Crystal structure of 2,4-dibromobenzene[1′,6′:6,7]-1-oxa-4-aza-cycloheptane[4,5-*a*]-benzimidazol, $C_{15}H_{10}Br_2N_2O$

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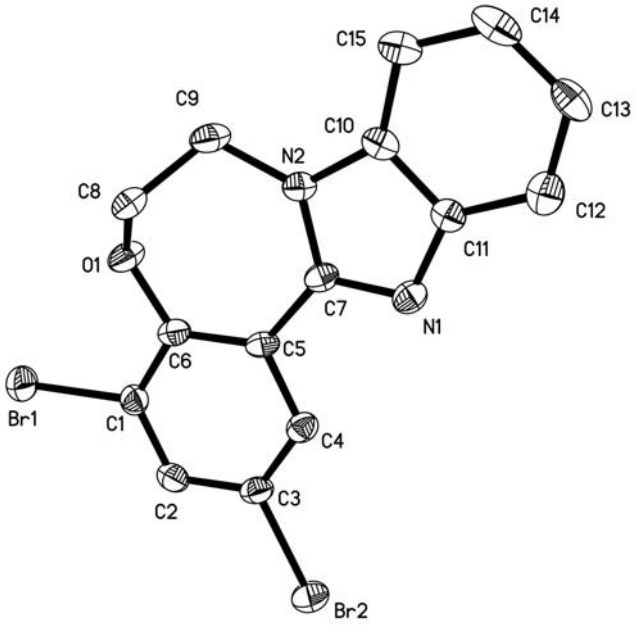

Abstract

$C_{15}H_{10}Br_2N_2O$, triclinic, $P\bar{1}$ (no. 2), a = 7.280(1) Å, b = 8.139(1) Å, c = 12.55(1) Å, α = 107.180(9)°, β = 98.07(1)°, γ = 101.9(1)°, V = 678.6 Å^3, Z = 2, $R_{gt}(F)$ = 0.036, $wR_{ref}(F^2)$ = 0.056, T = 293 K.

Source of material

All chemicals were purchased commercially and used without purification. 2-(2-Bromoethoxy)-3,5-dibromobenzaldehyde was prepared according to [1]. The title compound was synthesized by the following procedures. A mixture of 2-(2-bromoethoxy)-3,5-dibromobenzaldehyde (0.077 g, 0.02 mol), benzene-1,2-diamine (0.0108 g, 0.01 mol) and ethanol 10 mL was heated at 140 °C for 72 hours. The mixture was allowed to cool to room temperature. Colourless prism-shaped crystals were collected (yield 65 %).

Experimental details

All hydrogen atoms on carbons were generated geometrically with d(C—H) = 0.93 - 0.97 Å and U_{iso}(H) = 1.2 U_{eq}(C).

Discussion

Coordination polymers have attracted intense attention because of their unique applications for heterogeneous catalysis, molecular adsorptions, and ion exchange [2,3].

In the title crystal structure, the characteristic angles and distances are ∠O1–C8–C9 = 112.45°, d(O1—C3) = 1.351 Å and d(N2—C9) = 1.460 Å. Other bond distances and bond angles are all in normal ranges. The molecular structure is further extend into chains through π–π stacking interations with a centroid-centroid distance of 3.698 Å.

Table 1. Data collection and handling.

Crystal:	colourless prism, size 0.13 × 0.15 × 0.2 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	59.68 cm^{-1}
Diffractometer, scan mode:	Oxford Diffraction Gemini R Ultra, ω
$2\theta_{max}$:	58.54°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	4908, 3072
$N(param)_{refined}$:	181
Programs:	SHELXS-97, SHELXL-97, SHELXTL [4]

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

Atom	Site	x	y	z	U_{iso}
H(1)	2*i*	0.6901	0.1485	0.5753	0.048
H(5)	2*i*	0.8230	0.6102	0.8353	0.045
H(8A)	2*i*	0.8594	0.1986	1.0193	0.049
H(8B)	2*i*	0.7132	0.0441	1.0379	0.049
H(9A)	2*i*	0.5226	0.2489	1.0930	0.056
H(9B)	2*i*	0.7163	0.2885	1.1801	0.056
H(12)	2*i*	0.9219	1.0286	1.2045	0.058
H(13)	2*i*	0.9060	1.0820	1.3909	0.054
H(14)	2*i*	0.7884	0.8436	1.4521	0.068
H(15)	2*i*	0.6992	0.5513	1.3285	0.056

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}
C(1)	2*i*	0.7007(5)	0.2105(5)	0.6521(3)	0.042(2)	0.046(2)	0.035(3)	0.018(2)	0.012(2)	0.012(2)
C(2)	2*i*	0.6533(5)	0.1239(4)	0.7273(4)	0.035(2)	0.035(2)	0.043(3)	0.011(2)	0.011(2)	0.012(2)
C(3)	2*i*	0.6686(4)	0.2118(5)	0.8431(3)	0.031(2)	0.046(2)	0.037(3)	0.013(2)	0.012(2)	0.023(2)
C(4)	2*i*	0.7346(4)	0.3989(4)	0.8872(3)	0.024(2)	0.041(2)	0.029(2)	0.005(2)	0.007(2)	0.017(2)
C(5)	2*i*	0.7807(4)	0.4865(4)	0.8094(3)	0.030(2)	0.039(2)	0.046(3)	0.008(2)	0.009(2)	0.016(2)

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Table 3. Continued.

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(6)	2*i*	0.7650(5)	0.3944(5)	0.6963(3)	0.038(2)	0.048(2)	0.042(3)	0.014(2)	0.014(2)	0.025(2)
C(7)	2*i*	0.7556(4)	0.5143(5)	1.0048(3)	0.023(2)	0.040(2)	0.036(3)	0.003(2)	0.006(2)	0.017(2)
C(8)	2*i*	0.7249(5)	0.1515(4)	1.0177(3)	0.046(2)	0.040(2)	0.041(3)	0.006(2)	0.008(2)	0.023(2)
C(9)	2*i*	0.6614(5)	0.2853(5)	1.1043(3)	0.049(2)	0.056(2)	0.034(3)	0.002(2)	0.010(2)	0.021(2)
C(10)	2*i*	0.7584(4)	0.6179(5)	1.1905(3)	0.024(2)	0.047(2)	0.033(3)	0.009(2)	0.004(2)	0.010(2)
C(11)	2*i*	0.8203(5)	0.7556(5)	1.1498(3)	0.033(2)	0.045(2)	0.035(3)	0.013(2)	0.008(2)	0.014(2)
C(12)	2*i*	0.8775(5)	0.9339(5)	1.2294(4)	0.040(2)	0.045(2)	0.058(3)	0.013(2)	0.003(2)	0.019(2)
C(13)	2*i*	0.8677(5)	0.9660(5)	1.3395(4)	0.035(2)	0.052(3)	0.035(3)	0.016(2)	–0.002(2)	–0.003(2)
C(14)	2*i*	0.7980(5)	0.8207(6)	1.3762(4)	0.047(2)	0.084(3)	0.030(3)	0.025(2)	0.008(2)	0.001(3)
C(15)	2*i*	0.7436(5)	0.6457(5)	1.3032(4)	0.040(2)	0.064(3)	0.037(3)	0.014(2)	0.012(2)	0.018(2)
N(1)	2*i*	0.8179(3)	0.6913(4)	1.0352(3)	0.033(2)	0.041(2)	0.041(2)	0.004(2)	0.005(2)	0.016(2)
N(2)	2*i*	0.7190(4)	0.4638(4)	1.0969(3)	0.037(2)	0.038(2)	0.033(2)	0.008(2)	0.009(2)	0.013(2)
O(1)	2*i*	0.6158(3)	0.1075(3)	0.9056(2)	0.053(2)	0.042(1)	0.033(2)	–0.004(1)	0.003(1)	0.017(1)
Br(1)	2*i*	0.56896(6)	–0.12868(5)	0.66912(4)	0.0798(3)	0.0391(2)	0.0540(4)	0.0129(2)	0.0210(3)	0.0121(2)
Br(2)	2*i*	0.83474(6)	0.51746(5)	0.59530(4)	0.0942(4)	0.0597(3)	0.0517(4)	0.0235(3)	0.0339(3)	0.0323(3)

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References

1. Van der Made, A. W.; Van der Made, R. H.: A Convenient Procedure for Bromomethylation of Aromatic Compounds. Selective Mono-, Bis-, or Trisbromomethylation. J. Org. Chem. **58** (1993) 1262-1263.
2. Férey, G.; Mellot-Draznieks, C.; Serre, C.; Millange, F.: Crystallized Frameworks with Giant Pores: Are There Limits to the Possible? Acc. Chem. Res. **38** (2005) 217.
3. Bradshaw, D.; Claridge, J. B.; Cussen, E. J.; Prior, T. J.; Rosseinsky, M. J.: Design, Chirality, and Flexibility in Nanoporous Molecule-Based Materials. Acc. Chem. Res. **38** (2005) 273.
4. Sheldrick, G. M.: A short history of SHELX. Acta Crystallogr. **A64** (2008) 112-122.