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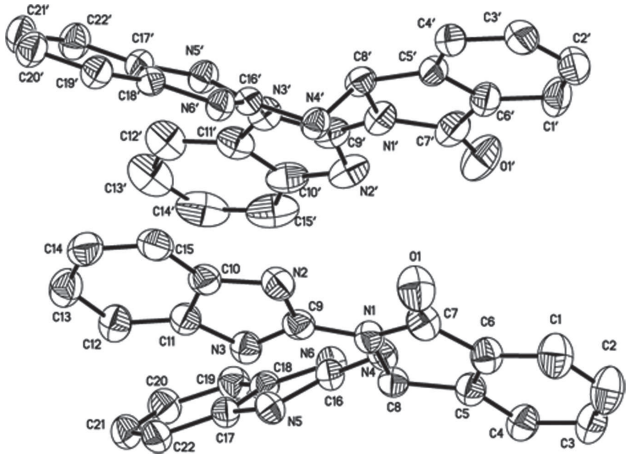
Crystal structure of 3-((1*H*-benzo[*d*]imidazol-2-yl) amino)-2-(1*H*-benzo[*d*]imidazol-2-yl)isoindolin-1-one, $C_{22}H_{16}N_6O$



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Abstract

$C_{22}H_{16}N_6O$, monoclinic, $P2_1/c$ (no. 14), $a = 13.2081(5)$ Å, $b = 23.3620(11)$ Å, $c = 12.2769(5)$ Å, $\beta = 102.345(4)°$, $V = 3700.7(3)$ Å^3, $Z = 8$, $R_{gt}(F) = 0.0648$, $wR_{ref}(F^2) = 0.1730$, $T = 293(2)$ K.

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The asymmetric unit of the title crystal structure containing two independent molecules is shown in the figure (hydrogen atoms are omitted for clarity). Tables 1 and 2 contain details on crystal structure and measurement conditions and a list of the atoms including atomic coordinates and displacement parameters.

Source of material

A Mixture of 2-aminobenzimidazole (10 mmol, 1.3315 g) and 2-carboxybenzaldehyde (10 mmol, 1.4912 g) in 25 mL ethanol was refluxed for 3 h. The resulting solution was cooled to room temperature and the light yellow precipitate was isolated. The crude product was recrystallized in a methanol-dimethylsulfoxide mixture and afforded colorless crystals suitable for crystal structure analysis. **IR** (cm^{-1}, KBr): 3368 (*m*), 2843 (br), 1716 (*s*), 1635(*s*), 1586 (*s*), 1550 (*s*), 1485 (*m*), 1450 (*s*), 1416 (*m*), 1362 (*s*), 1319 (*m*), 1305 (*m*), 1233 (w), 1209 (*m*), 1139 (*m*), 1108 (w), 1049 (*m*), 1028 (*m*), 1008 (w), 992 (w), 952 (w), 929 (w), 916 (w), 871 (w), 797 (*m*), 763 (*m*), 745 (*s*), 715 (w), 705 (*m*), 684 (*s*), 603 (*s*), 591(*s*), 583 (*s*), 560 (w), 537 (w), 481 (w), 441 (w), 416 (w).

Table 1: Data collection and handling.

Crystal:	Colorless prism
Size:	0.1 × 0.09 × 0.08 mm
Wavelength:	Cu $K\alpha$ radiation (1.54184Å)
μ:	0.72 mm^{-1}
Diffractometer, scan mode:	Xcalibur, ω-scans
θ_{max}, completeness:	67.1°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	16853, 6602, 0.039
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 3589
$N(param)_{refined}$:	544
Programs:	CrysAlis^{PRO} [1], SHELX [2], OLEX2 [3]

Experimental details

All hydrogen atoms were placed geometrically and refined using a riding model with U_{iso} set to $1.5U_{eq}$ of the parent atom.

Discussion

Isoindolinone derivatives with lactam five-membered heterocyclic skeleton have become a hot topic of current interest due to its high pharmacological and biological activities and potential applications in medicine, materials and organic pigments [4–7]. As a class of indole alkaloids, isoindolinone compounds can be extracted and isolated from plants [8–10]. In view of the structural diversity and important significance of isoindolinone derivatives, the construction of such a motif has attracted great attention over the past decades [11–13]. It is noteworthy that the traditional way of synthesizing the designed compounds would suffer from multistep reactions

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
N1	0.1148(2)	0.54550(11)	0.2065(2)	0.0592(7)
N2	0.1934(2)	0.48418(14)	0.0902(2)	0.0677(8)
H2	0.248(3)	0.5084(16)	0.093(3)	0.113(16)*
N3	0.0355(2)	0.46189(11)	0.1159(2)	0.0598(7)
N4	0.0062(2)	0.52544(11)	0.3452(2)	0.0596(7)
H4	0.0468	0.5281	0.4098	0.072*
N5	−0.1199(2)	0.46600(11)	0.2304(2)	0.0586(7)
H5	−0.080(3)	0.4685(15)	0.172(3)	0.101(13)*
N6	−0.10730(19)	0.46445(11)	0.4175(2)	0.0573(7)
N1′	0.4194(2)	0.48286(11)	0.3265(2)	0.0625(7)
N2′	0.2631(3)	0.45256(15)	0.3781(3)	0.0764(9)
H2′	0.254(3)	0.4847(14)	0.411(3)	0.081(13)*
N3′	0.3425(2)	0.39144(12)	0.2826(2)	0.0646(7)
N4′	0.4679(2)	0.47268(11)	0.1448(2)	0.0568(7)
H4′	0.471(2)	0.5046(12)	0.108(2)	0.063(10)*
N5′	0.4545(2)	0.37164(11)	0.1178(2)	0.0579(7)
H5′	0.425(2)	0.3693(15)	0.177(3)	0.082(12)*
N6′	0.50950(19)	0.42455(10)	−0.0116(2)	0.0553(7)
O1	0.2657(2)	0.58563(12)	0.1736(2)	0.0966(9)
O1′	0.3712(2)	0.54895(11)	0.4465(2)	0.1011(9)
C1	0.2005(3)	0.68776(17)	0.3055(3)	0.0926(13)
H1	0.2624	0.6976	0.2857	0.111*
C2	0.1527(4)	0.72488(17)	0.3658(4)	0.1004(14)
H2A	0.1821	0.7605	0.3864	0.121*
C3	0.0615(3)	0.70973(16)	0.3960(3)	0.0876(12)
H3	0.0309	0.7351	0.4376	0.105*
C4	0.0146(3)	0.65705(14)	0.3651(3)	0.0708(10)
H4A	−0.0466	0.6468	0.3860	0.085*
C5	0.0616(3)	0.62050(14)	0.3027(3)	0.0604(9)
C6	0.1536(3)	0.63508(15)	0.2749(3)	0.0684(10)
C7	0.1888(3)	0.58847(16)	0.2134(3)	0.0726(10)
C8	0.0265(2)	0.56201(13)	0.2593(3)	0.0577(8)
H8	−0.0367	0.5658	0.2011	0.069*
C9	0.1121(3)	0.49821(15)	0.1383(3)	0.0603(9)
C10	0.1661(3)	0.43344(15)	0.0332(3)	0.0645(9)
C11	0.0683(3)	0.41969(14)	0.0505(2)	0.0596(9)
C12	0.0196(3)	0.36940(15)	0.0080(3)	0.0714(10)
H12	−0.0458	0.3600	0.0191	0.086*
C13	0.0709(3)	0.33397(16)	−0.0510(3)	0.0804(11)
H13	0.0402	0.2997	−0.0791	0.096*
C14	0.1685(3)	0.34842(17)	−0.0696(3)	0.0831(12)
H14	0.2011	0.3237	−0.1106	0.100*
C15	0.2178(3)	0.39868(17)	−0.0285(3)	0.0769(11)
H15	0.2823	0.4086	−0.0417	0.092*
C16	−0.0725(2)	0.48660(13)	0.3331(3)	0.0549(8)
C17	−0.1854(2)	0.42250(13)	0.2513(3)	0.0568(8)
C18	−0.1784(2)	0.42272(13)	0.3669(3)	0.0553(8)
C19	−0.2358(2)	0.38377(14)	0.4137(3)	0.0667(9)
H19	−0.2335	0.3839	0.4899	0.080*
C20	−0.2967(3)	0.34473(15)	0.3440(3)	0.0729(10)
H20	−0.3349	0.3179	0.3744	0.087*
C21	−0.3020(3)	0.34466(15)	0.2295(3)	0.0732(10)
H21	−0.3429	0.3176	0.1850	0.088*
C22	−0.2475(3)	0.38419(15)	0.1809(3)	0.0687(10)
H22	−0.2524	0.3850	0.1042	0.082*
C1′	0.5709(4)	0.61141(15)	0.4154(3)	0.0849(12)
H1′	0.5414	0.6335	0.4635	0.102*
C2′	0.6610(4)	0.62809(18)	0.3852(3)	0.0923(13)
H2′A	0.6927	0.6623	0.4123	0.111*
C3′	0.7048(3)	0.59438(16)	0.3149(3)	0.0856(12)
H3′	0.7662	0.6061	0.2962	0.103*
C4′	0.6589(3)	0.54342(15)	0.2715(3)	0.0722(10)
H4′A	0.6883	0.5213	0.2234	0.087*
C5′	0.5693(3)	0.52668(13)	0.3015(3)	0.0599(9)
C6′	0.5255(3)	0.56030(14)	0.3714(3)	0.0655(9)
C7′	0.4312(3)	0.53296(15)	0.3886(3)	0.0734(10)
C8′	0.5037(2)	0.47405(13)	0.2649(3)	0.0572(8)
H8′	0.5428	0.4392	0.2909	0.069*
C9′	0.3445(3)	0.44209(16)	0.3286(3)	0.0644(9)
C10′	0.2023(3)	0.40383(18)	0.3621(3)	0.0757(11)
C11′	0.2509(3)	0.36669(17)	0.3015(3)	0.0683(10)
C12′	0.2088(3)	0.31345(18)	0.2679(3)	0.0876(12)
H12′	0.2417	0.2882	0.2283	0.105*
C13′	0.1156(4)	0.2997(2)	0.2959(3)	0.1013(15)
H13′	0.0842	0.2648	0.2729	0.122*
C14′	0.0682(3)	0.3367(2)	0.3574(4)	0.1057(17)
H14′	0.0060	0.3259	0.3753	0.127*
C15′	0.1106(3)	0.3893(2)	0.3931(4)	0.1007(15)
H15′	0.0793	0.4138	0.4357	0.121*
C16′	0.4794(2)	0.42445(13)	0.0847(3)	0.0520(8)
C17′	0.4671(2)	0.33389(13)	0.0353(3)	0.0548(8)
C18′	0.5025(2)	0.36689(13)	−0.0434(3)	0.0530(8)
C19′	0.5266(2)	0.34096(14)	−0.1366(3)	0.0631(9)
H19′	0.5530	0.3621	−0.1884	0.076*
C20′	0.5102(3)	0.28292(15)	−0.1497(3)	0.0727(10)
H20′	0.5246	0.2648	−0.2121	0.087*
C21′	0.4726(3)	0.25102(14)	−0.0717(3)	0.0751(10)
H21′	0.4616	0.2120	−0.0835	0.090*
C22′	0.4510(3)	0.27567(14)	0.0230(3)	0.0698(10)
H22′	0.4267	0.2541	0.0758	0.084*

and high cost. Thus, it is highly desirable to explore more efficient and convenient synthetic methods of isoindolinone derivatives. Herein we report the facile synthesis and crystal structure of a new isoindolinone-containing compound.

Single crystal structure analysis reveals that the title compound crystallizes in the monoclinic $P2_1/c$ space group with two crystallographically independent molecules in the asymmetric unit (*cf*. the figure). The molecule consists of a isoindolin-1-one group, a benzimidazole group and a 2-aminobenzimidazole group. The latter two groups lie to the same side of the plane through the isoindolin-1-one moiety and locate in 2- and 3-position of the isoindolin-1-one moiety, respectively. The isoindolin-1-one moiety is effectively planar with the maximum deviation from the least-squares plane through the atoms being 0.0262 Å for the C8 atom. The dihedral angles between the plane of isoindolin-1-one moiety and the planes through the C10—C15 and C17—C22

rings are 6.3° and 53.0°, respectively. The C8—N4 distance of 1.449 Å is consistent with a C—N single bond, as in 2-benzyl-3-(benzylamino)-6-nitroisoindolin-1-one (C—N: 1.434 Å) and 3-(Thiazol-2-ylamino)isobenzofuran-1(3*H*)-one (C—N: 1.418 Å) [14, 15].

The imidazole nitrogen atoms (N6 and N6′) are involved in the intermolecular hydrogen bond interactions with N4 and N4′ atoms [N4···N6: 2.107 Å, N4′···N6′: 2.083 Å], forming a dimer. Face to face π···π interactions are observed between adjacent dimers with the centroid-centroid distance between the other two imidazole rings of 3.50 Å. Thus a supramolecular chain structure which lies in the *ac* plane is constructed. Van der Waals forces are responsible to construct a three-dimensional architecture. All these interactions stabilize the solid state of the title compound.

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