Designation: D8392 – 22

Standard Practice for Calibration and Verification of Direct Imaging Analyzers Used for Particle Size and Shape Analysis of Catalytic Materials[1]

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 um 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

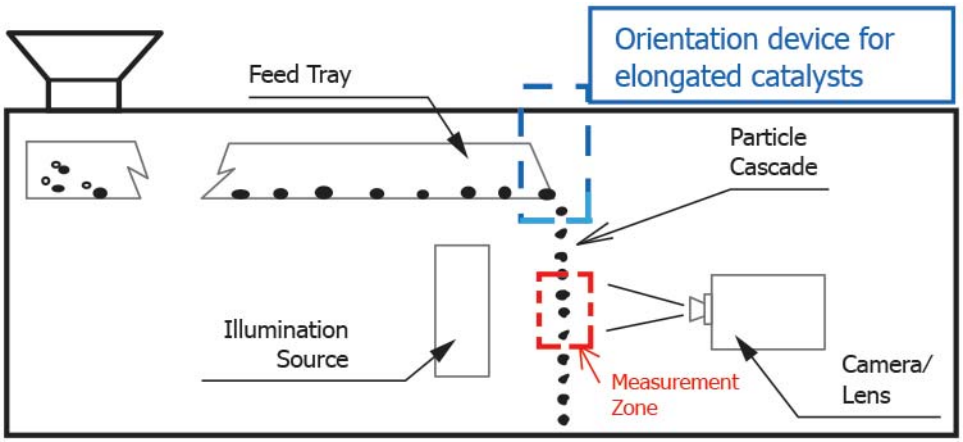

FIG. 1 Typical Analyzer Schematic

analyzed, δ is the relative error, and w is a calculated parameter based on the standard deviation of the distribution. An example calculation is given in Appendix X4.

9.2 Test samples should be obtained from larger composites by riffling or splitting in accordance with STP447A[5] or another similar means with the goal of obtaining samples with size distributions that are representative of the larger composites. Also refer to Guide E105 for constructing a sampling plan.

10. Apparatus Calibration and Verification

10.1 *Calibration:*

10.1.1 See the analyzer manufacturer's instructions for calibrating.

10.1.2 Calibration fixtures must be traceable to NIST.

10.2 *Verification, Procedure A:*

10.2.1 *Non-Spherical Elongated Shapes*

10.2.1.1 Use the cylindrical reference material (see 7.2) to verify the calibration of the analyzer. Select three sizes of spacer widths (see 3.3.2) that cover the range of measurement anticipated. Spacer widths in the following ranges are recommended: ≥ 0.5 mm and ≤ 2 mm, ≥ 4 mm and ≤ 6 mm, and ≥ 8 mm and ≤ 10 mm.

10.2.1.2 For the three widths chosen in 10.2.1.1, select three spacer lengths that will span the range of lengths expected to measure (up to 20 mm).

10.2.2 The minimum number of spacers of each size to use for the verification exercise is calculated by following the instructions in Section 9.

10.2.3 Select the reference particles to be used and measure the length and width of each. Calculate the average width and length of each size category. Mix the selected reference particles together in sufficient quantities to ensure the minimum number of particles of each size will be analyzed. Record the percentage of each in the mixture in order to check that the analyzer can detect in the proper proportions.

10.2.4 Following the procedure of Section 11, run the spacers through the analyzer and measure the particle width (see 3.3.2) and the particle length (see 3.3.1) of each. The average particle width determined should be within 0.05 mm of the average spacer diameter determined at 10.2.3. The average length may be outside the 0.05 mm value due to variations in the captured images when particles are not exactly perpendicular to the image sensor.

10.2.5 If the standard deviation in the detected width or length values by the analyzer is calculated to be greater than 0.05 mm, then the minimum number of particles must be recalculated per Eq 1 and the verification redone.

10.3 *Verification – Near Spherically Shaped Particles:*

10.3.1 To verify calibration for applications where catalyst materials are generally spherical in shape, repeat steps detailed in 10.2.2 – 10.2.4 using precision grade spheres (see 7.4) of various sizes that span the range of intended measurement. The spheres do not have to be measured manually; the nominal diameter indicated on the analysis certificate and the reported standard deviation will be used to calculate the minimum number of particles required per Section 9.

10.3.2 Analyze the selected sample per the instructions of 11.1.

10.4 *Verification, Procedure B*—It is also acceptable to perform verification studies using actual catalytic materials. The procedure is very close to that described in 10.2 and 10.3.

10.4.1 Estimate the standard deviation of particle width and length for the particular material to be tested. The distribution in the length and width will likely be characteristic of a normal curve, and so ISO13322-1 can be used to calculate the number of particles to be measured by the analyzer given the estimated standard deviation and a desired confidence level for these dimensions (see Appendix X4).

10.4.2 Obtain a representative sample of the catalyst material per 9.2.

10.4.3 Analyze the obtained sample per the procedure of Section 11.

11. Procedure

11.1 *Procedure A – Reference Materials:*

11.1.1 Place the sample of reference particles (see 7.2), obtained per Section 9, into the loading container of the analyzer. When catalytic materials to be analyzed are near spherical in shape, spherical reference particles (see 7.4) may be used instead of the cylindrical type (see 7.2).

11.1.2 Place a clean container (see 6.1.1.4) at the outlet of the measurement zone to catch spacers.

11.1.3 Follow the manufacturer's instructions to engage the analyzer to run the entire quantity of spacers through the measurement zone to obtain size and shape results.

11.1.4 Assess the results per Section 10 to confirm the verification is valid.

11.2 *Procedure B – Catalytic Materials:*

11.2.1 Place the sample of catalytic materials, obtained per 10.4.2, into the loading container of the analyzer.

11.2.2 Place a clean container (see 6.1.1.4) at the outlet of the measurement zone to catch tested materials.

11.2.3 Follow the manufacturer's instructions to engage the analyzer to run the entire quantity of the test specimen through the measurement zone to obtain size and shape results.

11.2.4 Assess the results per Section 10 to confirm the verification is valid.

11.2.5 Once the analyzer is verified, steps 11.2.1 – 11.2.3 may be used to analyze catalytic materials similar in size and shape to those used in the verification process.

12. Recommended Reporting

12.1 In order to compare results of measurements, the following parameters are recommended for reporting:

12.1.1 *Length*—Value and parameter used ($X_{FE\ max}$, X_{Length}, etc…).

12.1.2 *Width*—Value and parameter used ($X_{FE\ min}$, $X_{Ma\ min}$, etc…).

12.1.3 *Aspect Ratio.*

12.1.4 *Procedure Used (A or B).*

12.1.5 *Sample Identification.*

[5] STP 447A, *Manual on Test Sieving Methods*, ASTM International, West Conshohocken, PA 19428.

13. Keywords

13.1 catalytic materials; catalytic reference materials; direct imaging analyzer

APPENDIXES

(Nonmandatory Information)

X1. PARTICLE SHAPE FACTORS

X1.1 This appendix contains information to calculate particle shape parameters of two dimensionally imaged particles. Several parameters are included as they may be useful for different particle types (Figs. X1.1-X1.3).

X1.1.1 *Caution*—Terminology describing like parameters may differ throughout industry. The terms used in this practice, Aspect Ratio and Convexity, are defined in equation form, or by diagram, in this appendix so there is no confusion as to what they represent.

X1.2 For the particle in Fig. X1.2, the length is calculated as:

$$X_{Length} = SQRT(X^2_{FE\ max} - X^2_{Ma\ min}) \quad (X1.1)$$

X1.3 Aspect Ratio = $X_{Ma\ min}$ / Particle Length where particle length can be represented by various parameters such as X_{Fe}, X_{Length}, or $X_{Fe\ Rect}$. The preferred length parameter will likely depend on the expected shapes to be analyzed.

X1.4 Convexity, is calculated by the ratio of actual particle area to the area contained within the convex perimeter of the two dimensionally imaged particle (see Fig. X1.4).

NOTE X1.1—The convex perimeter can be envisioned as the perimeter a rubber band would make if stretched around the particle.

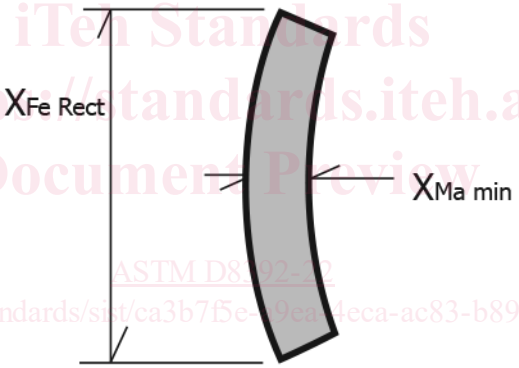

FIG. X1.1 Particle Length and Width, Extrudates

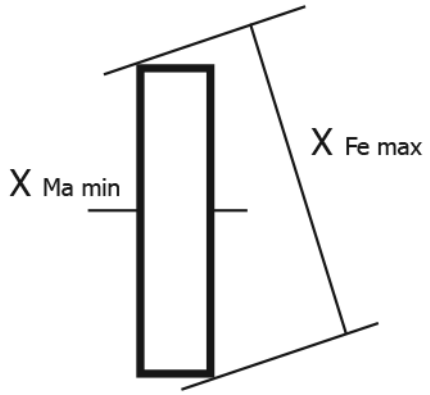

FIG. X1.2 Particle Length and Width, Extrudates

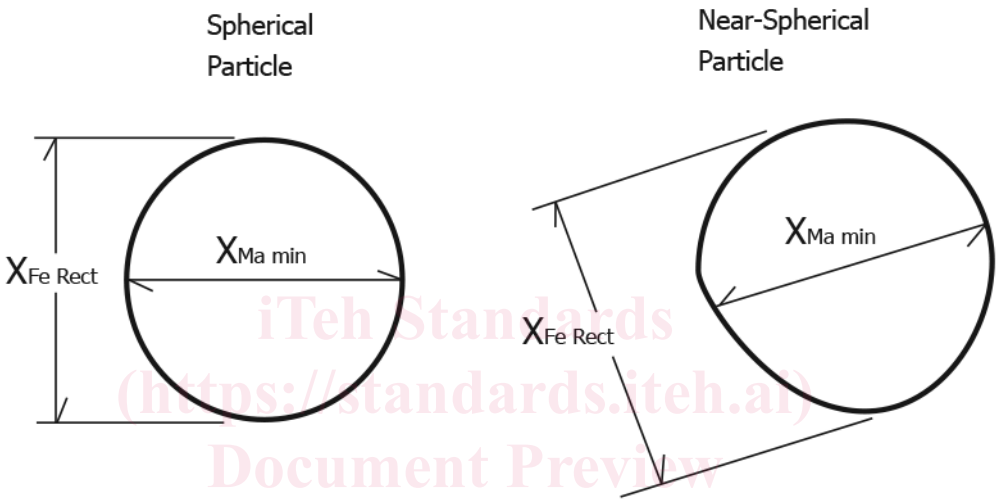

FIG. X1.3 Particle Length and Width, Spheres

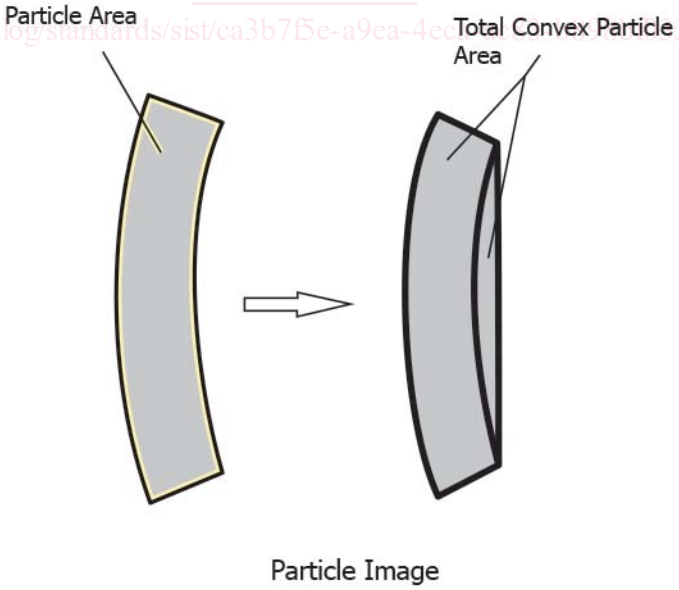

FIG. X1.4 Convexity = Particle Area / (Total Convex Particle Area)

X2. DEFINITION OF $X_{Ma\ min}$ AND X_{FeRect} FOR EACH TYPE OF CATALYST PARTICLE IMAGE

X2.1 See Figs. X2.1-X2.3.

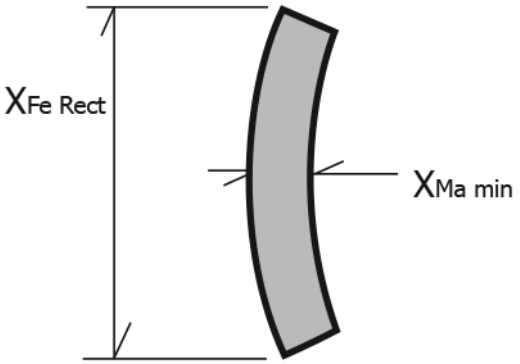

FIG. X2.1 Single Lobe Cylinder

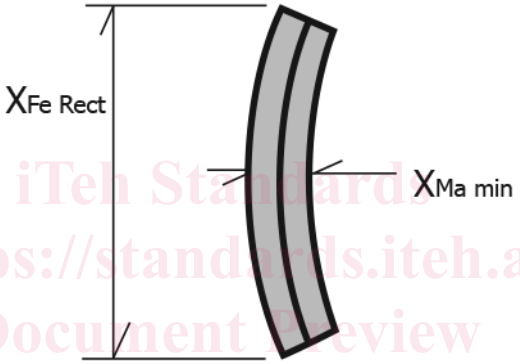

FIG. X2.2 Dual Lobe Cylinder

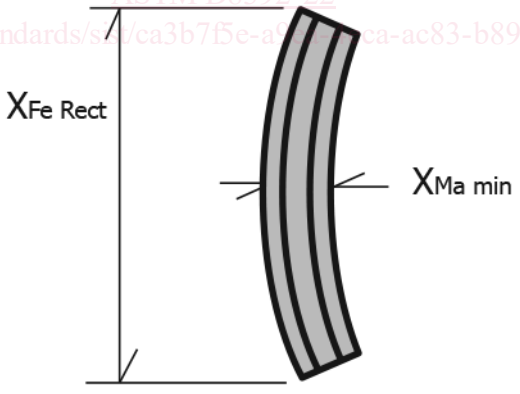

FIG. X2.3 Tri-Lobe Cylinder

X2.2 For these particle shapes, the base width, $X_{Ma\ min}$, of the image can be used to calculate the height per the following equations which are provided for reference only (Figs. X2.4-X2.6):

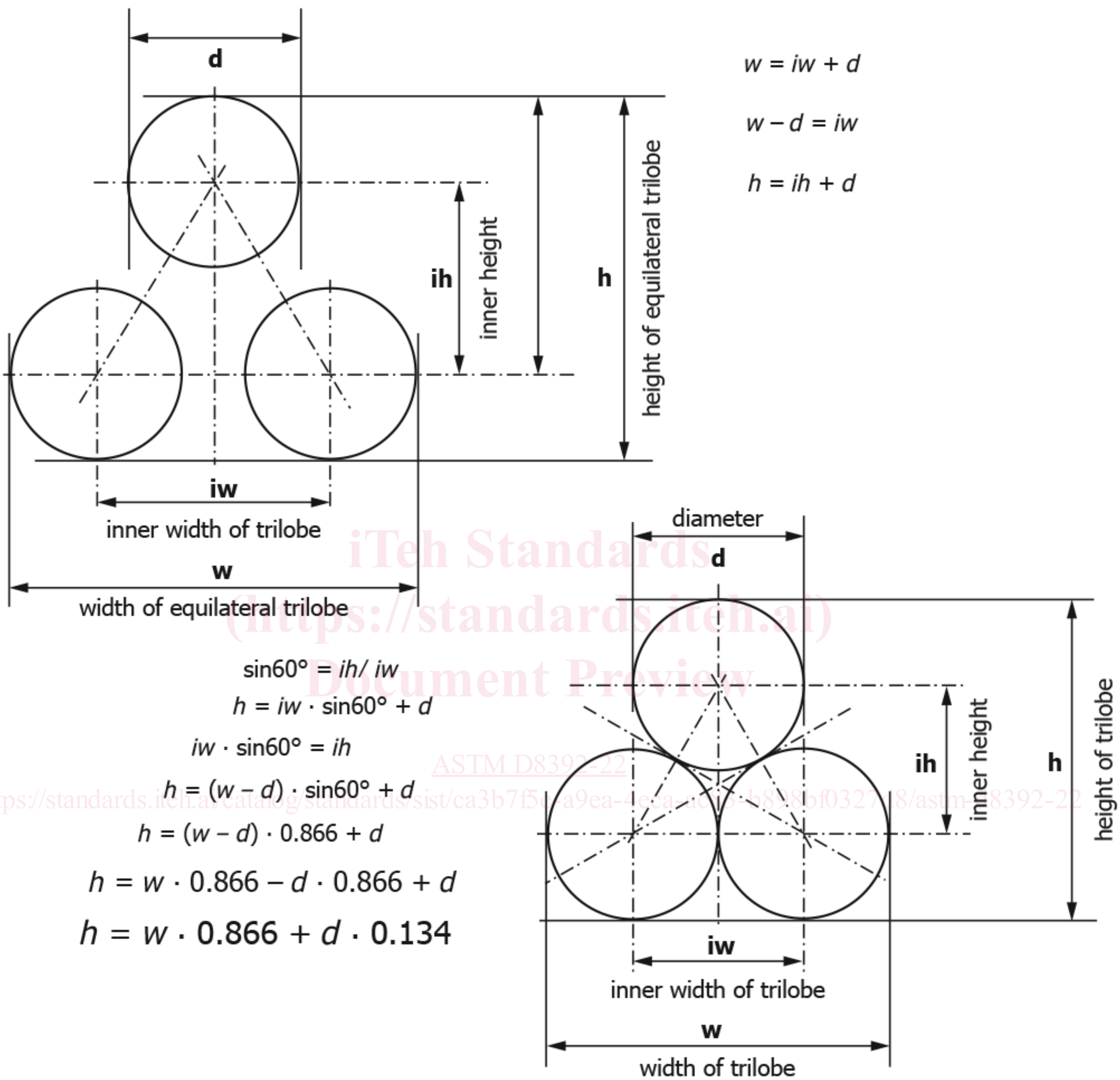

FIG. X2.4 Tri-Lobe Cylinder