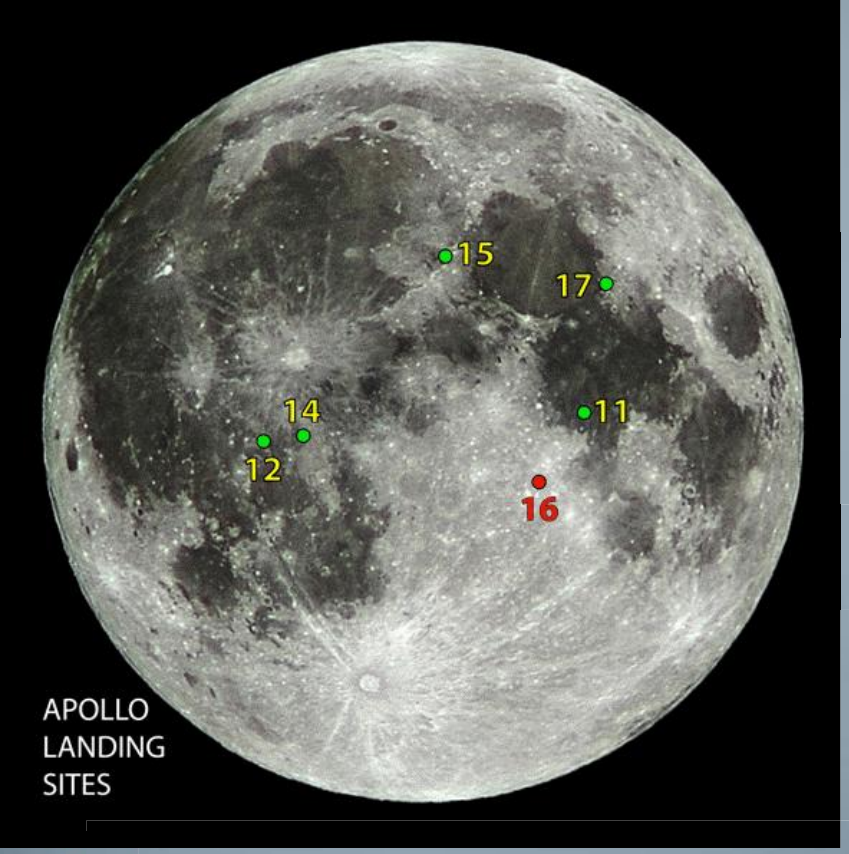
Production of Mineral Separates for Use in Lunar Simulants

Jacqueline Graham
Danielle Granlund
Janet Schweizerhof
Peter Rossiter

- Background
- Lunar Soil Mineralogy
- Project Statements & Highlights
- General Approach
- Geology of Stillwater Complex
- Available Technologies
- Current Results
- Conclusions
- Future Work
- Acknowledgements

Background

- **Lunar Soil (regolith) is extremely-fine, dust-like material**
- **All systems that go to the Moon will interact with this material, both intentionally and unintentionally:**
  - Can it be used for construction purposes?
  - Can it be used to grow plants?
  - Will it affect human health?
  - Is it detrimental to machinery, equipment, clothes, etc.?



Background

- In order to colonize the Moon, it must be known how the Lunar Soil will interact with the various systems
- Unfortunately, the Apollo missions did not bring back enough sample to study and characterize fully

- Emphasis has therefore been placed on making, studying, and characterizing "simulants" such as:
  - Minnesota Lunar Simulant (MLS-1) was developed in the 1970s and 1980s
  - Johnson Space Center (JSC-1) in 1990's
  - MLS-2, JSC-1A, JSC-1F, JSC-2, JSC-3, etc. were developed later but, like the others, all were consumed in various studies

Background

- International interest gains strength and a new "moon race" develops:
  - Japan (FJS-1 and MKS-1)
  - China (CAS-1)
- Previous studies were centered on using single resources of terrestrial igneous rock, predominantly from volcanic areas, with a focus on the elemental content and not necessarily the mineralogy and chemistry
- Most of the studies took these materials, added fluxes, heated to a molten state, allowed to solidify, and, after cooling, crushed, ground and pulverized the material
- Consequently, no simulant has accurately and adequately reproduced Lunar Soil

Background

"Simulants of lunar (rocks and) soils with appropriate properties, although difficult to produce in some cases, will be essential to meeting the system requirements for lunar exploration."

- McKay and Blacic (1991)

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Lunar Soil Mineralogy

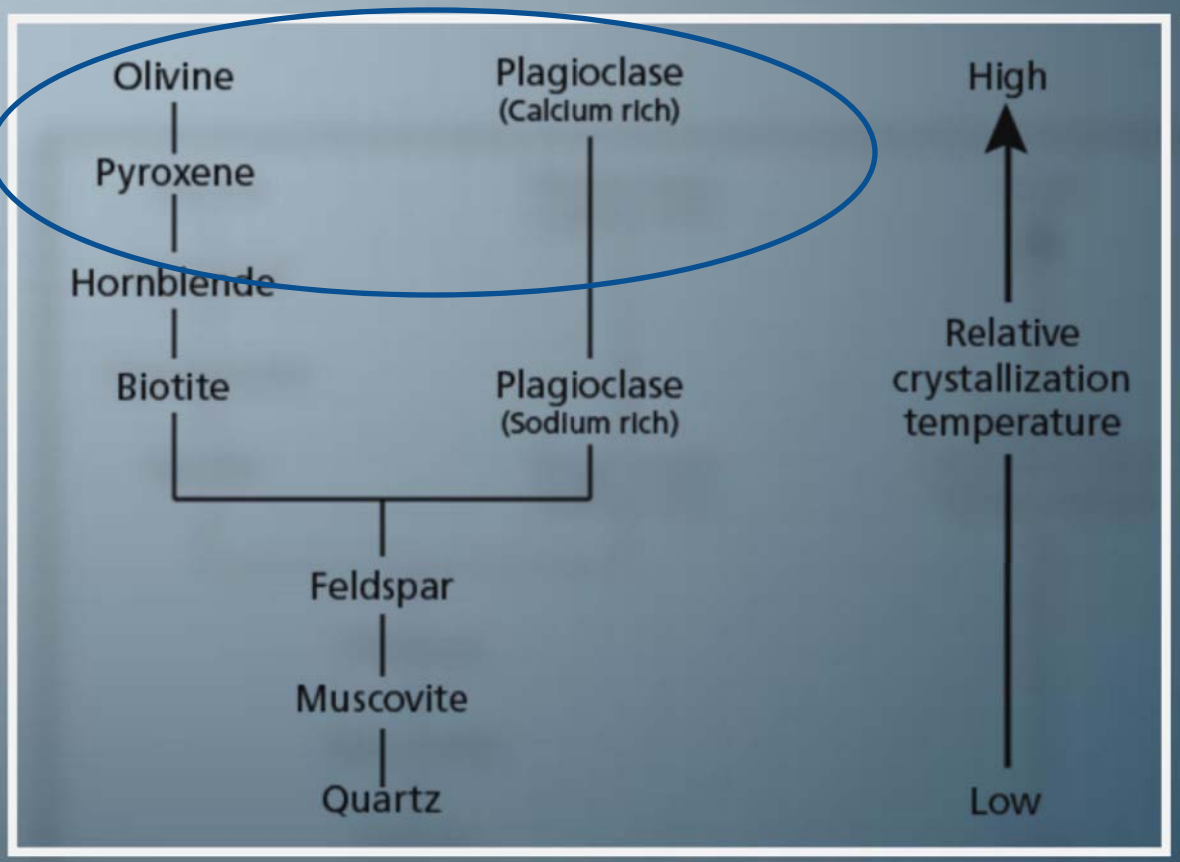
- **Over 95% of Lunar Soil is comprised of silicates belonging to 4 major mineral groups:**
  - High calcium Plagioclase [$(Na,Ca)(Si,Al)_4O_8$]
  - Orthopyroxene (Enstatite) [$(Mg,Fe,Ca)(Mg,Fe,Al)Si_2O_6$]
  - Clinopyroxene (Augite) [$(Ca,Na)(Mg,Fe)(Si,Al)_2O_6$]
  - Olivine [$(Mg,Fe)_2SiO_4$]
- **There are over 100 other minerals present, mostly oxides in minor amounts, including:**
  - Rutile (TiO_2)
  - Ilmenite ($FeTiO_3$)
  - Chromite ($FeCr_2O_4$)

Lunar Soil Mineralogy

Bowen's Reaction Series

Mafic

Felsic



Lunar Soil Mineralogy

Lunar Plagioclase

Name	An%	Ab%
Anorthite	90-100	0-10
Bytownite	70-90	10-30
Labradorite	50-70	30-50
Andesine	30-50	50-70
Oligoclase	10-30	70-90
Albite	0-10	90-100

Lunar Soil Mineralogy

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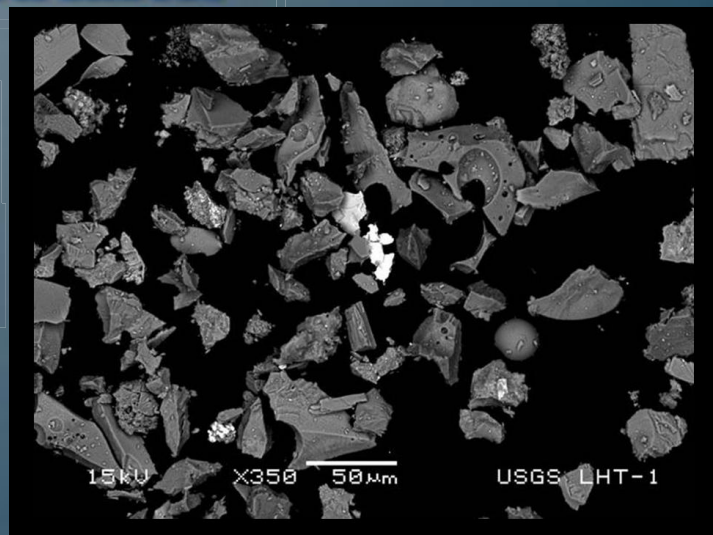
Project Statements and Highlights

- Develop a simulant by producing mineral "separates" from multiple resources
- Mix the separates in the appropriate ratios to match both the physical and chemical properties of the Lunar Soil
- Find appropriate resources at existing and permitted mines
- Develop processes to concentrate and purify the minerals into the "separates"
- Initial focus to be on the four major minerals and later on the minor minerals

LHT-2M

Project Statements and Highlights

- Making separates would allow impurities to be removed thereby allowing many terrestrial resources to be considered
- Mineral separates would allow bulk processing of rocks to make high grade feed stocks for later grinding and mixing
- Larger quantities would be produced faster
- Tremendous savings in time, labor and costs would be realized because the "pyrometallurgical" (i.e., thermal) processes would be avoided



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General Approach

Locate and analyze source material

- Identify appropriate terrestrial resources
- Obtain representative samples
- Determine the mineral content
- Develop tables listing the physical and chemical properties of the minerals

General Approach

Develop Production Processes

- Conceptualize processes that would produce separates using traditional mineral processing and extractive metallurgy techniques
- Conduct laboratory tests to evaluate the processes
- Design flow sheets with cost-estimates such that large amounts of separates ranging in size from 10 to 100 tons could be produced

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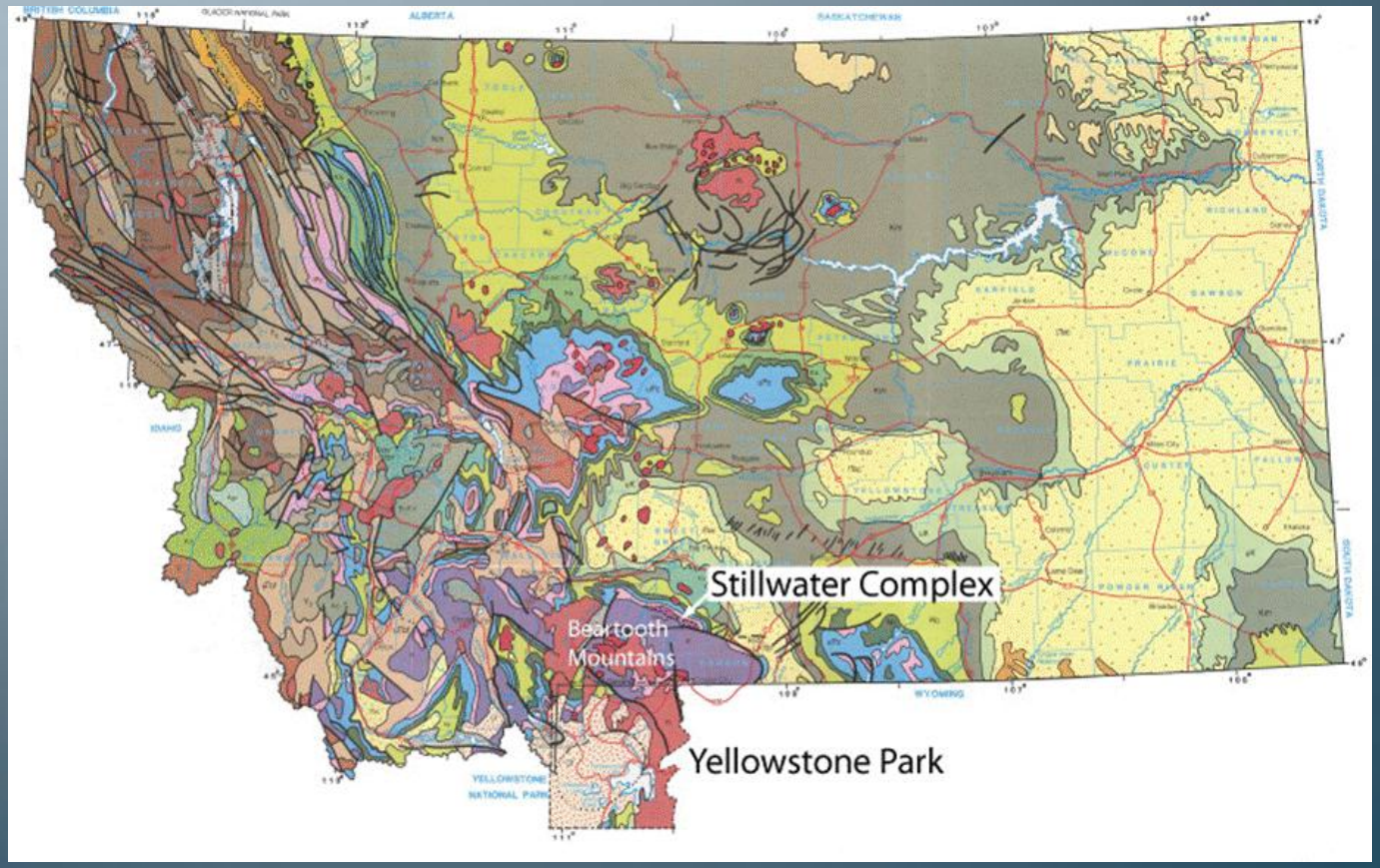
Geology of Stillwater Igneous Complex

Janet Schweizerhof

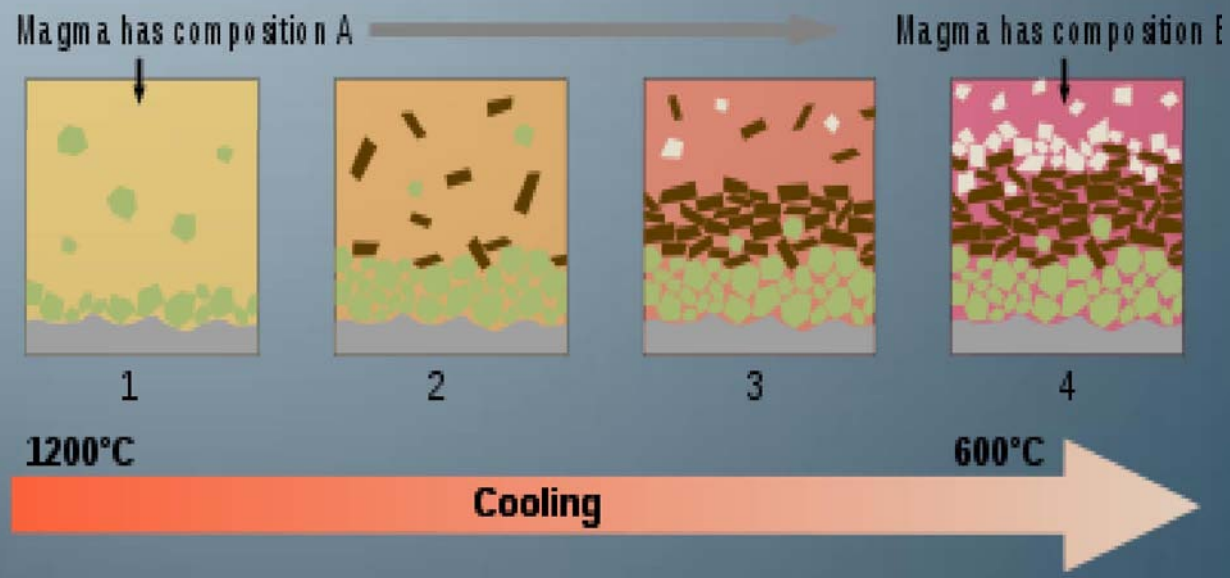


Terrestrial Counterparts

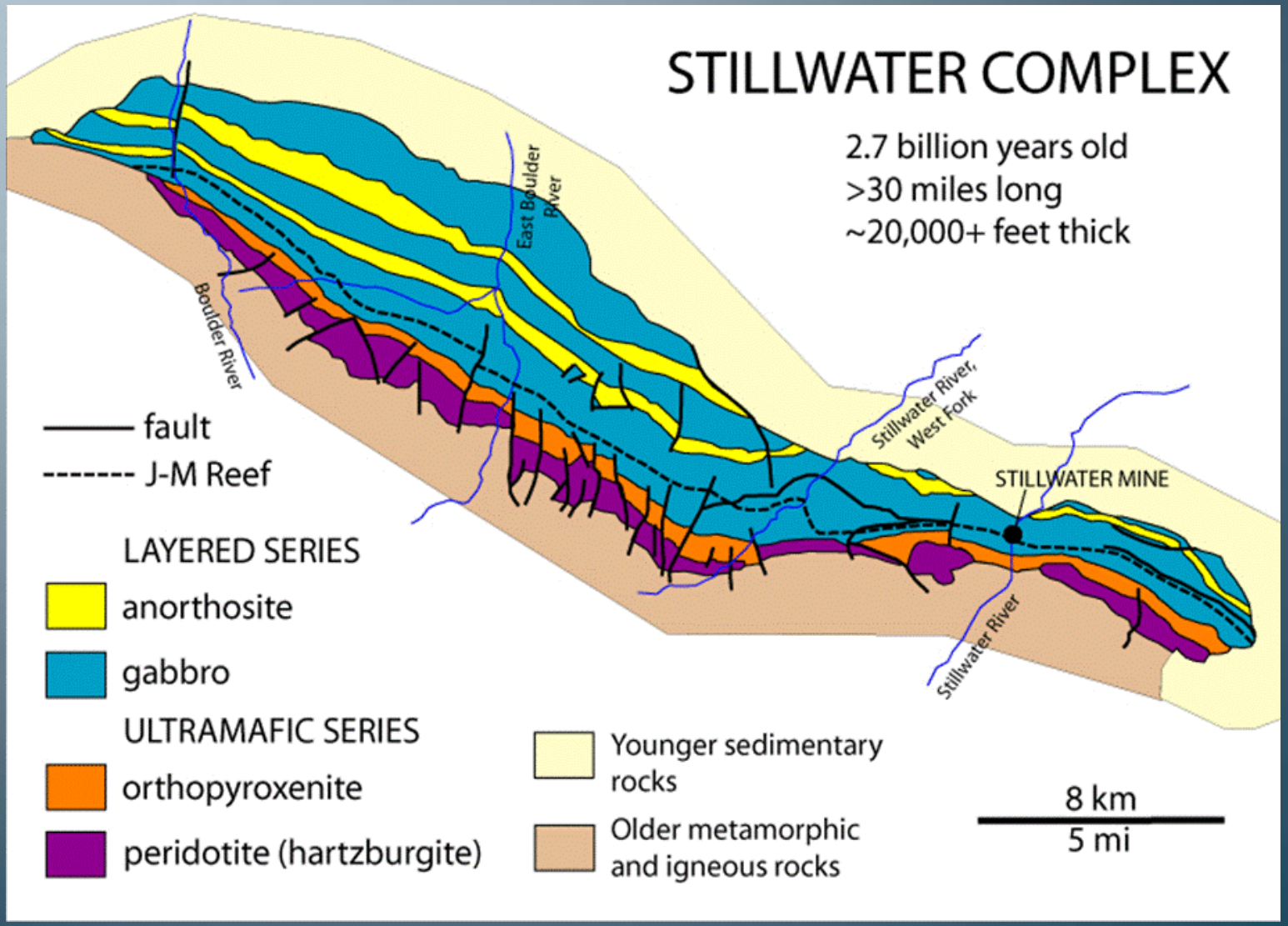
- The search for rocks similar to lunar rocks
- Archean Anorthosite
- Rare on earth
- Only formed during a particular era of earth's history

Fractional Crystallization

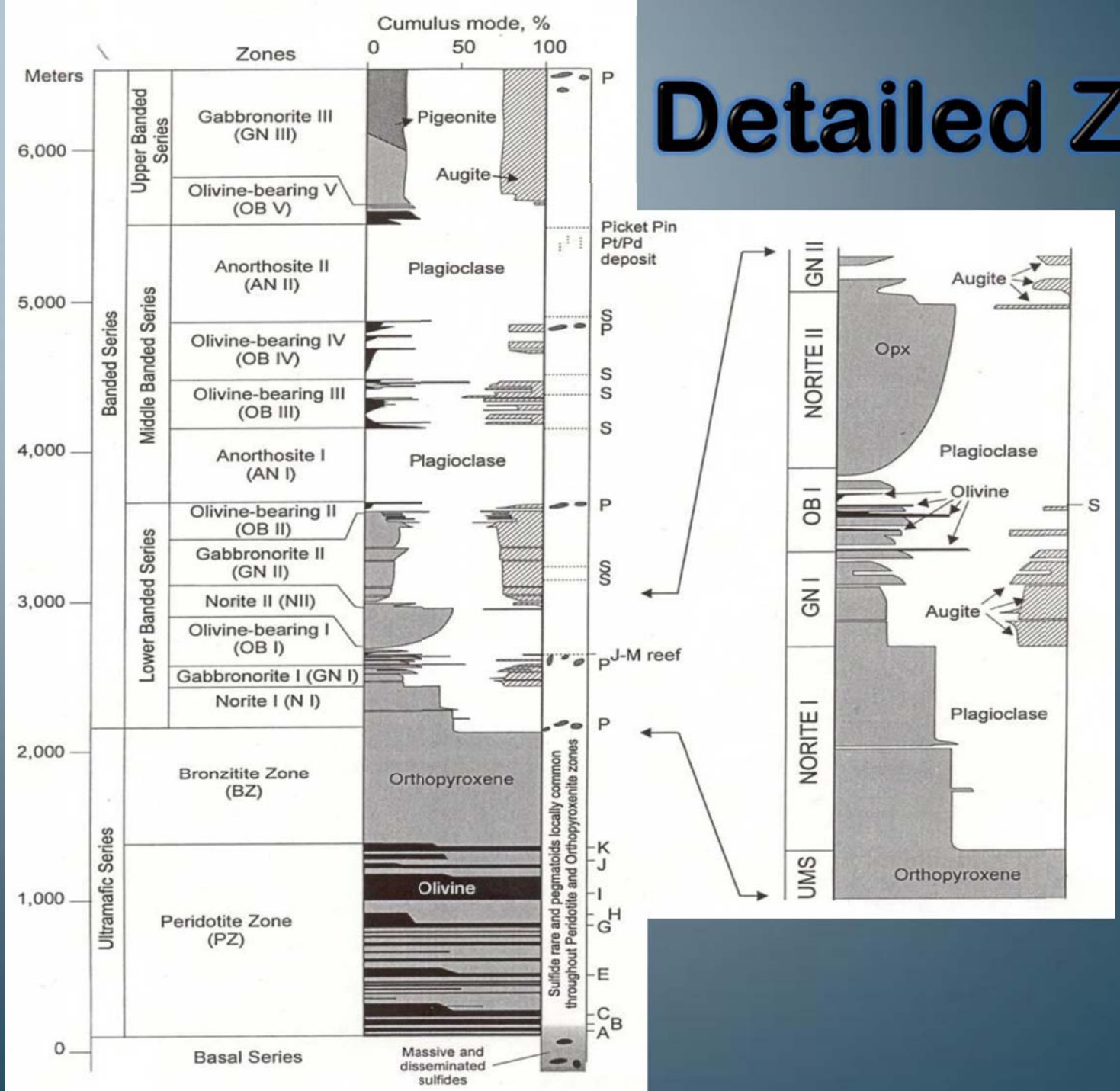
Creating a layered structure



Zones in Stillwater



Detailed Zones



Zones of Interest

- **Anorthosite Zones**
  - Plagioclase, Orthopyroxene, Clinopyroxene ratios roughly 85/5/10
  - Occasionally 100% plagioclase
  - Average An_{75}, Range An_{62-85}
  - Inadequate Ca/Na ratios

Zones of Interest

- Norite I Zone
  - Just above Ultra mafic zone
  - Plagioclase, Orthopyroxene, Clinopyroxene ratios roughly 60/30/10
  - Average An_{85}, Range An_{78-92}
  - Highest %Ca available
  - All of these are needed for the lunar simulant "ingredients"

Zones of Interest

- Ultramafic Zone
  - Composed mainly of Olivine below, Bronzite (pyroxene) above, and Chromite layers
  - Most of the Olivine has been serpentinized to Chrysotile
  - Other (OH) altered minerals i.e. Amphibole, Chlorite, Antigorite exist from altered Pyroxenes
  - Unusable for lunar simulant production

Mineral Separations

- **Plagioclase**
- Virtually the only diamagnetic mineral
- Easier to analyze once all magnetic minerals removed from samples
- Crush and sieve to remove fines
- Use of 80-120 mesh fraction good for magnetic separations

Mineral Separations

- **Pyroxenes**
- Trial runs in the .1 to .2 Amperes range recommended to determine cross-over point
- Augite less magnetic than Enstatite
- Few other detectable minerals as Chromite is in the Ultramafic Zone

Mineral References

- Hess, H.H., 1960, Stillwater Igneous Complex, pgs 21,73,80-87
- Hutchinson, Charles S., Laboratory Handbook of Petrographic Techniques, pg 119
- Wikipedia-general reference for mineral compositions
- Deer, Howie, Zussman, 1978, Single Chain Silicates

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Available Technologies in Metallurgical & Materials Engineering

- **Characterization Tools**
  - Scanning Electron Microscope/ Mineral Liberation Analyzer (SEM/MLA)
  - X-Ray Diffractometer (XRD)
  - X-Ray Fluorescence Spectrometer (XRF)
  - Thin Section and Ore Microscopy

Available Technologies in Metallurgical & Materials Engineering

- **Separation Equipment**
  - Magnetic (dry rolls, WHIMS, wet drums, rare earth, davis tube)
  - High-tension (dry rolls)
  - Gravity (shaking table, spiral, Knelson concentrator)
  - Hardness/Size (crushers, grinding mills, pulverizers, screens)
  - Flotation (batch)
  - Leaching (column, bottle roll, agitation, autoclave)
  - Ion-exchange (column, agitation, titration)

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} Norite

Norite Studies

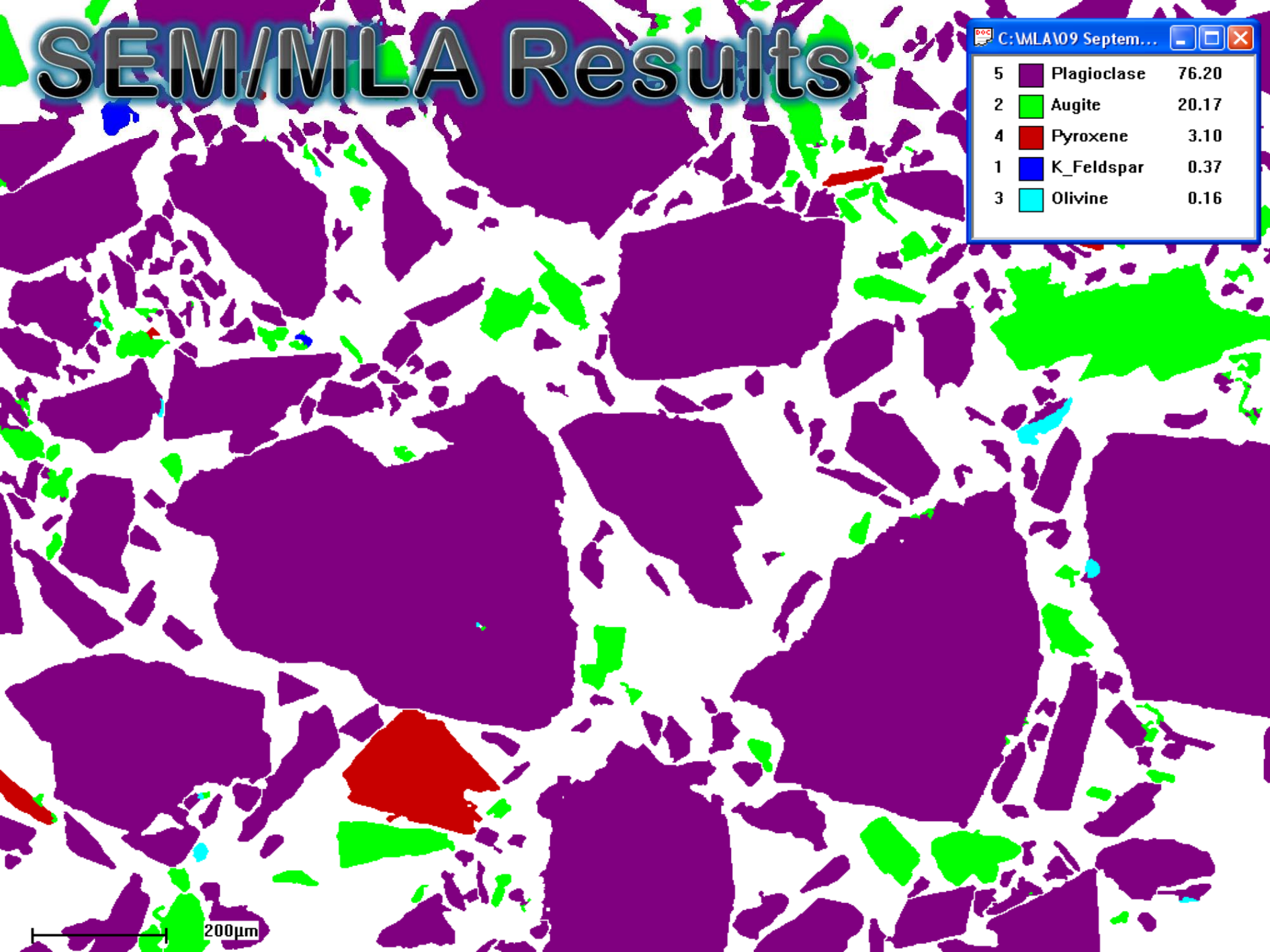
Jacqueline Graham

NORITE FEED STOCK

Progress

- Crushed
- Ground
- Pulverized
- SEM/MLA Analysis
- XRD
- XRF

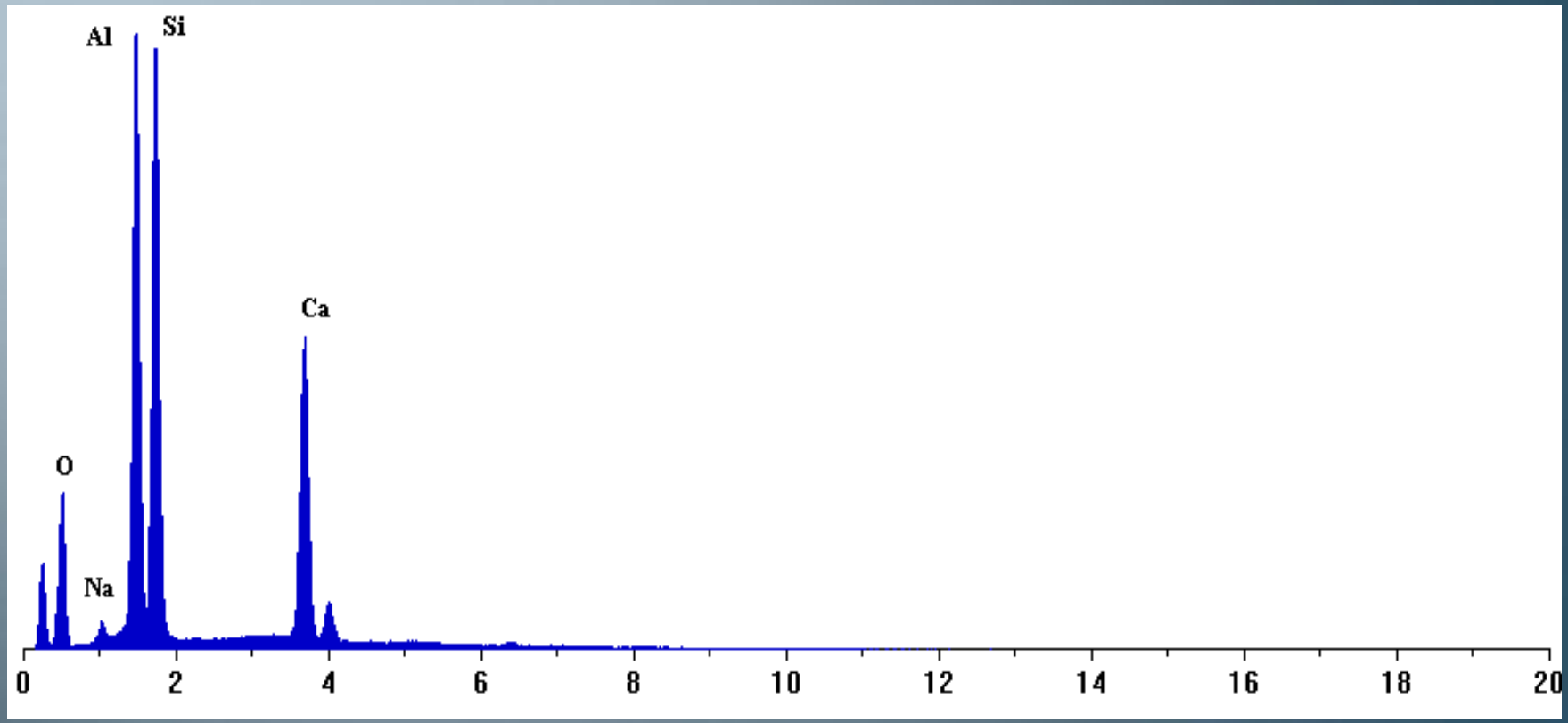
SEM/MLA Results



Minerals Present

- Plagioclase
- Augite
- Pyroxene
- Potassium Feldspar
- Olivine

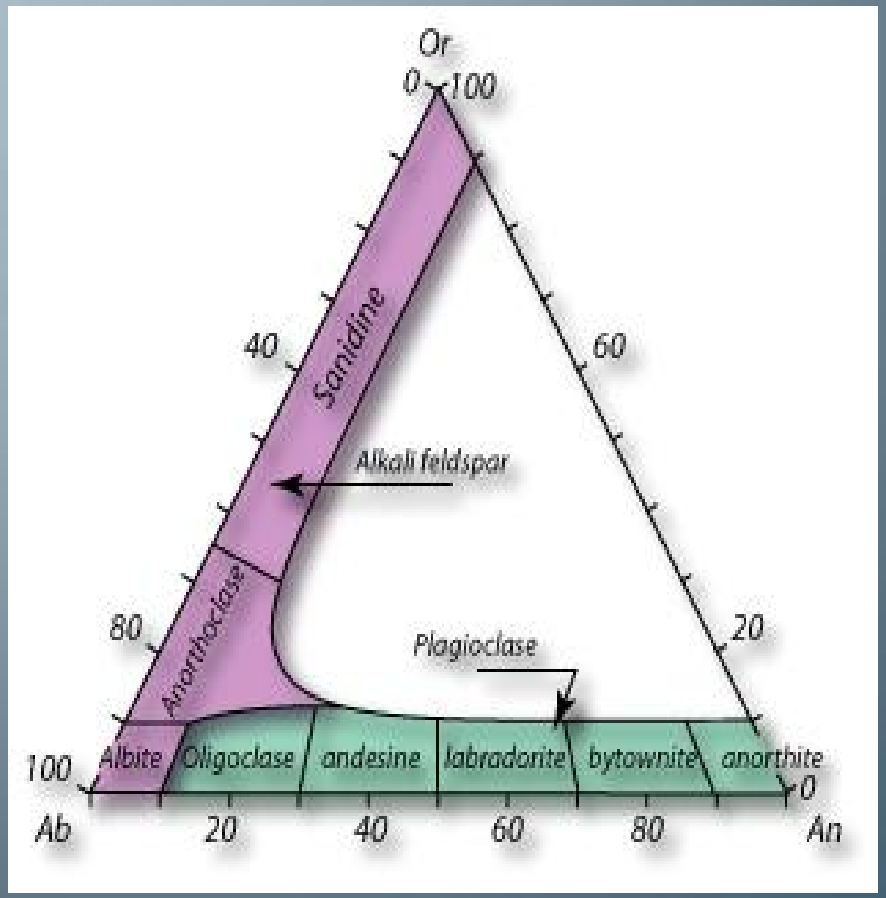
Plagioclase EDX Spectrum



- $(Na,Ca)(Si,Al)_4O_8$

High-Ca Plagioclase

% $KAlSi_3O_8$



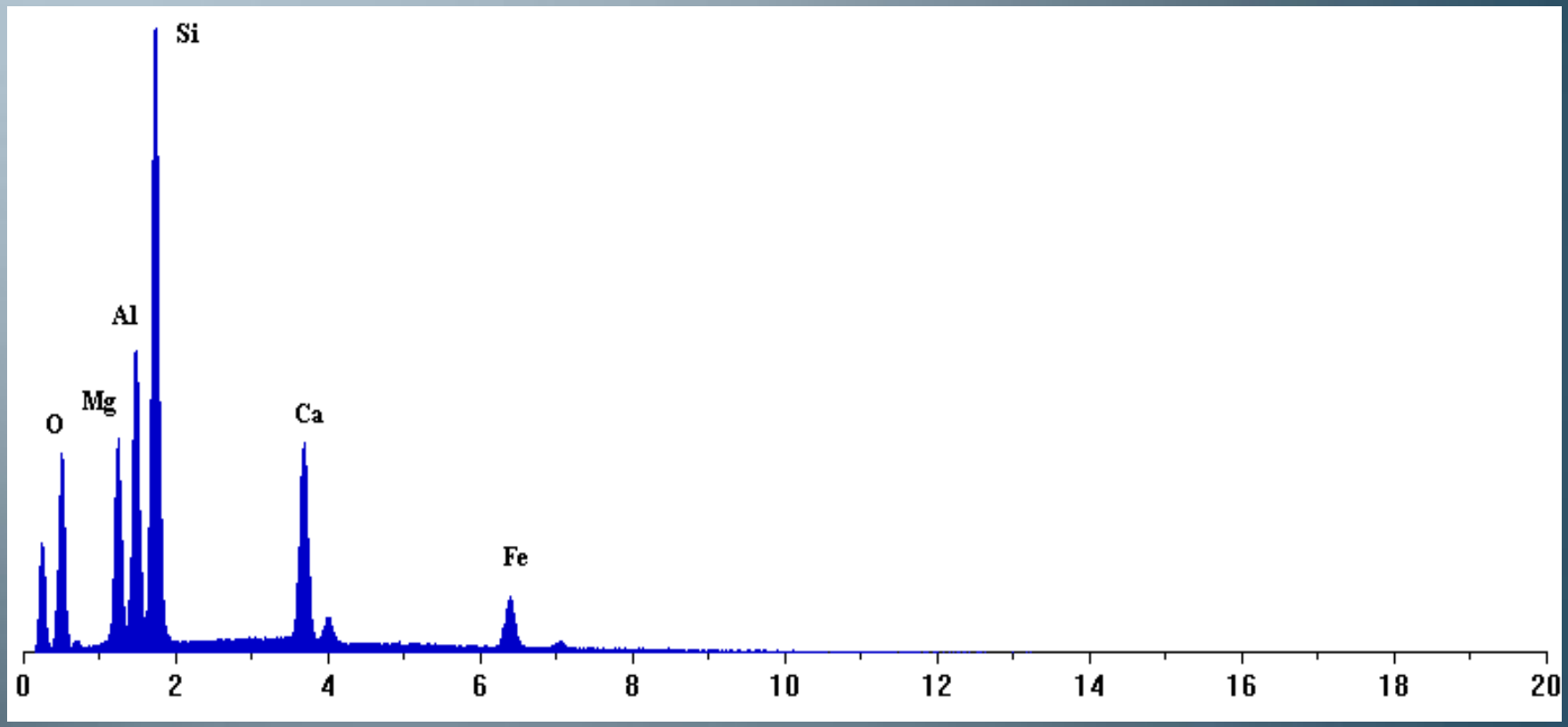
% $NaAlSi_3O_8$

% $CaAl_2Si_2O_8$

Minerals Present

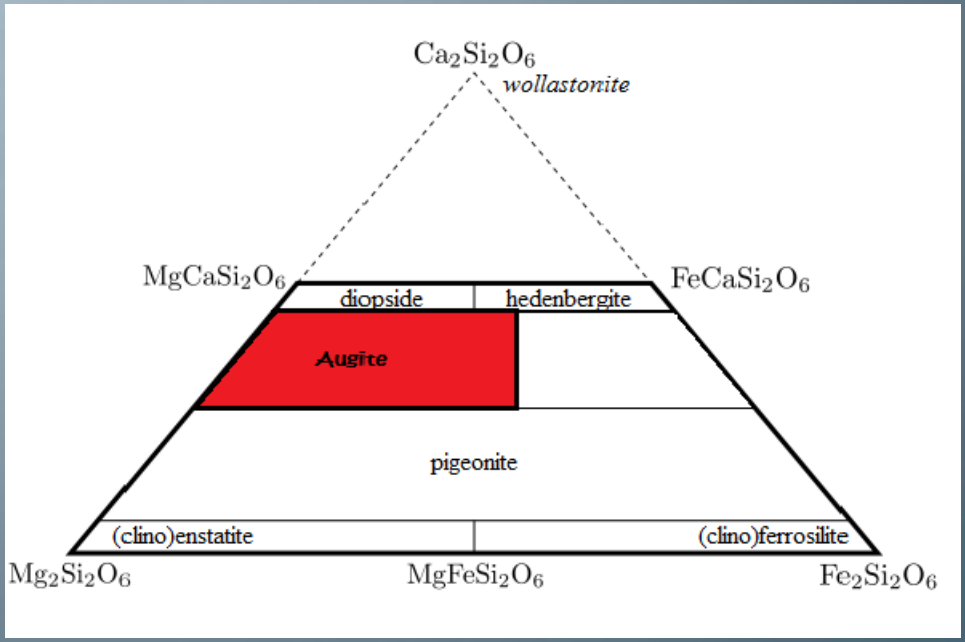
- Plagioclase
- **Augite**
- Pyroxene
- Potassium Feldspar
- Olivine

Augite EDX Spectrum



- $(Ca,Na)(Mg,Fe,Al)[(Si,Al)_2O_6]$

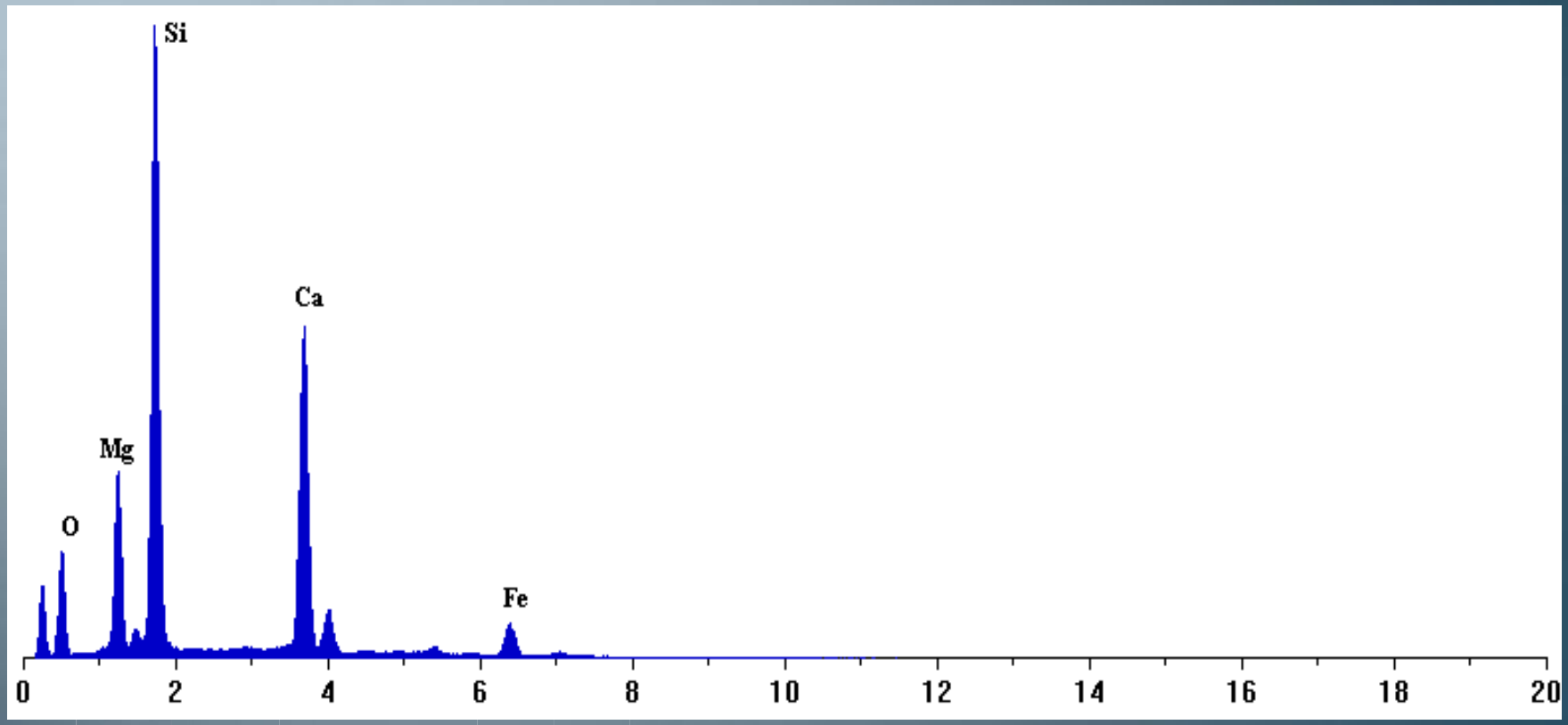
Pyroxene Group Compositions Augite



Minerals Present

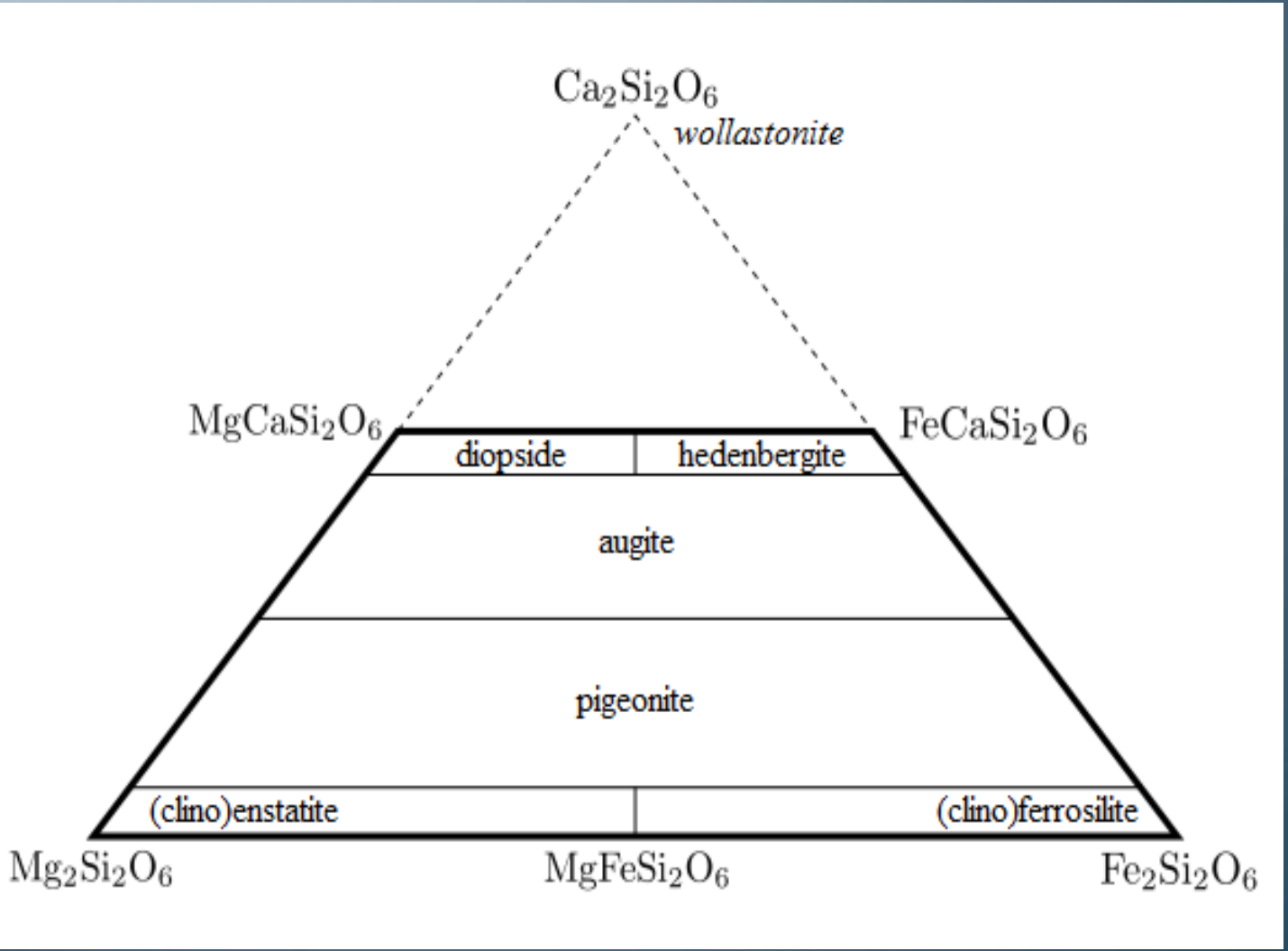
- Plagioclase
- Augite
- **Pyroxene**
- Potassium Feldspar
- Olivine

Pyroxene EDX Spectrum



- $(Ca,Mg,Fe)_2Si_2O_6$

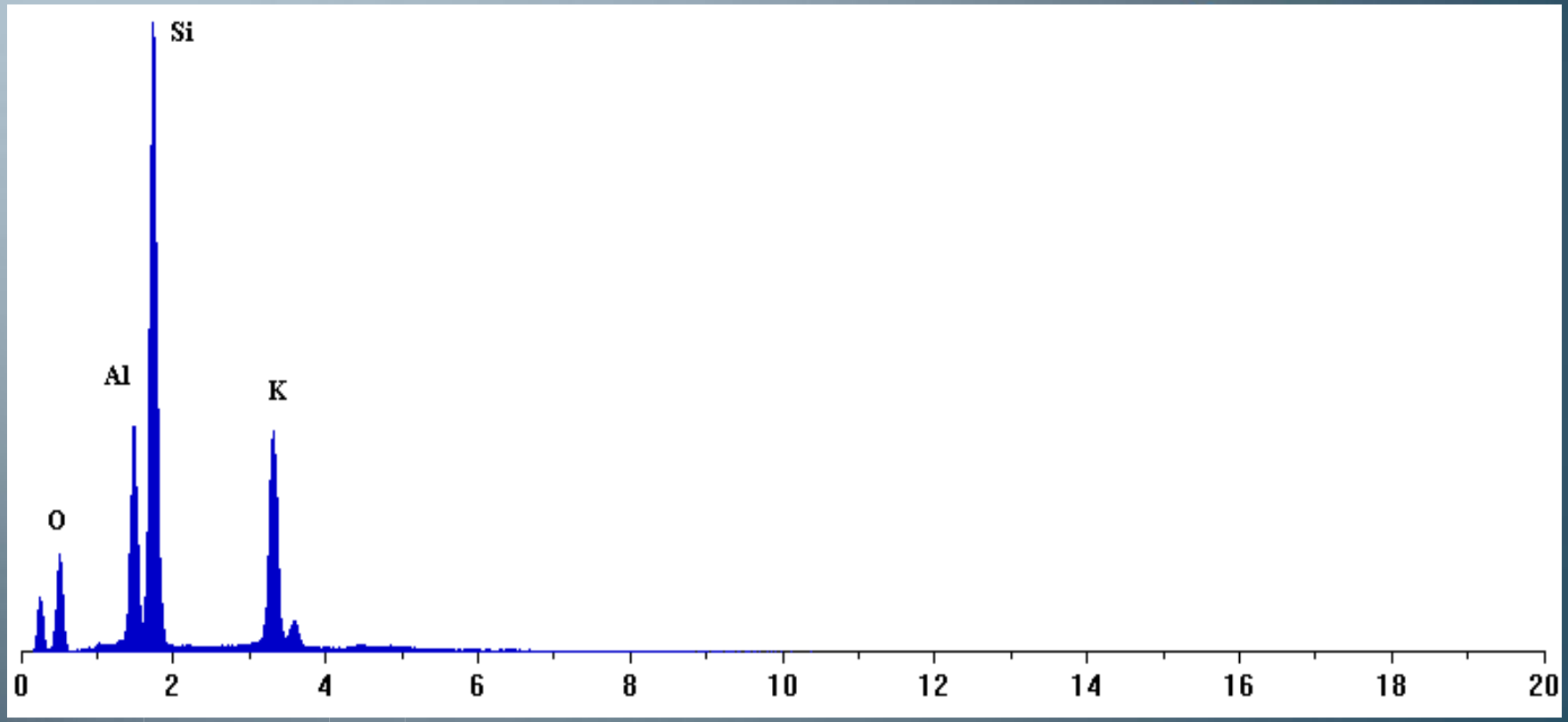
Pyroxene Group Compositions



Minerals Present

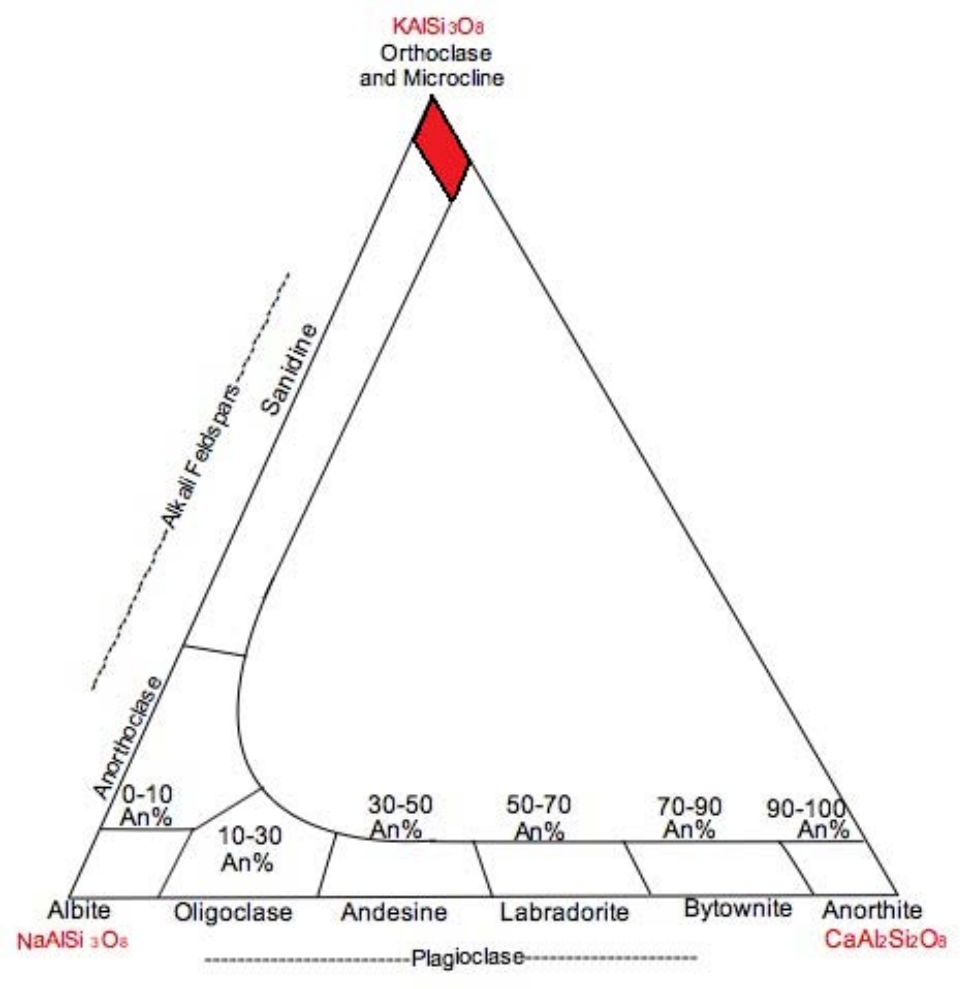
- Plagioclase
- Augite
- Pyroxene
- **Potassium Feldspar**
- Olivine

Potassium Feldspar EDX Spectrum



- $KAlSi_3O_8$

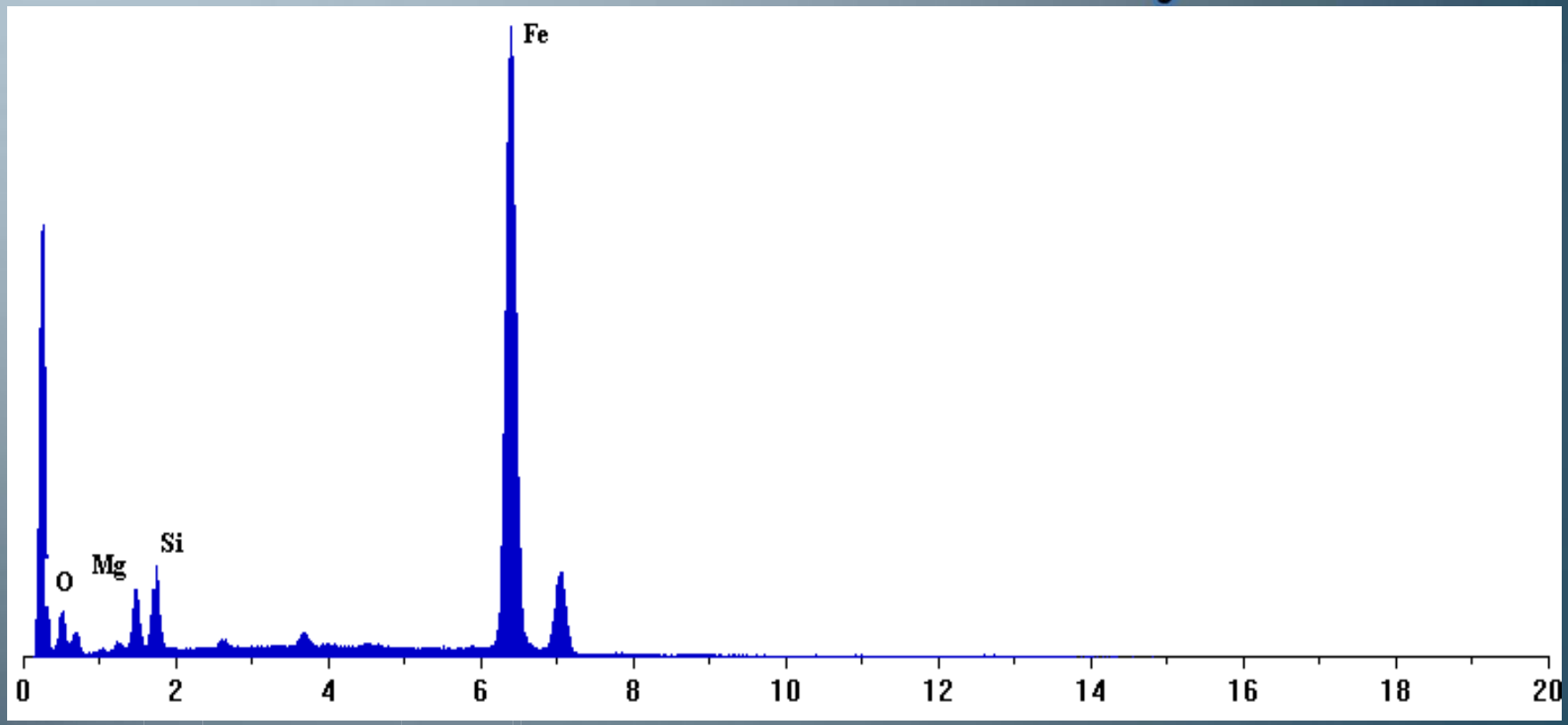
Potassium Feldspar (Orthoclase)



Minerals Present

- Plagioclase
- Augite
- Pyroxene
- Potassium Feldspar
- Olivine

Olivine EDX Spectrum



- $(Mg,Fe)_2SiO_4$

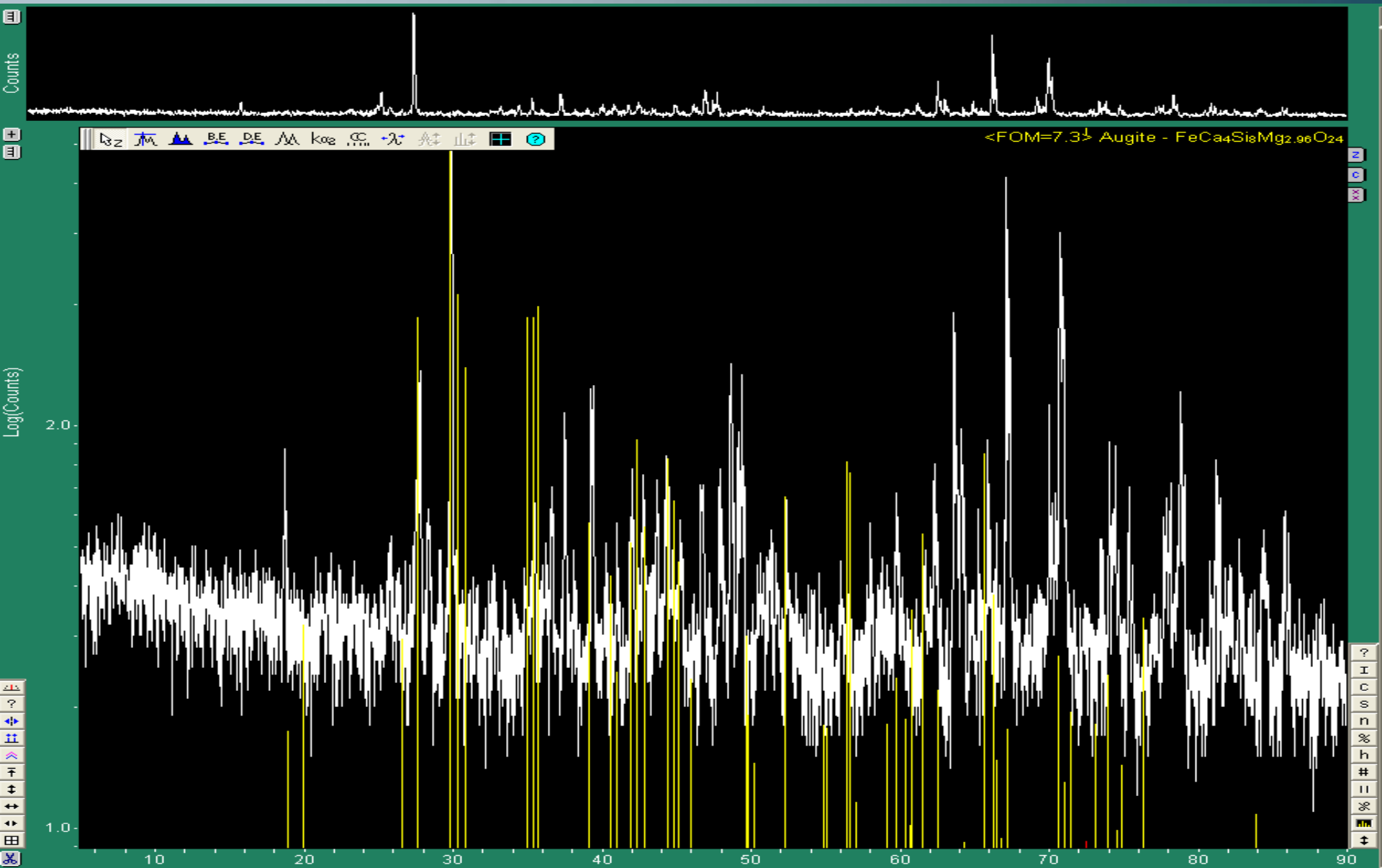
Physical Properties

Mineral	Chemical Formula	Specific Gravity	Magnetic Response	Hardness	Electrostatic Response
Plagioclase	$(Na, Ca)(Si,Al)_4O_8$	2.62-2.75	Non-Magnetic	6-6.5	Non-Conductive
Augite	$(Ca,Na)(Mg,Fe^{+2}, Al,Ti)[(Si,Al)_2O_6]$	3.2-3.5	Paramagnetic	5-6.5	Conductive*
Pyroxene	$(Mg,Fe^{+2})_2Si_2O_6$	3.1-3.6	Paramagnetic	5-6.5	Non-Conductive**
Potassium Feldspar	$KAlSi_3O_8$	2.6-2.8	Non-Magnetic	6	Non-Conductive
Olivine (Fayalite)	Fe_2SiO_4	4.392	Paramagnetic	6.5-7	Non-Conductive

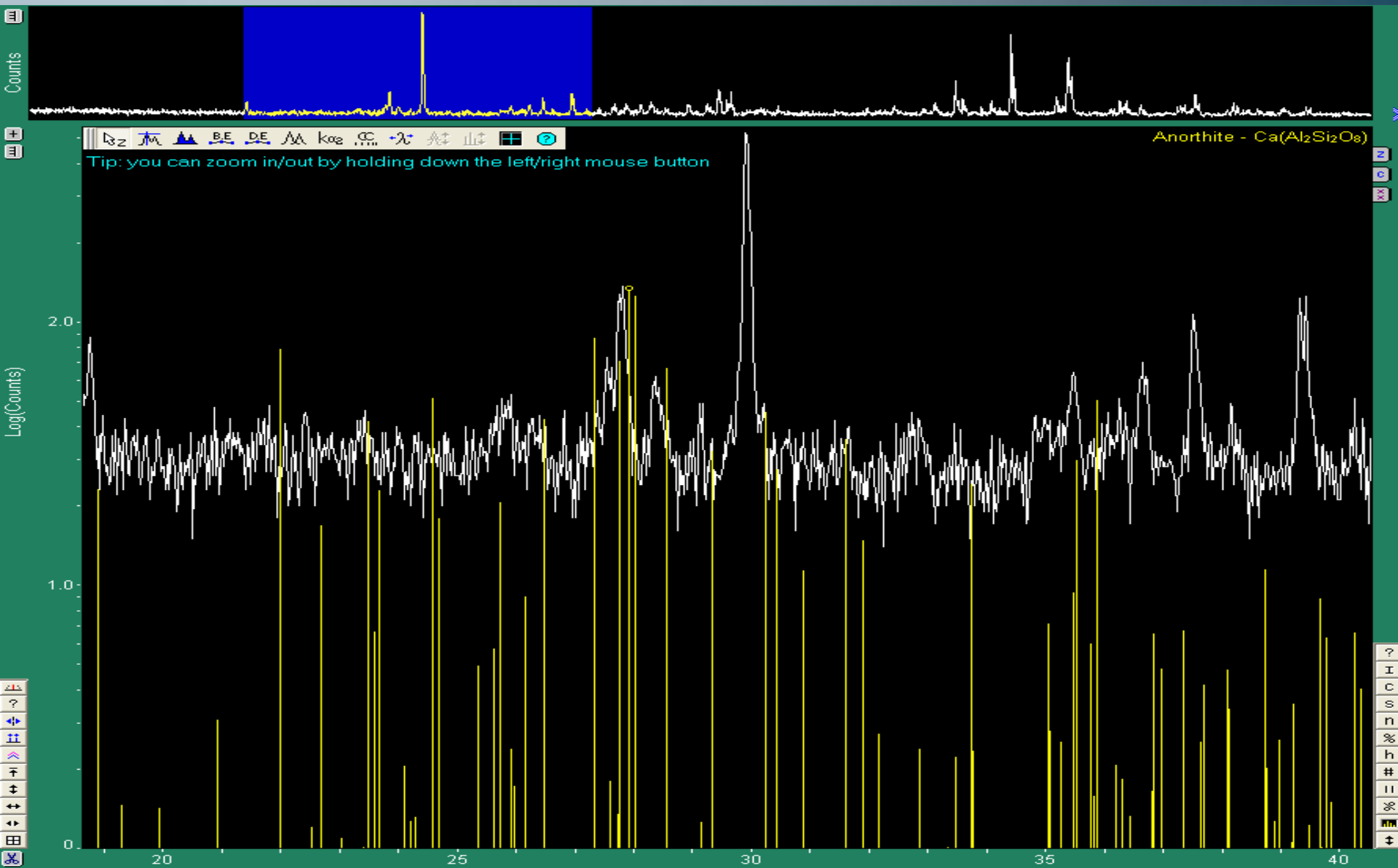
*Variations in response to electrostatic separation will occur based on the actual composition of the mineral from varying locations and the presence of inclusions.

**The response of some minerals can be varied with treatment at elevated temperature

Norite XRD Augite Peaks



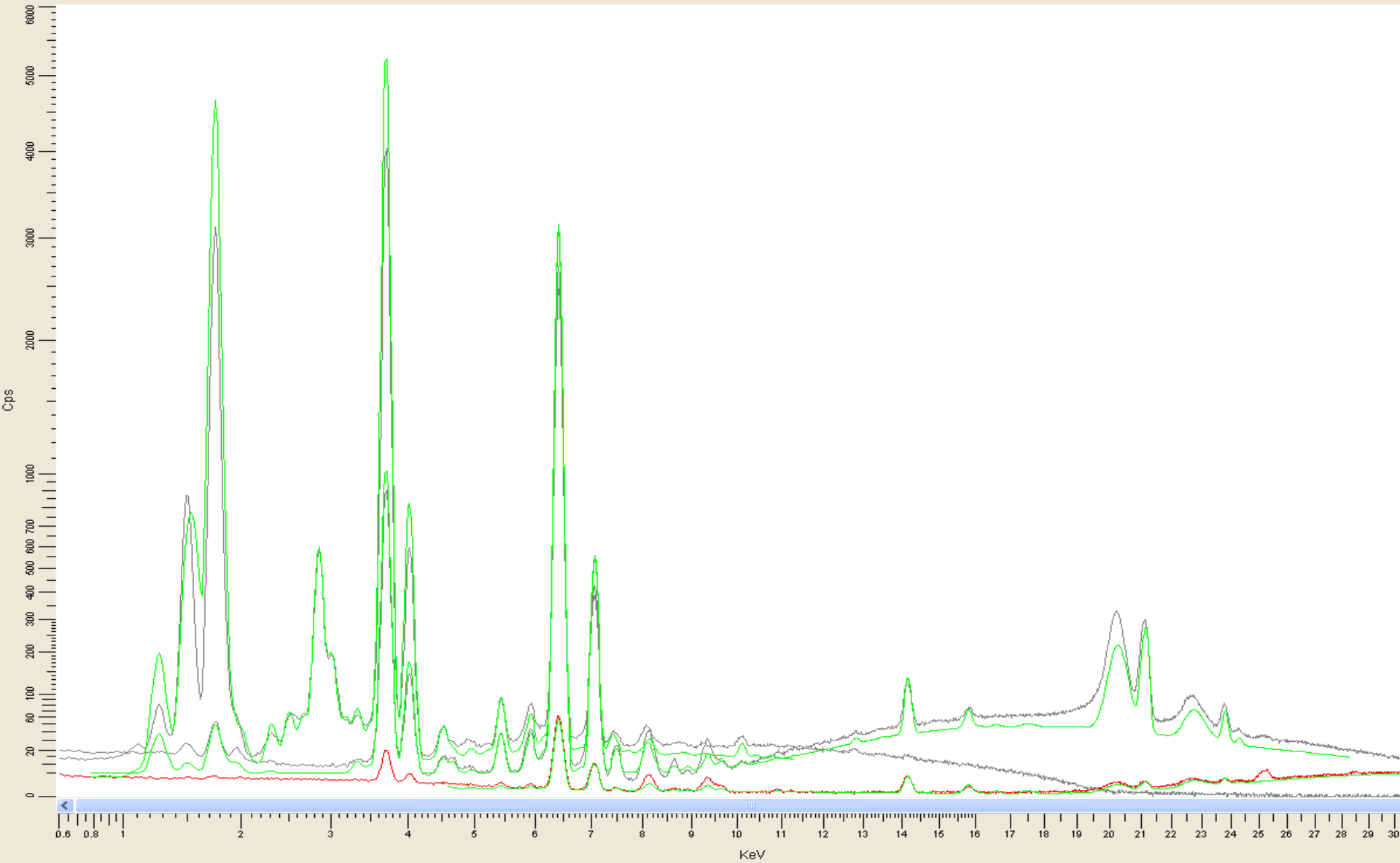
Norite XRD Anorthite Peaks



XRF Results

Formula	Concentration
Sample area density	0.75
SiO_2	50.23%
CaO	18.36%
Al_2O_3	14.28%
MgO	10.57%
Fe_2O_3	5.70%
SO_3	0.23%
K_2O	0.16%
Cr_2O_3	0.13%
P_2O_5	0.12%
TiO_2	0.12%
MnO	0.06%
SrO	0.02%
Ni_2O_3	0.02%
Cu_2O	0.01%
ZrO_2	0.00%
ZnO	0.00%
Nb_2O_5	0.00%
MoO_3	0.00%

Norite XRF Spectrum



Conclusion

- A magnetic separation needs to be performed
- From the separation a more accurate reading of the pyroxene levels and compositions can be determined
- A more accurate An% can also be determined

Future Work

- Magnetic Separation
- Wet Sieve Analysis
- Dry Sieve Analysis
- Shaking Table (Wilfley Table)
- Electrostatic Separation

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} Olivine

Olivine Studies

Peter Rossiter

Source:
Unimun Corporation
Hamilton WA, 98255

Progress

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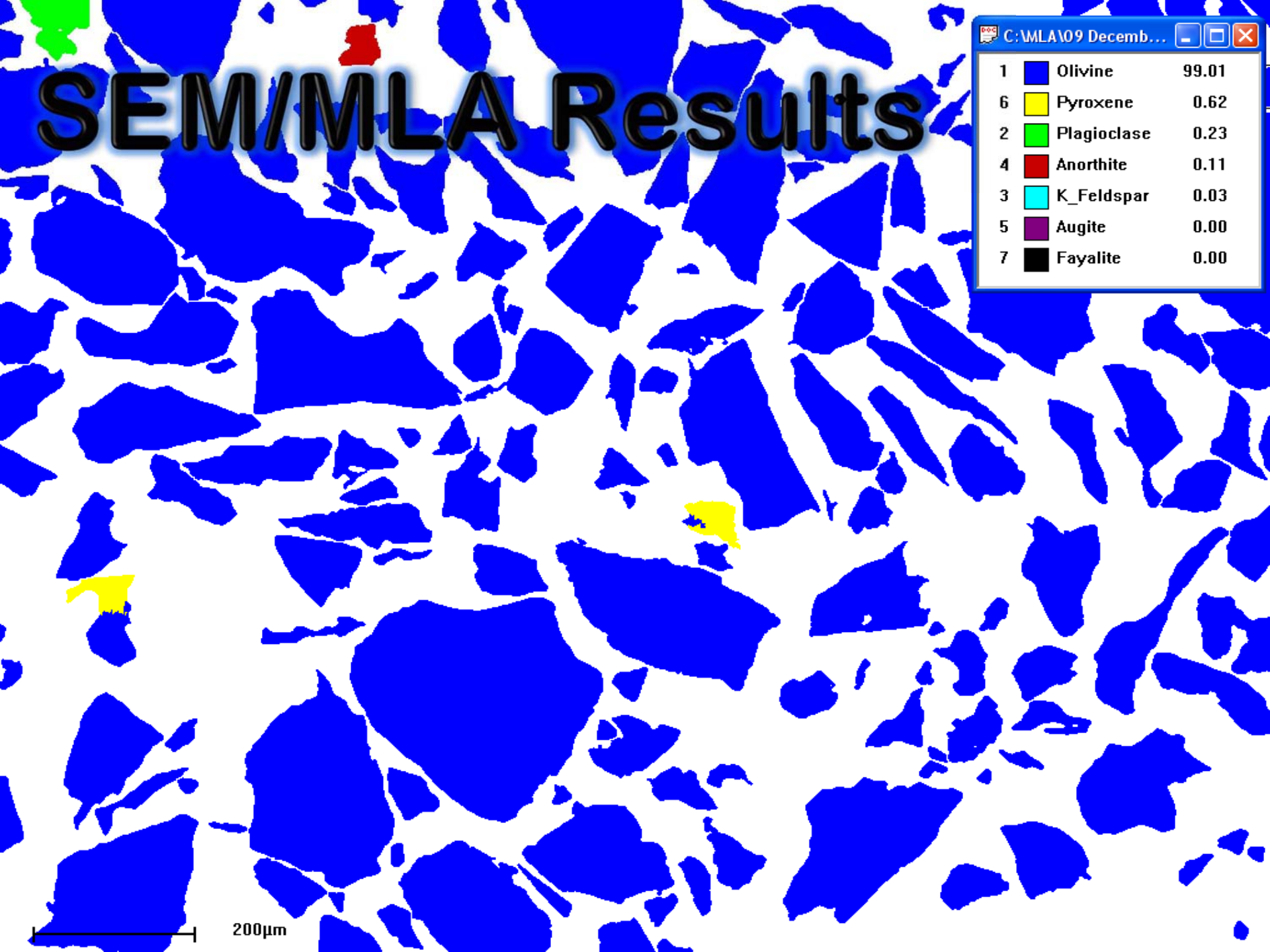
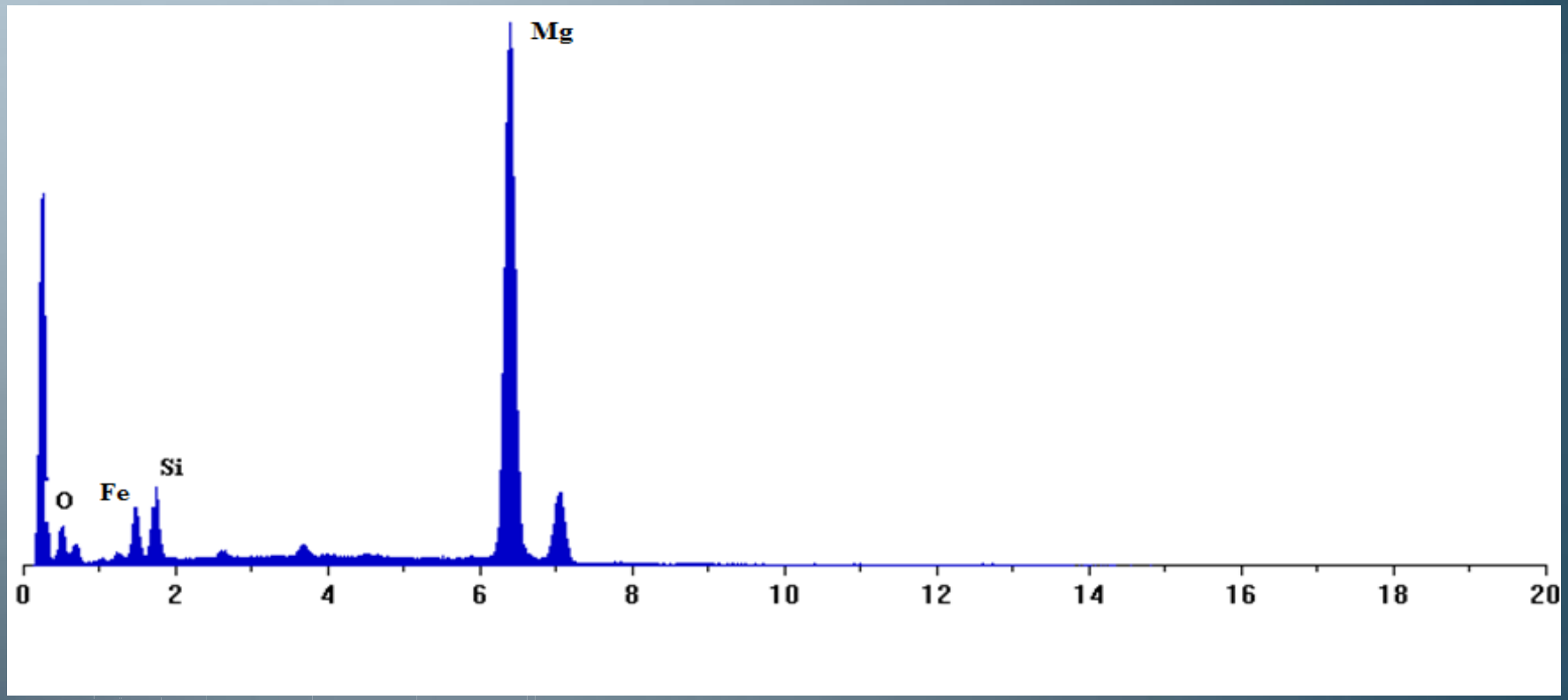
- Pulverized
- SEM/MLA Analysis
- XRD
- XRF

SEM/MLA Results

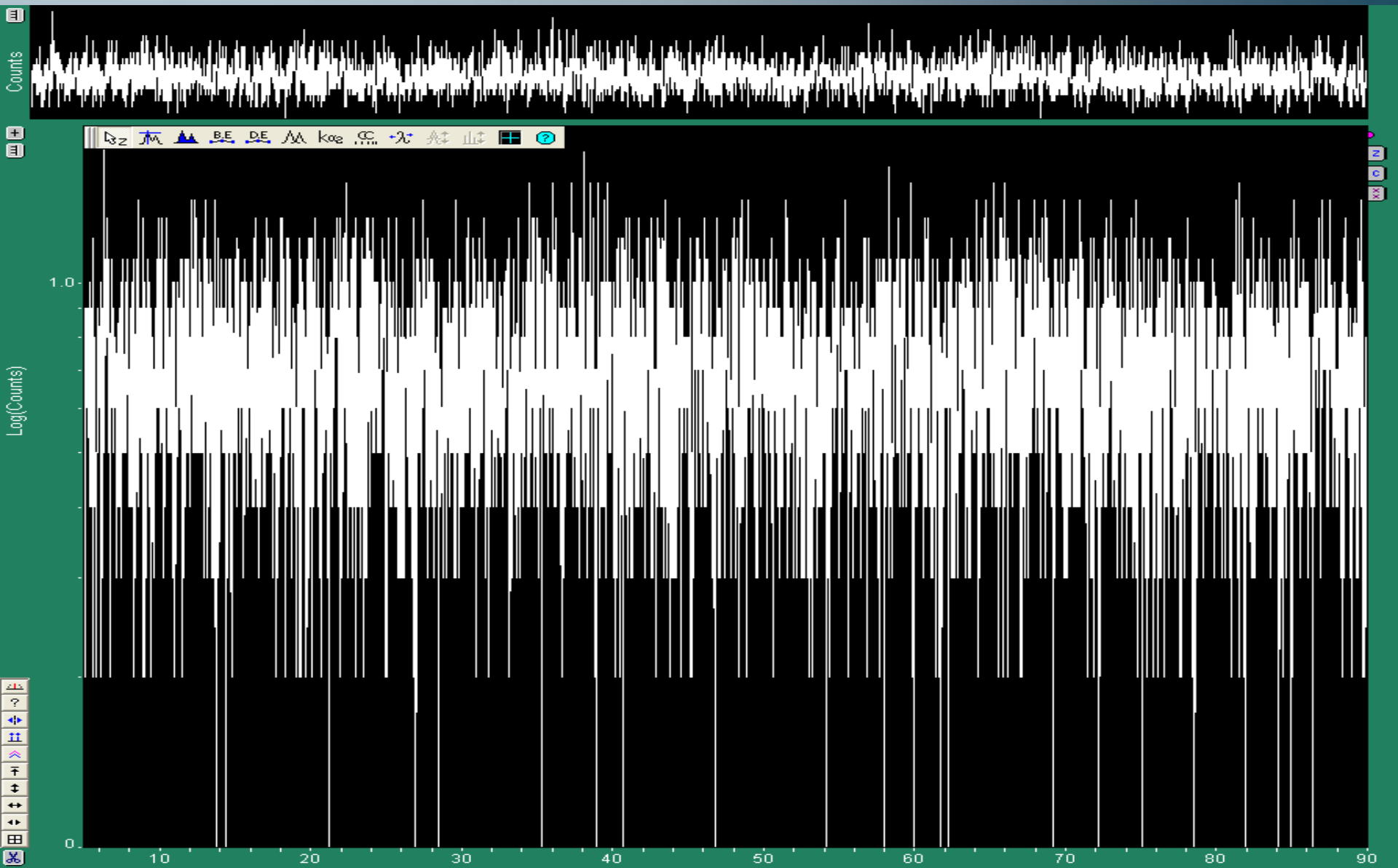


Olivine EDX Spectrum



- $(Mg,Fe)_2SiO_4$

Olivine XRD Results



Physical Properties

Mineral	Chemical Formula	Specific Gravity	Magnetic Response	Hardness	Electrostatic Response
Olivine	Mg_2SiO_4	4.392	Paramagnetic	6.5-7	Non-Conductive
Pyroxene	$(Mg,Fe^{+2})_2Si_2O_6$	3.1-3.6	Paramagnetic	5-6.5	Non-Conductive**
Plagioclase	$(Na, Ca)(Si,Al)_4O_8$	2.62-2.75	Non-Magnetic	6-6.5	Non-Conductive
Anorthite	$CaAl_2Si_2O_8$	2.72-2.75	Non-Magnetic	6	Non-Conductive
Potassium Feldspar	$KAlSi_3O_8$	2.6-2.8	Non-Magnetic	6	Non-Conductive

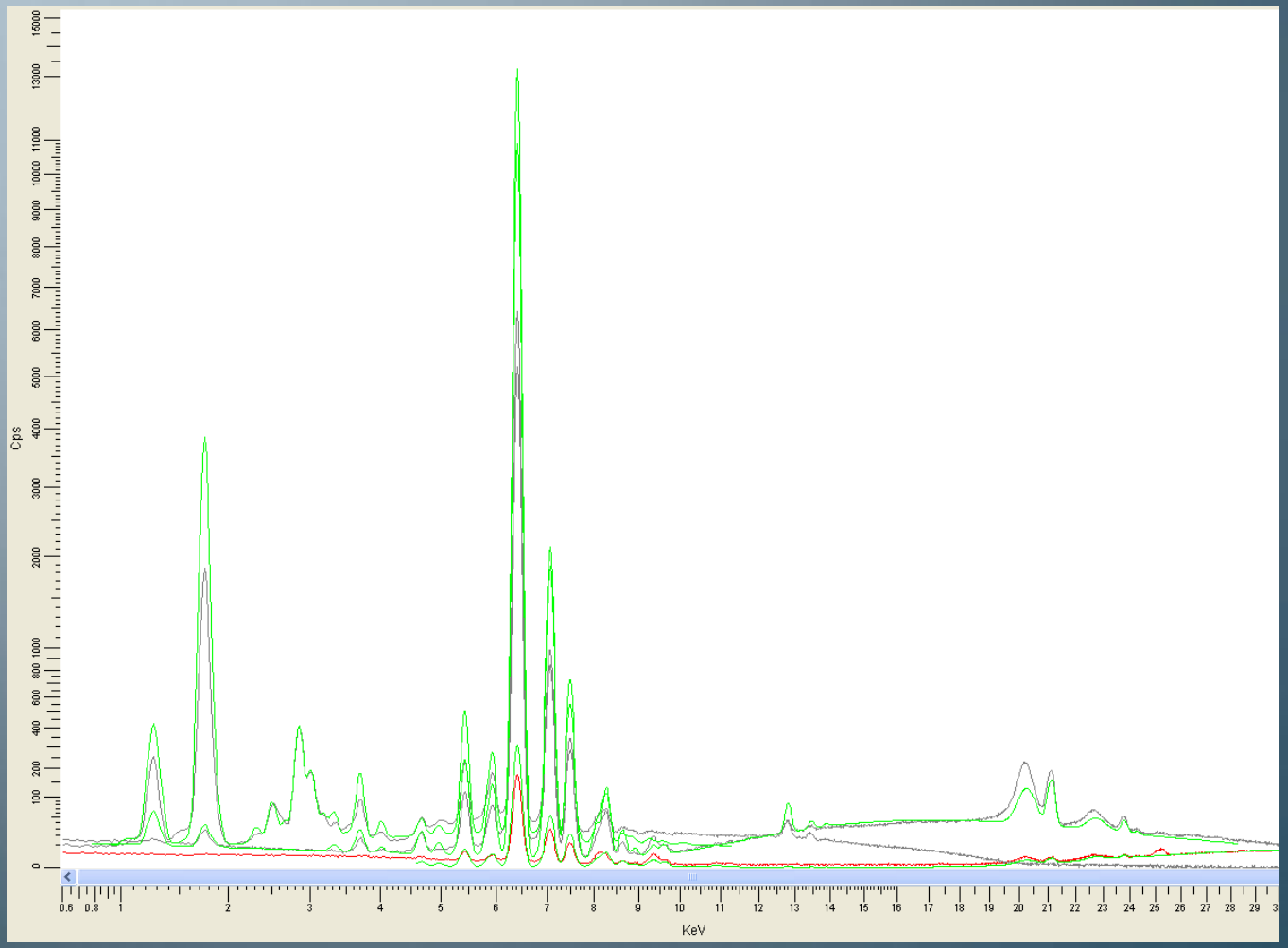
*Variations in response to electrostatic separation will occur based on the actual composition of the mineral from varying locations and the presence of inclusions.

**The response of some minerals can be varied with treatment at elevated temperature

XRF Results

Formula	Concentration (oxide %)
Sample area density	3.03
SiO2	45.61%
MgO	25.90%
Fe2O3	22.70%
Na2O	2.54%
Ni2O3	1.24%
Cr2O3	0.79%
CaO	0.54%
MnO	0.24%
K2O	0.19%
SO3	0.12%
Cu2O	0.04%
Co2O3	0.03%
V2O5	0.03%
ZnO	0.03%

Olivine XRF Spectrum



Future Work

- Magnetic Separation
- Wet Sieve Analysis
- Dry Sieve Analysis
- Shaking Table (Wilfley Table)
- Electrostatic Separation

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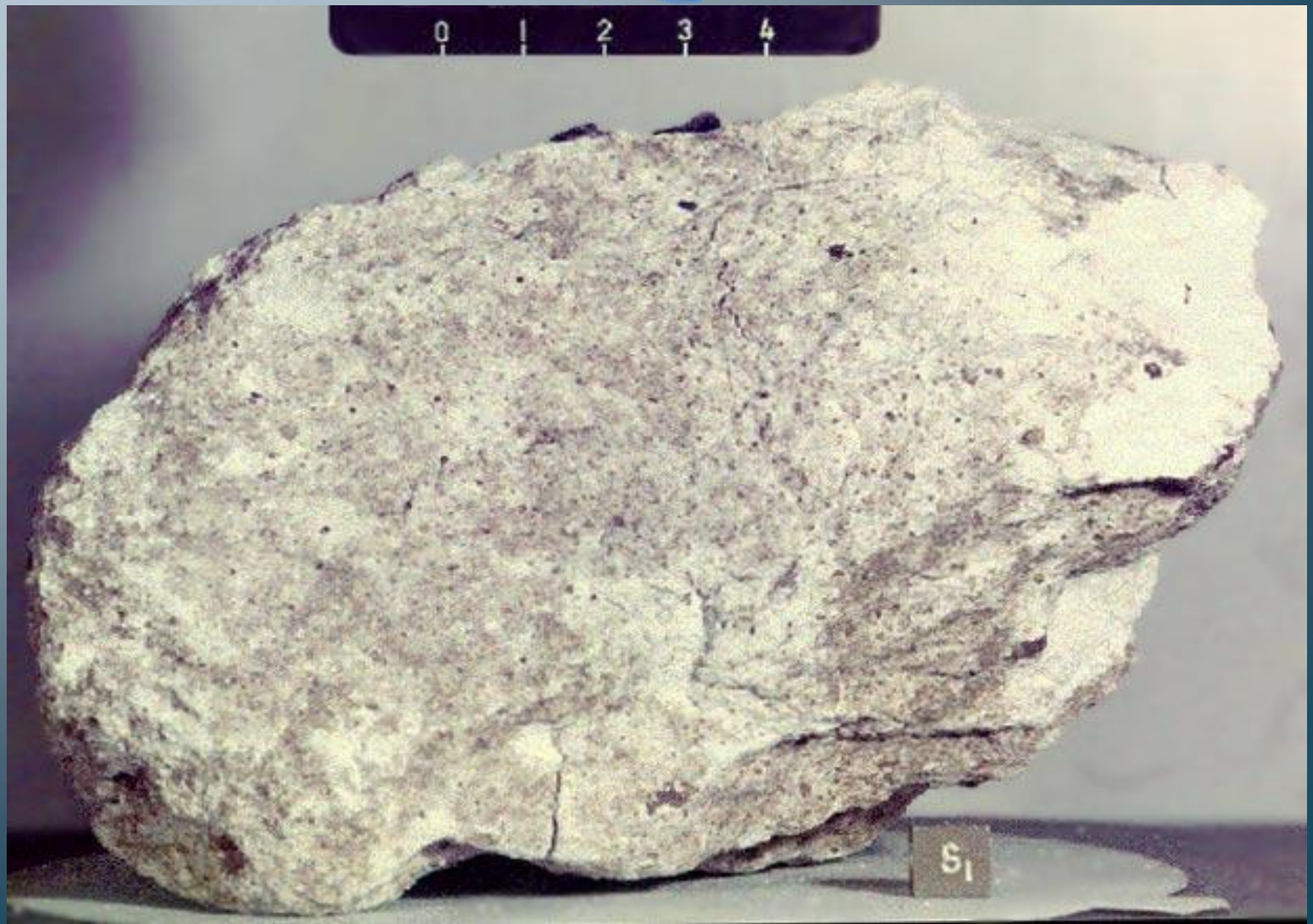
} Anorthite

Anorthite Studies

Danielle Granlund

Lunar Plagioclase



High Calcium Plagioclase

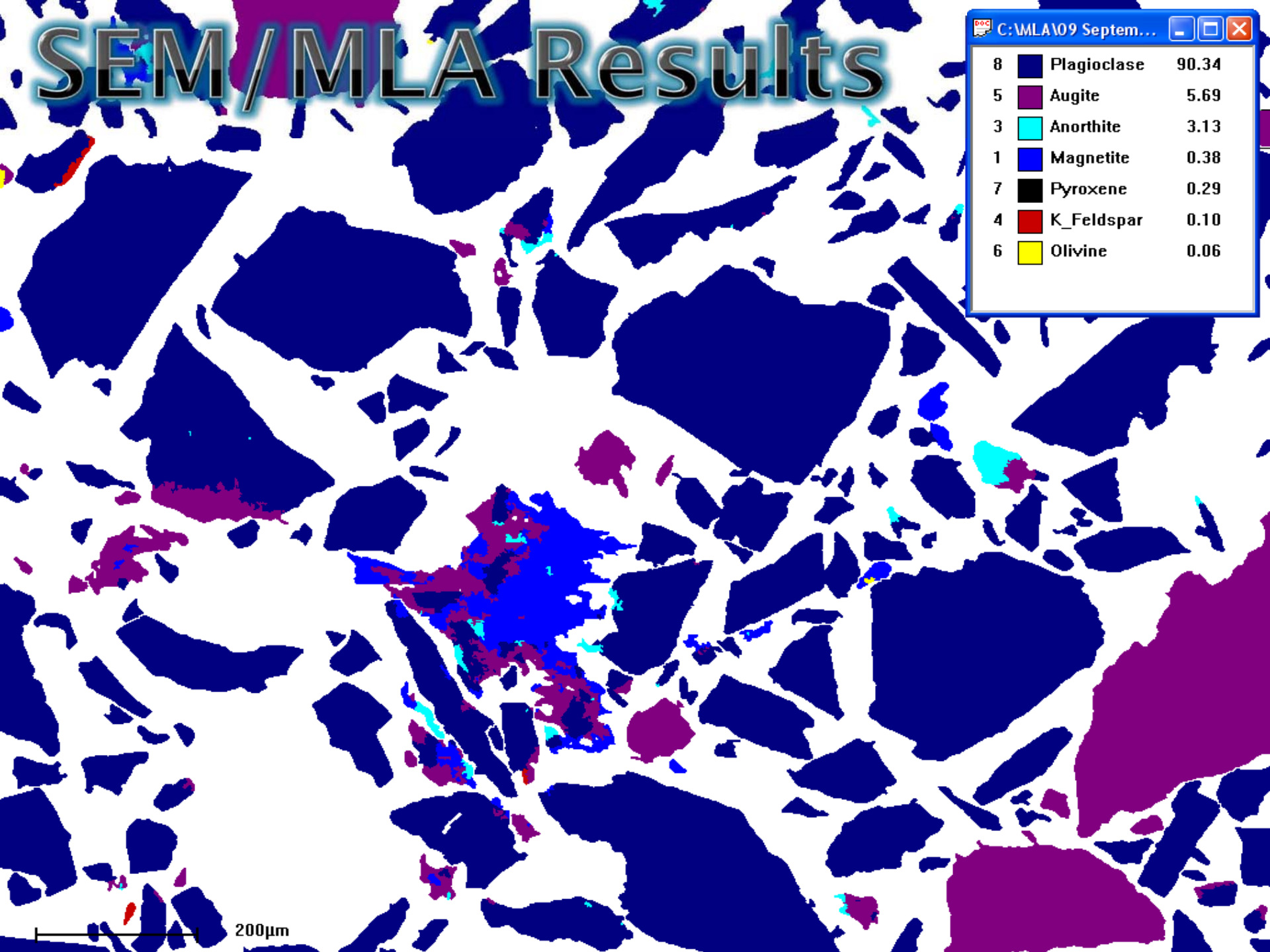
- Anorthite level of 80 or higher
- Samples from Stillwater
- Regolith has normal concentration of 90+ An
- Outcrop and Waste Pile Samples Analyzed

Progress

- Crushed
- Ground
- Pulverized
- SEM/MLA Analysis
- XRD
- XRF

Anorthite (outcrop)

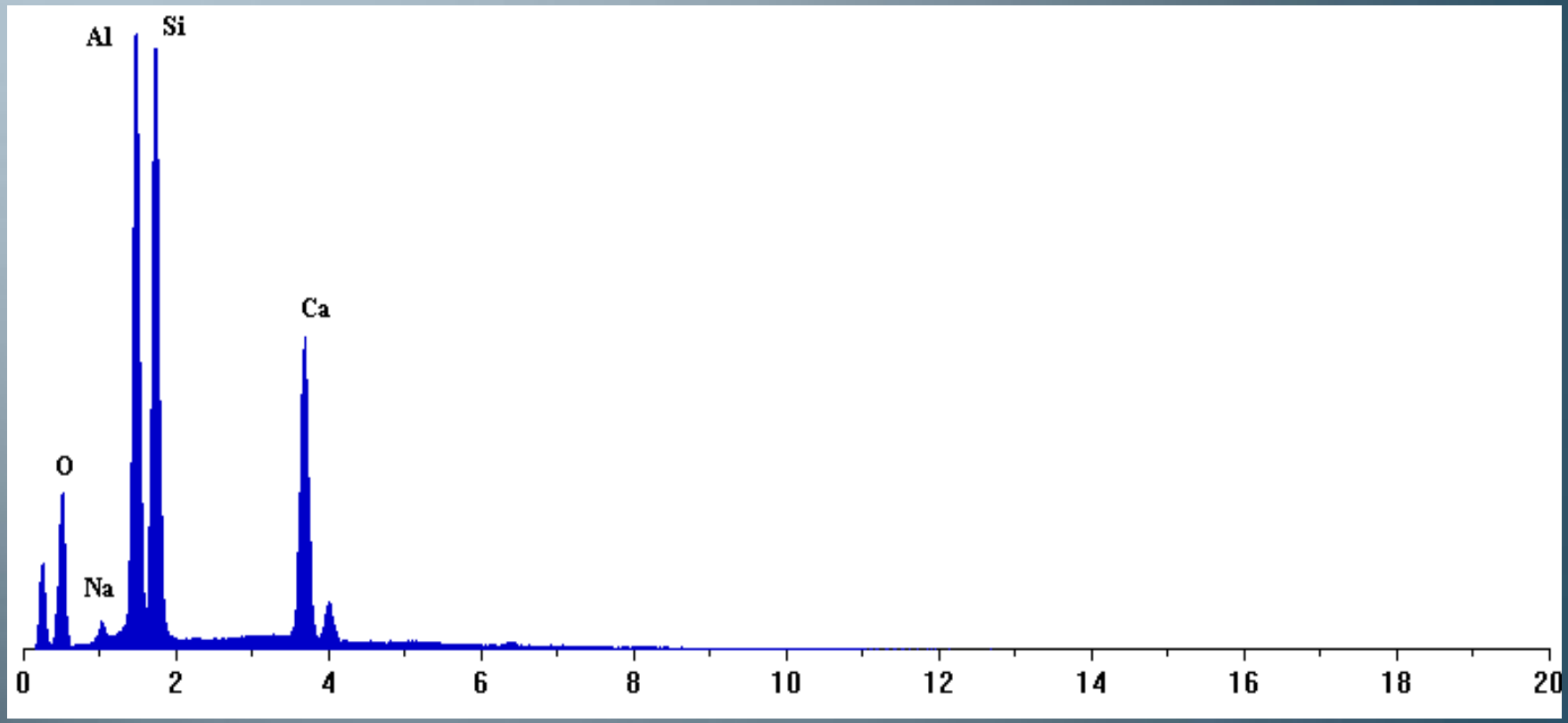
SEM/MLA Results



Minerals Present

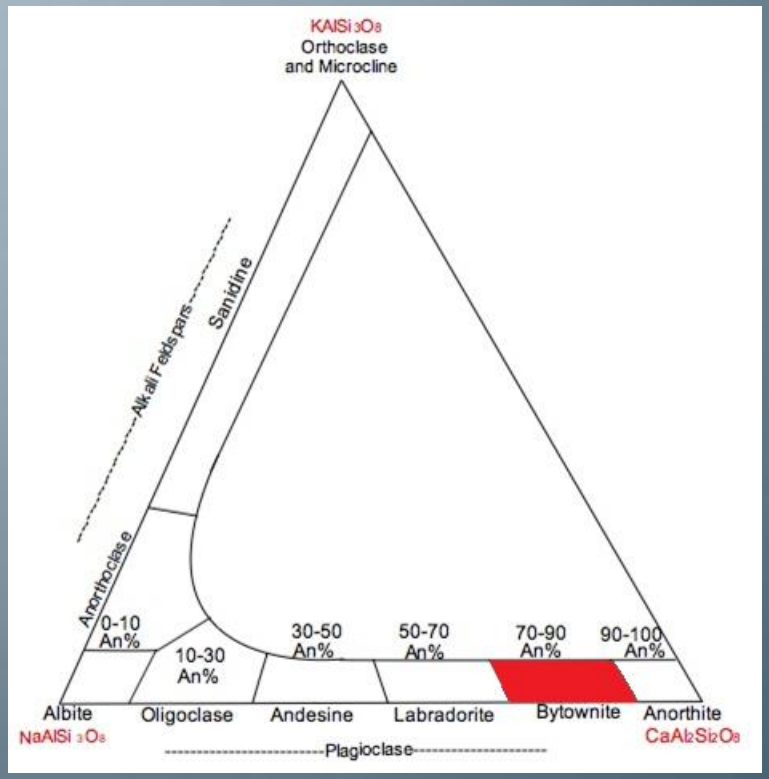
- Plagioclase
- Augite
- Anorthite

Plagioclase EDX Spectrum



- $(Na,Ca)(Si,Al)_4O_8$

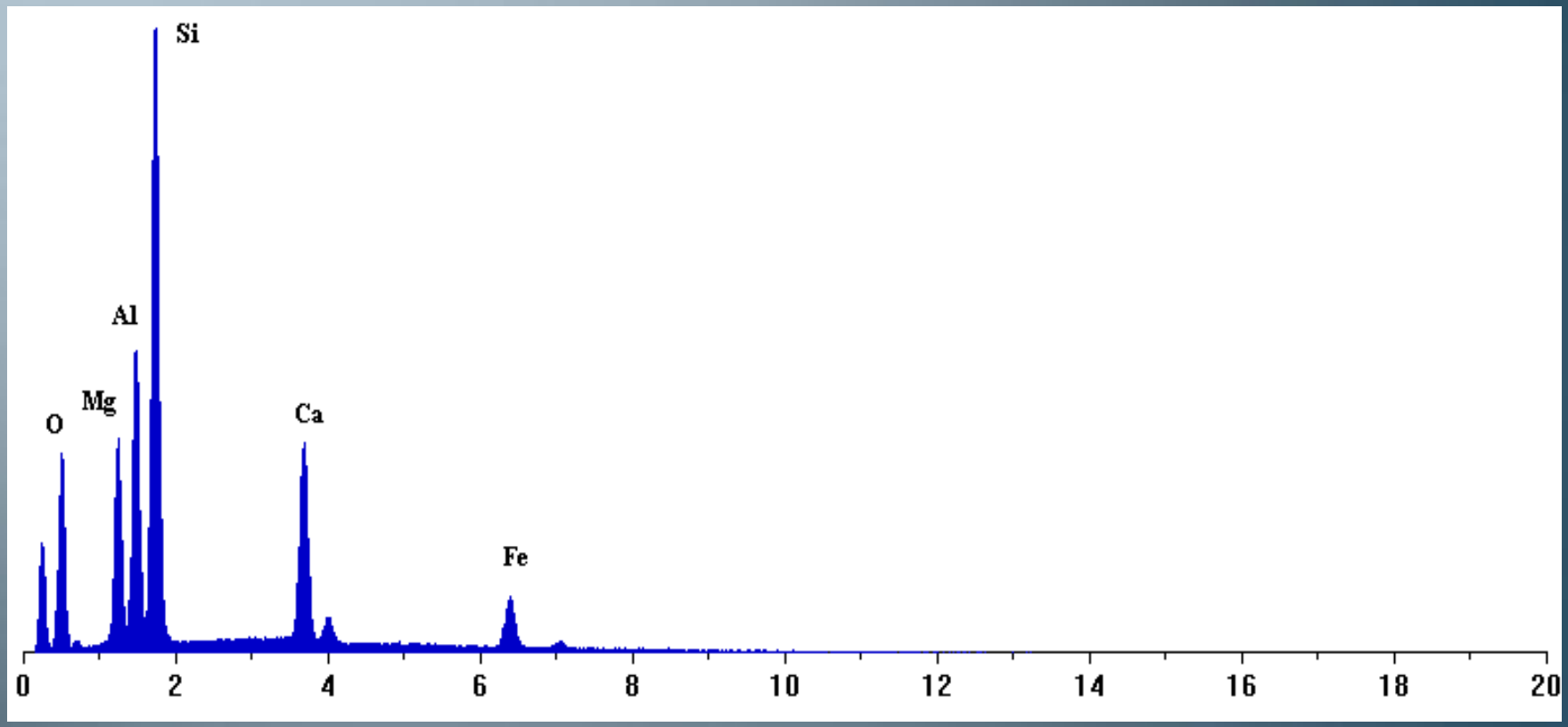
High-Ca Plagioclase



Minerals Present

- Plagioclase
- **Augite**
- Anorthite

Augite EDX Spectrum

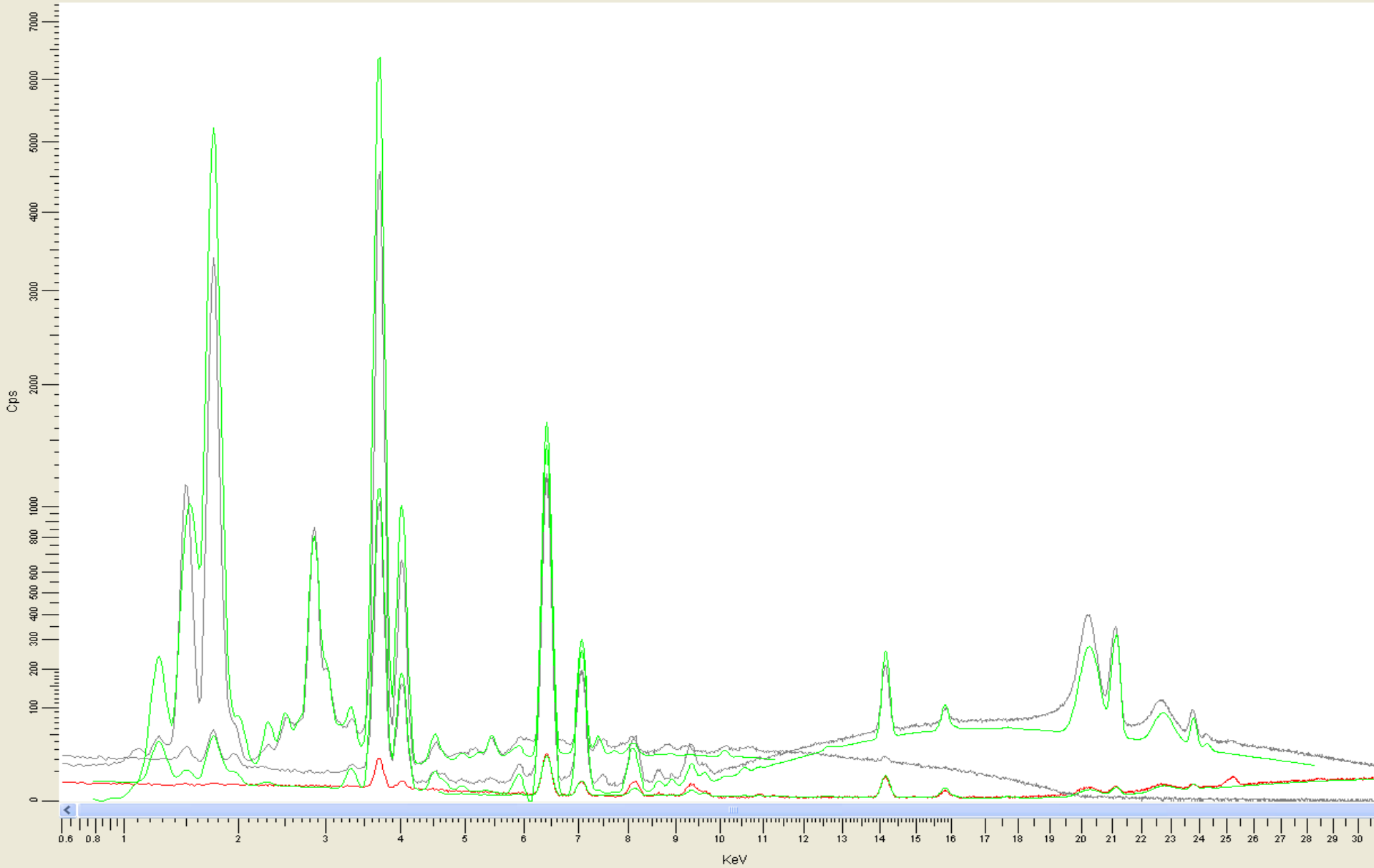


- $(Ca,Na)(Mg,Fe,Al)[(Si,Al)_2O_6]$

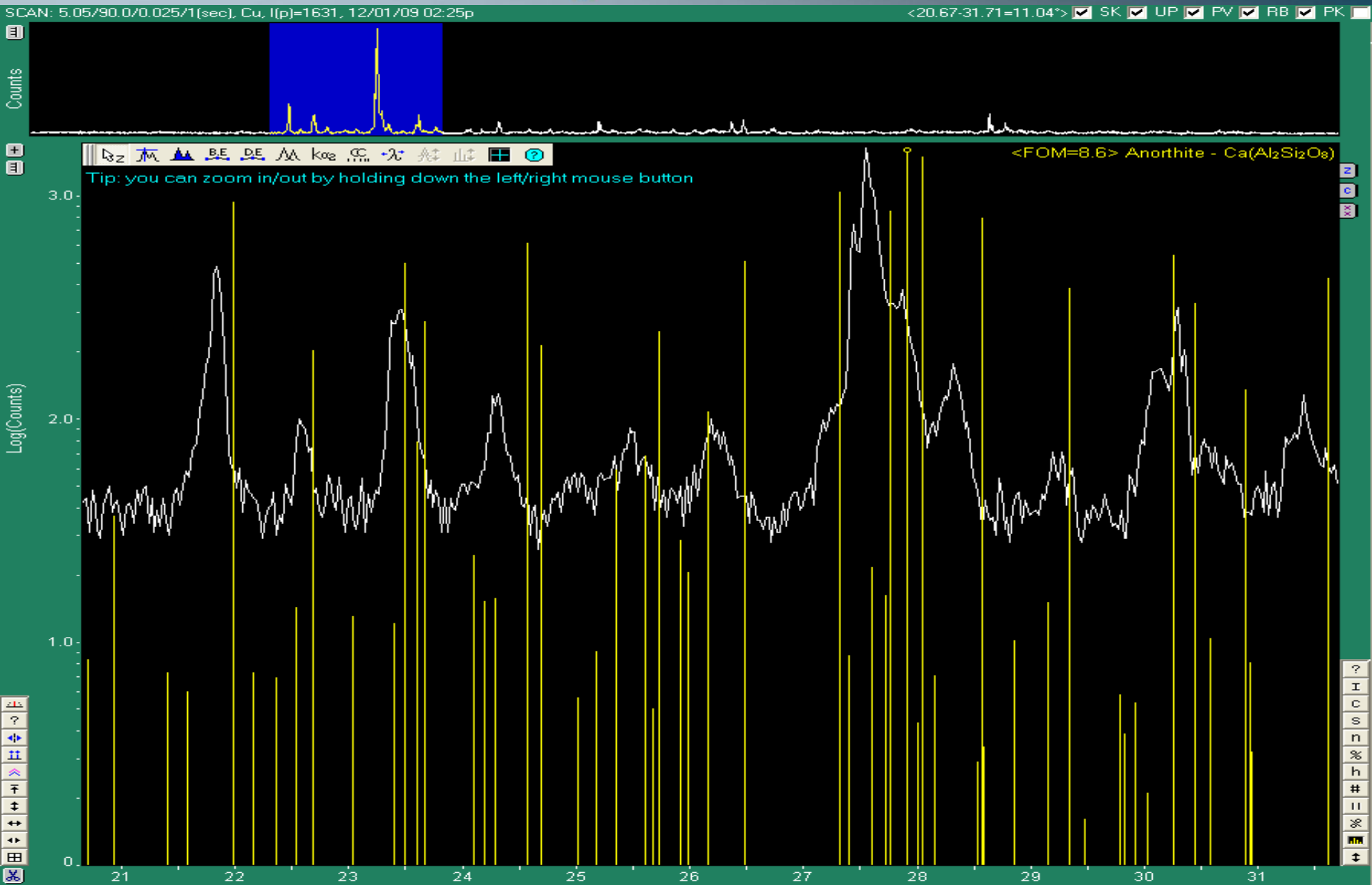
XRF Results of Outcrop

Formula	Concentration
Sample area density	2.6
SiO2	48.53%
CaO	24.88%
Al2O3	15.40%
MgO	5.90%
Fe2O3	4.78%
SO3	0.17%
TiO2	0.13%
P2O5	0.12%
SrO	0.03%
MnO	0.03%
Cu2O	0.02%
Ni2O3	0.01%
ZnO	0.01%

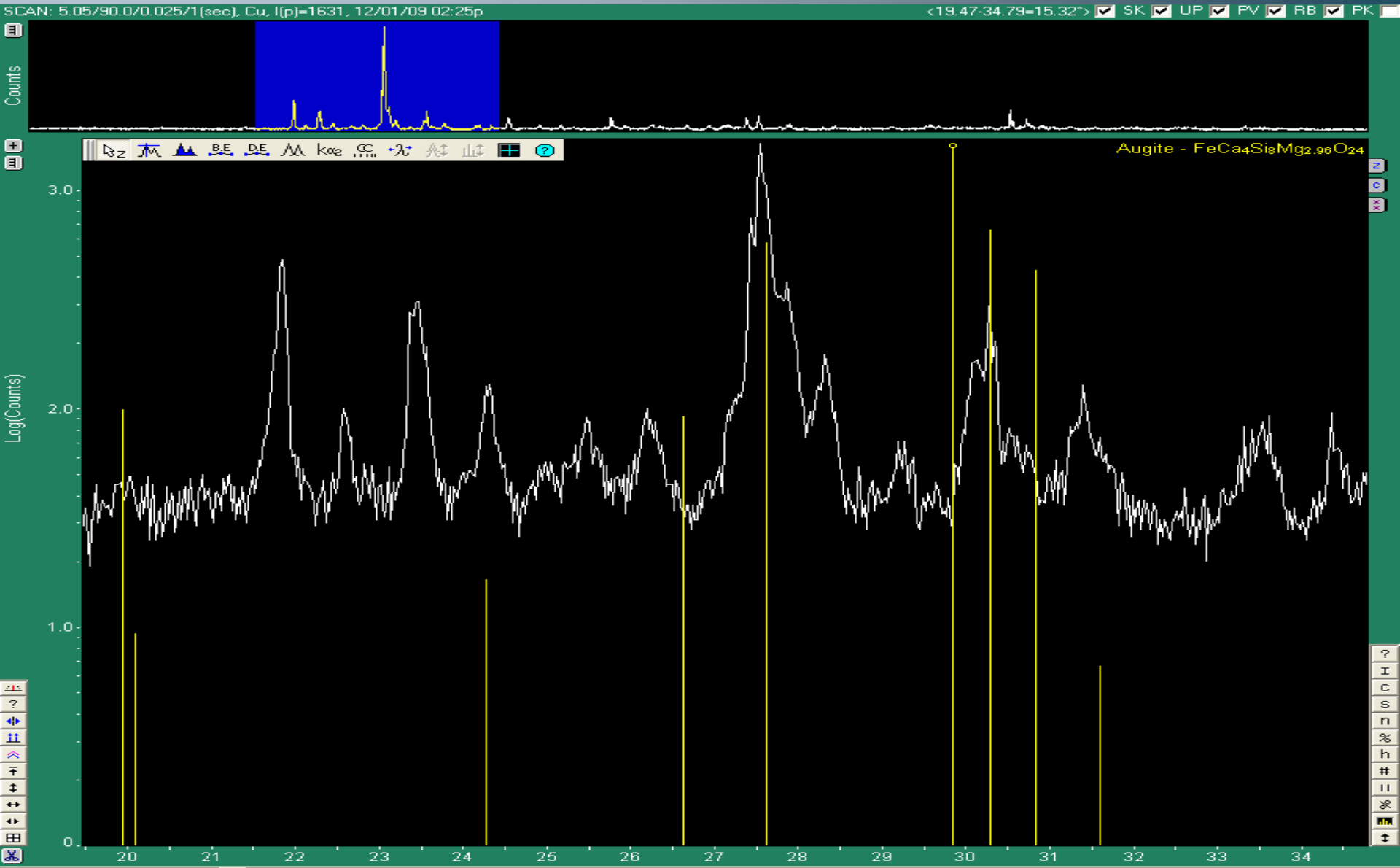
An Outcrop XRF



An Outcrop Anorthite Peaks



An Outcrop Augite Peaks



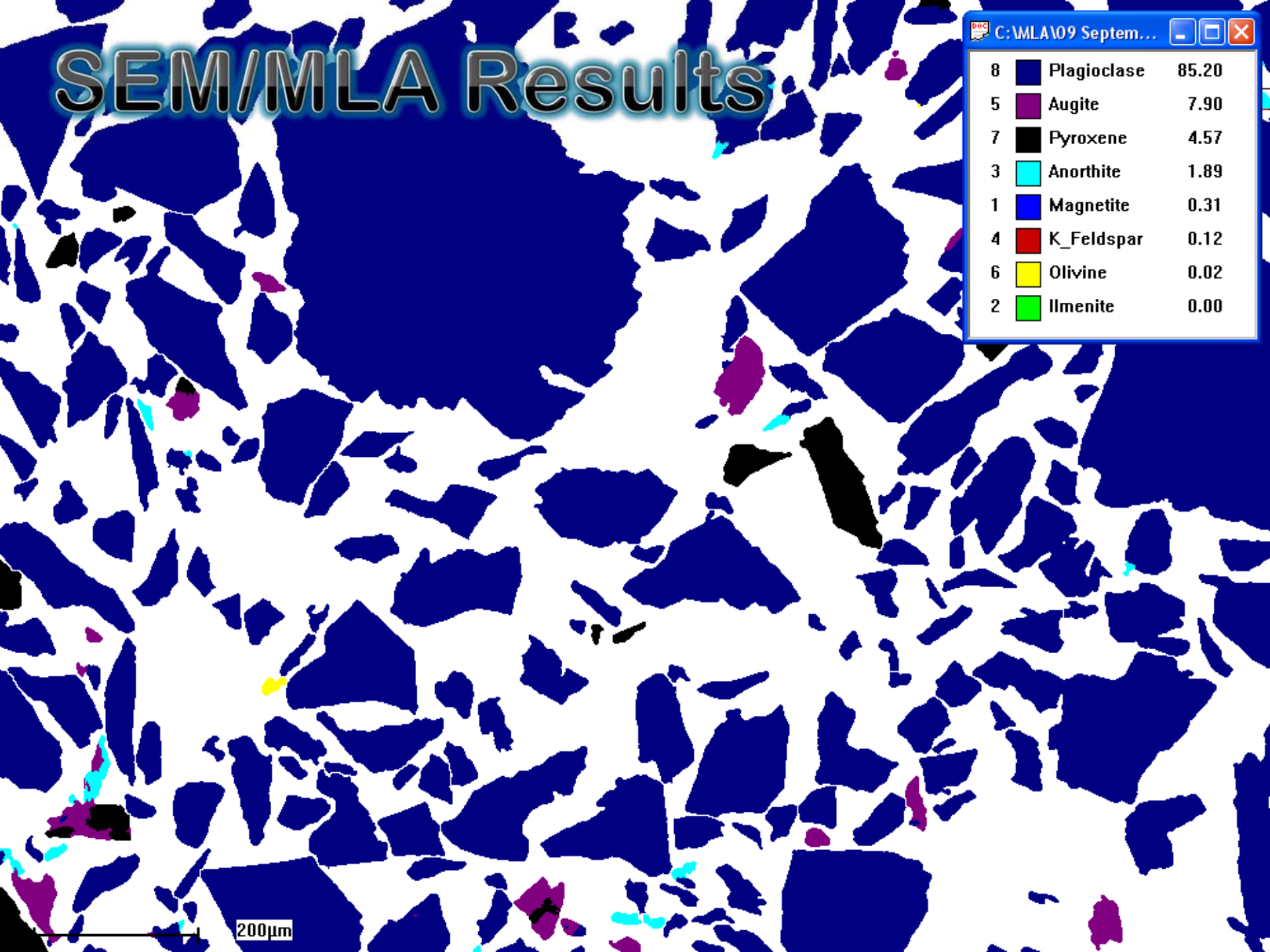
Physical Properties

Mineral	Chemical Formula	Specific Gravity	Magnetic Response	Hardness	Electrostatic Response
Plagioclase	$(Na, Ca)(Si,Al)_4O_8$	2.62-2.75	Non-Magnetic	6-6.5	Non-Conductive
Augite	$(Ca,Na)(Mg,Fe^{+2}, Al,Ti)[(Si,Al)_2O_6]$	3.2-3.5	Paramagnetic	5-6.5	Conductive*
Anorthite	$CaAl_2Si_2O_8$	2.72 - 2.75	Non-Magnetic	6	Non-Conductive**

**The response of some minerals can be varied with treatment at elevated temperature

Anorthite (waste)

SEM/MLA Results



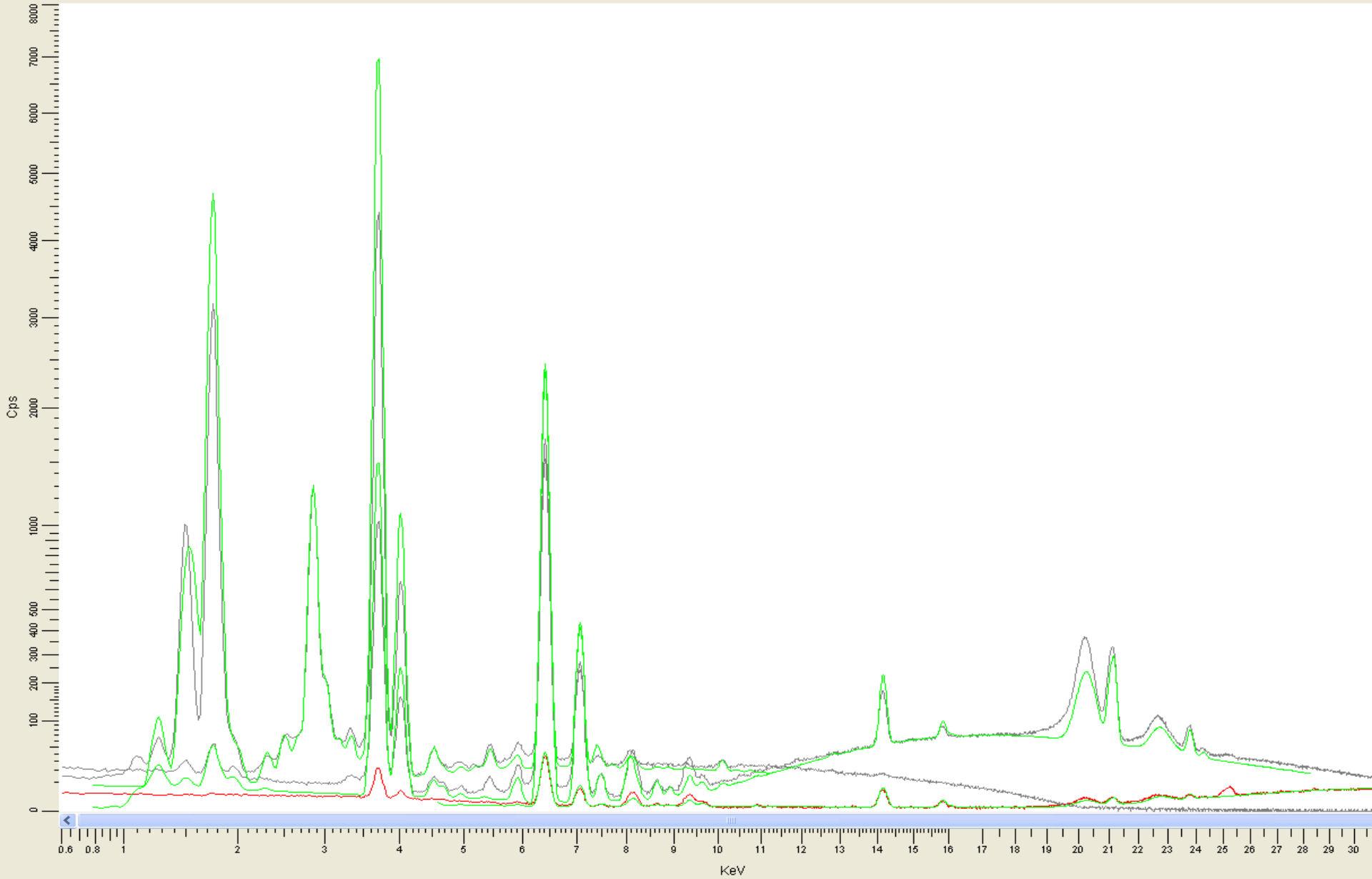
Minerals Present

- Plagioclase
- Augite
- Anorthite

XRF Results of Waste

Formula	Concentration
Sample area density	2.23
SiO2	49.36%
CaO	19.96%
Al2O3	15.78%
MgO	11.24%
Fe2O3	2.63%
SO3	0.31%
K2O	0.28%
P2O5	0.27%
TiO2	0.11%
SrO	0.03%
MnO	0.02%
Cu2O	0.01%

An Waste XRF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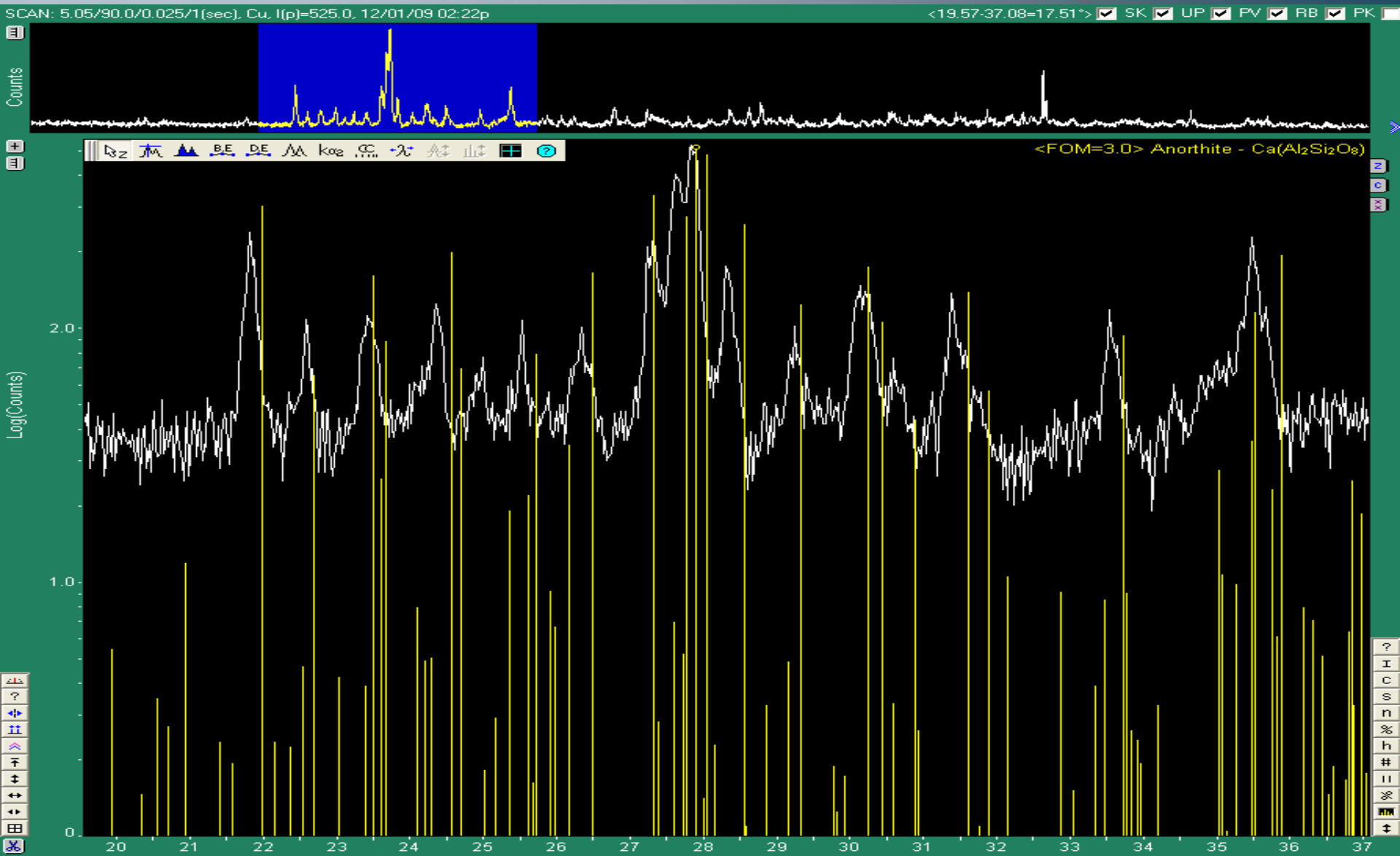
An Waste Anorthite Peaks



Physical Properties of Waste

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Augite	$(Ca,Na)(Mg,Fe^{+2}, Al,Ti)[(Si,Al)_2O_6]$	3.2-3.5	Paramagnetic	5-6.5	Conductive*
Pyroxene	$(Mg,Fe^{+2})_2Si_2O_6$	3.1-3.6	Paramagnetic	5-6.5	Non-Conductive**
Anorthite	$CaAl_2Si_2O_8$	2.72 - 2.75	Non-Magnetic	6	Non-Conductive**

*Variations in response to electrostatic separation will occur based on the actual composition of the mineral from varying locations and the presence of inclusions.

Conclusion

- A separation must be performed
- Too much background “noise”
- A more accurate An% can also be determined
- Appears that outcrop is a better source of Anorthite

Future Work

- Magnetic Separation
- Wet Sieve Analysis
- Dry Sieve Analysis
- Shaking Table (Wilfley Table)
- Electrostatic Separation

- Background
- Lunar Soil Mineralogy
- Project Statements & Highlights
- General Approach
- Geology of Stillwater Complex
- Available Technologies
- Current Results
- Conclusions
- Future Work
- Acknowledgements

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- Dr.'s Doug Stoeser & Steve Wilson, USGS
- Wikipedia, http://en.wikipedia.org/wiki/Wiki, 2009.