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X-RAY DIFFRACTION (XRD) ANALYSIS REPORT
16 Dec 2015

JOB NUMBER C0FSV221
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for

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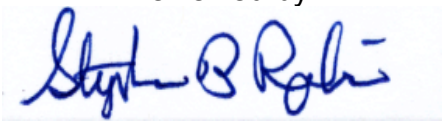

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Job Number: C0FSV221
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X-RAY DIFFRACTION ANALYSIS REPORT

Purpose: Use x-ray diffraction to determine the phases present in a sample of solids dried from a water sample.

Summary:

Table 1: Best Matches from the ICDD/ICSD data bases

Sample ID	Phases detected
Dried solids from water sample	Zincite ● ZnO Hexagonal, S.G: P63mc (186) Phase Info [01-070-8070] ZnO_2 ● Zinc Peroxide Cubic, S.G: Pa-3 (205) Phase Info [01-077-2414] $Zn_5(OH)_6(CO_3)_2$ ● Zinc Carbonate Hydroxide Crystal System: Unknown, S.G: Unknown Phase Info [00-054-0047] $CO(NH_2)_2$ ● Urea Tetragonal, S.G: P-421m (113) Phase Info [01-076-3886] Amorphous material

Results and Interpretations: The sample was received after XRF analysis where the solids were dried and pressed into a thin wafer-like sample. This sample was mounted using double-sided adhesive tape onto a glass-covered Al stub which was subsequently mounted in an x, y, z goniometric head. XRD data was collected in a standard Θ-2Θ configuration using a Bruker D8 Discover 6-axis microdiffractometer equipped with a Vantec-500 area detector, 500micron pinhole collimator and Copper micro-source X-ray tube. Unlike traditional point detectors, the 2D detector accepts diffraction from crystallites oriented in a wide variety of tilt angles with respect to the incident x-ray beam. This results in reasonable diffraction intensity even though the spot size analyzed is very small. With no collimation around the 2D detector, the peak

positions are very sensitive to sample height. To ensure that the sample height is correct, the GADDS employs a laser and camera system oriented so that the sample is in the correct position when the laser beam is in the center of the camera system. Photo 1 shows a high magnification alignment image of the dried solids sample with the analysis area located at the center of the crosshairs. The laser spot is clearly visible on the sample.

Figure 1 shows the best matches between the background-subtracted experimental data and the ICDD/ICSD diffraction database for the dried solids sample. The reference markers for the phases show the location in 2-Theta where a particular peak for a given phase should be located and the height of the marker indicates the expected intensity of the experimental peak, if the sample is a fine grained, randomly oriented sample.

This sample is composed of the hexagonal Zinc oxide phase known as Zincite, ZnO plus a cubic form of Zn peroxide, ZnO_2 and a tetragonal organic compound, Urea ($CO(NH_2)_2$). A fourth phase was found in the database, a Zinc Carbonate Hydroxide, but the database reference lists no crystal structure or space group for this phase. The Zinc peroxide phase should be considered a speculative match because of the large number of overlapping diffraction peaks. It also appears that there may be some amount of amorphous (non-crystalline) material in the sample. Amorphous material in XRD exhibits very broad, wide humps and there may be such amorphous humps in the 20-45° 2-Theta range and again in the 45-70° 2-theta range. However, XRD cannot determine the composition of amorphous materials. There may be other crystalline materials at low concentrations that XRD cannot observe or does not have enough diffraction peaks for identification. Typically XRD detection limits are about 1-3wt% and many of the elements in the XRF list (in the report from job C0FSD762) are well below XRD detection limits.

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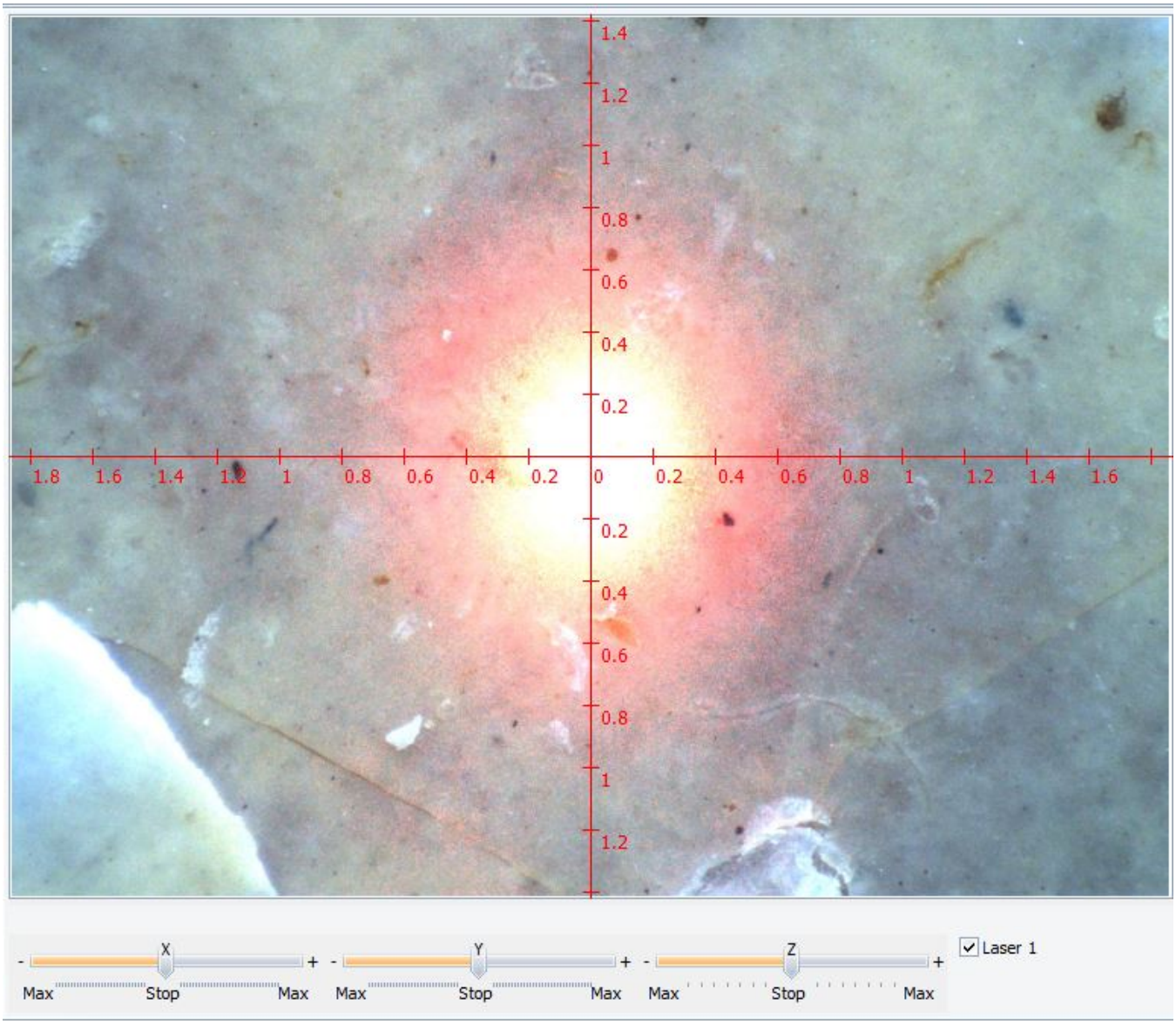

Photo 1: High magnification alignment image of dried solids from water sample.

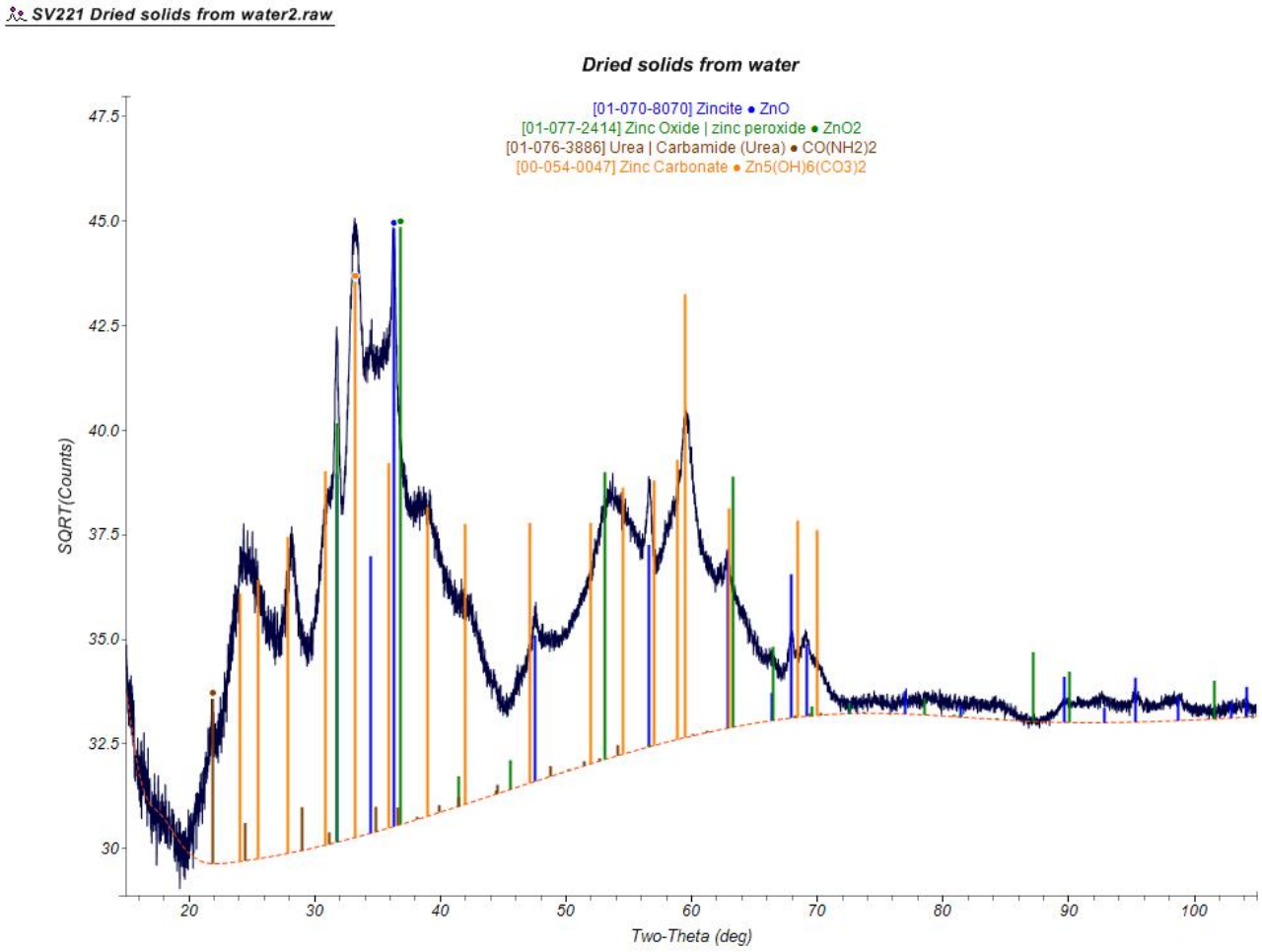

Figure 1: Phase identification from the dried solids from water sample.

Appendix

Measurement Uncertainty:

There are two types of uncertainty in XRD analysis; uncertainty in the number of x-ray counts at a particular angle and uncertainty in the diffraction angle. Because the arrival of X-ray quanta in the detector is random with respect to time, the accuracy of X-ray counting rate measurements is governed by the laws of probability. In particular, the size of the one sigma standard deviation in an X-ray measurement is equal to the square root of the number of X-rays counted. A conservative criterion for the detection of a weak peak in a XRD pattern must have amplitude of greater than three standard deviations above background. As a result, the more slowly a measurement is made, the lower the relative standard deviation in the number of counts measured and the more likely is detection of trace diffraction peaks. If X-ray data is acquired at a constant speed, the relative standard deviation for the major diffraction peaks in a pattern will be on the order of a few percent or less while the relative standard deviation for the weaker peaks in a pattern will be on the order of tens of percent or more. This also implies that the uncertainty in the concentrations of the major phases in a sample will be lower than for the trace phases. Please note that there are a number of sample related factors that can influence peak intensity. These include (but are not limited to): average crystallite size, preferred orientation (texture), strain, and absorption.

Uncertainty in the position of X-ray diffraction peaks is due to both instrumental and sample effects. Instrumental position uncertainty is primarily due to diffractometer misalignment. Repeat measurements of NIST standard reference materials has shown that the maximum positional uncertainty is less than +/- 0.05 degrees 2-Theta and is typically much less than that. Positional uncertainty due to sample effects are related to sample displacement (displacement of the sample surface either above or below the diffractometer focusing circle) and sample transparency (the effect gets larger as the sample matrix becomes more transparent to the incident X-rays. Through careful sample preparation, the uncertainty due to these two sample effects should be less than +/- 0.03 degrees 2-Theta. Please note that in addition to these factors, solid solution effects, where one element is partially substituted for another within a given crystal structure, can produce significant shifts in measured peak positions. Unlike sample and instrumental peak position effects, solid solution effects can result in phase misidentification.