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The Crystal and Molecular Structure of μ-Chloro-(dichloromethyltin)-2,2′-bipyridyltricarbonylmolybdenum, $(C_{10}H_8N_2)(CO)_3ClMoSnCH_3Cl_2$

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The crystal structure determination of bipy$(CO)_3ClMoSnCH_3Cl_2$ (bipy = 2,2′-bipyridyl) by single-crystal X-ray techniques is described. The discrepancy index for 1924 unique observations is $R_1 = 5.6\%$. Data were collected with a PAILRED fully automated diffractometer. The compound crystallizes in the monoclinic space group C_{2h}^5-$P2_1/c$ with $a = 6.73 \pm 0.01$ Å, $b = 11.14 \pm 0.01$ Å, $c = 24.46 \pm 0.03$ Å, and $\beta = 90.5 \pm 0.2°$. The monomeric molecule contains a molybdenum–tin bond of length 2.753 ± 0.003 Å, bridged by a chlorine atom. Thus the molybdenum atom is heptacoordinate and the tin is pentacoordinate. The stereochemistry around the molybdenum is that of a capped octahedron with the tin atom lying above an octahedral face formed by the bridging chlorine atom and two carbonyl groups. The bipyridyl group exhibits a small but significant deviation from planarity.

Introduction

This crystal structure determination of μ-chloro-(dichloromethyltin)-2,2′-bipyridyltricarbonylmolybdenum, bipy$(CO)_3ClMoSnCH_3Cl_2$, is the first of a series of structural investigations of organometallic compounds with metal–metal bonds. A number of related compounds have been prepared by reaction of molybdenum- and tungsten-substituted carbonyls with germanium and tin tetrahalides or organometallic halides.[1]

The unknown coordination geometry of these compounds together with present interest in the nature of seven-coordination[2] make them interesting problems for X-ray structure analysis. Further, it was hoped that structural knowledge would help elucidate the nature of the geometric isomers invoked by Kummer and Graham to account for infrared spectral evidence. The compound bipy$(CO)_3ClMoSnCH_3Cl_2$ was chosen because of the ready availability of crystals and their stability. It is a suitable choice, for, in addition to the above points, there is the interest provided by the molybdenum–tin bond length and by the coordination of the 2,2′-bipyridyl ligand for which there is little structural information.

Experimental Section

Crystals of bipy$(CO)_3ClMoSnCH_3Cl_2$ were prepared following the method described by Kummer and Graham.[1] They took the form of small orange needles with the needle axis corresponding to the short crystallographic axis. Preliminary Weissenberg photographs taken with Cu Kα radiation ($0kl$–$2kl$) and precession photographs taken with Mo Kα radiation ($h0l$, $hk0$) indicated a monoclinic space group. The systematic absences were consistent with space group C_{2h}^5-$P2_1/c$. The reciprocal cell parameters b^* and c^* were obtained from the diffractometer and refined by least squares.[3] Molybdenum Kα radiation (λ 0.71069 Å) was used. The parameters a^* and β^* were taken from a Mo Kα precession photograph of the ($h0l$) reciprocal lattice level. Final values were $a = 6.73 \pm 0.01$ Å, $b = 11.14 \pm 0.01$ Å, $c = 24.26 \pm 0.03$ Å, and $\beta = 90.5 \pm 0.2°$. The calculated density for four molecules per unit cell is 2.09 g cm^{-3}. An experimental density of 2.0 g cm^{-3} was measured using a pycnometer with water as the displacing liquid. The linear absorption coefficient is 28 cm^{-1} for Mo Kα radiation. The crystal used for data collection was a needle of length 0.3 mm and an average cross-sectional radius 0.053 mm. It was mounted on a thin glass fiber and showed no signs of decomposition during data collection.

Reciprocal lattice levels ($0kl$–$6kl$) were scanned using a PAILRED fully automated diffractometer. Molybdenum Kα radiation was employed, monochromatized from the [111] face of a silicon crystal. The pulse-height analyzer was centered on the Kα peak to include approximately 90% of the transmitted beam. A counter aperture of 1.5° was used. The equiinclination technique and a moving-crystal stationary-counter (ω-scan) scanning procedure were employed with a scan speed of 1°/min. Background counts were determined for 0.4 min immediately before and after each scan through a reciprocal lattice position, with the crystal stationary at either side of the scan range. The angular range of the scan varied from 0.8° for the zero layer to 4.0° for the sixth.

After each layer, reflections for which the two background readings differed significantly were repositioned centrally in the scan range and remeasured. Despite considerable care taken in crystal alignment, such variations were found to be associated with a small number of low θ reflections on each layer. They are presumed to be symptomatic of residual alignment errors magnified in their effect by the rapid change of ω with reciprocal lattice positions at low θ angles. A number of zero-layer reflections were checked after each layer. Their intensities showed no significant variation throughout the data collection, indicating both crystal and electronic stability. The criterion of significance of an observation was the ratio $\Delta I/I$, where I is given by $T - t(B_1 + B_2)$, and ΔI by $(T + t^2B)^{1/2}$. Here t is the ratio of the time of scan to the total background time, $B = B_1 + B_2$ is the total background count, and T is the scan count. Rejection of data for which $\Delta I/I > 0.38$ gave 1924 unique intensity maxima in the range $\sin\theta < 0.51$. The data are limited to $\sin\theta < 0.32$ in the a direction; the resolution parallel and perpendicular to a is thus 1.1 and 0.7 Å, respectively. This asymmetry in the data is discussed later.

Structure Refinement.—Lorentz and polarization corrections for Weissenberg geometry were applied to all data, but absorption corrections were not, in view of the size of the crystal ($\mu R_{av} = 0.15$) and the small variation in radius of the needle-shaped crystal. The structure was solved by Patterson and Fourier

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[1] R. Kummer and W. A. G. Graham, *Inorg. Chem.*, **7**, 1208 (1968).

[2] E. L. Muetterties and C. M. Wright, *Quart. Rev.* (London), **21**, 109 (1967).

[3] M. Elder and D. Hall, *Inorg. Chem.*, **8**, 1273 (1969).

methods.[4,5] Positional parameters and individual isotropic temperature factors for all nonhydrogen atoms, together with an over-all scale factor, were refined by full-matrix least-squares methods. After six cycles, refinement converged with the discrepancy factors $R_1 = \Sigma||F_o| - |F_c||/\Sigma|F_o|$ and $R_2 = [\Sigma w(|F_o| - |F_c|)^2/\Sigma w|F_o|^2]^{1/2}$ of 0.077 and 0.094, respectively. The function $\Sigma w[|F_o| - |F_c|]^2$ was minimized. The weights determined from counting statistics were used at first but these were subsequently discarded in favor of a scheme of the form

$$w = a^2/(a^2 + (|F_o| - b)^2)$$

where $a = b = 59$ on the absolute scale were chosen to minimize the variation of $\Sigma w(|F_o| - |F_c|)^2$ calculated for increasing ranges of $|F_o|$. A similar weighting scheme analysis indicated that the weights based on counting statistics were assigning too much weight to the more intense reflections. This was also observed in the reduction of the $0kl$ reflections for which the PAILRED produced two observations of each. Using standard error estimates based on the values of ΔI previously defined it was found that, although there were no major disagreements, the variations between two measurements of both the very weak and the strong reflections were greater than those that would be expected if the error estimates were true standard errors. This same observation was made during refinement of the topaz structure used in the initial trials of the PAILRED system.[6]

Atomic scattering factors for neutral tin and molybdenum,[7a] chlorine, oxygen, nitrogen, and carbon[7b] were used with the real dispersion corrections[7c] only applied to the scattering factor curves for tin, molybdenum, and chlorine.

A three-dimensional difference Fourier map computed at this point revealed some residual electron density around the heavy-atom positions. The refinement was continued with individual anisotropic temperature factors for the chlorine and metal atoms. The methyl carbon atom and the atoms of the three carbonyl groups were now given anisotropic temperature factors as well. This step was suggested by the relatively high isotropic temperature factors of these atoms compared with those of the atoms of the bipyridyl group. The two resulting cumulative decreases in both R_1 and R_2 were significant at better than $\alpha = 0.01$ judged by Hamilton's criteria.[8a] The final agreement factors were $R_1 = 0.056$ and $R_2 = 0.072$. The final positional parameters and temperature factors are listed in Table I. The estimated standard deviations of the parameters are derived from the inverse least-squares matrix. The orientations of the vibrational ellipsoids, indicated in Figure 1, confirm a physically reasonable pattern of vibration amplitudes. The observed and calculated structure factors are given in Table II. No correction for extinction was applied since the data of this table provide no indication that one is necessary.

The effect of the asymmetry in the data, caused by the limited data in the a direction, can be seen in the standard errors of the positional parameters. The estimated standard error of the x coordinates (in ångströms) tends to be about 10% greater than the corresponding errors in y and z. The associated effect upon the errors in the bond lengths is small enough to be unimportant: the C_{11}–O_1 bond has a considerably greater component in the a direction than the other two C–O bonds; it has an uncertainty of 0.019 Å compared with 0.016 and 0.018 Å. There is a similar effect associated with the standard errors in the anisotropic thermal parameters. The asymmetry must clearly increase the uncertainties associated with the thermal ellipsoids but there is no indication of any effect systematic in one direction.

TABLE I

POSITIONAL AND THERMAL PARAMETERS FOR $bipy(CO)_3ClMoSnCl_2CH_3$

Atom	x	y	z	B, Å^2
Sn	0.0320 (2)[a]	0.18243 (8)	0.38842 (4)	
Mo	0.0748 (2)	0.41013 (10)	0.34643 (5)	
Cl_1	−0.1244 (6)	0.3922 (3)	0.4346 (2)	
Cl_2	0.1961 (8)	0.0289 (4)	0.3344 (2)	
Cl_3	0.2203 (8)	0.1498 (5)	0.4688 (2)	
N_1	0.214 (2)	0.557 (1)	0.3956 (5)	2.5 (2)
N_2	−0.117 (2)	0.572 (1)	0.3373 (4)	2.3 (2)
C_1	0.380 (3)	0.547 (2)	0.4232 (6)	3.5 (3)
C_2	−0.467 (3)	0.639 (2)	0.4543 (7)	4.0 (3)
C_3	0.365 (3)	0.746 (3)	0.4556 (6)	3.6 (3)
C_4	0.197 (3)	0.760 (2)	0.4282 (7)	3.7 (3)
C_5	0.118 (2)	0.663 (2)	0.3989 (6)	2.7 (3)
C_6	−0.071 (2)	0.671 (1)	0.3675 (6)	2.6 (2)
C_7	−0.190 (3)	0.772 (2)	0.3677 (6)	3.6 (3)
C_8	−0.358 (3)	0.773 (2)	0.3359 (7)	3.9 (3)
C_9	−0.401 (3)	0.674 (2)	0.3043 (6)	3.5 (3)
C_{10}	−0.276 (3)	0.574 (1)	0.3064 (6)	3.4 (3)
C_{11}	0.330 (2)	0.327 (1)	0.3461 (6)	
O_1	0.487 (2)	0.289 (1)	0.3439 (6)	
C_{12}	0.203 (2)	0.458 (1)	0.2758 (6)	
O_2	0.219 (2)	0.485 (1)	0.2366 (5)	
C_{13}	−0.075 (2)	0.311 (1)	0.2922 (6)	
O_3	−0.153 (2)	0.263 (1)	0,2577 (4)	
C_{14}	−0.239 (3)	0.094 (2)	0.4068 (8)	

Atom	$10^4\beta_{11}$[b]	$10^4\beta_{22}$	$10^4\beta_{33}$	$10^4\beta_{12}$	$10^4\beta_{13}$	$10^4\beta_{23}$
Sn	167 (3)	39.6 (7)	10.6 (2)	2 (1)	1.1 (5)	1.9 (3)
Mo	124 (3)	35.7 (9)	9.0 (2)	0 (1)	0.7 (5)	0.1 (3)
Cl_1	237 (11)	58 (3)	12.7 (6)	2 (5)	22 (2)	−2 (1)
Cl_2	339 (15)	56 (3)	21.3 (9)	34 (6)	21 (3)	−7 (1)
Cl_3	250 (13)	127 (5)	16.3 (8)	15 (6)	−24 (3)	10 (2)
C_{11}	127 (43)	53 (11)	16 (3)	−10 (19)	−9 (7)	−1 (5)
O_1	135 (34)	86 (12)	30 (3)	5 (16)	−6 (7)	−2 (5)
C_{12}	202 (41)	41 (10)	10 (2)	−5 (16)	18 (8)	−9 (4)
O_2	276 (37)	89 (12)	15 (2)	−47 (17)	23 (7)	2 (4)
C_{13}	152 (39)	56 (12)	16 (3)	−4 (19)	6 (8)	1 (5)
O_3	308 (38)	82 (11)	14 (2)	−48 (17)	−20 (7)	−10 (4)
C_{14}	136 (42)	62 (14)	28 (4)	−21 (20)	6 (9)	−7 (6)

[a] Parenthetical numbers, here and in following tables, are estimated standard deviations in the least significant figures. [b] The anisotropic ellipsoid has the form: $\exp[-(\beta_{11}h^2 + \beta_{22}k^2 + \beta_{33}l^2 + 2\beta_{12}hk + 2\beta_{13}hl + 2\beta_{23}kl)]$.

Discussion

The structure consists of isolated molecular units of $bipy(CO)_3ClMoSnCH_3Cl_2$ lying on a set of four general positions per unit cell. The molecular structure is shown in the stereopair of Figure 1. Molecular packing is indicated in Figure 2. There are no intermolecular contacts less than the sum of appropriate van der Waals radii. Intramolecular distances and angles are given in Tables III and IV.

The tin atom is five-coordinate and the molybdenum is seven-coordinate, with one chlorine atom occupying a bridging position between the two metal atoms. The molybdenum–tin bond is short at 2.753 ± 0.003 Å; the sum of the covalent radii for the two atoms gives 3.0 Å.[9-12] An Mo–Sn distance of 2.891 ± 0.005 Å has been observed previously.[13] While it is probably unreasonable to put too much faith in a figure based upon a covalent radius estimate of a transition metal in a low oxidation state, the magnitude of the decrease and the comparison with O'Connor and Corey's value does suggest a bond order greater than single for this bond. In

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(4) Programs for an IBM 360/67 by F. R. Ahmed, *et al.*, Division of Pure Physics, National Research Council, Ottawa, were used in this work.

(5) In addition to various local programs, a modification of the Busing and Levy ORFLS least-squares program was used, together with ORFFE and Johnson's ORTEP thermal ellipsoid plotting program.

(6) J. Ladell, *Norelco Reptr.*, **12**, 34 (1965).

(7) "International Tables for X-Ray Crystallography," Vol III, The Kynoch Press, Birmingham, England, 1962: (a) p 211; (b) p 202; (c) p 216.

(8) W. C. Hamilton, "Statistics in Physical Science," Ronald Press, New York, N. Y., 1962: (a) p 160; (b) p 174.

(9) M. J. Bennett and R. Mason, *Proc. Chem. Soc.*, 273 (1963).

(10) R. J. Doedens and L. F. Dahl, *J. Am. Chem. Soc.*, **87**, 2576 (1965).

(11) F. C. Wilson and D. P. Shoemaker, *J. Chem. Phys.*, **27**, 809 (1957), suggest 1.61 Å for molybdenum.

(12) D. H. Olson and R. E. Rundle, *Inorg. Chem.*, **2**, 1310 (1963), give 1.39 Å for tin.

(13) J. E. O'Connor and E. R. Corey, *J. Am. Chem. Soc.*, **89**, 3930 (1967).

Table II

Observed and Calculated Structure Factors for bipy(CO)$_3$ClMoSnCH$_3$Cl$_2$ (×10)

K	L	FO	FC
	H = 0		
0	4	3262	-3378
0	6	317	364
0	8	650	647
0	10	1058	-1066
0	12	274	278
0	14	714	-693
0	16	854	-843
0	18	1025	1028
0	20	541	587
0	22	1289	-1356
0	24	379	-440
0	26	763	838
0	28	218	200
1	5	383	267
1	6	1177	-1084
1	7	125	47
1	9	1338	-1295
1	10	847	829
1	11	395	-372
1	12	324	295
1	13	1328	1320
1	14	408	400
1	15	621	555
1	16	717	-720
1	17	712	-712
1	18	287	-310
1	19	393	434
1	20	884	935
1	21	276	261
1	22	336	352
1	23	377	-358
1	24	817	-885
1	26	373	-415
1	28	293	348
1	29	243	-282
1	32	298	-292
1	35	322	-164
2	0	832	-819
2	1	199	-170
2	2	822	-800
2	3	1432	-1406
2	4	565	537
2	5	668	-613
2	6	834	840
2	7	1308	1245
2	8	995	-960
2	9	435	454
2	10	871	-853
2	11	2649	-2609
2	12	1175	1138
2	13	939	-917
2	14	861	937
2	15	1522	1514
2	16	595	-546
2	17	255	234
2	18	213	-181
2	19	335	-335
2	21	323	312
2	22	213	221
2	23	222	180
2	27	219	184
2	28	283	-285
2	31	365	-356
2	32	271	246
2	33	297	268
2	35	327	177
3	1	891	791
3	2	1337	1254
3	3	566	561
3	4	179	-224
3	5	1664	-1539
3	6	185	159
3	7	164	155
3	8	1639	1531
3	9	1799	1753
3	10	749	-724
3	11	254	221
3	12	1405	-1377
3	13	1458	-1473
3	14	608	614
3	15	517	-526
3	16	518	540
3	17	445	482
3	18	475	-484
3	19	204	205
3	21	438	-458
3	23	369	-367
3	24	200	-215
3	27	502	534
3	30	294	-306
3	31	240	-272
3	33	220	-194
4	0	794	-772
4	1	1014	1019
4	2	770	-897
4	3	1460	1464
4	4	722	706
4	5	345	-383
4	6	379	-360
4	7	1165	-1093
4	8	213	221
4	9	800	780
4	10	215	210
4	11	958	930
4	12	300	276
4	14	223	-247
4	15	406	-379
4	16	399	342
4	17	596	-595
4	19	243	255
4	20	287	-320
4	21	723	750
4	22	369	375
4	24	291	299
4	25	820	-851
4	26	254	-266
4	29	348	373
5	1	1849	-1843
5	2	646	-678
5	3	1527	1536
5	4	1082	-1065
5	5	950	920
5	6	196	165
5	7	872	-856
5	8	490	471
5	9	164	159
5	10	504	-508
5	12	711	-739
5	13	195	-214
5	14	460	435
5	15	529	534
5	16	269	303
5	17	385	-383
5	18	201	-184
5	19	648	-694
5	20	216	-257
5	21	339	405
5	23	960	958
5	24	293	313
5	25	271	-253
5	26	205	215
5	27	444	-471
5	29	257	167
5	30	216	-171
6	1	605	-599
6	2	366	376
6	3	1048	-1024
6	5	676	654
6	6	847	-826
6	7	412	382
6	8	854	858
6	9	353	341
6	10	1436	1422
6	12	1046	-1029
6	13	197	-206
6	14	1159	-1187
6	15	311	302
6	16	430	460
6	17	235	269
6	18	355	394
6	19	395	-420
6	20	404	-387
6	21	528	-552
6	25	323	325
6	32	324	-299
7	1	426	-426
7	2	487	467
7	3	609	636
7	5	577	-550
7	6	540	-544
7	7	484	-502
7	8	622	-615
7	9	618	600
7	10	711	762
7	11	241	262
7	12	1156	1174
7	13	559	-598
7	14	404	-424
7	15	330	312
7	16	835	-878
7	17	410	441
7	18	211	189
7	19	227	-188
7	20	417	459
7	22	306	-293
7	23	263	274
7	30	283	281
8	0	1033	-1055
8	1	1161	-1194
8	2	209	176
8	3	835	-838
8	4	1218	1222
8	5	853	847
8	7	407	-429
8	8	744	-735
8	9	573	-559
8	10	831	-848
8	11	608	595
8	12	331	337
8	14	509	533
8	15	323	-305
8	17	315	328
8	18	513	-529
8	21	477	-504
8	22	424	454
8	23	261	287
8	25	338	335
8	26	262	-235
9	1	516	533
9	2	1124	-1169
9	5	356	-395
9	6	820	840
9	7	211	276
9	10	269	-252
9	17	289	261
9	18	401	395
9	19	246	259
9	20	421	-479
9	21	240	-246
9	22	384	-376
9	23	329	-392
9	24	406	437
9	26	232	186
9	27	244	232
9	28	345	-358
10	0	613	648
10	1	312	314
10	3	550	536
10	4	661	-700
10	5	219	239
10	7	876	-935
10	8	211	187
10	9	232	-207
10	10	199	-187
10	11	774	837
10	12	252	154
10	15	664	-702
10	18	232	244
10	19	343	380
10	20	361	324
10	22	371	-401
10	26	254	239
11	2	425	398
11	3	310	-316
11	5	659	728
11	7	412	378
11	9	1018	-1081
11	10	266	-256
11	11	271	-219
11	13	874	852
11	17	563	-675
12	0	603	628
12	3	253	-262
12	4	467	-476
12	5	198	222
12	6	231	240
12	7	553	582
12	8	311	277
12	11	501	-552
12	15	400	446
12	19	234	-249
12	20	242	252
13	2	701	779
13	3	444	-461
13	6	439	-456
13	7	320	357
13	15	257	-289
13	16	239	-195
13	19	284	303
13	20	335	282
14	0	545	-589
14	1	327	317
14	3	257	198
14	4	351	392
14	5	310	-217
14	10	226	-195
14	11	220	-139
14	18	277	-131
15	8	370	452
15	10	248	-227
15	12	324	-230
16	6	355	-150
	H = 1		
0	4	2236	-2368
0	-4	2477	-2578
0	6	578	-617
0	8	2520	2570
0	-8	323	287
0	10	351	326
0	12	259	233
0	-12	339	-359
0	14	267	232
0	-14	1055	-1072
0	16	611	-643
0	18	267	255
0	-18	1695	1730
0	20	687	734
0	22	1164	-1186
0	-22	1019	-1033
0	24	778	-802
0	-24	276	-181
0	26	326	340
0	-26	760	759
0	28	484	540
1	4	1061	1087
1	-4	342	277
1	5	411	399
1	-5	596	549
1	6	1898	-1973
1	-6	1227	-1075
1	7	545	-546
1	-7	1763	1593
1	8	835	-877
1	9	838	-830
1	-9	1327	-1315
1	10	916	875
1	-10	462	-382
1	11	655	-677
1	-11	249	-271
1	12	774	772
1	-12	307	-268
1	13	1185	1237
1	-13	1332	1306
1	-14	478	433
1	15	512	512
1	-15	292	-323
1	-16	1239	1247
1	17	758	-787
1	-17	373	-362
1	18	239	-242
1	-19	574	550
1	20	764	798
1	-20	803	-817
1	21	317	313
1	22	504	539
1	-23	497	-495
1	24	642	-792
1	-24	732	729
1	26	445	-461
1	28	394	434
1	-29	334	-322
1	-31	322	314
1	32	307	-245
2	0	214	-228
2	1	798	839
2	-1	553	650
2	-2	799	-814
2	3	397	424
2	-3	1808	1846
2	4	728	-792
2	-4	1366	1313
2	-5	818	-734
2	6	1262	1230
2	-6	2150	1988
2	7	1059	1041
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view of the established π-acceptor properties of the –SnCl$_3$ group[14,15] (and hence presumably –SnCH$_3$Cl$_2$), it seems probable that the bond does possess some double-bond character. If this were so, then it is at least theoretically possible that it would cause a lengthening of the molybdenum to carbonyl bonds.[16] In fact, the experimental evidence for such bond lengthening is

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(14) W. Jetz, P. B. Simons, J. A. J. Thompson, and W. A. G. Graham, *Inorg. Chem.*, **5**, 2217 (1966).

(15) G. W. Parshall, *J. Am. Chem. Soc.*, **86**, 5367 (1964); **88**, 704 (1966).

(16) See, for example, a similar discussion of Co(SiCl$_3$)(CO)$_4$: W. T. Robinson and J. A. Ibers, *Inorg. Chem.*, **6**, 1208 (1967).

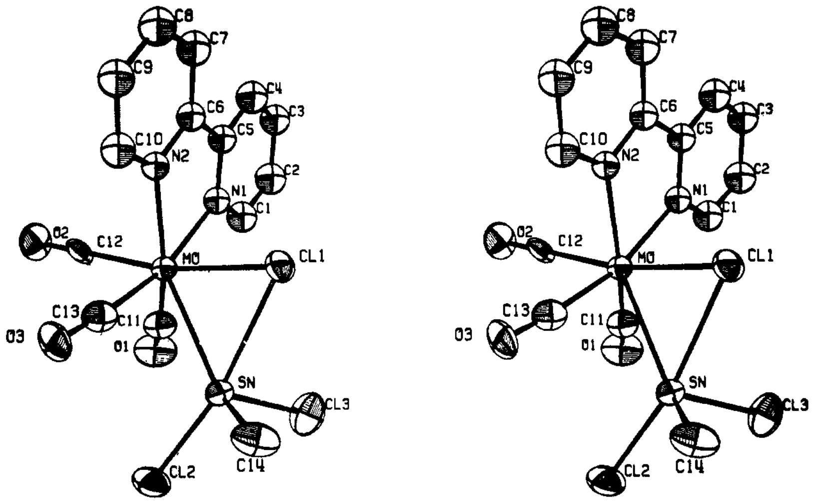

Figure 1.—Stereogram of a molecule of μ-chloro-(dichloromethyltin)-2,2′-bipyridyltricarbonylmolybdenum. Thermal ellipsoids are scaled to enclose 50% probability.

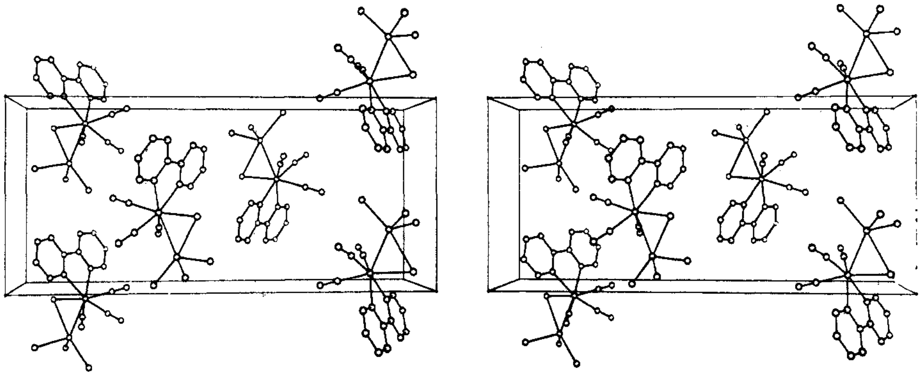

Figure 2.—Packing diagram for μ-chloro-(dichloromethyltin)-2,2′-bipyridyltricarbonylmolybdenum. The unit cell is outlined and the direction of view is along the reciprocal a^* axis. The c axis is horizontal.

TABLE III

SELECTED DISTANCES (Å)

		Intramolecular Bonded Distances			
Sn–Mo	2.753 (3)	C_{11}–O_1	1.15 (2)	C_4–C_5	1.40 (2)
Sn–Cl_1	2.805 (4)	C_{12}–O_2	1.14 (2)	C_5–N_1	1.34 (2)
Mo–Cl_1	2.557 (6)	C_{13}–O_3	1.13 (2)	C_5–C_6	1.48 (2)
Sn–Cl_2	2.433 (5)	Mo–N_1	2.23 (1)	N_2–C_6	1.36 (2)
Sn–Cl_3	2.358 (6)	Mo–N_2	2.23 (1)	C_6–C_7	1.38 (2)
Sn–C_{14}	2.12 (2)	N_1–C_1	1.31 (2)	C_7–C_8	1.37 (2)
Mo–C_{11}	1.95 (2)	C_1–C_2	1.40 (2)	C_8–C_9	1.37 (2)
Mo–C_{12}	2.01 (1)	C_2–C_3	1.38 (2)	C_9–C_{10}	1.40 (2)
Mo–C_{13}	1.99 (2)	C_3–C_4	1.32 (2)	C_{10}–N_2	1.31 (2)
		Intramolecular Nonbonded Distances			
Sn–C_{11}	2.78 (2)	Cl_1–C_{11}	3.83 (2)	N_1–C_{11}	2.94 (2)
Sn–C_{13}	2.84 (2)	Cl_1–C_{13}	3.62 (2)	N_1–C_{12}	3.13 (2)
Cl_1–Cl_3	3.65 (1)	C_{11}–C_{12}	2.40 (2)	N_2–C_{12}	2.93 (2)
Cl_2–Cl_3	3.56 (1)	C_{11}–C_{13}	3.02 (2)	N_2–C_{13}	3.12 (2)
Cl_1–C_{14}	3.47 (2)	C_{12}–C_{13}	2.52 (2)	N_1–N_2	2.64 (2)
Cl_2–C_{14}	3.51 (2)	N_1–Cl_1	3.08 (1)		
Cl_3–C_{14}	3.48 (2)	N_2–Cl_1	3.11 (1)		

inconclusive. The carbonyls are in all respects normal (C–O_{av} = 1.14 Å, Mo–C–O_{av} = 174.7°) and the molybdenum–carbon distances which average 1.98 Å lie at the end of the range of previously determined values: 1.93 Å in $(C_5H_5)_2Mo_2H[P(CH_3)_2](CO)_4$[10] to 1.99 Å in Mo$(C_5H_5)(CO)_3Cl$[17] with a number of values in between. Thus any lengthening of the bonds under discussion is of doubtful significance and would in any case be difficult to dissociate from effects arising from the differences in the coordination geometry of the molybdenum atoms in the various structures.

The tin to bridging chlorine distance is 2.805 ± 0.004 Å, considerably shorter than the sum of the van der

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[17] S. Chaiwasie and R. H. Fenn, *Acta Cryst.*, **B24**, 525 (1968).

TABLE IV

SELECTED ANGLES (DEG)

Mo–Sn–Cl_3	113.3 (1)	C_5–N_1–C_1	117.0 (1.3)
Mo–Sn–C_{14}	126.8 (5)	N_1–C_1–C_2	124.7 (1.6)
Cl_3–Sn–C_{14}	102.0 (6)	C_1–C_2–C_3	116.1 (1.6)
Cl_1–Sn–Mo	54.8 (1)	C_2–C_3–C_4	121.0 (1.6)
Cl_1–Sn–Cl_3	89.6 (2)	C_3–C_4–C_5	119.1 (1.6)
Cl_1–Sn–C_{14}	88.6 (5)	C_4–C_5–N_1	122.1 (1.4)
Cl_2–Sn–Mo	113.3 (2)	C_4–C_5–C_6	122.2 (1.3)
Cl_2–Sn–Cl_3	95.8 (2)	C_{10}–N_2–C_6	118.7 (1.2)
Cl_2–Sn–C_{14}	100.5 (5)	N_2–C_6–C_7	122.2 (1.3)
Cl_1–Sn–Cl_2	168.1 (1)	N_2–C_6–C_5	122.9 (1.3)
Mo–Cl_1–Sn	61.6 (1)	C_6–C_7–C_8	118.4 (1.5)
Sn–Mo–Cl_1	63.6 (1)	C_7–C_8–C_9	119.3 (1.6)
Sn–Mo–C_{11}	70.2 (4)	C_8–C_9–C_{10}	119.4 (1.5)
Sn–Mo–C_{13}	71.6 (4)	C_9–C_{10}–N_2	122.0 (1.6)
Cl_1–Mo–C_{11}	115.8 (5)	Mo–N_1–C_5	118.4 (1.0)
Cl_1–Mo–C_{13}	104.6 (5)	Mo–N_2–C_6	118.1 (0.9)
C_{11}–Mo–C_{13}	100.2 (6)	N_1–C_5–C_6	115.7 (1.3)
N_1–Mo–C_{12}	95.2 (5)	N_2–C_6–C_5	114.9 (1.2)
N_1–Mo–N_2	72.6 (4)	Mo–N_1–C_1	124.6 (1.0)
N_2–Mo–C_{12}	87.3 (5)	Mo–N_2–C_{10}	123.2 (1.1)

Waals radii, which is 4.0 Å if 2.2 Å is assumed for the tin atom.[18] There can be no doubt that the chlorine atom is coordinated at least weakly to the tin atom and thus bridges the metal–metal bond. Comparison with the other tin–chlorine distances, however, indicates that this bond is weaker than a tin–chlorine single bond. The situation is perhaps comparable with the Sn · · · Br contact in the substituted butadienyldimethyltin bromide[18] although there the tin to bromine distance is only 0.4 Å less than the sum of the van der Waals radii. The angle of 61.6° subtended at the chlorine by the molybdenum–tin bond agrees with the values for bridging chlorines in, for example, the trinuclear rhenium halide series (Re–Cl_μ–Re = 62°).[19] The molybdenum–chlorine length, 2.557 ± 0.006 Å, is close to the molybdenum to bridging chlorine distance of 2.52 Å in Mo_2Cl_{10}.[20]

The tin coordination can be described as distorted trigonal bipyramidal, although such descriptions for low-coordination polyhedra are of doubtful significance. The atoms Cl_1, Sn, and Cl_2 form the axis of the bipyramid (the angle Cl_1–Sn–Cl_2 is 168°) with the molybdenum, methyl carbon, and the third chlorine forming the equatorial plane. The major distortion is the displacement of the molybdenum from this plane toward the bridging chlorine such that the Mo–Cl_1 bond subtends 54.8° instead of 90° at the tin atom. There is a significant difference in the tin–chlorine distances for the two singly coordinated chlorine atoms, the difference between 2.433 and 2.358 Å being of the order of 14 times the estimated standard deviation in a tin–chlorine bond. Neither figure is unusual for Sn–Cl lengths.[21–24] Perhaps the difference can be explained by the different geometries of the two chlorine atoms in terms of trigonal-bipyramidal coordination. The longer tin–chlorine bond is one of the two axial bonds using a tin p orbital, while the short bond is in an equatorial position, utilizing an sp^2 hybrid.

The molybdenum atom exhibits a capped octahedral structure with the tin atom occupying the seventh position above an octahedral face defined by the bridging chlorine and two carbonyl groups. The angles subtended at the molybdenum atom by the three atoms determining the capped face are in the range 100–115°; between the atoms of the opposite face the angles are 72–95°. Thus there is distortion of octahedral symmetry in order to accommodate the tin atom. The Mo–Sn bond makes angles in the range 64–72° with the three nearest bonds. The form of the molybdenum atom coordination is thus very similar to the capped octahedral stereochemistry observed for $Y(BZA)_3 \cdot H_2O$.[25]

The Bipyridyl Group.—The mean C–N distance is 1.33 Å, C–C is 1.38 Å, and there are no significant deviations from these figures for any individual bond. The C–C bond joining the two rings is 1.48 Å, all bond angles are within 5° of 120°, the value for regular sp^2 hybridization. These figures are in close agreement with those found in 2,2′-bipyridyl itself[26] and in two other complexes containing 2,2′-bipyridyl groups.[27,28]

The major differences appear to lie in the planarity of the ligand and the angle it subtends at the metal atom. There is a correlation between the nitrogen–metal–nitrogen angle and the metal–nitrogen distance. In the present compound the molybdenum–nitrogen distance is 2.23 Å and the subtended angle is 72.6°, in $(CH_3)_3$-bipy[$C(COCH_3)_2$]Pt[26] the values are 2.15 Å and 77°, and in $Cu(bipy)_2I^+$ [27] they are 2.02 Å and 82°. Best least-squares planes have been calculated with a program from the source given in ref 4, which uses the method described by Blow.[29] Table V lists the relevant results. For the atoms of the two pyridine rings considered separately, there are no deviations from the planes greater than one esd in the corresponding atom. The χ^2 values confirm that there are no significant deviations from planarity.[8b] This is clearly not the case for the plane calculated for all 12 atoms, for which the χ^2 value indicates that the deviations are highly significant. The angle between the two pyridine rings is 4.7° which may be compared with 2 and 10° for the copper bis(bipyridyl) complex. The deviations from coplanarity of the two rings reveal a twisting about the C_5–C_6 bond such that atoms C_2, C_3, and C_4 of one ring and N_2, C_9, and C_{10} of the other are on the same side of the plane through both rings. The molybdenum atom lies on the plane through both rings. The molybdenum atom lies on the plane through ring 2 but is 0.16 Å from the other plane.

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(18) F. P. Boer, J. J. Flynn, H. H. Freedman, S. V. McKinley, and V. R. Sandel, *J. Am. Chem. Soc.*, **89**, 5068 (1967).

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(20) D. E. Sands and A. Zalkin, *Acta Cryst.*, **12**, 723 (1959).

(21) For example: 2.31–2.34 Å in the $SnCl_{4-n}(CH_3)_n$ series[22,23] and 2.43 Å in $[(\pi\text{-}C_5H_5)Fe(CO)_2]_2SnCl_2$[24] where the bond is postulated to contain greater p character than an sp^3 hybrid.

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TABLE V

LEAST-SQUARES PLANES FOR THE 2,2′-BIPYRIDYL GROUP[a]

Plane[b]	Atoms	Equation	χ^2 values
1	$N_1C_1C_2C_3C_4C_5$	$0.5071X + 0.3126Y - 0.8032Z + 5.1542 = 0$	$\chi^2 = 2.2$ $[\chi^2_{3,0.50} = 2.4]$
2	$N_2C_6C_7C_8C_9C_{10}$	$0.5359X + 0.3749Y - 0.7565Z + 4.3055 = 0$	$\chi^2 = 2.1$
3	$N_1C_1C_2C_3C_4C_5$ $N_2C_6C_7C_8C_9C_{10}$	$0.5151X + 0.3434Y - 0.7858Z + 4.7524 = 0$	$\chi^2 = 93.8$ $\chi^2_{9,0.01} = 21.7$

Deviation from Plane 3

Atom	Dev, Å	Esd, Å	Atom	Dev, Å	Esd, Å
N_1	−0.031	0.011	N_2	0.008	0.010
C_1	−0.025	0.016	C_6	−0.038	0.014
C_2	0.026	0.017	C_7	−0.069	0.016
C_3	0.056	0.016	C_8	−0.036	0.017
C_4	0.046	0.017	C_9	0.051	0.016
C_5	−0.021	0.014	C_{10}	0.064	0.016
Mo	−0.120	0.002	(Not included in plane calculation)		

[a] The equations are given in ångström units. The coordinate system used refers to three orthogonal axes such that X lies along the a axis, Y along b, and Z along c^*. [b] Interplanar angle is 4.7° for planes 1 and 2.

We believe that the angle between the two rings is significant in view of the physically feasible nature of the distortions from coplanarity.

This structural determination reveals the coordination geometry of "isomer II" in the series of compounds, discussed by Kummer and Graham.[1] The unexpected stereochemistry of the tin atom suggests that the difference between the two structural isomers may be related to the tin coordination number. However, we leave further discussion of this point until the results of related structures are available.

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CONTRIBUTION FROM THE DEPARTMENT OF CHEMISTRY,
UNIVERSITY OF ALBERTA, EDMONTON, ALBERTA, CANADA

The Crystal and Molecular Structure of μ-Chloro-(dichloromethyltin)-2,5-dithiahexanetricarbonyltungsten, $(C_4H_{10}S_2)(CO)_3ClWSnCH_3Cl_2$

BY M. ELDER AND D. HALL

Received November 27, 1968

The crystal structure determination of $DTH(CO)_3ClWSnCH_3Cl_2$ (DTH = $CH_3SCH_2CH_2SCH_3$, 2,5-dithiahexane) is reported. The compound crystallizes in space group C_{2h}^5-$P2_1/c$ with four molecules per unit cell of dimensions $a = 7.458 \pm 0.003$ Å, $b = 15.044 \pm 0.009$ Å, $c = 16.228 \pm 0.012$ Å, and $\beta = 110.75 \pm 0.02°$. The observed and calculated densities are, respectively, 2.4 and 2.46 g cm^{-3}. The structure was refined by a full-matrix least-squares method to a conventional R factor of 7.7% using 1743 observations above background collected on a PAILRED diffractometer. The dithiahexane ligand occupies two of seven coordination positions around the tungsten atom which has capped octahedral symmetry. The tungsten–tin distance is 2.759 ± 0.003 Å. The tin atom is effectively five-coordinate since there is a long Sn–Cl bond of 2.958 ± 0.010 Å between the tin atom and the chlorine atom which is attached to the tungsten.

Introduction

The crystal structure determination of μ-chloro-(dichloromethyltin)dithiahexanetricarbonyltungsten, $DTH(CO)_3ClWSnCH_3Cl_2$ (DTH = $CH_3SCH_2CH_2SCH_3$, 2,5-dithiahexane) has been undertaken as part of a group of crystallographic studies of molybdenum- and tungsten-substituted carbonyls with germanium and tin tetrahalides and alkyl halides. The structure of a closely related compound, $bipy(CO)_3ClMoSnCH_3Cl_2$, has been recently reported[1] (bipy = 2,2′-bipyridyl). That structure revealed seven- and five-coordinate molybdenum and tin, respectively, with a chlorine atom bridging the short molybdenum–tin bond. The present compound was undertaken in order to determine the effect of replacing the molybdenum with the third-row element tungsten. It was expected that any differences resulting from this replacement would be

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