Submitted to Acta Cryst.

RECEIVED
LAWRENCE
RADIATION LABORATORY

NOV 20 1972

LIBRARY AND
DOCUMENTS SECTION

LBL-1267
Preprint

INVESTIGATIONS OF ALKALINE-EARTH β-DIKETONE COMPLEXES. II. THE CRYSTAL AND MOLECULAR STRUCTURE OF BIS(1, 3-DIPHENYL-1, 3-PROPANEDIONATO) CALCIUM HEMIETHANOLATE

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

September 1972

AEC Contract No. W-7405-eng-48

For Reference

Not to be taken from this room

LBL-1267

DISCLAIMER

This document was prepared as an account of work sponsored by the United States Government. While this document is believed to contain correct information, neither the United States Government nor any agency thereof, nor the Regents of the University of California, nor any of their employees, makes any warranty, express or implied, or assumes any legal responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by its trade name, trademark, manufacturer, or otherwise, does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof, or the Regents of the University of California. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof or the Regents of the University of California.

0 0 0 0 3 8 0 7 7 7 8

LBL-1267

Investigations of Alkaline-earth β-diketone Complexes. II. The crystal and molecular structure of bis(1,3-diphenyl-1,3-propanedionato)calcium hemiethanolate*

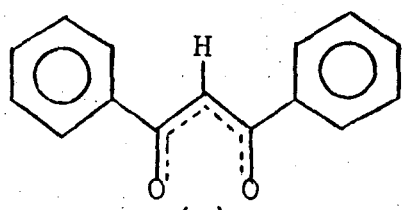
By Frederick J. Hollander, David H. Templeton and Allan Zalkin
Lawrence Berkeley Laboratory and Department of Chemistry,
University of California, Berkeley, California 94720 U.S.A.

Bis(1,3-diphenyl-1,3-propanedionato)calcium hemiethanolate, $Ca[(C_6H_5CO)_2CH]_2(C_2H_5OH)_{1/2}$, crystallizes in $\underline{P\bar{1}}$ with $\underline{a}$ = 15.247(1) Å, $\underline{b}$ = 13.555(1) Å, $\underline{c}$ = 14.097(1) Å, α = 74.64°(1), β = 95.97(1)°, γ = 113.59(1)°, $\underline{Z}$ = 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 $\underline{d_c}$ = 1.32 gm/cm^3. The structure was solved by direct methods and refined to a conventional $\underline{R}$ value of 0.040 for 4503 reflections collected by counter methods. The complex forms a novel tetrameric polymer around a crystallographic center of symmetry. There are two Ca atom environments, one six- and one seven-fold coordinated, with the ethanol providing the seventh member of the latter coordination. The form of the polymer is described in detail.

*Work performed under the auspices of the U.S. Atomic Energy Commission.

Introduction

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



(–)

We succeeded in crystallizing the calcium complex only when ethanol was present.

This paper reports the structure of bis(1,3-diphenyl-1,3-propanedionato)calcium hemiethanolate, $Ca(DPP)_2(C_2H_5OH)_{1/2}$. The complex exists as a novel tetrameric polymer around a crystallographic center of symmetry. The ethanol is bound to one of the two independent calcium atoms in the asymmetric unit.

Experimental Procedure

The complex was synthesized by mixing a solution of calcium acetate in aqueous ethanol with an ethanolic solution of 1,3-diphenyl-1,3-propanedione (HDPP) and adding concentrated NH_3/NH_4Cl, pH 10, buffer. A lemon yellow precipitate appeared immediately upon addition of the buffer solution. The mixture was heated to reflux for one hour to allow the reaction to go to completion. The precipitate dissolved on heating, but reappeared as the mixture was

cooled. It was filtered and dried in air and stored in a desiccator over Drierite. Attempts to recrystallize the complex generally yielded a viscous orange oil. However, well-formed clear yellow crystals were obtained by slow evaporation of a warm chloroform solution. The oil dissolved in many organic solvents, but formed a cloudy solid on addition of ethanol.

The crystals were mounted on glass fibers. Precession photographs indicated a triclinic unit cell, and preliminary cell dimensions were determined. Another crystal was mounted on a Picker/Nuclear four-circle diffractometer automated with a DEC PDP 8I computer and disc. Twelve high-angle reflections were carefully centered using monochromatized Cu $K\alpha_1$ radiation (λ = 1.54051 Å) at 23°C. The cell dimensions were refined on the 2θ measurements only, using program TTHCEL, a modified version of a program supplied to us by H. Hope. The program minimizes $\Sigma w(d_o^{*2} - d_c^{*2})^2$, where the weights are assigned subjectively by the user. The cell parameters obtained from this refinement were a = 15.247(1) Å, b = 13.555(1) Å, c = 14.097(3) Å, α = 74.64(1)°, β = 95.97(1)°, γ = 113.59(1)°. Calculation of the density of the compound for a cell volume of 2576 Å^3 gave, for the formula $Ca(DPP)_2$, d_c = 0.314 Z. Attempts to measure the density failed because a solution could not be found which neither dissolved the crystals nor caused them to decompose, but observations indicated

that the most reasonable value of $\underline{Z}$ was 4. This assumption was confirmed by the refinement of the structure, and the density based on the actual composition is $\underline{d_c}$ = 1.32 gm/cm^3.

A crystal approximately 0.12 mm in radius was used for data collection. Data were collected using monochromatized ($2\theta_{\underline{m}}$ = 26.42°) Cu $\underline{K}\alpha$ radiation and a θ-2θ scan technique. The peaks were scanned at a rate of 1°/min from 1.0° below the calculated $\underline{K}\alpha_1$ position to 1.0° above the calculated $\underline{K}\alpha_2$ position. Backgrounds were counted for 10 seconds at positions offset by 0.5° from each end of the scan (all angles 2θ). Aluminum attenuators were automatically inserted into the diffracted beam whenever the count rate exceeded 10000 cps and the peak was rescanned and backgrounds remeasured with the attenuators in place. The hemisphere of data $\pm\underline{h},\pm\underline{k},\pm\ell$ was collected for 2θ = 0°-100° ($\sin\theta/\lambda < 0.500$). Three reflections, (4 0 0), (0 3 0) and (0 0 3), were measured after every 100 data to monitor crystal decay. In the entire experiment 5824 reflections were measured, including remeasurements in connection with the decay correction. The standards showed a gradual fall in relative intensity from 1.00 to 0.942 over the first 3973 reflections, then a sharp drop to a relative intensity of 0.820 between reflections 3973 and 3974 caused by a malfunction in the equipment. The exact location of the change was determined by taking

the data in that region again, after the data set was completed. The data were processed as previously described (Hollander, Templeton and Zalkin, 1973a) to give 5294 unique reflections of which 4504 had $\underline{F}^2 > \sigma(\underline{F}^2)$. No correction was made for absorption. With μ = 23 cm^{-1}, a 33% deviation from spherical shape would result in a 15% variation in transmission factors, and the crystal is believed to have been more nearly spherical than that.

The atomic scattering factors of Doyle and Turner (1968) were used for Ca^{++}, O and C, along with the real and imaginary dispersion terms of Cromer and Liberman (1970). The spherical scattering factors of Stewart, Davidson and Simpson (1965) were used for the hydrogen atoms.

Our least-squares program minimizes the function $\Sigma \underline{w}(\Delta \underline{F})^2 / \Sigma \underline{w} \underline{F}_o^{\,2}$. Weights in the refinement were zero if $\underline{F}^2 < \sigma(\underline{F}^2)$ and $\underline{w} = 1/\sigma^2(\underline{F})$ otherwise; $\sigma(\underline{F})$ was calculated as noted in a previous paper (Hollander, Templeton and Zalkin, 1973a). The factor $\underline{p}$, used to reduce the weight of strong reflections, was 0.0 through most of the refinement but 0.05 in the last cycles.

Programs used in the structure analysis have been listed in the above referenced paper.

Structure Determination and Refinement

The assumption of the centric space group $P\bar{1}$ was made throughout, and is confirmed by the solution and successful refinement of the structure. Normalized structure factors, E_h, were generated in the usual manner. Phases were assigned to the 333 E values greater than 2.00 using R. E. Long's (1965) program. After some difficulty, an E-map was obtained which revealed two large peaks and a number of smaller peaks in configurations identifiable as portions of DPP molecules. The 41 atoms found on the E-map were then refined by full-matrix least-squares with isotropic thermal parameters. Two cycles of refinement brought $R_1 = \Sigma |\Delta F| / \Sigma |F_o|$ to 0.41, and the generated signs of the F's were used to phase a Fourier map of F_o. This Fourier showed 69 peaks and one shoulder in chemically reasonable positions for the expected formulation of $Ca(DPP)_2$ with two Ca atoms in the asymmetric unit. The seventy atoms were refined as before for three cycles, and for three cycles with the two Ca atoms and eight O atoms allowed anisotropic thermal parameters, to an R_1 of 0.21. A difference Fourier showed three major peaks with heights of 6 $e^-/Å^3$, 3 $e^-/Å^3$ and 3 $e^-/Å^3$, and no other peaks as high as 1 $e^-/Å^3$. The three peaks were linked together with the largest peak at a distance of 2.41 Å from Ca(1). The assumption made, which was subsequently confirmed by refinement, was that the peaks represented an ethanol

coordinated to the Ca. The atoms of the ethanol were put into least-squares with isotropic thermal parameters, together with the calculated positions of the 44 hydrogens on the DPP ligands. Each hydrogen was given an isotropic thermal parameter equal to the average thermal parameter of the ring it was bound to, and neither its position nor thermal parameter was allowed to change. The ethanol atoms refined well, and R_1 was reduced to 0.09.

Anisotropic refinement of the non-hydrogen atoms and isotropic refinement of the hydrogens proceeded by block-diagonal least-squares with three blocks to an R_1 of 0.048. Availability of a larger computer allowed further refinement in two blocks, the first containing the two "inner" DPP molecules and the second containing the two "outer" DPP molecules and the ethanol. The overall scale factor and the two Ca atoms were refined with each block.

Certain H atoms behaved poorly and some of their parameters were fixed to allow convergence (see Table 3). Two final cycles of each block brought the shifts of all parameters to less than 20% of their standard deviations and most to less than 10% of their standard deviations. The final R_1 was 0.040 for 4503 reflections, and $R_2 = (\Sigma w(\Delta F)^2/\Sigma wF_o^2)^{1/2}$ was 0.043. The standard deviation

of an observation of unit weight was 1.14. The data were examined for secondary extinction effects with negative results.

The observed structure factors, their standard deviations and the final differences are given in Table 1. The final coordinates and thermal parameters of the atoms are given in Tables 2 and 3.

Results and Discussion

The complex consists of clusters of composition $(Ca_2(DPP)_4(C_2H_5OH))_2$ around the symmetry at 0,1/2,1/2. There is only one such cluster per unit cell, and its neighbors are related to it by unit cell translations. There are no contact distances of less than 3.50 Å between non-hydrogen atoms in different clusters.

The two independent calcium atoms Ca(1) and Ca(2) are respectively six- and seven-fold coordinated by the oxygens of the DPP ligands and the ethanol (Fig. 1). Each calcium shares two oxygens with each of two neighboring calcium atoms of the other kind so that four of them are linked around the center of symmetry. The bridging oxygens, O(1) and O(2) of DPP(1), O(3) and O(4) of DPP(2), form a slightly distorted parallelopiped, whose edge lengths are listed in Table 4. Ca atoms cap each of four nearly rectangular faces, and DPP ligands project into the area above the two non-rectangular faces (Fig. 2).

The angles from the uncapped plane (core plane 1) to the two planes capped by Ca(1) and Ca(2) (core planes 2 and 3 respectively) are 89.1° and 89.4°. The angle between the two capped planes is 69.1°.

The non-bridging oxygens (O(5) and O(6) of DPP(3), O(7) and O(8) of DPP(4) and O(9) of the ethanol) and the calcium atoms lie on the outward sides of the core planes. Ca(1) is 1.03 Å above core plane 2, and the two oxygens of DPP(3) are 3.09 Å and 2.55 Å away from that plane. Ca(2) is 1.39 Å from core plane 3. The other three oxygens coordinated to Ca(2) are at distances of 2.90 Å, 2.80 Å and 2.82 Å from core plane 3 for O(7), O(8) and O(9) respectively, forming an almost parallel plane. The distances between core planes 1, 2 and 3 and the respective planes related by the center of symmetry are 2.90 Å for core plane 1, 2.60 Å for core plane 2 and 2.87 Å for core plane 3.

The Ca-O distances average 2.367 Å with a standard deviation of 0.026 Å calculated from the scatter of the measurements. The average compares well to the sum of the crystal radii (Pauling, 1960), 2.39 Å. The range of the Ca-O distances is from 2.256(2) Å to 2.508(2) Å. The Ca(1)-O distances are in general shorter than the Ca(2)-O distances. The distances from Ca to the unbridged oxygens are generally shorter than those to the bridging oxygens, but the Ca(1)-O(6) distance of 2.395 Å is an exception.

These variances from the averages are in keeping with the coordinations of the atoms involved, with longer bond distances to atoms with higher coordination.

The distances of the Ca atoms to the planes of the diketone portions of the inner DPP ligands are on the same order as the distances from the Ca atoms to the core planes. For the non-bridging DPP ligands the Ca atoms are much closer to the planes of the diketones. For DPP(3), the distance from the plane to Ca(1) is only 0.19 Å, while in the more crowded environment around Ca(2) the distance from Ca(2) to the diketone plane of DPP(4) is 0.24 Å. The planes of the diketone residues of DPP(1) and DPP(2) are spread away from one another, making angles with core plane 1 of 95.3° and 107.9° respectively, and an angle of 23.2° with respect to each other. DPP(1), which experiences crowding from DPP(4), is forced close to perpendicularity with core plane 1, while DPP(2) can bend more, away from DPP(1) into the emptier area around the ethanol. The DPP(3) diketone ring is almost perpendicular (89.2°) to core plane 2, reflecting its almost symmetrical surroundings with respect to core plane 1 and the symmetry-related core plane 1'. However, the distances of O(5) and O(6) to core plane 2 show that there is strong interaction between DPP(3) and DPP(4) forcing O(5) away from core plane 2 and O(6) towards it.

The diketone plane of DPP(4) comes in at a strong angle to core plane 3 (46.6°) in order to leave room for the ethanol, which makes an angle of 79.5° with core plane 3 and 53.9° with the diketone plane of DPP(4).

The DPP(n) molecules themselves can be analyzed in terms of three planes each, phenyl 1 and phenyl 2 (1-6 and 10-15; Fig. 3) and the diketone plane (7-9 and O's 1 and 2; Fig. 3). All the phenyl groups of the DPP ligands are planar to within five standard deviations of the coordinates of the atoms and most of them are within three standard deviations of planarity. The diketone planes of DPP(2) and DPP(3) are within six standard deviations of planarity, but the diketone portions of DPP(1) and DPP(4) are strongly twisted and are only planar to 15 standard deviations. The phenyl groups are rotated with respect to the diketone planes by angles ranging from 5.6° to 36.6° (Table 5). This rotation is due to packing forces between the clusters and between ligands within the clusters. The angles of rotation do not cause any significant variation in the phenyl-diketone bond length or the stressed interior angle adjacent to the diketone in the phenyl groups.

Interatomic distances in the DPP ligands and the ethanol are shown in Table 6. The average phenyl C-C distance is 1.374 Å, in reasonable agreement with literature

values, and the C-C distance in the diketone averages to 1.396 Å, again in good agreement. The average C-C distance between the phenyl groups and the diketone residue of 1.502 Å agrees well with other values for $\underline{sp}^2$-$\underline{sp}^2$ single bonds, and the C-O average distance of 1.273 Å is in reasonable agreement for a C-O bond order of 1.5 (Pauling, 1960). The standard deviations of these averages as calculated from the scatter of the various independent distances are all on the order of 0.003 Å.

There is a statistically but probably not chemically significant difference in the C-O bond lengths between those involving bridging oxygens and those involving non-bridging oxygens. The average C-O distance for the former is slightly longer at 1.281(2) Å than the average C-O distance for the latter at 1.264(3) Å. The phenyl C-C bonds also show a statistically significant variation. The bonds to the <u>para</u> carbon average to a low 1.363(2) Å compared to the averages of the <u>ortho</u>-<u>meta</u> bonds (1.382(2) Å) and the bonds to the carbon adjacent to the diketone (1.379(3) Å). The average of all C-H bonds in the DPP molecules is 0.94 Å with a spread of ±0.09 Å, in agreement with values for many X-ray studies.

The bond angles in the ligands are normal for complexed DPP molecules (Zalkin, Templeton and Karraker, 1969; Hollander, Templeton and Zalkin, 1973a, 1973b). The diketone ring is spread, with O-C-C and C-C-C bond angles

~125°, to give the ligand a bigger "bite," and the interior angle in the phenyl ring at the juncture to the diketone is reduced from 120° to ~118°.

The C-O and C-C bond distances in the ethanol appear greatly shortened, probably due to the very large amounts of anisotropic thermal motion (Fig. 2, Table 2).

References

Cromer, D. T. and Liberman, D. (1970). J. Chem. Phys. 53(5), 1891.

Doyle, P. A. and Turner, P. S. (1968). Acta Cryst. A24, 390.

Hollander, F. J., Templeton, D. T. and Zalkin, A. (1973a). In press. ($Mg(DPP)_2$)

Hollander, F. J., Templeton, D. T. and Zalkin, A. (1973b). In press. ($Sr(DPP)_2$)

Long, R. E. (1965). Ph.D. Thesis, University of California, Los Angeles, Calif.

Pauling, L. (1960). The Nature of the Chemical Bond, Third Ed., Cornell University Press, Ithaca, N. Y.

Stewart, R. F., Davidson, E. R. and Simpson, W. T. (1965). J. Chem. Phys. 42, 3175.

Zalkin, A., Templeton, D. T. and Karraker, D. G. (1969). Inorg. Chem. 8(12), 2680.

Table 1. Observed structure factors, standard deviations and final differences (x 10.0). Reflections marked with an * were given zero weight in least-squares.

(Three pages, to be reproduced photographically.)

OBSERVED STRUCTURE FACTORS, STANDARD DEVIATIONS, AND DIFFERENCES (X10.0) FOR
C41OHP12-172 C2H5OH F(O)=0.03 *10663
SG = ESTIMATED STANDARD DEVIATION OF FOB. DFL = /FOBS/-/FCALC/. * INDICATES ZERO WEIGHTED DATA.

OBSERVED STRUCTURE FACTORS FOR C4H9PP12-1/2 C2H5OH (CONTINUED)
PAGE 2

18

OBSERVED STRUCTURE FACTORS FOR CA(DPP)2-1/2 C2H5OH (CONTINUED)

L	FOB	SG	DEL
-3	226	9	1
-2	118	7	2
-1	187	8	10
0	59	14	-8
1	69	19	-4
2	85	15	3
3	46	48	10*
4	42	16	25
5	176	6	6
6	244	7	13
7	50	52	-2*
8	35	34	17*
9	63	28	-12
H,K= 8, 5			
-6	93	16	-2
-5	61	30	33
-4	60	31	-15
-3	534	13	-11
-2	242	7	2
-1	34	41	1*
0	27	38	-25*
1	156	10	-15
2	71	21	3
3	95	9	-13
4	139	6	-1
5	86	8	-13
6	371	10	0
7	257	9	-11
8	45	17	1
9	121	12	17
H,K= 8, 6			
-4	72	23	-27
-3	0	52	-34*
-2	208	9	-5
-1	290	6	-12
0	130	17	-11
1	46	52	45*
2	28	55	19*
3	70	30	-8
4	124	7	-1
5	36	52	-17
6	84	9	6
7	95	8	8
H,K= 8, 7			
-2	65	11	-9
-1	100	15	14
0	150	6	-3
1	94	16	-1
2	0	52	-56*
3	85	9	15
4	78	11	-18
5	153	7	-6
H,K= 9,-13			
-5	29	35	11*
-4	4	35	-9*
-3	18	35	-8*
-2	52	14	3
H,K= 9,-12			
-8	165	6	-8
-7	112	14	-24
-6	65	8	48
-5	193	6	-7
-4	143	11	3
-3	12	35	6*
-2	27	35	8*
-1	152	6	-4
0	147	11	-6
1	131	12	-5
H,K= 9,-11			
-9	194	6	2
-8	218	7	-17
-7	217	9	8
-6	0	52	-19*
-5	28	15	-3*
-4	0	38	-14*
-3	66	26	-12
-2	172	6	6
-1	17	26	10
0	176	6	18
1	0	52	-14*
2	73	10	1
3	212	9	2
H,K= 9,-10			
-10	138	14	-15
-9	298	9	-0
-8	23	52	-4*
-7	262	8	3
-6	165	10	-6
-5	119	7	-3
-4	51	13	11
-3	135	10	-2
-2	64	41	-12*
-1	0	37	-50*
0	74	9	15
1	276	9	-8
2	97	12	-3
3	40	22	4
4	329	9	-15
5	86	8	2
H,K= 9, -9			
-11	162	6	7
-10	176	7	-18
-9	31	52	16*
-8	34	9	7
-7	51	19	-3*
-6	0	49	-11*
-5	0	35	-3*
-4	72	31	-6
-3	63	9	12
-2	248	9	1
-1	72	13	12
0	219	7	-1

L	FOB	SG	DEL
1	162	9	-5
2	171	6	5
3	149	19	1
4	120	7	11
5	272	9	-17
6	109	7	-7
H,K= 9, -8			
-11	56	37	2
-10	159	10	11
-9	310	12	-4
-8	365	10	-15
-7	203	7	-19
-6	58	13	4
-5	92	13	0
-4	147	6	6
-3	258	7	3
-2	325	9	3
-1	196	8	-8
0	67	10	-5
1	269	8	4
2	70	9	6
3	208	13	4
4	50	51	-10*
5	377	11	-6
6	236	9	-13
7	0	55	-9*
H,K= 9, -7			
-11	79	20	6
-10	310	9	3
-9	725	19	-20
-8	478	11	-31
-7	92	8	17
-6	275	8	-5
-5	143	8	-11
-4	96	7	-4
-3	251	8	8
-2	301	9	6
-1	100	10	-13
0	0	63	-16*
1	20	64	16*
2	293	9	-6
3	289	8	27
4	243	8	-0
5	129	6	9
6	261	8	-8
7	75	9	-9
8	41	20	-11
H,K= 9, -6			
-12	40	52	0*
-11	123	13	-3
-10	0	42	-47*
-9	261	8	-8
-8	126	10	7
-7	361	10	-5
-6	55	12	42
-5	461	12	-15
-4	396	10	-3
-3	61	10	2
-2	375	10	6
-1	20	41	2*
0	0	33	-37*
1	46	28	9
2	255	8	4
3	155	6	8
4	208	12	-2
5	180	9	-6
6	55	35	-4
7	43	52	37*
8	143	10	-10
H,K= 9, -5			
-12	87	19	2
-11	52	14	26
-10	147	11	9
-9	215	9	-2
-8	0	39	-17*
-7	215	8	-1
-6	68	40	2
-5	433	11	3
-4	357	10	2
-3	90	7	1
-2	25	37	11*
-1	44	25	-19
0	151	7	4
1	135	5	-0
2	140	6	2
3	414	12	1
4	21	64	-18*
5	75	8	-0
6	64	10	14
7	190	6	3
8	177	6	-5
9	0	35	-15*
H,K= 9, -4			
-11	217	7	-10
-10	30	39	-14*
-9	32	51	3*
-8	93	14	11
-7	114	11	6
-6	60	62	-6*
-5	179	7	-8
-4	354	10	-17
-3	47	13	-11
-2	86	9	-12
-1	65	13	-22
0	160	6	-2
1	169	7	1
2	64	6	-1
3	122	6	10
4	233	11	1
5	108	11	-2
6	252	6	-19
7	212	7	-15

L	FOB	SG	DEL
8	252	8	-7
9	170	6	4
H,K= 9, -3			
-11	322	12	-15
-10	0	73	-77*
-9	68	71	27*
-8	34	15	34
-7	41	15	20
-6	290	9	-2
-5	69	15	-3
-4	304	9	-2
-3	51	10	6
-2	207	7	-6
-1	336	9	1
0	78	10	18
1	92	8	-5
2	72	7	7
3	48	11	-14
4	142	8	-1
5	117	10	-6
6	84	9	-15
7	146	10	4
8	66	25	8
9	140	6	4
10	102	7	-11
H,K= 9, -2			
-11	150	10	-2
-10	0	39	-7*
-9	0	41	-27*
-8	114	12	3
-7	29	48	4*
-6	230	8	-8
-5	81	13	2
-4	21	42	6*
-3	139	6	3
-2	107	6	10
-1	267	8	9
0	34	21	7
1	31	32	-15*
2	132	5	-7
3	146	6	4
4	35	43	0*
5	175	13	-6
6	157	9	15
7	14	37	-24*
8	57	12	7
9	121	7	-5
10	95	16	-11
H,K= 9, -1			
-10	59	32	-12
-9	138	7	5
-8	92	16	-12
-7	96	10	-12
-6	56	26	17
-5	360	10	7
-4	33	35	8*
-3	144	8	-1
-2	215	6	6
-1	595	15	1
0	160	6	-3
1	275	8	2
2	397	10	6
3	37	42	15*
4	17	33	-1*
5	96	12	-5
6	157	6	-6
7	286	9	0
8	28	35	-4*
9	37	35	-11*
10	55	38	-22
H,K= 9, 0			
-10	125	12	-3
-9	49	53	23*
-8	236	8	-6
-7	151	7	10
-6	213	13	15
-5	27	38	-23*
-4	92	12	5
-3	240	10	-0
-2	137	7	-0
-1	639	17	-3
0	414	11	0
1	173	8	-0
2	179	7	-3
3	92	11	5
4	25	44	15*
5	244	7	-4
6	139	6	9
7	11	50	-20*
8	101	7	-6
9	68	10	6
10	0	35	-20*
H,K= 9, 1			
-9	158	10	-6
-8	165	11	5
-7	213	7	3
-6	129	11	-9
-5	74	19	-4
-4	94	10	1
-3	213	8	-5
-2	30	36	-19*
-1	191	6	5
0	313	9	-6
1	79	29	-10
2	0	35	-56*
3	53	12	14
4	25	9	-1
5	106	12	-15
6	327	9	-7
7	151	6	6
8	74	9	-2
9	135	7	-7

L	FOB	SG	DEL
10	0	35	-22*
H,K= 9, 2			
-8	236	7	2
-7	217	9	-3
-6	81	9	17
-5	143	8	3
-4	312	11	4
-3	81	16	6
-2	178	8	-7
-1	41	20	19
0	40	19	11
1	224	8	-2
2	54	13	-4
3	0	65	-11*
4	235	12	-5
5	149	9	6
6	68	12	-13
7	149	6	-4
8	115	7	2
9	35	52	19*
H,K= 9, 3			
-7	42	52	-16*
-6	0	74	-14*
-5	74	12	-21
-4	95	19	-8
-3	70	11	16
-2	295	8	-4
-1	230	7	3
0	125	10	2
1	72	19	-4
2	0	36	-5*
3	40	18	11
4	92	9	4
5	289	9	2
6	296	8	-2
7	181	6	10
8	65	26	3
9	79	9	-1
H,K= 9, 4			
-6	37	52	-5*
-5	124	8	-13
-4	76	9	19
-3	285	8	-3
-2	294	9	1
-1	66	13	9
0	122	12	5
1	24	50	7*
2	98	14	18
3	170	9	-11
4	33	51	-2*
5	317	9	-1
6	292	8	2
7	68	10	16
8	48	16	2
H,K= 9, 5			
-4	132	6	-6
-3	123	13	4
-2	125	7	3
-1	176	9	-2
0	80	20	6
1	70	10	9
2	67	52	-10*
3	52	52	25*
4	156	6	8
5	13	35	-7*
6	87	8	-11
7	25	35	-25*
H,K= 9, 6			
-2	90	6	26
-1	30	35	5*
0	99	8	7
1	106	7	-2
2	119	7	7
3	202	7	16
4	110	7	8
5	107	7	-6
H,K= 10,-12			
-7	0	52	-7*
-6	50	15	-8
-5	109	14	4
-4	166	7	-13
-3	35	35	8*
-2	65	11	3
-1	300	9	-10
0	146	6	-4
H,K= 10,-11			
-9	225	7	-10
-8	237	9	-7
-7	91	8	2
-6	0	52	-14*
-5	32	35	13*
-4	170	10	2
-3	223	9	-11
-2	16	52	-33*
-1	44	24	-28
0	278	9	10
1	40	52	-24*
2	90	17	12
3	176	9	-2
H,K= 10,-10			
-10	84	10	-22
-9	206	7	-12
-8	118	7	15
-7	149	6	2
-6	144	20	-14
-5	190	6	-3
-4	145	7	-2
-3	157	9	3
-2	65	16	13
-1	44	51	7*
0	105	7	-1
1	43	18	20

L	FOB	SG	DEL
2	177	17	-18
3	130	8	-10
4	215	9	0
H,K= 10, -9			
-10	167	10	-10
-9	0	39	-27*
-8	85	41	-4
-7	258	9	1
-6	293	8	2
-5	0	37	-12*
-4	137	7	-7
-3	223	8	5
-2	258	8	-1
-1	159	6	-1
0	219	7	5
1	103	28	2
2	421	11	-5
3	43	16	10
4	414	11	-6
5	166	10	2
H,K= 10, -8			
-11	18	39	-20*
-10	119	13	6
-9	53	48	21
-8	355	10	-6
-7	328	12	-10
-6	0	40	-35*
-5	68	48	-2*
-4	11	47	-6*
-3	151	16	1
-2	240	8	3
-1	59	26	12
0	30	47	11*
1	258	12	4
2	37	90	7*
3	395	11	12
4	237	7	-3
5	196	9	12
6	34	35	2*
H,K= 10, -7			
-11	25	39	-16*
-10	316	9	-2
-9	24	39	10*
-8	116	12	-6
-7	219	18	0
-6	282	8	-12
-5	231	7	6
-4	174	6	9
-3	134	6	3
-2	254	7	-3
-1	115	6	-0
0	268	8	11
1	39	47	1*
2	178	8	3
3	187	6	6
4	328	9	1
5	233	7	-10
6	35	52	19*
7	25	35	-10*
H,K= 10, -6			
-10	79	20	14
-9	54	20	-22
-8	199	9	-8
-7	119	8	6
-6	274	8	0
-5	264	8	9
-4	187	8	8
-3	79	8	15
-2	71	35	17
-1	201	7	-1
0	265	8	-5
1	8	34	-26*
2	186	6	9
3	109	8	-0
4	307	9	4
5	53	8	12
6	59	12	-3
7	198	6	7
8	137	6	-5
H,K= 10, -5			
-11	30	52	9*
-10	33	34	-4*
-9	117	13	20
-8	61	16	6
-7	315	9	-2
-6	123	10	3
-5	243	8	-1
-4	179	8	-1
-3	171	6	1
-2	55	60	-0*
-1	366	10	5
0	393	11	16
1	197	6	13
2	32	45	-6*
3	68	18	0
4	154	9	-6
5	241	7	10
6	139	11	-2
7	291	8	-8
8	395	11	-18
H,K= 10, -4			
-11	176	9	-6
-10	0	39	-1*
-9	160	10	-1
-8	6	41	-13*
-7	428	11	-6
-6	171	6	-11
-5	363	8	-6
-4	41	21	6
-3	17	76	11*
-2	292	7	11

L	FOB	SG	DEL
-1	287	8	18
0	369	10	-0
1	130	8	-6
2	177	6	5
3	130	6	4
4	41	15	37
5	87	9	12
6	18	91	-18*
7	54	13	-1
8	374	10	-8
9	116	13	4
H,K= 10, -3			
-10	96	16	-12
-9	60	15	6
-8	138	7	6
-7	45	49	15*
-6	265	8	1
-5	56	14	7
-4	86	7	14
-3	76	8	24
-2	30	34	7*
-1	50	23	12
0	265	7	6
1	223	7	1
2	0	44	-20*
3	177	8	1
4	173	6	6
5	320	9	-4
6	164	6	8
7	153	7	-15
8	48	52	26*
9	47	57	11*
H,K= 10, -2			
-10	45	14	3
-9	238	7	-11
-8	82	10	13
-7	129	8	-16
-6	88	10	8
-5	0	47	-16*
-4	67	45	-4
-3	142	6	-2
-2	407	11	-14
-1	345	9	-1
0	322	9	9
1	107	10	21
2	71	15	-6
3	27	63	-10*
4	261	8	3
5	95	14	8
6	359	10	-6
7	56	34	28
8	136	11	22
9	48	52	5*
H,K= 10, -1			
-10	150	7	-10
-9	376	12	-32
-8	173	10	-11
-7	106	28	-19
-6	131	8	-2
-5	80	16	-9
-4	149	8	0
-3	175	8	-13
-2	455	13	5
-1	294	8	-14
0	78	14	-8
1	0	44	-6*
2	33	34	12*
3	172	8	-0
4	36	67	23*
5	86	34	3
6	256	12	-2
7	149	6	3
8	79	9	-9
9	38	23	-12
H,K= 10, 0			
-9	161	7	-8
-8	297	8	-8
-7	138	7	-2
-6	217	7	-4
-5	79	10	14
-4	72	19	12
-3	154	9	-5
-2	159	6	-5
-1	154	8	-3
0	41	45	-1*
1	26	34	10*
2	7	34	-9*
3	185	6	-5
4	106	12	5
5	256	8	-12
6	180	9	-9
7	0	52	-61*
8	56	36	7
9	95	16	-11
H,K= 10, 1			
-8	74	9	22
-7	0	39	-31*
-6	138	11	-6
-5	328	12	-8
-4	90	10	-12
-3	258	8	2
-2	357	10	-3
-1	20	47	-1*
0	0	47	-28*
1	214	8	-6
2	0	35	-37*
3	147	9	-7
4	172	7	-3
5	14	35	-10*
6	313	9	-6
7	155	10	-12
8	35	35	-9*

L	FOB	SG	DEL
9	124	6	-1
H,K= 10, 2			
-7	116	8	-15
-6	72	12	-13
-5	179	7	3
-4	445	12	-4
-3	236	8	3
-2	517	14	-4
-1	119	11	-7
0	63	12	-4
1	126	7	-9
2	75	9	11
3	74	18	10
4	283	12	-8
5	357	11	-19
6	204	9	-9
7	156	6	-4
8	34	35	-10*
H,K= 10, 3			
-6	151	10	-13
-5	143	7	-7
-4	45	19	-3
-3	237	7	-8
-2	614	16	4
-1	523	17	-10
0	44	51	16*
1	86	10	-2
2	112	7	3
3	36	72	13*
4	150	10	-14
5	217	7	9
6	0	33	-14*
7	100	7	1
8	41	20	1
H,K= 10, 4			
-4	98	15	3
-3	65	11	11
-2	57	13	33
-1	0	52	-8*
0	60	15	-19
1	40	22	17
2	171	10	6
3	238	9	10
4	14	35	2*
5	102	15	-11
6	60	31	-7
7	180	9	-4
H,K= 10, 5			
-2	24	52	-9*
-1	132	11	5
0	142	6	-4
1	35	52	-18*
2	302	7	-0
3	309	12	1
4	162	5	12
5	0	35	-16*
H,K= 11,-12			
-6	165	6	-3
-5	0	53	-19*
-4	134	7	1
-3	54	45	22
-2	9	53	-46*
-1	200	9	16
H,K= 11,-11			
-8	43	15	36
-7	152	6	-7
-6	93	8	-0
-5	74	10	17
-4	132	7	-17
-3	124	7	-1
-2	57	13	20
-1	93	17	-18
0	0	52	-31*
1	214	7	-5
2	0	35	-8*
H,K= 11,-10			
-9	29	52	-19*
-8	111	7	1
-7	274	9	-15
-6	54	13	12
-5	148	11	-0
-4	155	6	6
-3	187	9	11
-2	124	6	17
-1	55	33	-6
0	166	6	-2
1	257	13	-1
2	248	8	-19
3	119	6	-4
H,K= 11, -9			
-9	121	13	-11
-8	32	53	29*
-7	89	17	-3
-6	54	27	27
-5	101	30	-14
-4	121	12	-3
-3	374	10	-3
-2	94	15	-21
-1	30	51	3*
0	125	6	16
1	146	7	-9
2	173	7	10
3	74	10	19
4	90	8	6
H,K= 11, -8			
-10	50	52	-2*
-9	32	74	-19*
-8	147	7	-5
-7	300	9	-11
-6	50	18	22
-5	257	8	-14
-4	163	6	4

L	FOB	SG	DEL
-3	259	9	5
-2	96	7	6
-1	272	9	4
0	181	6	3
1	165	9	-6
2	311	9	9
3	185	9	-1
4	143	6	5
5	98	9	0
H,K= 11, -7			
-10	91	10	-1
-9	231	7	-6
-8	61	30	-21
-7	154	10	-6
-6	218	13	-14
-5	235	8	2
-4	78	11	-6
-3	158	9	1
-2	35	36	4*
-1	106	8	-11
0	129	7	2
1	43	49	17*
2	161	9	7
3	258	9	5
4	104	9	-6
5	108	14	-4
6	189	9	-15
H,K= 11, -6			
-10	65	24	-1
-9	89	17	15
-8	448	12	-14
-7	36	51	8*
-6	51	41	29
-5	121	11	-0
-4	281	8	6
-3	196	13	6
-2	0	46	-18*
-1	19	47	-21*
0	261	8	14
1	93	8	8
2	0	36	-32*
3	66	10	7
4	0	51	-9*
5	244	7	1
6	52	14	5
7	246	8	-6
H,K= 11, -5			
-10	0	52	-3*
-9	40	32	-3
-8	382	11	2
-7	314	9	-0
-6	91	15	-3
-5	60	13	37
-4	149	16	-9
-3	0	46	-30*
-2	181	8	5
-1	27	37	-1*
0	316	9	12
1	271	8	9
2	41	67	-19*
3	86	16	-2
4	0	50	-13*
5	182	7	-7
6	257	8	-1
7	354	10	-9
H,K= 11, -4			
-10	55	37	23
-9	81	11	-10
-8	99	15	-21
-7	502	14	-9
-6	151	10	17
-5	0	43	-15*
-4	284	11	-11
-3	360	10	11
-2	347	10	15
-1	70	17	8
0	351	9	13
1	243	8	10
2	165	9	-0
3	136	6	7
4	109	13	6
5	194	9	6
6	129	9	6
7	196	8	-8
8	175	6	2
H,K= 11, -3			
-10	115	13	25
-9	54	15	5
-8	93	11	-15
-7	110	11	2
-6	280	8	1
-5	126	9	2
-4	248	7	2
-3	279	11	-6
-2	285	9	1
-1	359	10	5
0	152	6	7
1	52	29	-3
2	136	5	-2
3	155	9	-8
4	159	9	-7
5	151	10	14
6	209	9	11
7	226	7	-7
8	0	35	-2*
H,K= 11, -2			
-9	251	9	-5
-8	0	51	-26*
-7	7[illegible]	11	-0
-6	5[illegible]	13	3
-5	34	53	-22*
-4	77	10	7

L	FOB	SG	DEL
-3	392	10	-5
-2	439	12	-11
-1	236	8	3
0	156	9	-3
1	175	6	5
2	176	6	6
3	290	9	10
4	121	7	-8
5	529	15	-15
6	210	9	9
7	68	10	12
8	34	52	19*
H,K= 11, -1			
-8	57	35	5
-7	0	52	-22*
-6	121	12	-16
-5	240	8	1
-4	354	10	-5
-3	287	9	1
-2	250	8	-7
-1	244	8	-5
0	128	6	-2
1	31	36	5*
2	165	9	9
3	177	9	9
4	209	7	3
5	10	38	-34*
6	124	7	4
7	12	35	-14*
8	44	18	-13
H,K= 11, 0			
-8	97	15	7
-7	32	19	4
-6	292	8	1
-5	138	8	-1
-4	63	71	15*
-3	150	7	-2
-2	478	13	-13
-1	89	15	-13
0	0	49	-16*
1	38	24	4
2	0	49	-24*
3	62	13	-14
4	164	17	-14
5	368	10	6
6	143	11	-3
7	79	9	-2
8	134	6	-0
H,K= 11, 1			
-7	0	39	-18*
-6	86	18	-10
-5	335	9	-8
-4	463	12	-5
-3	0	52	-28*
-2	150	10	-1
-1	41	25	20
0	170	9	-1
1	103	14	5
2	62	26	9
3	143	6	7
4	295	8	-4
5	319	9	-3
6	27	52	-24*
7	0	52	-41*
H,K= 11, 2			
-6	128	7	-13
-5	247	13	6
-4	32	52	16*
-3	252	13	-8
-2	425	12	1
-1	174	9	1
0	312	12	1
1	96	15	31
2	61	11	25
3	105	9	-4
4	119	7	1
5	12	35	-3*
6	79	20	8
7	82	44	-5
H,K= 11, 3			
-4	157	6	1
-3	96	16	22
-2	76	22	-10
-1	242	9	-4
0	59	12	6
1	245	9	-4
2	68	76	14*
3	39	22	17
4	161	6	2
5	42	52	14*
6	181	6	-7
H,K= 11, 4			
-2	97	16	-9
-1	119	13	-3
0	122	8	-17
1	120	13	-2
2	464	12	-6
3	271	9	-8
4	64	11	16
H,K= 12,-11			
-6	308	9	-1
-5	103	8	-1
-4	35	36	9*
-3	26	36	10*
-2	101	8	1
-1	0	53	-3*
0	109	7	10
H,K= 12,-10			
-8	0	53	-2*
-7	273	8	-5
-6	327	10	-8
-5	135	8	-7

L	FOB	SG	DEL
-4	0	53	-20*
-3	32	36	4*
-2	142	11	1
-1	121	7	4
0	207	7	11
1	164	10	2
2	226	7	-6
H,K= 12, -9			
-8	90	18	3
-7	250	8	-3
-6	113	8	2
-5	42	53	5*
-4	5	53	3*
-3	9	40	-10*
-2	188	6	8
-1	207	9	-9
0	47	17	14
1	118	8	-4
2	111	7	15
3	61	8	44
H,K= 12, -8			
-9	0	53	-51*
-8	115	14	-21
-7	0	53	-20*
-6	200	9	-8
-5	182	7	-7
-4	290	9	20
-3	161	7	7
-2	222	9	6
-1	309	9	10
0	200	9	11
1	54	44	1
2	56	13	12
3	47	17	-8
4	70	10	-12
H,K= 12, -7			
-9	301	9	-19
-8	281	8	-3
-7	233	7	-8
-6	80	20	-6
-5	116	13	-1
-4	122	12	11
-3	35	35	27*
-2	49	51	-3*
-1	269	9	10
0	292	8	11
1	162	7	4
2	23	74	15*
3	74	10	3
4	0	53	-35*
5	70	25	-1
H,K= 12, -6			
-9	184	7	-2
-8	312	9	-8
-7	56	38	20
-6	72	23	-3
-5	124	12	-4
-4	77	20	4
-3	67	12	20
-2	60	14	15
-1	156	7	2
0	262	8	9
1	95	13	31
2	219	9	-3
3	0	53	-16*
4	308	9	9
5	146	22	11
6	25	36	-1*
H,K= 12, -5			
-9	107	15	11
-8	85	11	5
-7	79	53	-13*
-6	69	24	42
-5	93	16	28
-4	90	16	-0
-3	255	9	-7
-2	239	13	7
-1	51	40	-4
0	153	10	-4
1	0	38	-46*
2	153	10	3
3	295	9	10
4	86	8	-0
5	266	9	-5
6	60	13	-16
H,K= 12, -4			
-9	51	33	1*
-8	70	11	-10
-7	92	17	6
-6	187	16	-18
-5	135	27	-12
-4	156	10	-3
-3	267	8	-3
-2	245	9	-6
-1	191	9	-0
0	94	15	2
1	67	23	-17
2	17[illegible]	9	4
3	35	52	-4*
4	92	8	4
5	305	9	2
6	272	9	-1
7	96	8	-5
H,K= 12, -3			
-8	32	40	-24*
-7	59	37	-2
-6	76	13	-4
-5	231	7	-4
-4	306	9	-4
-3	58	15	16
-2	264	8	-4

L	FOB	SG	DEL
-1	128	22	-3
0	84	17	3
1	33	51	-26*
2	211	9	-3
3	130	12	14
4	216	7	0
5	299	13	-8
6	250	7	-2
7	48	53	1*
H,K= 12, -2			
-8	162	7	3
-7	44	27	-22
-6	147	10	2
-5	152	7	15
-4	281	9	12
-3	173	9	-5
-2	155	7	-2
-1	0	41	-12*
0	143	7	-1
1	0	41	-20*
2	188	7	-2
3	217	7	-0
4	94	17	9
5	130	12	1
6	180	6	-7
7	153	11	-2
H,K= 12, -1			
-7	0	53	-6*
-6	93	17	8
-5	319	10	16
-4	445	12	6
-3	101	9	10
-2	145	9	4
-1	18	51	-9*
0	116	13	-17
1	0	38	-16*
2	42	52	11*
3	427	13	-2
4	373	11	0
5	111	7	3
6	154	6	-9
7	0	53	-21*
H,K= 12, 0			
-6	66	11	5
-5	176	11	4
-4	394	11	8
-3	236	14	-4
-2	341	9	-2
-1	60	74	-35*
1	35	39	-13*
2	75	10	-3
3	181	6	-8
4	252	9	-1
5	73	24	13
6	54	14	-3
H,K= 12, 1			
-5	0	40	-23*
-4	36	53	11*
-3	0	75	-14*
-2	270	8	-11
-1	132	8	-16
0	179	10	-3
1	127	13	-10
2	44	19	-7
3	28	53	-14*
4	84	9	11
5	26	36	-1*
6	144	6	1
H,K= 12, 2			
-4	111	14	-12
-3	0	40	-10*
-2	39	24	15
-1	69	13	-15
0	214	15	0
1	110	15	-9
2	0	53	-28*
3	98	16	13
4	0	53	-4*
5	60	12	-3
H,K= 12, 3			
-1	174	10	2
0	68	16	24
1	231	7	1
2	4	53	-14*
3	47	53	-24*
H,K= 13,-10			
-7	232	7	6
-5	131	12	[illegible]
-4	74	23	1
-3	121	13	-2
-2	3[illegible]7	10	-5
-1	1[illegible]4	10	-15
0	96	16	5
H,K= 13, -9			
-7	87	18	1
-6	121	13	-12
-5	0	53	-15*
-4	0	40	-15*
-3	35	10	-7
-2	147	10	2
-1	400	11	-5
0	122	7	0
1	52	53	37*
2	61	12	-6
H,K= 13, -8			
-8	155	7	-6
-7	49	21	-3
-6	71	8	1*
-5	142	7	2
-4	271	7	15
-3	[illegible]	53	26*
-2	241	8	8

L	FOB	SG	DEL
-1	460	12	8
0	209	7	4
1	83	9	-9
2	139	12	-1
3	26	53	-21*
H,K= 13, -7			
-8	214	9	3
-7	0	40	-34*
-6	41	53	-1*
-5	0	40	-23*
-4	206	9	-5
-3	36	53	-30*
-2	19	40	-3*
-1	161	7	-9
0	111	8	-5
1	65	28	24
2	131	12	10
3	161	10	4
4	131	7	1
H,K= 13, -6			
-8	218	9	-6
-7	15	53	-31*
-6	55	14	3*
-5	35	40	-5*
-4	196	9	-8
-3	245	8	13
-2	134	8	4
-1	61	30	13
0	17	53	1*
1	37	29	31
2	0	53	-15*
3	0	53	-17*
4	129	12	1
5	271	9	-9
H,K= 13, -5			
-8	30	40	-20*
-7	67	14	-16
-6	24	40	-16*
-5	51	20	-0
-4	31	53	-12*
-3	238	7	0
-2	175	17	10
-1	0	39	-15*
0	98	8	10
1	68	13	2
2	183	7	-1
3	94	8	-3
4	198	9	-3
5	144	11	18
H,K= 13, -4			
-8	0	53	-16*
-7	52	53	14*
-6	47	53	23*
-5	89	14	21
-4	106	7	10
-3	90	10	-10
-2	226	8	-1
-1	32	52	-21*
0	135	12	-5
1	41	53	16*
2	288	9	2
3	285	8	-3
4	197	9	-0
5	231	9	-2
H,K= 13, -3			
-7	290	9	-12
-6	117	14	3
-5	207	7	-5
-4	54	47	14
-3	0	53	-44*
-2	140	11	-10
-1	0	42	-5*
0	122	13	-6
1	51	53	24*
2	[illegible]	14	-5
3	239	9	1
4	134	12	7
5	111	7	-9
H,K= 13, -2			
-6	363	10	2
-5	251	9	-2
-4	159	10	7
-3	57	37	-13
-2	54	18	-2
-1	35	40	-6*
0	327	10	-9
1	248	8	12
2	127	13	-7
3	240	9	-3
4	97	8	-0
5	62	12	-15
H,K= 13, -1			
-5	234	9	10
-4	174	10	-1
-3	108	9	-8
-2	47	19	-6
-1	61	30	14
0	205	7	-9
1	245	9	-2
2	41	22	37
3	56	41	-4
4	86	12	-19
5	56	38	22
H,K= 13, 0			
-4	23	31	4
-3	27	75	-21*
-2	69	13	-14
-1	171	21	-5
0	171	7	9
1	240	9	6
2	73	23	30
3	20	36	15*

L	FOB	SG	DEL
4	91	8	-2
H,K= 13, 1			
-2	193	6	-5
-1	114	8	-4
0	428	12	2
1	216	9	1
2	148	10	-4
3	82	20	-5
H,K= 14, -9			
-5	265	8	-6
-4	160	10	12
-3	72	75	-5*
-2	420	11	-21
-1	194	6	-7
0	18	35	6*
H,K= 14, -8			
-6	24	74	-21*
-5	137	7	-8
-4	250	8	2
-3	104	9	-5
-2	216	9	-3
-1	296	9	3
0	39	24	7
1	0	53	-28*
H,K= 14, -7			
-6	77	12	-21
-5	106	15	10
-4	152	7	-4
-3	86	19	8
-2	252	8	3
-1	37	51	6*
0	41	71	15*
1	0	53	-17*
2	160	4	9
H,K= 14, -6			
-6	64	22	7
-5	27	40	-19*
-4	148	11	2
-3	192	6	24
-2	100	12	12
-1	115	14	-5
0	40	29	29
1	210	7	-9
2	35	36	20*
3	182	6	-8
H,K= 14, -5			
-6	181	10	-4
-5	0	40	-24*
-4	28	40	4*
-3	168	7	-3
-2	0	53	-49*
-1	107	9	-4
0	53	15	26
1	155	11	4
2	308	9	14
3	150	8	1
H,K= 14, -4			
-6	160	7	1
-5	219	8	15
-4	50	21	1
-3	85	13	-13
-2	63	12	29
-1	143	11	-2
0	50	53	27*
1	91	19	4
2	459	14	11
3	234	7	7
4	128	12	-16
H,K= 14, -3			
-5	218	9	-7
-4	0	75	-11*
-3	69	26	5
-2	44	10	-13
-1	126	13	-10
0	106	9	-3
1	73	10	5
2	180	5	4
3	104	7	-6
H,K= 14, -2			
-4	187	9	-5
-3	0	52	-19*
-2	21	40	-39*
-1	145	7	-13
0	89	16	11
1	102	5	1
2	54	44	-11
3	58	13	8
H,K= 14, -1			
-1	13	53	15*
-2	0	39	-6*
-1	[illegible]	55	-10*
0	[illegible]	12	[illegible]
1	301	9	7
2	182	11	-19
H,K= 15, -7			
-2	[illegible]	53	5*
H,K= 15, -6			
-3	56	36	-15
-2	[illegible]	53	4
-1	[illegible]	17	7
0	127	12	13
H,K= 15, -5			
-3	50	[illegible]	-4*
-2	147	7	-6
-1	1[illegible]	7	-0
0	[illegible]	16	21
H,K= 15, -4			
-2	[illegible]	[illegible]	-7
-1	12[illegible]	12	6
0	0	[illegible]	-24*

Table 2. Final parameters for non-hydrogen atoms. In this and the following tables the standard deviation of the least significant digit(s) is given in parentheses. The form of the temperature factor (B in units of $\mathring{A}^2$) is $T = \exp(-0.25(B_{11}h^2a^{*2} + 2B_{12}hka^*b^* + \ldots))$.

(To be reproduced photographically.)

ATOM	X	Y	Z	B11	B22	B33	B12	B13	B23
CA(1)	.1558(4)	.6274(2)(4)	.4448(4)	2.25(3)	2.55(3)	2.90(3)	.78(2)	.17(2)	-.48(2)
CA(2)	-.0689(4)	.5433(5)	.3024(4)	2.62(3)	2.80(3)	3.09(3)	1.23(2)	-.08(2)	-.64(2)
O(1)	-.0458(1)	.2998(1)	.6414(1)	3.1(1)	2.49(9)	3.8(1)	1.22(8)	.08(8)	-.95(7)
O(2)	.1235(1)	.4498(1)	.5458(1)	3.0(1)	2.57(9)	3.30(9)	1.30(8)	.29(7)	-.44(7)
C(1)	-.1316(2)	.0897(3)	.7620(3)	3.9(2)	2.9(2)	4.6(2)	.9(1)	.6(1)	-1.0(1)
C(2)	-.1835(3)	-.0168(3)	.8148(3)	4.2(2)	4.0(2)	5.1(2)	.9(2)	1.6(2)	-.4(2)
C(3)	-.1749(3)	-.1058(3)	.7934(3)	4.2(2)	3.1(2)	6.0(2)	.4(2)	.6(2)	-.0(2)
C(4)	-.1157(3)	-.0878(3)	.7186(3)	5.3(2)	2.7(2)	7.1(2)	1.4(2)	.5(2)	-1.7(2)
C(5)	-.0620(3)	.0195(3)	.6661(3)	4.7(2)	3.3(2)	4.9(2)	1.1(2)	.8(2)	-1.1(2)
C(6)	-.0703(2)	.1097(2)	.6873(2)	2.4(1)	2.8(2)	3.6(2)	.9(1)	-.1(1)	-1.1(1)
C(7)	-.0137(2)	.2274(2)	.6352(2)	3.0(2)	2.9(2)	3.1(1)	1.1(1)	-.2(1)	-.8(1)
C(8)	.0704(2)	.2544(3)	.5863(2)	3.4(2)	2.5(2)	4.0(2)	1.5(1)	.5(1)	-.7(1)
C(9)	.1328(2)	.3614(3)	.5407(2)	3.0(2)	3.3(2)	2.9(1)	1.4(1)	-.2(1)	-.7(1)
C(10)	.2177(2)	.3757(2)	.4859(2)	3.3(2)	3.1(1)	3.0(1)	1.8(1)	-.1(1)	-.3(1)
C(11)	.2181(3)	.2987(3)	.4391(2)	4.2(2)	3.7(2)	4.5(2)	1.5(2)	.7(2)	-1.0(1)
C(12)	.2966(3)	.3152(4)	.3885(3)	5.5(2)	5.7(2)	5.8(2)	2.9(2)	1.7(2)	-1.4(2)
C(13)	.3778(3)	.4085(4)	.3857(3)	4.7(3)	7.7(3)	6.8(3)	3.0(2)	2.0(2)	-1.3(2)
C(14)	.3801(3)	.4858(4)	.4325(3)	3.1(2)	6.5(3)	9.1(3)	.9(2)	1.2(2)	-2.5(2)
C(15)	.3000(3)	.4707(3)	.4822(3)	3.3(2)	4.7(2)	5.7(2)	1.4(2)	.3(2)	-1.8(2)
O(3)	.0862(1)	.5283(2)	.3235(1)	2.7(1)	3.03(9)	3.43(9)	1.20(8)	.02(7)	-1.04(8)
O(4)	-.0801(1)	.3648(1)	.4218(1)	3.1(1)	2.76(9)	3.18(9)	1.26(8)	.12(8)	-.91(8)
C(16)	.2412(3)	.5730(4)	.2109(3)	3.8(2)	8.3(3)	6.1(2)	-.2(2)	1.8(2)	-3.9(2)
C(17)	.3168(3)	.5991(6)	.1498(4)	4.6(3)	12.4(4)	9.2(3)	-1.0(3)	2.4(2)	-6.2(3)
C(18)	.3145(4)	.5289(6)	.0963(4)	5.1(3)	11.4(4)	6.7(3)	3.2(3)	2.5(2)	-1.8(3)
C(19)	.2357(5)	.4327(5)	.1006(3)	10.1(4)	6.8(3)	5.8(3)	4.8(3)	3.3(3)	-.5(2)
C(20)	.1605(3)	.4064(3)	.1618(3)	7.3(3)	4.3(2)	4.8(2)	2.3(2)	2.8(2)	-.1(2)
C(21)	.1632(2)	.4757(3)	.2191(2)	3.4(2)	3.9(2)	3.1(2)	1.8(1)	.1(1)	-.8(1)
C(22)	.0841(2)	.4495(2)	.2882(2)	3.2(2)	3.2(2)	2.8(1)	1.7(1)	-.2(1)	-1.0(1)
C(23)	.0136(2)	.3427(3)	.3121(2)	3.4(2)	2.8(2)	4.4(2)	1.1(1)	.7(1)	-1.3(1)
C(24)	-.0623(2)	.3029(2)	.3788(2)	3.1(2)	2.7(1)	2.9(1)	1.4(1)	-.5(1)	-.7(1)
C(25)	-.1268(2)	.1819(2)	.4006(2)	3.0(2)	3.0(1)	3.4(1)	1.4(1)	-.3(1)	-.9(1)
C(26)	-.1228(3)	.1104(3)	.3492(4)	7.1(3)	3.9(2)	9.5(3)	.5(2)	4.0(2)	-2.3(2)
C(27)	-.1845(4)	-.0002(3)	.3707(4)	8.4(3)	3.5(2)	10.7(4)	.4(2)	3.3(3)	-3.0(2)
C(28)	-.2510(3)	-.0412(3)	.4423(3)	5.1(2)	2.8(2)	6.4(2)	.7(2)	-.3(2)	-.4(2)
C(29)	-.2572(3)	.0272(3)	.4929(3)	5.6(2)	4.0(2)	6.1(2)	-.2(2)	1.7(2)	-1.5(2)
C(30)	-.1962(3)	.1370(3)	.4715(3)	5.3(2)	3.4(2)	6.1(2)	-.0(2)	2.0(2)	-1.8(2)
O(5)	.7142(2)	.2380(2)	.6385(1)	2.7(1)	4.4(1)	3.9(1)	.49(9)	.24(9)	-.63(9)
O(6)	.7190(1)	.3340(2)	.4360(1)	2.1(1)	3.9(1)	3.9(1)	.49(9)	.12(8)	-.80(8)
C(31)	.6125(3)	.0883(3)	.7993(3)	3.9(2)	4.5(2)	4.2(2)	1.5(2)	.8(2)	-.0(2)
C(32)	.5619(3)	.0089(3)	.8805(3)	5.5(3)	5.6(2)	4.4(2)	2.3(2)	1.1(2)	.6(2)
C(33)	.4679(3)	-.0577(3)	.8720(4)	5.0(3)	4.9(2)	6.8(3)	1.7(2)	2.9(2)	1.0(2)
C(34)	.4230(3)	-.0444(3)	.7835(4)	3.8(2)	4.7(2)	7.5(3)	.5(2)	1.5(2)	.1(2)
C(35)	.4720(3)	.0340(3)	.7022(3)	3.3(2)	4.5(2)	5.6(2)	.8(2)	.8(2)	-.0(2)
C(36)	.5683(2)	.1013(2)	.7089(2)	3.0(2)	3.0(1)	4.6(2)	.9(1)	1.2(1)	-.4(1)
C(37)	.6261(2)	.1862(2)	.6220(2)	2.7(2)	3.2(1)	4.3(2)	.8(1)	.7(1)	-1.0(1)
C(38)	.5833(3)	.2040(3)	.5298(3)	2.1(2)	4.4(2)	4.4(2)	.1(1)	-.1(2)	-.9(1)
C(39)	.6303(2)	.2758(3)	.4428(2)	2.9(2)	3.5(2)	3.8(2)	1.2(1)	-.3(1)	-1.3(1)
C(40)	.5716(2)	.2897(3)	.3516(2)	2.4(2)	4.1(2)	4.1(2)	1.0(1)	-.1(1)	-1.6(1)
C(41)	.4926(3)	.2027(4)	.3303(3)	3.6(2)	4.9(2)	5.9(2)	1.1(2)	.5(2)	-2.2(2)
C(42)	.4417(3)	.2371(5)	.2444(4)	3.8(2)	8.0(3)	6.9(3)	1.5(2)	-1.6(2)	-4.1(3)
C(43)	.4688(4)	.3206(5)	.1810(4)	5.9(3)	10.3(4)	5.0(3)	4.6(3)	-2.4(2)	-3.1(3)
C(44)	.5468(3)	.4089(5)	.2005(3)	5.7(3)	7.8(3)	4.5(2)	3.3(2)	-1.0(2)	-1.4(2)
C(45)	.5994(3)	.3923(3)	.2853(3)	3.4(2)	5.6(2)	4.2(2)	1.5(2)	-.8(2)	-1.7(2)
O(7)	-.1821(1)	.4180(2)	.2453(1)	3.4(1)	4.0(1)	3.6(1)	1.84(9)	.24(8)	-.08(9)
O(8)	-.0146(1)	.6543(2)	.1518(2)	4.0(1)	4.7(1)	3.8(1)	2.6(1)	.59(9)	.09(9)
C(46)	-.3111(3)	.7178(3)	.2481(3)	4.0(2)	5.3(2)	4.9(2)	2.4(2)	-.3(2)	-.8(2)
C(47)	-.3883(3)	.7475(3)	.2466(4)	4.4(2)	6.0(2)	7.6(3)	2.6(2)	1.0(2)	-1.2(2)
C(48)	-.4278(3)	.7786(3)	.1578(5)	3.2(2)	5.2(2)	11.3(4)	2.6(2)	-.6(3)	-2.1(2)
C(49)	-.3934(3)	.7776(3)	.0726(4)	4.9(2)	6.0(2)	7.6(3)	3.3(2)	-1.8(2)	-1.3(2)
C(50)	-.3151(3)	.7510(3)	.0755(3)	4.4(2)	5.2(2)	4.8(2)	2.7(2)	-.8(2)	-.9(2)
C(51)	-.2726(2)	.7209(2)	.1630(2)	2.9(2)	3.1(1)	4.0(2)	1.2(1)	.2(1)	-.4(1)
C(52)	-.1965(2)	.6913(3)	.1701(2)	2.8(2)	3.4(2)	3.6(2)	1.2(1)	-.4(1)	-.9(1)
C(53)	-.1169(2)	.7487(3)	.0968(3)	3.4(2)	4.0(2)	3.8(2)	1.9(2)	.4(2)	.5(1)
C(54)	-.0325(2)	.7324(3)	.0939(2)	3.1(2)	3.7(2)	3.2(2)	1.2(1)	.4(1)	-.9(1)
C(55)	.0445(2)	.8122(3)	.0703(2)	3.4(2)	3.9(2)	3.5(2)	1.2(1)	.2(1)	-1.3(1)
C(56)	.0375(3)	.9011(3)	-.0494(3)	4.3(2)	4.7(2)	4.7(2)	1.4(2)	1.0(2)	-.1(2)
C(57)	.1131(4)	.9747(4)	-.1114(3)	6.1(3)	5.0(2)	5.9(2)	1.1(2)	1.7(2)	.1(2)
C(58)	.1963(4)	.9602(4)	-.1032(4)	5.4(3)	5.8(3)	7.1(3)	-.1(2)	3.0(2)	-1.1(2)
C(59)	.2065(3)	.8728(4)	-.0348(4)	4.1(2)	6.9(3)	7.1(3)	1.3(2)	1.9(2)	-1.2(2)
C(60)	.1305(3)	.7990(4)	.0261(3)	5.2(3)	5.4(2)	5.3(2)	2.5(2)	1.1(2)	-.4(2)
O(9)	.8123(2)	.3902(2)	.2456(2)	4.5(1)	4.7(1)	4.6(1)	1.4(1)	-1.0(1)	-1.7(1)
C(61)	.7826(4)	.3822(5)	.1504(4)	8.2(3)	12.2(4)	5.5(3)	2.2(3)	-1.7(2)	-3.6(3)
C(62)	.8634(5)	.4059(5)	.0893(4)	13.7(5)	12.3(4)	5.9(3)	7.3(4)	-1.4(3)	-4.0(3)

Table 3. Final parameters for hydrogen atoms. Parameters without standard deviations were not refined. The form of the temperature factor is $T = \exp(-B \sin^2\theta/\lambda^2)$.

(To be reproduced photographically.)

Table 3

H(01)	-.134(2)	.150(3)	.777(2)	3.8(7)
H(02)	-.222(2)	-.028(2)	.868(2)	4.4(8)
H(03)	-.211(2)	-.182(3)	.827(2)	5.8(9)
H(04)	-.108(2)	-.145(3)	.702(2)	5.4(9)
H(05)	-.028(2)	.024(3)	.611(2)	5.3(9)
H(6)	.093(2)	.199(2)	.589(2)	3.1(7)
H(07)	.159(2)	.231(3)	.439(2)	3.9(7)
H(08)	.293(2)	.255(3)	.356(2)	5.9(9)
H(09)	.428(3)	.425(3)	.347(3)	7.4(11)
H(10)	.432(3)	.546(3)	.434(2)	5.5(10)
H(11)	.303(2)	.524(3)	.516(2)	4.9(8)
H(12)	.242(3)	.620(3)	.253(3)	7.0(10)
H(13)	.365(4)	.667(5)	.150(4)	12.3(18)
H(14)	.362(3)	.540(4)	.056(3)	9.0(13)
H(15)	.226(3)	.388(3)	.057(3)	6.5(11)
H(16)	.104(3)	.336(4)	.162(3)	9.2(13)
H(17)	.020(2)	.290(3)	.283(2)	4.4(8)
H(18)	-.078(3)	.135(4)	.297(3)	9.4(14)
H(19)	-.185(3)	-.043(4)	.335(3)	9.3(14)
H(20)	-.292(2)	-.116(3)	.455(2)	4.0(7)
H(21)	-.296(3)	.003(3)	.554(3)	7.6(11)
H(22)	-.206(3)	.179(4)	.506(3)	8.8(13)
H(23)	.679(3)	.137(3)	.809(2)	5.1(9)
H(24)	.596(2)	.000(3)	.939(3)	5.0(9)
H(25)	.429(3)	-.116(3)	.925(3)	5.6(9)
H(26)	.363(3)	-.094(3)	.774(3)	6.4(11)
H(27)	.440(2)	.037(3)	.643(3)	5.3(10)
H(28)	.518(2)	.167(2)	.520(2)	3.1(7)
H(29)	.473(2)	.130(3)	.371(2)	3.9(8)
H(30)	.386(3)	.151(3)	.238(2)	6.1(9)
H(31)	.440(3)	.339(3)	.126(3)	5.9(10)
H(32)	.569(3)	.477(3)	.156(3)	7.8(13)
H(33)	.653(2)	.454(2)	.301(2)	3.2(7)
H(34)	-.288(2)	.703(2)	.299(2)	1.9(7)
H(35)	-.416(3)	.737(3)	.307(3)	7.5(12)
H(36)	-.475(3)	.796(3)	.157(3)	6.3(11)
H(37)	-.419(3)	.796(3)	.007(3)	7.4(11)
H(38)	-.291(2)	.751(2)	.016(2)	3.8(8)
H(39)	-.127(2)	.798(2)	.050(2)	2.6(7)
H(40)	-.023(2)	.915(3)	-.058(2)	4.9(8)
H(41)	.102(3)	1.039(3)	-.159(3)	7.4(11)
H(42)	.249(3)	1.004(3)	-.142(3)	7.5(12)
H(43)	.262(3)	.862(3)	-.023(3)	7.6(13)
H(44)	.136(2)	.743(2)	.070(2)	3.1(8)
H(45)	.779(5)	.375(6)	.281(5)	15.6(28)
H(46)	.717(3)	.312(4)	.136(3)	10.6(14)
H(47)	.7497	.4344	.1232	12.000
H(48)	.8860	.3438	.113	14.000
H(49)	.930(4)	.469(5)	.089(5)	15.000
H(50)	.941(4)	.394(4)	.015(4)	12.8(16)

Table 4. Distances in the coordination cluster (Å). (See Fig. 1.)

Atoms		Distance	Atoms		Distance
Ca(1)	O(1)	2.353(2)	O(2)	O(4)	2.995(3)[b]
Ca(1)	O(2)	2.333(2)	O(3)	O(4)	2.804(3)[a,b]
Ca(1)	O(3)	2.358(2)	O(1)	O(5)	3.410[c]
Ca(1)	O(4)	2.353(2)	O(1)	O(8)	3.111
Ca(1)	O(5)	2.256(2)	O(2)	O(4)	3.333(3)[d]
Ca(1)	O(6)	2.395(2)	O(2)	O(6)	3.010
Ca(2)	O(1)	2.416(2)	O(2)	O(7)	2.999
Ca(2)	O(2)	2.411(2)	O(3)	O(5)	3.526
Ca(2)	O(3)	2.436(2)	O(3)	O(8)	3.205
Ca(2)	O(4)	2.508(2)	O(4)	O(6)	2.943
Ca(2)	O(7)	2.308(2)	O(4)	O(9)	2.851
Ca(2)	O(8)	2.266(2)	O(5)	O(6)	2.801(3)[a]
Ca(2)	O(9)	2.408(2)	O(6)	O(9)	2.941(4)
O(1)	O(2)	2.772(3)[a,b]	O(7)	O(8)	2.803(3)[a]
O(1)	O(3)	2.807(3)[b]	O(7)	O(9)	3.054(3)
O(1)	O(4)	3.037(3)[b]	O(8)	O(9)	3.491(4)
O(2)	O(3)	3.102(3)[b]			

(a) "Bite" of the ligand.

(b) Edges of central parallelopiped.

(c) Absence of a standard deviation indicates that atoms were refined in two different blocks of least-squares. Standard deviations are probably the same as others, ± .003 Å.

(d) Diagonal across the face of core plane 1.

Table 5. Rotation of phenyl ring planes with respect to diketone planes and each other (deg)

DPP	Phenyl 1 to diketone.	Phenyl 2 to diketone.	Phenyl 1 to Phenyl 2.
1	21.1	31.0	33.8
2	13.4	7.8	16.1
3	5.6	34.0	28.6
4	36.6	12.9	47.6

Table 6. Intra-molecular distances (Å)

DPP(1)			DPP(2)		
Atoms		Distance	Atoms		Distance
C(1)	C(2)	1.379(5)	C(16)	C(17)	1.385(6)
C(2)	C(3)	1.376(5)	C(17)	C(18)	1.350(7)
C(3)	C(4)	1.367(5)	C(18)	C(19)	1.367(7)
C(4)	C(5)	1.391(5)	C(19)	C(20)	1.378(6)
C(5)	C(6)	1.388(4)	C(20)	C(21)	1.379(5)
C(6)	C(1)	1.381(4)	C(21)	C(16)	1.364(5)
C(6)	C(7)	1.496(4)	C(21)	C(22)	1.500(4)
C(7)	O(1)	1.285(3)	C(22)	O(3)	1.280(3)
C(7)	C(8)	1.387(4)	C(22)	C(23)	1.391(4)
C(8)	C(9)	1.397(4)	C(23)	C(24)	1.403(4)
C(9)	O(2)	1.283(3)	C(24)	O(4)	1.277(3)
C(9)	C(10)	1.496(4)	C(24)	C(25)	1.502(4)
C(10)	C(11)	1.377(4)	C(25)	C(26)	1.379(5)
C(11)	C(12)	1.368(5)	C(26)	C(27)	1.384(6)
C(12)	C(13)	1.365(6)	C(27)	C(28)	1.349(6)
C(13)	C(14)	1.368(6)	C(28)	C(29)	1.348(5)
C(14)	C(15)	1.387(5)	C(29)	C(30)	1.373(5)
C(15)	C(10)	1.386(4)	C(30)	C(25)	1.368(5)
C(1)	H(1)	0.92(3)	C(16)	H(12)	0.97(4)
C(2)	H(2)	0.95(3)	C(17)	H(13)	0.92(5)
C(3)	H(3)	0.97(3)	C(18)	H(14)	0.90(5)
C(4)	H(4)	0.92(3)	C(19)	H(15)	0.93(4)
C(5)	H(5)	0.95(3)	C(20)	H(16)	1.00(4)
C(8)	H(6)	0.93(3)	C(23)	H(17)	0.95(3)
C(11)	H(7)	1.00(3)	C(26)	H(18)	0.95(4)
C(12)	H(8)	1.01(4)	C(27)	H(19)	0.86(4)
C(13)	H(9)	0.91(4)	C(28)	H(20)	0.94(3)
C(14)	H(10)	0.88(3)	C(29)	H(21)	1.00(4)
C(15)	H(11)	0.94(3)	C(30)	H(22)	0.90(4)

Table 6. (continued)

DPP(3)			DPP(4)		
Atoms		Distance	Atoms		Distance
C(31)	C(32)	1.389(5)	C(46)	C(47)	1.384(5)
C(32)	C(33)	1.367(6)	C(47)	C(48)	1.373(5)
C(33)	C(34)	1.364(6)	C(48)	C(49)	1.365(6)
C(34)	C(35)	1.378(5)	C(49)	C(50)	1.373(5)
C(35)	C(36)	1.393(5)	C(50)	C(51)	1.373(4)
C(36)	C(31)	1.381(5)	C(51)	C(46)	1.373(5)
C(36)	C(37)	1.506(4)	C(51)	C(52)	1.507(4)
C(37)	O(5)	1.272(3)	C(52)	O(7)	1.263(3)
C(37)	C(38)	1.399(4)	C(52)	C(53)	1.393(4)
C(38)	C(39)	1.399(4)	C(53)	C(54)	1.395(4)
C(39)	O(6)	1.262(3)	C(54)	O(8)	1.261(3)
C(39)	C(40)	1.502(4)	C(54)	C(55)	1.506(4)
C(40)	C(41)	1.381(4)	C(55)	C(56)	1.372(5)
C(41)	C(42)	1.386(6)	C(56)	C(57)	1.383(5)
C(42)	C(43)	1.354(6)	C(57)	C(58)	1.353(6)
C(43)	C(44)	1.374(6)	C(58)	C(59)	1.365(6)
C(44)	C(45)	1.388(5)	C(59)	C(60)	1.380(5)
C(45)	C(40)	1.382(5)	C(60)	C(55)	1.383(5)
C(31)	H(23)	0.98(3)	C(46)	H(34)	0.79(3)
C(32)	H(24)	0.93(3)	C(47)	H(35)	0.94(4)
C(33)	H(25)	0.97(4)	C(48)	H(36)	0.84(4)
C(34)	H(26)	0.92(4)	C(49)	H(37)	0.98(4)
C(35)	H(27)	0.92(3)	C(50)	H(38)	0.94(3)
C(38)	H(28)	0.93(3)	C(53)	H(39)	0.85(3)
C(41)	H(29)	0.94(3)	C(56)	H(40)	1.01(3)
C(42)	H(30)	0.99(4)	C(57)	H(41)	1.01(4)
C(43)	H(31)	0.88(4)	C(58)	H(42)	0.92(4)
C(44)	H(32)	0.92(4)	C(59)	H(43)	0.92(4)
C(45)	H(33)	0.96(3)	C(60)	H(44)	0.87(3)

Table 6. (continued)

Ethanol

Atoms		Distance
O(9)	C(61)	1.390(5)
C(61)	C(62)	1.464(7)
O(9)	H(45)	0.68(6)
C(61)	H(46)	1.12(5)
C(61)	H(47)[a]	1.00
C(62)	H(48)[a]	1.00
C(62)	H(49)	1.04(6)
C(62)	H(50)	1.09(6)

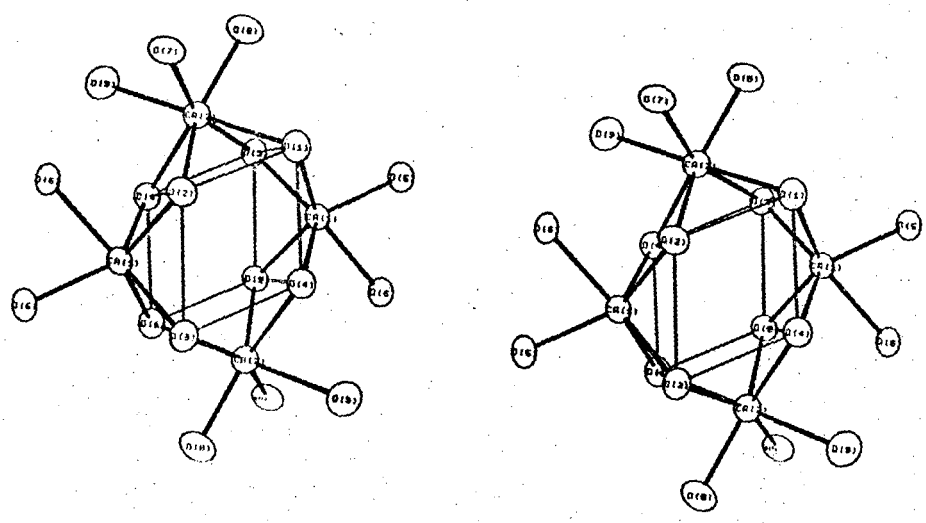
[a] Hydrogen atom restrained to a fixed position in the least-squares refinements.

Figure Captions

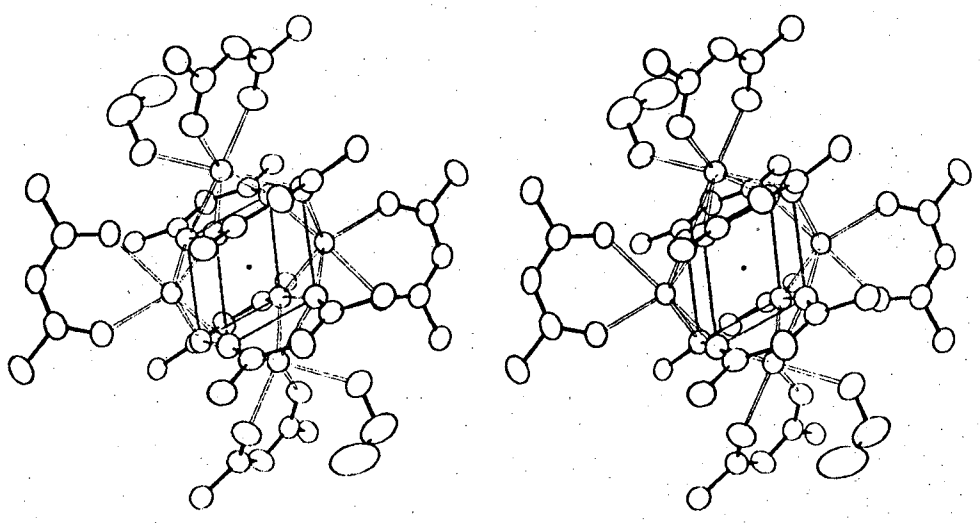
Fig. 1. Stereoscopic view of the coordination around the Ca atoms. The center of symmetry at 0,1/2,1/2 is in the center of the cluster. Thermal ellipsoids have been scaled to include 50% probability.

Fig. 2. Stereoscopic view of the complex cluster. Phenyl carbons not attached directly to the diketone and hydrogen atoms have been omitted for clarity. View direction and scaling of the thermal ellipsoids are the same as in Fig. 1.

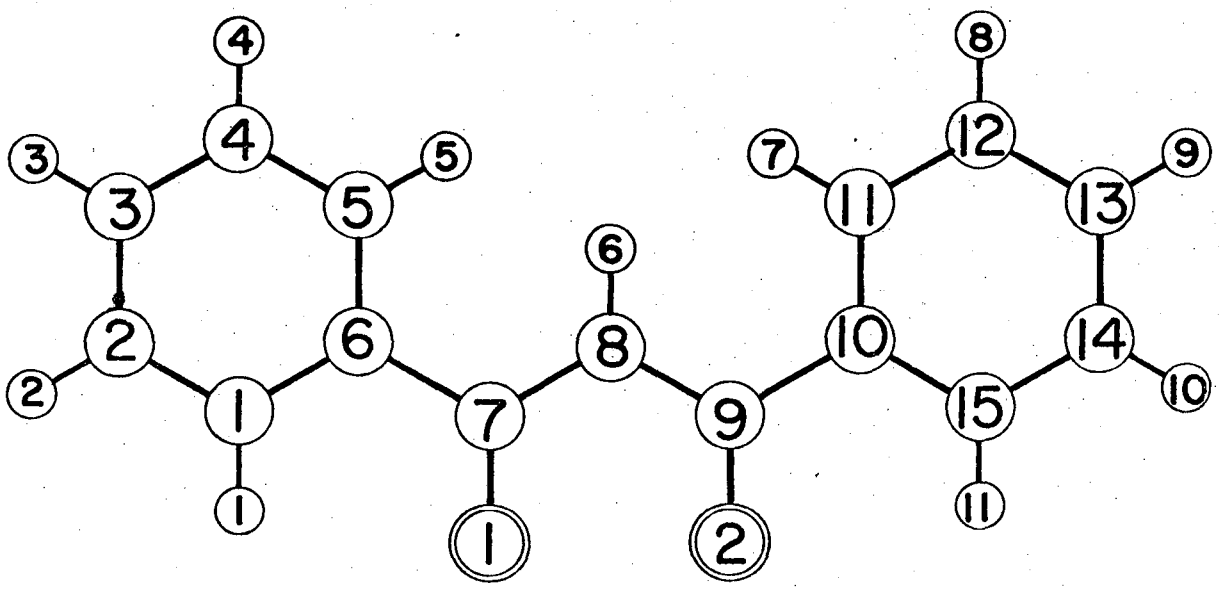
Fig. 3. Generalized numbering system for DPP ligands. Carbons are large single circles, oxygens are large double circles and hydrogens are small single circles. Numbering shown is for DPP(1), for successive DPP molecules add 2 to the oxygen numbers, 15 to the carbon numbers and 11 to the hydrogen numbers.



XBL 723-543



XBL 724-663


4
4
3
3
5
5
2
2
6
6
1
1
7
1
8
8
9
2
10
10
11
7
12
8
13
9
14
15
11

LEGAL NOTICE

This report was prepared as an account of work sponsored by the United States Government. Neither the United States nor the United States Atomic Energy Commission, nor any of their employees, nor any of their contractors, subcontractors, or their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness or usefulness of any information, apparatus, product or process disclosed, or represents that its use would not infringe privately owned rights.

TECHNICAL INFORMATION DIVISION
LAWRENCE BERKELEY LABORATORY
UNIVERSITY OF CALIFORNIA
BERKELEY, CALIFORNIA 94720