National Science Foundation (CHE-8514367) and the Texas Christian University and the University of North Texas Faculty Research Committees for financial support of this study.

References

ALLEN, D., LOCK, C. J. L., TURNER, G. & POWELL, J. (1975). *Can. J. Chem.* **53**, 2707–2713.

BAENZIGER, N. C., RICHARDS, G. F. & DOYLE, J. R. (1965). *Acta Cryst.* **18**, 924–926.

CHIZHEVSKY, I. T., KORIDZE, A. A., BAKHMUTOV, V. I., KOLOBOVA, N. E., ANDRIANOV, V. G. & STRUCHKOV, YU. T. (1981). *J. Organomet. Chem.* **215**, 403–414.

COTTON, F. A., DAY, V. W., FRENZ, B. A., HARDCASTLE, K. I. & TROUP, J. M. (1973). *J. Am. Chem. Soc.* **95**, 4522–4528.

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

MARCHAND, A. P., EARLYWINE, A. D. & HEEG, M. J. (1986). *J. Org. Chem.* **51**, 4096–4100.

MARCHAND, A. P., GOODIN, D. B., HOSSAIN, M. B. & VAN DER HELM, D. (1984). *J. Org. Chem.* **49**, 2897–2900.

MARCHAND, A. P. & HAYES, B. R. (1977). *Tetrahedron Lett.* pp. 1027–1030.

NAMETKINE, N. S., TYURINE, V. D., NEKHAEV, A. I., IVANOV, V. I. & BAYAOUOVA, F. S. (1976). *J. Organomet. Chem.* **107**, 377–391.

Nicolet Instrument Corporation (1986). *SHELXTL* for Desktop 30 (Microeclipse). Report PN269-1040340, April. Nicolet Instrument Corporation, WI, USA.

Acta Cryst. (1990). C**46**, 27–29

Structure of μ-1,2-Phenylene-bis(trifluoroacetato)dimercury(II)

BY SUZANNE DEGUIRE AND ANDRÉ L. BEAUCHAMP*

Département de Chimie, Université de Montréal, CP 6128, *Succ. A, Montréal, Québec, Canada H*3*C* 3*J*7

(*Received* 4 *April* 1989; *accepted* 12 *April* 1989)

Abstract. $[Hg_2(C_2F_3O_2)_2(C_6H_4)]$, $M_r = 703{\cdot}31$, monoclinic, $P2/n$, $a = 5{\cdot}071\,(4)$, $b = 11{\cdot}174\,(9)$, $c = 12{\cdot}750\,(5)$ Å, $\beta = 94{\cdot}10\,(5)^\circ$, $V = 720{\cdot}6$ Å^3, $Z = 2$, $D_x = 3{\cdot}241$ Mg m^{-3}, $\lambda(\text{Mo }K\alpha) = 0{\cdot}71069$ Å, $\mu = 21{\cdot}36$ mm^{-1}, $F(000) = 620$, $T = 175$ K, final $R = 0{\cdot}045$ for 989 unique observed reflections. One O atom of each CF_3CO_2 group forms a short Hg—O bond of 2·053 (12) Å, whereas the other takes part in bidentate Hg···O interactions [2·844 (11), 2·878 (13) Å] with both Hg atoms in a neighboring molecule.

Introduction. Bidentate Lewis acids containing the 1,2-phenylenedimercury unit can recognize and bind donor atoms in various organic molecules. For instance, dimethylformamide is bound in a cooperative manner by the adjacent Hg atoms of phenylenedimercury dichloride (Beauchamp, Olivier, Wuest & Zacharie, 1987). Tetrahydrofuran is similarly bound to pairs of Hg atoms in a tetradentate organomercury–perfluoroglutarate macrocycle (Wuest & Zacharie, 1987). The title trifluoroacetate compound attracted our attention because of its ability to activate thiocarbonyl groups towards nucleophilic attack (Wuest & Zacharie, 1985). This compound was expected to contain, like phenylmercury trifluoroacetate (Kamenar, Penavic & Hergold-Brundic, 1984), linear two-coordinate Hg atoms forming primary bonds with an aromatic C atom and one of the CF_3CO_2 O atoms. In order to determine how the residual Lewis acidity of the pair of Hg atoms would be expressed in this compound, its crystal structure was determined.

Experimental. Compound prepared by Wuest & Zacharie (1985), who kindly provided a sample of colorless crystals grown by recrystallization from acetonitrile. Plate, 0·33 × 0·27 × 0·019 mm. Enraf–Nonius CAD-4 diffractometer, reduced cell obtained from 25 centered reflections ($11 < \theta < 17^\circ$) found by the automatic search procedure, primitive monoclinic lattice deduced from the Niggli matrix, cell dimensions and monoclinic Laue symmetry checked with axial photographs, systematic absences determined from full data set ($h0l$, $h + l = 2n + 1$), consistent with space groups Pn and $P2/n$ (alternative definitions of Pc, No. 7, and $P2/c$, No. 13, respectively). Accurate cell dimensions by least-squares fit on the setting angles for the 25 reflections. Detailed procedure for data collection described elsewhere (Bélanger-Gariépy & Beauchamp, 1980), graphite-monochromatized Mo $K\bar{\alpha}$ radiation, $2\theta_{\max} = 50^\circ$, $0 \le h \le 6$, $0 \le k \le 13$, $-15 \le l \le 15$, orientation checked every 100 measurements, intensity of three standards checked every hour, max. fluctuation 3·7%, 1264 independent reflections measured, 989 with $I > 3{\cdot}0\sigma(I)$. Data corrected for Lp and absorption, transmission range 0·088–0·667.

---

* To whom correspondence should be addressed.

0108-2701/90/010027-03$03.00 © 1990 International Union of Crystallography

Table 1. *Refined coordinates* ($\times 10^3$, Hg $\times 10^5$) *and equivalent isotropic temperature factors* (Å^2 $\times 10^3$)

$U_{eq} = (1/3)\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	U_{eq}
Hg(1)	−453 (11)	9613 (6)	35548 (4)	37
F(1)	559 (4)	−230 (2)	509 (1)	124
F(2)	250 (2)	−295 (1)	411 (1)	89
F(3)	629 (5)	−257 (2)	352 (2)	171
O(1)	143 (2)	−62 (1)	417 (1)	54
O(2)	511 (2)	−33 (1)	340 (1)	55
C(1)	350 (4)	−98 (2)	391 (1)	68
C(2)	442 (3)	−226 (2)	412 (2)	61
C(11)	−139 (3)	258 (1)	291 (1)	65
C(12)	−56 (4)	366 (2)	330 (2)	59
C(13)	−142 (4)	466 (2)	288 (2)	66
H(12)†	69	371	389	76
H(13)†	−80	540	317	76

† Not refined.

Table 2. *Selected interatomic distances* (Å) *and bond angles* (°)

Hg(1)—O(1)	2·053 (12)	C(2)—F(3)	1·31 (3)
Hg(1)—C(11)	2·081 (16)	C(1)—O(1)	1·19 (2)
Hg(1)—O(2^{i})	2·844 (11)	C(1)—O(2)	1·30 (2)
Hg(1)—O(2^{ii})	2·878 (13)	C(1)—C(2)	1·52 (3)
Hg(1)—O(1^{iii})	3·057 (13)	C(11)—C(12)	1·36 (3)
C(2)—F(1)	1·33 (3)	C(11)—C(11^{iv})	1·48 (2)
C(2)—F(2)	1·24 (2)	C(12)—C(13)	1·30 (3)

O(1)—Hg(1)—C(11)	177·7 (6)	C(2)—C(1)—O(1)	122 (2)
O(1)—Hg(1)—O(2^{i})	83·1 (4)	C(2)—C(1)—O(2)	115 (2)
O(1)—Hg(1)—O(2^{ii})	83·1 (4)	F(1)—C(2)—F(2)	107 (2)
C(11)—Hg(1)—O(2^{i})	99·0 (5)	F(1)—C(2)—F(3)	104 (2)
C(11)—Hg(1)—O(2^{ii})	96·3 (5)	F(2)—C(2)—F(3)	116 (2)
O(2^{i})—Hg(1)—O(2^{ii})	74·3 (3)	C(1)—C(2)—F(1)	108 (2)
Hg(1)—O(1)—C(1)	119·2 (13)	C(1)—C(2)—F(2)	111 (2)
Hg(1)—C(11)—C(11^{iv})	119·3 (11)	C(1)—C(2)—F(3)	112 (2)
Hg(1)—C(11)—C(12)	122·8 (13)	O(1)—C(1)—O(2)	123 (2)
C(1)—O(2)—Hg(1^{v})	144·9 (12)	C(12)—C(11)—C(11^{iv})	117 (2)
C(1)—O(2)—Hg(1^{ii})	138·1 (12)	C(11)—C(12)—C(13)	122 (2)
Hg(1^{v})—O(2)—Hg(1^{ii})	76·2 (3)	C(12)—C(13)—C(13^{iv})	120 (2)

Symmetry code: (i) $-1+x, y, z$; (ii) $\frac{1}{2}-x, y, \frac{1}{2}-z$; (iii) $-x, -y, 1-z$; (iv) $-\frac{1}{2}-x, y, \frac{1}{2}-z$; (v) $1+x, y, z$.

Structure solved in the centrosymmetric *P*2/*n* space group, molecule on a twofold axis. Hg position determined from Patterson map, other non-H atoms from ΔF map. Structure refined anisotropically on $|F_o|$ by full-matrix least squares. H atoms fixed at idealized positions (C—H 0·95 Å, isotropic $B =$ 7 Å^2), repositioned after each least-squares cycle. Final $R = 0{\cdot}045$, $wR = 0{\cdot}056$ (weights based on counting statistics), $S = 1{\cdot}86$, (shift/σ), mean = 0·02, max. = 0·06. Residual electron density on final ΔF map: general background below $\pm 0{\cdot}6$ e Å^{-3}, a few peaks $\pm|0{\cdot}7$–$1{\cdot}4|$ e Å^{-3} near Hg. Absence of artificial disorder or unusual thermal motion rules out the lower-symmetry *Pn* space group.

Scattering factors of non-H atoms from Cromer & Mann (1968), of H from Stewart, Davidson & Simpson (1965). Anomalous-dispersion f' and f'' terms for Hg from Cromer & Liberman (1970). Programs used listed elsewhere (Authier-Martin & Beauchamp, 1977). Refined coordinates are given in Table 1.* Selected distances and angles are presented in Table 2.

Discussion. The compound consists of discrete molecules (Fig. 1) located on a crystallographic two-fold axis. Hg adopts a roughly linear coordination [O(1)—Hg(1)—C(11) = 177·7 (6)°]. The Hg—O(1) bond length [2·053 (12) Å] is shorter than in the two-coordinate phenylmercury acetate [2·08 (2) Å] and trifluoroacetate [2·12 (2) Å] molecules (Kamenar *et al.*, 1984). On the other hand, our Hg—C bonds [2·081 (16) Å] are slightly longer than those of the above monocarboxylates [2·04 (2), 2·05 (2) Å, respectively]. The aromatic ring is planar within 2σ

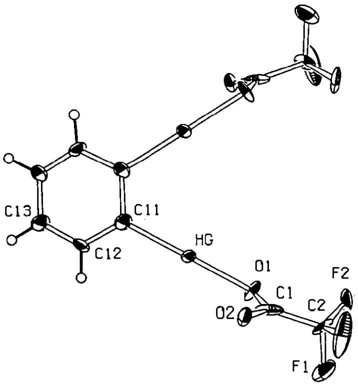

Fig. 1. *ORTEP* (Johnson, 1965) drawing of the molecule. The asymmetric unit corresponds to one-half of the molecule, the other half being generated by the crystallographic twofold axis. The ellipsoids correspond to 50% probability.

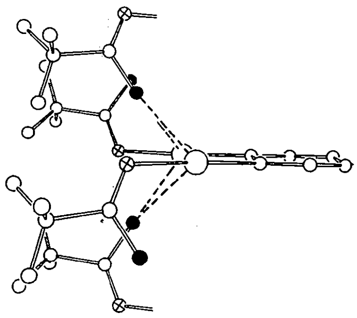

Fig. 2. Intermolecular Hg···O(2) interactions with 'free' carboxylate O atoms. Large atoms = Hg, black = O(2), crossed = O(1), H omitted.

---

* Lists of observed and calculated structure-factor amplitudes, anisotropic thermal parameters, and details of least-squares planes, as well as stereoviews of the unit cell, have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52141 (9 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

[0·04 Å], and the Hg atoms lie 0·0894 (6) Å above and below this plane, respectively.

The other trifluoroacetate O atom simultaneously forms secondary O(2)···Hg bonds of 2·844 (11) and 2·878 (13) Å with the pair of Hg atoms in the next molecule. Fig. 2 shows the environment of a molecule, including the Hg···O(2) interactions above and below the molecular plane. The O(2) atoms lie near the approximate mirror plane relating the two metal atoms, at 1·726 (12) Å from the molecular plane, and they define C(11)—Hg(1)—O(2) angles of 99·0 (5) and 96·3 (5)°. This arrangement probably optimizes the coordinative interactions with the Hg—Hg pair, while reducing steric hindrance to a minimum. The extra ligands were similarly positioned in the phenylenedimercury dichloride adducts with DMF or Cl^- (Beauchamp, Olivier, Wuest & Zacharie 1986, 1987) and in the THF complex with the Hg–perfluorosuccinate macrocycle (Wuest & Zacharie, 1987).

ORTEP drawings of the unit cell have been deposited.* Stacking along the *a* axis allows each pair of Hg atoms to interact simultaneously with carboxylate groups in the molecules above and below. Cohesion between these stacks originates from normal van der Waals contacts and complementary pairs of very long Hg···O(1) contacts of 3·057 (13) Å.

We wish to thank M. Simard for collecting the X-ray data.

References

AUTHIER-MARTIN, M. & BEAUCHAMP, A. L. (1977). *Can. J. Chem.* **55**, 1213–1217.

BEAUCHAMP, A. L., OLIVIER, M. J., WUEST, J. D. & ZACHARIE, B. (1986). *J. Am. Chem. Soc.* **108**, 73–77.

BEAUCHAMP, A. L., OLIVIER, M. J., WUEST, J. D. & ZACHARIE, B. (1987). *Organometallics*, **6**, 153–156.

BÉLANGER-GARIÉPY, F. & BEAUCHAMP, A. L. (1980). *J. Am. Chem. Soc.* **102**, 3461–3464.

CROMER, D. T. & LIBERMAN, D. (1970). *J. Chem. Phys.* **53**, 1891–1898.

CROMER, D. T. & MANN, J. B. (1968). *Acta Cryst.* **A24**, 321–324.

JOHNSON, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee, USA.

KAMENAR, B., PENAVIC, M. & HERGOLD-BRUNDIC, A. (1984). *Croat. Chem. Acta*, **57**, 145–152.

STEWART, R. F., DAVIDSON, E. R. & SIMPSON, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.

WUEST, J. D. & ZACHARIE, B. (1985). *J. Am. Chem. Soc.* **107**, 6121–6123.

WUEST, J. D. & ZACHARIE, B. (1987). *J. Am. Chem. Soc.* **109**, 4714–4715.

---

* See deposition footnote.

Acta Cryst. (1990). C**46**, 29–31

Structure of Potassium Hydrogen Pimelate

BY ALISTAIR L. MACDONALD AND ALISTAIR MURRAY

Chemistry Department, Clydebank High School, Shelley Drive, Clydebank G81 3EJ, Scotland

AND PAUL R. MALLINSON

Chemistry Department, University of Glasgow, Glasgow G12 8QQ, Scotland

(*Received* 11 *January* 1989; *accepted* 3 *May* 1989)

Abstract. $K^+.C_7H_{11}O_4^-$, $M_r = 198·27$, orthorhombic, *Cmma*, $a = 23·551\ (3)$, $b = 7·449\ (1)$, $c = 5·069\ (1)$ Å, $V = 889·3\ (2)$ Å^3, $Z = 4$, $D_m = 1·48$, $D_x = 1·48$ g cm^{-3}, $\lambda(\text{Mo } K\alpha) = 0·71069$ Å, $\mu = 5·65$ cm^{-1}, $F(000) = 416$, $T = 293$ K, $R = 0·029$, $wR = 0·041$ for 391 unique observed $[I/\sigma(I) > 2·0]$ reflections. Symmetrical hydrogen pimelate anions are linked into infinite chains parallel to **a** by symmetrical hydrogen bonds [OH···O 2·449 (3) Å]. The structure is that of a type A_2 acid salt and in accordance with this the IR spectrum is of Hadzi's type (ii).

Introduction. Acid salts (*MHY*) of dibasic carboxylic acids (H_2Y) with *M* a monovalent cation have been classified (Sime, Speakman & Parthasarathy, 1970) as type A_2 if they contain short and symmetric hydrogen bonds. These structures invariably show anomalous IR spectra. The cell constants of KH pimelate have been reported elsewhere (Speakman, 1972) with the suggestion that the salt was probably of type A_2, and isostructural with KH glutarate (Macdonald & Speakman, 1972). We report here the X-ray analysis of KH pimelate to validate this suggestion.

Experimental. Colourless, platy crystals were obtained by slow evaporation from a solution of pimelic acid in water/ethanol and potassium hydroxide in

0108-2701/90/010029-03$03.00 © 1990 International Union of Crystallography