Use of Standard Testing Methods in X-Ray Diffraction

Why Use Standards?

Standard testing methods (STMs) are often used in X-ray diffraction analysis. This helps to guarantee consistent testing procedures and results. Especially important is the fact that strict use of the STMs limits variability due to operator idiosyncracies, sample preparation procedures, and analysis procedures. The American Society for Testing and Materials (ASTM) has published a list of approved STMs for X-ray diffraction. The most common are listed below. H&M strives to adhere to these standards whenever possible.

Standard Test Methods Designated by ASTM

B761-97	Particle Size Distribution of Metal Powders and Related Compounds by X-ray Monitoring of Gravity Sedimentation
C958-92	Particle Size Distribution of Alumina or Quartz by X-ray Monitoring of Gravity Sedimentation
C1365-98	Determination of the Proportion of Phases in Portland Cement and Portland-Cement Clinker
D933-84	Reporting Results for Examination and Analysis of Water-Formed Deposits
D934-80	Identification of Crystalline Compounds in Water-Formed Deposits by X-Ray Diffraction
D3720-90	Ratio of Anatase to Rutile in Titanium Dioxide Pigments by X-ray Diffraction
D3906-91	Relative Zeolite Diffraction Intensities
D3942-91	Determination of the Unit Cell Dimension of a Faujasite Type Zeolite
D4926-89	Gamma Alumina Content in Catalysts Containing Silica and Alumina by X-ray Powder Diffraction
D5187-91	Determination of Crystallite Size (L_c) of Calcined Petroleum Coke by X-ray Diffraction
D5357-93	Determination of Relative Crystallinity of Zeolite Sodium by X-ray Diffraction
D5380-93	Identification of Crystalline Pigments and Extenders in Paint by X-ray Diffraction Analysis
D-5758-95	Determination of Relative Crystallinity of Zeolite ZSM-5 by X-ray Diffraction
E81-96	Preparing Quantitative Pole Figures
E82-91	Determining the Orientation of a Metal Crystal
E915-96	Verifying the Alignment of X-ray Diffraction Instrumentation for Residual Stress Measurement
E975-95	X-ray Determination of Retained Austenite in Steel with Near Random Crystallographic Orientation
E1426-94	Determining the Effective Elastic Parameter for X-ray Diffraction Measurements of Residual Stress
F26-87a	Determining the Orientation of a Semiconductive Single Crystal
F847-94	Measuring Crystallographic Orientation of Flats on Single Crystal Wafers by X-ray Techniques

In addition to the ASTM standard test methods, there are a number of other recommended procedures that are of use. These are issued by the International Centre for Diffraction Data (ICDD) and present many useful hints and guidelines on sample preparation, testing procedures, and analysis methods. These guidelines are especially useful for common diffraction applications such as phase identification, texture analysis, and quantitative analysis.

Recommended Methods and Practices Published by JCPDS-ICDD

5.2.1	Sample Preparation Methods in X-ray Powder Diffraction
6.1.1	Standard Reference Materials for X-ray Diffraction Part I: Overview of Current and Future Materials
6.2.1	Standard Reference Materials for X-ray Diffraction. Part II: Calibration Using d-Spacing Standards
7.1.1	Optimization of Stepsize in X-ray Powder Diffractogram Collection
8.2.1	A Practical Method for the Determination of the Instrumental Full Width at Half Maximum
10.2.1	"PC-PDF": A Search/Display System Utilizing the CD-ROM and the Complete Powder Diffraction File
11.1.1	Quantitative X-ray Powder Diffraction Method Using the Full Diffraction Pattern
11.2.1	RIR - Measurement and Use in Quantitative XRD
11.3.1	High Temperature X-ray Diffraction - A Primer
11.7.1	Reference Intensity Ratios (a listing)
11.8.1	The Reference Intensity Ratio: Its Measurement and Significance

For precision work and alignment of the diffractometer, a number of standard reference materials (SRM) are available from NIST. Among the materials used by H&M are those listed in the table below.

Standard Reference Materials Produced by NIST

SRM640b	Silicon powder for precision lattice parameter measurements
SRM656	Silicon nitride powder for quantitative analysis
SRM660	Lanthanum hexaboride for line profile analysis
SRM674a	Diffraction intensity standard
SRM675	Mica for precision determination of low diffraction angles
SRM676	Alumina (corundum) for quantitative analysis
SRM1878	Respirable quartz for determination of percentage of alpha quartz

Our Pledge

H & M Analytical Services has over 30 years experience in X-ray diffraction. With our state-of-the-art equipment, we will strive to apply our experience and knowledge to solve your most challenging problems. In most cases, we will provide turnaround of 24 hours on phase identification analyses at no additional cost. Sample preparation services are also available. Most samples can be run on a flat fee basis. For details, please contact us.

Services We Provide

We provide a wide range of X-ray diffraction services, including:

- residual stress analysis
- precision lattice parameter
- texture analysis
- phase identification
- Rietveld analysis
- particle size determination
- high temperature XRD
- analysis of modulated films
- misfit strains
- fiber analysis
- crystal orientation
- grazing incidence angle
- retained austenite analysis

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