Crystal structure of (*Z*)-2-amino-3-[(*E*)-4-(dimethylamino)-2-ethoxybenzylideneamino]-2-butenedinitrile, $C_{15}H_{17}N_5O$

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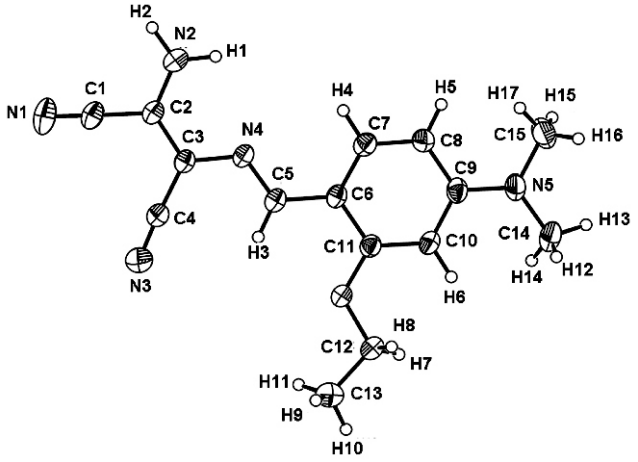

Abstract

$C_{15}H_{17}N_5O$, monoclinic, $P12_1/n1$ (no. 14), a = 11.4595(6) Å, b = 10.4624(5) Å, c = 14.1316(6) Å, β = 114.325(2)°, V = 1543.9 Å^3, Z = 4, $R_{gt}(F)$ = 0.052, $wR_{ref}(F)$ = 0.089, T = 296 K.

Source of material

(*Z*)-2-amino-3-[(*E*)-4-(dimethylamino)-2-ethoxybenzylideneamino]-2-butenedinitrile was synthesized through the reaction of diaminomaleonitrile and 4-(dimethylamino)-2-ethoxybenzaldehyde to form an azomethine bond [1]. Orange block-shaped crystals were grown by solvent diffusion method. Chloroform and *n*-hexane were used as a good and a poor solvent, respectively.

Experimental details

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

Discussion

In the title crystal structure, the ethoxy substituent is slightly deviating out of the mean plane of π-conjugated system making a torsion angle of 173.9(2)°. The torsion angles C14–N5–C9–C10 and C15–N5–C9–C8 are estimated to be 11.3(4)° and 2.8(4)°, respectively. The interatomic distances are in the normal range except for dicyanoethylene groups. Small bond alternation in electron-accepting dicyanoethylene groups are shown by d(C1—C2) = 1.437(4) Å and d(C3—C4) = 1.417(3) Å. The effective π-conjugation system for the title molecule is indicated between the terminal amino groups and the dicyanoethylene groups.Two different intermolecular H-bonds are formed (N2–H1 N1^{i} and N2–H2 N3^{ii}, i: $-x+\frac{5}{2},y+\frac{1}{2},-z+\frac{3}{2}$; ii: $x+\frac{1}{2},-y-\frac{1}{2},z+\frac{1}{2}$) with d(D A) = 3.117(3) Å and 2.983(3) Å, respectively. Both hydrogens of the amino group form H-bonds with nitrogen atoms of cyano groups of neighboring molecules. The molecules were stacked layer-by-layer with the shortest distance between two molecules of 3.403(3) Å in two-dimensional stacking layers. The molecular packing is characterized by a herring-bone arrangement with a gradient angle of 99.12° between the neighboring two-dimensional layers.

Table 1. Data collection and handling.

Crystal:	orange block, size 0.10 0.10 0.15 mm
Wavelength:	Cu K_α radiation (1.54187 Å)
μ:	6.56 cm ^{1}
Diffractometer, scan mode:	Rigaku RAXIS-RAPID, ω
$2\theta_{max}$:	136.48°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	14089, 2816
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 1520
$N(param)_{refined}$:	207
Program:	SIR92 [2]

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

Atom	Site	x	y	z	U_{iso}
H(1)	4*e*	1.1514	0.0369	0.7428	0.078
H(2)	4*e*	1.2830	0.1233	0.7836	0.079
H(3)	4*e*	0.8049	0.0911	0.4597	0.057
H(4)	4*e*	0.8867	0.1272	0.6731	0.063
H(5)	4*e*	0.7580	0.2818	0.6974	0.066
H(6)	4*e*	0.4655	0.1713	0.4285	0.060
H(7)	4*e*	0.4030	0.0089	0.3122	0.068
H(8)	4*e*	0.4715	0.1066	0.2698	0.068
H(9)	4*e*	0.4996	0.0318	0.1659	0.093
H(10)	4*e*	0.3745	0.0876	0.1664	0.093
H(11)	4*e*	0.5060	0.1452	0.2386	0.093
H(12)	4*e*	0.3669	0.3876	0.4551	0.098
H(13)	4*e*	0.3394	0.3717	0.5531	0.098
H(14)	4*e*	0.3542	0.2520	0.4942	0.097
H(15)	4*e*	0.5554	0.3861	0.7225	0.105
H(16)	4*e*	0.5237	0.5012	0.6468	0.105
H(17)	4*e*	0.6569	0.4360	0.6853	0.105

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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*	0.5924(2)	0.0049(2)	0.3789(1)	0.044(1)	0.064(1)	0.054(1)	0.0124(8)	0.0106(8)	0.0087(9)
N(1)	4*e*	1.2866(2)	0.3640(2)	0.6340(2)	0.080(2)	0.071(2)	0.120(2)	0.032(2)	0.053(2)	0.025(2)
N(2)	4*e*	1.1989(2)	0.1062(2)	0.7331(2)	0.049(1)	0.084(2)	0.063(2)	0.014(1)	0.006(1)	0.009(1)
N(3)	4*e*	0.9329(2)	0.2982(2)	0.4095(2)	0.063(2)	0.091(2)	0.087(2)	0.003(2)	0.013(2)	0.027(2)
N(4)	4*e*	0.9524(2)	0.0645(2)	0.5898(2)	0.038(1)	0.052(1)	0.048(1)	0.0063(9)	0.0171(9)	0.006(1)
N(5)	4*e*	0.5199(2)	0.3359(2)	0.5783(2)	0.053(1)	0.067(2)	0.083(2)	0.009(1)	0.029(1)	0.017(1)
C(1)	4*e*	1.2242(2)	0.2819(2)	0.6384(2)	0.049(2)	0.055(2)	0.068(2)	0.010(1)	0.026(1)	0.017(1)
C(2)	4*e*	1.1463(2)	0.1797(2)	0.6483(2)	0.041(1)	0.047(1)	0.050(2)	0.005(1)	0.017(1)	0.007(1)
C(3)	4*e*	1.0256(2)	0.1609(2)	0.5735(2)	0.039(1)	0.046(1)	0.049(1)	0.003(1)	0.020(1)	0.006(1)
C(4)	4*e*	0.9776(2)	0.2392(2)	0.4837(2)	0.044(2)	0.057(2)	0.057(2)	0.004(1)	0.015(1)	0.003(1)
C(5)	4*e*	0.8376(2)	0.0424(2)	0.5219(2)	0.040(1)	0.049(1)	0.052(2)	0.004(1)	0.020(1)	0.007(1)
C(6)	4*e*	0.7578(2)	0.0547(2)	0.5373(2)	0.039(1)	0.047(1)	0.050(1)	0.003(1)	0.021(1)	0.005(1)
C(7)	4*e*	0.8009(2)	0.1360(2)	0.6229(2)	0.044(1)	0.059(2)	0.053(2)	0.002(1)	0.019(1)	0.004(1)
C(8)	4*e*	0.7251(2)	0.2282(2)	0.6379(2)	0.047(2)	0.062(2)	0.057(2)	0.000(1)	0.021(1)	0.012(1)
C(9)	4*e*	0.5971(2)	0.2444(2)	0.5643(2)	0.045(2)	0.053(2)	0.064(2)	0.001(1)	0.030(1)	0.003(1)
C(10)	4*e*	0.5516(2)	0.1638(2)	0.4782(2)	0.037(1)	0.056(2)	0.055(2)	0.007(1)	0.018(1)	0.001(1)
C(11)	4*e*	0.6307(2)	0.0727(2)	0.4634(2)	0.041(1)	0.051(1)	0.049(2)	0.002(1)	0.020(1)	0.001(1)
C(12)	4*e*	0.4723(2)	0.0210(2)	0.2927(2)	0.043(2)	0.069(2)	0.057(2)	0.008(1)	0.012(1)	0.003(1)
C(13)	4*e*	0.4619(2)	0.0687(3)	0.2080(2)	0.068(2)	0.098(2)	0.066(2)	0.001(2)	0.017(2)	0.018(2)
C(14)	4*e*	0.3833(2)	0.3367(3)	0.5149(2)	0.056(2)	0.085(2)	0.101(2)	0.021(2)	0.029(2)	0.007(2)
C(15)	4*e*	0.5681(3)	0.4221(2)	0.6657(2)	0.086(2)	0.076(2)	0.100(2)	0.003(2)	0.049(2)	0.026(2)

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