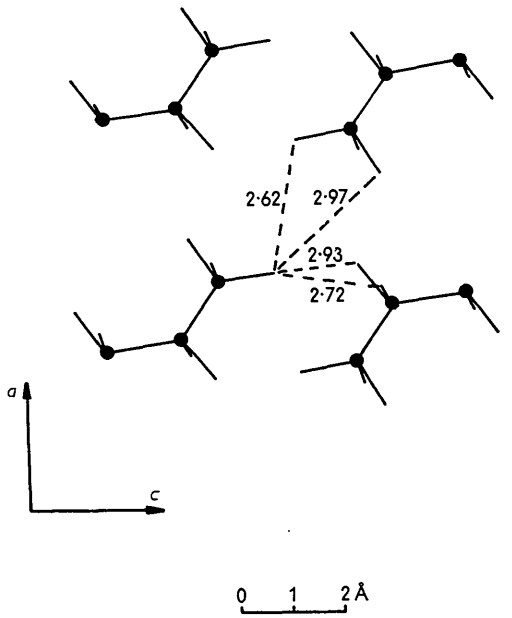

Fig. 5. Projection along the *b*-axis showing the methyl contact region. Non-equivalent hydrogen–hydrogen distances (in Å) less than 3·0 Å over the methyl gap are given.

The hydrogen–hydrogen contact distances over the methyl gap (Fig. 5) give a more informative picture of the packing of the methyl-group planes than the carbon–carbon distances discussed. The shortest distance is 2·62 Å, somewhat longer than the shortest lateral hydrogen–hydrogen contact between the chains, which is about 2·40 Å.

I wish to thank Prof. E. Stenhagen and Dr S. Abrahamsson for their interest in this work. I am also indebted to Dr C. Lagercrantz for providing laboratory space. Thanks are due to Mr L. Ahlquist for preparation of the sample and Mrs M. Innes for technical assistance. Grants from the National Institute of Arthritis and Metabolic Diseases, U.S. Public Health Service (A-4006) and from The Swedish Natural Science Research Council, The Swedish Medical Research Council and The Swedish Technical Research Council are gratefully acknowledged.

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The Crystal Structure of Monoaquobisacetylacetonatozinc

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(*Received* 18 *May* 1962)

The crystal structure of monoaquobisacetylacetonatozinc [$Zn(C_5H_7O_2)_2H_2O$] has been determined by three-dimensional methods. The cell dimensions are $a = 10{\cdot}48$, $b = 5{\cdot}37$, $c = 10{\cdot}94$ Å, $\beta = 93°48'$, space group $P2_1$, with $Z = 2$. The structure is made up of discrete molecules, containing 5-coordinate zinc with a coordination configuration intermediate between tetragonal pyramidal and trigonal bipyramidal.

Introduction

Lippert & Truter (1960) have recently published a determination of the crystal structure of monoaquobisacetylacetonatozinc, $Zn(C_5H_7O_2)_2.H_2O$. Since we had an independent structure determination in the final refinement stages at the time we learned of their work, and since there appeared to be some significant differences in the results, we have completed our determination.

Experimental

Monoaquobisacetylacetonatozinc crystallized from benzene, ethanol or benzene–ethanol mixtures in needles. The unit cell dimensions were determined from rotation and zero level Weissenberg photographs about the needle (b) axis using Cu $K\alpha$ radiation with superimposed reflections from a sodium chloride crystal ($a_0=5{\cdot}6387$ Å) for calibration. In addition, zero, first and second level Weissenberg exposures and zero level precession photographs (Mo $K\alpha$, $\lambda=0{\cdot}7107$ Å) were taken to establish the space group. The results were:

$$a_0=10{\cdot}48_0\pm0{\cdot}01,\ b_0=5{\cdot}37_0\pm0{\cdot}005,$$
$$c_0=10{\cdot}93_5\pm0{\cdot}01\text{ Å},\ \beta=93°\,48'\pm9'.$$

The space group was $P2_1$ or $P2_1/m$ ($0k0$ present only for $k=2n$). Calculated density for two molecules per unit cell 1·522 g.cm^{-3}; observed density 1·51 g.cm^{-3}. A further set of calibrated rotation exposures about b, using iron radiation ($\lambda=1{\cdot}9373$ Å), were taken to confirm the value of b_0 ($b_0=5{\cdot}373\pm0{\cdot}005$ Å).

The intensities for the four levels ($h0l$ to $h3l$) were recorded on Kodak medical X-ray film, using multiple films, in a Nonius integrating Weissenberg camera. Considerable difficulty was found in obtaining a crystal that would last through the series of exposures. The intensities were measured photometrically and corrected for Lorentz and polarization factors but not for absorption.

Determination and refinement of the structure

To carry out a structure determination, a choice had to be made between the two space groups $P2_1$ and $P2_1/m$. After experiments with models, the space group $P2_1$ was selected, since it proved very difficult to fit any reasonable model in the cell in a $P2_1/m$ arrangement. This choice was confirmed by the successful determination of the structure. Calculations were carried out for the $h0l$ projection first, using the heavy atom technique. Because of the short b axis all atoms were clearly resolved and accurate x and z coordinates were obtained. With the aid of these coordinates, the zinc and oxygen y parameters were derived from a three-dimensional Patterson function The first triple Fourier summation clearly showed all the atoms except one, and the structure determination was carried out by a series of difference (F_o-F_c) syntheses until the reliability index R was just below 0·10. In the difference maps anisotropy showed strongly in the zinc and oxygen peaks and a change was made to a least-squares cycle of refinement to allow the use of three-dimensional anisotropic temperature factors for these atoms.

The calculations for Fourier summations were carried out on an IBM type 650 computer, with programs devised by Brown, Lingafelter, Stewart & Jensen (1959). The least-squares refinement program of Busing & Levy (1959) was run on an IBM type 709 computer. The Hughes weighting scheme was used and $\Sigma w(|F_o|-|F_c|)^2$ was minimized. Atomic scattering factors for zinc were from Thomas & Umeda (1957) and for the other atoms from Berghuis, Haanappel, Potters, Loopstra, MacGillavry & Veenendaal (1955)

Table 1. *Final atomic parameters*

		x/a	y/b	z/c	B
	Zn	0·0892	0·0000	0·2156	*
	O(1)	0·0757	−0·2717	0·0904	*
	O(2)	0·1961	0·2675	0·1410	*
	O(3)	0·2507	−0·1235	0·3107	*
	O(4)	−0·0144	−0·1198	0·3517	*
	O(5)	−0·610	0·2122	0·1577	*
	C(2)	0·3746	0·4640	0·0583	3·78
	C(3)	0·4678	−0·1889	0·3708	4·03
	C(4)	−0·1584	−0·1246	0·5127	2·98
	C(5)	−0·2346	0·5040	0·1733	3·72
	C(22)	0·3203	0·2755	0·1400	2·54
	C(23)	0·4021	0·1200	0·2125	3·04
	C(33)	0·3667	−0·0596	0·2929	2·50
	C(44)	−0·1139	−0·0261	0·3936	2·23
	C(45)	−0·1822	0·1704	0·3354	2·68
	C(55)	−0·1530	0·2806	0·2246	2·17
C(23)	H(1)	0·5035	0·1810	0·2143	†
C(2)	H(2)	0·4100	0·6500	0·0900	†
	H(3)	0·3300	0·5000	−0·0100	†
	H(4)	0·5000	0·4300	0·0400	†
C(3)	H(5)	0·4600	−0·3750	0·3500	†
	H(6)	0·4300	−0·1600	0·4400	†
	H(7)	0·5500	−0·1500	0·3500	†
C(45)	H(8)	−0·2600	0·2560	0·3840	†
C(5)	H(9)	−0·2800	0·3800	0·0900	†
	H(10)	−0·1800	0·6700	0·2000	†
	H(11)	−0·3700	0·5240	0·2300	†
C(4)	H(12)	−0·1800	−0·1400	0·5200	†
	H(13)	−0·1700	−0·3250	0·5600	†
	H(13)	−0·1200	−0·0500	0·5500	†
O(1)	H(15)	0·0400	−0·2250	0·0100	†
	H(16)	0·1600	−0·4000	0·0800	†

* Anisotropic. See Table 2.
† $B=2{\cdot}50$ used for all hydrogen atoms.

Table 2. *Final anisotropic temperature factors*
($\times10^2$)

	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Zn	2·62	3·29	3·08	0·09	0·59	0·03
O(1)	5·02	4·36	2·69	0·16	0·25	0·07
O(2)	2·47	3·11	4·96	−0·10	1·19	0·70
O(3)	2·58	5·19	4·33	−0·35	0·49	0·57
O(4)	2·83	4·82	3·14	0·80	1·01	0·69
O(5)	3·37	3·81	2·82	1·42	0·78	0·68

Hydrogen atoms were located by examining the ΔF maps at distances about 1·05 Å from the carbon atoms and 0·95 Å from the water oxygen atom. A self consistent set of atomic parameters was chosen. Hydrogen positions were not refined and no great accuracy can be claimed for them. The final reliability factor R was 0·072 (1060 reflections). The results of the analysis are given in Tables 1, 2, and 3.

Table 3. *Comparison of observed and calculated structure factors*

(×10)

h	k	l	F_o	F_c	cos α
0	0	1	212	194	1.0000
0	0	2	555	786	1.0000-
0	0	3	347	353	1.0000--
0	0	4	129	85	1.0000-
0	0	5	228	250	1.0000
0	0	6	140	107	1.0000
0	0	7	359	374	1.0000-
0	0	8	57	35	1.0000-
0	0	9	183	176	1.0000
0	0	10	108	89	1.0000
0	0	11	140	150	1.0000-
0	0	12	94	101	1.0000-
0	0	13	79	86	1.0000
1	0	0	571	748	1.0000
1	0	1	338	379	1.0000-
1	0	-1	430	495	1.0000
1	0	2	493	609	1.0000-
1	0	-2	62	53	1.0000-
1	0	3	298	310	1.0000-
1	0	-3	404	456	1.0000-
1	0	4	48	10	.9999
1	0	-4	59	65	1.0000
1	0	5	112	95	1.0000
1	0	-5	399	381	1.0000
1	0	6	47	51	1.0000
1	0	-6	61	46	1.0000-
1	0	7	294	287	1.0000-
1	0	-7	267	263	1.0000-
1	0	8	133	121	1.0000
1	0	-8	102	106	1.0000-
1	0	9	208	225	1.0000
1	0	-9	124	133	1.0000
1	0	10	-29	17	.9999-
1	0	-10	158	151	1.0000
1	0	11	160	171	1.0000-
1	0	-11	30	27	1.0000
1	0	12	27	47	1.0000
1	0	-12	94	90	1.0000-
1	0	13	106	130	1.0000
1	0	-13	39	56	1.0000-
2	0	0	130	137	1.0000
2	0	1	45	37	1.0000
2	0	-1	68	30	1.0000-
2	0	2	355	361	1.0000-
2	0	-2	265	227	1.0000
2	0	3	247	234	1.0000
2	0	-3	178	170	1.0000-
2	0	4	233	244	1.0000
2	0	-4	286	248	1.0000-
2	0	5	61	36	1.0000
2	0	-5	466	515	1.0000
2	0	6	269	255	1.0000-
2	0	-6	-22	18	.9999
2	0	7	182	189	1.0000-
2	0	-7	323	329	1.0000-
2	0	8	298	301	1.0000
2	0	-8	253	253	1.0000-
2	0	9	143	146	1.0000
2	0	-9	-28	11	.9999
2	0	10	88	78	1.0000-
2	0	-10	131	128	1.0000
2	0	11	66	64	1.0000-
2	0	-11	140	139	1.0000
2	0	12	-24	31	1.0000-
2	0	-12	61	57	1.0000-
2	0	13	56	47	1.0000
2	0	-13	38	46	1.0000-
3	0	0	179	182	1.0000-
3	0	1	281	244	1.0000-
3	0	-1	598	654	1.0000
3	0	2	310	306	1.0000-
3	0	-2	356	331	1.0000
3	0	3	419	414	1.0000
3	0	-3	115	105	1.0000-
3	0	4	170	153	1.0000
3	0	-4	324	306	1.0000-
3	0	5	409	407	1.0000-
3	0	-5	300	268	1.0000
3	0	6	376	356	1.0000-
3	0	-6	429	410	1.0000
3	0	7	133	130	1.0000
3	0	-7	-26	27	1.0000
3	0	8	277	274	1.0000
3	0	-8	364	408	1.0000-
3	0	9	98	90	1.0000
3	0	-9	178	185	1.0000-
3	0	10	123	128	1.0000-
3	0	-10	62	59	1.0000
3	0	11	57	66	1.0000-
3	0	-11	171	181	1.0000
3	0	12	-23	5	.9993
3	0	-12	-25	14	1.0000-
3	0	13	71	82	1.0000
3	0	-13	106	119	1.0000-
4	0	0	311	332	1.0000-
4	0	1	420	397	1.0000-
4	0	-1	135	142	1.0000
4	0	2	19	17	1.0000-
4	0	-2	279	274	1.0000
4	0	3	503	558	1.0000
4	0	-3	-19	14	1.0000-
4	0	4	245	233	1.0000-
4	0	-4	376	384	1.0000-
4	0	5	390	364	1.0000-
4	0	-5	41	30	1.0000
4	0	6	-26	10	1.0001
4	0	-6	428	454	1.0000
4	0	7	203	216	1.0000
4	0	-7	160	191	1.0000
4	0	8	228	231	1.0000
4	0	-8	260	249	1.0000-
4	0	9	44	31	1.0000
4	0	-9	229	231	1.0000-
4	0	10	215	229	1.0000-
4	0	-10	-29	26	1.0000
4	0	11	27	31	1.0000
4	0	-11	203	195	1.0000
4	0	12	96	103	1.0000
4	0	-12	50	61	1.0000
4	0	-13	99	110	1.0000-
5	0	0	226	202	1.0000-
5	0	1	238	223	1.0000-
5	0	-1	42	30	1.0000
5	0	2	336	307	1.0000
5	0	-2	64	82	1.0000
5	0	3	336	341	1.0000
5	0	-3	125	78	1.0000
5	0	4	181	161	1.0000-
5	0	-4	152	143	1.0000-
5	0	5	361	337	1.0000-
5	0	-5	171	161	1.0000-
5	0	6	-28	22	.9999
5	0	-6	339	339	1.0000
5	0	7	219	231	1.0000
5	0	-7	97	95	1.0000
5	0	8	154	143	1.0000
5	0	-8	175	174	1.0000-
5	0	9	61	28	1.0000-
5	0	-9	226	219	1.0000-
5	0	10	178	191	1.0000-
5	0	-10	-28	30	1.0000-
5	0	11	-24	10	1.0001
5	0	-11	140	151	1.0000
5	0	12	76	84	1.0000
5	0	-12	73	68	1.0000
5	0	-13	96	88	1.0000-
6	0	0	344	307	1.0000-
6	0	1	131	118	1.0000-
6	0	-1	242	227	1.0000-
6	0	2	272	273	1.0000
6	0	-2	362	375	1.0000
6	0	3	164	139	1.0000
6	0	-3	282	275	1.0000
6	0	4	246	230	1.0000-
6	0	-4	102	92	1.0000-
6	0	5	302	292	1.0000-
6	0	-5	162	149	1.0000-
6	0	6	161	159	1.0000
6	0	-6	83	62	1.0000

h	k	l	F_o	F_c	cos α	sin α
6	0	7	195	195	1.0000	
6	0	-7	122	119	1.0000	
6	0	8	53	50	1.0000	
6	0	-8	208	199	1.0000	
6	0	9	59	65	1.0000-	
6	0	-9	173	171	1.0000-	
6	0	10	95	107	1.0000-	
6	0	-10	158	165	1.0000-	
6	0	11	-21	15	1.0001	
6	0	-11	89	93	1.0000	
6	0	12	54	75	1.0000	
6	0	-12	59	58	1.0000	
7	0	0	321	290	1.0000-	
7	0	1	76	66	1.0000	
7	0	-1	482	491	1.0000-	
7	0	2	200	201	1.0000	
7	0	-2	175	183	1.0000	
7	0	3	94	84	1.0000-	
7	0	-3	454	456	1.0000	
7	0	4	59	40	1.0000-	
7	0	-4	28	39	1.0000-	
7	0	5	43	32	1.0000	
7	0	-5	239	216	1.0000-	
7	0	6	160	174	1.0000	
7	0	-6	149	146	1.0000-	
7	0	7	103	99	1.0000	
7	0	-7	44	28	1.0000-	
7	0	8	103	108	1.0000-	
7	0	-8	291	279	1.0000	
7	0	9	109	117	1.0000-	
7	0	-9	51	51	1.0000	
7	0	10	57	58	1.0000	
7	0	-10	164	175	1.0000-	
7	0	11	50	50	1.0000	
7	0	-11	-22	3	.9988	
7	0	-12	72	78	1.0000	
8	0	0	58	46	1.0000-	
8	0	1	213	191	1.0000	
8	0	-1	448	434	1.0000-	
8	0	2	99	102	1.0000	
8	0	-2	72	79	1.0000-	
8	0	3	-28	24	1.0000	
8	0	-3	363	367	1.0000	
8	0	4	61	55	1.0000-	
8	0	-4	153	160	1.0000	
8	0	5	-29	27	1.0000	
8	0	-5	82	73	1.0000-	
8	0	6	143	132	1.0000	
8	0	-6	230	215	1.0000-	
8	0	7	-28	31	1.0000-	
8	0	-7	-29	25	1.0000	
8	0	8	184	176	1.0000-	
8	0	-8	148	143	1.0000	
8	0	9	53	64	1.0000-	
8	0	-9	-27	24	1.0000-	
8	0	10	106	117	1.0000	
8	0	-10	94	91	1.0000-	
9	0	0	31	22	1.0000	
9	0	1	217	194	1.0000	
9	0	-1	253	251	1.0000-	
9	0	2	154	141	1.0000	
9	0	-2	263	261	1.0000-	
9	0	3	112	105	1.0000-	
9	0	-3	148	132	1.0000	
9	0	4	237	236	1.0000-	
9	0	-4	196	206	1.0000	
9	0	5	135	132	1.0000	
9	0	-5	31	34	1.0000	
9	0	6	135	139	1.0000	
9	0	-6	202	209	1.0000-	
9	0	7	60	63	1.0000-	
9	0	-7	59	35	1.0000	
9	0	8	94	108	1.0000-	
9	0	-8	193	194	1.0000	
9	0	-9	25	41	1.0000	
9	0	-10	93	100	1.0000-	
10	0	0	107	125	1.0000	
10	0	1	196	193	1.0000	
10	0	-1	103	103	1.0000-	
10	0	2	92	92	1.0000-	
10	0	-2	123	123	1.0000-	
10	0	3	113	113	1.0000-	
10	0	-3	30	42	1.0000-	
10	0	4	-28	13	1.0000-	
10	0	-4	74	66	1.0000	
10	0	5	162	179	1.0000	
10	0	-5	83	83	1.0000	
10	0	6	62	54	1.0000	
10	0	-6	175	174	1.0000-	
10	0	7	92	107	1.0000-	
10	0	-7	98	102	1.0000-	
10	0	8	25	53	1.0000-	
10	0	-8	97	112	1.0000	
10	0	-9	150	153	1.0000	
11	0	0	175	182	1.0000	
11	0	1	-28	10	.9998	
11	0	-1	-28	1	1.0062-	
11	0	2	178	193	1.0000-	
11	0	-2	28	53	1.0000-	
11	0	3	-26	6	.9997	
11	0	-3	-27	27	1.0000-	
11	0	4	94	100	1.0000	
11	0	-4	-26	26	1.0000	
11	0	5	61	65	1.0000	
11	0	-5	64	70	1.0000	
11	0	6	-18	29	1.0000-	
11	0	-6	34	43	1.0000-	
11	0	7	83	105	1.0000-	
11	0	-7	123	139	1.0000-	
12	0	0	129	147	1.0000	
12	0	1	24	36	1.0000-	
12	0	-1	61	71	1.0000	
12	0	2	139	192	1.0000-	
12	0	-2	35	41	1.0000-	
12	0	3	-20	1	.9599	
12	0	-3	148	153	1.0000-	
12	0	4	121	133	1.0000	
12	0	-4	-21	18	1.0000	
12	0	5	22	27	1.0000	
12	0	-5	112	112	1.0000	
13	0	0	84	86	1.0000	
13	0	1	69	73	1.0000-	
13	0	-1	117	123	1.0000	
13	0	2	106	115	1.0000-	
13	0	-2	-17	10	1.0001	
13	0	-3	149	140	1.0000-	
0	1	1	487	793	.2294-	.9373
0	1	2	442	533	.8406-	.5146-
0	1	3	415	477	.2274	.9378-
0	1	4	436	428	.8785	.4777-
0	1	5	104	78	.9611	.2672
0	1	6	317	323	.1644	.9684
0	1	7	66	76	.8953-	.4545-
0	1	8	238	253	.3043-	.9256-
0	1	9	132	143	.7450-	.6760-
0	1	10	189	191	.3596-	.9331
0	1	11	148	146	.2397	.9079
0	1	12	109	101	.7640	.6542-
0	1	13	124	115	.1698	.9585-
1	1	0	263	301	.1988	.9080
1	1	1	409	551	.2734-	.9169
1	1	-1	272	352	.0350	.9994-
1	1	2	400	457	.1985-	.9081-
1	1	-2	459	704	.3422-	.9936-
1	1	3	395	458	.7800	.6527-
1	1	-3	224	230	.9995	.0332-
1	1	4	181	169	.9986	.0258
1	1	-4	366	415	.2629-	.9468
1	1	5	239	233	.1612	.9689
1	1	-5	157	164	.7843-	.6024
1	1	6	313	314	.0127-	.9999
1	1	-6	340	359	.2369-	.9175-
1	1	7	158	157	.7303-	.6381-
1	1	-7	84	78	.1186-	.7299-
1	1	8	302	314	.5224-	.8257-
1	1	-8	118	103	.0351	.9994
1	1	9	118	92	.1042-	.7496
1	1	-9	193	182	.5278	.8944
1	1	10	219	201	.1793	.9388

h	k	l	F_o	F_c	cos α	sin α
1	1	-10	-43	48	.5787	.8516-
1	1	11	43	34	.9241	.3280
1	1	-11	114	94	.1472	.9981-
1	1	12	129	139	.4115	.9114-
1	1	-12	-36	21	.5592-	.8920-
1	1	13	51	50	.3926	.9917-
1	1	-13	118	106	.1348	.9099
2	1	0	260	249	.7552	.6555
2	1	1	510	790	.5505	.8439
2	1	-1	88	72	.9959	.0094
2	1	2	232	236	.8189-	.5379-
2	1	-2	369	457	.2597-	.9567-
2	1	3	340	346	.4360	.8999-
2	1	-3	304	343	.9045-	.4624-
2	1	4	28	30	.1013	.9499-
2	1	-4	478	545	.1838-	.9380
2	1	5	283	280	.2517-	.9768
2	1	-5	309	333	.3737	.9725
2	1	6	133	106	.1018-	.9498
2	1	-6	325	334	.2295-	.9373-
2	1	7	276	277	.4328-	.9105-
2	1	-7	243	245	.0916	.7598-
2	1	8	218	201	.5614-	.8725-
2	1	-8	72	67	.7184	.6596
2	1	9	227	224	.5240	.8157
2	1	-9	176	171	.1063-	.9493
2	1	10	-43	54	.2191-	.9577
2	1	-10	-43	20	.8960-	.4441
2	1	11	58	56	.4080-	.9310-
2	1	-11	120	121	.0057	1.0000-
2	1	12	95	99	.3618	.9233-
2	1	-12	84	78	.0773	.9798-
2	1	13	27	29	.5176	.8556-
2	1	-13	74	81	.4683	.8386
3	1	0	247	280	.5600	.8825
3	1	1	337	353	.9483	.3713
3	1	-1	98	88	.8635	.5403
3	1	2	415	466	.0577-	.9893-
3	1	-2	364	424	.2910	.9657-
3	1	3	381	400	.6242-	.7182-
3	1	-3	273	291	.5755-	.8718-
3	1	4	375	363	.2776-	.9067
3	1	-4	384	401	.5504-	.8439
3	1	5	264	251	.0749-	.9792
3	1	-5	343	367	.1119-	.9397
3	1	6	184	178	.1862-	.9285-
3	1	-6	-35	49	.2805-	.9959-
3	1	7	220	215	.3062	.9250-
3	1	-7	320	320	.0645	.9799-
3	1	8	107	45	.9970-	.0778-
3	1	-8	146	132	.9978	.0667-
3	1	9	224	234	.1021-	.9498
3	1	-9	214	219	.2371-	.9175
3	1	10	62	63	.1430	.9987
3	1	-10	141	144	.4356-	.9001
3	1	11	89	86	.2885	.9755-
3	1	-11	102	103	.3150	.9941-
3	1	12	107	98	.2229	.9478-
3	1	-12	146	159	.1396	.9092-
3	1	13	24	37	.8909	.4451
3	1	-13	28	26	.6875	.7621
4	1	0	268	284	.4383	.8898
4	1	1	425	393	.8065	.5192-
4	1	-1	271	270	.0196	.9998
4	1	2	370	385	.2032-	.9971-
4	1	-2	228	205	.9335	.3857-
4	1	3	304	286	.9531-	.3206
4	1	-3	377	440	.4270-	.9402-
4	1	4	461	490	.0296-	.9996
4	1	-4	120	104	.6249-	.7087-
4	1	5	122	103	.9191-	.3399
4	1	-5	246	232	.1504-	.9886
4	1	6	195	195	.2582	.9661-
4	1	-6	153	134	.4369-	.8995
4	1	7	159	152	.5133	.8852-
4	1	-7	250	249	.0446	.9990-
4	1	8	88	74	.9316-	.3364
4	1	-8	131	108	.4922	.8075-
4	1	9	184	196	.1057-	.9494
4	1	-9	119	102	.7895-	.6318
4	1	10	43	51	.9937	.1212
4	1	-10	159	155	.0209-	.9998
4	1	11	141	157	.1344	.9099-
4	1	-11	-39	19	.7666	.6241
4	1	12	-30	26	.2446	.9966-
4	1	-12	157	124	.3073	.9156-
4	1	-13	52	52	.6960	.7811-
5	1	0	312	315	.6245	.7180
5	1	1	272	287	.1111-	.9398-
5	1	-1	317	324	.3338	.9247
5	1	2	254	238	.0190	.9998-
5	1	-2	190	191	.6192	.7582
5	1	3	180	163	.7969-	.6401
5	1	-3	248	276	.1756-	.9485-
5	1	4	350	330	.3407-	.9042
5	1	-4	237	244	.4202-	.9704-
5	1	5	119	108	.7923-	.6012-
5	1	-5	242	248	.2647-	.9463
5	1	6	225	213	.1645	.9684-
5	1	-6	264	274	.1748	.9486
5	1	7	98	86	.7246	.6982-
5	1	-7	148	141	.1937	.9181-
5	1	8	100	95	.4689	.8383
5	1	-8	172	169	.1934-	.9181-
5	1	9	97	85	.3746-	.9722
5	1	-9	110	97	.9076-	.4918-
5	1	10	40	44	.0036	1.0000-
5	1	-10	155	143	.0973	.9593
5	1	11	115	119	.0429-	.9991-
5	1	-11	54	52	.3681	.9928
5	1	12	62	59	.3027	.9351
5	1	-12	133	111	.0645	.9799-
6	1	0	78	62	.6602-	.7151-
6	1	1	384	387	.2113-	.9774-
6	1	-1	262	269	.3510	.9634
6	1	2	132	127	.3246-	.9540-
6	1	-2	195	178	.4206	.9702
6	1	3	275	250	.2115-	.9774
6	1	-3	260	258	.0606	.9892-
6	1	4	201	204	.2316-	.9278
6	1	-4	272	262	.2861	.9852-
6	1	5	188	183	.3482-	.9734-
6	1	-5	158	141	.1243-	.9292
6	1	6	206	188	.4323	.9107-
6	1	-6	237	218	.3003-	.9358
6	1	7	119	102	.4610	.8784
6	1	-7	88	85	.3416	.9939
6	1	8	154	155	.0709	.9795
6	1	-8	179	182	.3712-	.9825-
6	1	9	-40	11	.6767-	.7631
6	1	-9	159	146	.3211-	.9740-
6	1	10	111	80	.2511-	.9860-
6	1	-10	193	189	.2469	.9960
6	1	11	80	82	.0634-	.9890-
6	1	-11	140	145	.1429	.9987
6	1	-12	89	96	.0610-	.9891-
7	1	0	302	298	.1641-	.9685-
7	1	1	227	195	.0010-	1.0000-
7	1	-1	160	151	.2981-	.9455
7	1	2	194	176	.0899-	.9599
7	1	-2	337	348	.0763-	.9791
7	1	3	294	290	.1693	.9586
7	1	-3	107	83	.0768-	.9790-
7	1	4	-40	5	.3085	.9051-
7	1	-4	272	284	.2152	.9676-
7	1	5	176	164	.0899-	.9599-
7	1	-5	215	214	.9994	.0535
7	1	6	127	121	.3464-	.9831-
7	1	-6	-41	40	.3638-	.9135
7	1	7	126	117	.1591-	.9783
7	1	-7	184	175	.6425-	.7663
7	1	8	140	132	.3213	.9740
7	1	-8	118	103	.8034-	.5594-
7	1	9	85	84	.5455	.8831-
7	1	-9	194	194	.2381-	.9172-
7	1	10	107	103	.0295-	.9996-
7	1	-10	39	33	.0881	.9691
7	1	-11	143	158	.3755	.9628

h	k	l	F_o	F_c	cos α	sin α
8	1	0	245	262	.1030-	.9497-
8	1	1	187	168	.9930	.1811-
8	1	-1	95	89	.9687-	.2843-
8	1	2	97	91	.3924-	.9918
8	1	-2	308	322	.1281-	.9198
8	1	3	146	141	.2056-	.9876
8	1	-3	129	108	.4314-	.9202
8	1	4	62	58	.0364	.9993-
8	1	-4	220	215	.0464	.9899-
8	1	5	189	181	.5162-	.8655-
8	1	-5	195	184	.8469	.5138-
8	1	6	88	81	.7385-	.6473-
8	1	-6	100	96	.6459	.7364
8	1	7	151	148	.5602	.8824
8	1	-7	212	205	.0318-	.9995
8	1	8	78	65	.5956	.8302
8	1	-8	60	60	.9561-	.2390-
8	1	9	88	98	.4624	.8687-
8	1	-9	110	127	.3294-	.9442-
8	1	10	56	56	.0186-	.9998-
8	1	-10	68	55	.7357-	.6774-
8	1	-11	38	45	.1456-	.9983
9	1	0	172	167	.3055	.9252-
9	1	1	78	71	.9606	.2779
9	1	-1	127	119	.3955-	.9815-
9	1	2	127	129	.6432-	.7567
9	1	-2	162	149	.2533-	.9764
9	1	3	135	125	.3388-	.9049
9	1	-3	135	134	.1580-	.9784
9	1	4	146	134	.1573-	.9785-
9	1	-4	119	104	.2447	.9966-
9	1	5	129	120	.0316	.9995-
9	1	-5	223	237	.1377	.9095-
9	1	6	116	129	.3175	.9842
9	1	-6	44	33	.8792-	.4675-
9	1	7	124	139	.3930	.9915
9	1	-7	235	235	.1112	.9398
9	1	8	32	41	.1852-	.9287-
9	1	-8	54	68	.5397	.8149
9	1	9	84	86	.1206	.9297-
9	1	-9	127	125	.1703-	.9584-
9	1	-10	61	66	.0499	.9898-
10	1	0	154	151	.6183-	.7589-
10	1	1	171	161	.1990-	.9080
10	1	-1	140	134	.1244	.9292-
10	1	2	163	166	.1864	.9285
10	1	-2	76	75	.4713	.8280
10	1	3	85	66	.3197-	.9745-
10	1	-3	124	121	.1237-	.9293
10	1	4	170	166	.1677	.9588-
10	1	-4	136	126	.7149	.6993
10	1	5	-38	44	.6984	.7517-
10	1	-5	145	160	.1159-	.9393-
10	1	6	159	158	.0567	.9894
10	1	-6	144	138	.4181-	.9804-
10	1	7	31	38	.0685-	.9796
10	1	-7	91	102	.1699	.9585
10	1	8	91	97	.1721-	.9581-
10	1	-8	127	119	.0896	.9690
10	1	-9	71	56	.3530-	.9536-
11	1	0	109	102	.8369-	.5744-
11	1	1	162	177	.0062-	1.0000
11	1	-1	188	184	.3819-	.9422-
11	1	2	79	86	.6982	.7519
11	1	-2	-39	33	.9863	.1467-
11	1	3	119	138	.0452	.9990-
11	1	-3	89	98	.1871	.9283
11	1	4	61	63	.1655	.9582-
11	1	-4	128	128	.3908	.9525
11	1	5	-31	5	.5678-	.8420
11	1	-5	65	64	.0852	.9594-
11	1	6	66	87	.0132	.9999
11	1	-6	101	114	.1946-	.9089-
11	1	-7	-28	13	.0231-	.9998
11	1	-8	126	122	.0694	.9796
12	1	0	-32	41	.8092-	.5785
12	1	1	137	136	.0930-	.9597
12	1	-1	91	101	.2212-	.9572-
12	1	2	-31	27	.8317	.5553
12	1	-2	102	89	.0154-	.9999-
12	1	3	114	122	.2065	.9974-
12	1	-3	57	50	.0611	.9891
12	1	4	36	39	.9981	.0265-
12	1	-4	143	125	.0632	.9990
12	1	-5	40	32	.0697-	.9796-
12	1	-6	120	127	.1254	.9291-
13	1	0	91	92	.2148-	.9577
13	1	-1	72	20	.8937-	.4848-
13	1	-2	111	113	.0454	.9990-
0	2	1	196	233	.3294-	.9442
0	2	2	251	294	.9926-	.1121-
0	2	3	180	173	.1559-	.9788-
0	2	4	355	409	.9994	.0436
0	2	5	325	361	.9592	.2287
0	2	6	169	145	.8711-	.4191-
0	2	7	246	264	.9996-	.0923
0	2	8	126	110	.9580-	.2589-
0	2	9	118	112	.9255	.3877
0	2	10	144	141	.9989	.0645-
0	2	11	84	77	.9884-	.1157
0	2	12	125	123	.9831-	.1381
0	2	13	-17	7	.8958	.4447-
1	2	0	138	157	.9771	.2218-
1	2	1	331	405	.7792-	.6527
1	2	-1	51	44	.2446-	.9766-
1	2	2	199	214	.9589-	.2386-
1	2	-2	303	370	.9475-	.3918
1	2	3	198	196	.9993	.0330
1	2	-3	315	418	.9985-	.0556
1	2	4	234	216	.9662	.2757
1	2	-4	203	200	.6385	.7966-
1	2	5	188	178	.7281	.6585-
1	2	-5	410	533	.9993	.0533
1	2	6	285	293	.8933-	.4944-
1	2	-6	169	164	.8657	.5005
1	2	7	291	300	.8092-	.5786
1	2	-7	177	179	.9976-	.0964-
1	2	8	189	181	1.0000	.0503
1	2	-8	163	156	.9228-	.3582-
1	2	9	190	191	.9855	.1964-
1	2	-9	97	89	.8673	.4799-
1	2	10	-30	9	.5013-	.8564-
1	2	-10	176	174	.9998	.0919-
1	2	11	125	124	.9995-	.0233
1	2	-11	42	36	.9559-	.2398
1	2	12	82	77	.9707-	.2041-
1	2	-12	142	145	.9813-	.1297
1	2	13	75	72	.9293	.3763
1	2	-13	37	49	.9697-	.2442
2	2	0	140	138	.9234	.3389
2	2	1	305	355	.9628-	.2074
2	2	-1	289	355	.9728	.2137
2	2	2	274	299	1.0000-	.0007-
2	2	-2	97	86	.2724-	.9262-
2	2	3	330	358	.8751	.4480-
2	2	-3	390	512	.9816-	.1097-
2	2	4	402	439	1.0000	.0403
2	2	-4	271	306	.8143-	.5084
2	2	5	139	117	.2301	.9372-
2	2	-5	166	179	.9649	.2265-
2	2	6	279	278	.9996-	.0829-
2	2	-6	214	219	.9999	.0315-
2	2	7	173	158	.6523-	.7759
2	2	-7	66	54	.8854-	.4469
2	2	8	239	235	.9558	.2490-
2	2	-8	179	188	.9998-	.0029-
2	2	9	104	102	.9999	.0511
2	2	-9	122	118	.9402	.3046-
2	2	10	138	147	.9269-	.3572
2	2	-10	195	191	.9832	.1286
2	2	11	146	144	.9533-	.3109-
2	2	-11	51	51	.7124-	.7107-
2	2	12	-22	12	.7923-	.6011-
2	2	-12	133	132	.9999-	.0312
2	2	-13	92	86	.9798-	.1999
3	2	0	116	93	.8856	.4465-
3	2	1	398	495	.9614-	.2572-
3	2	-1	379	466	.9681	.2054
3	2	2	350	365	.9292-	.3965

Table 3 (*cont.*)

h	k	l	Fo	Fc	cosα	sinα
3	2	-2	283	291	•9960	•0984
3	2	-3	408	455	•9629	•2070-
3	2	-3	295	358	•7815-	•6329-
3	2	-4	321	344	•9959	•0896
3	2	-4	292	354	•9180-	•3697
3	2	-5	133	122	•9116-	•4110
3	2	-5	25	28	•8864	•4360
3	2	-6	196	201	•9766-	•2513-
3	2	-6	205	210	•9969	•0878-
3	2	-7	116	111	•9912-	•1235-
3	2	-7	79	73	•9740	•2624
3	2	-8	152	139	•7779	•6824
3	2	-8	128	122	•9906-	•1731-
3	2	-9	32	38	•9969	•0971
3	2	-9	73	73	•1679-	•9588-
3	2	-10	142	133	1•0000-	•0203
3	2	-10	149	139	•8417	•5939
3	2	-11	66	63	•8794-	•4671-
3	2	-11	58	61	•9966	•0888-
3	2	-12	57	58	•9988	•0847
3	2	-12	96	91	•9942-	•1702-
3	2	-13	128	124	•9776-	•2015
4	2	0	291	303	•9294	•3961-
4	2	1	258	275	•9984-	•0659
4	2	-1	260	298	•9945	•1404
4	2	-2	190	197	•0019	1•0000
4	2	-2	271	299	•9999	•0014-
4	2	-3	272	279	•8004	•5995-
4	2	-3	199	193	•9701-	•2246-
4	2	-4	188	163	•9943	•1608
4	2	-4	340	403	•9255-	•3877
4	2	-5	150	151	•9922-	•1520
4	2	-5	154	144	•9708-	•2938-
4	2	-6	118	99	•8639-	•5306-
4	2	-6	230	236	•9735	•2827-
4	2	-7	90	67	•9360	•3250
4	2	-7	174	170	•9565	•2198
4	2	-8	86	87	•7944	•6704
4	2	-8	72	59	•8952-	•4546-
4	2	-9	71	66	•4021-	•9516-
4	2	-9	86	72	•8483-	•5925-
4	2	-10	132	119	•9663-	•2753
4	2	-10	-29	9	•9535	•3008
4	2	-11	-24	8	•8109	•5584-
4	2	-11	134	127	•9887	•1948-
4	2	-12	111	106	•9919	•1722-
4	2	-12	39	37	•9291	•3969
5	2	0	257	265	•9965-	•0388-
5	2	-1	54	48	•8074-	•5989
5	2	-1	43	54	•9123	•4905-
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Discussion

The main features of the structure are as described by Lippert & Truter (1960). Differences in detail are primarily due to errors in their determination * To facilitate comparison we have adopted their notation for the several atoms (Fig. 1).

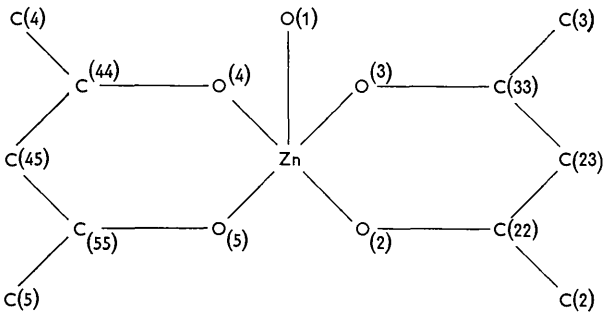

Fig. 1. Numbering of the atoms.

The bond lengths and angles, with their estimated standard deviations (Jeffrey & Cruickshank, 1953) are given in Table 4.

The five oxygen atoms coordinated to the zinc ion appear to be all at the same distance, 2·02 ± 0·02 Å. The arrangement of the oxygen atoms is intermediate between tetragonal pyramidal and trigonal bipyramidal, although somewhat nearer to the former. A comparison of the two models is shown by the bond angles in Table 5. The angles for the tetragonal pyramidal model were calculated with Zn–O distances of 2·02 Å and with the Zn atom 0·41 Å above the base plane.

The mean deviation of the bond angles is 2·9° from the tetragonal pyramid and 10·5° from the trigonal bipyramid. If a bipyramidal model is used in which the trigonal symmetry is not required (*i.e.* symmetry C_s rather than C_{3h}) the mean deviation of the 7 symmetry-fixed angles is 6·4°, while the mean deviation of all angles (taking the deviations in the equatorial plane to be zero) is 4·5°. The most obvious and important deviation of the molecule from the tetragonal pyramidal model is the deviation of the basal set of four O atoms from planarity, atoms O(3) and O(4) lying 0·1 Å above and O(2) and O(5) lying 0·1 Å below their mean plane.

The two acetylacetone groups are nearly planar, but not coplanar, each group being tilted about 12° from normality to the Zn–H_2O bond.

Table 4 *Bond lengths and angles in zinc acetylacetonate monohydrate*

Estimated standard deviation in parentheses

Bond lengths (Å)		Bond angles (°)	
Zn–O(1)	1·999 (0·02)	O(1)–Zn–O(2)	104·9 (0·8)
Zn–O(2)	2·026 (0·02)	O(1)–Zn–O(3)	97·5 (0·8)
Zn–O(3)	2·038 (0·02)	O(1)–Zn–O(4)	104·9 (0·8)
Zn–O(4)	2·005 (0·02)	O(1)–Zn–O(5)	100·3 (0·8)
Zn–O(5)	2·011 (0·02)	O(2)–Zn–O(3)	88·5 (0·8)
O(2)–C(22)	1·303 (0·048)	O(4)–Zn–O(5)	88·0 (0·8)
O(3)–C(33)	1·290 (0·048)	Zn–O(2)–C(22)	127·3 (1·3)
O(4)–C(44)	1·270 (0·048)	Zn–O(3)–C(33)	126·5 (1·3)
O(5)–C(55)	1·301 (0·048)	Zn–O(4)–C(44)	129·3 (1·3)
C(22)–C(23)	1·404 (0·058)	Zn–O(5)–C(55)	125·2 (1·3)
C(33)–C(23)	1·373 (0·058)	O(2)–C(22)–C(23)	123·3 (1·8)
C(44)–C(45)	1·404 (0·058)	O(3)–C(33)–C(23)	125·5 (1·8)
C(55)–C(45)	1·400 (0·058)	O(4)–C(44)–C(45)	122·7 (1·8)
C(2)–C(22)	1·488 (0·064)	O(5)–C(55)–C(45)	125·4 (1·8)
C(3)–C(33)	1·487 (0·064)	C(22)–C(23)–C(33)	126·8 (2·1)
C(4)–C(44)	1·508 (0·064)	C(44)–C(45)–C(55)	125·1 (2·1)
C(5)–C(55)	1·556 (0·064)	O(2)–C(22)–C(2)	116·7 (2·2)
		O(3)–C(33)–C(3)	115·6 (2·2)
		O(4)–C(44)–C(4)	118·7 (2·2)
		O(5)–C(55)–C(5)	115·2 (2·2)

Table 5. *Bond angles in* $Zn(acac)_2.H_2O$

Angle	Actual	Tetr. pyr. model	Diff.	Trig. bipyr. model	Diff.
O(1)–Zn–O(2)	104·9	101·7	3·2	120	15·1
O(1)–Zn–O(3)	97·5	101·7	4·2	90	7·5
O(1)–Zn–O(4)	104·9	101·7	3·2	120	15·1
O(1)–Zn–O(5)	100·3	101·7	1·4	90	10·3
O(2)–Zn–O(3)	88·5	87·8	0·7	90	1·5
O(2)–Zn–O(4)	150·1	156·7	6·6	120	30·1
O(2)–Zn–O(5)	85·0	87·8	2·8	90	5·0
O(3)–Zn–O(4)	89·4	87·8	1·6	90	0·6
O(3)–Zn–O(5)	162·0	156·7	5·3	180	18·0
O(4)–Zn–O(5)	88·0	87·8	0·2	90	2·0

This investigation was supported by the U.S. Public Health Service under Grant A-2241.

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* *Note by Mary R. Truter and E. L. Lippert*: We are grateful to the authors of this paper for pointing out that in Lippert & Truter (1960), the angle β is quoted as 93·6° whereas the structure factors quoted correspond to $\beta = 86·4°$. The refinement has been repeated with this error corrected and an empirical extinction correction has also been applied; the fractional coordinates do not differ significantly from those published and only for two values of y (for C(3) and C(44)) is the difference between our corrected results and those in this paper greater than twice the standard deviation. The effect of the correction of β (and a change in b_0 to 5·376 Å by a new Straumanis determination) is to alter the bond lengths round the zinc atom significantly but neither the general stereochemistry of the molecule nor the dimensions within the β-diketone have been changed significantly. Revised values of the Zn–O bond lengths are 2·00–2·02 Å for all bonds including that to the water molecule. The arrangement of the five ligand atoms about the zinc is essentially unchanged, that is it can be regarded as a distorted trigonal bipyramid or as a distorted tetragonal pyramid.