



Received 10 October 2015

Accepted 29 October 2015

Edited by J. Simpson, University of Otago, New Zealand

**Keywords:** arylboronic acid; hydrogen-bonding interactions; halogen-bonding interactions; biphenyl; THF solvate; crystal structure

CCDC reference: 1434205

**Supporting information:** this article has supporting information at journals.iucr.org/e

# Crystal structure of (2',3,6'-trichlorobiphenyl-2-yl)-boronic acid tetrahydrofuran monosolvate

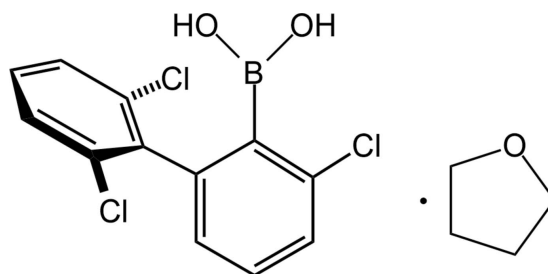
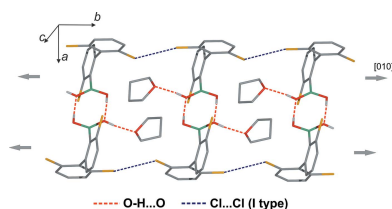
Krzysztof Durka,\* Tomasz Kliś and Janusz Serwatowski

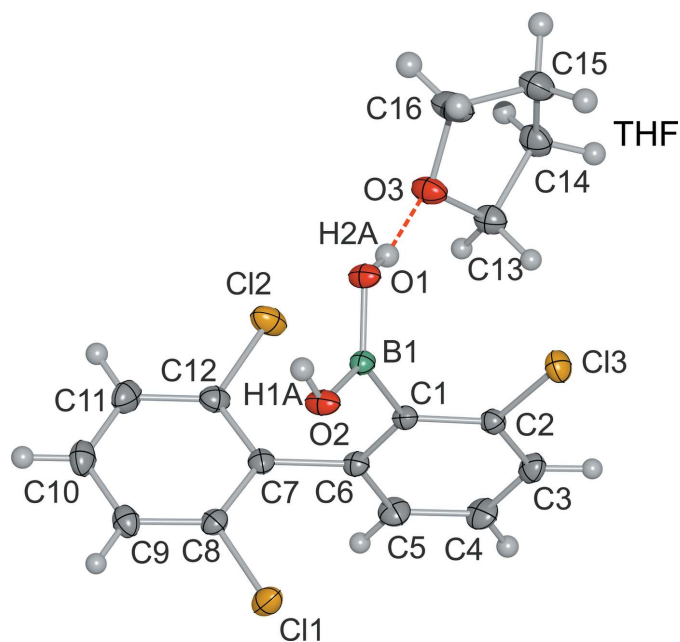
Physical Chemistry Department, Faculty of Chemistry, Warsaw University of Technology, Noakowskiego 3, 00-664 Warsaw, Poland. \*Correspondence e-mail: kdurka@ch.pw.edu.pl

The title compound,  $C_{12}H_8BCl_3O_2 \cdot C_4H_8O$ , crystallizes as a tetrahydrofuran monosolvate. The boronic acid group adopts a *syn-anti* conformation and is significantly twisted along the carbon–boron bond by  $69.2(1)^\circ$ , due to considerable steric hindrance from the 2',6'-dichlorophenyl group that is located *ortho* to the boronic acid substituent. The phenyl rings of the biphenyl are almost perpendicular to one another, with a dihedral angle of  $87.9(1)^\circ$  between them. In the crystal, adjacent molecules are linked *via*  $O-H \cdots O$  interactions to form centrosymmetric dimers with  $R_2^2(8)$  motifs, which have recently been shown to be energetically very favourable. The hydroxy groups are in an *anti* conformation and are also engaged in hydrogen-bonding interactions with the O atom of the tetrahydrofuran solvent molecule.  $Cl \cdots Cl$  halogen-bonding interactions [ $Cl \cdots Cl = 3.464(1) \text{ \AA}$ ] link neighbouring dimers into chains running along [010]. Further aggregation occurs due to an additional  $Cl \cdots Cl$  halogen bond [ $Cl \cdots Cl = 3.387(1) \text{ \AA}$ ].

## 1. Chemical context

Boronic acids and their derivatives have been studied intensively in recent years due to their numerous applications in organic, analytical and materials chemistry (Hall, 2011; Furukawa & Yaghi, 2009). They are widely used in medicine, for example, as antifungal and antibacterial agents (Adamczyk-Woźniak *et al.*, 2015; Kane *et al.*, 2003; Vogt *et al.*, 2013). Besides these applications, phenylboronic acids have also been studied in terms of crystal engineering (Nishiyabu *et al.*, 2011; Severin, 2009). In contrast, biphenyl-based boronic acids have been largely neglected. Exceptions to this include reports of the crystal structures of (2-biphenyl)boronic acid (Filthaus *et al.*, 2008) and (2-methoxy-3-biphenyl)boronic acid (Davies *et al.*, 2008). In this manuscript we focus our attention on a sterically hindered boronic acid derivative based on a biphenyl core with a boronic group located at the 2-position of one benzene ring with a Cl substituent at the 3-position. The second benzene ring of the biphenyl ring system carries chlorine substituents at the 2- and 6-positions. This molecule crystallized as a 1:1 solvate with THF, Fig. 1.

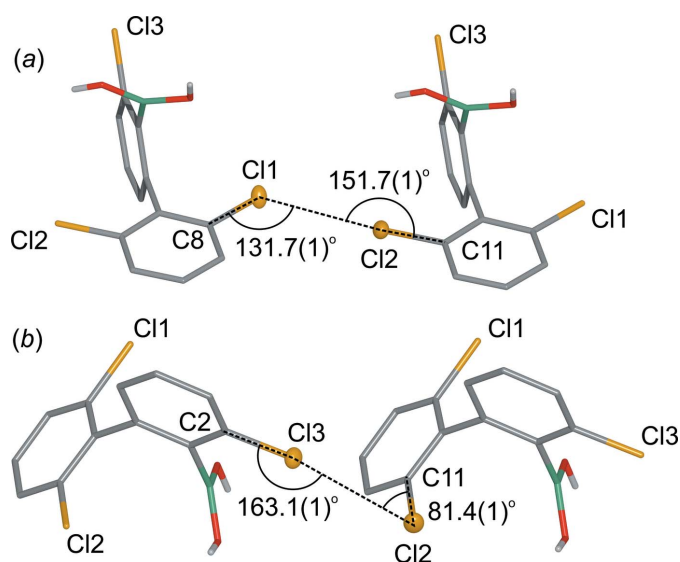




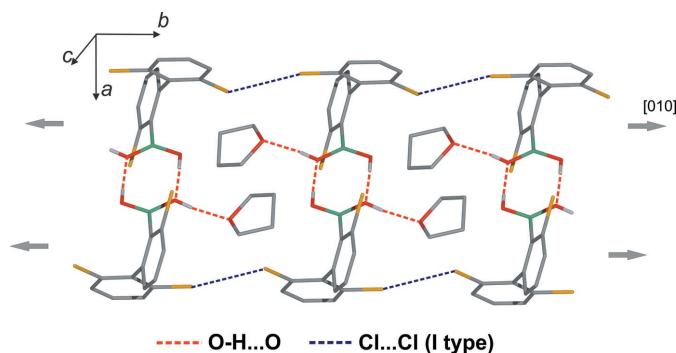
**Figure 1**  
The structure of **1**, showing the atom numbering, with displacement ellipsoids drawn at the 50% probability level.

## 2. Structural commentary

The B—C [1.5907 (16) Å] and B—O [1.3514 (14), 1.3641 (14) Å] bonds in the title compound (**1**) are within the expected range typically observed for boronic acids (Madura *et al.*, 2014; Luliński *et al.*, 2007; Maly *et al.*, 2006; Shimpi *et al.*, 2007; Durka *et al.*, 2012). The molecular structure shows that the B(OH)<sub>2</sub> group adopts the usual *syn-anti* conformation (Fig. 1). The boronic acid substituent is significantly rotated about the C—B bond in order to minimize the steric hindrance between the boronic group and the adjacent 2',6'-dichlorophenyl ring [ $\tau_{C2-C1-B1-O1} = 69.2 (2)^\circ$ ]. In the structure of the



**Figure 2**  
Type I (a) and II (b) Cl...Cl halogen bonds in (**1**).

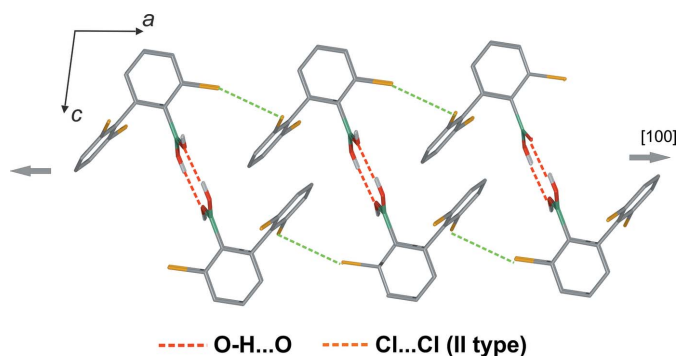


**Figure 3**  
Molecular chains formed along *b* by type I Cl...Cl halogen bonds. Also shown are inversion dimers and inclusion of the solvent through O—H...O hydrogen bonds. Aromatic H atoms have been omitted for clarity.

related (2-biphenyl)boronic acid (Filthaus *et al.*, 2008) this torsion angle is some 20° smaller, which clearly shows the influence of the three chlorine substituents on this structure. It is also notable that in (**1**) the phenyl rings of the biphenyl system are almost perpendicular to one another [ $\tau_{C1-C6-C7-C11} = 87.9 (1)^\circ$ ], whereas in (2-biphenyl)boronic acid they are rotated by only 48.4 or 45.4° for the two unique molecules in the asymmetric unit.

## 3. Supramolecular features

In the crystal, centrosymmetric O—H...O hydrogen-bonded dimers are formed. The *anti*-oriented OH group is engaged in an intermolecular O—H...O hydrogen bond (Table 1) with the oxygen atom from the tetrahydrofuran solvate molecule. Because all of the hydrogen-bond acceptor centres are saturated, the *syn* OH group is not involved in any side hydrogen-bond interactions. Neighbouring dimers are connected through Cl...Cl halogen bonds [ $d_{Cl...Cl} = 3.464 (1) \text{ Å}$ ; the sum of the van der Waals radii for Cl is 3.50 Å]. In terms of geometry of this contact, it can be classified as a type I halogen bond (Fig. 2a), (Metrangolo *et al.*, 2005; Nayak *et al.*, 2011). These contacts result in the formation of molecular chains propagating along [010] (Fig. 3). A three-dimensional network forms through additional Cl...Cl halogen bonds (Fig. 4) of type II [ $d_{Cl...Cl} = 3.387 (1) \text{ Å}$ ] (Fig. 2b).



**Figure 4**  
Crystal packing showing intermolecular O—H...O and type II Cl...Cl interactions.

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$O1-H1A\cdots O3$	0.82 (1)	1.84 (1)	2.6475 (12)	169 (2)
$O2-H2A\cdots O1^i$	0.84 (1)	1.96 (1)	2.7997 (12)	175 (2)

Symmetry code: (i)  $-x+2, -y+1, -z+1$ .

#### 4. Synthesis and crystallization

**Synthesis of (I) (Fig. 5):** A solution of 2-iodo-2',3,6'-trichlorobiphenyl (3.8 g, 10 mmol) in THF (50 mL) was added to a stirred solution of *n*-BuLi (10 mmol) in THF (30 mL) at 195 K. The resulting colorless solution was stirred for 1 h to give a colorless precipitate. The electrophile,  $B(OMe)_3$  (2.1 g, 20 mmol) was then added to the stirred mixture to give a colorless solution which was stirred for 1 h and then hydrolyzed with  $H_2O$  (100 mL). Dilute aq.  $H_2SO_4$  was added until the pH was slightly acidic.  $Et_2O$  (50 mL) was next added and the mixture stirred for 10 min. The organic phase was separated and the aqueous phase was extracted with  $Et_2O$  (20 mL). The combined organic solutions were dried over  $MgSO_4$  and evaporated to give a colorless precipitate, yield 2.0 g (66%).  $^1H$  NMR (400 MHz, acetone- $d_6$ ):  $\delta$  = 8.00 (2H, *s*, OH), 7.51 (2H, *m*), 7.39 (3H, *m*), 7.05 (1H, *m*);  $^{13}C\{^1H\}$  NMR (100.6 MHz, acetone- $d_6$ ):  $\delta$  = 141.49, 139.62, 139 (*br*), 136.17, 134.84, 130.49, 129.99, 128.24, 128.14, 127.53.

Crystals suitable for X-ray diffraction analysis were grown by slow evaporation of a THF solution.

#### 5. Refinement details

Crystal data, data collection and structure refinement details are summarized in Table 2. All CH hydrogen atoms were placed in calculated positions with C—H distances of 0.95 or 0.99 Å. They were included in the refinement in the riding-motion approximation with  $U_{iso}(\text{phenyl H}) = 1.2U_{eq}(C)$ . The positions of the OH hydrogen atoms were first found in a difference map. Then their bond lengths were restrained in the last least-squares cycles, with an O—H distance of 0.85 Å and their coordinates refined with  $U_{iso}(\text{hydroxyl H}) = 1.5U_{eq}(O)$ .

#### Acknowledgements

The X-ray measurements were undertaken in the Crystallographic Unit of the Physical Chemistry Laboratory at the Chemistry Department of the University of Warsaw. This

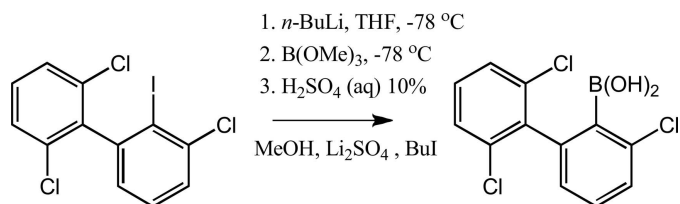


Figure 5

The synthesis of 2-chloro-6-(2',6'-dichlorophenyl)phenylboronic acid.

Table 2

Experimental details.

Crystal data	
Chemical formula	$C_{12}H_8BCl_3O_2 \cdot C_4H_8O$
$M_r$	373.45
Crystal system, space group	Triclinic, $P\bar{1}$
Temperature (K)	130
$a, b, c$ (Å)	8.3306 (3), 8.7122 (2), 12.4307 (4)
$\alpha, \beta, \gamma$ (°)	98.683 (3), 97.737 (3), 99.398 (3)
$V$ (Å <sup>3</sup> )	868.07 (5)
$Z$	2
Radiation type	Mo $K\alpha$
$\mu$ (mm <sup>-1</sup> )	0.54
Crystal size (mm)	0.15 × 0.12 × 0.10
Data collection	
Diffractometer	Agilent SuperNova Dual Source diffractometer with an Atlas detector
Absorption correction	Multi-scan (CrysAlis PRO; Agilent, 2014)
$T_{min}, T_{max}$	0.795, 1.000
No. of measured, independent and observed [ $I > 2\sigma(I)$ ] reflections	28647, 6107, 5162
$R_{int}$	0.032
$(\sin \theta/\lambda)_{max}$ (Å <sup>-1</sup> )	0.758
Refinement	
$R[F^2 > 2\sigma(F^2)], wR(F^2), S$	0.034, 0.085, 1.05
No. of reflections	6107
No. of parameters	214
No. of restraints	2
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}, \Delta\rho_{min}$ (e Å <sup>-3</sup> )	0.45, -0.36

Computer programs: CrysAlis PRO (Agilent, 2014), SHELXS97 (Sheldrick, 2015), SHELXL2013 (Sheldrick, 2015), DIAMOND (Brandenburg, 2005) and publCIF (Westrip, 2010).

work was supported by the Aldrich Chemical Company through the donation of chemicals and equipment, and by the Warsaw University of Technology.

#### References

- Adamczyk-Woźniak, A., Borys, K. M. & Sporyński, A. (2015). *Chem. Rev.* **115**, 5224–5247.
- Agilent (2014). *CrysAlis PRO*. Agilent Technologies, Santa Clara, USA.
- Brandenburg, K. (2005). *DIAMOND*. Crystal Impact GbR, Bonn, Germany.
- Davies, C. J., Gregory, A., Griffith, P., Perkins, T., Singh, K. & Solan, G. A. (2008). *Tetrahedron*, **64**, 9857–9864.
- Durka, K., Jarzemska, K. N., Kamiński, R., Luliński, S., Serwatowski, J. & Woźniak, K. (2012). *Cryst. Growth Des.* **12**, 3720–3734.
- Filthaus, M., Oppel, I. M. & Bettinger, H. F. (2008). *Org. Biomol. Chem.* **6**, 1201–1207.
- Furukawa, H. & Yaghi, O. M. (2009). *J. Am. Chem. Soc.* **131**, 8875–8883.
- Hall, D. G. (2011). In *Boronic Acids*. Weinheim: Wiley-VCH.
- Kane, R. C., Bross, P. F., Farrell, A. T. & Pazdur, R. (2003). *Oncologist*, **8**, 508–513.
- Luliński, S., Madura, I., Serwatowski, J., Szatyłowicz, H. & Zachara, J. (2007). *New J. Chem.* **31**, 144–154.
- Madura, I., Czerwińska, K. & Sołdańska, D. (2014). *Cryst. Growth Des.* **14**, 5912–5921.
- Maly, K. E., Maris, T. & Wuest, J. D. (2006). *CrystEngComm*, **8**, 33–35.

- Metrangolo, P., Neukirch, H., Pilati, T. & Resnati, G. (2005). *Acc. Chem. Res.* **38**, 386–395.
- Nayak, S. K., Reddy, M. K., Guru Row, T. N. & Chopra, D. (2011). *Cryst. Growth Des.* **11**, 1578–1596.
- Nishiyabu, R., Kubo, Y., James, T. D. & Fossey, J. S. (2011). *Chem. Commun.* **47**, 1124–1150.
- Severin, K. (2009). *Dalton Trans.* pp. 5254–5264.
- Sheldrick, G. M. (2015). *Acta Cryst.* **C71**, 3–8.
- Shimpi, M. R., SeethaLekshmi, N. & Pedireddi, V. R. (2007). *Cryst. Growth Des.* **7**, 1958–1963.
- Vogt, F. G., Williams, G. R. & Copley, R. C. B. (2013). *J. Pharm. Sci.* **102**, 3705–3716.
- Westrip, S. P. (2010). *J. Appl. Cryst.* **43**, 920–925.

## supporting information

*Acta Cryst.* (2015). E71, 1471-1474 [https://doi.org/10.1107/S205698901502054X]

## Crystal structure of (2',3,6'-trichlorobiphenyl-2-yl)boronic acid tetrahydrofuran monosolvate

Krzysztof Durka, Tomasz Kliś and Janusz Serwatowski

### Computing details

Data collection: *CrysAlis PRO* (Agilent, 2014); cell refinement: *CrysAlis PRO* (Agilent, 2014); data reduction: *CrysAlis PRO* (Agilent, 2014); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2015); program(s) used to refine structure: *SHELXL2013* (Sheldrick, 2015); molecular graphics: *DIAMOND* (Brandenburg, 2005); software used to prepare material for publication: *publCIF* (Westrip, 2010).

### (2',3,6'-Trichlorobiphenyl-2-yl)boronic acid tetrahydrofuran monosolvate

#### Crystal data

$C_{12}H_8BCl_3O_2 \cdot C_4H_8O$

$M_r = 373.45$

Triclinic,  $P\bar{1}$

$a = 8.3306$  (3) Å

$b = 8.7122$  (2) Å

$c = 12.4307$  (4) Å

$\alpha = 98.683$  (3)°

$\beta = 97.737$  (3)°

$\gamma = 99.398$  (3)°

$V = 868.07$  (5) Å<sup>3</sup>

$Z = 2$

$F(000) = 384$

$D_x = 1.429$  Mg m<sup>-3</sup>

Melting point: 411 K

Mo  $K\alpha$  radiation,  $\lambda = 0.71073$  Å

Cell parameters from 14113 reflections

$\theta = 2.4\text{--}32.4^\circ$

$\mu = 0.54$  mm<sup>-1</sup>

$T = 130$  K

Fragment, colourless

$0.15 \times 0.12 \times 0.10$  mm

#### Data collection

Agilent SuperNova Dual Source  
diffractometer with an Atlas detector  
Radiation source: SuperNova (Mo) X-ray  
Source

Mirror monochromator

Detector resolution: 5.2195 pixels mm<sup>-1</sup>

$\omega$  scans

Absorption correction: multi-scan  
(CrysAlis PRO; Agilent, 2014)

$T_{\min} = 0.795$ ,  $T_{\max} = 1.000$

28647 measured reflections

6107 independent reflections

5162 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.032$

$\theta_{\max} = 32.6^\circ$ ,  $\theta_{\min} = 2.4^\circ$

$h = -12 \rightarrow 12$

$k = -13 \rightarrow 13$

$l = -18 \rightarrow 18$

#### Refinement

Refinement on  $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.034$

$wR(F^2) = 0.085$

$S = 1.05$

6107 reflections

214 parameters

2 restraints

Hydrogen site location: mixed

H atoms treated by a mixture of independent  
and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0328P)^2 + 0.3549P]$

where  $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.001$

$\Delta\rho_{\max} = 0.45$  e Å<sup>-3</sup>

$\Delta\rho_{\min} = -0.36$  e Å<sup>-3</sup>

*Special details*

**Experimental.** Absorption correction: CrysAlisPro (Agilent Technologies, 2014), Empirical absorption correction using spherical harmonics, implemented in SCALE3 ABSPACK scaling algorithm.

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

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ( $\text{\AA}^2$ )*

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.77416 (13)	0.33187 (12)	0.22248 (8)	0.01544 (18)
C2	0.85513 (13)	0.33777 (12)	0.13113 (9)	0.01761 (19)
C3	0.77605 (15)	0.28436 (14)	0.02290 (9)	0.0221 (2)
H3	0.8355	0.2914	−0.0368	0.027*
C5	0.52304 (15)	0.21255 (15)	0.09167 (10)	0.0233 (2)
H5	0.4085	0.1690	0.0782	0.028*
C6	0.60414 (13)	0.26808 (13)	0.19972 (9)	0.01703 (19)
C7	0.50529 (13)	0.26201 (13)	0.29144 (9)	0.01742 (19)
C8	0.47656 (14)	0.12861 (13)	0.34104 (9)	0.0188 (2)
C9	0.38144 (14)	0.12012 (15)	0.42447 (10)	0.0230 (2)
H9	0.3646	0.0276	0.4565	0.028*
C10	0.31167 (15)	0.24852 (16)	0.46023 (11)	0.0263 (2)
H10	0.2466	0.2441	0.5172	0.032*
C11	0.43238 (14)	0.38827 (13)	0.33025 (10)	0.0211 (2)
C12	0.33610 (15)	0.38351 (16)	0.41341 (11)	0.0262 (2)
H12	0.2879	0.4714	0.4377	0.031*
C13	0.60897 (16)	0.22065 (15)	0.00366 (10)	0.0257 (2)
H13	0.5531	0.1825	−0.0697	0.031*
C14	0.82695 (16)	0.75012 (14)	0.14313 (10)	0.0241 (2)
H14A	0.7067	0.7154	0.1184	0.029*
H14B	0.8843	0.6702	0.1077	0.029*
C15	0.88574 (17)	0.91105 (15)	0.11452 (11)	0.0283 (3)
H15A	0.8014	0.9785	0.1202	0.034*
H15B	0.9142	0.9012	0.0393	0.034*
C16	1.03885 (18)	0.97668 (16)	0.20241 (12)	0.0325 (3)
H16A	1.1369	0.9378	0.1804	0.039*
H16B	1.0621	1.0937	0.2165	0.039*
C17	0.9899 (2)	0.91251 (17)	0.30259 (12)	0.0358 (3)
H17A	1.0868	0.8873	0.3471	0.043*
H17B	0.9443	0.9914	0.3495	0.043*
B1	0.86796 (14)	0.39672 (14)	0.34516 (10)	0.0160 (2)
O1	0.92407 (11)	0.55444 (9)	0.38238 (7)	0.02050 (16)
O2	0.89019 (11)	0.29285 (10)	0.41379 (7)	0.02293 (17)
O3	0.86646 (12)	0.77103 (10)	0.26144 (7)	0.02631 (19)
Cl1	0.56394 (4)	−0.03377 (3)	0.29838 (2)	0.02648 (7)
Cl2	0.46307 (4)	0.55927 (3)	0.27352 (3)	0.02936 (8)
Cl3	1.06696 (3)	0.41387 (4)	0.15296 (2)	0.02402 (7)

H1A	0.902 (2)	0.6121 (19)	0.3383 (13)	0.036*
H2A	0.941 (2)	0.336 (2)	0.4767 (12)	0.036*

*Atomic displacement parameters ( $\text{\AA}^2$ )*

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
C1	0.0179 (5)	0.0142 (4)	0.0137 (4)	0.0028 (3)	0.0005 (3)	0.0028 (3)
C2	0.0196 (5)	0.0163 (4)	0.0168 (5)	0.0029 (4)	0.0027 (4)	0.0034 (4)
C3	0.0289 (6)	0.0237 (5)	0.0148 (5)	0.0061 (4)	0.0047 (4)	0.0040 (4)
C5	0.0212 (5)	0.0264 (6)	0.0181 (5)	0.0002 (4)	−0.0030 (4)	0.0013 (4)
C6	0.0186 (5)	0.0169 (4)	0.0146 (4)	0.0025 (4)	0.0002 (4)	0.0028 (4)
C7	0.0156 (4)	0.0194 (5)	0.0152 (4)	0.0009 (4)	0.0001 (3)	0.0015 (4)
C8	0.0197 (5)	0.0202 (5)	0.0150 (5)	0.0025 (4)	0.0003 (4)	0.0014 (4)
C9	0.0208 (5)	0.0284 (6)	0.0192 (5)	0.0013 (4)	0.0022 (4)	0.0065 (4)
C10	0.0186 (5)	0.0363 (7)	0.0236 (6)	0.0033 (5)	0.0060 (4)	0.0044 (5)
C11	0.0176 (5)	0.0196 (5)	0.0243 (5)	0.0016 (4)	0.0010 (4)	0.0027 (4)
C12	0.0193 (5)	0.0296 (6)	0.0293 (6)	0.0065 (4)	0.0056 (4)	0.0005 (5)
C13	0.0305 (6)	0.0290 (6)	0.0138 (5)	0.0024 (5)	−0.0024 (4)	0.0013 (4)
C14	0.0267 (6)	0.0221 (5)	0.0226 (5)	0.0031 (4)	0.0000 (4)	0.0059 (4)
C15	0.0332 (7)	0.0258 (6)	0.0278 (6)	0.0044 (5)	0.0057 (5)	0.0114 (5)
C16	0.0333 (7)	0.0243 (6)	0.0375 (7)	−0.0023 (5)	0.0049 (6)	0.0068 (5)
C17	0.0411 (8)	0.0261 (6)	0.0328 (7)	−0.0066 (5)	−0.0064 (6)	0.0072 (5)
B1	0.0152 (5)	0.0170 (5)	0.0146 (5)	0.0022 (4)	0.0009 (4)	0.0015 (4)
O1	0.0278 (4)	0.0162 (4)	0.0149 (4)	0.0019 (3)	−0.0030 (3)	0.0027 (3)
O2	0.0302 (4)	0.0179 (4)	0.0170 (4)	0.0010 (3)	−0.0053 (3)	0.0036 (3)
O3	0.0328 (5)	0.0202 (4)	0.0233 (4)	−0.0005 (3)	−0.0017 (3)	0.0072 (3)
Cl1	0.04097 (17)	0.02093 (13)	0.02001 (13)	0.00986 (11)	0.00736 (11)	0.00481 (10)
Cl2	0.02627 (15)	0.02037 (13)	0.04284 (18)	0.00462 (10)	0.00663 (12)	0.00884 (12)
Cl3	0.01988 (13)	0.02816 (14)	0.02462 (14)	0.00239 (10)	0.00640 (10)	0.00636 (11)

*Geometric parameters ( $\text{\AA}$ ,  $^\circ$ )*

C1—C2	1.3999 (15)	C11—Cl2	1.7388 (12)
C1—C6	1.4081 (15)	C12—H12	0.9500
C1—B1	1.5907 (16)	C13—H13	0.9500
C2—C3	1.3902 (16)	C14—O3	1.4400 (15)
C2—Cl3	1.7498 (11)	C14—C15	1.5183 (17)
C3—C13	1.3859 (18)	C14—H14A	0.9900
C3—H3	0.9500	C14—H14B	0.9900
C5—C13	1.3900 (17)	C15—C16	1.532 (2)
C5—C6	1.3954 (15)	C15—H15A	0.9900
C5—H5	0.9500	C15—H15B	0.9900
C6—C7	1.4959 (15)	C16—C17	1.517 (2)
C7—C11	1.3959 (16)	C16—H16A	0.9900
C7—C8	1.3976 (15)	C16—H16B	0.9900
C8—C9	1.3908 (16)	C17—O3	1.4465 (16)
C8—Cl1	1.7395 (12)	C17—H17A	0.9900
C9—C10	1.3851 (18)	C17—H17B	0.9900



C9—H9	0.9500	B1—O2	1.3514 (14)
C10—C12	1.3869 (19)	B1—O1	1.3641 (14)
C10—H10	0.9500	O1—H1A	0.821 (14)
C11—C12	1.3924 (17)	O2—H2A	0.838 (14)
C2—C1—C6	116.31 (9)	C3—C13—C5	120.03 (11)
C2—C1—B1	121.95 (9)	C3—C13—H13	120.0
C6—C1—B1	121.73 (9)	C5—C13—H13	120.0
C3—C2—C1	123.29 (10)	O3—C14—C15	105.36 (10)
C3—C2—Cl3	117.77 (9)	O3—C14—H14A	110.7
C1—C2—Cl3	118.93 (8)	C15—C14—H14A	110.7
C13—C3—C2	118.82 (11)	O3—C14—H14B	110.7
C13—C3—H3	120.6	C15—C14—H14B	110.7
C2—C3—H3	120.6	H14A—C14—H14B	108.8
C13—C5—C6	120.36 (11)	C14—C15—C16	102.13 (10)
C13—C5—H5	119.8	C14—C15—H15A	111.3
C6—C5—H5	119.8	C16—C15—H15A	111.3
C5—C6—C1	121.18 (10)	C14—C15—H15B	111.3
C5—C6—C7	118.41 (10)	C16—C15—H15B	111.3
C1—C6—C7	120.40 (9)	H15A—C15—H15B	109.2
C11—C7—C8	116.15 (10)	C17—C16—C15	102.58 (11)
C11—C7—C6	121.66 (10)	C17—C16—H16A	111.3
C8—C7—C6	122.17 (10)	C15—C16—H16A	111.3
C9—C8—C7	122.68 (11)	C17—C16—H16B	111.3
C9—C8—Cl1	118.09 (9)	C15—C16—H16B	111.3
C7—C8—Cl1	119.23 (9)	H16A—C16—H16B	109.2
C10—C9—C8	119.03 (11)	O3—C17—C16	106.63 (11)
C10—C9—H9	120.5	O3—C17—H17A	110.4
C8—C9—H9	120.5	C16—C17—H17A	110.4
C9—C10—C12	120.49 (11)	O3—C17—H17B	110.4
C9—C10—H10	119.8	C16—C17—H17B	110.4
C12—C10—H10	119.8	H17A—C17—H17B	108.6
C12—C11—C7	122.63 (11)	O2—B1—O1	119.71 (10)
C12—C11—Cl2	118.30 (9)	O2—B1—C1	118.96 (9)
C7—C11—Cl2	119.07 (9)	O1—B1—C1	121.33 (9)
C10—C12—C11	119.01 (12)	B1—O1—H1A	115.3 (13)
C10—C12—H12	120.5	B1—O2—H2A	113.1 (12)
C11—C12—H12	120.5	C14—O3—C17	109.77 (10)
C6—C1—C2—C3	−0.15 (16)	Cl1—C8—C9—C10	179.55 (9)
B1—C1—C2—C3	−178.97 (10)	C8—C9—C10—C12	0.09 (18)
C6—C1—C2—Cl3	−179.42 (8)	C8—C7—C11—C12	−0.07 (17)
B1—C1—C2—Cl3	1.76 (14)	C6—C7—C11—C12	178.17 (11)
C1—C2—C3—C13	−0.56 (17)	C8—C7—C11—Cl2	179.60 (8)
Cl3—C2—C3—C13	178.72 (9)	C6—C7—C11—Cl2	−2.15 (15)
C13—C5—C6—C1	−0.63 (18)	C9—C10—C12—C11	−0.26 (19)
C13—C5—C6—C7	177.91 (11)	C7—C11—C12—C10	0.25 (18)
C2—C1—C6—C5	0.74 (16)	Cl2—C11—C12—C10	−179.42 (10)



B1—C1—C6—C5	179.56 (10)	C2—C3—C13—C5	0.69 (18)
C2—C1—C6—C7	−177.77 (9)	C6—C5—C13—C3	−0.11 (19)
B1—C1—C6—C7	1.05 (15)	O3—C14—C15—C16	33.84 (13)
C5—C6—C7—C11	−90.69 (13)	C14—C15—C16—C17	−35.53 (14)
C1—C6—C7—C11	87.86 (13)	C15—C16—C17—O3	25.23 (15)
C5—C6—C7—C8	87.44 (14)	C2—C1—B1—O2	−111.66 (12)
C1—C6—C7—C8	−94.00 (13)	C6—C1—B1—O2	69.59 (14)
C11—C7—C8—C9	−0.11 (16)	C2—C1—B1—O1	69.17 (14)
C6—C7—C8—C9	−178.35 (10)	C6—C1—B1—O1	−109.58 (12)
C11—C7—C8—C11	−179.56 (8)	C15—C14—O3—C17	−18.86 (14)
C6—C7—C8—C11	2.21 (14)	C16—C17—O3—C14	−4.30 (16)
C7—C8—C9—C10	0.10 (17)		

*Hydrogen-bond geometry (Å, °)*

<i>D</i> —H $\cdots$ <i>A</i>	<i>D</i> —H	H $\cdots$ <i>A</i>	<i>D</i> $\cdots$ <i>A</i>	<i>D</i> —H $\cdots$ <i>A</i>
O1—H1 <i>A</i> $\cdots$ O3	0.82 (1)	1.84 (1)	2.6475 (12)	169 (2)
O2—H2 <i>A</i> $\cdots$ O1 <sup>i</sup>	0.84 (1)	1.96 (1)	2.7997 (12)	175 (2)

Symmetry code: (i)  $-x+2, -y+1, -z+1$ .