

Tabelle 4. Abweichungen (Å) von 'besten Ebenen' durch die mit * gekennzeichneten Atome für (Ia) und (Ib)

(Ia) (*R* = Phenyl)

Ebene (1)							
C1*	C1'*	C2*	N1*	O1	C11	C3'	C21'
0.063(5)	-0.059(5)	0.030(4)	-0.032(4)	-0.477(4)	1.099(4)	1.509(4)	-0.833(4)

Ebene (2)							
C1*	C1'*	C2*	N1'*	O1'	C11'	C3	C21
0.060(6)	-0.062(6)	-0.007(3)	0.048(2)	0.414(2)	-1.005(3)	-1.457(3)	0.932(3)

Diederwinkel: 121,5°

(Ib) (*R* = *tert*-Butyl)

Ebene (1)							
C1*	C1'*	C2*	O1*	N1	C11	C3'	C21'
0.041(5)	-0.066(6)	0.067(6)	-0.016(3)	-0.288(5)	-1.685(6)	-1.089(6)	1.369(5)

Ebene (2)							
C1*	C1'*	C2*	O1'*	N1'	C11'	C3	C21
0.064(8)	-0.047(9)	-0.053(2)	0.028(6)	0.281(8)	1.688(8)	1.126(7)	-1.311(7)

Diederwinkel: 113,8°

Während das Dimere (IIIb) infolge starker Verspannungen deutliche Abweichungen der Bindungslängen und -winkel des Tetrahydroisoxazol-Rings von den üblichen Werten aufwies (Aurich, Baum, Massa &

Mogendorf, 1985) sind die für (Ia) und (Ib) ermittelten Werte als relativ normal anzusehen.

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Acta Cryst. (1989). C45, 763–766

6,8-Dichloro-2,4-bis(trichloromethyl)-1,3-benzodioxin

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(Received 18 July 1988; accepted 10 November 1988)

Abstract. C₁₀H₄Cl₈O₂, *M_r* = 439.76, orthorhombic, *P*2₁2₁2₁, *a* = 5.6553 (8), *b* = 16.331 (2), *c* = 17.046 (2) Å, *V* = 1574.3 (4) Å³, *Z* = 4, *D_x* = 1.86 Mg m⁻³, λ(Mo Kα) = 0.71069 Å, μ = 1.44 mm⁻¹, *F*(000) = 864, room temperature, final *R* = 0.078 for 1189 unique counter reflections with *F_o* > 4σ*F_o*. The distorted screw-boat heterocyclic ring is substituted pseudoequatorially at the 2-position and pseudoaxially at the 4-position, C–C_{eq} 1.52 (2) and C–C_{ax} 1.56 (2) Å. Endocyclic parameters in the heterocyclic ring are C_{Ar}–O 1.38 (2), (C_{Ar})O–C 1.39 (2), (C_{Ar})C–O 1.39 (2), O–C(C_{Ar}) 1.42 (1) Å; C_{Ar}–O–C 111 (1), (C_{Ar})O–C–O 111 (1), (C_{Ar})C–O–C 117.3 (9), O–C–C_{Ar} 113.5 (9)°.

Introduction. As part of a program of work on the structures of 1,3-benzodioxins we are investigating the

effect of changing the polarity and size of the 2,4-disubstituent on the conformation and endocyclic parameters of the heterocyclic ring. The structure of the title compound, (I), has been determined to investigate the way in which substitution of bulky polar groups in the heterocyclic ring of 6,8-dichloro-1,3-benzodioxin (Irving & Irving, 1989), (II), affects the molecular parameters and to determine whether these changes are the same as the results obtained for a series of 6-nitro-1,3-benzodioxins (Irving & Irving, 1988b).

Experimental. 6,8-Dichloro-2,4-bis(trichloromethyl)-1,3-benzodioxin was prepared by condensing 2,4-dichlorophenol (1 mol) with chloral hydrate [CCl₃-CH(OH)₂] (2½ mol) in concentrated sulfuric acid (4 d) as described by Backeberg (1950). The white solid obtained on pouring over ice was collected, dried and recrystallized repeatedly from boiling ethanol (animal charcoal) from which it separated as fine white prisms,

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Table 1. Atomic coordinates ($\times 10^4$) and equivalent isotropic temperature factors ($\text{\AA}^2 \times 10^4$)

	x	y	z	U_{eq}^*
O(1)	-3682 (16)	8668 (5)	2334 (5)	419 (26)
C(2)	-2028 (23)	8673 (7)	1735 (7)	376 (36)
H(2)	-856 (23)	9118 (7)	1842 (7)	648 (431)†
O(3)	-879 (16)	7923 (5)	1688 (5)	396 (25)
C(4)	146 (21)	7611 (7)	2387 (6)	362 (34)
H(4)	1897 (21)	7695 (7)	2402 (6)	668 (444)†
C(5)	543 (24)	7988 (8)	3823 (7)	420 (39)
H(5)	2015 (24)	7652 (8)	3866 (7)	387 (323)†
C(6)	-321 (25)	8390 (8)	4456 (7)	434 (39)
C(7)	-2330 (25)	8900 (7)	4433 (7)	404 (38)
H(7)	-2957 (25)	9181 (7)	4911 (7)	17 (203)
C(8)	-3394 (22)	8988 (7)	3694 (8)	403 (38)
C(9)	-2602 (22)	8563 (7)	3052 (7)	371 (39)
C(10)	-649 (21)	8050 (6)	3114 (7)	347 (38)
Cl(6)	1108 (9)	8321 (2)	5358 (2)	656 (13)
Cl(8)	-5791 (6)	9646 (2)	3612 (2)	554 (12)
C(21)	-3314 (24)	8800 (7)	962 (7)	412 (41)
Cl(2A)	-5026 (8)	9721 (2)	1039 (3)	697 (15)
Cl(2B)	-1179 (8)	8952 (3)	221 (2)	655 (13)
Cl(2C)	-5172 (7)	7981 (2)	728 (2)	607 (12)
C(41)	-539 (20)	6685 (7)	2378 (8)	381 (35)
Cl(4A)	908 (6)	6156 (2)	3152 (2)	511 (11)
Cl(4B)	377 (7)	6244 (2)	1484 (2)	577 (11)
Cl(4C)	-3605 (6)	6560 (2)	2496 (2)	499 (11)

* Equivalent isotropic U calculated from anisotropic U :

$$U_{eq} = \frac{1}{3} \sum_i \sum_j U_{ij} a_i^* a_j^* a_i a_j$$

† Isotropic U .

m.p. 396 K (lit. 396 K). Obtained by a different route, Ettel & Weichet (1948) report m.p. 396 K. Found: C, 27.40; H, 0.90%. Calc. for $C_{10}H_4Cl_8O_2$: C, 27.30; H, 0.92%. The 200 MHz 1H NMR spectrum in $CDCl_3$ gave $\delta(^1H)$ 5.26 [1H, s, H(2)]; 5.63 [1H, d, H(4)]; 7.51 [1H, d, H(7)]; 7.64 [1H, dd, H(5)]; $J_{4,5}$, 1.1; $J_{5,7}$, 2.4 Hz. Specimen with dimensions 0.16 \times 0.19 \times 0.32 mm used for X-ray work. Lattice parameters determined by least-squares fitting of setting angles of 24 reflections $16 \leq \theta \leq 17^\circ$, automatically centred on CAD-4 diffractometer. Intensities collected with graphite-monochromated Mo $K\alpha$ radiation, $\lambda = 0.71069 \text{ \AA}$. $\omega/2\theta$ scan mode, variable scan rate, scan width $(0.94 + 0.35 \tan \theta)^\circ$, aperture setting 4 mm, range of reflections $1 \leq \theta \leq 25^\circ$, 1654 reflections measured, 1451 unique, $R_{int} < 0.0001$, 1189 [$F_o > 4\sigma(F_o)$] used in refinement, index range $h 0/6, k 0/19, l 0/20$. Three intensity-control reflections (1,1,13, 463, 2,11,5) monitored after every hour of data collection showed an average decline in intensity of 12.0%. Data corrected for background, scan-speed, Lorentz and polarization factors; linear decay correction and empirical absorption correction applied (North, Phillips & Mathews, 1968), transmission factors 0.94 to 0.99. Structure solved by direct methods. Least-squares anisotropic refinement (based on F) of positions of non-H atoms and constrained isotropic refinement of H-atom positions gave final $R = 0.078$, $wR = 0.079$, $w = 1/[\sigma^2(F_o) + 0.00487(F_o)^2]$, $S = 1.62$, $\Delta/\sigma(\max.) < 0.020$, residual electron density $-0.54 \leq \Delta\rho \leq +0.99 \text{ e \AA}^{-3}$.

Steady decline in residual electron density with many peaks close to Cl atoms but not indicative of any disorder. Complex neutral-atom scattering factors from Stewart, Davidson & Simpson (1965) for H atoms, from Cromer & Mann (1968) for all other atom types; dispersion corrections from Cromer & Liberman (1970). Friedel pairs were not collected as the crystal decayed significantly. There are no differences in the bond lengths and angles between the enantiomers. Final atomic parameters are listed in Table 1.* Computer programs used: *SHELX76* (Sheldrick, 1978), *SHELX84* (Sheldrick, 1983), *PLUTO* (Motherwell, 1974) and *PARST* (Nardelli, 1983). All calculations performed at the Computer Centre of the University of Cape Town on a Univac 1100/81 computer.

Discussion. The title compound, (I), comprises an aromatic ring fused to a heterocyclic 1,3-dioxin ring in a distorted screw-boat conformation (Boeyens, 1978), see Fig. 1. Molecular parameters and Cremer-Pople parameters (Cremer & Pople, 1975) are given in Tables 2 and 3. The trichloromethyl group at the 2-position is pseudoequatorial whereas that at the 4-position is pseudoaxial as is expected for 2,4-bis(trichloromethyl)-1,3-benzodioxin derivatives (Irving & Irving, 1988a, and references therein). C(2) and O(3) are situated 0.80 (1) and 0.278 (9) \AA from the mean plane through O(1), C(9), C(10), C(4) and are on the same side of it and remote from the pseudoaxial C(41)Cl₃ group.

In a series of 6-nitro-1,3-benzodioxins the effect of increasing the polarity of the 2,4-disubstituent, by replacing $>CH_2$ successively by $>CH.CHCl_2$ and $>CH.CCl_3$, on the endocyclic bond lengths resulted in a decrease of the difference between the bond lengths O(1)-C(2) and C(2)-O(3) (Irving & Irving, 1988b)

* Lists of structure factors, anisotropic thermal parameters and equation of weighted least-squares plane have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51601 (7 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

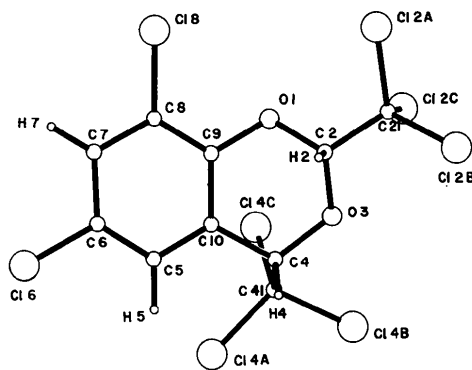


Fig. 1. A perspective view of a molecule of the title compound.

Table 2. Selected bond lengths (Å), angles (°), torsion angles (°) and Cremer-Pople parameters in the title compound, (I)

C(9)—O(1)	1.38 (2)	C(10)—C(5)—C(6)	119 (1)
O(1)—C(2)	1.39 (2)	C(5)—C(6)—C(7)	124 (1)
C(2)—O(3)	1.39 (2)	C(6)—C(7)—C(8)	116 (1)
O(3)—C(4)	1.42 (1)	C(7)—C(8)—C(9)	122 (1)
C(4)—C(10)	1.50 (2)	C(8)—C(9)—C(10)	120 (1)
C(10)—C(5)	1.39 (2)	C(9)—C(10)—C(5)	120 (1)
C(5)—C(6)	1.35 (2)	C(5)—C(6)—Cl(6)	120 (1)
C(6)—C(7)	1.41 (2)	C(7)—C(6)—Cl(6)	116 (1)
C(7)—C(8)	1.40 (2)	C(7)—C(8)—Cl(8)	118 (1)
C(8)—C(9)	1.37 (2)	C(9)—C(8)—Cl(8)	120 (1)
C(9)—C(10)	1.39 (2)	C(10)—C(4)—C(41)	113.4 (9)
C(6)—Cl(6)	1.74 (1)	O(3)—C(4)—C(41)	103.8 (9)
C(8)—Cl(8)	1.74 (1)	O(1)—C(2)—C(21)	108 (1)
C(2)—C(21)	1.52 (2)	O(3)—C(2)—C(21)	107.1 (9)
C(4)—C(41)	1.56 (2)		
C(21)—Cl(2A)	1.79 (1)	C(9)—C(10)—C(4)—C(41)	-104 (1)
C(21)—Cl(2B)	1.76 (1)	C(9)—C(10)—C(4)—H(4)	135 (1)
C(21)—Cl(2C)	1.75 (1)		
C(41)—Cl(4A)	1.78 (1)		
C(41)—Cl(4B)	1.76 (1)		
C(41)—Cl(4C)	1.76 (1)		

Cremer-Pople parameters			
	φ (°)	Q_T (Å)	θ (°)
Dioxin ring	-138 (1)	0.49 (1)	116 (1)
Aromatic ring	94 (16)	0.04 (1)	88 (17)

Table 3. Endocyclic angles (°) in the heterocyclic ring in compounds (I)–(V)

	(I)	(II)*	(III)*	(IV)*	(V)*
O(1)	111 (1)	112.7	113.5	113.3	110.2
C(2)	111 (1)	110.5	111.5	111.3	112.0
O(3)	117.3 (9)	110.1	110.3	113.6	115.5
C(4)	113.5 (9)	111.0	109.9	110.8	113.0
C(10)	118 (1)	118.3	119.8	120.0	118.6
C(9)	120 (1)	122.1	120.9	120.5	118.7

* Estimated standard deviations for compounds (II)–(V) are 0.2 to 0.3°.

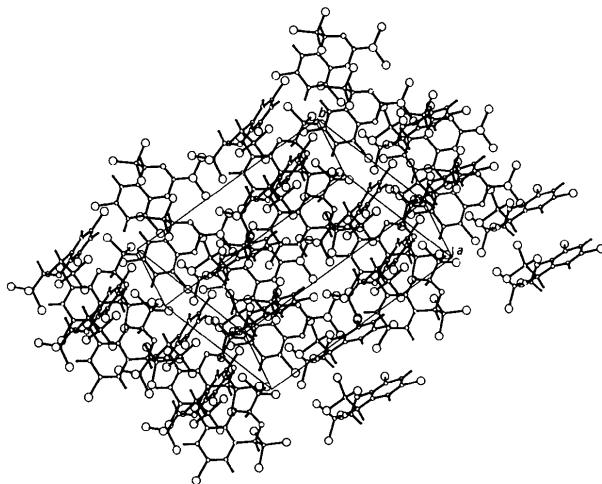


Fig. 2. Packing diagram viewed perpendicular to the plane through C(5), C(6), C(7) of the molecule whose coordinates are given.

while the remaining bond lengths in the heterocyclic ring were unchanged. Corresponding data for (II) [O(1)—C(2) 1.433 (6), C(2)—O(3) 1.401 (6) Å] (Irving & Irving, 1989) and for (I) (see Table 2) do not conflict with this trend.

In a pair of related 1,3-dioxanes, one with a pseudoequatorial 2-(*p*-bromophenyl) group and the other with a pseudoaxial 2-(*p*-trifluoromethylphenyl) group, the effect of changing a pseudoequatorial to a pseudoaxial substituent includes an increase in the endocyclic C—O—C angle and O—C(*C*_{ax})—O angle, and a decrease in the endocyclic C—C—C angle (Nader, 1975*a,b*). A similar effect for the relevant endocyclic angles, O(3), C(4), C(9), see Table 3, is observed in the pair of compounds 6-nitro-2,4-bis-(dichloromethyl)-1,3-benzodioxin (Irving & Irving, 1987), (IV), in which the C(41)HCl₂ group is pseudoequatorial, and 6-nitro-2,4-bis(trichloromethyl)-1,3-benzodioxin (Irving & Irving, 1986), (V), in which the C(41)Cl₃ group is pseudoaxial. In addition the endocyclic angle at O(1) in (V) is smaller relative to the value for (IV). These results are consistent with changing the substituent at C(4) from a bulky pseudoequatorial group in (IV) to a similar, bulky pseudoaxial group in (V). That the angle at O(3) is affected by the size as well as the position of the substituent group at C(4) is suggested by the further decrease in the angle O(3) in 6-nitro-1,3-benzodioxin (Irving & Irving, 1988*b*), (III), relative to the value in (IV) (see Table 3), whereas the other endocyclic angles in the heterocyclic ring are virtually unaffected by this change. Values of the torsion angle O(1)—C(2)—O(3)—C(4) for (III) 69.1 (4)°, and (V) 52.1 (4)°, compare well with those of 70.0 (4)° for (II) and 52 (1)° for the title compound.

Differences between corresponding endocyclic angles in the two 6,8-dichloro compounds (I) and (II) (see Table 3) present no new features beyond those previously noted in the series of 6-nitrobenzodioxins (Irving & Irving, 1988*b*). Packing is effected by normal van der Waals forces. A packing diagram is given in Fig. 2 and shows that unlike the situation in (II) there is no overlap of the aromatic rings.

We thank Dr M. L. Niven for the data collection and the UCT Research Committee for financial support.

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Acta Cryst. (1989). **C45**, 766–768

6,8-Dichloro-1,3-benzodioxin

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(Received 15 July 1988; accepted 10 November 1988)

Abstract. $C_8H_6Cl_2O_2$, $M_r = 205.04$, monoclinic, $P2_1/c$, $a = 13.895$ (6), $b = 4.0356$ (9), $c = 15.788$ (3) Å, $\beta = 109.51$ (2)°, $V = 834.5$ (5) Å³, $Z = 4$, $D_x = 1.63$ Mg m⁻³, $\lambda(Mo K\alpha) = 0.71069$ Å, $\mu = 0.73$ mm⁻¹, $F(000) = 416$, room temperature, final $R = 0.059$ for 1175 unique reflections with $F_o > 4\sigma(F_o)$. Endocyclic parameters in the heterocyclic ring are $C_{Ar}-O$ 1.365 (4), $(C_{Ar})O-C$ 1.433 (6), $(C_{Ar}O)C-O$ 1.401 (6), $O-C(C_{Ar})$ 1.422 (4) Å; $C_{Ar}-O-C$ 112.7 (3), $(C_{Ar})O-C-O$ 110.5 (4), $(C_{Ar}O)C-O-C$ 110.1 (3), $O-C-C_{Ar}$ 111.0 (3)°; $(C_{Ar})O-C-O-C(C_{Ar})-70.0$ (4)°.

Introduction. This paper continues the reports of a series of investigations into the effect of substituent groups, both in the heterocyclic ring and in the aromatic ring of 1,3-benzodioxins, on the conformation and molecular parameters of these compounds. Details of the background to these studies can be found in Irving & Irving (1988a,b,c, and references therein).

Experimental. 6,8-Dichloro-1,3-benzodioxin was prepared by the condensation of 2,4-dichlorophenol with formaldehyde in a mixture of concentrated hydrochloric and sulfuric acids following Buehler, Brown, Holbert, Fulmer & Parker (1941). The solid reaction product was collected, purified by steam distillation and recrystallized from boiling methanol, in which it is readily soluble, as colourless flat needles, m.p. 381.5 K (lit. 382–382.5 K). Better-formed and stouter needles

were obtained by slow growth from chloroform. Found: C, 46.9; H, 3.00%. $C_8H_6Cl_2O_2$ requires C, 46.86; H, 2.95%. The NMR spectrum (90 MHz in $CDCl_3$) gave $\delta(^1H)$ 4.84 [2H, t, H (4)], $J_{4,5}$ 8.6 Hz; 5.30 [2H, s, H (2)]; 6.85 [1H, td, H (5)]; 7.22 [1H, td, H (7)]. Specimen with dimensions 0.44 × 0.44 × 0.47 mm used for X-ray work. Lattice parameters determined by least-squares fitting of setting angles of 24 reflections $16 < \theta < 17^\circ$ automatically centred on CAD-4 diffractometer. Intensities collected with graphite-monochromated $Mo K\alpha$ radiation, $\lambda = 0.71069$ Å. $\omega/2\theta$ scan mode, variable scan rate, scan width $(1.73 + 0.35\tan\theta)^\circ$, aperture setting 4 mm, range of reflections $1 < \theta < 25^\circ$, 1761 reflections measured, 1325 unique, $R_{int} = 0.036$, 1175 [$F_o > 4\sigma(F_o)$] used in refinement, index range $h-16/16$, $k 0/4$, $l 0/18$. Three intensity-control reflections ($\bar{3}, 2, 10$, $\bar{8}27$, $\bar{2}35$) monitored after every hour of data collection, showed an average decline in intensity of 1.5%. Data corrected for background, scan-speed, Lorentz and polarization factors; empirical absorption correction applied (North, Phillips & Mathews, 1968), transmission factors 0.859 to 0.998. Structure solved by direct methods. Least-squares anisotropic refinement (based on F) of positions of non-H atoms and free isotropic refinement of H-atom positions gave final $R = 0.059$, $wR = 0.054$, $w = 1/[\sigma^2(F_o) + 0.000723F_o^2]$, $S = 7.8$, $\Delta/\sigma(\max.) < 0.001$, residual electron density $-0.72 \leq \Delta\rho \leq +0.49$ e Å⁻³. Complex neutral-atom scattering factors from Stewart, Davidson & Simpson (1965) for hydrogen and from Cromer & Mann (1968) for all other atom types; dispersion corrections from Cromer & Liberman (1970). Final atomic parameters are listed in

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