

Acta Crystallographica Section E

## Structure Reports

Online

ISSN 1600-5368

## 1,3,5-Tris(bromomethyl)-2,4,6-tris(2-methoxycarbonyl-2-methylpropyl)-benzene

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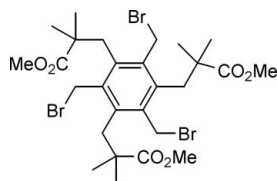
Received 13 November 2007; accepted 16 November 2007

Key indicators: single-crystal X-ray study;  $T = 213$  K; mean  $\sigma(\text{C}-\text{C}) = 0.003$  Å;  $R$  factor = 0.026;  $wR$  factor = 0.069; data-to-parameter ratio = 18.9.

In the title compound,  $\text{C}_{27}\text{H}_{39}\text{Br}_3\text{O}_6$ , static gearing of the methylene H atoms ensures that the  $\beta$ -groups alternate above (*a*) and below (*b*) the aromatic ring in a nearly trigonally symmetric conformation (*ababab*), resulting in the bromo substituents and ester groups being on opposite sides of the arene ring. A notable feature of the crystal packing is the intermolecular interactions involving Br atoms: in each molecule, two of the Br atoms are involved in intermolecular  $\text{Br} \cdots \text{Br}$  interactions [3.5911 (4) Å], while the third halogen makes two short contacts with the ester carbonyl O atom [ $\text{Br} \cdots \text{O}$  3.349 (2) Å] and one H atom of a  $\text{CH}_2$  group of an ester functionality of another molecule.

## Related literature

For related literature, see: Auffinger *et al.* (2004); Awwadi *et al.*, (2006); Desiraju (2002); Hennrich *et al.* (2002); Iverson *et al.* (1981); MacNicol *et al.* (1977); Marsau (1965); Metrangolo *et al.* (2005); Perreault *et al.* (1997); Stack *et al.* (1993); Steiner (2002); Szabo *et al.* (1998); Wiskur *et al.* (2001).



## Experimental

## Crystal data

 $\text{C}_{27}\text{H}_{39}\text{Br}_3\text{O}_6$  $M_r = 699.31$ Triclinic,  $P\bar{1}$  $a = 9.8160$  (9) Å $b = 10.9110$  (9) Å $c = 14.4422$  (13) Å $\alpha = 83.279$  (2)° $\beta = 70.934$  (2)° $\gamma = 89.364$  (2)° $V = 1451.3$  (2) Å<sup>3</sup> $Z = 2$ Mo  $K\alpha$  radiation $\mu = 4.21$  mm<sup>-1</sup> $T = 213$  (1) K

0.40 × 0.38 × 0.28 mm

## Data collection

Bruker SMART 1000/P4 diffractometer

Absorption correction: multi-scan

(SADABS; Sheldrick, 2004)

 $T_{\min} = 0.212$ ,  $T_{\max} = 0.313$ 

10173 measured reflections

6311 independent reflections

5523 reflections with  $I > 2\sigma(I)$  $R_{\text{int}} = 0.014$ 

## Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.026$  $wR(F^2) = 0.069$  $S = 1.04$ 

6311 reflections

334 parameters

H-atom parameters constrained

 $\Delta\rho_{\text{max}} = 0.62$  e Å<sup>-3</sup> $\Delta\rho_{\text{min}} = -0.34$  e Å<sup>-3</sup>

Table 1

Hydrogen-bond geometry (Å, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
$\text{C23}-\text{H23A} \cdots \text{Br3}^i$	0.98	2.90	3.7875 (19)	151

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

Data collection: SMART (Bruker, 1999); cell refinement: SAINT (Bruker, 2006); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: SHELXTL (Sheldrick, 2000); software used to prepare material for publication: SHELXTL.

Financial support by NSERC, Canada, and FQRNT, Québec, is gratefully acknowledged.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BG2134).

## References

- Auffinger, P., Hays, F. A., Westhof, E. & Ho, P. S. (2004). *Proc. Natl Acad. Sci. USA*, **101**, 16789–16794.
- Awwadi, F. F., Willett, R. D., Peterson, K. A. & Twamley, B. (2006). *Chem. Eur. J.* **12**, 8952–8960.
- Bruker (1999). SMART. Version 5.054. Bruker AXS Inc., Madison, Wisconsin, USA.
- Bruker (2006). SAINT. Version 7.23A. Bruker AXS Inc., Madison, Wisconsin, USA.
- Desiraju, G. R. (2002). *Acc. Chem. Res.* **35**, 565–573.
- Hennrich, G., Lynch, V. M. & Anslyn, E. V. (2002). *Chem. Eur. J.* **8**, 2274–2278.
- Iverson, J. D., Hunter, G., Blount, J. F., Damewood, J. R. Jr & Mislow, K. (1981). *J. Am. Chem. Soc.* **103**, 6073–6083.
- MacNicol, D. D., Hardy, A. D. U. & Wilson, D. R. (1977). *Nature (London)*, **266**, 611–612.
- Marsau, M. P. (1965). *Acta Cryst.* **18**, 851–854.
- Metrangolo, P., Neukirch, H., Pilati, T. & Resnati, G. (2005). *Acc. Chem. Res.* **38**, 386–395.
- Perreault, D. M., Cabell, L. A. & Anslyn, E. V. (1997). *Bioorg. Med. Chem.* **5**, 1209–1220.
- Sheldrick, G. M. (1997). SHELXS97 and SHELXL97. University of Göttingen, Germany.
- Sheldrick, G. M. (2004). SADABS. Bruker AXS Inc., Madison, Wisconsin, USA.
- Sheldrick, G. M. (2000). SHELXTL. Version 6.14. Bruker AXS Inc., Madison, Wisconsin, USA.
- Stack, T. D. P., Hou, Z. & Raymond, K. N. (1993). *J. Am. Chem. Soc.* **115**, 6466–6467.
- Steiner, T. (2002). *Angew. Chem. Int. Ed.* **41**, 48–76.
- Szabo, T., Hilmersson, G. & Rebek, J. Jr (1998). *J. Am. Chem. Soc.* **120**, 6193–6194.
- Wiskur, S. L., Ait-Haddou, H., Lavigne, J. J. & Anslyn, E. V. (2001). *Acc. Chem. Res.* **34**, 963–972.

**supplementary materials**

*Acta Cryst.* (2007). E63, o4905 [ doi:10.1107/S1600536807060072 ]

## 1,3,5-Tris(bromomethyl)-2,4,6-tris(2-methoxycarbonyl-2-methylpropyl)benzene

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### Comment

To the best of our knowledge, the first reported crystal structure of a hexasubstituted benzene was hexakisbromomethylbenzene (Marsau, 1965). Many other hexasubstituted benzenes have since been crystallized. The particularity of this class of compounds is their tendency to orient their 1,3,5 and 2,4,6 substituents respectively on the opposite side, above (*a*) and below (*b*), of the central ring due to static gearing (Iverson *et al.*, 1981), resulting in an ababab pattern.

Hexasubstituted benzene analogues in which the functional groups are observed or predicted to point alternately up and down around the ring perimeter have been extensively studied mainly due to their applications in supramolecular chemistry. For example, this kind of system, which offers a preorganized site, has been used in the design of clathrates (MacNicol *et al.*, 1977), artificial receptors (Stack *et al.*, 1993; Perreault *et al.*, 1997; Wiskur *et al.*, 2001) and self-assembling molecular capsules (Szabo *et al.*, 1998).

While there are numerous crystal structures reported containing hexasubstituted benzene systems with the same six functional groups or with three functional groups in the 2,4, and 6-positions, the 1,3,5 positions bearing simple methyl or ethyl groups, very few (Hennrich *et al.*, 2002) do not follow the ababab pattern. We report here the X-ray structure of a new molecular scaffold with alternating alkyl-bromo and alkyl-ester functions on either face of the six-membered ring.

The title compound, (C<sub>6</sub>(CH<sub>2</sub>Br)<sub>3</sub>(CH<sub>2</sub>C(CH<sub>3</sub>)<sub>2</sub>CO<sub>2</sub>CH<sub>3</sub>)<sub>3</sub>) **1**, is shown in Figure 1. As expected, the substituents adopt a three-up-three-down conformation (Figure 2). However, this result is contrary to the crystal structure of 1,3,5-tris(acetoxymethyl)-2,4,6-trithiophenyl benzene (Hennrich *et al.*, 2002) for which the three-dimensional arrangement of substituents is 1,3-up, 5-down tris-(acetoxymethyl), 2,4-down, 6-up-trithiophenyl benzene.

Although usually unpredictable, crystal packing is often guided and assisted by weak interactions like hydrogen bonds (Desiraju, 2002; Steiner, 2002) or halogen bonds (Metrangolo *et al.*, 2005). In our compound, both of these interactions are at work.

Thus, two bromine atoms per molecule (Figure 3) face two other bromine atoms of another molecule. Since the alignments are linear and the Br—Br distances are sufficiently short (Br1—Br2<sup>i</sup>/Br2—Br1<sup>i</sup> = 3.5911 (4) Å, i: -x, 1 - y, 1 - z), each pair of such neighboring bromine atoms is in fact brought together by a halogen bond (Awwadi *et al.*, 2006). The remaining Br atom forms Br...H—C (Br3—H23a<sup>ii</sup> = 2.904 Å, ii: -x, -y, 1 - z) and Br...O=C (Br3—O11<sup>iii</sup> = 3.349 (2) Å, iii: -1 - x, -y, 2 - z) bonds (Auffinger *et al.*, 2004) involving two different molecules.

Only one ester per molecule is part of such O—Br interactions.

In conclusion, the crystal structure of (C<sub>6</sub>(CH<sub>2</sub>Br)<sub>3</sub>(CH<sub>2</sub>C(CH<sub>3</sub>)<sub>2</sub>CO<sub>2</sub>CH<sub>3</sub>)<sub>3</sub>) shows an original arrangement in which the crystal packing displays halogen bonds that do not disturb the intuitive alternate three-up-three-down conformation.

## Experimental

$C_6(CH_3)_3(CH_2C(CH_3)_2CO_2CH_3)_3$  was treated with NBS and  $Bz_2O_2$  in  $CCl_4$  under reflux to yield compound **1**. A ether/pentane solution of  $C_6(CH_2Br)_3(CH_2C(CH_3)_2CO_2CH_3)_3$  was kept until complete evaporation of the solvent. Colourless, transparent crystals of **1** suitable for X-ray analysis were obtained.

## Refinement

Hydrogen atoms were placed in calculated positions and refined using a riding model.

## Figures

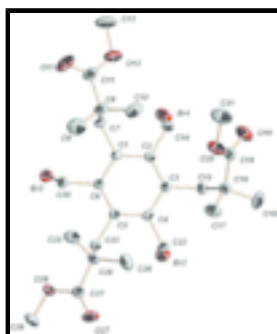


Fig. 1. A molecular view of the title compound with thermal ellipsoids at the 50% level. Hydrogen atoms have been omitted for clarity.

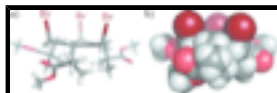


Fig. 2. Side view of **1** as a pipe and space filling model showing the ababab pattern of the substituents.



Fig. 3. Main interactions involving the bromine atoms. Symmetry codes: i:  $-x, 1 - y, 1 - z$ ; ii:  $-x, -y, 1 - z$ ; iii:  $-1 - x, -y, 2 - z$ .

## 1,3,5-Tris(bromomethyl)-2,4,6-tris(2-methoxycarbonyl-2-methylpropyl)benzene

### Crystal data

$C_{27}H_{39}Br_3O_6$

$M_r = 699.31$

Triclinic,  $P\bar{1}$

Hall symbol:  $-P\ 1$

$a = 9.8160(9)\ \text{\AA}$

$b = 10.9110(9)\ \text{\AA}$

$c = 14.4422(13)\ \text{\AA}$

$\alpha = 83.279(2)^\circ$

$\beta = 70.934(2)^\circ$

$Z = 2$

$F_{000} = 708$

$D_x = 1.600\ \text{Mg m}^{-3}$

Mo  $K\alpha$  radiation

$\lambda = 0.71073\ \text{\AA}$

Cell parameters from 5471 reflections

$\theta = 2.3\text{--}28.1^\circ$

$\mu = 4.21\ \text{mm}^{-1}$

$T = 213(1)\ \text{K}$

Parallelepiped, colourless

$\gamma = 89.364 (2)^\circ$   
 $V = 1451.3 (2) \text{ \AA}^3$   
 $0.40 \times 0.38 \times 0.28 \text{ mm}$

*Data collection*

Bruker SMART1000/P4 diffractometer	5523 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{\text{int}} = 0.014$
$T = 213(1) \text{ K}$	$\theta_{\text{max}} = 27.5^\circ$
$\varphi$ and $\omega$ scans	$\theta_{\text{min}} = 1.5^\circ$
Absorption correction: multi-scan (SADABS; Sheldrick, 2004)	$h = -12 \rightarrow 12$
$T_{\text{min}} = 0.212, T_{\text{max}} = 0.313$	$k = -14 \rightarrow 13$
10173 measured reflections	$l = -17 \rightarrow 18$
6311 independent reflections	

*Refinement*

Refinement on $F^2$	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.026$	H-atom parameters constrained
$wR(F^2) = 0.069$	$w = 1/[\sigma^2(F_o^2) + (0.0379P)^2 + 0.5054P]$
$S = 1.04$	where $P = (F_o^2 + 2F_c^2)/3$
6311 reflections	$(\Delta/\sigma)_{\text{max}} = 0.001$
334 parameters	$\Delta\rho_{\text{max}} = 0.62 \text{ e \AA}^{-3}$
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{\text{min}} = -0.34 \text{ e \AA}^{-3}$
	Extinction correction: none

*Special details*

**Experimental.** Crystal decay was monitored by repeating the initial 50 frames at the end of the data collection and analyzing duplicate reflections.

**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.

**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  $F^2 > 2\sigma(F^2)$  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 ( $\text{Å}^2$ )*

	$x$	$y$	$z$	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	-0.11458 (19)	0.17480 (16)	0.79133 (13)	0.0219 (4)

## supplementary materials

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C2	-0.01962 (19)	0.27822 (16)	0.76904 (13)	0.0224 (4)
C3	0.11470 (19)	0.28177 (16)	0.69291 (13)	0.0219 (4)
C4	0.15113 (19)	0.18209 (16)	0.63661 (13)	0.0221 (4)
C5	0.05705 (19)	0.07814 (16)	0.65758 (13)	0.0213 (4)
C6	-0.07767 (19)	0.07690 (16)	0.73304 (13)	0.0214 (3)
C7	-0.2565 (2)	0.17176 (18)	0.87601 (13)	0.0260 (4)
H7A	-0.3279	0.1230	0.8607	0.031*
H7B	-0.2912	0.2561	0.8806	0.031*
C8	-0.2474 (2)	0.1163 (2)	0.97943 (15)	0.0347 (5)
C9	-0.2373 (3)	-0.0242 (2)	0.98707 (18)	0.0523 (7)
H9A	-0.2363	-0.0561	1.0524	0.078*
H9B	-0.1494	-0.0463	0.9380	0.078*
H9C	-0.3199	-0.0594	0.9756	0.078*
C10	-0.1218 (3)	0.1692 (3)	1.00545 (17)	0.0474 (6)
H10A	-0.1342	0.2569	1.0100	0.071*
H10B	-0.0315	0.1566	0.9545	0.071*
H10C	-0.1204	0.1276	1.0683	0.071*
C11	-0.3887 (3)	0.1479 (3)	1.05471 (16)	0.0428 (6)
O11	-0.4844 (2)	0.0769 (2)	1.09979 (17)	0.0838 (8)
O12	-0.3940 (2)	0.2676 (2)	1.06339 (17)	0.0709 (6)
C13	-0.5216 (4)	0.3084 (4)	1.1352 (3)	0.0938 (14)
H13A	-0.6067	0.2796	1.1231	0.141*
H13B	-0.5197	0.3979	1.1296	0.141*
H13C	-0.5241	0.2751	1.2010	0.141*
C14	-0.0685 (2)	0.39069 (18)	0.82093 (15)	0.0288 (4)
H14A	0.0154	0.4369	0.8228	0.035*
H14B	-0.1301	0.3655	0.8890	0.035*
C15	0.2184 (2)	0.39274 (16)	0.67068 (15)	0.0256 (4)
H15A	0.1611	0.4666	0.6844	0.031*
H15B	0.2728	0.4024	0.5999	0.031*
C16	0.3291 (2)	0.38894 (18)	0.72834 (15)	0.0293 (4)
C17	0.3977 (3)	0.2633 (2)	0.73542 (18)	0.0419 (5)
H17A	0.4618	0.2654	0.7740	0.063*
H17B	0.4520	0.2444	0.6697	0.063*
H17C	0.3227	0.2003	0.7670	0.063*
C18	0.4480 (3)	0.4877 (3)	0.6719 (2)	0.0494 (6)
H18A	0.5162	0.4920	0.7073	0.074*
H18B	0.4045	0.5674	0.6667	0.074*
H18C	0.4978	0.4658	0.6064	0.074*
C19	0.2559 (2)	0.42944 (18)	0.83050 (16)	0.0310 (4)
O19	0.2168 (2)	0.53239 (15)	0.84482 (14)	0.0494 (4)
O20	0.2371 (2)	0.33924 (15)	0.90415 (12)	0.0485 (4)
C21	0.1737 (4)	0.3742 (3)	1.0022 (2)	0.0643 (8)
H21A	0.2344	0.4371	1.0125	0.097*
H21B	0.1654	0.3025	1.0504	0.097*
H21C	0.0787	0.4064	1.0094	0.097*
C22	0.2857 (2)	0.19154 (18)	0.54706 (14)	0.0277 (4)
H22A	0.3190	0.1087	0.5325	0.033*
H22B	0.3625	0.2361	0.5600	0.033*

C23	0.1000 (2)	-0.03083 (16)	0.59799 (14)	0.0231 (4)
H23A	0.1613	0.0003	0.5310	0.028*
H23B	0.0127	-0.0679	0.5929	0.028*
C24	0.1823 (2)	-0.13454 (17)	0.64124 (15)	0.0263 (4)
C25	0.0828 (3)	-0.20735 (19)	0.73649 (15)	0.0358 (5)
H25A	0.0034	-0.2452	0.7230	0.054*
H25B	0.0452	-0.1518	0.7861	0.054*
H25C	0.1369	-0.2710	0.7605	0.054*
C26	0.3150 (3)	-0.0854 (2)	0.6614 (2)	0.0426 (6)
H26A	0.3591	-0.1530	0.6901	0.064*
H26B	0.2855	-0.0238	0.7069	0.064*
H26C	0.3842	-0.0483	0.5999	0.064*
C27	0.2345 (2)	-0.22309 (17)	0.56309 (14)	0.0275 (4)
O27	0.35750 (17)	-0.24632 (17)	0.52245 (13)	0.0462 (4)
O28	0.12400 (17)	-0.27239 (14)	0.54482 (12)	0.0417 (4)
C29	0.1565 (3)	-0.3534 (2)	0.46845 (19)	0.0461 (6)
H29A	0.2180	-0.3095	0.4068	0.069*
H29B	0.0676	-0.3800	0.4603	0.069*
H29C	0.2058	-0.4249	0.4870	0.069*
C30	-0.1906 (2)	-0.02155 (17)	0.74423 (14)	0.0259 (4)
H30A	-0.2463	-0.0428	0.8142	0.031*
H30B	-0.1440	-0.0962	0.7186	0.031*
Br1	-0.17711 (3)	0.49697 (2)	0.750578 (19)	0.04397 (7)
Br2	0.24186 (3)	0.28042 (2)	0.433048 (15)	0.03893 (7)
Br3	-0.31992 (2)	0.04030 (2)	0.670707 (16)	0.03421 (6)

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

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
C1	0.0200 (9)	0.0256 (9)	0.0186 (8)	0.0032 (7)	-0.0046 (7)	-0.0017 (7)
C2	0.0222 (9)	0.0220 (8)	0.0224 (9)	0.0030 (7)	-0.0062 (7)	-0.0042 (7)
C3	0.0202 (9)	0.0208 (8)	0.0243 (9)	0.0008 (7)	-0.0074 (7)	-0.0014 (7)
C4	0.0177 (8)	0.0245 (8)	0.0214 (9)	0.0016 (7)	-0.0035 (7)	-0.0014 (7)
C5	0.0210 (9)	0.0212 (8)	0.0214 (9)	0.0034 (7)	-0.0066 (7)	-0.0024 (7)
C6	0.0198 (8)	0.0215 (8)	0.0212 (9)	0.0006 (7)	-0.0050 (7)	-0.0004 (7)
C7	0.0198 (9)	0.0337 (10)	0.0215 (9)	0.0026 (7)	-0.0021 (7)	-0.0053 (7)
C8	0.0272 (10)	0.0501 (13)	0.0227 (10)	-0.0002 (9)	-0.0034 (8)	-0.0024 (9)
C9	0.0676 (18)	0.0522 (15)	0.0321 (12)	0.0066 (13)	-0.0144 (12)	0.0087 (11)
C10	0.0324 (12)	0.0808 (18)	0.0285 (12)	0.0001 (12)	-0.0111 (10)	-0.0008 (11)
C11	0.0317 (12)	0.0701 (16)	0.0226 (11)	-0.0052 (11)	-0.0018 (9)	-0.0104 (11)
O11	0.0522 (13)	0.1063 (18)	0.0619 (14)	-0.0280 (12)	0.0254 (11)	-0.0134 (13)
O12	0.0459 (11)	0.0826 (15)	0.0690 (14)	0.0033 (10)	0.0124 (10)	-0.0407 (12)
C13	0.0494 (19)	0.138 (4)	0.091 (3)	0.020 (2)	0.0015 (18)	-0.075 (3)
C14	0.0270 (10)	0.0292 (9)	0.0309 (10)	0.0050 (8)	-0.0084 (8)	-0.0099 (8)
C15	0.0250 (9)	0.0222 (9)	0.0298 (10)	-0.0014 (7)	-0.0094 (8)	-0.0020 (7)
C16	0.0244 (10)	0.0302 (10)	0.0346 (11)	0.0010 (8)	-0.0112 (8)	-0.0044 (8)
C17	0.0408 (13)	0.0473 (13)	0.0459 (13)	0.0209 (10)	-0.0223 (11)	-0.0171 (11)
C18	0.0322 (12)	0.0621 (16)	0.0539 (15)	-0.0173 (11)	-0.0151 (11)	-0.0018 (12)

## supplementary materials

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C19	0.0296 (10)	0.0308 (10)	0.0389 (12)	0.0023 (8)	-0.0181 (9)	-0.0085 (8)
O19	0.0637 (12)	0.0342 (8)	0.0575 (11)	0.0153 (8)	-0.0264 (9)	-0.0167 (8)
O20	0.0722 (12)	0.0383 (9)	0.0334 (9)	0.0112 (8)	-0.0142 (8)	-0.0083 (7)
C21	0.088 (2)	0.0687 (19)	0.0360 (14)	0.0148 (16)	-0.0168 (15)	-0.0146 (13)
C22	0.0238 (9)	0.0285 (9)	0.0258 (10)	0.0005 (7)	-0.0017 (8)	-0.0028 (8)
C23	0.0241 (9)	0.0222 (8)	0.0224 (9)	0.0035 (7)	-0.0065 (7)	-0.0038 (7)
C24	0.0279 (10)	0.0230 (9)	0.0301 (10)	0.0056 (7)	-0.0110 (8)	-0.0070 (7)
C25	0.0486 (13)	0.0298 (10)	0.0280 (11)	0.0116 (9)	-0.0120 (10)	-0.0023 (8)
C26	0.0429 (13)	0.0336 (11)	0.0656 (16)	0.0105 (10)	-0.0352 (12)	-0.0133 (11)
C27	0.0290 (10)	0.0230 (9)	0.0284 (10)	0.0053 (7)	-0.0070 (8)	-0.0017 (7)
O27	0.0282 (8)	0.0646 (11)	0.0470 (10)	0.0181 (8)	-0.0093 (7)	-0.0223 (8)
O28	0.0315 (8)	0.0427 (9)	0.0515 (10)	-0.0007 (6)	-0.0065 (7)	-0.0289 (8)
C29	0.0538 (15)	0.0357 (12)	0.0518 (15)	0.0041 (10)	-0.0145 (12)	-0.0251 (11)
C30	0.0233 (9)	0.0239 (9)	0.0284 (10)	-0.0012 (7)	-0.0063 (8)	-0.0009 (7)
Br1	0.05016 (15)	0.03338 (12)	0.05592 (15)	0.01883 (10)	-0.02485 (12)	-0.01511 (10)
Br2	0.04493 (14)	0.03935 (12)	0.02538 (11)	-0.00344 (9)	-0.00353 (9)	0.00146 (8)
Br3	0.02797 (11)	0.04168 (12)	0.03524 (12)	0.00208 (8)	-0.01226 (9)	-0.00812 (9)

### *Geometric parameters (Å, °)*

C1—C6	1.408 (2)	C16—C18	1.545 (3)
C1—C2	1.410 (3)	C17—H17A	0.9700
C1—C7	1.522 (2)	C17—H17B	0.9700
C2—C3	1.412 (3)	C17—H17C	0.9700
C2—C14	1.505 (2)	C18—H18A	0.9700
C3—C4	1.407 (2)	C18—H18B	0.9700
C3—C15	1.526 (2)	C18—H18C	0.9700
C4—C5	1.411 (2)	C19—O19	1.202 (2)
C4—C22	1.511 (2)	C19—O20	1.329 (3)
C5—C6	1.411 (2)	O20—C21	1.443 (3)
C5—C23	1.524 (2)	C21—H21A	0.9700
C6—C30	1.509 (2)	C21—H21B	0.9700
C7—C8	1.575 (3)	C21—H21C	0.9700
C7—H7A	0.9800	C22—Br2	1.979 (2)
C7—H7B	0.9800	C22—H22A	0.9800
C8—C11	1.522 (3)	C22—H22B	0.9800
C8—C9	1.529 (3)	C23—C24	1.571 (2)
C8—C10	1.538 (3)	C23—H23A	0.9800
C9—H9A	0.9700	C23—H23B	0.9800
C9—H9B	0.9700	C24—C27	1.529 (3)
C9—H9C	0.9700	C24—C25	1.537 (3)
C10—H10A	0.9700	C24—C26	1.539 (3)
C10—H10B	0.9700	C25—H25A	0.9700
C10—H10C	0.9700	C25—H25B	0.9700
C11—O11	1.186 (3)	C25—H25C	0.9700
C11—O12	1.326 (3)	C26—H26A	0.9700
O12—C13	1.446 (3)	C26—H26B	0.9700
C13—H13A	0.9700	C26—H26C	0.9700
C13—H13B	0.9700	C27—O27	1.196 (2)



C13—H13C	0.9700	C27—O28	1.330 (2)
C14—Br1	1.979 (2)	O28—C29	1.446 (2)
C14—H14A	0.9800	C29—H29A	0.9700
C14—H14B	0.9800	C29—H29B	0.9700
C15—C16	1.568 (3)	C29—H29C	0.9700
C15—H15A	0.9800	C30—Br3	1.9719 (19)
C15—H15B	0.9800	C30—H30A	0.9800
C16—C17	1.528 (3)	C30—H30B	0.9800
C16—C19	1.530 (3)		
C6—C1—C2	119.19 (16)	C18—C16—C15	106.64 (17)
C6—C1—C7	121.01 (16)	C16—C17—H17A	109.5
C2—C1—C7	119.78 (16)	C16—C17—H17B	109.5
C1—C2—C3	120.94 (16)	H17A—C17—H17B	109.5
C1—C2—C14	119.17 (16)	C16—C17—H17C	109.5
C3—C2—C14	119.64 (16)	H17A—C17—H17C	109.5
C4—C3—C2	119.07 (16)	H17B—C17—H17C	109.5
C4—C3—C15	120.24 (16)	C16—C18—H18A	109.5
C2—C3—C15	120.68 (16)	C16—C18—H18B	109.5
C3—C4—C5	120.70 (16)	H18A—C18—H18B	109.5
C3—C4—C22	119.56 (16)	C16—C18—H18C	109.5
C5—C4—C22	119.50 (16)	H18A—C18—H18C	109.5
C4—C5—C6	119.43 (16)	H18B—C18—H18C	109.5
C4—C5—C23	120.09 (16)	O19—C19—O20	121.9 (2)
C6—C5—C23	120.47 (16)	O19—C19—C16	124.3 (2)
C1—C6—C5	120.50 (16)	O20—C19—C16	113.80 (17)
C1—C6—C30	118.93 (16)	C19—O20—C21	115.81 (19)
C5—C6—C30	120.17 (16)	O20—C21—H21A	109.5
C1—C7—C8	114.53 (16)	O20—C21—H21B	109.5
C1—C7—H7A	108.6	H21A—C21—H21B	109.5
C8—C7—H7A	108.6	O20—C21—H21C	109.5
C1—C7—H7B	108.6	H21A—C21—H21C	109.5
C8—C7—H7B	108.6	H21B—C21—H21C	109.5
H7A—C7—H7B	107.6	C4—C22—Br2	109.10 (13)
C11—C8—C9	108.2 (2)	C4—C22—H22A	109.9
C11—C8—C10	108.71 (19)	Br2—C22—H22A	109.9
C9—C8—C10	108.5 (2)	C4—C22—H22B	109.9
C11—C8—C7	105.47 (17)	Br2—C22—H22B	109.9
C9—C8—C7	111.31 (18)	H22A—C22—H22B	108.3
C10—C8—C7	114.43 (18)	C5—C23—C24	115.18 (15)
C8—C9—H9A	109.5	C5—C23—H23A	108.5
C8—C9—H9B	109.5	C24—C23—H23A	108.5
H9A—C9—H9B	109.5	C5—C23—H23B	108.5
C8—C9—H9C	109.5	C24—C23—H23B	108.5
H9A—C9—H9C	109.5	H23A—C23—H23B	107.5
H9B—C9—H9C	109.5	C27—C24—C25	108.36 (16)
C8—C10—H10A	109.5	C27—C24—C26	107.98 (17)
C8—C10—H10B	109.5	C25—C24—C26	108.85 (18)
H10A—C10—H10B	109.5	C27—C24—C23	106.26 (15)
C8—C10—H10C	109.5	C25—C24—C23	111.83 (16)

## supplementary materials

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H10A—C10—H10C	109.5	C26—C24—C23	113.36 (16)
H10B—C10—H10C	109.5	C24—C25—H25A	109.5
O11—C11—O12	123.0 (2)	C24—C25—H25B	109.5
O11—C11—C8	125.7 (3)	H25A—C25—H25B	109.5
O12—C11—C8	111.3 (2)	C24—C25—H25C	109.5
C11—O12—C13	116.5 (3)	H25A—C25—H25C	109.5
O12—C13—H13A	109.5	H25B—C25—H25C	109.5
O12—C13—H13B	109.5	C24—C26—H26A	109.5
H13A—C13—H13B	109.5	C24—C26—H26B	109.5
O12—C13—H13C	109.5	H26A—C26—H26B	109.5
H13A—C13—H13C	109.5	C24—C26—H26C	109.5
H13B—C13—H13C	109.5	H26A—C26—H26C	109.5
C2—C14—Br1	109.75 (13)	H26B—C26—H26C	109.5
C2—C14—H14A	109.7	O27—C27—O28	123.31 (19)
Br1—C14—H14A	109.7	O27—C27—C24	125.74 (19)
C2—C14—H14B	109.7	O28—C27—C24	110.95 (16)
Br1—C14—H14B	109.7	C27—O28—C29	117.52 (17)
H14A—C14—H14B	108.2	O28—C29—H29A	109.5
C3—C15—C16	116.98 (15)	O28—C29—H29B	109.5
C3—C15—H15A	108.1	H29A—C29—H29B	109.5
C16—C15—H15A	108.1	O28—C29—H29C	109.5
C3—C15—H15B	108.1	H29A—C29—H29C	109.5
C16—C15—H15B	108.1	H29B—C29—H29C	109.5
H15A—C15—H15B	107.3	C6—C30—Br3	109.25 (12)
C17—C16—C19	111.47 (18)	C6—C30—H30A	109.8
C17—C16—C18	109.34 (19)	Br3—C30—H30A	109.8
C19—C16—C18	106.46 (18)	C6—C30—H30B	109.8
C17—C16—C15	113.15 (17)	Br3—C30—H30B	109.8
C19—C16—C15	109.45 (16)	H30A—C30—H30B	108.3

### Hydrogen-bond geometry ( $\text{\AA}$ , $^\circ$ )

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C23—H23A $\cdots$ Br3 <sup>i</sup>	0.98	2.90	3.7875 (19)	151

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



Fig. 2

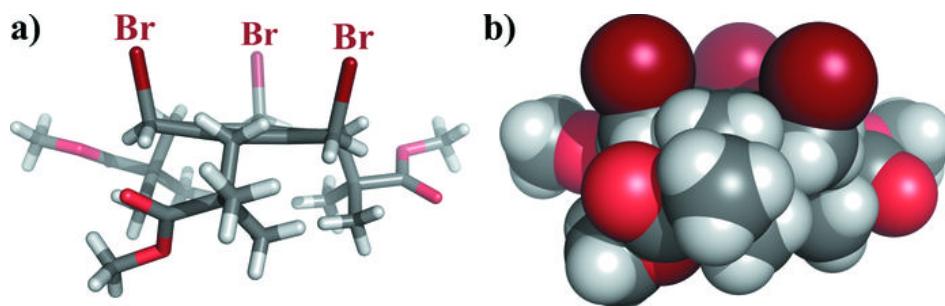


Fig. 3

