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AMERICAN SOCIETY FOR TESTING AND MATERIALS
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Standard Test Method for Determination of Relative Crystallinity of Zeolite Sodium A by X-Ray Diffraction¹

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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 *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:

D 3906 Test Method for Relative X-ray Diffraction Intensities of Faujasite-Type Zeolite Containing Materials²

E 177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods³

E 456 Terminology Relating to Quality and Statistics³

E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method³

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° 2 θ 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 since the 1960's as an active component in molecular sieves employed as desiccants for natural gas, process gas streams, and sealed insulated windows. In a more recent development, initiated commercially in the 1970's, zeolite NaA has achieved large-scale use 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 H₂O 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.

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.

5.2 *Drying Oven*, set at 100°C.

5.3 *Hydrator (Laboratory Desiccator)*, maintained at about 58 % relative humidity by a saturated solution of sodium bromide, NaBr.

5.4 *Planimeter*.

¹ This test method is under the jurisdiction of ASTM Committee D-32 on Catalysts and is the direct responsibility of Subcommittee D 32.05 on Zeolites.

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² *Annual Book of ASTM Standards*, Vol 05.03.

³ *Annual Book of ASTM Standards*, Vol 14.02.