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Structure of a Second Form of 1,2;2,3-Di-μ-hydrido-μ_3-tetracarbonylferrio-*cyclo*-tris (tricarbonylruthenium)(3*Ru*–*Ru*), $H_2FeRu_3(CO)_{13}$

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Abstract. $M_r = 725{\cdot}21$, monoclinic, $C2/c$, $a = 31{\cdot}418$ (6), $b = 9{\cdot}724$ (3), $c = 13{\cdot}816$ (1) Å, $\beta = 110{\cdot}83$ (1)°, $V = 3945{\cdot}0$ Å^3, $Z = 8$, $D_x = 2{\cdot}442$ g cm^{-3}, $F(000) = 2736$, Mo $K\alpha$, $\lambda = 0{\cdot}71073$ Å, $\mu = 29{\cdot}970$ cm^{-1}, room temperature, $R = 0{\cdot}043$ for 3546 reflections. Crystals were grown from a tetrahydrofuran (THF) solution at room temperature to which Et_2O was added. The molecule contains a distorted tetrahedral $FeRu_3$ cluster. The molecules are aligned parallel to the plane (111).

Introduction. From the attempted synthesis of $H_2Fe_3Ru_3(CO)_{18}$ by protonating with HCl an equimolar mixture of $K_2[Ru_3(CO)_{11}]$ and $Fe_3(CO)_{12}$ in THF a product was obtained which upon crystallization by adding Et_2O proved to be a previously unreported crystalline form of the known compound $H_2FeRu_3(CO)_{13}$ (Gilmore & Woodward, 1971). The previous description of this compound, form (I) (space group $P2_1/a$, two independent molecules in the asymmetric unit), differs markedly in shape and color from form (II) (space group $C2/c$, one independent molecule in the asymmetric unit) reported here. The more accurate and more extensive data obtained in the present study enable us to determine with greater accuracy and precision the molecular parameters of $H_2FeRu_3(CO)_{13}$ and to elucidate molecular features which were tentatively suggested in the earlier report. Furthermore, it is of interest to examine and compare the packing of molecules in each crystalline form.

Experimental. Crystal $0{\cdot}12 \times 0{\cdot}30 \times 0{\cdot}35$ mm, dark brown, plate-like. Enraf–Nonius CAD-4 diffractometer, graphite-monochromated Mo $K\alpha$ radiation. Cell dimensions: least-squares fitting from 24 reflections well distributed in reciprocal space, 2θ range 15–30°. Data collected by $2\theta{:}\theta$ scan mode, 2θ range 4–55°. Intensities in hkl and $hk\bar{l}$ octants measured. Total of 4885 reflections collected, 4512 unique; 3546 with $I > 3{\cdot}0\sigma(I)$ used in structure refinement. Six standard intensities: max. variation 0·03. Intensities corrected for Lorentz and polarization effects and also for absorption by using the method of Gaussian quadrature with grid $18 \times 14 \times 4$ points; max. and min. transmission coefficients 0·7352 and 0·3424. Analytical form of scattering factors for neutral atoms used, f' and f'' terms included for all atoms (Cromer & Waber, 1974). All crystallographic computations carried out on a PDP 11/44 computer using the Enraf–Nonius *Structure Determination Package*. Intensity statistics indicated the space group likely to be the centrosymmetric $C2/c$, rather than Cc. Structure solved by *MULTAN*11/82 (Frenz, 1982; Hull & Irwin, 1978; Yao, 1981). Full-matrix least-squares refinement (anisotropic thermal parameters) minimizing $\sum w(|F_o| - |F_c|)^2$; $w = [\sigma(I)^2 + (pI)^2]^{-1/2}$, with $p = 0{\cdot}06$ chosen to make $\sum w\Delta F^2$ uniformly distributed in $|F_o|$. A difference Fourier synthesis gave the coordinates of two bridging H atoms. These were included with isotropic $B = 4{\cdot}5$ Å^2. No H parameters were refined. Final $R = 0{\cdot}043$ and $R_w = 0{\cdot}064$. Final difference Fourier map showed no significant features, with a max. peak height of 0·66 e Å^{-3} in the vicinity of the Ru atom. $(\Delta/\sigma)_{max} = -0{\cdot}15$.

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Table 1. *Positional parameters and equivalent isotropic thermal parameters*

$$B_{eq} = \tfrac{4}{3}\textstyle\sum_i\sum_j\beta_{ij}\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	B_{eq}(Å^2)
Ru(1)	0·39662 (2)	0·43709 (6)	0·89029 (4)	2·76 (1)
Ru(2)	0·36233 (2)	0·21445 (5)	0·74264 (4)	2·460 (9)
Ru(3)	0·32704 (2)	0·27074 (7)	0·90417 (4)	3·45 (1)
Fe	0·41017 (3)	0·1723 (1)	0·94457 (7)	2·41 (2)
O(11)	0·4319 (2)	0·5298 (7)	1·1128 (4)	5·1 (1)
O(12)	0·4663 (2)	0·6000 (7)	0·8324 (5)	6·1 (2)
O(13)	0·3328 (3)	0·6802 (7)	0·8269 (6)	6·7 (2)
O(14)	0·4916 (2)	0·2969 (8)	0·9301 (5)	5·9 (2)
O(21)	0·3366 (3)	−0·0827 (7)	0·6903 (6)	6·7 (2)
O(22)	0·4440 (2)	0·1692 (8)	0·6801 (5)	5·9 (1)
O(23)	0·3009 (3)	0·3183 (8)	0·5307 (5)	6·7 (2)
O(31)	0·3523 (2)	0·3656 (6)	1·1222 (4)	4·6 (1)
O(32)	0·2516 (2)	0·0898 (9)	0·9143 (6)	7·0 (2)
O(33)	0·2580 (2)	0·4911 (9)	0·8204 (7)	7·7 (2)
O(34)	0·3492 (2)	−0·0412 (6)	0·9642 (6)	6·1 (2)
O(41)	0·4518 (2)	0·1633 (8)	1·1713 (5)	6·0 (2)
O(42)	0·4530 (3)	−0·0703 (7)	0·8988 (6)	7·4 (2)
C(11)	0·4185 (2)	0·4945 (8)	1·0301 (5)	3·6 (1)
C(12)	0·4402 (3)	0·5395 (9)	0·8535 (6)	4·2 (2)
C(13)	0·3554 (3)	0·5858 (8)	0·8495 (6)	4·3 (2)
C(14)	0·4558 (2)	0·2773 (8)	0·9295 (6)	3·4 (1)
C(21)	0·3464 (3)	0·0272 (8)	0·7145 (5)	3·6 (1)
C(22)	0·4148 (2)	0·1876 (8)	0·7031 (5)	3·5 (1)
C(23)	0·3234 (3)	0·2790 (8)	0·6097 (5)	3·7 (2)
C(31)	0·3430 (2)	0·3287 (8)	1·0399 (5)	3·1 (1)
C(32)	0·2790 (3)	0·157 (1)	0·9046 (6)	4·4 (2)
C(33)	0·2852 (3)	0·408 (1)	0·8498 (6)	4·7 (2)
C(34)	0·3638 (2)	0·0630 (7)	0·9481 (6)	3·3 (1)
C(41)	0·4362 (3)	0·1685 (8)	1·0831 (6)	3·7 (2)
C(42)	0·4358 (3)	0·0220 (9)	0·9165 (6)	4·2 (2)
H(1)	0·379	0·39	0·770	
H(2)	0·314	0·22	0·770	

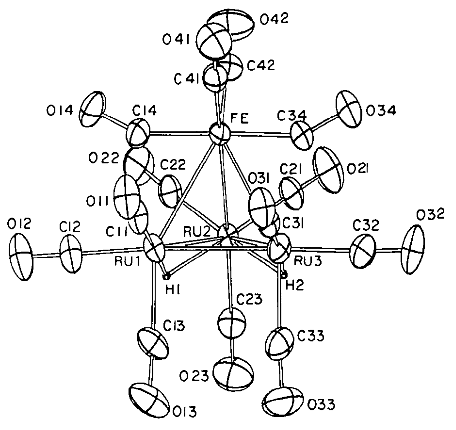

Fig. 1. Drawing of the molecular structure of $H_2FeRu_3(CO)_{13}$ with 50% probability ellipsoids.

Discussion. Final coordinates and equivalent isotropic thermal parameters are given in Table 1.* A projection of the molecular structure of $H_2FeRu_3(CO)_{13}$ with thermal ellipsoids and the atomic numbering scheme is shown in Fig. 1. Selected interatomic distances and bond angles are listed in Table 2.

Table 2. *Bond distances* (Å) *and angles* (°) *for* $H_2FeRu_3(CO)_{13}$, *with e.s.d.'s in parentheses*

Ru(1)–Ru(2)	2·906 (1)	Ru(1)–Fe	2·673 (1)
Ru(1)–Ru(3)	2·780 (1)	Ru(2)–Fe	2·689 (1)
Ru(2)–Ru(3)	2·876 (1)	Ru(3)–Fe	2·648 (1)
Ru(1)–C(11)	1·890 (5)	Ru(2)–C(23)	1·915 (5)
Ru(1)–C(12)	1·903 (5)	Ru(3)–C(31)	1·848 (5)
Ru(1)–C(13)	1·889 (6)	Ru(3)–C(32)	1·870 (6)
Ru(1)–C(14)	2·334 (5)	Ru(3)–C(33)	1·838 (6)
Ru(2)–C(21)	1·892 (5)	Ru(3)–C(34)	2·299 (5)
Ru(2)–C(22)	1·930 (5)	Fe–C(14)	1·831 (5)
Fe–C(41)	1·795 (5)	Fe–C(34)	1·818 (5)
Fe–C(42)	1·777 (5)	C(32)–O(32)	1·128 (7)
C(11)–O(1)	1·122 (6)	C(33)–O(33)	1·139 (7)
C(12)–O(12)	1·128 (6)	C(41)–O(41)	1·141 (7)
C(13)–O(13)	1·133 (7)	C(42)–O(42)	1·118 (7)
C(21)–O(21)	1·129 (6)	C(14)–O(14)	1·137 (6)
C(22)–O(22)	1·088 (6)	C(34)–O(34)	1·166 (6)
C(23)–O(23)	1·135 (6)	Ru(2)–H(2)	1·68
C(31)–O(31)	1·128 (6)	Ru(3)–H(2)	1·82
Ru(1)–H(1)	1·62	Ru(2)–H(1)	1·79

Ru(2)–Ru(1)–Ru(3)	60·71 (1)	Ru(1)–Ru(3)–Ru(2)	61·8 (1)
Ru(2)–Ru(1)–Fe	57·43 (2)	Ru(1)–Ru(3)–Fe	58·94 (2)
Ru(2)–Fe–Ru(3)	65·20 (2)	Ru(1)–Fe–Ru(2)	65·64 (2)
Ru(1)–Ru(2)–Ru(3)	57·48 (1)	Ru(1)–Fe–Ru(3)	63·00 (2)
Ru(1)–Ru(2)–Fe	56·92 (2)	Ru(2)–Ru(3)–C(32)	115·1 (2)
Ru(2)–Ru(1)–C(12)	108·9 (2)	Ru(2)–Ru(3)–C(33)	103·8 (2)
Ru(2)–Ru(1)–C(13)	108·5 (2)	Fe–Ru(3)–C(31)	90·3 (2)
Ru(3)–Ru(1)–C(11)	96·3 (1)	Fe–Ru(3)–C(32)	121·5 (2)
Ru(3)–Ru(1)–C(13)	89·6 (2)	Ru(1)–Fe–C(41)	106·6 (2)
Fe–Ru(1)–C(11)	91·9 (2)	Ru(1)–Fe–C(42)	140·4 (2)
Fe–Ru(1)–C(12)	121·4 (2)	Ru(1)–Fe–C(14)	58·9 (2)
Ru(1)–Ru(2)–C(22)	99·1 (2)	Ru(2)–Fe–C(42)	91·7 (1)
Ru(1)–Ru(2)–C(23)	112·3 (2)	Ru(2)–Fe–C(14)	88·2 (2)
Ru(3)–Ru(2)–C(21)	101·3 (2)	Ru(2)–Fe–C(34)	87·7 (2)
Ru(3)–Ru(2)–C(23)	113·0 (2)	Ru(3)–Fe–C(41)	105·9 (2)
Fe–Ru(2)–C(21)	94·4 (2)	Ru(3)–Fe–C(42)	137·8 (2)
Fe–Ru(2)–C(22)	93·1 (2)	Ru(3)–Fe–C(34)	58·5 (2)
Ru(1)–Ru(3)–C(31)	87·5 (1)	C(32)–Ru(3)–C(33)	88·3 (3)
Ru(1)–Ru(3)–C(33)	90·7 (2)	C(31)–Ru(3)–C(34)	94·0 (2)
C(11)–Ru(1)–C(12)	94·9 (2)	C(32)–Ru(3)–C(34)	79·1 (2)
C(11)–Ru(1)–C(13)	92·3 (2)	C(41)–Fe–C(42)	98·4 (3)
C(12)–Ru(1)–C(13)	90·1 (3)	C(41)–Fe–C(14)	93·1 (2)
C(11)–Ru(1)–C(14)	89·4 (2)	C(41)–Fe–C(34)	91·4 (2)
C(12)–Ru(1)–C(14)	79·7 (2)	C(42)–Fe–C(14)	90·0 (3)
C(21)–Ru(2)–C(22)	90·1 (2)	C(42)–Fe–C(34)	87·4 (2)
C(21)–Ru(2)–C(23)	94·9 (2)	C(14)–Fe–C(34)	175·0 (2)
C(22)–Ru(2)–C(23)	95·3 (2)	C(31)–Ru(3)–C(33)	95·1 (2)
C(31)–Ru(3)–C(32)	96·0 (2)	Ru(3)–C(34)–Fe	79·1 (2)
Ru(1)–C(14)–Fe	78·8 (2)	Ru(3)–C(32)–O(32)	173·8 (6)
Ru(1)–C(11)–O(11)	178·9 (4)	Ru(3)–C(33)–O(33)	175·5 (6)
Ru(1)–C(12)–O(12)	179·5 (5)	Ru(3)–C(34)–O(34)	128·2 (4)
Ru(1)–C(13)–O(13)	175·9 (6)	Fe–C(41)–O(41)	177·9 (5)
Ru(1)–C(14)–O(14)	126·4 (4)	Fe–C(42)–O(42)	178·0 (6)
Ru(2)–C(21)–O(21)	174·9 (5)	Fe–C(14)–O(14)	154·7 (5)
Ru(2)–C(22)–O(22)	178·3 (5)	Fe–C(34)–O(34)	152·5 (4)
Ru(2)–C(23)–O(23)	178·9 (5)	Ru(1)–Ru(2)–H(2)	86·9
Ru(3)–C(31)–O(31)	178·9 (5)	Fe–Ru(2)–H(2)	89·5
Ru(3)–Ru(1)–H(1)	85·3	Ru(1)–Ru(3)–H(2)	88·2
Fe–Ru(1)–H(1)	89·0	Fe–Ru(3)–H(2)	87·8
Ru(3)–Ru(2)–H(1)	79·7	C(21)–Ru(2)–H(2)	84·2
Fe–Ru(2)–H(1)	85·2	C(22)–Ru(2)–H(2)	173·9
C(11)–Ru(1)–H(1)	178·4	C(23)–Ru(2)–H(2)	83·0
C(12)–Ru(1)–H(1)	83·5	C(31)–Ru(3)–H(2)	175·7
C(13)–Ru(1)–H(1)	87·6	C(32)–Ru(3)–H(2)	88·2
C(14)–Ru(1)–H(1)	90·4	C(33)–Ru(3)–H(2)	84·6
C(21)–Ru(2)–H(1)	178·5	C(34)–Ru(3)–H(2)	87·2
C(22)–Ru(2)–H(1)	88·5	Ru(2)–H(2)–Ru(3)	110·7
C(23)–Ru(2)–H(1)	85·7	H(1)–Ru(2)–H(2)	97·3
Ru(1)–H(1)–Ru(2)	116·6		

The $H_2FeRu_3(CO)_{13}$ molecule contains a distorted tetrahedral $FeRu_3$ cluster which has a pseudomirror plane that passes through atoms Fe and Ru(2) and bisects the Ru(1)–Ru(3) bond. Two of the carbonyl groups bound to Fe function as semi-bridging groups to Ru atoms. The same gross configuration of $H_2FeRu_3(CO)_{13}$ was observed in the study of crystalline form (I). Differences between molecular parameters given for forms (I) and (II) probably arise from the less

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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 39083 (27 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

accurate data available from the earlier structure determination rather than from intermolecular interactions.

From a difference Fourier map the two H atoms in $H_2FeRu_3(CO)_{13}$ were located in bridging positions Ru(1)—H(1)—Ru(2) and Ru(2)—H(2)—Ru(3) in accord with the inferred H positions from the earlier study of Gilmore & Woodward. Distances between the H-bridged atoms [Ru(1)—Ru(2) = 2·906 (1) and Ru(2)—Ru(3) = 2·876 (1) Å] are longer than the distance between non-H-bridged Ru atoms [Ru(1)—Ru(3) = 2·780 (1) Å]. These observations are consistent with determinations of distances between H-bridged and non-H-bridged Ru atoms in $[(Ph_3P)_2N][HRu_3Fe(CO)_{13}]$ (Takusagawa *et al.*, 1981) and $H_2Ru_4(CO)_{13}$ (Yawney & Doedens, 1972). Fe—Ru bonds which are semi-bridged, Fe—Ru(1) = 2·673 (1) and Fe—Ru(3) = 2·648 (1) Å, are shorter than the unbridged bond Fe—Ru(2) = 2·689 (1) Å. This apparent contraction, possibly due to the semibridging system, is also observed in $H_2FeOs_3(CO)_{13}$ (Churchill, Bueno, Hsu, Plotkin & Shore, 1982).

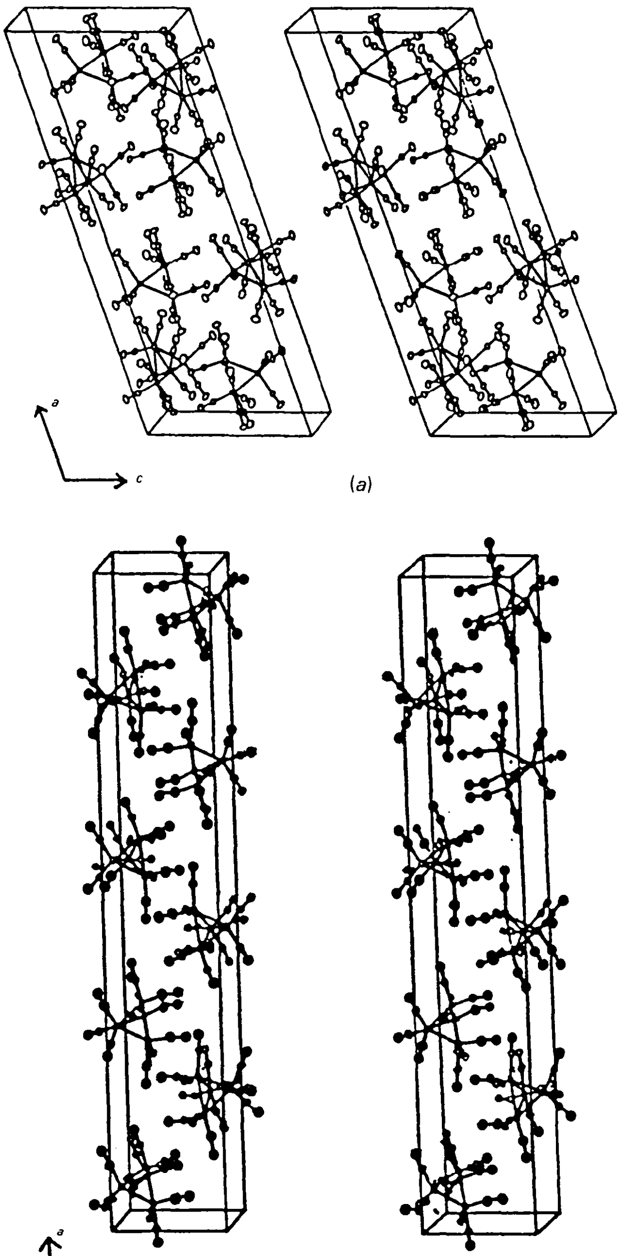

Fig. 2. Stereoviews of the packing of (*a*) $H_2FeRu_3(CO)_{13}$ molecules in crystalline form (I) and (*b*) $H_2FeRu_3(CO)_{13}$ molecules in crystalline form (II).

Table 3. *Intermolecular contacts* <3·0 Å *of crystalline form* (I)

$O(11)\cdots O(33^{i})$	2·91	$O(34)\cdots O(33^{iii})$	2·93
$O(41)\cdots O(41^{ii})$	2·98	$O(14')\cdots O(11^{ii})$	2·97
$O(13)\cdots O(21^{iv})$	2·90	$O(13)\cdots O(23^{v})$	2·86
$O(23)\cdots O(32^{vi})$	2·97	$O(41)\cdots O(21^{ii})$	2·99
$O(32)\cdots O(42^{viii})$	2·90	$O(12')\cdots O(41'^{ix})$	2·93

Roman numerals as superscripts refer to the following positions relative to the reference molecule at x, y, z: (i) $\frac{1}{2}-x, \frac{1}{2}+y, -z$; (ii) $\frac{1}{2}-x, y-\frac{1}{2}, -z$; (iii) $\frac{1}{2}-x, \frac{1}{2}+y, 1-z$; (iv) $x, y, z-1$; (v) $-x, 1-y, -z$; (vi) $-x, 1-y, 1-z$; (vii) $x, y-1, z$; (viii) $x, y-1, z$; (ix) $\frac{1}{2}-x, y-\frac{1}{2}, 1-z$.

Fig. 2 shows stereoviews of the molecular packing in crystalline forms (I) ($P2_1/a$) and (II) ($C2/c$). The packing arrangements are quite different. Form (I) is a red, needle-like crystal with elongation in the direction of the *b* axis (Gilmore & Woodward, 1971), while form (II) is a dark-brown plate-like crystal with (001) faces developed, and elongated in the direction [111]. Molecules in form (I) are stacked in a staggered way. There are 10 intermolecular contact distances <3·0 Å (Table 3), six more than listed in the original report. The shortest intermolecular contact distance is O(13)···O(23) = 2·86 Å between crystallographically dependent molecules related by symmetry $-x, 1-y, -z$. No intermolecular atom—atom distance is less than twice the van der Waals radius of O (2·8 Å). Therefore, it is presumed that crystal-packing forces do not affect the molecular geometries.

In crystalline form (II) the molecules are lined up parallel to the plane (111). No intermolecular contact distance is less than 3·0 Å. The shortest distance O(13)···O(21) = 3·008 Å is between molecules related by symmetry $x, 1+y, z$. No similarity between the two crystalline forms could be found. Within the intermolecular contact distances less than 3·2 Å there is only one corresponding atom—atom contact, *i.e.* crystalline form (I): O(11)···O(33′) = 2·900 Å between molecules (1) and (2) related by symmetry $-x, \frac{1}{2}+y, z$; form (II): O(11)···O(33) = 3·131 Å between molecules related by symmetry $x, 1-y, \frac{1}{2}+z$.

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Structures of Mercury Dichloride–Dipotassium Dichromate (1/1), $HgCl_2.K_2Cr_2O_7$, and Mercury Dichloride–Diammonium Dichromate (1/1), $HgCl_2.(NH_4)_2Cr_2O_7$

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Abstract. $HgCl_2.K_2Cr_2O_7$: $M_r = 565{\cdot}69$, $P2_1/n$, $a = 12{\cdot}372\,(8)$, $b = 10{\cdot}438\,(8)$, $c = 8{\cdot}191\,(5)$ Å, $\beta = 92{\cdot}32\,(5)°$, $V = 1056{\cdot}9$ Å^3, $Z = 4$, $D_x = 3{\cdot}555$ Mg m^{-3}, Ag $K\alpha$, $\lambda = 0{\cdot}5608$ Å, $\mu = 98{\cdot}76$ cm^{-1}, $F(000) = 1024$, room temperature, $R = 0{\cdot}042$ for 1470 independent reflexions. $HgCl_2.(NH_4)_2Cr_2O_7$: $M_r = 523{\cdot}56$, $P2_1/a$, $a = 14{\cdot}891\,(5)$, $b = 7{\cdot}604\,(5)$, $c = 9{\cdot}620\,(6)$ Å, $\beta = 96{\cdot}02\,(5)°$, $V = 1083{\cdot}2$ Å^3, $Z = 4$, $D_x = 3{\cdot}210$ Mg m^{-3}, Ag $K\alpha$, $\lambda = 0{\cdot}5608$ Å, $\mu = 92{\cdot}64$ cm^{-1}, $F(000) = 960$, room temperature, $R = 0{\cdot}032$ for 1369 independent reflexions. The structures comprise Cr_2O_7 groups which show no unusual features. Linkages of the mercury polyhedra are quite different in the two structures. In the ammonium salt, finite centrosymmetrical $Hg_2Cl_4O_8$ groups are present, located on the (110) planes, while infinite $HgCl_2O_2$ chains, running along **c**, exist in the potassium salt.

Introduction. These two adduct compounds were described as early as 1859 (Groth) but up to now never investigated from a structural point of view. This work is part of the investigation of condensed chromic anions: $(Cr_nO_{3n+1})^2$ (Durif & Averbuch-Pouchot, 1979; Blum & Tran Qui, 1979; Blum, Averbuch-Pouchot & Guitel, 1979; Blum, 1979).

Crystals of these two compounds are readily prepared by slow evaporation at room temperature of solutions containing mercury chloride and alkali dichromates in an equimolar ratio.

Experimental. $HgCl_2.K_2Cr_2O_7$: Cube-shaped crystal $0{\cdot}15 \times 0{\cdot}17 \times 0{\cdot}17$ mm, Philips PW 1100 diffractometer, graphite-monochromated Ag $K\bar{\alpha}$ radiation, ω scan, scan width 1·2°, scan speed 0·02° s^{-1}, total background measurement 20 s, 25 reflexions used for measuring lattice parameters, no absorption correction, $2\theta_{\max} = 48°$; $h_{\max} = 17$, $k_{\max} = 15$, $l_{\max} = 11$; intensity-reference reflexions ($\bar{9}32$ and $\bar{9}\bar{3}2$) showed no appreciable variation; 1895 independent reflexions, 1470 with $F_o > 2\,\sigma_F$; structure solved using classical methods: study of a three-dimensional Patterson function, followed by successive Fourier syntheses; $\sum w\Delta F^2$ minimized; atomic coordinates, anisotropic thermal parameters and a scale factor refined; final $R = 0{\cdot}042$, $R_w = 0{\cdot}047$, $S = 4{\cdot}248$; unit weights; max. Δ/σ 0·0 except for the scale factor (0·03) for the final refinement cycle; max./min. heights in final difference Fourier map $\pm 4{\cdot}8$ e Å^{-3}; correction for extinction $0{\cdot}19 \times 10^{-7}$; atomic scattering factors and f' and f'' values from *International Tables for X-ray Crystallography* (1974); *Enraf–Nonius Structure Determination Package* (Frenz, 1980).

$HgCl_2.(NH_4)_2Cr_2O_7$: Approximately cube-shaped crystal $0{\cdot}18 \times 0{\cdot}17 \times 0{\cdot}15$, Philips PW 1100 diffractometer, graphite-monochromated Ag $K\bar{\alpha}$ radiation, ω scan, scan width 1·30°, scan speed 0·02° s^{-1}, 18 reflexions used for measuring lattice parameters, no absorption correction, $2\theta_{\max} = 50°$, $h_{\max} = 22$, $k_{\max} = 11$, $l_{\max} = 14$; intensity-reference reflexions ($2\bar{4}2$ and $\bar{2}4\bar{2}$) showed no appreciable variation; 2484 independent reflexions, 1369 with $F_o > 3\,\sigma_F$; structure solved using classical methods: study of a three-dimensional

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