

Secondary Interactions in Bromomethyl-substituted Benzenes: Crystal Structures of Three α,α' -Bis-bromoxylenes, 1,2,3,5-Tetrakis(bromomethyl)benzene, and 1,2,4,5-Tetrakis(bromomethyl)benzene

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Z. Naturforsch. 2007, 62b, 725–731; received November 3, 2006

X-Ray structure determinations of all three isomers of bis(bromomethyl)benzene and of two isomeric tetrakis(bromomethyl)benzenes show that the packing of the molecules is determined principally by interactions of the bromomethyl groups ($\text{C-H}\cdots\text{Br}$ and $\text{Br}\cdots\text{Br}$), except for *ortho*-bis(bromomethyl)benzene, in which $\text{C-H}\cdots\pi$ interactions play a major role.

Key words: α,α' -Bis-bromoxylenes, X-Ray, “Weak” Hydrogen Bonds, Halogen-Halogen Contacts

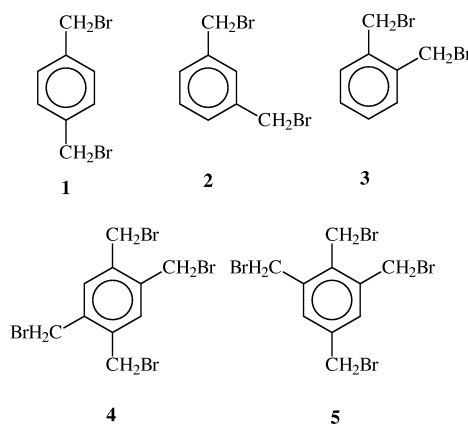
Introduction

Our continued interest in the influence of bromine atoms on the packing of organic molecules in crystal structures has led us to investigate systematically a series of poly(bromomethyl)-substituted arenes. Our previous reports concerned two bromine derivatives: 1,6,7-tris(bromomethyl)naphthalene [1] and 2,2''-bis(bromomethyl)-*p*-terphenyl [2], in which “weak” hydrogen bonds $\text{C-H}\cdots\text{Br}$ and (for the naphthalene derivative only) short $\text{Br}\cdots\text{Br}$ interactions were observed.

At r. t., the bis- and tetrakis-(bromomethyl)benzene derivatives are solids that readily form crystals suitable for X-ray studies. Here we report crystal structures of all three isomeric forms of the disubstituted compound (1–3), together with two of the three possible isomers (4, 5) of the tetrasubstituted compound.

Results and Discussion

The individual molecules of 1–5 are shown in Figs. 1–5. The *para* derivative 1 and the *ortho* derivative 3 crystallize with imposed inversion and twofold symmetry, respectively; compound 4 also displays inversion symmetry. The ring angles at the substituted atoms are consistently less than 120° (by *ca.* 1°) except for compound 5; otherwise, bond lengths and angles may be considered normal, *e. g.* the narrow



Formula scheme.

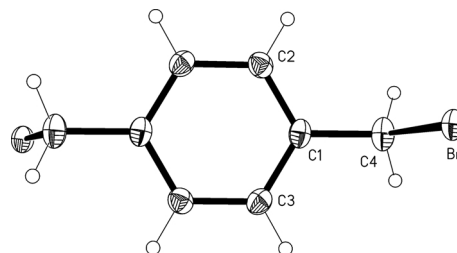


Fig. 1. The molecule of compound 1 in the crystal. Ellipsoids at the 50 % probability level.

range of C–Br bond lengths of 1.974–1.982 Å over all five compounds (for individual values, the Supplemen-

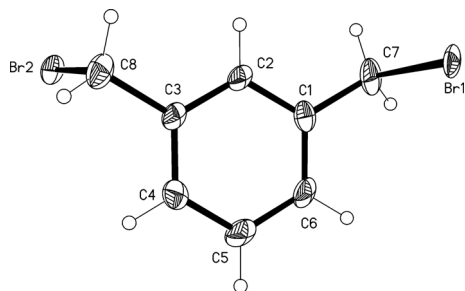


Fig. 2. The molecule of compound **2** in the crystal. Ellipsoids at the 50 % probability level.

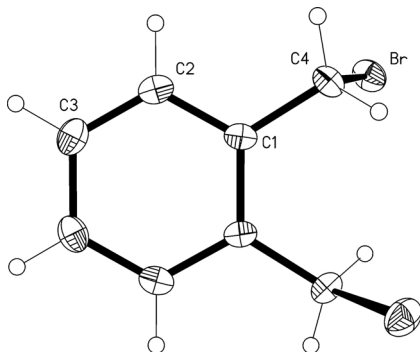


Fig. 3. The molecule of compound **3** in the crystal. Ellipsoids at the 50 % probability level. Intramolecular H bonds are omitted.

tary Material may be consulted). There are no significant deviations from planarity of the aromatic rings, in contrast to the previously (1965) reported structure of hexa(bromomethyl)benzene [3], where ring torsion angles were given as $\pm 6^\circ$, but this structure should be reinvestigated with modern methods. The bromine atoms in **1–3** are disposed to opposite sides of the ring plane (“*anti*”). On theoretical grounds, there is no obstacle to a “*syn*” configuration, and this was indeed described for 2,3-bis(bromomethyl)-1,4-dimethoxybenzene [4]. In compound **4**, the bromine atoms at C1,C3',C1',C3 (1,2,4,5-substitution) are disposed with respect to the ring plane in the manner $++--$ (one *ortho* pair is found above the ring plane and the other one below), and in compound **5** for C1,C2,C3,C5 with the pattern $+-+-$. All absolute torsion angles C–C–C–Br lie in the range $80–90^\circ$; a search of the Cambridge Database [5] revealed 15 hits for bromomethyl-substituted six-membered aromatic rings in compounds containing only C, H and Br, and all 49 absolute C–C–C–Br torsion angles lie within 20° of the ideal value 90° .

The main interest in these structures, however, centres on the molecular packing. Table 3 lists the “weak”

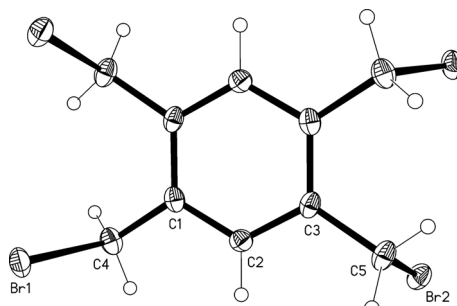


Fig. 4. The molecule of compound **4** in the crystal. Ellipsoids at the 50 % probability level.

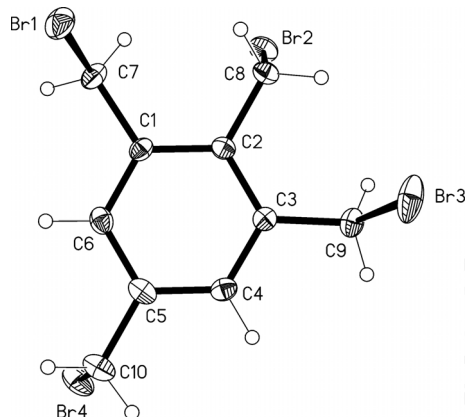


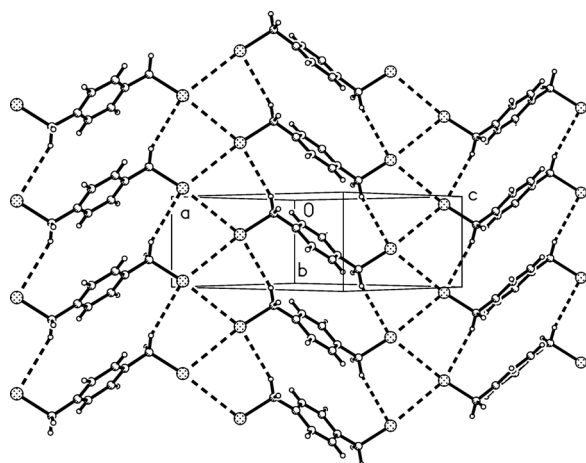
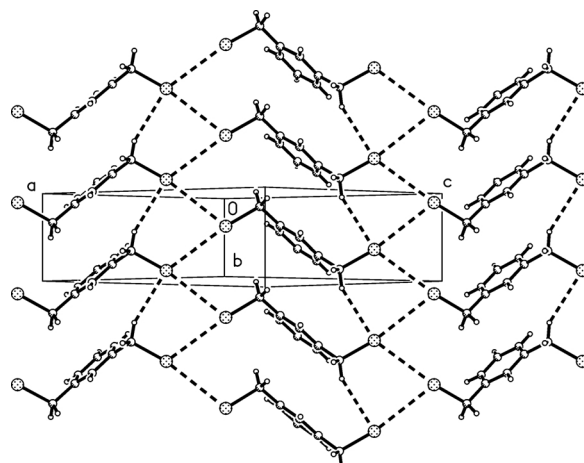
Fig. 5. The molecule of compound **5** in the crystal. Ellipsoids at the 50 % probability level.

C–H $\cdots$ Br hydrogen bonds and Table 4 the Br $\cdots$ Br interactions [6] (numbers given in parentheses in the present discussion refer to the numbered interactions in the Tables). In the *para* derivative **1** a layer structure parallel to [101] is produced, *via* 2_1 and translation symmetry. To a first approximation only the bromomethyl groups are involved, giving rise to one C–H $\cdots$ Br (1) and two Br $\cdots$ Br interactions (1, 2), in each case the shortest of each type*; the longer interactions (not shown in the Figure) link neighboring layers. The *meta* derivative **2**, despite its different substitution pattern and lack of crystallographic symmetry, displays a strikingly similar layer structure to that of **1** (Fig. 7), *cf.* the short *b* axes in both compounds. The two shortest Br $\cdots$ Br interactions (1, 2) are again in-

*We note that the Br $\cdots$ Br contacts fall into two groups according to length, namely 3.5–3.7 and 3.9 Å or longer (*cf.* double van der Waals' radius = 3.7 Å). In general, we discuss the structural role of the shorter contacts and merely present the longer ones in Table 3 for completeness, but a small net stabilization arising from the longer contacts cannot be ruled out.

Table 1. Crystallographic details for compounds **1**–**5**.

Compound	1	2	3	4	5
Formula	C ₈ H ₈ Br ₂	C ₈ H ₈ Br ₂	C ₈ H ₈ Br ₂	C ₁₀ H ₁₀ Br ₄	C ₁₀ H ₁₀ Br ₄
<i>M_r</i>	263.96	263.96	263.96	449.82	449.82
Crystal habit	colourless tablet	colourless block	colourless tablet	colourless tablet	colourless tablet
Size, mm ³	0.20 × 0.12 × 0.07	0.16 × 0.14 × 0.11	0.20 × 0.18 × 0.10	0.27 × 0.20 × 0.13	0.20 × 0.16 × 0.12
Crystal system	monoclinic	monoclinic	orthorhombic	monoclinic	monoclinic
Space group	<i>P</i> 2 ₁ / <i>c</i>	<i>P</i> 2 ₁ / <i>n</i>	<i>F</i> dd2	<i>P</i> 2 ₁ / <i>c</i>	<i>P</i> 2 ₁ / <i>n</i>
Cell constants:					
<i>a</i> , Å	8.7082(8)	12.9120(9)	13.5750(8)	6.8782(8)	11.7856(14)
<i>b</i> , Å	4.5987(4)	4.4794(3)	15.6938(11)	9.8783(11)	7.8904(11)
<i>c</i> , Å	10.5938(8)	14.9640(11)	7.9596(6)	9.1387(11)	13.980(2)
β , deg	99.688(4)	94.495(3)	90	103.536(4)	95.069(4)
<i>V</i> , Å ³	418.19(6)	862.83(10)	1695.7(2)	603.68(12)	1295.0(3)
<i>Z</i>	2	4	8	2	4
<i>D_x</i> , g cm ^{−3}	2.096	2.032	2.068	2.475	2.307
μ , mm ^{−1}	9.6	9.3	9.5	13.3	12.4
<i>F</i> (000), e	252	504	1008	420	840
<i>T</i> (K)	133(2)	133(2)	133(2)	133(2)	133(2)
2 θ _{max}	60	56.6	60	60	61
No. of reflections:					
Refl. meas.	8281	16242	7522	12406	26116
Refl. unique	1224	2122	1239	1754	3957
<i>R</i> _{int}	0.0388	0.0969	0.0301	0.0503	0.0325
Parameters	46	91	46	64	127
Restraints	0	0	1	0	0
<i>wR</i> (<i>F</i> ² , all refl.)	0.051	0.187	0.043	0.065	0.079
<i>R</i> [<i>F</i> ≥ 4 σ (<i>F</i>)]	0.022	0.070	0.017	0.025	0.034
<i>S</i>	1.008	1.152	1.008	1.022	1.059
Max. $\Delta\rho$, e Å ^{−3}	0.591	2.588	0.337	1.091	1.922

Fig. 6. Packing diagram of compound **1** viewed perpendicular to [101]. Dashed bonds indicate hydrogen bonding or bromine-bromine interactions (see text).Fig. 7. Packing diagram of compound **2** viewed perpendicular to [101]. Dashed bonds indicate hydrogen bonding or bromine-bromine interactions (see text).

volved and form an analogous pattern in both structures. The Br...Br contacts within the layers correspond well to type II according to the classification of Ref. [6], with C–Br...Br angles for a given contact of approximately 90° and 180°; this is thought to imply a

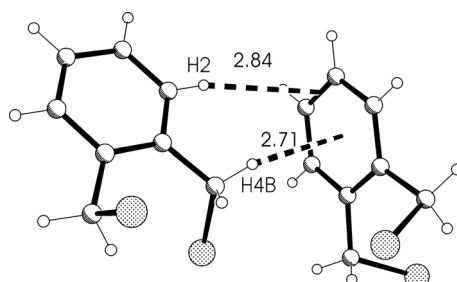
weak bonding attraction. The contacts between layers are type I (equal angles), which are not thought to be bonding in nature. The main topological difference between the two layers is that, for compound **2**, only the methylene group at C7 forms a C–H...Br hydrogen

Table 2. Hydrogen bonds (Å and deg) for compounds **1–5**^a.

Com- pound	H bond no.	D–H...A	<i>d</i> (H...A)	<i>d</i> (D...A)	∠(DHA)
1 ^b	1	C4–H4B...Br#1	3.04	3.914(2)	138
	2	C4–H4A...Br#2	3.13	4.077(2)	146
	3	C2–H2...Br#3 ^g	3.09	4.078(2)	152
	4	C2–H2...Br#4 ^g	3.15	3.956(2)	132
2 ^c	1	C7–H7B...Br1#1	2.96	3.915(9)	148
	2	C8–H8A...Br2#2	2.96	4.020(9)	168
	3	C5–H5...Br2#3 ^g	3.01	3.846(9)	135
	4	C5–H5...Br1#4 ^g	3.02	3.928(8)	142
3 ^d	1	C4–H4A...Br#1 ^h	2.85	3.653(2)	131
	2	C3–H3...Br#2 ^g	3.07	3.973(2)	141
	3	C3–H3...Br#3 ^g	3.05	3.726(2)	121
	4	C4–H4B...[f070]#4 ⁱ	2.71	3.72	156
	5	C2–H2...Midpt (C3–C3')#4	2.84	3.89	163
4 ^e	1	C4–H4B...Br1#1	2.87	3.940(3)	172
	2	C5–H5A...Br1#2 ^g	2.94	3.880(3)	146
	3	C5–H5B...Br1#3	3.10	3.958(3)	137
	4	C2–H2...Br2#4	3.11	4.066(3)	148
	5	C4–H4A...Br2#4	3.02	3.862(3)	135
	6	C5–H5A...Br2#5 ^g	3.15	3.958(3)	132
5 ^f	1	C7–H7A...Br1#1 ^j	3.10	3.672(3)	113
	2	C10–H10B...Br1#2	2.98	4.046(4)	170
	3	C7–H7A...Br3#3 ^j	2.93	3.541(3)	116
	4	C9–H9B...Br3#4 ^g	3.05	3.571(3)	110
	5	C9–H9B...Br2#7 ^g	3.22	4.238(3)	158
	6	C10–H10A...Br3#2	2.94	3.975(4)	160
	7	C7–H7A...Br4#5 ^j	2.97	3.848(3)	138
	8	C8–H8B...Br4#6	2.90	3.804(3)	141
	9	C9–H9A...Br4#7	2.86	3.909(3)	165

^a All C–H bond lengths normalized to 1.08 Å; ^b symmetry transformations used to generate equivalent atoms: #1 *x*, *y* + 1, *z*; #2 *x*, 1/2 – *y*, –1/2 + *z*; #3 –*x*, 1 – *y*, 1 – *z*; #4 *x*, 1/2 – *y*, –1/2 + *z*; ^c symmetry transformations used to generate equivalent atoms: #1 *x*, –1 + *y*, *z*; #2 1/2 – *x*, 1/2 + *y*, 1/2 – *z*; #3 1 – *x*, 1 – *y*, 1 – *z*; #4 1/2 – *x*, –1/2 + *y*, 1/2 – *z*; ^d symmetry transformations used to generate equivalent atoms: #1 1 – *x*, 1 – *y*, *z*; #2 *x*, *y*, –1 + *z*; #3 3/4 – *x*, –1/4 + *y*, –3/4 + *z*; #4 –1/4 + *x*, 3/4 – *y*, 1/4 + *z*; ^e symmetry transformations used to generate equivalent atoms: #1 *x*, 1/2 – *y*, –1/2 + *z*; #2 *x*, 1/2 – *y*, 1/2 + *z*; #3 –*x*, 1/2 + *y*, 1/2 – *z*; #4 1 – *x*, –1/2 + *y*, 1/2 – *z*; #5 *x*, 1/2 – *y*, 1/2 + *z*; ^f symmetry transformations used to generate equivalent atoms: #1 1/2 – *x*, 1/2 + *y*, 1/2 – *z*; #2 1 – *x*, 1 – *y*, 1 – *z*; #3 *x*, 1 + *y*, *z*; #4 1/2 – *x*, 1/2 + *y*, 1/2 – *z*; #5 1 – *x*, 2 – *y*, 1 – *z*; #6 1/2 + *x*, 1/2 – *y*, –1/2 + *z*; #7 1/2 – *x*, –1/2 + *y*, 1/2 – *z*; ^g components of three-centre system; ^h intramolecular; ⁱ π denotes the centroid of the six-membered ring; ^j components of four-centre system.

bond (1) within the layer; the hydrogen bond from the methylene group at C8 (2) is directed to a neighbouring layer. A natural explanation for the observed hydrogen bonding behaviour of the bromomethyl groups is that the inductive effect of bromine increases the acidity of methylene hydrogen atoms, thereby making them more attractive donors compared to hydrogen atoms of the benzene ring. Likewise, bromine atoms are better

Fig. 8. C–H... π interaction in compound **3** (see text). Distances are in Å.

acceptors than π electrons, and thus no strong hydrogen bonds of the type C–H... π are observed in these compounds (the contact H8B... π 2.88 Å in **2** via a *y* axis translation, identifiable but not explicitly shown in Fig. 7, has a very narrow angle of 116°).

For the *ortho* derivative (**3**), the space group *Fdd2* leads, as so often, to a complex packing diagram. Furthermore, the principles that seemed so logical for **1** and **2** are no longer valid. Of the two methylene hydrogen atoms, one is involved in an intramolecular C–H...Br interaction (1). There are no significant Br...Br contacts (the shortest is 4.46 Å), and the only intermolecular C–H...Br hydrogen bond is a three-centre system (2, 3) from H3. Hydrogen atoms H2 and H4B form short C–H... π contacts (4, 5) to an adjacent molecule (Fig. 8). The overall packing is a three-dimensional linkage of chains of molecules parallel to the polar *z* axis, best seen as a stereo view (Fig. 9).

In the tetrasubstituted compounds **4** and **5**, it is again the bromomethylene group that plays the major role in the packing (there are no significant H... π interactions). In compound **4** they combine (via translation and glide planes) to form a layer structure parallel to the *yz* plane (Fig. 10). The molecules lie approximately perpendicular (74°) to the layer, the top and bottom of which contain the bromine atoms, and which is accordingly quite thick. The predominant interactions are a very short Br...Br contact (1) of 3.50 Å, which is however type I in terms of its angles [6], and the two shortest C–H...Br hydrogen bonds (1, 2). Two further H bonds from C5 (3, 6) are observed within the layer (one as a weak component of a three-centre interaction from H5A; cf. Table 2) but have been omitted from Fig. 10 for clarity. The other three intermolecular Br...Br contacts and the remaining H bonds connect neighbouring layers.

For compound **5**, the presence of four independent bromomethyl groups leads to a complex three-dimen-

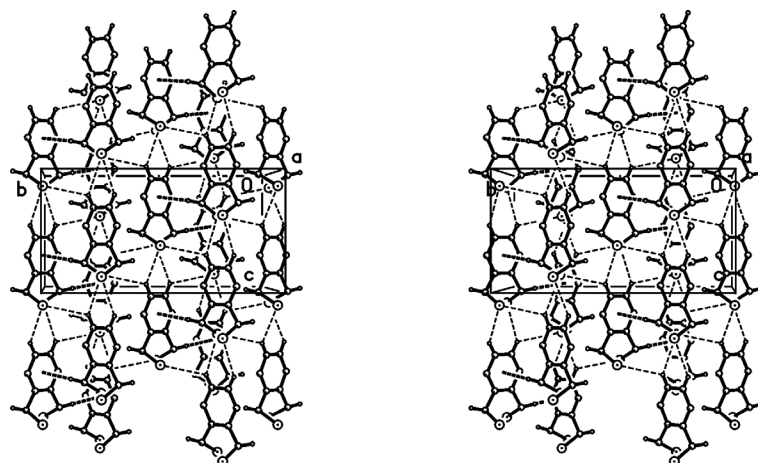


Fig. 9. Stereographic packing diagram of compound **3**. Thin dashed lines indicate C–H...Br and thick dashed lines C–H... π interactions. For clarity, the interactions from H2 are omitted (see text).

Table 3. Bromine-bromine contacts (Å and deg) for compounds **1–5**.

Compound	Contact no.	C–Br...Br–C	$d(\text{Br}\cdots\text{Br})$	$\angle(\text{C–Br}\cdots\text{Br})$	$\angle(\text{Br}\cdots\text{Br–C})$
1^a	1	C4–Br...Br#1–C4#1	3.6742(3)	108.29(6)	169.66(7) ^e
	2	C4–Br...Br#2–C4#2	3.6742(3)	169.66(7)	108.29(6) ^e
	3	C4–Br...Br#3–C4#3	3.9178(5)	76.77(7)	^f
2^b	1	C7–Br1...Br2#1–C8#1	3.6584(11)	114.1(3)	165.1(3)
	2	C7–Br1...Br2#2–C8#2	3.7389(11)	170.1(3)	110.5(3)
	3	C7–Br1...Br1#3–C7#3	3.9249(15)	75.1(3)	^f
4^c	1	C4–Br1...Br2#1–C5#1	3.4963(5)	167.50(8)	148.21(8)
	2	C4–Br1...Br2#2–C5#2	3.9079(5)	69.63(7)	135.58(8)
	3	C4–Br1...Br2#3–C5#3	4.0645(5)	69.92(8)	80.52(8)
	4	C4–Br1...Br2#4–C5#4 ^g	3.8769(6)	85.20(8)	68.72(8)
	5	C4–Br1...Br1#5–C4#5	4.0036(6)	120.85(7)	^f
5^d	1	C8–Br2...Br4#1–C10#1	3.5904(6)	91.38(10)	162.02(10)
	2	C7–Br1...Br2#2–C8#2	3.9401(7)	118.79(10)	151.07(9)
	3	C7–Br1...Br3#3–C9#3	3.9796(7)	147.16(10)	113.02(10)
	4	C7–Br1...Br4#4–C10#4	4.0451(7)	70.02(10)	134.36(11)
	5	C7–Br1...Br4#5–C10#5	4.0081(8)	82.71(10)	81.80(10)
	6	C8–Br2...Br2#6–C8#6	4.0744(7)	68.24(9)	^f
	7	C8–Br2...Br4#5–C10#5	4.1084(7)	67.13(9)	119.86(12)

^a Symmetry transformations used to generate equivalent atoms: #1 $-x, 1/2+y, 1/2-z$; #2 $-x, -1/2+y, 1/2-z$; #3 $-x, 1-y, 1-z$;

^b symmetry transformations used to generate equivalent atoms: #1 $1/2+x, 1/2-y, -1/2+z$; #2 $1/2+x, 1/2-y, -1/2+z$; #3 $1-x, 1-y, -z$;

^c symmetry transformations used to generate equivalent atoms: #1 $x, -1+y, z$; #2 $1-x, 1-y, 1-z$; #3 $1-x, -1/2+y, 1/2-z$; #4 $-x, 1-y, 1-z$; #5 $1-x, -y, 1-z$; ^d symmetry transformations used to generate equivalent atoms: #1 $1/2-x, -1/2+y, 1/2-z$; #2 $1/2+x, 1/2-y, 1/2+z$; #3 $1/2-x, 1/2+y, 1/2-z$; #4 $1-x, 2-y, 1-z$; #5 $1/2+x, 1/2-y, -1/2+z$; #6 $1-x, 1-y, -z$; ^e first two contacts equivalent by symmetry; ^f equal to first angle by symmetry; ^g intramolecular.

sional packing, which can be analyzed well in terms of its smaller components. Fig. 11 shows a layer of molecules, associated into dimers and then into chains parallel to the y axis by hydrogen bonds 2, 3, 6, 7, in the region $x \approx 0$. A symmetry-equivalent system of chains is found at $x \approx 1/2$. Fig. 11 shows the connections between the two regions *via* the short Br...Br contact and hydrogen bonds 4, 5, 8, 9. In this way the structural role of eight of the nine hydrogen bonds can be recognized. Of the eight methylene hydrogen atoms, six act as H bond donors; H7A is involved in a four-centre sys-

tem (1, 3, 7, of which H bond 1 is the only contact not shown in the Figures) and H9B in a highly asymmetric three-centre system (4, 5), whereby one component is short with a narrow angle and the other one long with a more linear angle.

We conclude that the most important structural role in these bromomethyl-substituted aromatic hydrocarbons is played by the $-\text{CH}_2\text{Br}$ groups, which can be both donor and acceptor in C–H...Br hydrogen bonds and can also be involved in Br...Br contacts. We are currently extending our studies to the structures

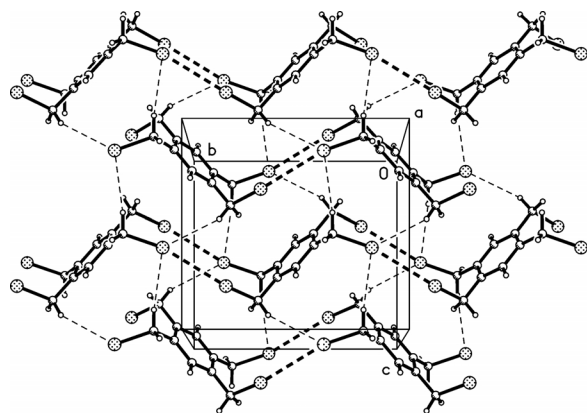


Fig. 10. Packing diagram of compound **4** viewed perpendicular to the yz plane in the region $x \approx 0$. Thin dashed lines indicate $C-H \cdots Br$ and thick dashed lines $Br \cdots Br$ interactions. For clarity, the interactions $H5B \cdots Br1$ and $H5A \cdots Br2$ are omitted (see text and Table 2).

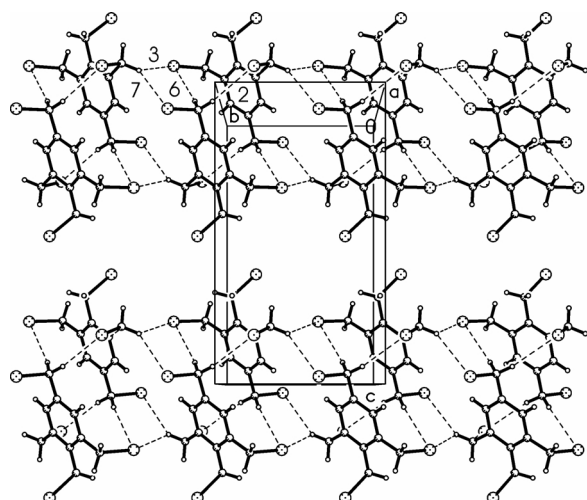


Fig. 11. Packing diagram of compound **5** viewed perpendicular to the yz plane in the region $x \approx 0$. Thin dashed lines indicate $C-H \cdots Br$ interactions. H bonds are numbered according to Table 2.

of a number of bromomethyl-substituted naphthalenes, tri(bromomethyl)benzenes and other related systems; corresponding reports are in preparation.

Experimental Section

The poly(bromomethyl)benzene were synthesized by bromination of the corresponding hydrocarbons with N-bromosuccinimide in tetrachloromethane as a solvent.

Compounds **1–3** were thus synthesized from *para*, *meta* and *ortho* xylenes, respectively. Compound **4** was synthe-

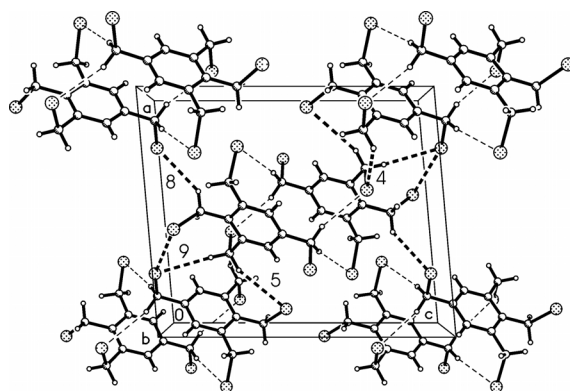


Fig. 12. Packing diagram of compound **5** viewed parallel to the y axis. Thin dashed lines indicate $C-H \cdots Br$ interactions already presented in Fig. 11 (see text); thick dashed lines indicate $C-H \cdots Br$ interactions (numbered according to Table 2) and the short $Br \cdots Br$ contact from the central dimeric unit to the peripheral molecules.

sized from durene, and **5** from isodurene. Analytical and spectroscopic properties of all compounds were in agreement with literature values [7–11]. Colourless single crystals were obtained by slow evaporation from chloroform (**1**), dichloromethane (**2** and **3**) or dichloromethane/hexane mixtures (**4** and **5**).

1,2,4,5-Tetrakis(bromomethyl)benzene (**4**)

1H NMR ($CDCl_3$): δ = 7.38 (s, 2H), 4.62 (s, 8H). – ^{13}C NMR ($CDCl_3$): δ = 29.02, 133.98, 137.96.

1,2,3,5-Tetrakis(bromomethyl)benzene (**5**)

1H NMR ($CDCl_3$): δ = 7.38 (s, 2H), 4.79 (s, 2H), 4.60 (s, 4H), 4.42 (s, 2H). – ^{13}C NMR ($CDCl_3$): δ = 24.29, 29.46, 31.70, 132.26, 136.82, 138.76, 139.44.

X-Ray structure determinations

Intensities were registered on a Bruker SMART 1000 CCD diffractometer using monochromated $MoK\alpha$ radiation (λ = 0.71073 Å). Absorption corrections were applied on the basis of multi-scans (program SADABS). Structures were refined on F^2 using SHELXL-97 (G. M. Sheldrick, University of Göttingen, Göttingen (Germany)). Hydrogen atoms were included using a riding model. For compound **3**, the Flack parameter x refined to 0.070(16).

Complete crystallographic data (excluding structure factors) have been deposited at the Cambridge Crystallographic Data Centre under the numbers CCDC 601764 (**1**), 601765 (**2**), 601766 (**3**), 601767 (**4**), 601768 (**5**). Copies may be obtained free of charge via www.ccdc.cam.ac.uk/data_request_cif.

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