Setting Up Your Lab Q&A

Q: If XRD was done at a very slow rate (eg: 0.5 step), is that incorrect? I did this for a sample that did not give peaks.

A: You need to balance step size and counting time. If you have noisy data, you may want to either 1) count for a longer time or 2) use a larger step size and so that you count at more time per point - but maybe at the same timeframe. In some complex (>8) phase samples, you may have a huge number of peaks. You would need to increase the count time per step so that you get significant peaks and shoulders, particularly in cluster areas to do the complete analysis.

Q: Could you give a brief introduction about XRD with PSD (position-sensitive detector)? In what situation is it suggested to use PSD XRD instead of conventional XRD?

A: I use the term position-sensitive detector to include a broad range of detectors that can detect multiple positions simultaneously; this includes strip and plate detectors (multi-chip) and wire detectors that use an ionized gas. These started to be mass-produced in the 1980s and now command the majority of the market, so I believe PSDs are part of today's conventional XRD. Since market introduction, these detectors have improved dramatically in counting efficiency, energy resolution, and dynamic count range. As a user, you can utilize the multiple position capability to either collect data faster or collect more data (sometimes 100-1000X) with better signal to noise. These detectors easily interface to a variety of optics and sources. There is a wide variety of available detectors and many are customized to particular types of analysis.

With the technical capabilities of these detectors today, I would personally use them in all situations.

I think the selection of the best PSD for your types of samples is one of the most important choices you can make when selecting a new instrument.

Q: Would data for reference patterns be accepted through GIA if an internal standard were used? In that case, which one is best?

A: In general, we require pure phase materials for reference patterns for Grant-in-Aid (GiA) submissions. For additional information about on GiA requirements, please see our website https://www.icdd.com/grant-in-aid/.

Q: Are PDF-4 and JADE free for students?

A: In practice, many students access these products via a licensing agreement the university or department has with the ICDD, which would be no direct cost to the student. However, the products are not freely available. Fees for the database are used to support the stated goals of the ICDD as a non-profit, charitable organization, which includes research grants to many universities to collect data on new materials, supporting editorial review, database and software development, as well as student scholarships.

Q: You mentioned the ICDD XRD Clinics. How do I find out more about the course?

A: Yes, this webinar just touched upon the information you would learn in the ICDD Powder X-ray Diffraction clinics. You can find more information at www.icdd.com/xrd/.

Setting Up Your Lab Q&A

Q: What is the minimum amount of material I should put in the sample holder?

A: **For phase identification:** At the ICDD, we have demonstrated multiphase analyses on specimens of ~1 mg using a zero-background holder and a 300W benchtop diffractometer equipped with a strip detector. The XRD laboratory at Texas A&M (Joseph Riebenspies and Nattamai Bhuvanesh), routinely analyzes single crystals using a variety of clever specimen holders and a focused beam. The general rule of thumb is that if you can see the specimen with your eye, you can analyze it – but it usually requires atypical sample holders, focusing your optics, and long counting times.

For quantitative analysis: Keep in mind particle statistics and the challenges of accurately assaying a large sample using a small size specimen (i.e. collecting a representative specimen from a sample). In the latter case, you may need to study and apply macro statistical sampling techniques that we did not discuss in the webinar. Larger specimens (100-400 mg) increase the chances that you have a representative sample and adequate particle statistics.

Q: Does peak width increase with grinding the sample?

A: It can, which is why we suggest analyzing specimens before and after grinding whenever you test a new sample matrix. When you grind, you impart energy into the sample. You want enough energy to break down the crystal size, which does involve breaking bonds, while not decomposing or phase changing the crystals. This can be a challenge. In some matrices, such as soil, you also have to be careful that hard phases, such as quartz, do not self-grind and destroy the soft phases, such as clays. This is the reason why in several analytical methods, there may be very specific instructions on the type of mill, media, and milling time, used to prepare a specific material for analysis. A change in peak width would be showing that you are changing the crystallite size.

Q: Can a pregnant woman use a diffractometer and/or be in a lab with diffractometers?

A: This will depend on your local state and country requirements. A fetus is more sensitive to radiation and there are generally lower detection limits and exposure limits for a pregnant woman in order to protect the fetus. In most situations, radiation falls off dramatically with distance from the source due to air absorption, therefore, distance is a major consideration. Being in a lab and 20 feet from a diffractometer, it is unlikely that you would have exposure.

To consider the risks in your particular situation, check on local regulations, and you may want a conversation with your management or local safety officer. Consider additional precautions, such as limiting your time near the diffractometer, putting your analysis PC in another room, wearing a lead apron (see a medical supply store), frequently checking for diffractometer radiation leakage with a hand monitor, always wearing a radiation badge.

Q: My diffractometer can display data in Counts and CPS, what is the difference?

A: This is often confusing. CPS, which stands for counts per second, is defined as the count rate referenced versus time. I believe counts are usually a display of counts per the defined step size of the experiment. Therefore, you may have a 0.01 degree step size and count for 0.5 seconds per step. In this example, the Counts scale would be half the CPS. To be definitive you would have to check how the manufacturer defines “Counts” and hopefully, they have this information in the help file.

Setting Up Your Lab Q&A

Q: How can I use corundum as a reference in quantitative analysis? I believe it was mentioned as the standard in RIR method.

A: In the Reference Intensity Ratio (RIR) method, the diffraction intensity of a material is referenced to a standard. This enables the user to develop a series of scale factors useful for quantitative analysis. While many materials can be used as a reference, corundum, for a number of reasons, is the most frequently used reference material, so the scale factor is I/I_c where the subscript c represents corundum. These scale factors can be theoretically calculated or experimentally derived and most references in the ICDD databases have an associated I/I_c value. There are many publications on the RIR method and published procedures for experimentally determining an I/I_c value if you have an unknown. If you search "RIR" on the ICDD website, you will find links to many of the relevant publications.

Q: Which different types of sample holders do you recommend to have in the lab?

A: The typical general all-purpose sample holders are a cavity mount (reflection) or a capillary (transmission). However, other types of holders will depend on the typical types and sizes of samples that you analyze. Having a zero background holder is useful if you study small or weak scattering specimens. There are additional specialized holders for air-sensitive materials and small specimens (1-10 mg). Grooved holders are used for asymmetric crystals to avoid orientation, as well as back and side loading holders that are used to reduce orientation. There are holders designed for spinning and rocking stages. There are angularly calibrated holders used to fix the orientation of a part when doing texture analysis.

ICDD hosts several workshops/sessions that include sample preparation. For more information, visit www.icdd.com/icdd-education/

Q: I would like to add the PDF database and JADE software to my lab. Where can I request more information?

A: There are great database and software options that will fit your lab. Check out the PDF Product Summary and the JADE software website or contact us today!

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