



Designation: D8392 – 22

Standard Practice for Calibration and Verification of Direct Imaging Analyzers Used for Particle Size and Shape Analysis of Catalytic Materials¹

This standard is issued under the fixed designation D8392; 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 practice covers the calibration and verification of Dynamic Imaging Analyzers (analyzers) using catalytic and non-catalytic reference materials. The measurement range of analyzers covers from 500 μm to 20 000 μm.

1.2 This practice may also be used to analyze catalytic materials once the analyzer has been calibrated and verified.

1.3 *Units*—The values stated in SI units are to be regarded as standard; however, English and mesh units are also acceptable with conversions provided in [Appendix X3](#).

1.4 *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.*

1.5 *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

[D3766 Terminology Relating to Catalysts and Catalysis](#)

[D6299 Practice for Applying Statistical Quality Assurance and Control Charting Techniques to Evaluate Analytical Measurement System Performance](#)

[E105 Guide for Probability Sampling of Materials](#)

[E2589 Terminology Relating to Nonsieving Methods of Powder Characterization](#)

2.2 *DIN Standard:*³

[66141 Representation of Particle Size Distributions – Basic Standard](#)

2.3 *ISO Standards:*⁴

[9276-6 Representation of Results of Particle Size Analysis – Part 6: Descriptive and Quantitative Representation of Particle Shape and Morphology](#)

[13322-1 Particle Size Analysis – Image Analysis Methods – Part 1: Static Image Analysis Methods](#)

3. Terminology

3.1 Reference Terminologies [D3766](#) and [E2589](#) for standard definitions.

3.2 *Non-standard Particle Shape Definitions and Definitions of Terms Specific to This Standard*

3.2.1 *aspect ratio, n*—equal to the particle width divided by the particle length (see [Appendix X1](#)).

3.2.2 *particle convexity, n*—a measurement, from 0 to 1, of the actual particle area to the area within the convex perimeter (see [X1.4](#)).

3.3 *Non-standard Particle Size Definitions and Definitions of Terms Specific to This Standard*

3.3.1 *particle length, n*—the maximum feret that is perpendicular to the particle width of the two-dimensionally imaged particle (see [Appendix X1](#)).

3.3.2 *particle width, n*—the minimum area bisector of the two-dimensionally imaged particle and signified by $X_{Ma\ min}$, minimum Martin diameter (see [Appendix X1](#)).

NOTE 1—Imaging terminology is known to have multiple phrasings throughout industry. [Appendix X1](#) and [Appendix X2](#) describe, in detail, what these terms represent with regard to this practice.

4. Summary of Practice

4.1 The general principle of operation of the analyzer is the transport of catalytic material from a loading container through

¹ This practice is under the jurisdiction of ASTM Committee [D32](#) on Catalysts and is the direct responsibility of Subcommittee [D32.02](#) on Physical-Mechanical 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.

³ Available from Deutsches Institut für Normung e.V.(DIN), Am DIN-Platz, Burggrafenstrasse 6, 10787 Berlin, Germany, <http://www.din.de>.

⁴ Available from International Organization for Standardization (ISO), ISO Central Secretariat, Chemin de Blandonnet 8, CP 401, 1214 Vernier, Geneva, Switzerland, <https://www.iso.org>.

a measurement zone where the particles can be dynamically imaged in order to determine their size (length and width) and shape. See Fig. 1 for a general diagram of the analyzer.

4.2 Catalytic materials are varied in shape, depending on application, and include spherical, or near spherical, particles, cylindrical and multilobe particles. No particle standards exist for the purpose of analyzer verification with regard to catalytic materials. This practice describes procedures to calibrate and verify calibrations using commercially available reference materials for verification because they are dimensionally controlled to small tolerances and inexpensive.

4.3 This practice also describes a procedure where actual catalytic materials can be used to verify calibrations in a similar manner as the procedure where reference materials are used.

5. Significance and Use

5.1 Particle size and shape are important in predicting the performance of catalytic materials. They influence the bulk density of the final product and thereby the effectiveness of performance.

5.2 Establishing a verification reference for the analyzer that is commercially available and dimensionally reliable to close tolerances enables different analyzers to be easily checked to equivalent standards.

5.3 This practice may also be followed to analyze catalytic materials for quality manufacturing purposes. Sections 9 and 10 instruct on sample count determination as well as sampling recommendations. Test Method D6299 may be utilized to monitor performance of the analyzer in measuring the size and shape of catalytic materials.

6. Apparatus

6.1 *Dynamic Flow Imaging Analyzer*—The diagram in Fig. 1 illustrates the general operating principle of the analyzer.

6.1.1 Major Components:

6.1.1.1 *Hopper/Feed Tray*—Moves particles into the measurement zone by mechanical vibration and gravity.

6.1.1.2 *Illumination Source*—A source that provides consistent illumination in order for the camera device to capture particle images for analysis which have high contrast.

6.1.1.3 *Camera/lens*—An imaging device with suitable resolution combined with a lens which provides suitable field of view for the particles to be analyzed.

6.1.1.4 *Appropriate size basin or catch container for capturing the specimen* (not pictured).

6.1.1.5 *Orientation Device*—A particle handling mechanism which aligns particles in order that the imaging device sees their full length and width. This is not required if a software algorithm is used to determine full particle length and width by capturing multiple images of the same particle in free fall.

7. Reagents and Materials

7.1 *Reticle*, NIST traceable, for calibration of the pixel array into units of measure.

7.2 *Cylindrical Reference Material*—Cylindrical particles of known size, similar in shape to catalyst particles, that can be used to verify the calibration of the analyzer. Electronic spacers have been used for this function (see Appendix X4).

7.3 *Caliper (or Similar Device)*, with calibration traceable to NIST to measure length and diameter of reference materials (see 7.2) with resolution of ± 0.01 mm.

7.4 *Precision Grade Glass Spheres*, with diameter tolerance of ± 20 μ m and a minimum width to length ratio of 0.99.

8. Preparation of Apparatus

8.1 Ensure the analyzer is prepared for use per the manufacturer's instructions.

8.2 Ensure the feed mechanism is clean and ready for use.

9. Sample Preparation

9.1 A minimum number of particles for analysis must be determined and is based on the analysis for a log normal distribution presented in ISO13322-1:

$$\log n^* = -2\log\delta + \log w \quad (1)$$

where n^* is the minimum number of particles required to be

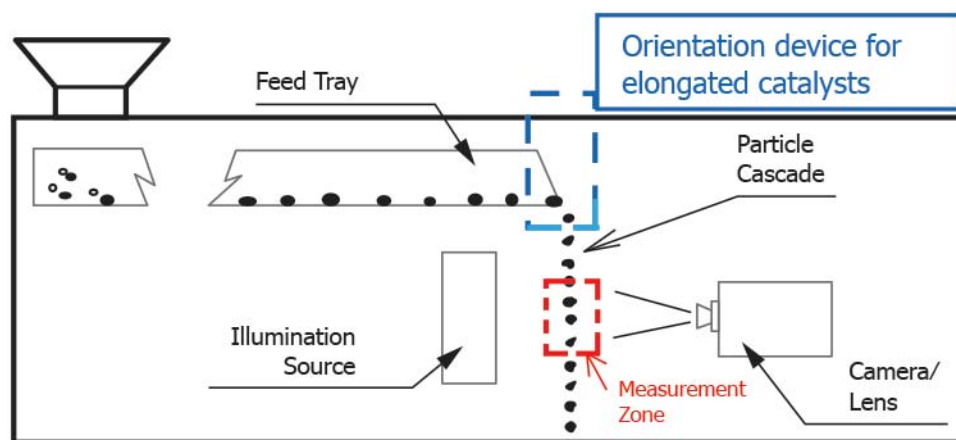


FIG. 1 Typical Analyzer Schematic