



Standard Test Method for Gamma Alumina Content in Catalysts Containing Silica and Alumina by X-ray Powder Diffraction¹

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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 $67^\circ 2\theta$ when copper $K\alpha$ radiation is employed.

1.2 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

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

E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

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 $76^\circ 2\theta$. The diffracted intensity above background for the peak occurring at about $67^\circ 2\theta$ is compared to that of a reference sample, after appropriate adjustments are made for scale settings and peak half-widths.

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 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.

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 $67^\circ 2\theta$ 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 $K\alpha$ radiation, monochromator, wide divergence and receiving slits (for example, 3° and 0.15° , respectively), goniometer speed of $0.5^\circ/\text{min}$ or equivalent, chart speed of about $0.5\text{ cm}/\text{min}$ or equivalent, and scale or gain factors to provide conveniently measurable peaks. Computerized data acquisition equipment may also be used.

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

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 .