

[2.250 (7)–2.294 (7) Å] are comparable with those [2.291 (5)–2.308 (5) Å] in [Pt<sub>2</sub>(CO)Cl( $\mu$ -dppm)<sub>2</sub>] (Manojlović-Muir, Muir & Solomun, 1979).

Perhaps the most interesting structural feature is the twisted configuration of the molecule as a whole. The two Pt<sub>2</sub>P<sub>2</sub>Cl coordination planes are mutually rotated about Pt(1)–Pt(2) to afford a dihedral angle of 38.6°. This, however, leads to only slight angular distortions in the Pt<sub>2</sub>P<sub>2</sub>C rings (see above). Thus the flexibility of the dppm ligands allows the molecule almost to minimize any antibonding interactions between filled interaxial *d* orbitals of the metal atoms. In the Pt<sup>I</sup> anion [Pt(CO)Cl<sub>2</sub>]<sub>2</sub><sup>2-</sup>, where the constraint of the bridging groups is not present, the corresponding dihedral angle is *ca* 60°; this has been interpreted as a compromise between the tendency of the filled interaxial metal *d* orbitals to be positioned as far from one another as possible (dihedral angle 45°) and the tendency of the ligands attached to the adjacent metal atoms to minimize their steric repulsions (dihedral angle 90°) (Modinos & Woodward, 1975).

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## Structure of (Chloromercurio)acetaldehyde

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**Abstract.** C<sub>2</sub>H<sub>3</sub>ClHgO, Hg(C<sub>2</sub>H<sub>3</sub>O)Cl, triclinic, *P* $\bar{1}$ , *a* = 8.147 (8), *b* = 6.377 (6), *c* = 4.511 (4) Å,  $\alpha$  = 92.0 (1),  $\beta$  = 81.3 (1),  $\gamma$  = 101.6 (1)°, *D*<sub>c</sub> = 4.09 Mg m<sup>-3</sup>, *Z* = 2; final *R* = 0.030 for 484 diffractometer-measured intensities (Mo *K* $\alpha$ ,  $\lambda$  = 0.7107 Å). Hg is coordinated to Cl [Hg–Cl = 2.326 (6) Å] and to C [Hg–C = 2.11 (2) Å] with the angle Cl–Hg–C = 172.2 (5)°, and also to O of *c*-related and centrosymmetrically related molecules [Hg–O = 2.87 (1) and 2.84 (2) Å respectively]. There is also an intermolecular Hg...Cl contact at 3.370 (6) Å.

**Introduction.** The title compound was supplied by Dr F. G. Thorpe, and had been prepared by the method of Makarova & Nesmayanov (1967). The crystals, used

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without further recrystallization, were thin laths twinned about *b*; with care the twins could be separated. The cell dimensions were obtained from Weissenberg photographs taken with Cu *K* $\alpha$  radiation ( $\lambda$  = 1.5418 Å); the crystal density was not measured as it was too high for the flotation method and the compound was not available in sufficient quantity for an alternative method. Intensities were collected on a Stoe STADI-2 automatic two-circle diffractometer with graphite-monochromatized Mo *K* $\alpha$  radiation. Although decomposition of the crystals occurred continuously under irradiation, it was sufficiently slow to permit all measurements to be made with one crystal with dimensions 0.31 × 0.03 × 1.13 mm. Of 537 intensities measured within the range  $2\theta$  = 4–40°, 484 had *I* >  $3\sigma(I)$  and were used for the structure determination. Absorption corrections ( $\mu$  = 33.095 mm<sup>-1</sup>) were applied with *SHELX* (Sheldrick, 1976), which was also

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used for data reduction. Subsequent calculations were carried out with XRAY 72 (Stewart, Kruger, Ammon, Dickinson & Hall, 1972). Patterson maps gave the coordinates of Hg which were refined with anisotropic  $U_{ij}$ ; these parameters were used to phase a Fourier map from which the coordinates of the Cl, C and O atoms were obtained. Least-squares refinement of all non-H atomic positions, anisotropic  $U_{ij}$  for Hg and Cl and isotropic  $U$  for C and O was alternated with refinement of the interlayer scale factors of the intensity data sets. Inclusion of H atoms (without refinement) at expected positions calculated with  $C-H = 1.0 \text{ \AA}$  and isotropic  $U$  values of  $0.05 \text{ \AA}^2$  was found to improve the agreement; convergence was reached at  $R = 0.030$ . Scattering factors for Hg (including anomalous dispersion) were those of Cromer & Mann (1968); values for all other atoms were taken from *International Tables for X-ray Crystallography* (1962). Final atomic coordinates and temperature factors are listed in Table 1.\*

**Discussion.** The satisfactory refinement of the structure in  $P1$  offers justification for the original assumption of that space group. The bond distances and angles within the molecule are shown in Fig. 1. The two covalent bonds from Hg to Cl and C(1) are slightly but significantly non-linear; each distance is within the typical covalent range for each type, e.g. of the 46 Hg-C bonds occurring in compounds for which quantitative structural data are available in the Cambridge Crystallographic Data File, 36 have values in the range  $2.0-2.2 \text{ \AA}$ . This file was accessed by the program CSSR (Machin, Mills, Mills & Elder, 1977). With the torsion angle  $Hg-C(1)-C(2)-O = 98.1^\circ$ , the O atom is twisted away from Hg with no intramolecular interaction.

\* A list of structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 34295 (5 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. Final atomic parameters ( $\times 10^3$ ; for Hg, Cl  $\times 10^4$ ) and temperature factors ( $U \times 10^3$ ;  $U_{ij}$  for Hg  $\times 10^4$ ;  $U_{ij}$  for Cl  $\times 10^3$ )

|      | $x$      | $y$      | $z$       | $U(\text{\AA}^2)$ |          |          |
|------|----------|----------|-----------|-------------------|----------|----------|
| C(1) | 215 (2)  | 212 (3)  | 466 (5)   | 25 (5)            |          |          |
| C(2) | 393 (3)  | 234 (4)  | 517 (6)   | 42 (6)            |          |          |
| O    | 441 (2)  | 313 (2)  | 760 (4)   | 40 (4)            |          |          |
| H(1) | 173      | 64       | 349       | 50                |          |          |
| H(2) | 132      | 213      | 679       | 50                |          |          |
| H(3) | 486      | 182      | 342       | 50                |          |          |
| Hg   | 2157 (1) | 4801 (1) | 2053 (2)  |                   |          |          |
| Cl   | 1808 (7) | 7512 (9) | 9032 (14) |                   |          |          |
|      | $U_{11}$ | $U_{22}$ | $U_{33}$  | $U_{12}$          | $U_{13}$ | $U_{23}$ |
| Hg   | 380 (6)  | 400 (6)  | 181 (6)   | 76 (4)            | -127 (4) | 20 (4)   |
| Cl   | 42 (3)   | 41 (3)   | 30 (3)    | 8 (3)             | -14 (3)  | 13 (3)   |

Table 2. Intermolecular distances ( $\text{\AA}$ ) and angles ( $^\circ$ ) around Hg

|   |           |                                    |          |
|---|-----------|------------------------------------|----------|
| Hg...O <sup>I</sup>                         | 2.84 (2)  | C(1)-Hg-O <sup>I</sup>             | 86.7 (6) |
| Hg...O <sup>II</sup>                        | 2.87 (1)  | C(1)-Hg-O <sup>II</sup>            | 96.3 (6) |
| Hg...Cl                                     | 3.370 (6) | Cl-Hg-O <sup>I</sup>               | 94.7 (4) |
| O <sup>I</sup> is at $x, y, z - 1$          |           | Cl-Hg-O <sup>II</sup>              | 91.4 (4) |
| O <sup>II</sup> is at $1 - x, 1 - y, 1 - z$ |           | O <sup>I</sup> -Hg-O <sup>II</sup> | 69.8 (4) |

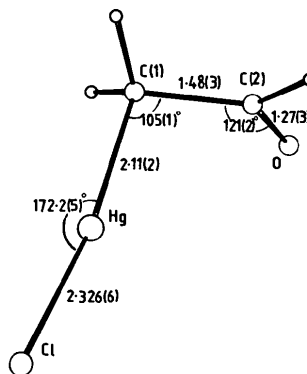


Fig. 1. The atom numbering, bond distances ( $\text{\AA}$ ) and angles ( $^\circ$ ) for  $C_2H_3ClHgO$ .

In the nomenclature of Grdenić (1965) Hg has a characteristic coordination of two, but within its effective coordination range are two O atoms of molecules related by  $c$  and a centre of symmetry respectively; the geometry of these contacts is listed in Table 2. A similar type of effective coordination has been reported in the 1:1 addition compound mercury(II) chloride-cyclononane (Dahl & Groth, 1971), in which each of two independent Hg atoms are bonded secondarily to two O atoms with a mean Hg-O distance of  $2.87 \text{ \AA}$ . Table 2 also lists a possible Hg...Cl contact; the distance involved is less than the sum of the van der Waals radii ( $3.55 \text{ \AA}$ ), taking that of Hg to be  $1.75 \text{ \AA}$  (Barr, Goldstein, Hairs, McPartlin & Markwell, 1974). Many similar contacts between centrosymmetrically related Hg-Cl pairs have been reported in chloromercury(II) organic compounds (e.g. Halfpenny & Small, 1978).

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## Structure of (2*R*,5*R*,8*S*,11*S*)-1,4,7,10-Tetrabenzyl-2,5,8,11-tetraethyl-1,4,7,10-tetraazacyclododecane

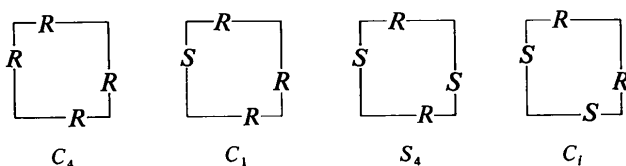
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**Abstract.** C<sub>44</sub>H<sub>60</sub>N<sub>4</sub>, monoclinic, *P*<sub>2</sub><sub>1</sub>/*c*, *a* = 9.345 (4), *b* = 9.327 (4), *c* = 22.358 (11) Å, β = 94.14 (8)°, *U* = 1944 (2) Å<sup>3</sup>, *D<sub>m</sub>* = 1.11, *D<sub>c</sub>* = 1.10 Mg m<sup>-3</sup>, *Z* = 2. The 12-membered ring forms an armchair conformation with the methylene groups at the top and the toe.

**Introduction.** This paper forms part of a series of investigations on the cyclic tetramers of chiral aziridines. These tetramers can be divided into four geometrical isomers as shown below (Tsuboyama, Tsuboyama, Uzawa & Higashi, 1974).



Recently, the two crystal structures with *C*<sub>4</sub> symmetry, *i.e.* (2*R*,5*R*,8*R*,11*R*)-2,5,8,11-tetraethyl-1,4,7,10-tetraazacyclododecane (TETA) (Sakurai, Kobayashi, Tsuboyama & Tsuboyama, 1978*a*), and (2*RS*,5*RS*,8*RS*,11*RS*)-1,4,7,10-tetrabenzyl-2,5,8,11-tetraethyl-1,4,7,10-tetraazacyclododecane (*RRRR*-type) (Sakurai, Kobayashi, Tsuboyama & Tsuboyama, 1978*b*) were reported. In this paper the structure of the title compound with *C*<sub>i</sub> symmetry (*RRSS*-type) is described.

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Triclinic single crystals were obtained from benzene solution (Tsuboyama *et al.*, 1974). However, many of these crystals were twinned or disordered and were not suitable for the structural work. Therefore they were recrystallized from dioxane solution, and a different crystal form was obtained. The new crystals belong to the monoclinic system *P*<sub>2</sub><sub>1</sub>/*c* with *Z* = 2, and possess intramolecular centrosymmetry. The size of the crystal used was 0.4 × 0.4 × 0.45 mm. X-ray diffraction data were measured on a Rigaku automated AFC four-circle diffractometer with graphite-monochromatized Mo *K*α radiation. Within the range 2θ ≤ 50°, 2132 independent reflections were observed. The intensities were corrected for Lorentz and polarization factors, but no absorption correction was applied.

The structure was solved by the direct phasing method, and all H atoms were deduced from successive difference Fourier syntheses. The structure was refined by the block-diagonal least-squares method. Unit weight was given to all reflections, and anisotropic temperature factors were used for all non-hydrogen atoms. The final *R* index was 4.8%. Atomic parameters are given in Table 1.†

**Discussion.** A stereoscopic drawing of the molecule is shown in Fig. 1. When projected on the mean plane of

† Lists of structure factors and anisotropic temperature factors have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 34202 (14 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.