

**X-RAY DIFFRACTION (XRD)
ANALYSIS REPORT
16 Oct 2017**

**JOB NUMBER F0HWZ477
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for

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X-RAY DIFFRACTION ANALYSIS REPORT

Purpose: Use X-ray diffraction to quantify the phase(s) present in a gypsum tile, as designated below.

Summary:

Table 1: Phase Identification and Concentrations

Sample ID	Phases present	Concentration (wt %) ± 5%
S1	Ca(SO ₄)(H ₂ O) ₂ – Gypsum Monoclinic, SG: I2/c (15) PDF# 01-074-1905	89.9
	Ca(CO ₃) – Aragonite Orthorhombic, SG: Pmcn (62) PDF# 01-078-4338	8.1
	Ca(SO ₄)(H ₂ O) _{0.5} – Bassanite Monoclinic, SG: I2 (5) PDF# 01-074-2787	0.9
	Ca(SO ₄) – Anhydrite Orthorhombic, SG: Amma (63) PDF# 01-072-0916	0.6
	C – Graphite Hexagonal, SG: P63/mmc (194) PDF# 01-083-6084	0.5
	Unknown material(s)	n/a

Results and Interpretations: The tile piece was ground lightly in a mortar and pestle and loaded into a glass sample holder. XRD data was collected by a coupled Theta-2Theta scan on a Rigaku SmartLab diffractometer equipped with copper X-ray tube with Ni beta filter, parafocusing optics, computer-controlled slits, and a D/teX Ultra 1D strip detector.

[Figure 1](#) and [Figure 2](#) show the coupled scan for the gypsum sample, superimposed with the reference patterns for the best matching phases from the ICDD/ICSD database. Any differences between the reference markers and the experimental peaks may be due to compositional differences. It is important to note that XRD is sensitive to crystal structure but relatively insensitive to elemental composition and/or chemical state. Peak height differences between the reference pattern and measured data can be explained by preferred crystallite orientation in the sample. Aside from gypsum, the primary phase in the sample, the phases have been identified by small, highly overlapping peaks and have more uncertainty than the gypsum phase identification. There are small peaks that have not been identified near 30° and 39.5° 2θ , and the small peak near 11° 2θ is believed to be an artifact from the high intensity of the gypsum peak to the right.

Semi-quantitative analysis was performed using WPF (whole pattern fitting), which is a subset of Rietveld Refinement that accounts for all areas above the background curve. This technique requires that either the structure factors and atomic locations or the reference intensity ratio (a way of comparing the diffracting power of different phases) are known for all phases identified. During this process, structure factor (which relates to concentration), lattice parameters (which relate to peak position), peak width and peak shape are refined for each phase to minimize the R value – an estimate of the agreement between the model and the experimental data over the entire pattern.

[Figure 3](#) shows the WPF results for S1 and the concentrations of the phases are listed in [Table 1](#). The R value for this refinement, 7.61%, is good for a pattern with a high number of overlapping peaks. The major sources of error are the many diffraction peaks in the patterns, several unidentified peaks, and the differences in relative peak intensities between the experimental data and the reference patterns.

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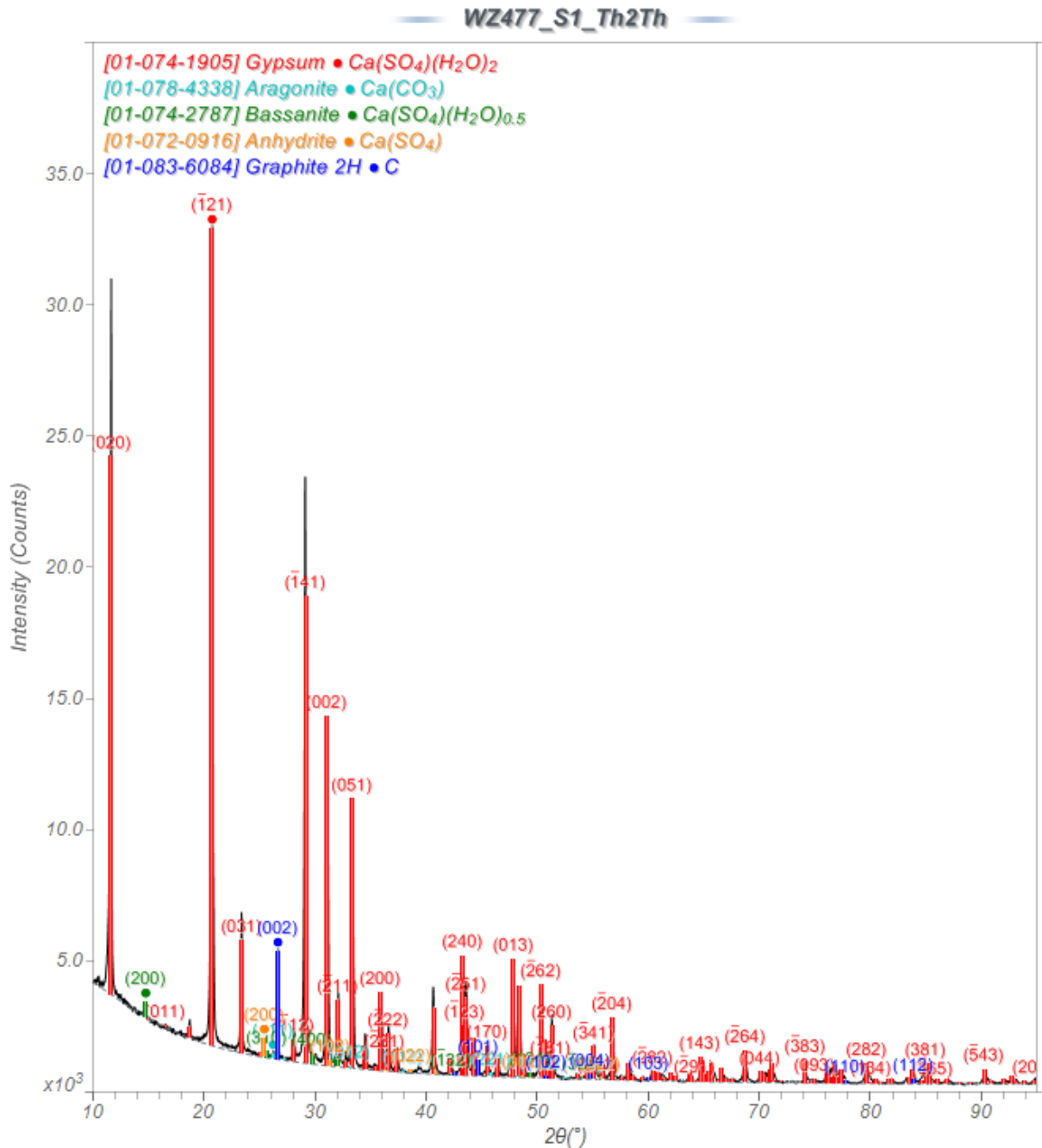


Figure 1: Phase identification for gypsum sample S1

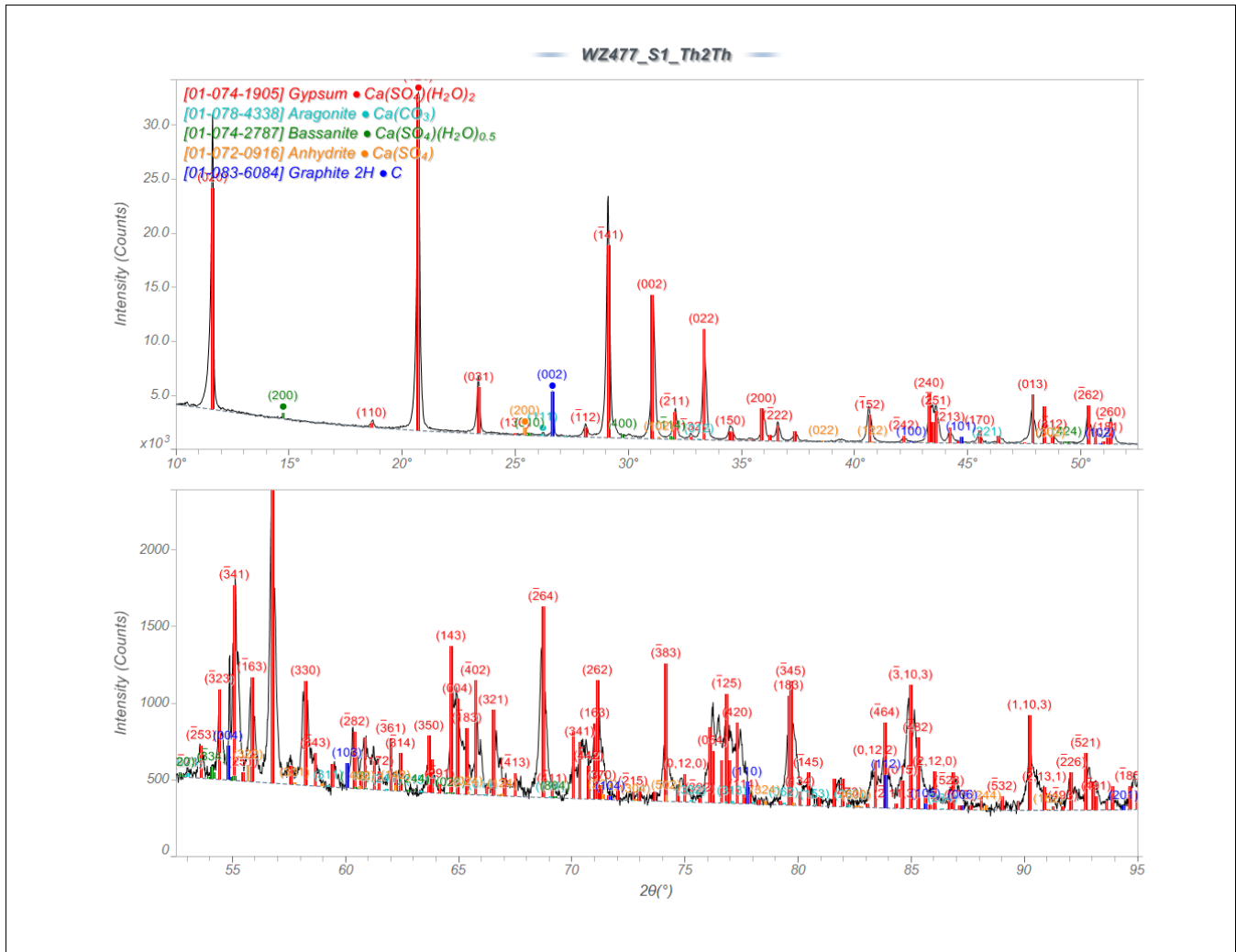


Figure 2: Phase identification for gypsum sample S1, expanded ranges

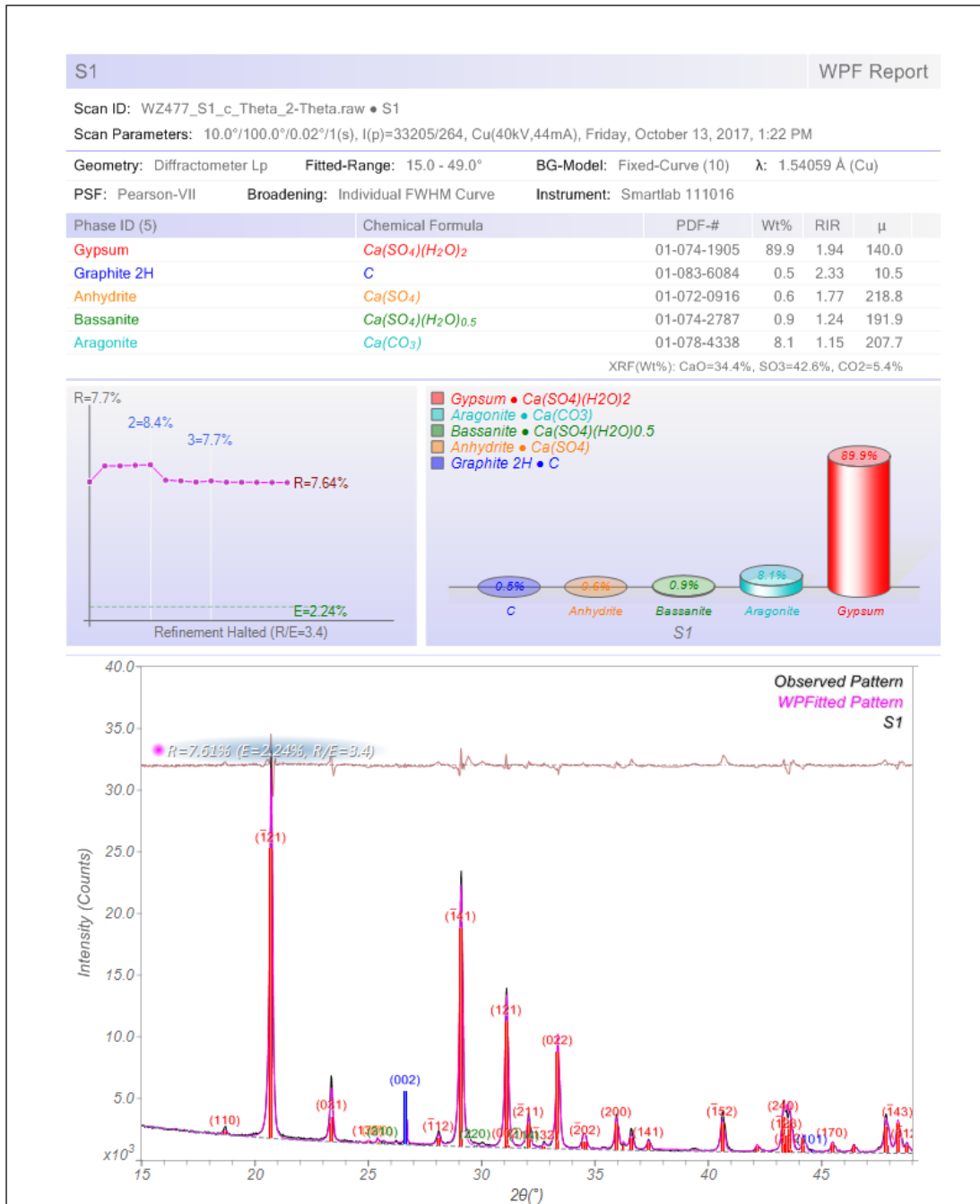


Figure 3: WPF results for gypsum sample S1

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.