Crystal structure of (*E*)-2,5-di-*tert*-butyl-3-(N-*tert*-butylimino)-2,3-dihydroindazyl, $C_7H_3N_2(NC_4H_9)(C_4H_9)_2$

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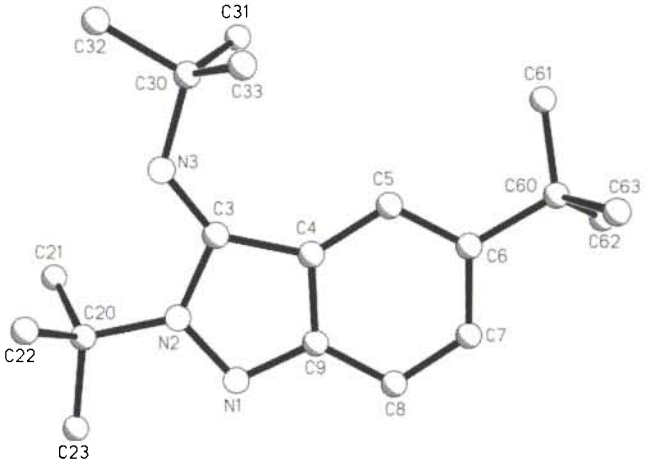

Abstract

$C_{19}H_{30}N_3$, monoclinic, $P12_1/c1$ (No. 14), $a = 14.378(1)$ Å, $b = 11.737(1)$ Å, $c = 11.750(1)$ Å, $\beta = 101.79(1)°$, $V = 1941.0$ Å^3, $Z = 4$, $R_{gt}(F) = 0.084$, $wR(F) = 0.082$, $T = 273$ K.

Source of material

The novel stable free radical was prepared, according to[1], by stirring of a suspension of 2,5-di-*tert*-butyl-3-(N-*tert*-butylamino)-2*H*-indazole (2 mmol), lead dioxide (20 mmol), and potassium carbonate (5 g) in dry benzene (60 ml) for 15 min at room temperature. The solid material was removed by filtration and the solvent distilled in vacuo to afford a deep red oil. Destillation of the oil at 293 K - 298 K/10^{-5} Torr gave a deep red oil which slowly crystallized. Recrystallization from acetonitrile yielded black-red, shining cubes, mp 375 K.

Table 1. Data collection and handling.

Crystal:	black red, size 0.55 × 0.50 × 0.30 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	0.60 cm^{-1}
Diffractometer, scan mode:	Bruker AXS P4, ω
$2\theta_{max}$:	55°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	5660, 4416
Criterion for F_{obs}, $N(hkl)_{gt}$:	$F_{obs} > 3\ \sigma(F_{obs})$, 3198
$N(param))_{refined}$:	199
Program:	SHELXTL-plus [2]

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

Atom	Site	*x*	*y*	*z*	U_{iso}
H(5)	4*e*	0.8105(2)	0.0920(2)	0.3373(2)	0.08
H(7)	4*e*	0.5256(2)	0.1365(3)	0.3043(3)	0.08
H(8)	4*e*	0.4946(2)	0.0094(3)	0.1486(3)	0.08
H(21A)	4*e*	0.8055(3)	–0.3791(3)	–0.0037(3)	0.08
H(21B)	4*e*	0.7557(3)	–0.3538(3)	0.1005(3)	0.08
H(21C)	4*e*	0.8516(3)	–0.2932(3)	0.0938(3)	0.08
H(22A)	4*e*	0.8003(3)	–0.2293(3)	–0.1585(3)	0.08
H(22B)	4*e*	0.8465(3)	–0.1395(3)	–0.0651(3)	0.08
H(22C)	4*e*	0.7480(3)	–0.1141(3)	–0.1466(3)	0.08
H(23A)	4*e*	0.6512(3)	–0.3297(4)	–0.1374(4)	0.08
H(23B)	4*e*	0.6058(3)	–0.2104(4)	–0.1232(4)	0.08
H(23C)	4*e*	0.6091(3)	–0.3039(4)	–0.0271(4)	0.08
H(31A)	4*e*	1.0199(2)	–0.0307(3)	0.3923(2)	0.08
H(31B)	4*e*	0.9730(2)	–0.1494(3)	0.3560(2)	0.08
H(31C)	4*e*	0.9092(2)	–0.0442(3)	0.3705(2)	0.08
H(32A)	4*e*	1.1013(2)	–0.0520(3)	0.2254(3)	0.08
H(32B)	4*e*	1.0409(2)	–0.0674(3)	0.0993(3)	0.08
H(32C)	4*e*	1.0472(2)	–0.1666(3)	0.1898(3)	0.08
H(33A)	4*e*	0.9013(2)	0.1231(3)	0.2234(3)	0.08
H(33B)	4*e*	0.9514(2)	0.1061(3)	0.1180(3)	0.08
H(33C)	4*e*	1.0125(2)	0.1245(3)	0.2433(3)	0.08
H(61A)	4*e*	0.8278(3)	0.2168(5)	0.4626(4)	0.08
H(61B)	4*e*	0.7948(3)	0.1280(5)	0.5459(4)	0.08
H(61C)	4*e*	0.7921(3)	0.2586(5)	0.5730(4)	0.08
H(62A)	4*e*	0.6346(4)	0.1163(6)	0.5481(4)	0.08
H(62B)	4*e*	0.5585(4)	0.1985(6)	0.4765(4)	0.08
H(62C)	4*e*	0.6356(4)	0.2462(6)	0.5794(4)	0.08
H(63A)	4*e*	0.7148(6)	0.3390(4)	0.3416(5)	0.08
H(63B)	4*e*	0.6853(6)	0.3802(4)	0.4562(5)	0.08
H(63C)	4*e*	0.6082(6)	0.3324(4)	0.3532(5)	0.08

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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}
N(1)	4*e*	0.6287(2)	–0.1089(2)	0.0687(2)	0.041(1)	0.060(2)	0.057(1)	0.002(1)	0.004(1)	–0.014(1)
N(2)	4*e*	0.7185(2)	–0.1366(2)	0.0658(2)	0.049(1)	0.055(1)	0.051(1)	0.003(1)	0.004(1)	–0.015(1)
C(3)	4*e*	0.7878(2)	–0.0750(2)	0.1466(2)	0.041(1)	0.042(1)	0.047(1)	0.000(1)	0.008(1)	–0.003(1)
N(3)	4*e*	0.8757(2)	–0.0944(2)	0.1454(2)	0.046(1)	0.053(1)	0.056(1)	0.004(1)	0.013(1)	–0.007(1)
C(4)	4*e*	0.7292(2)	–0.0040(2)	0.2072(2)	0.041(1)	0.040(1)	0.041(1)	0.004(1)	0.008(1)	0.001(1)
C(5)	4*e*	0.7465(2)	0.0737(2)	0.2998(2)	0.039(1)	0.047(2)	0.047(1)	0.002(1)	0.007(1)	–0.005(1)
C(6)	4*e*	0.6710(2)	0.1252(2)	0.3375(2)	0.049(1)	0.046(2)	0.046(1)	0.006(1)	0.010(1)	–0.004(1)
C(7)	4*e*	0.5777(2)	0.1000(3)	0.2793(3)	0.042(1)	0.063(2)	0.070(2)	0.012(1)	0.011(1)	–0.010(2)
C(8)	4*e*	0.5588(2)	0.0249(3)	0.1872(3)	0.037(1)	0.067(2)	0.067(2)	0.005(1)	0.004(1)	–0.013(2)
C(9)	4*e*	0.6354(2)	–0.0291(2)	0.1535(2)	0.041(1)	0.048(2)	0.047(1)	0.002(1)	0.006(1)	–0.005(1)
C(20)	4*e*	0.7367(2)	–0.2258(3)	–0.0176(2)	0.060(2)	0.061(2)	0.053(2)	0.006(1)	0.007(1)	–0.022(1)
C(21)	4*e*	0.7927(3)	–0.3216(3)	0.0491(3)	0.112(3)	0.058(2)	0.073(2)	0.012(2)	0.019(2)	–0.017(2)
C(22)	4*e*	0.7877(3)	–0.1725(3)	–0.1049(3)	0.139(4)	0.086(3)	0.067(2)	0.009(3)	0.036(2)	–0.012(2)
C(23)	4*e*	0.6423(3)	–0.2720(4)	–0.0827(4)	0.077(3)	0.133(4)	0.133(3)	0.006(2)	0.000(2)	–0.090(3)
C(30)	4*e*	0.9582(2)	–0.0377(2)	0.2191(2)	0.038(1)	0.051(2)	0.052(2)	0.000(1)	0.009(1)	–0.003(1)
C(31)	4*e*	0.9659(2)	–0.0684(3)	0.3462(2)	0.053(2)	0.076(2)	0.059(2)	0.004(2)	0.010(1)	0.003(2)
C(32)	4*e*	1.0449(2)	–0.0854(3)	0.1798(3)	0.049(2)	0.068(2)	0.077(2)	0.008(1)	0.020(1)	0.001(2)
C(33)	4*e*	0.9555(2)	0.0910(3)	0.1992(3)	0.056(2)	0.059(2)	0.077(2)	–0.004(1)	0.017(1)	0.001(2)
C(60)	4*e*	0.6874(2)	0.2105(3)	0.4379(3)	0.062(2)	0.064(2)	0.067(2)	0.012(2)	0.011(2)	–0.020(2)
C(61)	4*e*	0.7836(3)	0.2026(5)	0.5123(4)	0.105(3)	0.216(6)	0.153(4)	0.042(4)	–0.027(3)	–0.140(4)
C(62)	4*e*	0.6234(4)	0.1919(6)	0.5171(4)	0.166(5)	0.34(1)	0.147(5)	–0.125(6)	0.098(4)	–0.156(6)
C(63)	4*e*	0.6729(6)	0.3254(4)	0.3942(5)	0.46(1)	0.089(4)	0.125(5)	0.063(6)	–0.005(7)	–0.049(4)

References

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