radii (3·6 Å), indicating strong π-electron overlap. All other intermolecular distances are longer than the sums of the van der Waals radii.

References

Bastiansen, O. & Trætteberg, M. (1962). *Tetrahedron*, **17**, 147–154.

Cadogan, J. I. G. (1962). *J. Chem. Soc.* pp. 4257–4258.

Charbonneau, G. P. & Delugeard, Y. (1976). *Acta Cryst.* B**32**, 1420–1423.

Charbonneau, G. P. & Delugeard, Y. (1977). *Acta Cryst.* B**33**, 1586–1588.

Chattopadhyay, D., Banerjee, T., Majumdar, S. K., Podder, G., Kashino, S. & Haisa, M. (1987). *Acta Cryst.* C**43**, 482–484.

Frenz, B. A. (1978). *The Enraf–Nonius CAD-4 SDP – A Real-Time System for Concurrent X-ray Data Collection and Crystal Structure Solution.* In *Computing in Crystallography*, edited by H. Schenk, R. Olthof-Hazekamp, H. van Koningsveld & G. C. Bassi, pp. 64–71. Delft Univ. Press.

Hargreaves, A. & Rivzi, S. H. (1962). *Acta Cryst.* **15**, 365–373.

International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht.)

Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

Main, P., Fiske, S. J., Hull, S. E., Lessinger, L., Germain, G., Declercq, J.-P. & Woolfson, M. M. (1982). *MULTAN*11/82. *A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univs. of York, England, and Louvain, Belgium.

Motherwell, W. D. S. & Clegg, W. (1978). *PLUTO*78. Program for plotting crystal and molecular structures. Univ. of Cambridge, England.

Rømming, C., Seip, H. M. & Aanesen Øymo, I.-M. (1974). *Acta Chem. Scand. Ser. A*, **28**, 507–514.

Sutherland, H. H. & Hoy, T. G. (1968). *Acta Cryst.* B**24**, 1207–1213.

Trotter, J. (1961). *Acta Cryst.* **14**, 1135–1140.

Walker, N. & Stuart, D. (1983). *Acta Cryst.* A**39**, 158–166.

Young, D. W., Tollin, P. & Sutherland, H. H. (1968). *Acta Cryst.* B**24**, 161–167.

Acta Cryst. (1988). C**44**, 684–686

Structures of Chlorinated Methoxybiphenyls. II.
2,2',4,4',5',6-Hexachloro-3-methoxybiphenyl

By Kari Rissanen, Jussi Valkonen and Birgitta Mannila

Department of Chemistry, University of Jyväskylä, SF-40100 Jyväskylä, Finland

(*Received* 22 *September* 1987; *accepted* 17 *November* 1987)

Abstract. $C_{13}H_6Cl_6O$, $M_r = 390{\cdot}9$, monoclinic, $P2_1/c$ (No. 14), $a = 9{\cdot}821\,(2)$, $b = 22{\cdot}041\,(4)$, $c = 7{\cdot}259\,(1)$ Å, $\beta = 102{\cdot}53\,(2)°$, $V = 1533{\cdot}9$ Å^3, $Z = 4$, $D_x = 1{\cdot}69$ Mg m^{-3}, $\lambda(\text{Mo } K\alpha) = 0{\cdot}7107$ Å, $\mu = 1{\cdot}12$ mm^{-1}, $F(000) = 776$, $T = 296$ K, final $R = 0{\cdot}036$ for 1733 unique observed reflections. The three chlorine substituents occupying the *ortho* positions (2, 2' and 6) in the phenyl rings cause a twist angle of 82·72 (11)° between the rings. The methoxy group is perpendicular to the phenyl ring {deviation of the methoxy carbon [C(7)] from the least-squares plane is 1·253 (5) Å} owing to the chlorine substituents occupying the 2- and 4-positions.

Introduction. The degree of *ortho* substitution (2, 2', 6 and 6' positions) and the size of the substituents have been identified as the main causes of the twist angle observed in substituted biphenyls (Rissanen, Valkonen & Mannila, 1988; Chattopadhyay, Banerjee, Majumdar, Podder, Kashino & Haisa, 1987). Under some conditions π-electron overlap between adjacent molecules may sometimes contribute a small additional effect (Rissanen, Valkonen & Mannila, 1988). The orientation of the methoxy group is strongly affected by the substituents occupying the positions on either side of the methoxy-substituted benzene C atom. The methoxy group is coplanar or nearly coplanar with the phenyl ring when there is no or only one substituent present on the adjacent benzene C atoms (Rissanen, Valkonen & Mannila, 1988; Chattopadhyay *et al.*, 1987), but it assumes a perpendicular orientation when there are substituents on both adjacent benzene C atoms (Rissanen, Valkonen & Knuutinen, 1987).

Experimental. Colourless crystals synthesized by known method (Cadogan, 1962), $0{\cdot}25 \times 0{\cdot}15 \times 0{\cdot}05$ mm, mounted on a glass fibre, Enraf–Nonius CAD-4 diffractometer, graphite-monochromatized Mo $K\alpha$, ω–2θ method, lattice parameters from 25 reflections with $6 < \theta < 14°$, two standard reflections measured every hour, no loss of intensity, 4459 reflections (h: $-13 \to 13$, k: $-30 \to 0$, l: $0 \to 10$) with $\theta < 30°$, 4459 independent, 1733 with $I > 3\sigma(I)$, Lp correction, empirical absorption correction (Walker & Stuart, 1983); correction factors: max. = 1·218 and min. = 0·843, direct methods, refinement by full-matrix

0108-2701/88/040684-03$03.00

Table 1. *Fractional coordinates and equivalent isotropic temperature factors with e.s.d.'s in parentheses*

$B_{eq} = \frac{4}{3}[a^2B(1,1) + b^2B(2,2) + c^2B(3,3) + ab(\cos\gamma)B(1,2) + ac(\cos\beta)B(1,3) + bc(\cos\alpha)B(2,3)]$.

	x	y	z	B_{eq}(Å^2)
Cl(2)	0·5690 (1)	0·08335 (5)	1·0109 (2)	4·26 (3)
Cl(2′)	0·1480 (1)	0·10008 (6)	1·0477 (2)	5·24 (3)
Cl(4)	0·5819 (1)	0·32680 (5)	0·9681 (2)	4·36 (3)
Cl(4′)	0·0860 (1)	−0·09197 (6)	0·5865 (2)	5·06 (3)
Cl(5′)	0·2916 (2)	−0·03154 (6)	0·3581 (2)	5·41 (3)
Cl(6)	0·1226 (1)	0·20744 (6)	0·6260 (2)	5·43 (3)
O(3)	0·6955 (3)	0·2042 (2)	1·0799 (4)	4·08 (7)
C(1)	0·3526 (4)	0·1496 (2)	0·8189 (6)	2·78 (8)
C(1′)	0·2820 (4)	0·0907 (2)	0·7608 (6)	2·89 (8)
C(2)	0·4892 (4)	0·1508 (2)	0·9274 (5)	2·89 (8)
C(2′)	0·1898 (4)	0·0633 (2)	0·8563 (6)	3·25 (9)
C(3)	0·5626 (4)	0·2047 (2)	0·9706 (5)	2·99 (8)
C(3′)	0·1309 (4)	0·0078 (2)	0·8024 (6)	3·70 (10)
C(4)	0·4950 (4)	0·2590 (2)	0·9103 (5)	3·09 (9)
C(4′)	0·1619 (4)	−0·0225 (2)	0·6503 (6)	3·50 (10)
C(5)	0·3610 (5)	0·2594 (2)	0·8057 (6)	3·53 (9)
C(5′)	0·2504 (5)	0·0043 (2)	0·5508 (6)	3·41 (9)
C(6)	0·2909 (4)	0·2055 (2)	0·7613 (6)	3·22 (9)
C(6′)	0·3109 (4)	0·0599 (2)	0·6069 (6)	3·43 (9)
C(7)	0·8035 (5)	0·1984 (3)	0·9765 (8)	5·70 (10)
H(3′)	0·070 (4)	−0·012 (2)	0·864 (6)	5·0
H(5)	0·322 (4)	0·295 (2)	0·772 (6)	5·0
H(6′)	0·372 (4)	0·076 (2)	0·536 (6)	5·0
H(7*A*)	0·887 (4)	0·194 (2)	1·067 (6)	5·0
H(7*B*)	0·792 (4)	0·234 (2)	0·905 (6)	5·0
H(7*C*)	0·790 (4)	0·154 (2)	0·916 (6)	5·0

Table 2. *Bond distances* (Å) *and angles* (°) *with e.s.d.'s in parentheses*

Cl(2)–C(2)	1·728 (4)	C(3′)–C(4′)	1·380 (7)
Cl(4′)–C(4′)	1·723 (4)	C(5)–C(6)	1·375 (6)
O(3)–C(3)	1·373 (4)	C(5)–H(5)	0·88 (4)
C(1)–C(2)	1·402 (6)	C(7)–H(7*B*)	0·93 (4)
C(1′)–C(6′)	1·388 (6)	Cl(4)–C(4)	1·727 (4)
C(3)–C(4)	1·391 (6)	Cl(6)–C(6)	1·730 (4)
C(4′)–C(5′)	1·378 (7)	C(1)–C(1′)	1·490 (6)
C(3′)–H(3′)	0·94 (5)	C(1)–C(6)	1·396 (6)
C(7)–H(7*A*)	0·94 (4)	C(2′)–C(3′)	1·372 (6)
Cl(2′)–C(2′)	1·734 (5)	C(4)–C(5)	1·370 (6)
Cl(5′)–C(5′)	1·729 (5)	C(5′)–C(6′)	1·383 (6)
O(3)–C(7)	1·432 (7)	C(6′)–H(6′)	0·94 (5)
C(1′)–C(2′)	1·393 (6)	C(7)–H(7*C*)	1·07 (4)
C(2)–C(3)	1·389 (6)		

Cl(2)–C(2)–C(1)	119·3 (3)	C(1′)–C(1)–C(6)	122·7 (3)
Cl(2′)–C(2′)–C(3′)	119·1 (4)	C(1′)–C(6′)–H(6′)	121 (3)
Cl(4′)–C(4′)–C(3′)	119·2 (4)	C(2′)–C(3′)–C(4′)	120·5 (4)
Cl(5′)–C(5′)–C(6′)	118·8 (4)	C(3)–C(4)–C(5)	121·0 (4)
O(3)–C(3)–C(2)	120·3 (4)	C(4′)–C(3′)–H(3′)	116 (3)
O(3)–C(7)–H(7*B*)	101 (3)	C(4)–C(5)–H(5)	119 (3)
C(1)–C(1′)–C(6′)	119·4 (4)	H(7*A*)–C(7)–H(7*B*)	118 (4)
C(1′)–C(1)–C(2)	120·4 (3)	Cl(2′)–C(2′)–C(1′)	119·3 (3)
C(1′)–C(6′)–C(5′)	121·4 (4)	Cl(4)–C(4)–C(5)	119·6 (3)
C(2)–C(3)–C(4)	118·4 (3)	Cl(5′)–C(5′)–C(4′)	120·9 (3)
C(2′)–C(1′)–C(6′)	117·2 (4)	Cl(6)–C(6)–C(5)	118·7 (3)
C(3′)–C(4′)–C(5′)	119·0 (4)	O(3)–C(7)–H(7*A*)	107 (3)
C(4)–C(5)–C(6)	119·8 (4)	C(1)–C(1′)–C(2′)	123·4 (4)
C(6)–C(5)–H(5)	122 (3)	C(1)–C(6)–C(5)	121·9 (3)
H(7*A*)–C(7)–H(7*C*)	101 (3)	C(1′)–C(2′)–C(3′)	121·6 (4)
Cl(2)–C(2)–C(3)	118·9 (3)	C(2)–C(1)–C(6)	116·9 (4)
Cl(4)–C(4)–C(3)	119·4 (3)	C(2′)–C(3′)–H(3′)	123 (3)
Cl(4′)–C(4′)–C(5′)	121·8 (4)	C(3)–O(3)–C(7)	114·7 (3)
Cl(6)–C(6)–C(1)	119·5 (3)	C(4′)–C(5′)–C(6′)	120·3 (4)
O(3)–C(3)–C(4)	121·2 (4)	C(5′)–C(6′)–H(6′)	118 (3)
O(3)–C(7)–H(7*C*)	105 (3)	H(7*B*)–C(7)–H(7*C*)	124 (4)
C(1)–C(2)–C(3)	122·0 (4)		

least-squares method using unit weights and F's, all non-H atoms anisotropic, H atoms from difference map and refined with fixed isotropic temperature factor (B_{eq} = 5·0 Å^2), 206 parameters, max. shift/σ = 0·01 on final cycle, R = 0·036, wR = 0·036, S = 1·16, final difference map with no features greater than 0·22 (5) e Å^{-3}, scattering factors from *International Tables for X-ray Crystallography* (1974), computer programs *MULTAN*11/82 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982), *SDP* (Frenz, 1978), *PLUTO* (Motherwell & Clegg, 1978) and *ORTEP*II (Johnson, 1976).

Discussion. The atomic coordinates and isotropic temperature factors are listed in Table 1, and the bond distances and angles are given in Table 2.* A view of the molecule and the numbering scheme are shown in Fig. 1. A stereoscopic packing scheme is presented in Fig. 2.

Bond distances and angles are normal. The two halves of the molecule can be regarded as planar [max. deviation from least-squares plane is −0·072 (1) Å for Cl(2)], except for the methoxy group, which has a perpendicular orientation: deviations of C(7) and O(3) from the least-squares plane are 1·253 (5) and −0·029 (3) Å, respectively. This off-plane displacement is caused by the chlorine substituents occupying the 2- and 4-positions. A similar off-plane displacement, 1·182 (13) and −0·135 (9) Å for methoxy C and O atoms, respectively, was found in pentachloro-

---

* Lists of structure factors, anisotropic thermal parameters and least-squares-planes data have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44549 (23 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

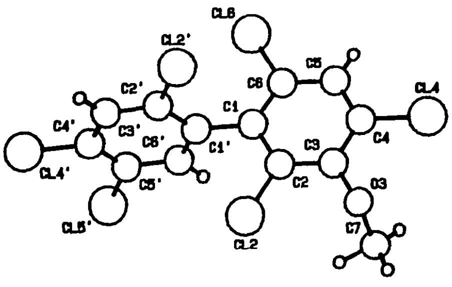

Fig. 1. *PLUTO* plot (Motherwell & Clegg, 1978) and numbering scheme for 2,2′,4,4′,5′,6-hexachloro-3-methoxybiphenyl.

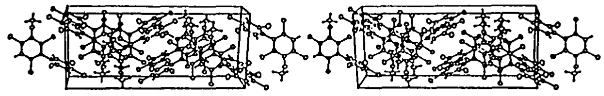

Fig. 2. Stereoscopic view of packing (*ORTEP*II; Johnson, 1976) for 2,2′,4,4′,5′,6-hexachloro-3-methoxybiphenyl. The a axis is vertical and the b axis horizontal.

methoxybenzene (Rissanen, Valkonen & Knuutinen, 1987). The angle C(3)—O(3)—C(7), 114·7 (3)°, differs from the angles observed in coplanar methoxy groups, *viz.* 117·6 (2)° in 2,2′,3,4′,5′-pentachloro-4-methoxybiphenyl (Rissanen, Valkonen & Mannila, 1988) and 117·4 (1)° in 2,2′,4,4′,5,5′-hexamethoxybiphenyl (Chattopadhyay *et al.*, 1987). But it is close to the value found in a similarly oriented methoxy group in pentachloromethoxybenzene, 112·2 (9)° (Rissanen, Valkonen & Knuutinen, 1987). The distance between the phenyl rings [C(1)—C(1′)], 1·490 (6) Å, and the narrowing of the endocyclic angle at C(1) and C(1′), indicating small inter-ring conjugation, have comparable values to those reported for similarly substituted biphenyls (Rissanen, Valkonen & Mannila, 1988; Chattopadhyay *et al.*, 1987). The twist angle between the phenyl rings, 82·72 (11)°, is caused by the three chlorine substituents occupying three *ortho* positions (2, 2′ and 6) and is larger than the value found in 2,2′,3,4′,5′-pentachloro-4-methoxybiphenyl (two *ortho* chlorine substituents) (Rissanen, Valkonen & Mannila, 1988). The two larger methoxy substituents occupying the *ortho* positions in 2,2′,4,4′,5,5′-hexamethoxybiphenyl induce almost the same twist angle, 81·2 (1)° (Chattopadhyay *et al.*, 1987), as the present three smaller chlorine *ortho* substituents. The perpendicular orientation of the methoxy group inhibits π-electron overlap between adjacent molecules and the packing is maintained by van der Waals forces. There are three intermolecular Cl—Cl distances shorter than the sum of the van der Waals radii: 3·456 (2), 3·418 (2) and 3·494 (2) Å for Cl(2)—Cl(5′), Cl(4′)—Cl(6) and Cl(4)—Cl(5′), respectively.

References

Cadogan, J. I. G. (1962). *J. Chem. Soc.* pp. 4257–4258.
Chattopadhyay, D., Banerjee, T., Majumdar, S. K., Podder, G., Kashino, S. & Haisa, M. (1987). *Acta Cryst.* C**43**, 482–484.
Frenz, B. A. (1978). *The Enraf–Nonius CAD-4 SDP – A Real-Time System for Concurrent X-ray Data Collection and Crystal Structure Solution.* In *Computing in Crystallography*, edited by H. Schenk, R. Olthof-Hazekamp, H. van Koningsveld & G. C. Bassi, pp. 64–71. Delft Univ. Press.
International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht.)
Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
Main, P., Fiske, S. J., Hull, S. E., Lessinger, L., Germain, G., Declercq, J.-P. & Woolfson, M. M. (1982). *MULTAN*11/82. *A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univs. of York, England, and Louvain, Belgium.
Motherwell, W. D. S. & Clegg, W. (1978). *PLUTO*78. Program for plotting crystal and molecular structures. Univ. of Cambridge, England.
Rissanen, K., Valkonen, J. & Knuutinen, J. (1987). *Acta Cryst.* C**43**, 1966–1968.
Rissanen, K., Valkonen, J. & Mannila, B. (1988). *Acta Cryst.* C**44**, 682–684.
Walker, N. & Stuart, D. (1983). *Acta Cryst.* A**39**, 158–166.

Acta Cryst. (1988). C**44**, 686–689

Structure of the Algal Metabolite *iso*-Strictaketal

By Vincenzo Amico and Mario Piattelli

Dipartimento di Scienze Chimiche dell'Università, Viale Doria 6, 95125 *Catania, Italy*

Francesca Cunsolo and Giuseppe Ruberto

Istituto del CNR per lo Studio delle Sostanze Naturali di Interesse Alimentare e Chimico-Farmaceutico, Viale Doria 6, 95125 *Catania, Italy*

and Giuseppe Bruno

Dipartimento di Chimica Inorganica e Struttura Molecolare dell'Università, Vill. S. Agata, salita Sperone 31, 98100 *Messina, Italy*

(*Received* 18 *May* 1987; *accepted* 18 *November* 1987)

Abstract. $C_{28}H_{40}O_5.H_2O$, $M_r = 474·64$, orthorhombic, $P2_12_12_1$, $a = 7·907$ (1), $b = 17·351$ (2), $c = 19·843$ (3) Å, $V = 2722·2$ (6) Å^3, $Z = 4$, $D_x = 1·16$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 0·74$ cm^{-1}, $F(000) = 1032$, $T = 296$ K, $R = 0·068$ for 1890 independent reflections. The X-ray study confirms that the structure of *iso*-strictaketal in the solid state is as deduced from spectroscopic evidence. The diterpenoid moiety of the molecule consists of four rings, which have the following conformations: cyclopentane distor-