The authors wish to thank the CAICYT (grant No. 2930|83) and the CSIC for financial support and a predoctoral fellowship for AF.

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Structure of an Orthorhombic Form of 2,2′-Biphenylenemercury

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(*Received* 14 *March* 1985; *accepted* 3 *December* 1985)

Abstract. 1,2;1,3;2,3-Tris(μ-2,2′-biphenylene)-trimercury, $[Hg_3(C_{12}H_8)_3]$, $M_r = 1058\cdot3$, orthorhombic, *Pbca*, $a = 26\cdot857$ (16), $b = 11\cdot458$ (6), $c = 19\cdot110$ (44) Å, $U = 5880$ Å^3, $Z = 8$, $D_m = 2\cdot398$, $D_x = 2\cdot391$ g cm^{-3}, $\lambda(\text{Mo } K\alpha) = 0\cdot7107$ Å, $\mu = 153\cdot44$ cm^{-1}, $F(000) = 3840$, $T = 293$ K, $R = 0\cdot053$ for 1898 reflexions with $I > 3\sigma(I)$. The biphenylenemercury molecule is trimeric, $(C_6H_4C_6H_4Hg)_3$, and chiral. The Hg atoms form a triangle with Hg$\cdots$Hg contacts in the range 3·461 (1) to 3·637 (2) Å, slightly greater than accepted van der Waals distances. The C—Hg—C angles are 176·3 (9)–178·9 (8)°.

Introduction. When Wittig first prepared 2,2′-biphenylenemercury he assumed it was a simple monomer (Wittig & Herwig, 1954) but later quoted ebullioscopic evidence to prove its tetrameric nature (Wittig & Lehmann, 1957). However, mass spectral analysis (Awad, Brown, Cohen, Humphries & Massey, 1977; Al-Jabar & Massey, 1984) and an X-ray study of triclinic crystals (Stender, Hinrichs, Kopf & Klar, 1981) showed some samples of the molecule to be trimeric. As both the trimeric and tetrameric species appear equally feasible we have searched for evidence of a tetramer. We found that the bulk of our biphenylenemercury samples separates from a variety of solvents as orthorhombic crystals which take on a surprising array of physical shapes from apparent cubes and truncated rods to perfectly formed hexagons. Again, even in this new crystal form biphenylenemercury is trimeric; so far we have been unable to obtain any evidence for a tetrameric species although the perfluorinated analogue has been detected mass spectroscopically (Al-Jabar & Massey, 1984).

Experimental. Preparation by the method of Neugebauer, Kos & Schleyer (1982); colourless crystals grown from toluene; D_m by flotation in hexane–dibromomethane; crystal (0·38 × 0·13 × 0·08 mm) mounted about **c**; Stoe Weissenberg diffractometer, $\sin\theta/\lambda < 0\cdot6$ Å^{-1}; lattice parameters from maximizing fit of axial row reflexions; 5123 reflexions measured, 1898 with $I > 3\sigma(I)$; h 0–34, k 0–14, l 0–15; standard check reflexions on each layer, no significant changes; absorption corrections applied, $t_{\min}$ 0·25, $t_{\max}$ 0·33; Hg positions found by direct methods and C

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atoms from subsequent difference maps, refined by full-matrix least squares on F to $R = 0{\cdot}053$, $wR = 0{\cdot}045$; Hg anisotropic, C isotropic, H atoms omitted; in final refinement cycles $w = 2{\cdot}1865/[\sigma^2(F) + 0{\cdot}000151F^2]$; max. $\Delta/\sigma = 0{\cdot}006$, $\Delta\rho$ excursions $+0{\cdot}8$ to $-0{\cdot}5$ e Å^{-3}; scattering factors from *International Tables for X-ray Crystallography* (1974); geometry calculations carried out using the *XRAY* system (1972) and all other calculations with *SHELX*76 (Sheldrick, 1976).

Discussion. The X-ray data show that orthorhombic 2,2′-biphenylenemercury is trimeric in agreement with previous mass spectral studies. It is possible to construct two Dreiding models of the trimer which, though they are mirror images of each other, are not superposable; the molecule is thus chiral. One of the enantiomers occurring in the unit cell is shown in Fig. 1, together with the numbering scheme. Final atomic coordinates are in Table 1;* bond lengths and angles are given in Table 2.

Table 1. *Final positional parameters* (×10^4) *and isotropic thermal parameters* (Å^2)

$B_{eq} = 1/3\sum_i B_{ii}$ for Hg atoms; $B_{iso} = 8\pi^2\bar{U}^{1/2}$ for C atoms.

	x	y	z	B_{eq}/B_{iso}
Hg(1)	1804·3 (0·4)	4590·2 (1)	9566·2 (0·5)	4·24 (1)
Hg(2)	1857·2 (0·4)	7754·7 (0·9)	9691·8 (0·6)	4·68 (1)
Hg(3)	729·9 (0·3)	6248·3 (0·9)	9678·4 (0·5)	3·73 (1)
C(1)	1240 (8)	3946 (21)	8937 (11)	3·4 (5)
C(2)	1312 (9)	3859 (26)	8210 (13)	5·4 (6)
C(3)	953 (11)	3455 (27)	7774 (14)	6·4 (7)
C(4)	496 (10)	3041 (24)	8054 (14)	5·2 (6)
C(5)	414 (8)	3080 (21)	8775 (11)	3·4 (5)
C(6)	790 (8)	3596 (21)	9194 (11)	3·4 (5)
C(7)	696 (8)	3631 (23)	9982 (11)	3·0 (4)
C(8)	589 (8)	2704 (21)	10366 (12)	3·6 (5)
C(9)	507 (10)	2728 (26)	11066 (14)	5·1 (6)
C(10)	530 (10)	3809 (29)	11393 (13)	5·7 (6)
C(11)	589 (11)	4844 (28)	11010 (16)	6·0 (7)
C(12)	684 (8)	4723 (18)	10297 (11)	2·9 (4)
C(13)	771 (8)	7760 (20)	9087 (11)	3·2 (5)
C(14)	696 (9)	7709 (24)	8342 (13)	4·6 (6)
C(15)	691 (9)	8791 (28)	8004 (13)	5·0 (6)
C(16)	696 (10)	9846 (27)	8325 (14)	5·8 (7)
C(17)	748 (9)	9920 (23)	9071 (13)	4·2 (6)
C(18)	806 (8)	8797 (23)	9452 (11)	3·7 (5)
C(19)	906 (8)	8849 (22)	10226 (11)	3·5 (5)
C(20)	538 (10)	9319 (23)	10649 (13)	4·6 (6)
C(21)	667 (9)	9257 (22)	11375 (12)	3·9 (5)
C(22)	1096 (11)	8717 (28)	11623 (14)	6·2 (7)
C(23)	1435 (10)	8272 (24)	11169 (13)	5·0 (6)
C(24)	1339 (8)	8332 (20)	10437 (12)	3·8 (5)
C(25)	2339 (8)	7103 (21)	8928 (13)	4·0 (5)
C(26)	2297 (9)	7394 (25)	8214 (14)	4·9 (6)
C(27)	2609 (10)	6943 (26)	7687 (14)	5·9 (7)
C(28)	2961 (10)	6126 (30)	7965 (15)	6·6 (7)
C(29)	3013 (9)	5798 (23)	8660 (13)	4·7 (6)
C(30)	2695 (10)	6281 (26)	9156 (14)	5·6 (6)
C(31)	2739 (10)	5964 (28)	9906 (15)	6·3 (7)
C(32)	3140 (9)	6463 (24)	10279 (14)	5·6 (6)
C(33)	3132 (10)	6185 (27)	10985 (15)	6·1 (7)
C(34)	2776 (12)	5592 (28)	11294 (15)	6·5 (7)
C(35)	2389 (10)	5065 (28)	10936 (16)	6·0 (7)
C(36)	2383 (9)	5208 (25)	10238 (14)	5·5 (6)

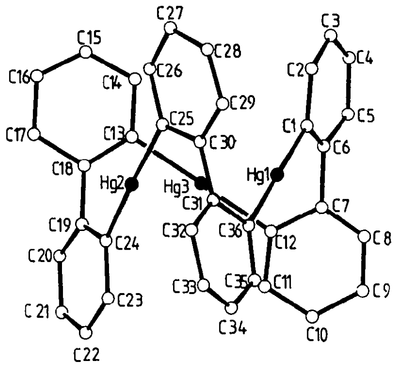

Fig. 1. Molecular structure and atom numbering for one of the enantiomers of the biphenylenemercury trimer.

Table 2. *Bond lengths* (Å) *and angles* (°)

Hg(1)–C(1)	2·07 (2)	C(15)–C(16)	1·35 (4)
Hg(1)–C(36)	2·14 (2)	C(16)–C(17)	1·43 (3)
Hg(2)–C(24)	2·10 (2)	C(17)–C(18)	1·48 (3)
Hg(2)–C(25)	2·09 (2)	C(18)–C(19)	1·50 (3)
Hg(3)–C(12)	2·11 (2)	C(19)–C(20)	1·38 (3)
Hg(3)–C(13)	2·07 (2)	C(19)–C(24)	1·36 (3)
C(1)–C(2)	1·40 (3)	C(20)–C(21)	1·43 (3)
C(1)–C(6)	1·36 (3)	C(21)–C(22)	1·39 (4)
C(2)–C(3)	1·35 (4)	C(22)–C(23)	1·36 (5)
C(3)–C(4)	1·42 (4)	C(23)–C(24)	1·42 (3)
C(4)–C(5)	1·39 (3)	C(25)–C(26)	1·41 (3)
C(5)–C(6)	1·42 (3)	C(26)–C(27)	1·41 (4)
C(6)–C(7)	1·52 (3)	C(27)–C(28)	1·43 (4)
C(7)–C(8)	1·32 (3)	C(28)–C(29)	1·39 (4)
C(7)–C(12)	1·39 (3)	C(29)–C(30)	1·39 (3)
C(8)–C(9)	1·35 (3)	C(30)–C(25)	1·41 (3)
C(9)–C(10)	1·39 (4)	C(30)–C(31)	1·48 (4)
C(10)–C(11)	1·40 (4)	C(31)–C(32)	1·41 (4)
C(11)–C(12)	1·39 (4)	C(31)–C(36)	1·44 (4)
C(13)–C(14)	1·44 (3)	C(32)–C(33)	1·38 (4)
C(13)–C(18)	1·38 (3)	C(33)–C(34)	1·31 (4)
C(14)–C(15)	1·40 (4)	C(34)–C(35)	1·38 (4)
		C(35)–C(36)	1·34 (4)
C(1)–Hg(1)–C(36)	178·1 (10)	C(17)–C(18)–C(19)	117·8 (21)
C(24)–Hg(2)–C(25)	176·3 (9)	C(18)–C(19)–C(20)	117·5 (20)
C(12)–Hg(3)–C(13)	178·9 (8)	C(18)–C(19)–C(24)	115·1 (20)
Hg(1)–C(1)–C(2)	119·8 (16)	C(20)–C(19)–C(24)	127·1 (21)
Hg(1)–C(1)–C(6)	122·9 (16)	C(19)–C(20)–C(21)	112·0 (22)
C(2)–C(1)–C(6)	117·1 (20)	C(20)–C(21)–C(22)	123·5 (23)
C(1)–C(2)–C(3)	122·2 (23)	C(21)–C(22)–C(23)	120·3 (26)
C(2)–C(3)–C(4)	119·8 (24)	C(22)–C(23)–C(24)	119·2 (35)
C(3)–C(4)–C(5)	119·8 (23)	Hg(2)–C(24)–C(19)	120·0 (16)
C(4)–C(5)–C(6)	117·3 (20)	Hg(2)–C(24)–C(23)	122·1 (23)
C(1)–C(6)–C(5)	123·3 (20)	C(19)–C(24)–C(23)	117·7 (26)
C(1)–C(6)–C(7)	119·5 (19)	Hg(2)–C(25)–C(26)	122·8 (17)
C(5)–C(6)–C(7)	116·7 (19)	Hg(2)–C(25)–C(30)	116·3 (18)
C(6)–C(7)–C(8)	124·2 (22)	C(26)–C(25)–C(30)	120·7 (22)
C(6)–C(7)–C(12)	117·0 (21)	C(25)–C(26)–C(27)	123·9 (24)
C(8)–C(7)–C(12)	118·5 (20)	C(26)–C(27)–C(28)	111·5 (24)
C(7)–C(8)–C(9)	124·5 (24)	C(27)–C(28)–C(29)	126·7 (26)
C(8)–C(9)–C(10)	117·0 (25)	C(28)–C(29)–C(30)	118·9 (24)
C(9)–C(10)–C(11)	121·6 (24)	C(25)–C(30)–C(29)	118·1 (24)
C(10)–C(11)–C(12)	116·5 (26)	C(25)–C(30)–C(31)	121·0 (24)
Hg(3)–C(12)–C(7)	120·0 (15)	C(29)–C(30)–C(31)	120·8 (25)
Hg(3)–C(12)–C(11)	118·4 (18)	C(30)–C(31)–C(32)	116·7 (25)
C(7)–C(12)–C(11)	121·1 (22)	C(30)–C(31)–C(36)	121·4 (24)
C(14)–C(13)–C(18)	122·9 (21)	C(32)–C(31)–C(36)	121·9 (25)
Hg(3)–C(13)–C(14)	119·9 (17)	C(31)–C(32)–C(33)	112·7 (25)
Hg(3)–C(13)–C(18)	116·6 (15)	C(32)–C(33)–C(34)	124·6 (26)
C(13)–C(14)–C(15)	115·0 (23)	C(33)–C(34)–C(35)	123·4 (28)
C(14)–C(15)–C(16)	125·5 (24)	C(34)–C(35)–C(36)	116·5 (26)
C(15)–C(16)–C(17)	120·2 (26)	Hg(1)–C(36)–C(31)	114·7 (19)
C(16)–C(17)–C(18)	116·4 (23)	Hg(1)–C(36)–C(35)	124·4 (20)
C(13)–C(18)–C(17)	119·3 (19)	C(31)–C(36)–C(35)	120·2 (25)
C(13)–C(18)–C(19)	122·9 (22)		

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* Lists of structure factors, anisotropic thermal parameters and mean-plane calculations and a figure showing another view of the enantiomer have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42687 (21 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

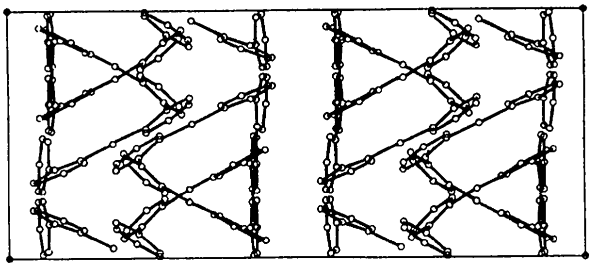

Fig. 2. A **c** projection of the unit-cell contents.

The angles between the ring planes, in the main, are considerably larger than the same angles in the triclinic molecule. The C—Hg bond lengths are within the normal values; for example, 2·09 Å in diphenylmercury (Grdenić, Kamenar & Nagl, 1977) and 2·07–2·13 Å in tribenzo[*b,e,h*][1,4,7]trimercuronin (Brown, Massey & Wickens, 1978) but slightly shorter than the distance (2·090–2·145 Å) found in the triclinic form. As expected for an organomercurial the C—Hg—C bond angles are close to 180°; there are no abnormal intermolecular contacts. The Hg···Hg distances within the heterocyclic ring are 3·461 (1) [Hg(1)—Hg(3)], 3·485 (1) [Hg(2)—Hg(3)] and 3·637 (2) Å [Hg(1)—Hg(2)]. As the van der Waals radius for Hg is thought to be 1·73 Å (Canty & Deacon, 1980) there will presumably be little or no steric strain in the ring.

A view of the unit cell is shown in Fig. 2, and a 'top view' of the molecule shown in Fig. 1 has been deposited.

Mrs Al-Jabar gratefully thanks the University of Salah Al-ddine (Iraq) for a Scholarship.

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Synthesis and X-ray Structure of a Two-Dimensional Thiocyanato-Bridged Mercury(II)–Copper(I) Complex with 5,7-Dimethyl[1,2,4]triazolo[1,5-*a*]pyrimidine, $[CuHg(\mu\text{-}SCN)_3(dmtp)_2]$

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Abstract. $[CuHg(\mu\text{-}SCN)_3(C_7H_8N_4)_2]$, $M_r = 734{\cdot}70$, triclinic, $P\bar{1}$, $a = 8{\cdot}681\,(3)$, $b = 17{\cdot}547\,(6)$, $c = 8{\cdot}331\,(3)$ Å, $\alpha = 99{\cdot}10\,(3)$, $\beta = 101{\cdot}64\,(2)$, $\gamma = 101{\cdot}62\,(3)°$, $V = 1191{\cdot}0\,(8)$ Å^3, $Z = 2$, $D_x = 2{\cdot}05$ g cm^{-3}, $\bar{\lambda}$(Mo $K\alpha$) = 0·71069 Å, $\mu = 76{\cdot}15$ cm^{-1}, $F(000) = 704$, room temperature, $R = 0{\cdot}068$ for 3443 independent observed reflections. The crystal structure consists of tetrahedral $[Hg^{II}(SCN)_3(dmtp)]$ and $[Cu^{I}(NCS)_3(dmtp)]$ coordination units, in which the thiocyanate groups, acting as bridges, are bound *via* S to Hg^{II} and *via* N to Cu^{I}, in such a way as to determine a two-dimensional arrangement of the units. Unlike the