Crystal structure of 4,5,2′,3′-tetrahydro-4,4,1′,3′-tetramethyl-1-(2-nitrophenyl)spiro-{[1*H*][1,2,3]-triazole-5,2′-benzimidazole}, $[C_6H_4N_2(CH_3)_2]C[CN_3(C_6H_4NO_2)(CH_3)_2]$

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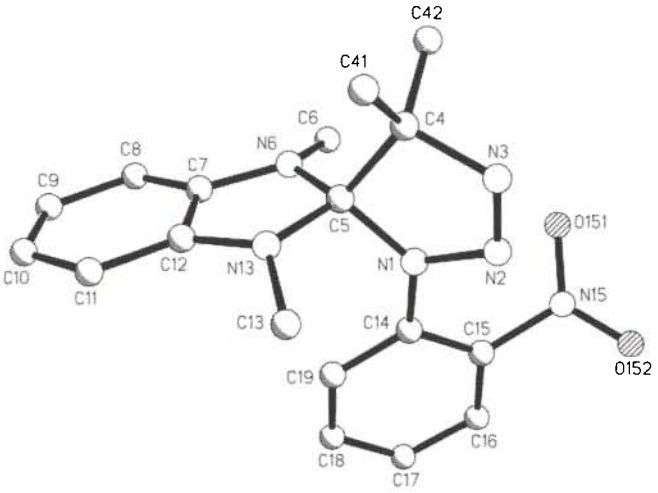

Abstract

$C_{18}H_{20}N_6O_2$, monoclinic, $P12_1/n1$ (No. 14), a = 16.520(7) Å, b = 8.346(2) Å, c = 13.160(6) Å, β = 99.24(3)°, V = 1790.9 Å^3, Z = 4, $R_{gt}(F)$ = 0.058, $wR(F)$ = 0.052, T = 293 K.

Source of material

The compound was prepared, according to [1], by the addition of 2-nitrophenyl azide to a stirred solution of 1,3-dimethyl-2-(1-methylethylidene)-2,3-dihydro-1*H*-benzimidazole [2] in dry toluene cooled at 278 K. The mixture immediately turned deep red. Distillation of the solvent in vacuo afforded a red solid, which was suspended in a small amount of pentane and kept at 253 K for two days to give a red solution and yellow crystals, mp 368 K – 369 K (decomposition with evolution of nitrogen).

Table 1. Data collection and handling.

Crystal:	yellow plate, size 0.5 × 1.0 × 0.2 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	0.90 cm^{-1}
Diffractometer, scan mode:	Siemens R3m/V, Wyckoff
$2\theta_{max}$:	55°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	4533, 4115
Criterion for F_{obs}, $N(hkl)_{gt}$:	$F_{obs} > 3\ \sigma(F_{obs})$, 2593
$N(param)_{refined}$:	236
Program:	SHELXTL-plus [3]

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

Atom	Site	x	y	z	U_{iso}
H(6A)	4*e*	0.5374(2)	0.3308(4)	0.4753(2)	0.08
H(6B)	4*e*	0.5356(2)	0.2549(4)	0.5839(2)	0.08
H(6C)	4*e*	0.5208(2)	0.1469(4)	0.4851(2)	0.08
H(8A)	4*e*	0.5904(2)	–0.0006(3)	0.6751(2)	0.08
H(9A)	4*e*	0.6754(3)	–0.2053(4)	0.7555(2)	0.08
H(10A)	4*e*	0.8048(3)	–0.2449(4)	0.7160(2)	0.08
H(11A)	4*e*	0.8538(2)	–0.0905(3)	0.5884(2)	0.08
H(13A)	4*e*	0.8184(2)	0.2167(4)	0.3665(2)	0.08
H(13B)	4*e*	0.8041(2)	0.0317(4)	0.3744(2)	0.08
H(13C)	4*e*	0.8734(2)	0.1158(4)	0.4514(2)	0.08
H(16A)	4*e*	0.4711(2)	0.0453(4)	0.0889(2)	0.08
H(17A)	4*e*	0.4957(2)	–0.2184(4)	0.1448(2)	0.08
H(18A)	4*e*	0.5883(2)	–0.2700(4)	0.2949(2)	0.08
H(19A)	4*e*	0.6590(2)	–0.0604(3)	0.3866(2)	0.08
H(41A)	4*e*	0.8037(2)	0.4557(4)	0.5831(2)	0.08
H(41B)	4*e*	0.8093(2)	0.5947(4)	0.5045(2)	0.08
H(41C)	4*e*	0.8381(2)	0.4216(4)	0.4810(2)	0.08
H(42A)	4*e*	0.6528(2)	0.5249(3)	0.5725(2)	0.08
H(42B)	4*e*	0.5969(2)	0.5274(3)	0.4643(2)	0.08
H(42C)	4*e*	0.6643(2)	0.6593(3)	0.4929(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}
N(1)	4*e*	0.6507(1)	0.2486(3)	0.3476(1)	0.063(1)	0.057(1)	0.049(1)	–0.008(1)	–0.0076(9)	0.014(1)
N(2)	4*e*	0.6706(1)	0.3776(3)	0.2908(2)	0.075(1)	0.072(2)	0.060(1)	–0.010(1)	–0.007(1)	0.021(1)
N(3)	4*e*	0.7070(2)	0.4855(3)	0.3473(2)	0.086(2)	0.065(1)	0.066(1)	–0.015(1)	–0.006(1)	0.015(1)
C(4)	4*e*	0.7129(2)	0.4434(3)	0.4573(2)	0.060(2)	0.055(2)	0.059(1)	–0.001(1)	–0.001(1)	0.006(1)
C(5)	4*e*	0.6904(1)	0.2600(3)	0.4573(2)	0.048(1)	0.054(1)	0.048(1)	0.002(1)	0.003(1)	0.005(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}
N(6)	4*e*	0.6381(1)	0.2059(2)	0.5284(1)	0.053(1)	0.059(1)	0.062(1)	0.003(1)	0.013(1)	0.005(1)
C(6)	4*e*	0.5505(2)	0.2373(4)	0.5172(2)	0.062(2)	0.084(2)	0.104(2)	0.002(2)	0.025(2)	0.002(2)
C(7)	4*e*	0.6733(2)	0.0760(3)	0.5861(2)	0.077(2)	0.048(1)	0.041(1)	–0.004(1)	0.006(1)	–0.004(1)
C(8)	4*e*	0.6441(2)	–0.0179(3)	0.6580(2)	0.122(3)	0.062(2)	0.055(2)	–0.019(2)	0.027(2)	–0.003(1)
C(9)	4*e*	0.6944(3)	–0.1381(4)	0.7050(2)	0.204(5)	0.065(2)	0.050(2)	–0.019(3)	0.016(2)	0.008(2)
C(10)	4*e*	0.7707(3)	–0.1626(4)	0.6806(2)	0.182(4)	0.057(2)	0.060(2)	0.014(3)	–0.029(2)	0.013(2)
C(11)	4*e*	0.8005(2)	–0.0713(3)	0.6060(2)	0.097(2)	0.061(2)	0.067(2)	0.017(2)	–0.021(2)	–0.001(2)
C(12)	4*e*	0.7500(2)	0.0479(3)	0.5585(2)	0.070(2)	0.049(1)	0.042(1)	0.005(1)	–0.008(1)	–0.003(1)
N(13)	4*e*	0.7616(1)	0.1566(2)	0.4829(1)	0.048(1)	0.061(1)	0.050(1)	0.010(1)	0.0066(9)	0.005(1)
C(13)	4*e*	0.8193(2)	0.1278(4)	0.4129(2)	0.062(2)	0.095(2)	0.071(2)	0.008(2)	0.014(1)	–0.004(2)
C(14)	4*e*	0.6070(1)	0.1214(3)	0.2949(2)	0.051(1)	0.065(2)	0.047(1)	–0.002(1)	0.001(1)	0.004(1)
C(15)	4*e*	0.5482(2)	0.1491(4)	0.2076(2)	0.053(1)	0.083(2)	0.057(1)	–0.005(2)	–0.006(1)	0.013(1)
N(15)	4*e*	0.5209(2)	0.3118(4)	0.1749(2)	0.078(2)	0.107(3)	0.094(2)	–0.014(2)	–0.031(2)	0.036(2)
O(151)	4*e*	0.5030(2)	0.4050(3)	0.2411(2)	0.101(2)	0.097(2)	0.159(3)	0.023(2)	–0.019(2)	0.022(2)
O(152)	4*e*	0.5155(2)	0.3450(4)	0.0843(2)	0.148(2)	0.167(3)	0.105(2)	–0.034(2)	–0.053(2)	0.072(2)
C(16)	4*e*	0.5087(2)	0.0237(4)	0.1508(2)	0.056(2)	0.118(3)	0.056(2)	–0.013(2)	–0.006(1)	–0.004(2)
C(17)	4*e*	0.5236(2)	–0.1315(4)	0.1833(2)	0.068(2)	0.091(2)	0.075(2)	–0.018(2)	0.008(2)	–0.024(2)
C(18)	4*e*	0.5787(2)	–0.1618(4)	0.2713(2)	0.068(2)	0.069(2)	0.072(2)	–0.004(2)	0.012(1)	–0.012(1)
C(19)	4*e*	0.6202(2)	–0.0371(3)	0.3259(2)	0.061(2)	0.064(2)	0.054(1)	–0.001(1)	0.001(1)	0.001(1)
C(41)	4*e*	0.7989(2)	0.4824(4)	0.5115(2)	0.076(2)	0.077(2)	0.085(2)	–0.014(2)	–0.006(2)	–0.003(2)
C(42)	4*e*	0.6510(2)	0.5485(3)	0.5008(2)	0.089(2)	0.057(2)	0.103(2)	0.006(2)	0.007(2)	–0.002(2)

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