Crystal structure of (2*Z*)-2-amino-3-[(*E*)-4-(diethylamino)-2-methoxybenzylideneamino]-2-butenedinitrile, $C_{16}H_{19}N_5O$

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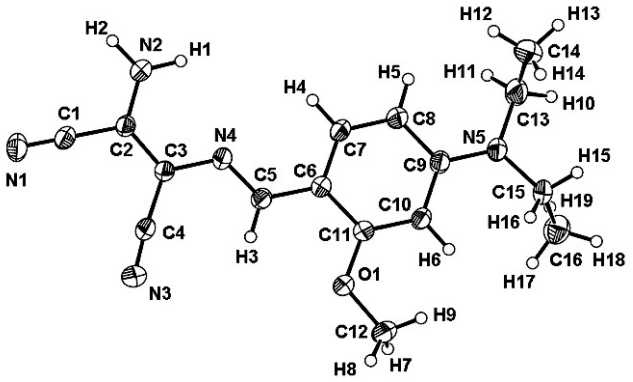

Abstract

$C_{16}H_{19}N_5O$, monoclinic, $P12_1/n1$ (no. 14), a = 10.9025(2) Å, b = 12.9499(2) Å, c = 11.8235(2) Å, β = 106.6478(7)°, V = 1599.4 Å^3, Z = 4, $R_{gt}(F)$ = 0.052, $wR_{ref}(F)$ = 0.080, T = 296 K.

Source of material

(*Z*)-2-Amino-3-[(*E*)-4-(diethylamino)-2-methoxybenzylideneamino]-2-butenedinitrile was synthesized according to the previously described method [1]. Reddish block-shaped crystals of this compound were grown by solvent diffusion method. Single crystals of the title compound were grown by using dichloromethane as good solvent and *n*-hexane as poor solvent.

Experimental details

The positions of all hydrogen atoms were calculated geometrically and not refined.

Discussion

In the title crystal structure, the two terminal ethyl substituents are projected in the inverted direction in the amino moiety. The torsion angles between the terminal ethyl substituents are estimated to be C9–N5–C13–C14 = 86.6(3)° and C9–N5–C15–C16 = 91.6(3)°, respectively. The methoxy substituent is in parallel with the almost planar molecular π-plane with a mean deviation of the component atoms of the chromophoric system in the range of 0.005 Å from 0.072 Å. The methoxy substituent makes the torsion angles of C12–O1–C11–C6 = –178.0(2)° and C12–O1–C11–C10 = 2.4(3)°, respectively. The intramolecular bond distances are in the normal range except for the diaminobutenedinitrile moiety. Small bond alternation within this moiety is shown by d(C1—C2) = 1.439(3) Å and d(C3—C4) = 1.437(3) Å, which clearly indicates that an effective π-conjugation is developed between the electro-donating amino group and the electro-accepting dicyanoethylene group. The crystal is formed by two different intermolecular H-bonds (N2–H1···N1^{i} and N2–H2···N3^{ii}, i: $-x+\frac{1}{2}$, $y+\frac{1}{2}$,$-z+\frac{3}{2}$; ii: $x-\frac{1}{2}$,$-y+\frac{1}{2}$,$z-\frac{1}{2}$) with d(D···A) = 3.335(3) Å and 3.031(2) Å, respectively. Hydrogen of the amino group form hydrogen bonds with nitrogen atoms of cyano groups of neighboring molecules. The crystal does not show a packed layer-by-layer arrangement of the molecules.

Table 1. Data collection and handling.

Crystal:	red block, size 0.15 × 0.15 × 0.20 mm
Wavelength:	Cu K_α radiation (1.54187 Å)
μ:	6.56 cm^{-1}
Diffractometer, scan mode:	Rigaku RAXIS-RAPID, ω
$2\theta_{max}$:	136.42°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	14631, 2876
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 1741
$N(param)_{refined}$:	218
Program:	SIR92 [2]

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

Atom	Site	x	y	z	U_{iso}
H(1)	4*e*	0.3232	0.4494	0.7257	0.059
H(2)	4*e*	0.1966	0.3770	0.6683	0.059
H(3)	4*e*	0.6550	0.3724	1.0218	0.048
H(4)	4*e*	0.5759	0.5864	0.8244	0.055
H(5)	4*e*	0.6920	0.7350	0.8249	0.059
H(6)	4*e*	0.9726	0.6243	1.1024	0.050
H(7)	4*e*	1.0491	0.4316	1.1780	0.069
H(8)	4*e*	0.9808	0.4185	1.2756	0.069
H(9)	4*e*	0.9994	0.5279	1.2295	0.069
H(10)	4*e*	0.9471	0.9029	0.8735	0.085
H(11)	4*e*	0.8335	0.8344	0.8047	0.085
H(12)	4*e*	0.6959	0.9186	0.8707	0.114
H(13)	4*e*	0.7910	1.0082	0.8729	0.114
H(14)	4*e*	0.7976	0.9476	0.9877	0.114
H(15)	4*e*	1.0305	0.8833	1.0780	0.060
H(16)	4*e*	1.0155	0.7781	1.1354	0.060
H(17)	4*e*	1.1671	0.7046	1.0772	0.084
H(18)	4*e*	1.2188	0.8169	1.0887	0.083
H(19)	4*e*	1.1413	0.7745	0.9664	0.084

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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}
O(1)	4*e*	0.8636(2)	0.4478(1)	1.1197(2)	0.0424(9)	0.048(1)	0.058(1)	–0.0063(8)	–0.0042(8)	0.0089(9)
N(1)	4*e*	0.1991(2)	0.1428(2)	0.7897(2)	0.055(1)	0.052(1)	0.075(2)	–0.015(1)	0.016(1)	–0.012(1)
N(2)	4*e*	0.2778(2)	0.3865(2)	0.7243(2)	0.044(1)	0.051(1)	0.053(1)	–0.0048(9)	–0.0020(9)	0.002(1)
N(3)	4*e*	0.5376(2)	0.1757(2)	1.0343(2)	0.055(1)	0.060(2)	0.084(2)	–0.001(1)	–0.009(1)	0.017(1)
N(4)	4*e*	0.5170(2)	0.4085(2)	0.8831(2)	0.037(1)	0.041(1)	0.047(1)	–0.0054(8)	0.0063(9)	–0.0037(9)
N(5)	4*e*	0.9135(2)	0.7865(2)	0.9684(2)	0.051(1)	0.047(1)	0.051(1)	–0.014(1)	0.002(1)	0.005(1)
C(1)	4*e*	0.2561(2)	0.2175(2)	0.7954(2)	0.037(1)	0.049(2)	0.047(2)	–0.002(1)	0.007(1)	–0.010(1)
C(2)	4*e*	0.3289(2)	0.3111(2)	0.8015(2)	0.037(1)	0.038(1)	0.043(1)	–0.002(1)	0.008(1)	–0.005(1)
C(3)	4*e*	0.4461(2)	0.3192(2)	0.8833(2)	0.037(1)	0.036(1)	0.044(1)	0.0003(9)	0.007(1)	–0.002(1)
C(4)	4*e*	0.4931(2)	0.2371(2)	0.9661(2)	0.036(1)	0.046(1)	0.059(2)	–0.005(1)	0.002(1)	–0.003(1)
C(5)	4*e*	0.6251(2)	0.4229(2)	0.9621(2)	0.037(1)	0.042(1)	0.043(1)	0.000(1)	0.008(1)	–0.002(1)
C(6)	4*e*	0.7007(2)	0.5141(2)	0.9615(2)	0.038(1)	0.041(1)	0.038(1)	–0.002(1)	0.007(1)	–0.002(1)
C(7)	4*e*	0.6566(2)	0.5941(2)	0.8818(2)	0.041(1)	0.052(1)	0.043(1)	–0.009(1)	0.000(1)	–0.001(1)
C(8)	4*e*	0.7250(2)	0.6827(2)	0.8819(2)	0.051(1)	0.049(2)	0.046(1)	–0.008(1)	–0.001(1)	0.008(1)
C(9)	4*e*	0.8453(2)	0.6972(2)	0.9661(2)	0.042(1)	0.045(1)	0.042(1)	–0.008(1)	0.009(1)	–0.001(1)
C(10)	4*e*	0.8915(2)	0.6172(2)	1.0455(2)	0.036(1)	0.047(1)	0.043(1)	–0.006(1)	0.005(1)	–0.001(1)
C(11)	4*e*	0.8218(2)	0.5279(2)	1.0441(2)	0.038(1)	0.040(1)	0.039(1)	0.000(1)	0.008(1)	0.001(1)
C(12)	4*e*	0.9831(2)	0.4574(2)	1.2081(2)	0.044(1)	0.067(2)	0.060(2)	–0.002(1)	–0.002(1)	0.012(2)
C(13)	4*e*	0.8732(3)	0.8663(2)	0.8784(2)	0.072(2)	0.071(2)	0.069(2)	–0.030(2)	0.001(2)	0.021(2)
C(14)	4*e*	0.7810(3)	0.9423(2)	0.9045(3)	0.072(2)	0.072(2)	0.140(3)	–0.004(2)	–0.008(2)	0.041(2)
C(15)	4*e*	1.0256(2)	0.8106(2)	1.0666(2)	0.044(1)	0.045(1)	0.060(2)	–0.011(1)	0.009(1)	–0.003(1)
C(16)	4*e*	1.1501(2)	0.7732(2)	1.0486(2)	0.055(2)	0.076(2)	0.076(2)	–0.005(2)	0.023(2)	–0.005(2)

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