

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

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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 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 g cm⁻³. $F(000) = 540$, μ_{Mo} = 66.6 cm⁻¹.

Experimental details

A hexagonal prism ~0.1 mm thick and ~0.1 mm in length was mounted on a Syntex P₂ 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 MoK α radiation source ($\lambda = 0.7106\text{\AA}$). 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. 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; 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-

¹ 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)

11, 4, L	3 20 102 62 4 59 32 39	7 0 23 78 8 94 101 32	13, 1, L	0 117 104 21 1 313 329 13 2 246 269 15 3 298 293 14 4 205 196 16 5 166 142 19 6 129 121 24 7 104 70 25 8 149 146 23	13, 7, L	0 73 66 34 1 274 250 15 2 146 166 21 3 168 192 21 4 179 181 19 5 217 197 15	14, 4, L	0 72 97 33 1 122 136 23 2 90 114 28 3 0 26 80 4 147 161 23 5 30 28 55 6 123 157 27	0 48 17 39 1 202 226 18 2 66 106 41 3 240 208 15 4 252 253 17 5 142 179 25 6 171 172 20		
11, 5, L	0 460 524 12 1 45 52 39 2 353 371 12 3 38 44 42 4 125 132 22 5 0 51 77 6 195 190 17 7 48 33 46 8 301 300 16 9 108 58 28 10 186 215 22	12, 5, L	0 135 149 20 1 177 228 20 2 67 86 34 3 176 152 18 4 0 4 78 5 0 36 74 6 20 61 59 7 163 158 21 8 0 56 77	13, 2, L	0 0 6 72 1 75 111 30 2 116 81 21 3 157 159 19 4 123 96 20 5 97 125 30 6 112 51 24 7 0 93 84 8 0 55 83	13, 8, L	0 0 57 72 1 0 74 81 2 91 55 24 3 143 148 22	14, 5, L	0 153 189 21 1 136 97 21 2 54 101 38 3 131 112 32 4 127 112 25 5 117 102 25 6 119 155 23	15, 4, L	0 58 52 33 1 76 74 30 2 108 72 23 3 66 66 35 4 0 71 78 5 152 134 18
11, 6, L	0 84 86 23 1 0 12 75 2 90 73 31 3 180 182 19 4 0 86 82 5 149 143 20 6 138 120 24 7 110 93 28 8 20 34 61	12, 6, L	0 183 189 17 1 114 77 24 2 175 209 19 3 0 50 76 4 0 33 78 5 94 91 28 6 178 155 17 7 118 65 23	13, 3, L	0 179 148 15 1 213 179 15 2 150 115 18 3 313 348 15 4 122 154 23 5 271 286 15 6 157 150 20 7 170 170 20 8 146 172 23	13, 9, L	0 164 138 19 1 155 158 21	14, 6, L	0 89 12 24 1 83 73 31 2 97 135 30 3 142 128 20 4 62 97 38	15, 5, L	0 235 215 17 1 155 152 22 2 85 123 31 3 224 219 17 4 118 97 24
11, 7, L	0 207 234 20 1 215 201 17 2 95 152 33 3 342 308 14 4 102 116 30 5 284 281 18 6 89 94 31 7 201 210 20	12, 7, L	0 0 19 72 1 49 91 42 2 138 93 19 3 160 177 21 4 80 84 31 5 191 156 19 6 0 33 77	13, 4, L	0 194 204 19 1 76 98 32 2 111 124 23 3 81 68 30 4 66 71 36 5 0 23 79 6 122 95 24 7 54 22 39	14, 0, L	0 261 231 12 1 124 76 20 2 148 165 19 3 0 83 78 4 310 313 14 5 0 66 76 6 167 158 18 7 129 97 21 8 145 156 23	14, 7, L	0 66 22 31 1 134 155 22 2 110 83 24 3 78 86 30	15, 6, L	0 139 146 21 1 90 77 27 2 0 71 79
11, 8, L	0 20 85 64 1 101 109 28 2 104 109 27 3 31 92 60 4 149 141 21 5 143 73 18 6 72 63 34	12, 8, L	0 147 133 20 1 104 138 26 2 122 149 25 3 0 123 84 4 142 114 22 5 116 91 24	14, 1, L	0 92 90 26 1 280 282 14 2 148 152 20 3 111 127 24 4 93 96 31 5 84 99 31 6 54 110 41 7 157 152 20 8 58 103 43	14, 2, L	0 257 231 13 1 133 106 20 2 268 260 14 3 0 48 76 4 0 24 77 5 85 98 29 6 165 189 21 7 99 79 26 8 171 187 21	15, 0, L	0 213 212 15 1 51 32 38 2 152 171 18 3 0 82 77 4 97 116 30 5 66 42 34 6 66 75 39 7 0 63 79 8 0 104 85	16, 0, L	0 80 76 30 1 92 51 25 2 191 192 18 3 111 66 25 4 110 153 26 5 58 69 39 6 142 160 21 7 138 87 21
11, 9, L	0 125 139 26 1 255 287 18 2 169 162 20 3 189 170 18 4 82 89 34 5 137 128 21	12, 9, L	0 163 138 18 1 54 80 39 2 94 114 29 3 94 86 27 4 75 70 32	14, 3, L	0 74 76 31 1 157 150 17 2 145 109 20 3 134 175 24 4 0 24 75 5 236 219 15 6 0 30 75 7 143 150 21	15, 1, L	0 158 193 22 1 242 254 14 2 210 188 14 3 147 178 24 4 19 70 59 5 153 160 22 6 119 93 24 7 195 236 20	15, 3, L	0 89 81 24 1 147 159 20 2 65 71 34 3 75 61 38 4 103 107 26 5 91 36 27 6 0 42 78 7 90 86 31	16, 1, L	0 102 41 22 1 180 142 14 2 98 130 30 3 158 138 19 4 145 148 20 5 179 165 19 6 125 114 22
11, 10, L	0 0 67 80 1 139 154 24 2 0 47 79	12, 10, L	0 0 46 72 1 0 65 75	13, 5, L	0 157 174 20 1 284 294 15 2 135 140 21 3 198 189 17 4 144 129 20 5 156 137 21 6 104 121 29 7 227 241 18	16, 2, L	0 82 106 32 1 19 57 61 2 108 139 25 3 126 116 23 4 152 128 21 5 88 109 31	16, 3, L	0 49 106 43 1 223 232 17 2 108 75 22 3 85 87 30 4 72 3 26 5 29 28 52		

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	155	133	18	0	142	126	20	2	133	172	23	0	121	113	24	0	29	50	52	
1	0	84	81	0	43	29	40	1	148	141	20	1	110	21	3	123	160	24	1	0	85	76	1	120	70	22		
2	0	85	81	1	0	79	78	2	144	203	24	2	115	150	2	18.0.L				2	82	74	30	2	113	85	24	
3	79	59	31	2	173	172	17	3	218	248	19	3	0	21	71	0	111	174	31	3	202	166	18					
16.5.L				3	170	175	22	4	72	19	31	4	37	50	52	1	85	82	28	18.2.L								
0	78	86	31	4	66	91	35	5	228	230	16	17.3.L				2	123	119	24	0	75	35	29					
1	0	13	76	5	136	135	22	0					3	0	35	77	3	0	35	77	1	90	113	29				
				6	121	124	23					4	37	45	47	4	37	45	47									