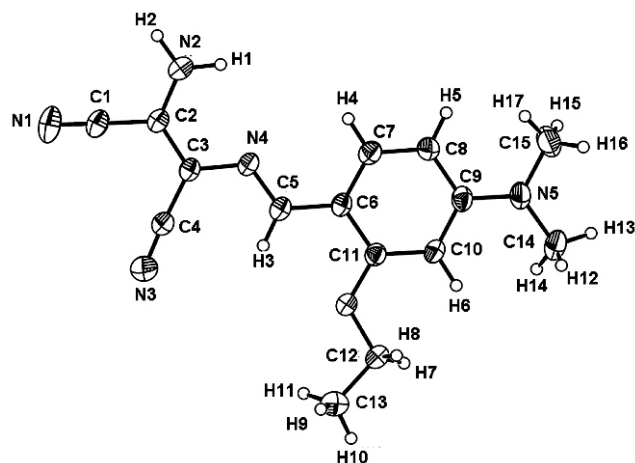


Crystal structure of (Z)-2-amino-3-[(E)-4-(dimethylamino)-2-ethoxybenzylideneamino]-2-butenedinitrile, C₁₅H₁₇N₅O

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Received February 25, 2011, accepted and available on-line July 1, 2011; CCDC no. 1267/3417



Abstract

C₁₅H₁₇N₅O, monoclinic, *P*2₁/*n*1 (no. 14), *a* = 11.4595(6) Å, *b* = 10.4624(5) Å, *c* = 14.1316(6) Å, β = 114.325(2)°, *V* = 1543.9 Å³, *Z* = 4, *R*_g(*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...N1ⁱ and N2–H2...N3ⁱⁱ, i: $-x+\frac{1}{2}, y+\frac{1}{2}, -z+\frac{1}{2}$; 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 hydro-

gens 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}
Diffraction, scan mode:	Rigaku RAXIS-RAPID, ω
$2\theta_{\text{max}}$:	136.48°
<i>N</i> (<i>hkl</i>) _{measured} , <i>N</i> (<i>hkl</i>) _{unique} :	14089, 2816
Criterion for <i>I</i> _{obs} , <i>N</i> (<i>hkl</i>) _{gt} :	<i>I</i> _{obs} > 2 σ (<i>I</i> _{obs}), 1520
<i>N</i> (<i>param</i>) _{refined} :	207
Program:	SIR92 [2]

Table 2. Atomic coordinates and displacement parameters (in Å²).

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

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Table 3. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> ₁₁	<i>U</i> ₂₂	<i>U</i> ₃₃	<i>U</i> ₁₂	<i>U</i> ₁₃	<i>U</i> ₂₃
O(1)	4e	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)	4e	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)	4e	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)	4e	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)	4e	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)	4e	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)	4e	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)	4e	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)	4e	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)	4e	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)	4e	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)	4e	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)	4e	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)	4e	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)	4e	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)	4e	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)	4e	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)	4e	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)	4e	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)	4e	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)	4e	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)

Acknowledgment. This research was financially supported by the research fund of the Overseas Visiting Scholarship Program by the Office of the Vice President for International Affairs at Chungnam National University.

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