Investigations of Alkaline-Earth β-Diketone Complexes.
I. The Crystal and Molecular Structure of
Bis(dimethylformamido)bis-(1,3-diphenyl-1,3-propanedionato)magnesium*

By Frederick J. Hollander, David H. Templeton and Allan Zalkin

Lawrence Berkeley Laboratory and Department of Chemistry, University of California, Berkeley, California 94720, U.S.A.

Bis(dimethylformamido)bis-(1,3-diphenyl-1,3-propanedionato)magnesium, $(DMF)_2(DPP)_2Mg$, $MgC_{36}H_{36}N_2O_6$, is monoclinic, space group $C2/c$, with $a=16\cdot893$ (3), $b=12\cdot853$ (2), $c=16\cdot927$ (3) Å, $\beta=117\cdot085$ (5)° at 23 °C, $Z=4$, $d_c=1\cdot10$ g cm^{-3}. The structure was determined by direct methods and refined to $R=0\cdot067$ for 1817 independent reflections measured with a scintillation counter using a θ–2θ scan. Each magnesium ion is octahedrally coordinated to the oxygen atoms of two DMF and two diketone molecules. The complex is monomeric and is situated on a twofold axis with the two DMF molecules *cis* to each other. Some remarks are made on distortions of ligand octahedra.

Introduction

Alkaline-earth ions are known to form complexes with various diketones, but few crystal structures of such compounds have been reported. We became interested in these substances and have investigated several complexes of 1,3-diphenyl-1,3-propanedione anion (DPP):

(−)
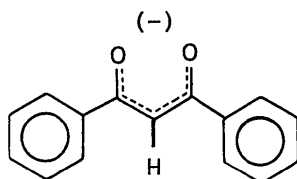
With Mg, Ca and Sr as cations we obtained suitable crystals only when certain solvent molecules were involved in the coordination.

This first paper reports the structure of Mg(DPP)₂(dimethylformamide)₂.

Experimental procedure

The compound was synthesized by combining an aqueous solution of magnesium acetate with an ethanolic solution of 1,3-diphenyl-1,3-propanedione (HDPP) in a strong NH₃/NH₄Cl, pH 10, buffer. All materials used were of reagent grade without further purification. A pale yellow precipitate formed immediately, and the mixture was stirred for several hours before the precipitate was filtered and dried in air. The filtrate, on standing, yielded another precipitate which was filtered and shown by its powder pattern to be identical with the first.

Attempts were made to recrystallize the compound from various organic solvents. Recrystallization from

ethanol yielded crystals which decomposed rapidly, even in sealed capillaries. Precession photographs of these crystals were so poor that they could not be indexed. Evaporation of dimethylformamide (DMF) solutions of the compound yielded crystals which were used throughout the rest of this investigation. These crystals tended to decompose when exposed to the atmosphere, but were stable in sealed capillaries and in a desiccator over Drierite. They were stable in air during one period of very low humidity, which suggests that moisture is responsible for the decomposition. A powder pattern of the recrystallized material was different from that of the original precipitate, but after exposure to air the diffraction lines of the original material slowly reappeared.

Several crystals were mounted in sealed quartz capillaries for study. Weissenberg and precession photographs indicated a monoclinic unit cell with absences hkl, $h+k\neq2n$ and $h0l$, $l\neq2n$. These absences are consistent with space groups Cc or $C2/c$, with b as the unique axis; solution of the structure established that $C2/c$ is the space group.

Further measurements were made with a General Electric XRD-5 manual three-circle diffractometer. Twelve high-angle reflections were carefully centered using Cu $K\alpha_1$ radiation ($\lambda=1\cdot54051$ Å). The cell dimensions were determined by refining the 2θ measurements, using program *TTHCEL*, a modification of a least-squares program supplied to us by H. Hope. The cell parameters and their standard deviations as given by least squares are $a=16\cdot893$ (3), $b=12\cdot853$ (2), $c=16\cdot927$ (3) Å, $\beta=117\cdot085$ (5)°, at 23 °C. The density was not measured because we failed to find a suitable flotation medium. The calculated density for an empirical formula Mg(DPP)₂ gave $Z=4$, $d_c=0\cdot96$ g cm^{-3} as the most reasonable result. The density based on the actual composition of Mg(DPP)₂(DMF)₂ with $Z=4$ is $d_c=1\cdot10$ g cm^{-3}.

Intensity data were collected from a crystal of approximate dimensions 0·25 × 0·25 × 0·30 mm sealed in a quartz capillary and mounted on a Picker/Nuclear four-circle automated diffractometer, using a scintillation counter and graphite-monochromated ($2\theta_m$ = 26·36°) Cu $K\alpha$ radiation. Intensities were collected

Table 1. F_o, $\sigma(F_o)$ *and final differences between* F_o *and* F_c *for* Mg(DPP)$_2$(DMF)$_2$ (×10·0)

Entries marked with an asterisk were given zero weight in least-squares calculations.

L	FOB	SG	DEL
H,K= 0, 0			
2	565	18	-71
4	881	27	-130
6	15	21	11*
8	380	12	-27
10	291	12	-12
12	151	7	-9
14	22	26	-13*
16	26	28	14*
H,K= 0, 2			
0	24	3	-7
1	689	34	13
2	731	98	-29
3	1557	217	-82
4	122	14	-9
5	347	45	-4
6	240	18	-19
7	348	14	-25
8	211	7	-9
9	130	5	-7
10	32	14	1
11	198	7	-1
12	28	33	27*
13	55	13	3
14	41	9	10
15	54	10	7
16	19	24	-4*
H,K= 0, 4			
0	88	4	-3
1	23	31	-7*
2	133	6	-3
3	414	42	-11
4	509	64	16
5	612	70	-1
6	508	40	-9
7	126	6	0
8	158	5	-6
9	23	25	8*
10	47	6	3
11	37	16	-11
12	56	10	7
13	31	32	-5*
14	76	7	0
15	33	36	12*
16	39	11	-1
H,K= 0, 6			
0	250	8	-2
1	95	4	3
2	576	19	37
3	260	10	33
4	74	10	-8
5	194	10	9
6	177	6	-2
7	89	5	6
8	204	8	-3
9	58	6	-13
10	61	6	6
11	20	31	7*
12	0	24	-11*
13	32	11	13
14	78	7	10
15	20	28	2*
H,K= 0, 8			
0	41	8	13
1	174	7	13
2	307	10	7
3	169	8	4
4	44	6	10
5	49	12	3
6	33	16	-8
7	115	6	-1
8	156	9	-1
9	99	8	8
10	30	33	19*
11	149	9	-3
12	18	23	16*
13	56	9	3
14	30	10	6
H,K= 0, 10			
0	142	5	11
1	404	14	21
2	83	10	1
3	238	20	6
4	122	8	5
5	127	11	3
6	96	12	-3
7	63	16	-5
8	60	8	1
9	61	5	6
10	100	5	-2
11	98	13	-3
12	0	20	-8*
H,K= 0, 12			
0	195	6	2
1	189	9	4
2	202	14	4
3	71	5	1
4	163	13	1
5	42	13	-5
6	135	19	-3
7	74	7	-0
8	82	10	-3
9	24	28	11*
H,K= 0, 14			
0	23	28	-24*
1	11	20	-11*
2	33	14	-12
3	66	9	-5
4	68	13	1
H,K= 1, 1			
-17	0	24	-19*
-16	72	6	3
-15	0	30	-2*
-14	31	16	-13
-13	0	29	-12*
-12	107	5	-2
-11	232	7	-4
-10	202	6	-10
-9	281	9	-18
-8	390	12	-35
-7	49	4	-3
-6	17	18	7*
-5	58	9	3
-4	30	39	25*
-3	540	17	1
-2	126	4	-6
-1	414	13	15
0	524	16	-13
1	265	8	-28
2	157	6	-5
3	17	25	-2*
4	553	17	-71
5	63	9	6
6	50	7	-19
7	104	4	-6
8	53	5	8
9	384	12	-7
10	169	6	-2
11	95	9	8
12	138	6	-1
13	48	26	7
14	0	30	-3*
15	27	30	-15*
16	0	27	-28*
H,K= 1, 3			
-17	13	23	1*
-16	38	9	23
-15	9	30	4*
-14	163	6	-3
-13	97	10	-1
-12	46	23	22
-11	30	38	-20*
-10	99	5	3
-9	162	6	-4
-8	170	6	-7
-7	0	27	-24*
-6	555	17	38
-5	333	10	26
-4	628	19	25
-3	347	11	28
-2	63	14	-4
-1	587	18	12
0	77	8	10
1	727	22	-22
2	118	11	-21
3	728	22	-93
4	509	15	-42
5	64	5	3
6	483	15	-18
7	129	4	-9
8	119	6	-8
9	103	5	4
10	19	27	-20*
11	121	5	6
12	114	6	-3
13	34	19	-10
14	99	6	15
15	52	9	-7
16	16	26	0*
H,K= 1, 5			
-16	43	10	-1
-15	93	10	-4
-14	72	7	14
-13	0	30	-17*
-12	69	7	22
-11	208	7	-13
-10	171	6	-4
-9	33	12	-3
-8	84	5	-1
-7	421	13	-27
-6	189	7	11
-5	368	11	10
-4	16	19	6*
-3	207	7	27
-2	46	18	-4
-1	0	23	-35*
0	100	3	6
1	169	5	-4
2	53	4	-3
3	386	12	14
4	60	4	2
5	111	4	-13
6	272	8	-2
7	34	10	-4
8	111	5	4
9	48	8	-2
10	37	14	36
11	85	5	14
12	40	9	-1
13	33	20	-2
14	49	22	11
15	133	7	9
H,K= 1, 7			
-15	37	12	8
-14	51	7	-12
-13	35	42	-8*
-12	103	5	-10
-11	115	9	3
-10	134	8	-6
-9	39	40	1*
-8	193	6	-6
-7	118	5	4
-6	45	7	7
-5	39	17	9
-4	30	11	-5
-3	190	6	8
-2	155	5	9
-1	184	6	16
0	461	14	48
1	173	6	-1
2	35	8	14
3	16	31	7*
4	33	25	-5
5	113	4	-6
6	109	5	5
7	105	5	-1
8	76	6	8
9	9	41	2*
10	43	9	-2
11	72	7	1
12	90	11	-1
13	24	29	-11*
14	0	27	-2*
H,K= 1, 9			
-13	32	16	8
-12	67	7	7
-11	23	26	-3*
-10	59	7	5
-9	53	10	9
-8	74	7	-2
-7	27	27	25*
-6	26	29	13*
-5	39	12	-9
-4	101	5	-2
-3	110	7	15
-2	229	7	9
-1	42	20	12
0	255	9	10
1	81	5	1
2	46	8	4
3	108	5	1
4	104	5	8
5	63	6	8
6	46	11	17
7	0	27	-20*
8	167	6	8
9	18	25	-2*
10	123	5	4
11	18	25	-9*
12	48	7	7
H,K= 1, 11			
-11	40	8	-2
-10	0	25	-21*
-9	89	6	-1
-8	5	42	-9*
-7	83	7	-0
-6	58	9	0
-5	86	5	3
-4	62	6	2
-3	84	12	-1
-2	43	32	19
-1	67	8	13
0	3	42	-8*
1	139	6	2
2	40	14	20
3	89	6	14
4	21	43	-1*
5	0	25	-14*
6	14	25	-2*
7	104	14	8
8	29	29	-10*
9	42	11	2
10	27	13	10
H,K= 1, 13			
-7	21	27	15*
-6	0	28	-23*
-5	93	6	-9
-4	71	5	-1
-3	59	6	3
-2	22	30	6*
-1	45	11	-12
0	65	6	2
1	24	30	-4*
2	59	8	17
3	87	6	12
4	136	5	11
5	50	7	1
6	64	5	10
H,K= 2, 0			
-16	66	8	-4
-14	88	11	8
-12	174	7	8
-10	151	5	0
-8	245	8	-16
-6	1418	44	-160
-4	1616	51	-252
-2	338	10	29
0	698	21	46
2	343	11	-26
4	134	7	-26
6	123	4	1
8	260	8	-14
10	132	12	6
12	35	59	-1*
14	47	11	10
H,K= 2, 2			
-17	28	39	-5*
-16	13	26	-11*
-15	58	19	22
-14	21	30	19*
-13	69	7	2
-12	47	10	1
-11	48	8	19
-10	45	15	-14
-9	195	6	-3
-8	232	8	-3
-7	432	13	12
-6	209	7	4
-5	153	6	21
-4	975	30	37
-3	159	5	1
-2	595	18	4
-1	740	23	2
0	192	7	1
1	562	18	-61
2	583	18	-84
3	207	6	-28
4	159	6	-18
5	53	4	-10
6	199	6	-10
7	51	10	1
8	54	6	-0
9	170	8	-2
10	99	8	11
11	108	9	-5
12	20	30	7*
13	41	43	-9*
14	95	10	3
15	65	7	8
H,K= 2, 4			
-17	47	7	10
-16	32	12	22
-15	0	30	-15*
-14	42	43	5*
-13	43	51	-10*
-12	87	6	3
-11	56	8	-10
-10	171	7	-4
-9	34	40	-1*
-8	8	22	-6*
-7	368	11	-9
-6	417	13	15
-5	419	13	33
-4	148	5	-4
-3	389	12	20
-2	183	6	-1
-1	301	9	22
0	185	6	-0
1	48	4	-11
2	112	4	3
3	609	19	-36
4	278	9	-12
5	124	4	1
6	161	5	-1
7	151	5	-6
8	52	7	-8
9	44	8	32
10	117	8	1
11	117	9	3
12	134	8	14
13	0	42	-1*
14	35	10	1
15	79	10	4
H,K= 2, 6			
-16	33	10	-6
-15	48	22	-6
-14	0	42	-20*
-13	105	6	-1
-12	47	9	-3
-11	127	11	-1
-10	182	6	-6
-9	57	6	9
-8	126	5	-4
-7	0	23	-5*
-6	58	5	8
-5	122	4	-2
-4	54	4	-3
-3	203	6	14
-2	23	10	17
-1	225	7	7
0	204	6	16
1	238	7	16
2	126	5	15
3	16	20	-5*
4	262	8	-18
5	60	5	-11
6	193	6	-2
7	67	6	-8
8	131	5	-1
9	38	14	25
10	12	30	9*
11	0	30	-2*
12	109	5	2
13	6	41	-2*
14	60	7	8
H,K= 2, 8			
-14	0	25	-3*
-13	0	30	-43*
-12	82	7	-9
-11	131	6	-1
-10	52	59	-0*
-9	72	21	-1
-8	155	6	-6
-7	91	5	-5
-6	143	5	-6
-5	91	5	-3
-4	72	9	9
-3	299	9	12
-2	57	6	1
-1	235	8	8
0	100	7	13
1	173	6	7
2	0	25	-15*
3	23	23	20*
4	122	5	1
5	38	9	13
6	86	15	2
7	125	8	1
8	17	30	-4*
9	92	6	-0
10	0	30	-18*
11	120	5	10
12	45	7	-4
13	28	19	17
H,K= 2, 10			
-13	0	36	-6*
-12	0	28	-10*
-11	20	25	10*
-10	24	30	19*
-9	110	5	-7
-8	26	26	-3*
-7	81	12	-6
-6	35	51	26*
-5	226	7	2
-4	110	6	0
-3	177	6	-1
-2	85	6	3
-1	0	29	-2*
0	102	5	1
1	77	6	21
2	43	9	9
3	56	9	17
4	66	15	3
5	0	30	-22*
6	21	43	-4*
7	24	25	-11*
8	106	6	9
9	101	9	-3
10	57	8	3
11	0	26	-14*
H,K= 2, 12			
-10	16	22	0*
-9	19	28	11*
-8	23	29	9*
-7	28	41	-2*
-6	47	8	-15
-5	109	5	-6
-4	63	8	-14
-3	80	7	-14
-2	74	6	-11
-1	21	30	20*
0	25	25	0*
1	0	52	-19*
2	49	11	10
3	0	30	-4*
4	102	5	-3
5	52	9	8
6	71	5	1
7	15	28	-1*
8	35	7	29
H,K= 2, 14			
-4	0	22	-16*
-3	49	6	5
-2	64	5	-5
-1	23	27	1*
0	36	12	21
1	25	38	15*
2	0	27	-25*
H,K= 3, 1			
-18	17	26	12*
-17	16	29	2*
-16	39	10	4
-15	27	27	-19*
-14	83	6	5
-13	52	19	-9
-12	50	8	-7
-11	187	6	-4
-10	52	6	-2
-9	117	4	3
-8	101	4	-1
-7	119	5	1
-6	15	22	-6*
-5	51	13	-13
-4	35	41	2*
-3	971	30	6
-2	150	6	-4
-1	295	9	19
0	1023	31	35
1	493	15	-25
2	428	13	-35
3	369	11	-18
4	59	5	6
5	52	7	-3
6	77	4	-2
7	150	5	-5
8	99	5	-3
9	115	5	-1
10	112	5	-4
11	82	18	13
12	89	11	8
13	0	30	-9*
14	0	29	-11*
15	52	7	6
H,K= 3, 3			
-17	25	28	9*
-16	57	7	-4
-15	23	26	15*
-14	79	13	5
-13	53	9	24
-12	70	12	-6
-11	84	5	6
-10	52	6	-0
-9	49	6	8
-8	171	6	-2
-7	49	8	8
-6	230	8	2
-5	165	5	12
-4	310	10	14
-3	35	42	-16*
-2	334	11	33
-1	311	10	6
0	345	11	-16
1	455	14	-23
2	308	9	-6
3	203	7	-6
4	386	12	-20
5	445	14	-10
6	99	4	-4
7	63	5	-2
8	33	43	-6*
9	41	25	2
10	27	29	25*
11	11	28	-1*
12	88	6	6
13	48	24	6
14	43	11	9
H,K= 3, 5			
-17	0	23	-1*
-16	20	24	18*
-15	40	9	7
-14	60	6	-2
-13	73	7	13
-12	74	7	6
-11	29	21	-27
-10	62	7	-9
-9	23	25	15*
-8	286	9	-17
-7	362	11	6
-6	25	10	1
-5	106	4	-4
-4	102	4	1
-3	96	3	4
-2	58	8	-2
-1	29	30	18*
0	345	11	30
1	195	7	15
2	581	18	4
3	706	22	-33
4	165	5	-16
5	0	22	-18*
6	0	24	-8*
7	105	5	8
8	50	18	-2
9	33	17	17
10	15	42	6*
11	24	26	-1*
12	0	30	-32*
13	63	6	8
14	35	13	21
H,K= 3, 7			
-15	29	24	2
-14	27	18	13
-13	82	7	6
-12	35	12	-11
-11	76	13	-2
-10	125	5	-8
-9	90	5	-6
-8	0	26	-11*
-7	57	7	-6
-6	31	10	1
-5	96	4	2
-4	0	23	-22*
-3	126	4	-0
-2	241	8	22
-1	239	7	25
0	305	9	36
1	46	6	8
2	92	7	12
3	56	6	-4
4	185	7	8
5	87	5	4
6	90	5	9
7	50	10	5
8	7	30	1*
9	43	34	18
10	13	52	-16*
11	54	6	-1
12	27	29	16*
13	37	12	5
H,K= 3, 9			
-14	40	10	-9
-13	27	40	-10*
-12	0	27	-22*
-11	0	30	-14*
-10	72	7	12
-9	35	18	-6
-8	91	16	7
-7	0	41	-10*
-6	82	6	3
-5	0	28	-11*
-4	95	9	6
-3	46	9	11
-2	307	9	-2
-1	36	11	-4
0	0	27	-7*
1	64	6	11
2	1	25	-12*
3	22	28	-20*
4	60	16	-9
5	24	50	-7*
6	8	42	4*
7	47	7	1
8	141	5	-1
9	12	25	-12*
10	82	6	2
11	0	27	-5*
H,K= 3, 11			
-12	25	26	21*
-11	17	24	1*
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-9	0	30	-5*
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-5	23	30	-2*
-4	40	52	-9*
-3	55	9	-4
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2	92	6	-0
3	12	43	-8*
4	25	30	15*
5	49	7	-1
6	30	13	-4
7	54	6	-3
8	36	14	7
9	26	26	11*
H,K= 3, 13			
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-2	67	15	-6
-1	0	41	-14*
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5	67	4	10
H,K= 4, 0			
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-6	255	8	3
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-2	267	92	-25
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8	124	5	-6
10	139	6	-1
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14	74	12	6
H,K= 4, 2			
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-12	28	16	17
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-8	301	9	4
-7	201	6	-5
-6	305	9	13
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-4	471	14	20
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-2	349	11	15
-1	129	8	4
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2	185	6	-11
3	53	9	-27
4	99	4	4
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7	105	12	-3
8	97	5	-2
9	132	6	-5
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11	0	30	-3*
12	41	14	11
13	0	27	-1*
14	64	7	15
H,K= 4, 4			
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-15	0	30	-7*
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-10	49	7	4
-9	129	5	-8
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-5	36	5	13
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-3	455	14	45
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-1	228	7	18
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2	173	6	-2
3	27	8	7
4	278	9	-16
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6	36	10	-9
7	13	26	5*
8	96	5	2
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11	27	30	3*
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14	12	23	9*
H,K= 4, 6			
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-7	38	19	-4
-6	98	4	-8
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1	129	4	3
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3	53	6	-4
4	189	8	4
5	16	36	-3*
6	95	5	-3
7	29	23	3
8	0	50	-18*
9	33	21	14
10	30	13	23
11	12	42	-12*
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13	25	27	-13*
H,K= 4, 8			
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-8	157	6	-4
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-6	114	5	3
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-3	86	5	-1
-2	149	5	7
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2	42	9	5
3	143	5	3
4	91	5	2
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6	46	27	19
7	93	5	13
8	0	30	-4*
9	47	11	22
10	66	6	-6
11	34	14	17
H,K= 4, 10			
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-8	29	30	5*
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-5	33	19	6
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-2	0	28	-31*
-1	92	15	7
0	39	14	0
1	42	50	6*
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3	49	8	-9
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5	0	30	-20*
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8	70	7	8
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H,K= 4, 12			
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-7	91	6	3
-6	54	9	7
-5	46	11	-8
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-3	13	24	-2*
-2	46	8	-17
-1	35	18	-14
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1	34	19	14
2	35	17	1
3	16	24	10*
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5	14	28	3*
6	0	23	-4*
H,K= 4, 14			
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H,K= 5, 1			
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-12	39	10	4
-11	86	5	7
-10	102	4	2
-9	350	11	-8
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-5	80	5	-9
-4	185	6	3
-3	306	9	32
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2	494	15	17
3	200	6	6
4	92	4	3
5	112	4	-8
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7	79	5	-3
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9	28	30	-3*
10	23	42	-2*
11	33	13	23
12	50	23	-1
13	87	10	7
14	59	6	1
H,K= 5, 3			
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-12	30	38	-4*
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-3	279	9	4
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1	209	8	-6
2	60	4	4
3	134	4	-1
4	333	10	-20
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6	100	5	3
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8	57	8	4
9	40	41	5*
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12	67	6	13
13	33	39	1*
H,K= 5, 5			
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-12	88	6	-4
-11	76	11	-16
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-9	94	5	-7
-8	23	40	18*
-7	31	37	16*
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0	58	4	15
1	333	10	-16
2	151	5	-6
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6	77	6	2
7	0	28	-28*
8	32	20	-0
9	50	8	-7
10	0	26	-8*
11	27	30	20*
12	22	24	13*
H,K= 5, 7			
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-14	21	30	18*
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-12	69	15	-7
-11	85	11	4
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-9	61	7	-2
-8	128	5	2
-7	30	36	21*
-6	9	42	-8*
-5	39	9	-0
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-3	26	15	23
-2	66	5	0
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3	26	37	0*
4	100	5	1
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6	29	30	-5*
7	28	18	8
8	89	5	-2
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10	16	25	2*
11	30	12	15
H,K= 5, 9			
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-11	25	30	10*
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-8	202	7	-5
-7	35	13	3
-6	53	19	7
-5	67	6	-1
-4	200	7	-9
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1	5	26	-7*
2	49	10	5
3	97	6	3
4	78	7	-3
5	98	10	-3
6	15	25	9*
7	25	30	5*
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9	39	12	32
10	0	26	-15*
H,K= 5, 11			
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-5	104	5	-6
-4	39	15	15
-3	34	10	2
-2	0	30	-2*
-1	69	8	7
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1	31	25	8
2	24	43	2*
3	51	10	-3
4	28	42	15*
5	0	29	-4*
6	27	28	-10*
7	45	7	-1
H,K= 5, 13			
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-6	55	5	14
-5	0	28	-15*
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-3	23	29	19*
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3	55	7	-8
H,K= 6, 0			
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-12	26	37	-13*
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-6	431	13	-2
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-2	30	9	-8
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4	236	8	-1
6	79	9	-14
8	85	6	-9
10	122	9	-5
12	30	17	-4
H,K= 6, 2			
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-16	67	8	-2
-15	27	30	15*
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-12	119	5	-3
-11	142	5	-4
-10	83	4	-2
-9	181	6	-4
-8	138	5	4
-7	111	5	-5
-6	146	5	7
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-3	279	9	9
-2	187	6	12
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1	33	6	10
2	236	7	-11
3	72	7	-2
4	106	4	-13
5	88	5	-3
6	31	36	2*
7	108	5	-6
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10	57	9	16
11	66	6	1
12	25	26	3*
13	0	26	-23*
H,K= 6, 4			
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-12	74	6	6
-11	112	5	0
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-9	149	5	-9
-8	99	4	-8
-7	466	14	-0
-6	104	4	-6
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-4	164	5	9
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4	304	9	-10
5	101	7	-6
6	134	5	-3
7	0	29	-7*
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9	10	26	0*
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11	92	6	5
12	0	28	-2*
H,K= 6, 6			
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-15	43	8	22
-14	23	26	-6*
-13	23	30	-1*
-12	0	42	-24*
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-8	136	5	-6
-7	47	6	1
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-1	0	23	-26*
0	187	6	9
1	13	24	-4*
2	91	8	-1
3	130	7	-3
4	134	5	-13
5	131	5	-7
6	148	6	-1
7	99	6	-3
8	140	8	-3
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10	108	6	-1
11	18	39	4*
H,K= 6, 8			
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-11	54	10	8
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-9	29	30	6*
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5	101	10	-2
6	69	5	-1
7	75	14	-3
8	33	10	12
9	0	28	-18*
10	33	14	-4
H,K= 6, 10			
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-7	5	30	-15*
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-5	100	6	2
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-3	0	30	-3*
-2	29	30	-5*
-1	164	6	5
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1	89	5	2
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3	55	9	-13
4	27	30	12*
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7	33	9	21
8	27	21	9
H,K= 6, 12			
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H,K= 7, 1			
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-8	61	4	-1
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-2	728	22	77
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2	240	8	17
3	151	5	6
4	121	5	-8
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6	121	5	0
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8	137	6	3
9	75	6	18
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12	28	38	20*
H,K= 7, 3			
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-15	43	42	-3*
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-8	196	7	-14
-7	119	5	15
-6	170	5	3
-5	144	5	16
-4	185	6	9
-3	105	5	-5
-2	48	4	-9
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0	31	8	-2
1	276	9	2
2	388	12	-8
3	128	5	2
4	209	7	-9
5	123	5	2
6	48	10	-12
7	85	11	3
8	28	30	-6*
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10	37	42	8*
11	40	9	7
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-3	156	5	4
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7	13	30	-7*
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9	98	6	5
10	90	5	3
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H,K= 7, 7			
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-9	138	5	-0
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-5	78	5	-4
-4	248	9	-9
-3	101	7	-3
-2	79	5	4
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0	118	5	-1
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2	13	26	5*
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5	93	11	-3
6	54	10	-4
7	84	5	2
8	107	6	-4
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10	23	27	-11*
H,K= 7, 9			
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-9	61	9	-5
-8	82	12	1
-7	53	38	-5
-6	135	8	-2
-5	44	11	17
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1	111	6	3
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4	148	6	-2
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7	0	28	-3*
8	99	4	3
H,K= 7, 11			
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-8	31	12	20
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-4	37	11	-14
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H,K= 7, 13			
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-3	20	27	12*
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-1	24	38	15*
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H,K= 8, 0			
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-6	637	20	58
-4	737	23	64
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2	149	5	5
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6	0	49	-4*
8	239	8	10
10	18	30	-20*
H,K= 8, 2			
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-16	29	30	-9*
-15	63	8	-8
-14	18	51	5*
-13	62	8	13
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-10	32	41	8*
-9	114	4	-1
-8	72	7	3
-7	233	7	2
-6	273	9	15
-5	420	13	24
-4	79	4	-1
-3	597	18	29
-2	70	4	9
-1	84	6	-1
0	34	7	14
1	356	11	9
2	124	5	-5
3	89	5	-6
4	188	6	2
5	222	7	-0
6	89	6	3
7	169	6	9
8	0	27	-6*
9	44	31	19
10	63	7	-6
11	74	17	-3
H,K= 8, 4			
-18	9	27	-7*
-17	42	8	2
-16	66	8	-4
-15	6	26	-11*
-14	71	14	-8
-13	18	28	-1*
-12	93	6	1
-11	160	6	-9
-10	154	7	-7
-9	223	7	-11
-8	308	10	-8
-7	133	5	-1
-6	27	11	-20
-5	33	8	11
-4	7	22	4*
-3	108	4	1
-2	127	4	5
-1	245	8	-6
0	143	5	1
1	211	7	-6
2	206	7	-2
3	90	9	-3
4	247	8	-3
5	39	40	11*
6	0	42	-5*
7	177	6	14
8	104	6	3
9	53	9	4
10	37	13	15
H,K= 8, 6			
-17	56	5	5
-16	0	29	-32*
-15	21	30	4*
-14	77	5	-4
-13	27	30	1*
-12	0	28	-24*
-11	0	29	-0*
-10	136	7	1
-9	110	5	-4
-8	134	5	-3
-7	17	26	2*
-6	120	5	-5
-5	100	4	3
-4	119	5	-13
-3	13	25	8*
-2	151	5	-6
-1	39	10	-6
0	245	8	-13
1	183	6	-3
2	165	7	-14
3	115	5	-8
4	175	6	4
5	24	26	15*
6	104	6	7
7	39	42	-2*
8	31	22	9
9	19	24	5*
10	27	11	2
H,K= 8, 8			
-15	86	4	5
-14	0	29	-25*
-13	106	5	-3
-12	0	30	-8*
-11	27	16	19
-10	31	42	-3*
-9	51	10	-13
-8	40	10	13
-7	93	10	-4
-6	7	27	6*
-5	178	6	-12
-4	153	5	-8
-3	242	8	-9
-2	69	13	2
-1	66	7	1
0	100	5	-4
1	62	8	4
2	63	8	2
3	55	6	-4
4	55	9	20
5	188	6	-0
6	144	5	8
7	14	29	-3*
8	77	6	1
H,K= 8, 10			
-13	21	24	-12*
-12	70	5	-2
-11	30	11	20
-10	72	7	-10
-9	46	12	-8
-8	49	11	10
-7	79	7	-3
-6	170	6	-3
-5	72	5	2
-4	81	5	3
-3	153	5	-9
-2	58	6	0
-1	103	6	-6
0	23	25	-10*
1	8	25	6*
2	82	12	-9
3	86	6	0
4	43	8	-11
5	47	9	-4
6	70	6	-3
H,K= 8, 12			
-9	43	6	5
-8	35	14	3
-7	69	7	-2
-6	109	4	-4
-5	28	15	-0
-4	26	17	-25
-3	69	7	-2
-2	49	9	-9
-1	97	5	-11
0	125	8	-15
1	41	10	-0
2	96	4	1
H,K= 9, 1			
-18	29	23	14
-17	87	5	4
-16	71	5	6
-15	102	5	4
-14	146	6	4
-13	24	29	10*
-12	19	38	15*
-11	78	5	-0
-10	23	35	13*
-9	37	7	7
-8	120	4	-3
-7	113	4	8
-6	44	5	1
-5	120	4	6
-4	96	4	8
-3	367	11	9
-2	0	21	-18*
-1	149	5	7
0	75	8	-4
1	32	11	11
2	46	8	6
3	96	5	-5
4	33	16	-3
5	147	10	3
6	38	14	-13
7	161	6	-2
8	81	7	5
9	8	30	-12*
10	107	5	2
H,K= 9, 3			
-18	23	23	-0*
-17	56	9	5
-16	37	16	-3
-15	13	26	-28*
-14	128	6	-3
-13	140	5	-9
-12	78	6	-7
-11	48	9	35
-10	231	7	-3
-9	116	6	6
-8	94	4	-8
-7	117	4	-6
-6	388	12	4
-5	22	31	6*
-4	24	12	-14
-3	23	17	9
-2	95	9	-0
-1	118	4	-1
0	242	8	-4
1	36	10	17
2	178	6	3
3	91	6	-8
4	169	6	5
5	16	41	12*
6	20	52	18*
7	88	6	5
8	42	40	8
9	25	40	0*
10	10	27	-16*
H,K= 9, 5			
-17	44	23	-14
-16	23	30	-14*
-15	88	5	6
-14	0	26	-6*
-13	31	14	-2
-12	54	65	-14*
-11	158	12	-9
-10	37	13	-12
-9	271	9	-14
-8	12	36	-0*
-7	189	6	-7
-6	110	5	3
-5	124	5	6
-4	86	5	-1
-3	176	6	4
-2	38	9	-9
-1	117	5	-6
0	0	25	-12*
1	184	6	-0
2	85	6	-9
3	0	29	-9*
4	191	6	1
5	0	42	-9*
6	78	13	-1
7	0	26	-5*
8	63	25	-7
9	73	6	10
H,K= 9, 7			
-16	0	38	-19*
-15	43	8	-0
-14	47	7	11
-13	40	14	12
-12	23	25	22*
-11	80	5	8
-10	77	7	-3
-9	76	7	-1
-8	103	6	-2
-7	67	20	-10
-6	152	5	-13
-5	50	8	-14
-4	95	5	4
-3	206	7	-5
-2	20	26	-1*
-1	108	5	-0
0	140	8	3
1	75	13	-3
2	46	11	-19
3	0	26	-17*
4	68	15	6
5	153	6	-2
6	23	30	-5*
7	0	28	-19*
8	47	19	-1
H,K= 9, 9			
-14	68	6	-4
-13	25	29	-24*
-12	76	13	3
-11	22	30	-1*
-10	61	6	2
-9	10	43	-12*
-8	52	10	-3
-7	86	5	-3
-6	166	6	-15
-5	114	6	-7
-4	166	6	-11
-3	142	5	-1
-2	51	10	11
-1	58	6	3
0	0	30	-3*
1	55	21	-12
2	48	7	3
3	38	10	0
4	78	5	-1
5	29	22	3
6	95	5	3
H,K= 9, 11			
-11	43	9	6
-10	53	8	-7
-9	52	7	-6
-8	20	29	-2*
-7	34	17	16
-6	22	25	-20*
-5	0	30	-23*
-4	0	30	-11*
-3	28	30	-9*
-2	59	6	-9
-1	48	10	6
0	28	12	26
1	39	12	-0
2	38	12	15
3	45	6	12
H,K= 10, 0			
-18	36	13	-3
-16	58	6	9
-14	116	5	-5
-12	171	6	-13
-10	112	5	4
-8	38	7	-4
-6	41	7	-2
-4	75	4	-0
-2	322	10	9
0	32	12	24
2	172	6	1
4	149	8	-6
6	105	10	3
8	74	20	1
H,K= 10, 2			
-18	8	39	-5*
-17	47	11	-0
-16	0	30	-1*
-15	26	30	21*
-14	27	30	-1*
-13	25	41	8*
-12	26	26	2*
-11	90	13	-7
-10	65	6	-6
-9	153	5	0
-8	142	5	-6
-7	88	4	-10
-6	251	8	-0
-5	202	6	-3
-4	57	5	4
-3	47	7	-7
-2	49	7	-14
-1	59	6	-12
0	64	6	1
1	44	9	18
2	54	8	3
3	38	48	-2*
4	158	10	2
5	65	16	-3
6	68	15	-6
7	37	12	-2
8	56	6	14
9	72	6	6
H,K= 10, 4			
-18	27	22	11
-17	14	40	-3*
-16	38	10	-7
-15	0	30	-1*
-14	71	7	6
-13	59	6	-2
-12	0	30	-31*
-11	134	5	3
-10	133	5	9
-9	170	8	-1
-8	99	4	2
-7	49	8	6
-6	51	14	9
-5	22	24	-6*
-4	146	5	3
-3	138	5	-8
-2	24	25	15*
-1	335	11	-13
0	47	7	14
1	339	10	-12
2	0	29	-37*
3	309	11	7
4	34	14	-1
5	64	16	8
6	72	7	7
7	81	12	10
8	20	28	1*
9	78	9	-4
H,K= 10, 6			
-17	25	26	-18*
-16	0	24	-14*
-15	39	14	-2
-14	47	7	6
-13	38	43	-2*
-12	78	5	-3
-11	94	6	2
-10	153	5	-3
-9	39	10	20
-8	42	9	-3
-7	112	5	-0
-6	41	11	-2
-5	42	9	5
-4	111	5	-7
-3	47	9	18
-2	216	7	-1
-1	60	8	2
0	485	15	-11
1	188	6	-2
2	231	8	3
3	56	9	1
4	146	5	0
5	66	8	5
6	145	6	9
7	34	10	16
8	92	8	-3
H,K= 10, 8			
-15	57	7	11
-14	38	13	0
-13	40	41	5*
-12	36	10	7
-11	17	25	15*
-10	41	9	-1
-9	41	14	7
-8	55	20	6
-7	141	8	-10
-6	66	8	7
-5	162	6	-4
-4	34	42	1*
-3	246	8	-5
-2	78	20	-16
-1	63	8	-8
0	94	5	2
1	68	8	2
2	134	6	-2
3	117	5	1
4	25	29	6*
5	173	6	4
6	64	5	4
H,K= 10, 10			
-12	27	38	25*
-11	43	8	1
-10	29	12	25
-9	32	20	8
-8	0	42	-17*
-7	23	24	2*
-6	148	6	0
-5	42	7	25
-4	35	10	-9
-3	43	13	7
-2	29	30	-4*
-1	40	8	5
0	0	29	-8*
1	93	4	8
2	0	28	-8*
3	44	7	-2
H,K= 10, 12			
-7	18	22	-11*
-6	33	8	26
-5	57	7	1
-4	49	8	-6
-3	50	29	0
-2	56	7	-8
H,K= 11, 1			
-18	0	23	-4*
-17	37	15	4
-16	73	7	-1
-15	50	11	19
-14	96	5	11
-13	6	30	-1*
-12	40	11	-2
-11	142	5	-1
-10	105	5	2
-9	9	24	-4*
-8	210	7	2
-7	125	5	1
-6	109	7	6
-5	316	10	6
-4	45	6	5
-3	18	35	10*
-2	119	5	6
-1	116	5	-0
0	26	27	8*
1	31	21	-28
2	36	15	15
3	28	30	-11*
4	0	42	-10*
5	130	5	4
6	76	6	-3
7	30	25	9
8	0	38	-6*
H,K= 11, 3			
-18	31	9	9
-17	20	29	3*
-16	19	42	12*
-15	0	26	-4*
-14	62	6	-9
-13	141	11	5
-12	36	41	12*
-11	36	40	21*
-10	25	39	2*
-9	28	14	18
-8	87	5	-1
-7	108	5	-7
-6	35	36	10*
-5	0	26	-5*
-4	28	17	21
-3	21	26	-14*
-2	189	6	1
-1	137	5	1
0	106	5	-5
1	0	29	-6*
2	16	30	5*
3	111	13	0
4	50	8	-3
5	3	26	-19*
6	51	7	12
7	56	8	3
8	98	5	3
H,K= 11, 5			
-17	0	27	-3*
-16	25	29	12*
-15	87	6	-5
-14	32	22	21
-13	34	11	2
-12	8	30	4*
-11	113	9	4
-10	98	6	-0
-9	101	9	3
-8	69	13	3
-7	0	28	-1*
-6	74	5	2
-5	0	26	-29*
-4	47	20	-13
-3	194	6	-7
-2	80	6	-6
-1	122	5	5
0	289	9	-4
1	195	6	5
2	18	42	-2*
3	10	30	3*
4	93	6	-1
5	0	30	-26*
6	32	10	16
7	29	12	11
H,K= 11, 7			
-16	0	26	-26*
-15	0	24	-3*
-14	8	29	-1*
-13	42	13	3
-12	50	6	8
-11	39	8	22
-10	36	10	-6
-9	33	11	19
-8	106	6	4
-7	12	30	-37*
-6	57	9	-6
-5	58	9	-7
-4	37	10	31
-3	243	12	-5
-2	106	5	-0
-1	66	6	4
0	51	8	-12
1	48	7	-1
2	96	5	5
3	98	5	-0
4	19	29	12*
5	73	6	6
6	41	6	13
H,K= 11, 9			
-13	28	23	-11
-12	46	23	9
-11	59	6	9
-10	27	30	-8*
-9	67	5	9
-8	62	8	3
-7	0	24	-2*
-6	163	6	-11
-5	33	13	-19
-4	62	6	-2
-3	28	15	-3
-2	36	8	15
-1	0	25	-1*
0	21	24	13*
1	15	23	12*
2	10	28	2*
3	13	27	12*
H,K= 11, 11			
-9	63	6	-1
-8	88	5	-2
-7	43	6	9
-6	0	28	-3*
-5	0	28	-17*
-4	16	24	-14*
-3	45	6	6
-2	26	12	24
-1	49	8	5
H,K= 12, 0			
-18	54	8	9
-16	73	5	1
-14	133	6	5
-12	184	6	-1
-10	220	7	-9
-8	15	37	13*
-6	149	5	0
-4	140	5	-1
-2	86	14	-15
0	119	5	-2
2	30	42	-8*
4	93	6	-9
6	0	41	-25*
H,K= 12, 2			
-18	12	27	9*
-17	59	8	6
-16	34	18	-10
-15	34	11	16
-14	37	16	13
-13	89	11	-17
-12	99	6	-1
-11	17	49	-3*
-10	0	40	-28*
-9	49	19	2
-8	81	6	4
-7	90	5	-1
-6	28	21	2
-5	46	8	-5
-4	21	25	17*
-3	102	5	-2
-2	0	27	-7*
-1	53	9	7

Table 1 (*cont.*)

L	FOB	SG	DEL	L	FOB	SG	DEL	L	FOB	SG	DEL	L	FOB	SG	DEL	L	FOB	SG	DEL	L	FOB	SG	DEL	L	FOB	SG	DEL	L	FOB	SG	DEL	L	FOB	SG	DEL	L	FOB	SG	DEL	L	FOB	SG	DEL	L	FOB	SG	DEL	L	FOB	SG	DEL
0	61	8	-11	-13	0	42	-12*	3	0	24	-22*	4	98	5	-1	-7	82	6	8	-8	0	29	-23*	-2	21	30	8*	-8	209	7	-2	-6	73	7	2	-9	9	42	-14*	-15	66	7	1	-11	23	28	14*	-4	95	4	-1
1	70	6	4	-12	50	10	8	H,K=		12,	10	5	25	24	3	-6	57	9	2	-7	22	41	17*	-1	68	8	16	-7	44	13	-14	-5	18	30	-9*	-8	20	26	-23*	-14	19	28	6*	-10	51	19	4	-3	35	39	20*
2	38	52	12*	-11	11	25	6*	-11	49	8	-13	6	31	15	-8	-5	72	7	2	-6	85	4	3	0	87	12	-8	-6	161	6	-4	-4	32	14	-22	-7	57	7	-4	-13	116	5	2	-9	0	28	-30*	-2	60	5	1
3	41	14	-2	-10	31	12	6	-10	47	9	-2	H,K=		13,	3	-4	87	11	-7	-5	29	12	11	1	60	6	-5	-5	13	25	-0*	-3	39	15	-6	-6	24	30	20*	-12	108	6	-2	-8	56	8	6	H,K=		17,	5
4	0	26	-11*	-9	20	30	6*	-9	66	7	-3	-17	27	47	12*	-3	4	30	-37*	-4	18	49	9*	2	24	25	7*	-4	54	10	3	-2	68	6	-13	-5	5	42	-3*	-11	38	9	13	-7	3	29	-29*	-12	0	37	-11*
5	9	30	-5*	-8	90	16	4	-8	30	11	10	-16	39	8	7	-2	69	8	3	-3	14	28	11*	3	27	29	-13*	-3	65	8	-8	-1	23	30	5*	-4	15	42	15*	-10	65	16	10	-6	119	5	3	-11	27	27	-8*
6	23	29	20*	-7	9	30	-3*	-7	59	5	3	-15	22	24	14*	-1	22	26	-8*	-2	19	24	-13*	4	0	24	-8*	-2	71	7	6	0	89	6	-5	-3	20	25	-5*	-9	12	30	-13*	-5	37	9	2	-10	0	23	-8*
7	37	38	-0*	-6	191	6	-6	-6	0	24	-27*	-14	59	6	3	0	102	10	-9	-1	19	23	-9*	H,K=		14,	4	-1	57	9	6	1	79	6	7	-2	0	25	-15*	-8	13	25	-3*	-4	87	6	1	-9	81	4	4
H,K=		12,	4	-5	143	5	3	-5	0	40	-11*	-13	36	17	16	1	131	5	-6	0	16	26	13*	-16	45	9	2	0	22	25	-2*	2	46	10	20	-1	87	6	11	-7	133	6	2	-3	33	9	17	-8	42	8	-4
-17	19	23	-6*	-4	277	9	-14	-4	45	7	3	-12	0	30	-11*	2	33	19	26	H,K=		14,	0	-15	61	15	-1	1	40	8	3	3	10	27	4*	0	16	39	-6*	-6	5	30	-15*	-2	68	6	7	-7	71	6	3
-16	0	29	-31*	-3	123	9	-3	-3	43	7	3	-11	95	6	5	3	53	9	6	-16	45	11	-1	-14	74	7	11	2	21	27	9*	H,K=		15,	3	1	41	10	7	-5	41	14	0	H,K=		17,	1	-6	77	6	-1
-15	29	30	-2*	-2	133	5	-7	-2	26	13	22	-10	71	7	-3	4	13	28	-23*	-14	66	8	6	-13	142	6	8	H,K=		14,	8	-16	37	12	-1	H,K=		15,	7	-4	39	9	20	-15	32	9	-3	-5	40	8	-8
-14	0	30	-16*	-1	87	6	5	-1	28	11	9	-9	44	10	-10	H,K=		13,	7	-12	99	5	-6	-12	46	29	-14	-12	17	27	-8*	-15	0	28	-29*	-13	12	26	6*	-3	34	42	-6*	-14	0	23	-12*	-4	33	14	4
-13	0	25	-8*	0	50	11	-15	0	0	26	-10*	-8	90	10	2	-15	49	8	10	-10	165	6	4	-11	214	7	1	-11	49	6	4	-14	99	6	-0	-12	42	10	3	-2	60	8	-3	-13	49	7	-1	-3	99	5	13
-12	0	30	-1*	1	84	5	5	H,K=		13,	1	-7	128	5	-2	-14	27	28	-8*	-8	219	7	-1	-10	53	10	-2	-10	93	6	-4	-13	0	30	-15*	-11	20	28	-4*	-1	74	5	-3	-12	34	9	21	H,K=		18,	0
-11	0	42	-39*	2	58	32	-9	-18	29	9	28	-6	102	5	-4	-13	28	29	12*	-6	177	7	16	-9	143	8	1	-9	60	16	-2	-12	130	5	6	-10	101	5	-10	0	53	17	2	-11	92	5	4	-12	71	5	0
-10	30	16	20	3	37	41	23*	-17	0	28	-2*	-5	91	6	4	-12	89	5	-1	-4	29	17	12	-8	100	5	1	-8	40	8	7	-11	58	6	-2	-9	15	29	14*	1	55	7	0	-10	84	6	3	-10	89	10	-1
-9	0	29	-6*	4	0	25	-6*	-16	0	29	-4*	-4	98	6	1	-11	39	14	-10	-2	0	42	-23*	-7	123	5	-2	-7	94	5	-1	-10	106	6	1	-8	51	7	0	H,K=		16,	4	-9	169	6	3	-8	12	28	-18*
-8	112	5	-2	5	28	12	8	-15	38	9	-6	-3	73	7	1	-10	38	15	-9	0	0	26	-6*	-6	83	12	-8	-6	29	12	15	-9	108	5	3	-7	49	7	-9	-15	0	37	-7*	-8	116	5	-1	-6	10	24	-1*
-7	200	7	-17	H,K=		12,	8	-14	49	11	-3	-2	142	5	-3	-9	22	25	-4*	2	100	6	-2	-5	71	7	2	-5	37	9	5	-8	108	10	-8	-6	24	29	-0*	-14	109	5	1	-7	74	13	4	-4	0	27	-5*
-6	28	28	-13*	-14	44	9	6	-13	37	16	-2	-1	38	15	5	-8	11	24	1*	4	69	6	8	-4	62	8	-17	-4	0	29	-25*	-7	60	31	2	-5	58	8	7	-13	24	28	1*	-6	131	6	-6	H,K=		18,	2
-5	246	8	-12	-13	24	25	-21*	-12	117	6	-5	0	19	30	6*	-7	0	30	-14*	H,K=		14,	2	-3	151	5	1	-3	0	40	-10*	-6	32	23	-7	-4	30	11	12	-12	76	6	5	-5	0	29	-34*	-13	52	15	-4
-4	109	5	1	-12	65	6	-6	-11	44	29	6	1	31	12	18	-6	19	30	-18*	-17	18	27	10*	-2	154	6	-1	-2	48	9	9	-5	38	15	7	-3	12	28	7*	-11	23	24	12*	-4	56	9	-8	-12	67	5	-2
-3	164	6	-11	-11	73	7	-2	-10	76	12	-3	2	97	6	1	-5	114	6	3	-16	46	6	9	-1	31	25	-2	-1	42	6	18	-4	151	6	6	-2	16	27	14*	-10	98	6	-6	-3	68	5	9	-11	0	23	-10*
-2	193	6	-3	-10	57	6	-0	-9	49	21	-12	3	49	11	-8	-4	102	5	-1	-15	18	30	5*	0	73	7	-4	0	26	11	15	-3	24	25	16*	-1	0	37	-8*	-9	38	14	24	-2	2	24	1*	-10	23	28	6*
-1	47	11	-13	-9	77	5	-2	-8	134	7	-0	4	24	41	19*	-3	55	6	-1	-14	109	5	3	1	51	6	18	H,K=		15,	1	-2	134	6	-6	H,K=		16,	0	-8	54	6	-1	-1	72	6	-2	-9	89	5	4
0	0	30	-30*	-8	50	7	-4	-7	190	7	-10	5	60	7	2	-2	23	24	16*	-13	69	8	-0	2	29	29	0*	-17	44	20	-0	-1	47	11	11	-16	76	4	1	-7	38	15	-2	H,K=		17,	3	-8	19	28	16*
1	25	26	1*	-7	15	25	9*	-6	0	28	-14*	H,K=		13,	5	-1	91	5	-5	-12	49	11	-5	3	55	6	2	-16	50	19	14	0	30	23	19	-14	54	6	2	-6	35	42	10*	-14	110	5	-1	-7	58	6	-0
2	96	6	-10	-6	147	5	-5	-5	106	8	12	-16	0	27	-10*	0	30	12	16	-11	0	30	-19*	4	26	22	17	-15	35	9	22	1	18	29	-13*	-12	29	30	8*	-5	106	5	4	-13	38	12	0	-6	0	38	-7*
3	57	9	-7	-5	20	25	6*	-4	71	6	-1	-15	38	13	4	1	0	25	-18*	-10	105	6	3	H,K=		14,	6	-14	39	14	-17	2	20	28	4*	-10	135	6	4	-4	101	5	-1	-12	121	5	1	-5	10	23	-1*
4	35	11	5	-4	148	6	-4	-3	115	5	-7	-14	73	5	4	2	82	5	-5	-9	0	27	-14*	-15	39	7	18	-13	73	7	-4	H,K=		15,	5	-8	131	5	3	-3	89	5	-3	-11	53	9	18	-4	59	7	7
5	30	15	-16	-3	28	15	1	-2	16	29	14*	-13	45	12	3	3	26	13	-3	-8	34	42	-1*	-14	83	5	-1	-12	78	7	-0	-15	88	4	4	-6	33	43	-24*	-2	46	10	1	-10	71	7	11	H,K=		18,	4
6	25	17	13	-2	80	4	10	-1	0	41	-28*	-12	0	30	-11*	H,K=		13,	9	-7	34	18	5	-13	44	7	7	-11	73	5	-7	-14	53	17	1	-4	61	9	6	-1	50	6	-1	-9	13	29	7*	-10	50	6	-2
H,K=		12,	6	-1	121	5	-11	0	34	14	-1	-11	6	25	-16*	-12	30	9	-1	-6	0	30	-19*	-12	114	6	0	-10	85	5	-4	-13	111	5	-9	-2	0	30	-14*	0	15	37	-2*	-8	21	29	9*	-9	46	9	24
-16	15	38	-12*	0	34	10	25	1	47	11	-0	-10	0	30	-24*	-11	28	24	1	-5	114	5	0	-11	44	8	9	-9	152	5	2	-12	63	6	-2	0	95	9	-3	H,K=		16,	6	-7	74	7	1	-8	38	8	-2
-15	71	7	-2	1	35	9	15	2	86	5	-9	-9	105	6	-4	-10	13	28	-2*	-4	0	30	-30*	-10	204	7	2	-8	0	42	-9*	-11	89	11	10	H,K=		16,	2	-13	12	26	9*	-6	101	5	2	-7	39	10	7
-14	13	25	-29*	2	0	28	-26*	3	106	5	7	-8	53	20	-4	-9	44	11	13	-3	0	27	-6*	-9	76	7	-9	-7	99	5	4	-10	0	30	-14*	-16	41	6	9	-12	40	11	-3	-5	38	14	5	-6	78	4	-1

using a θ–2θ scan technique at an X-ray tube takeoff angle of 3°. Peaks were scanned at a rate of 1°/min from 0·9° below the predicted $K\alpha_1$ position to 0·9° above the predicted $K\alpha_2$ position. Backgrounds were counted for 10 sec at positions offset 0·6° from each end of the scan interval (all angles in 2θ). Aluminum attenuators were automatically inserted in the diffracted beam whenever the count rate exceeded 10000 c.p.s., and the peak and backgrounds were remeasured with the attenuators in place. The reflections 400, $\bar{2}02$ and $04\bar{4}$ were monitored periodically during the data collection and exhibited no decrease in intensity. All reflections in the quadrant of reciprocal space $+h, +k, \pm l$ were measured out to a 2θ angle of 120° ($\sin\theta/\lambda < 0{\cdot}562$). 2433 unique reflections were measured, of which 614 had $I < \sigma(I)$. Net intensities and their standard deviations were calculated by the formulae:

$$I = C - \frac{t_c}{2t_b}(B_1 + B_2),\ \sigma^2(I) = C + \frac{t_c^2}{4t_b^2}(B_1 + B_2)$$

where C is the total counts recorded in scan time t_c and B_1 and B_2 are the background counts, each measured for time t_b. No absorption correction was applied ($\mu = 7{\cdot}7$ cm^{-1}). Intensities of equivalent reflections and those measured more than once were averaged. Standard deviations were set equal to the greater of $(1/n)\,(\sum\sigma_i^2)^{1/2}$ or $[1/(n-1)](\sum\Delta_i^2)^{1/2}$, where σ_i and Δ_i are the standard deviation of the ith measurement and the deviation of the ith measurement from the average respectively, and n is the number of reflections averaged. Lorentz and polarization corrections were applied.

The scattering factors of Doyle & Turner (1968) were used for neutral Mg, C and O, together with the real and imaginary dispersion terms of Cromer & Liberman (1970). The spherical hydrogen scattering factors of Stewart, Davidson & Simpson (1965) were used.

Our least-squares program minimizes the function $\sum w(\Delta F)^2/\sum wF_o^2$. The weighting scheme used throughout the refinement gave zero weight if $F^2 < \sigma(F^2)$, and $w = 1/\sigma(F)$ otherwise. Finite differences were used to calculate $\sigma(F)$ from $\sigma(F^2)$ and F^2:

$$\sigma(F) = F - [F^2 - \sigma(F^2)]^{1/2};\quad \sigma^2(F^2) = \sigma_c^2(F^2) + (pF^2)^2$$

where p is a factor (initially zero, but 0·06 in the final cycles) used to reduce the weights of intense reflections, which are more prone to undetected systematic errors, and $\sigma_c(F^2) = (\mathrm{Lp})^{-1}\sigma(I)$.

The following programs, written for our CDC 6600 computer, were also used in the solution and refinement of this structure: *MAGPIK*, a program for interpretation of raw data from the Picker/DEC system; *INCOR*, *EDIT*, and *ORDER*, general data reduction programs; *WILSON*, an unpublished program written by Maddox & Maddox for applying Wilson's (1942) statistics to data and calculation of normalized structure factors; *REL*, Long's (1965) program for direct determination of centric phases; *FORDAP*, A. Zalkin's Fourier analysis program; *LSLONG*, our modification of the Ganzel–Sparks–Trueblood least-squares program; *DISMAT*, a crystallographic distances and angles program which calculates standard deviations using the correlation matrix from least squares; *ORTEP*, Johnson's (1965) thermal ellipsoid plotting program; *LSPLAN*, our modification of the least-squares planes program from the University of Pittsburgh; and *LIST*1 and *LISTAP*, data presentation programs.

Structure determination and refinement

Normalized structure factors, E_h, were calculated using Wilson's (1942) method. Analysis of the average values of E and E^2-1 strongly indicated a centric space group [$\langle E\rangle = 0{\cdot}792$ *vs.* 0·792 (0·886) and $\langle|E^2-1|\rangle = 0{\cdot}973$ *vs.* 0·968 (0·736) theoretical values for the centric and (acentric) case]. Since the most probable number of molecules in the unit cell was four according to density considerations, this implied that the Mg atom had to lie either on the twofold axis at $x=0$, $z=\frac{1}{4}$ or on a center of symmetry (0,0,0 or $\frac{1}{4},\frac{1}{4},\frac{1}{2}$) in the eightfold space group.

REL, Long's (1965) sign-determination program was used to assign phases to 316 reflections with $E > 1.50$. After some failures, we found an E map which led us to the correct structure by a circuitous route. It was assumed to be a double image of a non-centric structure (in space group Cc) with the two images related by the center of symmetry. Plausible groups of atoms were refined by least-squares and Fourier methods until with 45 atoms (consisting of Mg(DPP)$_2$ and two molecules of DMF in the asymmetric unit of space group Cc) the residual $R_1 = \sum |\Delta F|/\sum |F_o|$ was reduced to 0.16, and a difference Fourier map showed no peaks greater than 0.5 e Å^{-3}. Refinement proceeded slowly.

The slow refinement was explained when it was noticed that the structure was actually very nearly centric, with the center displaced from the origin. After a shift of origin and redefinition of the parameters according to space group $C2/c$, refinement proceeded much more rapidly. Three cycles of least-squares refinement reduced R_1 to 0.154 with only small shifts. Assignment of anisotropic thermal parameters to all the atoms reduced R_1 to 0.141.

The hydrogen atoms were located in ΔF maps with the aid of bond geometry considerations. Those attached to C(17) did not respond well to least-squares refinement, and they were included in the last calcula-
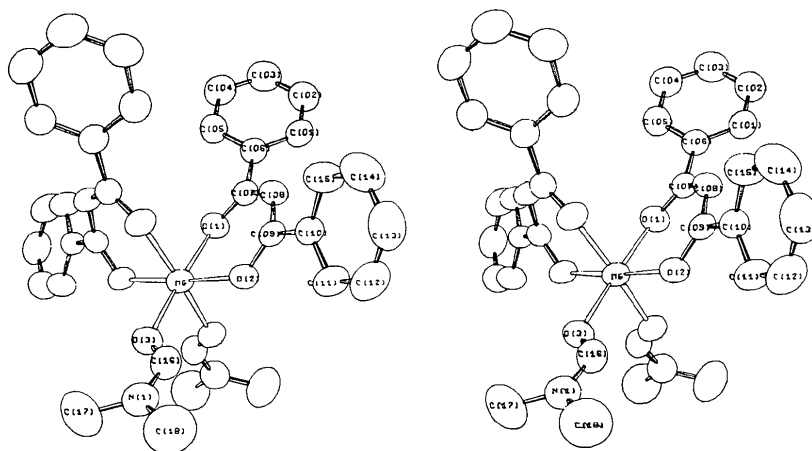

Fig. 1. Stereoscopic view of the complex, showing configuration and labelling. The twofold crystallographic axis runs vertically in the plane of the drawing. Hydrogen atoms have been omitted for clarity. Thermal ellipsoids are scaled to include 50 % probability.

Table 2. *Final coordinates and thermal parameters*

Standard deviations of the least significant digit(s) are given in parentheses. The form of the temperature factor (B in units of Å^2) is $T = \exp [-0.25 (B_{11}h^2a^{*2} + 2B_{12}hka^*b^* + ...)]$ for anisotropic, and $T = \exp (-B \sin^2 \theta/\lambda^2)$ for isotropic thermal parameters.

(a) Heavy atoms (values of coordinates are $\times 10^4$)

	x	y	z	B_{11}	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
Mg	0	3373 (1)	2500	3.68 (8)	3.44 (8)	4.85 (9)	0	2.14 (7)	0
O(1)	−439 (2)	4524 (2)	1553 (2)	3.8 (1)	4.1 (1)	5.3 (1)	−0.14 (9)	2.0 (1)	0.5 (1)
O(2)	1176 (1)	3437 (2)	2419 (2)	4.0 (1)	3.3 (1)	5.7 (1)	0.08 (9)	2.6 (1)	0.3 (1)
C(01)	−231 (3)	6479 (3)	215 (3)	5.1 (2)	4.5 (2)	4.8 (2)	0.3 (2)	2.3 (2)	−0.1 (2)
C(02)	−672 (3)	7344 (4)	−270 (3)	6.9 (3)	5.4 (2)	4.7 (2)	−0.2 (2)	2.3 (2)	0.8 (2)
C(03)	−1258 (3)	7868 (4)	−60 (4)	6.2 (3)	5.2 (2)	6.4 (3)	0.9 (2)	2.0 (2)	1.4 (2)
C(04)	−1402 (3)	7543 (4)	634 (3)	4.7 (2)	5.2 (2)	7.1 (3)	1.4 (2)	2.4 (2)	0.6 (2)
C(05)	−1006 (2)	6647 (3)	1085 (3)	4.3 (2)	5.2 (2)	5.1 (2)	0.5 (2)	2.2 (2)	0.7 (2)
C(06)	−398 (2)	6115 (3)	890 (2)	3.6 (2)	4.0 (2)	4.2 (2)	−0.2 (1)	1.5 (1)	0.0 (1)
C(07)	47 (2)	5173 (3)	1419 (2)	4.1 (2)	3.9 (2)	4.1 (2)	−0.1 (1)	1.9 (1)	−0.3 (1)
C(08)	974 (2)	5087 (3)	1741 (3)	3.7 (2)	3.5 (2)	5.5 (2)	−0.1 (1)	2.3 (2)	0.4 (2)
C(09)	1479 (2)	4250 (3)	2216 (2)	4.2 (2)	3.5 (2)	4.9 (2)	0.1 (1)	2.7 (2)	−0.5 (1)
C(10)	2463 (2)	4253 (3)	2503 (2)	3.6 (2)	3.7 (2)	4.4 (2)	0.1 (1)	2.0 (1)	−0.1 (1)
C(11)	2917 (3)	3328 (3)	2601 (3)	4.3 (2)	4.2 (2)	6.4 (2)	0.1 (2)	2.4 (2)	−0.5 (2)
C(12)	3813 (3)	3321 (5)	2873 (3)	5.0 (2)	6.3 (3)	7.5 (3)	2.2 (2)	3.0 (2)	0.6 (2)
C(13)	4285 (3)	4226 (5)	3074 (3)	3.6 (2)	9.2 (4)	6.8 (3)	0.5 (2)	2.4 (2)	1.0 (2)
C(14)	3860 (3)	5152 (5)	3003 (3)	4.3 (2)	6.2 (3)	7.8 (3)	−1.0 (2)	2.0 (2)	1.0 (2)
C(15)	2956 (3)	5167 (4)	2719 (3)	4.4 (2)	4.2 (2)	7.1 (3)	0.1 (2)	2.0 (2)	0.5 (2)
O(3)	579 (2)	2222 (2)	3473 (2)	4.5 (1)	4.3 (1)	5.5 (1)	0.7 (1)	2.4 (1)	1.0 (1)
C(16)	1330 (3)	1852 (3)	3767 (3)	5.1 (2)	5.5 (2)	4.9 (2)	1.0 (2)	2.5 (2)	0.7 (2)
N(1)	1725 (2)	1250 (3)	4443 (2)	5.6 (2)	5.4 (2)	4.6 (2)	1.5 (1)	1.6 (1)	0.8 (1)
C(17)	1281 (4)	975 (5)	4966 (4)	11.1 (4)	10.5 (4)	9.5 (4)	3.2 (3)	5.9 (3)	4.9 (3)
C(18)	2608 (6)	816 (10)	4699 (7)	7.3 (4)	11.3 (7)	10.4 (6)	4.5 (5)	3.0 (4)	3.3 (5)

Table 2 (cont.)

(b) Hydrogen atoms (values of coordinates are × 10³)

	x	y	z	B
H(01)	16 (3)	615 (3)	8 (2)	5·1 (10)
H(02)	−57 (2)	754 (3)	−75 (3)	6·5 (11)
H(03)	−153 (3)	838 (4)	−44 (3)	7·8 (13)
H(04)	−187 (4)	788 (4)	69 (3)	9·5 (15)
H(05)	−109 (2)	641 (3)	161 (2)	5·1 (9)
H(06)	127 (2)	560 (3)	158 (2)	4·7 (9)
H(07)	258 (2)	275 (3)	238 (2)	4·8 (9)
H(08)	406 (3)	272 (4)	290 (3)	7·9 (14)
H(09)	491 (3)	427 (3)	331 (3)	7·1 (11)
H(10)	416 (3)	573 (3)	314 (3)	5·7 (11)
H(11)	264 (2)	573 (3)	267 (2)	4·3 (9)
H(12)	162 (3)	197 (3)	347 (3)	5·6 (11)
H(13)	291 (4)	100 (5)	533 (5)	11·8 (23)
H(14)	251 (4)	21 (5)	462 (5)	9·4 (25)
H(15)	298 (5)	125 (6)	449 (5)	13·9 (28)
H(16)	181	95	573	12·000
H(17)	78	140	487	12·000
H(18)	109	21	488	12·000

tions with their positional and thermal parameters fixed. All other hydrogen atoms were refined independently with isotropic thermal parameters. In the last cycle of least-squares calculation no parameter shifted more than 10% of its standard deviation. The final R_1 was 0·067 for 1817 reflections. The final $R_2 = [\sum w(\Delta F)^2/\sum wF_0^2]^{1/2}$ was 0·065 and the standard deviation of an observation of unit weight was 1·30. An examination of the ratios of observed and calculated structure factors for strong reflections gave no evidence of secondary extinction effects. A Fourier of ΔF near the end of the refinement showed no peaks larger than 0·17 e Å^{-3}.

Values of F_0 and the final differences are given in Table 1. The final parameters of the atoms are given in Table 2.
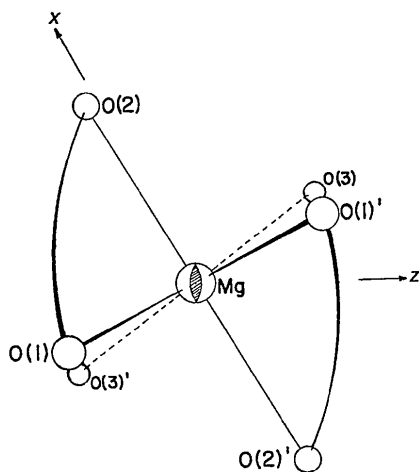

Fig. 2. Oxygen coordination of the Mg atom, showing distortion of the octahedron. The projection is down the twofold axis onto the ac plane.

Results and discussion

The structure is separated into groups with composition Mg(DPP)$_2$(DMF)$_2$. The magnesium ion is in a special position on the twofold axis, and is coordinated to two symmetry-related DPP and two symmetry-related DMF molecules. The two DPP ligands lie above and to either side of the magnesium ion in such a way that their mean planes form a propeller around a twofold axis. The planes of the two DMF molecules are so oriented that there is no indication of a propeller in their configuration (Fig. 1).

The six oxygen atoms coordinating the magnesium ion lie on the corners of a slightly distorted octahedron. The distances of 2·055 (2) and 2·057 (3) Å from the magnesium ion to the oxygen atoms of the DPP ligand are identical within one standard deviation, while the Mg–O distance to the DMF is slightly, but significantly, longer [2·095 (3) Å]. These distances fall well within the range for typical Mg–O distances in the literature (2·0–2·15 Å), with the Mg–O(DPP) distance being shorter than the Mg–O(DMF) as expected from electrostatic interactions. The angles of the octahedron are also distorted (Table 3 and Fig. 2). The angle O(2)–Mg–O(2′) is concave upwards in Fig. 1.

Table 3. Bond angles around magnesium

Standard deviation of each angle is ±0·1°.

O(1)–Mg–O(1)*	87·9°
O(1)–Mg–O(3)	174·0
O(1)–Mg–O(3)*	91·3
O(2)–Mg–O(1)	86·3
O(2)–Mg–O(1)*	90·4
O(2)–Mg–O(2)*	175·4
O(2)–Mg–O(3)	87·8
O(2)–Mg–O(3)*	95·5
O(3)–Mg–O(3)*	90·2

*Related by twofold axis through Mg.

The distances between those oxygen atoms that are coordinated to the Mg ion range from 2·81 to 3·07 Å, with the minimum distance being between the two oxygen atoms in the same DPP ligand. There are several close C···O and C···C contacts within the complex, specifically across the twofold axis, due to the close bonding of the oxygen atoms to the magnesium. Analysis of the least-squares planes through portions of the molecules involved shows that the DPP is tilted so that the Mg atom is 0·58 Å above the plane of the diketone and that the DMF molecules are spread away from the axis so that Mg is above that molecular plane by 0·29 Å. (The distances given are the perpendicular distances to the plane involved. 'Above' implies that the distance from the plane to the Mg has a component upward in Fig. 1.)

The two phenyl groups, the diketone moiety and the DMF molecule are each planar to within five standard deviations of the coordinates of the atoms involved. The two phenyl rings are twisted with respect to the

diketone plane by 47·6 and 30·9° for the first [C(1)–C(6)] and second [C(10)–C(15)] rings respectively. The first phenyl ring is also bent so that C(6) is out of the diketone plane by 0·08 Å.

The average C–C distance in the phenyl rings is 1·376 Å. Other bond distances in the DPP and DMF molecules are as expected (Williams, 1966; Hollander, 1972; Sutton, 1958). The O–C–C and C–C–C angles in the diketone are spread (average 125°) as expected, to give the ligand a bigger bite. In the phenyl groups the interior angle nearest the diketone is significantly less than the ideal 120° (118·8 and 117·5° for the first and second phenyls respectively). The phenyl C–C bond distances to the *para* carbon atoms are also systematically shorter than the C–C bond distances to the other carbons (Table 4).

The complexes themselves are packed in an inter-

Table 4. *Intramolecular and intracomplex distances* (Å)

Standard deviations are given in parentheses.

C(01)···C(02)	1·381 (6)	C(01)···H(01)	0·90 (4)
C(02)···C(03)	1·372 (7)	C(02)···H(02)	0·94 (4)
C(03)···C(04)	1·369 (7)	C(03)···H(03)	0·88 (5)
C(04)···C(05)	1·376 (6)	C(04)···H(04)	0·94 (5)
C(05)···C(06)	1·393 (5)	C(05)···H(05)	1·01 (4)
C(06)···C(01)	1·377 (5)	C(08)···H(06)	0·94 (4)
C(06)···C(07)	1·489 (5)	C(11)···H(07)	0·90 (4)
C(07)···O(1)	1·261 (4)	C(12)···H(08)	0·87 (5)
C(07)···C(08)	1·408 (5)	C(13)···H(09)	0·95 (5)
C(08)···C(09)	1·381 (5)	C(14)···H(10)	0·87 (4)
C(09)···O(2)	1·277 (4)	C(15)···H(11)	0·88 (3)
C(09)···C(10)	1·504 (5)	C(16)···H(12)	0·86 (4)
C(10)···C(11)	1·384 (5)	C(17)···H(16)†	1·19
C(11)···C(12)	1·367 (6)	C(17)···H(17)†	0·96
C(12)···C(13)	1·363 (7)	C(17)···H(18)†	1·03
C(13)···C(14)	1·366 (7)		
C(14)···C(15)	1·377 (6)	C(18)···H(13)	0·98 (6)
C(15)···C(10)	1·389 (5)	C(18)···H(14)	0·79 (6)
O(3)····C(16)	1·228 (5)	C(18)···H(15)	1·02 (8)
C(16)···N(1)	1·288 (5)		
N(1)····C(17)	1·441 (6)	Mg·····O(1)	2·057 (3)
N(1)····C(18)	1·461 (7)	Mg·····O(2)	2·055 (2)
O(1)····O(1)*	2·855 (5)	Mg·····O(3)	2·095 (3)
O(1)····O(2)	2·811 (3)	O(2)····O(3)	2·878 (3)
O(1)····O(2)*	2·917 (3)	O(2)····O(3)*	3·072 (3)
O(1)····O(3)*	2·967 (3)	O(3)····O(3)*	2·968 (5)

* Related by twofold axis through Mg.
† Positions not refined by least squares.

locking manner as shown in Fig. 3. The phenyl rings extending away from the twofold axis [C(10)–C(15)] project into the open space between the other two phenyl rings of a *C*-centering related complex and the DMF molecules of the complex related to the second by a translation in *y*. The phenyl rings directed up the twofold axis with respect to the Mg project into the area just below the DMF molecules of the complex above it. The phenyl moieties also project into the relatively open area to the side of a complex related to it by a center of symmetry, and the DMF ligands into the open side of a complex related by the *n* glide at $y=\frac{1}{4}$.

There are only three short ($< 3\cdot50$ Å) intercomplex contacts between non-hydrogen atoms. They are 3·39 Å from O(1) to C(18) of the complex related to the first by the *n* glide at $y=\frac{1}{4}$, 3·48 Å from O(2) to a C(2) of the complex related by the center of symmetry at $0,\frac{1}{2},\frac{1}{2}$ and 3·34 Å from C(4) to a C(4) of the complex related by the *n* glide at $y=\frac{3}{4}$.

The octahedral coordination of Mg in $Mg(DPP)_2(DMF)_2$ is similar to that exhibited by other β-diketone complexes of divalent metals, *e.g.* diaquo-bis(acetylacetonato)magnesium, $Mg(AA)_2(H_2O)_2$ (Morosin, 1967), $Co(AA)_2(H_2O)_2$ (Bullen, 1959), and $Ni(AA)_2(H_2O)_2$ (Montgomery & Lingafelter, 1964). In each case, the divalent metal cation is octahedrally coordinated by two β-diketone ligands and two solvent molecules in a monomeric unit. In all of these structures, the oxygen atoms of the solvent molecules are significantly further away from the metal ion than are those of the diketone. For the $Mg(DPP)_2$ complex, the distances (2·06, 2·06, 2·10 Å) may be compared with those in the acetylacetonato complexes of Mg (2·03, 2·04, 2·15 Å), Co (2·05, 2·06, 2·23 Å) and Ni (2·02, 2·01, 2·14 Å), the last distance in each case being that of the solvent. This effect is also noted in a dimeric situation with octahedral coordination in $Co(AA)_2(H_2O)$ (Cotton & Elder, 1966), and in cases where the coordination is not octahedral, as in the Ca and Sr DPP complexes (following two papers) and the seven-coordinate $Ho(DPP)_3(H_2O)$ complex (Zalkin, Templeton & Karraker, 1969).

The octahedral complex $Ni(HAA)_2(H_2O)_2(ClO_4)_2$ (Anzenhofer & Hewitt, 1971), in which all the ligands
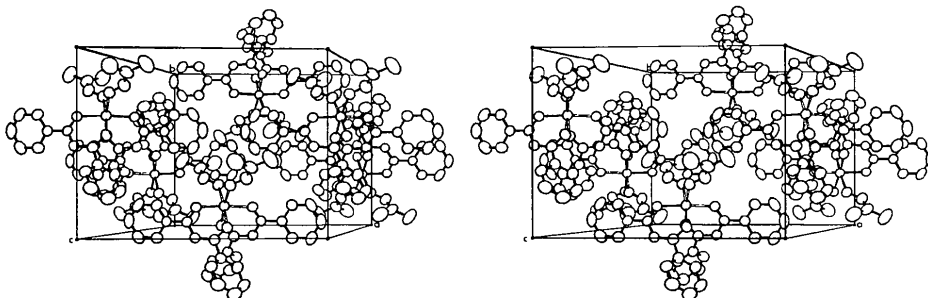

Fig. 3. Stereoscopic view of the unit cell, showing packing of complexes. Labelled axes are positive from the origin. Hydrogen atoms have been omitted for clarity.

on the Ni^{2+} are neutral, does not show this distortion. The distances are 2·07, 2·02, 2·04 Å and 2·04, 2·03, 2·05 Å for the three independent distances around the two different Ni ions, the distance to the water given last in each trio.

The $Mg(DPP)_2$ complex differs from the other octahedral complexes in that the solvent molecules are *cis* to one another on the coordination octahedron (Fig. 1). The solvent molecules are all *trans* to one another in the other monomeric complexes. The *cis* arrangement is not required by the space-group symmetry, which could just as easily accommodate the *trans* configuration, but, while all the other distorted octahedra could be described as tetragonally distorted, the octahedron in $Mg(DPP)_2(DMF)_2$ cannot.

Bullen explains the distortion of the $Co(AA)_2(H_2O)_2$ octahedron in terms of combination of the available *d* orbitals of the Co. The same explanation could hold for the Ni complex as well, but not for the magnesium complexes, since Mg^{2+} has no available *d* orbitals. Morosin concludes in his paper that the effect is due to packing forces rather than electronic ones since the diaquo Mg complex shows the same distortion as the Co and Ni complexes. The appearance of the same effect in $Mg(DPP)_2(DMF)_2$ and in other structures noted above, where the packing and coordination environments are radically different, as well as the absence of any effect in the octahedral nickel acetylacetone perchlorate complex, leads us to suspect that the effect is primarily electrostatic in origin, with secondary contributions from *d*-orbital hybridization and packing.

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Investigations of Alkaline-Earth β-Diketone Complexes.
II. The Crystal and Molecular Structure of
Bis-(1,3-diphenyl-1,3-propanedionato)calcium Hemiethanolate*

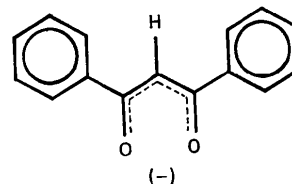BY FREDERICK J. HOLLANDER, DAVID H. TEMPLETON AND ALLAN ZALKIN

Lawrence Berkeley, Laboratory and Department of Chemistry, University of California, Berkeley California 94720, U.S.A.

Bis-(1,3-diphenyl-1,3-propanedionato)calcium hemiethanolate, $Ca[(C_6H_5CO)_2CH]_2(C_2H_5OH)_{1/2}$, crystallizes in space group $P\bar{1}$ with $a = 15\cdot247$ (1), $b = 13\cdot555$ (1), $c = 14\cdot097$ (1) Å, $\alpha = 74\cdot64$ (1), $\beta = 95\cdot97$ (1), $\gamma = 113\cdot59$ (1)°, $Z = 4$, at 23 °C. The density calculated for two units of empirical formula $Ca_2C_{62}H_{50}O_9$ in the unit cell is $d_c = 1\cdot32$ g cm^{-3}. The structure was solved by direct methods and refined to a conventional R value of 0·040 for 4503 reflections collected by counter methods. The complex consists of a centrosymmetric cluster containing four calcium atoms, two with six oxygen neighbors each and two with seven.

Introduction

Alkaline-earth metal cations form complexes with the anion of 1,3-diphenyl-1,3-propanedione: