Table 3 (*cont.*)

-4	100*	58
-5	-210	-116
-6	-100*	-115
-7	-140	-72
-8	-317	-328
-9	97*	17
-10	-95*	-101
-11	92*	39
-12	88*	97
-13	83*	64
-14	76*	91
-15	-67*	-16
-16	-54*	-76
• 20	4	
0	88*	138
1	85*	68
2	81*	97
3	76*	63
4	-70*	-47
5	61*	17
-1	252	164
-2	92*	90
-3	93*	113
-4	-94*	-127
-5	94*	46
-6	-93*	-37
-7	-92*	-18
-8	261	239
-9	-89*	-66
-10	86*	63
-11	-82*	-52
-12	-169	-176
-13	-71*	-37
-14	-177	-164
-15	99	70
• 22	4	
0	-100	-247
1	-67*	-33
2	-106	-173
3	-51*	-43
-1	-76*	-46
-2	-78*	-43
-3	80*	38
-4	81*	205
-5	-82*	-24
-6	81*	23
-7	-80*	-66
-8	-78*	-166
-9	133*	64
-10	-72*	-34
-11	67*	58
-12	61*	67
-13	-51*	-6
• 24	4	
-1	-48*	-84
-2	-54*	-57
-3	-58*	-88
-4	-60*	-124
-5	-61*	-18
-6	61*	32
-7	60*	26
-8	82	109
-9	-54*	-49
-10	48*	42
• 1	5	
0	-61	-79
1	-134	-160
2	-225	-262
3	46*	33
4	52*	7
5	247	229
6	283	315
7	-112	-130
8	397	503
9	-158	-175
10	103	109
11	-176	-134
12	-290	-280
13	-171	-84
14	-161	-153
15	86	17
-1	-78	-105
-2	187	231
-3	43*	44
-4	239	314
-5	157	136
-6	-195	-92
-7	-206	-192
-8	-395	-454
-9	-123	-148
-10	112	94
-11	125	18
-12	368	292
-13	116	29
-14	76*	24
-15	66*	51
-16	-104	-75
• 3	5	
0	292	427
1	176	194
2	334	412
3	52*	52
4	-255	-287
5	-211	-147
6	-206	-215
7	69*	59
8	-71*	-73
9	72*	34
10	133	67
11	-86*	-86
12	200	200
13	-75*	-45
14	66*	32
-1	105	109
-2	-100	-78
-3	150	147
-4	-278	-298
-5	189	227
-6	-209	-312
-7	-115	-86
-8	-63*	-51
-9	-341	-365
-10	-197	-228
-11	-89*	-79
-12	-309	-245
-13	-84*	-43
-14	78*	32
-15	-144	-104
-16	175	136
• 5	5	
0	-281	-340
1	89	55
2	-354	-378
3	-195	-183
4	223	167
5	111	48
6	238	175
7	230	144
8	72*	50
9	125	2
10	-87*	-69
11	83*	69
12	-77*	-34
13	97	108
14	112	110
-1	-132	-122
-2	272	298
-3	51*	38
-4	291	314
-5	402	521
-6	-283	-289
-7	361	351
-8	-388	-415
-9	71*	14
-10	102	55
-11	-329	-200
-12	294	174
-13	-84*	-51
-14	79*	78
-15	100	21
-16	-91	-65
• 7	5	
0	-82	-29
1	-189	-158
2	343	379
3	-65*	-39
4	-182	-103
5	-70*	-55
6	-317	-371
7	72*	82
8	-89*	-41
9	87*	93
10	83*	124
11	77*	11
12	69*	36
13	-58*	-22
-1	-134	-166
-2	-298	-291
-3	-297	-289
-4	-155	-140
-5	-309	-302
-6	263	281
-7	67*	36
-8	376	446
-9	217	220
-10	72*	72
-11	231	197
-12	-213	-65
-13	84*	23
-14	-111	-111
-15	-71*	-17
-16	61*	24
• 9	5	
0	-290	-283
1	205	225
2	-384	-512
3	185	155
4	-71*	-39
5	-72*	-47
6	103	143
7	-88*	-82
8	-121	-57
9	-82*	-50
10	-153	-151
11	-69*	-8
12	-81	-84
-1	-185	-167
-2	148	75
-3	-215	-269
-4	416	424
-5	-211	-180
-6	343	393
-7	-98	-89
-8	-110	-101
-9	-72*	-61
-10	-145	-174
-11	-125	-74
-12	86*	12
-13	83*	52
-14	162	141
-15	70*	27
-16	84	30
• 11	5	
0	188	262
1	-71*	-14
2	248	242
3	-189	-145
4	-103	-73
5	88*	38
6	-123	-104
7	119	142
8	160	97
9	121	45
10	168	157
11	-63	-84
-1	297	304
-2	-69*	-19
-3	69*	41
-4	-287	-317
-5	99	40
-6	-155	-151
-7	187	162
-8	145	111
-9	-73*	-20
-10	194	155
-11	87*	15
-12	84*	19
-13	160	21
-14	-188	-157
-15	-66*	-42
-16	-55*	-9
• 13	5	
0	-72*	-2
1	-103	-52
2	126	72
3	-88*	-17
4	174	197
5	158	114
6	214	234
7	-108	-10
8	99	9
9	-175	-132
10	-69	-91
-1	102	9
-2	-102	-57
-3	72*	52
-4	199	231
-5	102	12
-6	134	74
-7	126	105
-8	-249	-213
-9	151	86
-10	-219	-157
-11	-84*	-43
-12	-81*	-56
-13	-76*	-21
-14	70*	82
-15	-61*	-14
• 15	5	
0	124	71
1	-87*	-19
2	-86*	-16
3	-83*	-24
4	-169	-175
5	-108	-3
6	-71*	-66
7	-119	-53
8	113	137
-1	88*	44
-2	89*	33
-3	89*	28
-4	-194	-198
-5	88*	36
-6	-170	-150
-7	-215	-154
-8	134	94
-9	-132	-115
-10	227	167
-11	80*	81
-12	-76*	-11
-13	99	34
-14	-281	-234
-15	-52*	-48
• 17	5	
0	-134	-191
1	149	142
2	110	16
3	105	61
4	138	125
5	-89	-30
6	54*	42
-1	-83*	-8
-2	-231	-202
-3	-85*	-114
-4	85*	61
-5	-85*	-86
-6	151	110
-7	118	93
-8	-82*	-29
-9	80*	43
-10	-188	-184
-11	-73*	-52
-12	-68*	-24
-13	61*	63
-14	167	193
• 19	5	
0	101	81
1	-68*	-77
2	-90	-89
3	-82	-34
4	-49*	-94
-1	74*	26
-2	123	159
-3	76*	13
-4	-77*	-69
-5	-77*	-54
-6	-76*	-66
-7	-75*	-32
-8	103	67
-9	70*	29
-10	201	181
-11	-62*	-51
-12	161	128
-13	-44*	-16
• 21	5	
0	-104	-96
1	-45*	-36
-1	-57*	-37
-2	-170	-174
-3	-62*	-12
-4	63*	48
-5	-103	-65
-6	141	176
-7	61*	9
-8	59*	65
-9	55*	26
-10	-70	-70
-11	-40*	-15

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The Crystal Structure of Huntite, $Mg_3Ca(CO_3)_4$

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Huntite, $Mg_3Ca(CO_3)_4$, is an ordered rhombohedral double-carbonate, based, like the simple compositions, on a deformation of the NaCl face-centered cube. Its ordering, in $R32$, affords a true structural unit in the shape of the familiar cleavage rhomb, with $a = 6{\cdot}075$ Å and $\alpha = 102° 56'$. With Ca at the origin, three Mg are disposed about face centers, one CO_3 is unique at the body center, and three CO_3 are disposed about edge centers.

Although huntite is available only as powder (of about 1 micron particle size) and involves seven variable parameters, the precision with which pertinent bond lengths are known from the simpler carbonates permits a moderately accurate evaluation of the structure.

The model arranged to conform with these known bond lengths affords a series of calculated F values for the first 46 possible index combinations, from which observed amplitudes reduced from 41 powder lines observed in this range deviate by an average of only 11%. The greatest individual departure is 34%.

Huntite, $Mg_3Ca(CO_3)_4$, was first identified by Faust (1953) from magnesite deposits of Tertiary age in Nevada. Additional deposits, formed from surface waters in the weathering zone and in caves and mine workings, have been reported by Koblencz & Nemecz (1953), Baron *et al.* (1957), Skinner (1958), and Golvanov (1959). These huntites are all characterized by grain sizes of the magnitude of 1μ, frequently with no extraneous phases detectable in powder X-ray diffraction diagrams. Huntite analyses typically include about 1% H_2O (+110 °C.), but Stevens' analysis of a Nevada sample (in Faust, 1953) calculated on a water-free basis gives satisfactory mol ratios $MgO : CaO : CO_2 = 3{\cdot}07 : 1{\cdot}00 : 4{\cdot}03$. All observed diffraction features have been indexed on a rhombohedral cell containing only $Mg_3Ca(CO_3)_4$.

Table 1. *Comparison of observed and calculated d-spacings and structure factors for huntite,* $Mg_3Ca(CO_3)_4$

$h_rk_rl_r$	d_o	d_c	F_c	$F_c \exp(-B \sin^2 \theta/\lambda^2)$	Relative F_o	$\frac{F_c \exp(-B \sin^2 \theta/\lambda^2) - \text{Relative } F_o}{\text{Relative } F_o}$
100	$5{\cdot}66_5$	5·670	5·20	5·16	5·37	−0·04
$10\bar{1}$	$4{\cdot}75_3$	4·753	5·27	5·19	4·22	+0·23
$11\bar{1}$	$3{\cdot}64_0$	3·643	7·77	7·56	8·41	−0·10
110	$3{\cdot}53_3$	3·532	6·47	6·29	5·04	+0·25
$20\bar{1}$	$2{\cdot}88_8$	2·891	23·4	22·5	23·2	−0·03
200	$2{\cdot}83_3$	2·835	73·8	70·7	71·7	−0·01
$11\bar{2}$	$2{\cdot}74_4$	2·744	10·8	10·3	9·65	+0·07
111	2·604	2·607	43·0	40·9	44·5	−0·08
$21\bar{1}$	2·432	2·435	15·7	14·8	18·2	−0·19
$20\bar{2}$	2·375	2·377	30·3	28·5	25·1	−0·14
210	2·284	2·286	14·6	13·7	14·8	−0·07
$21\bar{2}$	2·190	2·192	18·4	17·1	17·6	−0·03
$3\bar{1}\bar{1}$	1·991	1·990	33·9	31·1	33·0	−0·06
$30\bar{1}$	1·972	1·972	37·3	34·1	40·7	−0·15
211	1·896	1·902	7·88	7·16	10·8	−0·34
300		1·890	9·37	8·28	9·26	−0·11
$22\bar{1}$			8·86			
$30\bar{2}$	1·835	1·836	17·6	15·9	14·2	+0·12
$22\bar{2}$	1·821	1·821	22·6	20·4	17·2	+0·19
$21\bar{3}$	1·796	1·797	15·9	14·3	14·1	+0·01
220	1·765	1·766	54·7	49·0	55·2	−0·11
$31\bar{1}$	1·757	1·756	39·0	34·9	39·2	−0·11
$31\bar{2}$	1·700	1·701	16·6	14·7	14·0	+0·05
310	1·656	1·656	8·68	7·66	6·70	+0·14
$22\bar{3}$	Present	1·611	10·2	8·93	8·20	+0·09
$30\bar{3}$	1·584	1·584	46·0	40·1	44·5	−0·10
221	Present	1·537	5·57	4·81	Not measurable	
$31\bar{3}$	1·526	1·526	20·2	17·4	14·0	+0·24
$11\bar{4}$	1·518	1·518	16·2	14·0	15·3	−0·08
$32\bar{1}$	1·481	1·485	28·1	24·1	22·4	+0·08
$32\bar{2}$		1·479	18·8	16·3	17·5	−0·07
$40\bar{1}$			19·4			
311	Present	1·462	10·3	8·79	Not measurable	
$4\bar{1}\bar{2}$	1·453	1·453	20·4	17·4	15·3	+0·14
$40\bar{2}$	1·445	1·446	22·1	18·8	18·2	+0·03
400	1·418	1·418	22·6	19·1	20·8	−0·08
320	1·397	1·398	17·0	14·3	17·7	−0·19
$32\bar{3}$	1·383	1·383	12·9	10·8	12·2	−0·11
$4\bar{2}\bar{2}$	1·372	1·372	34·6	28·9	24·2	+0·19
$41\bar{1}$	1·354	1·358	9·55	7·96	Not measurable	
$41\bar{2}$		1·354	16·6	13·8	14·5	−0·05
$40\bar{3}$	1·333	1·334	12·7	10·5	10·4	+0·01
$31\bar{4}$	1·318	1·318	21·4	17·6	18·7	−0·06
222	1·304	1·304	74·1	60·7	59·0	+0·03
410	1·290	1·290	10·8	8·80	7·40	+0·19
$41\bar{3}$	1·279	1·279	13·9	11·3	11·7	−0·03
						Average 0·11

A value of $B \times 10^{16} = 1{\cdot}4$ was arbitrarily chosen as a reasonable value for a relatively plastic solid, and one which afforded both + and − amplitude deviations throughout the angular range of the data.

The list of observed *d*-spacings given in Table 1 was measured from a powder diffraction film taken of a sample from Currant, Nevada, using filtered Fe radiation and a 114·59 mm. diameter Straumanis-mount camera. The cell constants obtained for this material, from a least squares analysis involving a drift error term of the form $\sin^2 2\theta[(1/\sin\theta)-(1/\theta)]$, are $a_0 = 9{\cdot}505$, $c_0 = 7{\cdot}821$ Å ($a_{rh} = 6{\cdot}075$ Å, $\alpha = 102° 56'$). The quality of the agreement between calculated and observed *d*-spacings substantiates the correctness of the indexing. A more extensive discussion of huntite cell constants will be presented elsewhere.

The reflections considered in the structure analysis extended only to $\sin\theta/\lambda$ values of about 0·35. Preliminary inspection of the data showed strong resemblance to the powder diagrams of the simple rhombohedral carbonates, which in turn are deformations of the NaCl cube. An ordered arrangement for cations in three to one ratio requires that the simplest unit be at least the cleavage-rhomb-shaped 4-cation cell (on which the indices of Table 2 are based), and the number of variable parameters is patently too great to allow a straight-forward structural analysis from powder data. The assumption has therefore been made that C–O, Mg–O, and Ca–O bond lengths must be closely comparable with those that have been established in

Table 2. *Parameters in R32*

Rhombohedral cell	Hexagonal cell $(0, 0, 0;\ \frac{1}{3}, \frac{2}{3}, \frac{2}{3};\ \frac{2}{3}, \frac{1}{3}, \frac{1}{3})+$
1 Ca at 0, 0, 0	3 Ca at 0, 0, 0
1 C_I at $\frac{1}{2}, \frac{1}{2}, \frac{1}{2}$ (basal-plane carbonate group)	3 C_I at $0, 0, \frac{1}{2}$
3 Mg in (*d*) $0, x, \bar{x}$, etc. with $x=0{\cdot}541$	9 Mg in (*d*) $x, 0, 0$, etc. with $x=0{\cdot}541$
3 C_{II} in (*e*) $\frac{1}{2}, x, \bar{x}$, etc. with $x=-0{\cdot}039$ (tilted carbonate groups)	9 C_{II} in (*e*) $x, 0, \frac{1}{2}$, etc. with $x=0{\cdot}461$
3 O_I in (*e*) $\frac{1}{2}, x, \bar{x}$, etc. with $x=0{\cdot}365$ (basal-plane carbonate group)	9 O_I in (*e*) $x, 0, \frac{1}{2}$, etc. with $x=-0{\cdot}135$
3 O_{II} in (*e*) $\frac{1}{2}, x, \bar{x}$, etc. with $x=0{\cdot}096$ (in-plane oxygens of tilted carbonate groups)	9 O_{II} in (*e*) $x, 0, \frac{1}{2}$, etc. with $x=-0{\cdot}404$
6 O_{III} in (*f*) x, y, z, etc. with $x=-0{\cdot}033$, $y=0{\cdot}180$, $z=0{\cdot}371$ (out-of-plane oxygens of tilted carbonate groups)	18 O_{III} in (*f*) x, y, z, etc. with $x=0{\cdot}461$, $y=0{\cdot}135$, $z=0{\cdot}506$

the pertinent simple rhombohedral carbonate compositions (Steinfink & Sans, 1959; Sass *et al.*, 1957; Chessin & Post, 1958).

Such an arrangement is easily arrived at in *R*32. In a rhombohedral analogue to the NaCl face centered cube, Ca is placed at the origin, and the carbon atom of the unique CO_3 group at the body center. The three Mg are then displaced cyclically from the face centers with positive parameter increments, and the carbons of the other three CO_3 groups are displaced cyclically from the edge centers with negative parameter increments. Maintenance of known bond lengths renders the several variable parameters interdependent. The parameters of Table 2, taken as a working model, afford the pertinent interatomic distances listed in Table 3.

Table 3. *Interatomic distances in huntite model*

	Cation–oxygen	
	$C–O_{I,II,III}$	1·28 Å
	$Mg–O_I$	2·10
	$Mg–O_{II}$	2·09
	$Mg–O_{III}$	2·10
	$Ca–O_{III}$	2·35
	Oxygen–oxygen in octahedra	
in basal plane	$O_I–O_{II}$	2·56 (the shared edge)
	$O_I–O_{III}$	3·10
	$O_{II}–O_{III}$	2·89
out of basal plane	$O_I–O_{II}$	3·09
	$O_I–O_{III}$	3·06
	$O_{II}–O_{II}$	2·86
	$O_{III}–O_{III}$	3·30 (av. for octahedra 2·96)
	in prism	
in basal plane	$O_{III}–O_{III}$	3·32
out of basal plane	$O_{III}–O_{III}$	2·70

Calculated *F*-values for this model are listed in Table 1 and compared with relative observed amplitudes for those powder reflections (41 of the first 44 possibilities) which yield integrated intensity measurements of reasonable accuracy on a slow-speed diffractometer track. Except for one value of 34%, no deviations exceed 25% and the absolute average, analogous to an *R*-factor, is 11%. The level of agreement is such that the present parameters may be considered a reasonably accurate description of the structure. Any further refinement would require terms for higher angle reflections, which overlap in the powder diagrams and are doubtfully indexed.

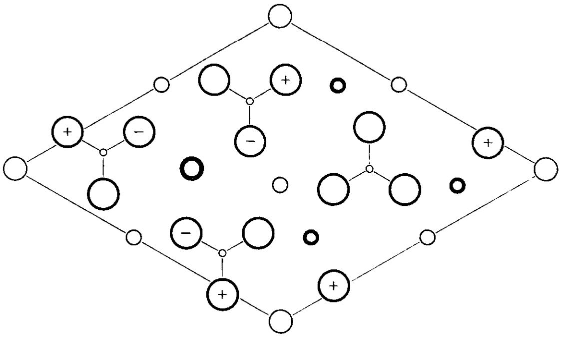

Fig. 1. A section of the huntite structure parallel to the hexagonal base. C, Mg, Ca, and O are represented schematically by successively larger circles. The unique carbonate group lies in the plane and the other three carbonate groups are slightly inclined to it with oxygens marked '+' 0·05 Å above and those marked '−' 0·05 Å below the plane. Light Ca and Mg circles are 1·305 Å below the plane and heavy Ca and Mg 1·305 Å above it. Successive layers shifted one third to the right build the rhombohedral array.

The carbonate groups of one layer of the structure, in a slice cut perpendicular to the unique axis, and their associated cations are illustrated in Fig. 1. The whole structure is built up by successive shifts to the right of one-third the long diagonal of the figure for each succeeding superposed layer. In contrast with dolomite and the single-cation rhombohedral carbonates, in which all carbonate groups in a given layer have one orientation and which must therefore be described on the basis of the steep rhombohedron as a unit cell, this structure has at the body center the one unique carbonate which is disposed in 180° rotation with respect to the other three, and the cleavage rhomb itself as the structural unit. All unique carbonates have one orientation and lie in the basal plane; all edge-centered have the other and are slightly inclined to the base. Each unique carbonate has 6 Mg neighbors, and each edge-centered has 2 Ca and 4 Mg. The environment of each Mg is an octahedron, distorted by foreshortening of its two shared edges, as illustrated in Fig. 2. The environment of each Ca is a nearly right trigonal prism. The assumptions made in setting up the model concentrate probable errors in the prism shape, but it is unlikely that the rotation of prism bases toward an octahedral configuration is in error by more than 5°, or that the prism height of 2·70 Å differs by more than 0·05 Å from the true value. The height of the Ca octahedron in CaO is 2·77 Å and O–O edges are 3·40 Å.

If the assumed cation–oxygen bond articulation is retained, the agreements with observed amplitudes for several low index reflections are deleteriously affected by trials for shared edge lengths 0·02 Å from the

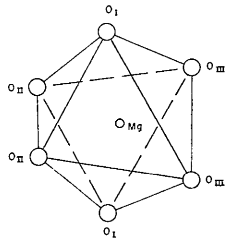

Fig. 2. The octahedral environment of Mg. The O_I–O_{II} edges lying in basal planes are shared with equivalent octahedra.

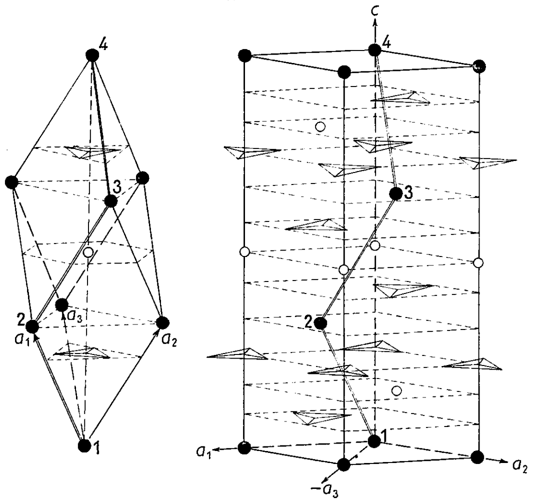

Fig. 3. The contents of the rhombohedral and hexagonal unit cells of dolomite. The orientation of the two cells relative to each other is shown by the position of the common line 1–2–3–4. Mg are schematically represented by small open circles, Ca by larger filled circles, carbonate groups by triangular symbols. Ca, Mg, and C in huntite are essentially distributed among the same total group of positions as in dolomite, except for slight shifts of Mg and the carbons of tilted carbonate groups.

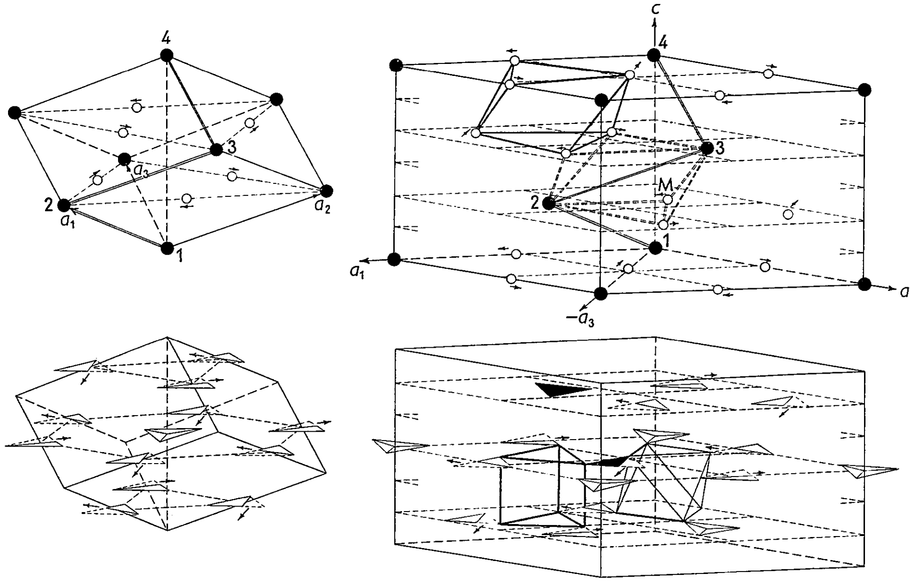

Fig. 4. The rhombohedral and hexagonal unit cells of huntite, drawn to the same scale as those of dolomite (Fig. 3). The orientation of the two cells relative to each other is shown by the position of the common line 1–2–3–4. Small open circles are Mg; larger filled circles, Ca; solid-line triangles, carbonate groups lying in basal planes. The partially dotted triangles represent carbonate groups tilted out of basal planes about the indicated angular bisectrices as axes, with the dotted portion in each case lying below the basal plane. Small arrows indicate the hexagonal axial directions along which Mg are shifted from dolomite cation positions. Arrows similarly show directions of shifts of the C of tilted carbonate groups. The C shifts have been ignored in drafting the rhombohedral cell.

Heavy solid lines connect the six Mg that surround a basal-plane carbonate group, colored black for reference. Dashed double lines similarly indicate the four Mg and two Ca that surround a tilted carbonate group, half-black for reference. Another set of heavy solid lines connects the six O forming a nearly right trigonal prism about a Ca atom ('2'). Thin solid lines outline the six O forming an octahedron about a Mg atom ('M'); the two shared edges are shown as heavier lines.

chosen 2·56 Å value. The chosen value is reasonably consistent with Kamb's (1960) recent elaborate analysis of zunyite in which Al octahedra with two adjacent shared edges each are found to have 2·53 Å shared edge lengths, and with Newnham's (1960) determination of 2·36 Å for the 3 non-adjacent shared edges in the Al octahedra of dickite. The authors are not aware of any evaluations of shared edge lengths in Mg octahedra with so few shared edges, but the 2·56 Å figure seems to bear a proper relation to both kinds of shared edges determined for the Al octahedra. Other O–O distances probably are less precise, especially the O_{III}–O_{III} edge, which has a component complementary to the Ca prism height.

It seems probable that Mg are actually displaced somewhat from effective octahedral centers, but it is considered unjustifiable to make such a test from powder data.

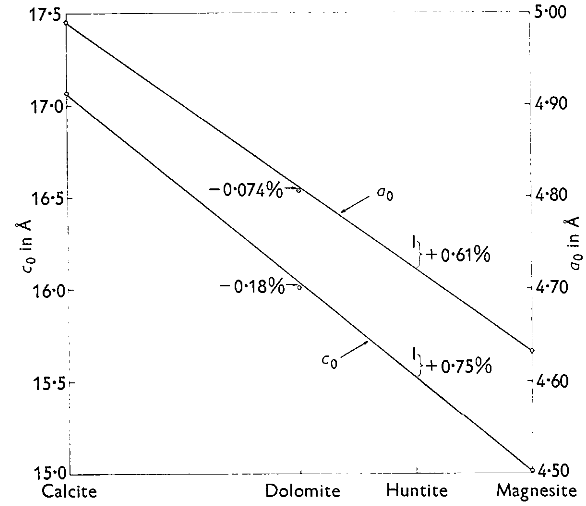

Fig. 5. A comparison of a_0 and c_0 values of calcite, dolomite, and magnesite with those of the equivalent hexagonal pseudo-cell of huntite.

These geometrical relationships within the huntite unit cell, and the relation of the huntite cell to that of dolomite, are further illustrated in Figs. 3 and 4.

The a- and c-axis lengths of dolomite, calcite, and magnesite are compared in Fig. 5 with values for the analogous hexagonal pseudo-cell of huntite. The bars plotted for huntite indicate the maximum uncertainty among measurements of several samples by various experimental techniques. Whereas the dolomite a_0 and c_0 are very slightly less than predicted from averages of calcite and magnesite values (Goldsmith & Graf, 1958), the huntite values deviate 5 to 8 times as much in the direction of inefficient packing. Huntite is thus an ordered, relatively low-density phase. It has not been observed to form in experimental work in CaMg carbonate systems from 500 to 1250 °C., and higher pressures would obviously not help. A small stability field therefore is probably to be sought in the complex region of low partial pressures of CO_2 and earth-surface temperatures where hydrates, basic carbonates, and hydroxides as well as anhydrous carbonates occur. This is precisely the natural environment in which huntite has been found.

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