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7 Key factors when considering a benchtop X-ray diffractometer

White paper

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Having managed X-ray diffraction labs at previous jobs and in grad school, I know that there are a number of factors to consider when the time comes to add or replace an X-ray diffraction (XRD) system in your lab. Benchtop XRD systems may be a compact and cost-effective alternative to floor-standing systems. Deciding which factors are key requirements for your needs will help you select a benchtop system ideal for your situation.

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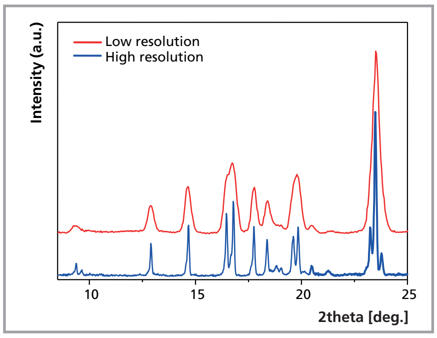
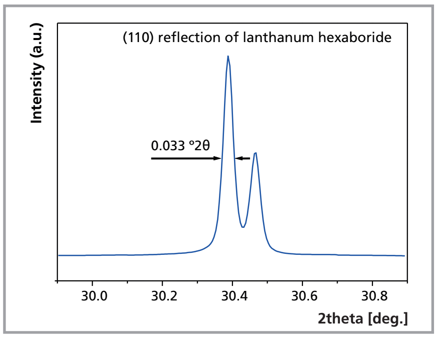
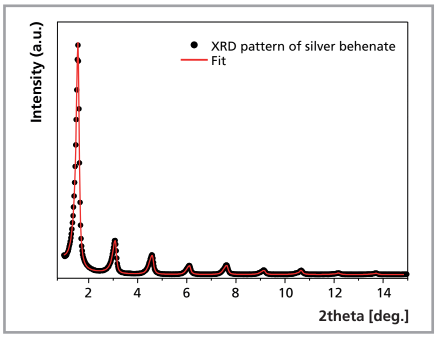
1. Determine what level of performance is acceptable for your application







One of the key aspects of system performance to consider when buying a benchtop XRD system is resolution. Compact XRD systems typically have a smaller radius at which the X-ray tube and detector sit from the sample being measured, and this will impact resolution. There is a range of resolution performance available from the benchtop XRD market, so it is important to determine what will meet your needs. If your work is routine, involves a low number of phases in your samples, or you are looking at primarily nanomaterials, resolution may not be critical to obtaining data of sufficient quality to meet your analysis goals.

But if you are looking at complex mixtures or looking for the presence of low amounts of polymorphs or trace phases, resolution should be an important factor in your evaluation process. The ability to distinguish small peaks will be critical for indicating the presence of unexpected or unwanted phases, in particular with patterns that have many peaks present. Resolution is reported as full width at half maximum (FWHM) of an early reflection from a suitable XRD standard (e.g. NIST SRM 660c - lanthanum hexaboride). The lower the FWHM, the better is the resolution. Excellent resolution for a benchtop XRD system is less than 0.04 degrees 2theta FWHM.

Another measure of performance is the linearity of the goniometer. A highly linear goniometer provides accurate peak positions over the entire 2theta range of the goniometer. This is important if you are making critical calculations from your XRD data, such as unit cell size of cracking catalyst. Linearity is determined using a line position standard such as NIST 640e – silicon powder. For the good peak position accuracy 2theta linearity should be within ±0.02 degrees 2theta.

Low-angle performance also varies from system to system. If you need to analyze pharmaceuticals or other organic materials, clays, or mesoporous materials, low-angle performance should be high on your criteria list. Low-angle reflections will be key to correct phase identification and a requirement for accurate standardless quantitative analysis such as Rietveld analysis. Low-angle performance can be demonstrated with an appropriate low-angle standard such as silver behenate. Excellent low-angle performance would show clearly discernible peaks with correct intensity ratio down to 1 degree 2theta.

2. Match your work load with the throughput of the system

A benchtop XRD system can have varying capacity to measure samples within a day. If throughput time is an important factor for you it is worth exploring the possible options and select a benchtop diffractometer optimal for your needs.

The allowed power settings of the instrument have a significant impact on throughput – operating at 40 kV instead of 30 kV improves measurement speed and thus throughput by 25%. Another way to reduce the measurement time is to use a high-speed line detector instead of a point detector. With optimized instrument setup measurement times can be as low as 5 - 10 minutes. With such quick measurements, you then may want to consider a sample changer on the system to alleviate the human sample changer. At a certain work load, it may also make sense to be able to use automation such as a robotic arm or a belt to feed samples into the system, or join the XRD system with sample preparation or complementary technologies such as an X-ray fluorescence (XRF) system.

Automation may also refer to analysis software, which can be tailored for your needs and execute data processing, quantification, report results to printer, network, or LIMS system. If automation is desired for your lab, ensure it is a possibility with the XRD system you are evaluating.

3. Determine the true cost of ownership

Of course an XRD system has an initial price tag, but there may be additional one-time or repeated costs that contribute to a true cost of ownership. Factors to consider are that XRD systems may require:

- a water chiller to provide cooling of the system's X-ray tube – this will be another piece of equipment to maintain, it will consume resources like water and electricity, and also could be a source of instrument downtime if it needs repair.
- lab or bottled gases or compressed air to operate components in the system.
- an external computer to operate the system.
- a lab environment with a controlled range of temperature and humidity.

Also XRD systems may have power requirements requiring the installation of a new electrical service. Additionally, a maintenance agreement would be a recurring cost, but may be something to consider to keep your system in top running condition, or cover you for unexpected repairs.

4. Evaluate ease of use

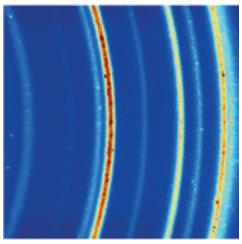
With X-ray diffraction, there are users who can collect data, and then there are diffractionists who can translate desired material properties to be analyzed into appropriate measurement programs and accurate analyses. Many labs will have people with a range of XRD knowledge, and a diffraction system should be able to accommodate all. Ease of use applies to instrument care and operation, the software for data collection as well as data analysis and also reporting, and all of these factors should be evaluated. In the case of a site with a transient work force such as a university, an easy-to-use benchtop system with good documentation will be desirable.

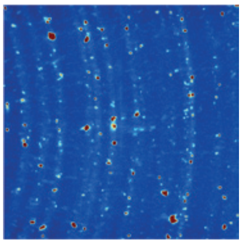
5. Assess the safety of the system

The obvious safety concern with an XRD system is radiation safety. According to the International Commission on Radiological Protection (ICRP) the absorbed dose of radiation should not exceed 1 mSv/year for a general public. When choosing a benchtop XRD you should check if the instrument complies with this requirement.

The system should also be safe for the operator, no possibility of bodily harm from the moving components, electricity, etc. This is assured by the compliance with the Machine Directive 2006/42/EC and EMC Directive 2004/108/EC, CE mark, and UL/CSA 61010-1 and 61010-2-091. It is also important that the instrument itself is protected from harmful intrusion from objects and even dust and debris. If dusty environment is an issue in your lab, pay attention to an Intrusion Protection (IP) rating of a chosen diffractometer; the higher the IP number the better. Ensure that the selected benchtop XRD system complies with the safety requirements in your laboratory.

6. Availability of options for your benchtop XRD system





A benchtop XRD system is a tool optimized for simple routine measurements and does not offer the flexibility of a floor-standing instrument. However, depending on a brand you choose, a number of additional options can be available. Those options may include a sample changer, a different X-ray tube, stages for heating and cooling your sample, linear and even area detectors. While an area detector may not be a well-known option on a benchtop XRD, its benefits, particularly for research and teaching labs, are clear. An area or 2-D detector allows you to visualize the Debye rings diffracting from a sample at characteristic positions in 2theta. This will immediately illustrate if the sample preparation was sufficient to produce fine particles (<50 μms), which then produce solid Debye rings (top image), or if the particle size is too coarse, resulting in spotty Debye rings (bottom image).

It may also become apparent if the crystallites are oriented either by poor sample preparation, or perhaps by growth of crystals preferentially in certain orientations during heating. Preferred orientation would exhibit as variations in the intensity along the Debye rings (top blue image) versus a consistent intensity all along the ring, indicating randomness of the crystallite orientations (bottom blue image). When converted into linear scans, one can see that the ratio of peak intensities before and after heating is quite different.

7. Availability of application support and service support

An XRD vendor should have available application scientists to give advice on how to properly analyze your materials and answer questions you may have, as well as educate their customers through courses, seminars, conference presentations and webinars. Just as important is an XRD vendor with sufficient service engineers to maintain and repair systems in the field. Local support is key to highest uptime and best use of your investment – ask how many service engineers are trained to service your benchtop XRD system, where the nearest service engineer is located, and whether the instrument is serviced on site or needs to be shipped to the manufacturer for depot service.

Acknowledgments
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