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Standard Test Method for Determination of Relative X-ray Diffraction Intensities of Faujasite-Type Zeolite-Containing Materials¹

This standard is issued under the fixed designation D 3906; 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 the determination of relative X-ray diffraction intensities of zeolites having the faujasite crystal structure, including synthetic *Y* and *X* zeolites, their modifications such as the various cation exchange forms, and the dealuminized, decationated, and ultrastable forms of *Y*. These zeolites have cubic symmetry with a unit cell parameter usually within the limits of 24.2 and ~~25.0 Å~~ 25.0 Å (2.42 and 2.50 nm).

1.2 The samples include zeolite preparations in the various forms, and catalysts and adsorbents containing these zeolites.

1.3 The term “intensity of an X-ray powder diffraction (XRD) peak” is the “integral intensity,” either the area of counts under the peak or the product of the peak height and the peak width.

1.4 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, NaY. (Laboratories may use a modified *Y* or *X*, for example, REY as a secondary standard.) The intensity ratio, expressed as a percentage, is then labeled “% XRD intensity/NaY.”

1.5 Under certain conditions such a ratio is the percent zeolite in the sample. These conditions include:

1.5.1 The zeolite in the sample is the same as the reference zeolite.

1.5.2 The absorption for the X-rays used is the same for the zeolite and the nonzeolite portions of the sample.

1.6 *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.*

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[†] This test method is under the jurisdiction of ASTM Committee D-32 on Catalysts and is the direct responsibility of Subcommittee D32.05 on Zeolites. Current edition approved March 10, 1997. Published October 1997. Originally published as D3906-80. Last previous edition D3906-91.

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. Current edition approved April 1, 2008. Published May 2008. Originally approved in 1980. Last previous edition approved in 2003 as D 3906-03.

2. Referenced Documents

2.1 ASTM Standards:²

- E 177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods
- E 456 Terminology Relating to Quality and Statistics
- E 691 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 containing sample and the reference sample (NaY), are obtained under the same conditions. Intensities of the (533) peak (23.5° with Cu K α radiation) are compared to provide “% XRD intensity/NaY (533).” If the XRD pattern of the zeolite is sufficiently strong, a comparison of the sums of intensities of eight peaks is used to give “% crystallinity.”

3.1 The XRD patterns of the zeolite containing sample and the reference sample (NaY), are obtained under the same conditions. If the XRD pattern of the zeolite is sufficiently strong, a comparison of intensities of eight peaks is used to give % XRD intensity/NaY. For lower zeolite content intensities of the (533) peak (23.5° with Cu K α radiation) are compared to provide “% XRD intensity/NaY (533).”

4. Significance and Use

4.1 Zeolites *Y* and *X*, particularly for catalyst and adsorbent applications, are a major article of manufacture and commerce, use of which has developed since the 1960s. Catalysts and adsorbents comprising these zeolites in various forms plus binder and other components have likewise become important in this period. *Y*-based catalysts are used for fluid catalytic cracking (FCC) and hydrocracking of petroleum, while *X*-based adsorbents are used for desiccation, sulfur compound removal, and air separation.

4.2 This X-ray procedure is designed to monitor these *Y* and *X* zeolites and catalysts and adsorbents, providing a number more or less closely related to percent zeolite in the sample. This number has proven useful in technology, research, and specifications.

4.3 Drastic changes in intensity of individual peaks in the XRD patterns of *Y* and *X* can result from changes of distribution of electron density within the unit cell of the 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 cations in *Y* and *X* are exchanged by other cations.

4.3.1 Because of the factors mentioned in 4.3 that could vary the intensities of the XRD peaks, this XRD method will provide the best determination of relative crystallinity when the reference and sample have a similar history of preparation and composition.

4.4 Corrections are possible that can make this XRD method accurate for measuring percent zeolite in many specific situations. These corrections are well known to those skilled in X-ray diffraction. It is not practical to specify those corrections here.

5. Apparatus

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

5.2 *Drying Oven*, set at 110°C .

5.3 *Hydrator (Glass Laboratory Desiccator)*, maintained at 35 % relative humidity by a saturated solution of salt, such as CaCl₂·6H₂O.

5.4 *Planimeter*.

NOTE 1—This planimeter may not be needed if the XRD instrument is equipped with a digital integrating circuit. Planimeter or Appropriate Peak Profile Analysis or Digital Integration Software—If XRD is not equipped with appropriate software data analysis capability.

6. Reagents and Materials

6.1 *NaY Powder and RE Exchanged Y Powder*, as reference standards.³

7. Sampling

7.1 Conduct sampling by splitting a large portion of the sample and reference material homogeneously.

7.2 Divide the sample and reference finely to permit packing of the materials into XRD sample holders.

NOTE 2—The 1—The best test to determine if grinding is required is to try to pack the sample in the holder. Overgrinding can lead to breaking up of fine crystals and even destruction of zeolite.

8. Procedure

8.1 Carry out the following steps, 8.2 through 8.5, in an identical manner for both the sample and the reference material, NaY.

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* Vol 14.02-volume information, refer to the standard's Document Summary page on the ASTM website.

³ Available from ASTM Committee D-32.

³ Available from National Institute of Standards and Technology (NIST), 100 Bureau Dr., Stop 3460, Gaithersburg, MD 20899-3460.

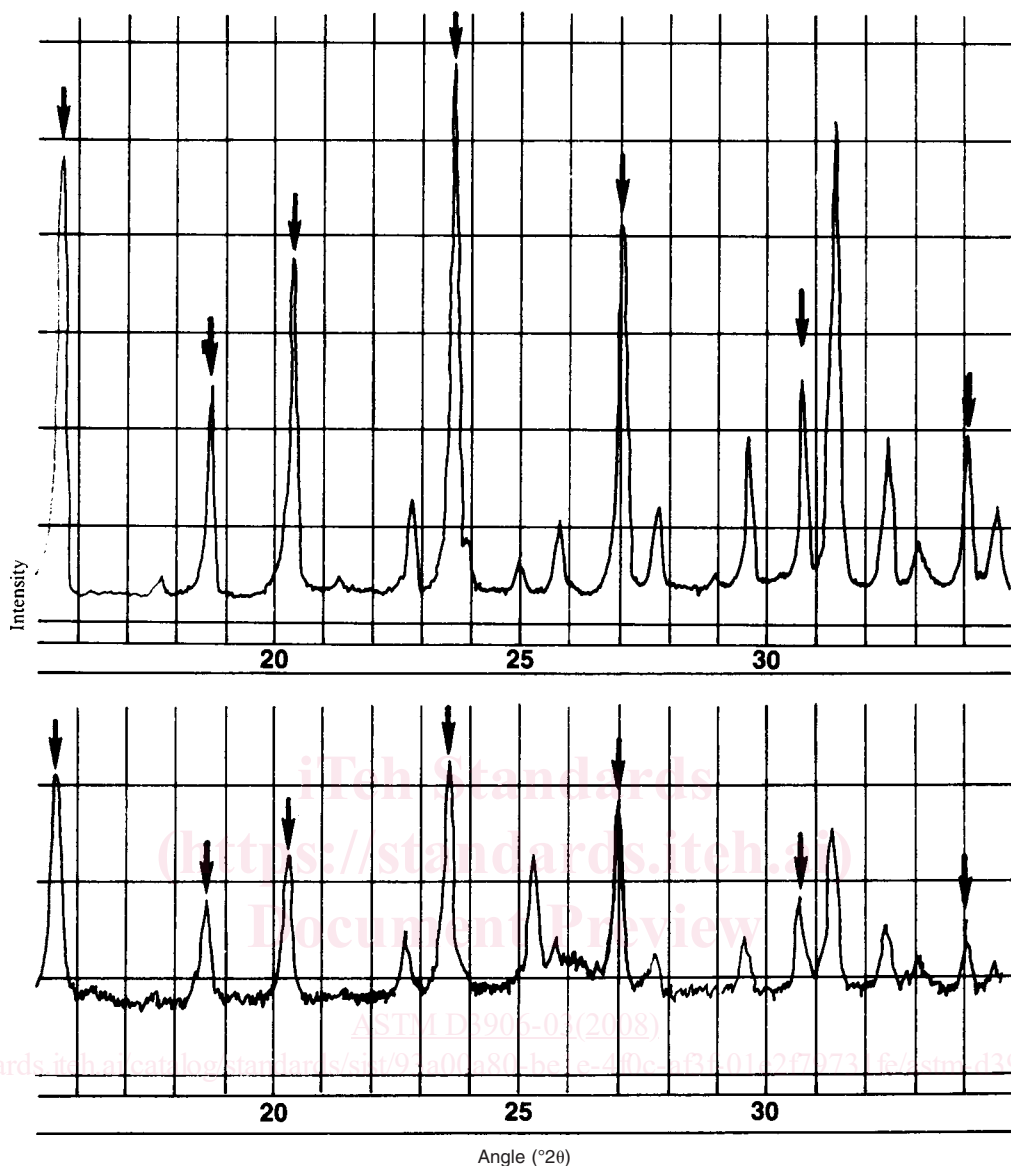


FIG. 1 X-Ray Diffraction Patterns of ASTM Zeolite Samples Upper—NaY; Lower—Cracking Catalyst Intensity

8.2 Place about 3 to 5 g of the sample in the drying oven at 110°C for 1 h. Cool the sample in the hydrator and hold at room temperature and 35 % relative humidity for at least 16 h.

NOTE 32—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.

8.3 Pack the humidity-conditioned sample into an XRD sample holder.

8.4 Obtain a first XRD pattern by scanning over the angle range from 14 to 35° 2θ at about 1°/min and using other instrument parameters best suited to the diffractometer.

8.4.1 If a strip chart recorder is used, set the chart drive at 10 mm/min. Select the scale factor (amplification) for the NaY reference pattern so that the strong (533) peak at 23.6° is between 50 and 100 % of full scale. For the sample the scale factor may be reduced (amplification increased) to provide reasonable peak heights. If possible the height of the (533) peak for the sample should be at least 10 % of full scale. Fig. 1 shows such patterns for the reference NaY and for a zeolite-containing catalyst.

8.5 If this first pattern of the sample contains XRD peaks of some nonfaujasite components, it must be established whether this may cause interference in the following steps. (Fig. 2 is a complete diffractometer scan for NaY.)

8.6 Obtain a second XRD pattern by scanning over a small angle range at ¼ °/min.

8.6.1 If a strip chart recorder is used, set the drive at 20 mm/min. The same scale factors used in Step 8.4 are suitable. The preferred angle range is from 22.5 to 25° 2θ, the (533) peak.

NOTE 3—Longer scan times will be required for samples having a lower content of zeolite. For example 0.02° 2θ/step for 1 s may be acceptable for a pure NaY while 10 to 20 s counting times per step may be required for a low level of zeolite samples.

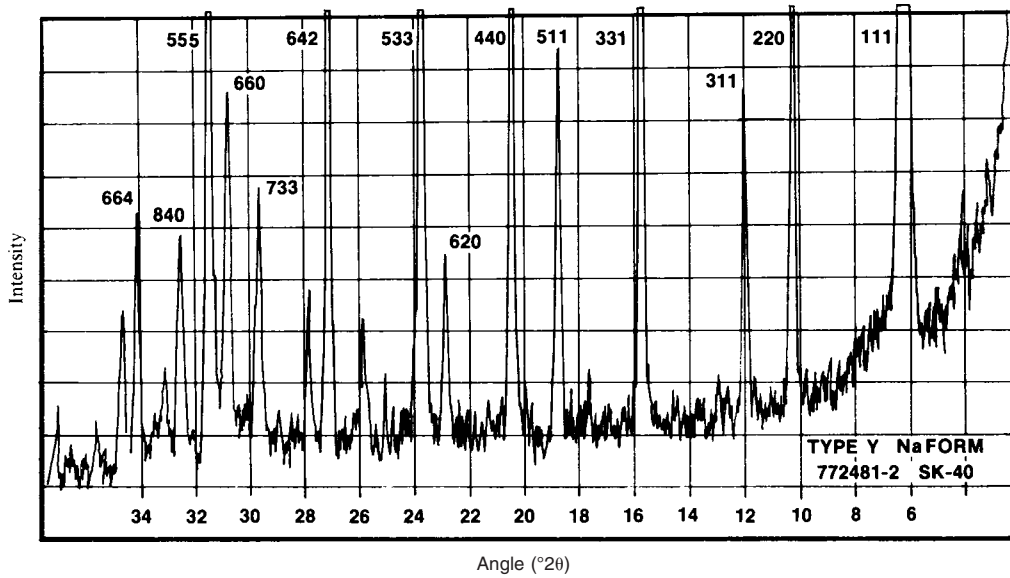


FIG. 2 NaY—Complete Diffractometer Scan Intensity

8.6.1 The preferred angle range is from 22.5 to 25° 2θ, the (533) peak. Fig. 3 shows such a pattern for NaY. If interference rules

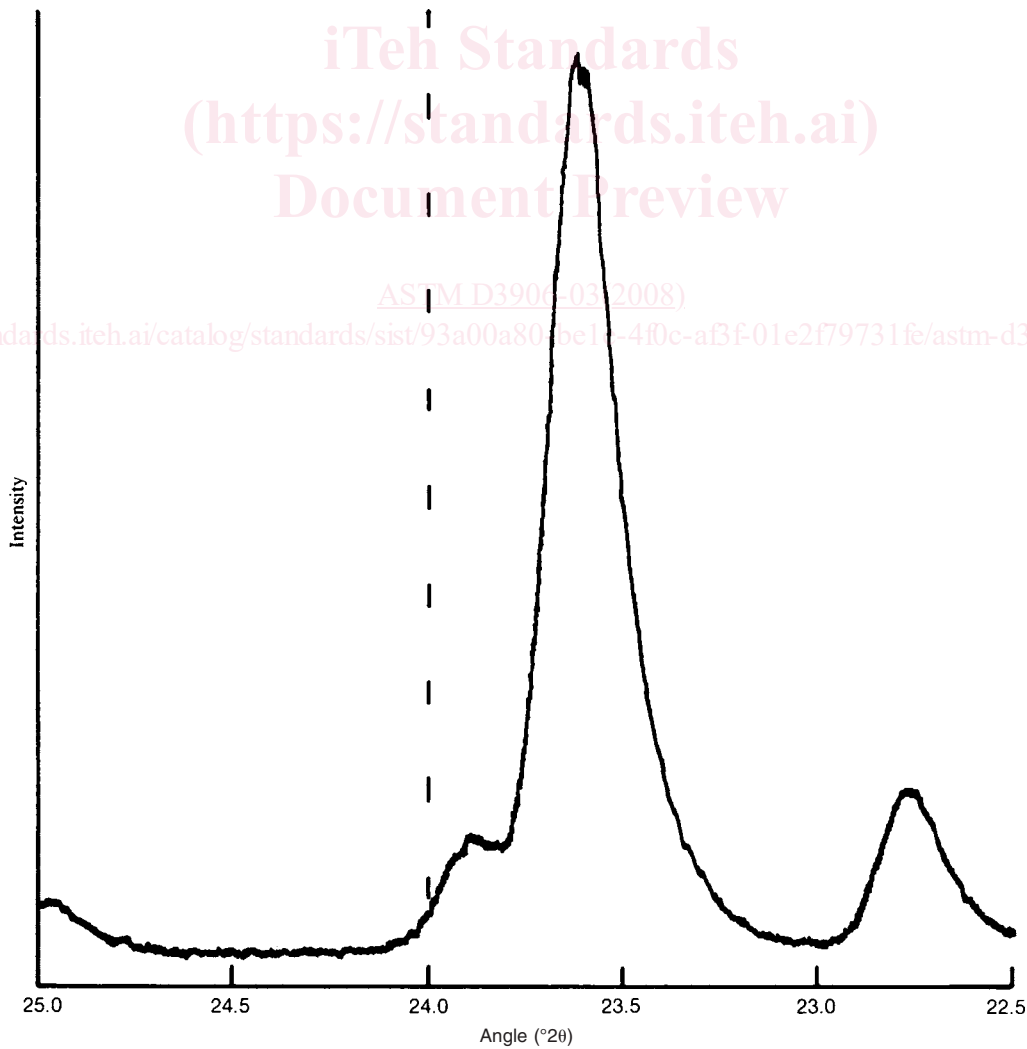


FIG. 3 XRD ASTM NaY Intensity