

2,2',6,6'-Tetrachloro-4,4'-propane-2,2'-diylidiphenol, 2,2',6-tribromo-4,4'-propane-2,2'-diylidiphenol and 2,2',6,6'-tetrabromo-4,4'-propane-2,2'-diylidiphenol

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Three flame retardants with very similar molecular structures showing three different packing patterns have been studied. The crystal structure of 2,2',6,6'-tetrachloro-4,4'-propane-2,2'-diylidiphenol, $C_{15}H_{12}Cl_4O_2$, can be described as a packing of sheets. The packing shows a very short intermolecular $Cl \cdots Cl$ contact distance of 3.094 (2) Å between pairs of molecules inside each sheet. The crystal structure of 2,2',6-tribromo-4,4'-propane-2,2'-diylidiphenol, $C_{15}H_{13}Br_3O_2$, can be described as a packing of doubly stranded helical square tubes. These square helices are interconnected through $Br \cdots Br$ contacts between different helices. Finally, a previously known structure, 2,2',6,6'-tetrabromo-4,4'-propane-2,2'-diylidiphenol [Simonov, Cheban, Rotaru & Bels'skii (1986). *Kristallografiya*, **31**, 397–399], $C_{15}H_{12}Br_4O_2$, which is the most commonly used flame retardant and which has twofold rotational symmetry, has been refined in the correct absolute configuration. The structure shows large differences from the chloro analogue with regard to packing, van der Waals distances and hydrogen-bond distances.

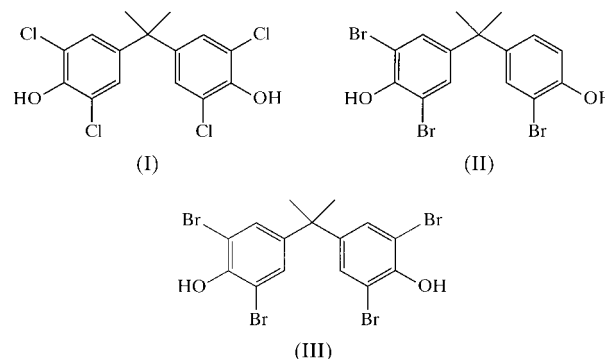
Comment

In recent years, several reports have indicated that the widespread use of flame retardants is responsible for the bioaccumulation of these compounds in nature. The decomposition of flame retardants in the natural environment and the products obtained from these is a long-running project in our department. The goal is a better understanding of the reactivity of these compounds in the environment.

The different substances observed in decomposition experiments that mimic realistic circumstances in the natural environment indicate large differences among the decomposition products, depending on the halogen substitution pattern and on whether the reactant is in aqueous solution or in the solid state.

The title compounds are members of a class collectively known as 'halogenated bisphenols', which are used as reactive flame retardants. This means that they are not only mixed together with a prefabricated plastic material but also take part in the polymerization process, where they are covalently bound into the polymer. The salient feature of reactive flame retardants is thus that they migrate less easily to the environment due to this covalent bonding to the polymer. The corresponding additive flame retardants, which are simply mixed into a pre-produced polymer, migrate more easily (Kuryla & Papa, 1979).

The three title compounds, 2,2',6,6'-tetrachloro-4,4'-propane-2,2'-diylidiphenol, (I), 2,2',6-tribromo-4,4'-propane-2,2'-diylidiphenol, (II) and 2,2',6,6'-tetrabromo-4,4'-propane-2,2'-diylidiphenol, (III), are three flame retardants with very similar molecular structures (Figs. 1–3), but which exhibit very different crystal structures.



The first two title compounds, (I) and (II), represent previously unknown structures, while the third, (III), has been reported previously by Simonov *et al.* (1986). Diffraction data have been remeasured for (III) and we have changed the absolute configuration of the molecule and also the space group from $P4_12_12$ (92) to $P4_32_12$ (96), as these two constitute an enantiomorphic space-group pair. Some anomalous bond distances observed with the refinement in $P4_12_12$ are found to be absent when refining the structure model with the correct absolute configuration in $P4_32_12$.

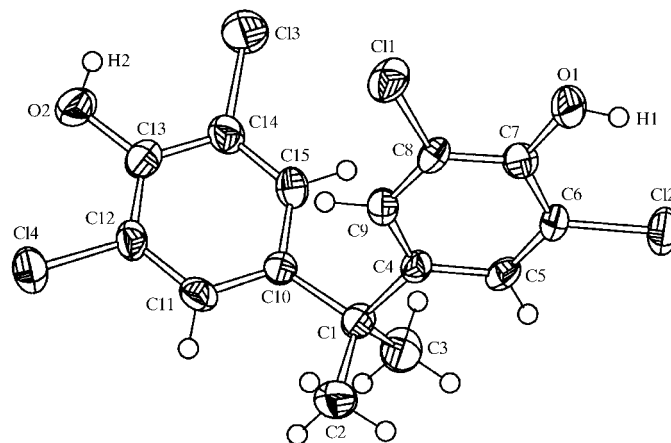


Figure 1
The molecular structure of (I) showing 50% probability displacement ellipsoids. H atoms are shown with an arbitrary radius.

The crystal structure of (I) is built up by a packing of sheets of hydrogen-bonded molecules, further stabilized by a very short intermolecular Cl \cdots Cl contact [Cl1 \cdots Cl1ⁱ = 3.094 (2) Å; symmetry code: (i) 1 - x, -y, -z]. This is shorter than is observed in most of the well determined ($R \leq 10\%$) and non-disordered similar structures in the Cambridge Structural Database (CSD; Allen & Kennard, 1993), for instance, triphenylchloromethane (Dunand & Gerdil, 1982), where the shortest intermolecular Cl \cdots Cl distance is 3.210 Å, which is itself considered to be very short (Desiraju, 1989). A similarly short intermolecular interhalogen distance does not occur in the bromo analogue, (III).

The shortest intermolecular O \cdots O contact distance in (I) [O1 \cdots O2ⁱⁱ = 2.723 (3) Å; symmetry code: (ii) x, y - 1, z] is also shorter than the corresponding shortest intermolecular O \cdots O contact distance in (III) [O \cdots O = 3.11 (1) Å], thus indicating stronger hydrogen bonding between the molecules of (I) along the *b* axis.

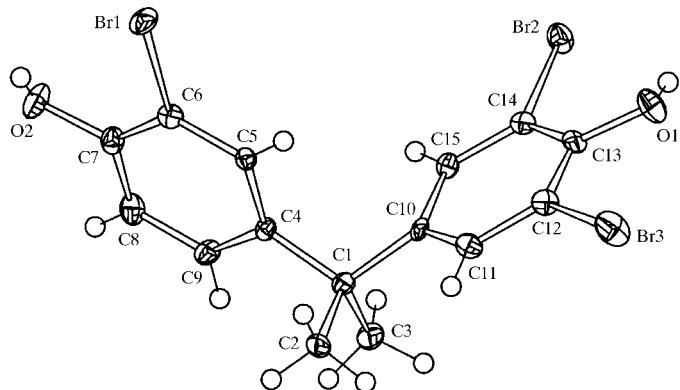


Figure 2
The molecular structure of (II) showing 50% probability displacement ellipsoids. H atoms are shown with an arbitrary radius.

Regarding the close similarity of the molecular structures, a reasonable conjecture would be that tetrachlorobisphenol (I) should have a rather similar structure to the brominated analogue (III). The present investigation shows that this is not the case, neither concerning the space group nor the packing of the molecules. Further investigations will be carried out to elucidate the physical background to the close Cl \cdots Cl contact in (I), and whether it is a consequence or a reason for the packing of (I).

The hydrogen-bonding scheme in (I) cannot be deduced with certainty, since at least two schemes are possible (Fig. 4). The conformations of the four hydroxyl groups are restricted by the space-group symmetry. One can speculate on other hydrogen-bond schemes which break the space-group symmetry, *e.g.* with the four hydroxyl groups pointing towards each other in a circular pattern, or a disordered hydrogen-bond scheme. This possible violation of the space-group symmetry by the H atoms cannot be detected in the diffraction data.

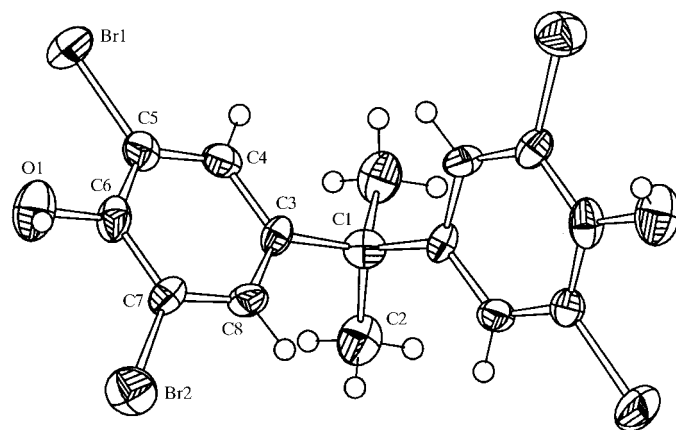


Figure 3
The molecular structure of (III) showing 50% probability displacement ellipsoids. H atoms are shown with an arbitrary radius.

The second compound, (II), differs from the most commonly used flame retardant, (III), only by the lack of one Br atom, yet it crystallizes in a totally different structure. The crystal structure of (II) can be described as a packing of square helices running along the *c* axis. Each helix is built of two strands of hydrogen-bonded molecules of (II), further stabilized by interactions between the halogens and the aromatic ring systems. Three molecules of each strand in the helices are shown in Fig. 5. The two strands fit together to make up a square-type double helix in the *ab* plane. The shortest Br \cdots Br distances (3.71–4.21 Å) correspond to intermolecular Br \cdots Br contacts between different helices.

In contrast with the plausible hydrogen-bonding pattern of (II), no appreciable hydrogen bonding can be deduced from the packing of (III). Here, the molecules preferentially pack in long chains, with interactions between the aromatic rings and

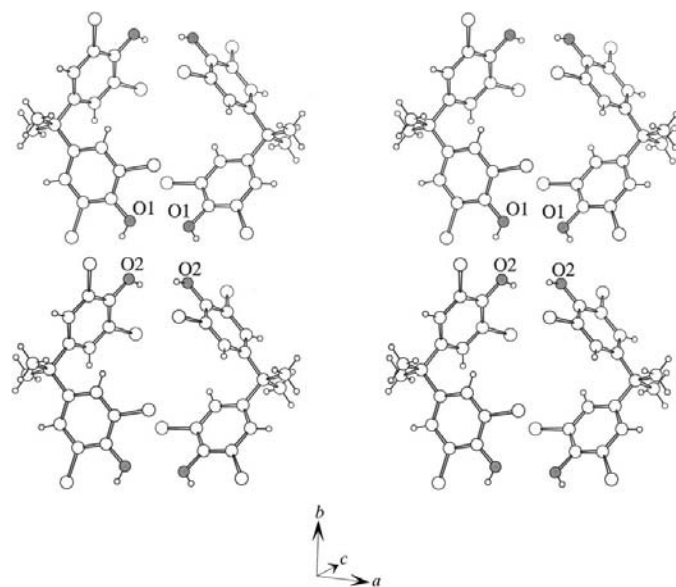


Figure 4
A stereoview of the square pattern of the O atoms from four molecules of (I), illustrating the possibilities for hydrogen bonding. The H-atom positions shown were optimized from the geometrically calculated positions.

the aliphatic central part of the molecule, to give the packing pattern shown in Fig. 6, which is completely different from that in (II). The rather long intermolecular O...O contacts in (III) (≥ 3.1 Å) may contribute to a minor stabilization of the structure of (III). The reported position for atom H1 involved in the possible hydrogen bond is that derived from the least-squares calculations. A geometrically computed position for H1 (0.2458, 0.2955, 0.0487) gives a linear link to the plausible acceptor O1ⁱⁱⁱ [symmetry code: (iii) $y, x, -z$]. Atom H1 and the symmetry-related H1 atom cannot both be linearly directed to the corresponding acceptor, but there is a possibility of a H-atom positional disorder around atom O1, giving some slight stabilization due to intermolecular hydrogen bonds in (III).

Compound (II) exhibits much shorter intermolecular O...O distances compared with (III), so the contribution from hydrogen bonds to the stabilization of the structure is probably much larger for (II) than for (III). Similar Br...Br contact distances exist in both (II) and (III). In (II), the intermolecular Br...Br distances are greater than 3.71 Å; in (III), these distances are greater than 3.93 Å. This also indicates slightly stronger intermolecular bonding involving the Br...Br contacts for (II) compared with (III).

Both rings of (I) are planar, to within 0.013 Å for C4–C9 and 0.010 Å for C10–C15. Atom Cl1, which is involved in the very short intermolecular Cl...Cl contact, is within 0.007 (5) Å of the plane defined by the C atoms of the aromatic ring. Atom O1 of the C4–C9 ring has the largest deviation from the ring plane. This can be interpreted as originating from effects by the short intermolecular O1...O2ⁱⁱ

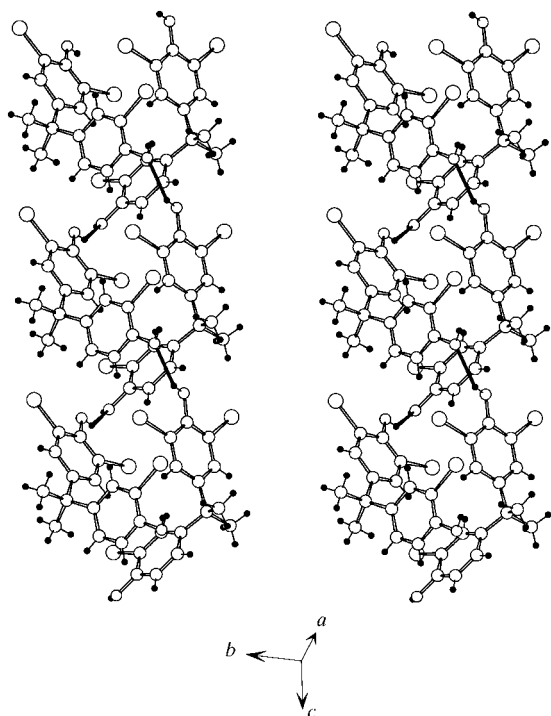


Figure 5

A stereoview of the three molecules of (II) in each strand of the two-stranded helix.

contact. The substituents of the second ring deviate more from the ring plane; deviations are 0.103 (5) Å for Cl4, 0.037 (5) Å for O2 and 0.098 (6) Å for Cl3. The angle between the two ring planes is 64.0 (1)° and this is smaller than corresponding interplanar angles in most of the similar structures from the CSD. There are seven structures with the 4,4'-propane-2,2-diyldiphenol skeleton available in the CSD. One exhibits an interplanar angle of 64°, but the others have interplanar angles in the range 72–96°. The compound with the smallest angle, 2,2',6,6'-tetranitro-4,4'-isopropylidenediphenol (CSD refcode BIDJED; Wang *et al.*, 1982) is also heavily affected by intermolecular bonding effects, similar to those present in (I). Smaller interplanar angles shown by similar compounds from the CSD are only present when either heavily steric interactions occur or a direct covalent bond restricts the conformation of the different rings.

Both rings of (II) are planar, to within 0.007 Å for C4–C9 and 0.02 Å for C10–C15. The ring with two Br substituents is more puckered than that with only one Br substituent. Most noteworthy are the deviations from the ring plane of the two hydroxyl O atoms, *viz.* 0.108 (7) Å for O1 and 0.019 (7) Å for O2. These deviations can be described as a function of steric interaction. Atom O2 is pushed away by the close contact from Br1 and stays approximately in the ring plane, while atom O1 is pushed out of the ring plane, as it is situated between two close Br substituents (Br2 and Br3). It is rather strange that the angular distortion found for O2 is also present for O1; the difference is mostly the out-of-plane deviation of O1. Both ring planes are defined solely by their C atoms; the angle between the two ring planes is 81.8 (2)°.

The ring of (III) is planar to within 0.011 Å (C3–C8). The two atoms deviating most from this plane are Br1 [0.022 (1) Å] and Br2 [0.118 (10) Å]. In this compound, the angular

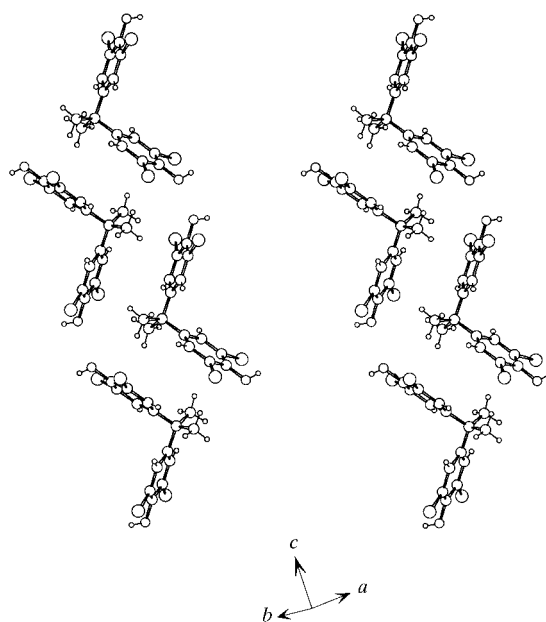


Figure 6

A stereoview of one of the chains of (III) dominated by hydrophobic interactions.

distortion of atom O1 with respect to the ring is much less than the corresponding distortions in the dibromo-substituted ring of (II). The ring plane is defined solely by the C atoms of the ring. The angle between the ring plane and the plane of the symmetry-related ring is 80.2 (2)°.

Experimental

Compounds (I) and (III) are commercially available (Aldrich Chemical Co., Milwaukee, USA). Compound (II) was synthesized by the bromination of bisphenol A (4,4'-propane-2,2-diylidiphenol). All three compounds were recrystallized from ethanol at ambient temperature.

Compound (I)

Crystal data

C₁₅H₁₂Cl₄O₂
M_r = 366.05
 Orthorhombic, *Pbcn*
a = 25.675 (9) Å
b = 11.602 (3) Å
c = 10.530 (4) Å
V = 3136.6 (17) Å³
Z = 8
D_x = 1.550 Mg m⁻³

Data collection

Stoe AED-2 diffractometer
 $\theta/2\theta$ scans
 Absorption correction: numerical
 (*X-RED*; Stoe, 1997)
T_{min} = 0.843, *T_{max}* = 0.906
 3572 measured reflections
 2771 independent reflections
 1267 reflections with *I* > 2σ(*I*)

Refinement

Refinement on *F*²
R[*F*² > 2σ(*F*²)] = 0.054
wR(*F*²) = 0.075
S = 1.07
 2771 reflections
 191 parameters
 H-atom parameters constrained

Mo *K*α radiation
 Cell parameters from 56 reflections
 θ = 7.5–10.7°
 μ = 0.75 mm⁻¹
T = 293 (2) K
 Prism, light yellow
 0.23 × 0.16 × 0.13 mm

*R*_{int} = 0.061
 θ_{\max} = 25°
h = -1 → 30
k = -1 → 13
l = -1 → 12
 4 standard reflections
 frequency: 90 min
 intensity decay: <1%

$w = 1/[\sigma^2(F_o^2) + (0.01P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
 (Δ/σ)_{max} = 0.001
 $\Delta\rho_{\max} = 0.28 \text{ e } \text{Å}^{-3}$
 $\Delta\rho_{\min} = -0.32 \text{ e } \text{Å}^{-3}$
 Extinction correction: *SHELXL97*
 (Sheldrick, 1997)
 Extinction coefficient: 0.00102 (11)

Table 1

Selected geometric parameters (Å, °) for (I).

C11–C8	1.734 (4)	C13–C14	1.736 (4)
C12–C6	1.732 (4)	C14–C12	1.732 (4)
C4–C1–C10	108.1 (3)	C9–C4–C1	120.5 (3)
C4–C1–C3	112.3 (4)	O1–C7–C8	120.3 (4)
C10–C1–C3	109.2 (3)	O1–C7–C6	123.0 (4)
C4–C1–C2	106.8 (3)	C11–C10–C15	117.9 (4)
C10–C1–C2	111.7 (3)	C11–C10–C1	124.0 (4)
C3–C1–C2	108.8 (3)	C15–C10–C1	118.1 (3)
C5–C4–C9	116.4 (4)	O2–C13–C12	120.1 (4)
C5–C4–C1	123.0 (3)	O2–C13–C14	122.8 (4)
C10–C1–C4–C5	-148.3 (4)	C2–C1–C4–C9	-87.5 (4)
C3–C1–C4–C5	-27.8 (5)	C9–C4–C5–C6	-2.3 (6)
C2–C1–C4–C5	91.4 (5)	C1–C4–C5–C6	178.8 (3)
C10–C1–C4–C9	32.9 (5)	C15–C10–C11–C12	-2.8 (6)
C3–C1–C4–C9	153.3 (4)	C1–C10–C11–C12	174.8 (4)

Table 2

Hydrogen-bonding geometry (Å, °) for (I).

<i>D</i> –H... <i>A</i>	<i>D</i> –H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> –H... <i>A</i>
O1–H1...O2 ⁱⁱ	0.82	2.113	2.723 (3)	131
O2–H2...O2 ^{iv}	0.82	2.221	2.773 (5)	125

Symmetry codes: (ii) *x*, *y* – 1, *z*; (iv) 1 – *x*, *y*, $\frac{1}{2}$ – *z*.

Compound (II)

Crystal data

C₁₅H₁₃Br₃O₂
M_r = 464.98
 Orthorhombic, *Fdd2*
a = 19.545 (3) Å
b = 30.971 (4) Å
c = 10.0335 (19) Å
V = 6073.6 (16) Å³
Z = 16
D_x = 2.034 Mg m⁻³

Mo *K*α radiation
 Cell parameters from 1230 reflections
 θ = 1.7–26.1°
 μ = 7.97 mm⁻¹
T = 293 (2) K
 Prism, colourless
 0.31 × 0.27 × 0.22 mm

Data collection

Stoe IPDS area-detector diffractometer
 Area detector scans
 Absorption correction: numerical
 (*X-RED*; Stoe, 1997)
T_{min} = 0.086, *T_{max}* = 0.167
 10 060 measured reflections

1559 independent reflections
 1500 reflections with *I* > 2σ(*I*)
*R*_{int} = 0.066
 θ_{\max} = 25.9°
h = -23 → 23
k = -36 → 37
l = -12 → 12

Refinement

Refinement on *F*²
R[*F*² > 2σ(*F*²)] = 0.026
wR(*F*²) = 0.063
S = 1.16
 1559 reflections
 185 parameters

H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.04P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
 (Δ/σ)_{max} = 0.001
 $\Delta\rho_{\max} = 1.50 \text{ e } \text{Å}^{-3}$
 $\Delta\rho_{\min} = -0.41 \text{ e } \text{Å}^{-3}$

Table 3

Selected geometric parameters (Å, °) for (II).

Br1–C6	1.903 (5)	C1–C4	1.528 (7)
Br2–C14	1.905 (5)	C1–C3	1.537 (7)
Br3–C12	1.900 (5)	C1–C2	1.541 (7)
C1–C10	1.520 (7)		
C10–C1–C4	112.0 (4)	C5–C4–C1	121.0 (5)
C10–C1–C3	106.2 (4)	O2–C7–C8	117.1 (5)
C4–C1–C3	111.8 (5)	O2–C7–C6	124.4 (5)
C10–C1–C2	112.3 (4)	C15–C10–C11	117.1 (5)
C4–C1–C2	106.6 (4)	C15–C10–C1	120.1 (4)
C3–C1–C2	107.9 (5)	C11–C10–C1	122.6 (5)
C9–C4–C5	117.4 (5)	O1–C13–C12	118.8 (5)
C9–C4–C1	121.4 (5)	O1–C13–C14	124.4 (5)
C10–C1–C4–C9	144.8 (5)	C2–C1–C4–C5	81.8 (6)
C3–C1–C4–C9	25.8 (7)	C9–C4–C5–C6	1.0 (7)
C2–C1–C4–C9	-92.0 (6)	C1–C4–C5–C6	-173.1 (5)
C10–C1–C4–C5	-41.3 (6)	C15–C10–C11–C12	-3.1 (8)
C3–C1–C4–C5	-160.4 (5)	C1–C10–C11–C12	171.1 (5)

Table 4

Hydrogen-bonding geometry (Å, °) for (II).

<i>D</i> –H... <i>A</i>	<i>D</i> –H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> –H... <i>A</i>
O1–H1...O2 ^v	0.82	2.18	2.818 (5)	134

Symmetry code: (v) $\frac{1}{2}$ – *x*, $\frac{1}{2}$ – *y*, *z* – 1.

Compound (III)

Crystal data

$C_{15}H_{12}Br_4O_2$	Mo $K\alpha$ radiation
$M_r = 543.89$	Cell parameters from 1720 reflections
Tetragonal, $P4_32_12$	$\theta = 1.7\text{--}26.1^\circ$
$a = 12.0038$ (16) Å	$\mu = 9.62$ mm ⁻¹
$c = 11.618$ (3) Å	$T = 293$ (2) K
$V = 1674.1$ (5) Å ³	Prism, colourless
$Z = 4$	$0.15 \times 0.14 \times 0.12$ mm
$D_x = 2.158$ Mg m ⁻³	

Data collection

Stoe IPDS area-detector diffractometer	990 independent reflections (plus 635 Friedel-related reflections)
Area-detector scans	915 reflections with $I > 2\sigma(I)$
Absorption correction: numerical (<i>X-RED</i> ; Stoe, 1997)	$R_{int} = 0.171$
$T_{min} = 0.236$, $T_{max} = 0.302$	$\theta_{max} = 25.9^\circ$
13 032 measured reflections	$h = -14 \rightarrow 14$
	$k = -14 \rightarrow 14$
	$l = -14 \rightarrow 14$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.01P)^2]$
$R[F^2 > 2\sigma(F^2)] = 0.039$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.060$	$(\Delta/\sigma)_{max} = 0.001$
$S = 0.81$	$\Delta\rho_{max} = 0.42$ e Å ⁻³
1625 reflections	$\Delta\rho_{min} = -0.39$ e Å ⁻³
98 parameters	Absolute structure: Flack (1983)
H-atom parameters constrained	Flack parameter = 0.03 (3)

Table 5

Selected geometric parameters (Å, °) for (III).

Br1—C5	1.877 (7)	C1—C3	1.532 (9)
Br2—C7	1.862 (7)	C1—C2	1.533 (9)
C3—C1—C3 ^{vi}	108.3 (8)	C4—C3—C1	123.6 (6)
C3—C1—C2 ^{vi}	113.1 (4)	C8—C3—C1	120.5 (6)
C3—C1—C2	107.9 (5)	O1—C6—C7	121.1 (8)
C2 ^{vi} —C1—C2	106.6 (10)	O1—C6—C5	119.6 (8)
C4—C3—C8	115.8 (7)		
C3 ^{vi} —C1—C3—C4	111.5 (8)	C2 ^{vi} —C1—C3—C8	172.6 (8)
C2 ^{vi} —C1—C3—C4	-8.0 (10)	C2—C1—C3—C8	54.9 (9)
C2—C1—C3—C4	-125.7 (7)	C8—C3—C4—C5	-1.7 (11)
C3 ^{vi} —C1—C3—C8	-67.9 (6)	C1—C3—C4—C5	178.9 (7)

Symmetry code: (vi) $y, x, 1 - z$.

Table 6

Hydrogen-bonding geometry (Å, °) for (III).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
O1—H1 ⁱⁱⁱ —O1 ⁱⁱⁱ	0.82	3.13	3.11 (1)	81

Symmetry code: (iii) $y, x, -z$.

For (I), the H atoms were placed geometrically. The hydroxyl groups were allowed to rotate freely around the C—O bond using the *AFIX83* instruction in *SHELXL97* (Sheldrick, 1997). The hydroxyl H atoms converged to a position that could be interpreted as a favourable conformation for the formation of a hydrogen-bonded chain of molecules. For (II), the space group was determined from reflection conditions, indicating the unique space group *Fdd2* (43). The Flack (1983) parameter was refined as a scale factor of this model and of the inverted model. The refined value of the Flack parameter in this model was 0.49 (2); thus, a merging of Friedel-related reflections was performed for the final model. The total

number of 2936 reflections gave 1559 unique reflections plus 1377 Friedel-related equivalents. The merging of reflection data improved *wR2* and *R1* without affecting s.u.'s by more than approximately 10%. All coordinates and derived distances *etc.* were equal to within 1 s.u. of the corresponding quantities. The highest residual electron-density peak (1.50 e Å⁻³) was located at (0.0814, 0.0417, 0.4149), 1.16 Å from H8, and should not be interpreted as an additional atom. Compound (II) is a strong absorber of Mo $K\alpha$ radiation. Thus, a possible cause for this residual peak is an imperfect absorption correction. The applied absorption correction decreased the internal *R* value from 0.25 to 0.06, but the remaining absorption effects could be an explanation for the positive ghost peak. However, the residual peak can also be interpreted as a ripple in the residual density map. For (III), the high internal *R* value is, to a large extent, dependent on the weak scattering from the crystal. Thus, R_{int} is dominated by a large fraction of weak reflections. Of 1625 unique reflections in total, only 539 fulfil the criterion $I \geq 3\sigma(I)$. The internal *R* value calculated from these 539 reflections is 0.063; the corresponding *wR2* value of 0.046 and *R1* value of 0.042 indicate insignificant differences to the refinement with all reflections present. The applied absorption correction did not affect the internal *R* value as much, but *wR2* and *R1* were significantly lowered, as expected. The total number of reflections (1625) was composed of 990 unique reflections and 635 Friedel equivalents. The Friedel equivalents were not averaged, as the four Br atoms of each molecule give a considerable contribution to anomalous dispersion effects and no signs of twinning were detectable. The present model was refined in *P4₃2₁2* (96), instead of the inverse space group *P4₁2₁2* (92) previously used by Simonov *et al.* (1986). The Flack parameter clearly indicates that *P4₃2₁2* is the correct space group. This change in space group eliminates some of the anomalous bonding distances previously reported by Simonov *et al.* (1986). For all three compounds, C—H distances were constrained to 0.93–0.96 Å, O—H distances to 0.82 Å and $U_{iso}(H)$ to 1.2 or 1.5 times U_{eq} of the parent atom.

For compound (I), data collection: *DIF4* (Stoe, 1988); cell refinement: *DIF4*; data reduction: *REDU4* (Stoe, 1988). For compounds (II) and (III), data collection: *EXPOSE* (Stoe, 1997); cell refinement: *CELL* (Stoe, 1997); data reduction: *INTEGRATE* (Stoe, 1997). For all three compounds, program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97*; molecular graphics: *DIAMOND* (Bergerhoff, 1996).

Supplementary data for this paper are available from the IUCr electronic archives (Reference: OS1139). Services for accessing these data are described at the back of the journal.

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