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The Crystal and Molecular Structure of 1,3-Diphenyl-1,3-propanedione-(1,6-dichloro-1,5-cyclooctadiene)rhodium(I)

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The crystal and molecular structure of 1,3-diphenyl-1,3-propanedione-(1,6-dichloro-1,5-cyclooctadiene)-rhodium(I) has been determined by single-crystal X-ray methods. The unit cell is orthorhombic with $a=9{\cdot}56$ (1), $b=20{\cdot}50$ (2), $c=10{\cdot}65$ (1) Å, $V=2087$ Å^3, and contains four formula units. The space group is *Pnma*. The crystal structure has been determined from single-crystal diffractometer data by symbolic addition methods and refined by the least-squares method. The final R value is 0·040. The molecule is discussed in terms of a conjugated system.

The investigation of the chemical and physical properties of the coordination compounds of Rh(I) with various donors such as ethylene, dienes, or CO has resulted in a number of relevant findings in the field of the catalysis of industrially important reactions, such as the oxo process, isomerization, dimerization and polymerization of unsaturated compounds (Rinehart, Smith, Witt & Romeyn, 1962). The catalytic activity of these complexes is strongly dependent on the character of ligands coordinated with the central metal atom. In the series of 1,3-substituted propanedione rhodium(I) chelates the catalytic activity depends on the character of the substituents of the propanedione ring (Bouchal, Škramovská, Čoupek, Pokorný & Hrabák, 1972). An analysis of the n.m.r. spectra allowed a qualitative interpretation of this dependence on the basis of the pseudoconjugated character of the chelate ring (Bouchal, Škramovská, Schmidt & Hrabák, 1972). The changes in electron density caused by substitution are transferred in the hybrid orbitals of the central metal atom, thus affecting also the bond with the donor. A quantitative interpretation necessitates a knowledge of the structure of these complexes, which has led to their study by X-ray diffraction methods (Huml & Ječný, 1971).

Experimental

Yellow crystals of 1,3-diphenyl-1,3-propanedione-(1,6-dichloro-1,5-cyclooctadiene)rhodium(I) were obtained in the laboratory headed by Dr Hrabák in this Institute (Bouchal, Škramovská, Schmidt & Hrabák, 1972).

Crystal data

1,3-Diphenyl-1,3-propanedione-(1,6-dichloro-1,5-cyc-looctadiene)rhodium(I), $C_{23}H_{21}O_2Cl_2Rh$,
F.W. 503·22; m.p. 216–218°C; orthorhombic;
$a=9{\cdot}56$ (1); $b=20{\cdot}50$ (2); $c=10{\cdot}65$ (1) Å; $V=2087$ Å^3.
(The cell dimensions are weighted averages obtained from single-crystal diffractometer measurements of axial reflexions.)
$Z=4$; $F(000)=1016$;
$D_x=1{\cdot}594$; $D_m=1{\cdot}59$ g cm^{-3} (flotation in aqueous CsCl solution at room temperature);
$\mu(\text{Cu } K\alpha)=94{\cdot}79$ cm^{-1}.
Absent reflexions were determined from single-crystal photographs:

$0kl$ for $k+l$ odd,
$hk0$ for h odd.

Space group: $Pna2_1$ or *Pnma*. The latter was indicated by statistical tests and confirmed by the subsequent refinement. The crystal, ground to a spherical shape, radius $r=0{\cdot}095$ mm, was mounted along the c axis on a manual four-circle diffractometer provided with a scintillation counter and pulse-height analyser. The intensity data were collected by the θ–2θ scan technique. The following scan intervals were used:

2° for $2\theta \le 40°$,
3° for $40° < 2\theta \le 90°$,
4° for $2\theta > 90°$.

The background was measured at the beginning and end of each scan. Ni-filtered Cu $K\alpha$ radiation was employed. If the pulse number exceeded 20 000 c.p.s., Al attenuation filters were used. The standard reflexion 080 was measured after every 25 reflexions and used for data scaling.

No evidence of crystal decomposition could be detected during the measurement of the intensity data. 1792 independent reflexions with $2\theta < 128°$ were measured, of which 1482 were above the threshold value (*i.e.* net count higher than 30 pulses, or net count higher than 10% of the background). The intensities were corrected for the Lp factors. Absorption corrections for a sphere, ranging from 2·88 to 3·61, were applied to the measured intensities. No extinction correction was considered. The absolute scale and approximate overall temperature factor were determined from all three-dimensional data by Wilson's method (Ahmed, Hall, Pippy & Huber, 1966). All calculations were carried out with an IBM 7040 computer.

Structure determination and refinement

The approximate structure was readily deduced by symbolic addition procedures, and refined by block-diagonal least-squares methods with the programs of Ahmed, Hall, Pippy & Huber (1966). The quantity minimized was $\sum w(|F_c|-|F_o|)^2$. The weighting scheme, chosen to ensure reasonable constancy of $w\Delta F^2$ with respect to $|F_o|$ and $\sin^2\theta$, was $w=w_1w_2$, where

$$\begin{aligned} w_1 &= 1{\cdot}0 && \text{for } |F_o| \le 50{\cdot}0\,, \\ &= (50{\cdot}0/F_o)^2 && \text{for } |F_o| > 50{\cdot}0\,, \\ w_2 &= 1{\cdot}0 && \text{for } \sin^2\theta < 0{\cdot}30\,, \\ &= (\sin^2\theta/0{\cdot}30)^2 && \text{for } \sin^2\theta \ge 0{\cdot}30\,. \end{aligned}$$

The scattering factor curves were taken from *International Tables for X-ray Crystallography* (1962). The curve for rhodium was corrected for anomalous scattering. Isotropic least-squares refinement, followed by anisotropic refinement, reduced the R value ($=\sum||F_o|-|F_c||/\sum|F_o|$, for all observed reflexions) from 0·220 to 0·045.

A difference synthesis for all reflexions with $\sin\theta/\lambda <$ 0·4 Å^{-1} was calculated at this point and the H(C2)–H(C7), H(C11) and H(C121) hydrogen atoms were found at the expected positions. Their contributions were subsequently included in the structure-factor calculations. The parameters of H(C7), H(C11) and H(C121) with initial isotropic temperature factors made equal to those of the corresponding carbon atoms were also refined during the calculation procedure. Several attempts to localize the remaining hydrogen atoms H(C91), H(C92) and H(C122) failed and these atoms were not included in the final calculation of structure factors. It was not possible to locate the missing hydrogen atoms on the final difference map. The refinement was complete when R was 0·040. None of the shifts indicated in the final cycle exceeded $\frac{1}{3}$ of the corresponding e.s.d. The resulting atomic parameters with the corresponding e.s.d. are given in Tables 1 and 2. The observed and calculated structure factors are in Table 3.

Table 2. *Parameters and e.s.d.'s of hydrogen atoms*

Fractional coordinates are $\times 10^4$. Parameters of H(C2) to H(C6) were not refined.

	x/a	y/b	z/c	B (Å^2)
H(C2)	3650	790	4572	5·58
H(C3)	2307	−241	4858	6·97
H(C4)	566	−303	6577	6·96
H(C5)	138	637	7955	6·07
H(C6)	1452	1665	7648	4·40
H(C7)	1919	2500	6385	2·61
	101	0	88	1·77
H(C11)	7200	1455	5269	4·25
	95	41	81	1·66
H(C121)	9567	2040	4378	6·98
	133	64	114	2·56

H(C91), H(C92) and H(C122) omitted from the F_c calculation.

Table 1. *Final parameters and e.s.d.'s of non-hydrogen atoms*

Temperature factor $=\exp[-(\beta_{11}h^2+\beta_{22}k^2+\beta_{33}l^2+\beta_{12}hk+\beta_{13}hl+\beta_{23}kl)]$. B_i (Å^2) are the temperature factors corresponding to the principal radii of the thermal motion ellipsoids. All the other values are $\times 10^5$.

	x/a	y/b	z/c	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}	B_1	B_2	B_3
C(1)	26389	12903	60819	862	247	907	56	−213	223	5·10	3·59	2·67
	50	26	49	50	13	47	42	84	42			
C(2)	28719	7537	53251	1569	243	1326	−20	324	158	5·33	6·61	3·80
	74	29	66	77	14	63	57	131	54			
C(3)	21204	1824	54712	2311	243	2145	−270	476	207	8·30	10·22	3·62
	107	34	98	121	16	113	74	205	71			
C(4)	11571	1432	64277	2047	317	2515	−314	556	653	7·98	12·56	3·55
	101	41	112	116	20	136	82	216	88			
C(5)	9034	6716	72030	1511	426	2004	−152	693	880	6·23	12·12	3·31
	85	42	91	89	23	106	79	167	85			
C(6)	16419	12435	70303	1125	313	1145	34	387	370	4·25	6·96	3·28
	62	31	60	63	17	57	55	103	51			
C(7)	28502	25000	61174	744	253	786	0	220	0	2·51	4·23	3·74
	69	0	66	66	18	60	0	115	0			
C(8)	34432	18973	58494	830	260	602	1	−76	130	3·05	4·53	2·50
	50	25	43	47	13	36	41	73	36			
C(9)	70857	21440	23663	2800	488	1158	838	2063	212	2·69	14·31	6·54
	115	46	76	153	22	67	105	184	72			
C(10)	70487	18270	35183	937	352	881	383	301	−89	6·59	4·30	2·38
	57	31	51	54	16	47	52	88	48			
C(11)	76147	18226	47154	858	446	1063	441	−119	134	2·44	8·12	4·81
	57	36	59	56	20	54	56	100	63			
C(12)	89605	21372	50967	903	633	2047	58	−930	250	2·70	11·27	9·15
	69	54	98	64	29	104	74	142	95			
Cl	59956	11629	30472	1588	373	1468	398	−16	−540	6·00	9·16	3·48
	20	9	18	21	5	18	18	35	15			
O	46395	18052	53328	802	238	920	85	324	85	2·61	4·74	3·70
	34	17	33	34	8	32	28	59	30			
Rh	59746	25000	46654	594	259	643	0	78	0	2·12	4·33	2·94
	5	0	4	4	1	4	0	8	0			

Table 3. *Observed and calculated structure factors* (× 10)

An asterisk indicates an unobserved reflexion, for which the threshold value is given.

H	FO	FC
K= 0, L= 0		
2	1006	-971
4	1137	-1091
6	1453	-1418
8	290	269
10	572	573
K= 0, L= 1		
1	1832	1888
2	644	-614
3	401	-353
4	358	-366
5	683	-666
6	46*	88
7	404	-404
8	346	359
9	373	367
10	45*	-61
11	269	273
K= 0, L= 2		
0	1549	1612
1	2694	-2823
2	148	73
3	743	-733
4	124	103
5	218	-212
6	647	-626
7	434	413
8	176	160
9	193	184
10	306	295
11	72	-84
K= 0, L= 3		
1	794	762
2	1262	-1162
3	151	167
4	40*	-51
5	563	-521
6	49*	-53
7	53*	-35
8	237	234
9	53*	65
10	47*	-9
K= 0, L= 4		
0	1543	1584
1	567	-580
2	498	510
3	192	176
4	517	-518
5	439	-427
6	645	-631
7	220	227
8	117	104
9	52*	65
10	288	297
K= 0, L= 5		
1	654	691
2	832	-866
3	221	229
4	824	-791
5	751	-731
6	396	408
7	179	-164
8	257	261
9	223	214
10	42*	44
K= 0, L= 6		
0	532	545
1	847	-846
2	304	299
3	1278	-1294
4	347	-318
5	151	-140
6	310	-304
7	486	465
8	52*	25
9	310	301
K= 0, L= 7		
1	104	-120
2	972	-993
3	51*	-13
4	745	-749
5	187	211
6	299	298
7	97	-94
8	449	459
9	42*	-9
K= 0, L= 8		
0	184	-202
1	416	-430
2	232	-235
3	438	-448
4	284	288
5	281	-284
6	54*	-11
7	342	339
8	44*	20
K= 0, L= 9		
1	219	-227
2	371	-367
3	55*	25
4	318	-304
5	166	171
6	77	76
7	45*	38
K= 0, L= 10		
0	122	-132
1	311	-305
2	95*	-73
3	326	-311
4	52*	28
5	170	-163
6	91	107
K= 0, L= 11		
1	199	-193
2	367	-341
3	48*	-26
4	255	-232
5	170	166
K= 0, L= 12		
0	369	-354
1	223	-223
2	109	-109
K= 1, L= 0		
2	1569	-1572
4	904	-917
6	951	879
8	835	830
10	230	245
K= 1, L= 1		
0	732	-706
1	852	-808
2	25*	-61
3	1530	-1467
4	269	257
5	41*	-32
6	508	486
7	1144	1107
8	75	-45
9	554	543
10	240	-239
11	217	-211
K= 1, L= 2		
1	697	-672
2	279	292
3	356	337
4	377	-374
5	797	746
6	465	441
7	206	187
8	537	532
9	223	-212
10	140	135
11	241	-247
K= 1, L= 3		
0	403	312
1	800	-822
2	502	-498
3	476	-468
4	608	574
5	44*	11
6	394	382
7	724	715
8	54*	12
9	242	240
10	272	-265
K= 1, L= 4		
1	161	189
2	680	-701
3	173	-142
4	334	-321
5	722	706
6	471	461
7	130	136
8	373	351
9	229	-235
10	99	93
K= 1, L= 5		
0	581	-609
1	802	-830
2	63	-60
3	614	-622
4	506	492
5	50*	16
6	716	705
7	565	569
8	168	-161
9	201	181
10	302	-303
K= 1, L= 6		
1	822	-838
2	527	-503
3	484	474
4	110	106
5	967	975
6	55*	84
7	209	206
8	130	158
9	327	-332
K= 1, L= 7		
0	979	-1005
1	299	-289
2	49*	-57
3	106	113
4	632	623
5	54*	58
6	614	628
7	54*	11
8	136	-134
9	42*	-5
K= 1, L= 8		
1	452	-465
2	253	253
3	362	360
4	81	92
5	480	490
6	54*	-22
7	161	159
8	145	-153
K= 1, L= 9		
0	579	-594
1	245	257
2	83	96
3	55*	29
4	319	316
5	53*	79
6	272	272
7	128	-122
K= 1, L= 10		
1	328	-309
2	177	183
3	171	173
4	52*	66
5	406	382
6	71	-57
K= 1, L= 11		
0	240	-229
1	199	188
2	172	-148
3	213	195
4	302	292
5	68	-40
K= 1, L= 12		
1	164	-149
2	321	318
K= 2, L= 0		
0	2597	-2489
2	180	-143
4	575	552
6	759	742
8	174	-155
10	420	-425
K= 2, L= 1		
1	1716	-1768
2	950	904
3	512	485
4	182	159
5	769	756
6	109	-97
7	306	302
8	154	-158
9	328	-319
10	49*	14
11	285	-299
K= 2, L= 2		
0	1749	-1929
1	456	430
2	1436	-1388
3	178	179
4	1175	1149
5	81	75
6	857	830
7	124	-117
8	186	-185
9	142	-132
10	395	-399
K= 2, L= 3		
1	670	-684
2	1240	1197
3	168	-193
4	200	209
5	725	700
6	149	-145
7	151	144
8	314	-323
9	195	-179
10	47*	9
K= 2, L= 4		
0	1010	-1044
1	684	726
2	574	-575
3	1106	1121
4	568	577
5	132	154
6	615	586
7	478	-471
8	192	-181
9	182	-188
10	249	-248
K= 2, L= 5		
1	331	-337
2	850	874
3	142	-129
4	736	727
5	425	412
6	376	-387
7	89	86
8	330	-335
9	96	-105
10	72	-74
K= 2, L= 6		
0	189	-172
1	270	267
2	201	-189
3	994	997
4	150	148
5	93	82
6	246	232
7	438	-428
8	52*	-32
9	204	-195
K= 2, L= 7		
1	67	-32
2	733	745
3	51*	5
4	515	521
5	55*	-10
6	245	-244
7	53*	50
8	356	-363
9	41*	-46
K= 2, L= 8		
0	123	-93
1	395	396
2	153	145
3	596	593
4	94	-101
5	221	217
6	54*	28
7	397	-399
8	44*	8
K= 2, L= 9		
1	167	169
2	534	515
3	55*	-17
4	427	417
5	120	-134
6	211	-199
7	44*	-45
K= 2, L= 10		
0	275	265
1	241	229
2	119	109
3	396	384
4	129	-108
5	67	83
6	161	-170
K= 2, L= 11		
1	258	256
2	300	285
3	48*	-17
4	205	183
5	237	-231
K= 2, L= 12		
0	257	245
1	136	139
2	141	124
K= 3, L= 0		
2	430	-394
4	234	268
6	216	-206
8	525	-518
10	66	-43
K= 3, L= 1		
0	681	646
1	1593	1532
2	546	-519
3	938	898
4	254	248
5	199	167
6	47*	-46
7	845	-815
8	54*	-9
9	417	-404
10	49*	23
11	185	174
K= 3, L= 2		
1	239	-249
2	1998	1971
3	125	121
4	722	702
5	133	-122
6	513	-486
7	143	-137
8	757	-745
9	173	156
10	121	-110
K= 3, L= 3		
0	632	705
1	1152	1189
2	215	220
3	1186	1196
4	415	-415
5	134	127
6	649	-624
7	802	-798
8	83	99
9	384	-393
10	277	290
K= 3, L= 4		
1	1214	1268
2	424	434
3	503	-496
4	284	263
5	1044	-1021
6	261	-260
7	304	-309
8	312	-301
9	349	342
10	44*	-45
K= 3, L= 5		
0	1224	1290
1	358	352
2	42*	82
3	280	281
4	523	-521
5	132	-145
6	657	-639
7	150	-126
8	54*	50
9	161	-156
10	369	376
K= 3, L= 6		
1	345	375
2	171	158
3	136	-151
4	51*	-65
5	480	-492
6	55*	-17
7	183	-195
8	92	-98
9	278	278
K= 3, L= 7		
0	634	664
1	195	186
2	81	89
3	163	139
4	424	-421
5	55*	31
6	336	-332
7	165	-168
8	48*	17
9	41*	-8
K= 3, L= 8		
1	661	674
2	54*	93
3	339	-329
4	55*	33
5	549	-549
6	54*	-64
7	191	-196
8	43*	51
K= 3, L= 9		
0	771	776
1	110	-120
2	128	115
3	102	-92
4	425	-416
5	53*	-25
6	421	-423
7	99	78
K= 3, L= 10		
1	371	365
2	374	-363
3	156	-148
4	146	-162
5	372	-362
6	97	117
K= 3, L= 11		
0	271	265
1	210	-202
2	49*	52
3	307	-297
4	173	-169
K= 3, L= 12		
1	126	109
2	232	-233
K= 4, L= 0		
0	830	754
2	303	282
4	640	-585
6	611	-595
8	78	76
10	499	480
K= 4, L= 1		
1	423	419
2	593	569
3	33*	-45
4	277	264
5	870	-854
6	91	-70
7	171	-177
8	159	-144
9	260	260
10	88	75
K= 4, L= 2		
0	2143	2353
1	99	72
2	848	820
3	331	-319
4	1466	-1414
5	46*	42
6	810	-808
7	102	120
8	142	119
9	177	173
10	510	523
K= 4, L= 3		
1	1211	1262
2	789	-784
3	269	-250
4	636	-623
5	1212	-1210
6	394	378
7	244	-258
8	439	452
9	405	406
10	65	44
K= 4, L= 4		
0	621	649
1	1013	-1043
2	38*	33
3	1133	-1148
4	278	-266
5	48*	37
6	493	-491
7	561	558
8	131	125
9	338	354
10	256	251
K= 4, L= 5		
1	66	-51
2	916	-949
3	78	-86
4	598	-601
5	88	-78
6	465	458
7	55*	-19
8	454	448
9	49*	54
10	94	75
K= 4, L= 6		
0	91	118
1	184	-182
2	92	94
3	370	-384
4	151	-144
5	127	-138
6	167	-148
7	394	391
8	51*	61
9	229	228
K= 4, L= 7		
1	131	127
2	519	-530
3	52*	5
4	298	-308
5	175	-170
6	245	251
7	53*	-48
8	346	351
9	96	94
K= 4, L= 8		
0	222	217
1	525	-534
2	54*	23
3	623	-617
4	155	-154
5	55*	-48
6	53*	16
7	444	439
8	42*	6
K= 4, L= 9		
1	142	-141
2	573	-573
3	55*	-7
4	424	-428
5	187	194
6	277	286
7	64	64
K= 4, L= 10		
0	428	-435
1	194	-207
2	54*	-61
3	419	-419
4	216	215
5	47*	27
6	202	203
K= 4, L= 11		
1	242	-245
2	233	-239
3	71	58
4	44*	-75
K= 4, L= 12		
0	186	-185
1	93	-65
2	91	-96
K= 5, L= 0		
2	920	-895
4	1085	-1065
6	343	332
8	487	480
10	46*	51
K= 5, L= 1		
0	279	311
1	296	-315
2	169	142
3	1620	-1542
4	155	-152
5	73	-88
6	79	-83
7	563	545
8	54*	-3
9	276	266
10	90	62
K= 5, L= 2		
1	437	-452
2	1476	-1442
3	36*	-53
4	602	-600
5	439	422
6	342	335
7	83	93
8	457	466
9	112	-120
10	40*	37
K= 5, L= 3		
0	863	-875
1	602	-640
2	746	-751
3	1493	-1507
4	641	639
5	47*	-64
6	536	526
7	551	555
8	103	-127
9	355	350
10	197	-223
K= 5, L= 4		
1	1061	-1106
2	515	-527
3	396	365
4	496	-488
5	632	615
6	139	142
7	155	160
8	321	315
9	244	-253
10	42*	24
K= 5, L= 5		
0	951	-990
1	42*	-38
2	395	-408
3	382	-382
4	561	570
5	52*	42
6	388	376
7	77	66
8	53*	12
9	156	154
K= 5, L= 6		
1	639	-682
2	134	-149
3	49*	58
4	200	-185
5	521	524
6	72	66
7	249	260
8	94	105
9	249	-254
K= 5, L= 7		
0	541	-576
1	50*	-44
2	480	-480
3	235	-241
4	514	504
5	76	-72
6	359	362
7	155	174
8	47*	-17
K= 5, L= 8		
1	640	-646
2	54*	20
3	122	116
4	55*	-17
5	544	562
6	53*	73
7	153	138
8	66	-51
K= 5, L= 9		
0	524	-527
1	78	71
2	332	-330
3	235	233
4	390	383
5	52*	37
6	374	366
7	128	-111
K= 5, L= 10		
1	344	-344
2	276	272
3	52*	40
4	191	189
5	345	344
6	121	-119
K= 5, L= 11		
0	241	-245
1	82	85
2	88	-97
3	294	271
4	152	158
K= 5, L= 12		
1	162	-144
K= 6, L= 0		
0	819	-806
2	645	-661
4	1198	1139
6	942	934
8	97	-58
10	598	-585
K= 6, L= 1		
1	918	-901
2	331	331
3	726	663
4	235	237
5	939	937
6	220	-179
7	443	438
8	229	-208
9	514	-512
10	46*	-10
K= 6, L= 2		
0	355	-395
1	92	-32
2	114	-105
3	531	508
4	901	859
5	79	79
6	485	485
7	346	-336
8	105	-93
9	264	-248
10	437	-454
K= 6, L= 3		
1	410	-400
2	669	675
3	602	573
4	251	250
5	376	399
6	204	-195
7	297	294
8	514	-519
9	312	-320
10	44*	-18
K= 6, L= 4		
0	829	-860
1	200	211
2	110	-115
3	386	380
4	688	679
5	50*	-69
6	396	392
7	397	-398
8	54*	-53
9	285	-298
10	325	-323
K= 6, L= 5		
1	361	-375
2	542	578
3	258	269
4	119	130
5	378	372
6	175	-174
7	55*	94
8	502	-519
9	169	-170
K= 6, L= 6		
0	251	-233
1	482	496
2	138	-136
3	776	787
4	326	324
5	54*	-53
6	143	124
7	458	-503
8	75	-62
9	355	-367
K= 6, L= 7		
1	118	-89
2	755	783
3	70	59
4	310	320
5	134	130
6	318	-325
7	82	-81
8	502	-495
K= 6, L= 8		
0	133	123
1	278	283
2	73	72
3	529	538
4	89	-81
5	106	-106
6	125	-119
7	366	-374
8	40*	-13
K= 6, L= 9		
1	136	163
2	435	443
3	55*	-42
4	186	167
5	195	-212
6	245	-246
7	70	-63
K= 6, L= 10		
0	141	130
1	205	203
2	100	76
3	311	301
4	211	-188
5	69	-66
K= 6, L= 11		
1	163	161
2	303	286
3	102	-104
4	68	68
K= 6, L= 12		
0	255	243
K= 7, L= 0		
2	1894	1879
4	1261	1257
6	597	-583
8	412	-415
10	88	-102
K= 7, L= 1		
0	740	763
1	607	623
2	774	745
3	1939	1868
4	745	-712
5	46*	-14
6	370	-358
7	466	-451
8	85	88
9	309	-311
10	159	142
K= 7, L= 2		
1	836	879
2	766	747
3	35*	-23
4	556	576
5	690	-670
6	123	-138
7	76	57
8	312	-321
9	69	45
10	44*	26
K= 7, L= 3		
0	545	547
1	445	425
2	451	439
3	358	407
4	470	-456
5	104	94
6	52*	-70
7	75	-90
8	54*	-15
9	207	-204
10	125	131
K= 7, L= 4		
1	490	511
2	826	829
3	44*	35
4	416	416
5	293	-297
6	53*	-43
7	112	-91
8	407	-421
9	140	138
10	59	50
K= 7, L= 5		
0	1061	1091
1	407	437
2	261	264
3	718	723
4	357	-366
5	249	248
6	265	-289
7	394	-405
8	52*	-32
9	198	-206
K= 7, L= 6		
1	1065	1088
2	305	306
3	229	-240
4	268	272
5	532	-603
6	88	-80
7	297	-299
8	208	-205
9	298	308
K= 7, L= 7		
0	984	1012
1	193	-189
2	432	434
3	54*	27
4	456	-458
5	55*	-12
6	548	-556
7	51*	12
8	77	75
K= 7, L= 8		
1	466	484
2	308	-304
3	55*	65
4	55*	-51
5	440	-450
6	51*	36
7	176	-183
K= 7, L= 9		
0	300	300
1	160	-156
2	249	257
3	141	-124
4	222	-226
5	79	-65
6	216	-210
K= 7, L= 10		
1	280	287
2	52*	-56
3	51*	17
4	157	-161
5	254	-258
K= 7, L= 11		
0	271	270
1	47*	-63
2	184	195
3	208	-191
4	210	-215
K= 8, L= 0		
0	1214	1207
2	223	248
4	1238	-1240
6	855	-853
8	157	216
10	489	491
K= 8, L= 1		
1	553	556
2	502	-477
3	188	-190
4	645	-622
5	1123	-1111
6	252	256
7	367	-369
8	388	394
9	480	486
10	66	-22
K= 8, L= 2		
0	101	124
1	593	-568
2	169	168
3	525	-509
4	468	-464
5	48*	-27
6	591	-555
7	450	495
8	54*	43
9	145	145
10	374	368
K= 8, L= 3		
1	234	247
2	84	67
3	383	-388
4	108	-58
5	385	-411
6	95	96
7	95	-96
8	328	323
9	193	196
10	41*	30
K= 8, L= 4		
0	225	275
1	101	79
2	258	251
3	221	-215
4	698	-692
5	131	-144
6	152	-166
7	361	366
8	52*	-40
9	265	293
K= 8, L= 5		
1	606	614
2	624	-638
3	231	-232
4	366	-371
5	733	-731
6	353	338
7	54*	-66
8	491	499
9	190	207
K= 8, L= 6		
0	230	235
1	414	-414
2	211	207
3	831	-839
4	464	-466
5	197	215
6	55*	-44
7	446	445
8	48*	72
9	316	330
K= 8, L= 7		
1	100	-100
2	623	-640
3	54*	-40
4	429	-435
5	55*	79
6	438	440
7	124	138
8	387	382
K= 8, L= 8		
0	160	-173
1	82	-68
2	55*	-67
3	588	-592
4	156	159
5	154	168
6	123	109
7	305	308
K= 8, L= 9		
1	55*	-46
2	315	-315
3	54*	-5
4	146	-124
5	140	154
6	244	233
K= 8, L= 10		
0	52*	43
1	210	-215
2	114	-121
3	292	-283
4	168	161
5	84	70
K= 8, L= 11		
1	152	-152
2	308	-297
3	85	80
K= 9, L= 0		
2	1725	-1704
4	508	-504
6	428	446
8	371	382
10	80	28
K= 9, L= 1		
0	37*	-26
1	372	-370
2	625	-564
3	1270	-1240
4	432	429
5	49*	50
6	99	104
7	341	345
8	54*	-45
9	241	248
10	72	-84
K= 9, L= 2		
1	636	-645
2	1048	-1042
3	246	227
4	476	-483
5	401	407
6	85	66
7	86	-76
8	479	471
9	49*	-52
10	41*	10
K= 9, L= 3		
0	368	-393
1	670	-675
2	373	-351
3	589	-601
4	427	419
5	137	-135
6	100	66
7	287	279
8	53*	19
9	233	231
K= 9, L= 4		
1	636	-664
2	737	-725
3	147	116
4	193	-200
5	298	288
6	100	-37
7	247	248
8	404	427
9	177	-160
K= 9, L= 5		
0	1044	-1093
1	353	-358
2	325	-325
3	314	-310
4	500	504
5	261	-236
6	415	418
7	312	329
8	50*	-40
9	130	123
K= 9, L= 6		
1	795	-801
2	168	-181
3	324	318
4	181	-189
5	309	354
6	93	79
7	266	279
8	173	175
K= 9, L= 7		
0	694	-701
1	167	163
2	207	-205
3	55*	26
4	239	253
5	54*	-2
6	420	420
7	76	-41
8	84	-88
K= 9, L= 8		
1	401	-420
2	165	159
3	55*	20
4	54*	-38
5	340	350
6	49*	4
7	222	221
K= 9, L= 9		
0	360	-355
1	108	91
2	239	-239
3	71	46
4	253	243
5	48*	49
6	247	260
K= 9, L= 10		
1	307	-305
2	118	107
3	48*	46
4	137	125
5	259	260
K= 9, L= 11		
0	241	-233
1	109	101
2	158	-144
3	220	217
K= 10, L= 0		
0	196	-217
2	329	-310
4	492	491
6	464	464
8	53*	-62
10	330	-346
K= 10, L= 1		
1	666	-676
2	124	-95
3	305	311
4	47*	-60
5	672	669
6	53*	-27
7	292	302
8	53*	-56
9	378	-385
10	40*	32
K= 10, L= 2		
0	743	-784
1	70	-32
2	243	-248
3	104	100
4	576	570
5	84	90
6	602	590
7	176	-189
8	53*	-54
9	89	-117
K= 10, L= 3		
1	761	-801
2	517	534
3	272	282
4	330	343
5	694	704
6	134	-145
7	289	301
8	395	-401
9	285	-300
K= 10, L= 4		
0	490	-496
1	312	332
2	253	-248
3	673	684
4	562	559
5	79	54
6	222	229
7	410	-417
8	50*	-59
9	265	-275
K= 10, L= 5		
1	167	-164
2	577	598
3	168	162
4	368	371
5	175	174
6	161	-195
7	53*	-31
8	435	-446
9	77	-85
K= 10, L= 6		
0	52*	-65
1	262	281
2	53*	-29
3	573	565
4	112	96
5	55*	-63
6	54*	22
7	374	-369
8	44*	-27
K= 10, L= 7		
1	153	-150
2	395	404
3	88	83
4	188	205
5	105	106
6	240	-236
7	46*	-16
K= 10, L= 8		
0	55*	-83
1	195	193
2	55*	23
3	492	491
4	54*	7
5	51*	-63
6	47*	-32
7	246	-253
K= 10, L= 9		
1	72	56
2	486	490
3	52*	-5
4	244	228
5	118	-114
6	175	-181
K= 10, L= 10		
0	198	177
1	275	272
2	109	102
3	300	301
4	191	-190
K= 10, L= 11		
1	208	208
2	202	208
K= 11, L= 0		
2	351	363
4	509	489
6	325	-310
8	299	-313
K= 11, L= 1		
0	307	-287
1	460	466
2	132	-109
3	595	602
4	49*	18
5	52*	-32
6	185	195
7	411	-401
8	52*	-3
9	187	-192
K= 11, L= 2		
1	43*	-37
2	975	1003
3	108	97
4	607	584
5	128	-145
6	474	-473
7	54*	-18
8	422	-440
9	46*	20
K= 11, L= 3		
0	951	952
1	397	417
2	182	163
3	809	811
4	448	-441
5	115	102
6	453	-449
7	457	-479
8	83	107
9	269	-271
K= 11, L= 4		
1	744	766
2	496	497
3	248	-253
4	321	331
5	688	-681
6	231	-227
7	175	-98
8	338	-332
9	188	204
K= 11, L= 5		
0	555	565
1	51*	-45
2	190	192
3	88	-45
4	386	-398
5	55*	-48
6	339	-341
7	74	-43
8	96	65
K= 11, L= 6		
1	293	303
2	74	-58
3	55*	-22
4	82	84
5	404	-405
6	53*	-23
7	140	-148
8	76	-80
K= 11, L= 7		
0	404	407
1	55*	-11
2	112	115
3	218	245
4	286	-287
5	53*	1
6	249	-257
7	96	-93
K= 11, L= 8		
1	355	353
2	142	136
3	54*	-65
4	53*	6
5	397	-395
6	70	-47
K= 11, L= 9		
0	413	402
1	70	-68
2	230	235
3	138	-154
4	338	-344
5	44*	-22
K= 11, L= 10		
1	229	253
2	231	-219
3	44*	-66
4	139	-130
K= 11, L= 11		
0	128	144
K= 12, L= 0		
0	568	585
2	296	286
4	228	-247
6	365	-365
8	85	-46
K= 12, L= 1		
1	789	791
2	78	81
3	191	-187
4	163	139
5	485	-482
6	145	-135
7	252	-254
8	70	-34
9	228	236
K= 12, L= 2		
0	1287	1299
1	140	-107
2	125	138
3	182	-168
4	474	-466
5	53*	-70
6	494	-494
7	106	73
8	125	104
9	127	132
K= 12, L= 3		
1	757	775
2	547	-544
3	151	-151
4	383	-391
5	624	-651
6	140	140
7	175	-182
8	308	301
9	279	292
K= 12, L= 4		
0	396	407
1	545	-563
2	133	148
3	659	-670
4	124	-138
5	54*	-8
6	274	-295
7	337	353
8	103	77
K= 12, L= 5		
1	215	222
2	498	-503
3	119	-106
4	273	-275
5	92	-98
6	144	137
7	50*	-48
8	269	266
K= 12, L= 6		
0	199	208
1	265	-256
2	55*	-50
3	248	-252
4	55*	29
5	219	-216
6	78	-90
7	279	285
8	40*	14
K= 12, L= 7		
1	169	156
2	472	-468
3	55*	-25
4	269	-272
5	146	-137
6	98	119
7	55	-63
K= 12, L= 8		
0	85	73
1	411	-417
2	54*	22
3	442	-424
4	51*	-101
5	48*	-34
6	65	42
K= 12, L= 9		
1	133	-142
2	374	-403
3	68	16
4	216	-212
5	126	123
K= 12, L= 10		
0	338	-337
1	195	-208
2	44*	-38
3	208	-226
K= 13, L= 0		
2	684	-697
4	281	-282
6	256	243
8	535	539
K= 13, L= 1		
0	132	-127
1	368	-367
2	84	-49
3	439	-440
4	52*	70
5	100	-60
6	101	94
7	491	488
8	49*	52
9	228	237
K= 13, L= 2		
1	250	-254
2	706	-714
3	166	148
4	257	-262
5	232	210
6	350	362
7	151	159
8	414	418
9	135	-126
K= 13, L= 3		
0	349	-359
1	171	-173
2	50*	20
3	397	-410
4	219	228
5	87	80
6	345	345
7	374	391
8	103	-121
K= 13, L= 4		
1	320	-326
2	223	-238
3	220	218
4	123	-150
5	389	387
6	158	161
7	120	116
8	224	237
K= 13, L= 5		
0	281	-276
1	177	-195
2	149	-161
3	208	-213
4	405	413
5	54*	6
6	298	294
7	165	147
8	41*	-92
K= 13, L= 6		
1	245	-222
2	262	-255
3	80	85
4	55*	-84
5	327	339
6	126	119
7	149	158
K= 13, L= 7		
0	449	-470
1	55*	-50
2	132	-108
3	54*	-6
4	348	332
5	50*	17
6	317	325
K= 13, L= 8		
1	382	-377
2	53*	-10
3	238	235
4	102	66
5	343	355
6	40*	-2
K= 13, L= 9		
0	286	-284
1	50*	26
2	48*	-34
3	177	176
4	225	228

Table 3 (*cont.*)

H	FO	FC
K= 13, L= 10		
1	115	-129
2	194	193
K= 14, L= 0		
0	1007	-1018
2	412	-395
4	441	443
6	411	409
8	63	-14
K= 14, L= 1		
1	839	-850
2	445	442
3	79	105
4	210	182
5	571	584
6	54*	-30
7	211	156
8	178	-166
9	190	-197
K= 14, L= 2		
0	450	-649
1	397	368
2	261	-239
3	343	339
4	208	226
5	87	57
6	276	298
7	149	-137
8	101	-89
K= 14, L= 3		
1	318	-335
2	325	310
3	53*	-87
4	185	178
5	325	352
6	154	-111
7	50*	-20
8	79	-91
K= 14, L= 4		
0	218	-232
1	178	167
2	284	-294
3	304	322
4	185	198
5	54*	37
6	205	225
7	156	-150
8	82	-72
K= 14, L= 5		
1	360	-382
2	387	381
3	55*	-21
4	366	350
5	357	352
6	162	-139
7	107	119
K= 14, L= 6		
0	306	-331
1	328	302
2	55*	-75
3	506	524
4	113	125
5	114	116
6	176	164
7	299	-295
K= 14, L= 7		
1	54*	7
2	423	426
3	53*	27
4	388	396
5	48*	-17
6	139	-155
K= 14, L= 8		
0	219	212
1	233	232
2	77	45
3	396	394
4	98	-103
5	42*	60
K= 14, L= 9		
1	114	95
2	232	226
3	44*	-7
4	149	147
K= 14, L= 10		
0	121	109
1	89	86
K= 15, L= 0		
2	727	713
4	392	365
6	357	-358
8	480	-476
K= 15, L= 1		
0	356	347
1	469	472
2	145	138
3	391	393
4	270	-269
5	55*	45
6	246	-238
7	460	-463
8	44*	29
K= 15, L= 2		
1	233	225
2	201	203
3	87	-102
4	249	247
5	261	-279
6	276	-260
7	118	-108
8	258	-275
K= 15, L= 3		
0	213	220
1	191	199
2	121	-90
3	54*	76
4	119	-115
5	54*	4
6	179	-189
7	199	-204
8	41*	25
K= 15, L= 4		
1	54*	-51
2	406	407
3	55*	50
4	113	153
5	259	-255
6	204	-207
7	46*	-75
K= 15, L= 5		
0	314	311
1	340	338
2	103	41
3	217	225
4	240	-233
5	52*	30
6	314	-308
7	250	-259
K= 15, L= 6		
1	420	409
2	271	269
3	209	-215
4	81	27
5	452	-462
6	78	-90
K= 15, L= 7		
0	560	566
1	71	53
2	85	-67
3	51*	-55
4	240	-265
5	79	-73
K= 15, L= 8		
1	193	190
2	152	-138
3	142	-154
4	95	-88
K= 15, L= 9		
0	289	289
1	131	-106
2	43*	-54
3	69	-37
K= 16, L= 0		
0	1191	1171
2	53*	76
4	385	-390
6	346	-352
8	116	110
= 16, L= 1		
1	619	615
2	260	-246
3	170	-186
4	55*	-43
5	384	-344
6	52*	23
7	154	-154
8	150	142
K= 16, L= 2		
0	455	447
1	471	-453
2	172	116
3	166	-165
4	54*	-90
5	54*	-21
6	260	-265
7	151	154
8	40*	48
K= 16, L= 3		
1	361	366
2	323	-314
3	54*	-23
4	105	-65
5	288	-299
6	50*	17
7	45*	-58
K= 16, L= 4		
0	296	298
1	157	-158
2	286	275
3	234	-226
4	249	-269
5	101	-102
6	206	-203
7	136	141
K= 16, L= 5		
1	202	229
2	417	-394
3	54*	25
4	256	-261
5	297	-301
6	123	125
K= 16, L= 6		
0	109	96
1	292	-287
2	54*	18
3	500	-500
4	67	-32
5	47*	-17
6	41*	-67
K= 16, L= 7		
1	52*	-24
2	304	-308
3	49*	7
4	255	-267
5	57	38
K= 16, L= 8		
0	70	-81
1	81	-78
2	46*	-60
3	219	-210
4	102	103
K= 16, L= 9		
1	83	-60
K= 17, L= 0		
2	450	-457
4	285	-271
6	265	278
K= 17, L= 1		
0	172	184
1	54*	41
2	113	-89
3	421	-427
4	119	60
5	53*	7
6	50*	23
7	256	275
K= 17, L= 2		
1	122	-105
2	259	-256
3	54*	34
4	311	-316
5	188	185
6	180	191
7	44*	20
K= 17, L= 3		
0	96	-75
1	206	-218
2	190	-188
3	393	-402
4	218	200
5	51*	-14
6	178	172
7	236	249
K= 17, L= 4		
1	185	-182
2	329	-349
3	101	-36
4	145	-122
5	327	318
6	130	132
K= 17, L= 5		
0	278	-267
1	164	-179
2	176	-182
3	53*	-83
4	284	306
5	47*	10
6	278	264
K= 17, L= 6		
1	281	-257
2	77	-86
3	133	141
4	47*	31
5	263	266
K= 17, L= 7		
0	296	-310
1	45*	-46
2	48*	-20
3	46*	6
4	175	183
K= 17, L= 8		
1	163	-170
2	43*	-29
3	111	91
K= 18, L= 0		
0	459	-452
2	54*	57
4	163	145
6	188	175
K= 18, L= 1		
1	327	-336
2	55*	15
3	175	154
4	106	-89
5	267	268
6	47*	23
7	76	90
K= 18, L= 2		
0	569	-577
1	173	165
2	106	-85
3	54*	-29
4	255	276
5	50*	25
6	256	265
7	40*	-56
K= 18, L= 3		
1	349	-356
2	297	301
3	53*	76
4	136	131
5	359	364
6	57	-83
K= 18, L= 4		
0	207	-190
1	309	311
2	117	-117
3	352	343
4	191	201
5	47*	10
6	106	126
K= 18, L= 5		
1	53*	-31
2	329	311
3	50*	-19
4	179	181
5	106	97
K= 18, L= 6		
0	97	41
1	157	122
2	49*	-12
3	224	206
4	44*	-20
K= 18, L= 7		
1	46*	-27
2	164	146
3	42*	-6
K= 18, L= 8		
0	56	-60
1	104	116
K= 19, L= 0		
2	86	97
4	201	216
6	44*	-48
K= 19, L= 1		
0	54*	16
1	273	246
2	54*	9
3	285	291
4	51*	12
5	106	83
6	44*	-13
K= 19, L= 2		
1	54*	-24
2	446	453
3	52*	98
4	276	283
5	90	-69
6	163	-170
K= 19, L= 3		
0	296	282
1	243	242
2	117	96
3	284	286
4	122	-134
5	46*	43
6	196	-201
K= 19, L= 4		
1	336	331
2	223	223
3	106	-92
4	47*	77
5	282	-287
K= 19, L= 5		
0	308	304
1	80	98
2	82	70
3	47*	51
4	146	-144
5	40*	-12
K= 19, L= 6		
1	170	148
2	46*	53
3	44*	-26
4	40*	48
K= 19, L= 7		
0	282	272
1	42*	42
2	41*	46
K= 20, L= 0		
0	118	56
2	181	178
4	252	-237
6	172	-177
K= 20, L= 1		
1	193	212
2	52*	-56
3	51*	-102
4	49*	-13
5	300	-283
6	39*	-7
K= 20, L= 2		
0	286	286
1	52*	31
2	150	121
3	129	-144
4	333	-338
5	44*	12
K= 20, L= 3		
1	153	158
2	168	-166
3	49*	-92
4	103	-116
5	218	-231
K= 20, L= 4		
0	75	58
1	129	-122
2	48*	40
3	268	-267
4	43*	-95
K= 20, L= 5		
1	72	62
2	195	-180
3	67	-26
4	94	-75
K= 20, L= 6		
0	44*	71
1	64	-30
2	42*	27
K= 21, L= 0		
2	361	-354
4	256	-245
K= 21, L= 1		
0	51*	-81
1	180	-158
2	50*	-46
3	319	-301
4	45*	19
5	40*	-63
K= 21, L= 2		
1	149	-171
2	273	-269
3	47*	12
4	140	-111
5	142	141
K= 21, L= 3		
0	159	-174
1	138	-123
2	100	-79
3	214	-212
4	98	91
K= 21, L= 4		
1	159	-156
2	130	-133
3	84	62
K= 21, L= 5		
0	239	-234
1	75	-67
2	94	-86
K= 22, L= 0		
0	339	-318
2	94	-75
4	248	255
K= 22, L= 1		
1	149	-152
2	83	72
3	44*	77
4	83	68
K= 22, L= 2		
0	121	-123
1	46*	81
2	45*	25
3	156	146
4	130	120
K= 22, L= 3		
1	83	-80
2	101	78
3	89	77
K= 22, L= 4		
0	139	-160
1	90	54
2	40*	-9
K= 23, L= 0		
2	259	264
K= 23, L= 1		
0	109	92
1	117	106
2	42*	64
K= 23, L= 2		
1	100	100
2	131	116
K= 23, L= 3		
0	119	109
1	79	72

Description of structure and discussion

A perspective view of the structure is shown in Fig. 1. The thermal motion ellipsoids (program by Huml & Hlavatá, 1971; Šoler, 1973) of the independent atoms can be seen in Fig. 2. The intramolecular geometry is summarized in Fig. 3 and Table 4. The bond lengths and angles have not been corrected for thermal

Table 4. *Some intramolecular distances and bond angles, with e.s.d.'s*

(See also Fig. 3.)

H(C7)—C(7)	0·93 (9) Å
H(C11)—C(11)	1·03 (9)
H(C121)-C(12)	0·98 (12)
C(10)—Rh—C(10$^{\mathrm{i}}$)	81·8 (3)°
C(11)—Rh—C(11$^{\mathrm{i}}$)	83·2 (3)
C(10)—Rh—C(11)	38·5 (2)
C(9)—Rh—C(12)	63·3 (3)
H(C7)—C(7)—C(8)	117 (5)
H(C11)—C(11)-C(10)	112 (5)
H(C11)—C(11)-C(12)	119 (5)
H(C121)-C(12)-C(11)	102 (7)
H(C121)-C(12)-C(12$^{\mathrm{i}}$)	102 (7)

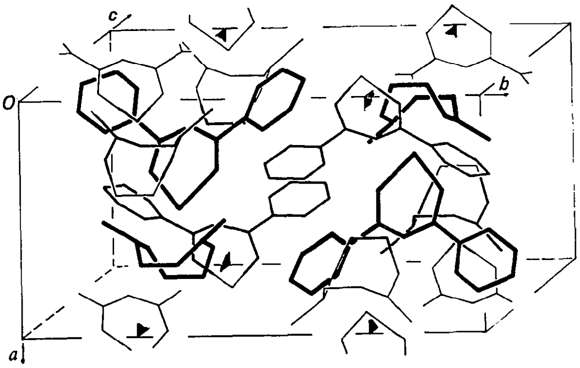


Fig. 1. Perspective view of the structure.

Table 5. *Equations of weighted least-squares planes*

Equations are given in the form $pX+qY+rZ-s=0$, where X, Y, and Z are coordinates in Å referred to the directions of the cell axes and origin. The deviations of certain atoms from these planes are given in square brackets. The lower-case roman numeral superscript i to the arabic numeral is to distinguish the mirror-related atoms and planes. The χ^2 value is to test the planarity.

Plane	Defined by	p	q	r	s	χ^2
1	C(9), C(10), C(11), C(12) [C(9), 0·0021, C(10) −0·0017, C(11) 0·0022, C(12) −0·0021]	0·4884	−0·8471	−0·2097	−0·6703	0·27
2	C(9), C(9$^{\mathrm{i}}$), C(12)	0·9226	0·0000	−0·3858	5·7801	0
3	C(7), C(8), C(8$^{\mathrm{i}}$), O, O$^{\mathrm{i}}$, Rh [C(7) 0·0312, C(8) 0·0182, O −0·0306, Rh 0·0004, H(7) 0·1960, C(1) 0·1588]	−0·4687	0·0000	−0·8833	−7·0472	208·90
4	C(1), C(2), C(3), C(4), C(5), C(6) [C(1) 0·0013, C(2) −0·0072, C(3) 0·0150, C(4) −0·0074, C(5) −0·0029, C(6), 0·0019, C(8) 0·0256]	−0·7034	0·3279	−0·6306	−4·9800	4·11

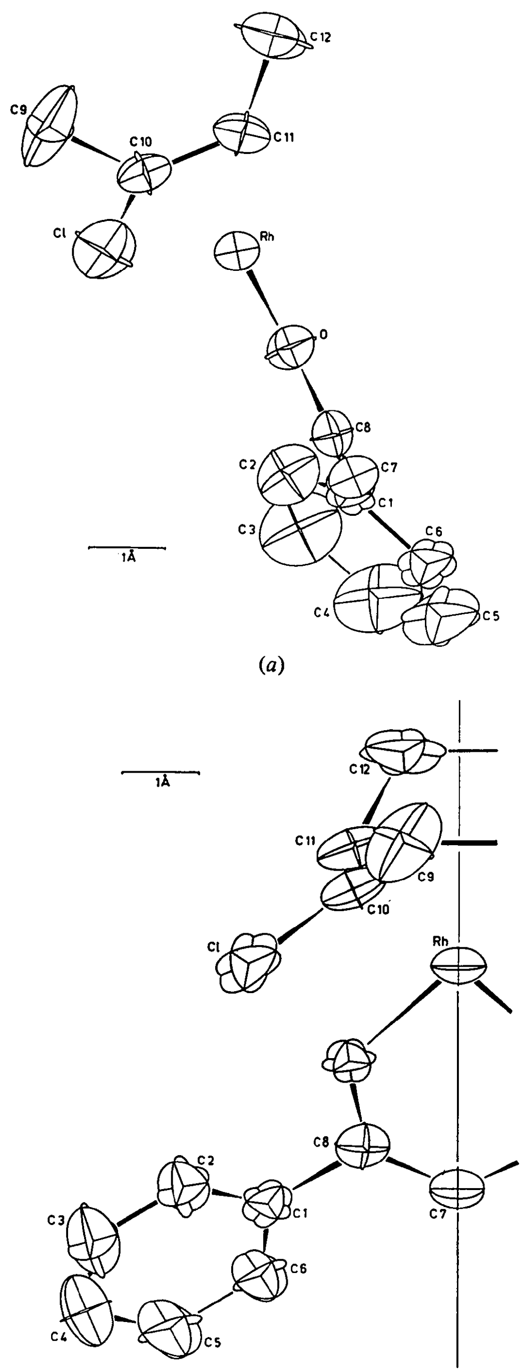
Some dihedral angles between the planes

∠1−2	∠2−3	∠3−4	∠4−4$^{\mathrm{i}}$
57·9°	95·3°	27·5°	38·1°

motion. The equations of weighted mean planes calculated by the least-squares method and some dihedral angles of these planes are given in Table 5.

Central atom

The coordination bonds Rh–O, 2·034 (3) Å, and distances Rh–C(10), 2·104 (6), and Rh–C(11), 2·088 (6) Å, correspond to the usual values found in the literature (Thomas & Stanko, 1973). This result is also in good accordance with the n.m.r. spectra where no spin interaction with the Rh nucleus has been observed for any of the chelates measured (Kříž & Bouchal, 1973). The angle O–Rh–O^{i}, 88·6 (2)° and the rhodium angle formed with the centres of the double bonds C(10)=C(11) and C(10^{i})=C(11^{i}), 88·5 (6)° are close to 90° (*cf.* Ibers & Snyder, 1962) and indicate that the prevailing orbitals that participate in the hybridization of the Rh orbitals are $4p_x$, $4p_y$ and $4d$ of a suitable symmetry; the orbital $5s$ participates to a much smaller extent (superscript i designates an atom mirror-coupled with the independent atom).



(*a*)

(*b*)

Fig. 2. Thermal motion ellipsoids of 50% probability. (*a*) Projection along the *y* axis. (*b*) Projection along the *z* axis.

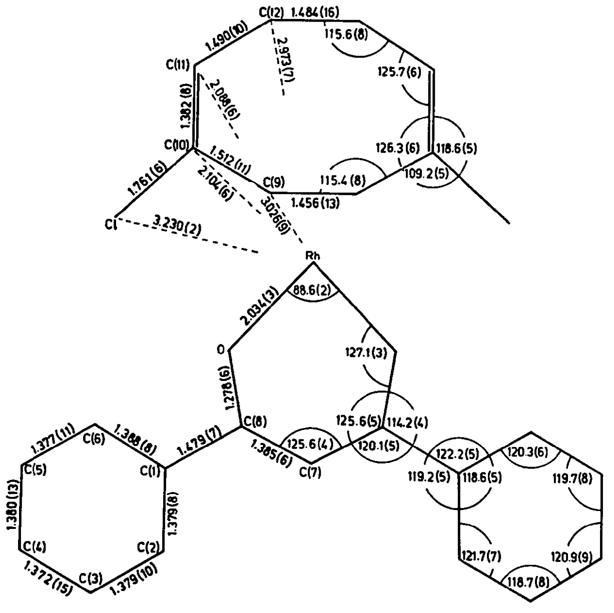


Fig. 3. The bond lengths (Å) and some valence angles (°) of the non-hydrogen atoms. Dashed lines are the C–Rh distances.

Chelate ring

A slight shifting of the Rh atom away from the plane of the chelate residue [O, O^{i}, C(8), C(8^{i}), C(7)] which allows a better overlapping of non-hybridized $4p_z$, $4d_{xz}$ and $4d_{yz}$ orbitals with the π-system of the chelate ring is in agreement with the hybridization of the Rh orbitals described above. Although small (0·09 Å), this deviation still seems to be significant. The value of the χ^2 test for the whole chelate ring is 208·90, although for the chelate residue it is $\chi^2 = 1{\cdot}90$. The extension of the C(8)=O, 1·278 (6) Å, bond and shortening of the C(7)–C(8), 1·385 (6) Å, bond indicate a strong conjugation which forms a pseudoaromatic system from the chelate ring. The slight widening of the valence angles C(7)–C(8)–O, 125·6 (5)°, and C(8)–O–Rh, 127·1 (3)°, is probably necessitated by the tendency of the orbitals of the central atom to attain symmetry (the coordination bond length Rh–O, 2·034 (3) Å, and the distance of Rh from the centre of the double bond of diene, 1·979 (6) Å, are close to each other). The fact that the Rh–diene bond obviously dictates to a certain extent the configuration of the chelate suggests that this bond is at least as strong as that between Rh and the chelating agent. This is also in accordance with the fragmentation of the mass spectrum (Ryska, Bouchal & Hrabák, 1973).

Phenyl substituents

The weighted average value of the C–C bond in the phenyl ring is 1·383 (2) Å, which is less than the normal value 1·394 Å (Sutton, 1965) for the aromatic C–C bond. The fact that $\chi^2=1{\cdot}61$ and $P=90\%$ ($\upsilon=5$) for the phenyl ring clearly indicate that the observed differences between the bonds are insignificant. However, some of the differences between the angles of the phenyl ring lie in the significance range [weighted average 119·9 (3)°, maximum deviation 1·8° ($\sigma=0{\cdot}7°$), $\chi^2=17{\cdot}36$, $\upsilon=5$, $P<0{\cdot}5\%$]. The planarity test ($\chi^2=4{\cdot}11$) shows that the σ values calculated by the least-squares method are realistic and may be used for testing the other optimal planes in the structure. The normal of the plane of the phenyl ring is somewhat inclined to the direction of the C(8)–C(1) bond forming with it an angle of 89·1°. The angle between the direction of the C(8)–C(1) bond and the direction of the connecting line C(1)–C(4) is 2·0°. The angle between the normal of the chelate ring and the direction of the C(8)–C(1) bond is 95·5°. A slight shortening of the C(8)–C(1) bond indicates a certain, though incomplete conjugation. This incomplete conjugation is reflected in a marked rotation of the planes of the aromatic rings against the plane of the chelate ring (27·5°) and in an angle of 38·1° formed between the planes of the aromatic rings. However, as can be proved by calculations, the measure of conjugation is still high enough (approximately 90%).

The phenyl rings form zigzag ribbons in the structure. Some closest intermolecular distances are: C(1)–C(5^{ii}), 3·819 (10); C(2)–C(5^{ii}), 3·907 (11); C(3)–C(4^{iii}), 3·775 (15); C(4)–C(4^{iii}), 3·796 (16) Å (superscript ii means an atom related to an independent atom by a glide $x+\frac{1}{2},y,\frac{1}{2}-z$; iii stands for an atom related by a centre of symmetry).

Diene ring

The angle between the C(10)–C(11) double bond and the normal of the chelate ring is only 5·1°, which makes it possible for almost all 4*p* and 4*d* orbitals of the central metal atom to take part in the overlapping with the π-electrons of the C=C bond. The C=C bond is slightly extended, which corresponds to a decrease in the density of the π-electrons while preserving at the same time the sp^2 hybridization of the orbitals, as is also confirmed to a certain degree by the valency angles.

The distance of the Cl atom from the C(9), C(10), C(11), C(12) plane (plane 1) is 0·58 Å in the outwards direction. The direction of the Cl–C(10) bond forms an angle of 64·2° with the normal of plane 1. The distance of Cl from the plane of the phenyl ring is 0·30 Å; all intramolecular contacts of Cl with the C atoms of the phenyl ring are longer than 3·75 Å.

To illustrate the ordering of molecules in the structure, some shortest intermolecular contacts between the atoms of the diene ring are given: Cl–C(3^{iii}), 3·642 (9), Cl–C(4^{iii}), 3·846 (9); Cl–C(4^{iv}), 3·781 (10); C(8)–C(9^{ii}), 3·530 (9); C(8)–O^{i}, 2·939 (6); C(9)–Rh^{ii}, 3·873 (10) Å (superscript iv designates an atom conjugated with an independent one by a twofold screw axis).

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