Designation: D 3942 – 03

Standard Test Method for Determination of the Unit Cell Dimension of a Faujasite-Type Zeolite[1]

This standard is issued under the fixed designation D 3942; 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 the unit cell dimension 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 ultra stable forms of *Y*. These zeolites have cubic symmetry with a unit cell parameter usually within the limits of 24.2 and 25.0 Å (2.42 and 2.50 nm).

1.2 The samples include zeolite preparation in the various forms, and catalysts and adsorbents containing these zeolites. The zeolite may be present in amounts as low as 5 %, such as in a cracking catalyst.

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:*

E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method[2]

3. Summary of Test Method

3.1 A sample of the zeolite *Y* or *X*, or catalyst containing zeolite is mixed with powdered silicon. The zeolite unit cell dimension is calculated from the X-ray diffraction pattern of the mixture, using the silicon reflections as a reference.

4. Significance and Use

4.1 Zeolites *Y* and *X*, particularly for catalyst and adsorbent applications, are a major article of manufacture and commerce. Catalysts and adsorbents comprising these zeolites in various forms plus binder and other components have likewise become important. *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 The unit cell dimension of a freshly synthesized faujasite-type zeolite is a sensitive measure of composition which, among other uses, distinguishes between the two synthetic faujasite-type zeolites, *X* and *Y*. The presence of a matrix in a *Y*-containing catalyst precludes determination of the zeolite framework composition by direct elemental analysis.

4.3 Users of the method should be aware that the correlation between framework composition and unit cell dimension is specific to a given cation form of the zeolite. Steam or thermal treatments, for example, may alter both composition and cation form. The user must therefore determine the correlation that pertains to his zeolite containing samples.[3] In addition, one may use the method solely to determine the unit cell dimension, in which case no correlation is needed.

4.4 Other crystalline components may be present in the sample whose diffraction pattern may cause interference with the selected faujasite-structure diffraction peaks. If there is reason to suspect the presence of such components, then a full diffractometer scan should be obtained and analyzed to select faujasite-structure peaks free of interference.

5. Apparatus

5.1 *X-Ray Diffractometer*, able to scan at 0.25° 2θ/min. 2θ values in the following discussions were based on data obtained with a copper tube, although other tubes such as molybdenum can be used.

NOTE 1—A step-scanning accessory, to scan at a rate of 0.25° or less 2θ/min, will increase the accuracy of the determination and will facilitate measurement in samples of low zeolite content.

5.2 *Drying Oven*, set at 110°C.

[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] *Annual Book of ASTM Standards*, Vol 14.02.

[3] Three correlations have been published for pure synthetic faujasite-type zeolites in the sodium or calcium form: Breck, D. W. and Flanigen, E. M. in" Molecular Sieves", *Society of Chemical Industry*, London, 1968, p. 47, Wright A. C., Rupert, J. P. and Granquist W. T. Amer. Mineral., Vol 53, 1968, p. 1293; and Dempsy, E., Kuehl, G. H., and Olson, D. H., *Journal of the Physical Chemistry*, Vol 73, 1968, p. 387.