1952; Cahn, 1954) twinning due to phase inversion in feldspars occurs the more easily the smaller the degree of triclinicity. It is therefore concluded that the K feldspars studied in the present work have crystallized (or recrystallized) directly with triclinic symmetry and have not inverted from a primary monoclinic disordered phase through ordering due to a diffusive process. It follows that the various degrees of order of the Adamello crystals studied here, and presumably of the Spencer U, Pontiskalk and Pellotsalo microclines studied previously, represent the order corresponding to the equilibrium conditions at the moment of crystallization or of a possible subsequent recrystallization.

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The Crystal Structure of Strontium Uranyl Tetraformate Hydrate: $SrUO_2(HCOO)_4.(1 + x)H_2O$

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The crystal structure of strontium uranyl tetraformate hydrate has been established by X-ray diffraction at room temperature. This compound crystallizes in the orthorhombic space group *Pbca* with $a = 6{\cdot}449$ (1), $b = 16{\cdot}393$ (3) and $c = 20{\cdot}740$ (3) Å; $Z = 8$. The structure has been determined by the heavy-atom method from 2352 Mo $K\bar{\alpha}$ intensities measured on a four-circle automatic diffractometer and refined by full-matrix least-squares computations. The final weighted residual is 0·0468 (conventional $R = 0{\cdot}0463$). The U atom is surrounded by a pentagonal bipyramid of O atoms. The apical O atoms are those of the uranyl group, which is not quite linear [O—U—O = 175·5 (2)°]. The formato groups all bridge either two U atoms or U and Sr atoms. The O atom of the water molecule (O*W*) belongs to the coordination polyhedron of the Sr atom [Sr—O*W* = 2·509 (5) Å], which is a capped octahedron (seven-coordinate polyhedron) of O atoms. The structure reveals the presence of two kinds of channels running parallel to the [100] direction; one of them allows the presence of interstitial water (xH_2O) as shown by chemical analysis.

Introduction

During the structural studies on photosensitive uranyl formato complexes undertaken at the Laboratoire de Cinétique et Génie Chimiques, Lyon, (Mentzen, 1977; Mentzen, Puaux & Loiseleur, 1977) strontium uranyl tetraformate hydrate $SrUO_2(HCOO)_4.(1+x)H_2O$ with $x = 0{\cdot}38$ has been synthesized. This compound is also sensitive to visible, ultra-violet and X radiations. As shown by chemical analysis (Claudel, Mentzen, Puaux & Sautereau, 1977) the water content of the investigated complex corresponds to 1·38 H_2O per formula unit and, in order to explain the presence of this fractional water and obtain some information on the action of water during the photolytic process, the structural study of this complex has been undertaken.

Results concerning the preliminary IR and powder-diffraction studies have already been published (Claudel, Mentzen, Puaux & Sautereau, 1977). The interpretation of the $\nu_{as}(OH)$ vibration at 3600 cm^{-1} indicates the presence of a strongly polarized water molecule; this phenomenon has also been observed in the case of $NaUO_2(HCOO)_3.H_2O$ (Mentzen, 1977). The presence of five δ(OCO) bands at 798, 772, 766, 754 and 749 cm^{-1} is a result of the presence of different formato groups in the solid.

Experimental results

Using the preparation already described (Claudel, Mentzen, Puaux & Sautereau, 1977) we obtained small prismatic plates of rectangular shape. We selected a crystal (0·25 × 0·35 × 0·13 mm) and mounted it on the four-circle Enraf–Nonius CAD-4 automatic diffractometer of the Centre de Diffractométrie Automatique, Lyon. 3576 intensities corresponding to hkl, $h\bar{k}l$, $hk\bar{l}$ and $h\bar{k}\bar{l}$ (half the reflexion sphere) were collected with the automatic 'flat' program, for $1 < \theta < 35°$ and $I > 2\sigma(I)$. After data reduction 2352 reflexions were obtained, among which 692 are unique, and their intensities corrected for Lorentz and polarization factors as well as for absorption ($\mu R = 1{\cdot}66$). Furthermore, the intensities were also corrected for the weakening of the intensity-control reflexion resulting from the photolytic effect of the X-rays on the crystal. Inspection of the data collected shows that the conditions limiting possible reflexions are consistent with the space group *Pbca* previously ascribed (Claudel, Mentzen, Puaux & Sautereau, 1977), *i.e.* $0kl$: $k = 2n$, $h0l$: $l = 2n$, $hk0$: $h = 2n$; the unit-cell parameters are: $a = 6{\cdot}449\ (1)$, $b = 16{\cdot}393\ (3)$, $c = 20{\cdot}740\ (3)$ Å, $Z = 8$, $U = 2192{\cdot}5$ Å^3; $M_r = 555{\cdot}74$ (for $x = 0$); $D_m = 3{\cdot}303$, $D_x = 3{\cdot}367$ g cm^{-3}; $F(000) = 1984$; $\mu(\text{Mo } K\bar{\alpha}) = 187{\cdot}9$ cm^{-1}; $t = 18$ °C.

Table 1. *Relative atomic coordinates, equivalent isotropic thermal parameters and standard deviations for the uranium, strontium, oxygen and carbon atoms*

	x	y	z	B_{eq} (Å^2)
U	0·18694 (4)	0·14975 (1)	0·09635 (1)	1·5
Sr	0·23110 (9)	0·37288 (3)	0·18029 (3)	1·9
OU(1)	0·0383 (8)	0·1120 (2)	0·0330 (2)	4·4
OU(2)	0·3226 (7)	0·1941 (3)	0·1593 (2)	2·9
O(1)	0·0997 (7)	0·0358 (2)	0·1640 (2)	0·5
O(2)	0·437 (1)	0·0495 (3)	0·0797 (3)	1·7
O(3)	0·1219 (6)	0·2913 (2)	0·0709 (2)	2·5
O(4)	−0·1142 (7)	0·1867 (2)	0·1498 (2)	3·5
O(5)	0·4238 (8)	0·2056 (3)	0·0136 (3)	0·8
O(6)	0·4019 (7)	0·2941 (2)	0·2815 (2)	2·1
O(7)	0·0392 (7)	0·4532 (2)	0·2644 (2)	3·7
O(8)	−0·0838 (7)	0·4454 (3)	0·1238 (2)	2·9
C(1)	0·049 (1)	0·3261 (4)	0·0221 (3)	1·1
C(2)	0·313 (1)	0·2334 (4)	0·3052 (3)	2·8
C(3)	−0·086 (1)	0·5083 (4)	0·0911 (3)	4·1
C(4)	0·031 (1)	0·5204 (4)	0·2925 (3)	3·6
O*W*	0·5467 (8)	0·3569 (3)	0·1107 (2)	3·3

Structure determination

The interpretation of the Patterson function gave the atomic positions of the heavy atoms. A Fourier synthesis with the structure factors and the phases corresponding to the heavy U and Sr atoms revealed the positions of all but the hydrogen atoms.* The atomic coordinates and the isotropic thermal parameters of the 17 atoms of the asymmetric unit were refined by minimizing $\sum w(|F_o| - |F_c|)^2$, the weighting function being $w = (2|F_o|_{\min} + |F_o| + 2F_o^2/|F_o|_{\max})^{-1}$. A difference-Fourier synthesis did not reveal the location of the non-stoichiometric $x(H_2O)$. After eight refinement iterations the anisotropic thermal parameters and the weighting function $w = (a + b|F_o|)^2$ (Stout & Jensen, 1968) were introduced. Correction for anomalous dispersion did not improve the model and was therefore rejected. The final residual $R_w = [\sum w(|F_o| - |F_c|)^2/\sum w|F_o|^2]^{1/2}$ is 0·0468, the conventional $R = \sum(||F_o| - |F_c||/\sum|F_o|)$ being 0·0463. The refined parameters are listed in Table 1; the atomic scattering factors used were those given in *International Tables for X-ray Crystallography* (1974).

Description of the structure

The main interatomic distances and angles are given in Table 2. As is the case for many uranyl complexes (Drew, 1977), the seven-coordinated uranium atom is at the center of a pentagonal bipyramid (Fig. 1). The

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* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 33511 (13 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

apical OU(1) and OU(2) oxygen atoms are those of the uranyl group. The oxygen atoms O(1) through O(5) belong to the formato groups and the fact that each of these oxygen atoms corresponds to a different formato group explains why the pentagonal base is relatively undistorted. It might also be noticed that the uranyl group is far from being strictly linear [OU(1)–U–OU(2) = 175·5 (2)°].

Table 2. *Interatomic distances* (Å) *and angles* (°)

U–O(1)*	2·403 (4)	OU(1)–U–OU(2)	175·5 (2)
U–O(2)	2·328 (6)	O(1)–U–O(2)	72·5 (2)
U–O(3)	2·416 (3)	O(2)–U–O(5)	74·4 (2)
U–O(4)	2·317 (4)	O(5)–U–O(3)	66·4 (2)
U–O(5)	2·473 (6)	O(3)–U–O(4)	73·0 (1)
U–OU(1)	1·740 (4)	O(4)–U–O(1)	74·2 (1)
U–OU(2)	1·732 (4)	OU(2)–U–O(1)	90·3 (2)
OU(2)···O(1)	2·968 (6)	OU(2)–U–O(2)	93·3 (2)
OU(2)···O(2)	2·981 (7)	OU(2)–U–O(5)	93·2 (2)
OU(2)···O(5)	3·097 (7)	OU(2)–U–O(3)	81·3 (2)
OU(2)···O(3)	2·752 (6)	OU(2)–U–O(4)	87·0 (2)
OU(2)···O(4)	2·826 (6)	OU(1)–U–O(1)	92·0 (2)
OU(1)···O(1)	3·016 (6)	OU(1)–U–O(2)	91·1 (2)
OU(1)···O(2)	2·932 (8)	OU(1)–U–O(5)	87·0 (2)
OU(1)···O(5)	2·949 (7)	OU(1)–U–O(3)	94·6 (2)
OU(1)···O(3)	3·090 (5)	OU(1)–U–O(4)	89·6 (2)
OU(1)···O(4)	2·887 (6)	O(6^{xii})–O(8^{i})–O(7^{i})	56·1 (1)
Sr^{i}–O(6^{xii})	2·608 (4)	O(8^{i})–O(7^{i}) –O(6^{xii})	64·4 (1)
Sr^{i}–O(8^{i})	2·629 (5)	O(7^{i})–O(6^{xii})–O(8^{i})	59·5 (1)
Sr^{i}–O(7^{i})	2·512 (4)	O(6^{i})–O*W*^{i}–O(7^{viii})	48·9 (1)
Sr^{i}–O(7^{viii})	2·645 (4)	O*W*^{i}–O(7^{viii})–O(6^{i})	79·5 (2)
Sr^{i}–O(6^{i})	2·699 (4)	O(7^{viii})–O(6^{i})–O*W*^{i}	51·6 (1)
Sr^{i}–O*W*^{i}	2·509 (5)	O(6^{xii})–Sr^{i}–O(8^{i})	74·4 (1)
Sr^{i}–O(3^{i})	2·726 (4)	O(6^{xii})–Sr^{i}–O(7^{i})	69·4 (1)
Sr^{i}–O(1^{vi})	2·905 (4)	O(7^{i})–Sr^{i}–O(8^{i})	72·1 (1)
Sr^{i}–OU(2^{i})	3·021 (5)	O(6^{i})–Sr^{i}–O(7^{viii})	66·1 (1)
O(6^{i}···O(7^{viii})	2·914 (5)	O(6^{i})–Sr^{i}–O*W*^{i}	93·8 (1)
O(7^{i})···O(6^{xii})	2·914 (5)	O*W*^{i}–Sr–O(7^{viii})	72·1 (1)
O(7^{i})···O(8^{i})	3·025 (6)	O(3^{i})–C(1)–O(5^{ix})	123·2 (6)
O*W*···O(7^{viii})	3·034 (6)	O(4^{i})–C(2^{xii})–O(6^{xii})	126·0 (6)
O(8^{i})···O(6^{xii})	3·165 (6)	O(2^{i})–C(3^{xi})–O(8^{xi})	124·8 (6)
O(7^{i})···O(7^{viii})	3·279 (8)	O(1^{i})–C(4^{x})–O(7^{x})	122·7 (6)
C(1^{i})–O(3^{i})	1·253 (8)		
C(1^{i})–O(5^{ix})	1·213 (9)	Averaged values	
C(2^{xii})–O(4^{i})	1·295 (8)	U–O (equatorial plane)	2·387 (5)
C(2^{xii})–O(6^{xii})	1·249 (8)	Sr–7(O) (monocapped octahedron)	2·618 (5)
C(3^{xi})–O(2^{i})	1·198 (9)		
C(3^{xi})–O(8^{xi})	1·234 (8)	OU···O	2·950 (6)
		C–O	1·244 (8)
C(4^{x})–O(1^{i})	1·260 (8)		
		O–U–O	72·1 (2)
C(4^{x})–O(7^{x})	1·247 (7)		
		OU(1)–U–O	89·1 (2)
		OU(2)–U–O	90·9 (2)
		O–C–O	124·2 (6)

Symmetry code

(i)	x	y	z	(viii)	$\frac{1}{2}+x$	y	$\frac{1}{2}-z$
(ii)	$\frac{1}{2}+x$	$\frac{1}{2}-y$	$-z$	(ix)	$-\frac{1}{2}+x$	$\frac{1}{2}-y$	$-z$
(iii)	$-x$	$\frac{1}{2}+y$	$\frac{1}{2}-z$	(x)	$-x$	$-\frac{1}{2}+y$	$\frac{1}{2}-z$
(iv)	$\frac{1}{2}-x$	$-y$	$\frac{1}{2}+z$	(xi)	$\frac{1}{2}-x$	$-\frac{1}{2}+y$	z
(v)	$-x$	$-y$	$-z$	(xii)	$-\frac{1}{2}+x$	y	$\frac{1}{2}-z$
(vi)	$\frac{1}{2}-x$	$\frac{1}{2}+y$	z	(xiii)	$1+x$	y	z
(vii)	x	$\frac{1}{2}-y$	$\frac{1}{2}+z$				

* For the sake of clarity the superscripts for the equivalent positions have been omitted for the bond angles and distances involving the U atom and its environment.

The Sr atom has an environment of nine nearest oxygen atoms [average Sr–9(O) = 2·695 (5) Å], but if we exclude the two farthest oxygen atoms O(1^{vi}) and OU(2^{i}) at respectively 2·905 (4) and 3·021 (5) Å, the Sr appears to be at the center of a capped octahedron (seven-coordination), the average Sr–7(O) distance being 2·618 (5) Å. The O*W* atom of the stoichiometric water molecule belongs to the coordination polyhedron of the Sr atom and corresponds to the shortest Sr–O distance [2·509 (5) Å]. The Sr polyhedra are interlinked through the O(6) and O(7) oxygen atoms and form infinite zigzag chains which run parallel to the [100] direction.

The bidentate formato groups all bridge heavy atoms: (*a*) two symmetry-related O(3)–C(1)–O(5) formato groups link two neighboring U atoms thus forming twins of U polyhedra; (*b*) these twins are in turn linked to the Sr polyhedra chains through the remaining three formato groups.

Another feature of this complex (Fig. 2) is the presence of planes parallel to ($x0z$) and containing all the U, Sr and O*W* atoms for y = 0·14 (and the symmetry-related y coordinates). On Fig. 2 a partial projection of the structure viewed along the [100] axis reveals the presence of two kinds of channels in the structure. Each of them has its axis running parallel to the [100] direction: (*a*) the CN(1) channels which have a nearly cylindrical shape (mean diameter 3·8 Å) are centered at $y = 0·13$, $z = \frac{1}{4}$; (*b*) the CN(2) channels which have an elliptical section (3·2 × 6 Å) are centered at $y = 0$, $z = \frac{1}{2}$, their major axis being nearly parallel to the {012} form.

Discussion

In $SrUO_2(HCOO)_4.(1+x)H_2O$ the uranyl group is far from the linear $D_{\infty h}$ symmetry and, on inspection of the

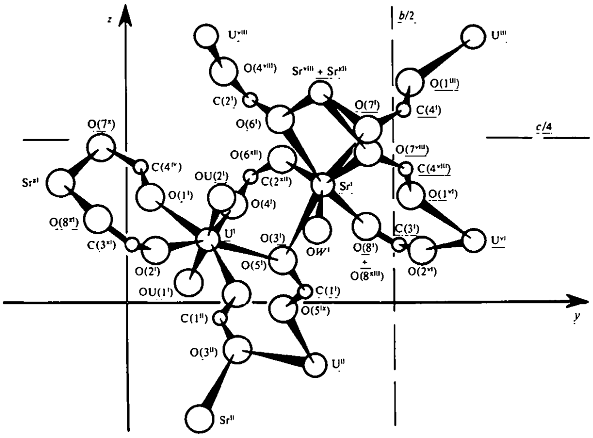

Fig. 1. Projection along the [100] direction of the structure of $SrUO_2(HCOO)_4.(1+x)H_2O$ showing the asymmetric unit and its near environment.

standard deviations, the U—OU(1) and U—OU(2) bond lengths [respectively 1·740 (4) and 1·732 (4) Å] may be considered as identical; accordingly, the molecular symmetry of the uranyl group is C_{2v}, its site symmetry being of course C_1, since U is on a general position. The deformation of the uranyl group might be due to a small interaction of the Sr atom with the OU(2) atom [Sr—OU(2) = 3·021 (5) Å], the result being a bending of the OU(1)—U—OU(2) group towards Sr [O(4)—U—OU(2) = 87·0 (2)°].

The five oxygen atoms O(1) through O(5) around the U atom are very close to the mean equatorial plane (Fig. 3) which has a nearly regular pentagonal shape.

As mentioned previously, the five δ(OCO) vibrations of the formato group result from the four formato groups of the asymmetric unit which have their C—O bond lengths in the 1·198 (9) to 1·295 (8) Å range, the mean C—O distance being 1·244 (8) Å. It is worthwhile noting that in this structure there is no chelating formato group, as has been observed in the case of $(NH_4)_2UO_2(HCOO)_4$ (Mentzen, Puaux & Sautereau, 1978).

On comparing the crystal structures of the present complex and $Sr(OH)_2$ (Grueninger & Bärnighausen, 1969), it appears that the environment of the Sr atom is in both cases a monocapped octahedron, the Sr—O bond lengths being respectively in the 2·509 (5)–2·726 (4) and 2·496–2·767 Å ranges. On the other hand, in $Sr(OH)_2.H_2O$ (Bärnighausen & Weidlein, 1967), the Sr atom is surrounded by eight oxygen atoms, at distances from 2·59 (Sr—OH bond) to 2·74 Å (Sr—H_2O bond). For both strontium hydroxides, the longest Sr—O bond lengths are close to 2·75 Å, and this is the reason why, from the nine oxygen atoms in the vicinity of the Sr atom in $SrUO_2(HCOO)_4.(1 + x)H_2O$, we rejected $O(1^{vi})$ and $OU(2^{i})$, the remaining seven oxygen atoms thus yielding the monocapped octahedron around Sr. The short Sr—O*W* bond distance [2·509 (5) Å] is a consequence of the strong polarization of the stoichiometric water molecule, and explains the presence of the $\nu_{as}(OH)$ IR band at 3600 cm^{-1}. A similar phenomenon has already been reported in the case of $NaUO_2(HCOO)_3.H_2O$ (Mentzen, 1977).

If we consider the environment of the stoichiometric water molecule, the nearest oxygen atom $O(8^{xiii})$ is at 2·803 (7) Å, and it is reasonable to consider that one of the hydrogen atoms, H*W*(1), of this water molecule is located on this OW^{i}—H*W*(1)—$O(8^{xiii})$ hydrogen bond, since the length of this bond is comparable to those in $NaHC_2O_4.H_2O$ (Tellgren, Thomas & Olovsson, 1977), and corresponds to a bond of normal strength (Guillermet, 1974). For the time being no attempts have been made to locate the second hydrogen atom of the O*W* water molecule.

As mentioned above, the chemical analysis of $SrUO_2(HCOO)_4.(1 + x)H_2O$ reveals the presence of excess water (x = 0·38 in the present case). The existence of this non-stoichiometric water is supported by differential thermal analysis results which exhibit two endotherms at 213 and 254 °C corresponding to the two dehydration steps of the solid prior to its decarboxylation. The first DTA peak at 213 °C corresponds to the departure of the stoichiometric water molecule; the second peak at 254 °C, the area of which is nearly three times less than the former peak, is attributed to the departure of the fractional water, the latter being apparently trapped in the structure and evolving only after the stoichiometric water. The CN(1) and CN(2) channels (Fig. 2) could be the only locations for the fractional water. On Fig. 2 we have drawn the approximate positions of the hydrogen atoms H(1)–H(4) of the formato groups. The distribution of the H(2) and H(4) atoms in CN(1) excludes the presence of excess water in this channel. On the other hand, channel CN(2) is relatively free of hydrogen atoms and the non-stoichiometric water might in this case be somewhere in this channel, possibly hydrogen bonded to the stoichiometric O*W* water. It must be noted that it is impossible to locate the xH_2O on the inversion centers, since they are at 1·886 (5) Å from the O(2)

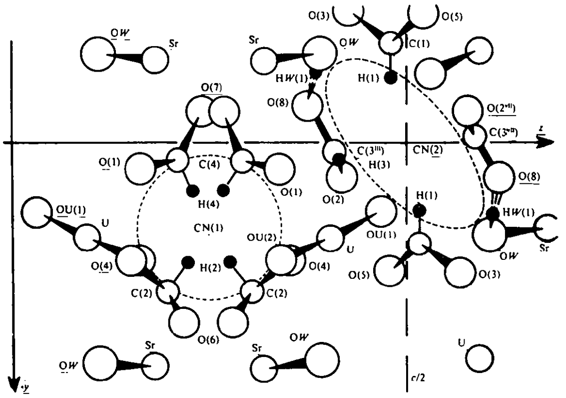

Fig. 2. Projection along the [100] direction showing the CN(1) and CN(2) channels and the U, Sr, O*W* planes.

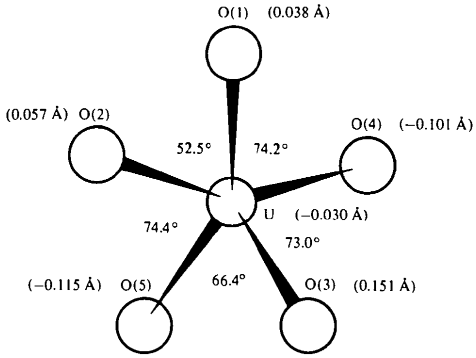

Fig. 3. Environment of the U atom: the mean equatorial plane of the pentagonal bipyramid (numbers in parentheses indicate the distances of the atoms to the mean plane).

atoms, this distance being inconceivable for an O · · · O interaction in this type of structure. In any case, these assumptions have to be supported by additional data and further investigations are planned for the location of the hydrogen atoms of the water molecules by neutron diffraction. Nevertheless, our assumptions are consistent with the interpretation of the DTA, TG and DTG experiments.

All the computations have been performed on an IBM 370-168 computer at Orsay (CIRCE), through the terminal of the Institut de Recherches sur la Catalyse, Lyon, and by means of programs available at the Laboratoire de Chimie Analytique II, Université Claude-Bernard, Lyon I.

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The Crystal Structure of Phenylmercury Cyanide–Phenanthroline

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The title compound, $C_{19}H_{13}N_3Hg$, crystallizes in the monoclinic system, space group $P2_1/a$, with unit-cell dimensions $a = 10{\cdot}152\ (1)$, $b = 18{\cdot}502\ (2)$, $c = 8{\cdot}861\ (1)$ Å, $\beta = 99{\cdot}59\ (1)°$ and $Z = 4$. The structure was solved by the heavy-atom method and successive Fourier syntheses and refined by the full-matrix least-squares method to a final R of 0·071 for 3355 observed reflections. The Hg atom presents fourfold coordination. The three rings of the phenanthroline are in the same plane, forming an angle of 89° with the plane of the phenyl group.

Introduction

It is well known that the Hg atoms in organomercury derivatives show very little tendency to increase their coordination number by interaction with donor molecules. However, the organomercury derivatives of the type Ph—Hg—C≡X (X = N, C—R; Ph = C_6H_5) should have an acceptor capacity intermediate between that of Ph_2Hg and that of the corresponding $(X{\equiv}C)_2Hg$. A strong donor ligand, such as 1,10-phenanthroline (phen), is capable of forming adducts with the organomercury derivative Ph—Hg—C≡N and with zinc, cadmium and mercury dicyanides (Cano Esquivel, Santos & Ballester, 1977).

In the present paper the structure of the complex of Ph—Hg—CN with phenanthroline is reported.

Experimental

Samples of phenylmercury cyanide–phenanthroline were provided by Drs M. Cano and A. Santos of the Instituto de Química Inorgánica 'Elhuyar' del CSIC, Madrid, Spain.

Colourless crystals were obtained by the reaction of phenylmercury cyanide with phenanthroline and by slow evaporation from a solution in methanol.

The single crystal (approximately 0·3 × 0·4 × 0·2