



Standard Test Method for Determination of Relative Crystallinity of Zeolite ZSM-5 by X-Ray Diffraction¹

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1. Scope

1.1 This test method covers a procedure for determination of the relative crystallinity of zeolite ZSM-5 using selected peaks from the X-ray diffraction 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 ZSM-5 to intensity of the corresponding portion of the pattern of a reference ZSM-5. The intensity ratio, expressed as a percentage, is then labeled percent XRD relative crystallinity/ZSM-5. 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 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](#)

[D5357 Test Method for Determination of Relative Crystallinity of Zeolite Sodium A by X-ray Diffraction](#)

[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 XRD patterns of the sample ZSM-5 and the reference ZSM-5 are obtained under the same conditions. From these

patterns, there is a choice from two procedures for calculation of relative crystallinity/ZSM-5.

3.1.1 *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 23.1 and 24.3° 2 θ .

3.1.2 *Procedure B (Peak Height Method)*—A comparison is made of the absolute peak heights (sample versus reference) of the 24.3° 2 θ peak.

4. Significance and Use

4.1 ZSM-5 is a siliceous zeolite that can be crystallized with SiO₂/Al₂O₃ ratio in the range of 20 to greater than 1000. ZSM-5, upon modification to the H-cation form (HZSM-5) in a post-crystallization step, has been used since the 1970s as a shape selective, acid-site catalyst for petroleum refining and petrochemicals production, including such processes as alkylation, isomerization, fluid cracking catalysis (FCC), and methanol-to-gasoline. The most siliceous member of the ZSM-5 family, sometimes called silicalite, is hydrophobic and it is used for selective sorption of organic molecules from water-containing systems.

4.2 This X-ray procedure is designed to allow a reporting of the relative degree of crystallization upon manufacture of ZSM-5. The relative crystallinity/ZSM-5 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 ZSM-5 can result from changes in distribution of electron density within the unit cell of the ZSM-5 zeolite. 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 ZSM-5 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,

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.