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The crystal structure of picropharmacolite, $Ca_4Mg(HAsO_4)_2(AsO_4)_2 \cdot 11H_2O$

MICHELE CATTI, GIOVANNI FERRARIS AND GABRIELLA IVALDI

Istituto di Mineralogia, Cristallografia e Geochimica 'G. Spezia'
Università di Torino, via S. Massimo 22, 10123 Torino, Italy

Abstract

X-ray diffraction intensities were measured by single-crystal diffractometry (Mo$K\alpha$ radiation) on picropharmacolite from Sainte-Marie-aux-Mines (Alsace), and the following structure parameters were determined: $a = 13.547(3)$, $b = 13.500(3)$, $c = 6.710(1)$Å, $\alpha = 90.85(1)$, $\beta = 96.41(2)$, $\gamma = 91.60(1)°$; $Z = 2$, space group $P\bar{1}$. The structure was solved by direct methods; the mixed isotropic (oxygen atoms) and anisotropic (heavier atoms) refinement converged to $R = 0.087$ (1611 reflections). As, Ca, and Mg coordination polyhedra sharing edges and vertices form corrugated (100) layers, which are linked by hydrogen bonding only. Four independent water molecules are sandwiched between adjacent layers, and build up [001] hydrogen-bonded chains. The Mg coordination octahedron and the Ca polyhedra show typical bond distances, so that no significant Ca/Mg substitution should occur in any cation site. The formula of picropharmacolite can then be written as $Ca_4Mg(H_2O)_7(AsO_3OH)_2(AsO_4)_2 \cdot 4H_2O$. A close relationship is observed between this structure and those of the two dimorphs guerinite and ferrarisite, $Ca_5(HAsO_4)_2(AsO_4)_2 \cdot 9H_2O$; in these minerals the layers of polyhedra are also present, but are linked by Ca–O bonds in addition to hydrogen bonds. Cleavage and possible twinning are discussed on structural grounds.

Introduction

Picropharmacolite is a member of a family of mineral acid arsenates of divalent cations (Pierrot, 1964), and occurs as globular crusts of tiny crystals whose poor quality prevented a crystal structure determination until now. The known phases of the family, whose chemical formula can be written as $M_5^{2+}H_2(AsO_4)_4 \cdot nH_2O$, can be divided into two groups: species such as sainfeldite, $Ca_5H_2(AsO_4)_4 \cdot 4H_2O$, the dimorphs guerinite and ferrarisite, $Ca_5H_2(AsO_4)_4 \cdot 9H_2O$ (Bari *et al.*, 1980), and chudobaite, $(Mg,Zn)_5H_2(AsO_4)_4 \cdot 10H_2O$, with only one type of M^{2+} (except for isomorphous substitution); minerals such as irhtemite, $Ca_4MgH_2(AsO_4)_4 \cdot 4H_2O$ (Pierrot and Schubnel, 1972), and picropharmacolite, $Ca_4MgH_2(AsO_4)_4 \cdot 11H_2O$ where two different, and presumably ordered, types of M^{2+} are present. The Ca and Ca/Mg phases often occur together as accessory minerals which originated by reactions of arsenic ores with surrounding calcareous rocks. The crystal structures of sainfeldite (Ferraris and Abbona, 1972), guerinite (Catti and Ferraris, 1974), ferrarisite (Catti *et al.*, 1980), and chudobaite (Dorner and Weber, 1976) have been determined.

Different crystallochemical formulae have been proposed for picropharmacolite (Abbona and Ferraris, 1976). In most chemical analyses, the CaO/As_2O_5 ratio is slightly higher than 2.0 and the MgO/As_2O_5 ratio fluctuates significantly around 0.5. On the basis of (1) the results obtained by Guérin *et al.* (1967) on synthetic picropharmacolite, (2) the criticism of the available chemical analyses, and (3) a close similarity with the layered structure of guerinite, Abbona and Ferraris favored the hypothesis that there was no solid solution between Ca and Mg in picropharmacolite; a limited substitution involving Ca^{2+}, Mg^{2+}, and H^+ was, however, not completely excluded. A structural study of picropharmacolite was undertaken to clarify its crystallochemical features and to throw light on its relationships with other members of the family.

Experimental

A sample of picropharmacolite from Sainte-Marie-aux-Mines (Alsace) was kindly supplied by H. Bari (BRGM, Orléans). After a difficult search (which followed several unsuccessful attempts made previously at our institute on other samples), a tiny needle-like

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OBSERVED AND CALCULATED STRUCTURE FACTORS FOR PICROPHARMACOLITE CA4MGH2(ASO4)4.11H20

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6	0	0	45	43	-11	3	0	115	120	5	5	0	130	126	2	8	0	82	79	4	12	0	115	-110
9	0	0	81	-80	-7	3	0	62	-56	6	5	0	51	-57	3	8	0	196	-193	5	12	0	116	121
10	0	0	106	104	-3	3	0	66	70	7	5	0	41	53	4	8	0	116	121	6	12	0	68	-63
-16	1	0	49	33	-2	3	0	220	-223	-13	6	0	48	-57	9	8	0	51	-45	-7	13	0	48	-59
-13	1	0	54	58	-1	3	0	73	66	-8	6	0	65	63	12	8	0	56	59	-4	13	0	81	91
-12	1	0	91	-92	0	3	0	130	123	-7	6	0	100	-98	13	8	0	53	-56	-3	13	0	58	-38
-11	1	0	49	52	1	3	0	68	-64	-6	6	0	95	99	-9	9	0	81	83	8	13	0	69	-79
-8	1	0	131	-129	2	3	0	75	-70	-4	6	0	77	80	-8	9	0	60	-63	-5	14	0	66	-44
-7	1	0	38	29	6	3	0	74	68	-3	6	0	49	-56	-5	9	0	136	137	-4	14	0	62	64
-5	1	0	46	40	7	3	0	39	29	-2	6	0	103	-94	-4	9	0	42	-44	-1	14	0	73	-81
-2	1	0	43	-43	8	3	0	91	-94	0	6	0	59	59	2	9	0	52	-43	0	14	0	45	47
-1	1	0	60	-69	9	3	0	116	-116	1	6	0	104	101	3	9	0	91	-85	-3	15	0	51	42
1	1	0	129	132	10	3	0	110	110	4	6	0	49	-34	6	9	0	121	120	-1	15	0	47	-49
2	1	0	28	-35	14	3	0	46	47	5	6	0	78	-71	7	9	0	49	-48	3	15	0	71	-75
3	1	0	159	-161	-12	4	0	48	-36	6	6	0	71	82	9	9	0	57	-52	-2	-15	1	49	52
4	1	0	168	175	-10	4	0	55	61	8	6	0	93	-82	-6	10	0	65	-70	-1	-15	1	49	-56
5	1	0	58	53	-9	4	0	62	64	11	6	0	62	62	-5	10	0	70	80	-4	-14	1	61	-62
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12	1	0	65	63	-7	4	0	59	52	-10	7	0	98	103	-2	10	0	134	-130	-1	-14	1	65	58
13	1	0	87	-85	-6	4	0	50	-51	-9	7	0	85	-76	-1	10	0	65	49	0	-14	1	91	-87
-15	2	0	46	48	-5	4	0	44	-50	-8	7	0	46	-39	0	10	0	62	60	-7	-13	1	63	56
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-12	2	0	39	35	1	4	0	327	293	-2	7	0	94	-85	7	10	0	70	72	-2	-13	1	81	-61
-9	2	0	77	73	2	4	0	281	-265	-1	7	0	151	-147	8	10	0	47	-53	-1	-13	1	97	97
-8	2	0	93	-97	3	4	0	145	133	0	7	0	53	64	-9	11	0	54	58	0	-13	1	74	-66
-4	2	0	37	-35	7	4	0	85	82	1	7	0	57	59	-8	11	0	70	-83	2	-13	1	60	-68
-3	2	0	105	-115	9	4	0	51	-41	2	7	0	48	-54	-2	11	0	48	-45	3	-13	1	78	77
-1	2	0	31	-32	10	4	0	57	-52	7	7	0	72	79	1	11	0	93	106	5	-13	1	56	-48
0	2	0	53	53	11	4	0	71	84	9	7	0	88	-94	3	11	0	59	-61	-10	-12	1	60	61
4	2	0	44	-40	-11	5	0	90	-86	11	7	0	48	57	4	11	0	71	70	-9	-12	1	58	-55
5	2	0	67	76	-10	5	0	88	80	-10	8	0	49	50	-9	12	0	50	40	-8	-12	1	68	52
7	2	0	91	-107	-8	5	0	53	52	-7	8	0	112	-114	-5	12	0	97	98	-4	-12	1	74	-65
8	2	0	46	-50	-7	5	0	53	-56	-6	8	0	153	162	-4	12	0	94	-98	-3	-12	1	90	95
9	2	0	38	-33	-6	5	0	82	84	-5	8	0	80	-79	-3	12	0	60	55	-1	-12	1	52	-34
10	2	0	135	139	-4	5	0	49	-52	-2	8	0	44	-54	-1	12	0	70	-71	0	-12	1	47	43

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
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3	-12	1	60	58	7	-8	1	56	-53	15	-5	1	56	40	0	-2	1	92	110	10	0	1	75	80
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-6	-11	1	45	44	-14	-7	1	52	-51	-11	-4	1	51	-50	2	-2	1	59	-67	-5	1	1	65	60
-2	-11	1	48	38	-10	-7	1	71	-66	-7	-4	1	118	-132	3	-2	1	102	-114	-4	1	1	65	55
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-12	-10	1	83	77	2	-7	1	91	85	0	-4	1	170	-171	14	-2	1	42	33	1	1	1	49	-41
-11	-10	1	56	-58	3	-7	1	60	-59	3	-4	1	87	79	15	-2	1	49	37	2	1	1	44	-50
-8	-10	1	46	47	8	-7	1	61	68	4	-4	1	89	93	-11	-1	1	43	-51	4	1	1	74	82
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-7	-8	1	102	102	1	-5	1	77	63	13	-3	1	86	92	-4	0	1	240	-251	-6	3	1	60	54
-5	-8	1	60	-39	2	-5	1	84	-92	-10	-2	1	38	38	1	0	1	95	-93	-5	3	1	44	52
-2	-8	1	122	120	3	-5	1	66	67	-9	-2	1	99	-100	2	0	1	70	65	-4	3	1	205	-210
-1	-8	1	55	-53	5	-5	1	82	76	-8	-2	1	71	73	3	0	1	33	-39	-3	3	1	52	42
0	-8	1	64	-61	6	-5	1	162	-172	-7	-2	1	48	-47	5	0	1	86	94	-1	3	1	135	125
2	-8	1	68	-63	7	-5	1	53	55	-6	-2	1	89	89	7	0	1	89	-87	0	3	1	64	-53

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
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3	3	1	134	-121	-5	6	1	93	-91	7	9	1	105	-101	2	-15	2	45	-38	-6	-9	2	68	-64
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7	4	1	50	-47	5	7	1	55	46	-3	11	1	92	-93	2	-12	2	57	45	-7	-7	2	63	68
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-8	5	1	58	45	12	7	1	55	57	-9	12	1	61	-75	-2	-11	2	48	57	1	-7	2	50	53
-6	5	1	95	95	-11	8	1	98	105	-8	12	1	46	41	4	-11	2	91	92	2	-7	2	58	49
-3	5	1	54	35	-10	8	1	82	-81	0	12	1	123	124	5	-11	2	120	-120	6	-7	2	141	-146
0	5	1	71	73	-2	8	1	92	-93	1	12	1	96	-82	-11	-10	2	48	-30	7	-7	2	122	121
5	5	1	43	52	-1	8	1	170	165	3	13	1	80	-83	-4	-10	2	41	-41	-14	-6	2	65	51
6	5	1	176	-169	4	8	1	85	68	4	13	1	54	58	-3	-10	2	88	103	-12	-6	2	44	47
7	5	1	64	81	10	8	1	45	46	-3	14	1	86	-94	-1	-10	2	99	-106	-11	-6	2	91	-93
8	5	1	46	-44	-8	9	1	47	-50	-2	14	1	54	50	0	-10	2	42	36	-2	-6	2	101	106
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12	5	1	45	-55	-4	9	1	70	-69	5	14	1	61	51	8	-10	2	65	77	0	-6	2	108	-113
-11	6	1	42	43	-2	9	1	41	-29	-2	15	1	47	-34	9	-10	2	75	-73	1	-6	2	145	145
-10	6	1	43	-31	0	9	1	93	90	0	15	1	54	64	-13	-9	2	57	45	2	-6	2	75	78
-8	6	1	85	-97	1	9	1	65	62	-5	-15	2	56	64	-8	-9	2	46	56	3	-6	2	86	97

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
4	-6	2	44	-41	-3	-3	2	43	-54	8	-1	2	113	112	-11	2	2	54	-71	3	4	2	53	-41
8	-6	2	40	33	-2	-3	2	211	228	9	-1	2	50	-56	-9	2	2	61	-60	4	4	2	108	97
9	-6	2	62	58	-1	-3	2	187	-206	10	-1	2	75	67	-8	2	2	120	123	8	4	2	39	51
10	-6	2	119	-122	0	-3	2	113	121	14	-1	2	45	39	-7	2	2	44	-48	10	4	2	45	-53
11	-6	2	51	55	1	-3	2	45	-57	-13	0	2	66	70	-4	2	2	46	-52	-14	5	2	44	-31
-12	-5	2	82	77	6	-3	2	39	-45	-12	0	2	71	-66	-3	2	2	75	67	-12	5	2	51	41
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-1	-6	4	67	-73	4	-3	4	42	-42	5	0	4	44	37	-3	3	4	44	46	-11	7	4	54	-60
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H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
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-4	7	4	46	29	0	-10	5	80	93	-10	-4	5	56	-52	3	-1	5	52	-41	7	2	5	51	-54
-3	7	4	52	59	1	-10	5	42	-40	-8	-4	5	62	-54	4	-1	5	45	-48	-12	3	5	54	37
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6	7	4	59	-67	-7	-9	5	47	45	-3	-4	5	43	-45	11	-1	5	44	36	-3	3	5	54	-59
7	7	4	56	71	1	-9	5	65	48	-1	-4	5	44	46	-7	0	5	81	-76	-2	3	5	50	46
9	7	4	46	-46	2	-9	5	66	-64	0	-4	5	54	-63	-6	0	5	56	59	-1	3	5	70	76
-7	8	4	91	-96	4	-9	5	64	61	1	-4	5	45	52	-2	0	5	53	-46	0	3	5	44	-43
-10	9	4	58	36	-6	-8	5	60	66	2	-4	5	86	-89	0	0	5	44	40	3	3	5	45	-48
-8	9	4	46	54	-4	-8	5	50	-35	3	-4	5	101	114	3	0	5	78	-75	6	3	5	74	72
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5	10	4	97	-102	3	-7	5	45	-28	10	-3	5	46	56	-3	1	5	50	-44	0	4	5	65	51
-1	11	4	67	-67	4	-7	5	57	51	-10	-2	5	45	50	0	1	5	103	98	2	4	5	60	-59
1	11	4	50	40	6	-7	5	47	57	-9	-2	5	39	-15	2	1	5	62	-54	5	4	5	91	86
2	11	4	48	27	8	-7	5	44	-45	-7	-2	5	48	-47	3	1	5	109	-103	6	4	5	70	-66
-1	-14	5	59	55	-8	-6	5	62	-57	-6	-2	5	158	165	4	1	5	100	92	7	4	5	77	79
2	-13	5	48	-50	-7	-6	5	102	108	-5	-2	5	71	-73	7	1	5	52	-48	-6	5	5	80	87
3	-13	5	45	35	-4	-6	5	54	-62	2	-2	5	86	-94	8	1	5	56	48	0	5	5	43	45
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-1	-11	5	78	90	-6	-5	5	95	93	-6	-1	5	41	49	-4	2	5	145	-146	7	5	5	54	56
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4	-11	5	47	-37	5	-5	5	56	40	-3	-1	5	38	-38	-1	2	5	50	-37	-1	6	5	82	-83

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
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5	6	5	56	55	-5	-6	6	65	66	-11	-1	6	52	36	1	3	6	100	89	-6	-4	7	68	-75
-7	7	5	50	-66	-4	-6	6	64	-66	-9	-1	6	54	-57	2	3	6	105	-106	1	-4	7	44	-40
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3	7	5	56	-47	8	-6	6	49	38	-1	-1	6	79	-84	-3	4	6	46	-37	-5	-3	7	64	-64
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0	8	5	48	53	-2	-5	6	81	-79	8	-1	6	69	80	-9	5	6	60	-58	4	-3	7	65	56
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-2	-9	6	75	75	1	-3	6	52	-54	-8	2	6	53	58	4	-7	7	48	-45	4	2	7	45	-39
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-4	-8	6	70	-67	-5	-2	6	59	-60	-1	2	6	93	97	-7	-5	7	80	-78	-4	3	7	68	71
-3	-8	6	73	67	-4	-2	6	43	52	1	2	6	56	-55	-6	-5	7	51	46	-3	3	7	57	-63
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-4	-7	6	80	-7[illegible]	-2	-2	6	62	65	-7	3	6	46	55	0	-5	7	65	-53	-2	5	7	45	47
4	-7	6	47	54	-1	-2	6	74	70	-1	3	6	72	73	2	-5	7	59	74	-1	5	7	45	-37
7	-7	6	48	-38																				