Crystal structure of 2-((1*H*-benzo[d]imidazol-1-yl)-methyl)isoindoline-1,3-dione, $C_{16}H_{11}N_3O_2$

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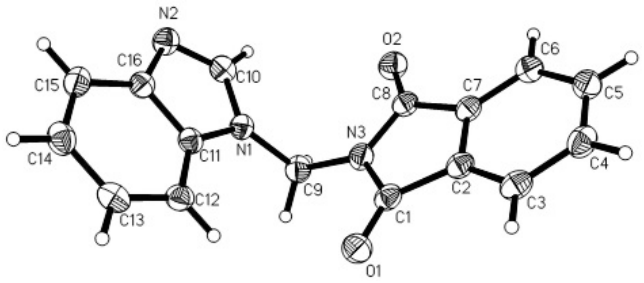

Abstract

$C_{16}H_{11}N_3O_2$, orthorhombic, $Pna2_1$ (no. 33), a = 10.568(1) Å, b = 23.030(2) Å, c = 5.1985(5) Å, V = 1265.2 Å^3, Z = 4, $R_{gt}(F)$ = 0.030, $wR_{ref}(F^2)$ = 0.074, T = 293 K.

Source of material

All commercially available reagents were used as supplied. 2-(bromomethyl)isoindoline-1,3-dione (4.8 g, 0.02 mol) and benzimidazole (2.4 g, 0.02 mol) were dissolved in 15ml chloroform. The solution was cooled to 283 K. Then, triethylamine (2.04 g, 0.02 mol) was added dropwise via cannula into the well-stirred solution at 283 K. The reaction mixture was stirred at 283 K for 7 h. Then the solution was continued to stir at room temperature about 16 h. 20 ml water was added into the solution, the organic phase was seperated and dryed with anhydrous potassium carbonate,The colorless organic phase was evaporated. The title compound is afforded in 86 % yield. colorless crystals of suitable for X-ray determination were obtained from anhydrous ethanol at room temperature after two days. Chemical analysis — found: C, 68.98 %; H, 4.05 %; N, 15.16 %; calculated for $C_{16}H_{11}N_3O_2$: C, 69.29 %; H, 4.00 %; N, 15.16 %.

Experimental details

All hydrogen atoms were positioned geometrically and allowed to ride on their parent atoms with $d(Csp^2—H)$ = 0.93 Å or $d(Csp^3—H)$ = 0.97 Å and $U_{iso}(H) = 1.2\,U_{eq}(C)$. The absolute configuration could not be confirmed from X-ray data. The Flack parameter x = –10(10) has no evidence, because the part of anomalous dispersion is too small (Mo-source and C, H, N, O elements).

Discussion

The efficency of substituted benzimidazoles in the application of chemical and biology are well known [1]. In addition benzimidazole and its derivatives are unidentate ligands and form complexes with metal ions through their tertiary N atom [2].

In the title crystal structure , the dihedral angles formed by the ring II (N3,C1,C2,C7,C8) and the ring III (C2,C3,C4,C5,C6,C7) was 0.5(3)°. The dihedral angles formed by the ring II and ring IV (C11,C12,C13,C14,C15,C16) with the ring I (N1,N2,C6 and C7) are 61.8(8)° and 1.5(8)°, respectively. The C=O bond lengths (1.206(7) Å, 1.204(0) Å) and the C—N bond lengths (1.451(2) Å, 1.448(2) Å) are in agreement with those observed before [3]. There is a C–H···O intermolecular interaction. The crystal structure is also stabilized by intermolecular C–H···π interaction.

Table 1. Data collection and handling.

Crystal:	colorless block, size 0.10 × 0.15 × 0.20 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	0.99 cm^{-1}
Diffractometer, scan mode:	Bruker SMART CCD, φ/ω
$2\theta_{max}$:	56.52°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	7857, 1698
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 1464
$N(param)_{refined}$:	191
Programs:	WinGX [4], NRCVAX [5], SHELXS-97 [6], SHELXL-97 [7], SHELXTL [8]

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

Atom	Site	x	y	z	U_{iso}
H(3A)	4*a*	1.0690	0.8959	0.6474	0.058
H(4A)	4*a*	0.9473	0.9799	0.6775	0.063
H(5A)	4*a*	0.7845	0.9975	0.3970	0.063
H(6A)	4*a*	0.7331	0.9303	0.0792	0.056
H(9A)	4*a*	0.9682	0.7383	–0.2500	0.056
H(9B)	4*a*	1.0698	0.7212	–0.0437	0.056
H(10A)	4*a*	0.7554	0.6859	–0.2328	0.054
H(12A)	4*a*	1.0916	0.6674	0.3901	0.054
H(13A)	4*a*	1.0871	0.5891	0.6712	0.061
H(14A)	4*a*	0.9249	0.5221	0.6573	0.062
H(15A)	4*a*	0.7600	0.5320	0.3706	0.058

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}
O(1)	4*a*	1.1143(1)	0.78082(6)	0.3782(3)	0.0529(8)	0.0527(8)	0.066(1)	0.0003(6)	–0.0139(8)	0.0131(8)
O(2)	4*a*	0.7843(1)	0.81468(6)	–0.1680(3)	0.0545(8)	0.0572(8)	0.0511(8)	–0.0042(6)	–0.0141(8)	–0.0028(7)

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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(1)	4*a*	0.9024(1)	0.68437(6)	0.0170(3)	0.0434(8)	0.0380(7)	0.0386(8)	–0.0008(6)	0.0014(8)	0.0002(7)
N(2)	4*a*	0.7435(1)	0.61977(7)	0.0072(4)	0.0491(9)	0.0466(9)	0.0439(9)	–0.0029(7)	–0.0039(8)	–0.0048(8)
N(3)	4*a*	0.9574(1)	0.78566(6)	0.0693(3)	0.0466(9)	0.0389(7)	0.0458(9)	–0.0035(6)	–0.0031(8)	0.0024(8)
C(1)	4*a*	1.0277(2)	0.80639(8)	0.2793(4)	0.0424(9)	0.0438(9)	0.043(1)	–0.0092(8)	–0.0012(9)	0.0100(9)
C(2)	4*a*	0.9706(2)	0.86333(7)	0.3453(4)	0.0406(9)	0.0444(9)	0.038(1)	–0.0084(7)	0.0020(9)	0.0064(8)
C(3)	4*a*	1.0021(2)	0.90263(8)	0.5350(4)	0.047(1)	0.058(1)	0.040(1)	–0.0114(9)	–0.004(1)	0.003(1)
C(4)	4*a*	0.9292(2)	0.95274(8)	0.5505(5)	0.057(1)	0.052(1)	0.048(1)	–0.0121(9)	0.005(1)	–0.010(1)
C(5)	4*a*	0.8306(2)	0.96330(9)	0.3826(4)	0.051(1)	0.050(1)	0.057(1)	–0.0019(9)	0.007(1)	–0.004(1)
C(6)	4*a*	0.7995(2)	0.92346(8)	0.1927(4)	0.045(1)	0.048(1)	0.047(1)	–0.0023(8)	–0.0011(9)	0.002(1)
C(7)	4*a*	0.8705(2)	0.87337(7)	0.1786(4)	0.041(1)	0.0415(9)	0.038(1)	–0.0076(7)	0.0028(9)	0.0041(8)
C(8)	4*a*	0.8597(2)	0.82347(8)	0.0019(4)	0.0406(9)	0.0439(9)	0.039(1)	–0.0059(7)	0.0017(9)	0.0038(9)
C(9)	4*a*	0.9818(2)	0.73192(8)	–0.0676(4)	0.051(1)	0.044(1)	0.046(1)	–0.0014(8)	0.0098(9)	0.0002(9)
C(10)	4*a*	0.7924(2)	0.66649(8)	–0.0952(4)	0.049(1)	0.048(1)	0.039(1)	0.0042(8)	–0.0041(9)	–0.0054(9)
C(11)	4*a*	0.9278(2)	0.64517(7)	0.2120(4)	0.0408(9)	0.0371(8)	0.0364(9)	0.0042(7)	0.0046(9)	–0.0044(8)
C(12)	4*a*	1.0261(2)	0.64043(8)	0.3855(4)	0.0395(9)	0.047(1)	0.049(1)	0.0021(8)	0.0013(9)	–0.0043(9)
C(13)	4*a*	1.0225(2)	0.59376(9)	0.5515(5)	0.048(1)	0.060(1)	0.046(1)	0.0119(9)	–0.003(1)	–0.000(1)
C(14)	4*a*	0.9240(2)	0.55333(8)	0.5439(4)	0.062(1)	0.047(1)	0.048(1)	0.0079(9)	0.008(1)	0.006(1)
C(15)	4*a*	0.8259(2)	0.55872(8)	0.3727(4)	0.054(1)	0.0417(9)	0.050(1)	–0.0038(8)	0.007(1)	–0.0014(9)

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