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Standard Test Method for Determination of Relative Crystallinity of Zeolite Sodium A by X-ray Diffraction[1]

This standard is issued under the fixed designation D5357; 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.

ε[1] NOTE—The units statement in subsection 1.4 was corrected editorially in May 2008.

1. Scope

1.1 This test method covers a procedure for determining the relative crystallinity of zeolite sodium A (zeolite NaA) using selected peaks from the X-ray diffraction pattern of the zeolite.

1.2 The term "intensity of an X-ray powder diffraction (XRD) peak" refers to the "integral intensity," either the area or counts under the peak or the product of the peak height and the peak width at half height.

1.3 This test method provides a number that is the ratio of intensity of portions of the XRD pattern of the sample to intensity of the corresponding portion of the pattern of a reference zeolite NaA. The intensity ratio, expressed as a percentage, is then labeled relative crystallinity of NaA.

1.4 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.5 *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 and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D3906 Test Method for Determination of Relative X-ray Diffraction Intensities of Faujasite-Type Zeolite-Containing Materials

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

E456 Terminology Relating to Quality and Statistics

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

3. Summary of Test Method

3.1 The XRD patterns of the zeolite NaA or zeolite NaA-containing sample and the reference sample (NaA) are obtained under the same conditions. A comparison of the sums of intensities of six strong peaks in the 11–32° 2θ range is made, giving relative crystallinity of NaA. This type of comparison is commonly used in zeolite technology and is often referred to as "% crystallinity."

4. Significance and Use

4.1 Zeolite NaA has been used as an active component in molecular sieves employed as desiccants for natural gas, process gas streams, sealed insulated windows, and as a builder (water softener) in household laundry detergents.

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

4.3 Drastic changes in intensity of individual peaks in the XRD pattern of NaA can result from changes in distribution of electron density within the unit cell of the NaA zeolite. The electron density distribution is dependent upon the extent of filling of pores in the zeolite with guest molecules, and on the nature of the guest molecules. In this XRD method, the guest molecule H_2O completely fills the pores. Intensity changes may also result if some or all of the sodium cations in NaA are exchanged by other cations.

4.4 Drastic changes in overall intensity can result from changes in X-ray absorption attributed to non-crystalline phases, if present, in a NaA sample. If non-zeolite crystalline phases are present, their diffraction peaks may overlap with some of the NaA diffraction peaks selected for this test method. If there is reason to suspect the presence of such components, then NaA peaks free of interference should be chosen for analysis.

[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.