Journal of Organometallic Chemistry, 319 (1987) 1–8
Elsevier Sequoia S.A., Lausanne – Printed in The Netherlands

TRIMERCURATED ACETALDEHYDE. THE CRYSTAL STRUCTURES OF $2[OHg_3CCHO]NO_3 \cdot HNO_3$ AND $[HOHg_2(NO_3Hg)CCHO]NO_3$

D. GRDENIĆ, D. MATKOVIĆ-ČALOGOVIĆ and M. SIKIRICA

Laboratory of General and Inorganic Chemistry, Faculty of Science, University of Zagreb, P.O. Box 153, 41001 Zagreb (Yugoslavia)

(Received July 15th, 1986)

Summary

Three new compounds $2[OHg_3CCHO]NO_3 \cdot HNO_3$ (**A**), $[HOHg_2(NO_3Hg)CCHO]NO_3$ (**B**) and $(NO_3Hg)_3CCHO$ (**C**), have been successively obtained by mercuration of acetaldehyde with mercuric nitrate containing increasing concentrations of nitric acid. All of them contain trimercurated acetaldehyde. The orthorhombic crystals of **A** built up of parallel infinite networks of the polymeric trimercurated oxonium $[OHg_3CCHO]_n^{n+}$ cation, while the monoclinic crystals of **B** contain polymeric dimercurated oxonium $[HOHg_2(NO_3Hg)CCHO]_n^{n+}$ cation in the form of an infinite helical chain. The structures were determined by X-ray diffraction and refined by full-matrix least-squares to the R value of 0.050 for **A** and 0.063 for **B**. The Hg–C and Hg–O distances are in the ranges 2.04 to 2.11 Å and 2.11 to 2.18 Å, respectively. The crystals of **C** were not suitable for X-ray structure analysis.

Introduction

Addition of an ethanolic solution of acetaldehyde to an aqueous solution of mercuric nitrate gave two products [1]. The crystal structure of one of them, $[OHg_3CCHO]NO_3 \cdot H_2O$, has been reported previously [2]. In our further investigations we have now used various concentrations of nitric acid and established the conditions for separate crystallization of these two products as well as for isolation of two new derivatives of trimercurated acetaldehyde.

Results and discussion

Mercuration of acetaldehyde with mercuric nitrate in aqueous nitric acid always leads to trimercurated acetaldehyde, which separates from the solution in form of various nitrates whose type and composition depends upon the acid concentration. It is known that the organomercuric oxyacid salts, e.g. RHgX with X = NO_3, ClO_4, SO_4 etc., are hydrolyzed in dilute aqueous solutions, but do not separate as hydroxides, RHgOH, but condense to "basic" salts, actually salts of the mercurated

0022-328X/87/$03.50

TABLE 1

DEPENDENCE OF THE FORMATION OF DIFFERENT TRIMERCURATED ACETALDEHYDES ON THE MASS FRACTION OF NITRIC ACID

Mass fraction of nitric acid $w(HNO_3)$ (%)	Compound
$w \leq 3$	$[OHg_3CCHO]NO_3 \cdot H_2O$, tetragonal [2]
$3 \leq w \leq 5$	$2[OHg_3CCHO]NO_3 \cdot HNO_3$, orthorhombic (**A**)
$5 \leq w < 60$	$[HOHg_2(NO_3Hg)CCHO]NO_3$, monoclinic (**B**)
$w = 60$	$(NO_3Hg)_3CCHO$, monoclinic (**C**)

oxonium, i.e. $(RHg)_2OH^+$ and $(RHg)_3O^+$ [3,4]. The condensation of tri- or tetramercurated organic compounds (a polymeric dimercurated species has not yet been observed) involves polymerisation, as in the case with the nitrate of trimercurated acetaldehyde [2], trimercurated acetic acid [5], and Hofmann's base and its salts [6,7]. The normal organomercuric salt, RHgX, is obtained when X = Cl, Br, I, CN and AcO, or when the oxyacid concentration is proportionally high. But when the mercuration product is insoluble the oxonium polymer persists even in highly concentrated oxyacid, e.g., the nitrate of Hofmann's base, $CHg_4O(NO_3)_2 \cdot xH_2O$ [6,8]. Analogous conditions were found to result in formation of inorganic mercurous and mercuric oxonium salts [9] distinguished by various Hg–O–Hg linkages in their crystal structure [10].

Thirty preparations of trimercurated acetaldehyde with various concentrations of nitric acid and mercuric nitrate were studied. Mercuric nitrate and acetaldehyde were always added in 3/1 molar ratio. Four different compounds were found to crystallize in the mass fraction range of nitric acid $w(HNO_3)$ given in Table 1. The nitrate-to-mercury ratio increases from 1/3 for the first two members of the series to 2/3 for the third, and to 3/3 for the last member. The trimercurated oxonium is formed at low nitric acid concentrations, the dimercurated oxonium and one nitratomercury group in a higher concentration of nitric acid as in **B** and finally use of concentrated nitric acid results in formation of three nitratomercury groups, to give **C**. The analogous effect of the nitric acid concentration on the separation of the nitrates of trimercurated acetic acid from nitric acid solutions has been observed previously [5].

Descriptions of the structures

The crystal structure of compound **A**, $2[OHg_3CCHO]NO_3 \cdot HNO_3$, projected along the *a*-axis direction is shown in Fig. 1(a). The infinite network of oxonium cations, $[OHg_3CCHO]_n^{n+}$ shown separately in Fig. 1(b), belongs to the $[Hg_3O_2]_n^{2n+}$ type [10,11] except that every second oxygen atom is replaced by a carbon atom of the aldehyde group, with the same conformation of the net. The aldehyde groups are directed outwards on the same side of the net. The bond angles at the oxygen and the carbon are close to the tetrahedral, the mean values being 108.7 and 109.9° (the interatomic distances and bond angles are given in Table 2). In the structure there are two crystallographically different cation nets stacked in pairs and parallel to each other with their CHO groups pointing in opposite directions. Since the carbonyl oxygen atoms were found to have disordered positions they are assumed to

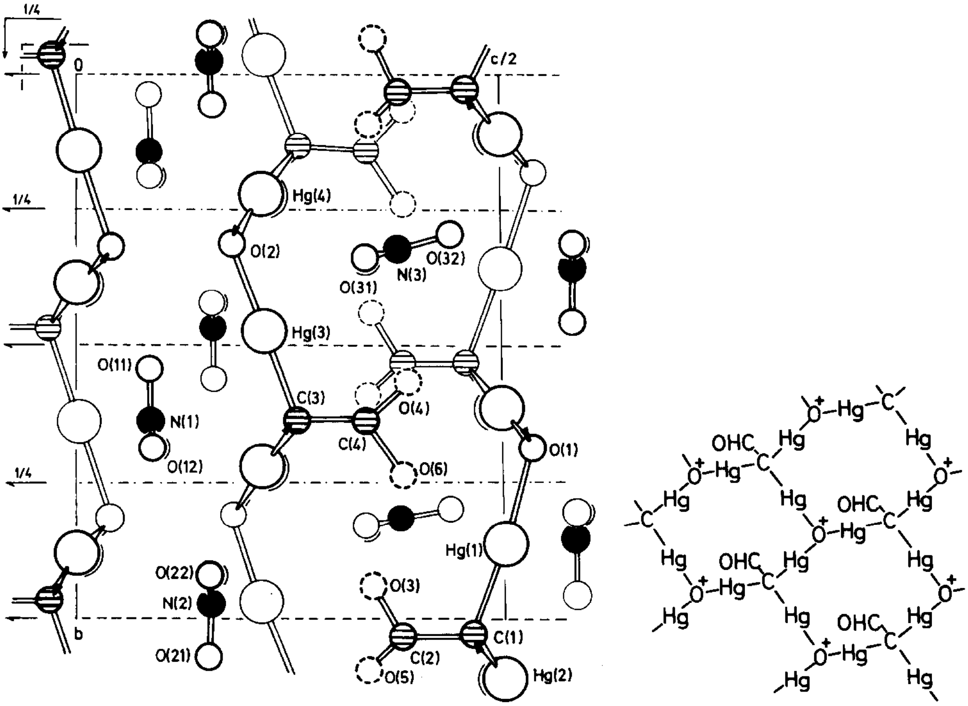

Fig. 1. (a) The structure of $2[OHg_3CCHO]NO_3 \cdot HNO_3$ (**A**) projected along the *a*-axis direction; (b) $[OHg_3CCHO]_n^{n+}$ oxonium cation.

come in pairs O(3) and O(4) in one net pair and O(5) and O(6) in the other. The oxonium Hg_3O^+ pyramids, with atoms O(1) and O(2), point outwards from the double-net layer with the closest $O \cdots O$ contact of 2.61 Å between the layers. The nitrate ions are in the vacancies between these double-net layers. The nitric acid molecules occupy the vacancies within the layer. The hydrogen atom of the nitric acid molecule, probably in disordered positions, has not been located but, from the $O \cdots O$ distances of 2.76, 2.87 and 2.89 Å, it is assumed to take part in the hydrogen bonding with the aldehyde oxygen or with one of the nitrate ion oxygen. It is interesting to compare the cations in the two nitrates of trimercurated acetaldehyde; the tetragonal [2] and the present orthorhombic one. The $[OHg_3CCHO]_n^{n+}$ stoichiometry is the same for both of them because the column in the former is actually a coiled net from the structure of the latter.

The crystal structure of **B**, $[HOHg_2(NO_3Hg)CCHO]NO_3$, projected along the *b*-axis direction is shown in Fig. 2(a). The cation is an infinite helical chain in which the units of trimercurated acetaldehyde are connected through the $(-Hg-OH-Hg-)^+$ oxonium link by means of a 2_1 symmetry operation along the *b*-axis direction, as shown separately in Fig. 2(b). One free organomercuric cationic centre, $C-Hg^+$, is neutralized by the N(1) nitrate ion which functions as a nitrato ligand, while the other N(2) nitrate is present as an anion in the cavities between the cationic polymers. The interatomic distances and angles, shown in Table 2, have normal values [10,12]. The hydrogen atoms of the aldehyde group and the oxonium species

TABLE 2

SELECTED INTERATOMIC DISTANCES (Å) AND BOND ANGLES (°) (e.s.d.s are given only for the refined atom positions)

*Compound **A***					
Hg(1)–C(1)	2.11	Hg(1)···O(3)	2.99	C(1)–Hg(1)–O(1)	179.3
Hg(2)–C(1)	2.05	Hg(1)···O(6)	2.78	C(1)–Hg(2)–O(1)	177.3
Hg(3)–C(3)	2.12	Hg(1)···O(12$^{\text{viii}}$)	2.88(4)	C(3)–Hg(3)–O(2)	176.6
Hg(4)–C(3$^{\text{i}}$)	2.10	Hg(1)···O(21$^{\text{ix}}$)	2.89(9)	O(2)–Hg(4)–C(3$^{\text{i}}$)	174.9
Hg(1)–O(1)	2.18	Hg(2)···O(4$^{\text{ii}}$)	2.76	Hg(1)–O(1)–Hg(2$^{\text{iii}}$)	108.5
Hg(2)–O(1$^{\text{ii}}$)	2.16	Hg(2)···O(11$^{\text{viii}}$)	2.90(6)	Hg(2$^{\text{iii}}$)–O(1)–Hg(2$^{\text{iv}}$)	107.7
Hg(3)–O(2)	2.13	Hg(2)···O(12$^{\text{ix}}$)	2.94(4)	Hg(3)–O(2)–Hg(4)	110.2
Hg(4)–O(2)	2.16	Hg(2)···O(22$^{\text{ix}}$)	2.96(6)	Hg(4)–O(2)–Hg(4$^{\text{v}}$)	108.3
C(1)–C(2)	1.50	Hg(3)···O(22$^{\text{iii}}$)	2.78(6)	Hg(1)–C(1)–Hg(2)	110.1
C(3)–C(4)	1.46	Hg(3)···O(11)	2.81(8)	Hg(2)–C(1)–Hg(2$^{\text{v}}$)	110.7
C(2)–O(3)	1.22	Hg(3)···O(31)	2.81(6)	Hg(1)–C(1)–C(2)	111
C(2)–O(5)	1.18	Hg(4)···O(31)	2.79(6)	Hg(2)–C(1)–C(2)	107
C(4)–O(4)	1.19	Hg(4)···O(12$^{\text{iv}}$)	2.57(4)	C(1)–C(2)–O(3)	119
C(4)–O(6)	1.27	Hg(4)···O(22$^{\text{iv}}$)	2.80(6)	C(1)–C(2)–O(5)	125
N(1)–O(11)	1.15(13)	Hg(4)···O(21$^{\text{x}}$)	2.77(7)	Hg(3)–C(3)–Hg(4$^{\text{vi}}$)	108.3
N(1)–O(12)	1.29(6)	O(1)···O(2$^{\text{xi}}$)	2.61	Hg(4$^{\text{vi}}$)–C(3)–Hg(4$^{\text{vii}}$)	105.8
N(2)–O(21)	1.22(11)	O(3)···O(6)	2.62	Hg(3)–C(3)–C(4)	110
N(2)–O(22)	1.21(7)	O(31)···O(3$^{\text{i}}$)	2.76	Hg(4$^{\text{vi}}$)–C(3)–C(4)	112
N(3)–O(31)	1.20(9)	O(31)···O(6$^{\text{i}}$)	2.87	C(3)–C(4)–O(4)	133
N(3)–O(32)	1.22	O(32)···O(12$^{\text{xi}}$)	2.89	C(3)–C(4)–O(6)	123
				O(11)–N(1)–O(12)	123(4)
				O(12)–N(1)–O(12$^{\text{v}}$)	113(7)
				O(21)–N(2)–O(22)	119(4)
				O(22)–N(2)–O(22$^{\text{v}}$)	119(7)
				O(31)–N(3)–O(32)	126
				O(31)–N(3)–O(31$^{\text{v}}$)	108(9)
*Compound **B***					
Hg(1)–C(1)	2.07(2)	Hg(1)···O(22)	2.82(4)	C(1)–Hg(1)–O(1)	173.5(9)
Hg(2)–C(1)	2.04(3)	Hg(1)···O(21$^{\text{xiv}}$)	2.82(4)	C(1)–Hg(2)–O(11)	170.9(9)
Hg(3)–C(1$^{\text{xii}}$)	2.04(2)	Hg(1)···O(11$^{\text{xvi}}$)	2.92(3)	C(1$^{\text{xii}}$)–Hg(3)–O(1)	173.7(8)
Hg(1)–O(1)	2.11(2)	Hg(1)···O(13$^{\text{xviii}}$)	2.99(5)	Hg(1)–O(1)–Hg(3)	121(1)
Hg(3)–O(1)	2.04(2)	Hg(1)···O(2$^{\text{xvii}}$)	2.71(4)	Hg(1)–C(1)–Hg(2)	109(1)
Hg(2)–O(11)	2.18(3)	Hg(2)···O(12)	2.86(6)	Hg(1)–C(1)–Hg(3$^{\text{xiii}}$)	110(1)
C(1)–C(2)	1.57(4)	Hg(2)···O(22$^{\text{xv}}$)	2.96(3)	Hg(2)–C(1)–Hg(3$^{\text{xiii}}$)	112(1)
C(2)–O(2)	1.31(4)	Hg(2)···O(1$^{\text{xv}}$)	2.93(4)	Hg(1)–C(1)–C(2)	106(2)
N(1)–O(11)	1.29(4)	Hg(3)···O(23$^{\text{xvi}}$)	2.93(3)	Hg(3$^{\text{xiii}}$)–C(1)–C(2)	103(1)
N(1)–O(12)	1.20(3)	Hg(3)···O(13$^{\text{xviii}}$)	2.92(3)	Hg(2)–C(1)–C(2)	115(2)
N(1)–O(13)	1.28(3)	O(1)···O(2$^{\text{xvi}}$)	3.03(5)	C(1)–C(2)–O(2)	123(2)
N(2)–O(21)	1.30(3)	C(2)···O(21$^{\text{xviii}}$)	2.94(5)	O(11)–N(1)–O(12)	122(2)
N(2)–O(22)	1.24(4)			O(11)–N(1)–O(13)	112(2)
N(2)–O(23)	1.24(3)			O(12)–N(1)–O(13)	124(2)
				O(21)–N(2)–O(22)	119(2)
				O(21)–N(2)–O(23)	115(2)
				O(22)–N(2)–O(23)	125(2)

Transformation of the asymmetric unit (x, y, z): (i) $x+0.5$, $y-0.5$, z; (ii) $x+0.5$, $y+0.5$, z; (iii) $x-0.5$, $y-0.5$, z; (iv) $0.5-x$, $y-0.5$, z; (v) $-x$, y, z; (vi) $x-0.5$, $y+0.5$, z; (vii) $0.5-x$, $y+0.5$, z; (viii) $0.5-x$, $1.5-y$, $z+0.5$; (ix) $-x$, $2-y$, $z+0.5$; (x) x, $y-1$, z; (xi) $-x$, $1-y$, $z+0.5$; (xii) $-x$, $y-0.5$, $0.5-z$; (xiii) $-x$, $y+0.5$, $0.5-z$; (xiv) $x-1$, y, z; (xv) $1-x$, $y+0.5$, $0.5-z$; (xvi) $1-x$, $y-0.5$, $0.5-z$; (xvii) x, $1.5-y$, $z+0.5$; (xviii) $x-1$, $1.5-y$, $z-0.5$

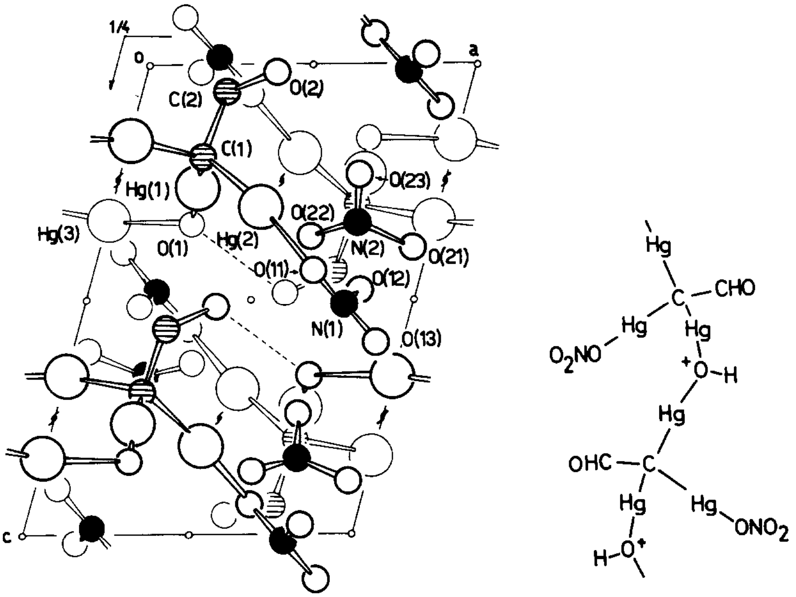

Fig. 2. (a) The structure of [HOHg$_2$(NO$_3$Hg)CCHO]NO$_3$ (**B**) projected along the *b*-axis direction; (b) [HOHg$_2$(NO$_3$Hg)CCHO]$_n^{n+}$ oxonium cation.

were not located. The O(1) · · · O(2) distance of 3.03(5) Å between the carbonyl and oxonium oxygen atoms can be associated with a hydrogen bridge.

The crystal structure of compound **C**, $(NO_3Hg)_3CCHO$, obtained from the solution in 60% nitric acid, could not be determined because the crystals available gave inadequate reflections. By the analogy with tris(nitratomercury)acetic acid [5], which is formed under similar conditions, the above formula is assumed. This formula is supported also by the good agreement between calculated and observed density and by the chemical analysis.

Experimental

Preparations

2[OHg$_3$CCHO]NO$_3$ · HNO$_3$ (A). Acetaldehyde (0.214 g) was added to Hg $(NO_3)_2 \cdot H_2O$ (5 g) in 5% HNO_3 (10 ml). After a few days at room temperature colourless crystals were obtained. Found: Hg, 80.05; N, 2.67. calc.: Hg, 80.00; N, 2.39%.

[HOHg$_2$(NO$_3$Hg)CCHO]NO$_3$ (B). When a similar solution to that used to give **A** was left for several days the initial crystals of **A** progressively dissolved and colourless crystals of **B** suitable for single crystal diffraction were formed. When solutions of higher concentrations of $Hg(NO_3)_2 \cdot H_2O$ and higher concentration of HNO_3 were used, only **B** is obtained after only a few hours at room temperature, but the crystals were much smaller.

(NO$_3$Hg)$_3$CCHO (C). Acetaldehyde (0.064 g) was added to $Hg(NO_3)_2 \cdot H_2O$ (1.5 g) in 60% HNO_3 (5 ml). The solution was left at a temperature of 4°C and after 2 d colourless prismatic crystals were formed. Found: Hg, 72.3; calc: 72.6%.

X-ray diffraction experiments

For identification of the various crystalline products X-ray powder diffraction was used. From suitable crystals of **A** and **B** the preliminary data were determined by oscillation and Weissenberg techniques and the final data by using Philips PW 1100 automatic diffractometer (graphite-monochromatized Mo-K_α radiation, λ 0.7107 Å).

Single crystal data. (**A**) $C_4H_3Hg_6N_3O_{13}$, mol. wt. 1504.62, orthorhombic, a 6.853(2), b 12.043(3), c 18.508(12) Å, V 1527.48 Å^3, D_o 6.57 g cm^{-3}, D_c 6.543 g cm^{-3}, $Z = 4$, space group $Cmc2_1$, $F(000) = 2528$, Mo-K_α radiation, λ 0.7107 Å, μ(Mo-K_α) 580.3 cm^{-1}, crystal dimensions (mm from centroid): (001) 0.041; $(00\bar{1})$ 0.047; $(02\bar{1})$, $(0\bar{2}\bar{1})$ 0.058; $(\bar{1}1\bar{1})$, $(1\bar{1}\bar{1})$, $(11\bar{1})$, $(\bar{1}\bar{1}\bar{1})$ 0.105; maxium and minimum transmission coeficients, 0.086 and 0.015.

(**B**) $C_2H_2Hg_3N_2O_8$, mol wt. 783.81, monoclinic, a 7.215, b 11.643, c 10.821 Å, β 105.00(3)°, V 877.36 Å^3, D_c 5.934 g cm^{-3}, $Z = 4$, space group $P2_1/c$, $F(000) = 1328$, Mo-K_α radiation, λ 0.7107 Å, μ(Mo-K_α) 505.3 cm^{-1}, crystal dimensions (mm from centroid): (100), $(\bar{1}00)$, (001), $(00\bar{1})$, 0.118; (010), $(0\bar{1}0)$ 0.044; maximum and minimum transmission coefficients, 0.068 and 0.005.

(**C**) $C_2HHg_3N_3O_{10}$, mol. wt. 828.81, monoclinic, a 10.57, b 12.42, c 8.15 Å, β 104.6°, V 1035.4 Å^3, D_o 5.31 g cm^{-3}, D_c 5.32 g cm^{-3}, $Z = 4$, (probably one of the C-centered space groups).

Intensity measurements. Intensities were measured by the θ–2θ technique, with scan range 1.2°, scan speed 0.04° s^{-1} in the range $2 < \theta < 30°$ for both **A** and **B**. Rapid decomposition of compound **C** allowed measurements of only 25 reflections, of which 12 were used for determining the cell parameters. (The others resulted from crystal twinning.) Data for **A** and **B** were corrected for Lorentz, polarization and absorption effects. For **A** 2060 reflections were measured, of which independent 1024 reflections with $I > 3\sigma(I)$, were used for the solution of the structure. Crystals of **A** darkened during data collection as a result of slow decomposition, the intensities of the standard reflections weakening by 12.4%. For **B** no significant variation of intensities was found, and 1742 unique (out of 1989 collected) reflections with $I > 3\sigma(I)$ were used in the refinement.

Determination and refinement of the structures. Mercury atoms in **A** and **B** were located by a combination of the heavy atom method and direct methods (MULTAN 80 [13]). Atom parameters were refined by the full-matrix least-squares method using SHELX 76 program [14]. For **A** three cycles of refinement of the Hg positions with isotropic and three with anisotropic thermal parameters gave an R value of 0.088. Other non-hydrogen atoms were found from a three-dimensional difference Fourier map. Positional and isotropic thermal parameters of all the nitrate atoms (except for one oxygen atom of the nitric acid molecule whose positions were fixed) were included in the refinement, to give $R = 0.054$. The carbonyl oxygen atoms were located in two positions and were fixed with half of the normal occupancy and were given an isotropic factor of 0.05. Atom O(5) is also disordered, being statistically located above and below the mirror plane. The positions of the oxonium oxygen and the acetaldehyde carbon atoms were fixed (as found from the difference Fourier map) and only their isotropic temperature factors were refined. The final R value was 0.050. In the structure of **B** refinement of the mercury atom positions with isotropic and then anisotropic temperature factors gave $R = 0.100$. All of the non-hydrogen atoms were found from a difference Fourier map and were included

TABLE 3

ATOMIC COORDINATES AND THERMAL PARAMETERS (Å^2) (with e.s.d.s in parentheses for the refined parameters)

Atom	x	y	z	U_{eq} or U
*$2[OHg_3CCHO]NO_3 \cdot HNO_3$ (**A**)*				
Hg(1)	0.0	0.8605(2)	0.5022(3)	0.0129(6) [a]
Hg(2)	0.2461(3)	1.1064(1)	0.5	0.0128(4) [a]
Hg(3)	0.0	0.4716(2)	0.2263(2)	0.0144(6) [a]
Hg(4)	0.2556(3)	0.2186(1)	0.2190(1)	0.0140(4) [a]
O(1)	0.0	0.690	0.542	0.01(1)
O(2)	0.0	0.308	0.183	0.01(1)
C(1)	0.0	1.026	0.465	0.02(1)
C(2)	0.0	1.031	0.384	0.03(2)
C(3)	0.0	0.638	0.263	0.02(2)
C(4)	0.0	0.641	0.342	0.02(2)
O(3)	0.0	0.944	0.350	0.05
O(4)	0.0	0.570	0.387	0.05
O(5)	0.082	1.098	0.349	0.05
O(6)	0.0	0.731	0.378	0.05
N(1)	0.0	0.635(8)	0.087(6)	0.07(2)
O(11)	0.0	0.540(7)	0.081(4)	0.04(2)
O(12)	0.157(5)	0.694(3)	0.085(2)	0.02(1)
N(2)	0.0	0.973(6)	0.161(4)	0.02(1)
O(21)	0.0	1.073(7)	0.152(5)	0.06(2)
O(22)	0.153(9)	0.923(5)	0.155(3)	0.05(1)
N(3)	0.0	0.325(9)	0.377(6)	0.08(2)
O(31)	0.142(8)	0.340(5)	0.340(3)	0.05(1)
O(32)	0.0	0.295	0.440	0.06
*$[HOHg_2(NO_3Hg)CCHO]NO_3$ (**B**)*				
Hg(1)	0.2496(1)	0.6177(1)	0.2735(1)	0.0213(3) [a]
Hg(2)	0.4693(1)	0.8747(1)	0.3114(1)	0.0280(3) [a]
Hg(3)	0.0093(1)	0.3574(1)	0.3264(1)	0.0220(3) [a]
C(1)	0.252(3)	0.781(2)	0.197(2)	0.021(4)
C(2)	0.267(4)	0.763(3)	0.056(3)	0.036(6)
O(1)	0.257(3)	0.445(2)	0.333(2)	0.031(4)
O(2)	0.408(3)	0.804(2)	0.014(2)	0.043(5)
N(1)	0.795(4)	0.932(2)	0.516(2)	0.035(5)
O(11)	0.665(3)	0.992(2)	0.439(2)	0.036(5)
O(12)	0.831(3)	0.835(2)	0.492(2)	0.038(5)
O(13)	0.904(3)	0.994(2)	0.602(2)	0.034(4)
N(2)	0.764(3)	0.623(2)	0.335(2)	0.027(4)
O(21)	0.945(3)	0.609(2)	0.392(2)	0.039(5)
O(22)	0.643(4)	0.567(2)	0.372(2)	0.048(6)
O(23)	0.729(4)	0.692(2)	0.244(2)	0.049(6)

[a] Equivalent isotropic thermal parameters, $U_{eq} = (\Sigma_i \Sigma_j \beta_{ij} \mathbf{a}_i \cdot \mathbf{a}_j)/6\pi^2$.

in the refinement with isotropic temperature factors giving the final $R = 0.063$ and $R_w = 0.068$ $(w = 1/\sigma^2(F) + 0.0176F^2)$. Scattering factors and correction for anomalous dispersion were taken from Cromer and Mann [15] and Cromer and Liberman [16]. The atomic coordinates and thermal parameters are listed in Table 3. A list of observed and calculated structure factors can be obtained from the author on request. Calculations were carried out on the UNIVAC 1110 of the University Computing Center in Zagreb.

Acknowledgements

We thank Mrs. Dr. B. Korpar-Čolig for help in the chemical analysis of the mercuration products and Mr. M. Bruvo MSc for the diffraction data collection. The investigation was supported by the Foundation for Scientific Research of SR Croatia.

References

1 K.A. Hofmann, Ber. Deut. Chem. Gesel., 31 (1898) 2212.

2 D. Grdenić, M. Sikirica, D. Matković-Čalogović and A. Nagl, J. Organomet. Chem., 253 (1983) 283.

3 D. Grdenić and F. Zado, J. Chem. Soc., (1962) 521.

4 J.H.R. Clarke and L.A. Woodward, Trans. Faraday Soc., 62 (1966) 3022.

5 D. Grdenić, M. Sikirica and D. Matković-Čalogović, J. Organomet. Chem., 306 (1986) 1.

6 D. Grdenić, B. Kamenar, B. Korpar-Čolig, M. Sikirica and G. Jovanovski, J. Chem. Soc., Chem. Commun., (1974) 646.

7 D. Grdenić, M, Sikirica and B. Korpar-Čolig, J. Organomet. Chem., 153 (1978) 1.

8 K.A. Hofmann, Ber. Deut. Chem. Gesel., 31 (1898) 1904; 33 (1900) 1328.

9 G. Johansson, Acta Chem. Scand., 25 (1971) 1905, 2787, 2799.

10 D. Grdenić, in D. Dodson, J.P. Glusker and D. Sayre (Eds.), Connections in the Crystal Structures of Mercury Compounds, Structural Studies of Molecules of Biological Interest, a Volume in Honour of Professor Dorothy Hodgkin, Clarendon Press, Oxford, 1981, p. 207.

11 H. Behm, Acta Cryst., C39 (1983) 1319.

12 L.G. Kuz'mina and Yu.T. Struchkov, Croat. Chim. Acta, 57 (1984) 701.

13 P. Main, S.J. Fiske, S.E. Hull, L. Lesinger, G. Germain, J.-P. Declercq and M.M. Woolfson, MULTAN 80. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data, Univs. of York, England, and Louvain, Belgium, 1980.

14 G.M. Sheldrick, SHELX76. Program for Crystal Structure Determination, Univ. of Cambridge, England.

15 D.T. Cromer and J.B. Mann, Acta Cryst., A24 (1968) 321.

16 D.T. Cromer and D. Libermann, J. Chem. Phys., 53 (1970) 1891.