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The Crystal Structure of Wolfram's Red Salt

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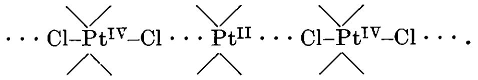
Wolffram's red salt is shown to be correctly formulated as dichlorotetraethylamine platinum (IV) tetraethylamine platinum (II) tetrachloride tetrahydrate, $[Pt(C_2H_5NH_2)_4Cl_2]^{2+}[Pt(C_2H_5NH_2)_4]^{2+}Cl_4.4\,H_2O$. The crystal structure is tetragonal, or pseudo-tetragonal, containing chains parallel to *c*,



$$\cdots \text{Cl–Pt}^{IV}\text{–Cl} \cdots \text{Pt}^{II} \cdots \text{Cl–Pt}^{IV}\text{–Cl} \cdots .$$

There is no long-range order in the structure because of occasional slips in the packing of the chains: a chain may attain an alternative position of low potential energy by a translation along its length of half the chain-repeating distance. The chloride ions lie in the cylindrical holes between the chains. The positions of the water molecules remain undetermined.

Introduction

Wolffram's red salt has been formulated by Drew & Tress (1935) as

$$[Pt^{III}(C_2H_5NH_2)_4Cl]Cl_2.2\,H_2O$$

and alternatively by Reihlen & Flohr (1934) as

$$[Pt^{IV}(C_2H_5NH_2)_4Cl_2][Pt^{II}(C_2H_5NH_2)_4]Cl_4.4\,H_2O.$$

Various optical, magnetic and crystallographic investigations of such apparently trivalent complexes of platinum and palladium have supported a mixed valence formula (Brosset, 1948; Cohen & Davidson, 1951; Watt & McCarley, 1957) and specifically the polarized absorption spectrum of Wolffram's red salt (Yamada & Tsuchida, 1956) suggests the above formula of Reihlen & Flohr. We have shown that the crystal structure is consistent with this, being similar to the disordered structure of

$$Pt(NH_3)_2Br_2.Pt(NH_3)_2Br_4$$

(Hall & Williams, 1958).

Experimental

Wolffram's red salt was prepared by the method of Reihlen & Flohr and recrystallized from very dilute hydrochloric acid. Either needles of square cross section or tetragonal pyramids (or bipyramids asymmetric with respect to the basal plane) were obtained according to the speed of crystallization.

Under the polarizing microscope, the crystals appeared to be single, but X-ray photographs showed them invariably to be multiply twinned with the crystallites slightly disoriented, but retaining a common [001] direction. All attempts to grow single crystals by varying the rate of crystallization were unsuccessful. Single crystals were obtained only as very small irregularly shaped fragments produced by crushing larger crystals.

The unit-cell parameters were measured from rotation photographs and the intensity data for the (001) and (100) projections were recorded on Weissenberg photographs, using the multifilm technique. The intensities were estimated visually.

All X-ray data were recorded using copper $K\alpha$ radiation for which the irregularly shaped crystals had a high absorption factor. Since absorption corrections were impracticable, the accuracy of the intensity data was severely limited. The intensities of all symmetrically related reflections were estimated and the average taken.

The crystal data

Wolffram's red salt. M.W. 1036·2.

$$[Pt^{IV}(C_2H_5NH_2)_4Cl_2][Pt^{II}(C_2H_5NH_2)_4]Cl_4.4\,H_2O\ .$$

Tetragonal:

The subcell data:

$$a = 13{\cdot}28 \pm 0{\cdot}05\ \text{Å},\ c = 5{\cdot}39 \pm 0{\cdot}05\ \text{Å},\ V = 951\ \text{Å}^3.$$
$$Z = 1\ \text{formula unit/cell},$$
$$d_c = 1{\cdot}81\ \text{g.cm.}^{-3},\ d_o = 1{\cdot}80\ \text{g.cm.}^{-3}.$$

Diffraction symmetry: $4/mmm$.
Absent spectra: hkl when $h+k+l=2n+1$.
Space group (from structure analysis): $I4mm$.

It was expected from the previous investigations of $Pt(NH_3)Br_2.Pt(NH_3)Br_4$ that there would be some evidence of long-range disorder in the crystal structure of Wolffram's red salt and this was found to be the

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case. On rotation and oscillation photographs taken with crystals mounted about *c*, very faint streaks were observed interleaving the usual layer lines, thus doubling the repeating distance along *c*. Long exposure equi-inclination Weissenberg photographs of these layers failed to reveal any weak diffuse reflections. Thorough examination did not reveal any other structural disorder effects in the diffraction pattern.

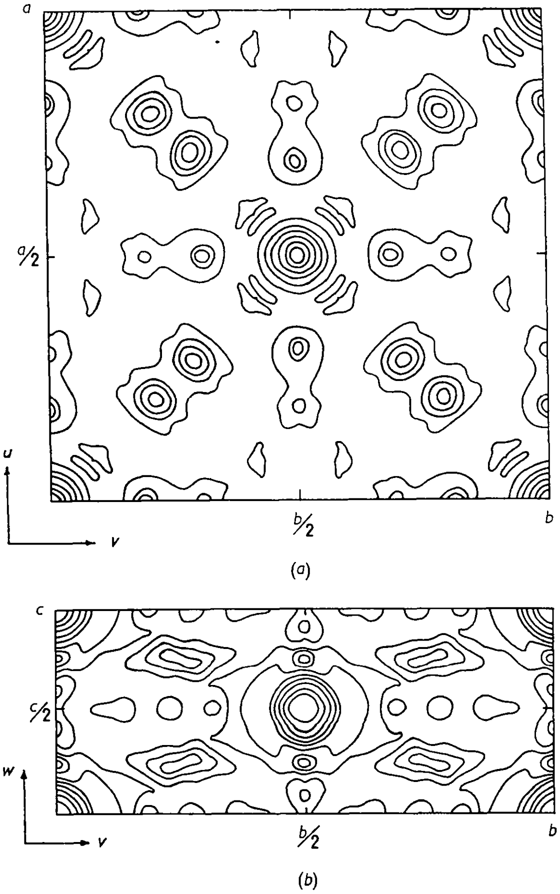
The determination of the subcell structure

The streaking was at first ignored and the structure of the subcell, taken as an ordered 'average' cell, was considered.

If Wolffram's red salt exists as the dimeric form, as postulated by Reihlen & Flohr (1934), then there is only one formula unit in the subcell, i.e. only one $\mathrm{Pt^{IV}}$ and one $\mathrm{Pt^{II}}$. However, the subcell, being body centred, can accommodate only two equivalent platinum atoms, one associated with each lattice point. In considering the subcell structure, the monomeric form

$$[\mathrm{Pt(C_2H_5NH_2)_4Cl]Cl^2.2\,H_2O}^*$$

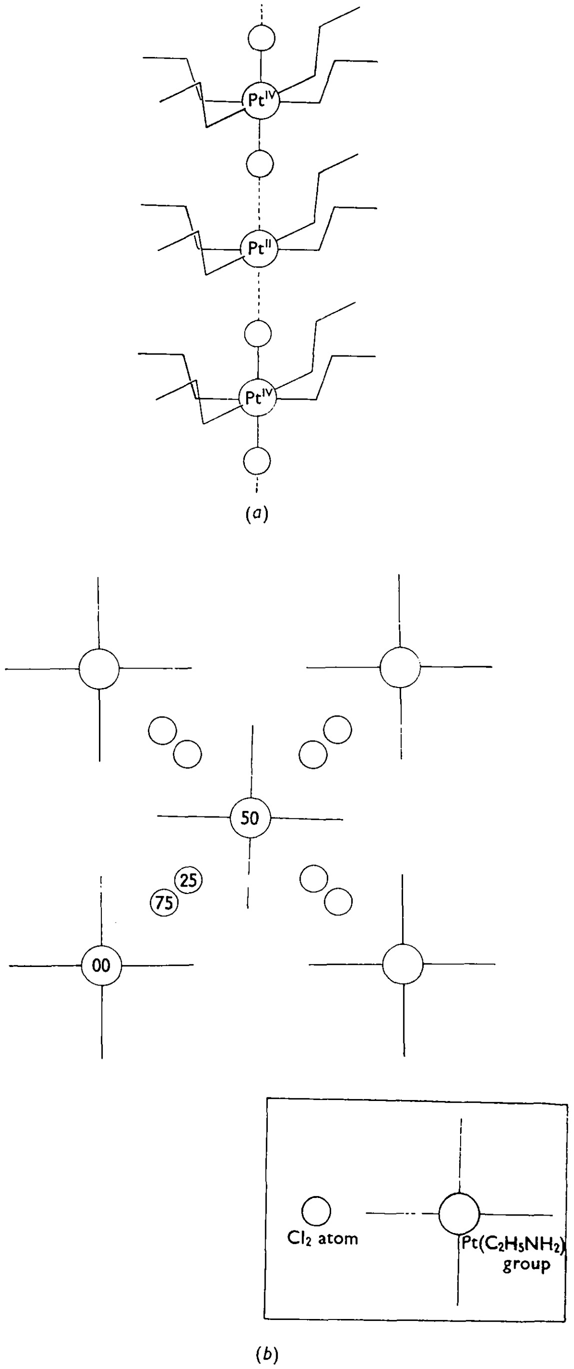
must be assumed with all Pt equivalent and two formula units in the subcell.



(a)

(b)

Fig. 1. Patterson projections on (a) (001) and (b) (100).



(a)

(b)

Fig. 2. (a) A chain of alternating $\mathrm{Pt^{IV}}$ and $\mathrm{Pt^{II}}$ atoms. (b) The structure projected on to (001), showing the packing of the chains and the chloride ions. The *z* coordinates of the platinum atoms and chloride ions are shown. Both of the alternative chloride sites are included, but only one may be occupied in any quadrant.

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* Reihlen & Flohr showed that one of the three chlorine atoms is chemically different from the other two, and we assume that this chlorine atom will also differ crystallographically from the other two.

There are five possible space groups consistent with the observed diffraction symmetry and systematic spectral absences. These are,

$$I\bar{4}2m,\ I4mm,\ I42,\ I4/mmm,\ I\bar{4}m2$$

with $I4mm$ the most probable on crystal morphological grounds.

The Patterson projections on (001) and (100) were calculated, (Fig. 1(*a*) and (*b*)) in the hope that they would enable the space group to be determined uniquely. The Patterson projections should be readily interpretable since the interatomic vectors involving the Pt atoms greatly outweigh all other vectors.

The Pt and the unique chlorine atom (Cl_1) must lie in twofold positions at ($00z$) and ($\frac{1}{2}, \frac{1}{2}, \frac{1}{2}+z$). The Pt atom may be assumed to be at (000) in order to determine the origin. Cl_1 must lie close to $z=\frac{1}{2}$ since c is only 5·39 Å. Chains of alternating Pt and Cl atoms thus run parallel to c at $x=0, y=0$ and $x=\frac{1}{2}, y=\frac{1}{2}$.

The (001) Patterson projection shows peaks at $(u, 0)$, $(0, u)$ etc., which can be attributed to Pt–N and Pt–C vectors, but only one other set of peaks at (u, u), $(u, \bar{u})$ etc., with which to account for vectors to the other chlorine atom (Cl_2) and the water molecule.

In the (100) Patterson projection, the large unresolved peak at $(v, \frac{1}{4})$ is attributed to Pt–C and Pt–Cl_2 vectors. It is evident from the partly resolved peaks surrounding $v=0, w=\frac{1}{2}$, that Cl_1 does not lie exactly at $z=\frac{1}{2}$, but is slightly closer to one flanking Pt than the other. There are two possible chain configurations to consider,

$$\cdots\mathrm{Pt}\overset{\mathrm{III}}{—}\mathrm{Cl}\cdots\mathrm{Pt}\overset{\mathrm{III}}{-\,-}\mathrm{Cl}\cdots\mathrm{Pt}\overset{\mathrm{III}}{—}\mathrm{Cl} \qquad (A)$$

$$\mathrm{Cl}—\mathrm{Pt}\overset{\mathrm{IV}}{—}\mathrm{Cl}\cdots\mathrm{Pt}^{\mathrm{II}}\cdots\mathrm{Cl}—\mathrm{Pt}\overset{\mathrm{IV}}{—}\mathrm{Cl}\,. \qquad (B)$$

$$|\longleftarrow 2c \longrightarrow|$$

Case (*B*) with non-equivalent Pt atoms is possible if the streaking which doubles the repeat distance c is taken into account. In the subcell, case (*B*) is represented by placing a half chlorine atom at both z and $-z$, thus retaining all Pt atoms as equivalent.

Table 1. *Atomic parameters*

Atom	x	y	z	B_{xy}	B_{yz}
Pt	0	0	0	0·8 Å²	1·0 Å²
Cl_1	0	0	0·418 0·582	6·0	5·0
Cl_2	0·213	0·213	0·745	6·0	5·0
N	0·148	0	0	6·0	4·0
C_1	0·190	0	0·240	6·0	6·0
C_2	0·308	0	0·240	6·0	6·0

Table 2. *Structure factors*

$hk0$	F_o	F_c	$hk0$	F_o	F_c	$hk0$	F_o	F_c	$hk0$	F_o	F_c
2 0 0	103	152	2 2 0	130	148	8	96	92	7 7 0	123	122
4	176	222	4	80	92	10	107	95	9	98	79
6	168	184	6	126	137	12	90	71	11	83	80
8	119	118	8	106	101	14	64	69	13	63	68
10	122	120	10	84	82	5 5 0	147	145	8 8 0	103	81
12	98	79	12	99	84	7	96	98	10	78	73
14	95	79	14	62	63	9	117	102	12	71	67
16	46	60	3 3 0	143	140	11	89	76	14	49	57
1 1 0	148	220	5	106	103	13	74	72	9 9 0	94	80
3	123	152	7	142	135	15	50	62	11	61	65
5	136	162	9	96	85	6 6 0	127	134	13	48	62
7	100	134	11	98	85	8	110	100	10 10 0	62	73
9	126	109	13	77	73	10	103	89	12	45	60
11	97	88	15	47	62	12	87	76	11 11 0	50	62
13	89	78	4 4 0	122	132	14	59	65	12 12 0	30	58
15	55	65	6	128	130						

$0kl$	F_o	F_c	A_0	B_0	$0kl$	F_o	F_c	A_0	B_0	$0kl$	F_o	F_c	A_0	B_0
020	89	151	89	0	002	89	80	89	10	2	69	74	69	1
4	153	225	153	0	2	171	174	171	−1	4	97	96	97	−3
6	147	181	147	0	4	93	86	93	4	6	90	86	89	−2
8	106	123	106	0	6	125	114	125	2	8	76	70	76	0
10	107	118	107	0	8	100	109	100	0	10	77	70	77	−2
12	86	78	86	0	10	64	66	64	2	12	52	54	52	0
14	83	80	83	0	12	54	80	54	0					
16	40	56	40	0	14	63	58	63	1	015	101	83	101	2
										3	98	77	98	10
011	115	160	113	18	013	129	120	129	−8	5	93	73	93	−4
3	142	132	125	66	3	106	103	103	−27	7	78	73	78	9
5	130	119	130	−13	5	102	102	102	9	9	61	60	61	−3
7	106	124	100	35	7	98	97	94	−23					
9	83	88	83	−9	093	79	79	79	6	006	46	51	46	3
11	78	77	77	14	11	63	67	63	−8	2	65	62	65	−1
13	77	69	77	1	13	59	63	59	0	4	46	50	46	1
15	57	58	57	1						6	46	54	46	1
					004	143	128	143	−6					

The following atomic coordinates were deduced directly from the Patterson projections:

	x	y	z
Pt	0	0	0
Cl_1	0	0	±0·40
Cl_2	0·21	0·21	±0·25
N	0·15	0	0
C_1	0·19	0	±0·25
C_2	0·30	0	±0·25

It is possible from packing considerations to reduce these to a single set of coordinates (except for Cl_1) and thus determine the space group for the subcell.

(i) Let there be a Cl_2 at (0·21, 0·21, 0·75) as in Fig. 2. There cannot be another Cl_2 at (0·21, 0·21, 0·25), (0·29, 0·29, 0·25) or (0·29, 0·29, 0·75) since these would be distant only 2·7 Å, 3·1 Å and 1·1 Å respectively, and these distances are too short for non-bonding interactions.

(ii) If there is a Cl_2 at (0·21, 0·21, 0·75) then there must be C_1 and C_2 at (0·18, 0, 0·25), (0, 0·18, 0·25) etc., and (0·30, 0, 0·25), (0, 0·30, 0·25) etc., because if any of the carbon atoms of the ethylamine groups associated with the Pt atom at (000) are at $z=0·75$, this will give rise to Cl_2–C_1 and Cl_2–C_2 distances of only 2·9 Å and 3·1 Å respectively and these are too short for non-bonding interactions. A similar result is obtained regardless of the initially postulated Cl_2 position. The point symmetry at all Pt atoms must therefore be $4mm$ and the space group is thus $I4mm$.

In the space group $I4mm$, the Cl_2 position (0·21, 0·21, 0·75) is one of eightfold multiplicity. These eight sites must accommodate the four Cl_2 in some sort of disorder.

The positions of the four water molecules remain undetermined. The only position of fourfold multiplicity in this space group is at $(0, \frac{1}{2}, z)$ etc. There are holes in the deduced structure centred at $(0, \frac{1}{2}, \frac{1}{2})$ etc., but the radius of these is only 3·1 Å, with atoms C_2 as nearest neighbours, and hence they are rather small to accommodate the water molecules. The water molecule must, like the chloride ions, be distributed with disorder in positions of at least eightfold multiplicity, and since the oxygen scattering power for X-rays is thereby dispersed the water molecules should be very difficult to find.

Table 3. *Interatomic distances and angles*

Pt–Cl_1	2·26 Å	Pt · · · Cl_1	3·13 Å
Pt–N_1	1·97	Cl_2 · · · N	3·29
N–C_1	1·41	Cl_2 · · · C_1	3·94
C_1–C_2	1·57	Cl_2 · · · C_2	4·12
		Cl_1 · · · C_1	2·70 3·12
Pt–N–C_1	113°		
N–C_1–C_2	113°		

Structure factors were calculated with the approximate atomic coordinates which had been deduced. Because of the Pt and Cl_1 atoms both at $x=0$, $y=0$ in the centrosymmetric (001) projection, all structure factors F_{hk0} were of positive sign. The reliability factor was $R=0·14$. A Fourier synthesis was calculated, and, as expected, appeared very similar to the (001) Patterson projection. The agreement was improved to give $R=0·10$† by two cycles of difference syntheses and structure-factor calculations, during which there were small adjustments in atomic coordinates and individual isotropic temperature factors were assigned (see Table 1). The structure factors F_{0kl} calculated using Cl_1 positions according to the alternative type (A) and type (B) chains showed, at first, equally good agreement with F_o, with R approximately 0·15 in both cases. However, only the type (B) case showed improving agreement after several cycles of difference syntheses and structure-factor calculations. The possibility of type (A) chains was thus discarded. The final atomic coordinates and anisotropic temperature factors obtained after four cycles are listed in Table 1. These values gave the agreement $R=0·087$.† A final (100) Fourier synthesis is shown in Fig. 3. Some bond lengths and interatomic distances are listed in Table 3 and the observed and calculated structure factors in Table 2. The atomic scattering factors used in these calculations were those of Thomas & Fermi for platinum (*Internationale Tabellen zur Bestimmung von Kristallstrukturen*, 1935), of Berghuis *et al.* (1955) for chlorine, and of McWeeny (1951) for carbon and nitrogen.

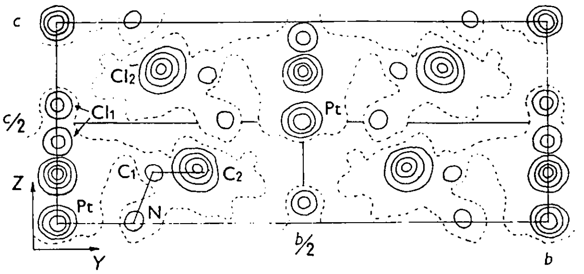

Fig. 3. The final Fourier projection on (100), with the platinum atom subtracted to eliminate series termination effects. The broken contour is at 1 e.Å^{-2} and the full contours at 3, 6, 9 and 12 e.Å^{-2}.

There was no evidence of the water-molecule positions of any of the difference syntheses.

Determination of the disordered structure

The continuous streaking which is observed interleaving the layer lines of normal Bragg reflections in photographs taken of crystals mounted about c shows that the c cell parameter should be doubled and that in reciprocal space there are continuous sheets inter-

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† The observed amplitudes for the planes 200, 400, 110 and 011 were assumed to be affected by extinction and were not included in the agreement factors.

leaving the normal lattice nets perpendicular to c^*. Such sheets in reciprocal space arise if the structure contains ordered lines of atoms parallel to c with a repeating distance of $2c$, and also if there is no long-range order in the structure in any direction other than c. The extremely weak intensity of the observed streaking shows that the electron density in the ordered or 'average' subcell is not greatly different from the electron-density distribution in the actual disordered structure.

Consideration of the sharp Bragg reflections $0kl$ showed the Pt–Cl–Pt chains to be of type (B) with a repeating distance of $2c$ or 10·78 Å. The failure to detect any intensity maxima in the diffuse layers perpendicular to c^* indicates that there is very little, if any, short-range order in the packing of the chains. Since the nearest-neighbour chain axes are separated by 9·4 Å, there will be only small Coulombic interactions between atoms of neighbouring chains. Whatever the lowest energy configuration may be, there will be others almost as favourable, energetically, formed from the occasional slipping of chains parallel to their length by a distance of c, thus replacing Pt^{II} by Pt^{IV} and vice versa. Geometrically, a given chain could be translated along its length by a distance of c without affecting the overall packing in the structure, since such a translation is effectively only a translation of the Cl_1 atoms flanking a given Pt^{IV} (0·8 Å in opposite senses).

The loss of long-range order, arising from the 'mistakes' in the stacking of the chains, can thus explain satisfactorily both the presence and the weak intensity of the observed streaking on the photographs.

The four Cl_2 ions of the subcell cannot be distributed completely at random in the eightfold positions (0·21, 0·21, 0·75) etc., since the positions such as (0·21, 0·21, 0·75) and (0·29, 0·29, 0·25) are separated by only 3·1 Å and this is too close for both to be occupied in the same subcell by chloride ions. The four cylindrical holes parallel to the Pt–Cl chain axes must each contain one chloride ion per subcell in one of two possible ordered arrangements, e.g. in the hole at $x=\frac{1}{4}$, $y=\frac{1}{4}$, all Cl_2 must be at either (0·21, 0·21, 0·75) or at (0·29, 0·29, 0·25). In a given hole, the chloride ions thus lie on one of two straight lines parallel to, but slightly displaced from the central axis of the cylindrical hole, and the separation between chloride ions along these lines is c or 5·4 Å.

If there is no long-range order in the arrangement of the chloride ions in directions perpendicular to c, diffuse diffraction phenomena should be observed on layer lines for all values of l. Such phenomena were not observed, even on oscillation photographs with exposures exceeding 1500 milliamp. hours, and hence the chloride ions must lie on an ordered lattice. The symmetry of the structure is then not truly tetragonal, and reflections such as hkl and khl should differ slightly in intensity. Because the intensity measurements were essentially of low accuracy it is not possible to confirm or refute this deduction. Moreover it is feasible that the apparently single crystal obtained was in fact an accurately aligned twin, with a and b interchanged, the individual crystallites being sufficiently large to give sharp reflections. The departure from tetragonal diffraction symmetry might then be very small.

It is suggested that the displacement of the chloride ions from the more symmetrical arrangement at (0·25, 0·25, 0·75) etc., results because the water molecules have to be accommodated in the same cylindrical holes. Although there is no direct evidence from the X-ray data for the water-molecule positions, they could be present at or near the vacant Cl_2 sites, where they would be in a position with respect to the chloride ions compatible with a weak hydrogen bonding interaction. Their effect would then be to decrease the intensity of any diffuse reflections or deviations from tetragonal symmetry due to the Cl_2 arrangement.

Discussion

Since the platinum atom contribution to all the observable structure factors is completely in phase, the light atom contributions are always heavily outweighed. The chlorine atoms are distributed throughout the 'average' structure as half atoms so that their scattering is only comparable to that of nitrogen and carbon atoms. This, together with the marked absorption errors in the observed intensity data, has limited the accuracy with which the atomic parameters can be determined. The present structure analysis can thus only claim to present a general outline of the crystal structure of Wolffram's red salt.

However, the dimeric formulation of Wolffram's red salt by Reihlen & Flohr (1934) has been established, rather than the monomeric formulation of Drew & Tress (1935), the crystal structure containing chains of alternating Pt^{IV} and Pt^{II} with octahedral and square planar coordination respectively.

Each chlorine atom in the chain may be regarded as forming a covalent type bond to a Pt^{IV} with length 2·26 Å and also taking part in a weaker interaction with Pt^{II} at a distance of 3·13 Å. The two Pt^{IV}–Cl_1 bonds appear to be of equal length, regardless of the C_1–Cl_1 repulsions, which should be considerably greater for one Cl_1 atom than the other.

The crystal-structure determination of the anhydrous bromine analogue of Wolffram's red salt has been undertaken as it is hoped that with the heavier bromine atom, a more precise investigation will be possible of the stereochemistry of the platinum–halogen chains which seem characteristic of this class of compound.

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Smith and the staff of the Adolph Basser Computing Laboratory, University of Sydney, for performing some of the calculations, and for the use of the IBM 650 computer of the Computation and Data Processing Center, University of Pittsburgh, and the structure-factor programme of R. Shiono (1957), for other calculations.

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The Crystal Structure of Diazoaminobenzene Copper (I)

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Crystals of diazoaminobenzene copper(I) are monoclinic, space group $C_{2h}^{6}(I2/a)$ with

$$a = 16\cdot00,\ b = 5\cdot61,\ c = 24\cdot01\ \text{Å};\ \beta = 99^\circ\ 20'.$$

The structure has been determined by standard X-ray diffraction methods and refined by least-squares analysis of the three-dimensional intensity data ($R = 0\cdot09$).

The crystal contains discrete, centrosymmetric, approximately planar, dimeric molecules in which pairs of diazoamino groups are linked through linearly coordinated copper atoms to form an eight-membered ring (N–Cu, 1·90, 1·94 Å). The trans-annular Cu–Cu separation, 2·45 Å, is unusually small.

Introduction

Diazoaminobenzene copper (I),

$$(C_6H_5{-}N{=}N{-}N{-}C_6H_5)^{-}Cu^{+},$$

hereinafter referred to as DAB.Cu(I), is one of a series of stable complexes formed between diazoaminobenzene and transition metals (Dwyer, 1941). Little direct evidence from which a convincing structure could be proposed for DAB.Cu(I) has hitherto been available, although Meunier & Rigot (1900), who first prepared the compound, Dwyer (1941), and recently Harris, Hoskins & Martin (1959), have suggested possible atomic arrangements. The present paper, which describes the determination of its crystal structure, forms part of a wider study of compounds containing copper(I) and azo groups (Brown & Dunitz, 1960).

Crystal data

Diazoaminobenzene copper(I), $C_{12}H_{10}N_3Cu$.
Monoclinic,

$$a = 16\cdot00 \pm 0\cdot05,\ b = 5\cdot61 \pm 0\cdot05,\ c = 24\cdot01 \pm 0\cdot05\ \text{Å};$$
$$\beta = 99^\circ\ 20' \pm 11'.$$

Volume of unit cell = 2127 Å^3.
Density (measured) = $1\cdot65 \pm 0\cdot03$ g.cm.$^{-3}$.
Density (calculated) = $1\cdot62 \pm 0\cdot03$ g.cm.$^{-3}$.
Eight formula units per unit cell.
Systematic absences: hkl if $h+k+l$ is odd,
$h0l$ if h or l is odd.

Space group: $C_s^4(Ia)$ or $C_{2h}^6(I2/a)$. The analysis leads to a structure based on the latter, centrosymmetric, space group which is conventionally described in terms of the orientation $C2/c$.

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