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The Structure of 2,2′-Methylenebis(4-chloro-5-isopropyl-3-methylphenol)

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Abstract

$C_{21}H_{26}Cl_2O_2$, $M_r = 381$, m.p. 448 (1) K, crystallizes in space group $P2_12_12_1$ with $a = 25{\cdot}284$ (3), $b = 8{\cdot}972$ (1), $c = 17{\cdot}593$ (2) Å and $Z = 8$; $V = 3991{\cdot}07$ Å^3, $F(000) = 1616$, $\mu(\mathrm{Cu}\ K\alpha) = 3{\cdot}093$ mm^{-1}, D_m (293 K, pycnometry) = 1·30, $D_c = 1{\cdot}27$ Mg m^{-3}; $(\sin\theta/\lambda)_{\max} = 0{\cdot}61$ Å^{-1}. A single-crystal X-ray analysis of data (1748 observed reflexions) collected on an automatic Nonius CAD-4 diffractometer allowed the structure to be determined. The structure was solved by direct methods and refined by least-squares calculations to an R of 0·057. The molecular geometry of the two molecules in the asymmetric unit shows that the C—C—C valency angle (116°) at the methylene group differs significantly from the tetrahedral value. Two different molecules *A* and *B* linked by hydrogen bonds at the hydroxyl groups form dimers, and the dimers are held together by van der Waals contacts.

Introduction

A previous paper (Rantsordas, Perrin & Thozet, 1978) reported the crystal and molecular structures of 2,2′-methylenebis(4-chloro-6-isopropyl-3-methylphenol). This is the second in a series of reports describing X-ray diffraction studies of bridged bis(phenol) compounds, linked by CH_2 in an *ortho* position to the hydroxyl group. The present paper describes the crystal structure and molecular configuration of 2,2′-methylenebis(4-chloro-5-isopropyl-3-methylphenol) (BIPCMT).

Because of its fungicidal and bactericidal activity, a patent for the title compound has been registered by Perrin, Aureille-Salvadori, Vincent-Falquet & Collange in France (1972), in Great Britain (1973), in West Germany (1973) and in the USA (1979). The title compound, synthesized by Vincent-Falquet *et al.* (1974), was recrystallized by slow evaporation from a saturated solution in a mixture of solvents: diethyl ether, carbon tetrachloride, 1,2-dichlorethane. The melting points of the crystals and the pure product were identical: 448(1)K. It was concluded from this that solvent was not trapped. The crystals are transparent colourless prisms elongated along the b axis. For data collection, a nearly equidimensional crystal, obtained by cleaving, was sealed in a glass capillary. Preliminary rotation and Weissenberg photographs indicated that the crystals are orthorhombic with space group $P2_12_12_1$. Cell parameters were accurately determined by least-squares refinement of 25 general reflections measured on an automatic diffractometer. The crystal data are listed in the *Abstract*. Intensity data were collected, at 293 K, on an automatic Enraf–Nonius CAD-4 four-circle diffractometer with Cu $K\alpha$ radiation ($\lambda = 1{\cdot}54178$ Å). 4248 reflections within the range $1° < \theta < 70°$ were measured. The crystal was a poor diffractor and only 1748 reflections had $I > 3\sigma(I)$ and were used in the structure determination. This number is rather low relative to the number of refined parameters (607), implying a low precision for the molecular geometry. For this reason the C—H and O—H bond lengths are not given. The structure was solved by direct methods with the program *MULTAN* (Germain, Main & Woolfson, 1971). The atomic scattering factors for C, O, and Cl were taken from *International Tables for X-ray Crystallography* (1962) and those for H from Stewart, Davidson & Simpson (1965). A Wilson (1942) statistical calculation gave an overall temperature factor of 4·99 Å^2. An initial E map based on 100 reflections ($E > 2{\cdot}07$) revealed 33 non-hydrogen atoms of the two molecules. A Fourier synthesis with these calculated positions and with all reflections showed the structure. Refinement, with isotropic temperature factors, by the least-squares method with a block-diagonal-matrix approximation (Ahmed, Hall, Pippy & Huber, 1966) and the weighting scheme $\sqrt{w} = 1$ if $|F_o| < P_1$; $\sqrt{w} = P_1/|F_o|$ if $|F_o| > P_1$ with $P_1^2/F_{\max}^2 = 0{\cdot}1$, reduced R to 0·087. A difference map calculated at this stage contained peaks; most were interpretable as H atoms. These atoms were included during further refinement with the isotropic temperature factors of the atoms to which they are bonded. Refinement with anisotropic thermal parameters for non-hydrogen atoms and isotropic for H atoms reduced R to a final value of 0·0575. The final

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Table 1. *Final fractional coordinates* ($\times 10^4$) *for non-hydrogen atoms, with corresponding e.s.d.'s in parentheses, and equivalent isotropic thermal parameters* (Å^2)

	x	y	z	B_{eq}*		x	y	z	B_{eq}*
Molecule A					Molecule B				
C(1)	4219 (6)	8324 (19)	2594 (8)	3·32	C(31)	857 (6)	3651 (20)	5115 (9)	4·19
C(2)	4140 (6)	6841 (17)	2785 (9)	3·36	C(32)	710 (6)	2260 (17)	4907 (9)	3·33
C(3)	3702 (7)	6126 (18)	2406 (9)	4·08	C(33)	962 (6)	1590 (19)	4268 (9)	3·82
C(4)	3363 (7)	6921 (21)	1965 (10)	4·97	C(34)	1329 (6)	2460 (21)	3897 (9)	4·92
C(5)	3418 (7)	8409 (22)	1814 (9)	4·98	C(35)	1448 (6)	3929 (20)	4067 (10)	4·12
C(6)	3871 (8)	9094 (20)	2108 (10)	5·41	C(36)	1226 (6)	4510 (20)	4722 (10)	4·27
C(7)	3022 (8)	9349 (22)	1364 (10)	6·43	C(37)	1829 (7)	4872 (21)	3596 (11)	5·55
C(8)	3302 (10)	10540 (29)	854 (14)	9·34	C(38)	2123 (8)	6057 (25)	4055 (15)	8·10
C(9)	2630 (9)	10073 (30)	1907 (15)	10·19	C(39)	1495 (11)	5630 (27)	2961 (14)	8·10
C(10)	3613 (9)	4460 (22)	2495 (12)	6·32	C(40)	887 (7)	−8 (20)	4042 (12)	5·12
C(11)	4894 (6)	7598 (18)	4400 (9)	3·76	C(41)	−473 (6)	3013 (17)	5925 (9)	3·57
C(12)	4476 (6)	6770 (17)	4143 (8)	2·75	C(42)	−267 (6)	2219 (19)	5326 (9)	3·72
C(13)	4037 (6)	6557 (19)	4608 (9)	3·89	C(43)	−601 (7)	2097 (19)	4651 (10)	4·02
C(14)	4069 (6)	7220 (19)	5363 (9)	3·40	C(44)	−1091 (6)	2801 (18)	4654 (9)	4·01
C(15)	4487 (6)	8053 (19)	5641 (9)	4·03	C(45)	−1307 (6)	3531 (16)	5304 (9)	3·02
C(16)	4919 (6)	8209 (17)	5139 (8)	3·16	C(46)	−980 (5)	3630 (18)	5933 (8)	3·26
C(17)	4486 (8)	8662 (21)	6426 (8)	5·74	C(47)	−1871 (6)	4128 (21)	5289 (10)	4·41
C(18)	4904 (10)	9901 (23)	6528 (13)	8·52	C(48)	−1938 (7)	5450 (20)	5851 (11)	5·65
C(19)	4624 (8)	7453 (24)	7021 (10)	6·24	C(49)	−2241 (7)	2973 (20)	5434 (13)	6·44
C(20)	3571 (7)	5635 (19)	4391 (10)	4·44	C(50)	−414 (6)	1214 (19)	3974 (10)	3·94
C(21)	4476 (7)	6086 (18)	3362 (9)	3·59	C(51)	270 (6)	1496 (20)	5353 (10)	4·16
Cl(4)	2823 (2)	6010 (7)	1542 (3)	7·06	Cl(34)	1623 (2)	1671 (6)	3079 (3)	5·56
Cl(14)	3494 (2)	7013 (6)	5939 (3)	5·42	Cl(44)	−1476 (2)	2663 (6)	3844 (3)	6·10
O(1)	4658 (4)	9085 (11)	2851 (6)	3·53	O(31)	663 (4)	4331 (12)	5783 (6)	4·07
O(11)	5322 (4)	7854 (12)	3926 (6)	4·15	O(41)	−160 (4)	3151 (13)	6562 (6)	4·22

* $B_{eq} = \frac{4}{3}\sum_i\sum_j \beta_{ij}\,\mathbf{a}_i\cdot\mathbf{a}_j$.

Table 2. *Final fractional coordinates* ($\times 10^3$) *for the* H *atoms, with e.s.d.'s in parentheses, and isotropic thermal parameters* (Å^2)

	x	y	z	B		x	y	z	B
Molecule A					Molecule B				
H(11)	459 (5)	1011 (15)	284 (7)	3·9	H(311)	35 (4)	435 (14)	591 (7)	3·5
H(111)	530 (5)	886 (15)	373 (6)	3·5	H(411)	−39 (4)	322 (15)	695 (7)	3·8
H(61)	393 (4)	1010 (14)	202 (7)	3·6	H(361)	133 (5)	549 (15)	480 (7)	3·9
H(71)	278 (5)	868 (16)	102 (8)	5·0	H(371)	214 (5)	414 (15)	341 (7)	4·6
H(81)	311 (6)	1071 (20)	39 (9)	8·4	H(381)	189 (6)	677 (19)	412 (9)	7·4
H(82)	365 (6)	1051 (19)	76 (9)	8·4	H(382)	247 (6)	659 (18)	372 (8)	7·4
H(83)	330 (6)	1153 (19)	112 (9)	8·4	H(383)	227 (6)	560 (18)	463 (9)	7·4
H(91)	233 (6)	1052 (17)	164 (9)	7·1	H(391)	133 (5)	509 (17)	269 (8)	6·4
H(92)	284 (6)	1098 (18)	207 (9)	7·1	H(392)	165 (5)	619 (17)	260 (8)	6·4
H(93)	248 (6)	937 (17)	227 (9)	7·1	H(393)	141 (5)	645 (18)	303 (8)	6·4
H(101)	389 (5)	384 (17)	253 (8)	5·5	H(401)	122 (5)	−44 (16)	389 (8)	5·3
H(102)	334 (5)	412 (15)	283 (8)	5·5	H(402)	70 (5)	−13 (16)	363 (8)	5·3
H(103)	346 (5)	398 (16)	202 (8)	5·5	H(403)	70 (5)	−82 (16)	443 (8)	5·3
H(161)	525 (4)	865 (15)	536 (7)	3·4	H(461)	−113 (4)	405 (15)	630 (7)	3·5
H(171)	413 (5)	898 (16)	655 (8)	4·9	H(471)	−201 (5)	439 (14)	473 (7)	3·7
H(181)	493 (6)	1070 (19)	616 (9)	7·9	H(481)	−230 (5)	573 (16)	574 (8)	5·2
H(182)	524 (6)	964 (19)	622 (9)	7·9	H(482)	−173 (5)	621 (16)	585 (7)	5·2
H(183)	502 (6)	1005 (19)	705 (9)	7·9	H(483)	−190 (5)	515 (16)	644 (8)	5·2
H(191)	504 (5)	699 (16)	688 (7)	5·1	H(491)	−216 (5)	218 (16)	529 (7)	5·0
H(192)	433 (5)	689 (16)	703 (8)	5·1	H(492)	−258 (5)	309 (16)	521 (7)	5·0
H(193)	461 (5)	776 (16)	753 (7)	5·1	H(493)	−235 (5)	301 (15)	601 (8)	5·0
H(201)	361 (4)	478 (14)	417 (7)	3·8	H(501)	−10 (5)	177 (15)	369 (7)	4·1
H(202)	326 (4)	618 (14)	408 (7)	3·8	H(502)	−76 (5)	105 (15)	361 (7)	4·1
H(203)	345 (5)	561 (14)	481 (7)	3·8	H(503)	−18 (5)	67 (14)	404 (7)	4·1
H(211)	440 (4)	510 (14)	341 (7)	3·0	H(511)	23 (4)	45 (14)	518 (7)	3·3
H(212)	487 (4)	607 (14)	314 (6)	3·0	H(512)	37 (4)	140 (14)	589 (7)	3·3

parameters of non-hydrogen and H atoms are, respectively, listed in Tables 1 and 2.*

Discussion

The asymmetric unit consists of two molecules, *A* and *B*. Fig. 1 shows a general view of each of these molecules with the atom and plane numbering schemes adopted. Fig. 2 is the stereoscopic view obtained with *ORTEP* (Johnson, 1965) for molecule *A*. To conserve space in the tables, only atom labels for molecule *A* are listed; the corresponding labels for molecule *B* can be obtained by adding 30 for each non-H atom (see Fig. 1). Bond distances and angles with their corresponding estimated standard deviations are listed in Tables 3 and 4 respectively. These values have low precision owing to the limited number of reflections used, but they do not differ significantly from values found in other phenols (Bavoux, Perrin, Thozet, Bertholon & Perrin, 1976) and the bis(phenol) (Rantsordas, Perrin & Thozet, 1978). The abnormally short value for C(47)–C(49) may be caused by the combined effect of high thermal motion for C(49) and limited size of data.

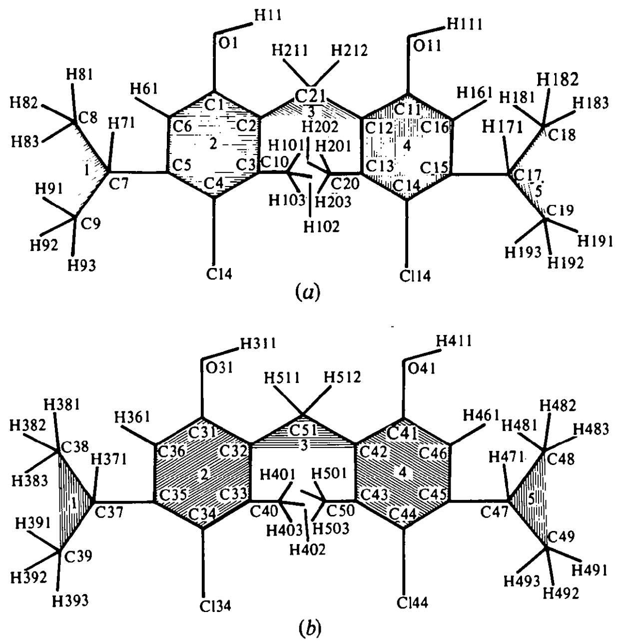

Fig. 1. Atomic and plane numbering schemes for (*a*) molecule *A* and (*b*) molecule *B*.

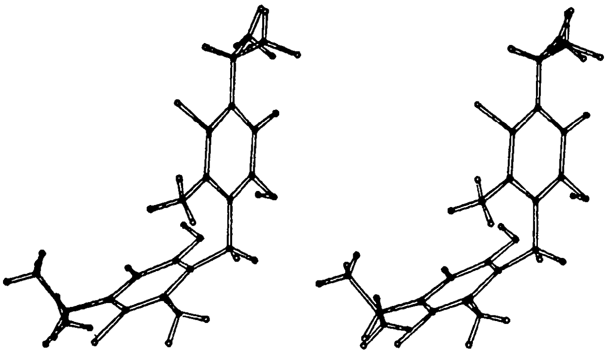

Fig. 2. A stereoscopic view of molecule *A*.

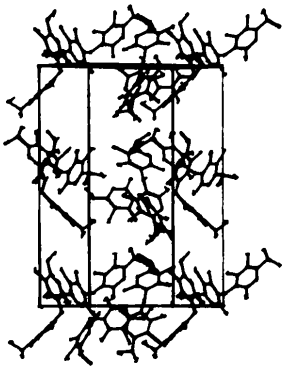

Fig. 3. An illustration of the crystal structure seen along [010].

Table 3. *Bond distances* (Å) *with e.s.d.'s in parentheses* (*see Fig.* 1 *for atom labelling*)

	Molecule *A*	Molecule *B*		Molecule *A*	Molecule *B*		Molecule *A*	Molecule *B*
C(1)–C(2)	1·39 (2)	1·35 (2)	C(5)–C(6)	1·40 (3)	1·38 (2)	C(13)–C(14)	1·46 (2)	1·39 (2)
C(1)–C(6)	1·41 (3)	1·40 (2)	C(5)–C(7)	1·53 (3)	1·53 (3)	C(13)–C(20)	1·49 (2)	1·51 (3)
C(1)–O(1)	1·38 (2)	1·41 (2)	C(7)–C(8)	1·56 (3)	1·53 (3)	C(14)–C(15)	1·38 (2)	1·43 (2)
C(2)–C(3)	1·44 (2)	1·43 (2)	C(7)–C(9)	1·52 (3)	1·56 (3)	C(14)–Cl(14)	1·78 (2)	1·73 (2)
C(2)–C(21)	1·49 (2)	1·52 (2)	C(11)–C(12)	1·37 (2)	1·38 (2)	C(15)–C(16)	1·41 (2)	1·39 (2)
C(3)–C(4)	1·36 (2)	1·38 (2)	C(11)–C(16)	1·41 (2)	1·40 (2)	C(15)–C(17)	1·49 (2)	1·52 (2)
C(3)–C(10)	1·52 (3)	1·50 (3)	C(11)–O(11)	1·39 (2)	1·38 (2)	C(17)–C(18)	1·55 (3)	1·55 (3)
C(4)–C(5)	1·37 (3)	1·38 (3)	C(12)–C(13)	1·39 (2)	1·46 (2)	C(17)–C(19)	1·55 (3)	1·42 (3)
C(4)–Cl(4)	1·76 (2)	1·77 (2)	C(12)–C(21)	1·50 (2)	1·51 (2)			

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* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 35831 (30 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

The dihedral angles between the mean planes are expected to be less influenced by the low precision of the individual atom positions. All the benzene rings are planar (Table 5), the displacements of the atoms from the mean plane being less than 0·05 Å. The dihedral angles are given in Table 6. Only the dihedral angles between planes (2) and (3), 61 and 64° for *A* and *B* respectively, are similar; all others are different. The isopropyl planes (1) and (5) are not equally inclined to the benzene rings where they are attached; the 33° discrepancy between these two angles is identical but inverted in *A* and *B*. Torsion angles concerning isopropyl groups are given in Table 7.

The two benzene rings are linked by a methylene group: the valency angles C(2)—C(21)—C(12) and C(32)—C(51)—C(42) of 116° are appreciably different from the tetrahedral value; this can be compared with the value of 119° found in similar compounds (Whittaker, 1953; Chaudhuri & Hargreaves, 1956; Rantsordas, Perrin & Thozet, 1978) and could be a common feature in bis(phenols) with a C_{Ar}—C_{CH_2}—C_{Ar} linkage.

Table 4. *Bond angles* (°) *with e.s.d.'s in parentheses*

	Molecule *A*	Molecule *B*		Molecule *A*	Molecule *B*		Molecule *A*	Molecule *B*
C(2)—C(1)—C(6)	121·8 (1·5)	124·1 (1·6)	C(4)—C(5)—C(7)	124·8 (1·6)	123·3 (1·5)	C(12)—C(13)—C(20)	123·8 (1·4)	120·0 (1·4)
C(2)—C(1)—O(1)	120·8 (1·4)	121·9 (1·4)	C(6)—C(5)—C(7)	119·0 (1·6)	119·9 (1·5)	C(14)—C(13)—C(20)	120·2 (1·4)	121·4 (1·5)
C(6)—C(1)—O(1)	117·3 (1·4)	113·9 (1·4)	C(1)—C(6)—C(5)	121·4 (1·6)	118·4 (1·5)	C(13)—C(14)—C(15)	125·8 (1·5)	123·6 (1·5)
C(1)—C(2)—C(3)	115·1 (1·4)	118·7 (1·5)	C(5)—C(7)—C(8)	112·1 (1·6)	113·9 (1·6)	C(13)—C(14)—Cl(14)	115·4 (1·2)	117·7 (1·2)
C(1)—C(2)—C(21)	121·4 (1·4)	118·5 (1·4)	C(5)—C(7)—C(9)	109·7 (1·6)	106·8 (1·6)	C(15)—C(14)—Cl(14)	118·6 (1·2)	118·5 (1·2)
C(3)—C(2)—C(21)	123·5 (1·4)	122·8 (1·4)	C(8)—C(7)—C(9)	111·3 (1·8)	109·8 (1·7)	C(14)—C(15)—C(16)	115·1 (1·4)	116·1 (1·4)
C(2)—C(3)—C(4)	120·9 (1·5)	115·8 (1·5)	C(12)—C(11)—C(16)	123·3 (1·4)	124·1 (1·5)	C(14)—C(15)—C(17)	121·7 (1·5)	120·5 (1·3)
C(2)—C(3)—C(10)	120·1 (1·5)	123·8 (1·5)	C(12)—C(11)—O(11)	119·7 (1·3)	116·9 (1·4)	C(16)—C(15)—C(17)	123·1 (1·5)	123·4 (1·4)
C(4)—C(3)—C(10)	118·9 (1·6)	120·1 (1·5)	C(16)—C(11)—O(11)	117·0 (1·3)	118·9 (1·3)	C(11)—C(16)—C(15)	120·2 (1·4)	121·0 (1·4)
C(3)—C(4)—C(5)	124·0 (1·6)	125·8 (1·6)	C(11)—C(12)—C(13)	119·7 (1·4)	116·2 (1·4)	C(15)—C(17)—C(18)	111·8 (1·5)	111·1 (1·4)
C(3)—C(4)—Cl(4)	119·2 (1·4)	116·3 (1·3)	C(11)—C(12)—C(21)	121·5 (1·3)	122·7 (1·4)	C(15)—C(17)—C(19)	111·8 (1·5)	110·9 (1·5)
C(5)—C(4)—Cl(4)	116·8 (1·3)	117·7 (1·3)	C(13)—C(12)—C(21)	118·8 (1·3)	121·0 (1·4)	C(18)—C(17)—C(19)	105·8 (1·5)	111·8 (1·5)
C(4)—C(5)—C(6)	116·1 (1·6)	116·7 (1·5)	C(12)—C(13)—C(14)	115·8 (1·4)	118·6 (1·5)	C(2)—C(21)—C(12)	115·9 (1·3)	116·5 (1·4)

Table 5. *Least-squares planes and atom deviations* (Å × 10^3) *from the planes, with corresponding e.s.d.'s in parentheses*

Asterisks indicate atoms not included in the plane calculations.

Molecule *A*

Plane (1) [through C(7), C(8), C(9)]

$$-0\cdot7229X - 0\cdot0960Y - 0\cdot6842Z + 7\cdot9710 = 0$$

Plane (2)

$$0\cdot5530X - 0\cdot2471Y - 0\cdot7957Z - 0\cdot4138 = 0$$

C(1)	9 (15)	C(7)*	−171 (19)
C(2)	−41 (15)	C(8)*	671 (25)
C(3)	35 (17)	C(9)*	−1639 (25)
C(4)	4 (17)	C(10)*	156 (21)
C(5)	−38 (16)	O(1)*	94 (10)
C(6)	31 (19)	Cl(4)*	42 (6)

Plane (3) [through C(2), C(21), C(12)]

$$0\cdot7719X + 0\cdot5808Y - 0\cdot2584Z - 10\cdot3798 = 0$$

C(10)*	−2138 (21)	O(11)*	2316 (10)
C(20)*	−2471 (17)	Cl(4)*	−2438 (6)
O(1)*	2149 (10)	Cl(14)*	−2607 (5)

Plane (4)

$$-0\cdot4155X + 0\cdot8376Y - 0\cdot3546Z + 2\cdot1906 = 0$$

C(11)	14 (16)	C(17)*	−21 (19)
C(12)	8 (15)	C(18)*	407 (22)
C(13)	3 (17)	C(19)*	−1446 (21)
C(14)	−4 (16)	C(20)*	−65 (17)
C(15)	9 (17)	O(11)*	52 (10)
C(16)	−14 (15)	Cl(14)*	86 (5)

Plane (5) [through C(17), C(18), C(19)]

$$0\cdot5910X - 0\cdot4543Y - 0\cdot6666Z + 4\cdot3637 = 0$$

Molecule *B*

Plane (1) [through C(37), C(38), C(39)]

$$0\cdot7763X - 0\cdot0652Y - 0\cdot6270Z + 0\cdot6611 = 0$$

Plane (2)

$$-0\cdot7342X + 0\cdot3561Y - 0\cdot5780Z + 5\cdot6127 = 0$$

C(31)	−13 (16)	C(37)*	117 (18)
C(32)	27 (16)	C(38)*	−517 (23)
C(33)	−48 (16)	C(39)*	1626 (26)
C(34)	−31 (17)	C(40)*	−147 (19)
C(35)	45 (16)	O(31)*	−115 (11)
C(36)	−23 (16)	Cl(34)*	3 (5)

Plane (3) [through C(32), C(51), C(42)]

$$-0\cdot2319X - 0\cdot5452Y - 0\cdot8056Z + 8\cdot4771 = 0$$

C(40)*	2232 (20)	O(41)*	−2270 (11)
C(50)*	2494 (18)	Cl(34)*	2344 (5)
O(31)*	−2227 (11)	Cl(44)*	2592 (5)

Plane (4)

$$0\cdot3898X + 0\cdot8521Y - 0\cdot3493Z + 1\cdot8286 = 0$$

C(41)	25 (16)	C(47)*	−110 (18)
C(42)	−11 (16)	C(48)*	489 (18)
C(43)	−19 (17)	C(49)*	−1446 (19)
C(44)	35 (16)	C(50)*	−95 (17)
C(45)	−20 (15)	O(41)*	47 (11)
C(46)	−9 (16)	Cl(44)*	48 (6)

Plane (5) [through C(47), C(48), C(49)]

$$-0\cdot6482X + 0\cdot4305Y - 0\cdot6281Z + 1\cdot1841 = 0$$

Hydrogen bonds and packing

The distances O(1)···O(11) and O(31)···O(41) are, respectively, 2·76 Å and 2·71 Å, indicating intramolecular hydrogen bonds. In the crystal the two independent molecules *A* and *B* are also linked by hydrogen bonds: O(1)···O(41) and O(11)···O(31) with respective values of 2·73 and 2·72 Å. So the four O atoms participate in intra- and intermolecular hydrogen bonds to form a dimer with two of their benzene rings (planes 4) superimposed. These dimers are repeated by the 2_1 axis and distributed in sheets parallel to the (100) plane. The contacts between dimers seem very large and Table 8 gives some concerning Cl atoms.

The packing of the molecules is shown in Fig. 3.

Table 6. *Dihedral angles* (°) *between mean planes and angles* (°) *of straight lines* C(8)–C(9) *with corresponding ring planes*

Dihedral angles

Planes	Molecule *A*	Molecule *B*
(1)–(2)	81 (5)	104 (5)
(2)–(3)	61 (5)	64 (5)
(2)–(4)	99 (5)	78 (5)
(3)–(4)	75 (5)	107 (5)
(4)–(5)	114 (5)	71 (5)

Straight-line angles

	Molecule *A*	Molecule *B*
[C(8)–C(9)]–plane (2)	65 (4)	58 (4)
[C(18)–C(19)]–plane (4)	48 (4)	51 (4)

Table 7. *Pertinent torsion angles* (°) *with e.s.d.'s in parentheses*

	Molecule *A*	Molecule *B*
C(3)–C(4)–C(5)–C(6)		8 (3)
C(4)–C(5)–C(7)–C(8)	−143 (2)	−150 (2)
C(6)–C(5)–C(7)–C(8)	38 (2)	27 (2)
C(4)–C(5)–C(7)–C(9)	93 (2)	89 (2)
C(6)–C(5)–C(7)–C(9)	−86 (2)	−95 (2)
C(14)–C(15)–C(17)–C(18)	−163 (2)	−154 (1)
C(16)–C(15)–C(17)–C(18)	20 (2)	28 (2)
C(14)–C(15)–C(17)–C(19)	78 (2)	81 (2)
C(16)–C(15)–C(17)–C(19)	−99 (2)	−97 (2)
C(1)–C(2)–C(21)–C(12)	59 (2)	63 (2)
C(11)–C(12)–C(21)–C(2)	−104 (2)	−105 (2)
C(3)–C(2)–C(21)–C(12)	−119 (2)	−115 (2)
C(13)–C(12)–C(21)–C(2)	75 (2)	75 (2)

Table 8. *Intermolecular distances* (Å) *less than* 4·0 Å, *with e.s.d.'s in parentheses*

Symmetry code

(I) $\frac{1}{2}-x, 1-y, \frac{1}{2}+z$
(II) $x-\frac{1}{2}, 1\cdot5-y, 1-z$
(III) $x-\frac{1}{2}, \frac{1}{2}-y, 1-z$
(IV) $1-x, \frac{1}{2}+y, \frac{1}{2}-z$
(V) $\frac{1}{2}+x, 1\cdot5-y, 1-z$
(VI) $\frac{1}{2}-x, 1-y, z-\frac{1}{2}$
(VII) $x, y-1, z$
(VIII) $\frac{1}{2}+x, \frac{1}{2}-y, 1-z$
(IX) $\bar{x}, y-\frac{1}{2}, \frac{1}{2}-z$

O(31)–C(1^{I})	3·99 (2)	O(41)–O(1^{I})	3·28 (2)
O(31)–C(2^{I})	3·71 (2)	O(41)–O(1^{II})	2·73 (2)
O(31)–C(3^{I})	3·30 (2)	O(41)–O(11^{II})	3·88 (2)
O(31)–C(4^{I})	3·41 (2)	O(1)–C(21^{IV})	3·55 (2)
O(31)–C(5^{I})	3·84 (2)	O(1)–C(41^{V})	3·39 (2)
O(31)–C(11^{II})	3·39 (2)	O(1)–C(46^{V})	3·37 (2)
O(31)–C(16^{II})	3·32 (2)	O(11)–C(10^{IV})	3·95 (2)
O(31)–O(1^{II})	3·77 (2)	O(11)–C(31^{V})	3·81 (2)
O(31)–O(11^{II})	2·72 (2)	Cl(34)–Cl(14^{VI})	3·96 (1)
O(41)–C(1^{I})	3·27 (2)	Cl(34)–C(9^{VII})	3·58 (2)
O(41)–C(1^{II})	3·83 (2)	Cl(34)–C(49^{VIII})	3·90 (2)
O(41)–C(2^{I})	3·36 (2)	Cl(34)–C(19^{VI})	3·74 (2)
O(41)–C(6^{I})	3·95 (2)	Cl(44)–Cl(4^{IX})	3·78 (1)
O(41)–C(21^{I})	3·67 (2)	Cl(44)–C(39^{IX})	3·66 (3)
O(41)–C(21^{III})	3·91 (2)	Cl(44)–C(9^{IX})	3·87 (3)

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