Designation: E3338 – 22

Standard Guide for Size and Shape of Solid Particles, Liquid Droplets, and Gas Bubbles, Dynamically Conveyed, Using a Dynamic Imaging Analyzer[1]

This standard is issued under the fixed designation E3338; 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 guide provides information for determining particle size and shape using Dynamic Imaging Analyzers (DIA) in multiple application points including in-line, at-line and stand alone, lab based or portable, configurations. This guide focuses on concepts and strategies for applying imaging techniques to process applications in a way that improves the knowledge of the particles contained in dynamic flows, dry and wet, which can lead to more improved control of manufacturing processes.

1.2 Analyzers may be configured for open, dry or wet analysis, or enclosed, dry or wet analysis, as appropriate for analysis of the process or test specimen. Particles in liquid borne flows can be analyzed at least up to 1000 µm and dry particle flows can be analyzed up to several cm if equipment is appropriate for the size. Limitations will be discussed in Section 6.

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

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]

B215 Practices for Sampling Metal Powders

B821 Guide for Liquid Dispersion of Metal Powders and Related Compounds for Particle Size Analysis

D4057 Practice for Manual Sampling of Petroleum and Petroleum Products

D6323 Guide for Laboratory Subsampling of Media Related to Waste Management Activities

D7596 Test Method for Automatic Particle Counting and Particle Shape Classification of Oils Using a Direct Imaging Integrated Tester

E2589 Terminology Relating to Nonsieving Methods of Powder Characterization

E2651 Guide for Powder Particle Size Analysis

2.2 *API Standard:*[3]

API STD 19C Measurement of and Specifications for Proppants Used in Hydraulic Fracturing and Gravel-packing Operations

2.3 *DIN Standard:*[4]

DIN 66141 Representation of Particle Size Distributions – Basic Standard

2.4 *ISO Standards:*[5]

ISO 13322-1 Particle sizing analysis – Image analysis methods – Part 1: Static image analysis methods

ISO 13322-2 Particle size analysis – Image analysis methods – Part 2: Dynamic image analysis methods

[1] This guide is under the jurisdiction of ASTM Committee E29 on Particle and Spray Characterization and is the direct responsibility of Subcommittee E29.02 on Non-Sieving Methods.

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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 American Petroleum Institute (API), 200 Massachusetts Ave. NW, Suite 1100, Washington, DC 20001, http://www.api.org.

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

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

ISO 14488 Particulate materials – Sampling and sample splitting for the determination of particulate properties
ISO 14887 Sample preparation – Dispersing procedures for powders in liquids

3. Terminology

3.1 *Definitions*—For definitions of terms used in this guide, refer to Terminology E2589 and ISO 13322-2.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *bounding box, n—in image analysis*, the narrowest fit of a rectangular box enclosing a particle defined by the minimum Feret diameter, $X_{FE\ Min}$.

3.2.2 *dynamic imaging analyzer (DIA), n—in image analysis*, a type of instrument where a test specimen is conducted through an illuminated measurement volume where images of the particles (solid particles, liquid droplets or gaseous bubbles) of the specimen are captured and their size and shape characteristics are determined as appropriate to the application.

3.2.3 *pixel, n—in image analysis*, the smallest addressable element of an image display.

3.2.4 *smoothness, n—in image analysis*, with reference to the perimeter of the particle projection, the measure of the roundness of a particle's contours and is defined by the ratio of the area of the particle projection (Ap) to the area of the smallest ellipse which encloses the particle projection (Ae); Smoothness = Ap / Ae.

3.2.5 *transparent circularity, n—in image analysis*, refers to a modification of the traditional circularity calculation by replacing the outer perimeter with the sum of the outer and inner perimeters of particles with transparent centers.

3.3 *Symbols:*

3.3.1 A—area of the two-dimensional particle projection

3.3.2 $X_{FE\ Max}$—maximum Feret diameter

3.3.3 $X_{FE\ Min}$—minimum Feret diameter

3.3.4 X_{Length}—calculated particle length

3.3.5 $X_{Ma\ Min}$—minimum Martin diameter

4. Significance and Use

4.1 This guide is intended to inform those who have need for particle analysis data of their product or process, how imaging technology, in the form of a DIA, can be employed to provide the required information for a wide range of processes and material types. It expands on dynamic imaging information provided in Guide E2651 which is a broad view of particle analysis methods.

4.2 This guide can be used to assess the suitability of the technology to particular applications as well as any limitations that may be encountered. It is also intended to help the user make an informed decision on how to best use the technology to make the measurement(s) most important in providing data that best describes the process or product.

4.3 Determining particle shape of materials such as proppants, catalysts, additive manufacturing powders, and many more materials, is critical to their performance. Imaging technology can provide a consistent assessment of shape factors based on objective criteria and a statistically significant number of particles analyzed. Human visual methods generally compare a small number of particles to a standard leaving room for subjective interpretation.

4.4 Determining particle count, size and shape are important in assessing contamination of fluids such as fuels, lubricating oils, water, injectables, and other liquids where particle contamination can affect their performance. Particle shape can point to the type and source of these particles which can help analysts improve process control.

4.5 Shape information is also advantageous in categorizing particles detected so as to not skew particle analysis results. For instance, if a flowing mixture of solid particles in liquid also contains gas bubbles or water droplets, it is important to be able to identify the bubbles and droplets and not count them as solid particles.

5. Sampling and Sample Preparation

5.1 Analyzers may be installed directly into a pipe with flowing liquid, at-line by sampling liquid or off-line by sampling liquids for test. Analyzers, differently configured, can also be used to measure size and shape of dry particles conveyed through the measurement volume.

5.1.1 Obtaining a representative sample for analysis is a critical first step in achieving good results. Guide E2651 discusses standards available for sampling and sample handling (reduction, dilution, etc.) that are used in industries where particle analysis is performed. One should refer to ASTM standards for sampling practices and methods whenever possible. A good reference for sampling of liquids, stationary and flowing, is Practice D4057. Good references for sampling dry powders are Practice B215 and ISO 14488.

5.1.2 Preparation of the test specimen may be required in order to ensure the analyzer sees a true representation of the sample. For instance, inspection of fluids which contain solid particles require the test specimen to be agitated to disperse the particulate in the fluid and to avoid settling out of particles prior to analysis. The particle concentration of a mixture may also cause particles to be overlapping in the image capture which can lead to error in assessing size and shape.

5.1.3 Dry solid analyzers generally have a hopper and vibratory feed mechanism. There is a tendency for solids of different sizes to segregate with the smallest particles collecting at the bottom of the hopper and being analyzed first. It is important that the entire test specimen be analyzed in order to avoid errors that could arise from partial analysis of the test specimen.

5.1.4 Dry solids in powder form may more easily be analyzed in slurry form (see 6.2). Guide B821 and ISO 14887 describe methods that may be used to slurry metal powders for analysis. This practice is included as a general reference as these methods may be applicable to other materials.

5.1.5 Samples obtained may require further reduction in volume in order to create a test specimen that is suitable for the analyzer. Guide D6323 provides an overview of standards available to accomplish this function. In addition to creating a sample size appropriate for the physical restrictions of the

analyzer, the user must also ensure that a proper volume of material is assessed in order to yield repeatable results. ISO 13322-1 can inform on determining a sufficient sample size for particle distributions.

6. Limitations

6.1 Two phase flows that tend to separate, solid-liquid or liquid-liquid, can be difficult to sample accurately. Following the advice in Practice D4057 is recommended in order to maximize sampling efficacy.

6.2 Very small particles, such as those that make up powders, may tend to agglomerate. Guide B821 offers recommendations for dispersing particles in base liquids prior to analysis. Dry solid flows can also experience agglomeration of particles due to Van der Waal's forces between particles or moisture, which can lead to error in size and shape analysis without treatment of the material to mitigate the cause. Some instruments offer a pressurized air dispersion system to disperse particles prior to dry analysis.

6.3 Too many particles in the field of view can lead to the detection of apparent doublets and agglomerations. In many instances this can be recognized by visual inspection of the images captured. A reduction in the particle concentration, through dilution or reduction in feed rate (for dry solid particles falling through air), can resolve this.

6.4 Non-spherical particles may present themselves in random orientations to the imaging device which can lead to variation in the analysis result. In liquid based laminar flows this is much less an issue, versus turbulent flows, as the particles tend to flow in a manner that exposes their broadest dimension to the imaging device for analysis. For dry solid analyzers, many analyzer manufacturers have proprietary methods to align particles in a consistent manner to the imaging device, but not all applications and size ranges may be resolved (see Fig. 3).

6.5 Particle motions can cause images to be blurred thereby masking particle features and limiting the accuracy of the size and shape analysis. To alleviate this effect the aperture setting of the camera, or the time interval in which it accepts light rays, can be reduced in order to reduce the effect of motion. When this is done the image appears in better focus, however the shorter time exposure also limits the amount of light that enters the camera for each image with the unwanted effect of dimming the image. ISO 13322-2 contains recommendations on maximum particle velocities and aperture settings.

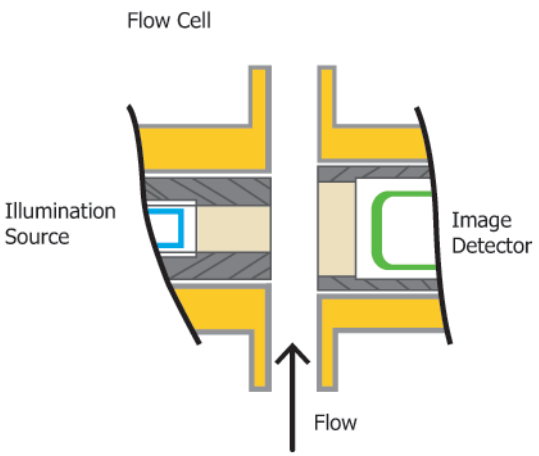

FIG. 1 Typical Flow Cell Cross Sectional

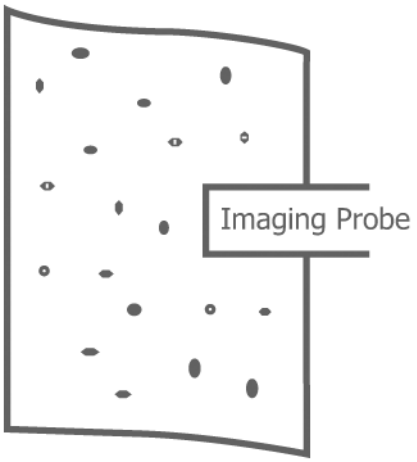

FIG. 2 Single Probe Imaging Device

6.6 Particle shape analysis requires a particle image to consist of several pixels. The minimum recommended pixel count per particle image for shape analysis would be 81, or 9 × 9, per guidance of ISO 13322-2. Where particle distributions include particle images represented by less than the minimum count there will be uncertainty in the shape analysis of the entire distribution. For example, in the case where shape is used to identify particles such as water droplets from solids, droplet images of less than 9 × 9 pixels in size will likely be identified as solids since no shape measurement is available.

6.7 The technology applies down to approximately 0.4 µm, the lower bounds of the visible spectrum. The upper end is unlimited, however with fixed equipment settings the field of view limits the total range of particle size that can be captured.

7. Apparatus

7.1 Analysis of liquid-based flows are often conducted through a cell with the imaging device on one side and the illumination device on the other. The space between them is the measurement volume where the focal plane of the image must be located. It is generally ideal for the focal plane to be centered in the measurement volume to avoid flow variations which can occur, especially in laminar flows. Fig. 1 shows a typical flow cell schematic. Single probe type devices, where illumination and image detection are combined, are also common configurations where front lighting of the particles works well (see Fig. 2).

7.2 Dry solid flows are most often mechanically conveyed to a point above the measurement volume where they then free fall through it. The movement of the particles from the hopper to the feed tray is controlled in order to create a thin layer of particles that can be individually imaged. Fig. 3 shows a typical schematic. The focal plane of the imaging sensor must be of sufficient depth to capture the particle field.

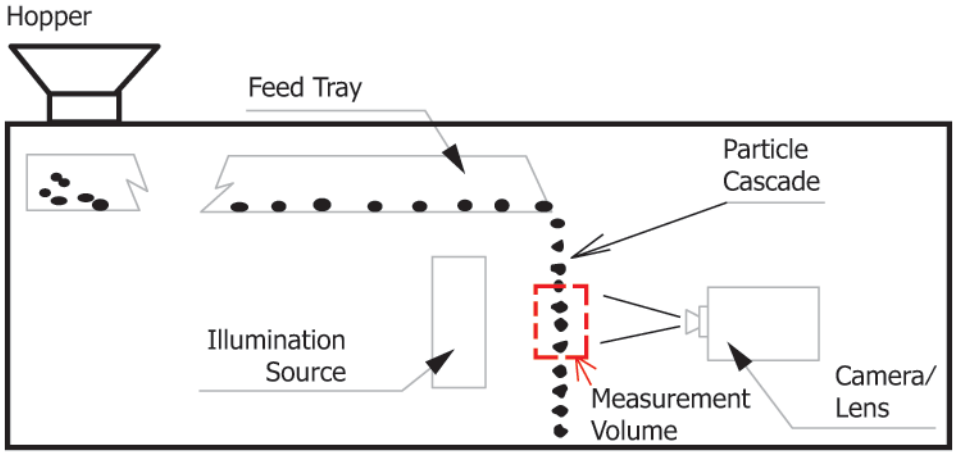

FIG. 3 Typical Dry Solids Analyzer

8. Imaging Fundamentals

8.1 *Sensor*—High resolution, digital technology is ideal for imaging work. Currently, mega pixel arrays allow for good resolution down to 0.5 µm when paired with an appropriate lens. Resolution can be defined by the pixel scale factor (psf) which defines the number of micrometres of view per pixel. Practical applications of the technology range from analysis of particles <1 µm size to particles >100 000 µm in size. It is good practice that the size of the largest particle to be sized, when using a dynamic analyzer, be no more than ⅕ th the span of the view to avoid partial imaging of particles on the borders of the view. As an example, assume the CCD array is 4000 h × 3000 v and the lens magnification is set so the psf = 1 µm per pixel. Using the horizontal direction our field of view is 1 µm/pixel × 4000 pixel = 4000 µm. Our largest particle that could reasonably be expected to be captured in a dynamic situation would be 800 µm. On the low end of the scale there should be some resolution to the smallest particles. ISO 13322-1 contains recommendations for the minimum number of pixels that make up a particle to be analyzed for count, size and shape. For particle count one pixel can be used. For size measurement, a particle should have a minimum dimension of 3 pixels, and for shape a minimum dimension of 9 particles. In each case calibration and verification standards should be used to confirm the selected pixel counts per particle image.

NOTE 1—Refer to ISO 13322-1 and ISO 13322-2 for in-depth information on static and dynamic imaging fundamentals.

8.2 *Illumination*—In order to consistently detect particles, they must be illuminated evenly across the field of view and over time. Uneven lighting over space and time creates inconsistent detection of particles because detection algorithms rely on the intensity difference between the particle and the background.

NOTE 2—The continued development of high intensity, long life LED technology, along with imaging normalization algorithms, has resolved these issues and illumination across the field of view is generally ±1 % average intensity.

8.2.1 The high intensity of the LED and the ability to strobe it has a dual benefit for fast moving dynamic applications. Synchronizing the strobe with the camera aperture directs high intensity light into the aperture during its brief opening. The effect is to capture moving objects so they appear almost still. High intensity is required when the shutter is open for such a short period of time otherwise the image is dark and with no detail. A second benefit is to prolong the life of the LED. Strobing, as opposed to constant-on operation, greatly reduces heat generation by the LED which is the leading cause of failure.

8.3 *Particle Size,* often expressed as a mean value of particle diameters. It can also be expressed as a distribution of particle diameters. The traditional method of determining size of a dry material is by sieve analysis which does not directly measure any particle parameter, rather it measures the relevant amount of a mass of product that falls through, or is retained on, screens with various size openings. For non-visual instruments, particle size has often been expressed in terms of a single parameter which is diameter. For example, light obscuration devices measure the area of a particle projection, assume the area is circular in form and determine the diameter of the circle which becomes the particle's size descriptor. Laser diffraction instruments measure the diffracted light patterns of particles, and based on these, calculate a particle diameter. Imaging technology attempts to determine the actual minimum and maximum dimensions, as well as shape, of each two-dimensional particle image.

NOTE 3—This discussion of particle dimensions actually refers to particle image dimensions which represent the projections of the actual particles from which the images are created.

8.3.1 *Feret Diameter (Terminology E2589)*—There are various particle descriptors available to imaging algorithms that can be used to describe particle dimensions. A common representation of particle dimensions is through a rotating bounding box type algorithm. This method determines the minimum of the maximum Feret diameters (Terminology E2589) by measuring the Feret diameters around the particle in all 360°. Fig. 4 shows the particle and the bounding box. The diameter is usually denoted by $X_{FE\ Min}$.

8.3.1.1 The $X_{FE\ Min}$ would be very close to the opening size of a sieve that the particle would fall through. Other parameters are also used as approximations to the sieve including the minimum elliptical diameter and the maximum inscribed disk diameter to name just two.

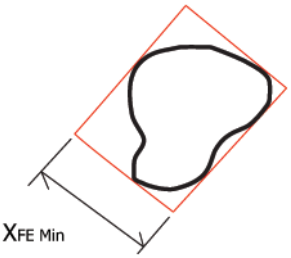

FIG. 4 Minimum Feret Diameter

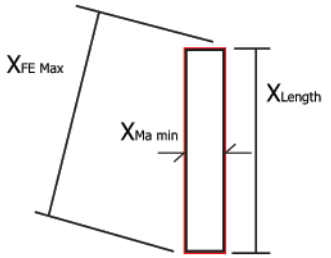

FIG. 6 X_{Length} Parameter

8.3.2 *Martin Diameter (Terminology E2589)*—Where particles display a bent shape such as in Fig. 5 the minimum width of the bounding box does not accurately describe the particle. In these instances, the minimum area bisector, or minimum Martin diameter, can be used. This is helpful in describing catalyst, fiber and other similarly shaped materials.

8.3.3 X_{Length}—For particles with a rectangular projection such as in Fig. 6, a length dimension matching the bounding box length is useful. In these cases, if a maximum Feret were used, the length dimension would be from corner-to-corner which is not likely the value that is desired. The value of X_{Length} equals SQRT($X_{FE\ Max}^{2} - X_{Ma\ Min}^{2}$).

NOTE 4—Other particle dimensions can be used depending on their value in describing parameters of interest. Reference DIN 66141 for a more complete catalog of particle descriptors.

NOTE 5—A closer approximation to how a particle would fall through a sieve could be made if a 3rd dimension were available. Some instruments do provide an orthogonal measurement to the 2-D image plane to accomplish this. Others gather multiple images of the particle as it traverses the measurement volume in order to determine its maximum and minimum dimensions in 3 dimensions. Regardless, correlations are required to compare to sieve results because the measurement methodologies are different and the exact particle volumes cannot be precisely determined (sieves measure particle distribution by weight at designated sieve sizes which are not continuous). Where particulate weight densities vary, size distributions in relation to sieves becomes difficult if not impossible. The value of particle shape and size information remains unchanged.

8.4 *Particle Presentation:*

8.4.1 Consider flat or rod-shaped particles shown in Fig. 7 and Fig. 8. If the imaging detector sees them from the edge, or the end, the analysis of the particle is quite different than if the broadest dimensions are seen. Controlling the orientation of non-spherical particles is often desired in order to detect their largest dimensions. Many instrument manufacturers have proprietary systems to accomplish this. ISO 13322-2 shows examples of various methods of viewing particles.

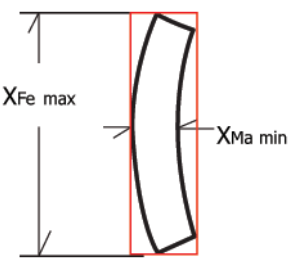

FIG. 5 Minimum Martin Diameter

FIG. 7 Flat/Flake Shaped Particle

FIG. 8 Rod Shaped Particle

8.4.2 Solid-fluid mixtures in many cases involve very small particles. Sometimes as large as grains of sand, but often much smaller in order that they remain suspended in the liquid for a sufficient period of time to flow through the instrument and analyze. These analyses are accomplished using high magnification lenses and very small flow cell dimensions. Flow through these cells is often laminar which has a large benefit in consistently presenting the particles to the detector. Laminar flows tend to order particles in the broadest orientation to the detector which is quite convenient and requires no secondary handling methods to accomplish. This is not the case with turbulent flows and so in those cases further work may be needed to either influence the particle orientation or perhaps consider a three-dimensional imaging scheme.

9. Particle Imaging Analysis Techniques

9.1 This section will be divided into two general categories of particle analysis: mathematical and image comparison. Basic mathematical formulae can be used to identify particle characteristics such as aspect ratio and circularity and a number of other particle parameters that are useful in classifying them. More advanced mathematical treatments are also available that can determine more subtle shape characteristics. Image comparison can be used to 'teach' the software what particles look like and classify them according to their similarities.

9.2 *Basic Mathematical Functions:*

9.2.1 *Aspect Ratio*—Defined in Terminology E2589 and is the ratio of the length (L) to the width (w) of the 2-D particle image. In many cases the inverse of this equation is used which confines the value to a maximum of 1:

$$\text{Aspect Ratio} = L / w \tag{1}$$

9.2.2 *Circularity*—Traditionally defined as a ratio of the particle image area (A) to the particle image perimeter (P) squared. A solid, circular particle image would have a circularity of 1 while a needle shaped particle would be << 1.