



Designation: D8352 – 20

Standard Test Method for Determination of Relative Crystallinity of Zeolite Beta by X-Ray Diffraction¹

This standard is issued under the fixed designation D8352; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers a procedure for determination of the relative crystallinity of zeolite Beta containing samples using selected peaks from the X-ray diffraction (XRD) pattern of the zeolite.

1.2 The test method provides a number that is the ratio of intensity of a portion of the XRD pattern of the sample zeolite Beta to intensity of the corresponding portion of the pattern of a reference zeolite Beta. The intensity ratio, expressed as a percentage, is then labeled percent XRD relative crystallinity of zeolite Beta. This type of comparison is commonly used in zeolite technology and is often referred to as percent crystallinity.

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.4 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*²

E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods

E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

3. Summary of Test Method

3.1 This method applies to zeolite Beta samples with average crystallite size >150 nm. XRD patterns of the sample

zeolite Beta and the reference zeolite Beta are obtained under the same conditions. From these patterns, there is a choice from two procedures for calculation of relative crystallinity of zeolite Beta.

3.2 *Procedure A (Integrated Peak Area Method)*—A comparison is made of the sums of intensities (sample versus reference) of the strong peaks, having maxima between about 20.0 and 24.0° 2 θ .³

3.3 *Procedure B (Peak Height Method)*—A comparison is made of the absolute peak heights (sample versus reference) of the 22.5° 2 θ peak.

4. Significance and Use

4.1 Zeolite Beta is a siliceous zeolite that can be crystallized with SiO₂/Al₂O₃ ratio greater than 20. Zeolite Beta, upon modification to the H-cation form in a post-crystallization step, has been used in catalytic NO_x reduction, isomerization of waxes, alkylation of aromatics, hydrocarbon adsorption from exhaust gas emission, etc.

4.2 This X-ray procedure is designed to allow a reporting of the relative degree of crystallinity upon manufacture of zeolite Beta. The relative crystallinity of zeolite Beta number has proven useful in technology, research, and specifications.

4.3 The Integrated Peak Area Method (Procedure A) is preferred over the Peak Height Method (Procedure B) since it calculates XRD intensity as a sum from several peaks rather than utilizing just one peak. Drastic changes in intensity of individual peaks in the XRD pattern of zeolite Beta can result from changes in distribution of electron density within the unit cell of the zeolite Beta. The electron density distribution is dependent upon the following factors:

4.3.1 Extent of filling of pores with guest molecules and the nature of these guest molecules.

4.3.2 Type of cations and extent of their presence (these cations may also affect the absorption of X rays by the zeolite Beta sample).

4.3.3 In this XRD method, the guest molecule H₂O completes the filling of the pores. Other guest molecule types may also be present, including one of numerous amines, diamines, and quaternary ammonium cations that can function as a template for crystallization of the zeolite Beta structure.

¹ This test method is under the jurisdiction of ASTM Committee D32 on Catalysts and is the direct responsibility of Subcommittee D32.05 on Zeolites.

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² For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

4.3.4 Because of the factors mentioned in 4.3.1 – 4.3.3 that could vary the intensities of the XRD peaks in zeolite Beta, this XRD method will provide the best determination of relative crystallinity when the reference zeolite Beta and sample zeolite Beta have a similar history of preparation and composition.

4.4 If crystalline phases other than zeolite Beta are present in the sample, their diffraction peaks may overlap with some of the zeolite Beta peaks selected for the Integrated Peak Area Method (Procedure A). If there is reason to suspect the presence of such components, then the Peak Height Method (Procedure B) should be chosen for analysis, provided that there is no interference with the $22.5^\circ 2\theta$ peak that is used for the calculation.

5. Apparatus

5.1 *X-ray Diffractometer*, equipped with computerized data acquisition and reduction capability, or with a strip chart recorder, and using copper K-alpha radiation (0.1541 nm).

5.2 *Drying Oven*, set at 110°C .

5.3 *Hydrator (Laboratory Desiccator)*, maintained at about 35 % relative humidity by a saturated solution of salt such as $\text{CaCl}_2 \cdot 6\text{H}_2\text{O}$

5.4 *Planimeter or Appropriate Peak Profile Analyzer or Digital Integration Software*, if XRD instrument is not equipped with appropriate software data analysis capability.

6. Reagents and Materials

6.1 *Zeolite Beta Powder*, with average crystallite size $>150\text{ nm}$. For crystallinity calculation, the reference zeolite Beta sample, ASTM 2200-45, is available from ASI Standards at 27635 Commerce Oaks Drive, Suite 100, Oak Ridge North, TX 77385 for crystallinity calculation.

7. Procedure

7.1 Carry out steps 7.2 – 7.4, in an identical manner, for both the sample zeolite Beta and the reference zeolite Beta.

7.2 Place about 1 to 3 g of finely divided zeolite Beta in the drying oven at 110°C for 1 h. Cool the sample in the hydrator and hold there at room temperature and about 35 % relative humidity for at least 16 h.

NOTE 1—Grinding of coarse-textured samples should be done gently. Overgrinding can lead to breaking up of fine crystals and destruction of the zeolite.

NOTE 2—Drying, followed by rehydration, results in filling the zeolite pores with water of hydration but without an excess of moisture residing on the surface of the zeolite particles.

7.3 Pack the sample into an XRD sample holder.

7.4 Obtain an XRD pattern of the reference zeolite Beta and also obtain a pattern of the sample zeolite Beta (in the same day) by scanning over the angle range from 5 to $50^\circ 2\theta$ using instrument parameters best suited to the X-ray diffractometer. The collected data points above the full width half maximum (FWHM) of each peak is preferably 4 to 5 so that there are enough data points to describe the peak shape. The scan range includes the diffraction peaks that are to be used in the calculation for relative crystallinity. The XRD pattern of the sample can also be used to check for crystalline phases (other than zeolite Beta) that might be present and might interfere with the utility of the calculation of Procedure A. Figs. 1 and 2 show the pattern for the reference zeolite Beta.

8. Calculation or Interpretation of Results

8.1 Planimeter or appropriate Peak Profile Analysis or Digital Integration Software can be used for data analysis.

8.2 From the patterns obtained, there is a choice from two procedures for calculation of relative crystallinity of zeolite Beta.

8.3 *Procedure A (Integrated Peak Area Method)*—A comparison is made of the sums of integrated intensities (sample versus reference) of the strong peaks having maxima between about 20 and $24^\circ 2\theta$.

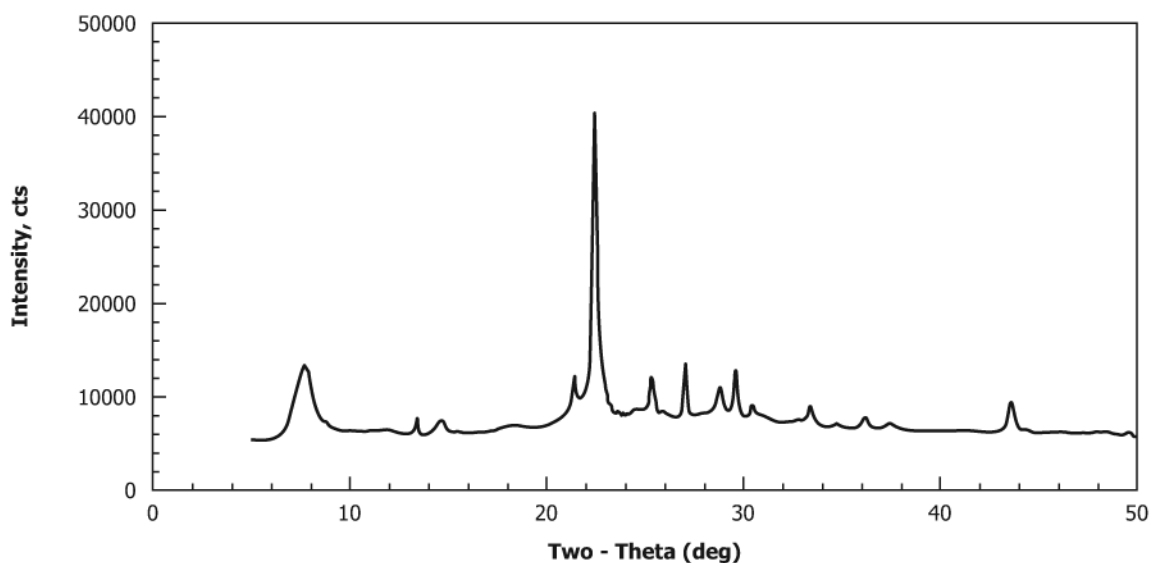


FIG. 1 X-Ray Diffraction Wide Scan Pattern of Reference Zeolite Beta