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).

TRENT OLSON

PETROLOGY

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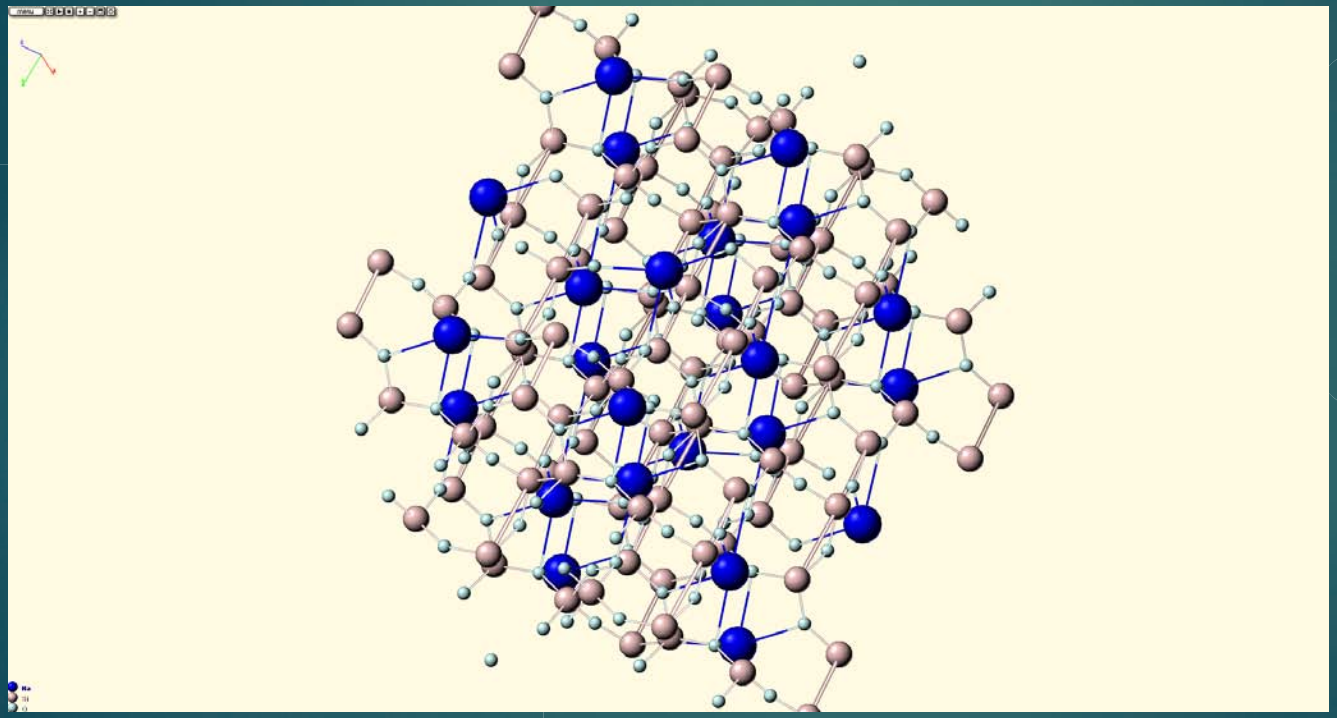
Outline

- Introduction
- Methods
- Crystal analyses
- Conclusions

Plagioclase

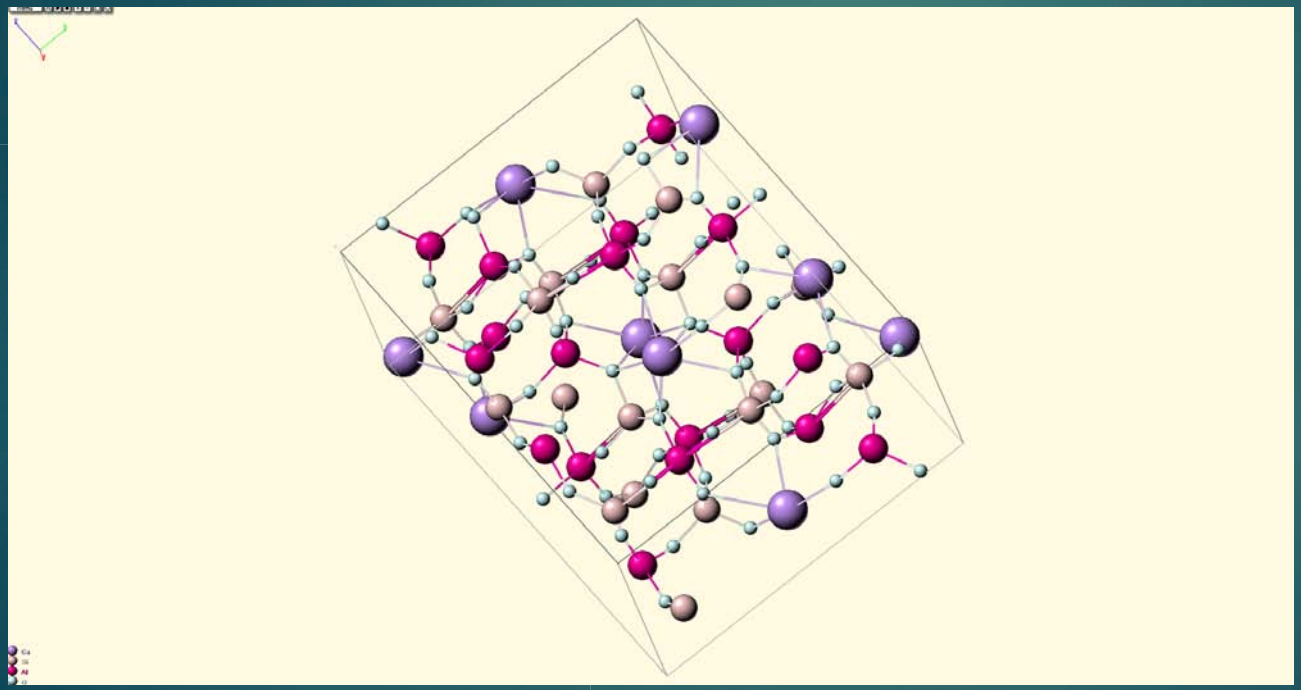
- Complete solid solution of $NaAlSi_3O_8$ (albite) to $CaAl_2Si_2O_8$ (anorthite)
- Zonation prevalent due to substitution chemistry
- Crystal Structure distorts depending on chemistry

Albite Structure



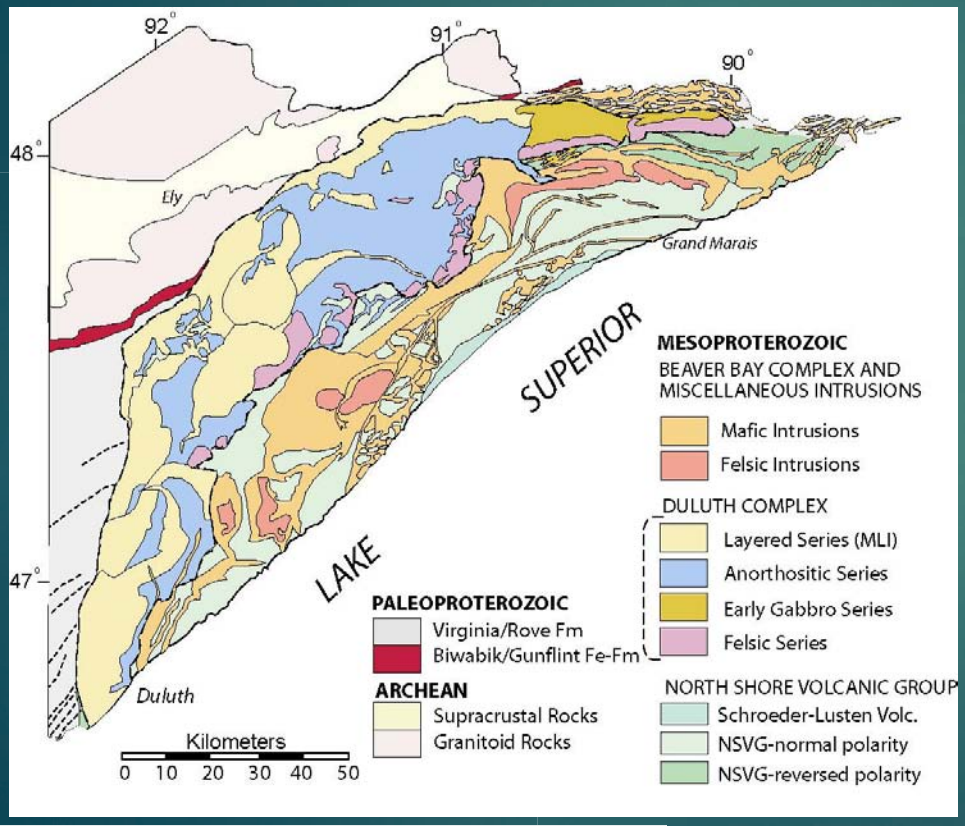
Space Group: C1
Cell Parameters:
a = 8.16 □, b = 12.87 □,
c = 7.11 □
□ = 93.45°, □ = 116.4°,
□ = 90.28°

Anorthite Structure



Space Group: *P*1
Cell Parameters:
a = 8.1768 Å, b = 12.8768 Å,
c = 14.169 Å
α = 93.17°, β = 115.85°,
γ = 92.22°

Duluth Complex Anorthosites



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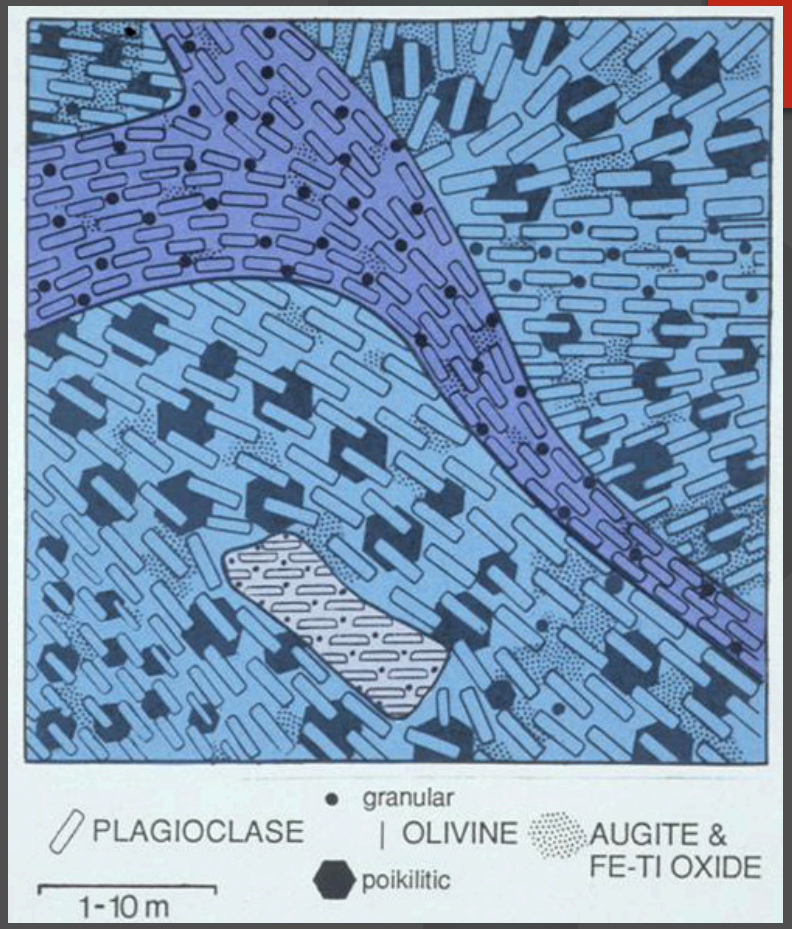
Anorthositic Series Texture



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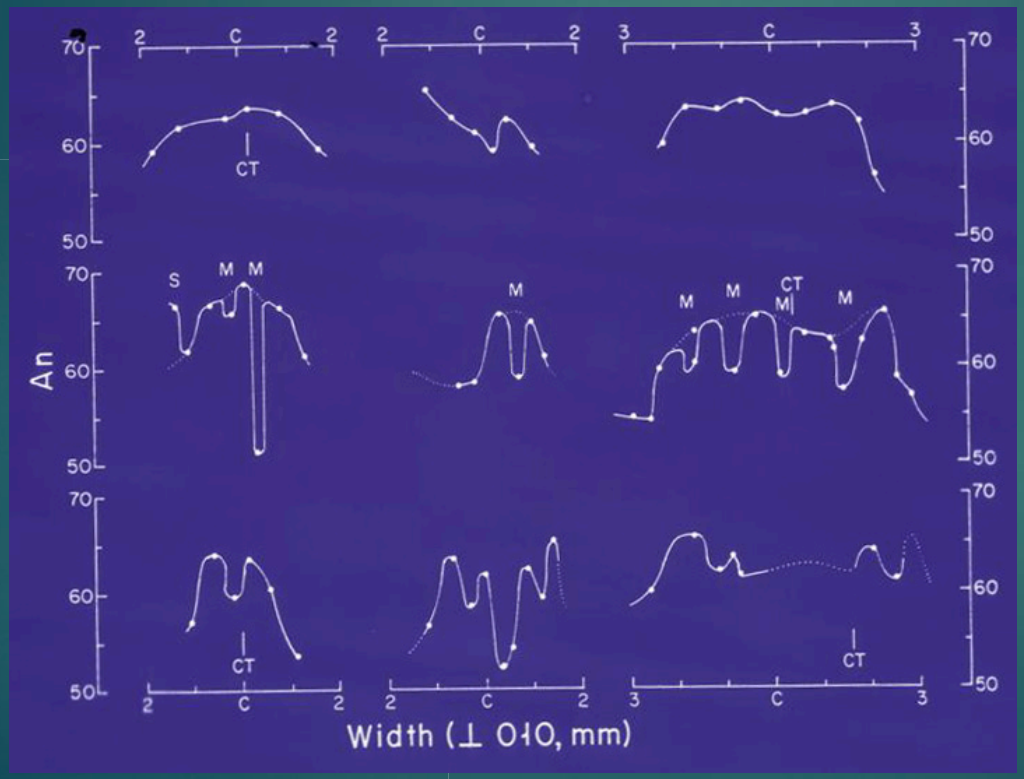
Plagioclase Zonation Patterns



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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

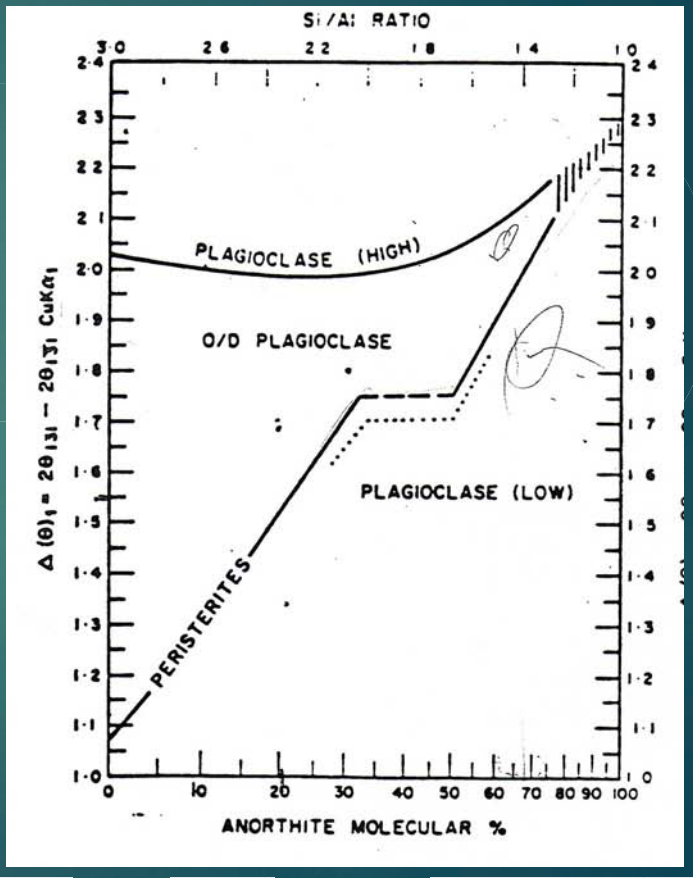
- Polished samples were mounted using conductive XYZ tape (Ted Pella Inc., Redding, California) and then coated with a conductive layer of carbon in a high-vacuum evaporative coater (Cressington 208c, Ted Pella Inc., Redding, California). Images were obtained with a JEOL JSM-6490LV scanning electron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

XRD equipment

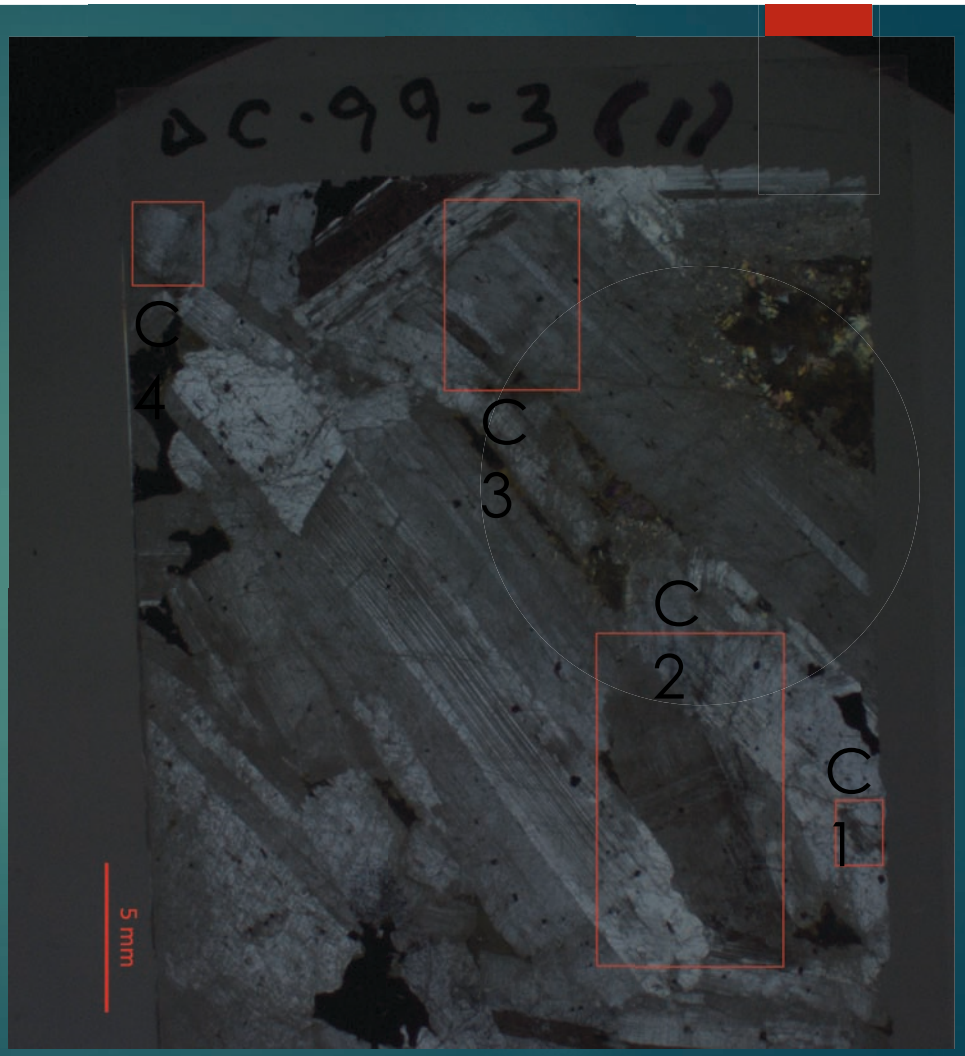
- Analysis preformed on a Bruker D8 Discover

□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°

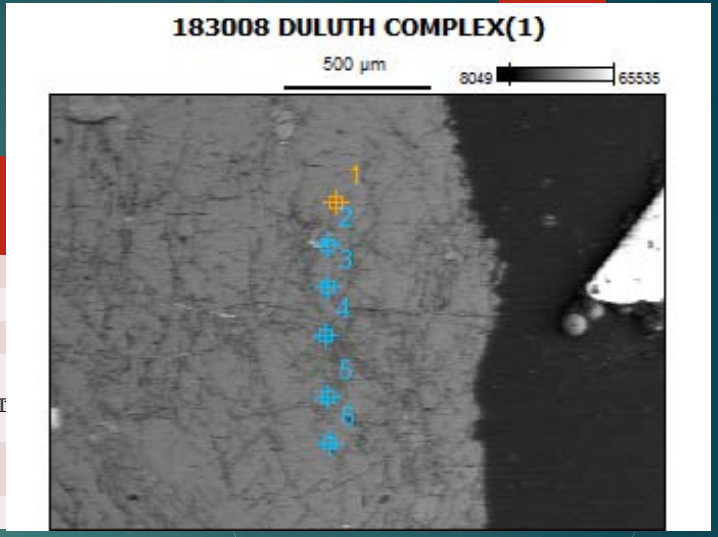


Crystal Locations



Crystal 1 SEM Data and Analysis

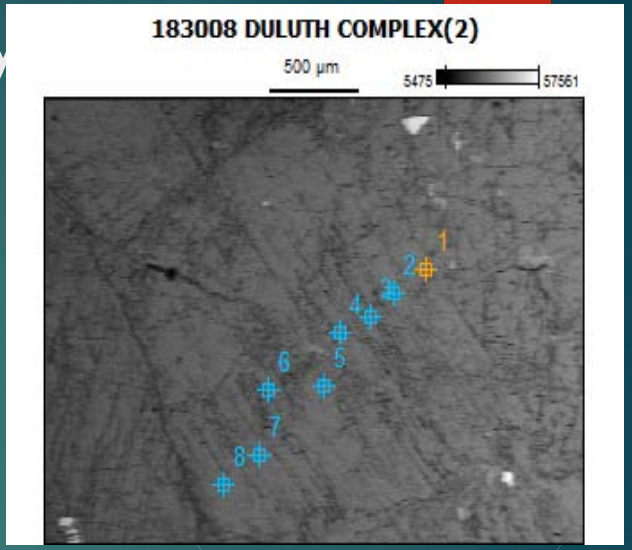
Point	Na (atom %)	Ca (atom %)	An %	Comment



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 COMPLEX(1)_pt1	58.00	3.37		12.79	20.39		0.17	0.40	4.88		
183008 DULUTH COMPLEX(1)_pt2	59.58	3.39		12.55	19.81			0.17	4.52		
183008 DULUTH COMPLEX(1)_pt3	59.93	3.27		12.37	19.72			0.18	4.54		
183008 DULUTH COMPLEX(1)_pt4	55.31	1.83	6.49	10.18	17.27		1.03	2.30	1.08		4.51
183008 DULUTH COMPLEX(1)_pt5	52.65	2.51	0.34	13.99	21.72	0.22	1.32	3.46	2.93	0.42	0.44
183008 DULUTH COMPLEX(1)_pt6	59.05	2.88		13.03	19.53			0.18	5.11		0.22

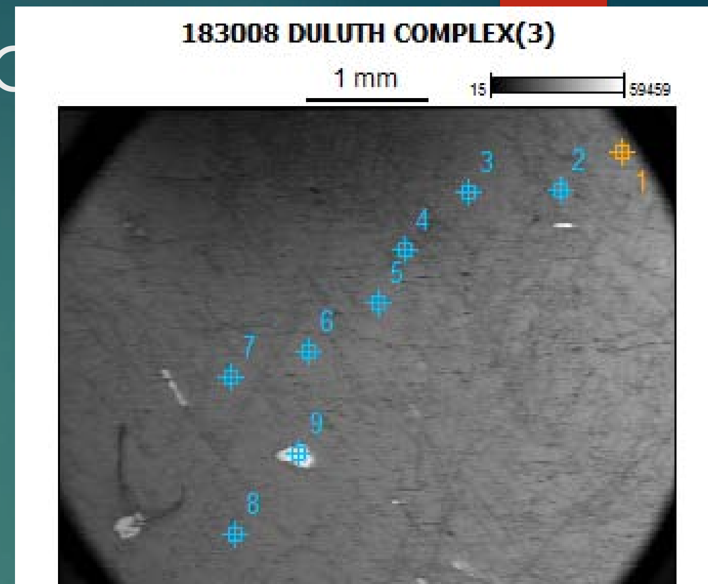
Crystal 2 SEM Data and Analy

Point	Na (atom %)	Ca (atom %)	An %	Comment
□	□□□.	3□□.	□1□8	
□	□□33	□□□3	1□□□ 8	
□	□□□□	□□□□	13□1. 8	
□	□□ □	□□ □	1□□1□8	
□	□□□□	□□3.	1□□3□8	
1	□□□□	□□	□. □3 8	
	□□ .	□□3	1□□1□8	
3	□□□.	□□	1□□□□8	



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

Crystal 3 SEM Data and Ana

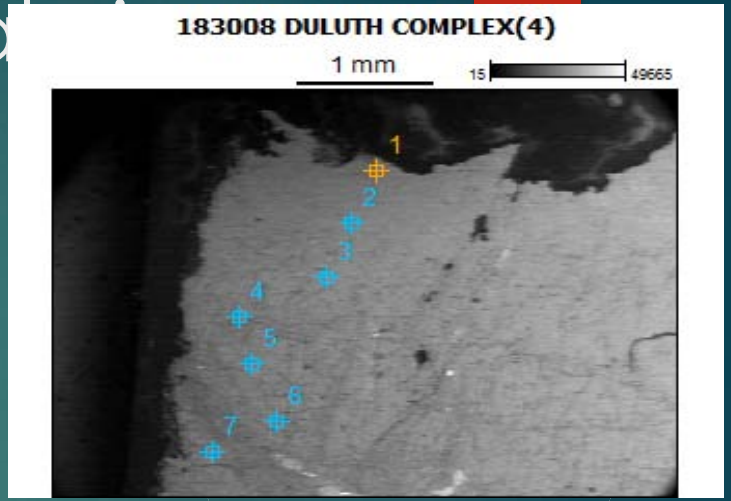


Point	Na (atom %)	Ca (atom %)	An %	Comment
□	□□	□□3	□□□ □8	
□	□□ □	□□1□	1□□□8	
□	□□1	□□1	□□□□8	
□	□□□	□□1	1□□ □8	
□	□□.	□□3	□. □ □8	
1	□□ □	□□ □	1□□1□8	
	□	□□1	1□□3. 8	
3	□□ □	□□	1□□ . 8	

	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		

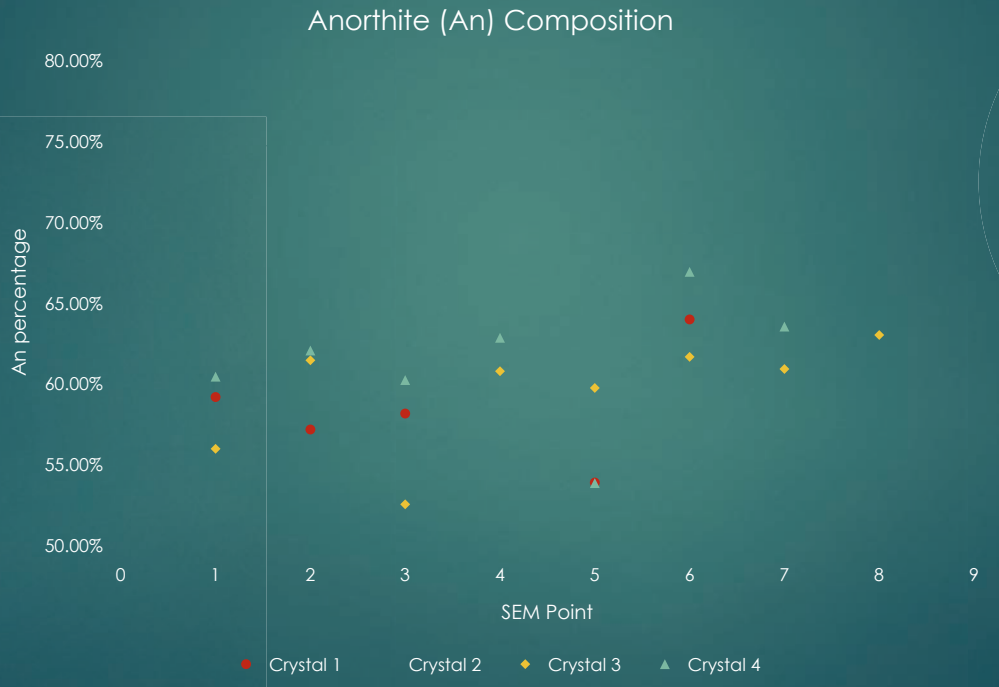
Crystal 4 SEM Data and Ana

Point	Na (atom %)	Ca (atom %)	An %	Comment

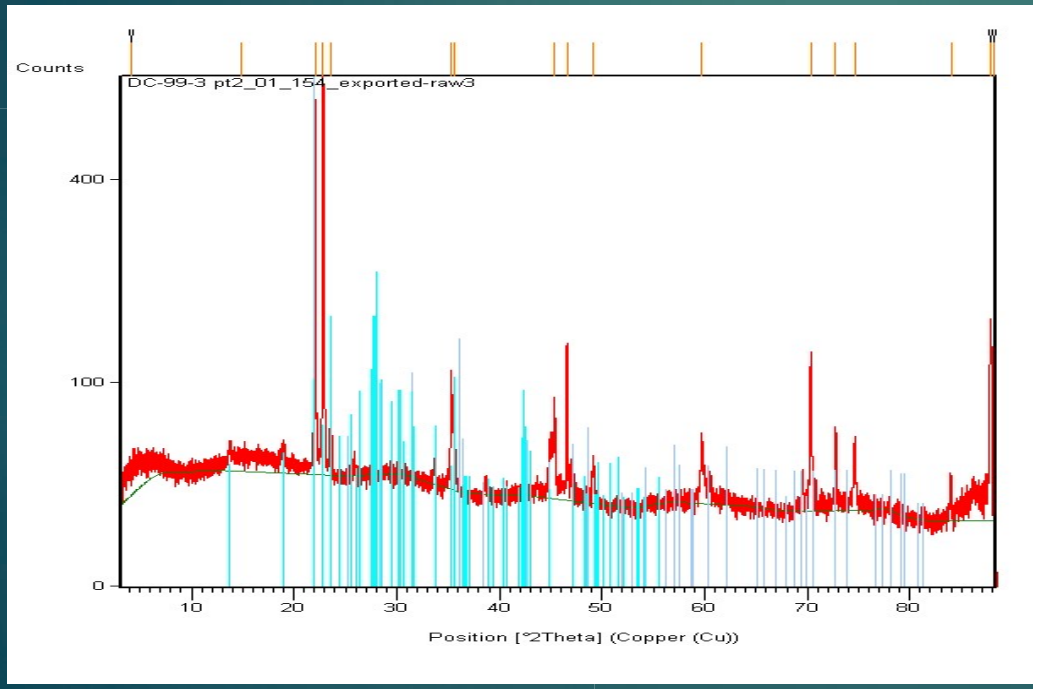


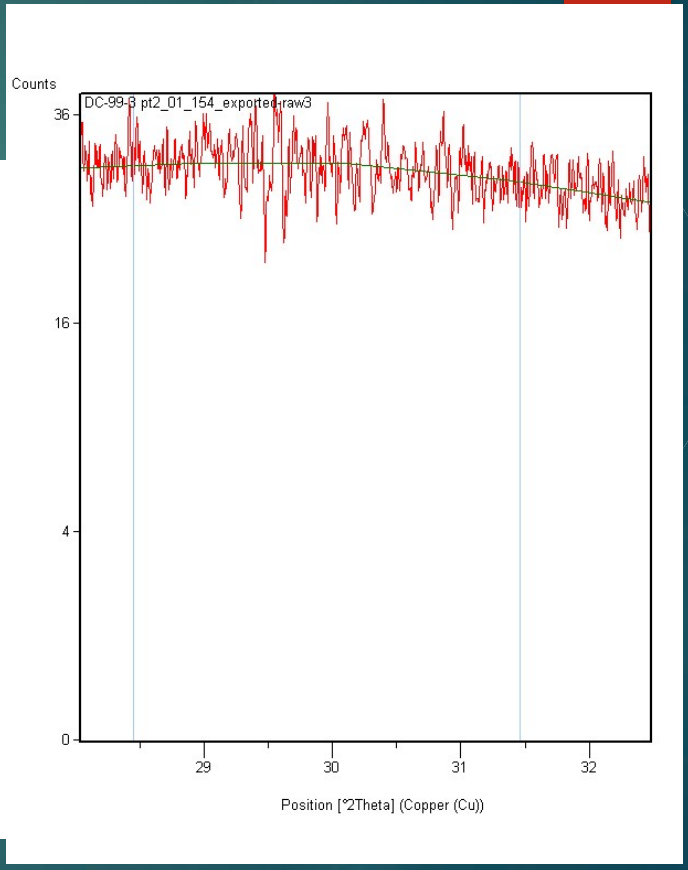
	O-K	Na-K	Mg-K	Al-K	Si-K	Cl-K	K-K	Ca-K	Fe-K
183008 DULUTH COMPLEX(4)_pt1	60.63	3.06		12.47	19.02		0.16	4.67	
183008 DULUTH COMPLEX(4)_pt2	60.54	2.84		12.48	19.29		0.20	4.64	
183008 DULUTH COMPLEX(4)_pt3	60.46	3.12		12.26	19.22		0.23	4.72	
183008 DULUTH COMPLEX(4)_pt4	50.83	3.48	0.64	13.46	22.27	0.69	0.76	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

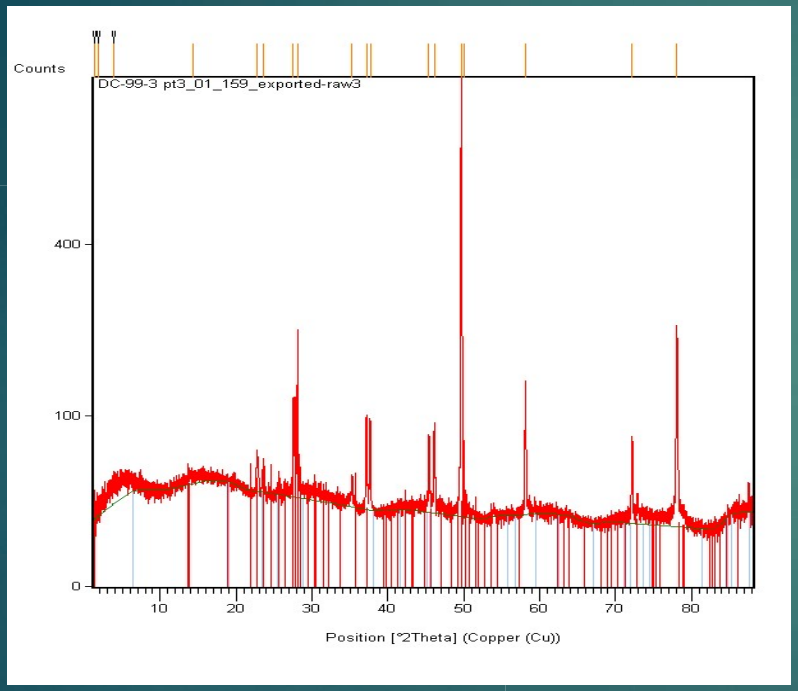


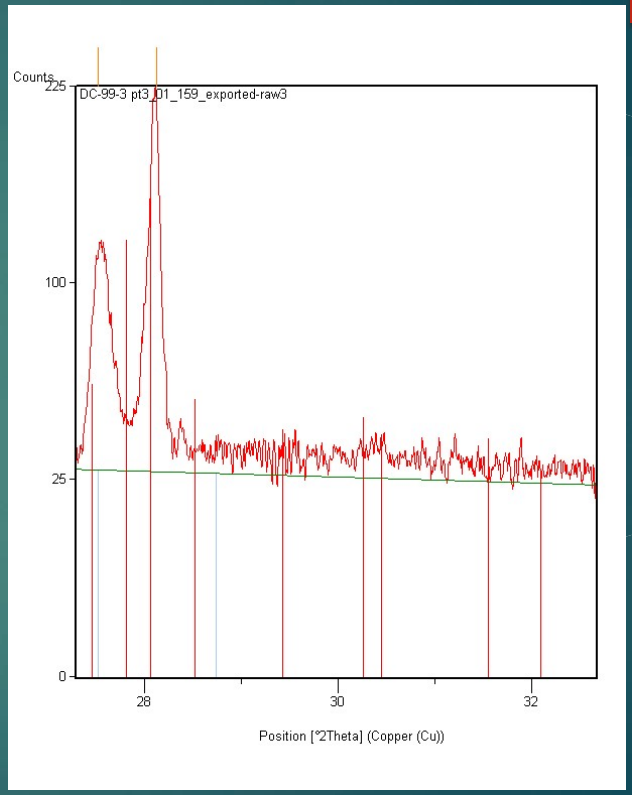
XRD Point 2


Counts
DC-99-3 pt2_01_154_exported-raw3
400
100
0
10
20
30
40
50
60
70
80
Position [°2Theta] (Copper (Cu))


Counts
DC-99-3 pt2_01_154_exported-raw3
36
16
4
0
29
30
31
32
Position [°2Theta] (Copper (Cu))

XRD Point 3


Counts
DC-99-3 pt3_01_159_exported-raw3
400
100
0
10
20
30
40
50
60
70
80
Position [°2Theta] (Copper (Cu))


Counts
225
DC-99-3 pt3_01_159_exported-raw3
100
25
0
28
30
32
Position [°2Theta] (Copper (Cu))

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

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