TRIMERCURATED ACETIC ACID. THE CRYSTAL STRUCTURES OF [Hg(H$_2$OHg)(NO$_3$Hg)CCOO]NO$_3$ AND 2(NO$_3$Hg)$_3$CCOOH · HNO$_3$

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

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

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Summary

Two nitrates of trimercurated acetic acid, obtained from the solution of mercuretin in nitric acid, were identified as [Hg(H$_2$OHg)(NO$_3$Hg)CCOO]NO$_3$ (A) and 2(NO$_3$Hg)$_3$CCOOH · HNO$_3$ (B) by X-ray diffraction. The cation of A is an infinite chain composed of the –Hg(H$_2$OHg)(NO$_3$Hg)CCOO– units joined through the C–Hg–O linkages with mean bond lengths of 2.08(3) (C–Hg) and 2.11(3) Å (Hg–O). The chains are connected by hydrogen bonding between the water molecule and the carboxyl oxygen with O–H · · · O distances of 2.61(4) Å. The structure of B consists of discrete molecules of (NO$_3$Hg)$_3$CCOOH. One half of the molecules in the unit cell join up to give centrosymmetrical pairs through the carboxyl groups, with O–H · · · O bonds of 2.61(7) Å, and the other half are hydrogen-bonded to the nitric acid via the carboxyl group, with an O–H · · · O distance of 2.59(8) Å.

Introduction

The formulae as well as the structure of some highly mercurated aliphatic compounds, i.e., tetramercurated methane [1], trimercurated acetaldehyde [2] and trimercurated acetic acid [3], have been determined recently by means of X-ray diffraction, but the formulae of some derivatives of mercurated acetic acid were still uncertain. We have now examined the nitrates formulated as NO$_3$Hg(OHg$_2$)CCOOH [4] and NO$_3$Hg(HgOH)$_2$CCOOH [5,6], and those formulated as (NO$_3$Hg)$_3$CCOOH- ·(NO$_3$Hg)$_2$CHCOOH and Hg$_5$(NO$_3$)$_2$C$_2$O$_4$ [7]; the last two formulae were proposed for the products of hydrolysis of mercuretin with concentrated and diluted nitric acid, respectively. Since mercuretin is a condensation polymer of tris(acetoxymercuri)acetic acid [3], we expected the two compounds to be nitrates of trimercurated acetic acid.

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Results and discussion

Trimercurated acetic acid was described as a microcrystalline powder when isolated from the crude mercuration product of ethanol [6], acetaldehyde [4], or sodium acetate [5] either by acidifying the alkaline filtrate or by diluting the nitric-acid extract of the solid obtained. In both cases the precipitation was too fast to allow formation of crystals suitable for structure analysis. But when the nitric-acid extract [5] was only moderately diluted with water, as in our experiments, then left overnight, transparent well developed crystals separated (**A**). They were shown to have the formula $\{[Hg(H_2OHg)(NO_3Hg)CCOO]NO_3\}_n$ by the X-ray structure analysis reported below.

The infinite $-Hg(Hg)(Hg)CCOO-$ chain, found in the structure of the cation of **A** had been already assumed to be present in the structure of mercuretin [3]. We also obtained the crystals of compound **A** from the solution of mercuretin in dilute nitric acid, thus showing that **A** was actually previously prepared in this way but described as $Hg_5(NO_3)_2C_2O_4$ [7].

Well-developed crystals which we separated from the solution of mercuretin in concentrated nitric acid, described previously as a double nitrate of di- and trimercurated acetic acid [7], are actually of the solvate of tris(nitratomercuri)acetic acid, $2(NO_3Hg)_3CCOOH \cdot HNO_3$ (**B**), as shown below.

Description of the structures

The crystal structure of compound **A**, $[Hg(H_2OHg)(NO_3Hg)CCOO]NO_3$, projected along the a-axis direction is shown in Fig. 1. The interatomic distances and bond angles are given in Table 1. The $-Hg(Hg)HgCCOO-$ units of the mercuretin-type chain are connected through $Hg(3)-O(3)^{iv}$ bonds of 2.11(3) Å between mercury and carboxyl and repeated along the c-glide plane. One of the two remaining mercury atoms is bound to the nitrate ion, $NO_3(1)$, and the other, $Hg(2)$, is bonded to the water molecule, $Hg(1)-O(11)$ distance being 2.12(2) Å and the $Hg(2)-O(1)$ distance 2.17(3) Å. Both values are close to the sum of the correspond-

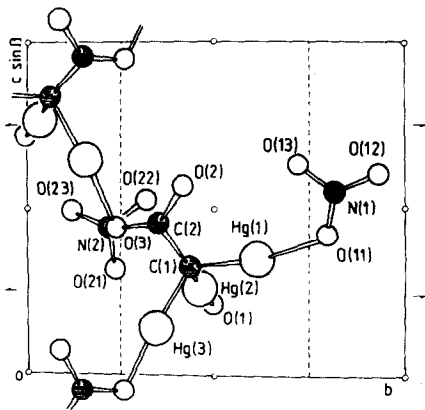Fig. 1. The structure of $[Hg(H_2OHg)(NO_3Hg)CCOO]NO_3$ (A) projected along the c-axis direction. Only about one half of the cell content is shown.

TABLE 1

SELECTED INTERATOMIC DISTANCES (Å) AND BOND ANGLES (°), WITH E.S.D.'S IN PARENTHESES

Compound A

Hg(1)–C(1)	2.05(3)	Hg(1) $\cdots$ Hg(2)	3.288(3)	C(1)–Hg(1)–O(11)	172(1)
Hg(2)–C(1)	2.11(3)	Hg(2) $\cdots$ Hg(3)	3.223(3)	C(1)–Hg(2)–O(1)	175(1)
Hg(3)–C(1)	2.08(3)	Hg(1) $\cdots$ Hg(3)	3.521(3)	C(1)–Hg(3)–O(3)^{iv}	171(1)
Hg(1)–O(11)	2.12(2)	Hg(1) $\cdots$ O(2)^{i}	2.91(3)	Hg(1)–C(1)–C(2)	112(2)
Hg(2)–O(1)	2.17(3)	Hg(1) $\cdots$ O(12)^{iii}	2.99(3)	Hg(1)–C(1)–Hg(2)	104(1)
Hg(3)–O(3)^{iv}	2.11(3)	Hg(1) $\cdots$ O(13)	2.77(3)	Hg(1)–C(1)–Hg(3)	117(1)
C(1)–C(2)	1.51(4)	Hg(1) $\cdots$ O(21)^{v}	2.75(3)	Hg(2)–C(1)–Hg(3)	101(1)
C(2)–O(2)	1.25(4)	Hg(2) $\cdots$ O(21)	2.69(3)	Hg(2)–C(1)–C(2)	107(2)
C(2)–O(3)	1.26(4)	Hg(2) $\cdots$ O(22)	2.98(4)	Hg(3)–C(1)–C(2)	114(2)
		Hg(2) $\cdots$ O(23)^{iv}	2.74(4)	C(1)–C(2)–O(3)	116(3)
		Hg(3) $\cdots$ O(2)^{iv}	2.87(2)	C(1)–C(2)–O(2)	119(3)
		Hg(3) $\cdots$ O(12)^{iii}	3.01(3)	O(3)–C(2)–O(2)	125(3)
		O(1) $\cdots$ O(2)^{ii}	2.61(4)	C(2)–O(3)–Hg(3)^{vii}	109(2)
		O(1) $\cdots$ O(11)^{vi}	2.87(3)		

Compound B

Hg(1)–C(1)	2.08(5)	Hg(1) $\cdots$ Hg(2)	3.369(4)	C(1)–Hg(1)–O(11)	175(2)
Hg(2)–C(1)	2.06(5)	Hg(1) $\cdots$ Hg(3)	3.327(4)	C(1)–Hg(2)–O(21)	178(2)
Hg(3)–C(1)	2.07(5)	Hg(2) $\cdots$ Hg(3)	3.380(3)	C(1)–Hg(3)–O(31)	172(2)
Hg(4)–C(3)	2.05(5)	Hg(4) $\cdots$ Hg(5)	3.446(4)	C(3)–Hg(4)–O(41)	177(2)
Hg(5)–C(3)	2.08(5)	Hg(4) $\cdots$ Hg(6)	3.274(3)	C(3)–Hg(5)–O(51)	174(2)
Hg(6)–C(3)	2.08(5)	Hg(5) $\cdots$ Hg(6)	3.332(4)	C(3)–Hg(6)–O(61)	173(2)
Hg(1)–O(11)	2.15(3)	Hg(1) $\cdots$ O(12)	2.70(6)	Hg(1)–C(1)–Hg(2)	109(2)
Hg(2)–O(21)	2.14(5)	Hg(1) $\cdots$ O(33)^{xiv}	2.82(5)	Hg(1)–C(1)–Hg(3)	107(2)
Hg(3)–O(31)	2.13(5)	Hg(2) $\cdots$ O(13)^{vi}	2.99(7)	Hg(1)–C(1)–C(2)	112(3)
Hg(4)–O(41)	2.10(5)	Hg(2) $\cdots$ O(23)	2.77(6)	Hg(2)–C(1)–Hg(3)	110(2)
Hg(5)–O(51)	2.14(5)	Hg(2) $\cdots$ O(63)^{x}	2.93(5)	Hg(2)–C(1)–C(2)	110(4)
Hg(6)–O(61)	2.03(5)	Hg(3) $\cdots$ O(21)^{viii}	2.86(5)	Hg(3)–C(1)–C(2)	110(4)
C(1)–C(2)	1.57(8)	Hg(3) $\cdots$ O(22)^{viii}	2.98(3)	C(1)–C(2)–O(1)	113(5)
C(2)–O(1)	1.32(9)	Hg(3) $\cdots$ O(32)	2.80(6)	C(1)–C(2)–O(2)	115(6)
C(2)–O(2)	1.12(8)	Hg(3) $\cdots$ O(62)^{x}	2.80(5)	O(1)–C(2)–O(2)	131(7)
C(3)–C(4)	1.55(8)	Hg(4) $\cdots$ O(23)^{xiii}	2.83(6)	Hg(4)–C(3)–Hg(5)	113(2)
C(4)–O(3)	1.28(8)	Hg(4) $\cdots$ O(42)	2.82(5)	Hg(4)–C(3)–Hg(6)	105(2)
C(4)–O(4)	1.21(7)	Hg(5) $\cdots$ O(53)	2.68(3)	Hg(4)–C(3)–C(4)	113(4)
		Hg(5) $\cdots$ O(61)^{ix}	2.95(5)	Hg(5)–C(3)–Hg(6)	107(2)
		Hg(5) $\cdots$ O(62)^{ix}	2.86(5)	Hg(5)–C(3)–C(4)	114(3)
		Hg(5) $\cdots$ O(73)	2.88(5)	Hg(6)–C(3)–C(4)	104(4)
		Hg(6) $\cdots$ O(43)^{xi}	2.87(6)	C(3)–C(4)–O(3)	117(5)
		Hg(6) $\cdots$ O(63)	2.84(5)	C(3)–C(4)–O(4)	117(5)
		O(4) $\cdots$ O(3)^{xii}	2.61(7)	O(3)–C(4)–O(4)	125(6)
		O(4) $\cdots$ O(1)	2.81(8)		
		O(73) $\cdots$ O(2)	2.74(8)		
		O(72) $\cdots$ O(2)	2.59(8)		
		O(71) $\cdots$ O(72)^{xiii}	2.64(7)		

Transformation of the asymmetric unit (x, y, z): (i) $2 - x$, $1 - y$, $1 - z$; (ii) $1 - x$, $1 - y$, $1 - z$; (iii) x, $1.5 - y$, $z - 0.5$; (iv) x, $0.5 - y$, $z - 0.5$; (v) $1 - x$, $0.5 + y$, $0.5 - z$; (vi) $1 - x$, $y - 0.5$, $0.5 - z$; (vii) x, $0.5 - y$, $0.5 + z$; (viii) x, $1 + y$, z; (ix) x, $y - 1$, z; (x) $x - 1$, $y - 1$, z; (xi) $2 - x$, $1 - y$, $- z$; (xii) $1 - x$, $1 - y$, $- z$; (xiii) $1 - x$, $- y$, $- z$; (xiv) $- x$, $y - 0.5$, $0.5 - z$.
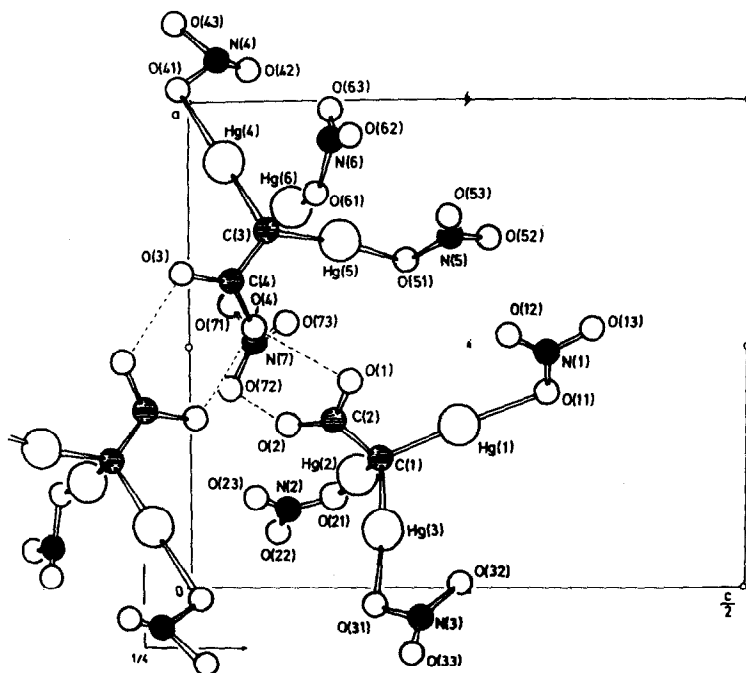
Fig. 2. The asymmetric unit in the structure of $2(NO_3Hg)_3CCOOH \cdot HNO_3$ viewed along the *b*-axis direction. Hydrogen bonds are indicated by dashed lines.

ing covalent radii [8] and, according to the conventional criteria [9], both ligands, the water molecule and the nitrate ion, must be considered as belonging to the chain and are therefore put in square brackets of the formula A. The other nitrate ion, $NO_3(2)$, does not belong to the chain, since the shortest oxygen-to-mercury distance of 2.69(3) Å is typical of a weak coordinative interaction to mercury [8]. The geometry of the Hg_3CC tetrahedron is fully consistent with previous data [2,3,10], the Hg–C interatomic distances and the Hg–C–Hg bond angles (mean values) being 2.08(3) Å and 109(2)°, respectively. The chains are linked through the mercurated oxonium ion (water molecule) and the carboxyl oxygen by $O(1)H \cdots O(2)^{ii}$ hydrogen bonds of 2.61(4) Å. The N–O distances and O–N–O angles in the nitrate ions are normal, with the mean values of 1.25(5) Å and 120(3)°, respectively, and are therefore not given in Table 1.

The crystal structure of **B** is built up (Fig. 2) of two crystallographically different types of molecule, and the contents of the unit cell can be conveniently formulated as $2((NO_3Hg)_3CCOOH)_2 \cdot 4((NO_3Hg)_3CCOOH \cdot HNO_3)$. The molecules in the first moiety are connected centrosymmetrically in pairs through hydrogen bonding with $O(3)^{xiii} \cdots O(4)$ distance of 2.61(7) Å, like the double molecules in the acetic acid vapour [11]. The other moiety consists of the same $(NO_3Hg)_3CCOOH$ molecules still solvated with nitric acid through hydrogen bonding, with an $O(72) \cdots O(2)$ distance of 2.59(8) Å. The moieties are interconnected by a $O(1)H \cdots O(4)$ hydrogen bond of 2.81(8) Å. The nitrate ion is bound to mercury at the mean Hg–O distance of 2.12(5) Å, i.e., virtually as in **A**. The mean Hg–C distance of 2.07(5) Å is also similar to that in **A**. The N–O distance in all nitrate ions is in the range

1.13(7)–1.34(7) Å, with a mean value of 1.24(8) Å. The O–N–O angle is 120(6)°. There is a lengthening of the N–O bond at the oxygen bound to the mercury atom, the mean distance being 1.28(7) Å.

Experimental

Preparation

[Hg(H$_2$OHg)(NO$_3$Hg)CCOO]NO$_3$ (A). A solution of mercuretin (0.5 g) in 20% nitric acid (10 ml) was covered with a layer of water and left overnight at room temperature. The colourless prism-shaped crystals were filtered off and dried.

2(NO$_3$Hg)$_3$CCOOH · HNO$_3$ (B). A solution of mercuretin (3.0 g) in conc. nitric acid (20 ml) was heated on a water bath for 1 h. The solution was then left at room temperature for several hours, and the colourless crystals which separated were filtered off, washed in conc. nitric acid, and air-dried. Analysis. Found: Hg, 68.8, calcd.: Hg, 68.7%.

X-ray diffraction experiments

X-ray single crystal diffractometry. Preliminary crystal data were determined by oscillation and Weissenberg techniques and finally on a Philips PW 1100 computer-controlled diffractometer (graphite-monochromatized Mo-K_α radiation, λ 0.7107 Å). Since crystals of compound **B** were not stable in air or under X-rays, two crystals of similar size were used for data collection.

Single crystal data. (A) C$_2$H$_2$Hg$_3$N$_2$O$_9$, mol. wt. 799.83, monoclinic, *a* 9.233(9), *b* 10.706(4), *c* 9.608(4) Å, β 104.24(4)°, *V* 920.6 Å^3, D_c 5.77 g cm^{-3}, *Z* = 4 formula units, space group *P*2$_1$/*c* (No. 14), *F*(000) = 1360, Mo-K_α radiation, λ 0.7107 Å, μ(Mo-K_α) 510.4 cm^{-1}, crystal dimensions (mm from centroid): (011), (0$\bar{1}\bar{1}$) 0.0380; (01$\bar{1}$), (0$\bar{1}$1) 0.0450; ($\bar{3}$23), (3$\bar{2}\bar{3}$), ($\bar{3}$23), (3$\bar{2}\bar{3}$) 0.0975, maximum, minimum transmission coefficients 0.083, 0.020.

(B) C$_4$H$_3$Hg$_6$N$_7$O$_{25}$, mol. wt. 876.32, monoclinic, *a* 11.555(6), *b* 7.670(3), *c* 27.004(9) Å, β 90.23(1)°, *V* 2393.3 Å^3, D_c 4.86 g cm^{-3}, *Z* = 4 formula units, space group *P*2$_1$/*c* (No. 14). *F*(000) = 3024, Mo-K_α radiation, λ 0.7107 Å, μ(Mo-K_α) 370.8 cm^{-1}, crystal dimensions (mm from centroid): crystal I (010), (0$\bar{1}$0) 0.1982; (100), ($\bar{1}$00) 0.0439; (10$\bar{4}$), ($\bar{1}$04) 0.0396; (102), ($\bar{1}$0$\bar{2}$) 0.0545; crystal II (010), (0$\bar{1}$0) 0.1682; (100), ($\bar{1}$00) 0.0311; (10$\bar{4}$), ($\bar{1}$04) 0.0361; (102), ($\bar{1}$0$\bar{2}$) 0.0481; maximum, minimum transmission coefficients 0.102, 0.041 for I and 0.169, 0.087 for II.

Intensity measurements. Integrated intensities were measured using the Θ–2Θ technique with scan range 1.6° (scan speed 0.04° s^{-1}, 2 < Θ < 30° for **A** and 0.08° s^{-1}, 2 < Θ < 26° for **B**). 1814 reflections of which 1710 unique with $I > 3\sigma(I)$ were used for solving the structure of **A**. Lorentz, polarization and absorption corrections were applied. For structure **B** 1800 reflections from crystal I (up to h,k,l = 7,0,12) and 1500 reflections from crystal II (collection started at h,k,l = 11,1,11) were used. Each set was separately corrected for decay and Lorentz, polarization, and absorption factors, and then brought on the same scale. A total of 2108 reflections with $I > 3\sigma(I)$ were used for solving the structure.

Determination and refinement of the structures. Mercury atoms were located by the heavy atom method (**A**), and direct methods using MULTAN 80 [12] and the heavy atom method (**B**). Other non-hydrogen atoms were found from a three-dimensional difference Fourier map. All atoms were refined by a full-matrix least-squares

TABLE 2

ATOMIC COORDINATES AND THERMAL PARAMETERS (Å²) WITH E.S.D.'S IN PARENTHESES

Atom	x	y	z	U_{eq} or U	Atom	x	y	z	U
1 [(H₂OHg)(NO₃Hg)HgCCOO]NO₃ (A)									
Hg(1)	0.8346(1)	0.6118(1)	0.3639(1)	0.0278(4) [a]	N(1)	0.923(3)	0.818(3)	0.550(3)	0.031(6)
Hg(2)	0.5153(1)	0.4604(1)	0.2743(1)	0.0267(4) [a]	O(11)	0.900(3)	0.799(2)	0.417(3)	0.032(5)
Hg(3)	0.7748(1)	0.3463(1)	0.1408(1)	0.0236(3) [a]	O(12)	0.949(3)	0.925(3)	0.601(3)	0.041(6)
O(1)	0.278(3)	0.503(2)	0.225(3)	0.038(6)	O(13)	0.924(3)	0.728(3)	0.629(3)	0.042(6)
O(2)	0.842(3)	0.406(2)	0.578(3)	0.030(5)	N(2)	0.428(4)	0.222(3)	0.444(4)	0.038(7)
O(3)	0.766(3)	0.240(2)	0.441(3)	0.028(5)	O(21)	0.396(3)	0.239(3)	0.316(3)	0.047(7)
C(1)	0.749(3)	0.435(3)	0.326(3)	0.023(6)	O(22)	0.472(4)	0.317(3)	0.527(4)	0.062(9)
C(2)	0.789(3)	0.355(3)	0.460(3)	0.019(5)	O(23)	0.401(3)	0.124(3)	0.500(4)	0.054(8)
2(NO₃)₃CCOOH·HNO₃ (B)									
Hg(1)	0.3413(2)	0.1817(4)	0.2407(1)	0.0466(10) [a]	O(23)	0.193(5)	-0.230(8)	0.061(2)	0.07(2)
Hg(2)	0.2274(2)	-0.0806(3)	0.1531(1)	0.0407(9) [a]	N(3)	-0.059(4)	0.555(7)	0.207(2)	0.04(1)
Hg(3)	0.1199(2)	0.3334(3)	0.1726(1)	0.04038(7) [a]	O(31)	-0.039(4)	0.462(6)	0.166(1)	0.05(1)
Hg(4)	0.8817(2)	0.3494(3)	0.0330(1)	0.0355(8) [a]	O(32)	0.003(5)	0.528(7)	0.242(2)	0.08(1)
Hg(5)	0.7095(2)	0.2858(4)	0.1353(1)	0.0399(8) [a]	O(33)	-0.137(4)	0.663(6)	0.203(2)	0.06(1)
Hg(6)	0.7832(2)	0.6822(3)	0.0960(1)	0.0397(8) [a]	N(4)	1.092(5)	0.173(8)	0.016(2)	0.06(1)
C(1)	0.269(4)	0.174(7)	0.170(2)	0.03(1)	O(41)	1.029(4)	0.279(8)	-0.008(2)	0.07(2)
C(2)	0.354(6)	0.249(9)	0.130(2)	0.05(2)	O(42)	1.065(4)	0.121(7)	0.056(2)	0.06(1)
O(1)	0.428(5)	0.364(8)	0.148(2)	0.08(2)	O(43)	1.172(5)	0.115(8)	-0.009(2)	0.11(2)
O(2)	0.335(5)	0.210(9)	0.091(2)	0.12(2)	N(5)	0.718(4)	0.164(7)	0.234(2)	0.05(1)
C(3)	0.740(4)	0.433(7)	0.072(2)	0.05(1)	O(51)	0.668(5)	0.117(7)	0.196(2)	0.06(1)
C(4)	0.632(5)	0.461(8)	0.039(2)	0.03(1)	O(52)	0.707(4)	0.074(7)	0.269(2)	0.07(1)
O(3)	0.649(4)	0.492(6)	-0.007(2)	0.04(1)	O(53)	0.762(4)	0.299(6)	0.232(1)	0.04(1)
O(4)	0.538(4)	0.439(6)	0.058(2)	0.04(1)	N(6)	0.914(4)	0.966(7)	0.131(2)	0.04(1)
N(1)	0.493(4)	0.257(7)	0.318(2)	0.04(1)	O(61)	0.804(4)	0.934(7)	0.118(2)	0.06(1)
O(11)	0.402(3)	0.181(6)	0.316(1)	0.04(1)	O(62)	0.928(4)	1.117(6)	0.148(2)	0.05(1)
O(12)	0.524(5)	0.344(8)	0.282(2)	0.07(1)	O(63)	0.982(4)	0.860(6)	0.132(2)	0.05(1)
O(13)	0.541(6)	0.252(9)	0.359(2)	0.09(2)	N(7)	0.517(4)	-0.066(6)	0.062(2)	0.10(1)
N(2)	0.162(4)	-0.357(7)	0.087(2)	0.05(1)	O(71)	0.578(4)	-0.173(6)	0.038(2)	0.12(1)
O(21)	0.189(4)	-0.345(6)	0.134(2)	0.06(1)	O(72)	0.416(3)	-0.043(7)	0.038(2)	0.10(1)
O(22)	0.123(4)	-0.494(6)	0.075(1)	0.05(1)	O(73)	0.548(4)	0.052(6)	0.088(2)	0.11(1)

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

method using programs X-RAY76 [13] (A) and SHELX 76 [14] (B). The final refinement with anisotropic thermal parameters for the mercury atoms and isotropic for all the other atoms resulted in values of $R = 0.070$, $R_w = 0.087$ ($w = 1/\sigma(F)^2$) for A and $R = 0.088$, $R_w = 0.092$ ($w = 1.0536/(\sigma(F)^2 + 0.008185\,F^2)$) for B. Scattering factors and corrections for anomalous dispersion were taken from Cromer and Mann [15] and Cromer and Libermann [16]. The atomic coordinates and thermal parameters are listed in Table 2. A list of observed and calculated structure factors can be obtained from the authors on request. Calculations were carried out on the UNIVAC 1110 of the University Computing Center, Zagreb.

Acknowledgements

References

1 (a) D. Grdenić, B. Kamenar, B. Korpar-Čolig, M. Sikirica and G. Jovanovski, J. Chem. Soc., Chem. Commun., (1974) 646; Cryst. Struct. Commun., 11 (1982) 565; (b) D. Grdenić, M. Sikirica and B. Korpar-Čolig, J. Organomet. Chem., 153 (1978) 1; (c) D. Grdenić and M. Sikirica, Z. Kristallogr., 150 (1979) 107; (d) D.K. Breitinger, G. Petrikowski, G. Liehr and R. Sendelbeck, Z. Naturforsch. B, 38 (1983) 357; (e) J. Mink, Z. Meić, M. Gál and B. Korpar-Čolig, J. Organomet. Chem., 256 (1983) 203.

2 (a) D. Grdenić, B. Korpar-Čolig, M. Sikirica and M. Bruvo, J. Organomet. Chem., 238 (1982) 327; (b) M. Sikirica and D. Grdenić, Cryst. Struct. Commun., 11 (1982) 1571; (c) D. Grdenić, M. Sikirica, D. Matković-Čalogović and A. Nagl, J. Organomet. Chem., 253 (1983) 283.

3 D. Grdenić, B. Korpar-Čolig and M. Sikirica, J. Organomet. Chem., 276 (1984) 1.

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

5 K.A. Hofmann, Ber. Deut. Chem. Gesel., 32 (1899) 870.

6 K.A. Hofmann, Ber. Deut. Chem. Gesel., 33 (1900) 1328.

7 J.E. Marsh and R. de J. Fleming Struthers, J. Chem. Soc., (1927) 2658.

8 D. Grdenić, Quart. Rev., 19 (1965) 303.

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

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

11 L. Pauling, The Nature of the Chemical Bond, 3rd Ed., Cornell University Press, Itaka N.Y., 1960, p. 477.

12 P. Main, S.J. Fiske, S.E. Hull, L. Lesinger, G. Germain, J.-P. Declercq, and M.M. Woolfson, MULTAN80. 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.

13 J.M. Stewart, P.A. Machin, C.W. Dickinson, H.L. Ammon, H. Heck and H. Flack, The XRAY76 system, Tech. Rep. TR-446, Computer Science Center, Univ. of Maryland, College Park, Maryland.

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

15 D.T. Cromer and J.B. Mann, Acta Cryst., A, 24 (1968) 321.

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