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

This standard is issued under the fixed designation D5758; 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 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, 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

[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](#)

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.

[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.

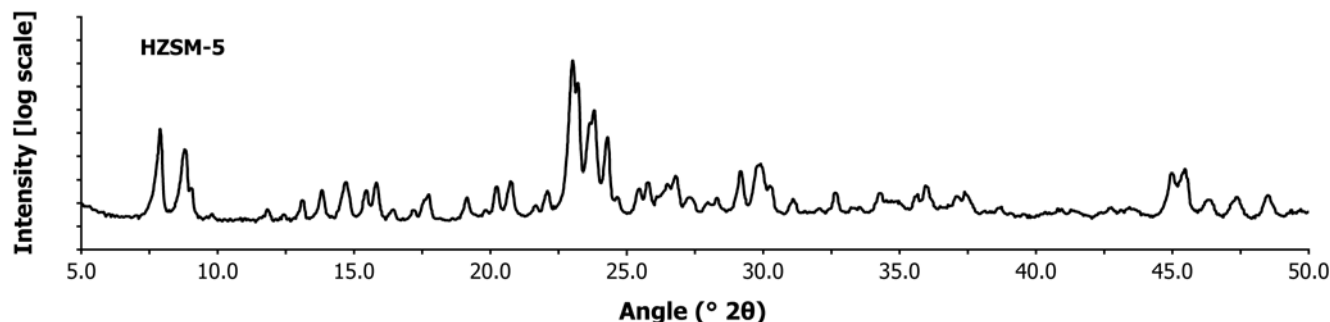


FIG. 1 X-Ray Diffraction Wide Scan Pattern of Zeolite ZSM-5—ASTM Z-20 (Reference)

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 H_2O completes the filling of the pores. Other guest molecule types may also be present, including one of numerous amines, diamines, and quarternary ammonium cations that can function as a template for crystallization of the ZSM-5 structure.

4.3.4 Because of the factors mentioned in 4.3.1 to 4.3.3 that could vary the intensities of the XRD peaks in ZSM-5, this XRD method will provide the best determination of relative crystallinity when the reference ZSM-5 and sample ZSM-5 have a similar history of preparation and composition.

4.4 ZSM-5 can exist with either orthorhombic or monoclinic symmetry, depending upon the composition of the precursor gel or post-crystallization modification conditions, or both. In the orthorhombic type, the XRD peaks centered at about 23.1° and 23.8° 2θ are usually split into doublets, whereas the less symmetric monoclinic type may show a further split of these peaks into triplets. The peak area intensities of these peaks are unaffected by the crystalline form. The XRD peak at 24.3° 2θ for the orthorhombic form is a singlet and hence is the most suitable for the Peak Height Method (Procedure B). If the 24.3° peak is split (doublet in the monoclinic form), then the Integrated Peak Area Method (Procedure A) should be used.

4.5 If crystalline phases other than ZSM-5 are present in the sample, their diffraction peaks may overlap with some of the ZSM-5 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 24.3° 2θ 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.

5.2 *Drying Oven*, set at $105 \pm 5^\circ C$.

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

5.4 *Planimeter*.

NOTE 1—The planimeter will not be needed if the XRD instrument is equipped with computerized data acquisition and reduction capability.

6. Reagents and Materials

6.1 *ZSM-5 Powder*,³ as reference standard, preferably with a mean particle diameter of less than 10 μm .

7. Procedure

7.1 Carry out steps 7.2 through 7.4, in an identical manner, for both the sample ZSM-5 and the reference ZSM-5.

7.2 Place about 1.5 g of finely divided ZSM-5 in the drying oven at $105^\circ C$ for 2 h. Cool the sample in the hydrator and hold there at room temperature and about 58 % relative humidity for at least 16 h.

NOTE 2—Grinding of coarse-textured samples should be done gently. Overgrinding can lead to breaking up of fine crystals and destruction of the zeolite.

NOTE 3—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 humidity-conditioned sample into an XRD sample holder.

7.4 Obtain an XRD pattern of the reference ZSM-5 and also obtain a pattern of the sample ZSM-5 (in the same day), by scanning over the angle range from 11° to 32° 2θ using instrument parameters best suited to the X-ray diffractometer. The scan rate should not be greater than $1.0^\circ/min$. 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 ZSM-5 that might be present and might interfere with the utility of the calculation of Procedure A (see 4.5). Fig. 1 shows a pattern for the reference ZSM-5 (H^+ cation form) used in the testing of this test method.

7.4.1 If a strip chart recorder is used, set the chart drive at 20 mm/min. Select the scale factor (for amplification) for the reference ZSM-5 pattern so that the strongest peak at 23.1° 2θ is between 50 and 100 % of full scale. The same scale factor should be used for the sample ZSM-5 pattern. However, if the sample gives considerably lower peak intensities, the scale factor may be reduced (amplification increased) in order to provide reasonable peak heights.

³ Available from NIST as RM8852.