

Testing the resolution of point X-ray
Diffraction (XRD) using zoned
Plagioclase crystals from the Duluth
Complex against a chemical
composition baseline set by Scanning
Electron Microscopy (SEM).

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PETROLOGY

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Outline

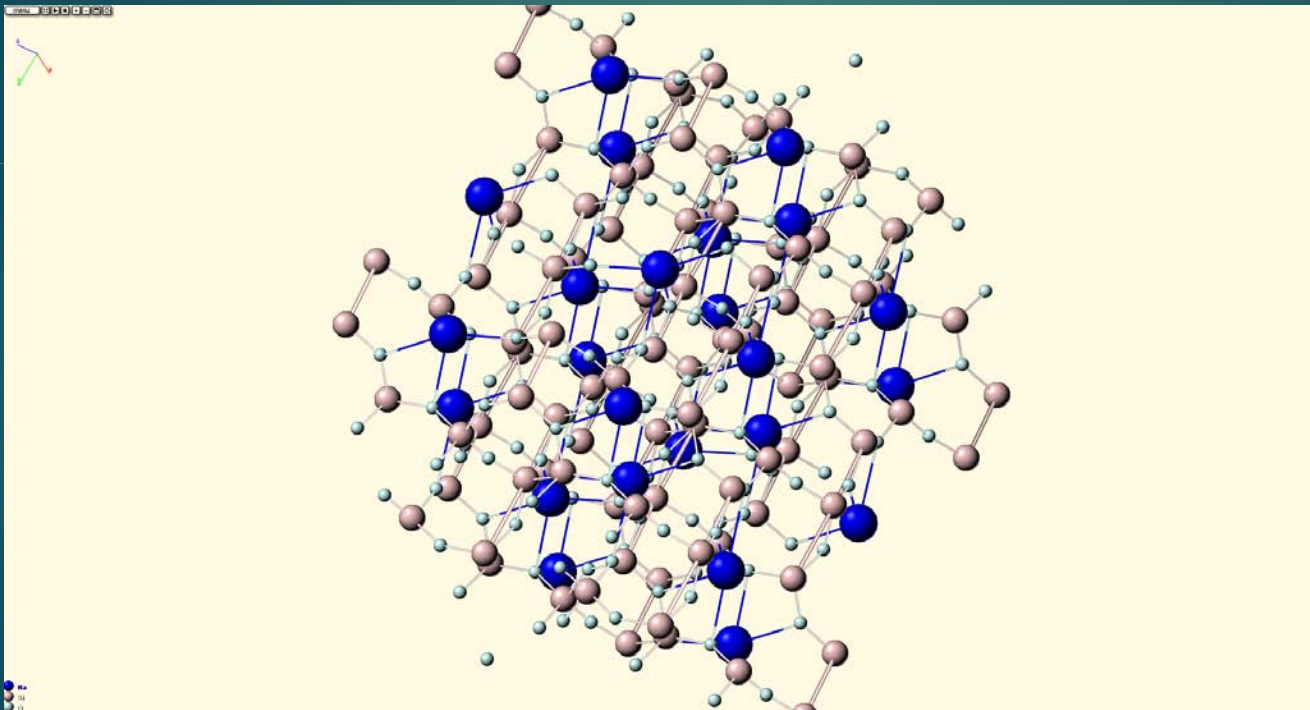
- ▶ Introduction
- ▶ Methods
- ▶ Crystal analyses
- ▶ Conclusions



Plagioclase

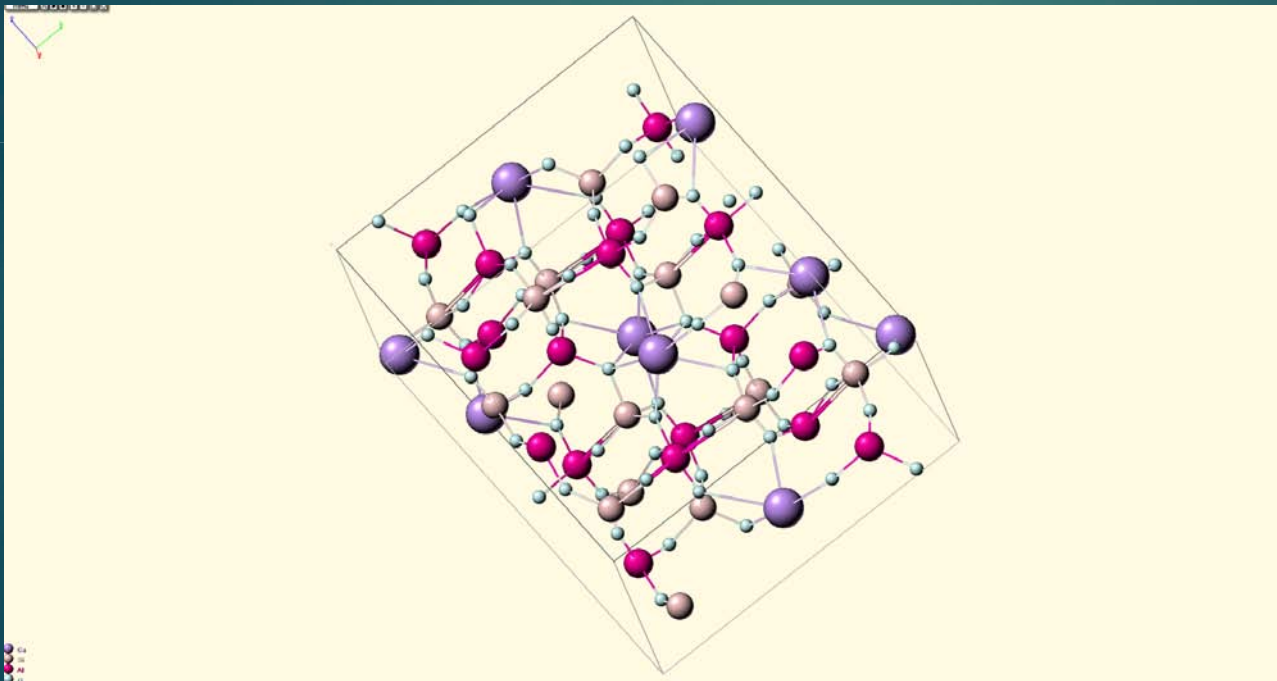
- ▶ Complete solid solution of $\text{NaAlSi}_3\text{O}_8$ (albite) to $\text{CaAl}_2\text{Si}_2\text{O}_8$ (anorthite)
- ▶ Zonation prevalent due to substitution chemistry
- ▶ Crystal Structure distorts depending on chemistry

Albite Structure



Space Group: C1
Cell Parameters:
 $a = 8.16 \text{ \AA}$ $b = 12.87 \text{ \AA}$
 $c = 7.11 \text{ \AA}$
 $\alpha = 93.45^\circ$, $\beta = 116.4^\circ$,
 $\gamma = 90.28^\circ$

Anorthite Structure



Space Group: $P1$

Cell Parameters:

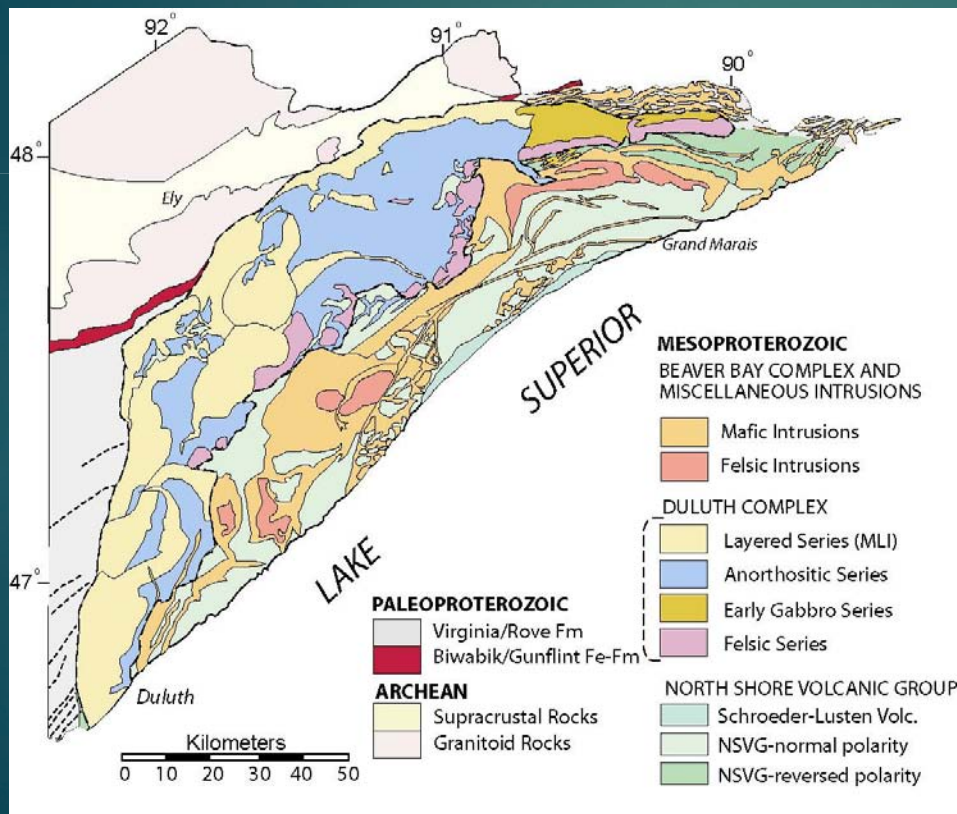
$a = 8.1768 \text{ \AA}$ $b = 12.8768 \text{ \AA}$

$c = 14.169 \text{ \AA}$

$\alpha = 93.17^\circ$, $\beta = 115.85^\circ$,

$\gamma = 92.22^\circ$

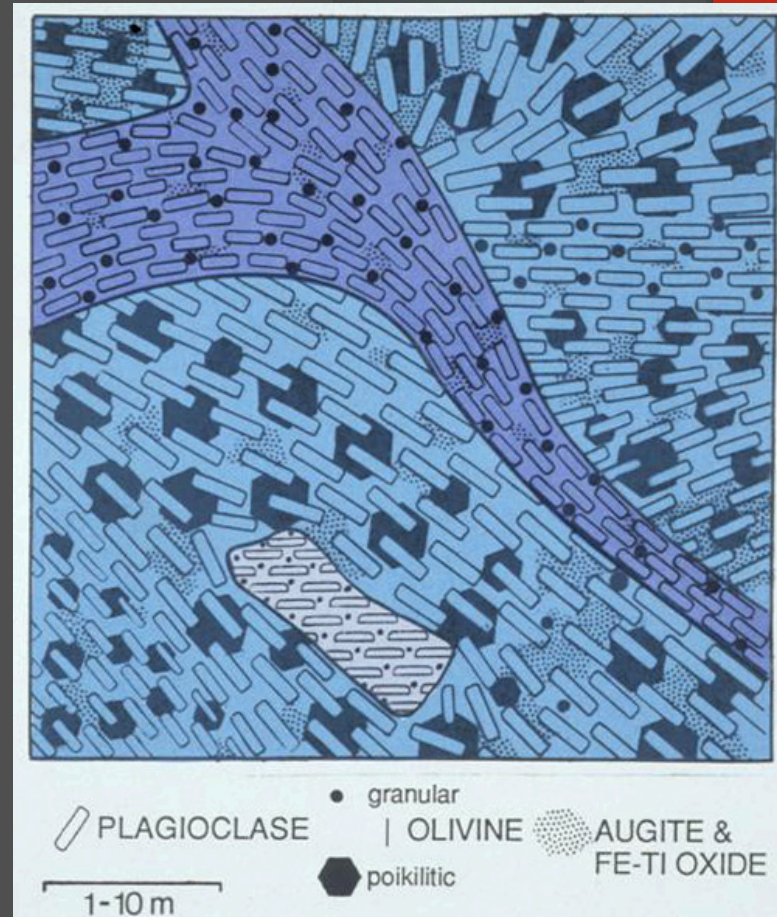
Duluth Complex Anorthositic



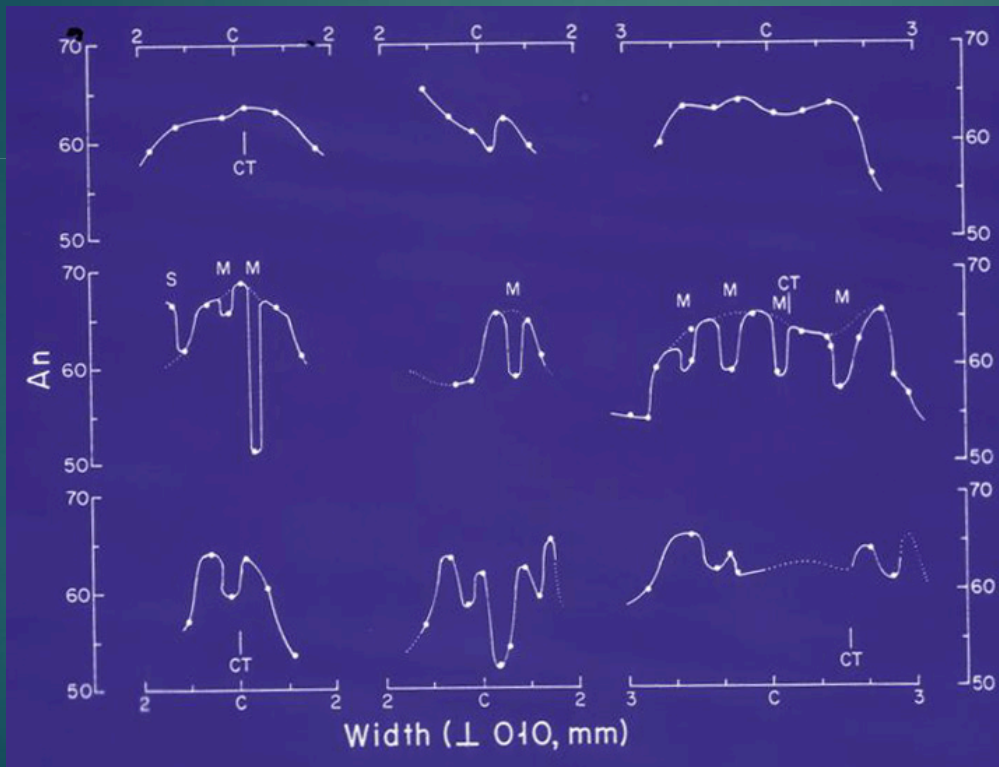
Courtesy of:
JIM MILLER
MINNESOTA GEOLOGICAL SURVEY
PRECAMBRIAN RESEARCH CENTER
UNIVERSITY OF MINNESOTA DULUTH

Anorthositic Series Texture

Courtesy of:
JIM MILLER
MINNESOTA GEOLOGICAL SURVEY
PRECAMBRIAN RESEARCH CENTER
UNIVERSITY OF MINNESOTA DULUTH




Plagioclase Zonation Patterns



Courtesy of:
JIM MILLER
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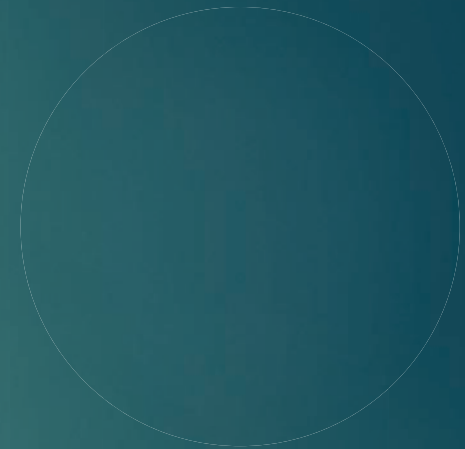
Hypothesis



- ▶ Is it possible to detect chemical changes within zoned plagioclase feldspars using point X-Ray diffraction (XRD)?
 - ▶ How does this method of analysis compare to Scanning Electron Microprobe/Energy-Dispersive X-ray Spectroscopy (SEM-EDS) chemical data?
- 

Methods

- ▶ Scanning Electron Microprobe/Energy-Dispersive X-ray Spectroscopy (SEM-EDS)
- ▶ Point X-ray diffraction (XRD)
- ▶ Chemical Analysis calculations in MS excel
- ▶ XRD analysis through X'Pert High Score



SEM equipment

- ▶ Polished samples were mounted using conductive XYZ tape (Ted Pella Inc., Redding, California) and then coated with a conductive layer of carbon in a high-vacuum evaporative coater (Cressington 208c, Ted Pella Inc., Redding, California). Images were obtained with a JEOL JSM-6490LV scanning electron microscope operating at 15 kV (JEOL USA Inc., Peabody, Massachusetts).

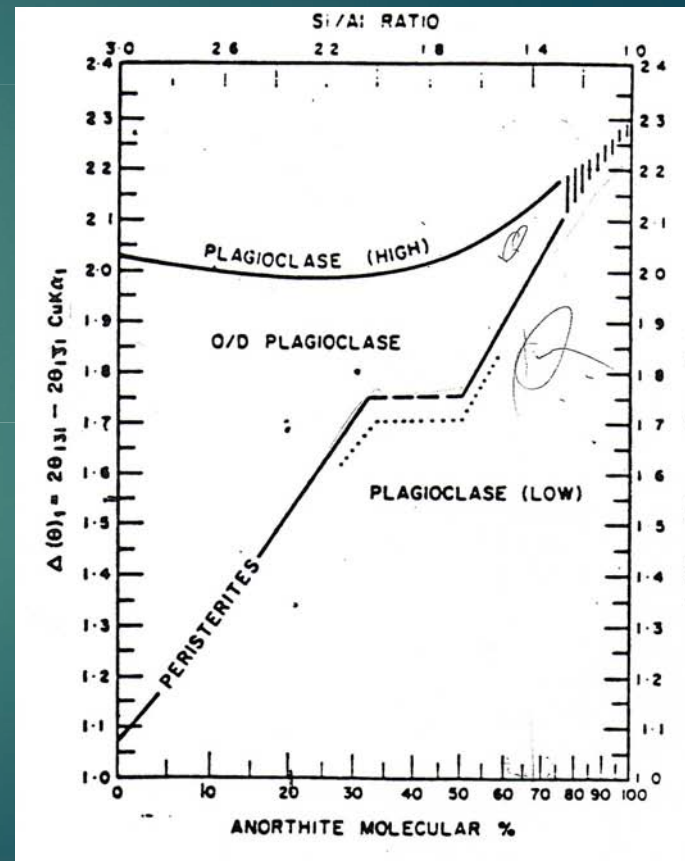
XRD equipment

- ▶ Analysis performed on a Bruker D8 Discover

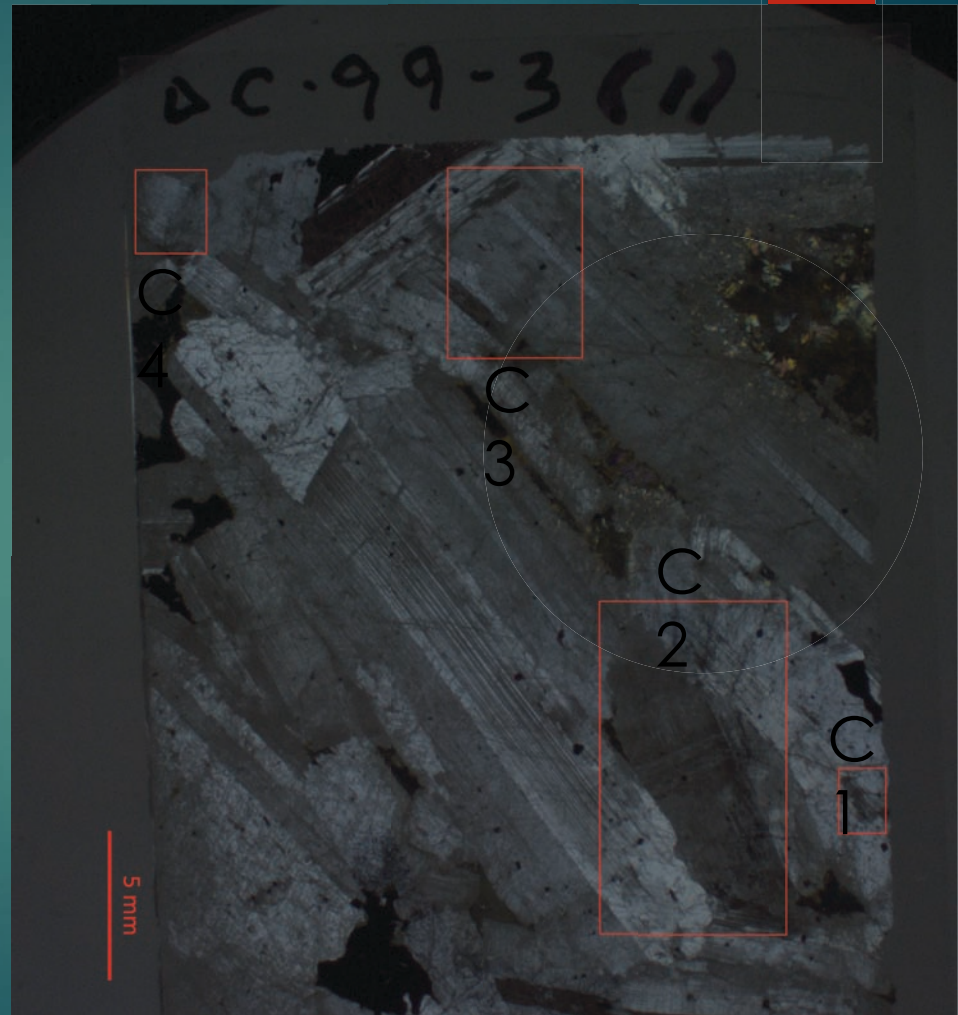


□ 131 XRD analysis method

- ▶ $\Delta 131 = 2\theta(131) - 2\theta(1\bar{3}1)$
- ▶ Typically occurs in the 2θ range of 29° to 32°

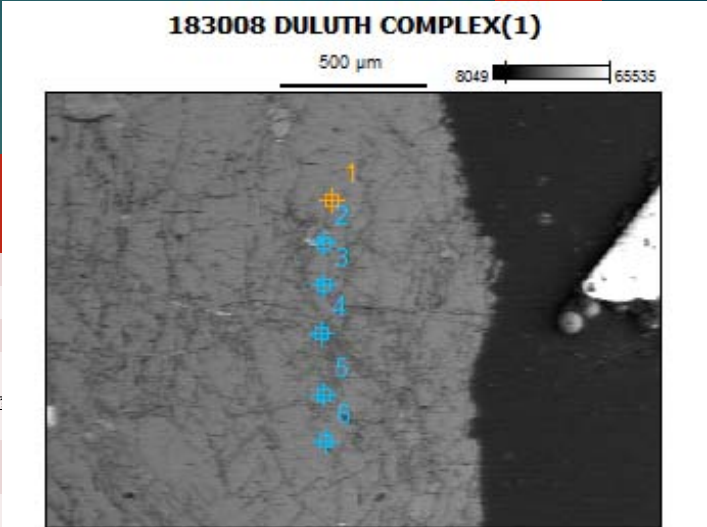


Crystal Locations



Crystal 1 SEM Data and Analysis

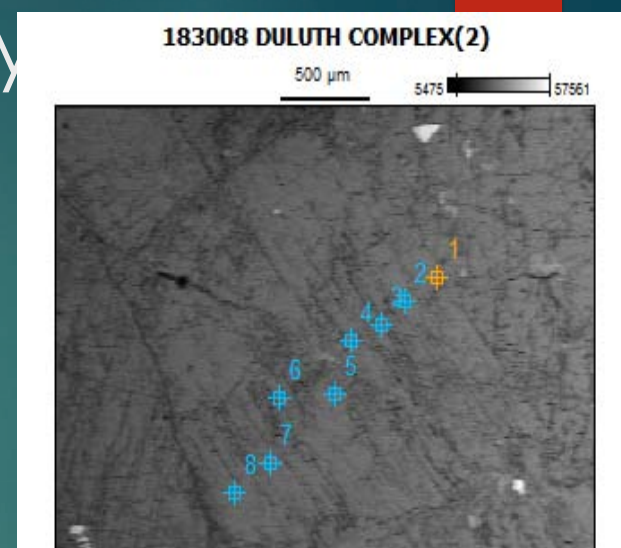
Point	Na (atom %)	Ca (atom %)	An %	Comment
1	2.77	2.33	2.778	
2	2.77	2.33	2.778	
3	2.77	2.33	2.778	
4	2.77	2.33	2.778	
5	2.77	2.33	2.778	
6	2.77	2.33	2.778	
7	2.77	2.33	2.778	
8	2.77	2.33	2.778	
9	2.77	2.33	2.778	
10	2.77	2.33	2.778	
11	2.77	2.33	2.778	
12	2.77	2.33	2.778	
13	2.77	2.33	2.778	
14	2.77	2.33	2.778	
15	2.77	2.33	2.778	
16	2.77	2.33	2.778	
17	2.77	2.33	2.778	
18	2.77	2.33	2.778	
19	2.77	2.33	2.778	
20	2.77	2.33	2.778	
21	2.77	2.33	2.778	
22	2.77	2.33	2.778	
23	2.77	2.33	2.778	
24	2.77	2.33	2.778	
25	2.77	2.33	2.778	
26	2.77	2.33	2.778	
27	2.77	2.33	2.778	
28	2.77	2.33	2.778	
29	2.77	2.33	2.778	
30	2.77	2.33	2.778	
31	2.77	2.33	2.778	
32	2.77	2.33	2.778	
33	2.77	2.33	2.778	
34	2.77	2.33	2.778	
35	2.77	2.33	2.778	
36	2.77	2.33	2.778	
37	2.77	2.33	2.778	
38	2.77	2.33	2.778	
39	2.77	2.33	2.778	
40	2.77	2.33	2.778	
41	2.77	2.33	2.778	
42	2.77	2.33	2.778	
43	2.77	2.33	2.778	
44	2.77	2.33	2.778	
45	2.77	2.33	2.778	
46	2.77	2.33	2.778	
47	2.77	2.33	2.778	
48	2.77	2.33	2.778	
49	2.77	2.33	2.778	
50	2.77	2.33	2.778	
51	2.77	2.33	2.778	
52	2.77	2.33	2.778	
53	2.77	2.33	2.778	
54	2.77	2.33	2.778	
55	2.77	2.33	2.778	
56	2.77	2.33	2.778	
57	2.77	2.33	2.778	
58	2.77	2.33	2.778	
59	2.77	2.33	2.778	
60	2.77	2.33	2.778	
61	2.77	2.33	2.778	
62	2.77	2.33	2.778	
63	2.77	2.33	2.778	
64	2.77	2.33	2.778	
65	2.77	2.33	2.778	
66	2.77	2.33	2.778	
67	2.77	2.33	2.778	
68	2.77	2.33	2.778	
69	2.77	2.33	2.778	
70	2.77	2.33	2.778	
71	2.77	2.33	2.778	
72	2.77	2.33	2.778	
73	2.77	2.33	2.778	
74	2.77	2.33	2.778	
75	2.77	2.33	2.778	
76	2.77	2.33	2.778	
77	2.77	2.33	2.778	
78	2.77	2.33	2.778	
79	2.77	2.33	2.778	
80	2.77	2.33	2.778	
81	2.77	2.33	2.778	
82	2.77	2.33	2.778	
83	2.77	2.33	2.778	
84	2.77	2.33	2.778	
85	2.77	2.33	2.778	
86	2.77	2.33	2.778	
87	2.77	2.33	2.778	
88	2.77	2.33	2.778	
89	2.77	2.33	2.778	
90	2.77	2.33	2.778	
91	2.77	2.33	2.778	
92	2.77	2.33	2.778	
93	2.77	2.33	2.778	
94	2.77	2.33	2.778	
95	2.77	2.33	2.778	
96	2.77	2.33	2.778	
97	2.77	2.33	2.778	
98	2.77	2.33	2.778	
99	2.77	2.33	2.778	
100	2.77	2.33	2.778	



Atom %	O-K	Na-K	Mg-K	Al-K	Si-K	S-K	Cl-K	K-K	Ca-K	Ti-K	Fe-K
183008 DULUTH COMPL EX(1)_p1	58.00	3.37		12.79	20.39		0.17	0.40	4.88		
183008 DULUTH COMPL EX(1)_p2	59.58	3.39		12.55	19.81			0.17	4.52		
183008 DULUTH COMPL EX(1)_p3	59.93	3.27		12.37	19.72			0.18	4.54		
183008 DULUTH COMPL EX(1)_p4	55.31	1.83	6.49	10.18	17.27		1.03	2.30	1.08		4.51
183008 DULUTH COMPL EX(1)_p5	52.65	2.51	0.34	13.99	21.72	0.22	1.32	3.46	2.93	0.42	0.44
183008 DULUTH COMPL EX(1)_p6	59.05	2.88		13.03	19.53			0.18	5.11		0.22

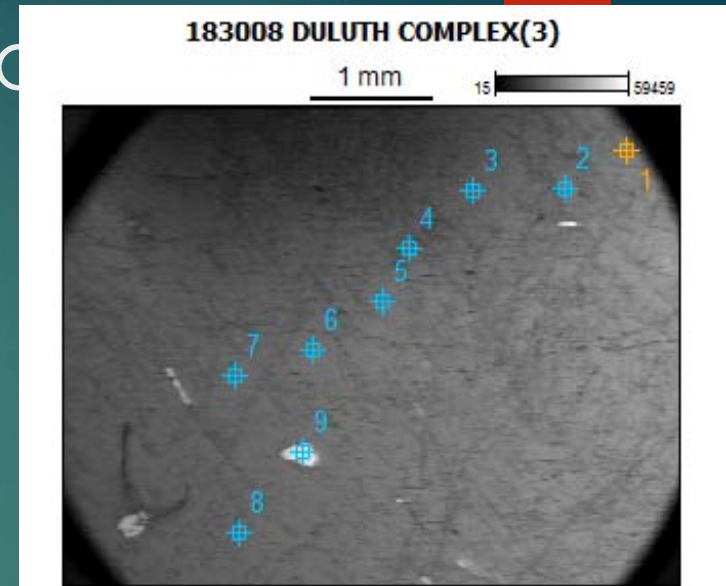
Crystal 2 SEM Data and Analy

Point	Na (atom %)	Ca (atom %)	An %	Comment
2	22.1	31.1	1.08	
2	23.3	27.3	1.08	
2	22.2	27.2	131.8	
2	22.2	27.2	1.08	
2	22.2	27.3	1.08	
1	22.2	27.2	1.08	
	22.1	27.2	1.08	
3	22.1	27.2	1.08	



Atom %	O-K	Na-K	Al-K	Si-K	Cl-K	K-K	Ca-K	Fe-K
183008 DULUTH C OMPLEX(2)_pt1	48.68	2.39	14.60	23.24	0.66	2.14	8.29	
183008 DULUTH C OMPLEX(2)_pt2	58.40	2.88	13.30	19.80		0.22	5.18	0.22
183008 DULUTH C OMPLEX(2)_pt3	59.02	2.43	13.24	19.36	0.12	0.51	5.33	
183008 DULUTH C OMPLEX(2)_pt4	59.07	2.95	12.87	19.88		0.29	4.94	
183008 DULUTH C OMPLEX(2)_pt5	58.99	3.15	12.75	19.99		0.24	4.89	
183008 DULUTH C OMPLEX(2)_pt6	60.27	3.15	12.46	19.20		0.22	4.70	
183008 DULUTH C OMPLEX(2)_pt7	59.86	2.99	12.75	19.42		0.18	4.80	
183008 DULUTH C OMPLEX(2)_pt8	59.95	3.09	12.66	19.44		0.16	4.70	

Crystal 3 SEM Data and Analysis

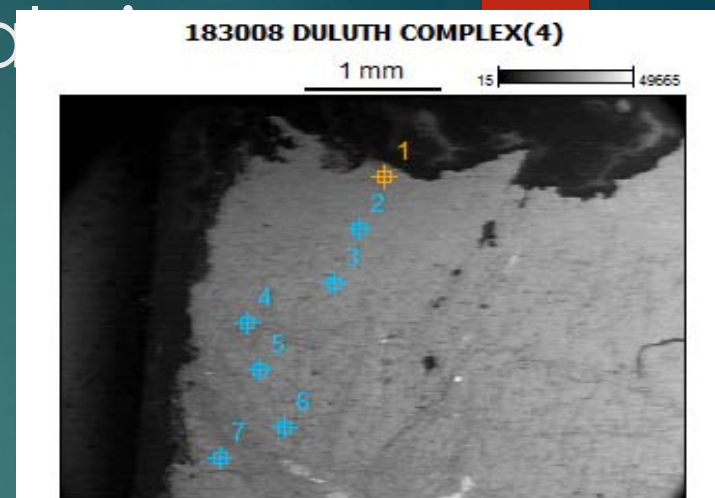


Point	Na (atom %)	Ca (atom %)	An %	Comment
2	27.7	27.3	27.7 28	
2	27.2	27.2	127.7 28	
2	27.1	27.1	227.7 28	
2	27.2	27.1	127.7 28	
2	27.1	27.3	2. 7 28	
1	27.2	27.2	127.7 28	
	27.2	27.1	127.7 28	
3	27.2	27.2	127.7 28	

	O-K	Na-K	Mg-K	Al-K	Si-K	Cl-K	K-K	Ca-K	Ti-K	Fe-K
183008 DULUTH COMPLEX(3)_pt1	59.85	3.37		12.20	19.72		0.24	4.28		0.34
183008 DULUTH COMPLEX(3)_pt2	59.95	2.92		12.55	19.61		0.32	4.65		
183008 DULUTH COMPLEX(3)_pt3	58.30	4.16		12.46	19.63	0.33	0.52	4.60		
183008 DULUTH COMPLEX(3)_pt4	59.49	3.14		12.67	19.68		0.17	4.86		
183008 DULUTH COMPLEX(3)_pt5	59.41	3.09		12.79	19.90		0.23	4.58		
183008 DULUTH COMPLEX(3)_pt6	59.31	2.95		12.74	19.83		0.23	4.74		0.20
183008 DULUTH COMPLEX(3)_pt7	60.46	3.00		12.32	19.25		0.30	4.67		
183008 DULUTH COMPLEX(3)_pt8	59.26	2.92		13.04	19.82			4.97		

Crystal 4 SEM Data and Analysis

Point	Na (atom %)	Ca (atom %)	An %	Comment
2	27.71	27.1		127778
2	28.2	27.2		127778
2	27.72	27.2		127778
2	27.3	28.3		127778
2	27.71	27.72		222328
1	28.2	27.2		11228
	22	22		127778



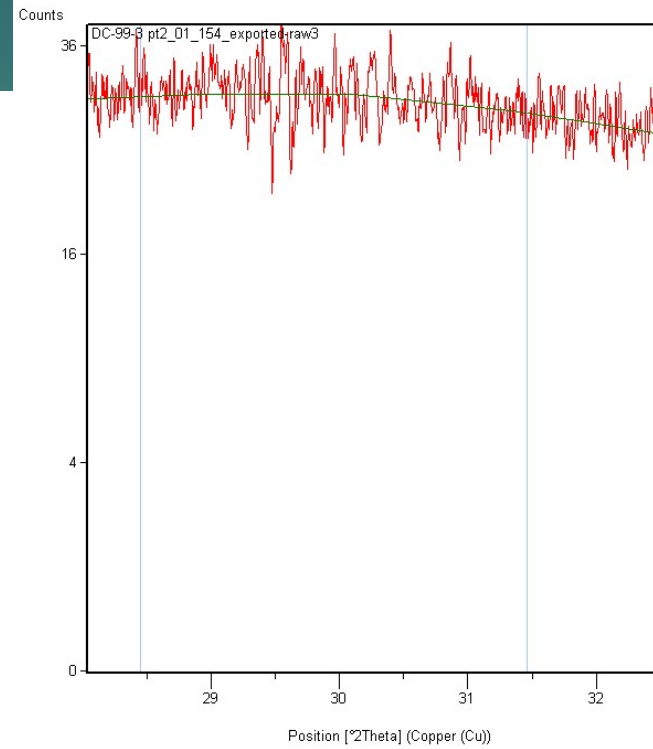
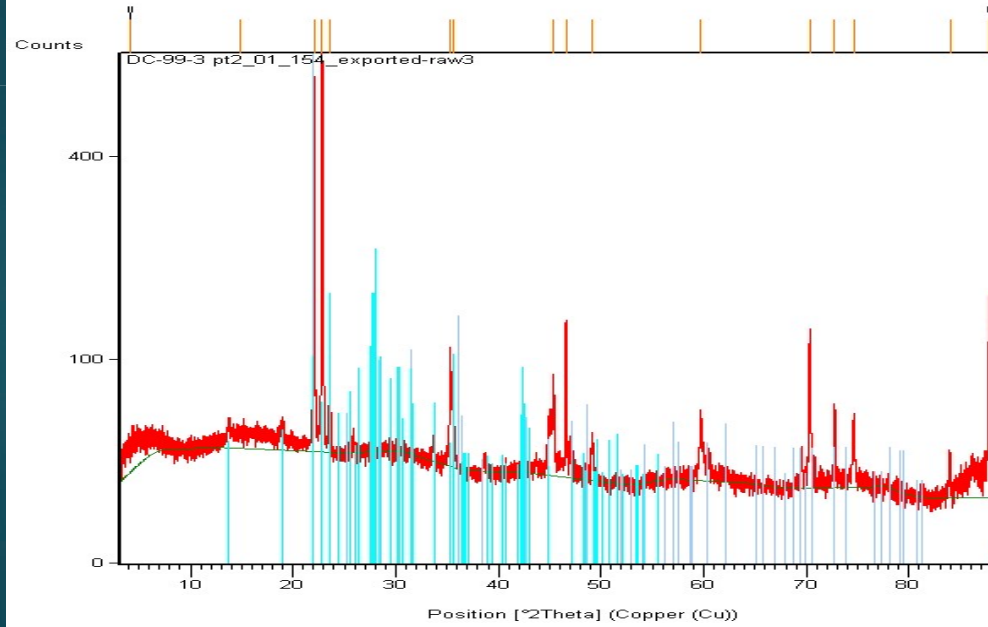
	O-K	Na-K	Mg-K	Al-K	Si-K	Cl-K	K-K	Ca-K	Fe-K
183008 DULUTH COMPLEX(4)_pt1	60.63	3.06		12.47	19.02		0.16	4.67	
183008 DULUTH COMPLEX(4)_pt2	60.54	2.84		12.48	19.29		0.20	4.64	
183008 DULUTH COMPLEX(4)_pt3	60.46	3.12		12.26	19.22		0.23	4.72	
183008 DULUTH COMPLEX(4)_pt4	50.83	3.48	0.64	13.46	22.27	0.69	0.76	5.88	2.00
183008 DULUTH COMPLEX(4)_pt5	60.48	3.56		11.79	19.78		0.24	4.15	
183008 DULUTH COMPLEX(4)_pt6	57.67	2.83		12.99	20.25		0.54	5.72	
183008 DULUTH COMPLEX(4)_pt7	61.05	2.70		12.48	18.83		0.24	4.70	

Graphical Trend of the Plagioclase Composition

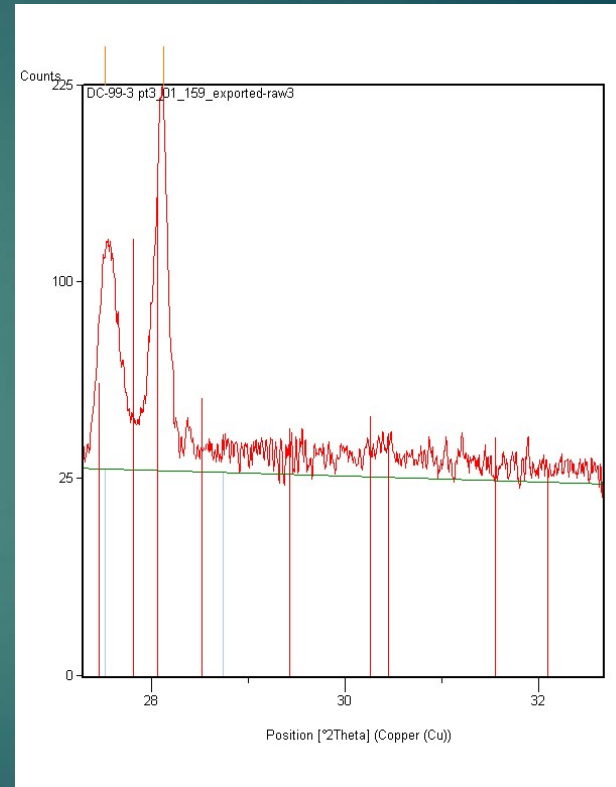
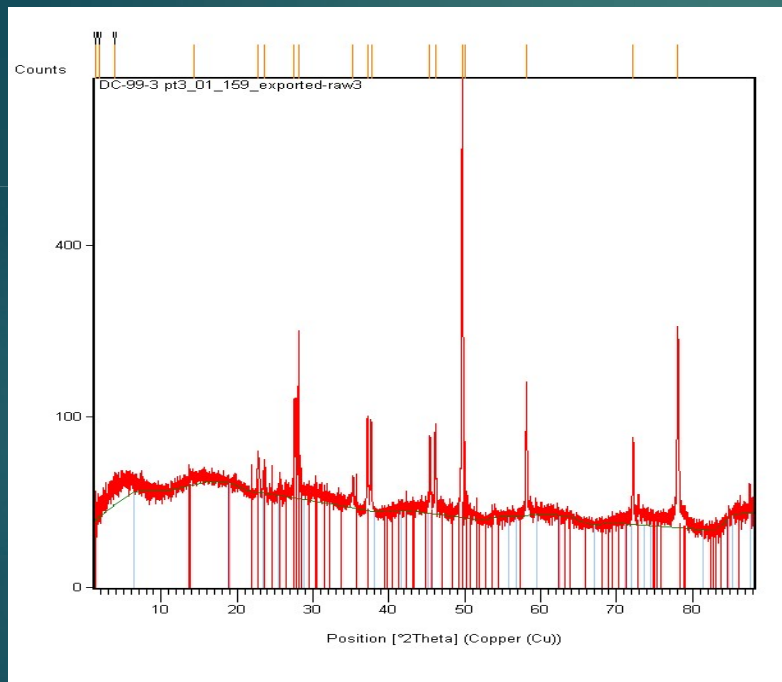
Anorthite (An) Composition



XRD Point 2



XRD Point 3



Conclusion

- ▶ Changes in chemical composition minimal across the crystals
- ▶ Point XRD to dependent on crystal orientation to detect differences in chemical composition across the crystals

Acknowledgements



- ▶ In publications and presentations, please acknowledge the NDSU Electron Microscopy Center core facility and include the following statement as required by the National Science Foundation:

This material is based upon work supported by the National Science Foundation under Grant No. 0619098 and 0923354.

