Crystal structure of 4,8-dimethyl-3,7-diphenylcycloocta-1,3,5,7-tetraene-1,5-dicarbonitrile, $C_8H_2(C_6H_5)_2(CH_3)_2(CN)_2$

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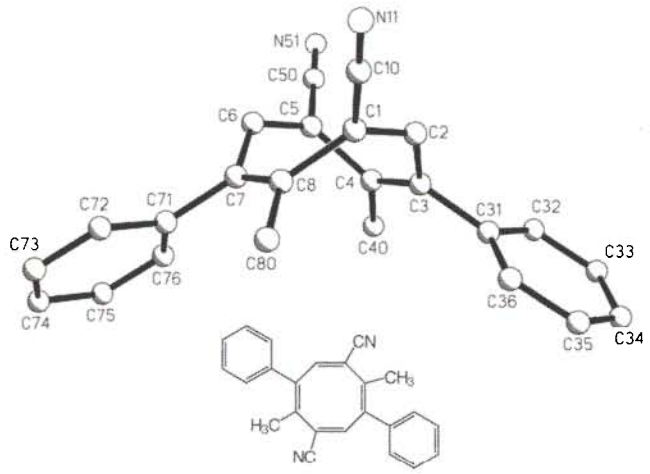

Abstract

$C_{24}H_{18}N_2$, monoclinic, $P12_1/n1$ (No. 14), a = 14.193(2) Å, b = 14.741(2) Å, c = 9.405(1) Å, β = 98.68(1)°, V = 1945.2 Å^3, Z = 4, $R_{gt}(F)$ = 0.052, $wR(F)$ = 0.053, T = 293 K.

Source of material

The title compound was prepared, according to [1], by heating a solution of 1,5-dimethyl-4,8-diphenylsemibullvalene-2,6-dicarbonitrile [2], in ethyl acetate under argon and reflux [3], followed by medium-pressure liquid chromatography of the product on silicagel with petroleum ether/ethyl acetate (92:8). Recrystallization of the product from dichloromethane afforded coarse, pale yellow crystals, mp 421 K – 423 K.

Table 1. Data collection and handling.

Crystal:	pale yellow prism, size 0.8 × 0.9 × 0.5 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	0.70 cm^{-1}
Diffractometer, scan mode:	Siemens R3m/V, Wyckoff
$2\theta_{max}$:	55°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	4820, 4471
Criterion for F_{obs}, $N(hkl)_{gt}$:	$F_{obs} > 3\ \sigma(F_{obs})$, 3736
$N(param)_{refined}$:	236
Program:	SHELXTL-plus [4]

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

Atom	Site	x	y	z	U_{iso}
H(2)	4e	0.2886(1)	0.5500(1)	0.3531(2)	0.08
H(6)	4e	0.1772(1)	0.2635(1)	0.2303(2)	0.08
H(32)	4e	0.1998(2)	0.6466(1)	–0.0292(2)	0.08
H(33)	4e	0.2687(2)	0.7791(2)	–0.1059(2)	0.08
H(34)	4e	0.4316(2)	0.8065(2)	–0.0373(3)	0.08
H(35)	4e	0.5253(2)	0.7019(2)	0.1035(3)	0.08
H(36)	4e	0.4590(2)	0.5683(2)	0.1779(3)	0.08
H(40A)	4e	0.2300(1)	0.5082(1)	–0.1413(2)	0.08
H(40B)	4e	0.1293(1)	0.4674(1)	–0.1309(2)	0.08
H(40C)	4e	0.2148(1)	0.4029(1)	–0.1474(2)	0.08
H(72)	4e	0.4255(1)	0.1439(1)	0.3124(2)	0.08
H(73)	4e	0.4559(1)	0.0119(1)	0.1889(3)	0.08
H(74)	4e	0.3953(2)	–0.0048(2)	–0.0535(3)	0.08
H(75)	4e	0.3048(2)	0.1110(2)	–0.1748(2)	0.08
H(76)	4e	0.2722(1)	0.2424(1)	–0.0540(2)	0.08
H(80A)	4e	0.5060(1)	0.2800(1)	0.2811(2)	0.08
H(80B)	4e	0.5072(1)	0.3283(1)	0.4298(2)	0.08
H(80C)	4e	0.5194(1)	0.3854(1)	0.2931(2)	0.08

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}
C(1)	4e	0.3463(1)	0.4289(1)	0.3633(2)	0.0475(9)	0.055(1)	0.0447(9)	–0.0032(8)	0.0018(7)	–0.0041(8)
C(2)	4e	0.3046(1)	0.5005(1)	0.2949(2)	0.0491(9)	0.052(1)	0.0491(9)	–0.0034(8)	0.0075(7)	–0.0056(8)
C(3)	4e	0.2806(1)	0.5109(1)	0.1367(2)	0.0500(9)	0.051(1)	0.049(1)	0.0045(8)	0.0078(8)	0.0020(8)
C(4)	4e	0.2221(1)	0.4536(1)	0.0564(2)	0.0517(9)	0.052(1)	0.0462(9)	0.0067(8)	0.0046(8)	0.0019(8)
C(5)	4e	0.1740(1)	0.3781(1)	0.1237(2)	0.0463(9)	0.053(1)	0.0443(9)	0.0001(8)	0.0025(7)	–0.0063(8)
C(6)	4e	0.2166(1)	0.3076(1)	0.1925(2)	0.0481(9)	0.051(1)	0.0457(9)	–0.0017(8)	0.0078(7)	–0.0024(8)
C(7)	4e	0.3210(1)	0.2915(1)	0.2156(2)	0.0482(9)	0.0486(9)	0.0450(9)	0.0031(7)	0.0064(7)	0.0033(7)
C(8)	4e	0.3815(1)	0.3465(1)	0.2954(2)	0.0488(9)	0.055(1)	0.049(1)	0.0036(8)	0.0049(8)	0.0015(8)
C(10)	4e	0.3658(1)	0.4321(1)	0.5186(2)	0.062(1)	0.069(1)	0.054(1)	0.002(1)	–0.0010(9)	–0.005(1)
N(11)	4e	0.3798(2)	0.4331(2)	0.6413(2)	0.103(2)	0.120(2)	0.052(1)	0.007(1)	–0.004(1)	–0.007(1)

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Table 3. Continued.

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(31)	4*e*	0.3227(1)	0.5940(1)	0.0803(2)	0.057(1)	0.054(1)	0.054(1)	–0.0008(8)	0.0139(8)	–0.0005(8)
C(32)	4*e*	0.2669(2)	0.6572(1)	–0.0025(2)	0.072(1)	0.059(1)	0.059(1)	0.003(1)	0.018(1)	0.0029(9)
C(33)	4*e*	0.3074(2)	0.7359(2)	–0.0472(2)	0.109(2)	0.060(1)	0.072(1)	0.008(1)	0.033(1)	0.011(1)
C(34)	4*e*	0.4035(2)	0.7519(2)	–0.0071(3)	0.114(2)	0.075(2)	0.101(2)	–0.026(2)	0.051(2)	0.007(1)
C(35)	4*e*	0.4585(2)	0.6903(2)	0.0756(3)	0.074(2)	0.100(2)	0.123(2)	–0.022(1)	0.033(2)	0.009(2)
C(36)	4*e*	0.4194(2)	0.6113(2)	0.1199(3)	0.061(1)	0.080(2)	0.089(2)	–0.006(1)	0.015(1)	0.010(1)
C(40)	4*e*	0.1967(1)	0.4585(1)	–0.1056(2)	0.079(1)	0.068(1)	0.049(1)	0.006(1)	0.002(1)	0.0027(9)
C(50)	4*e*	0.0709(1)	0.3805(1)	0.1007(2)	0.053(1)	0.061(1)	0.057(1)	0.0015(9)	–0.0007(8)	–0.0052(9)
N(51)	4*e*	–0.0100(1)	0.3824(1)	0.0828(2)	0.051(1)	0.105(2)	0.092(1)	0.004(1)	–0.0040(9)	–0.010(1)
C(71)	4*e*	0.3468(1)	0.2071(1)	0.1416(2)	0.0487(9)	0.051(1)	0.0497(9)	0.0006(8)	0.0104(8)	0.0010(8)
C(72)	4*e*	0.4004(1)	0.1378(1)	0.2122(2)	0.052(1)	0.059(1)	0.060(1)	0.0051(8)	0.0121(8)	0.0066(9)
C(73)	4*e*	0.4183(1)	0.0595(1)	0.1391(3)	0.060(1)	0.056(1)	0.087(2)	0.0103(9)	0.024(1)	0.010(1)
C(74)	4*e*	0.3828(2)	0.0496(2)	–0.0034(3)	0.081(2)	0.063(1)	0.086(2)	0.006(1)	0.032(1)	–0.015(1)
C(75)	4*e*	0.3293(2)	0.1177(2)	–0.0745(2)	0.098(2)	0.077(2)	0.059(1)	0.010(1)	0.013(1)	–0.015(1)
C(76)	4*e*	0.3106(1)	0.1956(1)	–0.0033(2)	0.075(1)	0.062(1)	0.053(1)	0.011(1)	0.006(1)	–0.0018(9)
C(80)	4*e*	0.4883(1)	0.3339(1)	0.3278(2)	0.052(1)	0.074(1)	0.084(1)	0.001(1)	–0.001(1)	–0.012(1)

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