Microanalysis across the Curriculum – Integrating Electron Microprobe and Micro-XRF Instruments into Undergraduate Teaching and Research

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CONCORD UNIVERSITY

1 Abstract

In 2010, Concord University, a 2800-student, predominantly undergraduate institution in southern West Virginia, installed an ARL SEMQ microprobe as the first stage of a plan to establish a microanalytical facility that is open to outside commercial and academic users, with emphasis on undergraduate teaching and research. This instrument is also one of the exceedingly few research-grade electron microprobes located at an undergraduate institution.

The instrument recently received its first major upgrade since installation - a new high-sensitivity energy-dispersive (EDS) x-ray system based on a 30mm^2 active area silicon drift detector (SDD) with light-element capability. The detector is complemented by a comprehensive software package which allows for rapid X-ray and phase mapping, spectral imaging, and automated particle characterization.

Concord University also houses a Horiba XGT-5000 micro-X-ray fluorescence (XRF) analytical microscope for qualitative elemental mapping of areas as large as 100 cm^2 with a resolution of 0.01-0.1 mm.

This equipment affords outstanding opportunities for our students who have taken to the new capabilities with enthusiasm. We have begun the process of incorporating microanalysis into the curriculum at all levels from introductory general education to advanced major courses in multiple disciplines, including the earth sciences, chemistry, and physics/material science. Initial enrollment in our new microanalysis course, first offered in spring 2012, exceeded expectations. Both instruments are also used in an active and growing student-faculty research program. We are currently working microanalysis into additional courses and course sections, and we are formulating plans to extend this to our satellite campus by developing remote-operation and videoconferencing capabilities.

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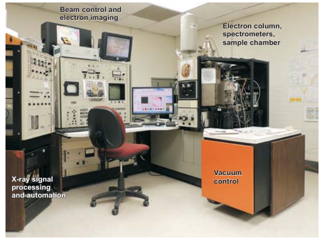
The West Virginia Research Trust Fund supported the installation and initial operation of the electron microprobe.

A West Virginia EPSCoR Innovation Grant funded 2/3 of the EDS upgrade.

2 The Instruments

ARL SEMQ Electron Microprobe



Capable of electron imaging, point analysis (EDS and WDS), and X-ray mapping/imaging (including spectral imaging/hypermapping)

Higher-sensitivity EDS detector for very rapid analysis.

Can detect elements as light as beryllium; Can analyze for oxygen and carbon

Higher resolution / limited to smaller samples

Significant sample preparation typically required

Horiba XGT-5000 micro-XRF

Capable of point analysis and X-ray mapping/imaging (including transmitted X-rays)

Can detect elements only as light as sodium (limited sensitivity to sodium); Unable to detect oxygen or carbon.

Lower resolution / larger samples possible

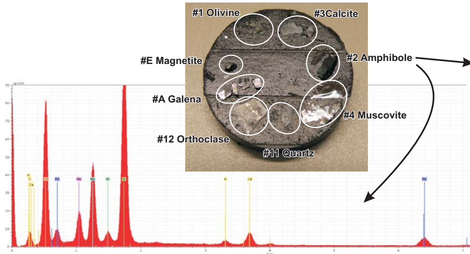
Little or no sample preparation; Hand-samples can be loaded and analyzed immediately.

Mineral and rock samples mounted, carbon-coated, and ready for the microprobe

Mineral samples ready for the XRF

3 Microanalysis in Geology 101

Mineral Specimens - Spot Analyses: *Chemistry as a tool in mineral identification; and Thinking of minerals as compounds.*



Spectrum: Mineral 2.spx

Element	wt.%	at.%	Oxide	wt.%
Oxygen	40.3	56.6		
Aluminium	1.0	0.9	Al2O3	2.0
Silicon	22.5	18.0	SiO2	48.2
Sodium	4.3	4.2	Na2O	5.8
Magnesium	8.7	8.0	MgO	14.5
Potassium	1.6	0.9	K2O	1.9
Calcium	5.1	2.9	CaO	7.1
Titanium	0.3	0.1	TiO2	0.5
Iron	12.8	5.2	FeO	16.5
Manganese	0.5	0.2	MnO	0.6

Rocks - Spot Analyses, X-ray Maps, & Area Analyses: *Understanding the hierarchy of elements, minerals / compounds, and aggregates (rocks); Identifying minerals in context; Using minerals to describe & identify rocks*

Spot analyses

Spectrum: Rock M, Crystal 1

Element	wt.%	at.%	Oxide	wt.%
Oxygen	41.8	56.8		0.0
Magnesium	27.5	24.6	MgO	45.6
Silicon	17.5	13.5	SiO2	37.4
Iron	13.2	5.1	FeO	17.0

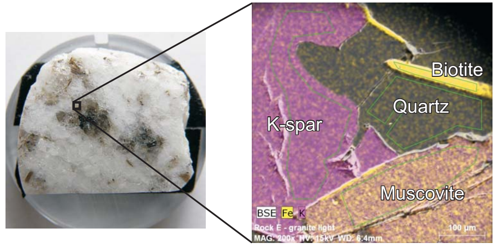
Identification: Olivine

Spectrum: Rock M, Crystal 2

Element	wt.%	at.%	Oxide	wt.%
Oxygen	43.4	59.7		
Magnesium	9.7	8.8	MgO	16.1
Iron	4.3	1.7	FeO	5.5
Silicon	22.5	17.6	SiO2	48.2
Calcium	15.7	8.6	CaO	22.0
Aluminium	4.4	3.5	Al2O3	8.2

Identification: Pyroxene

X-ray mapping



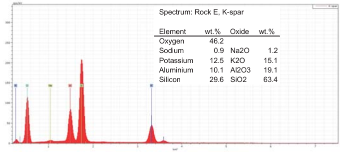
One of many possible views of an X-ray hypermap (full EDS spectrum for every pixel). Here, Fe and K are overlain on an electron image. Acquisition time was only 2-minutes.

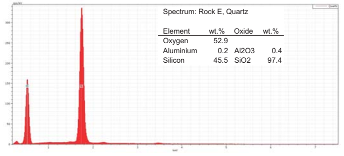
The green outlines mark the regions used to generate composite spectra and analyses shown in the next box.

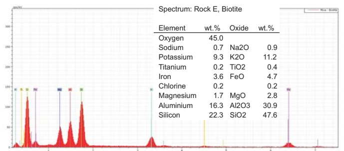
4 Microanalysis in Geology 101

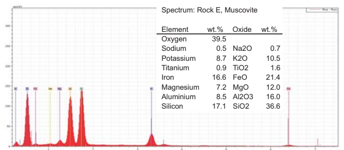
Rocks - Spot Analyses, X-ray Maps, & Area Analyses - *continued*

Composite EDS spectra and semi-quantitative analyses extracted from selected regions of the X-ray map. Together these show that elements combine to form minerals which in turn form rocks.









5 Microanalysis in Student Research

Example: chemical fingerprinting of volcanic ash for tephrochronology

CU1169 – Tephra sample from Carp Lake, WA (Normalized values)

Point No.	SiO2	TiO2	Al2O3	FeO	MnO	MgO	CaO	Na2O	K2O	P2O5	Cl	Total	H2Odiff
66	71.65	0.40	14.90	2.08	0.04	0.52	1.70	5.64	2.86	0.06	0.19	100.00	4.70
67	71.47	0.43	14.45	2.17	0.07	0.49	1.65	6.26	2.90	-0.05	0.21	100.00	8.47
68	72.37	0.43	14.15	1.91	0.04	0.51	1.72	5.74	2.86	0.11	0.19	100.00	1.98
69	72.75	0.45	14.40	2.04	0.07	0.55	1.67	5.05	2.78	0.08	0.21	100.00	2.65
71	72.67	0.42	13.82	1.94	0.05	0.50	1.65	5.93	2.82	0.07	0.18	100.00	5.15
72	71.49	0.43	14.42	2.03	0.06	0.55	1.62	6.04	3.17	0.04	0.18	100.00	7.64
73	72.67	0.41	13.84	2.03	0.03	0.50	1.69	5.84	2.80	0.04	0.20	100.00	5.40
75	71.47	0.48	14.27	2.09	0.05	0.49	1.86	6.15	2.87	0.10	0.20	100.00	7.00
76	73.86	0.34	13.17	1.99	0.06	0.50	1.55	5.47	2.85	0.09	0.19	100.00	2.36
77	72.51	0.45	13.88	2.00	0.02	0.48	1.70	5.77	3.02	0.04	0.18	100.00	2.06
78	72.72	0.39	13.81	2.06	0.07	0.52	1.56	5.80	2.84	0.08	0.20	100.00	5.82
AVERAGE	**72.33**	**0.42**	**14.10**	**2.03**	**0.05**	**0.51**	**1.67**	**5.79**	**2.89**	**0.06**	**0.19**	**100.00**	**4.84**
StDev	0.75	0.04	0.46	0.07	0.02	0.02	0.08	0.33	0.11	0.04	0.01	0.00	2.32

Reference analysis:

Sample CB-39 (by J.O. Davis, 1982) in Eruptive history of Mt. Mazama and Crater Lk Caldera by C.R. Bacon

	SiO2	TiO2	Al2O3	FeO	MnO	MgO	CaO	Na2O	K2O	P2O5	Cl	Total	H2Odiff
Average	72.70	0.44	14.66	2.03		0.48	1.61	5.22	2.71		0.18	100.00	

Result: Sample identified as Mt. Mazama (Crater Lake, OR) tephra, ~7630 cal yr BP