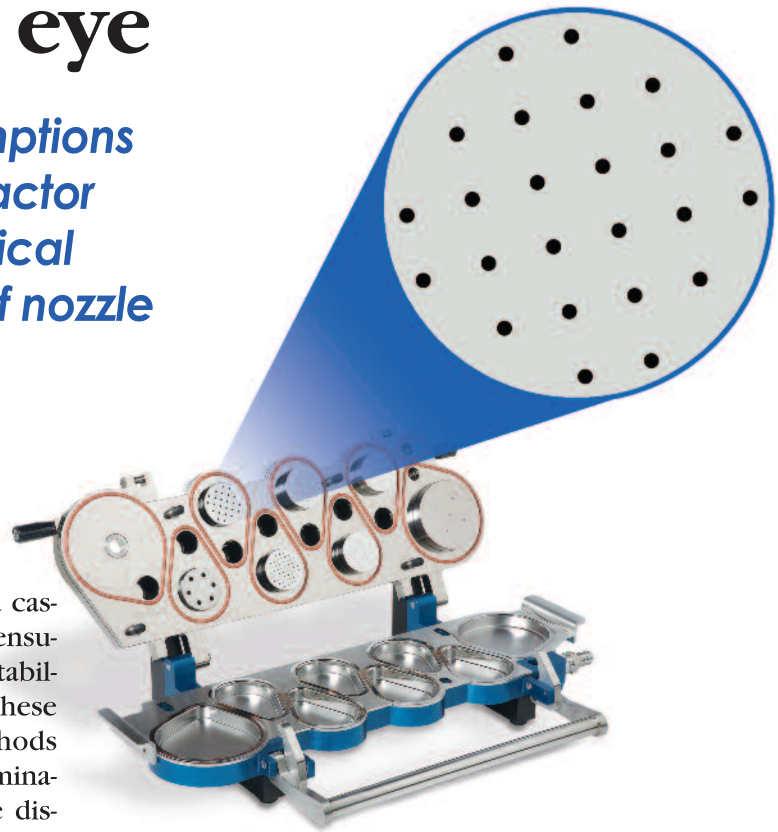


Accuracy in the optical inspection of impactor nozzles—More to it than meets the eye

This article addresses assumptions in optical inspection of impactor nozzles, then compares optical and non-optical methods of nozzle measurement.



Daryl L. Roberts and Christian A. Lavarreda
MSP Corporation

Periodic optical inspection of the nozzles of a cascade impactor, known commonly as "stage mensuration," is widely accepted for determining suitability for continued use of impactors. Tools for these measurements have been studied,[1] and methods exist for handling the data and making a determination of the suitability for use.[2,3] However, the discussion of optical inspection in the impactor nozzle measurement community has so far relied on two key assumptions. The first of these is that the focused, two-dimensional image of the nozzle properly reflects the shape of the three-dimensional surface that constitutes the walls of the nozzle. The second assumption is that a single parameter, namely diameter, is meaningful and sufficiently characterizes the nozzle dimensions. Stated more colloquially, the second assumption is simply that the nozzle is round.

The current article addresses these assumptions quantitatively by describing the basics of the way in which a state-of-the-art optical system quantifies nozzle dimensions, then comparing these optical nozzle measurements with a non-optical method of nozzle measurement. The results bear directly on the question of *accuracy* of the nozzle diameter data gathered with an optical system. The *repeatability* of optical system measurements is a separate question that is also addressed briefly in the context of the accuracy determination. Dimensional accuracy and the presumption of roundness in nozzle measurements are important because impactor nozzle specifications for new and used impactors hinge on single-digit micron boundaries. If the object studied is not round, then "diameter" begins to lose its meaning as a single-parameter method of quantifying nozzle dimensions. Indeed, the pharmacopeial specifications for impactor nozzles presume roundness but with no definition of its meaning. The following discussion explains a path forward for remedying this oversight.

The basis of optical inspection accuracy—Defining an edge

It is important to describe how an optical system quantifies the dimension(s) of an object so that the principles can be understood and properly applied to the questions at hand. Whereas the 'lingo' that describes a properly operating optical system can vary from manufacturer to manufacturer, the terms herein are those that apply to the Mitutoyo (Aurora, IL, US) vision systems that MSP maintains in its laboratory[4] (http://ecatalog.mitutoyo.com/Vision-Measuring-

Systems-C102.aspx). These systems conduct automated two-dimensional (2-D) microscopic image analysis, a subset of an increasingly wide class of image analysis equipment currently on the market.

The fundamental accuracy of the optical system derives from the calibration reticle supplied by the manufacturer of the image analysis system (Figure 1). The key attributes of this chrome-on-glass reticle are that all the features are square (no curved shapes) and that the length-scale of the chrome components covers the range of sizes of the optical tools necessary to measure the intended objects under the user-selected magnification levels.

Figure 1

Calibration reticle for the Mitutoyo QV-404 and QV-ELF Pro Vision Systems

The calibration reticle that MSP employs has all-square features with dimensions ranging from 20 microns to 4,000 microns. These dimensions are determined every five years with NIST-traceable laser interferometry at a local laboratory, yielding accurate values to within 0.6 micron (95% confidence interval).

The calibration routine optimizes the lighting and focus by maximizing the contrast between adjacent gray pixels across the edges of each given chrome element. Then, to locate each edge, it determines which pixels correspond to the factory-specified edge-contrast level (a grayscale definition). Because the distance between these edges are known lengths, the system can then count the number of pixels across each of the chrome elements, giving a quantified length-scale to each pixel.

The stated accuracy of the calibration reticle defines the *best accuracy* of the optical measurements that one will achieve when measuring the dimensions of any object. The user has manual control of the optical settings if he or she so desires, namely light intensity, speed of traversing an edge and the definition of contrast (bright pixel versus dark pixel). However, the key to achieving the *best accuracy* in practice is discovering a set of optical settings (the independent variables) that yield sensible interpretations of the reticle object dimensions and then ***keeping these settings*** memorized by the optical system so that they are not subject to lamp fade, user focus and other such sources of error and variability.

The notion of "sensible interpretations" is purposely vague—so as to emphasize that there are a range of independent variables (optical settings) that one could choose, if one wishes, so long as the settings yield at least reproducible results. However, to save the user a potentially frustrating and lengthy process of finding such viable optical settings, the Mitutoyo software automatically runs a search routine with the calibration reticle in place which identifies optical settings that optimally define an edge. These settings are then maintained when the system is operated in the measurement of an object.

In this search routine, focus takes place automatically by changing the focal length (the Z position) so as to minimize the number of gray pixels (a gray pixel is defined to be one that fails to fall into the category of "dark" or "bright" in the user-defined contrast test). The X-Y contrast controller moves along only one axis at a time as it crosses each chrome/glass edge on the calibration reticle. In doing so, it identifies the best conditions for edge detection for the X direction separately from the best conditions for edge detection for the Y direction. It also distinguishes the meaning of an edge when crossed from a dark area to a bright area separately from an edge crossed from a bright area to a dark area. The critical role of the calibration reticle in achieving accuracy is that the conditions for edge detection are identified by forcing the distance from edge to edge of a reticle feature to be the known stated dimension for that feature.

Quantifying the edge-contrast diameter

The settings memorized by the optical system through the use of the calibration reticle allow confident detection of an edge feature on a real object and, in particular, locating the edge of a nozzle, even though the nozzle itself is (at least approximately) round. The Mitutoyo software contains a method called the "circle tool" that, with the edge definition now available to it, can determine the circumference of a circle. In the circle tool method, the search for the edge of the nozzle takes place on hundreds of rays pointing in the direction from the dark area outside the nozzle to the center of the lighted area inside the nozzle. The focus at each edge determination is made by minimizing the number of gray pixels. The curvature of the nozzle is inconsequential to finding its edge because each pixel is about 0.3 micron in size (square) and therefore the tangent to the nozzle is indistinguishable from the actual nozzle edge. Once these hundreds of edge locations are identified, the best-fit perfect circle based on these edge locations is computed by a routine that is considered proprietary by Mitutoyo, but which is believed to be essentially a least-square fitting routine such as that described by Shakarji[5] (section 3.4). The edge-contrast diameter (ECD) is simply the circumference of this best-fit perfect circle divided by π.[4]

The power of the edge-contrast "memorized settings" and the logic of the circle tool with its "least-square" fitting routine is demonstrated in Table 1, where the measured edge-contrast diameter of glass dots on a chrome-on-glass reticle are compared to the diameters stated by the reticle manufacturer (denoted as D-ref; Klarmann Rulings, Litchfield, NH, US). The ECD repeatability is no larger than 0.1 micron and the ECD values agree with the D-ref values within the stated accuracy of the reticle.

Table 1

Accuracy and repeatability of edge-based diameter measurements

Nozzle	D-ref[1] (mm)	ECD 1 (mm)	ECD 2 (mm)	ECD 3 (mm)	ECD Avg (mm)	ECD SD (mm)
S3	2.1822	2.1821	2.1820	2.1820	2.1820	0.0001
S4	1.2074	1.2078	1.2078	1.2078	1.2078	0.0000
S5	0.6084	0.6084	0.6084	0.6084	0.6084	0.0000
S6	0.3252	0.3250	0.3250	0.3251	0.3250	0.0001
S7	0.2060	0.2060	0.2060	0.2060	0.2060	0.0000
MOC	0.0670	0.0668	0.0667	0.0668	0.0668	0.0001

[1] Chrome-on-glass reticle diameters measured independently by reticle manufacturer with 0.5-micron accuracy

Quantifying the area-based diameter, enabling a roundness measure

The Mitutoyo optical system has a completely separate method, known as the centroid tool, for measuring the area of a lighted feature. This method relies on a calibration with a separate reticle with circular objects. For this purpose, MSP uses a chrome-on-glass reticle with clear glass circular dots that are approximately the nominal size of the nozzles for stages 0 through 7 of the Andersen impactor (2.55 mm to 0.254 mm). With accurately known ECD optical settings, the centroid tool is now employed to force the area-based diameters to equal the ECD values (within the one-micron total uncertainty intrinsic to the measurement equipment). These centroid tool optical settings, once developed, are the same for all nozzle sizes that fit entirely in the field of view. The centroid tool essentially counts the number of lighted pixels in the field of view, and with the known magnification settings, each (square) pixel has a known area. The total area divided by π is the square of the assumed-perfectly-circular nozzle radius, and the area-based diameter (ABD) is therefore simply two times this calculated radius.[4]

The power of these memorized settings for area measurements is demonstrated in Table 2, where the measured area-based diameter of glass dots on a chrome-on-glass reticle are compared to the ECD measurements. The ECD repeatability is typically 0.1 micron or less, and the ABD values are in agreement with the ECD measurements within 1.0 micron plus the stated uncertainty of the glass-dot reticle standard. As with the ECD determinations, the repeatability of the optical measurement is an order of magnitude better than the accuracy; in practice, this result means that the total uncertainty is dominated by accuracy uncertainty.

Table 2

Accuracy and repeatability of area-based diameter measurements via centroid tool

Nozzle	ECD Avg (mm)	ECD RSD	ABD Avg (mm)	ABD RSD	Difference between ECD Avg and ABD Avg (mm)
S0	2.5493	1.7E-05	2.5484	2.6E-05	0.0009
S1	1.8894	2.4E-05	1.8881	5.6E-05	0.0013
S2	0.9130	1.9E-05	0.9124	2.3E-05	0.0006
S3	0.7099	4.0E-05	0.7096	5.9E-05	0.0003
S4	0.5325	2.2E-05	0.5316	7.0E-05	0.0009
S5	0.3426	3.1E-05	0.3421	4.4E-05	0.0005
S6/7	0.2535	5.4E-05	0.2531	7.9E-05	0.0004

For a perfectly round nozzle, ABD should equal ECD. Now, with an established method of measuring ECD and ABD, an adequate definition of roundness would simply test whether ABD equals ECD. Either a ratio or a difference would be a sufficient quantitative measure of roundness. MSP has adopted the requirement for new individual NGI nozzles that ABD must be within 3.0 microns of ECD and that for all the nozzles on a stage the root mean square difference must be no more than 2.0 microns. These exact specifications, *per se*, are not being recommended for adoption in pharmacopeial guidelines because the differences between ECD and ABD can depend on the particular optical system. However, it is recommended that the pharmacopeial guidelines require that all nozzle optical systems *quantify some measure of roundness* and that the user be required to define *his or her own specification for roundness.* This practice would cause each user to provide an operational meaning of a nozzle's diameter and to validate that he or she is achieving his or her operational meaning on a routine basis. Absent an operational meaning of "diameter" (which is the current state of affairs in the industry), a test of whether nozzle diameters are within pharmacopeial specifications is fundamentally open to interpretation.

A real nozzle—What can go wrong

This somewhat belabored explanation of the method by which the optical system chooses its optical settings, and therefore the basis of the accuracy of ECD and ABD determinations, gives insight as to why these settings may deceive the user if the object being examined is three-dimensional and possibly not round. Simply stated, real nozzle edges are not as clean and sharp as chrome/glass edges, introducing uncertainty in the accuracy of the location of an edge. Secondly, real nozzles are not right-angle circular cylinders, as much as one might hope that one's machining is perfect. So, the diameter of the nozzle may not be constant along the length of the nozzle; light will be constrained to pass through the minimum diameter, diffusing outwards to the diameter at the nozzle exit plane, unless the minimum diameter is actually ***at*** the nozzle exit plane, a question that cannot be addressed optically. Both of these factors introduce more "gray pixels" when the optical system attempts to focus, so the exact focal plane may not be the same for the ECD determination as for the ABD determination. A determination of roundness (or lack thereof) is therefore not as certain with a three-dimensional (3-D) nozzle as for a chrome-on-glass reticle.

A non-optical method of defining the 3-D surface of real nozzles

A non-optical method of measuring real nozzle dimensions is necessary if one is going to inquire about the accuracy of the optical measurements. Fiber touch probe methods are well suited to this application and such tools are commercially available. The National Institutes for Standards and Technology (NIST, Gaithersburg, MD, US) maintains and operates one such fiber touch probe device known as the Ultrasonic Micro and Accurate Probe (UMAP) Model Ultra 350 (http://ecatalog.mitutoyo.com/UMAP-Vision-System-VisionMicro-Stylus-Measuring-Systems-C1577.aspx) inside its Dimensional Metrology Group. This group has also developed several advanced fiber deflection probe devices.[6]

For these non-optical measurements, NIST used the UMAP Fiber Probe Model 103 that has a 30-micron-diameter probe tip on the end of a 20-micron-diameter shaft, which can be inserted to a depth of up to two millimeters into a nozzle. In operation, the fiber is vibrated piezoelectrically; when it touches the wall of an object, the vibration is partially dampened. This change in the shape of the vibration curve can be detected readily and the position of the probe is then recorded. With precise control of the X-Y coordinates, the probe can be brought into contact with the wall of a nozzle in multiple locations. The probe can then be positioned further into the nozzle (Z direction), so that the diameter can be determined as a function of the distance from the nozzle exit plane.

To test this fiber "touch probe" method and compare it to the optical inspection method, MSP machined six nozzle pieces, each with one nozzle approximately the size and three-dimensional shape of the nozzles on the NGI stages 2 to 7 and mounted them in a fixture (Figure 2) that could undergo examination in the UMAP Model 130. NIST personnel determined the diameter of these

Figure 2

Prototypical NGI nozzles for fiber probe measurements

(a) Back-side

(b) Front-side

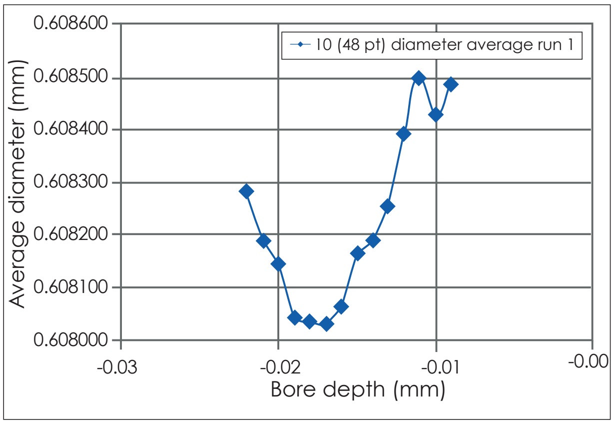
nozzles at several locations ***inside*** the nozzles, mapping the nozzle shape by physical touch of the fiber probe to the wall of the nozzles, contacting 48 touch points around the nozzle circumference and repeating these measurements 10 times at each depth position. In this way, the fiber touch probe method gives a true physical measurement of the coordinates of the inner wall of each nozzle so examined, leading immediately to a least-square calculation of circumference, diameter and area.

Figure 3 is a typical plot of the diameter of the nozzle as a function of depth ***into*** the nozzle. The fiber probe entered each nozzle from the front side noted in Figure 2b. For all six of the prototypical nozzles studied, it was found that the minimum nozzle diameter was about 20 microns to 60 microns deep into the nozzle. In addition, the nozzle exit plane diameter was typically 0.5 micron larger than the minimum nozzle diameter.

Table 3 compares the physical touch probe diameter to the optically determined diameters. Considering the details of the touch probe and the ECD techniques, one might expect full agreement between the touch probe diameter and ECD, within +/- one micron. In fact, for the smaller nozzles, the agreement is within this expected uncertainty window. However, for the nozzles larger than a millimeter (S3 and S4), the difference is outside this uncertainty window. It is logical, based on light-scattering principles, that the nozzle exit plane focal point will have more diffuse "gray pixels" than will be found for a chrome-on-glass reticle and that the optical ECD is smaller than the touch probe diameter. However, it would take a more complete and repeated study of this phenomenon to establish fully the statistical significance of the two-micron or three-micron difference found between the touch probe and the ECD measurements for the S3 and S4 nozzles. Nevertheless, in practice, it is a wise course of action to keep new impactor nozzles several microns smaller than the pharmacopeial upper limit on nozzle sizes so as to eliminate this potential problem from having any influence on aerodynamic particle size distribution measurements.

Figure 3

Diameter of nozzle S5 at various depths below the nozzle exit plane



The optical results themselves show that the nozzles are not quite as round as the clear dots on the chrome-on-glass reticle, as evidenced by the single-digit-micron differences between ECD and ABD. The magnitude of these roundness measures would, however, be within the specification of roundness that is used by MSP for all new NGI nozzles. The fact that ECD is greater than ABD is consistent with the preponderance of the approximately one million NGI nozzles that MSP has examined in the manufacture of new NGIs. So, overall, there is no concern about roundness when looking only at the optical results.

If anything may be disturbing about the data, the comparison of the touch probe results to the ABD causes concern that the nozzles may be more out-of-round than one might think based on the optical

Table 3

Comparison of touch probe and optical measurement methods

Nozzle[1]	Touch probe diameter[2] (mm)	Optical ECD (mm)	Optical ABD (mm)	Roundness measure ECD-ABD (mm)	Touch/optical offset; Touch-ECD (mm)	Touch/optical offset; Touch-ABD (mm)
S3	2.1853	2.1821	2.1795	0.0026	0.0032	0.0058
S4	1.2069	1.2047	1.2022	0.0025	0.0022	0.0047
S5	0.6080	0.6078	0.6059	0.0019	0.0002	0.0021
S6	0.3228	0.3227	0.3210	0.0017	0.0001	0.0018
S7	0.2053	0.2045	0.2040	0.0005	0.0008	0.0013

[1] No S1 or S2 data presented because the ABD measurement method is only possible for nozzles that fit entirely in the field of view, and S1 and S2 nozzles are too large to fit entirely in the field of view.

[2] The minimum value, determined approximately 20 microns to 60 microns below the nozzle exit plane.

results alone. Even comparing touch probe diameter to ABD for S5, S6, and S7, the roundness measure is just barely outside of the +/- one micron uncertainty window. But for S3 and S4, the difference would be outside the MSP roundness specification for new NGI nozzles. These data are influenced by the exact three-dimensional quality of the nozzle bore and it begs the question of a more rigorous approach to nozzle bore smoothness and diameter control during manufacture. Although the magnitude of the effect is "single-digit microns," these factors are currently under consideration at MSP with respect to continual improvement of the NGI nozzle quality.

As a final remark, the exact quantitative results described here may not be the case for all Mitutoyo systems nor for all optical systems, simply because the independent variables of the optical inspection methods can vary. However, the techniques described here for determining the significance of any offset can and should be applied as part of the validation of any optical nozzle inspection method.

Summary

The means by which chrome-on-glass reticles form the basis for the accuracy of optical measurements of impactor nozzle diameters and one non-optical method for measuring the physical dimensions of a nozzle surface have been described. The physical dimensions of the nozzle were shown to agree better with the optically-determined ECD rather than with the ABD, which tends to be systematically smaller than ECD. Careful understanding of the means by which the optical system quantifies the nozzle circumference and the nozzle area lends credibility to the finding of a small disagreement between nozzle physical dimensions and the optically-determined diameters, at least for nozzles larger than about one millimeter in diameter. This small discrepancy between the physical dimension and the ECD indicates that, in best practice, new impactor nozzles measured optically should steer clear by a few microns from the upper boundary of the pharmacopeial specifications, simply to leave room for this discrepancy. Used impactors, assessed according to "effective diameter,"[2] will in most cases not be affected by this discrepancy.

The difference between the ECD and ABD provides a sound quantitative basis for a roundness specification. Each user should have a specification for roundness so that he or she can decide quantitatively when a single parameter (namely, diameter) satisfactorily characterizes the nozzle, since all pharmacopeial specifications for nozzles are expressed in terms of the nozzle diameters. However, each user should have the discretion to establish his or her own quantitative definition of roundness, as opposed to a single, industry-wide roundness specification, because optical systems vary in their detailed methods.

Following the approaches outlined here, modern optical systems can be trusted to give sufficiently accurate results to the single-digit-micron level for meaningful comparison to pharmacopeial specifications.

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Daryl L. Roberts is Vice President and Christian A. Lavarreda is Product Manager, Pharma and Biotech, MSP Corporation, 5910 Rice Creek Parkway, Suite 300, Shoreview, MN, 55126, US, Tel: +1 651 287-8110, droberts@mspcorp.com. www.mspcorp.com.