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The Crystal Structure of Mercury(I) *o*-Phthalate*

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Mercury(I) *o*-phthalate, $C_6H_4(COOHg)_2$, crystallizes in the monoclinic system (space group $C2/c$) with $a = 26.33$ Å, $b = 6.255$ Å, $c = 12.896$ Å, and $\beta = 116.94°$. The structure has been studied from complete three-dimensional X-ray intensity data within the Cu$K\alpha$ range. The positions of all atoms, except the hydrogen atoms, have been determined. The molecule is approximately planar, with exception for one oxygen atom in each carboxylic group. An almost linear array of atoms O—Hg—Hg—O was found with a Hg—Hg distance of 2.519 ± 0.004 Å. Every molecule of $C_6H_4(COOHg)_2$ is joined to two other molecules by this short Hg—Hg bond, thus forming infinite chains running parallel to the monoclinic axis.

The only known crystal structures of mercury(I) compounds are those of the halogenides [1–3] Hg_2X_2 (X = F—I), the nitrate [4] $Hg_2(NO_3)_2 \cdot 2H_2O$, the perchlorate [5] $Hg_2(ClO_4)_2 \cdot 4H_2O$, and the bromate [6] $Hg_2(BrO_3)_2$. In all these structures there exists a linear arrangement X—Hg—Hg—X, where X stands for a halogen or an oxygen atom. The Hg—Hg distance seems to increase with decreasing electronegativity of the X atom. The main aim of the present investigation was to determine the length of the Hg—Hg bond, and also to see, if a linear arrangement O—Hg—Hg—O exists in the structure. It was also of interest to find out if the compound is a chelate.

Preparation and analysis. Crystals of mercury(I) *o*-phthalate were synthesized by adding a slightly acid solution of mercury(I) nitrate to a phthalic acid solution. The precipitate consisted of colourless, needleshaped crystals. The compound is insoluble in water, benzene, alcohol, and ether. The pure sample was analysed for mercury and water and was found to be anhydrous. The mercury contents were determined electrolytically and the water contents according to Penfield.[7] The results are in good agreement with the values calculated for the composition $C_6H_4(COOHg)_2$; Hg found 70.68 %, calc. 70.97 %.

STRUCTURE DETERMINATION

Cell dimensions and density. Powder photographs were taken in a Guinier focusing camera of 80 mm diameter using monochromatic Cu$K\alpha_1$ radiation and potassium chloride as an internal standard. The powder pattern is given

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elsewhere.[8] It could be interpreted by means of a monoclinic cell. A least-squares program [9] for refining the unit cell dimensions was used, giving the following cell dimensions and standard deviations:

$$\begin{aligned} a &= 26.33 \pm 0.02 \text{ Å} \\ b &= 6.254 \pm 0.002 \text{ Å} \\ c &= 12.90 \pm 0.01 \text{ Å} \\ \beta &= 116.94 \pm 0.03° \end{aligned}$$

The density of the sample, 4.03 g/cm³, was determined from its loss of weight in benzene. The unit cell contains eight formula units, which gives a calculated density of 3.96 g/cm³.

Single-crystal work. The crystals were mostly twinned but some single crystals could be picked out. One of them was cut to suitable size (0.02 × 0.17 × 0.04 mm³) and Weissenberg photographs (*h*0*l*—*h*3*l*,*hk*0—*hk*6) were taken with Cu$K\alpha$ radiation, using the multiple film (three films) technique. In total 852 independent reflections were obtained. In the photographs the following spectra are systematically absent:

$$\begin{aligned} hkl \text{ with } h + k &= 2n + 1 \\ h0l \text{ with } \quad l &= 2n + 1 \end{aligned}$$

which is characteristic of the space groups $C2/c$ (No. 15) and Cc (No. 9).

The intensities of the reflections in the photographs were estimated visually by comparison with a calibrated scale and corrected in the usual way for Lorentz and polarization effects. All intensities were corrected for absorption.[10] The linear absorption coefficient $\mu = 626.1 \cdot \text{cm}^{-1}$ was derived from the atomic absorption coefficients given in the *International Tables for X-ray Crystallography*.[11]

Positions of the mercury atoms. To derive preliminary positions of the mercury atoms the projections $P(upw)$ and $P(uvp)$ of the Patterson function were calculated and also the generalized projection $P_1(uw)$. The projections could be interpreted by assuming an arrangement in space group $C2/c$ of

$$\begin{gathered} 8 \text{ Hg(1) and } 8 \text{ Hg(2) in } 8(f)\text{: } (\tfrac{1}{2}\ \tfrac{1}{2}\ 0) + \\ x, y, z;\ \bar{x}, \bar{y}, \bar{z};\ \bar{x}, y, \tfrac{1}{2} - z;\ x, \bar{y}, \tfrac{1}{2} + z. \end{gathered}$$

This metal atom arrangement explained the positions and heights of all the major maxima of the Patterson function. When the parameter values of the mercury atoms had thus been obtained, the electron density sections $\varrho(xpz)$ and $\varrho(xyp)$ were calculated and more exact values for the coordinates were determined. These were then refined, using all observed 852 structure factors by means of the method of least squares.[12] In the calculation of the structure factors, the scattering factors given by Cromer and Waber [13] were used for the mercury atoms. These factors also included corrections for the anomalous dispersion. For the oxygen and carbon atoms the scattering factors were taken from Hanson *et al.*[14] The value of the discrepancy factor $R = \sum||F_{obs}| - |F_{calc}||/\sum|F_{obs}|$ was then 0.21. The resulting positions of the mercury atoms and their temperature factors were

8 Hg(1) in 8(*f*): $x = 0.0866$, $y = 0.9268$, $z = 0.2989$, $B = 4.19$
8 Hg(2) in 8(*f*): $x = 0.0153$, $y = 0.7796$, $z = 0.1049$, $B = 4.24$.

Positions of the oxygen and carbon atoms. It was not possible to identify any maxima of the electron density projections as due to oxygen or carbon atoms. This was obviously due to the predominant contribution of the mercury atoms. In order to increase the resolution of the picture and to remove the influence of the heavy metal atoms, sections in the three-dimensional electron density function were calculated after subtracting the mercury contributions from the observed structure factors. This evidently implied an assumption of the light nonhydrogen atoms also possessing the centrosymmetric symmetry. Several electron density sections were systematically made at different values of y at intervals $\Delta y \approx 0.3$ Å. On combining the various sections preliminary positions of the oxygen and carbon atoms could be deduced. The oxygen atoms as well as the carbon atoms were found to occupy the point position 8(f) of space group $C2/c$. After having identified peaks as due to oxygen and carbon atoms, a number of peaks, probably caused by diffraction effects, remained in the difference sections. A couple of these were of the same height as the carbon peaks and the rest of them smaller. They could under no conditions be due to oxygen or carbon atoms, as they were located at coordinates, giving impossible bond lengths between the atoms in the phthalate groups. The resulting positional parameters gave an R-value of 0.18.

Refinement of the structure. A refinement by the method of least-squares applying isotropic temperature factors reduced the R-value to 0.17. The electron density difference synthesis described above showed, however, that anisotropic temperature factors should be used for the mercury atoms. Thus, in the final refinement using the program LALS [15] the isotropic temperature factors for the mercury atoms were converted into anisotropic ones. All together 72 parameters were refined: 42 positional parameters, 12 anisotropic and 12 isotropic temperature factors and 6 interlayer scale factors. The function $\sum w(|F_{obs}|-|F_{calc}|)^2$ was minimized and the weighting factor, w, recommended by Cruickshank was used. Reflections too weak to be measured were given zero weight in the calculations and are omitted when calculating the R-value. The refinement was considered to be complete when the shifts in the coordinates of all atoms were below 1 % of their standard deviations. The R-value was then 0.145. The largest changes of the positional coordinates from the values found from the Fourier maps were $\Delta x = 0.01$, $\Delta y = 0.04$, and $\Delta z = 0.02$ for a carbon atom. The other shifts were all smaller. In Table 1 are given the final fractional positional parameters and their corresponding standard deviations and also the temperature factors and their standard deviations. Isotropic thermal parameters B of the form $\exp[-B \sin \theta/\lambda^2]$ are given in Å^2. The anisotropic thermal parameters β (dimensionless) are based on the expression

$$\exp\{-(h^2\beta_{11} + k^2\beta_{22} + l^2\beta_{33} + hk\beta_{12} + hl\beta_{13} + kl\beta_{23})\}$$

The observed and calculated structure factors are given in Table 2.

By introducing anisotropic thermal parameters the number of refinement parameters increased. Thus a drop in the R-value could be expected. The statistical method [17] which tests the significance of this drop could not be used in this case, as this requires a specially calculated weighted R-value, which was not yielded by the least-squares program used. However, the ob-

Table 1. Final fractional positional parameters and temperature factors and their corresponding standard deviations.

	x	y	z	B
	$\sigma(x)$	$\sigma(y)$	$\sigma(z)$	$\sigma(B)$
Hg(1)	0.0866	0.9275	0.2989	—
	0.0001	0.0006	0.0003	—
Hg(2)	0.0154	0.7804	0.1054	—
	0.0001	0.0006	0.0003	—
O(1)	0.146	0.100	0.450	5.2
	0.002	0.009	0.004	1.1
O(2)	0.071	0.160	0.454	5.9
	0.002	0.010	0.005	1.2
O(3)	0.047	0.645	0.547	5.6
	0.002	0.010	0.004	1.1
O(4)	0.105	0.633	0.460	7.6
	0.003	0.012	0.006	1.5
C(1)	0.120	0.178	0.499	3.2
	0.002	0.012	0.005	1.1
C(2)	0.148	0.263	0.613	4.5
	0.003	0.013	0.006	1.4
C(3)	0.194	0.121	0.715	6.2
	0.004	0.016	0.008	1.9
C(4)	0.214	0.214	0.825	5.3
	0.003	0.015	0.007	1.6
C(5)	0.196	0.413	0.842	4.3
	0.003	0.012	0.006	1.4
C(6)	0.156	0.543	0.746	5.3
	0.003	0.014	0.007	1.6
C(7)	0.135	0.472	0.647	4.7
	0.003	0.014	0.006	1.5
C(8)	0.094	0.610	0.540	3.6
	0.003	0.011	0.005	1.2

	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
	$\sigma(\beta_{11})$	$\sigma(\beta_{22})$	$\sigma(\beta_{33})$	$\sigma(\beta_{12})$	$\sigma(\beta_{13})$	$\sigma(\beta_{23})$
Hg(1)	0.0024	0.025	0.0080	−0.0029	0.0049	−0.0081
	0.0001	0.004	0.0003	0.0005	0.0002	0.0009
Hg(2)	0.0021	0.029	0.0082	−0.0023	0.0045	−0.0091
	0.0001	0.004	0.0003	0.0005	0.0002	0.0010

tained β_{ij} values were transformed to U_{ij} values, and the principal axes of the vibration ellipsoids were computed.[18] The root mean square amplitudes showed that the ellipsoids just slightly deviate from spheres. The difference between the smallest and the greatest amplitudes is of the order of 3σ. The ellipsoids are, within the standard deviations, the same for the mercury atoms in different positions. It is, however, not possible to conclude from the present data whether the thermal vibrations of the mercury atoms are significantly anisotropic.

Table 2. Observed and calculated structure factors for mercury(I) *o*-phthalate.

h k l	F_{obs}	F_{calc}	h k l	F_{obs}	F_{calc}	h k l	F_{obs}	F_{calc}	h k l	F_{obs}	F_{calc}	h k l	F_{obs}	F_{calc}
-12 2 4	141	141	- 6 2 9	114	87	1 3 2	99	- 85	13 3 7	60	91	- 2 4 4	198	-194
-10 2 4	113	-121	- 4 2 9	116	84	5 3 2	100	74				0 4 4	208	-185
- 8 2 4	149	-155	0 2 9	204	-193	7 3 2	77	67	-19 3 8	77	72	2 4 4	144	-108
- 6 2 4	106	96	2 2 9	131	-128	9 3 2	51	47	-17 3 8	101	105	4 4 4	88	- 71
- 4 2 4	378	409	6 2 9	77	78	11 3 2	53	31	-15 3 8	129	139	6 4 4	173	-142
- 2 2 4	453	502				15 3 2	64	59	-13 3 8	110	110	8 4 4	212	-163
0 2 4	417	416	-24 2 10	88	97	17 3 2	90	91	- 5 3 8	91	84			
2 2 4	227	185	-22 2 10	77	96	19 3 2	67	85	1 3 8	88	- 80	-22 4 5	189	-128
4 2 4	128	80	-18 2 10	71	-106				3 3 8	119	- 89	-20 4 5	186	-197
6 2 4	322	221	-16 2 10	130	-158	-19 3 3	210	147	5 3 8	77	- 67	-18 4 5	103	-144
8 2 4	301	333	-14 2 10	97	-101	-17 3 3	92	38				-10 4 5	116	-240
10 2 4	252	306	- 8 2 10	220	-197	-15 3 3	197	-229	-29 3 9	54	68	- 8 4 5	388	-317
12 2 4	126	160	- 6 2 10	320	-297	-13 3 3	351	-389	-19 3 9	144	146	- 6 4 5	203	-144
			- 4 2 10	261	-220	-11 3 3	220	-251	-17 3 9	235	285	- 4 4 5	134	125
-26 2 5	131	101	- 2 2 10	155	-115	- 9 3 3	35	- 23	-15 3 9	243	276	- 2 4 5	239	177
-24 2 5	120	112	0 2 10	89	- 51	- 7 3 3	36	18	-11 3 9	87	- 59	2 4 5	215	-162
-16 2 5	207	219	4 2 10	84	- 82	- 5 3 3	183	-179	- 9 3 9	97	77	4 4 5	174	-128
-14 2 5	269	307	6 2 10	95	-111	- 3 3 3	444	-517	- 7 3 9	215	214	8 4 5	165	181
-12 2 5	141	128	8 2 10	57	- 65	- 1 3 3	531	-602	- 5 3 9	259	264	10 4 5	112	167
-10 2 5	87	- 94				1 3 3	296	-287	- 3 3 9	118	134			
- 8 2 5	193	-202	-30 2 11	79	- 62	3 3 3	104	74	- 1 3 9	116	- 67	-12 4 6	121	-152
- 6 2 5	99	69	-28 2 11	83	- 91	5 3 3	130	94	1 3 9	171	-141	-10 4 6	202	-199
- 4 2 5	378	336	-18 2 11	252	-195	7 3 3	111	- 77				- 8 4 6	178	-176
- 2 2 5	277	237	-16 2 11	108	-100	9 3 3	196	-216	- 9 3 10	58	- 37	- 6 4 6	137	-121
2 2 5	252	-206	-12 2 11	101	95	11 3 3	108	-152	- 7 3 10	77	- 70	- 2 4 6	111	- 89
4 2 5	264	-190	- 8 2 11	117	-104	13 3 3	57	46	- 5 3 10	118	-111	0 4 6	138	-116
8 2 5	105	90	- 6 2 11	123	-110	15 3 3	138	167	- 3 3 10	127	-110			
12 2 5	77	- 88	- 2 2 11	130	111	17 3 3	107	141	- 1 3 10	89	- 76			
14 2 5	135	-161	0 2 11	128	119							3 5 0	181	165
16 2 5	75	- 86				-25 3 4	54	- 44	-25 3 11	144	121	5 5 0	230	241
			-26 2 12	63	- 72	-23 3 4	61	- 47	-23 3 11	166	164	7 5 0	167	165
-28 2 6	65	- 58	-24 2 12	119	-137	-21 3 4	90	- 78	-21 3 11	68	85			
-26 2 6	86	- 76	-22 2 12	113	-128	-19 3 4	85	- 97	-13 3 11	142	130	- 7 5 1	119	-147
-22 2 6	161	127	-16 2 12	79	- 92	-17 3 4	63	- 48	-11 3 11	100	104	- 5 5 1	100	- 98
-20 2 6	221	195	-14 2 12	191	-170	- 5 3 4	103	84	- 7 3 11	188	-175	- 3 5 1	77	71
-18 2 6	109	113	-12 2 12	183	-182	- 3 3 4	215	216	- 5 3 11	187	-174	- 1 5 1	212	218
-16 2 6	62	69	-10 2 12	172	-131	- 1 3 4	218	209	3 3 11	90	- 94	9 5 1	153	135
-14 2 6	78	86				1 3 4	155	131	5 3 11	89	-123	11 5 1	156	166
-12 2 6	237	262	-26 2 13	45	- 65	3 3 4	105	78	7 3 11	57	- 82			
-10 2 6	440	445	-24 2 13	73	- 65	5 3 4	79	61				-13 5 2	146	195
- 8 2 6	436	458	- 8 2 13	146	129	7 3 4	121	83	-25 3 12	81	- 36	-11 5 2	150	186
- 6 2 6	305	290	- 6 2 13	111	94	9 3 4	126	143	-23 3 12	83	- 56	- 3 5 2	158	164
- 4 2 6	104	79				11 3 4	109	146	-21 3 12	78	- 52	- 1 5 2	196	188
- 2 2 6	103	88	-24 2 14	74	- 14	13 3 4	80	90	-15 3 12	142	- 42	1 5 2	103	96
0 2 6	222	194	-18 2 14	70	- 76				-13 3 12	183	- 81	3 5 2	109	- 91
2 2 6	363	278	-14 2 14	80	78	-23 3 5	110	- 88	-11 3 12	118	- 92	5 5 2	190	-143
4 2 6	192	182	0 2 14	58	76	-21 3 5	358	-256						
6 2 6	102	- 11				-19 3 5	308	-268	- 3 3 13	102	-111	- 9 5 3	122	168
8 2 6	102	-122	-14 2 15	70	101	-17 3 5	134	-121	- 1 3 13	78	-100	- 7 5 3	181	225
10 2 6	89	-103	-12 2 15	108	46	-11 3 5	252	-227				- 5 5 3	127	129
18 2 6	56	- 73				- 9 3 5	421	-403				3 5 3	132	142
20 2 6	46	- 73	-18 2 16	19	66	- 7 3 5	362	-324	0 4 0	93	67	5 5 3	160	165
						- 5 3 5	69	67	2 4 0	166	138			
-22 2 7	176	177				- 3 3 5	306	321	4 4 0	228	231	- 9 5 4	125	83
-20 2 7	109	136	-27 3 0	62	21	- 1 3 5	155	146	6 4 0	249	231	- 7 5 4	115	63
-16 2 7	139	-165	1 3 0	99	-149	1 3 5	94	- 76	8 4 0	135	133	- 5 5 4	127	-116
-14 2 7	149	-147	3 3 0	120	-127	3 3 5	178	-128				- 3 5 4	237	-204
-10 2 7	189	187	5 3 0	243	-236	7 3 5	240	253	-24 4 1	130	101	- 1 5 4	183	-150
- 8 2 7	90	83	7 3 0	251	-219	9 3 5	209	305	-22 4 1	149	88			
- 6 2 7	195	-190	9 3 0	143	-123	11 3 5	113	156	-12 4 1	234	261	-15 5 5	116	157
- 4 2 7	270	-279				17 3 5	44	57	-10 4 1	116	114	-13 5 55	109	136
- 2 2 7	188	-152	-25 3 1	124	112	19 3 5	104	86	- 8 4 1	115	-140	- 3 5 5	169	139
2 2 7	142	88	-23 3 1	174	158				- 6 4 1	305	-274	- 1 5 5	95	68
6 2 7	208	-187	-21 3 1	114	121	-19 3 6	73	65	- 4 4 1	90	-105	1 5 5	143	- 96
8 2 7	166	-180	-15 3 1	124	126	-13 3 6	58	73	- 2 4 1	142	140	3 5 5	183	-154
			-13 3 1	386	309	-11 3 6	136	141	0 4 1	156	148	5 5 5	125	-126
-30 2 8	100	68	-11 3 1	418	356	- 9 3 6	187	183	4 4 1	386	-385			
-28 2 8	163	119	- 7 3 1	358	-335	- 7 3 6	205	198	6 4 1	361	-364	-11 5 6	146	-170
-26 2 8	174	124	- 5 3 1	265	-283	- 5 3 6	176	164	8 4 1	137	-159	- 9 5 6	193	-198
-24 2 8	97	101	- 1 3 1	138	192	- 3 3 6	105	112	18 4 1	145	-143	- 7 5 6	146	-148
-20 2 8	63	98	3 3 1	456	-399	- 1 3 6	90	75				1 5 6	241	-164
-18 2 8	170	229	5 3 1	655	-589	1 3 6	109	95	-12 4 2	141	178	3 5 6	180	-149
-16 2 8	238	299	7 3 1	458	-420	3 3 6	124	117	-10 4 2	158	181			
-14 2 8	205	228	9 3 1	64	- 65	5 3 6	82	42	- 8 4 2	114	106			
-10 2 8	91	- 74	13 3 1	122	-114				- 4 4 2	125	129	0 6 0	179	-183
- 6 2 8	92	70	15 3 1	204	-194	-29 3 7	76	- 85	- 2 4 2	130	143	2 6 0	128	- 94
- 4 2 8	136	114	17 3 1	190	-199	-27 3 7	116	-130						
0 2 8	213	-205	19 3 1	89	- 87	-25 3 7	126	-112	-14 4 3	187	-256	- 8 6 2	146	-134
2 2 8	303	-263	23 3 1	50	50	-21 3 7	85	82	-12 4 3	184	-246	- 6 6 2	140	-140
4 2 8	178	-157				-17 3 7	119	-125	- 4 4 3	229	-248	- 2 6 2	124	106
10 2 8	81	- 88	-17 3 2	130	-107	-15 3 7	148	-174	- 2 4 3	441	-402	0 6 2	113	109
12 2 8	88	110	-15 3 2	208	-162	-11 3 7	288	285	0 4 3	310	-331			
14 2 8	71	- 93	-13 3 2	191	-206	- 9 3 7	383	340	2 4 3	83	- 60	- 8 6 4	139	144
16 2 8	35	- 47	-11 3 2	153	-184	- 7 3 7	211	202	4 4 3	104	-108	- 6 6 4	85	80
			- 9 3 2	110	-123	- 1 3 7	230	206	8 4 3	165	-149	- 2 6 4	150	131
-22 2 9	95	-112	- 7 3 2	59	- 65	1 3 7	410	337	10 4 3	204	-193	4 6 4	108	132
-16 2 9	58	72	- 5 3 2	43	- 34	3 3 7	344	284						
-12 2 9	239	-239	- 3 3 2	138	-138	5 3 78	101	95	- 6 4 4	79	- 84	1 7 0	146	-155
-10 2 9	262	-268	- 1 3 2	120	-132	11 3 7	52	63	- 4 4 4	149	-170	3,7 0	91	- 68
- 8 2 9	62	- 70												

Table 2. Continued.

h k l	F_{obs}	F_{calc}
-12 2 4	141	141
-10 2 4	113	-121
- 8 2 4	149	-155
- 6 2 4	106	96
- 4 2 4	378	409
- 2 2 4	453	502
0 2 4	417	416
2 2 4	227	185
4 2 4	128	80
6 2 4	322	221
8 2 4	301	333
10 2 4	252	306
12 2 4	126	160
-26 2 5	131	101
-24 2 5	120	112
-16 2 5	207	219
-14 2 5	269	307
-12 2 5	141	128
-10 2 5	87	- 94
- 8 2 5	193	-202
- 6 2 5	99	69
- 4 2 5	378	336
- 2 2 5	277	237
2 2 5	252	-206
4 2 5	264	-190
8 2 5	105	90
12 2 5	77	- 88
14 2 5	135	-161
16 2 5	75	- 86
-28 2 6	65	- 58
-26 2 6	86	- 76
-22 2 6	161	127
-20 2 6	221	195
-18 2 6	109	113
-16 2 6	62	69
-14 2 6	78	86
-12 2 6	237	262
-10 2 6	440	445
- 8 2 6	436	458
- 6 2 6	305	290
- 4 2 6	104	79
- 2 2 6	103	88
0 2 6	222	194
2 2 6	363	278
4 2 6	192	182
6 2 6	402	- 11
8 2 6	102	-122
10 2 6	89	-103
18 2 6	56	- 73
20 2 6	46	- 73
-22 2 7	176	177
-20 2 7	109	136
-16 2 7	139	-165
-14 2 7	149	-147
-10 2 7	189	187
- 8 2 7	90	83
- 6 2 7	195	-190
- 4 2 7	270	-279
- 2 2 7	188	-152
2 2 7	142	88
6 2 7	208	-187
8 2 7	166	-180
-30 2 8	100	68
-28 2 8	163	119
-26 2 8	174	124
-24 2 8	97	100
-20 2 8	63	98
-18 2 8	170	229
-16 2 8	238	299
-14 2 8	205	228
-10 2 8	91	- 74
- 6 2 8	92	70
- 4 2 8	136	114
0 2 8	213	-205
2 2 8	303	-263
4 2 8	178	-157
10 2 8	81	- 88
12 2 8	88	110
14 2 8	71	- 93
16 2 8	35	- 47
-22 2 9	95	-112
-16 2 9	58	72
-12 2 9	239	-239
-10 2 9	262	-268
- 8 2 9	62	- 70
- 6 2 9	114	87
- 4 2 9	116	84
0 2 9	204	-193
2 2 9	131	-128
6 2 9	77	78
-24 2 10	88	97
-22 2 10	77	96
-18 2 10	71	-106
-16 2 10	130	-158
-14 2 10	97	-101
- 8 2 10	220	-197
- 6 2 10	320	-297
- 4 2 10	261	-220
- 2 2 10	155	-115
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-12 2 11	101	95
- 8 2 11	117	-104
- 6 2 11	123	-110
- 2 2 11	130	111
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-24 2 12	119	-137
-22 2 12	113	-128
-16 2 12	79	- 92
-14 2 12	191	-170
-12 2 12	183	-182
-10 2 12	172	-131
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-24 2 13	73	- 65
- 8 2 13	146	129
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5 3 0	243	-236
7 3 0	251	-219
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-15 3 2	208	-162
-13 3 2	191	-206
-11 3 2	153	-184
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- 3 3 2	138	-138
- 1 3 2	120	-132
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5 3 3	130	94
7 3 3	111	- 77
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- 1 3 4	218	209
1 3 4	155	131
3 3 4	105	78
5 3 4	79	61
7 3 4	121	83
9 3 4	126	143
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- 5 3 6	176	164
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5 3 8	77	- 67
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- 9 3 9	97	77
- 7 3 9	215	214
- 5 3 9	259	264
- 3 3 9	118	134
- 1 3 9	116	- 67
1 3 9	171	-141
- 9 3 10	58	- 37
- 7 3 10	77	- 70
- 5 3 10	118	-111
- 3 3 10	127	-110
- 1 3 10	89	- 76
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-21 3 11	68	85
-13 3 11	142	130
-11 3 11	100	104
- 7 3 11	188	-175
- 5 3 11	187	-174
3 3 11	90	- 94
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-25 3 12	81	- 36
-23 3 12	83	- 56
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- 2 4 1	142	-140
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- 4 4 5	134	125
- 2 4 5	239	177
2 4 5	215	-162
4 4 5	174	-128
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10 4 5	112	167
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-10 4 6	202	-199
- 8 4 6	178	-176
- 6 4 6	137	-121
- 2 4 6	111	- 89
0 4 6	138	-116
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3 5 2	109	- 91
5 5 2	190	-143
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5 5 3	160	165
- 9 5 4	125	83
- 7 5 4	115	65
- 5 5 4	127	-116
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2 6 0	128	- 94
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- 6 6 2	140	-140
- 2 6 2	124	106
0 6 2	113	109
- 8 6 4	139	144
- 6 6 4	85	80
- 2 6 4	150	131
4 6 4	108	132
1 7 0	146	-155
3 7 0	91	- 68

DESCRIPTION AND DISCUSSION OF THE STRUCTURE

The metal-metal distances of the mercury atom doublets, which are present in all the known structures of mercury(I) compounds, vary within rather wide limits (*cf.* Table 3). In mercury(I) *o*-phthalate the Hg—Hg distance in the pair is 2.519 ± 0.004 Å, which fits in the series of the halogenides just before the mercury(I) chloride. This corresponds well with a tendency of increasing bond distance with decreasing electronegativity.

Each mercury atom of the *o*-phthalate has an oxygen atom as its nearest neighbour (at 2.16 ± 0.05 Å and 2.08 ± 0.05 Å, respectively) in such a

Table 3. Interatomic distances in some mercury(I) compounds.

Compound	Shortest Hg—Hg distance	Shortest Hg—O distance	Ref.
Hg_2F_2	2.43 ± 0.04 Å		3
Hg_2Cl_2	2.53 Å		1
Hg_2Br_2	2.58 Å		1
Hg_2I_2	2.72 Å		2
$Hg_2(NO_3)_2 \cdot 2H_2O$	2.54 ± 0.01 Å	2.15 ± 0.10 Å	4
$Hg_2(ClO_4)_2 \cdot 4H_2O$	2.50 ± 0.01 Å	2.14 ± 0.10 Å	5
$Hg_2(BrO_3)_2$	2.507 ± 0.006 Å	2.16 ± 0.04 Å	6

Table 4. Interatomic distances (Å units), bond angles and their corresponding standard deviations in mercury(I) *o*-phthalate.

Bond	Dist.	s.d.	Angle	(°)	s.d.
Hg(1)—Hg(2)	2.519	0.004	Hg(2)—Hg(1)—O(1)	171	2
			Hg(1)—Hg(2)—O(3)	175	2
Hg(1)—O(1)	2.16	0.05			
Hg(2)—O(3)	2.08	0.05	Hg(1)—O(1)—C(1)	108	4
			Hg(2)—O(3)—C(8)	111	4
C(1)—O(1)	1.22	0.08			
C(8)—O(3)	1.30	0.08	O(1)—C(1)—O(2)	118	6
C(1)—O(2)	1.17	0.08	O(1)—C(1)—C(2)	122	6
C(8)—O(4)	1.21	0.09	O(2)—C(1)—C(2)	119	6
C(1)—C(2)	1.42	0.10	C(1)—C(2)—C(3)	119	7
C(8)—C(7)	1.57	0.10	C(1)—C(2)—C(7)	124	7
C(2)—C(3)	1.60	0.12	C(3)—C(2)—C(7)	116	6
C(3)—C(4)	1.40	0.12	C(2)—C(3)—C(4)	114	8
C(4)—C(5)	1.39	0.12	C(3)—C(4)—C(5)	122	8
C(5)—C(6)	1.46	0.11	C(4)—C(5)—C(6)	122	7
C(6)—C(7)	1.22	0.11	C(5)—C(6)—C(7)	120	8
C(7)—C(2)	1.46	0.12	C(2)—C(7)—C(6)	125	8
			C(6)—C(7)—C(8)	121	8
			C(2)—C(7)—C(8)	113	6
			C(7)—C(8)—O(3)	110	5
			C(7)—C(8)—O(4)	118	6
			O(3)—C(8)—O(4)	130	6

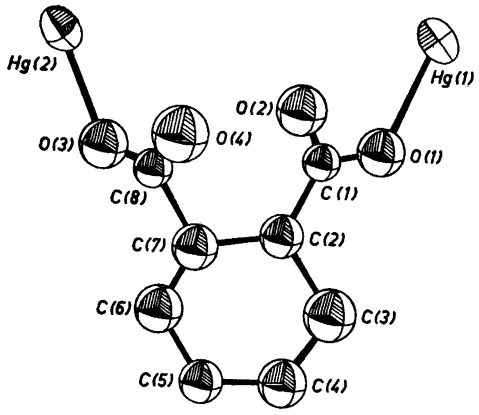

Fig. 1. One formula unit $C_6H_4(COOHg)_2$.

way that an almost linear O—Hg—Hg—O group is formed. The angles are $172 \pm 2°$ and $174 \pm 2°$. This arrangement is evidently in concordance with the structural principle displayed in all the compounds listed in Table 3.

The bond lengths and bond angles present in the structure are listed in Table 4. They were calculated by the program DISTAN.[19] The accuracy of the positional parameters of the non-metal atoms is not very high due to systematic errors in the intensity data, such as the difficulties associated with the absorption correction. However, within the standard deviations thus obtained, the interatomic distances and angles are in fair agreement with corresponding data previously reported in the literature.

In particular, the appearance of the *o*-phthalate group may be compared with the structure of *o*-phthalic acid reported by Nowacki and Jaggi.[20] The *o*-phthalic acid is approximately planar except for one oxygen atom in

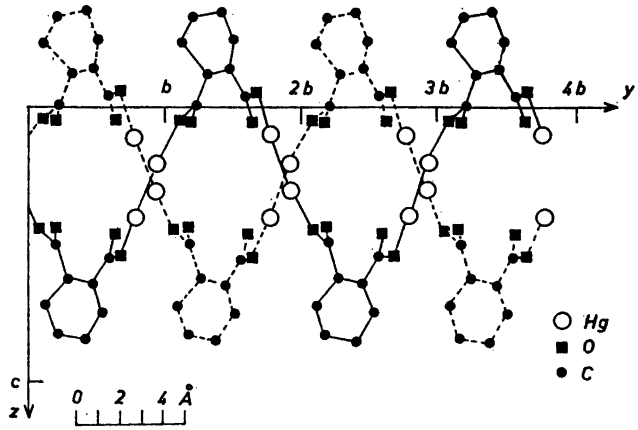

Fig. 2. Parts of two infinite helical chains —*o*-phthalate—Hg—Hg—*o*-phthalate—.

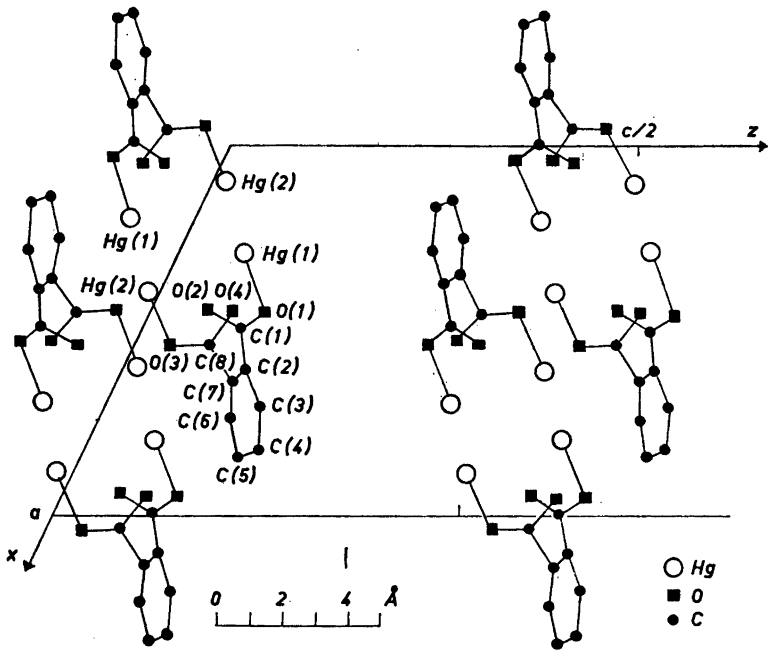

Fig. 3. Projection of the structure on the *xz*-plane.

each carboxylic group. These two oxygen atoms deviate 0.67 Å from the plane. All atoms in the mercury(I) *o*-phthalate, except for the two carboxylic oxygen atoms, O(2) and O(4), fit a plane (Fig. 1). The equation for this plane, defined by the eight carbon atoms and two of the oxygen atoms, is the following $0.676\ x + 0.566\ y - 0.479\ z = 0.409$. The carboxylic oxygen atoms deviate from the plane by 1.1 Å and 1.2 Å, respectively. All the other atoms are at distances from the plane, which are within the calculated standard errors.

The Hg—Hg doublets link different *o*-phthalate groups, which means that the compound is not a chelate. On the contrary, this mechanism of linking leads to the formation of endless zigzag chains —*o*-phthalate—Hg—Hg—*o*-phthalate—. In each unit cell there are eight crystallographically equivalent chains, combined in pairs. One of these four pairs is shown in Fig. 2, which also shows that these pairs of zigzag chains run parallel to the *y*-axis, thus being extended in the longest direction of the needleformed crystal. The chains are combined in pairs, having the same *y*-coordinate but being rotated 180° around an axis parallel to the *y*-axis. In Fig. 3, which is a projection of the structure along the monoclinic axis, can be seen how the chains are spread out in the *xz*-plane. Thus, the chains run perpendicular to this projection and the phthalate groups are inclined in relation to the *xz*-plane.

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