Practical Aspects of Calibration of Airborne Particle Counters According to ISO 21501-4 Standard

I Ye Kovbasyuk, O Yu Maslakov, M N Shakhov

NRC Kurchatov Institute, Moscow, Academic Kurchatov Sq., 1

Corresponding author: I Ye Kovbasyuk, e-mail: kovbasyuk.ivan@gmail.com

Abstract: The article reviews the practical aspects of calibration of airborne particle counters according to GOST R ISO 21501-4 (Russian equivalent of the international ISO 21501-4) standard. The authors share their gained experience and explain what difficulties can be faced during the procedure described in the standard, how to avoid and how to revise them. The article may be useful for specialists as an introduction to the standard or for particle counters’ users who want to learn more about practical metrology aspects of these devices without going into the specifics and the basis.

GOST R ISO 21501-4 standard "Determination of particle size distribution – Single particle light interaction methods – Part 4: Light scattering airborne particle counter for clean spaces" [1] (GOST R ISO 21501-4 is an equivalent of the international ISO standard translated to Russian, so we use ISO 21501 title and designations in this paper) was first introduced in Russia in 2012 as a translation of the international standard ISO 21501-4 [2], however it attracted the attention of the community only six years later, after the adoption of the current version of the standard GOST R ISO 14644-1-2017 (Russian equivalent of international ISO 14644-1:2015) [3,4]. This standard, in turn, specifies the classification of air cleanliness in terms of concentration of airborne particles in cleanrooms and clean zones, and has a direct and only reference to the standard ISO 21501 in matters of calibration of light scattering airborne particle counters (LSAPC, or shortly APC) as the instruments used for monitoring of the airborne particles’ concentration. The ISO 21501 standard is not the first standard regulating the calibration of APC and is a further development of several national and international standards as well as generalization of the ideas embedded in them.

So, what issues of calibration of APCs are considered in this standard? The standard describes size calibration, conducting size setting error, determining the size resolution, counting efficiency, false count, sampling flow rate and time error, maximum allowable particle number concentration and response rate.

Imagine we are given a task to calibrate a particle counter, where should we start? First, before proceeding directly to the size calibration of the APC, it makes sense to conduct a preliminary check of the performance condition of its main components, check the condition of the device service parameters, such as laser or background voltage, etc., and if possible, compare the current values with the previous ones. Changes in these values beyond the limits specified by the manufacturer may indicate that service is required for the instrument. Secondly, instruments false count should be preliminary verified. The overwhelming majority of the modern particle counters have zero false count in five minutes as declared by manufacturers. Thus, a negative result of this test may indicate contamination of the optical camera of the APC, under which further calibration is meaningless until the contamination is eliminated. Finally, the sampling flow rate should be checked and adjusted if necessary. The deviation of the sampling flow rate by more or less than 5% directly affects the count of the number of particles and the resulting size distributions.

In accordance with the requirements of the standard, when carrying out size setting, it is necessary to use monodisperse spherical polystyrene latex particles (PSL) with a relative standard uncertainty equal to or less than 2.5% and use at least three particle sizes to plot the calibration curve. When
determining the size channel threshold values, the size setting error according to the calibration curve should not exceed 10%. In fact, this regulation requires to use calibration particles of sizes as close to the size channels of the particle counter under test as possible during size setting. To ensure the same principle, the number of suspensions of calibration particles for plotting a calibration curve will most often be equal to the number of size channels of the particle counter under test. It should be noted, that when obtaining pulse height distributions for the test aerosol the median voltage of the distribution is assigned to the certified average size of the test particles but not the maximum.

When performing size calibration, the size resolution is evaluated for one of the particle sizes lying above the minimum detectable particle size of the APC under test. It is in fact the ability of the particle counter to distinguish particles of different sizes. The size resolution (Fig. 1) should not exceed 15%, and is calculated from the standard deviation of the size distribution of the calibration particles of particular size observed by the particle counter. But what to do if the calculated size resolution value turns out to be close to the limit, or even exceeds it? Several reasons may lead to such result. First of all, it is necessary to check the suspension of the test aerosol and the parameters of its generation: an excessive concentration of test aerosol particles fed to the input of the counter can lead to a blurring of the distribution peak. Or vice versa, there may not be enough test particles in the suspension to form a distinctive peak. Among the reasons for the insufficient resolution of the obtained distribution of test particles, it is worth noting the contamination of the optical camera and its elements, as well as overtime, during the long-term operation, deviations in the alignment of optical elements, mainly of the light source, i.e., the laser diode, which inevitably lead to distortion of the entire optical system function (Fig. 2). In this case, the optical camera needs to be cleaned or the laser needs to be adjusted again, after which the calibration procedure starts from the beginning.

![Fig. 1. Determination of size resolution of the obtained distribution, where $V_M$ is the median voltage value, lower $V_L$ and upper $V_U$ voltage limits corresponding to the distribution density of 61%.](image)

The formula for calculating the resolution is given in clause 4.4. of the GOST R ISO 21501-4 standard [1] (7.3 in ISO 21501-4 [2]).

![Fig. 2. Examples of distributions obtained using a multichannel pulse height analyzer (PHA) for counters with good (a) and unsatisfactory (b) size resolution.](image)
After drawing a new calibration curve (Fig. 3), new voltage values of the threshold channels of the particle counter can be determined, and it is possible to proceed to the counting efficiency test. To determine the counting efficiency, a reference particle counter with a confirmed 100% efficiency is required for particles corresponding to the first channel of the counter under test. In other words, the reference particle counter must have a better sensitivity than the tested counter. The counting efficiency of the APC under test will be equal to the ratio of the particle concentration measured by unit under test to the one measured by the reference unit. The counting efficiency is determined by two sizes for calibration particles: the first one should be close to the minimum detectable particle size of the counter; the other size is selected 1.5 to 2 times larger than this value. In the first case, the efficiency of the counter under test should be (50 ± 20) %, and (100 ± 10) % in the second. In this case, the comparison of the values of the number concentrations obtained takes place for the first channel of the particle counter under test and for the channel of the reference counter corresponding to the particle of 1.5 times smaller size than the first size channel of the tested counter. This is since, thus, the particle distribution corresponding to the particle size of the first channel of the unit under test will lie entirely inside the size channel of the reference unit. In the second case, the comparison takes place for the first channel of the unit under test and an equivalent channel of the reference unit. Consequently, both values of the counting efficiency refer to the first size channel of the particle counter under test and determine the correctness of the voltage threshold value setting of the first size channel according to the obtained calibration curve, so that the distribution of particles equal to the size of the first channel of the APC fits in by half, and the distribution of particles 1.5 to 2 times larger fits in completely.

![Fig. 3. An example of plotting a calibration curve (the dependence of the amplitude A of the pulses in volts on the particle size D in microns) using a set of PSL calibration particles with diameters D.<sup>i</sup>](image)

As well as in the case of size resolution, if the obtained counting efficiency values do not fit within the specified limits, then, first, the concentration of test particles distributed to the input of both counters should be checked. It is necessary to ensure the stability and uniformity of the supply of the test aerosol. It is important to remember that the number concentration of particles in the test sample should not exceed 25% of the maximum permissible number concentration of both the counter under test and the reference instrument. At the same time, the number concentration value should be sufficient for the representativeness of the data obtained, and the results themselves should ensure repeatability. The sampling tubes connecting the distributing box, where the test aerosol is delivered, and both instruments should have the same length and the least amount of bends with the greatest possible curvature of these bends (with radius larger than 10 times the diameter of the tube), and the material of the tubes should ensure the least settling of the aerosol particles on the inner surface of the tube. In this case, the number
concentration in a volume delivered to the input of both counters will be as close as possible. The condition of the measuring camera and the correct alignment of the optical elements of the LSAPC under test can also play a significant role in the obtained result. A counter with a low resolution is highly likely to show unsatisfactory results for the counting efficiency.

At the end of the calibration test, a final check of the false (background) count of the instrument under test is carried out. The probability of a false count obeys the Poisson distribution, and the upper 95% confidence limit per 1 m$^3$ is indicated as the value of false count in calibration protocol. The ISO 21501-4 standard provides a table for the lower and upper confidence limits depending on the actual number of observed particles. Surprisingly, there is no explicit restriction in the standard that the actual count must be zero when sampling with an absolute filter at the input, which does not correspond to the requirements of previous standards, such as Japanese JIS B 9921 [4]. Also, the standard puts counters with different flow sampling rates in an unequal position. For example, if two LSAPCs with sampling flow rates equal to 28.3 (1 CFM) and 2.8 l/min, respectively, took samples for 15 minutes with an absolute filter installed at the input of each of them, and no particles were observed in the selected samples, this would mean that with a probability of 5% it is possible to observe 3 particles in each of the selected samples. But when normalized to 1 m$^3$, we obtain 7 particles for the counter with a sampling flow rate of 28.3 l/min and 71 particles for the counter with a sampling flow rate of 2.8 l/min. Unfortunately, since in practice the end users of APCs, as well as the audit that verifies their work, are usually far from the basics of statistical physics, then such results in the calibration protocol may raise questions or even put them in a dead end. Therefore, in practice, two lines can often be seen in the calibration protocol at the point where the false count is indicated: the actual zero count of the instrument per 5 minutes according to the JIS B 9921 standard and the calculated upper confidence limit according to the ISO 21501-4 standard.

At the end of calibration and all the tests, a protocol with all the obtained results is prepared. The LSAPC is calibrated in accordance with the GOST R ISO 21501-4 standard if a positive result was obtained during all the tests described above. An astute reader will probably pay attention to the fact that at the very beginning of this article we mentioned two more tests and didn’t talk about after: the maximum particle number concentration and the response rate. The maximum particle number concentration is provided by the manufacturer for the appropriate model of the instrument. The coincidence loss (i.e., the simultaneous presence of two or more particles in the measuring volume) according to the ISO 21501-4 standard should not exceed 10%. With an increase in the aerosol number concentration supplied to the input of the particle counter, the probability of several particles entering the measuring volume at the same time increases statistically. In this case, the counter will count them as one particle, but of a larger size. As the number of matches increases, the count and size distribution are distorted, in other words, the particle counter begins to "lie". During periodic calibration, this check is not confirmed due to the risk of contaminating the optical camera. The response rate is checked to assess the ability of the instrument to cleanse itself after getting a high, close to the maximum number concentration of particles. Exactly for this reason, due to the risk of contamination of the optical camera, this check is also not mandatory for periodic calibration.

Separately, as question about the counting error of particle counters is often raised up, it should be noted that the GOST R ISO 21501-4 standard does not define the counting error of the counter when setting particle sizes, does not describe a procedure for finding it and does not set acceptable limits. Therefore, this parameter cannot be found either in the particle counter specifications provided by its manufacturer, nor the primary calibration certificate, or in subsequent calibration certificates made in accordance with GOST R ISO 21501-4 or the international equivalent ISO 21501-4. The standard operates different terms of APC specifications (all mentioned above) that can be presented to the customer or operator of an APC.

And finally, good calibration results cannot be achieved without proper qualification of personnel and perfection of the equipment by means of which the calibration will be performed. For example, one of the particle counters manufacturers Lighthouse (USA) offers a PDS (Particle Delivery System) unit.
for LSAPC calibration and specially designed to match ISO 21501-4 standard. It combines units for size calibration and for evaluating the counting efficiency in a single package (Fig. 4). Vertical orientation of the channel connecting the particle generator and the instrument under test replaces the usual distributing box, provides better mixing of the test aerosol, and minimizes the loss of test particles of large (more than a micron) sizes due to their sedimentation. In addition, the residual effect of particles used in previous measurements on the current is minimized.

Fig. 4. Appearance of the PDS unit for calibration of LSAPCs.

Thus, in GOST R ISO 21501-4 standard, high skill of the personnel and the high technical level of the used measuring instruments have been established. So that, in order to achieve the best results, it is necessary to increase the personnel skill level and improve the characteristics of the equipment used in the calibration, as well as use advanced measuring stands and more precision measuring instruments. At the same time, there are some aspects of this standard needed to be made clear for both who use the standard for calibration and for the users of particle counters calibrated in accordance with this standard. Yet, this matter should be solved during the public discussion.

References
[1] GOST R ISO 21501-4 2012 Determination of particle size distribution — Single particle light interaction methods — Part 4 Light scattering airborne particle counter for clean spaces
[2] ISO 21501-4 2018 Determination of particle size distribution — Single particle light interaction methods — Part 4 Light scattering airborne particle counter for clean spaces
[3] GOST R ISO 14644-1 2017 Cleanrooms and associated controlled environments — Part 1 Classification of air cleanliness by particle concentration
[4] ISO 14644-1 2015 Cleanrooms and associated controlled environments — Part 1 Classification of air cleanliness by particle concentration
[5] JSA - JIS B 9921:2010 Light scattering airborne particle counter for clean spaces