Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

1,3-Bis[(3-chloropyrazin-2-yl)oxy]-benzene

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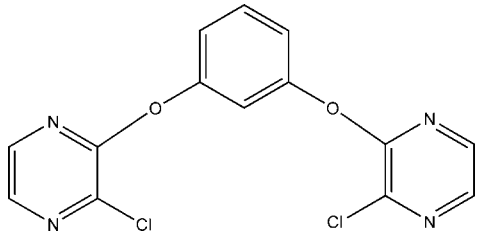
Received 6 April 2013; accepted 25 April 2013

Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.002 Å; R factor = 0.036; wR factor = 0.113; data-to-parameter ratio = 17.6.

The asymmetric unit of the title compound, $C_{14}H_8Cl_2N_4O_2$, contains one half-molecule, the complete molecule being generated by the operation of a twofold rotation axis. The Cl atom deviates significantly from the plane of the pyrazine ring [0.0215 (4) Å]. The central benzene ring makes a dihedral angle of 72.82 (7)° with the plane of the pyrazine ring.

Related literature

For applications of the pyrazine ring system in drug development, see: Du *et al.* (2009); Dubinina *et al.* (2006); Ellsworth *et al.* (2007); Mukaiyama *et al.* (2007). For background to the fluorescence properties of compounds related to the title compound, see: Kawai *et al.* (2001); Abdullah (2005). For a related structure, see: Nasir *et al.* (2010).



Experimental

Crystal data

$C_{14}H_8Cl_2N_4O_2$
M_r = 335.14
Monoclinic, $C2/c$
a = 9.9618 (3) Å
b = 10.2196 (4) Å
c = 14.6010 (6) Å
β = 106.231 (2)°
V = 1427.22 (9) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.47 mm^{-1}
T = 293 K
0.30 × 0.25 × 0.20 mm

Data collection

Bruker SMART APEXII area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2008)
T_{min} = 0.873, T_{max} = 0.912
6736 measured reflections
1781 independent reflections
1552 reflections with $I > 2\sigma(I)$
R_{int} = 0.026

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.113
S = 1.00
1781 reflections
101 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.25 e Å^{-3}
$\Delta\rho_{min}$ = −0.31 e Å^{-3}

Data collection: *APEX2* (Bruker, 2008); cell refinement: *SAINT* (Bruker, 2008); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012) and *Mercury* (Macrae *et al.*, 2008); software used to prepare material for publication: *SHELXL97*and *PLATON* (Spek, 2009).

The authors thank the TBI X-ray facility, CAS in Crystallography and Biophysics, University of Madras, India, for the data collection. TS and DV thank the UGC (SAP–CAS) for the departmental facilties. TS also thanks the DST Inspire program for financial assistance.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: KP2450).

References

Abdullah, Z. (2005). *Intl J. Chem. Sci.* **3** , 9–15.
Bruker (2008). *APEX2*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Du, X. H., Gustin, D. J., Chen, X. Q., Duquette, J., McGee, L. R., Wang, Z. L., Ebsworth, K., Henne, K., Lemon, B., Ma, J., Miao, S. C., Sabalan, E., Sullivan, T. J., Tonn, G., Collins, T. L. & Medina, J. C. (2009). *Bioorg. Med. Chem. Lett.* **19**, 5200–5204.
Dubinina, G. G., Platonov, M. O., Golovach, S. M., Borysko, P. O., Tolmachov, A. O. & Volovenko, Y. M. (2006). *Eur. J. Med. Chem.* **41**, 727–737.
Ellsworth, B. A., Wang, Y., Zhu, Y. H., Pendri, A., Gerritz, S. W., Sun, C. Q., Carlson, K. E., Kang, L. Y., Baska, R. A., Yang, Y. F., Huang, Q., Burford, N. T., Cullen, M. J., Johnghar, S., Behnia, K., Pelleymounter, M. A., Washburn, W. N. & Ewing, W. R. (2007). *Bioorg. Med. Chem. Lett.* **17**, 3978–3982.
Farrugia, L. J. (2012). *J. Appl. Cryst.* **45**, 849–854.
Kawai, M., Lee, M. J., Evans, K. O. & Norlund, T. (2001). *J. Fluoresc.* **11**, 23–32.
Macrae, C. F., Bruno, I. J., Chisholm, J. A., Edgington, P. R., McCabe, P., Pidcock, E., Rodriguez-Monge, L., Taylor, R., van de Streek, J. & Wood, P. A. (2008). *J. Appl. Cryst.* **41**, 466–470.
Mukaiyama, H., Nishimura, T., Kobayashi, S., Ozawa, T., Kamada, N., Komatsu, Y., Kikuchi, S., Oonota, H. & Kusama, H. (2007). *Bioorg. Med. Chem. Lett.* **15**, 868–885.
Nasir, S. B., Abdullah, Z., Fairuz, Z. A., Ng, S. W. & Tiekink, E. R. T. (2010). *Acta Cryst.* E**66**, o2187.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.

supporting information

Acta Cryst. (2013). E**69**, o815 [https://doi.org/10.1107/S160053681301129X]

1,3-Bis[(3-chloropyrazin-2-yl)oxy]benzene

Thothadri Srinivasan, Venkatesan Kalpana, Perumal Rajakumar and Devadasan Velmurugan

S1. Comment

The pyrazine ring system is a useful structural element in medicinal chemistry and has found broad applications in drug development which can be used as antiproliferative agent (Dubinina *et al.*, 2006), potent CXCR3 antagonists (Du *et al.*, 2009), CB1 antagonists (Ellsworth *et al.*, 2007), and c-Src inhibitory (Mukaiyama *et al.*, 2007). On-going structural studies of heterocyclic N-containing derivatives (Nasir *et al.*, 2010) are motivated by an investigation of their fluorescence properties (Kawai *et al.*, 2001; Abdullah, 2005). In view of different applications of this class of compounds, we have undertaken the single crystal structure determination of the title compound.

The title compound C_{14} H_8 Cl_2 N_4 O_2, contains a half of the molecule in an asymmetric unit; the complete molecule is generated by two the fold rotation axis along the direction [0 1 0] with the symmetry code: -x, y, -z+1/2. X-ray analysis confirms the molecular structure and atom connectivity of the compound (Fig. 1). The deviation of the atom Cl1 from the pyrazine ring (C1/N1/C2/C3/N2/C4) is -0.0215 (4) Å.

The central phenyl ring (C5/C6/C7/C8/C5^i/C7^i) forms the dihedral angle of 72.82 (7) ° with the pyrazine ring (C1/N1/C2/C3/N2/C4). The dihedral angle between the pyrazine rings (C1/N1/C2/C3/N2/C4) and (C1^i/N1^i/C2^i/C3^i/N2^i/C4^i) is 68.38 (3) ° (Macrae *et al.*, 2008). The crystal packing is via van der Waals interactions, only.

S2. Experimental

To a stirred solution of Cs_2CO_3/K_2CO_3 (22 mmol) in CH_3CN (50 mL), resorcinol (10 mmol) was added and stirred for 5 min. 2,3-dichloropyrazine (20 mmol) in CH_3CN (100 mL) was added dropwise to the above reaction mixture and allowed for stirring at refluxing condition for 12 h. After the reaction was complete, the reaction mixture was allowed to attain room temperature and then evaporated to dryness. The residue obtained was extracted with CH_2Cl_2 (3 x 100 mL), washed with water (3 x 100 mL), brine and then dried over Na_2SO_4. Evaporation of the organic layer gave a residue, which on purification using column chromatography with hexane/$CHCl_3$ (1:1) as an eluent gave the corresponding compound. Single crystals suitable for X-ray diffraction were obtained by slow evaporation of a solution of the title compound in hexane at room temperature.

S3. Refinement

The hydrogen atoms were placed in calculated positions with C—H = 0.93 Å, refined in the riding model with fixed isotropic displacement parameters:U_{iso}(H) = 1.2U_{eq}(C).

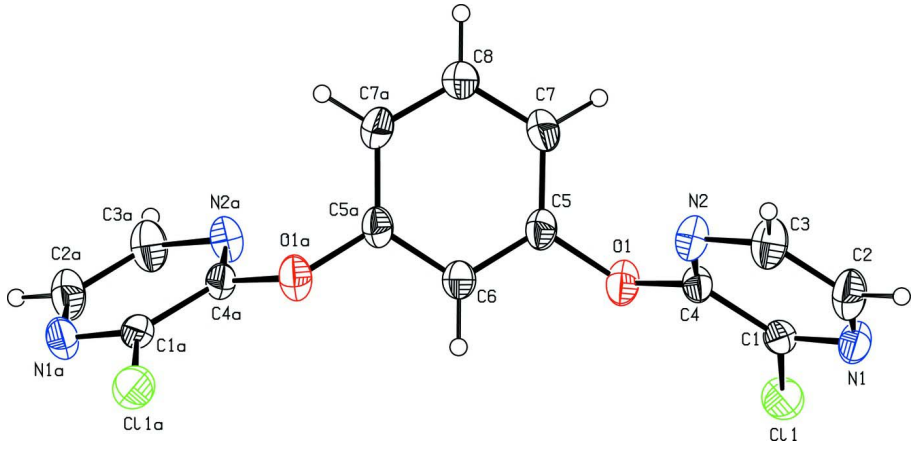


Figure 1

The molecular structure of the title compound, showing displacement ellipsoids drawn at the 30% probability level. H atoms are presented as small spheres of arbitrary radius. The related atoms have the symmetry code: (a) -*x*, *y*, -*z*+1/2.

1,3-Bis[(3-chloropyrazin-2-yl)oxy]benzene

Crystal data

$C_{14}H_8Cl_2N_4O_2$
$M_r = 335.14$
Monoclinic, *C*2/*c*
Hall symbol: -C 2yc
$a = 9.9618$ (3) Å
$b = 10.2196$ (4) Å
$c = 14.6010$ (6) Å
$\beta = 106.231$ (2)°
$V = 1427.22$ (9) Å^3
$Z = 4$
$F(000) = 680$
$D_x = 1.560$ Mg m^{-3}
Mo *Kα* radiation, $\lambda = 0.71073$ Å
Cell parameters from 1781 reflections
$\theta = 2.9$–28.4°
$\mu = 0.47$ mm^{-1}
$T = 293$ K
Block, colourless
0.30 × 0.25 × 0.20 mm

Data collection

Bruker SMART APEXII area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω and φ scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2008)
$T_{min} = 0.873$, $T_{max} = 0.912$
6736 measured reflections
1781 independent reflections
1552 reflections with $I > 2\sigma(I)$
$R_{int} = 0.026$
$\theta_{max} = 28.4°$, $\theta_{min} = 2.9°$
$h = -13 \rightarrow 13$
$k = -13 \rightarrow 12$
$l = -19 \rightarrow 14$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.036$
$wR(F^2) = 0.113$
$S = 1.00$
1781 reflections
101 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0685P)^2 + 0.5533P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.25$ e Å^{-3}
$\Delta\rho_{min} = -0.31$ e Å^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.10770 (15)	0.37514 (13)	−0.02670 (10)	0.0461 (3)
C2	−0.07466 (18)	0.3213 (2)	−0.15100 (11)	0.0644 (4)
H2	−0.1203	0.3246	−0.2159	0.077*
C3	−0.13428 (17)	0.25363 (19)	−0.09216 (11)	0.0618 (4)
H3	−0.2196	0.2121	−0.1178	0.074*
C4	0.04654 (14)	0.30517 (13)	0.03426 (9)	0.0417 (3)
C5	0.05477 (14)	0.23121 (15)	0.18814 (9)	0.0454 (3)
C6	0.0000	0.3013 (2)	0.2500	0.0436 (4)
H6	0.0000	0.3923	0.2500	0.052*
C7	0.05777 (19)	0.09686 (17)	0.18801 (10)	0.0597 (4)
H7	0.0979	0.0516	0.1470	0.072*
C8	0.0000	0.0305 (2)	0.2500	0.0694 (7)
H8	0.0000	−0.0605	0.2500	0.083*
N1	0.04795 (16)	0.38337 (13)	−0.11809 (9)	0.0580 (3)
N2	−0.07257 (13)	0.24545 (13)	0.00221 (8)	0.0515 (3)
O1	0.11655 (11)	0.30189 (12)	0.12853 (7)	0.0537 (3)
Cl1	0.26460 (5)	0.45456 (4)	0.01959 (4)	0.06883 (19)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0581 (7)	0.0383 (6)	0.0511 (8)	0.0030 (5)	0.0308 (6)	−0.0006 (5)
C2	0.0681 (10)	0.0875 (12)	0.0390 (7)	0.0107 (9)	0.0175 (7)	0.0107 (7)
C3	0.0527 (8)	0.0885 (12)	0.0432 (8)	−0.0002 (8)	0.0121 (6)	0.0067 (7)
C4	0.0491 (6)	0.0447 (7)	0.0363 (6)	0.0042 (5)	0.0201 (5)	0.0004 (5)
C5	0.0493 (7)	0.0565 (8)	0.0298 (6)	−0.0035 (5)	0.0104 (5)	0.0000 (5)
C6	0.0461 (9)	0.0492 (10)	0.0340 (8)	0.000	0.0085 (7)	0.000
C7	0.0861 (11)	0.0587 (9)	0.0385 (7)	0.0055 (8)	0.0240 (7)	−0.0053 (6)
C8	0.119 (2)	0.0472 (12)	0.0468 (12)	0.000	0.0308 (13)	0.000
N1	0.0756 (8)	0.0596 (8)	0.0484 (7)	0.0084 (6)	0.0332 (6)	0.0117 (6)
N2	0.0489 (6)	0.0689 (8)	0.0387 (6)	−0.0036 (5)	0.0157 (5)	0.0063 (5)
O1	0.0553 (6)	0.0703 (7)	0.0374 (5)	−0.0130 (5)	0.0162 (4)	−0.0022 (4)
Cl1	0.0778 (3)	0.0608 (3)	0.0815 (4)	−0.02286 (19)	0.0447 (2)	−0.01437 (19)

Geometric parameters (Å, º)

C1—N1	1.303 (2)	C5—C7	1.373 (2)
C1—C4	1.4064 (18)	C5—C6	1.3789 (17)
C1—Cl1	1.7229 (15)	C5—O1	1.3991 (17)
C2—N1	1.340 (2)	C6—C5^{i}	1.3789 (17)
C2—C3	1.362 (2)	C6—H6	0.9300
C2—H2	0.9300	C7—C8	1.379 (2)
C3—N2	1.345 (2)	C7—H7	0.9300
C3—H3	0.9300	C8—C7^{i}	1.379 (2)
C4—N2	1.2996 (18)	C8—H8	0.9300
C4—O1	1.3584 (16)		
N1—C1—C4	121.76 (14)	C6—C5—O1	117.57 (14)
N1—C1—Cl1	118.42 (11)	C5—C6—C5^{i}	117.45 (19)
C4—C1—Cl1	119.81 (11)	C5—C6—H6	121.3
N1—C2—C3	121.90 (14)	C5^{i}—C6—H6	121.3
N1—C2—H2	119.0	C5—C7—C8	118.51 (15)
C3—C2—H2	119.0	C5—C7—H7	120.7
N2—C3—C2	121.51 (15)	C8—C7—H7	120.7
N2—C3—H3	119.2	C7^{i}—C8—C7	121.1 (2)
C2—C3—H3	119.2	C7^{i}—C8—H8	119.4
N2—C4—O1	120.78 (11)	C7—C8—H8	119.4
N2—C4—C1	121.62 (12)	C1—N1—C2	116.50 (13)
O1—C4—C1	117.60 (12)	C4—N2—C3	116.71 (13)
C7—C5—C6	122.18 (14)	C4—O1—C5	116.90 (10)
C7—C5—O1	120.12 (13)		
N1—C2—C3—N2	−0.1 (3)	C4—C1—N1—C2	−0.2 (2)
N1—C1—C4—N2	0.0 (2)	Cl1—C1—N1—C2	−179.30 (12)
Cl1—C1—C4—N2	179.08 (11)	C3—C2—N1—C1	0.3 (3)
N1—C1—C4—O1	179.94 (13)	O1—C4—N2—C3	−179.81 (14)
Cl1—C1—C4—O1	−0.95 (17)	C1—C4—N2—C3	0.2 (2)
C7—C5—C6—C5^{i}	−1.04 (11)	C2—C3—N2—C4	−0.1 (3)
O1—C5—C6—C5^{i}	−176.94 (13)	N2—C4—O1—C5	0.4 (2)
C6—C5—C7—C8	2.0 (2)	C1—C4—O1—C5	−179.61 (12)
O1—C5—C7—C8	177.84 (11)	C7—C5—O1—C4	75.09 (18)
C5—C7—C8—C7^{i}	−0.99 (11)	C6—C5—O1—C4	−108.92 (13)

Symmetry code: (i) −*x*, *y*, −*z*+1/2.