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Standard Test Method for Gamma Alumina Content in Catalysts Containing Silica and Alumina by X-Ray Powder Diffraction[1]

This standard is issued under the fixed designation D 4926; 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 gamma alumina and related transition aluminas in catalysts containing silica and alumina by X-ray powder diffraction, using the diffracted intensity of the peak occurring at about 67°2θ when copper Kα radiation is employed.

1.2 *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 catalyst is calcined and ground, and an X-ray powder diffraction pattern is obtained under specified conditions over the approximate range from 52 to 76°2θ. The diffracted intensity above background for the peak occurring at about 67°2θ is compared to that of a reference sample, after appropriate adjustments are made for scale settings and peak half-widths.

4. Significance and Use

4.1 This test method is for estimating the relative amount of gamma alumina in calcined catalyst samples, assuming that the X-ray powder diffraction peak occurring at about 67°2θ is attributable to gamma alumina. Gamma alumina is defined as a transition alumina formed after heating in the range from 500 to 550°C, and may include forms described in the literature as eta, chi, and gamma aluminas. Delta alumina has a diffraction peak in the same region, but is formed above 850°C, a temperature to which most catalysts of this type are not heated. There are other possible components which may cause some interference, such as alpha-quartz and zeolite Y, as well as aluminum-containing spinels formed at elevated temperatures. If the presence of interfering material is suspected, the diffraction pattern should be examined in greater detail. More significant interference may be caused by the presence of large amounts of heavy metals or rare earths, which exhibit strong X-ray absorption and scattering. Comparisons between similar materials, therefore, may be more appropriate than those between widely varying materials.

5. Apparatus

5.1 *X-Ray Powder Diffractometer Unit*, with standard sample mount, Cu Kα radiation, monochromator, wide divergence and receiving slits (for example, 3° and 0.15°, respectively), goniometer speed of 0.5°/min or equivalent, chart speed of about 0.5 cm/min or equivalent, and scale or gain factors to provide conveniently measurable peaks.

NOTE 1—For diffractometers employing step scanning, convenient corresponding conditions include a step size of 0.02° and a counting time of 2.4 s/step, which is equivalent to a scanning rate of 0.5°/min.

5.2 *Calcination Furnace*.

5.3 *Grinding Equipment*, suitable for preparing samples for mounting in the sample holder.

6. Procedure

6.1 Calcine the catalyst sample for 3 h at 500°C.

6.2 Grind the sample sufficiently (for example, 200 to 400 mesh) to enable it to be packed into a standard X-ray powder diffractometer sample holder and mounted on the diffractometer.

6.3 Obtain diffraction patterns for three samples over the approximate range from 52 to 76°2θ, using the conditions described in 5.1.

6.4 Measure the height of the 67°2θ peak above background for the sample and the reference material to give *H* (sample) and *H* (reference), respectively.

NOTE 2—Reference material may be prepared by calcining a high-purity sample of fine-particle boehmite for 3 h at 550°C.

7. Calculation

7.1 The relative X-ray powder diffraction intensity of three samples, compared to a reference standard and expressed as

[1] This test method is under the jurisdiction of ASTM Committee D32 on Catalysts and is the direct responsibility of Subcommittee D32.01 on Physical-Chemical Properties.

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

percent, is calculated by use of the following equation:

$$100\,\frac{I(\text{sample})}{I(\text{ref})} = \frac{H(\text{sample})}{H(\text{ref})} \times \frac{W(\text{sample})}{W(\text{ref})} \times \frac{G(\text{ref})}{G(\text{sample})} \times 100$$

where: *H* (sample) and *H* (ref) are as defined in 6.4, *W* (sample)/*W* (ref) is the ratio of the peak width at half-height for the sample compared to that for the reference, and *G* (ref)/*G* (sample) is the ratio of the instrument gain factor used to record the peak for the sample to the gain factor used to record the peak for the reference. (Gain factor is often defined as the inverse of the product of the diffractometer scale setting and rate multiplier setting.)

7.2 The average of the three values calculated in 7.1 can be considered a measure of gamma alumina content in the catalyst if the measured sample peak is attributable to gamma alumina, the reference material is essentially pure gamma alumina, and the sample does not contain large amounts of heavy metals or rare earths. Caution should be observed in comparing results for widely varying materials.

7.3 A working or secondary reference material can be used as a matter of practical convenience. Should such a secondary reference material be available, results as calculated in 7.1 relative to the first or primary reference material can be transformed to relate to the working reference material. Determine the relative X-ray powder diffraction intensity of the primary reference material relative to the secondary reference material, using an equation corresponding to that shown in 7.1 and expressing the ratio I_{R1}/I_{R2} in fractional form. Multiply the result relative to the primary reference material (100 I/I_{R1}) by the factor just determined above (I_{R1}/I_{R2}) to get the result (100 I/I_{R2}) relative to the secondary reference material. Identify the reference materials when reporting the results.

8. Precision and Bias [3]

8.1 *Precision*—Based on the results of a multilaboratory, multisample study and using Practice E 691 noted in 2.1, the within-laboratory repeatability was found to be ±15 % (2*S* %) of the measured value, and the between-laboratory reproducibility was found to be ±24 % (2*S* %) of the measured value.

8.2 *Bias*—No estimate of the bias of this test method is possible.

9. Keywords

9.1 alumina; catalyst; gamma alumina; gamma alumina content; x-ray powder diffraction

[3] Supporting data are available from ASTM Headquarters. Request RR: D32-1027.