Acta Cryst. (1964). **17**, 1481

The crystal structure of diaquobisacetylacetonatonickel(II).

By H. MONTGOMERY and E. C. LINGAFELTER,
Department of Chemistry, University of Washington, Seattle, Washington 98105, *U.S.A.*

(*Received* 12 *November* 1963 *and in revised form* 11 *May* 1964)

Bullen (1959) has reported the crystal structure of diaquobisacetylacetonatocobalt(II), $Co(C_5H_7O_2)_2.2H_2O$. Cell dimensions and general distribution of intensities in the diffraction patterns indicate that the corresponding nickel compound has essentially the same structure. Because of our interest in the coordination polyhedra of transition metal ions, we have determined the crystal structure of diaquobisacetylacetonatonickel(II).

The unit cell has dimensions

$$a = 10{\cdot}954 \pm 0{\cdot}01,\quad b = 5{\cdot}361 \pm 0{\cdot}006,\quad c = 11{\cdot}245 \pm 0{\cdot}01\ \text{Å};\quad \beta = 106°\ 48' \pm 9',$$

and contains two molecules. The space group is $P2_1/c$. The structure was refined finally to $R = 0{\cdot}07$ by three-dimensional least squares on the IBM 709 computer, using 937 independent reflections from integrated equi-inclination Weissenberg photographs, *h0l* through *h3l*, taken with Cu $K\alpha$ radiation.

The final atomic positions and thermal parameters are given in Tables 1 and 2 and the bond lengths and angles, with their estimated standard deviations, in Table 3. The numbering system used by Bullen (1959) has been followed. The final values of observed and calculated structure factors are given in Table 4.

Table 1. *Final atomic parameters*

		x/a	*y/b*	*z/c*
	Ni	0·0000	0·0000	0·0000
	O(1)	0·1445	0·2147	0·1013
	O(2)	0·1137	−0·1741	−0·0861
	O(3)	0·0453	−0·2849	0·1390
	C(1)	0·3564	0·3371	0·2024
	C(2)	0·2613	0·1696	0·1122
	C(3)	0·3081	−0·0154	0·0485
	C(4)	0·2346	−0·1755	−0·0458
	C(5)	0·3024	−0·3645	−0·1019
C(1)	H(1)	0·3440	0·5077	0·1656
C(1)	H(2)	0·3396	0·3372	0·2851
C(1)	H(3)	0·4453	0·2784	0·2122
C(5)	H(4)	0·2826	−0·3234	−0·1844
C(5)	H(5)	0·2718	−0·5364	−0·0915
C(5)	H(6)	0·3965	−0·3549	−0·0623
C(3)	H(7)	0·4009	−0·0217	0·0788
O_3	H(8)	0·0876	−0·4385	0·1449
O_3	H(9)	0·0152	−0·2465	0·2128

Table 2. *Anisotropic temperature factors*

Each term is multiplied by 10^2

	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Ni	3·17	7·86	2·65	0·04	0·69	−0·37
O(1)	2·36	5·63	2·33	−0·12	0·34	−1·10
O(2)	2·94	6·29	1·77	0·11	0·26	−1·13
O(3)	6·50	5·62	2·15	0·64	1·00	0·05
C(1)	3·27	5·73	3·00	−1·38	0·36	−0·13
C(2)	2·39	5·81	1·53	0·00	0·28	0·46
C(3)	2·16	5·69	2·71	0·73	0·23	0·18
C(4)	2·78	5·11	1·69	0·28	0·87	0·25
C(5)	3·67	6·60	3·18	0·69	1·47	−0·24
H	3·17	3·17	3·17	0·00	0·92	0·00

The structure is essentially the same as that reported by Bullen (1959) for the cobalt compound, the only significant difference being in the coordination polyhedron. In the cobalt compound, the cobalt ion is surrounded by a tetragonally distorted octahedron, with acetylacetone oxygen atoms at 2·05 and 2·06 Å and water oxygen atoms at 2·23 Å. In the nickel compound, the nickel ion has acetylacetone oxygen atoms at 2·01 and 2·02 Å and water oxygen atoms at 2·14 Å. This tetragonal distortion is not observed in diaquobis-(salicylaldehydato)nickel (Stewart, Lingafelter & Breazeale, 1961), in which the salicylaldehyde oxygen atoms are at 2·02 Å and the water oxygen atoms are at 2·04 Å, nor in the hexakisaquo metal ions in the Tutton salts (Montgomery & Lingafelter, 1964), which show Co–O = 2·08, 2·09, 2·11 Å and Ni–O = 2·04, 2·08, 2·09 Å.

Table 3. *Bond lengths and angles*

Estimated standard deviation in parentheses

Bond lengths (Å)			Bond angles (°)		
Ni–O(1)	2·021	(0·010)	O(1)–Ni–O(2)	92·4	(0·4)
Ni–O(2)	2·014		O(3)–Ni–O(1)	90·9	
Ni–O(3)	2·139		O(3)–Ni–O(2)	88·6	
O(1)–C(2)	1·273	(0·022)	Ni–O(1)–C(2)	123·2	(0·7)
O(2)–C(4)	1·270		Ni–O(2)–C(4)	123·9	
C(2)–C(3)	1·404	(0·027)	O(1)–C(2)–C(3)	126·2	(0·9)
C(3)–C(4)	1·420		O(2)–C(4)–C(3)	124·8	
C(1)–C(2)	1·499		O(1)–C(2)–C(1)	115·3	(1·0)
C(4)–C(5)	1·520		O(2)–C(4)–C(5)	116·5	
			C(2)–C(3)–C(4)	126·6	(1·1)

The acetylacetone residue is planar, with a mean deviation of the seven atoms from the least-squares plane of 0·04 Å ($\sim 1{\cdot}5\sigma$). As noted for other acetylacetonates, the nickel atom does not lie in the plane of the acetylacetone residue, but 0·32 Å from it. Corresponding displacements in other compounds are 0·36 Å in diaquobisacetylacetonatocobalt(II) (Bullen, 1959), 0·40 and 0·25 Å in monoaquobisacetylacetonatozinc (Montgomery & Lingafelter, 1963), and 0·17 Å in bisacetylacetonatocopper(II) (Dahl, 1963).

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

References

BULLEN, G. J. (1959). *Acta Cryst.* **12**, 703.
DAHL, L. F. (1963). Private communication.
MONTGOMERY, H. & LINGAFELTER, E. C. (1963). *Acta Cryst.* **16**, 748.
MONTGOMERY, H. & LINGAFELTER, E. C. (1964). *Acta Cryst.* **17**, 1478.
STEWART, J. M., LINGAFELTER, E. C. & BREAZEALE, J. D. (1961). *Acta Cryst.* **14**, 888.

Table 4. *Calculated and observed structure factors* (× 10)

H,0,0		
1	547E	884
2	154	-58
3	651	773
4	234	261
5	75	7
6	280	304
7	401	400
8	331	331
9	215	212
10	128	82
11	33•	38
12	89	58
13	106	102
H,0,2		
0	713E	854
1	403	395
2	47	69
3	95	110
4	338	351
5	128	154
6	82	93
7	216	220
8	52	56
9	75	75
10	220	235
11	108	123
12	98	97
H,0,-2		
1	683E	851
2	160	175
3	419	430
4	509	548
5	197	208
6	344	372
7	568	558
8	391	425
9	382	393
10	144	162
11	72	-76
12	78	81
13	110	124
H,0,4		
0	297	-211
1	151	190
2	390	412
3	515	518
4	403	408
5	414	454
6	325	353
7	35•	-38
8	36•	-9
9	230	243
10	159	177
11	118	122
H,0,-4		
1	285	-197
2	364	364
3	257	274
4	321	339
5	462	466
6	190	204
7	175	204
8	361	338
9	203	213
10	140	164
11	121	139
12	57	79
13	106	116
H,0,6		
0	78	-14
1	319	312
2	727	722
3	374	371
4	257	266
5	501	518
6	235	238
7	52	-55
8	113	114
9	185	181
10	80	74
H,0,-6		
1	139	153
2	182	193
3	553	520
4	627	589
5	282	251
6	180	161
7	159	152
8	33•	24
9	37	-38
10	105	94
11	234	260
12	154	151
13	36	41
H,0,8		
0	245	242
1	365	346
2	268	253
3	76	91
4	91	87
5	132	140
6	154	163
7	107	112
8	121	105
H,0,-8		
1	355	344
2	351	344
3	381	359
4	577	557
5	275	263
6	95	111
7	362	341
8	144	137
9	134	-123
10	123	112
11	218	250
12	90	111
13	36	52
H,0,10		
0	261	264
1	65	78
2	35•	43
3	33•	38
4	46	-47
5	28•	38
6	147	151
H,0,-10		
1	253	257
2	204	204
3	189	184
4	83	83
5	163	112
6	189	187
7	230	225
8	240	248
9	132	142
10	88	89
11	126	160
H,C,12		
C	30•	42
1	119	125
2	167	151
3	39	49
H,0,-12		
1	189	203
2	231	225
3	32•	-7
4	96	-91
5	31•	33
6	139	121
7	83	79
8	108	108
9	179	207
10	127	122
H,0,-14		
2	141	226
3	80	77
4	30	-39
5	163	147
6	143	143
H,1,0		
1	73	67
2	249	237
3	138	113
4	322	290
5	333	306
6	79	58
7	34•	6
8	74	-68
9	125	-122
10	87	97
11	89	111
12	30•	-23
13	21•	22
H,1,1		
0	607E	744
1	429	457
2	284	311
3	472	497
4	330	343
5	181	185
6	308	329
7	265	258
8	160	154
9	238	239
10	161	154
11	63	65
12	114	120
H,1,-1		
1	516	568
2	185	201
3	105	131
4	338	358
5	268	295
6	133	151
7	324	322
8	310	334
9	178	174
10	149	142
11	105	91
12	67	69
13	102	101
H,1,2		
0	598	-633
1	330	310
2	389	364
3	258	245
4	262	244
5	182	160
6	34•	27
7	228	-207
8	268	-260
9	39•	-33
10	39	59
11	32•	20
12	25•	10
H,1,-2		
1	213	195
2	702	-708
3	315	-287
4	263	-223
5	287	-254
6	30•	15
7	116	98
8	93	76
9	94	71
10	39•	1
11	38•	5
12	33•	12
13	26•	6
H,1,3		
0	614	665
1	86	125
2	558	573
3	608	624
4	200	208
5	270	273
6	318	333
7	106	92
8	59	65
9	186	190
10	152	147
11	87	73
12	93	88
H,1,-3		
1	584	627
2	54	8
3	418	422
4	514	514
5	202	211
6	228	246
7	315	318
8	247	242
9	152	158
1C	138	140
11	100	98
12	43	58
13	86	95
H,1,4		
C	419	-413
1	266	-242
2	137	109
3	56	-57
4	200	-189
5	51	41
6	38•	-3
7	159	-147
8	39•	-10
9	37•	36
10	32•	-12
H,1,-4		
1	411	376
2	23•	-33
3	480	-453
4	313	-290
5	195	-177
6	217	-187
7	34•	-17
8	149	133
9	147	133
10	39•	-29
11	164	-169
12	34•	-39
13	27•	33
H,1,5		
0	204	219
1	248	256
2	382	386
3	294	309
4	244	242
5	240	248
6	223	224
7	92	85
8	38•	36
9	152	150
1C	117	121
H,1,-5		
1	321	333
2	202	243
3	434	454
4	550	565
5	249	261
6	249	258
7	342	346
8	136	132
9	77	75
10	189	201
11	130	124
12	49	61
13	134	118
H,1,6		
0	92	-22
1	33	-24
2	61	-62
3	165	-117
4	73	-71
5	70	-63
6	71	64
7	176	186
8	89	88
9	30•	16
H,1,-6		
1	29•	14
2	203	189
3	113	97
4	31•	0
5	198	178
6	35•	10
7	127	-132
8	114	97
9	137	120
10	87	-77
11	96	-96
12	32•	-44
13	25•	-16
H,1,7		
0	179	182
1	242	247
2	201	202
3	153	155
4	159	160
5	222	229
6	180	178
7	78	72
8	119	123
9	133	120
H,1,-7		
1	126	125
2	273	280
3	353	359
4	277	287
5	243	243
6	209	212
7	206	210
8	159	166
9	65	69
10	120	123
11	179	187
12	99	101
13	72	66
H,1,8		
0	298	295
1	68	47
2	53	-52
3	83	77
4	59	-50
5	102	-106
6	106	102
7	135	145
8	26	32
H,1,-8		
1	230	-220
2	95	-90
3	106	109
4	247	249
5	129	132
6	57	53
7	39•	22
8	39•	-31
9	39•	-11
10	55	1
11	34•	27
12	37	54
H,1,9		
0	142	148
1	202	204
2	211	212
3	70	57
4	104	103
5	187	189
6	91	97
7	109	103
H,1,-9		
1	246	259
2	238	249
3	155	157
4	180	178
5	158	156
6	96	106
7	163	145
8	179	173
9	105	105
1C	64	67
11	145	143
12	126	124
H,1,10		
0	39•	42
1	39•	-34
2	39	54
3	36•	13
4	33•	-37
5	29•	-39
H,1,-10		
1	207	-211
2	155	-136
3	39•	8
4	92	80
5	39•	-16
6	117	-100
7	39•	16
8	38•	-18
9	157	-157
1C	32•	-33
11	105	101
12	57	59
H,1,11		
0	104	108
1	156	153
2	159	155
3	72	80
4	63	58
5	82	76
H,1,-11		
1	215	225
2	242	250
3	39•	60
4	99	96
5	179	184
6	71	69
7	97	100
8	187	187
9	92	98
10	57	60
11	108	106
H,1,12		
C	144	-138
1	30•	-26
2	27•	9
H,1,-12		
1	62	73
2	37	-31
3	74	63
4	35•	9
5	116	-116
6	36	-45
7	32•	12
8	30•	-16
H,1,13		
C	59	61
1	81	76
H,1,-13		
1	111	112
2	118	128
3	85	79
4	54	54
5	110	116
6	120	135
7	71	82
8	98	89
H,1,-14		
2	96	112
4	57	-51
H,2,0		
1	67	-21
2	235E	264
3	429	460
4	364	363
5	241	232
6	266	270
7	170	175
8	35•	22
9	152	144
10	221	229
11	109	109
12	79	85
H,2,1		
0	86	-79
1	281	-281
2	32	36
3	239	234
4	124	-103
5	27•	-10
6	131	120
7	151	-136
8	179	-172
9	36•	12
1C	62	66
11	31	41
12	22•	-2
H,2,-1		
1	262	251
2	150	138
3	133	-134
4	29	-20
5	52	-44
6	81	-78
7	49	-45
8	73	-61
9	36•	5
10	38	27
11	32•	-2
12	27•	20
H,2,2		
0	364	375
1	268	280
2	460	498
3	512	561
4	255	271
5	243	243
6	290	314
7	198	206
8	164	179
9	197	203
10	96	91
11	28	35
12	59	54
H,2,-2		
1	143	160
2	189	193
3	388	429
4	304	328
5	191	201
6	223	224
7	147	142
8	45	-33
9	36•	-3
10	195	191
11	177	174
12	70	68
13	112	103
H,2,3		
0	321	-334
1	55	-39
2	146	131
3	27	28
4	116	108
5	148	127
6	62	40
7	36•	13
8	76	-84
9	34•	-36
10	84	86
11	24•	-9
H,2,-3		
1	327	324
2	55	42
3	261	-242
4	50	-40
5	122	113
6	136	-118
7	41	-32
8	228	222
9	56	55
10	80	-77
11	33•	6
12	28•	-20
13	20•	-36
H,2,4		
0	570	584
1	343	354
2	311	341
3	216	206
4	80	82
5	93	86
6	175	171
7	205	212
8	174	175
9	109	104
10	27•	22
H,2,-4		
1	613	652
2	245	255
3	309	301
4	350	350
5	201	211
6	254	266
7	278	280
8	122	107
9	72	60
10	140	124
11	110	103
12	60	59
13	76	73
H,2,5		
0	27•	19
1	182	175
2	75	-61
3	31•	-27
4	75	54
5	35•	2
6	54	48
7	65	63
8	33•	14
9	29•	-2
10	22•	-34
H,2,-5		
1	178	160
2	234	-233
3	228	-212
4	71	64
5	29•	-2
6	49	-36
7	34•	29
8	118	103
9	110	101
10	36•	-32
11	73	-67
12	27•	-13
13	20•	-4
H,2,6		
0	311	334
1	104	111
2	82	79
3	120	108
4	35•	22
5	54	55
6	168	168
7	156	141
8	110	110
9	81	91
H,2,-6		
1	448	491
2	255	277
3	35	48
4	173	185
5	215	227
6	171	174
7	258	270
8	277	300
9	190	191
10	139	138
11	75	67
12	27•	21
13	66	70
H,2,7		
0	108	100
1	34•	-36
2	35•	-20
3	84	-78
4	110	-107
5	36•	-28
6	34•	-9
7	46	63
8	76	82
9	45	-46
H,2,-7		
1	170	-174
2	94	-83
3	34	-26
4	49	40
5	35	23
6	35•	-24
7	36•	-7
8	38	-48
9	38	-40
10	34•	20
11	39	-47
12	25•	-34
H,2,8		
0	113	103
1	65	48
2	181	183
3	166	161
4	137	129
5	184	188
6	106	111
7	54	44
8	96	89
H,2,-8		
1	243	244
2	161	149
3	34•	35
4	89	84
5	148	142
6	96	80
7	93	90
8	207	211
9	197	206
10	115	110
11	79	74
12	23•	29
H,2,9		
0	38	-34
1	66	-59
2	36•	24
3	52	17
4	49	-52
5	77	-78
6	25•	23
7	60	58
H,2,-9		
1	178	-167
2	38	-36
3	192	199
4	38	42
5	36•	-14
6	54	64
7	46	-40
8	80	-79
9	42	-44
10	55	-50
11	26•	4
12	49	45
H,2,10		
0	57	55
1	186	184
2	195	207
3	137	147
4	167	190
5	153	156
H,2,-10		
1	139	115
2	174	158
3	120	109
4	185	193
5	188	180
6	74	56
7	67	46
8	84	74
9	74	68
10	74	68
11	74	64
H,2,11		
0	32•	12
1	56	-46
2	65	67
3	125	122
H,2,-11		
1	34•	-21
2	36	23
3	44	47
4	50	52
5	34•	-20
6	42	-40
7	33	21
8	29•	17
9	27•	-25
1C	36	-37
H,2,12		
C	94	97
1	133	131
2	89	82
H,2,-12		
1	30	33
2	92	90
3	142	157
4	176	193
5	184	203
6	112	111
7	56	70
8	84	89
9	30	27
H,2,-13		
4	38	-57
5	23•	-28
6	69	-68
H,3,0		
1	80	84
2	17	-14
3	57	-41
4	65	-66
5	113	-116
6	118	-121
7	20•	-10
8	46	47
9	30	-4
10	18•	-8
11	15•	8
H,3,1		
C	131	133
1	82	67
2	303	308
3	315	311
4	164	153
5	176	184
6	183	182
7	116	113
8	38	29
9	49	42
1C	109	109
11	26	38
H,3,-1		
1	133	128
2	321	292
3	550	569
4	282	277
5	155	152
6	340	350
7	129	129
8	100	-108
9	81	76
1C	162	169
11	71	79
12	60	53
H,3,2		
C	35	9
1	29	25
2	74	-70
3	122	-129
4	59	-55
6	20•	2
7	69	78
8	68	77
9	19•	9
1C	17•	-11
H,3,-2		
1	65	54
2	98	82
3	159	150
4	120	127
5	17•	-16
6	48	-43
7	20•	-23
8	33	-34
9	22	-18
10	20•	19
11	17•	5
12	13•	-19
H,3,3		
0	162	157
1	209	194
2	69	50
3	88	74
4	136	118
5	20•	43
5	62	43
6	148	139
7	205	208
8	134	137
9	105	105
10	33	29
H,3,-3		
1	201	188
2	284	280
3	216	202
4	194	188
5	155	167
6	161	166
7	165	182
8	54	44
9	48	43
10	176	184
11	113	124
12	62	54
H,3,4		
0	120	106
1	34	26
2	17•	-8
3	18•	14
4	20•	24
5	62	64
6	78	82
7	21	35
8	19•	1
9	16•	-5
1C	12	-21
H,3,-4		
1	142	-136
2	38	-21
3	74	63
4	86	80
5	91	101
6	66	59
7	20•	-1
8	22	-32
9	20•	-20
10	20•	12
11	17•	29
12	14•	16
H,3,5		
0	361	347
1	90	71
2	143	135
3	176	170
4	34	28
5	97	87
6	143	149
7	140	145
8	179	193
9	82	79
H,3,-5		
1	411	396
2	125	115
3	17•	-3
4	72	63
5	102	96
6	58	45
7	105	94
8	189	196
9	105	112
1C	66	62
11	94	108
12	27	31
H,3,6		
0	83	61
1	20	-27
2	51	-5
3	30	38
4	26	30
5	20•	12
6	20•	-1
7	32	-35
8	56	-55
9	23	-18
H,3,-6		
1	96	-89
2	82	-70
3	119	-98
4	69	-74
5	20	29
6	64	43
7	35	-25
8	20•	-19
9	26	-9
10	34	-34
11	16•	9
12	35	50
H,3,7		
0	182	177
1	88	93
2	236	232
3	161	166
4	86	85
5	78	76
6	67	64
7	84	71
8	60	57
H,3,-7		
1	446	432
2	144	144
3	48	-53
4	178	179
5	142	142
6	48	39
7	133	125
8	150	158
9	126	138
1C	85	84
11	15•	4
H,3,8		
0	31	-37
1	35	-32
2	20•	-21
3	20•	30
4	19•	20
5	17•	-30
6	15•	-28
7	23	-27
H,3,-8		
1	76	72
2	45	39
3	69	-78
4	78	-79
5	20•	-26
6	38	-37
7	20•	-24
8	20•	0
9	18•	-17
10	42	-41
11	13•	-25
H,3,9		
C	84	68
1	82	82
2	114	119
3	138	151
4	75	85
5	56	60
6	51	41
H,3,-9		
1	104	88
2	122	119
3	78	71
4	134	141
5	202	224
6	116	119
7	82	79
8	103	118
9	77	92
10	82	86
11	33	35
H,3,10		
0	19•	-23
1	20	17
2	31	32
3	23	28
4	13•	11
H,3,-10		
1	64	72
2	69	66
3	20•	26
4	20•	-8
5	20•	-17
6	19•	-17
7	18•	2
8	17•	26
9	14•	19
10	11•	3
H,3,11		
0	36	29
1	80	79
2	68	71
3	37	33
H,3,-11		
1	17•	-26
2	17•	-4
3	101	116
4	110	129
5	97	104
6	97	117
7	32	47
8	25	25
9	57	61
H,3,12		
0	18	25
1	42	41
H,3,-12		
1	27	-34
2	21	-21
3	14•	16

* Signifies unobserved reflections, *E* reflections treated as extinctions