New standards for assessing the suitability of equipment and materials for use in cleanrooms: ISO 14644 Parts 14 and 15

Isabelle Tovena-Pecault¹, Delphine Faye²

Abstract
One of the main difficulties facing a cleanroom manager is whether or not to accept a particular material or piece of equipment in the cleanroom without risk to production. In the absence of any international standard, different industries, such as space and lasers, have chosen their own specifications. This paper gives an overview of two international standards, currently in the latter stages of development, for assessing the suitability of equipment and materials for use in cleanrooms. ISO 14644-14 and ISO 14644-15 will consider particulate and chemical contamination, respectively.

Keywords: cleanroom, material, equipment, suitability, ISO 14644-14, 14644-15

Introduction
Materials suitable for ISO 5° – who amongst us has never come across these words in a technical specification sheet or cleanroom product catalogue? Yet, until now, there have been no universal standards that have allowed us to define what makes a material or piece of equipment ‘cleanroom suitable’. Furthermore, cleanroom suitability is a notion which varies significantly from sector to sector – microelectronics, space engineering, automobile engineering, optics etc. – hence our proposal, in 2011, to establish a new classification system for assessing the suitability of equipment and materials for use in cleanrooms. This proposal was accepted by ISO TC (Technical Committee) 209 on behalf of the international community in July 2012. The aim of these new standards will be to set out specifications and guidelines for assessing the suitability of equipment and/or materials for use in cleanrooms and associated controlled environments, focusing on air and surface particle and chemical cleanliness. The standards will provide guidelines for measuring particle and chemical emission rates with reference to ISO 14644-1 and ISO 14644-8 respectively.

The first proposed standard (ISO 14644-14) will be concerned with measuring the suitability of equipment for use in cleanroom environments with regard to particle emissions. The FDIS (final draft international standard) is currently being prepared for formal approval, i.e. voting for progression to full standard. The second standard (ISO 14644-15) will be concerned with measuring the suitability of equipment and materials in terms of their chemical emissions. This standard is at a slightly earlier stage – the DIS (draft international standard) is being voted on for progression to FDIS. Both are being developed by ISO TC 209 Working Group 11.

Before going into further detail regarding the content of these standards, we will begin with a review of some of the risks, which particles and chemicals may pose for the environment and for products that are handled or fabricated in cleanrooms, by looking at two practical examples. We will then set out definitions of certain terms which are essential when discussing the suitability of equipment and materials for use in cleanrooms and associated controlled environments. Finally, the two new standards are briefly described.

Background and two examples of methodologies currently in application
The criteria for surface contamination (by particles or chemical compounds) may be very different in different sectors of activity, with a wide range of possible specifications.

Example 1: The space industry
Special efforts are required to minimise contamination, in order to avoid compromising the performance of satellites and on-board instruments throughout the entire duration of a mission. In order to meet the requirements of space equipment standards in environments which are sensitive to contamination, chemical and particle concentration specifications need to be established and monitored, with the implementation of a dedicated cleanliness strategy for each instrument as well as the satellite as a whole in the final assembly phase.

Risk prevention at every stage of the development process requires certain basic precautions, not only in the design and fabrication phases, but also and especially during assembly and testing in ISO 8 cleanrooms, or even ISO 5 if the sensitive surfaces of the equipment are not protected. Potential sources of contamination are many and varied and, as such, these basic precautions cover both personnel and all types of material present in the cleanroom. Furthermore, special care is taken to use only consumables which generate no (or next to no) contaminants. To this end, a list of consumables or construction materials judged to be unsuitable for use in the cleanroom for reasons of cleanliness, or for the health and safety of personnel, is generally issued to all involved. This list can either be general or, for certain projects, specific.

Until recently, little had been done to quantify emissions from process equipment, this generally being kept...
isolated to separate it from sensitive products. Greater priority has been afforded to analysing emissions of chemical contaminants by materials. Here, applying a methodology similar to that used to certify materials for use in space, (in a thermal vacuum chamber), tests are conducted in the laboratory to determine the mass lost by all new materials (primarily during the construction period) left at room temperature, at 40°C or even at 60°C, for between 48 and 72 hours. The results are then measured against acceptance criteria such as TML ≤ 1.0% and CVCM ≤ 0.1%, or else RML and CVCM ≤ 0.1% where:

- TML = total mass lost by the material in %
- RML = relative mass lost in % after rehydration of the material
- CVCM = mass (as a %) of collected volatile condensable material at 25°C

However, unlike the methodology used to certify materials for use in space, standardized in the ECSS (European Cooperation for Space Standardization) format, the methodology used to select cleanroom materials is not really standardized, and its criteria are not directly comparable with the cleanliness levels specified in ISO 14644-8 and ISO-10 (D. Faye, J. Mary, 2008).

The time was therefore considered ripe for the development and implementation of standards to establish the suitability of materials and equipment by measuring particle and chemical emissions in the air, and the corresponding surface contamination levels resulting from deposition or condensation.

### Example 2: High-power lasers such as the Megajoule Laser (LMJ)

High-power lasers are physical instruments which are immensely sensitive to the cleanliness of the environments in which they are located, in terms of both particle and chemical concentrations (I. Tovena Pecault, 2014). It has already been demonstrated that the cleanliness of high-power lasers is primarily a question of the surface particle concentrations on the laser’s optical-mechanical components, along with the degassing of the organic materials used in the laser (G. Guéhenneux et al., 2006).

With regard to chemical contamination, numerous studies allowed us to ascertain that the chemical nature of the molecules generated is at least as important as the total quantity of material degassed. This led us to put forward our own materials classification test for the LMJ laser (see I. Tovena, 2008). This involved testing the materials in an ultra-clean test chamber, in temperature conditions comparable to those in which they will be used, i.e. between 30 and 50°C. The gases emitted by the material are then analysed by mass spectrometer, allowing us to identify the chemical profile of each molecule (I. Tovena et al., 2014).

Over the past 12 years we have built up our own database listing all materials deemed acceptable for use in the LMJ, based on the ‘degassing’ criteria. A standardized test at international level would allow us to build up a more wide-ranging standardised database of ultra-clean materials, allowing us to limit contamination risks due to changes in material composition and/or fabrication process. This would be a considerable asset for the high-power laser sector and all industrial sectors where chemical contamination presents a serious risk.

### Approach

Nowadays, as the definitions for clean environments have been updated to take all sorts of contaminants into account, a controlled environment or cleanroom is no longer defined solely in terms of its airborne particle concentration (as per ISO 14644-1) or airborne chemical concentration (as per ISO 14644-8). Measurements of surface particle cleanliness (SPC as per ISO 14644-9) and surface chemical cleanliness (SCC as per ISO 14644-10) may also be incorporated.

It is therefore only logical that efforts to measure the suitability of materials and equipment for use in cleanrooms should also take these different aspects and classifications of cleanliness into account. Table 1 shows how the new standards relate to existing standards, with ISO 14644-14 focusing on air cleanliness by particle concentration for equipment and ISO 14644-15 focussing on air cleanliness by chemical concentration for materials and equipment.

A few definitions are required for the purposes of the new standards:

- Emission: contaminants that are discharged into the environment (ISO 14644-15)
- Emission rate: rate describing the mass of one or more volatile chemical(s) emitted from equipment or material per unit time (ISO 14644-15)

### Table 1: Relationship between chemical and particle cleanliness classification standards and the new assessment standards

<table>
<thead>
<tr>
<th>Contamination type</th>
<th>Category</th>
<th>Subject of evaluation</th>
<th>Applicable assessment standard</th>
<th>Classification standard</th>
<th>Designation</th>
</tr>
</thead>
<tbody>
<tr>
<td>particle</td>
<td>air</td>
<td>equipment</td>
<td>14644-15</td>
<td>14644-1</td>
<td>ISO N</td>
</tr>
<tr>
<td>particle</td>
<td>air</td>
<td>materials</td>
<td>- a</td>
<td>14644-1</td>
<td>ISO N</td>
</tr>
<tr>
<td>particle</td>
<td>surface</td>
<td>equipment</td>
<td>- a</td>
<td>14644-9</td>
<td>ISO-SCP</td>
</tr>
<tr>
<td>particle</td>
<td>surface</td>
<td>materials</td>
<td>- a</td>
<td>14644-9</td>
<td>ISO-SCP</td>
</tr>
<tr>
<td>chemical</td>
<td>air</td>
<td>equipment</td>
<td>14644-15</td>
<td>14644-8</td>
<td>ISO-ACC</td>
</tr>
<tr>
<td>chemical</td>
<td>air</td>
<td>materials</td>
<td>14644-15</td>
<td>14644-8</td>
<td>ISO-ACC</td>
</tr>
<tr>
<td>chemical</td>
<td>surface</td>
<td>equipment</td>
<td>- a</td>
<td>14644-10</td>
<td>ISO-SCC</td>
</tr>
<tr>
<td>chemical</td>
<td>surface</td>
<td>materials</td>
<td>- a</td>
<td>14644-10</td>
<td>ISO-SCC</td>
</tr>
</tbody>
</table>

* There may be applicable assessment standards for these categories in the future.
• Chemical emission rate: rate corresponding to the mass of one or more volatile chemical compounds emitted by a product over a fixed period of time
• Particle emission rate: rate corresponding to the number of particles emitted by a product over a fixed period of time
• Equipment: system designed for specific function(s), integrating materials, components and/or controls (ISO 14644-15)
• Material: single substance or composite (ISO 14644-15)

ISO 14644:14

Objective
ISO 14644 – Part 14, Assessment of the cleanroom suitability of equipment based on airborne particle concentration, will set out a methodology which allows us to assess the suitability of equipment (e.g.: control and processing equipment, tools etc.) for use in cleanrooms and associated controlled environments that are classified in accordance with ISO 14644-1. Classification of air cleanliness by particle concentration. It should be remembered that the ISO 14644-1 classification covers particles of between 0.1 and 5 μm in size.

Among other things, Part 14 will not address the following items:
• Assessing compatibility in terms of biocontamination
• Compatibility of decontamination agents and techniques
• Cleanability of materials and equipment
• Requirements relating to the design of equipment and the selection of materials
• Physical properties of materials (e.g.: electrostatic and thermal properties)
• Selection and use of statistical methods for testing purposes

Method for assessing the suitability of equipment based on particle emissions

General observations:
• The standardized method consists of measuring the airborne particle contamination at critical points at or in immediate proximity to areas of high particle concentration (HPC).
• As the distribution of particle emissions cannot be known in advance, it is advisable to select three widely spread particle size ranges.
• The particle concentrations thus measured at critical points on the equipment are then compared with the respective classification limits specified in ISO 14644-1.
• Among the additional, optional tests available, it may also be useful to test the equipment at different phases in its operational life cycle, and to measure the quantity of sediment or total particle emissions in the air.

Assessment of the testing environment
In order to assess the suitability of an item of equipment, we must begin by clearly defining the appropriate testing environment. In this context, it has been agreed that the testing environment must be at least one ISO cleanliness class superior to the cleanroom or environment in which the equipment is intended to be installed. Information on testing means and methods are given in ISO 14644-1 and ISO 14644-3.

The following parameters will need to be measured in advance:
• the concentration of particles in suspension in the air
• the airflow velocity (guidance range for vertical velocity is 0.3 m/s – 0.5 m/s)
• temperature (guidance range is 18°C – 25°C)
• atmospheric humidity (guidance range is 30% – 70% relative humidity)
  Further tests may also be performed for information purposes (e.g. visual representation of airflows, electrostatic testing, particle deposition tests etc.).

Precise identification of HPC locations
After an initial estimate, the precise position(s) of HPC locations can be determined using a discrete particle counter. During this measurement phase, the geometric positioning of the counter’s measuring probe must be adjusted to achieve optimal sampling of the HPC locations.

The positioning of the probe must be recorded. The operation should be repeated for each of the previously-identified HPC locations.

Processing the results
The data processing method described hereunder should be applied to each HPC location identified. The procedure must cover the whole of the specified particle size range.

A statistical distribution model for airborne particles can then be calculated in order to obtain meaningful results based on the on the number of discrete particles detected.

A distinction is made between two different types of distribution:

a) Student’s t test
This procedure will be applied for measurements where the number counted (mean value) for the particle size under consideration is assumed to be larger than 10 particles per measurement:

The mean value, standard deviation and upper confidence limit are calculated using equations 1, 2 and 3.

Equation 1
\[ \bar{x} = \frac{x_1 + x_2 + \ldots + x_n}{n} \]
Where:
\( \bar{x} \) is the mean value
\( x_i \) is a single reading in the consecutive measuring of particle size range i
\( n \) is the number of readings

Equation 2
\[ S = \sqrt{\frac{\sum (x_i - \bar{x})^2}{n-1}} \]
Where:
\( S \) is the standard deviation
\( x_i \) is a single reading in the consecutive measuring of particle size range i
\( \bar{x} \) is the mean value
\( n \) is the number of readings

Equation 3
\[ P_u = \bar{x} + 1.66 \times \frac{S}{\sqrt{n}} \]
Where:
\( P_u \) is the upper confidence limit for a confidence level of \( (1 - \alpha) = 95\% \)
\( \bar{x} \) is the mean value according to Equation 1
\( S \) is the standard deviation according to Equation 2
1.66 is the factor from Student’s t distribution
\( n \) is the number of readings
A detailed example will be given in ISO 14644:14

b) Poisson distribution
This procedure will be applied for measurements where the number counted (mean value) for the particle size under consideration is assumed to be a maximum of 10 particles per measurement.

The mean value is again calculated according to Equation 1. A table for upper confidence limit (based on the Poisson distribution for 95% confidence limit) shows the upper confidence level for mean values $X$ between 0 and 10.

As with procedure a), an example will be given in ISO 14644-14

Establishing cleanroom suitability
The suitability of equipment for use in cleanrooms must be declared on the basis of an assessment conducted using the method set out above, combined with a visual inspection.

Example of declaration: Equipment Z is suitable for use in cleanrooms of ISO X (Y μm) standard, where
- $X$ is the number of the ISO category, rounded up to the nearest whole number (as per 14644-1)
- $Y$ is the range of particle sizes measured
- $Z$ is the designation and unique identification information of the equipment (e.g. type, serial number, manufacturer)

ISO 14644:15

Objective
ISO 14644-15, Assessment of suitability of equipment and materials for cleanrooms by airborne chemical concentration, will include requirements and recommendations for assessing the cleanroom compatibility of equipment and materials on the basis of their chemical emissions, as per ISO 14644-8. Although ISO 14644-15 is at an earlier stage of drafting than ISO 14644-14, and thus not yet definitive, we can still give some indication as to its likely content.

Test configurations
Test configurations may be adapted to suit the size of the object to be tested and/or the pre-estimated emission rate of chemical contaminants.

Calculating the specific emission rate
To begin with, the specific emission rate is calculated as the normalized mass flow of chemical contaminants emitted by a test subject (material or equipment) and any background noise present in the test environment is subtracted. This ensures that the emission rate is derived purely from the mass of chemical contaminants emitted into the test environment. Closed and open test designs are described.

The specific emission rate for a closed design test is calculated according to Equation 4.

$$q_o = \frac{m_o - m_b}{X \cdot t_o}$$

Where
- $q_o$ is the specific emission rate of the test object in g/m²s or g/s/unit
- $m_o$ is the total sampled mass from the test environment with the test object in g
- $m_b$ is the total sampled mass from the test environment without the test object in g
- $t_o$ is the sampling duration time in s
- $X$ is the specific metric of the test object or unit

The specific emission rate is always stated with the chemical or chemical group for which it has been assessed.

A corresponding formula is given for an open test design.

The chemical mass concentration in a cleanroom can be calculated using

$$p_c = \frac{p_m \cdot n_r \sqrt{\frac{V_o}{V_c}}}{n_m + n_r \cdot \alpha}$$

Where
- $q_o$ is the specific emission rate of all considered objects (material or equipment) in g/(m² s) or g/(unit s)
- $p_c$ is the chemical mass concentration in the cleanroom in g/m³
- $p_m$ is the chemical mass concentration of the make-up air in g/m³
- $n_m$ is the make-up air change rate in 1/s
- $n_r$ is the recirculated air change rate in 1/s
- $V_o$ is the volume of air in the cleanroom in m³
- $x$ is the quantity related to the assessed object in m² or unit
- $\alpha$ is the efficiency of the chemical filtration system (if there is no filtration then $\alpha = 0$)

If the concentration is within the limits for the respective ISO ACC class number (ISO 14644-10) then the materials or equipment tested are suitable for use in that cleanroom.

Conclusions
The ISO 14644-14 standard will set out a methodology for assessing the suitability of equipment (e.g. control and processing equipment, tools etc.) for use in cleanrooms with regard to airborne particle contamination. The standardized method consists of measuring the airborne particle contamination at critical points at or in the immediate proximity of areas of high particle concentration (HPC) in accordance with ISO 14644-1 (airborne particle cleanliness for particle sizes between 0.1 and 5 μm).

The ISO 14644-15 standard will set out a methodology for assessing the suitability of equipment and materials for use in cleanrooms based on airborne chemical contamination. It will include requirements and recommendations for assessing the cleanroom compatibility of equipment and materials on the basis of their chemical emissions, as per ISO 14644-8.

These two standards will help to clarify which materials and items of equipment can be considered ‘cleanroom compatible’, in relation to users’ required levels of particle and chemical contamination, and establish testing conditions for materials and equipment which are representative of the conditions in which they will be used.

References
1. ISO 14644-1:2015, Cleanrooms and associated controlled environments
   - Part 1: Classification of air cleanliness by particle concentration
2. ISO 14644-3:2005, Cleanrooms and associated controlled environments
   - Part 3: Test Methods
3. ISO 14644-8:2013, Cleanrooms and associated controlled environments
   - Part 8: Classification of air cleanliness by chemical concentration (ACC)
   - Part 9: Classification of surface cleanliness by particle concentration
5. ISO 14644-10:2013, Cleanrooms and associated controlled environments
   - Part 10: Classification of surface cleanliness by chemical concentration
6. ISO/FDIS 14644-14, Cleanrooms and associated controlled environments - Part 14: Assessment of suitability for use of equipment by airborne particle concentration

7. ISO/DIS 14644-15, Cleanrooms and associated controlled environments - Part 15: Assessment of suitability for use of equipment and materials by airborne chemical concentration


9. ISO 2889 : 2010 : Échantillonnage des substances radioactives contenues dans l’air dans les conduits et émissaires de rejet des installations nucléaires


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