TECH TALK

ISO/TC 209 Outreach Series: ISO 14644-12

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Monitoring of Nanoscale Particles in Cleanrooms: ISO 14644-12

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The *Journal of the IEST* presents the second in a series of outreach articles by ISO/TC 209 leaders to foster promotion and education of the expanding body of ISO/TC 209 Standards

Keywords

ISO, TC 209, 14644, cleanrooms, standards, nanoscale particles, ultrafine particles, nanoparticles, processes, monitoring

Abstract

Within the International Organization for Standardization (ISO), Technical Committee (TC) 209 is chartered with standardization of cleanrooms and associated controlled environments. A series of 15 international standards (thirteen parts under ISO 14644 and ISO 14698 Parts 1–2) has been established for controlling contamination by means of cleanroom technology. The standards address design, classification, and monitoring, and support operation of cleanrooms.

The committee's most recently published standard, ISO 14644-12, *Cleanrooms and associated controlled environments—Part 12: Specifications for monitoring air cleanliness by nanoscale particle concentration*, provides specifications for the cleanroom monitoring of nanoscale particles (nanoparticles) smaller than 100 nm with a condensation particle counter or equivalent. The standard provides guidance for air monitoring for the purpose of identifying the contributions of sources to the cleanroom burden of particles.

Sources of nanoscale particles are primarily from the processes in the cleanroom. The standard includes example specifications for instrumentation performance and information on how to apply the data. Intended users include process engineers and cleanroom specialists. It is anticipated that as the nanotechnology field advances, this standard may find extensive use.

Introduction

International standards facilitate global trade by providing a common basis of communicating specifications in purchase transactions. The responsibility for cleanroom standardization within the International Organization for Standardization (ISO) is held by Technical Committee (TC) 209, Cleanrooms and associated controlled environments. In 1992, United States ISO Member ANSI proposed the formation of the technical committee to ISO at the recommendation of IEST and delegated the responsibility for the administration to IEST. ISO/TC 209 currently publishes standards as parts of the ISO 14644 and 14698 series. These standards are available from IEST in the United States and from ISO member bodies globally.

The objective of this article is to foster understanding regarding a newly available ISO 14644 standard developed by ISO/TC 209 Working Group (WG) 10 Nanotechnology. ISO 14644-12[1], *Cleanrooms and associated controlled environments—Part 12: Specifications for monitoring air cleanliness by nanoscale particle concentrations* provides specifications for the monitoring of airborne particles smaller than 100 nm with a condensation particle counter (CPC) or equivalent. Although nanotechnology standardization in ISO has generally fallen under ISO/TC 229, Nanotechnologies, much of the production involved in nanotechnology requires the use of cleanrooms.

The first international consensus cleanroom standard was developed in 1999 as ISO 14644-1:1999,[2] *Cleanrooms and associated controlled environments—Part 1: Classification of air cleanliness*. For classification purposes, the smallest particle size was 0.1 µm, but the standard also included sections on "ultrafine" particles of less than 0.1 µm. During the development of the second edition of 14644-1[3] in 2015, the sections on ultrafine particles were removed and earmarked for transfer into a pending document (ISO 14644-12) on nanotechnology. (The terms "ultrafine" from the early standards have evolved into the generally accepted "nanoparticle and "nanoscale particles." Nanoparticle is defined by size and a spherical shape. Nanoscale particle is a more general term and may refer to an equivalent diameter determined by a particle counting instrument.)

The initial drafts of the nanotechnology standard extended the cleanroom classification tables from ISO 14644-1:1999 below the 0.1 µm threshold. However, it was felt that this had the potential to create unrealistic technical conditions and confusion in the industry. The draft classification tables were removed when it became clear that current understanding of nanoparticles in cleanrooms limited the scope of ISO 14644-12 to providing guidance on monitoring—rather than classification—of cleanrooms. For example, nanoscale airborne particles appear to be generated mainly by intermittent emissions from process tools in semiconductor cleanrooms. At the present time, open literature is not available from other industries. Monitoring includes obtaining time-dependent concentration trends of nanoscale particles and developing monitoring goals for troubleshooting of the processes in the cleanroom.

Background of nanoscale particle measurement

The measurement of nanoscale airborne particles dates back to 1875. A French investigator, Coulier, performed experiments with water supersaturation of air to simulate the formation of clouds and found that particles in the atmosphere promoted the formation of cloud droplets. Over the years, a number of different concepts were employed to detect particles by creating supersaturated atmospheres including adiabatic expansion or continuous flow vapor condensation. The early investigators termed the particles "nuclei" because the particles had the chemical properties to serve as nuclei or sites for water condensation. Modern instruments have the capability to detect particles regardless of the chemical composition. Currently, condensation particle counting is an established measurement method for nanoscale particles.[4] One of the first applications of CPCs to cleanrooms is described in Ensor and Donovan.[5]

Particle size distribution data encompassing nanoscale particles is very limited in current cleanroom literature. The absence is due to the very low concentrations and that measurements are normally conducted to establish compliance with the classification levels in ISO 14644-1:2015. Ensor et al.[6] reported the measurement of particle size distributions in a variety of cleanrooms in various operational states.

Sem[7] analyzed cleanroom nanoscale airborne particle behavior based on established aerosol dynamics found in Hinds[8] and data shared by Texas Instruments, Inc. Based on the well-established tri-modal size distribution found in the ambient atmosphere, Sem suggested similar airborne particle size distributions' behavior would be mirrored in cleanrooms. To summarize, the behavior of aerosols in the nanoscale mode is formed from reactions or condensation (less than 0.1 µm); the accumulation mode is formed by coagulation of particles from the nanoscale mode or direct industrial emissions and is shaped by larger particle deposition between 0.1–1 µm; and coarse mode greater than 1 µm is formed from suspended dust. The main difference between the ambient atmosphere and cleanrooms is that the particle concentration in the 0.1–1 µm decade would be shaped by penetration of ambient particles at the most penetrating particle size through the filters. The industrial semiconductor cleanroom nanoscale particle concentration was observed to occur in "bursts." This phenomenon was interpreted to indicate small point sources of particles or particle precursors were being emitted from processing tools and were detected when the plume passed the sampling inlet of the instrument.

Ensor et al.[9] reported measurements in an ISO Class 5 cleanroom from 0.05–5 µm using a parallel array of two CPCs with inlet diffusion batteries and two optical particle counters. The reason for using a parallel array of instruments was to average the bursts of nanoscale particles. The characteristic "at rest" state (during the night) curve followed the predicted earlier by Ensor et al.[6] where very few nanoscale particles were observed. However, when the cleanroom was in an "operational" state (during the day when the processes were operating) the shape and slope of the curve approached ISO 14644-1:1999 classes from nanoscale emissions from the process and operating personnel.

Ahonen el al.[10] reported the measurement of nanoscale particles in a contemporary semiconductor cleanroom using advanced condensation particle detection equipment with a size cutoff of near 1 nm. The study found sub 2 nm particles formed from the condensation of vapor from processing tools (atomic layer deposition [ALD], Indium Tin Oxide [ITO]-sputtering, lithography) tend to rapidly coagulate into larger particles. The emissions from processing tools appeared in bursts similar to the phenomena (probably coagulated nanoparticles) reported by Sem many years earlier. Some of these bursts from the ALD exceeded $>10^5$ cm^{-3} (10^{11} m^{-3}). However, between the bursts, the particles of 1.4 nm were very low—less than 10 cm^{-3}. In the 1.1–1.4 nm particle size range, a constant concentration between 200–700 cm^{-3} was measured. The study found the concentration did not appear to be related to the process in the cleanroom or have time-dependent properties but may have been due to particle or cluster formation from radiation such as cosmic rays or from the earth.[10]

One additional explanation for the high concentration of 1.1–1.4 nm particles might be thermal rebound from fibers within the high efficiency ventilation filters. It was hypothesized by Wang and Kasper[11] that particles below a very small particle size (thought to be in the 1 nm diameter range) have sufficient velocity from Brownian motion to bounce and not stick to fiber surfaces.

Givehchi and Tan[12] reviewed the thermal rebound literature (over 20 investigators had failed to detect the phenomena) and advanced a new theory identifying relative humidity as an unrecognized factor in explaining some cases with positive experimental results. In a recent paper, Givehchi et al.[13] found evidence of thermal rebound of particles smaller than 1.17 nm at low relative humidity from thin electrospun polymer fiber filters. Therefore, it is possible that thermal rebound may exist in the filtration systems under the environmentally controlled conditions found in cleanrooms.

As the nanotechnology field advances, additional research may be undertaken to more fully understand nanoscale airborne particle behavior in the cleanroom environment. Standardized monitoring data gathered through broader use of ISO 14644-12 may also lead to additional observations that merit further investigation.

How ISO 14644-12 fits into the ISO 14644 family of standards

Using the concept presented earlier that airborne particle size regions have distinctly different physical behavior from sources of particles and removal mechanisms, a comparison table (Table 1) can be generalized.

Table 1. Comparison of the application of ISO 14644 standards relating to particle size range

ISO/TC 209 Standard (under the title "Cleanrooms and associated controlled environments")	Size Range	Particle Mechanisms	Application
ISO 14644-8:2013[14] **Part 8: Classification of air cleanliness by chemical concentration (ACC)** **(Note: In the future "classification" will be reserved for ISO 14644-1.)**	Molecular Smaller than 0.001 µm (1 nm)	Sources: Off-gassing from materials and deposited organic test aerosols; atmospheric contaminants entering through filters. Mechanisms: Condensable and reactive precursors for nanoparticles and deposition on surfaces.	Attributes used in design of facilities and monitoring
ISO 14644-12:2018 **Part 12: Specifications for monitoring air cleanliness by nanoscale particle concentration**	Nano scale 0.001 µm to 0.1 µm (1 nm to 100 nm)	Sources: Emissions from equipment, corona discharge or radiation induced clusters, filter leaks or possibly thermal rebound from filter fibers. Mechanisms: Brownian motion drives movement and coagulation.	Monitoring
ISO 14644-1:2015 **Part 1: Classification of air cleanliness by particle concentration**	Micro scale 0.1 µm to 5 µm	Sources: Emissions from processes and personnel, and resuspension from surfaces. Leaks and penetration of filters. Mechanism: Movement by air convection.	Classification
ISO 14644-2:2015[15] **Part 2: Monitoring to provide evidence of cleanroom performance related to air cleanliness by particle concentration**	Micro scale 0.1 µm to 5 µm	Companion document to ISO 14644-1.	Monitoring Contains specifications for a risk-based program
ISO 14644-17[16] **Part 17: Specification of requirements for particle deposition monitoring [Currently under development.]**	Macro scale > 5 µm	Sources: Resuspension from surfaces and emissions from personnel. Mechanisms: Movement dominated by gravity; air convection.	Monitoring
ISO 14644-14:2016[17] **Part 14: Assessment of suitability for use of equipment by airborne particle concentration**	Uses ISO 14644-1 to determine suitability of equipment for specified ISO classes	In the future, principles in ISO 14644-14 might be extended to other particle size ranges, if required by application, e.g. ISO 14644-12.	Suitability

Targeted users of ISO 14644-12

Process Engineering

ISO 14644-12 is intended to support nanotechnology research, development, and manufacturing. One example of a growth area of nanotechnology may be the semiconductor industry, due to shrinking feature sizes over the past decades. Monitoring requirements may be in the nanoscale region. A recent presentation[18] suggested that applications of nanoscale particle monitoring might include:

- Monitoring nanoscale particles from processing tools.
- Monitoring cleanrooms to identify problem areas.
- Monitoring ultrapure inert gases with a pressure reduction device on the inlet.
- Detecting nanoscale particles below the critical flying height in hard disk drives.

One potential reason for monitoring process tools is to provide an independent indication of the process such as the integrity of the process vessel. In addition, there is a possibility of cross-contamination by nanoscale particles between processes within the cleanroom. Remiarz[19] described a CPC designed with low background for cleanrooms and with a lower particle cut-off of 10 nm using the water-based laminar flow concept invented by Hering et al.[20] As reported by Ahonen et al., semiconductor processing tools including chemical and thermal process may be greater sources of nanoscale particles than previously suspected.

Monitoring and surveys for industrial hygiene purposes

Airborne nanoparticles are receiving increased attention as a health concern in nanotechnology manufacturing environments including cleanrooms. Particle counting instruments are being used because traditional filter sampling methods have insufficient sensitivity and time resolution. Shepard and Brenner[21] described the use of a CPC and other instruments in semiconductor wafer polishing areas within a cleanroom. Ahonen et al. considered whether process emissions from processing tools were potential hazards but was inconclusive.

Another application for ISO 14644-12 could be monitoring of nanoparticles released from drug compounds in cleanrooms and isolators (separative devices).

As reported by Dutton:[22]

> According to Dr. Steven Oldenburg, “Nanoparticles are enablers”. “It is not the size that sets them apart but how their properties change at the nanoscale that makes them useful”.

Nanoparticles are being used as active ingredients and carriers in a wide range of newly formulated therapeutics in various stages of introduction to the market place. The use of high-potency active pharmaceutical ingredients (HPAPI) is increasing as companies develop more effective and better targeted medicines.[23] Monitoring for HPAPI nanoparticles may become a critical parameter in many areas of the operations, including the cleaning of an isolator after operations. Some of these therapeutics might be emitted as nanoscale particles during manufacturing. Data could be generated during validation to determine the recovery of the isolator and the safety margin for opening these units after operations. ISO 14644-12 could benefit this industry in the monitoring effort.

Content of ISO 14644-12

For the purposes of monitoring, airborne nanoscale particle counting can be carried out most effectively by CPC. The reference test method for CPC monitoring is given in Annex A of ISO 14644-12. Table A.1 in the Standard contains an example instrumentation specification. The justification for including only example specifications is that instrumental requirements such as the nanoscale particle size and detection limits may evolve as process monitoring needs change. The method for performing the monitoring of air cleanliness by nanoscale particle concentration should provide a systematic plan, well-defined procedure, and identify how the assessment should be performed. In general, the establishment of alert and action limits is based on a risk assessment. (ISO 14644-2 is suggested for an example of monitoring plan guidelines.) If there is a requirement by buyer-seller agreement, the limits may be established per agreement.

Criteria for determining the counting method will include:

- nanoscale particle size to be measured;
- time dependence of sampling and analysis;
- sample volume;
- location of sampling;
- number of samples;
- criticality of process/product;
- design/layout of clean zone.

Alternative methods and/or instrumentation, with documented evidence of having at least comparable performance to CPC measurement may be specified. If no alternative is specified or agreed upon, the reference method shall be used.

The nanoscale particle size to be measured is critical to the specification. The CPC measures the cumulative concentrations above the 50% cutoff particle size (sometimes called the size resolution). The 50% cutoff particle diameter and the shape of the cutoff curve will determine the size dependent response of the instrument. Typically, the cutoff curve is “S” shaped. Annex B of ISO 14644-12 contains information on the minimum sharpness of the cutoff curve.

Tests performed to demonstrate compliance to the standard shall be conducted using calibrated instruments. Reporting requires complete documentation of the characteristic of the instrument such as cutoff and zero counts, description of the sampling plan, sampling results and any other information required by the buyer of the information.

Summary

ISO 14644-12 includes requirements for nanoscale airborne particles in a standalone document. Nanoscale particles within cleanrooms have limited references in open literature because the traditional focus in cleanrooms has been on microscale particles.

Major sources of nanoscale particles in an operating semiconductor cleanroom are emissions from process tools, often in short-term bursts. ISO 14644-12 supports process monitoring and provides instrumentation guidance for industrial hygiene studies for airborne nanoparticle measurement in cleanrooms.

Specifically, ISO 14644-12 provides:

- Consensus of application of CPCs and other instruments in the airborne nanoscale particle range.
- Potential for wide-spread use as instrumentation technology improves.
- An example instrument specification intended as the impetus to start further dialogue on the requirements for instruments for cleanroom use.
- Support of the historical trend for the dimensions of features of manufacturing to be reduced. Nanotechnology is the most recent expression of that trend. Nanotechnology-enabled products will likely be manufactured in cleanrooms, possibly in reactors or processes.

About ISO/TC 209

The use of cleanrooms and associated controlled environments is becoming more and more common and a key enabling technology for production. In response, ISO/TC 209 working groups (WGs) have contributed standards for design, testing and use of cleanrooms and associated controlled environments to aid in the acceptance of this beneficial technology by different user groups and regions.

There are currently 24 participating member (P members) countries, which are eligible to nominate experts for WGs and vote on standards in development or systematic review. There are currently 21 countries (O members) that can observe the work of ISO/TC 209.

ISO/TC 209 standards are written generically in that they can be applied for testing and monitoring, or in a broader sense to control cleanliness in various industries such as

- automotive,
- aerospace,
- electronics,
- semiconductors,
- food,
- life sciences (e.g. pharmaceuticals, health care, hospitals),
- scientific research.

In addition, industry or national standards and guidelines are sometimes used to provide deviating or more specific requirements and aspects.

ISO/TC 209 has established formal liaisons with five other ISO TCs and the International Confederation of Contamination Control Societies (ICCCS) to ensure transparency and consistency in its standardization efforts. In 2017, ISO/TC 209 revised its business plan and scope to capture and address current and future standardization needs of consumers, regulators, and industry regarding cleanrooms. The revised scope reflects technical progress and the recognition that cleanroom technology has become more widely applied in various industries and the applications have become more diverse.

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IEST is the leading global nonprofit contamination control society and Secretariat for ISO Technical Committee 209 (ISO/TC 209), the committee developing the ISO 14644 Standards. IEST has served as the Secretariat for ISO/TC 209 for more than 25 years with an established international leadership role based on more than 45 years of expertise in cleanrooms and controlled environments.