The first equipment for protection from nanoparticles

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Abstract. How can we guarantee the containment of ultrafine particles but also implement the ergonomic and handling constraints voiced by researchers? This is the equation that the engineers at FAURE INGENIERIE had to resolve to develop the first barrier protection equipment for nanoparticle research.

1. Introduction

In 2005, FAURE Ingénierie (FI) decided to develop its presence in the fields of nanotechnology consulting and engineering and notably participated in the Minatex project in Grenoble, France. Initial specialized demand came mainly from research laboratories developing the first artificial nanoparticles and nanostructures. With the problems linked to asbestos fresh in everyone’s memories, researchers were the first to be aware of the knowledge gap concerning the effects on health and the potential dispersal of such substances.

FI R&D department is focused on understanding and controlling indoor air quality. Amongst our priorities, controlling nanoparticles has become one our most important challenges for the management of specialized environments (laboratories and production rooms), but also for indoor air conditioning in the widest possible sense.

R&D work focused on evaluating the limits of traditional calculation tools used for designing rooms and work stations particularly for nanoparticles. Through partnerships with CEA (LITEN in Grenoble) and LEPTAB (La Rochelle) research teams, it became clear that a lot of progress was needed before the dispersal of nanoparticles could be controlled in view of their particular behavior (high dispersal rate, sensitivity to electrostatic forces, rapid development of particle size, etc.). This was the case whether nanoparticles came in the form of individual particles, aggregates or agglomerates.

As a result, FI is currently in a design and validation process that should lead to the production of the first work stations and rooms specifically designed and validated for this type of use. They present a solution for controlling risk by primary prevention.

This range of work stations called PSPN (Safety cabinets for nanostructured particles) meet the expectations of the different parties involved in the use of nanomaterials and those requesting safety cabinets. These first safety cabinets are specifically designed for research and development work.
2. Presentation
The PSPN is a reinforced safety cabinet designed to protect personnel handling harmful particles, especially nanoparticles and nanostructured particles. Made of stainless steel, this robust cabinet can be installed in a laboratory with or without dedicated air conditioning. This equipment has a unidirectional vertical airflow and controlled exhausting (like a PSM). It is easy to handle and assemble in a protected space in response laboratory requirements.

This document presents the general characteristics of the safety cabinet range which is to expand shortly. The following characteristics can be modified according to use: dimensions, filtering efficiency, chemical filtering and filter type, alarm type. Each new model will undergo the same thorough initial qualification procedure as the prototype, as containment efficiency depends on several different factors.

2.1. Functionalities
This equipment is designed for working on dry powders and or suspensions in a clean and contained environment (dynamic containment) while ensuring that the operator, handled product and environment are protected. As we are dealing with air contamination, the equipment must be effective for a wide range of particle size from 'standard' dust (D<100 µm) to nanometric particles (D<100 nm).

When in operation, the equipment must completely control the risk of particle dispersal:

- Via the equipment’s front opening.
- Via leaks throughout the ductwork and air plenums.
- During disassembly, maintenance or controls on parts of the equipment that need replacing (filters, fans, joints, sensors, etc.).
- While cleaning the inside surfaces of the equipment.
- To the outside of the building (air extracted from the cabinet through the rooftop).
- To the inside room environment and possibly any areas adjacent to where the equipment is placed.

2.2. General presentation
The inside dimensions of the safety cabinet are: depth = 700 mm, width = 1300 mm and height = 950 mm. This provides enough space for laboratory equipment (scales, cyclone, mill, etc…).

The choice and preparation of the stainless steel used makes cleaning easy (minimum roughness, chemical resistance and good static dissipation) and gives it the sturdiness needed for the potentially abrasive nature of certain products.

The particle level class in the operating space is set according to the product’s contamination risk level. Filtering systems can be adapted depending on the size of the aerosols to be contained (Filters for the PSPN range are manufactured especially to obtain filtering efficiency and the homogeneous pressure loss required. The homogeneity of speeds obtained guarantees a balance of flow between the different parts of the work surface). These systems concern both the quality of the operating space and
the air discharged into the environment around the safety cabinet. Adsorption modules can be added to the filtering equipment to reduce any possible emission of chemical substances used in the operating space.

The work surface is perforated at the edges. It can be removed making it easier to disassemble pre-filters. The main work surface is shaped to collect liquids if these are to be used.

Fittings for glassware are included and can be modified on each cabinet. Injection and measurement points for integrity testing are situated at each filtering level to make such operations easier to carry out.

2.3. Airflow
Modeling has been used to design the cabinet’s airflow to decide or analyze:

- The size and position of exhaust openings.
- The size of the work surface (non-perforated).
- The effects of having a sink.
- The disturbances caused by equipment used in the work surface area.
- The influence of external airflow conditions that can be very prominent.
- The limits for safe operation in order for level 1 (malfunction leading to corrective action) and level 2 (immediate evacuation) alarm thresholds to be set.
- The horizontal speed in the air barrier taking into account the influence of the position of the operator when working.

Particular attention was paid to the path taken by airflows under the work surface to reduce the risks of back-diffusion. The speeds at which air flows through exhaust openings must be minimal to avoid turbulence.

We also simulated operating two cabinets one next to the other to verify that there was no exchange between the two.

2.4. Ergonomics
These safety cabinets provide the universal and flexible qualities required for handling and testing operations in the context of research and development: large front opening and modular work surface in function of operational requirements. Safety cabinets can be set up side by side to ensure maximum protection during handling operations.

The sides are equipped with tracks for the front plate glass access panel, with two lateral sliding glass panels to allow communication between the two cabinets and with points reserved for passing fluids.

There are in-built fittings for glassware and other laboratory accessories.

2.5. Safety
When the cabinet operates normally, the operator is protected. If the safety cabinet malfunctions, there are two alarm levels:

1. Failure without consequence for operator protection
2. Malfunction with possibility of leakage (immediate evacuation of unprotected operators).
The operator has access to information and alarms on the front of the safety cabinet. The cabinet is also equipped for transmitting alarm information to a Building Management System if necessary.

3. Qualification procedure
A formal approach to qualification was followed for safety cabinet qualification in order to meet the requirements for minimizing risks needed by laboratories handling nanoparticles. All qualification tests include a report for each safety cabinet giving the operational capacity and the overall protection level corresponding to the primary protection from exposure criteria.

Qualification has two stages:
• Initial qualification (standard test) of a prototype
• Operational qualification when standard manufactured equipment is received.

Below is a description of the initial qualification stage which is the more interesting stage.
This consists of a thorough characterization of properties including all the latest modifications made to the system and optimal settings. This particular qualification required tools that are not normally used to qualify safety cabinets: An ISO 5 class clean room, a generator and a nanoparticle analyzer.

A few tests concerning comfort of use are carried out before the performance tests:
• Lighting efficiency in the work surface area
• Noise level at the front of the cabinet
• Vibration level of the structure measured over 3 axes

The first qualification stage then begins which corresponds to the standard tests we carry out on all MBSC (Microbiological safety cabinets) in conformity with the NF EN 12469 standard:
• Validation of modelling
• Measurement of blowing speeds
• Measurement of air barrier speeds
• Control of absence of particle collection and leakage to outside environment using smoke
• Evaluation of air movements outside of the safety cabinet (smoke)
• Absolute filter integrity testing using aerosols equivalent to the ISO particle class range.
• Inside and outside particle level testing
• Control of automatic alarm functions

This first stage corresponds to the operational qualification tests that will have to be carried out on standard manufactured safety cabinets.
The second qualification stage is only started once the preceding tests give satisfactory results (after setting blown and exhausted airflow rates).

3.1. Performance tests with nanoparticles:
This type of test needs to be conducted in a clean room as a nanoparticle source cannot be detected in an environment that is naturally full of them. As a result, tests were carried out in an ISO 4 to 5 area thus eliminating most of the standard nanoparticles found in an everyday environment.
The nanoparticle aerosol used was made up of graphite particles with a median diameter of 41nm (selected according to the characteristics required for the first experiments to be carried out in these cabinets).
Particles were measured by an SMPS (Scanning Mobility Particle Sizer) consisting of a DMA (Differential Mobility Analyser) and a CNC (Condensation nucleus counter) with up to 44 channels.
with a range of 5.5 to 350 nm at a rate of 0.3 per minute. This system gives a precise image of particle size but requires a large amount of aerosols (rate 100 times lower than that of a particle counter).

- **Particle level control**
  Here, we simply needed to adapt the sample volume for measurements to be relevant. We decided to take samples every hour from each sample point when we used an SMPS.
  An additional measurement made with just the CNC confirmed the results given by the SMPS (verification of accuracy of SMPS value correction algorithm whose overall measurement is only made up of some of the sampled aerosols).

- **Filter system integrity test**
  The testing protocol is similar to a standard integrity test (see ISO 14644-3). The sensor speed just needed to be adapted to take the detector’s slow rate (1.5 l/min) into account. Particle size development for such a test aerosol (high concentration = greater interactions) required controlling just above the filter to be tested (and not at generator outlet).

- **Containment test with nanoparticles**
  We created a diffuser with the same dimensions as the work surface for this test. This diffuser was perforated at regular intervals all over its upper face to reconstitute what could be contamination occurring in an identical manner at any point on the work surface.
  The test consisted of searching for any nanoparticle that could escape from the cabinet via the front opening by sweeping it with the cabinet in a standard operating mode and then deteriorating the operating conditions (opening the front panel wider, reducing the extraction rate, creating obstacles, etc.).

4. **Performance results**

Once the blowing and exhausting rates had been adjusted and some of the parts modified to obtain a completely airtight cabinet (particular attention was paid to all welding, the joint system used and cable installment), we concluded that the prototype was in compliance for the following criteria:

- **Dispersion of blowing speeds**
  We obtained a less than 20% dispersion level. The aim was to reduce dispersion as much as possible. We checked that the position of the blowing fan did not have any adverse effect on the results.

- **Air barrier horizontal speed dispersion**
  We obtained a less than 20% dispersion level.
  This criterion is very important for controlling containment. We checked the influence of airflows situated in front of the safety cabinet by varying the blowing speeds in the clean room: as expected, a high outside air speed can disrupt air barrier homogeneity. This needs to be taken into account in clean room design and during qualification checks.

- **Inside air flow paths**
  The visual test using smoke showed good exhaust speed and good containment efficiency. Tests were carried out by placing obstacles in front of the air barrier and on the work surface to test airflow behavior in unfavorable conditions.

- **Filter and structure integrity**
  Localized leakage rate for filters and structures less than 0.001% MPPS (stricter criterion than the ISO 14644-3 standard that gives 0.01% as the maximum penetration value for absolute filters).
  The acceptable leakage rate was very low as we went on the basis that nanoparticles handled could be toxic and weak in concentration.

- **Cleanliness class**
Class lower than ISO 5 for particles greater than 0.1 µm. 
Less than 5 particles per m$^3$ between 5 and 100nm (measured 15 cm above the work surface). 
It is worth noting that the level of cleanliness required for a product can vary immensely and that it will often be unknown for new products.

- **Containment efficiency**
  
  Sweeping of the front of the cabinet showed that the leakage rate by back-diffusion to the outside space was less than 0.001% MPPS (identical criterion to integrity test).

5. **Conclusion**

The following conclusions were drawn from this series of tests:

- Manufacturing quality is of utmost importance to ensure that the cabinet is sufficiently airtight in view of the operational pressure attained.
- The results obtained by simulating air barrier speeds were validated by the tests.
- The environment in which the cabinet is placed must take into account any interfering air flows.

Here are some further remarks we can add concerning the installment of safety cabinets for handling harmful nanoparticles or nanostructured particles:

- It is very important for the future users to participate in the project to ensure both the ergonomic aspect of using the cabinet (whether laboratory apparatus fits into the workspace and the risks involved with the activity undertaken) and an understanding of how to use the cabinet (maintenance, monitoring of indicators, hand movements, etc.).
- The prototype stage is indispensable. This stage must include case-specific qualification with the aerosol that is closest to the materials that are to be handled in the standard manufactured safety cabinet.

FI projects in this domain are to develop the PSPN range in order to adapt cabinets to the different requirements and practical constraints of laboratories (size, price, level of risk involved). As a result, new prototypes are to be developed over the next two years and will undergo the same design procedure: Simulation/Prototype/Case-specific qualification.