Tableau 5 (*suite*)

H	K	L	F(OBS)	F(CALC)	H	K	L	F(OBS)	F(CALC)	H	K	L	F(OBS)	F(CALC)	H	K	L	F(OBS)	F(CALC)
4	13	-3	8.45	8.66	4	11	0	23.80	35.06	4	1	5	6.48	0.23	5	7	-5	17.43	16.62
4	0	-2	12.18	13.29	4	1	1	33.60	37.92	4	2	5	10.16	7.45	5	8	-5	17.33	13.91
4	1	-2	52.27	59.84	4	2	1	24.78	28.85	4	3	5	11.40	13.74	5	1	-4	43.52	31.39
4	2	-2	38.22	40.12	4	3	1	32.93	41.61	4	4	5	22.92	27.83	5	2	-4	67.02	54.94
4	3	-2	62.23	64.53	4	4	1	22.35	29.30	4	1	-9	11.57	6.30	5	3	-4	6.36	2.23
4	4	-2	3.94	5.82	4	5	1	38.58	48.46	5	2	-9	29.82	18.89	5	4	-4	15.67	13.89
4	5	-2	4.45	2.66	4	6	1	17.06	19.06	5	3	-9	27.29	18.61	5	5	-4	20.87	12.04
4	7	-2	24.89	25.87	4	7	1	26.75	33.22	5	4	-9	9.55	4.56	5	6	-4	17.99	19.79
4	6	-2	25.77	24.55	4	8	1	6.68	1.86	5	5	-9	45.94	35.88	5	8	-4	30.43	31.08
4	8	-2	8.81	8.73	4	9	1	6.84	7.84	5	0	-8	16.37	11.24	5	10	-4	30.98	30.59
4	9	-2	59.48	61.15	4	10	1	8.91	8.19	5	1	-8	37.10	27.83	5	1	-3	20.97	26.64
4	10	-2	13.79	10.21	4	0	2	36.97	44.34	5	2	-8	11.92	7.09	5	2	-3	92.45	99.71
4	11	-2	23.90	20.54	4	1	2	18.77	27.27	5	3	-8	52.11	38.91	5	3	-3	11.22	11.81
4	12	-2	6.79	7.60	4	2	2	32.20	42.63	5	5	-8	14.91	8.50	5	4	-3	28.96	32.11
4	13	-2	18.56	16.51	4	3	2	24.01	27.93	5	7	-8	37.91	27.10	5	6	-3	27.39	29.50
4	1	-1	44.96	48.49	4	4	2	16.64	16.86	5	1	-7	7.78	2.98	5	8	-3	35.83	40.21
4	2	-1	15.60	19.66	4	5	2	13.53	18.18	5	2	-7	13.80	10.35	5	0	-2	25.52	34.17
4	3	-1	65.03	68.95	4	6	2	6.63	8.71	5	3	-7	45.14	32.66	5	2	-2	62.83	76.97
4	4	-1	24.89	24.51	4	7	2	22.19	27.64	5	4	-7	15.11	12.90	5	3	-2	18.04	22.64
4	5	-1	59.58	63.31	4	8	2	6.84	3.55	5	5	-7	69.20	55.85	5	5	-2	12.18	17.98
4	6	-1	23.75	24.16	4	9	2	17.11	26.46	5	6	-7	30.93	22.50	5	6	-2	16.07	17.60
4	7	-1	43.66	47.41	4	10	2	15.97	25.32	5	7	-7	40.33	32.07	5	8	-2	11.47	14.12
4	8	-1	6.11	6.11	4	1	3	12.23	13.22	5	8	-7	31.94	22.81	5	1	-1	23.80	32.60
4	9	-1	11.30	12.20	4	2	3	14.98	19.92	5	0	-6	18.55	17.50	5	2	-1	12.33	21.22
4	10	-1	6.74	5.57	4	3	3	6.68	7.42	5	1	-6	68.79	53.27	5	3	-1	10.26	15.08
4	11	-1	7.83	8.17	4	4	3	22.71	31.83	5	2	-6	35.99	25.75	5	4	-1	42.31	52.61
4	12	-1	6.74	2.18	4	5	3	21.72	28.55	5	3	-6	29.06	21.74	5	6	-1	38.21	46.67
4	13	-1	19.49	22.50	4	6	3	18.41	24.22	5	4	-6	61.72	48.19	5	7	-1	8.64	10.53
4	0	0	11.82	14.99	4	7	3	16.69	27.66	5	5	-6	13.19	7.99	5	8	-1	14.81	24.75
4	1	0	97.85	106.99	4	8	3	12.39	15.87	5	6	-6	13.29	13.53	5	0	0	15.01	23.67
4	2	0	8.60	11.08	4	0	4	7.83	13.46	5	7	-6	16.58	10.37	5	1	0	20.22	27.49
4	3	0	6.22	8.96	4	1	4	7.83	10.28	5	8	-6	56.96	53.53	5	2	0	29.62	42.51
4	4	0	6.68	7.43	4	2	4	21.83	27.31	5	9	-6	41.45	38.27	5	3	0	16.83	20.37
4	5	0	10.63	12.43	4	3	4	6.84	6.01	5	1	-5	48.42	35.55	5	4	0	21.53	31.24
4	6	0	5.65	4.86	4	4	4	17.11	20.17	5	2	-5	30.68	24.98	5	6	0	12.99	15.87
4	7	0	17.32	17.91	4	5	4	6.74	7.76	5	3	-5	43.01	32.93	5	2	1	13.29	21.04
4	8	0	8.34	10.88	4	6	4	6.58	6.64	5	4	-5	57.37	46.74	5	3	1	13.59	20.22
4	9	0	26.60	39.69	4	7	4	6.32	6.59	5	5	-5	27.75	21.02	5	4	1	23.96	32.03
4	10	0	6.84	6.99	4	8	4	15.09	20.48	5	6	-5	72.53	63.14	5	5	1	33.81	43.25

courte à l'anion Br^- du feuillet voisin devient $C(9,I)\cdots Br(1,II-c)=3{,}88$ Å alors que l'atome terminal C(10) est lié à un atome de soufre (dans le feuillet) par une liaison C–H···S forte (3,40 Å). Ce type de liaison se rencontre dans d'autres structures (Gauthier, 1966; Sutor, 1962, 1963).

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The Crystal Structure of π-Cyclopentadienyl Molybdenum Tricarbonyl Chloride, π-$C_5H_5Mo(CO)_3Cl$

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The crystal structure of π-cyclopentadienyl molybdenum tricarbonyl chloride, π-$C_5H_5Mo(CO)_3Cl$, has been determined from three-dimensional data collected photographically ($R=0{\cdot}11$). The crystals are monoclinic, space-group $P2_1/c$, with cell dimensions $a=7{\cdot}90\pm0{\cdot}02$, $b=10{\cdot}723\pm0{\cdot}004$, $c=15{\cdot}11\pm0{\cdot}04$ Å and $\beta=130°22'\pm8'$, with four formula units per cell. The complex forms a 'sandwich' with the planar cyclopentadienyl ring on one side of the molybdenum and the remaining atoms on the other with the chlorine towards an apex of the ring. The average bond lengths are C–C (ring) 1·36 Å, Mo–C (ring) 2·35 Å, Mo–C (carbonyl) 1·99 Å, C–O 1·16 Å and Mo–Cl 2·54 Å.

Introduction

In recent years there has been great interest in the compounds formed between transition metals and the cyclopentadienyl group, as reviewed for example by Wilkinson & Cotton (1959). The crystal structure of the dimer $[\pi$-$C_5H_5Mo(CO)_3]_2$, has been determined by Wilson & Shoemaker (1957) and it has been found that the metal–metal bond in this compound can be replaced by chlorine to give the derivative π-$C_5H_5Mo(CO)_3Cl$ (Piper & Wilkinson, 1956). The possibility of localized metal–cyclopentadienyl bonding in complexes lacking cylindrical symmetry has been discussed by Bennett, Churchill, Gerloch & Mason (1964) and this investigation adds further data on this type of complex.

Experimental

Red, 'lath-shaped' crystals were formed with faces parallel to the non-unique c axis of the monoclinic system. The accurate unit-cell dimensions were meas-

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ured by a procedure developed by Powell (1967) in which calibration was obtained by superimposing the powder pattern from gold wire on to a Weissenberg photograph of the *hk*0 reflexions and a precession photograph of the *h*0*l* reflexions. The lattice parameter for gold was taken from *International Tables for X-ray Crystallography* (1962) to be 4·078504 Å at 25 °C, not corrected for refraction (Weyerer, 1956), with coefficient of linear expansion $14{\cdot}1\times10^{-6}\,°C^{-1}$ and refraction correction $1{\cdot}86\times10^{-4}$ Å. Photographs were taken at 20 °C. The density was measured by flotation in a mixture of zinc bromide and alcohol.

Crystal data

Monoclinic with space group $P2_1/c$ from systematic absences of *h*0*l* when $l=2n+1$ and 0*k*0 when $k=2n+1$.

$a=\ 7{\cdot}90\ \pm0{\cdot}02$ Å
$b=10{\cdot}723\pm0{\cdot}004$
$c=15{\cdot}11\ \pm0{\cdot}04$
$\beta=130°22'\pm8'$

using the following wavelengths taken from *International Tables* (1962):

Cu $K\alpha_1=1{\cdot}54051$ Å
Cu $K\alpha_2=1{\cdot}54433$
Cu $K\bar{\alpha}\ =1{\cdot}54178$
Cu $K\beta\ =1{\cdot}39217$
Mo $K\alpha=0{\cdot}71069$
Mo $K\beta=0{\cdot}63225$.

Measured density $1{\cdot}97\pm0{\cdot}15$ g.cm^{-3}.
Calculated density $1{\cdot}910\pm0{\cdot}007$ g.cm^{-3}.
Molecular weight 280·535 a.m.u.
Number of molecules in one unit cell, 4.
Linear absorption coefficient for Cu $K\alpha$, $\mu_{\mathrm{Cu}\ K\alpha}$, $139{\cdot}7\pm0{\cdot}5$ cm^{-1}.
Linear absorption coefficient for Mo $K\alpha$, $\mu_{\mathrm{Mo}\ K\alpha}$, $16{\cdot}94\pm0{\cdot}06$ cm^{-1}.

Intensity data were collected with molybdenum radiation and a crystal chosen so that the shape would not introduce large absorption variation. Data from eleven levels were collected with a non-integrating Weissenberg camera, *hk*0 to *hk*10, and three levels, *h*0*l*, *h*1*l* and 0*kl*, were obtained with a Buerger precession camera. All intensities were measured visually and Lorentz, polarization and Weissenberg spot extension corrections were applied (Waser, 1951; Phillips, 1954, 1956), but no absorption or extinction corrections were made.

Layer scale factors were computed from the 73 reflexions occurring on more than one layer by the method described by Hamilton, Rollett & Sparks (1965). Seven hundred and ninety-nine unique reflexions were observed and the unobserved reflexions were not included in any way in the data used for the structure determination.

Structure determination

A three-dimensional Patterson synthesis yielded both the molybdenum and the chlorine positions. The parameters of these atoms were refined by a block-diagonal least-squares method in which the quantity being minimized was $\Sigma w(KF_o-F_c)^2$, where the weighting factor, w, was given by

$$w=\left[1+\frac{(KF_o-b)^2}{a^2}\right],$$

with parameters $a=2900$ and $b=2012$ on a scale of fifty times absolute. The atomic scattering factors were obtained from *International Tables* (1962). A three-dimensional electron density distribution allowed the carbonyl groups to be located, and after further refine-

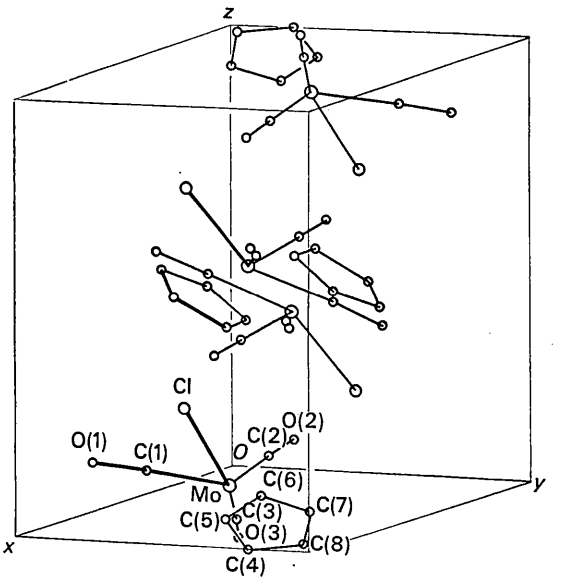

Fig. 1. Clinographic projection of structure excluding hydrogen atoms.

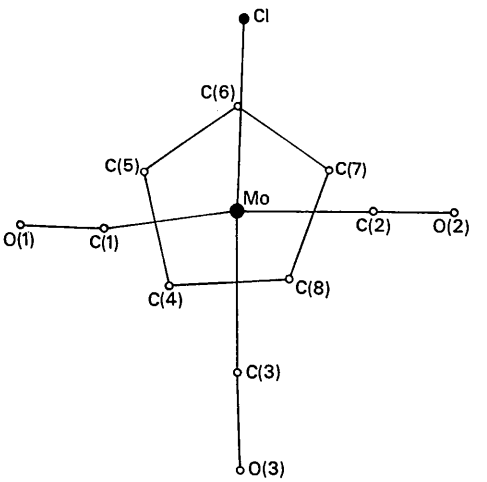

Fig. 2. Projection of molecule on to plane of cyclopentadienyl ring.

Table 1. *Observed and calculated structure factors* (×5)

h	k	l	Fo	Fc
0	2	0	643	-878
0	4	0	605	715
0	6	0	228	-222
0	8	0	113	-64
0	10	0	203	169
0	12	0	151	-148
0	14	0	151	179
1	0	0	759	1074
1	2	0	569	-763
1	3	0	196	-168
1	4	0	309	295
1	5	0	154	107
1	6	0	260	-251
1	7	0	174	-168
1	8	0	161	-108
1	10	0	190	157
1	11	0	116	-106
1	12	0	174	-189
1	14	0	158	187
2	0	0	209	87
2	2	0	412	-433
2	3	0	116	92
2	4	0	199	173
2	6	0	113	-68
2	8	0	109	-121
2	10	0	145	207
2	12	0	164	-234
2	14	0	154	185
3	0	0	466	470
3	1	0	280	-263
3	2	0	431	-398
3	3	0	338	299
3	4	0	437	401
3	5	0	196	-165
3	7	0	129	98
3	10	0	193	197
3	12	0	183	-199
3	14	0	164	149
4	0	0	444	443
4	1	0	257	-233
4	2	0	463	-505
4	3	0	212	166
4	4	0	322	326
4	5	0	167	-139
4	6	0	180	-140
5	0	0	360	347
5	1	0	116	-98
5	2	0	325	-351
5	4	0	203	188
6	0	0	203	197
6	1	0	183	-141
6	2	0	186	-149
7	0	0	141	105
7	1	0	145	-149
0	2	1	299	-299
0	4	1	469	573
0	5	1	363	-374
0	6	1	444	-473
0	7	1	254	261
0	8	1	315	367
0	9	1	219	-232
0	10	1	193	-202
0	11	1	100	91
0	12	1	106	100
1	1	1	215	201
1	2	1	67	-37
1	3	1	418	517
1	4	1	280	259
1	5	1	318	-297
1	6	1	267	-281
1	7	1	309	329
1	8	1	193	207
1	9	1	170	-206
1	10	1	158	-184
2	1	1	80	-52
2	2	1	164	-137
2	3	1	293	245
2	4	1	209	192
2	5	1	302	-325
2	6	1	260	-265
2	7	1	209	208
2	8	1	154	153
2	9	1	222	-230
2	10	1	164	-156
2	11	1	113	147
3	1	1	231	-197
3	2	1	212	-184
3	3	1	235	201
3	4	1	267	256
3	5	1	309	-323
3	6	1	212	-216
3	7	1	212	223
3	8	1	222	221
3	9	1	190	-208
4	1	1	77	-71
4	2	1	113	-104

h	k	l	Fo	Fc
4	3	1	228	218
4	4	1	119	123
4	5	1	170	-161
4	6	1	125	-138
4	7	1	228	275
4	8	1	138	153
4	9	1	145	-154
5	3	1	129	111
5	5	1	190	-165
5	6	1	96	-97
5	7	1	203	234
6	3	1	100	112
6	5	1	174	-189
-1	2	1	277	-295
-1	3	1	190	-153
-1	4	1	370	378
-1	5	1	209	-192
-1	6	1	418	-475
-1	7	1	186	194
-1	8	1	312	341
-1	9	1	154	-166
-1	10	1	206	-241
-1	11	1	145	143
-2	1	1	122	106
-2	2	1	167	-114
-2	3	1	338	277
-2	4	1	334	331
-2	5	1	222	-202
-2	6	1	309	-314
-2	7	1	212	181
-2	8	1	264	290
-2	9	1	96	-96
-2	10	1	212	-214
-2	11	1	103	105
-3	1	1	129	-125
-3	2	1	231	-186
-3	3	1	203	167
-3	4	1	389	456
-3	5	1	206	-176
-3	6	1	302	-332
-3	8	1	267	300
-3	10	1	193	-198
-4	1	1	109	-83
-4	2	1	215	-205
-4	3	1	77	77
-4	4	1	322	305
-4	6	1	289	-332
-4	10	1	164	-199
-5	2	1	177	-194
-5	4	1	209	194
-5	6	1	254	-287
-5	8	1	206	255
-5	10	1	141	-149
-6	4	1	148	153
-6	6	1	183	-219
-6	8	1	145	180
-7	2	1	113	-66
-7	4	1	116	127
0	0	2	154	-111
0	1	2	691	800
0	2	2	199	169
0	3	2	219	-177
0	4	2	244	-200
0	5	2	158	109
0	7	2	122	119
0	9	2	302	-293
0	11	2	199	231
0	13	2	203	-225
1	0	2	318	273
1	1	2	576	642
1	2	2	447	-423
1	3	2	495	-460
1	4	2	219	131
1	5	2	286	213
1	6	2	167	-141
1	7	2	151	53
1	9	2	206	-161
1	11	2	164	225
1	13	2	183	-194
2	0	2	141	84
2	1	2	498	523
2	3	2	556	-592
2	4	2	251	202
2	5	2	341	307
2	11	2	170	143
2	13	2	186	-142
3	0	2	177	-137
3	1	2	373	452
3	2	2	170	91
3	3	2	428	-442
3	5	2	206	173
3	11	2	125	135
3	13	2	138	-137
4	1	2	286	261
4	3	2	193	-195
4	13	2	145	-139

h	k	l	Fo	Fc
5	0	2	209	200
5	1	2	212	210
5	2	2	151	-112
5	3	2	154	-162
5	4	2	113	99
6	0	2	119	129
6	1	2	212	217
6	2	2	119	-123
6	3	2	170	-181
7	0	2	132	43
7	1	2	132	138
7	3	2	132	-106
-1	0	2	203	-309
-1	1	2	730	860
-1	2	2	453	494
-1	3	2	170	-169
-1	4	2	376	-346
-1	5	2	386	343
-1	6	2	254	211
-1	7	2	125	-88
-1	9	2	193	-145
-1	11	2	145	134
-1	13	2	183	-162
-1	15	2	141	160
-2	0	2	74	-35
-2	1	2	759	956
-2	2	2	122	90
-2	3	2	514	-542
-2	4	2	109	-90
-2	5	2	428	431
-2	7	2	141	-129
-2	9	2	129	-52
-2	10	2	109	-54
-2	11	2	145	115
-2	13	2	161	-149
-3	0	2	129	-90
-3	1	2	437	506
-3	2	2	119	100
-3	3	2	453	-495
-3	5	2	293	202
-3	6	2	113	-95
-3	8	2	100	97
-3	10	2	113	-99
-3	11	2	170	180
-3	13	2	132	-158
-4	0	2	322	-283
-4	1	2	164	167
-4	2	2	206	189
-4	3	2	267	-268
-4	4	2	151	-140
-4	9	2	141	-131
-4	11	2	154	192
-5	0	2	350	-330
-5	1	2	260	272
-5	2	2	299	290
-5	3	2	222	-244
-5	4	2	203	-218
-5	6	2	154	134
-6	0	2	270	-264
-6	1	2	244	208
-6	2	2	215	213
-6	3	2	154	-208
-6	4	2	158	-146
-6	5	2	116	116
-7	0	2	129	-133
-7	1	2	167	142
0	1	3	141	153
0	3	3	476	-555
0	4	3	264	203
0	5	3	543	579
0	6	3	244	-196
0	7	3	450	-423
0	8	3	163	177
0	9	3	270	253
0	10	3	174	-162
0	11	3	183	-170
1	1	3	87	59
1	2	3	254	-184
1	3	3	293	-284
1	4	3	412	450
1	5	3	412	434
1	6	3	289	-276
1	7	3	328	-362
1	8	3	190	203
1	9	3	286	284
1	10	3	151	-153
2	2	3	164	-137
2	3	3	177	-163
2	4	3	257	238
2	5	3	241	232
2	6	3	180	-168
2	7	3	270	-299
2	8	3	180	171
2	9	3	222	233
3	1	3	119	102
3	2	3	145	-158

h	k	l	Fo	Fc
3	3	3	177	-173
3	4	3	129	118
3	5	3	190	214
3	6	3	177	-197
3	7	3	183	-221
3	8	3	193	189
3	9	3	158	154
4	2	3	138	-156
4	3	3	138	-142
4	4	3	141	121
4	5	3	174	167
4	6	3	215	-228
4	7	3	158	-150
4	8	3	164	202
5	4	3	158	125
5	6	3	164	-172
-1	2	3	199	143
-1	3	3	399	-347
-1	5	3	502	484
-1	6	3	138	-113
-1	7	3	373	-374
-1	8	3	129	121
-1	9	3	273	279
-1	11	3	196	-186
-2	1	3	250	227
-2	3	3	395	-405
-2	4	3	264	222
-2	5	3	441	482
-2	6	3	158	-163
-2	7	3	379	-429
-2	8	3	129	102
-2	9	3	338	339
-2	11	3	183	-184
-3	2	3	125	-70
-3	3	3	450	-493
-3	4	3	158	127
-3	5	3	412	436
-3	6	3	138	-129
-3	7	3	395	-458
-3	9	3	341	321
-3	11	3	161	-169
-4	1	3	103	87
-4	2	3	132	-90
-4	3	3	244	-201
-4	5	3	379	402
-4	7	3	299	-321
-4	9	3	299	290
-4	11	3	164	-140
-5	1	3	151	128
-5	3	3	248	-246
-5	5	3	331	341
-5	7	3	315	-313
-5	9	3	222	240
-6	3	3	254	-246
-6	5	3	225	224
-6	7	3	260	-287
-6	9	3	170	181
-7	5	3	161	172
-7	7	3	170	-181
0	0	4	572	-634
0	1	4	344	-312
0	2	4	630	801
0	3	4	228	200
0	4	4	392	-377
0	6	4	293	266
0	7	4	129	-102
0	12	4	177	138
1	0	4	624	-723
1	1	4	357	-383
1	2	4	521	605
1	3	4	248	180
1	4	4	424	-398
1	5	4	145	-165
1	7	4	96	69
1	10	4	161	-146
1	12	4	180	155
2	0	4	251	-247
2	1	4	209	-169
2	2	4	280	260
2	3	4	122	89
2	10	4	215	-151
2	12	4	186	172
3	0	4	241	-228
4	0	4	305	-310
4	2	4	296	296
4	4	4	222	-227
5	0	4	235	-230
5	2	4	219	224
5	4	4	174	-170
-1	0	4	441	-495
-1	1	4	193	121
-1	2	4	90	114
-1	3	4	183	152
-1	4	4	209	-202
-1	6	4	273	218
3	2	4	254	249

h	k	l	Fo	Fc
-1	8	4	135	84
-1	9	4	145	139
-2	0	4	630	-745
-2	1	4	270	-248
-2	2	4	212	-92
-2	3	4	328	301
-2	4	4	190	-175
-2	5	4	289	-237
-2	8	4	193	168
-2	10	4	219	-219
-2	12	4	174	176
-3	0	4	379	-371
-3	1	4	608	-683
-3	2	4	505	503
-3	3	4	466	480
-3	4	4	190	-137
-3	5	4	305	-262
-3	6	4	174	145
-3	8	4	138	86
-4	0	4	341	-395
-4	1	4	338	-395
-4	2	4	498	504
-4	3	4	334	319
-4	4	4	354	-300
-5	0	4	309	-291
-5	1	4	177	-267
-5	2	4	251	226
-5	3	4	180	165
-5	4	4	231	-206
-6	1	4	302	-289
-6	3	4	193	180
-6	5	4	148	-142
-7	1	4	228	-248
-7	3	4	193	190
-7	5	4	164	-166
-8	0	4	170	-123
-8	1	4	209	-194
-8	2	4	170	88
-8	3	4	206	150
0	1	5	190	-170
0	2	5	244	239
0	3	5	100	-65
0	4	5	402	-414
0	6	5	466	492
0	8	5	302	-315
0	10	5	228	252
1	1	5	148	123
1	2	5	170	163
1	4	5	318	-326
1	5	5	244	244
1	6	5	322	362
1	8	5	293	-316
1	9	5	129	124
1	10	5	254	178
2	2	5	125	93
2	3	5	183	-167
2	4	5	186	-189
2	5	5	138	135
2	6	5	206	212
2	7	5	215	-222
2	8	5	225	-249
3	2	5	141	136
3	3	5	113	-99
3	4	5	186	-188
3	6	5	199	218
3	7	5	164	-181
4	4	5	161	-162
4	6	5	170	170
-1	1	5	203	-190
-1	2	5	235	215
-1	3	5	260	-236
-1	4	5	457	-422
-1	5	5	109	70
-1	6	5	373	379
-1	7	5	129	-87
-1	8	5	312	-322
-1	10	5	222	234
-2	1	5	260	233
-2	2	5	392	403
-2	4	5	473	-466
-2	5	5	322	245
-2	6	5	402	418
-2	8	5	334	-333
-3	1	5	177	-53
-3	2	5	347	343
-3	4	5	366	-368
-3	6	5	453	467
-3	8	5	360	-348
-3	10	5	219	242
-4	1	5	135	-112
-4	2	5	154	123
-4	3	5	106	-49
-4	4	5	376	-374
-4	6	5	379	383
-4	8	5	350	-382
-2	10	5	203	202

h	k	l	Fo	Fc
-4	10	5	244	243
-5	2	5	154	126
-5	4	5	376	-356
-5	6	5	334	333
-5	8	5	299	-306
-5	10	5	170	192
-6	2	5	158	139
-6	4	5	199	-199
-6	5	5	145	-143
-6	6	5	209	241
-6	8	5	193	-198
-7	2	5	148	143
-7	4	5	154	-156
-7	6	5	164	194
-8	2	5	167	116
-8	6	5	180	176
0	0	6	479	500
0	1	6	228	-249
0	2	6	450	-499
0	3	6	231	196
0	4	6	251	225
1	0	6	129	163
1	1	6	350	-407
1	2	6	174	-166
1	3	6	296	299
2	1	6	373	-426
2	3	6	296	340
2	5	6	199	-179
3	1	6	264	-287
3	2	6	164	-155
3	3	6	231	263
3	5	6	177	-185
-1	0	6	582	636
-1	1	6	457	-450
-1	2	6	402	-380
-1	3	6	392	350
-1	4	6	466	471
-1	5	6	277	-280
-1	6	6	138	-125
-2	0	6	344	304
-2	1	6	585	-664
-2	2	6	473	-433
-2	3	6	415	402
-2	4	6	309	230
-2	5	6	418	-373
-2	7	6	180	148
-2	9	6	161	106
-3	0	6	302	282
-3	1	6	325	-329
-3	2	5	395	-404
-3	4	6	161	132
-3	5	6	251	-203
-3	8	6	154	-134
-3	10	6	174	188
-4	0	6	508	556
-4	1	6	231	-189
-4	2	6	431	-429
-4	4	6	328	324
-5	0	6	506	609
-5	1	6	244	-214
-5	2	6	514	-555
-5	3	6	238	217
-5	4	6	338	350
-5	6	6	151	-154
-6	0	6	347	380
-6	1	6	167	-141
-6	2	6	322	-322
-6	3	6	206	191
-6	4	6	212	217
-7	0	6	199	239
-7	2	6	161	-168
-8	0	6	254	273
-8	2	6	183	-202
-9	0	6	199	222
-9	2	6	196	-190
0	2	7	109	-126
0	3	7	257	266
0	5	7	334	-290
0	7	7	286	316
0	9	7	222	-230
1	1	7	122	-118
1	3	7	280	263
1	5	7	309	-333
1	9	7	209	-234
2	3	7	141	157
2	5	7	231	-242
2	7	7	206	223
2	9	7	177	-190
-1	1	7	87	-76
-1	2	7	93	-63
-1	3	7	225	203
-1	5	7	338	-364
-1	7	7	296	301
-1	9	7	235	-249
-1	11	7	170	161
1	7	7	280	312

Table 1 (*cont.*)

h	k	l	Fo	Fc
-2	1	7	186	-177
-2	3	7	405	346
-2	4	7	103	-65
-2	5	7	441	-451
-2	7	7	367	393
-2	9	7	277	-263
-2	11	7	199	180
-3	1	7	190	-184
-3	3	7	312	293
-3	5	7	341	-348
-3	7	7	350	377
-3	9	7	260	-262
-3	11	7	167	155
-4	1	7	129	-115
-4	2	7	96	-93
-4	3	7	289	268
-4	4	7	109	99
-4	5	7	334	-308
-4	6	7	125	-98
-4	7	7	331	324
-4	9	7	235	-248
-5	2	7	170	-155
-5	3	7	254	264
-5	4	7	235	224
-5	5	7	309	-305
-5	6	7	190	-158
-5	7	7	270	285
-5	8	7	151	143
-5	9	7	199	-227
-6	3	7	154	134
-6	4	7	190	177
-6	5	7	257	-249
-6	6	7	174	-155
-6	7	7	215	202
-6	9	7	167	-198
-7	3	7	145	147
-7	5	7	180	-184

h	k	l	Fo	Fc
-7	7	7	193	178
0	0	8	325	348
0	1	8	251	209
0	2	8	344	-349
0	3	8	148	-138
0	4	8	260	235
0	6	8	141	-122
0	9	8	138	-139
1	0	8	328	406
1	1	8	270	326
1	2	8	408	-354
1	3	8	277	-254
1	4	8	251	234
1	5	8	158	139
1	6	8	129	-102
2	0	8	228	219
2	1	8	215	210
2	2	8	180	-198
2	3	8	199	-171
3	0	8	138	114
3	2	8	138	-129
4	0	8	180	170
4	2	8	154	-153
-1	0	8	225	221
-1	1	8	235	194
-1	2	8	241	-228
-1	3	8	148	-142
-1	4	8	138	117
-1	9	8	132	-106
-1	11	8	148	156
-2	0	8	199	173
-2	1	8	598	564
-2	2	8	225	-193
-2	3	8	463	-466
-2	5	8	215	178
-2	8	8	154	-115
-2	10	8	138	135

h	k	l	Fo	Fc
-3	0	8	457	455
-3	1	8	633	635
-3	2	8	212	-170
-3	3	8	514	-546
-3	4	8	251	218
-3	5	8	260	222
-3	6	8	212	-166
-3	11	8	145	118
-3	13	8	161	-145
-4	0	8	354	512
-4	1	8	408	433
-4	2	8	296	-246
-4	3	8	331	-326
-4	4	8	277	235
-4	5	8	186	160
-4	6	8	154	-132
-4	9	8	132	-114
-4	11	8	174	186
-4	13	8	161	-162
-5	1	8	354	342
-5	2	8	183	-146
-5	3	8	257	-266
-5	5	8	129	107
-5	9	8	170	-169
-5	11	8	190	197
-6	0	8	145	-68
-6	1	8	264	335
-6	3	8	289	-301
-6	5	8	167	173
-7	1	8	273	379
-7	3	8	273	-274
-7	5	8	183	196
-8	1	8	293	308
-8	3	8	190	-182
-9	1	8	170	180
-9	3	8	116	-108
0	2	9	177	-160

h	k	l	Fo	Fc
0	4	9	260	273
0	5	9	113	108
0	6	9	207	-258
0	8	9	228	258
1	2	9	158	-151
1	4	9	219	251
1	6	9	235	-256
1	8	9	193	210
2	4	9	163	179
2	6	9	196	-208
3	6	9	148	-159
-1	2	9	199	-186
-1	3	9	132	-85
-1	4	9	309	305
-1	8	9	248	262
-1	10	9	193	-192
-2	1	9	219	172
-2	2	9	199	-176
-2	4	9	331	312
-2	5	9	135	116
-2	6	9	354	-373
-2	7	9	158	-156
-2	8	9	238	265
-2	10	9	186	-190
-3	1	9	122	101
-3	2	9	154	-141
-3	4	9	289	273
-3	5	9	212	180
-3	6	9	315	-334
-3	7	9	222	-212
-3	8	9	264	315
-3	9	9	174	147
-3	10	9	186	-181
-4	2	9	260	-239
-4	3	9	251	-216
-4	4	9	302	266
-4	5	9	177	147

h	k	l	Fo	Fc
-4	6	9	325	-353
-4	7	9	222	-236
-4	8	9	267	299
-4	9	9	177	143
-5	1	9	164	134
-5	2	9	193	-190
-5	3	9	251	-196
-5	4	9	267	223
-5	5	9	286	259
-5	6	9	277	-259
-5	7	9	203	-190
-5	8	9	174	212
-6	3	9	177	-162
-6	4	9	212	214
-6	5	9	222	222
-6	6	9	164	-153
-6	7	9	170	-172
-7	3	9	161	-161
-7	4	9	235	211
-7	5	9	170	187
-7	6	9	206	-181
-7	7	9	183	-184
-8	5	9	183	211
0	0	10	338	-350
0	2	10	289	266
0	4	10	251	-243
1	0	10	161	-212
1	2	10	164	174
2	1	10	164	192
-1	0	10	309	-332
-1	1	10	251	246
-1	2	10	370	380
-1	3	10	219	-238
-1	4	10	228	-221
-1	5	10	135	117
-1	6	10	203	171
-2	0	10	363	-328

h	k	l	Fo	Fc
-2	1	10	270	234
-2	2	10	289	298
-2	3	10	238	-200
-2	4	10	196	-164
-2	5	10	177	125
-3	0	10	347	-386
-3	4	10	270	-221
-4	0	10	450	-465
-4	4	10	350	-278
-5	0	10	447	-483
-5	2	10	505	478
-5	4	10	373	-313
-5	6	10	190	153
-6	0	10	263	-299
-6	2	10	238	219
-6	4	10	215	-214
-7	0	10	254	-248
-7	2	10	225	235
-7	4	10	141	-128
-8	0	10	270	-311
-8	1	10	164	-133
-8	2	10	267	249
-8	4	10	170	-149
-9	0	10	231	-258
-9	2	10	107	185
-10	0	10	180	-153
-2	1	12	395	-440
-3	1	12	395	-438
-4	1	12	280	-333
-5	1	12	280	-276
-6	1	12	280	-357
-7	1	12	341	-383
-1	0	14	138	294
-3	0	14	270	274
-4	0	14	402	385
-5	0	14	347	316
-7	0	14	264	286
-8	0	14	231	267

ment a difference Fourier synthesis revealed the cyclopentadienyl ring and also indicated that anisotropic temperature factors were required for the molybdenum and chlorine atoms.

Further refinement on all atoms in which the parameters being refined were the overall scale factor, K, layer scale factors, positional parameters and isotropic or anisotropic temperature factors, gave a residual

$$R=\frac{\Sigma\,|KF_o-F_c|}{\Sigma KF_o}=0{\cdot}11\,.$$

A final difference Fourier synthesis gave no significant indications of the positions of hydrogen atoms. An analysis of the average $\Sigma w(KF_o-F_c)^2$ for different ranges of KF_o did not indicate that the parameters of the weighting factor expression needed modification. The observed and calculated structure factors on a scale five times absolute are given in Table 1. The final positional parameters and the standard deviations from the least squares method are given in Table 2 and the thermal parameters are listed in Table 3.

Table 2. *Final fractional positional parameters with their estimated standard deviations* (*all* $\times 10^4$)

	x	$\sigma(x)$	y	$\sigma(y)$	z	$\sigma(z)$
Mo	165	4	2152	2	3586	2
Cl	−2344	14	2459	8	1400	8
O(1)	−2949	47	−196	25	2638	27
O(2)	3432	38	1954	21	3093	23
O(3)	3507	44	108	24	5456	27
C(1)	−1641	56	590	32	3029	34
C(2)	2227	47	1976	27	3258	28
C(3)	2211	55	849	31	4715	33
C(4)	92	62	2843	35	5035	39
C(5)	−1723	59	3356	33	4012	35
C(6)	−1132	54	4163	31	3555	32
C(7)	1077	56	4232	32	4268	34
C(8)	1792	60	3445	34	5164	36

Table 3

(*a*) Thermal parameters (Å²) where the temperature factor is given by

$\exp[-10^{-4}(B_{11}h^2+B_{22}k^2+B_{33}l^2+B_{23}kl+B_{13}hl+B_{12}hk)]$ or $\exp[-B\sin^2\theta/\lambda^2]$.

	B_{11}	B_{22}	B_{33}	B_{23}	B_{13}	B_{12}
Mo	151	35	32	5	105	23
Cl	251	83	51	23	135	77

	B
O(1)	5·93
O(2)	4·44
O(3)	5·82
C(1)	4·04
C(2)	2·82
C(3)	3·92
C(4)	4·83
C(5)	4·15
C(6)	3·64
C(7)	3·86
C(8)	4·32

(*b*) Magnitudes $(\overline{U^2})^{1/2}$ and direction cosines l relative to **a***, **b***, **c*** of the principal axes of the vibrational ellipsoids.

	$(\overline{U^2})^{1/2}\times 10^3$ (Å)	$l_1\times 10^4$	$l_2\times 10^4$	$l_3\times 10^4$
Mo	171	134	3870	7111
	125	9197	4711	3576
	138	−3931	−5222	8499
Cl	248	−2832	3404	6689
	176	−5687	−8879	4605
	203	7723	3096	5842

Description of structure

The structure is of the 'sandwich' type in which the molybdenum is coordinated to the cyclopentadienyl ring on one side and to the chlorine and carbonyl

Table 4. *Intramolecular bond lengths and angles with the estimated standard deviations*

		σ			σ
Mo–Cl	2·542 Å	0·009 Å	Cl—Mo–C(1)	79·5°	1·1°
Mo–C(1)	1·999	0·037	Cl—Mo–C(2)	76·4	1·0
Mo–C(2)	1·996	0·037	C(2)–Mo–C(3)	78·7	1·5
Mo–C(3)	1·976	0·036	C(1)–Mo–C(3)	74·5	1·6
C(1)–O(1)	1·156	0·049	Mo–C(1)–O(1)	169·8	3·5
C(2)–O(2)	1·129	0·047	Mo–C(2)–O(2)	175·5	2·6
C(3)–O(3)	1·203	0·047	Mo–C(3)–O(3)	175·9	3·1
C(4)–C(5)	1·370	0·060	C(4)–C(5)–C(6)	112·1	4·0
C(5)–C(6)	1·363	0·056	C(5)–C(6)–C(7)	109·0	3·5
C(6)–C(7)	1·333	0·058	C(6)–C(7)–C(8)	104·4	3·7
C(7)–C(7)	1·369	0·055	C(7)–C(8)–C(4)	114·0	4·0
C(8)–C(4)	1·386	0·065	C(8)–C(4)–C(5)	100·4	3·7
Mo–C(4)	2·348	0·046			
Mo–C(5)	2·355	0·043			
Mo–C(6)	2·374	0·035			
Mo–C(7)	2·365	0·033			
Mo–C(8)	2·299	0·040			

groups on the other. Fig. 1 shows a clinographic projection of the atomic arrangement, excluding hydrogen atoms. Intramolecular bond lengths and angles are given in Table 4 where the estimated standard deviations are calculated from the coordinate standard deviations with use of the expressions given by Darlow (1960). The standard deviations from the least-squares method are probably underestimates but it is suggested that the molybdenumcarbonyl configuration is non-linear, which is in agreement with the results of Bennett & Mason (1963) for the compound tricarbonyl-π-cyclopentadienylethylmolybdenum. The average C–O distance is 1·16 Å, which agrees well with the values found by previous authors, but the average Mo–C (carbonyl) distance is 1·99 Å, which is slightly greater than the previously published values though not significantly so in view of the estimated standard deviations.

The average C–C (ring) distance is 1·36 Å without significant variation and the ring atoms are at an average distance of 2·36 Å from the molybdenum except Mo–C(8) at 2·30 Å. The cyclopentadienyl ring is planar with the equation of the mean plane

$$0{\cdot}4403X-0{\cdot}7498Y-0{\cdot}4938Z+7{\cdot}2978=0\,,$$

where X, Y and Z are orthogonal coordinates related to the fractional coordinates in the monoclinic cell by $X=xa+zc\cos\beta$, $Y=yb$ and $Z=zc\sin\beta$. The distances of the ring atoms and the molybdenum from this plane are given in Table 5. If all the atomic positions in one formula unit are projected on to this plane, the arrangement is as shown in Fig. 2. Thus the chlorine takes up a position towards one of the apices of the cyclopentadienyl ring.

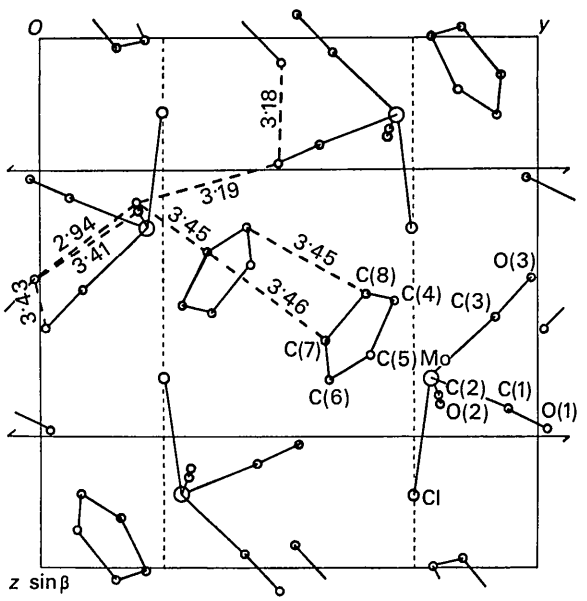

Fig. 3. [100] projection showing intermolecular contacts less than 3·5 Å.

Table 5. *Distances of atoms from mean plane of cyclopentadienyl ring,*

$$0{\cdot}4403X-0{\cdot}7498Y-0{\cdot}4938Z+7{\cdot}2978=0$$

C(4)	0·012 Å		
C(5)	−0·010		
C(6)	0·004	Mo	2·041 Å
C(7)	0·004		
C(8)	−0·010		

The shortest intermolecular contacts are between the oxygen atoms of the carbonyl groups which have the values 2·94, 3·18 and 3·19 Å. The separation of the two cyclopentadienyl rings across a centre of symmetry is not less than 3·45 Å. Fig. 3 shows a projection of the structure, excluding hydrogen atoms, with the contacts less than 3·5 Å illustrated.

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