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## Crystal structure of kambaldaite, $\text{Na}_2\text{Ni}_8(\text{CO}_3)_6(\text{OH})_6 \cdot 6\text{H}_2\text{O}$

LUTZ M. ENGELHARDT, SYDNEY R. HALL AND ALLAN H. WHITE

Department of Physical and Inorganic Chemistry and Crystallography Centre  
University of Western Australia, Nedlands, W.A. 6009.

### Abstract

A new carbonate mineral, kambaldaite, with the ideal formula  $\text{Na}_2\text{Ni}_8(\text{CO}_3)_6(\text{OH})_6 \cdot 6\text{H}_2\text{O}$ , is hexagonal,  $P6_3$ ,  $a = 10.340(3)$ ,  $c = 6.097(2)\text{\AA}$ ;  $Z = 1$ . The structure has been determined from single crystal X-ray diffractometer data, and refined by full matrix least squares to a residual of 0.048 for 1157 independent "observed" reflections. Distorted  $\text{NiO}_6$  "octahedra" are linked in a three-dimensional edifice with bridging carbonate and hydroxyl groups; Ni-O range between 2.022(5)–2.191(5)\AA with O-Ni-O angles as small as 80.5(2)°. Columnar tunnels through the structure with a free pore diameter of ~5\AA about the principal cell axis are occupied by arrays of sodium atoms with octahedral environments of bridging water molecules; Na-O are 2.312(8) and 2.321(8)\AA with O-Na-O angles as small as 81.2(4)°.

### Introduction

The description of kambaldaite, a new hydrated sodium/nickel basic carbonate mineral from Kambalda in Western Australia, has been presented in the preceding paper by Nickel and Robinson (1985). The present paper reports the determination of its crystal structure by single crystal X-ray diffraction methods.

### Structure determination

#### Crystal data

$\text{Na}_2\text{Ni}_8(\text{CO}_3)_6(\text{OH})_6 \cdot 6\text{H}_2\text{O} \equiv \text{C}_6\text{H}_{18}\text{Na}_2\text{Ni}_8\text{O}_{30}$ ,  $M = 1085.7$ . Hexagonal, space group  $P6_3$  ( $C_6^6$ , No. 173),  $a = 10.340(3)$ ,  $c = 6.097(2)\text{\AA}$ ,  $U = 564.6(3)\text{\AA}^3$ ,  $D_c$  ( $Z = 1$ ) =  $3.19 \text{ g cm}^{-3}$ .  $F(000) = 540$ ,  $\mu_{\text{Mo}}$  =  $66.6 \text{ cm}^{-1}$ .

#### Experimental details

A hexagonal prism ~0.1 mm thick and ~0.1 mm in length was mounted on a Syntex  $P2_1$  four-circle diffractometer after examination by Weissenberg photographic methods. Cell parameters were obtained from 12 axial reflections with  $2\theta \sim 40^\circ$ . A unique data set was measured at 295 K within the limit  $2\theta_{\text{max}} = 100^\circ$  using a conventional  $2\theta/\theta$  scan mode, with a monochromatic  $\text{MoK}\alpha$  radiation source ( $\lambda = 0.71069\text{\AA}$ ). 2265 independent reflections were measured; of these 1157 with  $I > 3\sigma(I)$  were used in the structure determination after the application of analytical absorption correction. The structure was solved by the heavy atom method, and refined by full matrix least squares using anisotropic thermal parameters for the non-hydrogen atoms. Hydrogen atoms were located in difference maps and refined in  $(x, y, z)$  with  $U(\text{isotropic})$  constrained at estimated values. At convergence, residuals  $R, R'$  were 0.048, 0.051, reflection weights being  $(\sigma^2(F_o) + 0.0005(F_o)^2)^{-1}$ ; for the alternative chirality  $R$  was 0.048.  $\text{Na}^+$  excepted, neutral complex scattering factors were used (Ibers and Hamilton, 1974). Computation used

the X-RAY program system (Stewart, 1976) implemented on a Perkin-Elmer 3240 computer. Observed and calculated structure factor amplitudes are given in Table 1;<sup>1</sup> atomic coordinates and thermal parameters are presented in Table 2. A projection of the structure down the  $c$  axis is shown in Figure 1, while a projection of the infinite  $\text{Na}_2(\text{H}_2\text{O})_6$  column along the non-unique axis is given in Figure 2.

### Description of the structure

The structure determination suggests the  $\text{Na}_2\text{Ni}_8(\text{CO}_3)_6(\text{OH})_6 \cdot 6\text{H}_2\text{O}$  stoichiometry for kambaldaite. The deviation of the stoichiometry from this ideal composition suggested by the analysis reported in the previous paper (Nickel and Robinson, 1985) was explored during refinement by allowing the population of the most deviant element, sodium, to refine as an unconstrained variable. Since the population did not deviate significantly from one, it was restored and constrained at that value. Note, however, that the "impurity" element present in the greatest proportion is magnesium, and since this is isoelectronic with sodium, its presence replacing Na might be expected to have little effect on the refinement. In this case some disorder may be introduced into the structure which would probably be only reflected as higher than normal thermal parameters in consequence of different metal-oxygen distances. Some "streaking" of the reflections was evident in the profiles observed both in Weissenberg photographs and diffractometer data, and we note that the highest non-hydrogen atom thermal param-

<sup>1</sup> To receive a copy of Table 1, order Document Am-85-257 from the Business Office, Mineralogical Society of America, 2000 Florida Avenue, N.W., Washington, D.C. 20009. Please remit \$5.00 in advance for the microfiche.

BETA1 S.F. AMPLITUDES FOR KAMBALDAITE (10FO 10FC 10SIGMAF)

0	0, 0, L	10	321	313	15	1	183	170	12	0	216	231	16	11	136	160	24	1, 5, L	5	323	316	12
1		11	104	139	32	2	292	296	10	1	53	76	38	12	157	127	24		6	162	185	19
2		12	282	259	16	3	21	85	50	2	163	165	19	13	101	77	33	0	7	109	131	27
3	4		830	824	8	4	162	211	17	3	0	83	77	0				1	8	88	110	32
4	6		251	229	12	5	137	115	19	4	296	313	15	1				2	9	19	67	70
5	8		762	763	10	6	152	155	19	5	43	66	48	2				3	10	186	173	22
6	10		315	296	15	7	146	171	23	6	138	158	24	0				4	11	148	132	23
7	12		378	375	16	8	209	211	18	7	131	97	24	1				5				
8						9	0	27	79	8	136	156	26	2				6				
9						10	119	88	26	9				3				7				
10						11	0	28	87	10				4				8				
11						12				11				5				9				
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BETA1 S. F. AMPLITUDES FOR KAMBALDAITE (10FO 10FC 10SIGMAF)

1, 14, L	8	82	72	32	2, 6, L	7	99	110	29	1	153	124	18	1	377	373	6	8	135	125	21		
	9	138	167	25		8	172	209	21	2	38	127	55	2	561	554	6	9	233	230	18		
6	20	79	65	10	0	1	0	102	86	3	63	58	36	3	302	307	8	10	235	179	17		
7	162	204	24	11	0	2	128	142	26	4	144	151	23	4	625	631	8	11	152	161	24		
8	165	175	21	12	0	3				5	107	85	26	5	248	257	11						
					2, 2, L	3								6	373	379	10						
1, 15, L	0	1408	1466	12	2, 11, L	0	159	149	15	2, 17, L	0	146	132	20	7	160	165	19					
0	149	184	22	0	4	452	445	11	0	8	38	8	47	10	71	94	36	0	373	366	10		
1	149	141	22	1	5	153	200	22	1	9	163	181	17	11	173	145	18	1	515	524	9		
2	129	131	25	2	6	441	437	13	2	10	140	158	23	12	114	106	28	2	271	285	11		
3	209	214	17	3	7	129	138	29	3	11	0	12	80	11	83	82	41	3	224	238	13		
4	86	61	28	4	8	180	193	23	4	12	0	32	78	12	128	146	29	4	102	90	21		
5	212	245	19	5	9	50	66	92	5	13				13				5	36	38	43		
6	87	83	35	6	10	228	227	20	6	14				14				6	116	125	23		
7	133	121	25	7	11				7	15				15				7	140	182	23		
				8	12				8	16				16				8	36	85	54		
1, 16, L	0	417	434	15	2, 7, L	0	336	335	9	2, 18, L	0	0	6	74	0	125	131	12	9	151	136	21	
0	109	42	23	0	1	249	246	10	1	1	136	105	22	2	200	195	9	10	84	116	38		
1	199	173	17	1	2	242	252	11	2	2				3	424	409	7	11	169	167	20		
2	157	165	21	2	3	59	81	28	3	3				4	486	497	8						
3	169	171	20	3	4	143	133	17	4	4				5	198	194	11						
4	184	155	20	4	5	86	67	25	5	5				6	312	311	10						
5	196	170	18	5	6	0	103	78	6	6				7	161	158	16	0	60	72	26		
6	182	187	21	6	7	155	164	8	7	7				8	65	104	36	0	303	337	12		
				7	8	587	570	7	8	8				9	97	114	31	1	213	218	13		
1, 17, L	0	596	592	7	2, 3, L	0	345	336	6	2, 12, L	0	358	335	5	10	103	63	26	2	289	301	12	
0	166	134	18	0	1	297	302	10	1	1	240	232	13	11	167	181	21	3	307	303	12		
1	189	184	19	1	2	313	319	10	2	2	130	98	18	12	105	95	33	4	218	220	16		
2	68	67	39	2	3	260	245	11	3	3	253	251	13	13	0	68	90	5	5	293	282	14	
3	169	145	18	3	4	62	88	36	4	4	77	153	36	14	111	122	25	6	170	186	20		
4	117	73	22	4	5	161	163	21	5	5	140	102	22	15	0	38	79	7	0	11	81		
5	71	92	37	5	6	70	101	39	6	6	105	129	33	16	0	48	86	8	8	138	138	23	
				6	7	159	139	23	7	7	0	72	82	17	10	0	48	86	9	9	119	73	26
1, 18, L	0	0	40	6	2, 8, L	0	821	825	9	2, 13, L	0	0	6	74	11	70	88	40	10	119	73	26	
0	101	76	26	7	1	37	66	35	1	1	155	169	19	11	185	176	21	11					
1	237	249	15	8	2	554	543	9	2	2	121	101	21	12				12					
2	157	149	19	9	3	56	63	30	3	3	66	91	34	13				13					
3	158	147	19	10	4	257	253	12	4	4	144	121	21	14				14					
				11	5	35	19	43	5	5	157	139	20	15				15					
2, 0, L	0	1319	1305	10	2, 4, L	0	355	320	13	2, 14, L	0	376	381	14	16				16				
0	639	619	9	1	1	390	378	14	1	1	0	6	74	17				17					
1	1193	1209	13	2	2	554	543	9	2	2	155	169	19	18				18					
2	226	231	8	3	3	56	63	30	3	3	121	101	21	19				19					
3	1082	1098	11	4	4	257	253	12	4	4	66	91	34	20				20					
4	250	251	11	5	5	35	19	43	5	5	144	121	21	21				21					
5	747	768	9	6	6	355	320	13	6	6	157	139	20	22				22					
6	232	221	13	7	7	0	20	78	7	7	127	76	23	23				23					
7	509	492	11	8	8	390	378	14	8	8	30	87	60	24				24					
8	0	101	81	9	9	0	18	81	9	9	30	41	56	25				25					
9	309	319	17	10	10	289	282	18	10	10	0	41	56	26				26					
10	99	12	28	11	11	0	18	92	11	11	0	41	56	27				27					
11	275	258	19	12	12	0	18	92	12	12	0	41	56	28				28					
12	0	49	94	13	13	0	18	92	13	13	0	41	56	29				29					
				14	14	0	18	92	14	14	0	41	56	30				30					
2, 5, L	0	418	382	7	2, 9, L	0	106	136	20	2, 15, L	0	84	51	31	15				31				
0	360	355	8	1	1	279	287	11	1	1	172	178	27	16				32					
1	77	72	22	2	2	119	104	18	2	2	83	3	28	17				33					
2	617	609	8	3	3	116	91	18	3	3	0	72	82	18				34					
3	213	211	12	4	4	106	73	27	4	4	106	99	23	19				35					
4	146	153	18	5	5	147	139	22	5	5	147	139	22	20				36					
5	171	161	18	6	6	54	11	42	6	6	54	11	42	21				37					
6	108	124	28	7	7	119	156	18	7	7	214	199	17	22				38					
7	75	18	33	8	8	194	188	24	8	8	214	199	17	23				39					
8	0	51	88	9	9	81	103	37	9	9	0	51	31	24				40					
9	113	94	29	10	10	103	112	33	10	10	84	51	31	25				41					
10	0	94	38	11	11	0	55	87	11	11	90	136	32	26				42					
11	96	146	38	12	12	71	24	40	12	12	93	50	27	27				43					
				13	13	0	55	87	13	13	122	64	21	28				44					
2, 1, L	0	136	162	9	2, 10, L	0	369	373	10	2, 16, L	0	124	98	23	14				45				
1	649	648	7	1	1	143	142	15	1	1	103	119	25	15				46					
2	1057	1055	11	2	2	371	370	11	2	2	118	89	22	16				47					
3	558	541	7	3	3	133	120	17	3	3	129	69	22	17									









BETA1 S.F. AMPLITUDES FOR KAMBALDAITE (10FO 10FC 10SIGMAF)

	16,4,L	2	100	62	22		17,1,L		17,2,L	1	227	260	17		18,1,L		19,0,L	
0	231	251	18				17,0,L	0	142	126	20	133	172	23	0	121	113	24
1	0	84	81					1	110	24	21	123	160	24	1	0	85	76
2	0	85	81	0	43	29	40	2	115	150	25			2	82	74	30	
3	79	59	31	1	0	79	78	3	0	21	71	18,0,L		3	202	166	18	
				2	173	172	17	4	37	50	52	0	111	174	31			
	16,5,L			3	170	175	22	5				1	85	82	28			
0	78	86	31	4	66	91	35		17,3,L			2	123	119	24			
1	0	13	76	5	136	135	22		0	137	128	21	3	0	35	77		
				6	121	124	23						4	37	45	47		
															0	75	35	29
															1	90	113	29