XRD Technique, Reporting, and Sample Policies

The submission of samples for testing constitutes acknowledgment of the information, sample requirements, and financial responsibility of the submitter which are provided in this document.

Quantitative Analysis with spiking for Amorphous content:

- This analysis includes a description of the process and scan parameters, an image of the diffraction pattern before and after spiking, a Rietveld refinement to quantify any identified phases and a list of relative percentages of each identified phase as well as the calculated amorphous content derived from a new refinement of a spiked sample. If the pattern cannot be identified or refined, the order will be changed to a qualitative analysis. If spiking cannot be performed based on the initial scan, but the phases can be quantified, the order will be changed to quantitative without spiking.

General Sample requirements:

- Preferred sample quantity is 0.5g or more. Less than 0.1g is not generally suitable for spiking but may be possible if requested.
- Coarse powders that can be easily ground will be ground with a mortar and pestle prior to analysis.
- It is essential that the material is a very fine powder for this analysis. Samples that consist of hard materials, such as carbides, that cannot be ground using a mortar and pestle will be ground in a ball mill prior to analysis and a milling fee will apply.
- Samples with a high plasticity such as polymers and metals that cannot be ground to a fine powder are not suitable for this analysis.
- Samples that are received wet/damp will be dried prior to analysis. If air drying is insufficient, the sample(s) will be oven dried at a suitable temperature and an oven drying fee will be applied. If the sample is damp/wet and temperature sensitive, inform the lab prior to sending to avoid potential issues with improper handling.

Technique Limitations:

- The sample must contain an identifiable crystalline component in order for this technique to be applicable, samples that are mostly or entirely amorphous will be reported as such and the customer will be charged for quantitative analysis without spiking instead.
- The lower detection limit of XRD is on the order of 1% for standard scans but may be much higher or lower depending on the sample matrix and compound of interest. This test is not suitable for trace-level analysis.
- The identification of a substance is dependent upon the pattern it produces matching a database entry. Crystalline substances that do not have a database entry or a provided reference will be reported as an “unidentified phase” and cannot be accurately quantified.
- If a pattern cannot be identified, the technique cannot be used to estimate the amorphous content since it is dependent upon knowing the values of the crystalline substances present.
- The estimated error range for an ideal sample containing 10-80% amorphous content with identifiable crystalline phases is on the order of ±5%. This error range greatly increases if: unidentified phases are present, the sample cannot be ground to a fine (1-10µm) powder, and/or the sample contains less than 10% or more than 80% amorphous content.
- XRD provides crystalline *compound* identification but does not provide *elemental* composition. The technique is subject to a higher degree of identification error if elemental composition is not determined or provided prior to analysis as many elements can be substituted in different compounds that produce similar patterns.

Please submit your samples with the required documents to the following address:

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Suite 101
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