O_2 and O_3 atoms of the upper square faces of the antiprisms become his O_1 and O_7 atoms.

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The Crystal Structure of $MoCl_5$*

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$MoCl_5$ has the $NbCl_5$ type structure. The crystal system is monoclinic with

$$a = 17{\cdot}31,\ b = 17{\cdot}81,\ c = 6{\cdot}079\ \text{Å},\ \beta = 95{\cdot}7°.$$

The space group is $C2/m$, and there are 12 $MoCl_5$ units in the unit cell. The structure consists of dimers (Mo_2Cl_{10}) with the chlorine atoms forming two octahedra which share a common edge. The molybdenum atoms occupy the centers of the octahedra and are joined by two chlorine bridge bonds. The Mo–Cl bridge bond length is 2·53 Å; the Mo–Cl non-bridge bond length is 2·24 Å.

Introduction

The structural unit of $MoCl_5$ in the vapor phase, determined by electron diffraction (Ewens & Lister, 1938), is the trigonal bipyramid. The trigonal bipyramid unit is also present in gaseous $NbCl_5$ (Skinner & Sutton, 1940); however, solid $NbCl_5$ consists of Nb_2Cl_{10} dimers (Zalkin & Sands, 1958). Measurements of the Trouton constants of the niobium and tantalum pentahalides (Fairbrother & Frith, 1951), and of the electrical conductivities of NbF_5 and TaF_5 (Fairbrother, Frith & Woolf, 1954) suggest dimerization in these compounds in the liquid state.

Vapor density measurements by Debray (Remy, 1956) at 350 °C. support the molecular formula $MoCl_5$ in the gaseous state. Mesnage (1939) found two spectra for $MoCl_5$, *A* occurring between 100 and 200 °C., and *B* above 300 °C.; the transition from *A* to *B* is attributed to depolymerization.

Experimental

The molybdenum pentachloride was obtained from the Climax Molybdenum Company. Samples of the extremely hygroscopic, bluish-black powder were transferred in an argon-filled dry box to 0·3 mm. quartz capillaries. Crystals suitable for diffraction studies were grown by heating the capillaries to about 230 °C. and cooling over a period of several hours to room temperature.

X-ray measurements revealed that single crystals of at least three phases were obtained by this technique. The $MoCl_5$ was identified by color, density, melting point, and the similarity of the X-ray patterns to $NbCl_5$; a specimen of this material was selected for the structural investigation. The other materials present were believed to be oxychlorides.

Oscillation, Weissenberg, and precession photographs, using Cu $K\alpha$ radiation ($\lambda = 1{\cdot}5418$ Å) show a monoclinic unit cell with:

$$a = 17{\cdot}31 \pm 0{\cdot}01,\ b = 17{\cdot}81 \pm 0{\cdot}01,\ c = 6{\cdot}079 \pm 0{\cdot}005\ \text{Å};\ \beta = 95{\cdot}7 \pm 0{\cdot}1°.$$

* This work was performed under the auspices of the U.S. Atomic Energy Commission.

The specimen being studied was a flat plate of dimensions $0{\cdot}016 \times 0{\cdot}091 \times 2{\cdot}4$ mm., the unique axis being perpendicular to the plate and c parallel to the long edge. The crystal was oriented about c.

The measured density is 2·928 g.cm.$^{-3}$ (*Lange's Handbook of Chemistry*, 1956); the density calculated from the cell constants is 2·936 g.cm.$^{-3}$. There are 12 $MoCl_5$ formula units per unit cell. The diffraction symmetry and extinction conditions were characteristic of space group $C2$, Cm, and $C2/m$. In addition to the hkl, $h+k \neq 2n$, extinctions required by the space group, $hk0$ reflections with $h \neq 3n$ were very weak or missing. The crystal was twinned on (100), with the twins sharing a common c-axis.

The relative intensities of the reflections on the $l=0$, 1, 2, 3, and 4 equi-inclination Weissenberg layers, made with Cu $K\alpha$ radiation and the usual multiple-film technique, were estimated by visual comparison with a standard scale. Values of $|F|^2$ were obtained by correction for the Lorentz, polarization, and velocity factors. Only those reflections amenable to unambiguous indexing and ease of measurement were included, for a total of 676 reflections of which 185 were too weak to be observed.

The $hk0$ data were corrected for absorption by the method of Busing & Levy (1957) applied to the IBM 650 (Sands, 1958).

Table 1. *Observed and calculated structure factors for the hk0 reflections of* $MoCl_5$

h	k	l	F_o	F_c	h	k	l	F_o	F_c	h	k	l	F_o	F_c	h	k	l	F_o	F_c
0	2	0	17	15	4	8	0	<2	<1	9	1	0	13	12	14	2	0	<4	1
0	4	0	44	-43	4	10	0	<1	<1	9	3	0	10	-10	14	4	0	<4	-1
0	6	0	20	-20	4	12	0	<1	<1	9	5	0	29	-32	14	6	0	<4	-1
0	8	0	14	14	4	14	0	<1	<1	9	7	0	4	4	14	8	0	<3	<1
0	10	0	59	65	4	16	0	<4	-1	9	9	0	18	18	14	10	0	<3	<1
0	12	0	5	5	4	18	0	<3	<1	9	11	0	<5	-1	14	12	0	<4	1
0	14	0	24	-22	4	20	0	<1	<1	9	13	0	16	-13	14	14	0	<4	<1
0	16	0	<1	<1	5	1	0	<1	<1	9	15	0	16	-12	14	16	0	<3	-1
0	18	0	9	9	5	3	0	<1	<1	9	17	0	10	7	15	1	0	22	23
0	20	0	23	16	5	5	0	2	-1	9	19	0	12	9	15	3	0	11	-10
0	22	0	<1	<1	5	7	0	<3	-1	10	0	0	<1	<1	15	5	0	12	-12
1	1	0	<1	<1	5	9	0	<1	<1	10	2	0	<1	<1	15	7	0	<4	-1
1	3	0	<1	<1	5	11	0	<1	<1	10	4	0	<3	2	15	9	0	22	18
1	5	0	<1	<1	5	13	0	<1	<1	10	6	0	3	3	15	11	0	13	12
1	7	0	<1	<1	5	15	0	<3	<1	10	8	0	<1	<1	15	13	0	12	-10
1	9	0	<1	<1	5	17	0	<3	<1	10	10	0	<2	<1	15	15	0	<4	-5
1	11	0	<3	<1	5	19	0	<1	<1	10	12	0	<1	<1	16	0	0	<4	<1
1	13	0	<3	<1	5	21	0	<1	<1	10	14	0	<3	<1	16	2	0	<4	<1
1	15	0	<4	<1	6	0	0	25	-25	10	16	0	<4	1	16	4	0	<4	-2
1	17	0	<2	<1	6	2	0	4	4	10	18	0	<2	<1	16	6	0	<4	-3
1	19	0	<2	<1	6	4	0	26	-30	11	1	0	<1	<1	16	8	0	<4	<1
1	21	0	<3	<1	6	6	0	21	-20	11	3	0	<1	<1	16	10	0	<4	<1
2	0	0	<1	<1	6	8	0	29	35	11	5	0	3	2	16	12	0	<4	<1
2	2	0	<1	<1	6	10	0	15	-11	11	7	0	<3	<1	16	14	0	<2	<1
2	4	0	<2	<1	6	12	0	19	-17	11	9	0	<1	<1	17	1	0	<4	<1
2	6	0	<2	<1	6	14	0	22	-17	11	11	0	<2	<1	17	3	0	<4	<1
2	8	0	<1	<1	6	16	0	7	-5	11	13	0	<1	<1	17	5	0	<4	-2
2	10	0	<1	<1	6	18	0	22	15	11	15	0	<2	<1	17	7	0	<3	<1
2	12	0	<4	1	6	20	0	7	-3	11	17	0	<1	<1	17	9	0	<4	<1
2	14	0	<2	<1	7	1	0	<3	<1	12	0	0	35	52	17	11	0	<4	<1
2	16	0	<3	<1	7	3	0	<3	<1	12	2	0	10	8	17	13	0	<4	<1
2	18	0	<3	<1	7	5	0	<3	<1	12	4	0	20	-22	18	0	0	<4	2
2	20	0	<5	<1	7	7	0	<3	<1	12	6	0	13	-13	18	2	0	<4	<1
2	22	0	<2	<1	7	9	0	<4	<1	12	8	0	11	10	18	4	0	13	-10
3	1	0	28	43	7	11	0	<4	<1	12	10	0	35	33	18	6	0	9	-6
3	3	0	20	-22	7	13	0	<3	<1	12	12	0	<5	3	18	8	0	12	8
3	5	0	29	-34	7	15	0	<3	<1	12	14	0	13	-14	18	10	0	<3	1
3	7	0	<1	<1	7	17	0	<3	<1	12	16	0	<3	-1	18	12	0	9	-6
3	9	0	33	31	7	19	0	<3	<1	12	18	0	6	7	19	1	0	<2	<1
3	11	0	15	13	8	0	0	<1	<1	13	1	0	<2	<1	19	3	0	<2	<1
3	13	0	22	-18	8	2	0	<3	<1	13	3	0	<2	<1	19	5	0	<2	<1
3	15	0	14	-12	8	4	0	2	2	13	5	0	<1	<1	20	0	0	<2	<1
3	17	0	9	8	8	6	0	2	1	13	7	0	<1	<1	20	2	0	<2	<1
3	19	0	16	12	8	8	0	<3	-1	13	9	0	<2	<1	20	4	0	<1	<1
3	21	0	<1	<1	8	10	0	<3	<1	13	11	0	<3	<1	21	1	0	<4	2
4	0	0	<1	<1	8	12	0	<3	<1	13	13	0	<3	<1					
4	2	0	<1	<1	8	14	0	<2	<1	13	15	0	<2	<1					
4	4	0	2	-1	8	16	0	<3	<1	13	17	0	<1	<1					
4	6	0	<3	-2	8	18	0	<2	<1	14	0	0	<2	<1					

Determination of the structure

Comparison of the $hk0$ Weissenberg film of $MoCl_5$ with that of $NbCl_5$ indicated that, at least as far as this projection is concerned, the two structures are isomorphous. The x and y parameters of $NbCl_5$ (Zalkin & Sands, 1958) were used as the initial parameters in least-squares refinements of the $MoCl_5$ $hk0$ data. Individual isotropic temperature factors were assigned to each atom and were included in the refinements. The scattering factors of James & Brindley (1931) were used. A total of 194 independent reflections, of which 128 were too weak to be observed, were included in the computation. ($hk0$ reflections with $h \neq 3n$ accounted for 6 of the observed and 117 of the unobserved reflections.) A reliability factor of 15·6% was attained. The observed $hk0$ structure factors, corrected for absorption, are listed with the calculated values in Table 1. Fig. 1 shows the $\varrho(x, y)$ Fourier projection calculated from these data.

Refinement of the z parameters required a choice between the parameters directly analogous to those of $NbCl_5$ and the parameters derived from the unit cell with $\beta' = 180° - \beta$. Both sets of parameters were tested, and only in the latter case did the data refine. That is, for direct comparison with the $NbCl_5$ structure we should consider the $MoCl_5$ unit cell having $\beta = 84{\cdot}3°$. The convention of $\beta > 90°$ has been followed throughout this work, however.

The molybdenum atoms occupy positions 4(g) and 8(j) of space group $C2/m$ (*International Tables for X-ray Crystallography*, 1952); the chlorine atoms occupy three sets of 4(i) and six sets of 8(j) positions. These positions are as follows:

Table 2. *Atomic parameters in* $MoCl_5$

Position	Atom	x	y	z	B (Å^2)
4(g)	Mo_1	0·0000	0·1078 ± 0·0007	0·0000	2·1
8(j)	Mo_2	0·3333 ± 0·0004	0·1085 ± 0·0004	0·4348 ± 0·0012	2·2
4(i)	Cl_1	0·079 ± 0·002	0·000	0·867 ± 0·005	2·2
8(j)	Cl_2	0·077 ± 0·002	0·192 ± 0·002	0·855 ± 0·005	4·8
8(j)	Cl_3	0·075 ± 0·001	0·094 ± 0·001	0·323 ± 0·004	2·8
4(i)	Cl_4	0·260 ± 0·002	0·000	0·243 ± 0·006	3·5
8(j)	Cl_5	0·259 ± 0·002	0·193 ± 0·002	0·238 ± 0·005	4·0
8(j)	Cl_6	0·253 ± 0·001	0·093 ± 0·001	0·698 ± 0·004	3·4
4(i)	Cl_7	0·407 ± 0·002	0·000	0·628 ± 0·006	2·7
8(j)	Cl_8	0·407 ± 0·002	0·193 ± 0·002	0·629 ± 0·005	4·1
8(j)	Cl_9	0·414 ± 0·001	0·094 ± 0·001	0·167 ± 0·004	3·2

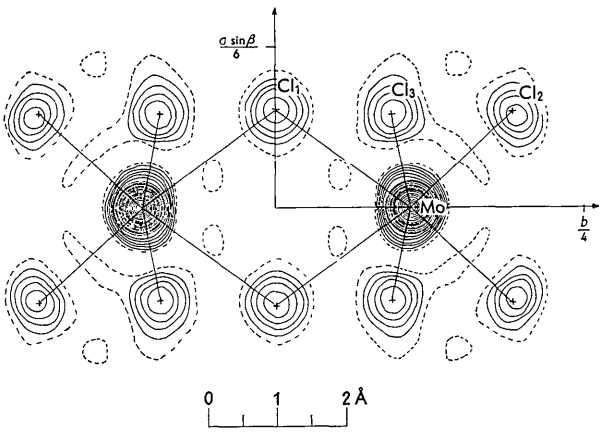

Fig. 1. Fourier projection of the Mo_2Cl_{10} molecule onto (001). Contours are at arbitrary intervals. The atomic positions as obtained from the least-square refinement are indicated by crosses.

4(*g*) $0, y, 0$; $0, \bar{y}, 0+C$ centering
4(*i*) $x, 0, z$; $\bar{x}, 0, \bar{z}+C$ centering
8(*j*) x, y, z; $x, \bar{y}, z$; $\bar{x}, y, \bar{z}$; $\bar{x}, \bar{y}, \bar{z}+C$ centering.

Least-squares refinement of all the measured data was started using the x and y parameters from the $hk0$ refinements and z parameters of $\frac{1}{2}$ for Mo and $\frac{1}{4}$ or $\frac{3}{4}$ for Cl. The final reliability factor was 16·5%. The data used in these refinements have been previously published (Sands & Zalkin, 1959).

The atomic parameters are listed in Table 2. The standard deviations of these parameters were computed by the method of Cruickshank (1949). Table 3 lists the distances between nearest neighbors; the probable errors in these distances were calculated from the standard deviations in the atomic parameters (Cruickshank & Robertson, 1953).

Discussion

Two independent configurations of the Mo_2Cl_{10} group are given by the structure, one of which is constrained to $2/m$ symmetry while the other is required to possess only a mirror plane. The Mo–Mo distances of the two molecules agree to within 0·7% and the Mo–Cl distances agree to about 1·0% in the worst case. Fig. 2

Table 3. *Nearest neighbor distances in* $MoCl_5$

Mo_1–1 Mo_1	3·817 ± 0·017 Å	Cl_3–1 Cl_2	3·66 ± 0·04* Å	Cl_6–1 Cl_6	3·31 ± 0·04 Å
–2 Cl_1	2·525 ± 0·024	–1 Cl_2	3·25 ± 0·04	–1 Cl_7	3·20 ± 0·04
–2 Cl_2	2·240 ± 0·033	–1 Cl_3	3·54 ± 0·04*	–1 Cl_8	3·26 ± 0·04
–2 Cl_3	2·257 ± 0·023	–1 Cl_3	3·34 ± 0·03	–1 Cl_9	3·78 ± 0·03*
		–1 Cl_4	3·68 ± 0·04*		
Mo_2–1 Mo_2	3·842 ± 0·011	–1 Cl_5	3·72 ± 0·04*	Cl_7–2 Mo_2	2·534 ± 0·022
–1 Cl_4	2·526 ± 0·024	–1 Cl_6	3·65 ± 0·03*	–1 Cl_4	3·29 ± 0·05
–1 Cl_5	2·243 ± 0·029	–1 Cl_8	3·78 ± 0·04*	–2 Cl_6	3·20 ± 0·04
–1 Cl_6	2·234 ± 0·027			–1 Cl_7	3·70 ± 0·05*
–1 Cl_7	2·534 ± 0·022	Cl_4–2 Mo_2	2·526 ± 0·024	–2 Cl_8	3·42 ± 0·03
–1 Cl_8	2·233 ± 0·029	–1 Cl_1	3·69 ± 0·05*	–2 Cl_9	3·67 ± 0·04*
–1 Cl_9	2·258 ± 0·027	–2 Cl_3	3·68 ± 0·04*	–2 Cl_9	3·27 ± 0·04
		–2 Cl_5	3·43 ± 0·03	–2 Cl_9	3·62 ± 0·04*
Cl_1–2 Mo_1	2·525 ± 0·024	–2 Cl_6	3·69 ± 0·04*		
–1 Cl_1	3·30 ± 0·05	–2 Cl_6	3·24 ± 0·04	Cl_8–1 Mo_2	2·233 ± 0·029
–2 Cl_2	3·40 ± 0·03	–1 Cl_7	3·29 ± 0·05	–1 Cl_2	3·73 ± 0·04*
–2 Cl_3	3·24 ± 0·04	–2 Cl_9	3·22 ± 0·04	–1 Cl_2	3·73 ± 0·04*
–2 Cl_3	3·70 ± 0·04*			–1 Cl_2	3·61 ± 0·04*
–2 Cl_3	3·26 ± 0·04	Cl_5–1 Mo_2	2·243 ± 0·029	–1 Cl_3	3·78 ± 0·04*
–1 Cl_4	3·69 ± 0·05*	–1 Cl_2	3·73 ± 0·04*	–1 Cl_5	3·32 ± 0·04
–2 Cl_6	3·68 ± 0·04*	–1 Cl_2	3·57 ± 0·04*	–1 Cl_5	3·66 ± 0·04*
		–1 Cl_3	3·72 ± 0·04*	–1 Cl_6	3·26 ± 0·04
Cl_2–1 Mo_1	2·240 ± 0·033	–1 Cl_4	3·43 ± 0·03	–1 Cl_7	3·42 ± 0·03
–1 Cl_1	3·40 ± 0·03	–1 Cl_5	3·51 ± 0·04*	–1 Cl_8	3·71 ± 0·04*
–1 Cl_2	3·35 ± 0·05	–1 Cl_5	3·80 ± 0·04*	–1 Cl_9	3·70 ± 0·04*
–1 Cl_3	3·33 ± 0·04	–1 Cl_6	3·72 ± 0·04*	–1 Cl_9	3·32 ± 0·04
–1 Cl_3	3·66 ± 0·04*	–1 Cl_6	3·32 ± 0·04	–1 Cl_9	3·67 ± 0·04*
–1 Cl_3	3·25 ± 0·04	–1 Cl_6	3·80 ± 0·04*		
–1 Cl_5	3·73 ± 0·04*	–1 Cl_8	3·32 ± 0·04	Cl_9–1 Mo_2	2·258 ± 0·027
–1 Cl_5	3·57 ± 0·04*	–1 Cl_8	3·66 ± 0·04*	–1 Cl_2	3·80 ± 0·04*
–1 Cl_6	3·71 ± 0·04*	–1 Cl_9	3·27 ± 0·04	–1 Cl_4	3·22 ± 0·04
–1 Cl_8	3·73 ± 0·04*			–1 Cl_5	3·27 ± 0·04
–1 Cl_8	3·73 ± 0·04*	Cl_6–1 Mo_2	2·234 ± 0·027	–1 Cl_6	3·78 ± 0·03*
–1 Cl_8	3·61 ± 0·04*	–1 Cl_1	3·68 ± 0·04*	–1 Cl_7	3·67 ± 0·04*
–1 Cl_9	3·80 ± 0·04*	–1 Cl_2	3·71 ± 0·04*	–1 Cl_7	3·27 ± 0·04
		–1 Cl_3	3·65 ± 0·03*	–1 Cl_7	3·62 ± 0·04*
Cl_3–1 Mo_1	2·257 ± 0·023	–1 Cl_4	3·69 ± 0·04*	–1 Cl_8	3·70 ± 0·04*
–1 Cl_1	3·24 ± 0·04	–1 Cl_4	3·24 ± 0·04	–1 Cl_8	3·32 ± 0·04
–1 Cl_1	3·70 ± 0·04*	–1 Cl_5	3·72 ± 0·04*	–1 Cl_8	3·67 ± 0·04*
–1 Cl_1	3·26 ± 0·04	–1 Cl_5	3·32 ± 0·04	–1 Cl_9	3·77 ± 0·04*
–1 Cl_2	3·33 ± 0·04	–1 Cl_5	3·80 ± 0·04*	–1 Cl_9	3·33 ± 0·04

* Intermolecular distance.

shows the average Mo_2Cl_{10} molecule, calculated on the assumption that all Mo_2Cl_{10} molecules are identical and have $2/m$ symmetry. The bond lengths in Fig. 2 were obtained by weighting the distances given in Table 3 according to the inverse squares of their standard deviations and averaging. The low probable errors in Fig. 2 reflect the small deviations of the individual distances of Table 3 from their weighted means.

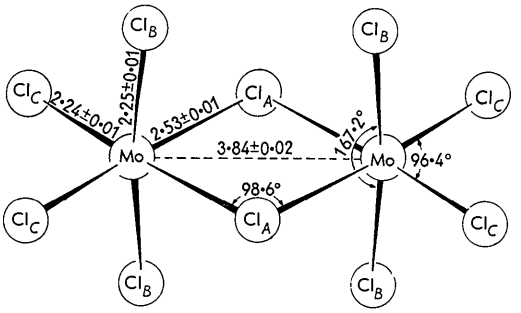

Fig. 2. Configuration of the Mo_2Cl_{10} molecule.

The Mo_2Cl_6 group formed by the two Mo atoms, the two Cl_A bridge atoms, and the four Cl_C atoms is planar to within the precission of the parameters. The four Mo–Cl_B bonds of molybdenum pentachloride are not perpendicular to the plane of the Mo_2Cl_6, but are bent toward each other to form an angle of about 84° with a line connecting the two molybdenum atoms. The bending of these bonds may be attributed to the steric effect of the chlorines in the planar Mo_2Cl_6 group, Cl_B becoming nearly equidistant from Cl_A and Cl_C; this distance (3·3 Å) is less than the normal van der Waals distance (3·6 Å). The molecular symmetry of Mo_2Cl_{10} is D_{2h}–mmm.

Table 4. *Comparison of the molecular structure of $MoCl_5$ in the gas and solid phases*

	Gas*	Solid
Molecular formula	$MoCl_5$	Mo_2Cl_{10}
Molecular configuration	Trigonal bipyramid	Two octahedra sharing an edge
Mo–Cl distances	2·27 ± 0·02 Å	2·24 ± 0·01 Å 2·53 ± 0·01 Å (bridge bond)
Cl–Cl distances (intramolecular)	3·21 Å	3·21 to 3·43 Å

* Ewens & Lister (1938) data.

Table 4 compares the configuration and size of molybdenum pentachloride in the gaseous and solid state.

$TaCl_5$ is also of the $NbCl_5$ type (Zalkin & Sands, 1958). Data on $NbBr_5$ indicate a similar structure, but with a modified packing of the molecules into a smaller orthorhombic unit cell (Rolsten, 1958). The planar Nb_2Cl_6 dimer is present in $NbOCl_3$ (Sands, Zalkin & Elson, 1959).

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