Powder Diffraction Data for Synthetic Pargasite, Scandian Pargasite and Their Fluorine Analogues: $NaCa_2Mg_4(Al,Sc)Si_6Al_2O_{22}(OH,F)_2$

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Abstract
Indexed X-ray powder diffraction data and crystal data are reported for:
synthetic pargasite (P: $NaCa_2Mg_4AlSi_6Al_2O_{22}(OH)_2$),
fluor-pargasite (FP: $NaCa_2Mg_4AlSi_6Al_2O_{22}F_2$),
scandian pargasite (SP: $NaCa_2Mg_{4.4}Sc_{0.6}Si_6Al_2O_{22}(OH)_2$)
and scandian fluor-pargasite
(SFP: $NaCa_2Mg_{4.2}Sc_{0.8}Si_6Al_2O_{22}F_2$).

Introduction
Amphiboles are common rock-forming silicate minerals, found in a wide variety of igneous and metamorphic rocks. They can be orthorhombic or monoclinic, and have the general formula $A_{0-1}B_2C_5T_8O_{22}W_2$, where A = Na, K;B = Ca, Na, Li, Mg, Fe^{2+}, Mn; C = Mg, Fe^{2+}, Mn, Al, Fe^{3+}, Ti, Li; T = Si, Al, Ti; W = OH, F, Cl, O^{2-}. The structural chemistry of natural amphiboles is often extremely complex, and its interpretation is facilitated by a knowledge of the physical and chemical characteristics of end-members.

Pargasite is a common calcic amphibole with the ideal formula $NaCa_2Mg_4AlSi_6Al_2O_{22}(OH)_2$; other trivalent cations (Cr^{3+}, Fe^{3+}, Ga, Sc) can substitute for Al in octahedral coordination (see Hawthorne, 1983, for structural details). Here we report the synthesis and X-ray powder diffraction data for pargasite, scandian pargasite and their fluorine analogues.

Synthesis
Dry mixtures of amphibole stoichiometry were prepared from commercial reagent-grade chemicals. Pargasites were synthesized in sealed Au capsules using conventional cold-seal hydrothermal apparatus at 800-900°C and 2kb (200Mpa) water pressure. Fluorine analogues of pargasites were reacted at 1000°C, 1atm, in sealed Pt capsules suspended in a vertical furnace. Run products were examined by X-ray powder diffraction and scanning electron microscopy to identify any extraneous phases.

X-ray studies
Samples were ground under ethanol to less than 5μm grain size and were front-loaded into standard aluminum sample holders with a glass insert to support the powder. The slurry was worked with a probe so that it was evenly distributed and dried with its surface slightly rough, but precisely flush with the top of the slide. Preferred orientation was minor. Powder diffractograms were obtained at room temperature on a Philips Automated Powder Diffractometer System PW1710 using CuKα radiation (λ= 1.54178 Å). The instrument was equipped with a theta-compensating slit and graphite diffracted-beam monochromator. Generator settings were 40kV and 40mA. BaF_2 [a = 6.19860(5) Å] calibrated against Si, [NBS SRM 640a, (1962), *a* = 5.43083(4) Å], was used as an internal standard. Kα_1 peak positions were calculated by a five-point parabolic fit to each peak, followed by correction against the internal calibration pattern and allowance for Kα_1-Kα_2 splitting as a function of 2θ. Cell dimensions (Table 1) were refined and the pattern was indexed using the least-squares program of Appleman and Evans (1973). Indexed X-ray powder patterns are given in Tables 2-5; 2θ to *d* conversions were done using the wavelength for CuKα_1 (λ = 1.5406 Å. Relative intensities were corrected to fixed-slit values.

The crystal structures of three of these phases (excluding pargasite) were refined by the Rietveld method (Rietveld, 1969); full details are given elsewhere (Raudsepp *et al.*, 1987). This work confirmed the pattern indexing given Table 2, and also showed that the crystal structures could be refined satisfactorily in the space group C2/*m*.

Table 1.
Cell dimensions for Synthetic Pargasitic Amphiboles

	a (Å)	b (Å)	c (Å)	β (°)	V (Å^3)
P	9.899(2)	17.945(4)	5.279(1)	105.60(2)	903.3(2)
FP	9.816(2)	17.918(3)	5.292(1)	105.18(2)	898.2(2)
SP	9.941(2)	18.090(3)	5.2952(9)	105.39(2)	918.1(2)
SFP	9.878(2)	18.151(3)	5.315(1)	105.23(2)	919.5(2)

Table 2.
X-ray Powder Diffraction Data for Pargasite
$F_{30} = 26(0.018, 63)$

$2\theta_{exp}$ (°)	I/I_O	d_{exp} (Å)	*h k l*	$\Delta 2\theta$* (°)
9.865	34	8.9588	0 2 0	-0.0153
10.493	53	8.4240	1 1 0	+0.0049
17.491	14	5.0662	0 0 1	-0.0635
			1 3 0	-0.0032
18.100	23	4.8971	$\bar{1}$ 1 1	-0.0013
18.572	9	4.7737	2 0 0	+0.0247
19.779	30	4.4850	0 4 0	-0.0058
21.088	12	4.2095	2 2 0	-0.0028
21.831	11	4.0678	$\bar{2}$ 0 1	+0.0112
22.446	17	3.9578	1 1 1	+0.0069
22.913	23	3.8781	$\bar{1}$ 3 1	+0.0081
26.514	66	3.3590	1 3 1	+0.0143
27.275	67	3.2670	2 4 0	-0.0011
28.483	100	3.1311	3 1 0	+0.0148
29.286	15	3.0471	$\bar{3}$ 1 1	-0.0277
30.569	70	2.9220	2 2 1	+0.0228
31.861	27	2.8064	3 3 0	-0.0026
32.538	38	2.7496	$\bar{3}$ 3 1	+0.0096
33.317	68	2.6870	1 5 1	-0.0113
34.774	50	2.5777	0 6 1	-0.0027
35.228	54	2.5433	$\bar{2}$ 0 2	+0.0031
			0 0 2	+0.0473
36.693	9	2.4472	$\bar{2}$ 2 2	-0.0233
			0 2 2	+0.0194
37.308	15	2.4082	$\bar{2}$ 6 1	-0.0152
37.771	16	2.3798	3 5 0	+0.0067
38.413	51	2.3415	$\bar{3}$ 5 1	-0.0398
			$\bar{4}$ 2 1	+0.0360
39.292	24	2.2911	$\bar{3}$ 1 2	-0.0254
			$\bar{1}$ 7 1	+0.0261
40.172	9	2.2429	0 8 0	-0.0039
			3 3 1	+0.0136
40.730	14	2.2135	$\bar{2}$ 4 2	-0.0068
			0 4 2	+0.0322
42.016	31	2.1486	1 3 2	-0.0755
			2 6 1	+0.0056
42.456	10	2.1274	$\bar{1}$ 5 2	+0.0220
44.571	28	2.0312	$\bar{4}$ 0 2	-0.0382
			2 8 0	+0.0358
			2 0 2	+0.0707
45.192	15	2.0047	3 5 1	-0.0231
46.446	8	1.9535	1 9 0	+0.0460
46.647	8	1.9455	$\bar{3}$ 5 2	+0.0570
47.952	14	1.8956	5 1 0	-0.0177
49.132	12	1.8528	$\bar{4}$ 4 2	+0.0326
49.533	8	1.8387	$\bar{1}$ 7 2	-0.0063
50.153	8	1.8174	5 3 0	+0.0177
51.886	7	1.7607	$\bar{5}$ 1 2	-0.0573
			3 1 2	+0.0723
54.132	7	1.6929	0 0 3	-0.0671
			3 3 2	-0.0591
			0 10 1	+0.0233
54.505	11	1.6821	$\bar{2}$ 8 2	-0.0250
			0 8 2	+0.0063
			$\bar{1}$ 3 3	+0.0343
55.089	9	1.6657	0 2 3	+0.0122
55.969	16	1.6416	4 6 1	-0.0196
			$\bar{2}$ 10 1	-0.0044
57.303	7	1.6065	1 11 0	-0.0583
57.990	8	1.5891	1 1 3	-0.0385
			6 0 0	-0.0009
			$\bar{5}$ 5 2	+0.0335
58.576	12	1.5746	$\bar{1}$ 5 3	+0.0123

*$2\theta_{exp} - 2\theta_{calc}$

Table 3
X-ray Powder Diffraction Data for Fluor-pargasite
$F_{30} = 27(0.020, 57)$

$2\theta_{exp}$ (°)	I/I_O	d_{exp} (Å)	*h k l*	$\Delta 2\theta$* (°)
9.865	31	8.9588	0 2 0	-0.0004
10.532	41	8.3929	1 1 0	+0.0227
17.339	5	5.1103	0 0 1	+0.0113
17.518	10	5.0584	1 3 0	+0.0212
18.121	25	4.8915	$\bar{1}$ 1 1	-0.0039
19.787	24	4.4832	0 4 0	+0.0163
21.239	21	4.1799	2 2 0	-0.0388
21.991	11	4.0386	$\bar{2}$ 0 1	-0.0076
22.357	14	3.9733	1 1 1	+0.0143
22.939	26	3.8738	$\bar{1}$ 3 1	+0.0098
26.453	93	3.3666	1 3 1	+0.0170
27.342	78	3.2592	2 4 0	+0.0390
28.666	76	3.1116	3 1 0	+0.0162
29.491	11	3.0264	$\bar{3}$ 1 1	+0.0128
30.529	82	2.9258	$\bar{1}$ 5 1	-0.0387
			2 2 1	+0.0266
32.022	19	2.7927	3 3 0	+0.0128
32.764	43	2.7311	$\bar{3}$ 3 1	+0.0162
33.273	100	2.6905	1 5 1	+0.0039
34.782	64	2.5771	0 6 1	-0.0109
35.260	80	2.5433	$\bar{2}$ 0 2	+0.0065
36.535	11	2.4574	0 2 2	+0.0270
37.410	15	2.4019	$\bar{4}$ 0 1	+0.0009
			$\bar{2}$ 6 1	+0.0074
37.929	13	2.3702	3 5 0	+0.0174
38.590	30	2.3311	$\bar{3}$ 5 1	+0.0013
38.784	15	2.3203	$\bar{4}$ 2 1	+0.0048
39.392	39	2.2855	$\bar{1}$ 7 1	-0.0166
			$\bar{3}$ 1 2	+0.0427
40.136	7	2.2448	3 3 1	+0.0729
			0 8 0	+0.0952
40.777	17	2.2110	$\bar{2}$ 4 2	-0.0070
42.037	42	2.1476	2 6 1	-0.0125
			$\bar{3}$ 3 2	-0.0058
42.409	14	2.1296	$\bar{1}$ 5 2	+0.0250
44.474	22	2.0354	2 0 2	-0.0134
44.838	25	2.0197	$\bar{4}$ 0 2	-0.0053
45.218	18	2.0036	3 5 1	-0.0118
45.612	9	1.9872	3 7 0	-0.0292
			2 2 2	+0.0453
46.137	5	1.9658	$\bar{3}$ 7 1	+0.0024
46.588	7	1.9479	1 9 0	-0.0116
			1 5 2	+0.0439
46.876	7	1.9366	$\bar{3}$ 5 2	-0.0049
48.286	12	1.8833	5 1 0	-0.0238
49.132	14	1.8528	2 4 2	-0.0208
			$\bar{1}$ 9 1	-0.0310
49.486	13	1.8404	4 4 2	-0.0299
			$\bar{1}$ 7 2	+0.0216
50.468	6	1.8068	5 3 0	+0.0264
52.272	10	1.7486	$\bar{1}$ 1 3	-0.0137
			$\bar{5}$ 1 2	-0.0171
53.295	4	1.7175	1 7 2	-0.0200
54.502	12	1.6822	3 9 0	-0.0652
			0 8 2	-0.0540
			$\bar{2}$ 8 2	+0.0530
54.880	11	1.6715	0 2 3	-0.0292
			$\bar{3}$ 9 1	0.0464
56.083	18	1.6385	4 6 1	-0.0347
			$\bar{2}$ 10 1	+0.0219
57.376	8	1.6046	1 11 0	-0.0283
58.463	18	1.5773	6 0 0	-0.0615
			$\bar{1}$ 5 3	-0.0232
			$\bar{5}$ 5 2	-0.0263

*$2\theta_{exp} - 2\theta_{calc}$

Table 4.
X-ray Powder Diffraction Data for Scandian Pargasite
$F_{30} = 21(0.021,68)$

$2\theta_{exp}$ (°)	I/I_O	d_{exp} (Å)	*h k l*	$\Delta 2\theta$* (°)
9.777	43	9.0392	0 2 0	-0.0060
10.442	62	8.4650	1 1 0	-0.0056
17.383	11	5.0974	1 3 0	-0.0221
			0 0 1	-0.0272
18.051	30	4.9103	$\bar{1}$ 1 1	-0.0028
19.601	35	4.5253	0 4 0	+0.0130
20.967	14	4.2335	2 2 0	-0.0063
22.322	18	3.9795	1 1 1	+0.0009
22.804	24	3.8964	$\bar{1}$ 3 1	+0.0007
26.339	85	3.3809	1 3 1	+0.0179
27.089	70	3.2890	2 4 0	-0.0010
28.340	100	3.1466	3 1 0	+0.0030
29.174	29	3.0585	$\bar{3}$ 1 1	-0.0066
30.382	70	2.9396	2 2 1	+0.0130
31.661	34	2.8237	3 3 0	+0.0065
32.413	51	2.7599	$\bar{3}$ 3 1	+0.0020
33.063	80	2.7071	1 5 1	+0.0052
34.505	54	2.5972	0 6 1	+0.0163
35.137	86	2.5519	$\bar{2}$ 0 2	+0.0086
36.532	13	2.4576	0 0 2	+0.0143
			$\bar{2}$ 2 2	+0.0326
37.075	17	2.4228	$\bar{2}$ 6 1	-0.0132
37.495	18	2.3967	3 5 0	+0.0303
38.245	48	2.3514	$\bar{3}$ 5 1	-0.0733
			$\bar{4}$ 2 1	+0.0555
39.134	27	2.3000	$\bar{1}$ 7 1	-0.0955
			$\bar{3}$ 1 2	+0.0411
39.904	10	2.2574	0 8 0	-0.0698
			3 3 1	+0.0133
40.540	15	2.2234	0 4 2	+0.0083
			$\bar{2}$ 4 2	+0.0251
41.703	37	2.1640	1 3 2	+0.0013
			$\bar{3}$ 3 2	+0.0341
			2 6 1	+0.0081
42.257	10	2.1369	$\bar{1}$ 5 2	+0.0081
44.400	33	2.0386	2 0 2	-0.0217
			$\bar{4}$ 0 2	+0.0251
44.855	16	2.0190	3 5 1	-0.0043
45.665	13	1.9851	$\bar{4}$ 2 2	-0.0647
			$\bar{3}$ 7 1	-0.0219
46.102	10	1.9673	1 9 0	+0.0034
46.502	9	1.9513	1 5 2	-0.0185
			$\bar{3}$ 5 2	+0.0116
46.998	9	1.9318	4 2 1	+0.0163
47.683	14	1.9057	5 1 0	-0.0169
48.049	8	1.8920	$\bar{4}$ 6 1	-0.0174
49.182	10	1.8510	$\bar{1}$ 7 2	+0.0495
49.922	9	1.8253	5 3 0	-0.0456
50.306	8	1.8123	4 4 1	+0.0290
51.675	9	1.7674	3 1 2	-0.0486
			$\bar{5}$ 1 2	+0.0070
53.024	7	1.7256	1 7 2	-0.0033
			$\bar{3}$ 7 2	+0.0240
54.159	15	1.6921	$\bar{3}$ 1 3	-0.0454
			2 10 0	-0.0099
			0 8 2	-0.0174
			$\bar{2}$ 8 2	-0.0039
54.856	8	1.6722	0 2 3	-0.0067
55.532	17	1.6534	$\bar{2}$ 10 1	+0.0041
			4 6 1	+0.0124
56.754	10	1.6207	1 11 0	-0.0025
57.668	11	1.5972	1 1 3	-0.0175
			6 0 0	-0.0113
58.318	13	1.5809	$\bar{1}$ 5 3	-0.0043
58.915	8	1.5663	$\bar{1}$ 11 1	+0.0467

$*2\theta_{exp} - 2\theta_{calc}$

Table 5.
X-ray Powder Diffraction Data for Scandian Fluor-pargasite
$F_{30} = 34(0.014,62)$

$2\theta_{exp}$ (°)	I/I_O	d_{exp} (Å)	*h k l*	$\Delta 2\theta$* (°)
9.764	45	9.0512	0 2 0	-0.0263
10.489	60	8.4272	1 1 0	-0.0137
17.362	9	5.1035	0 0 1	-0.0849
			1 3 0	-0.0153
18.036	29	4.9143	$\bar{1}$ 1 1	-0.0198
19.549	33	4.5372	0 4 0	-0.0023
19.836	7	4.4722	0 2 1	+0.0330
21.061	9	4.2148	2 2 0	-0.0216
22.273	12	3.9881	1 1 1	-0.0104
22.761	18	3.9037	$\bar{1}$ 3 1	-0.0040
26.276	95	3.3889	1 3 1	+0.0039
27.118	46	3.2856	2 4 0	-0.0051
28.492	100	3.1302	3 1 0	+0.0081
30.372	60	2.9406	2 2 1	+0.0068
31.787	31	2.8128	3 3 0	+0.0013
32.529	46	2.7503	$\bar{3}$ 3 1	-0.0031
32.971	97	2.7144	1 5 1	-0.0068
34.386	51	2.6059	0 6 1	+0.0042
35.088	66	2.5554	$\bar{2}$ 0 2	-0.0015
36.458	8	2.4624	$\bar{2}$ 2 2	+0.0400
37.021	10	2.4263	$\bar{2}$ 6 1	-0.0046
37.595	12	2.3905	3 5 0	-0.0025
38.242	31	2.3516	$\bar{3}$ 5 1	-0.0106
38.478	20	2.3377	$\bar{4}$ 2 1	+0.0355
38.930	21	2.3116	$\bar{1}$ 7 1	-0.0148
39.209	17	2.2958	$\bar{3}$ 1 2	-0.0024
39.948	8	2.2550	3 3 1	+0.0014
40.474	12	2.2269	$\bar{2}$ 4 2	+0.0041
41.637	36	2.1673	2 6 1	-0.0060
42.057	8	2.1466	$\bar{1}$ 5 2	+0.0479
44.252	17	2.0451	2 0 2	+0.0035
44.570	16	2.0313	$\bar{4}$ 0 2	-0.0114
44.831	18	2.0200	3 5 1	-0.0169
45.466	8	1.9728	1 9 0	-0.0074
			4 0 1	+0.0211
46.515	7	1.9507	$\bar{3}$ 5 2	-0.0206
			$\bar{5}$ 1 1	-0.0219
47.046	7	1.9300	4 2 1	+0.0781
47.943	12	1.8959	5 1 0	+0.0068
49.045	10	1.8559	$\bar{1}$ 7 2	-0.0004
			4 4 2	-0.0428
50.185	7	1.8163	5 3 0	-0.0489
			0 10 0	+0.0377
51.575	5	1.7706	3 1 2	-0.0225
51.901	6	1.7603	$\bar{5}$ 1 2	+0.0117
53.003	5	1.7262	3 7 2	-0.0148
			$\bar{5}$ 5 1	-0.0159
53.635	5	1.7074	3 3 2	-0.0039
54.067	11	1.6947	$\bar{3}$ 1 3	-0.0496
			$\bar{1}$ 3 3	-0.0096
54.561	8	1.6806	0 2 3	-0.0229
55.570	19	1.6524	4 6 1	-0.0172
56.538	10	1.6264	1 11 0	-0.0194
58.051	31	1.5875	6 0 0	-0.0342
			$\bar{1}$ 5 3	-0.0062
58.963	5	1.5651	$\bar{5}$ 7 1	-0.0029
			6 2 0	-0.0219

$*2\theta_{exp} - 2\theta_{calc}$

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