Short Communications

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Acta Cryst. (1968). B**24**, 156

Confirmation and refinement of the structure of $Hg_3S_2Cl_2$.

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(*Received* 24 *February* 1967 *and in revised form* 19 *September* 1967)

α–$Hg_3S_2Cl_2$ is isometric, with $a = 8{\cdot}949 \pm 0{\cdot}002$ Å, space group $I2_13$ (199) and with the atoms in the following special positions: 12 Hg in 12(*b*); $x, 0, \frac{1}{4}$; with $x = 0{\cdot}3029 \pm 0{\cdot}0005$; 8 S in 8(*a*); x,x,x; with $x = 0{\cdot}2709 \pm 0{\cdot}0029$; 8 Cl in 8(*a*); x,x,x; with $x = 0{\cdot}0107 \pm 0{\cdot}0028$.

Puff & Kuster (1962) determined the crystal structure of $Hg_3S_2Cl_2$ from powder diffraction data. More recently Carlson (1967) in a series of vapor-transport experiments in the system HgS–HCl obtained single crystals of three polymorphs of the compound. The diffraction of the α-phase corresponded to the phase described by Puff & Kuster. The morphology of the single crystals indicated the crystal class $2/m\bar{3}$, which was in disagreement with the non-centrosymmetric space group $I2_13$ of the earlier determination. Close examination revealed fine striations on the cube faces in the directions [110] and [100], indicating that mirror twinning perpendicular to the twofold axis may cause the apparent centrosymmetric morphology.

A Geiger-counter goniometer with Cu $K\alpha$ radiation was used to record 255 independent reflections from three levels on a ground sphere. The data were corrected for absorption, Lorentz and polarization and the following parameters were found.

Table 1. *F observed, F calculated, A calculated and B calculated for all observed reflections of* $Hg_3S_2Cl_2$

H	K	L	F(O)	F(C)	A(C)	B(C)	H	K	L	F(O)	F(C)	A(C)	B(C)	H	K	L	F(O)	F(C)	A(C)	B(C)
2	0	0	216.6	210.5	-208.3	-3C.4	6	7	1	94.2	102.1	102.1	0.2	3	9	2	63.6	51.4	33.7	38.8
4	0	0	638.9	639.3	634.2	80.7	8	7	1	87.9	90.8	-64.7	63.8	-1	9	2	85.4	72.4	28.0	66.8
6	0	0	92.6	77.3	76.3	12.3	-2	7	1	106.6	88.5	-77.2	-43.4	-3	9	2	65.0	59.4	56.9	-16.9
8	0	0	197.7	181.1	179.1	27.2	-4	7	1	183.1	179.3	97.5	150.5	0	10	2	72.2	82.8	81.4	13.5
10	0	0	90.9	85.1	83.4	17.0	-6	7	1	108.4	95.9	-89.0	-35.8	4	10	2	105.4	111.2	109.7	18.4
1	1	0	198.2	175.1	-24.2	173.4	-8	7	1	95.9	85.9	33.6	79.1	-4	10	2	113.7	112.7	109.5	26.8
3	1	0	150.5	159.7	-27.0	157.4	1	8	1	72.8	80.8	-59.5	54.6	3	0	3	329.8	305.3	47.6	-301.5
5	1	0	111.8	99.3	25.5	-96.0	3	8	1	177.0	190.3	-1.9	190.3	5	0	3	134.6	119.4	-14.5	118.5
11	1	0	67.2	83.5	-17.3	81.7	5	8	1	63.5	43.4	-37.5	-21.8	7	0	3	183.7	178.7	31.5	-175.9
2	2	0	91.3	70.6	-61.6	-34.4	7	8	1	109.8	119.4	-6.1	119.2	0	1	3	460.1	479.7	-66.2	475.1
4	2	0	308.6	307.7	-303.3	-52.0	-1	8	1	72.8	81.8	31.5	75.4	2	1	3	207.1	151.7	-134.4	-70.4
6	2	0	108.4	117.0	116.8	7.7	-3	8	1	214.8	186.5	-74.2	171.1	4	1	3	454.6	401.7	113.4	385.4
8	2	0	247.0	269.4	-262.8	-59.2	-7	8	1	129.1	114.9	-44.9	105.8	6	1	3	160.9	131.5	-121.6	-50.1
10	2	0	74.7	82.8	81.7	13.5	0	9	1	113.2	99.3	25.5	-96.0	8	1	3	192.6	190.3	-1.9	190.3
1	3	0	476.7	479.7	-66.2	475.1	2	9	1	85.5	91.5	28.5	86.9	-2	1	3	151.9	145.3	142.8	-26.7
3	3	0	269.3	305.3	47.6	-301.5	4	9	1	99.5	100.6	-32.0	-95.4	-4	1	3	428.1	396.2	-228.7	323.6
5	3	0	148.9	160.8	-23.2	159.1	6	9	1	76.7	68.0	21.9	64.4	-6	1	3	116.7	127.8	127.8	-1.0
4	4	0	326.3	353.1	349.9	47.1	-2	9	1	95.3	84.2	-54.9	63.9	-8	1	3	170.5	186.5	-74.2	171.1
6	4	0	173.5	162.5	159.7	30.2	-4	9	1	105.0	99.0	74.4	-62.9	1	2	3	293.8	252.9	115.4	225.1
8	4	0	98.8	78.2	77.8	8.4	-6	9	1	79.6	57.4	-42.0	39.2	3	2	3	151.2	127.0	-120.3	-40.7
10	4	0	100.2	116.9	114.0	20.1	0	11	1	63.7	83.5	-17.3	81.7	5	2	3	259.9	239.7	53.1	233.8
3	5	0	110.0	119.4	-14.5	118.5	6	0	2	248.1	248.7	-241.3	-60.3	7	2	3	81.7	70.9	-70.7	4.8
5	5	0	155.2	153.7	28.5	-151.1	8	0	2	112.3	128.8	126.4	24.8	9	2	3	65.3	51.4	33.7	38.8
7	5	0	174.8	195.7	-36.4	192.3	10	0	2	138.5	160.8	-154.6	-44.4	-1	2	3	228.3	253.4	-177.2	181.1
9	5	0	128.7	125.8	31.7	-121.7	1	1	2	405.0	358.1	206.6	-292.5	-3	2	3	122.9	138.5	138.5	0.5
2	6	0	237.0	248.7	-241.3	-60.3	3	1	2	286.4	252.9	115.4	225.1	-5	2	3	252.6	243.1	-140.8	198.1
8	6	0	110.6	94.9	-91.6	-24.6	5	1	2	117.6	103.1	103.0	4.7	-7	2	3	75.1	84.5	77.3	34.0
1	7	0	275.4	264.4	-44.6	260.6	9	1	2	94.9	91.5	28.5	86.9	-9	2	3	59.4	59.4	-56.9	16.9
3	7	0	178.3	178.7	31.5	-175.9	-1	1	2	325.1	356.8	110.6	339.3	2	3	3	140.2	127.0	-120.3	-40.7
5	7	0	116.5	99.2	-14.5	98.1	-3	1	2	257.5	253.4	177.2	-181.1	4	3	3	285.3	290.3	207.2	-203.3
2	8	0	131.4	128.8	126.4	24.8	-5	1	2	104.3	95.5	89.6	32.9	6	3	3	132.2	127.7	-121.5	-39.5
4	8	0	104.5	78.2	77.8	8.4	-9	1	2	81.5	84.2	54.9	-63.9	8	3	3	131.6	134.8	76.0	-111.3
6	8	0	172.1	176.6	172.2	39.2	0	2	2	89.0	70.6	-61.6	-34.4	-2	3	3	115.2	138.5	138.5	0.5
2	10	0	152.9	160.8	-154.6	-44.4	2	2	2	627.3	568.8	562.2	86.6	-4	3	3	285.7	289.7	-124.3	-261.6
1	11	0	58.2	54.4	-7.3	53.9	4	2	2	96.0	75.9	75.5	-8.0	-6	3	3	111.2	135.5	135.4	7.1
1	0	1	185.4	175.1	-24.2	173.4	6	2	2	156.0	178.7	175.4	34.3	-8	3	3	128.6	131.8	-21.3	-130.1
3	0	1	452.8	479.7	-66.2	475.1	8	2	2	134.9	140.0	138.9	17.6	1	4	3	217.4	192.9	-185.9	-51.4
7	0	1	258.1	264.4	-44.6	260.6	10	2	2	43.9	52.5	51.2	11.5	3	4	3	274.0	290.3	207.2	-203.3
2	1	1	383.5	358.1	206.6	-292.5	-2	2	2	558.8	568.5	562.3	84.0	5	4	3	164.0	165.0	-150.9	66.9
4	1	1	237.4	240.0	-221.2	93.1	-4	2	2	88.3	75.4	75.3	-2.7	7	4	3	160.3	170.8	120.7	-120.8
6	1	1	234.2	233.9	170.0	-160.7	-6	2	2	174.5	178.0	175.5	29.9	9	4	3	70.8	65.6	-64.3	-13.0
10	1	1	90.0	87.4	3.3	-87.4	-8	2	2	148.7	140.7	138.7	23.9	-1	4	3	181.4	199.3	199.0	11.3
-2	1	1	330.2	356.8	-110.6	-339.3	-10	2	2	33.7	51.9	51.3	7.9	-3	4	3	289.8	289.7	-124.3	-261.6
-4	1	1	218.6	239.7	179.0	159.5	1	3	2	164.3	151.7	-134.4	-70.4	-5	4	3	152.9	172.3	125.6	118.0
-6	1	1	243.9	234.1	-97.0	-213.0	3	3	2	135.9	127.0	-120.3	-40.7	-7	4	3	165.1	174.8	-65.8	-161.9
-10	1	1	86.8	89.9	38.8	-81.1	5	3	2	171.3	170.8	-114.8	126.5	-9	4	3	65.5	77.5	75.3	18.6
3	2	1	156.2	151.7	-134.4	-70.4	7	3	2	160.0	161.7	-43.4	-155.8	0	5	3	174.5	160.8	-23.2	159.1
5	2	1	320.1	319.9	161.3	-276.2	9	3	2	136.0	156.4	-72.1	138.8	2	5	3	154.4	170.8	-114.8	126.5
7	2	1	98.2	105.1	-71.6	-76.9	-1	3	2	149.0	145.3	-142.8	26.7	4	5	3	173.7	175.2	89.5	150.6
9	2	1	61.4	80.5	67.8	-43.4	-3	3	2	118.7	138.5	-138.5	-0.5	6	5	3	104.2	124.3	-97.9	76.5
-3	2	1	149.8	145.3	142.8	-26.7	-5	3	2	160.1	172.9	-61.1	-161.8	8	5	3	60.7	74.6	0.2	74.6
-5	2	1	317.8	317.1	-43.2	-314.1	-7	3	2	160.0	169.9	-113.5	126.3	-2	5	3	166.1	172.9	61.1	161.8
-7	2	1	111.3	96.0	84.3	-45.8	-9	3	2	142.3	160.5	-6.2	-160.4	-4	5	3	171.3	164.5	-129.9	100.9
-9	2	1	75.9	72.4	-28.0	-66.8	0	4	2	297.4	307.7	-303.3	-52.0	-6	5	3	129.5	128.9	57.0	115.7
2	3	1	262.0	252.9	115.4	225.1	4	4	2	116.0	116.0	-114.3	-19.6	-8	5	3	84.3	65.5	-26.8	59.8
4	3	1	176.2	192.9	-185.9	-51.4	6	4	2	88.5	111.9	-104.9	-38.9	1	6	3	186.8	188.0	114.7	149.0
6	3	1	159.8	188.0	114.7	149.0	10	4	2	76.3	92.2	-86.4	-32.1	3	6	3	118.2	127.7	-121.5	-39.5
10	3	1	62.5	56.7	-14.4	54.8	-4	4	2	108.5	117.1	-114.1	-26.2	5	6	3	162.7	176.1	62.6	164.6
-2	3	1	238.6	253.4	-177.2	181.1	-6	4	2	103.7	109.0	-105.2	-28.7	7	6	3	60.6	77.5	-77.2	-6.9
-4	3	1	179.2	199.3	199.0	11.3	-10	4	2	88.1	89.9	-86.7	-23.7	-1	6	3	169.9	189.8	-161.7	99.3
-6	3	1	173.6	189.8	-161.7	99.3	1	5	2	322.7	319.9	161.3	-276.2	-3	6	3	111.2	135.5	135.4	7.1
-10	3	1	72.1	61.1	-12.7	59.8	3	5	2	223.5	239.7	53.1	233.8	-5	6	3	202.8	179.5	-129.4	124.4
3	4	1	373.6	401.7	113.4	385.4	5	5	2	87.1	107.8	89.9	-59.4	-7	6	3	87.5	86.4	82.4	26.2
5	4	1	134.9	153.0	-140.8	-59.8	7	5	2	59.4	69.1	54.9	41.9	2	7	3	150.3	161.7	-43.4	-155.8
7	4	1	208.9	238.6	57.4	231.6	-1	5	2	327.2	317.1	43.2	314.1	4	7	3	109.9	110.3	109.4	14.0
9	4	1	87.3	87.4	-86.2	14.5	-3	5	2	253.9	243.1	140.8	-198.1	6	7	3	105.0	128.9	-62.5	-112.8
-3	4	1	393.6	396.2	-228.7	323.6	-5	5	2	98.1	101.3	46.3	90.0	8	7	3	20.1	43.4	43.1	4.7
-5	4	1	150.0	145.0	144.9	-9.6	-7	5	2	87.0	69.6	67.6	-10.4	-2	7	3	188.8	169.9	113.5	-126.3
-7	4	1	234.3	231.1	-134.9	187.9	0	6	2	107.2	117.0	116.8	7.7	-4	7	3	109.9	107.0	-103.3	-27.9
-9	4	1	86.4	77.2	60.3	48.1	4	6	2	164.5	184.5	183.3	20.3	-6	7	3	135.1	140.2	115.8	-79.1
2	5	1	109.6	103.1	103.0	4.7	8	6	2	168.6	179.2	172.7	29.0	-8	7	3	36.2	40.2	-39.1	-9.3
4	5	1	140.1	136.8	-114.0	-75.5	-4	6	2	199.5	185.6	183.1	30.5	1	8	3	90.9	59.2	-55.0	-22.0
6	5	1	84.6	86.6	86.0	10.8	-8	6	2	188.7	177.2	172.4	41.1	3	8	3	133.0	134.8	76.0	-111.3
-2	5	1	115.9	95.5	-89.6	-32.9	1	7	2	101.1	105.1	-71.6	-76.9	5	8	3	53.8	67.6	-55.5	38.6
-4	5	1	156.9	138.5	136.7	-22.2	3	7	2	64.7	70.9	-70.7	4.8	7	8	3	69.2	81.5	43.4	-69.0
-6	5	1	106.6	81.8	-75.8	-30.9	5	7	2	87.0	85.4	-70.7	48.0	-1	8	3	47.3	63.7	63.7	-1.5
3	6	1	140.9	131.5	-121.6	-50.1	-1	7	2	78.1	83.7	-25.2	-79.8	-3	8	3	132.3	131.8	-21.3	-130.1
5	6	1	192.5	221.2	137.7	-175.1	-1	7	2	116.2	96.0	-84.3	45.8	-5	8	3	69.8	67.8	39.1	55.4
9	6	1	59.9	66.7	60.2	-28.7	-3	7	2	72.9	84.5	-77.3	-34.0	-7	8	3	91.4	82.3	-7.4	-82.0
-3	6	1	151.8	127.8	127.8	-1.0	-5	7	2	81.0	82.4	-38.0	-73.1	2	9	3	140.4	156.4	-72.1	138.8
-5	6	1	239.8	220.6	-47.8	-215.4	-7	7	2	95.1	92.0	-70.6	58.9	4	9	3	64.0	55.8	50.0	24.6
-9	6	1	73.7	62.3	-29.8	-54.7	0	8	2	283.4	269.4	-262.8	-59.2	-2	9	3	147.6	160.5	6.2	160.4
0	7	1	137.0	159.7	-27.0	157.4	4	8	2	142.8	153.1	-149.5	-33.1	-4	9	3	68.6	46.7	-40.4	-5.7
2	7	1	100.8	95.2	94.4	-11.9	-4	8	2	162.6	154.7	-149.3	-40.5	1	10	3	59.5	56.7	-14.4	54.8
4	7	1	171.6	178.9	-144.5	105.5	1	9	2	93.8	80.5	67.8	-43.4	-1	10	3	60.9	61.1	-12.7	59.8

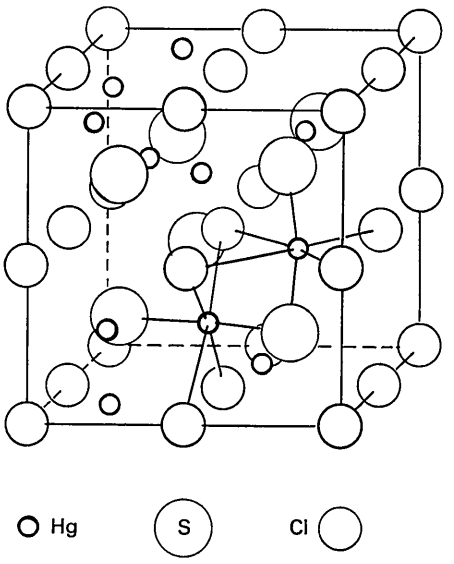

Fig. 1. The crystal structure of $Hg_3S_2Cl_2$.

Isometric: $a=8{\cdot}949\pm0{\cdot}002$ Å, $U=716{\cdot}7$ Å^3, $D_m=6{\cdot}895\pm0{\cdot}086$ g.cm^{-3}. $Z=4$, $D_x=6{\cdot}827$ g.cm^{-3}.
Space group $I2_13$ (no. 199).

Atomic positions

12 Hg in 12(*b*); x, 0, ¼; with $x=0{\cdot}3029\pm0{\cdot}0005$
8 S in 8(*a*); x, x, x; with $x=0{\cdot}2709\pm0{\cdot}0029$
8 Cl in 8(*a*); x, x, x; with $x=0{\cdot}0107\pm0{\cdot}0028$

In the structure (Fig. 1) the Hg is octahedrally (*trans*) coordinated to:

2 S at $2{\cdot}45\pm0{\cdot}02$ Å
2 Cl at $2{\cdot}87\pm0{\cdot}01$
2 Cl at $3{\cdot}38\pm0{\cdot}04$

The bond angles within the octahedra are:

S–Hg–S $166\pm1°$
Cl–Hg–Cl $78{\cdot}7\pm0{\cdot}1°$
(2) Cl–Hg–Cl $86{\cdot}2\pm0{\cdot}6°$
Cl–Hg–Cl $109\pm1°$

Each octahedron shares a face with an adjoinning octahedron, the Hg–S–Hg angle between octahedra being $92\pm1°$. This results in the nearest Hg–Hg distance being $3{\cdot}546\pm0{\cdot}004$ Å.

The structure was refined by least-squares using isotropic temperature coefficients and correcting for anomalous scattering, until the R value for 255 *hkl* reflections was 0·08 (weighted, $R=0{\cdot}077$). The resulting isotropic temperature factors are $B_{Hg}=2{\cdot}74\pm0{\cdot}03$; $B_S=1{\cdot}5\pm0{\cdot}3$ and $B_{Cl}=1{\cdot}8\pm0{\cdot}2$. The F calculated are compared with F observed in Table 1.

This work was supported by National Research Council of Canada Grant A-165.

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Acta Cryst. (1968). B**24**, 157

Trimethylplatinum(IV) iodide and its misrepresentation as hexamethyldiplatinum*.

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(*Received* 10 *July* 1967)

Transparent, tan-colored trimethylplatinum(IV) iodide crystals have cell dimensions $17{\cdot}7_{7\pm5}$, $19{\cdot}3_{9\pm6}$, $17{\cdot}1_{2\pm5}$ Å, $\beta=115°41\pm5'$, space group $P2_1/a$, and 32 molecules per cell. These data duplicate the ones erroneously ascribed to hexamethylplatinum by Illuminati & Rundle [*J. Amer. Chem. Soc.* (1949) **71**, 3575], whose published (010) Patterson projection enables eight tetramers of the molecule to be located in the crystal structure. This tetramer has a configuration similar to that reported for the tetramer of trimethylplatinum(IV) chloride.

Trimethylplatinum(IV) iodide has been assumed (Kite, Smith & Wilkins, 1966) to be cubic and isostructural with trimethylplatinum(IV) chloride (Rundle & Sturdivant, 1947), although Burovaya (1949) studied its morphology, pointed out that it is monoclinic, point group $2/m$, and observed forms 001, 110, 100, 011, leading to axial elements $a:b:c=1{\cdot}778:1:1{\cdot}826$; $\beta=116°02'$. Our own data follow.

On recrystallizing $Pt(CH_3)_3I$, two distinct phases are obtained. One phase, recrystallized from benzene, is solvated; the other, recrystallized from toluene, is not. The solvated phase loses its benzene molecules within minutes when exposed to air. The change in crystal phase is readily observed because, although the crystal faces are perfectly preserved, the tan-colored transparent crystals turn yellow and opaque. Such a yellow pseudomorph gives a spotty X-ray powder pattern, showing a grain size of at least 1000 Å and little preferred orientation. This powder pattern is identical with that of the $Pt(CH_3)_3I$ recrystallized from toluene, thus showing that no toluene molecules are captured in the phase obtained from toluene. A combustion analysis, performed by J. Walter, gave C 9·83, H 2·65%; calculated for $Pt(CH_3)_3I$: C 9·8; H 2·5%.

The unsolvated crystals are brittle, 2 to 3 mm in largest dimension, transparent, tan-colored, thick-tabular (100) with point-group symmetry $2/m$. Optical extinction is sharp parallel to an edge which is chosen as the b axis. The observed forms, measured with excellent signals on the optical goniometer, are indexed 100, 021, 001, and 120. The cell to which these indices refer has dimensions: $a=17{\cdot}77\pm5$, $b=19{\cdot}39\pm6$, $c=17{\cdot}12\pm5$ Å and $\beta=115°41'\pm5'$, cell volume = 5316 Å^3 (precession films, Cu $K\alpha$, $\lambda=1{\cdot}5418$ Å), and axial ratios $a:b:c=0{\cdot}9165:1:0{\cdot}8829$, requiring a transformation matrix 001/020/100 to be applied to Burovaya's morphological cell. The observed forms thus indicate a

* Work supported in part by National Science Foundation Grant no. GP-4947, awarded to The Johns Hopkins University.