044(1)	0.038(1)	0.004(1)	0.0067(9)	0.0011(9)
O(1)	4*e*	0.1313(2)	0.1709(3)	0.02438(8)	0.075(1)	0.060(1)	0.034(1)	0.015(1)	0.0116(9)	0.0090(9)
N(2)	4*e*	0.2113(2)	–0.0250(3)	0.09489(9)	0.052(1)	0.046(1)	0.035(1)	0.010(1)	0.0076(9)	0.0025(9)
O(2)	4*e*	0.4514(2)	–0.3509(3)	–0.0618(1)	0.072(2)	0.086(2)	0.063(1)	0.015(1)	0.030(1)	–0.011(1)
O(3)	4*e*	0.3874(2)	–0.5482(3)	0.30983(9)	0.079(2)	0.045(1)	0.050(1)	0.006(1)	0.000(1)	0.0079(9)
C(3)	4*e*	0.2167(2)	–0.0399(3)	0.1547(1)	0.047(1)	0.046(2)	0.033(1)	0.003(1)	0.008(1)	0.001(1)
N(4)	4*e*	0.1628(2)	0.0850(3)	0.17485(9)	0.055(1)	0.044(1)	0.039(1)	0.006(1)	0.009(1)	0.001(1)
O(4)	4*e*	–0.1130(2)	0.7675(3)	0.1594(1)	0.102(2)	0.054(1)	0.059(1)	0.027(1)	0.034(1)	0.008(1)
C(5)	4*e*	0.1231(2)	0.1853(3)	0.1278(1)	0.046(1)	0.042(2)	0.035(1)	0.002(1)	0.006(1)	0.001(1)
C(6)	4*e*	0.2745(2)	–0.1070(3)	0.0547(1)	0.045(1)	0.042(2)	0.038(1)	0.004(1)	0.009(1)	0.001(1)
C(7)	4*e*	0.4050(3)	–0.1097(4)	0.0659(1)	0.048(2)	0.050(2)	0.047(2)	–0.004(1)	0.003(1)	–0.003(1)
C(8)	4*e*	0.4684(3)	–0.1911(4)	0.0282(1)	0.042(2)	0.057(2)	0.058(2)	–0.001(1)	0.012(1)	–0.002(1)
C(9)	4*e*	0.4000(3)	–0.2670(4)	–0.0215(1)	0.058(2)	0.050(2)	0.046(2)	0.006(1)	0.020(1)	0.002(1)
C(10)	4*e*	0.2674(3)	–0.2604(4)	–0.0334(1)	0.056(2)	0.064(2)	0.042(2)	0.000(1)	0.008(1)	–0.011(1)
C(11)	4*e*	0.2053(3)	–0.1816(4)	0.0047(1)	0.044(2)	0.056(2)	0.043(1)	–0.002(1)	0.006(1)	–0.004(1)
C(12)	4*e*	0.5859(4)	–0.3558(5)	–0.0532(2)	0.075(2)	0.080(3)	0.113(3)	0.013(2)	0.053(2)	0.000(2)
C(13)	4*e*	0.2683(3)	–0.1755(3)	0.1917(1)	0.046(1)	0.043(2)	0.040(1)	0.002(1)	0.008(1)	0.002(1)
C(14)	4*e*	0.2820(3)	–0.3283(3)	0.1697(1)	0.049(2)	0.049(2)	0.040(1)	0.000(1)	0.006(1)	–0.001(1)
C(15)	4*e*	0.3225(3)	–0.4555(3)	0.2076(1)	0.050(2)	0.040(2)	0.044(1)	–0.000(1)	0.006(1)	–0.003(1)
C(16)	4*e*	0.3499(3)	–0.4312(4)	0.2686(1)	0.049(2)	0.046(2)	0.043(1)	0.003(1)	0.007(1)	0.004(1)
C(17)	4*e*	0.3390(3)	–0.2788(4)	0.2911(1)	0.078(2)	0.052(2)	0.037(1)	0.009(2)	0.008(1)	–0.000(1)
C(18)	4*e*	0.2987(3)	–0.1532(4)	0.2532(1)	0.072(2)	0.045(2)	0.038(1)	0.007(1)	0.007(1)	–0.002(1)
C(19)	4*e*	0.3814(3)	–0.7105(4)	0.2899(2)	0.066(2)	0.045(2)	0.067(2)	0.005(2)	0.007(2)	0.008(2)
C(20)	4*e*	0.0578(2)	0.3364(3)	0.1316(1)	0.046(1)	0.043(2)	0.037(1)	0.000(1)	0.008(1)	–0.002(1)
C(21)	4*e*	0.0227(3)	0.3746(4)	0.1853(1)	0.063(2)	0.048(2)	0.042(1)	0.005(1)	0.016(1)	0.008(1)
C(22)	4*e*	–0.0341(3)	0.5172(4)	0.1929(1)	0.069(2)	0.053(2)	0.045(2)	0.006(2)	0.025(1)	–0.000(1)
C(23)	4*e*	–0.0581(3)	0.6283(4)	0.1467(1)	0.054(2)	0.046(2)	0.047(2)	0.006(1)	0.015(1)	0.000(1)
C(24)	4*e*	–0.0259(3)	0.5925(4)	0.0926(1)	0.055(2)	0.045(2)	0.038(1)	0.004(1)	0.010(1)	0.003(1)
C(25)	4*e*	0.0327(3)	0.4483(3)	0.0855(1)	0.053(2)	0.046(2)	0.036(1)	0.001(1)	0.008(1)	0.001(1)
C(26)	4*e*	–0.1393(4)	0.8877(4)	0.1148(2)	0.080(2)	0.053(2)	0.067(2)	0.018(2)	0.024(2)	0.010(2)

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