measurements, the following structure is proposed for an N-substituted 2-carbamoyldimedone–metal complex with a metal–ligand ratio of 1:2.

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Five-Coordinated Structure of Bis(N-methylsalicylaldiminato)zinc(II); Isomorphism with the Cobalt(II) and Manganese(II) Analogs

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The crystal structure of bis(N-methylsalicylaldiminato)zinc(II) has been determined by three-dimensional X-ray analysis. The structure consists of dimers with symmetry C_i, in which the metal atom shows the coordination number of five. The configuration around the metal atom can be described as a distorted trigonal bipyramid. The Mn(II) and Co(II) derivatives are isomorphous with the Zn(II) complex.

Introduction

The ligand N-methylsalicylaldimine forms complexes with many bivalent 3d metals, having the general formula bis(N-methylsalicylaldiminato)metal(II): $(C_6H_4OCHNHCH_3)_2M^{II}$. So far only the structure of the β form of the nickel(II) complex[1] and of the α form of the copper(II) complex[2] have been determined by X-ray analysis. Both complexes have a square-planar configuration.

For the Co(II) and Zn(II) complexes a tetrahedral structure would be expected, on the basis of previous knowledge of the stereochemistry of four-coordinated complexes of these two metals with salicylaldimines.[3] Nevertheless, the reflectance spectrum of bis(N-methylsalicylaldiminato)cobalt(II) could not be interpreted on the basis of a tetrahedral structure.[4] For this reason we have undertaken an X-ray investigation of these complexes, in order to establish their molecular stereochemistry.

In a previous communication[5] we have reported preliminary results on the structure of bis(N-methylsalicylaldiminato)zinc(II) and we have shown the cobalt(II) and manganese(II) analogs to be isomorphous with the zinc complex. We wish now to report the final results of the three-dimensional X-ray analysis of the zinc(II) complex.

This compound has been chosen in preference to the others because it is insensitive to air exposure, while the others tend to decompose.

Experimental Section

The compounds were prepared by reaction between the metal salicylaldehydates and methylamine in methanol solution. Suitable crystals were obtained by slow evaporation from solutions in chloroform. The crystals of the three complexes are identical in shape, but different in color (Mn, orange; Co, emerald-green; Zn, straw yellow).

Cell dimensions were determined from rotation and Weissenberg photographs using Fe Kα radiation (λ 1.9373 A) for the Mn(II) and Co(II) complexes and Cu Kα radiation (λ 1.5418 A) for the Zn(II) complex. The NaCl rotation pattern (a = 5.6387 A) was superimposed on all the photographs for calibration. The results are shown in Table I. The space group was assumed to be $P\bar{1}$ and confirmed by the structural analysis. Visual examination of the X-ray photographs of the three complexes revealed no detectable differences in intensities.

Three-dimensional X-ray analysis was undertaken on the zinc compound, using Cu Kα radiation. The crystal chosen had dimensions: 0.20 × 0.15 × 0.40 mm. Double integrated Weissenberg photographs $hk0$ through $hk6$ were taken on a Nonius integrating camera, using the multiple film equiinclination technique. The intensities of the reflections were measured on a Nonius microdensitometer, the density being assumed proportional to the intensity of the reflections; 2478 independent reflections were examined, about 75% of the total number in the Cu sphere of reflection.

Of these, 617 were found to be too weak to be measured and were assigned half of the minimum observable intensity. All intensities were corrected for Lorentz and polarization effects.

The three reflections $2\bar{2}1$, $\bar{1}21$, and 101 were later considered affected by secondary extinction and not included in the least-squares refinement. No correction was made for absorption.

The atomic scattering factors were taken from Cromer.[6] The zinc scattering factor was corrected for the real part of the anomalous dispersion ($\Delta f' = -1.7$ for Cu Kα).

All calculations were performed on an IBM 1620 with a set of programs written by Albano, Bellon, Pompa, and Scatturin.[7]

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TABLE I

CRYSTAL DATA FOR BIS(N-METHYLSALICYLALDIMINATO)METAL(II) (M = Mn, Co, Zn)

Metal	a, A	b, A	c, A	α	β	γ	V, A^3	Calcd density, g/cm^3	Z^a
Mn	9.35	10.76	8.45	100° 28′	92° 29′	117° 51′	731.6	1.47	2
Co	9.40	10.72	8.30	96° 23′	95° 16′	118° 10′	722.3	1.50	2
Zn	9.48	10.53	8.45	99° 45′	92° 58′	117° 58′	725.7	1.53	2

[a] Number of formula units $(C_6H_4OCHNCH_3)_2Zn^{II}$ in the unit cell.

TABLE II

POSITIONAL PARAMETERS, TEMPERATURE FACTORS, AND THEIR ESTIMATED STANDARD DEVIATIONS

Atom	x/a	$10^5\sigma_x$	y/b	$10^5\sigma_y$	z/c	$10^5\sigma_z$	B, A^2	σB, A^2
Zn	0.0531	8	0.1726	8	0.0505	11	3.59	0.02
O(1)	0.2178	55	0.3598	51	0.0080	71	4.78	0.11
O(2)	−0.0979	54	−0.0332	49	0.1011	70	4.55	0.11
N(1)	−0.1296	67	0.1979	60	−0.0544	86	4.52	0.13
N(2)	0.1307	60	0.2392	54	0.2979	79	4.02	0.12
C(1)	0.0459	78	0.4113	70	−0.1612	103	4.27	0.15
C(2)	0.1983	75	0.4338	68	−0.0952	100	4.13	0.14
C(3)	0.3384	84	0.5471	76	−0.1370	110	4.67	0.17
C(4)	0.3267	100	0.6332	91	−0.2403	129	5.75	0.22
C(5)	0.1743	105	0.6093	95	−0.3053	134	6.03	0.24
C(6)	0.0379	97	0.5019	87	−0.2651	124	5.51	0.21
C(7)	−0.1046	83	0.3014	75	−0.1342	110	4.62	0.16
C(8)	−0.2980	99	0.0943	91	−0.0438	127	5.66	0.21
C(9)	−0.1158	76	0.0526	68	0.3695	100	4.07	0.14
C(10)	−0.1803	73	−0.0514	67	0.2272	99	3.97	0.14
C(11)	−0.3326	87	−0.1750	79	0.2123	114	4.94	0.18
C(12)	−0.4213	100	−0.1934	91	0.3394	127	5.73	0.22
C(13)	−0.3600	106	−0.0891	97	0.4817	135	6.19	0.25
C(14)	−0.2057	103	0.0309	94	0.4998	130	5.96	0.23
C(15)	0.0429	79	0.1813	71	0.4003	105	4.34	0.15
C(16)	0.2865	92	0.3715	83	0.3603	118	5.24	0.19

Structure Determination

A three-dimensional Patterson synthesis showed clearly the Zn–Zn vectors, from which the coordinates of the Zn atoms were easily determined. The short distance between pairs of Zn atoms (3.22 A) was already suggestive of a dimeric structure.

A series of four three-dimensional Fourier syntheses, interpreted with the aid of a model, showed very clearly the position of all the nonhydrogen atoms. At this point the R factor was 21.5% with the temperature factors of the atoms as follows: $B_{Zn} = 3.00\ A^2$, $B_{O,N,C} = 3.50\ A^2$.

R is throughout defined as $\Sigma||F_o| - |F_c||/\Sigma|F_o|$, where the sums are over the independent, observed reflections. Nine cycles of block-diagonal least-squares with individual isotropic temperature factors reduced R to 9.8%. The weighting scheme was as follows.[8] For observed reflections: $w = 1/(a + F_o + cF_o^2)$ with $a = 10.0$ and $c = 0.05$; for unobserved reflections (included in the last three cycles): constant weight, adjusted to give average $w\Delta^2 = 1$.

Atomic coordinates and temperature factors and their estimated standard deviations, from the last cycle, are listed in Table II.

A final three-dimensional ΔF synthesis was calculated and 15 out of 16 hydrogen atoms were located. Search for hydrogen peaks was done on the basis of geometrical considerations. The mean C–H distance is 1.02 A.

TABLE III

COORDINATES OF HYDROGEN ATOMS

Atom	x/a	y/b	z/c
H(3)	0.450	0.540	−0.110
H(4)	0.420	0.680	−0.280
H(5)	0.160	0.630	−0.420
H(7)	−0.200	0.300	−0.210
H′(8)	−0.400	0.120	−0.080
H″(8)	−0.280	0.020	−0.120
H‴(8)	−0.270	0.080	0.050
H(11)	−0.380	−0.260	0.120
H(12)	−0.550	−0.270	0.320
H(13)	−0.400	−0.160	0.540
H(14)	−0.150	0.130	0.590
H(15)	0.080	0.210	0.520
H′(16)	0.310	0.430	0.280
H″(16)	0.390	0.360	0.380
H‴(16)	0.270	0.410	0.480

Introduction of hydrogen coordinates with isotropic temperature factor of 4.50 A^2 reduced R to 9.4%. Table III lists the hydrogen coordinates. The observed and calculated structure factors are listed in Table IV. Unobserved reflections have not been reported in order to reduce the size of the table.

Description of the Structure

The structure consists of dimers formed by two units of bis(N-methylsalicylaldiminato)zinc(II) (Figure 1). In these dimers the zinc atoms achieve the coordination number of five, by sharing two oxygen atoms.

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TABLE IV

OBSERVED AND CALCULATED STRUCTURE FACTORS

H	K	10FO	10FC
	L = 0		
3	0	193	192
4	0	165	162
5	0	114	86
6	0	48	48
7	0	134	132
8	0	148	138
9	0	55	48
10	0	35	42
1	1	93	54
2	1	47	38
3	1	289	308
4	1	383	390
5	1	384	384
6	1	298	304
7	1	144	134
8	1	84	74
9	1	49	34
0	2	72	86
1	2	212	226
2	2	302	336
3	2	473	486
4	2	230	228
5	2	60	46
6	2	83	72
7	2	88	92
0	3	332	344
1	3	293	282
2	3	483	514
3	3	242	256
7	3	81	56
8	3	91	68
0	4	195	212
1	4	201	164
2	4	226	214
3	4	77	86
4	4	113	122
5	4	181	162
6	4	120	96
7	4	113	88
8	4	76	62
0	5	414	412
1	5	327	324
2	5	114	106
3	5	111	98
4	5	160	144
5	5	132	108
6	5	57	38
0	6	251	234
1	6	125	118
2	6	154	152
3	6	68	58
5	6	51	44
6	6	67	52
0	7	50	50
3	7	90	62
4	7	90	64
5	7	81	68
0	8	177	182
1	8	168	134
2	8	148	108
3	8	106	80
4	8	71	54
0	9	162	144
1	9	141	112
2	9	113	86
2	10	32	34
0	11	63	48
1	11	41	44
2	-1	339	370
3	-1	295	318
4	-1	107	130
5	-1	238	254
6	-1	227	202
7	-1	133	112
2	-2	316	320
3	-2	522	560
4	-2	364	410
5	-2	160	174
6	-2	283	306
7	-2	140	150
8	-2	62	54
9	-2	72	68
10	-2	45	42
1	-3	338	344
2	-3	536	562
3	-3	550	558
4	-3	144	126
5	-3	77	74
6	-3	136	124
7	-3	49	58
8	-3	116	118
9	-3	127	118
10	-3	105	104
11	-3	66	92
1	-4	131	152
2	-4	103	80
3	-4	590	632
4	-4	536	578
5	-4	431	480
6	-4	248	248
7	-4	73	98
9	-4	64	76
1	-5	212	206
2	-5	147	134
3	-5	106	92
4	-5	246	260
5	-5	279	300
6	-5	242	272
7	-5	165	196
8	-5	137	162
9	-5	77	100
10	-5	91	80
11	-5	40	36
1	-6	383	372
2	-6	309	306
3	-6	309	324
4	-6	261	254
5	-6	130	142
6	-6	94	98
7	-6	131	134
8	-6	128	132
9	-6	110	110
10	-6	90	94
11	-6	67	80
1	-7	115	108
2	-7	213	200
3	-7	211	222
4	-7	261	256
5	-7	253	258
6	-7	157	170
7	-7	166	106
9	-7	70	76
10	-7	59	58
11	-7	40	48
1	-8	107	94
2	-8	68	70
3	-8	116	96
4	-8	279	282
5	-8	146	150
6	-8	144	136
7	-8	196	218
8	-8	136	146
9	-8	51	54
1	-9	156	148
2	-10	80	72
3	-9	125	118
4	-9	99	98
5	-9	65	56
6	-9	104	88
8	-9	81	82
9	-9	86	92
10	-9	65	74
11	-9	58	74
4	-10	100	106
5	-10	138	140
6	-10	126	144
8	-10	49	40
3	-11	48	42
6	-11	62	74
7	-11	101	114
8	-11	65	82
10	-11	36	54
1	-12	56	58
2	-12	81	78
3	-12	78	80
4	-12	58	56
5	-12	56	56
8	-12	53	50
9	-12	62	76
4	-13	59	72
	L = 1		
1	0	667	898
3	0	115	140
4	0	59	34
5	0	194	204
6	0	49	54
7	0	96	82
8	0	145	140
9	0	72	72
10	0	30	34
-1	0	293	324
-2	0	328	378
-3	0	134	136
-4	0	154	160
-6	0	134	138
-7	0	194	194
-8	0	81	72
-10	0	49	46
0	1	173	140
2	1	74	84
3	1	40	68
4	1	245	240
5	1	378	376
6	1	328	336
7	1	142	136
8	1	56	56
-1	1	83	40
-2	1	484	518
-3	1	252	238
-4	1	222	244
-5	1	244	238
-6	1	344	318
-7	1	260	264
1	2	172	196
2	2	122	148
3	2	85	74
4	2	237	238
5	2	142	120
9	2	42	48
-1	2	834	1110
-2	2	710	806
-3	2	69	34
-4	2	125	136
-5	2	171	186
-6	2	210	192
-7	2	181	172
-8	2	80	64
-9	2	64	60
0	3	341	346
1	3	149	146
2	3	181	152
3	3	153	128
4	3	120	114
5	3	126	86
7	3	81	68
8	3	81	64
-2	3	108	84
-3	3	313	330
-4	3	256	258
-5	3	93	84
-6	3	60	58
-9	3	65	72
-10	3	127	116
-11	3	59	70
1	4	45	40
2	4	74	74
3	4	121	112
4	4	148	128
5	4	132	126
6	4	125	112
7	4	90	60
8	4	39	26
-1	4	66	58
-2	4	37	4
-3	4	200	178
-4	4	438	562
-5	4	503	508
-6	4	252	256
-7	4	151	152
-10	4	69	68
-11	4	64	70
0	5	318	290
1	5	355	328
2	5	148	128
3	5	108	102
4	5	98	92
5	5	72	52
-1	5	238	214
-2	5	243	208
-6	5	172	200
-7	5	277	296
-8	5	123	136
-9	5	108	124
-10	5	78	74
0	6	190	176
1	6	77	50
4	6	67	56
5	6	71	46
6	6	75	64
-1	6	315	258
-2	6	337	368
-3	6	194	170
-5	6	135	130
-6	6	105	108
-7	6	67	74
-8	6	110	110
-9	6	90	88
-10	6	111	114
0	7	113	90
1	7	75	66
2	7	113	94
3	7	120	96
4	7	126	100
5	7	73	62
-1	7	131	114
-2	7	130	112
-3	7	146	144
-4	7	107	100
-5	7	155	142
-6	7	168	172
-7	7	112	130
0	8	98	96
1	8	194	178
2	8	112	78
3	8	70	54
4	8	44	28
-3	8	63	48
-6	8	60	48
-7	8	79	86
-8	8	85	94
-9	8	75	92
0	9	113	100
1	9	110	94
2	9	57	50
-2	9	61	58
-3	9	94	96
-4	9	89	92
-5	9	76	76
-6	9	61	42
-10	9	75	98
-11	9	70	68
2	10	27	36
-1	10	59	56
-3	10	81	72
-4	10	89	86
-5	10	176	176
-6	10	109	116
-7	10	58	52
-8	10	77	80
0	11	66	60
1	11	47	40
-2	11	56	52
-10	11	38	62
-2	12	49	52
-6	12	50	52
-9	12	39	50
1	-1	379	384
2	-1	474	576
3	-1	248	282
4	-1	73	94
6	-1	117	84
7	-1	48	42
8	-1	74	74
9	-1	129	116
10	-1	76	70
-1	-1	288	420
-2	-1	96	68
-3	-1	140	134
-5	-1	378	348
-6	-1	203	166
-8	-1	59	38
-9	-1	73	52
0	-2	386	432
1	-2	371	380
2	-2	846	1108
3	-2	465	560
4	-2	512	566
5	-2	207	228
6	-2	42	38
7	-2	102	104
8	-2	71	70
9	-2	47	32
-1	-2	119	126
-2	-2	362	406
-3	-2	427	472
-4	-2	289	292
-5	-2	148	144
-6	-2	119	118
-7	-2	80	60
0	-3	342	352
1	-3	338	372
2	-3	545	572
3	-3	119	130
4	-3	192	228
5	-3	55	46
6	-3	89	78
7	-3	75	94
8	-3	118	124
9	-3	154	128
10	-3	97	68
11	-3	54	64
-1	-3	509	572
-2	-3	693	752
-3	-3	214	224
-7	-3	76	54
-8	-3	80	62
-9	-3	60	58
0	-4	293	292
1	-4	415	420
2	-4	141	188
3	-4	125	134
4	-4	400	414
5	-4	152	150
6	-4	128	130
8	-4	70	58
9	-4	75	64
10	-4	64	74
11	-4	59	84
-1	-4	260	240
-3	-4	82	72
-4	-4	68	62
-5	-4	76	56
-6	-4	125	106
-7	-4	143	108
-8	-4	66	60
0	-5	100	60
1	-5	144	134
2	-5	90	76
3	-5	97	90
4	-5	418	446
5	-5	300	318
6	-5	227	238
7	-5	181	202
9	-5	49	58
10	-5	73	60
-1	-5	96	108
-2	-5	187	172
-3	-5	123	116
-4	-5	96	82
-5	-5	112	78
-6	-5	112	68
0	-6	288	290
1	-6	394	392
2	-6	126	128
3	-6	88	92
4	-6	42	2
5	-6	177	172
6	-6	136	136
7	-6	169	170
8	-6	118	156
9	-6	80	94
10	-6	48	46
11	-6	37	46
-1	-6	173	174
-2	-6	141	134
-3	-6	78	60
0	-7	53	54
1	-7	222	210
2	-7	344	344
3	-7	303	306
4	-7	253	278
5	-7	128	160
9	-7	66	92
10	-7	85	100
-4	-7	71	50
-5	-7	72	52
-6	-7	44	42
0	-8	172	154
1	-8	58	60
2	-8	76	76
3	-8	162	160
4	-8	291	302
5	-8	228	236
6	-8	138	134
7	-8	165	170
8	-8	106	108
9	-8	51	54
-1	-8	151	132
-2	-8	115	98
-3	-8	125	100
-4	-8	81	66
-5	-8	32	30
1	-9	125	104
2	-9	188	156
3	-9	66	74
4	-9	63	66
6	-9	83	92
9	-9	86	98
10	-9	75	84
-1	-9	119	96
-2	-9	103	76
0	-10	75	60
1	-10	81	72
2	-10	96	96
3	-10	132	114
4	-10	149	144
5	-10	108	112
7	-10	48	42
8	-10	90	86
9	-10	46	46
10	-10	37	34
0	-11	43	38
2	-11	105	92
3	-11	49	52
5	-11	78	68
6	-11	54	60
7	-11	105	106
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3	-4	152	164
5	-4	185	210
6	-4	128	144
7	-4	132	150
8	-4	97	106
10	-4	43	48
-1	-4	246	262
-2	-4	171	190
-3	-4	204	210
-7	-4	64	64
-8	-4	40	36
0	-5	175	188
1	-5	170	172
2	-5	386	406
3	-5	322	334
4	-5	245	274
5	-5	285	222
6	-5	121	114
8	-5	88	78
-1	-5	56	52
-2	-5	68	64
-4	-5	67	50
-5	-5	126	104
-6	-5	112	100
-7	-5	54	52
0	-6	144	140
2	-6	56	54
3	-6	116	114
4	-6	116	124
5	-6	59	60
6	-6	64	78
7	-6	153	178

TABLE IV (*Continued*)

H	K	10FO	10FC
8	-6	83	92
-1	-6	175	164
-3	-6	125	126
-4	-6	182	162
-5	-6	89	86
-6	-6	61	90
1	-7	146	160
2	-7	160	160
3	-7	109	104
4	-7	96	110
5	-7	138	132
6	-7	128	136
8	-7	86	100
9	-7	94	100
10	-7	46	50
-2	-7	108	92
-3	-7	114	98
0	-8	164	158
1	-8	109	124
2	-8	134	134
3	-8	155	162
4	-8	149	166
5	-8	86	88
10	-8	43	52
-1	-8	67	70
-4	-8	62	54
-5	-8	31	28
0	-9	52	44
5	-9	100	98
6	-9	112	128
7	-9	159	196
8	-9	97	124
9	-9	43	50
10	-9	34	38
-1	-9	59	58
-2	-9	72	68
-3	-9	69	62
0	-10	87	82
1	-10	95	98
2	-10	73	82
4	-10	52	60
7	-10	63	66
8	-10	92	118
9	-10	42	68
-1	-10	55	50
1	-11	63	72
2	-11	69	60
3	-11	77	72
4	-11	83	98
5	-11	47	52
7	-11	50	60
4	-12	40	56
5	-12	68	76
6	-12	42	52
7	-12	39	52
L = 5			
0	0	137	106
1	0	149	140
2	0	180	182
3	0	135	146
4	0	85	82
5	0	97	128
6	0	75	72
7	0	62	48
8	0	41	42
-1	0	64	72
-2	0	219	174
-3	0	340	350
-4	0	283	298
-5	0	193	200
-6	0	120	130
-7	0	62	82
0	1	233	222
1	1	270	270
2	1	246	266
3	1	161	154
4	1	138	136
5	1	84	74
6	1	44	48
7	1	40	34
-1	1	129	102
-2	1	178	156
-3	1	87	64
-4	1	89	88
-5	1	158	152
-6	1	179	204
-7	1	155	142
-8	1	99	102
-9	1	40	42
-10	1	41	52
0	2	181	154
1	2	46	42
5	2	46	40
6	2	70	66
7	2	53	56
-1	2	230	222
-2	2	325	290
-3	2	313	304
-4	2	168	172
-5	2	163	170
-7	2	101	104
-8	2	51	64
-9	2	42	56
-10	2	45	54
0	3	64	82
1	3	73	62
3	3	84	122
4	3	133	120
5	3	88	78
6	3	48	44
-1	3	162	154
-2	3	190	154
-3	3	263	248
-4	3	250	254
-5	3	111	128
-6	3	79	86
0	4	63	78
1	4	97	98
2	4	156	154
3	4	151	134
4	4	116	102
5	4	44	48
-1	4	123	110
-2	4	107	110
-3	4	44	20
-4	4	96	94
-6	4	110	122
-7	4	108	110
-8	4	132	152
-9	4	133	158
-10	4	53	64
0	5	142	148
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4	5	35	30
5	5	31	40
-1	5	98	84
-2	5	182	188
-3	5	268	278
-4	5	89	88
-5	5	104	106
-8	5	112	108
-10	5	53	74
-11	5	41	68
2	6	61	78
3	6	49	50
4	6	28	22
-1	6	55	66
-3	6	48	36
-5	6	111	118
-6	6	194	204
-7	6	138	134
-8	6	83	88
0	7	79	94
1	7	79	78
3	7	22	16
-1	7	66	54
-2	7	73	72
-6	7	142	146
-7	7	146	156
-8	7	97	112
-9	7	69	90
0	8	67	66
-1	8	97	96
-2	8	113	98
-3	8	128	132
-4	8	100	88
-5	8	45	52
-9	8	34	52
-10	8	25	46
-3	9	74	62
-4	9	65	66
-5	9	66	66
-6	9	54	72
-9	9	35	50
0	-1	279	224
-1	-1	216	188
-2	-1	145	132
3	-1	41	36
4	-1	54	48
7	-1	58	38
8	-1	97	94
9	-1	44	50
-1	-1	308	262
-2	-1	383	356
-3	-1	274	268
-4	-1	181	174
-5	-1	109	90
-6	-1	69	70
-7	-1	65	64
-8	-1	57	56
-9	-1	58	54
0	-2	38	14
1	-2	136	116
2	-2	253	286
3	-2	137	136
4	-2	82	82
5	-2	97	92
7	-2	64	76
9	-2	36	48
-1	-2	153	134
-2	-2	147	124
-3	-2	44	34
-5	-2	94	106
-6	-2	155	152
-7	-2	77	66
-8	-2	92	78
-9	-2	52	52
0	-3	140	120
1	-3	221	188
2	-3	171	144
3	-3	135	142
4	-3	172	198
5	-3	173	190
6	-3	80	94
7	-3	74	92
8	-3	70	66
-1	-3	235	212
-2	-3	153	158
-3	-3	170	190
-4	-3	62	74
-5	-3	82	56
-6	-3	81	62
-7	-3	63	66
-8	-3	41	32
0	-4	363	380
1	-4	267	266
3	-4	114	114
4	-4	131	138
5	-4	112	134
6	-4	179	198
7	-4	101	112
8	-4	95	110
9	-4	49	46
10	-4	23	28
-1	-4	262	264
-2	-4	268	266
-3	-4	67	62
-5	-4	88	74
-6	-4	23	30
0	-5	227	220
1	-5	343	346
2	-5	278	316
3	-5	218	238
4	-5	83	96
5	-5	122	122
6	-5	69	68
8	-5	45	52
-2	-5	126	136
-3	-5	199	192
-4	-5	86	88
-5	-5	69	66
-6	-5	68	56
0	-6	105	94
2	-6	123	130
3	-6	165	156
4	-6	138	146
5	-6	47	64
7	-6	48	62
8	-6	45	58
-1	-6	47	32
-2	-6	66	76
-3	-6	231	200
-4	-6	111	104
-5	-6	78	78
-6	-6	38	36
0	-7	72	78
1	-7	116	120
2	-7	182	176
3	-7	117	112
5	-7	104	110
6	-7	129	134
7	-7	74	70
8	-7	78	80
9	-7	70	76
-1	-7	87	90
-2	-7	150	134
-3	-7	100	92
-4	-7	55	54
0	-8	102	96
1	-8	135	132
2	-8	87	90
3	-8	194	190
4	-8	162	172
5	-8	62	56
0	-9	74	74
5	-9	113	128
6	-9	130	142
7	-9	63	72
-1	-9	43	46
-2	-9	63	48
-3	-9	31	40
0	-10	78	66
1	-10	101	98
2	-10	78	76
6	-10	44	50
7	-10	41	48
8	-10	37	50
9	-10	37	52
2	-11	46	56
3	-11	72	72
4	-11	92	66
5	-11	62	66
6	-11	42	46
9	-11	23	44
4	-12	32	40
5	-12	33	60
6	-12	44	66
7	-12	58	70
L = 6			
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2	0	154	158
3	0	123	134
4	0	142	138
5	0	123	100
6	0	67	94
7	0	42	44
-3	0	111	90
-4	0	192	198
-5	0	211	212
-6	0	135	130
-7	0	94	106
-8	0	63	76
-9	0	32	34
0	1	235	204
1	1	208	216
2	1	139	132
3	1	134	124
4	1	85	78
6	1	68	58
7	1	81	54
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-5	1	97	82
-6	1	93	96
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-8	1	71	84
-9	1	70	74
-10	1	32	40
0	2	89	88
1	2	66	74
3	2	48	44
4	2	72	76
5	2	90	84
6	2	81	76
-1	2	190	176
-2	2	289	286
-3	2	123	92
-4	2	143	138
-5	2	180	192
-6	2	51	72
0	3	64	74
1	3	135	136
2	3	161	148
3	3	140	126
4	3	94	88
5	3	51	58
-2	3	166	150
-3	3	118	114
-6	3	87	88
-7	3	110	126
-8	3	96	98
-9	3	41	54
0	4	58	44
1	4	101	112
2	4	123	116
3	4	70	64
4	4	38	34
-1	4	51	52
-3	4	50	50
-4	4	148	120
-5	4	51	34
-8	4	124	138
-9	4	72	96
-10	4	32	48
0	5	69	72
1	5	45	48
2	5	58	40
3	5	46	50
4	5	34	28
-1	5	51	30
-2	5	156	146
-3	5	169	180
-4	5	190	164
-5	5	158	146
-6	5	47	54
-10	5	31	50
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1	6	69	76
2	6	90	66
3	6	85	86
-4	6	79	58
-5	6	143	150
-6	6	76	90
-7	6	72	78
-8	6	59	64
-9	6	50	60
-10	6	46	60
0	7	74	76
1	7	39	44
2	7	17	24
-1	7	118	114
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-6	7	42	58
-8	7	37	36
-1	8	53	60
-2	8	91	96
-3	8	85	92
-4	9	37	42
-5	9	47	58
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-7	9	41	50
-8	9	32	64
0	-1	98	78
1	-1	145	144
3	-1	46	24
4	-1	66	72
5	-1	79	82
6	-1	110	98
7	-1	83	76
8	-1	54	54
-1	-1	70	52
-2	-1	237	254
-3	-1	239	252
-4	-1	221	236
-5	-1	151	146
-7	-1	44	32
0	-2	182	120
1	-2	138	124
2	-2	214	208
3	-2	159	150
7	-2	64	68
-1	-2	294	262
-2	-2	163	152
-3	-2	109	94
-4	-2	65	66
-6	-2	46	40
-7	-2	92	70
-8	-2	55	54
0	-3	178	150
1	-3	243	202
2	-3	155	144
3	-3	177	162
4	-3	233	244
5	-3	159	156
6	-3	73	84
7	-3	47	48
8	-3	42	56
-1	-3	90	90
-2	-3	89	86
-3	-3	172	168
-4	-3	161	176
-5	-3	119	132
-6	-3	112	112
-7	-3	80	62
0	-4	107	106
1	-4	89	82
2	-4	62	64
3	-4	48	48
4	-4	65	58
6	-4	110	124
7	-4	112	120
8	-4	96	104
9	-4	68	86
-1	-4	241	254
-2	-4	145	154
-3	-4	67	76
-4	-4	173	178
-5	-4	92	100
0	-5	175	180
1	-5	287	288
2	-5	255	282
3	-5	132	140
7	-5	45	46
8	-5	64	70
9	-5	62	66
-2	-5	51	50
-3	-5	55	32
-5	-5	56	42
-7	-5	37	36
0	-6	47	38
1	-6	87	94
2	-6	274	284
3	-6	181	186
4	-6	94	94
5	-6	126	144
6	-6	102	114
7	-6	52	52
8	-6	46	48
-1	-6	52	48
-2	-6	115	104
-3	-6	110	118
-4	-6	90	92
-5	-6	67	70
-6	-6	25	38
0	-7	113	118
1	-7	106	110
2	-7	68	54
4	-7	85	84
5	-7	110	116
6	-7	114	132
7	-7	69	66
8	-7	39	42
9	-7	54	50
-1	-7	213	212
-2	-7	163	166
-3	-7	76	86
-4	-7	56	44
0	-8	127	140
1	-8	92	102
2	-8	76	76
3	-8	65	68
4	-8	88	88
-1	-8	158	146
-2	-8	42	36
2	-9	75	58
3	-9	76	74
4	-9	64	100
5	-9	92	98
6	-9	80	86
7	-9	54	66
-2	-9	47	46
0	-10	39	48
1	-10	93	88
2	-10	62	52
5	-10	57	66
6	-10	56	76
7	-10	36	52
0	-11	24	32
1	-11	80	74
2	-11	65	68
3	-11	49	66
4	-11	44	62
8	-11	23	26
3	-12	47	56
4	-12	56	56
5	-12	49	60
6	-12	34	60

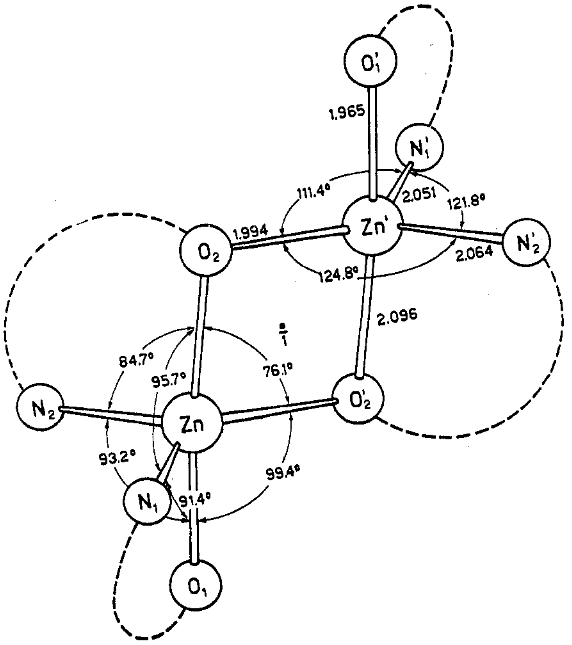

Figure 1.—Schematic view of the structure. The salicylaldimines are represented by dotted lines.

TABLE V

DISTANCES AND ANGLES IN THE COORDINATION GROUP WITH THEIR ESTIMATED STANDARD DEVIATIONS

Zn–O(1)	1.965 A	0.003 A
Zn–O(2)	2.096 A	0.003 A
Zn–O′(2)	1.994 A	0.003 A
Zn–N(1)	2.051 A	0.004 A
Zn–N(2)	2.064 A	0.005 A
Zn–Zn′	3.222 A	0.001 A
O(2)–O′(2)	2.523 A	0.006 A
O(1)–Zn–O(2)	172.5°	0.2°
N(1)–Zn–N(2)	121.8°	0.2°
N(1)–Zn–O′(2)	111.4°	0.2°
N(2)–Zn–O′(2)	124.8°	0.2°
O(1)–Zn–N(1)	91.4°	0.2°
O(1)–Zn–N(2)	93.2°	0.2°
O(1)–Zn–O′(2)	99.4°	0.2°
O(2)–Zn–N(1)	95.7°	0.2°
O(2)–Zn–N(2)	84.7°	0.2°
O(2)–Zn–O′(2)	76.1°	0.2°
Zn–O′(2)–Zn′	103.9°	0.2°

The arrangement of the ligands around the metal can be described as a distorted trigonal bipyramid. The equatorial positions are occupied by one oxygen and two nitrogen atoms; two oxygens are in the polar positions. The Zn atom is 0.166 A out of the equatorial plane. The dimers have a center of symmetry.

Distances and angles in the coordination group with their standard deviations are given in Table V.

It is interesting to notice that the bridging O–Zn intrachelate distance of 2.096 A is more than three standard deviations longer than the corresponding O–Zn interchelate distance of 1.994 A. The nonbridging O–Zn distance is obviously shorter than the other two.

The average Zn–O distance of 2.02 A is in agreement with the analogous distance of 2.02 A found in monoaquobisacetylacetonatozinc[9] and the average Zn–N distance of 2.06 A is similar to the 2.08 A in (N,N′-disalicylideneethylenediaminato)zinc(II) monohydrate.[10]

Bond lengths and angles in the salicylaldimine residues are given in Tables VI and VII, respectively, with their standard deviations. The labeling of the atoms is clear from Figure 2. All distances and angles appear to be normal in the nonbridging salicylaldimine,

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[9] H. Montgomery and E. C. Lingafelter, *Acta Cryst.*, **16**, 748 (1963).

[10] D. Hall and F. H. Moore, *Proc. Chem. Soc.*, 256 (1960).

Table VI

Intramolecular Distances (A) in the Salicylaldimine Groups and Their Estimated Standard Deviations

C(1)–C(2)	1.415	0.008	C(9)–C(10)	1.367	0.009
C(2)–C(3)	1.420	0.008	C(10)–C(11)	1.398	0.008
C(3)–C(4)	1.396	0.008	C(11)–C(12)	1.381	0.012
C(4)–C(5)	1.407	0.011	C(12)–C(13)	1.368	0.012
C(5)–C(6)	1.367	0.011	C(13)–C(14)	1.391	0.011
C(6)–C(1)	1.425	0.007	C(14)–C(9)	1.410	0.011
C(1)–C(7)	1.414	0.009	C(9)–C(15)	1.447	0.008
C(2)–O(1)	1.323	0.006	C(10)–O(2)	1.340	0.008
C(7)–N(1)	1.312	0.006	C(15)–N(2)	1.261	0.008
N(1)–C(8)	1.469	0.009	N(2)–C(16)	1.462	0.008

Table VII

Bond Angles (deg) in the Salicylaldimine Groups and Their Estimated Standard Deviations

C(1)–C(2)–C(3)	117.8	0.5	C(9)–C(10)–C(11)	120.2	0.7
C(2)–C(3)–C(4)	121.3	0.6	C(10)–C(11)–C(12)	120.9	0.7
C(3)–C(4)–C(5)	120.3	0.7	C(11)–C(12)–C(13)	119.7	0.7
C(4)–C(5)–C(6)	119.4	0.6	C(12)–C(13)–C(14)	119.7	0.9
C(5)–C(6)–C(1)	121.6	0.7	C(13)–C(14)–C(9)	121.0	0.8
C(6)–C(1)–C(2)	119.6	0.6	C(14)–C(9)–C(10)	118.4	0.6
O(1)–C(2)–C(3)	118.3	0.6	O(2)–C(10)–C(11)	120.1	0.6
O(1)–C(2)–C(1)	123.9	0.6	O(2)–C(10)–C(9)	119.6	0.5
C(7)–C(1)–C(6)	115.6	0.6	C(15)–C(9)–C(14)	116.7	0.7
C(7)–C(1)–C(2)	124.7	0.5	C(15)–C(9)–C(10)	124.8	0.7
C(1)–C(7)–N(1)	127.2	0.6	C(9)–C(15)–N(2)	127.9	0.7
C(7)–N(1)–C(8)	117.4	0.5	C(15)–N(2)–C(16)	117.4	0.6
C(2)–O(1)–Zn	127.0	0.4	C(10)–O(2)–Zn	123.9	0.3
C(7)–N(1)–Zn	123.3	0.5	C(15)–N(2)–Zn	123.1	0.5
C(8)–N(1)–Zn	119.2	0.3	C(16)–N(2)–Zn	119.0	0.5

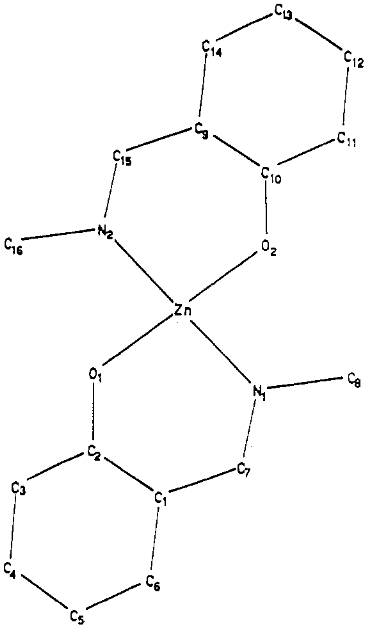

Figure 2.—Bis(N-methylsalicylaldiminato)zinc(II).

compared with the results from other metal salicylaldiminates. On the other hand, the bridging chelate ring shows some anomalies. The N(2)–Zn–O(2) angle of 84.7° is smaller than the usual values ranging between 90 and 94° in most metal salicylaldiminates. Also the C(9)–C(10)–O(2) and C(10)–O(2)–Zn angles are smaller than the corresponding angles in analogous structures. This is certainly due to the particular situation of the bridging oxygen which is involved in all the three angles. Distortion in the bridging chelate ring is even more evident if we consider the deviations of the atoms from the least-squares planes through the salicylaldimine groups. The results are given in Table VIII.

Table VIII

Distances (A) from Least-Squares Planes of Salicylaldimine Residues

C(1)	0.0398	C(9)	−0.0293
C(2)	0.0322	C(10)	−0.0163
C(3)	−0.0028	C(11)	0.0005
C(4)	−0.0351	C(12)	0.0225
C(5)	−0.0304	C(13)	0.0349
C(6)	0.0189	C(14)	−0.0397
C(7)	0.0454	C(15)[a]	−0.1007
C(8)	−0.0284	C(16)	0.0264
N(1)	−0.0395	N(2)[a]	0.1544
O(1)[a]	0.1116	O(2)	0.0010
Zn	−0.2213	Zn[a]	0.9631

[a] Atoms not included in the calculation of the least-squares plane.

The equations of the planes, referred to the triclinic axes, are: $-0.37076x + 0.58935y + 0.69617z - 1.40300 = 0$ for salicylaldimine no. 1 and $-0.74894x + 0.85740y - 0.41093z - 0.04297 = 0$ for salicylaldimine no. 2, with x, y, z in A.

As found in most metal salicylaldiminates, the metal atom is considerably out of the planes of the salicylaldimine groups. In the bridging chelate ring, N(2) and C(15) are clearly out of the plane. The angle between the mean planes through the salicylaldimines is 119° 48′. It may be noted that the angle between the coordination planes, defined by the atoms O(1), Zn, N(1), and, respectively, O(2), Zn, N(2) is 135° 57′.

Atom O(1) in the nonbridging salicylaldimine is about 0.1 A out of the plane. A possible explanation can be found by considering the molecular contacts within a dimer.

Table IX lists all the molecular contacts less than 3.6 A, within and between the dimers. It can be seen that atom O(1) has very short molecular contacts with O(2), C(10), and C(11) in $\bar{x}$, $\bar{y}$, $\bar{z}$. This might cause some repulsion which could slightly shift O(1) out of the plane.

All the other molecular contacts within and between the dimers appear to be normal. The distance between the zinc atom and C(10) in $\bar{x}$, $\bar{y}$, $\bar{z}$ is so short because C(10) is bound to the bridging oxygen. C(10) is by no means in a coordinating position.

A view of the structure down the *c* axis is given in Figure 3. An interesting feature of the structure is the existence of planes passing through both bridging salicylaldimines in a dimer.

These planes leave out the zinc atoms by 1.02 A, the nitrogen atoms by 0.21 A, and the methyl carbons by 0.10 A. The planes pass through the origin and are parallel to the (2$\bar{2}$1) crystallographic plane. This might suggest that crystal packing plays an important role in establishing this rather peculiar structure.

Five-coordination in Zn(II) complexes has been re-

TABLE IX

MOLECULAR CONTACTS LESS THAN 3.6 A

Atom 1 (in x, y, z)	Atom 2	Distance, A	Molecule position
C(1)	C(1)	3.422	$\bar{x}, \bar{y} + 1, \bar{z}$
C(1)	C(7)	3.378	$\bar{x}, \bar{y} + 1, \bar{z}$
C(2)	C(11)	3.534	$\bar{x}, \bar{y}, \bar{z}$
C(4)	C(8)	3.540	$\bar{x}, \bar{y} + 1, \bar{z}$
C(5)	C(8)	3.592	$\bar{x}, \bar{y} + 1, \bar{z}$
C(6)	C(7)	3.501	$\bar{x}, \bar{y} + 1, \bar{z}$
C(8)	C(8)	3.579	$\bar{x} - 1, \bar{y}, \bar{z}$
C(14)	C(7)	3.569	$x, y, z + 1$
C(14)	C(15)	3.438	$\bar{x}, \bar{y}, \bar{z} + 1$
N(1)	C(5)	3.519	$\bar{x}, \bar{y} + 1, \bar{z}$
N(1)	C(6)	3.510	$\bar{x}, \bar{y} + 1, \bar{z}$
N(1)	O(2)	3.342	$\bar{x}, \bar{y}, \bar{z}$
O(1)	C(10)	3.358	$\bar{x}, \bar{y}, \bar{z}$
O(1)	C(11)	3.066	$\bar{x}, \bar{y}, \bar{z}$
O(1)	O(2)	3.020	$\bar{x}, \bar{y}, \bar{z}$
O(2)	N(2)	3.598	$\bar{x}, \bar{y}, \bar{z}$
Zn	C(10)	3.051	$\bar{x}, \bar{y}, \bar{z}$
Zn	C(11)	3.537	$\bar{x}, \bar{y}, \bar{z}$

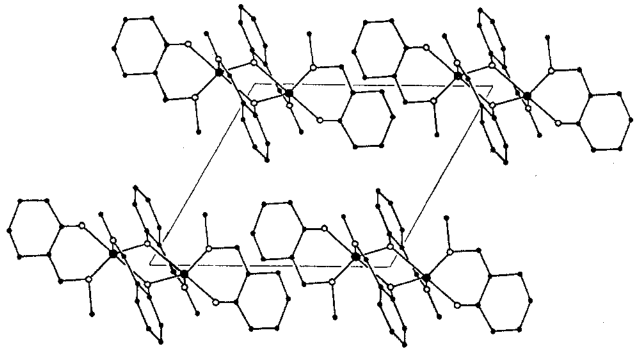

Figure 3.—The crystal structure of bis(N-methylsalicylaldiminato)zinc(II) viewed down the c axis.

ported by other authors.[9–11] On the basis of the isomorphism with the zinc(II) complex, bis(N-methylsalicylaldiminato)cobalt(II) and -manganese(II) very likely contain high-spin five-coordinated cobalt(II) and manganese(II).

It has been reported[12] that bis(N-β-diethylamineethyl-5-chlorosalicylaldimine)cobalt(II) is isomorphous with the nickel(II) analog, which has been shown by three-dimensional X-ray analysis to contain high-spin five-coordinated nickel(II).

More recently[13] Pauling, *et al.*, have reported preliminary X-ray results on the high-spin five-coordinated cobalt(II) complex with diphenylmethylarsine oxide. No structure has been so far described for five-coordinated Mn(II) complexes.

It might have been expected that the dimeric arrangement would afford a good electronic framework for a superexchange process between the metal atoms *via* the bridging oxygens.

Magnetic measurements on the Co(II) and Mn(II) complexes have shown no anomalous behavior in the magnetic properties of these complexes.[4] The same behavior has been found in the trimeric bis(acetylacetonato)nickel(II), where the magnetic susceptibility follows the Curie–Weiss law.[14]

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Structure of Chlorocarbonyl(sulfur dioxide)bis(triphenylphosphine)iridium[1]

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The crystal and molecular structure of chlorocarbonyl(sulfur dioxide)bis(triphenylphosphine)iridium, $IrCl(CO)(SO_2)(P(C_6H_5)_3)_2$, has been determined from three-dimensional X-ray data. The material crystallizes in space group C_{2h}^5-$P2_1/c$ of the monoclinic system, with four molecules in a cell of dimensions $a = 12.11$, $b = 16.79$, $c = 17.09$ A, $\beta = 103.0°$. The individual monomeric units are well separated, the closest Ir–Ir distance being greater than 8 A. The coordination geometry around the Ir is that of a tetragonal pyramid, with CO, Cl, and *trans* P atoms in the base and the S of the SO_2 group at the apex. The O (of the CO), Cl, and two P atoms are coplanar; the Ir lies 0.21 A above this plane toward the S. The basal interatomic distances are normal. However, the Ir–S bond length of 2.49 A is very long and the Ir, S, and two O atoms of the SO_2 group are not coplanar. Rather the Ir–S vector makes an angle of 32° with the normal to the SO_2 plane. The dimensions of the SO_2 group are not significantly different from those found in solid SO_2. The basic geometry of the SO_2 complex differs markedly from that of the analogous O_2 complex, $Ir(O_2)Cl(CO)(P(C_6H_5)_3)_2$, in which the Ir is in a trigonal bipyramidal configuration with *trans* P atoms at the apices.

Introduction

Vaska has found that several molecular species interact *reversibly* in benzene solution with $IrCl(CO)(P(C_6H_5)_3)_2$ to form stable adducts that can be crystallized. Among these species are molecular oxygen[3] and molecular sulfur dioxide.[4] A complete structure determina-

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[3] L. Vaska, *Science*, **140**, 809 (1963).

[4] L. Vaska, unpublished results.