Crystal structure of 4-[(4-*N*,*N*-dimethylaminophenylene)amino]-3-ethoxy-3-cyclobutene-1,2-dione, $C_{14}H_{16}N_2O_3$

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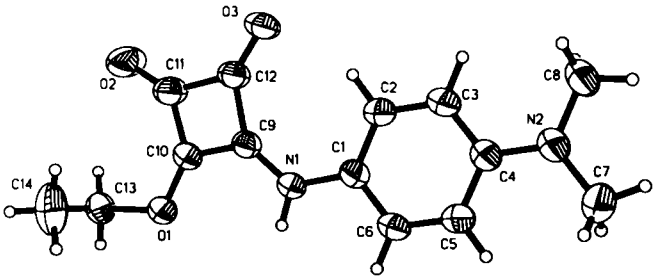

Abstract

$C_{14}H_{16}N_2O_3$, triclinic, $P\bar{1}$ (No. 2), $a = 6.3066(1)$ Å, $b = 7.5559(2)$ Å, $c = 14.7551(4)$ Å, $\alpha = 88.5449(9)°$, $\beta = 82.533(1)°$, $\gamma = 69.205(1)°$, $V = 651.6$ Å^3, $Z = 2$, $R_{gt}(F) = 0.046$, $wR_{ref}(F^2) = 0.134$, $T = 291$ K.

Source of material

The title compound was prepared according to the general procedure described by Tietze et al. [1]. Diethylester of squaric acid (3,4-diethoxy-3-cyclobutene-1,2-dione, 1.21 g, 7.1 mmol) was dissolved in 10 ml ethanol. 4-Amino-*N*,*N*-dimethylaniline (0.97 g, 7.1 mmol) was dissolved in 50 ml ethanol and added to the first solution at continuous stirring at room temperature. The reaction mixture was stirred for 6 hours. The obtained yellow precipitate was filtered off, dried in vacuo and recrystallized from ethanol (mp 413 K – 415 K). Crystals were grown by slow evaporation from ethyl acetate.

Discussion

In the course of our investigations on organic nonlinear optical (NLO) materials we now determined the crystal structure of the title compound. The asymmetric unit of the crystal contains one formula unit $C_{14}H_{16}N_2O_3$. The molecule except the ethoxy group is nearly planar. The molecules are linked by a N—H···O hydrogen bond (d(N1—H1···O3) = 3.007(2) Å, ∠(N–H···O) = 171°). A short intramolecular H···O contact (2.21 Å) indicates a intramolecular hydrogen bond (d(C2—H2···O3) = 3.130(2) Å, ∠(C–H···O) = 155°).

Table 1. Data collection and handling.

Crystal:	yellow plate, size 0.1 × 0.4 × 0.5 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	0.94 cm^{-1}
Diffractometer, scan mode:	Nonius KappaCCD, 410 frames, $\Delta\omega = 1°$
$2\theta_{max}$:	54.86°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	8182, 2910
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 1957
$N(param)_{refined}$:	236
Programs:	SHELXS-97 [2], SHELXTL-Plus [3], SHELXL-97 [4], PARST95 [5]

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

Atom	Site	x	y	z	U_{iso}
H(1)	2*i*	0.788(3)	0.314(2)	0.368(1)	0.056(5)
H(2)	2*i*	0.270(3)	0.295(3)	0.479(1)	0.078(6)
H(3)	2*i*	0.211(4)	0.216(3)	0.634(1)	0.078(6)
H(5)	2*i*	0.869(3)	0.133(2)	0.658(1)	0.069(5)
H(6)	2*i*	0.924(3)	0.220(3)	0.506(1)	0.073(5)
H(7A)	2*i*	0.734(4)	0.176(3)	0.816(2)	0.095(7)
H(7B)	2*i*	0.817(4)	–0.037(3)	0.783(1)	0.090(6)
H(7C)	2*i*	0.625(3)	0.038(3)	0.868(2)	0.082(6)
H(8A)	2*i*	0.278(3)	0.101(3)	0.858(2)	0.085(6)
H(8B)	2*i*	0.212(3)	0.044(3)	0.765(1)	0.082(6)
H(8C)	2*i*	0.167(3)	0.258(3)	0.790(1)	0.069(5)
H(13A)	2*i*	0.875(4)	0.493(3)	0.079(1)	0.093(7)
H(13B)	2*i*	0.610(3)	0.521(2)	0.078(1)	0.069(5)
H(14A)	2*i*	0.988(5)	0.168(4)	0.050(2)	0.14(1)
H(14B)	2*i*	0.717(5)	0.197(4)	0.051(2)	0.14(1)
H(14C)	2*i*	0.850(3)	0.279(3)	–0.029(2)	0.081(6)

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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)	2*i*	0.7564(2)	0.3975(2)	0.18661(7)	0.0455(6)	0.0931(8)	0.0379(6)	–0.0384(6)	–0.0022(4)	0.0066(5)
O(2)	2*i*	0.2471(2)	0.4309(2)	0.13762(9)	0.0582(7)	0.143(1)	0.0621(8)	–0.0505(8)	–0.0272(6)	0.0428(8)
O(3)	2*i*	0.1186(2)	0.3383(2)	0.34999(7)	0.0371(6)	0.0937(9)	0.0564(7)	–0.0311(6)	–0.0032(5)	0.0152(6)
N(1)	2*i*	0.6538(2)	0.3054(2)	0.38560(8)	0.0359(7)	0.0629(8)	0.0382(7)	–0.0261(6)	–0.0006(5)	0.0026(5)
N(2)	2*i*	0.5009(2)	0.1128(2)	0.75254(9)	0.0480(8)	0.105(1)	0.0464(8)	–0.0306(8)	–0.0050(6)	0.0229(7)
C(1)	2*i*	0.6077(2)	0.2600(2)	0.47827(9)	0.0379(7)	0.0471(8)	0.0392(8)	–0.0191(6)	–0.0007(6)	–0.0002(6)
C(2)	2*i*	0.3960(3)	0.2619(3)	0.5170(1)	0.0427(9)	0.090(1)	0.0467(9)	–0.0349(8)	–0.0090(7)	0.0113(8)
C(3)	2*i*	0.3615(3)	0.2148(3)	0.6071(1)	0.0413(9)	0.089(1)	0.0501(9)	–0.0335(8)	–0.0035(7)	0.0118(8)
C(4)	2*i*	0.5365(2)	0.1638(2)	0.6622(1)	0.0411(8)	0.0545(8)	0.0421(8)	–0.0192(7)	–0.0030(6)	0.0062(6)
C(5)	2*i*	0.7475(3)	0.1655(2)	0.6221(1)	0.0380(8)	0.077(1)	0.0493(9)	–0.0229(7)	–0.0088(7)	0.0113(8)
C(6)	2*i*	0.7817(3)	0.2131(2)	0.5320(1)	0.0333(8)	0.069(1)	0.0472(9)	–0.0218(7)	–0.0013(6)	0.0074(7)
C(7)	2*i*	0.6814(4)	0.0692(3)	0.8087(1)	0.067(1)	0.083(1)	0.049(1)	–0.033(1)	–0.0149(9)	0.0125(9)
C(8)	2*i*	0.2748(3)	0.1321(3)	0.7935(1)	0.054(1)	0.072(1)	0.048(1)	–0.0253(9)	0.0043(8)	0.0095(9)
C(9)	2*i*	0.5199(2)	0.3416(2)	0.32008(9)	0.0357(7)	0.0426(7)	0.0412(8)	–0.0169(6)	–0.0017(6)	–0.0011(6)
C(10)	2*i*	0.5659(2)	0.3829(2)	0.22867(9)	0.0382(7)	0.0504(8)	0.0392(8)	–0.0215(6)	–0.0002(6)	0.0029(6)
C(11)	2*i*	0.3443(3)	0.3981(2)	0.2059(1)	0.0412(8)	0.065(1)	0.0533(9)	–0.0243(7)	–0.0091(7)	0.0133(7)
C(12)	2*i*	0.2854(2)	0.3542(2)	0.3046(1)	0.0346(7)	0.0544(9)	0.0488(9)	–0.0181(6)	–0.0046(6)	0.0068(6)
C(13)	2*i*	0.7632(3)	0.4328(3)	0.0884(1)	0.052(1)	0.075(1)	0.0365(8)	–0.0306(9)	–0.0034(7)	0.0119(7)
C(14)	2*i*	0.8329(6)	0.2542(3)	0.0359(2)	0.122(2)	0.082(1)	0.043(1)	–0.029(2)	–0.007(1)	0.004(1)

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