

Project Summary

Question:

"Does the speciation of Sulfur into Elemental Sulfur, Sulfide, Sulphate and Phosphorus into Phosphate change in the soil of Northern Alberta at various depths when exposed to extreme heat caused by wildfire, If so, what are the relative concentrations of each species before and after the fire at the same depth."

Scan List:

- 1) XRF Wildfire source 1, 2cm
- 2) XRF No Wildfire source 1, 2cm
- 3) XRF Wildfire source 1, 5cm
- 4) XRF No Wildfire source 1, 5cm
- 5) XRF Wildfire source 1, 10cm
- 6) XRF No Wildfire source 1, 10cm
- 7) XANES Sulfur Wildfire source 1, 2cm
- 8) XANES Sulfur No Wildfire source 1, 2cm
- 9) Standards Sulfur Ferric Sulphate
- 10) Standards Sulfur Iron(II) Sulfide
- 11) Standards Sulfur Elemental Sulfur
- 12) Standards Sulfur Potassium Sulphate
- 13) Standards Sulfur Copper Sulfide
- 14) Standards Sulfur Zinc Sulfide
- 15) Standards Sulfur Calcium Sulphate
- 16) XANES Sulfur Wildfire source 1, 5cm
- 17) XANES Sulfur No Wildfire source 1, 5cm
- 18) XANES Sulfur Wildfire source 1, 10cm
- 19) XANES Sulfur No Wildfire source 1, 10cm
- 20) XANES Phosphorous Wildfire source 1, 2cm
- 21) XANES Phosphorous No Wildfire source 1, 2cm
- 22) Standards Phosphorous Potassium Phosphate monobasic
- 23) Standards Phosphorous Ferric Phosphate
- 24) XANES Phosphorous Wildfire source 1, 5cm
- 25) XANES Phosphorous No Wildfire source 1, 5cm
- 26) XANES Phosphorous Wildfire source 1, 10cm
- 27) XANES Phosphorous No Wildfire source 1, 10cm
- 28) *Repeat for Second Wildfire and no wildfire source if there is extra time*

Oxidation States

Sulphate (+6) ex; Ferric Sulphate

Sulfide (-2) ex; Iron(II) Sulfide

Elemental Sulfur (0) ex; Elemental Sulfur

Phosphate (+5) ex; Ferric Phosphate

Our Standards (Bold=Using)

Potassium Phosphate monobasic

Potassium Sulphate

Ferric Sulphate

Calcium Sulphate

Elemental Sulfur

Copper Sulfide

Ferric Phosphate

Iron (II) Sulfide

Zinc Sulfide

Potassium Phosphate dibasic

Calcium Phosphate

Aluminum Phosphate

X-ray Critical Absorption and Emission Energies

Sulfur: K shell emission (eV)

Ka₁	Ka₂	Kb₁
2,307.84	2,306.64	2,464.04

Phosphorus : K shell emission (eV)

Ka₁	Ka₂	Kb₁
2,013.7	2,012.7	2,139.1

Phosphorus XRF ~2.142eV

Sulphur XRF ~2.470eV

XANES Sulphur Standards ~2.4825 KeV (Monochromator calibration)

XANES Phosphorus Standards ~2.1541 KeV (Monochromator calibration)

Notes On Techniques

X-ray Fluorescence (XRF)

XRF is a spectroscopy technique that allows us to view the relative concentration of elements in a sample. A beam of photons(light particles) is sent at the sample at a specific energy level. Electrons that exist at an energy level within the atom but not the highest energy level in the atom may absorb these photons and have enough energy to eject the electrons from the atom. When this happens the atom is left in an excited state and an electron from a higher energy level is sent down to occupy the “core hole,” left by the ejected electron. When that happens a photon is emitted from the atom to conserve energy, this is known as the fluorescent photon, the fluorescent photon has an energy that is equal to the difference in the energy levels of the electron that came down to fill the core hole and the electron that was ejected from the atom. There are different types of fluorescent photons that correspond to the difference between these energy levels. There is a letter assigned that is dependent on what energy level the core hole is present at. If the core hole is at the lowest energy level then the fluorescent photon is from the edge, the next level is called the edge, and so on. There is then a subscript that is assigned to the letter and further subscripts after that, but the naming is quite tedious. The energy levels of these fluorescent photons is well known which is why they are often times called characteristic X-Rays. The idea behind XRF is to measure the amount of photons absorbed by a detector at certain energies, so you measure counts of photons vs energy of photons. This will create a graph that has many different peaks that correspond to different fluorescent photons and different elements. There will also be a peak at the energy level of the photons that you sent in, this is known as the scatter peak. The scatter peak is the count of photons that were not absorbed by the sample. Here is an example of an XRF graph. (The scatter peak is labeled: Compton)

The higher the peak(more counts) of an element means the more of that element you have in a sample. A common technique used to compare XRF graphs of different samples to evaluate relative concentration is normalizing to the scatter peak. Essentially you divide the counts by the scatter peak counts of both graphs and then graph again so that the scatter peak of both graphs is 1.

X-Ray Absorption Near Edge Structure Spectroscopy (XANES)

XANES is a spectroscopy technique that allows us to see the relative concentration of different oxidation states of a single element. An oxidation state is the charge of an atom due to the number of electrons that are either gained or lost in a bond. Electrons usually exist at well defined energy levels, however when an atom has an oxidation state the electrons exist at a slightly shifted energy level. The idea of XANES is to find the shift and figure out the oxidation state(s) of an element in the sample. To do this, photons are slowly sent in over a range of energy. When the energy of the incoming photons reaches the required energy to eject an electron from a specific energy level from the atom there will be a large increase in the amount of photons absorbed. A XANES graph plots the counts of the photons that are absorbed against the energy level of the photons. Peaks with more counts correspond to oxidation states of higher concentration in the sample.

Linear Combination Fitting (LCF)

LCF is a data analysis technique that is used to find the relative concentrations of chemical species within a sample. In order to accomplish this there are many XANES scans that are needed, a XANES scan of the sample, and XANES scans of standards (standards are known chemical species). LCF is an attempt to add up all of the XANES scans of the standards in such a way that they recreate the XANES scan of the sample. This is done on a computer program(Athena is commonly used) the scans of the standards are given a weighting such that the sum of the weights of the standards are equal to 1. Comparing the weighting that the computer program gives to each standard gives you an idea of the relative concentrations of each species. Larger weighting means a higher concentration.