Crystal structure of (*Z*)-2-amino-3-[(*E*)-2-(benzyloxy)-4-(diethylamino)-benzylideneamino]-2-butenedinitrile, $C_{22}H_{23}N_5O$

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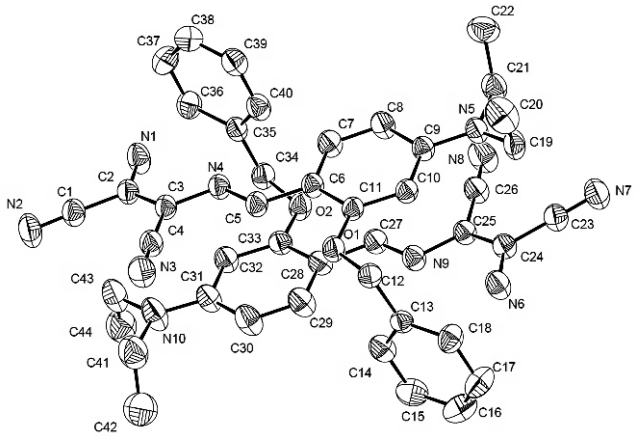

Abstract

$C_{22}H_{23}N_5O$, monoclinic, $P12_1/c1$ (no. 14), a = 14.2290(3) Å, b = 18.1985(3) Å, c = 16.0679(3) Å, β = 101.3791(7)°, V = 4079.0 Å^3, Z = 8, $R_{gt}(F)$ = 0.053, $wR_{ref}(F)$ = 0.084, T = 296 K.

Source of material

(*Z*)-2-amino-3-[(*E*)-2-(benzyloxy)-4-(diethylamino)benzylideneamino]-2-butenedinitrile was synthesized according to a previously described method [1] with some modification. Reddish block-shaped crystals of the title compound, suitable for X-ray diffraction measurements, were grown by solvent-diffusion method, using chloromethane and *n*-hexane as a good and a poor solvent, respectively.

Discussion

The title crystal structure comprises two molecules arranged in opposite directions. The bond lengths of part of the terminal diethyl amino group show small bond alternation (d(N10—C41) = 1.505(4) Å and d(C41—C42) = 1.423(5) Å) as compared with the given values (d(ArC—N) = 1.46 Å and d(ArCN—C) = 1.50 Å) by Allen [2]. The diaminomaleonitrile moiety of the title compound also exhibited little bond alternation in d(C1—C2) = 1.418(3) Å. Some small bond alternation may be caused in almost all π-conjugated systems. The small bond alternation was developed within the electro-donating amino groups and the electro-accepting dicyanoethylene groups. The title compound has an almost planar π-conjugated system and the subsituents are projected out of the planar π-conjugated molecular plane (∠O1–C12–C13 = 113.2(2)° and ∠O2–C34–C35 = 112.8(2)°). The terminal two ethyl substituents are projected out of the molecular plane and oriented in the same direction (∠N5–C19–C20 = 113.0(2)°, ∠N5–C21–C22 = 115.5(2)° and ∠(N10–C41–C42 = 110.6(2)°, ∠N10–C43–C44 = 110.8(2)°). π—π interaction was found between two composition molecules. Two molecules were stacked layer-by-layer with the shortest distance between two molecules about d(C9—C25) = 3.539(2) Å. This interaction displays the usual slipped stacking geometry, but with no interacting π-system parallelly displaced because of their conversed molecular direction.

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Table 1. Data collection and handling.

Crystal:	red block, size 0.15 × 0.30 × 0.30 mm
Wavelength:	Cu K_α radiation (1.54187 Å)
μ:	6.21 cm^{-1}
Diffractometer, scan mode:	Rigaku RAXIS-RAPID, ω
$2\theta_{max}$:	136.42°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	37768, 7439
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 3925
$N(param)_{refined}$:	551
Programs:	SIR92 [3], PLATON [4]

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

Atom	Site	x	y	z	U_{iso}
H(1)	4*e*	0.9214	0.1084	0.6316	0.090
H(2)	4*e*	0.9807	0.0329	0.6189	0.090
H(3)	4*e*	0.5341	0.4041	0.9313	0.090
H(4)	4*e*	0.5914	0.3260	0.9279	0.090
H(5)	4*e*	0.6192	0.1161	0.6593	0.067
H(6)	4*e*	0.7856	0.2613	0.6468	0.079
H(7)	4*e*	0.7639	0.3847	0.6643	0.080
H(8)	4*e*	0.5036	0.3422	0.7205	0.062
H(9)	4*e*	0.3984	0.2636	0.6941	0.071
H(10)	4*e*	0.3734	0.1811	0.7010	0.071
H(11)	4*e*	0.5438	0.1637	0.8536	0.081
H(12)	4*e*	0.5499	0.1779	0.9996	0.098
H(13)	4*e*	0.4344	0.2472	1.0469	0.114
H(14)	4*e*	0.3160	0.3058	0.9510	0.110
H(15)	4*e*	0.3113	0.2945	0.8053	0.083
H(16)	4*e*	0.4959	0.4516	0.7463	0.094
H(17)	4*e*	0.5292	0.5301	0.7282	0.094
H(18)	4*e*	0.4596	0.5245	0.5937	0.128
H(19)	4*e*	0.3901	0.4730	0.6294	0.128
H(20)	4*e*	0.4714	0.4399	0.5895	0.128
H(21)	4*e*	0.6804	0.5480	0.7272	0.087
H(22)	4*e*	0.7504	0.4844	0.7185	0.086
H(23)	4*e*	0.7304	0.4973	0.5828	0.119
H(24)	4*e*	0.7129	0.5782	0.6059	0.119
H(25)	4*e*	0.6265	0.5265	0.5747	0.119
H(26)	4*e*	0.8898	0.3099	0.8852	0.067

Table 2. Continued.

Atom	Site	*x*	*y*	*z*	U_{iso}
H(27)	4*e*	0.7215	0.1655	0.8989	0.086
H(28)	4*e*	0.7427	0.0416	0.8786	0.096
H(29)	4*e*	1.0111	0.0827	0.8405	0.073
H(30)	4*e*	1.1423	0.2410	0.8585	0.073
H(31)	4*e*	1.1150	0.1586	0.8601	0.072
H(32)	4*e*	1.1992	0.1431	0.7400	0.085
H(33)	4*e*	1.1837	0.1409	0.5929	0.104
H(34)	4*e*	1.0546	0.1999	0.5071	0.094
H(35)	4*e*	0.9415	0.2601	0.5698	0.088
H(36)	4*e*	0.9577	0.2616	0.7165	0.076

Table 2. Continued.

Atom	Site	*x*	*y*	*z*	U_{iso}
H(37)	4*e*	0.7541	–0.0595	0.8122	0.134
H(38)	4*e*	0.8258	–0.1216	0.8026	0.134
H(39)	4*e*	0.8508	–0.1498	0.9319	0.169
H(40)	4*e*	0.7462	–0.1218	0.9224	0.169
H(41)	4*e*	0.8312	–0.0701	0.9581	0.169
H(42)	4*e*	0.9773	–0.1015	0.8036	0.108
H(43)	4*e*	1.0113	–0.0217	0.7930	0.108
H(44)	4*e*	1.0879	–0.0153	0.9229	0.130
H(45)	4*e*	1.0985	–0.0992	0.9102	0.130
H(46)	4*e*	1.0183	–0.0697	0.9541	0.130

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*	0.5082(1)	0.19894(8)	0.6991(1)	0.0578(9)	0.0519(9)	0.089(1)	–0.0041(7)	0.0361(8)	–0.0080(8)
O(2)	4*e*	1.0085(1)	0.22440(8)	0.8669(1)	0.0543(9)	0.057(1)	0.086(1)	–0.0072(7)	0.0300(8)	–0.0148(8)
N(1)	4*e*	0.9227(1)	0.0566(1)	0.6244(1)	0.057(1)	0.052(1)	0.116(2)	0.003(1)	0.029(1)	–0.009(1)
N(2)	4*e*	0.8555(2)	–0.1228(1)	0.6036(2)	0.094(2)	0.056(2)	0.140(2)	0.008(1)	0.037(2)	–0.011(1)
N(3)	4*e*	0.6078(2)	–0.0354(1)	0.6267(2)	0.070(1)	0.071(2)	0.129(2)	–0.010(1)	0.042(1)	–0.011(1)
N(4)	4*e*	0.7504(1)	0.1210(1)	0.6423(1)	0.052(1)	0.050(1)	0.066(1)	0.0054(9)	0.0167(9)	–0.0011(9)
N(5)	4*e*	0.6169(1)	0.4519(1)	0.7047(1)	0.052(1)	0.047(1)	0.079(1)	–0.0005(9)	0.017(1)	–0.002(1)
N(6)	4*e*	0.5905(1)	0.3781(1)	0.9259(1)	0.054(1)	0.054(1)	0.116(2)	0.002(1)	0.031(1)	0.004(1)
N(7)	4*e*	0.6547(2)	0.5569(1)	0.9106(2)	0.094(2)	0.057(1)	0.104(2)	0.004(1)	0.019(1)	–0.002(1)
N(8)	4*e*	0.8967(2)	0.4643(1)	0.8893(2)	0.062(1)	0.078(2)	0.144(2)	–0.007(1)	0.029(1)	0.011(2)
N(9)	4*e*	0.7605(1)	0.3079(1)	0.9054(1)	0.049(1)	0.052(1)	0.070(1)	0.0057(9)	0.0128(9)	0.0036(9)
N(10)	4*e*	0.8927(2)	–0.0254(1)	0.8415(2)	0.063(1)	0.050(1)	0.161(2)	–0.004(1)	0.027(2)	0.002(1)
C(1)	4*e*	0.8500(2)	–0.0605(2)	0.6110(2)	0.058(1)	0.054(2)	0.085(2)	0.007(1)	0.022(1)	–0.003(1)
C(2)	4*e*	0.8425(2)	0.0166(1)	0.6216(2)	0.052(1)	0.044(1)	0.067(2)	0.001(1)	0.019(1)	–0.004(1)
C(3)	4*e*	0.7568(2)	0.0453(1)	0.6306(1)	0.052(1)	0.046(1)	0.063(2)	0.002(1)	0.017(1)	–0.003(1)
C(4)	4*e*	0.6756(2)	–0.0015(1)	0.6287(2)	0.058(2)	0.054(2)	0.081(2)	0.005(1)	0.026(1)	–0.006(1)
C(5)	4*e*	0.6710(2)	0.1484(1)	0.6572(1)	0.050(1)	0.054(1)	0.065(2)	0.005(1)	0.017(1)	–0.002(1)
C(6)	4*e*	0.6589(2)	0.2256(1)	0.6700(1)	0.044(1)	0.049(1)	0.062(2)	0.004(1)	0.014(1)	–0.002(1)
C(7)	4*e*	0.7275(2)	0.2778(1)	0.6612(2)	0.044(1)	0.062(2)	0.093(2)	0.009(1)	0.021(1)	0.002(1)
C(8)	4*e*	0.7150(2)	0.3512(1)	0.6716(2)	0.048(1)	0.052(2)	0.101(2)	–0.002(1)	0.024(1)	0.002(1)
C(9)	4*e*	0.6307(2)	0.3778(1)	0.6935(1)	0.044(1)	0.051(1)	0.058(2)	0.001(1)	0.008(1)	–0.002(1)
C(10)	4*e*	0.5612(2)	0.3261(1)	0.7048(1)	0.044(1)	0.052(1)	0.060(2)	0.003(1)	0.015(1)	–0.003(1)
C(11)	4*e*	0.5749(2)	0.2522(1)	0.6925(1)	0.047(1)	0.048(1)	0.055(1)	–0.002(1)	0.015(1)	–0.003(1)
C(12)	4*e*	0.4191(2)	0.2184(1)	0.7211(2)	0.046(1)	0.059(2)	0.072(2)	–0.003(1)	0.022(1)	–0.004(1)
C(13)	4*e*	0.4258(2)	0.2279(1)	0.8150(2)	0.046(1)	0.049(1)	0.059(2)	–0.005(1)	0.015(1)	0.001(1)
C(14)	4*e*	0.4975(2)	0.1930(1)	0.8733(2)	0.057(2)	0.062(2)	0.083(2)	0.003(1)	0.014(1)	0.008(1)
C(15)	4*e*	0.5002(2)	0.2008(2)	0.9599(2)	0.072(2)	0.100(2)	0.073(2)	0.003(2)	0.001(2)	0.019(2)
C(16)	4*e*	0.4328(2)	0.2429(2)	0.9877(2)	0.090(2)	0.131(2)	0.062(2)	–0.008(2)	0.017(2)	–0.006(2)
C(17)	4*e*	0.3625(2)	0.2767(2)	0.9311(2)	0.080(2)	0.123(2)	0.074(2)	0.020(2)	0.023(2)	–0.017(2)
C(18)	4*e*	0.3596(2)	0.2700(1)	0.8447(2)	0.059(1)	0.081(2)	0.068(2)	0.018(1)	0.014(1)	–0.002(1)
C(19)	4*e*	0.5227(2)	0.4809(1)	0.7080(2)	0.074(2)	0.050(2)	0.111(2)	0.009(1)	0.037(2)	–0.007(2)
C(20)	4*e*	0.4543(2)	0.4794(2)	0.6222(2)	0.058(2)	0.102(2)	0.159(3)	0.018(2)	0.003(2)	0.018(2)
C(21)	4*e*	0.6897(2)	0.5054(1)	0.6955(2)	0.076(2)	0.055(2)	0.085(2)	–0.011(1)	0.021(2)	–0.006(1)
C(22)	4*e*	0.6901(2)	0.5292(2)	0.6068(2)	0.088(2)	0.095(2)	0.113(2)	–0.005(2)	0.038(2)	0.024(2)
C(23)	4*e*	0.6616(2)	0.4943(2)	0.9139(2)	0.057(1)	0.059(2)	0.070(2)	0.003(1)	0.014(1)	–0.002(1)
C(24)	4*e*	0.6693(2)	0.4158(1)	0.9160(1)	0.051(1)	0.051(1)	0.061(2)	0.001(1)	0.012(1)	0.001(1)
C(25)	4*e*	0.7539(2)	0.3837(1)	0.9075(1)	0.047(1)	0.054(1)	0.059(2)	–0.002(1)	0.010(1)	0.002(1)
C(26)	4*e*	0.8323(2)	0.4303(1)	0.8979(2)	0.050(1)	0.056(2)	0.081(2)	0.002(1)	0.014(1)	0.003(1)
C(27)	4*e*	0.8384(2)	0.2782(1)	0.8909(1)	0.048(1)	0.058(2)	0.062(2)	–0.000(1)	0.011(1)	0.004(1)
C(28)	4*e*	0.8516(2)	0.2005(1)	0.8830(2)	0.047(1)	0.054(1)	0.064(2)	0.004(1)	0.012(1)	0.006(1)
C(29)	4*e*	0.7805(2)	0.1488(1)	0.8866(2)	0.045(1)	0.062(2)	0.110(2)	0.004(1)	0.018(1)	0.012(2)
C(30)	4*e*	0.7930(2)	0.0750(2)	0.8750(2)	0.053(2)	0.060(2)	0.128(2)	–0.004(1)	0.023(2)	0.010(2)
C(31)	4*e*	0.8791(2)	0.0482(1)	0.8562(2)	0.053(1)	0.052(2)	0.098(2)	0.002(1)	0.013(1)	0.008(1)
C(32)	4*e*	0.9518(2)	0.0989(1)	0.8526(2)	0.053(1)	0.053(2)	0.078(2)	0.001(1)	0.018(1)	0.001(1)
C(33)	4*e*	0.9388(2)	0.1725(1)	0.8662(1)	0.047(1)	0.053(1)	0.062(2)	–0.003(1)	0.014(1)	0.002(1)
C(34)	4*e*	1.0943(2)	0.2056(1)	0.8380(2)	0.046(1)	0.054(1)	0.082(2)	–0.003(1)	0.022(1)	–0.006(1)
C(35)	4*e*	1.0800(2)	0.2023(1)	0.7430(2)	0.047(1)	0.040(1)	0.071(2)	–0.000(1)	0.020(1)	0.000(1)
C(36)	4*e*	1.1461(2)	0.1667(1)	0.7051(2)	0.061(2)	0.071(2)	0.081(2)	0.018(1)	0.021(1)	0.005(1)
C(37)	4*e*	1.1371(2)	0.1657(2)	0.6175(2)	0.086(2)	0.088(2)	0.086(2)	0.014(2)	0.041(2)	–0.003(2)
C(38)	4*e*	1.0611(2)	0.2006(2)	0.5670(2)	0.089(2)	0.075(2)	0.071(2)	–0.004(2)	0.024(2)	0.008(2)
C(39)	4*e*	0.9944(2)	0.2359(1)	0.6043(2)	0.074(2)	0.061(2)	0.085(2)	0.006(1)	0.010(2)	0.009(2)
C(40)	4*e*	1.0038(2)	0.2369(1)	0.6913(2)	0.056(1)	0.053(2)	0.081(2)	0.007(1)	0.018(1)	–0.000(1)
C(41)	4*e*	0.8138(2)	–0.0813(2)	0.8366(2)	0.110(2)	0.084(2)	0.141(3)	0.005(2)	0.043(2)	0.003(2)

Table 3. Continued.

Atom	Site	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(42)	4*e*	0.8098(2)	–0.1084(2)	0.9188(2)	0.139(3)	0.126(3)	0.157(3)	0.038(2)	0.064(2)	0.029(2)
C(43)	4*e*	0.9858(2)	–0.0542(2)	0.8291(2)	0.089(2)	0.054(2)	0.128(2)	–0.001(2)	0.018(2)	–0.001(2)
C(44)	4*e*	1.0541(2)	–0.0604(2)	0.9112(2)	0.094(2)	0.096(2)	0.133(3)	0.009(2)	0.039(2)	0.004(2)

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