

## LABORATORY REPORT

Pennsylvania Department of  
Environmental Protection  
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Report Date: May 20, 2021  
Samples Received: May 06, 2021  
RJ Lee Group Job No.: PA070520210015  
Client Job No.: N/A  
Purchase Order No.: N/A

### **ANALYSIS: X-ray diffraction (XRD) for crystalline phases**

### **METHOD: Qualitative Phase Identification and Expansive Clay Determination**

Samples were received at RJ Lee Group in good condition. A portion of each sample was pipetted onto zero-background holders in order to preferentially orient the platy minerals and exaggerate the (00 $\ell$ ) basal spacing. The slides were allowed to air dry at room temperature. After drying, the samples were scanned on a PANalytical X'Pert Pro diffractometer using copper radiation. The samples were next placed in a desiccator filled with ethylene glycol. This step serves to expand any potential swelling clays. After removal from the desiccator, the samples were again scanned by XRD. The various scans were overlaid, the reflections were examined and the evolution of each was compared to the USGS Clay Mineral Identification Flow Diagram to determine which mineral each peak corresponds to. Results are presented below.

A portion of each dried sample was ground and scanned on a PANalytical X'Pert Pro diffractometer using copper radiation and standard run parameters. The resulting diffraction patterns were then search-matched with PANalytical X'Pert HighScore software against phases in the ICDD PDF4+ database. Concentrations presented below are estimated based on peak intensities of identified crystalline phases only. Major concentrations denote phases that are estimated to make up more than 20% of the material by weight, minor concentrations estimate concentrations in the material between 20% and 5% by weight and trace concentration estimates a phases present in the sample at concentrations less than 5% by weight. Estimations may vary, as factors such as preferred orientation and the ability of each material to diffract x-rays, as well as phased concentration will affect peak intensities. Additionally, amorphous material may not necessarily be detected by XRD. In certain cases where amorphous material is present in major concentrations, its presence is evidenced by a broad hump in the background signal of an XRD scan, however minor concentrations of amorphous material may be present in a material with no evidence in the scan. Further, XRD is generally accepted to have a detection limit of approximately a few weight percent, depending on phase. It is possible that trace phases are present in the samples that remain unidentified.

**Table 1. Phase identification of "1102-001" (RJLG Sample 001) by XRD**

Phase*	Approximate Composition**	Estimated Concentration <sup>+</sup>
Quartz	SiO <sub>2</sub>	Major
Chlorite Group	(Mg,Al,Fe,Ni,Mn) <sub>6</sub> Al(Al,Si <sub>3</sub> )O <sub>10</sub> (OH) <sub>8</sub>	Trace
Mica/Illite	K(Al,Mg,Fe) <sub>2</sub> (AlSi <sub>3</sub> O <sub>10</sub> )(F,OH) <sub>2</sub>	Trace
Feldspar(s)	(K,Na)AlSi <sub>3</sub> O <sub>8</sub>	Trace
Monoclinic Amphibole***	(Na,Ca,Fe,Mg) <sub>7</sub> Si <sub>8</sub> O <sub>22</sub> (OH) <sub>2</sub>	Trace

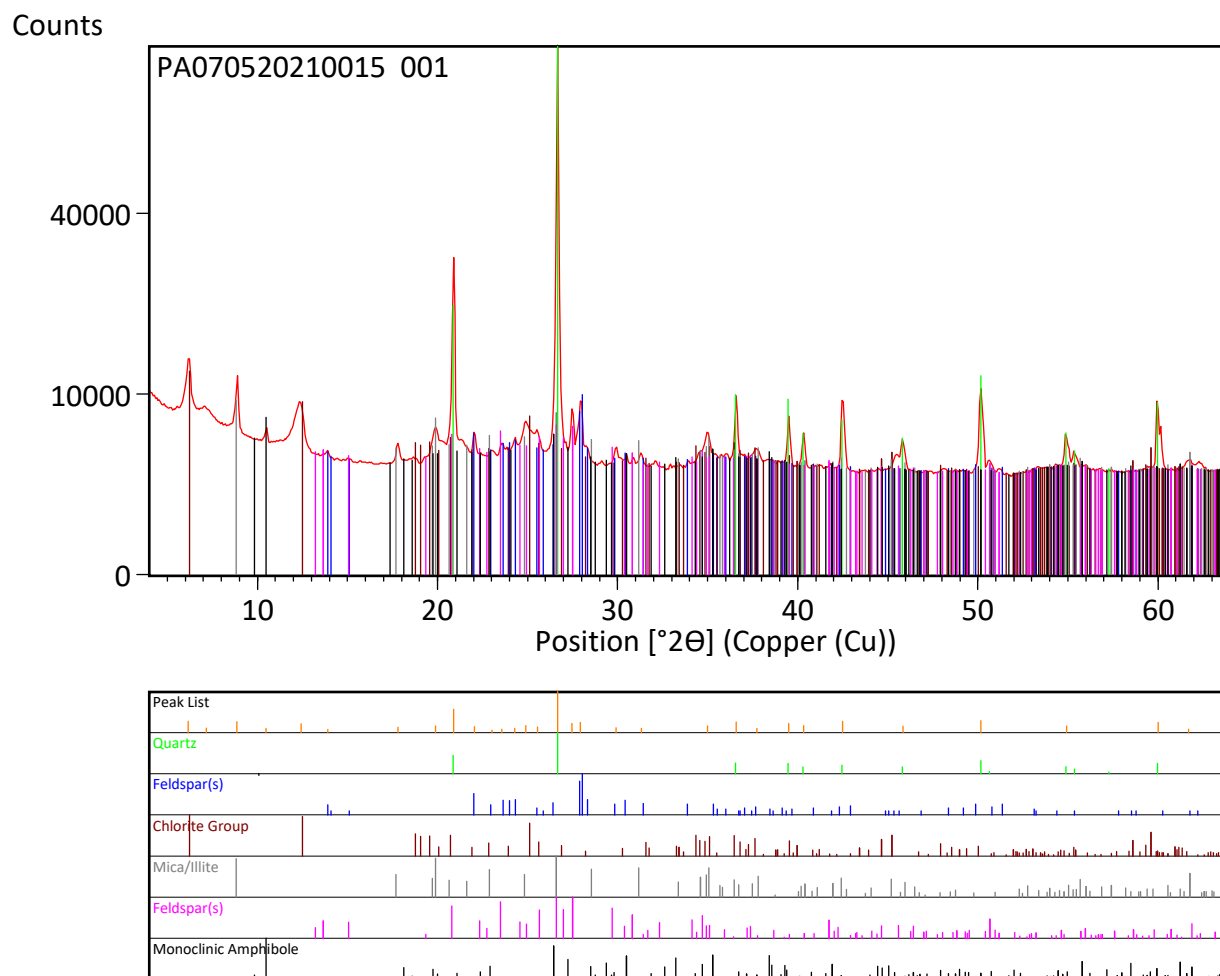
\*Amorphous content, crystalline phases present at trace levels and phases that are not currently part of the ICDD PDF 4+ database may remain unidentified.

\*\*Compositions are approximate and represent an idealized formula for that structure, not including possible elemental substitutions into that crystal structure.

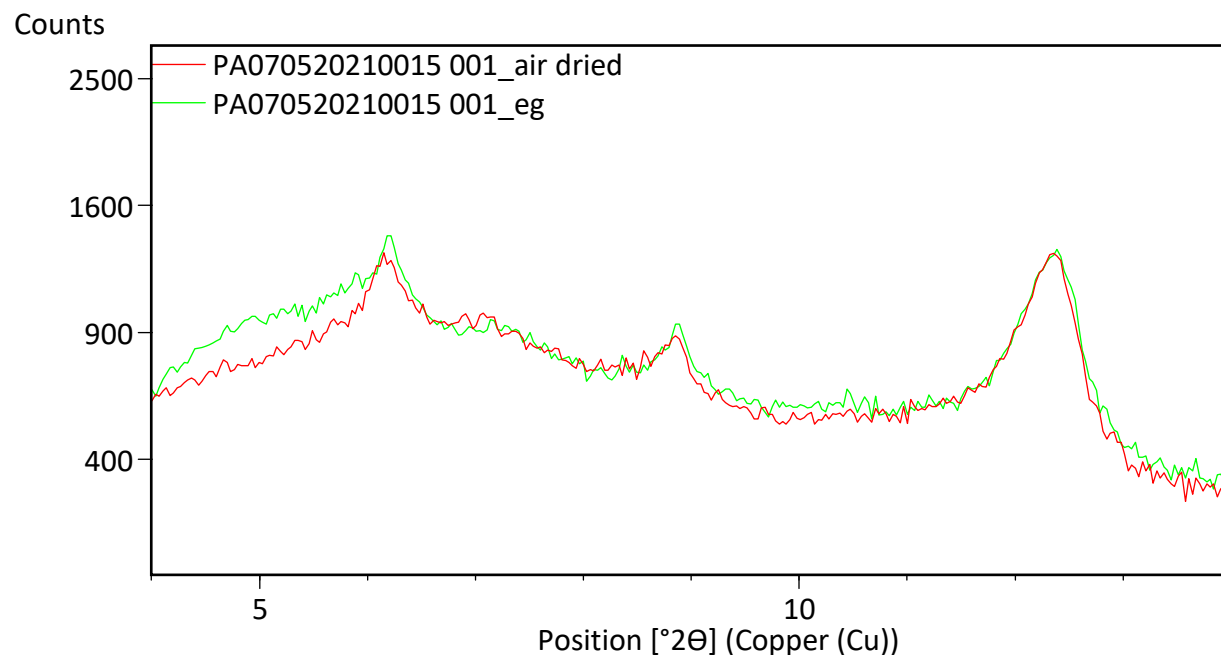
\*\*\* Further testing is necessary to confirm amphibole phases.

+Estimated concentration is based off of the dried solid material.

Note: Bentonite/montmorillonite was not detected.



**Figure 1 –X-ray diffraction pattern of as-received specimen "1102-01", with position (degrees 2θ) along the x-axis and intensity (counts) along the y-axis. Corresponding legend denoting phase matches (bottom).**



**Figure 2 – Overlay of x-ray diffraction patterns for specimens: “1102-01\_air dried,” (in red), “1102-01\_eg,” (in green), with position (degrees 2θ) along the x-axis and intensity (counts) along the y-axis. The patterns have been zoomed to the region between 4-14°2θ to show detail.**

**Table 2. Phase identification of "1102-02" (RJLG Sample 002) by XRD**

Phase*	Approximate Composition**	Estimated Concentration <sup>+</sup> (weight %)
Quartz	SiO <sub>2</sub>	Major
Chlorite Group	(Mg,Al,Fe,Ni,Mn) <sub>6</sub> Al(Al,Si <sub>3</sub> )O <sub>10</sub> (OH) <sub>8</sub>	Trace
Mica/Illite	K(Al,Mg,Fe) <sub>2</sub> (AlSi <sub>3</sub> O <sub>10</sub> )(F,OH) <sub>2</sub>	Trace
Feldspar(s)	(K,Na)AlSi <sub>3</sub> O <sub>8</sub>	Trace
Monoclinic Amphibole***	(Na,Ca,Fe,Mg) <sub>7</sub> Si <sub>8</sub> O <sub>22</sub> (OH) <sub>2</sub>	Trace

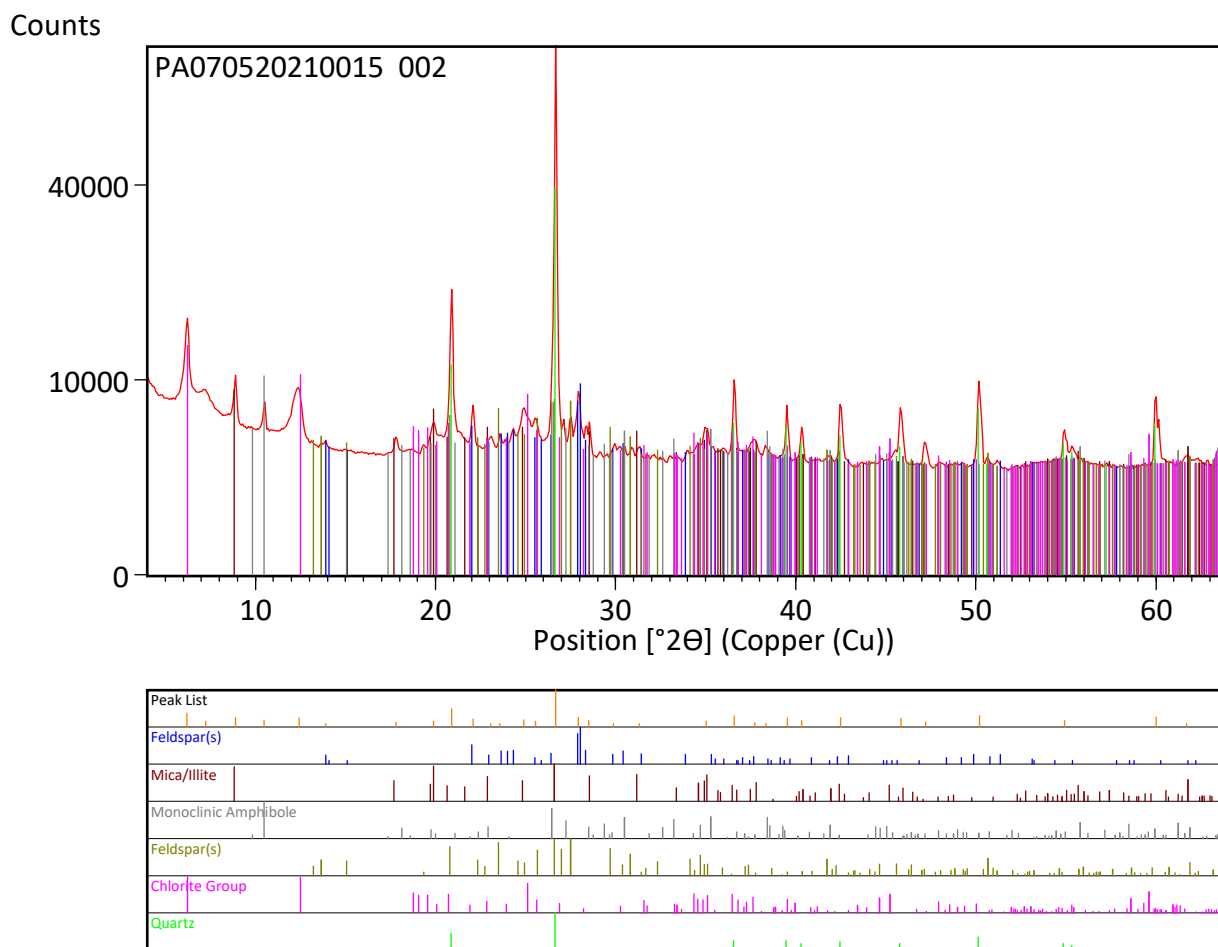
\*Amorphous content, crystalline phases present at trace levels and phases that are not currently part of the ICDD PDF 4+ database may remain unidentified.

\*\*Compositions are approximate and represent an idealized formula for that structure, not including possible elemental substitutions into that crystal structure.

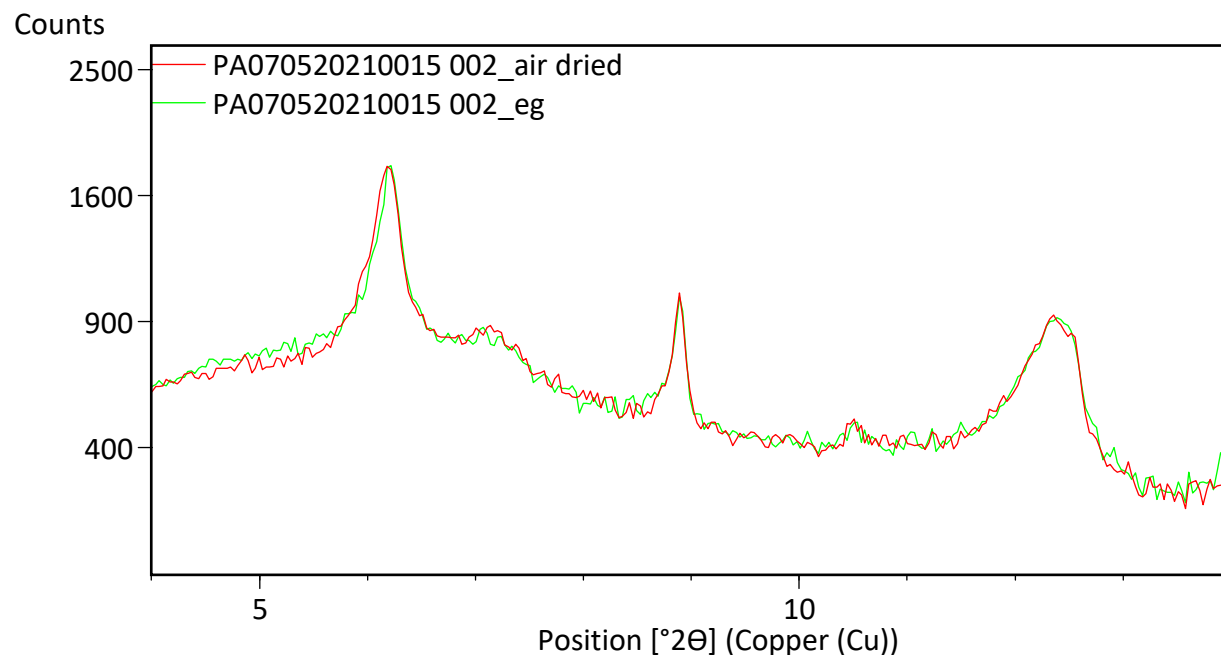
\*\*\* Further testing is necessary to confirm amphibole phases.

+Estimated concentration is based off of the dried solid material.

Note: Bentonite/montmorillonite was not detected.



**Figure 3 –X-ray diffraction pattern of as-received specimen "1102-02", with position (degrees 2θ) along the x-axis and intensity (counts) along the y-axis. Corresponding legend denoting phase matches (bottom).**



**Figure 4 – Overlay of x-ray diffraction patterns for specimens: “1102-02\_ air dried,” (in red), “1102-02\_eg,” (in green), with position (degrees 2θ) along the x-axis and intensity (counts) along the y-axis. The patterns have been zoomed to the region between 4-14°2θ to show detail.**

**Table 3. Phase identification of "1102-03" (RJLG Sample 003) by XRD**

Phase*	Approximate Composition**	Estimated Concentration+ (weight %)
Quartz	SiO <sub>2</sub>	Major
Chlorite Group	(Mg,Al,Fe,Ni,Mn) <sub>6</sub> Al(Al,Si <sub>3</sub> )O <sub>10</sub> (OH) <sub>8</sub>	Trace
Mica/Illite	K(Al,Mg,Fe) <sub>2</sub> (AlSi <sub>3</sub> O <sub>10</sub> )(F,OH) <sub>2</sub>	Trace
Feldspar(s)	(K,Na)AlSi <sub>3</sub> O <sub>8</sub>	Trace
Monoclinic Amphibole***	(Na,Ca,Fe,Mg) <sub>7</sub> Si <sub>8</sub> O <sub>22</sub> (OH) <sub>2</sub>	Trace

\*Amorphous content, crystalline phases present at trace levels and phases that are not currently part of the ICDD PDF 4+ database may remain unidentified.

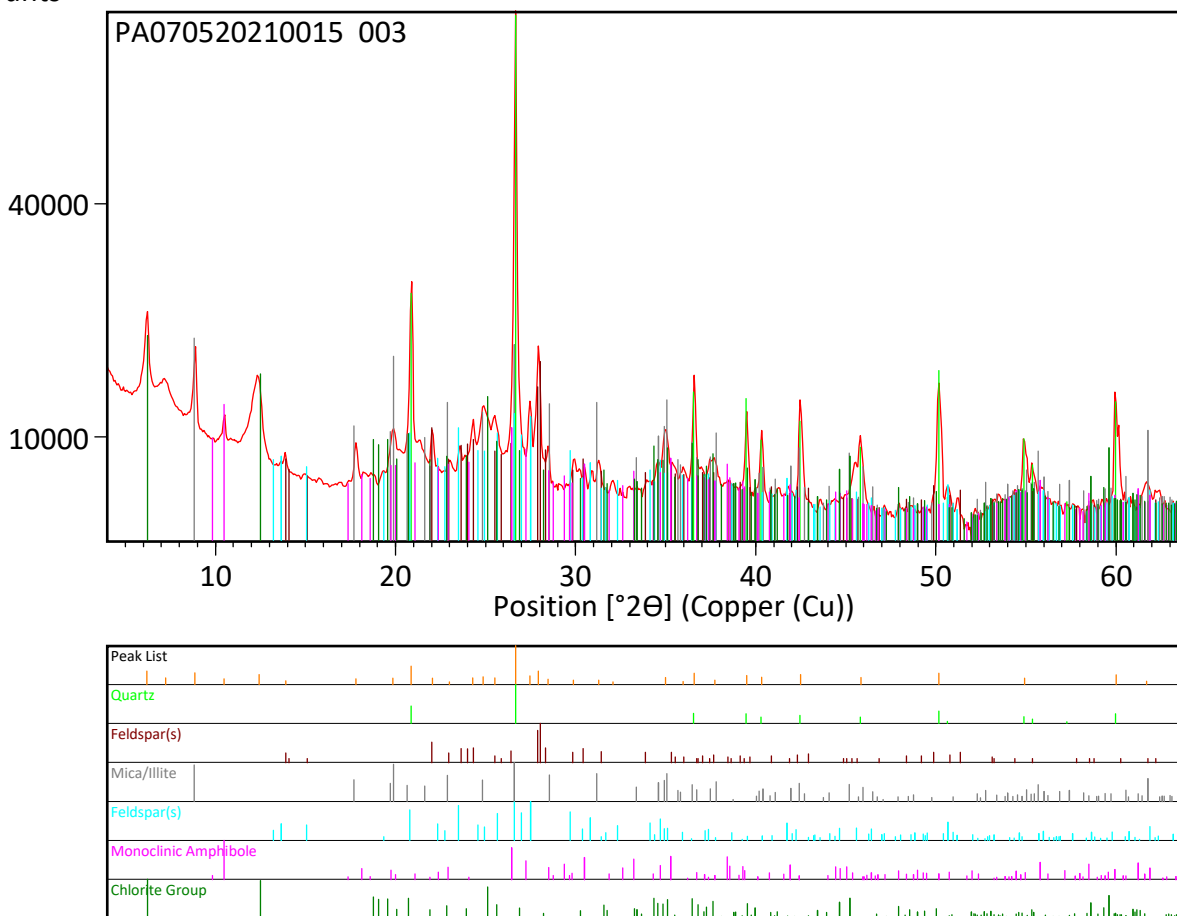
\*\*Compositions are approximate and represent an idealized formula for that structure, not including possible elemental substitutions into that crystal structure.

\*\*\* Further testing is necessary to confirm amphibole phases.

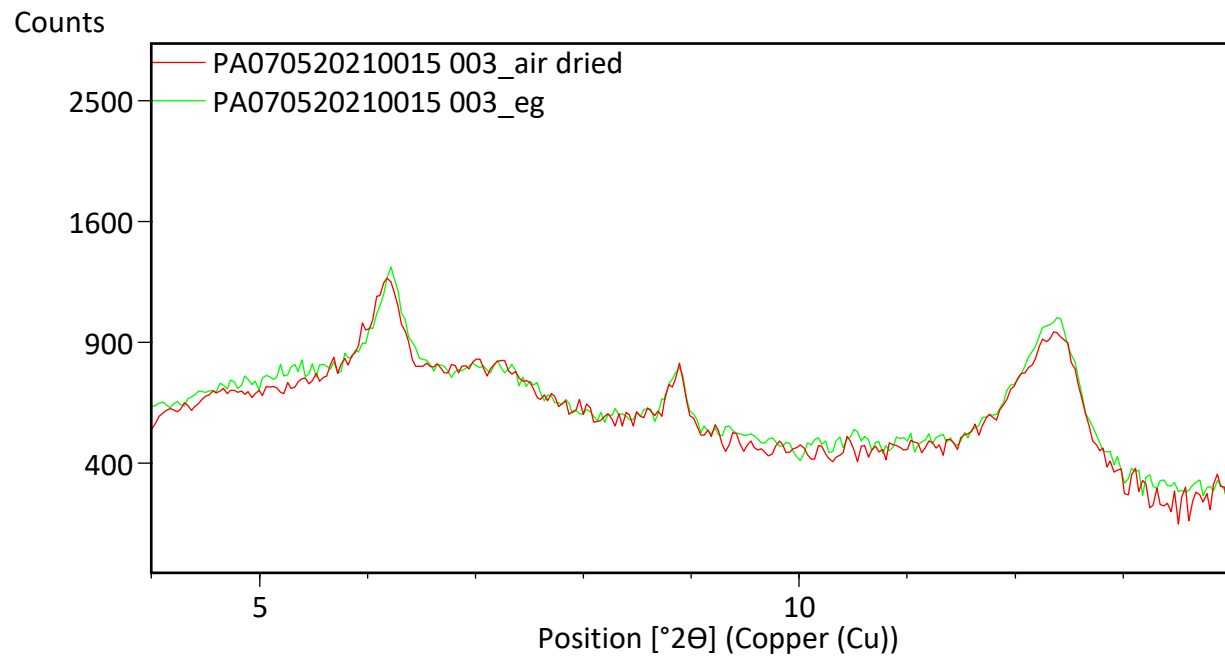
+Estimated concentration is based off of the dried solid material.

Note: Bentonite/montmorillonite was not detected.

Counts



**Figure 5 –X-ray diffraction pattern of as-received specimen "1102-03", with position (degrees 2θ) along the x-axis and intensity (counts) along the y-axis. Corresponding legend denoting phase matches (bottom).**



**Figure 6 – Overlay of x-ray diffraction patterns for specimens: “1102-03\_air dried,” (in red), “1102-03\_eg,” (in green), with position (degrees 2θ) along the x-axis and intensity (counts) along the y-axis. The patterns have been zoomed to the region between 4-14°2θ to show detail.**

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This laboratory operates in accord with ISO 17025:2017 guidelines and holds a limited scope of accreditation. Please refer to <http://www.rjlg.com/about-us/accreditations/> for more information and current status.

Please feel free to contact us should you have any questions regarding this analysis or if we can be of further assistance to you.

Sincerely,



Sarah Candiello, Scientist

