Gamal A. El-Hiti*, Keith Smith, Amany S. Hegazy, Ali M. Masmali and Benson M. Kariuki

Crystal structure of 3-*tert*-butyl-7-azadioxindole, $C_{11}H_{14}N_2O_2$

Abstract

$C_{11}H_{14}N_2O_2$, orthorhombic, $P2_12_12_1$ (no. 19), $a = 7.3227(3)$ Å, $b = 10.9955(4)$ Å, $c = 26.3709(9)$ Å, $V = 2123.30(14)$ Å³, $Z = 8$, $R_{\text{gt}}(F) = 0.0372$, $wR_{\text{ref}}(F^2) = 0.0907$, $T = 150$ K.

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The asymmetric unit of the title crystal structure is shown in the figure. Tables 1 and 2 contain details of the measurement method and a list of the atoms including atomic coordinates and displacement parameters.

Table 1: Data collection and handling.

Crystal:	Colourless blocks
	Size $0.16 \times 0.11 \times 0.05$ mm
Wavelength:	Cu K_{α} radiation (1.54184 Å)
μ :	7.4 cm^{-1}
Diffractometer, scan mode:	SuperNova, ω
$2\theta_{\text{max}}$, completeness:	147.4° , >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	5568, 3536, 0.032
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 3130
$N(\text{param})_{\text{refined}}$:	279
Programs:	SHELX [4], CrysAlis [5], WinGX [6]

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

Atom	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.8177(3)	−0.0167(2)	0.00679(9)	0.0180(5)
C2	0.8535(4)	−0.0951(2)	0.05516(10)	0.0182(5)
C3	0.9099(3)	−0.2187(2)	−0.01211(10)	0.0175(5)
C4	0.8486(4)	−0.1108(2)	−0.03438(9)	0.0181(5)
C5	0.9294(4)	−0.3168(2)	−0.08722(10)	0.0245(6)
H5	0.9587	−0.3875	−0.1062	0.029*
C6	0.8645(4)	−0.2166(3)	−0.11299(10)	0.0259(6)
H6	0.8480	−0.2199	−0.1487	0.031*
C7	0.8232(4)	−0.1100(2)	−0.08633(10)	0.0229(5)
H7	0.7791	−0.0397	−0.1033	0.027*
C8	0.9555(4)	0.0909(2)	0.00494(10)	0.0192(5)
C9	1.1511(4)	0.0422(2)	0.00972(11)	0.0243(5)
H9A	1.2378	0.1095	0.0059	0.036*
H9B	1.1732	−0.0185	−0.0168	0.036*
H9C	1.1672	0.0047	0.0431	0.036*
C10	0.9321(4)	0.1586(2)	−0.04574(10)	0.0234(6)
H10A	0.9975	0.2362	−0.0443	0.035*

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***Corresponding author: Gamal A. El-Hiti**, Cornea Research Chair, Department of Optometry, College of Applied Medical Sciences, King Saud University, P. O. Box 10219, Riyadh 11433, Saudi Arabia, e-mail: gelhiti@ksu.edu.sa

Keith Smith, Amany S. Hegazy and Benson M. Kariuki: School of Chemistry, Cardiff University, Main Building, Park Place, Cardiff CF10 3AT, UK

Ali M. Masmali: Cornea Research Chair, Department of Optometry, College of Applied Medical Sciences, King Saud University, P. O. Box 10219, Riyadh 11433, Saudi Arabia

Table 2 (continued)

Atom	x	y	z	U _{iso} */U _{eq}
H10B	0.8021	0.1737	−0.0519	0.035*
H10C	0.9818	0.1088	−0.0733	0.035*
C11	0.9177(4)	0.1794(2)	0.04858(11)	0.0246(6)
H11A	0.9399	0.1384	0.0810	0.037*
H11B	0.7903	0.2064	0.0470	0.037*
H11C	0.9988	0.2500	0.0456	0.037*
C12	0.3424(4)	0.1409(2)	0.25738(10)	0.0229(5)
C13	0.3155(4)	0.2718(3)	0.27937(10)	0.0237(6)
C14	0.1976(4)	0.1413(2)	0.33760(9)	0.0196(5)
C15	0.2753(4)	0.0648(2)	0.30120(9)	0.0189(5)
C16	0.1191(4)	−0.0143(2)	0.38892(10)	0.0236(6)
H16	0.0617	−0.0437	0.4188	0.028*
C17	0.1976(4)	−0.0976(2)	0.35654(10)	0.0250(6)
H17	0.1963	−0.1817	0.3649	0.030*
C18	0.2791(4)	−0.0585(2)	0.31133(10)	0.0238(6)
H18	0.3346	−0.1144	0.2886	0.029*
C19	0.2249(5)	0.1254(3)	0.20802(10)	0.0285(6)
C20	0.0259(5)	0.1585(3)	0.21818(12)	0.0394(8)
H20A	0.0169	0.2456	0.2258	0.059*
H20B	−0.0192	0.1115	0.2471	0.059*
H20C	−0.0477	0.1397	0.1881	0.059*
C21	0.2321(5)	−0.0081(3)	0.19097(11)	0.0383(8)
H21A	0.1717	−0.0593	0.2164	0.057*
H21B	0.3597	−0.0335	0.1873	0.057*
H21C	0.1694	−0.0168	0.1583	0.057*
C22	0.3015(7)	0.2052(4)	0.16542(12)	0.0508(10)
H22A	0.2264	0.1954	0.1349	0.076*
H22B	0.4274	0.1806	0.1580	0.076*
H22C	0.2998	0.2906	0.1761	0.076*
N1	0.9177(3)	−0.20598(19)	0.04049(8)	0.0193(4)
H1	0.9585	−0.2621	0.0614	0.023*
N2	0.9539(3)	−0.32013(19)	−0.03619(9)	0.0211(5)
N3	0.2166(3)	0.26218(19)	0.32306(8)	0.0227(5)
H3	0.1707	0.3243	0.3398	0.027*
N4	0.1194(3)	0.10754(19)	0.38051(8)	0.0211(5)
O1	0.6386(2)	0.03186(15)	0.00681(7)	0.0201(4)
H1A	0.5639	−0.0229	0.0147	0.030*
O2	0.8290(3)	−0.06328(16)	0.09886(6)	0.0241(4)
O3	0.5333(3)	0.12938(19)	0.24819(8)	0.0293(4)
H3A	0.5574	0.0573	0.2400	0.044*
O4	0.3692(3)	0.36629(17)	0.26086(8)	0.0335(5)

Source of material

3-*tert*-Butyl-7-azadioxindole was prepared from lithiation of 2-pivaloylaminopyridine with 2.1 mole equivalents of *n*-BuLi for 15 min at −78 °C and 2 h at 0 °C in anhydrous tetrahydrofuran followed by reaction with carbon monoxide for 2 h [1]. Crystallization of the crude product using a mixture of diethyl ether and ethyl acetate (in 1:2 ratio by volume) gave the title compound as colourless crystals in 62% yield [1].

Experimental details

H atoms were placed in calculated positions and refined using a riding model, with $U_{\text{iso}}(\text{H})$ set to $1.2U_{\text{eq}}(\text{C})$ and C—H and N—H distances of 0.93 and 0.86 Å, respectively. The exceptions were the methyl (C—H = 0.96 Å) and hydroxyl (O—H = 0.86 Å) groups which were allowed to rotate around the C—C bond (HFIX 137 in SHELX[4]) and C—O bond (HFIX 147), with $U_{\text{iso}}(\text{H})$ set to $1.5U_{\text{eq}}(\text{C/O})$.

Discussion

Azaindoles represent an important class of pharmacologically active compounds [2, 3]. The asymmetric unit of the title structure comprises two independent molecules. The two molecules are linked by N—H···N (angle = 144.5°, N···N distance = 2.936(3) Å) and N—H···O (angle = 150.5°, C···O distance = 2.835(3) Å) hydrogen bonds forming rings which can be classified by the R₂²(8) graph set. Additional O—H···O (angle = 165.7°, O···O distance = 2.989(3) Å) and O—H···N (angle = 163.2°, O···N distance = 2.802(3) Å) hydrogen bonds lead to the formation of a three-dimensional network.

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References

- Smith, K.; El-Hiti, G. A.; Pritchard, G. J.; Hamilton, A.: Carbonylation of various organolithium reagents. A novel approach to heterocycles *via* intramolecular trapping of aromatic acyllithiums. *J. Chem. Soc., Perkin Trans. 1* (1999) 2299–2303.
- Manchester, J. I.; Dussault, D. D.; Rose, J. A.; Ann Boriack-Sjodin, P.; Uria-Nickelsen, M.; Ioannidis, G.; Bist, S.; Fleming, P.; Hull, K. G.: Discovery of a novel azaindole class of antibacterial agents targeting the ATPase domains of DNA gyrase and Topoisomerase IV. *Bioorg. Med. Chem. Lett.* **22** (2012) 5150–5156.
- Popowycz, F.; Routier, S.; Joseph, B.; Mérou, J.-Y.: Synthesis and reactivity of 7-azaindole (1*H*-pyrrolo[2,3-*b*]pyridine). *Tetrahedron* **63** (2007) 1031–1064.
- Sheldrick, G. M.: A short history of SHELX. *Acta Crystallogr.* **A64** (2008) 112–122.
- Agilent. CrysAlis PRO. Agilent technologies, Yarnton, England, 2014.
- Farrugia, L. J.: WinGX and ORTEP for Windows: an update. *J. Appl. Crystallogr.* **45** (2012) 849–854.