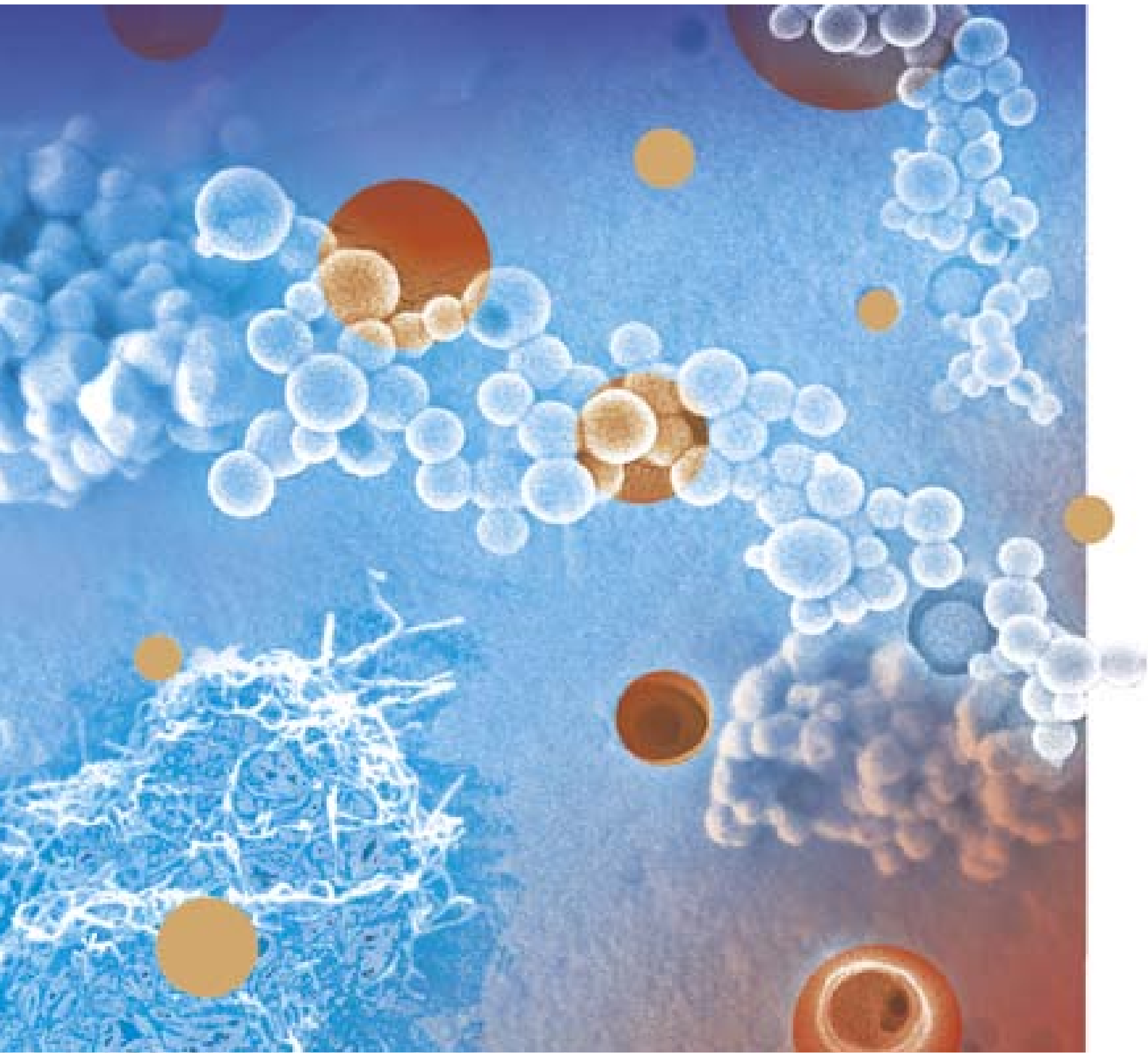
Institut National de Recherche et de Sécurité



Nanomaterials

L’Institut national de recherche et de sécurité (INRS)

In the field of occupational risk prevention, INRS is a scientific and technical body that works on an institutional level with the *Caisse nationale d’assurance maladie des travailleurs salariés (CNAMTS), Caisses d’Assurance Retraite et de la Santé au Travail (CARSATs), Caisses régionales d’assurance maladie (CRAMs)* and *Caisses Générales de Sécurité Sociale (CGSSs)*. As the need arises, it also works for Government departments as well as for any other body that deals with occupational risk prevention.

It has developed a body of multidisciplinary know-how that is available to all those responsible for prevention in businesses, such as employers, occupational physicians, *Comités d’hygiène, de sécurité et des conditions de travail (CHSCTs)*, and workers. To help deal with such complex issues, INRS has scientific, technical and medical skills covering a very wide range of disciplines, all aimed at controlling occupational risks.

For instance, INRS draws up and distributes a number of documents concerning occupational health and safety, such as publications (periodical or not), posters, audiovisual aids, multimedia and a web site. INRS publications are distributed by the CARSATs. They may be obtained from the S*ervice Prévention of the Caisse régionale* or *Caisse générale* of your area, whose address is provided at the end of this brochure.

INRS is a non-profit organization (law of 1901) set up under the aegis of CNAMTS and subject to the financial control of the State. It is run by a joint Board of Directors made up of two colleges representing in equal numbers employers and employees, and chaired in turn by a representative of each college. It is almost entirely funded by the National Fund for the Prevention of Occupational Accidents and Diseases.

Caisses d’Assurance Retraite et de la Santé au Travail (CARSATs), Caisses Régionales d’Assurance Maladie (CRAMs) and Caisses Générales de Sécurité Sociale (CGSSs)

In order to help reduce occupational risks in their region, CARSATs, CRAMs and CGSSs have a prevention department made up of consulting engineers and safety inspectors. They are specifically trained in occupational risk prevention disciplines and use their everyday experience of businesses to advise, and under certain conditions back up, the company stakeholders (management, occupational physicians, CHSCT, etc.) in implementing the most suitable prevention approaches and tools for each situation. All the documents published by INRS are available from them.

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Nanomaterials

Definitions, toxicological risk, characterisation of occupational exposure and prevention measures

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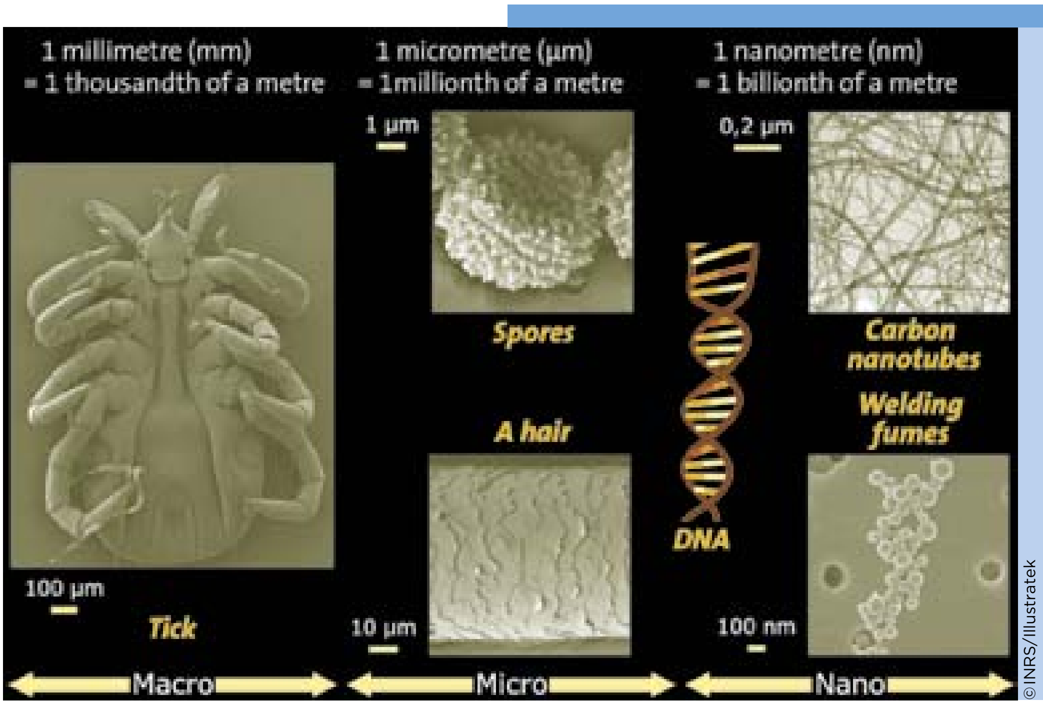
Terminology and definitions

The nanoworld

At a conference organised in 1959, the American physicist Richard Feynman stated that the principles of physics allowed the handling and controlled positioning of atoms and molecules, individually, rather like Lego building bricks. With this statement, Feynman invited the scientific community to explore the world of the infinitesimally small.

The term 'nanotechnology' was first used in 1974. In the 1980s, the discovery of the scanning tunnelling microscope and then of the atomic force microscope truly opened up the nanoworld to researchers.

The reference unit of the nanoworld is the nanometre (or nm for short). The prefix 'nano' comes from the Greek *nannos*, which means 'dwarf'. A nanometre equals one billionth of a metre (1 nm = 10^{-9} m = 0.000 000 001 m), which is approximately 1/50 000th the thickness of a human hair (see Figure 1). This is the scale of the atom, the basic building block of all matter. There is the same difference in size between an atom and a tennis ball as there is between a tennis ball and the planet Earth.



▲ Figure 1. The scale of sizes: from the visible to the invisible

Nanotechnologies and nanosciences

The nanotechnologies make up a multidisciplinary field of research and development based on understanding and controlling the infinitesimally small. More specifically, they bring together all the techniques used to produce, handle and characterise matter in the nanoscale.

The nanotechnologies are the formalisation of concepts and processes from the nanosciences, i.e. the sciences that aim to study and understand the properties of matter on the atomic and molecular scale.

Nanomaterials

There are many definitions of the term ‘nanomaterial’.

In October 2011, the European Commission proposed in a recommendation[1] a definition for the term ‘nanomaterial’: “a nanomaterial is a natural, incidental or manufactured material containing particles, in an unbound state or as an aggregate or as an agglomerate and where, for 50 % or more of the particles in the number size distribution, one or more external dimensions is in the size range 1 nm - 100 nm”.

The recommendation states that in specific cases and where warranted by concerns for the environment, public health, safety or competitiveness the number size distribution threshold of 50 % may be replaced by a threshold between 1 and 50 %.

It is also stated that, by derogation from the above, fullerenes, graphene flakes and single-wall carbon nanotubes with one or more external dimensions below 1 nm should be considered as nanomaterials.

According to ISO standard TS 80004-1, a nanomaterial is a material with any external dimension in the nanoscale, i.e. approximately between 1 and 100 nm, or having internal structure or surface structure in the nanoscale.

There are two major families of nanomaterials (see Figure 2):

1. Nano-objects are materials with one, two or three external dimensions in the nanoscale, i.e. approximately between 1 and 100 nm.

Nano-objects can be divided into three categories:

- nanoparticles, which designate nano-objects with three external dimensions in the nanoscale, such as nanoparticles of latex, zinc, iron and cerium oxides, alumina, titanium dioxide, calcium carbonate, etc. (see Figure 3);
- nanofibres, nanotubes, nanofilaments and nanorods, which refer to nano-objects with two external dimensions in the nanoscale and the third dimension significantly larger (carbon nanotubes, polyester nanofibres, boron nanotubes, etc.). These terms refer to elongated nano-objects ranging from one to a few tens of nm in cross-section and 500 to 10,000 nm in length (see Figure 4);

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[1] http://ec.europa.eu/environment/chemicals/nanotech/pdf/commission_recommendation.pdf.

- nanoplates, which refer to nano-objects with one external dimension in the nanoscale and the two other external dimensions significantly larger (clay nanoplates, cadmium selenide nanoplates, etc.).

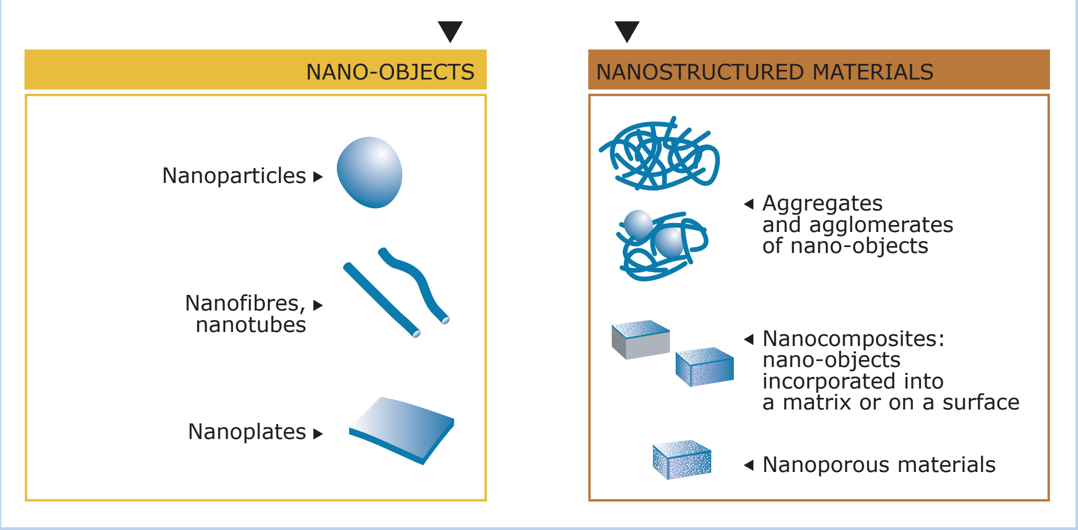
Nano-objects can be used as such in the form of a powder, liquid suspension, or gel.

2. Nanostructured materials are materials that have an internal or surface structure in the nanoscale. Among nanostructured materials, several families may be distinguished, including:

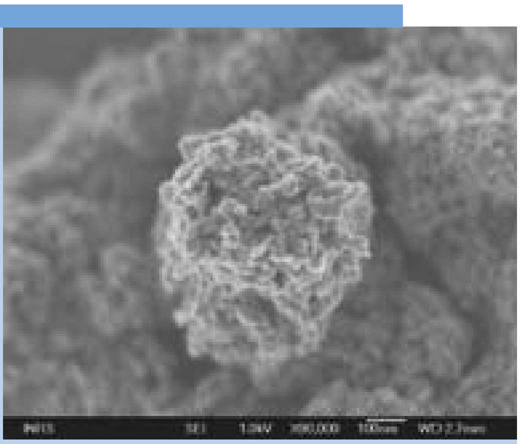
- aggregates and agglomerates of nano-objects: the nano-objects may either be in an individual form (i.e. in the form of primary particles), or in the form of aggregates or agglomerates whose size is significantly greater than 100 nm;
- nanocomposites: these materials are composed, either entirely or in part, of nano-objects that give them improved properties, or properties that are specific to the nanoscale. Nano-objects are incorporated into a matrix or a surface in order to provide it with a new functionality or to alter its mechanical, magnetic, thermal, etc. properties. Carbon nanotube-filled polymers used in the sports equipment sector in order to improve mechanical resistance and reduce weight are one example of nanocomposites;
- nanoporous materials: these materials have nanometre-sized pores. Silica aerogels are nanoporous materials that have excellent thermal insulation properties.

Nanomaterials produced intentionally by humans for specific applications and having specific properties are called manufactured nanomaterials. Among such manufactured nanomaterials, some have been produced in large quantities for many years, such as titanium dioxide, carbon black, alumina, calcium carbonate and amorphous silica. Others that are more recent are manufactured in smaller quantities, such as carbon nanotubes, quantum dots and dendrimers. There also exist nanomaterials produced by humans unintentionally—sometimes called ultrafine particles—in certain thermal and mechanical processes, such as welding and thermal projection fumes, internal combustion engine emissions, etc.

Lastly, natural ultrafine particles are present in the environment, such as volcanic fumes or viruses.



▲ Figure 2. Classification of nanomaterials according to ISO standard TS 80004-1



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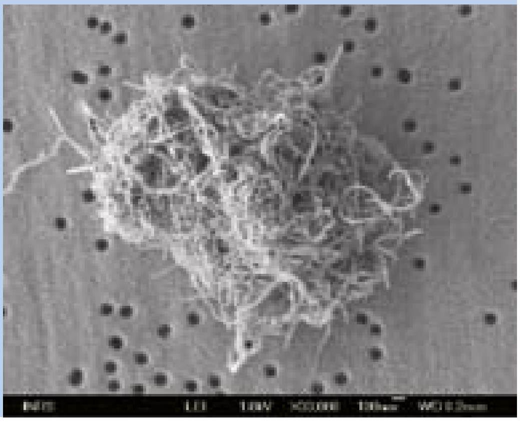
◂ Figure 3. Titanium dioxide nanoparticles seen through a scanning electron microscope



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◂ Figure 4. Multi-walled carbon nanotubes seen through a scanning electron microscope

Applications

In the nanoscale, matter takes on unexpected properties that are often completely different from the properties of the same materials on micro- or macroscopic scales, especially regarding mechanical resistance, chemical reactivity, electrical conductivity and fluorescence. Nanotechnologies thus lead to the development of materials whose fundamental (chemical, mechanical, optical, biological, etc.) properties may have been altered. For instance, gold is completely unreactive on micrometre scales, whereas it becomes an excellent catalyst of chemical reactions in the nanoscale.

All the major families of materials are concerned: metals, ceramics, dielectrics, magnetic oxides, polymers, carbons, etc.

Because of their varied and often novel properties, nanomaterials have a wide range of potential applications, and their use opens up a host of new avenues.

Nanotechnologies are therefore leading to incremental and radical innovations in many industry sectors such as healthcare, the automotive and building industries, agrifood and electronics.

Applications of nanotechnologies and nanomaterials according to industry sector

Industry sector	Examples of current and potential applications
Car industry, aviation and space	Stronger and lighter materials; brighter exterior paintwork with colour effects, and scratch, corrosion and dirt resistant; sensors to optimise engine performance; ice sensors on aircraft wings; diesel fuel additives for enhanced combustion; longer-lasting and recyclable tyres.
Electronics and communications	High-density storage devices and miniaturised processors; solar cells; e-book readers; ultra-fast computers and computer games; wireless technologies; flat screens
Agrifood	Active packaging; additives; dyes, anticaking agents, emulsifying agents
Chemistry and materials	Pigments; fillers; ceramic powders; corrosion inhibitors; multifunctional catalysts; antibacterial and ultra-resistant fabrics and coatings
Building industry	Self-cleaning and pollution-resistant cement, dirt-resistant and self-cleaning windows; paints; varnishes; glues; sealants
Pharmaceuticals and healthcare	Active drugs and agents; medical anti-allergic adhesive surfaces; bespoke drugs that target specific organs; biocompatible surfaces for implants; oral vaccines; medical imaging
Cosmetics	Transparent sun creams; abrasive toothpastes; longer lasting make-up
Energy	Next-generation photovoltaic cells; new types of battery; smart windows; more efficient insulating materials; storage of hydrogen fuel
Environment and ecology	Reduction of carbon dioxide emissions; production of ultra-pure water from sea water; more efficient and less harmful pesticides and fertilisers; specific chemical analysers
Defence	Detection and control of chemical and biological agents; miniaturised monitoring systems; more accurate guidance systems; light, self-repairing fabrics

Some examples of nanomaterials and their applications

Titanium dioxide

Since the nineteen twenties, titanium dioxide has been one of the most widely used synthetic mineral pigments in the world (especially in paints, inks, plastics, bitumen, etc.), together with iron oxides and carbon black. This white pigment is today incorporated into cements, and also into glass, due to its photocatalytic properties which enable it to break down a wide variety of organic and inorganic materials as well as micro-organisms (NO_X, CO, O_3, etc.). It gives cement self-cleaning properties (beneficial for the maintenance and durability of buildings) as well as pollution-resistant properties. Nano-titanium dioxide, like zinc oxide, is also currently used in sunscreen products as a UV filter.

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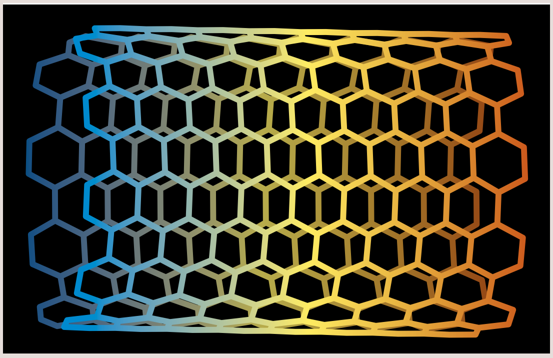
▲ Figure 5. Façade of a building made of glass with titanium dioxide nanoparticles

Carbon nanotubes

Carbon nanotubes, together with other molecules called fullerenes, make up the third crystalline form of carbon. Their structure can be represented by one or more sheets of graphene rolled up on themselves (single-walled carbon nanotubes) or around each other (multi-walled carbon nanotubes). These hollow cylinders show remarkable mechanical and electrical properties (a carbon nanotube is 100 times more resistant and six times lighter than steel with an equivalent cross-section), which can lead to many applications, such as the development of high-performance composite materials, conducting polymers, and smart fabrics. For instance, they are used in the aviation industry (aircraft wings), sports equipment (tennis rackets, bicycles), and electronics (diodes, transistors, etc).



▲ Figure 6. Structure of a single-walled carbon nanotube

Quantum dots

Quantum dots are semi-conducting nanocrystals such as cadmium selenide which have fluorescent properties that can be adjusted by controlling their size. When illuminated by white or ultraviolet light, these inorganic crystals fluoresce, emitting light whose colour depends on their composition and diameter (the colour can vary from blue to red). These materials can in particular be used in biological imaging, for instance for tagging and monitoring living cells, imaging live animals, fluorescent microscopy, etc.

Figure 7. Fluorescent ▶ inorganic nanocrystals

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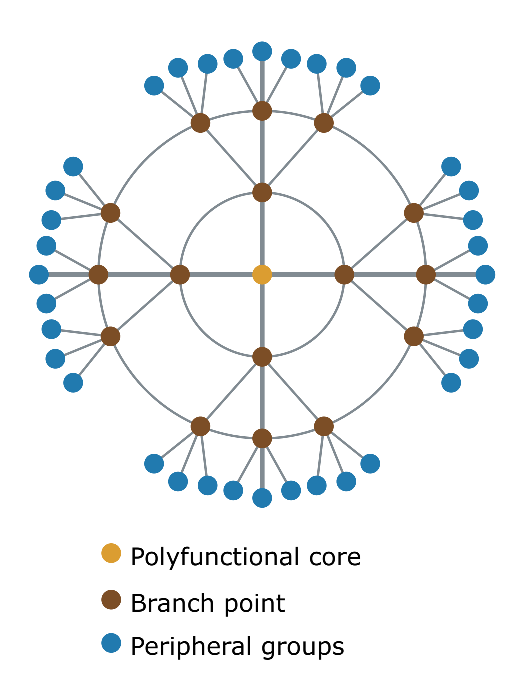
Dendrimers

Dendrimers are nanometre-size macromolecules characterised by a branched 3-D structure. They are related to multifunctional polymers and have specific properties of solubility, viscosity, thermal stability, etc. They are usually globular in shape. Dendrimers have many potential uses, and these are related to their unusual topology which consists of three highly specific regions: the core, the branches that form the dendritic matrix, and the periphery, which is made up of a wide variety of functional groups. Applications include the carrying and controlled release of active principles, as well as gene therapy, catalysis and biological sensors.



▲ Figure 8. Structure of a dendrimer

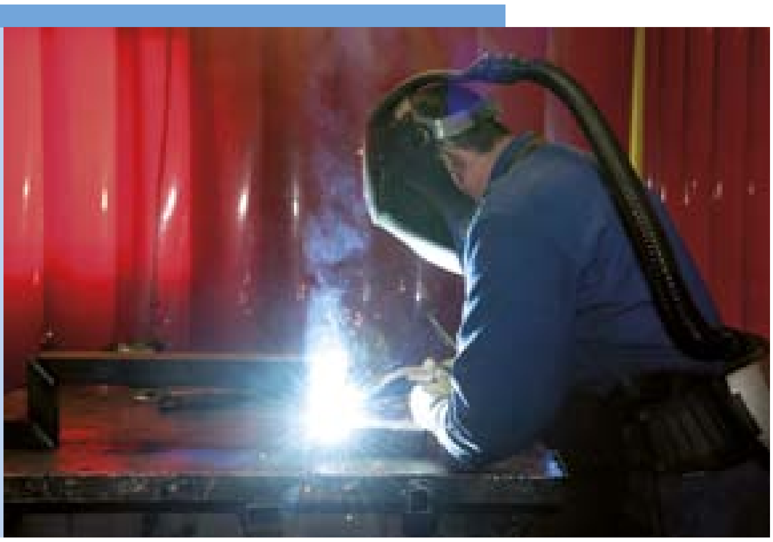
Occupational exposure situations

Two types of occupational exposure to nanomaterials may be distinguished:

- exposure related to processes whose purpose is not the production of nanomaterials, but whose implementation generates such materials: thermal and mechanical processes such as the welding, cutting and polishing of metals (see Figure 9), the smoking of foodstuffs, etc.

Processes generating nanomaterials unintentionally

Type of process	Examples of processes
Thermal processes	Smelting and refining of metals (steel, aluminium, iron, etc.) Metallisation (galvanisation, etc.) Welding and gouging of metals Cutting of metals (laser, thermal lance, etc.) Thermal surface treatment (laser, thermal projection, etc.) Application of resins, waxes, etc.
Mechanical processes	Machining Sanding Drilling Polishing
Combustion	Diesel, gasoline or gas engine exhaust Incineration plant, thermal power plant, crematorium Smoking of foodstuffs Gas heating



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◄ Figure 9. Arc welding operation

- **exposure related to the production and use of manufactured nanomaterials**

At every stage of production, from the reception and storage of raw materials to the packaging and dispatch of finished products, and including the possible transfer of intermediate products, workers may be exposed to nanomaterials. Similarly, the use of nanomaterials, their incorporation into various matrices, and the machining of composites that contain them, as well as the cleaning and upkeep of workplaces and equipment, and waste disposal, are additional sources of exposure.

Examples of occupational exposure situations

- Transfer, sampling, weighing, preparation of a liquid suspension and incorporation in a matrix of nanopowders (formation of aerosols)
- Transfer, shaking, mixing and drying of liquid suspensions containing nanomaterials (formation of droplets)
- Filling or emptying of reaction vessels
- Machining of nanocomposites: cutting, polishing, drilling, etc.
- Packing and packaging, storage and transportation of products
- Cleaning of equipment and workplaces: cleaning vessels, glove boxes, benchtops, etc.
- Upkeep and maintenance of equipment and premises: dismantling vessels, changing used filters, etc.
- Collection, packaging, storage and transportation of waste
- Degraded mode or incidents: leak in a reaction vessel or closed system

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▲ Figure 10. Filling a reaction vessel during formulation of a glue

Manufacturing processes for nanomaterials

Manufactured nanomaterials may be synthesised using two different approaches, known as the bottom-up approach and the top-down approach.

The **bottom-up approach** originated in research laboratories and in nanoscience. It consists in building nanomaterials atom by atom, molecule by molecule or aggregate by aggregate. Assembly or positioning of atoms, molecules or aggregates is carried out in a precise, controlled and exponential manner, thus making it possible to produce functional materials whose structure is completely controlled.

The **top-down approach** originated in microelectronics. It consists in reducing, and more specifically miniaturising, existing systems (usually microstructured materials) by optimising current industrial technologies. The systems or structures are thus gradually reduced in size or divided until they reach nanometre scales.

The two approaches tend to converge in terms of the range of object sizes. However, the bottom-up approach appears to be richer in terms of the type of material, diversity of architecture and control of the nanometre state, whereas the top-down approach is capable of providing larger quantities of material, but the control of the nanometre state is trickier.

The bottom-up approach uses chemical and physical production methods (gas phase reactions, sol-gel techniques, laser induced pyrolysis, microwave plasma, etc.), whereas the top-down approach mainly requires the use of mechanical methods (mechanosynthesis, strong deformation by torsion, etc.).

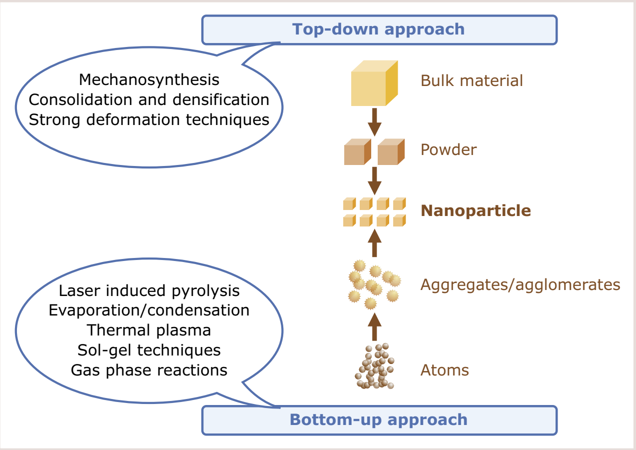


Figure 11. The two approaches to producing nano-objects and nanomaterials ▶

Health effects

Our understanding of the toxicity of manufactured nanomaterials is still fragmentary, even though much research has been carried out in this field. Most toxicological data comes from studies, usually concerning the onset of acute effects, carried out on cells or on animals, and is therefore difficult to extrapolate to humans. However, it has already been shown that the ultrafine particle components in air pollution, especially in that emitted by factories and diesel engines, show toxic properties that are likely to have harmful effects on human health (respiratory allergic diseases, such as rhinitis, asthma and bronchitis, and cardiovascular disorders, particularly in vulnerable people). These specific properties may apply to manufactured nanomaterials.

Routes of exposure

The toxicological risks of nanomaterials are linked to three potential routes of exposure: inhalation, ingestion and skin contact.

The respiratory system is the primary route of entry of nanomaterials into the human body. Penetration is all the greater if the individual is engaged in physical activity or has impaired lung function. Once nanomaterials have been inhaled, they can either be exhaled (rejected) or deposited in various regions of the respiratory tree. Deposition is not generally uniform throughout the entire respiratory tract: it varies considerably according to the diameter, the extent of aggregation and agglomeration, and the behaviour of the nano-objects in air (see Figure 12). Objects with a diameter between 10 and 100 nm are mostly deposited in the alveolar region (deep lung), in a significantly higher proportion than that of micrometre-scale objects. Smaller objects are mainly deposited in the upper airway and, to a lesser extent, in the tracheobronchial region.

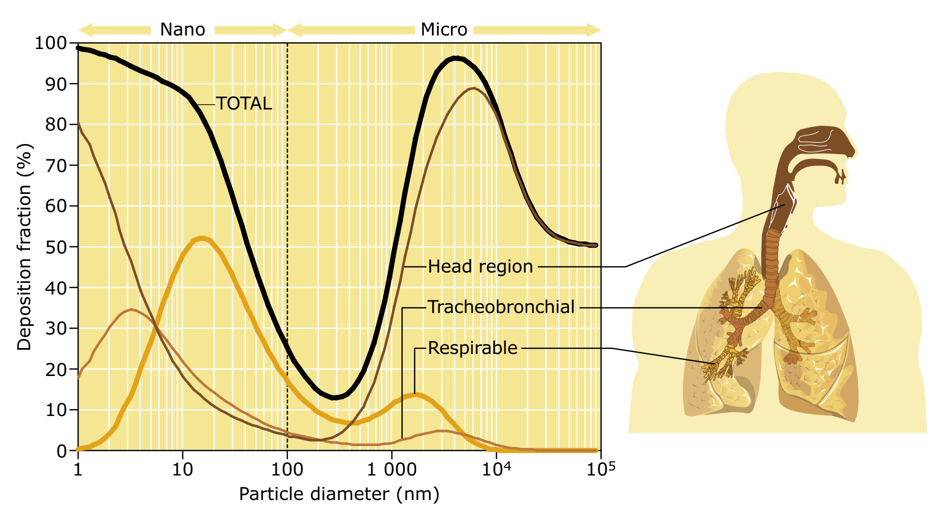


Figure 12. Predicted total and regional deposition of particles in the human respiratory tract, according to particle size (model of the International Commission on Radiological Protection, ICRP)

Nanomaterials may also be found in the gastro-intestinal system after being ingested, or after swallowing when they have been inhaled.

Transcutaneous absorption of nanomaterials is a possibility that is still being investigated. However, it has already been shown that nanomaterials are able to penetrate more deeply than micrometre-scale objects (which generally remain on the surface of the upper layers of the skin), and increasingly so as their size becomes smaller. The surface and elastic properties of nano-objects, as well as sebum, sweat, pores, local irritation and repeated flexion of the skin are also factors which might promote their transcutaneous absorption.

Fate in the body and potential health effects

The toxicity of inhaled nanomaterials partly depends not only on their deposition in the respiratory tree (region, quantity, etc) but also on the latter's ability to eliminate them partially or totally (clearance process). Two processes are involved:

The toxicity of inhaled nanomaterials partly depends not only on their deposition in the respiratory tree (region, quantity, etc) but also on the latter's ability to eliminate them partially or totally (clearance process). Two processes are involved:

- **chemical elimination,** which consists in the dissolving of soluble nanomaterials in biological fluids. Chemical elimination processes take place in all regions of the respiratory system;
- **physical elimination,** which consists in the transport of non-soluble or poorly-soluble nanomaterials to one or more other sites in the body, especially to the mouth and nose. The mechanisms involved in physical elimination vary according to the regions of the respiratory system concerned. Insoluble nanomaterials that are deposited in the upper airway and in the tracheobronchial tree are mainly eliminated by mucociliary transport towards the nose and mouth. They may then be either swallowed (and reach the digestive system) or be expelled (sneezing, nose blowing). In the pulmonary alveoli, purifying cells called macrophages usually take care of the elimination of insoluble nanomaterials via a phagocytosis mechanism. However, several studies appear to show that individual nano-objects, i.e. that are not aggregated or agglomerated, are not phagocytosed efficiently by macrophages. This may result in significant build-up of nano-objects in the pulmonary alveoli. This overload is likely to cause inflammation, which may lead to the development of certain pulmonary diseases.

Moreover, due to their size, inhaled or ingested nanomaterials are probably able, unlike other kinds of dust, to cross biological barriers—nasal, bronchial, alveolar, intestinal and placentary—and migrate to different sites in the body (the translocation process) via the blood and lymph. They can then reach various organs, especially those which are the most highly irrigated such as the liver, heart and spleen. They can also pass through the nasal membrane and be transported via the olfactive and cranial nerves to the ganglia and the central nervous system. The dissemination and build-up of nanomaterials throughout the whole body could play a major role in the development of certain diseases of the heart and central nervous system (see Figure 13).

Lastly, a certain amount of evidence clearly shows that nanometre-scale objects have greater toxicity and cause more serious inflammatory effects than micro- and macroscopic objects with the same chemical composition. It is therefore necessary to remain vigilant, even with regard to chemicals that are said to be inert on micro- and macro-scales.

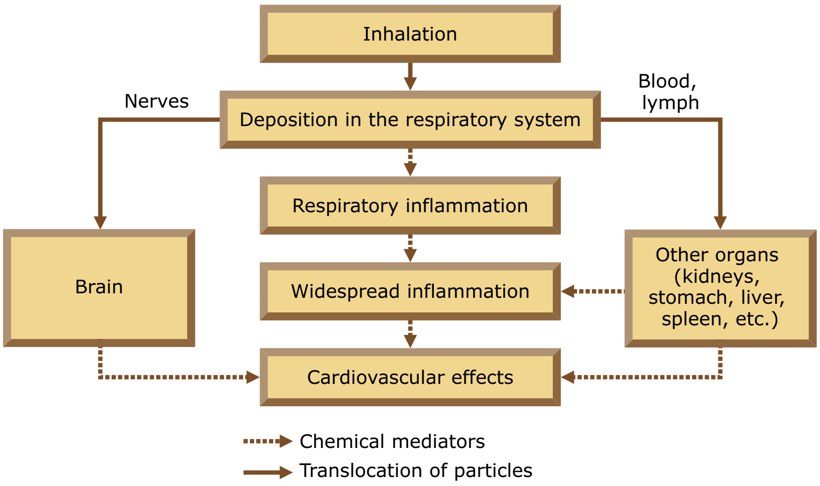


Figure 13. Fate and potential effects on the body of inhaled nano-objects

Principal factors responsible for health effects

The principal factors that determine the toxicological effects of nanomaterials on the body are as follows:

- factors **related to exposure:** routes of entry into the human body, duration and frequency of exposure;
- factors **related to the body of the exposed person:** individual susceptibility, carrying out of physical exercise, sites of deposition, evolution and translocation of nanomaterials after they enter the body;
- factors **related to the nanomaterials:** apart from chemical composition and the possible presence of adsorbed substances (metals, polycyclic aromatic hydrocarbons, etc), several physico-chemical characteristics are also involved in the degree of toxicity of nanomaterials, such as size, particle-size distribution, specific surface area, surface reactivity, number, shape, dustiness, porosity, crystallinity, solubility, electric charge and the degrees of aggregation and agglomeration. Similarly, production methods, surface treatment and ageing are likely to affect the toxicity of nanomaterials. Each nanomaterial therefore has its own potential toxicity.

Explosion and fire

There is little data currently available in the literature about the explosion risk associated with nanomaterials. It should nonetheless be possible to predict their behaviour by extrapolating from what is already known about fine and ultrafine powders. However, this approach cannot be implemented with any guarantee of success in view of the fact that physical and chemical properties are generally altered once nanometre scales are reached. As a general rule, the violence and severity of an explosion as well as the likelihood of causing one tend to increase as the size of the particles decreases. The finer the powder, the greater is the increase in pressure and the lower is the required activation energy. **Nanomaterials therefore tend to be more reactive, or indeed more explosive, than coarser dust with the same chemical composition.**

Several conditions need to be fulfilled at the same time for an explosion to occur: the presence of combustible particles in suspension in the air and in sufficient concentration (concentration within the explosibility range), the presence of an oxidising agent (usually oxygen in the air), a confined space, and an ignition source (spark, hot surface, friction, lightning, etc).

The characteristics of the particles (chemical composition, particle-size distribution, etc.) and the environmental conditions (temperature, humidity, etc.) affect the explosibility range. In addition, a number of factors are likely to facilitate airborne suspension of nanomaterials, and thus create the right conditions for an explosion to occur: poor ventilation, unsuitable working methods (for instance, infrequent cleaning or cleaning with a compressed air gun), leaking equipment, accidental spillage, build-up in pipes, etc.

Some easily oxidisable metals, such as aluminium, magnesium and lithium, as well as some organic substances such as carbon nanotubes, are especially high-risk.

There is also little available information about nanomaterial fire risks. However, it is always possible to refer to published data about coarser powders. Three factors need to be combined for a fire to occur: the presence of combustible particles, an oxidising agent (usually oxygen in the air), and a source of energy. Given that an oxidising agent and combustible material is very often present in the workplace, the risk of fire is extremely high if there are energy sources present. Among these, work with heat sources is a major cause of accidents.

Characterisation of occupational exposure

The characterisation of potential emissions and exposures in workplaces during operations involving nanomaterials is a difficult task, but one that is key to documenting exposure to nanomaterials and the effectiveness of technical prevention measures.

Indicators to be considered

For nearly fifty years, workplace exposure to a chemical agent in aerosol form (airborne particles) has been characterised quantitatively by the time-weighted average particle mass concentration in air (mg/m^3 or µg/m^3 of air). For fibres, exposure is expressed as the number of fibres per unit volume of air (fibres/m^3 of air). Aerosols are sampled by particle-size fraction (inhalable, thoracic and respirable fractions) according to the extent of penetration into the respiratory tree and the resulting health effects. Since the aim of measuring is to assess the presence of a chemical agent in the air in a worker's breathing zone, it should be carried out using personal air samplers. Until now, this approach has been used for all chemical agents in aerosol form, whatever the size of the particles they are composed of. It is included in a large number of standards and regulations relating to occupational health. The choice of indicators to be taken into account (aerosol fraction related to a health effect, mass concentration in air, chemical composition) results from the correlations established between the indicators and toxic effects in animals (by toxicological inhalation studies) or harmful effects on humans (by epidemiological studies).

This conventional approach to quantitative exposure assessment has been called into question for aerosols in operations involving nanomaterials. In the current state of knowledge resulting from toxicological and epidemiological studies, it seems increasingly evident that, for nanomaterials made up of insoluble or poorly soluble substances, exposure cannot be assessed solely by the two indicators of mass and chemical composition. However, defining how they should be assessed today remains an ambitious goal, since the list of risk factors is long and the number of nanomaterials studied is still limited. Nonetheless, it appears that measuring the:

- **particle mass concentration** (i.e. in mg/m^3) continues to be a useful measurement as long as a particle-size selection is carried out. In addition, since this measurement remains the standard method for aerosols, it ensures continuity with past exposure data;
- **particle surface concentration** (i.e. in µm^2/m^3) is an appropriate measurement in many circumstances, although it cannot be generalised to all situations;

- **particle number concentration** (i.e. in 1/cm^3) is a suitable measurement when it is not the 'surface-area' factor that is mainly responsible for toxicity. In addition, since this method highlights the finest fraction of a polydisperse aerosol, it is useful for identification.

The size of aerosol particles that should be taken into account ranges from several nm to approximately 10 µm. Although there is no standard definition in this regard, there are several arguments in favour of this recommendation, in particular the fact that:

- nano-objects in their free, agglomerated or aggregated form need to be taken into account;
- nano-objects in their free form can disseminate by heterogeneous coagulation on the submicrometre- and micrometre-size particles making up the background aerosol (ambient aerosol);
- mechanical operations on nanocomposites can cause emissions of particles within the size range corresponding to the alveolar fraction.

As for all chemicals in particle form, chemical composition continues to be a key characteristic to determine. This is also true for morphology when nano-objects are poorly soluble or insoluble and have a high aspect ratio (length/diameter ratio) (nanotubes, nanofibres, etc), or are characterised by a structure with an irregular shape or broken-up. Other characteristics may also be relevant in certain cases, such as crystal lattice, surface reactivity, electrostatic charge, solubility, etc.

Lastly, it is currently agreed that:

- all sampling of aerosols with a view to testing their chemical composition should be carried out at the very least on the basis of the respirable fraction;
- deposition of particles in the respiratory tree should be taken into account when interpreting the results. In practice, this can be done via suitable measurements (particle-size distribution, concentration) and by calculating deposition with the help of a model, such as the model of the International Commission on Radiological Protection, which represents recent data in humans; nevertheless, there also exist other models.

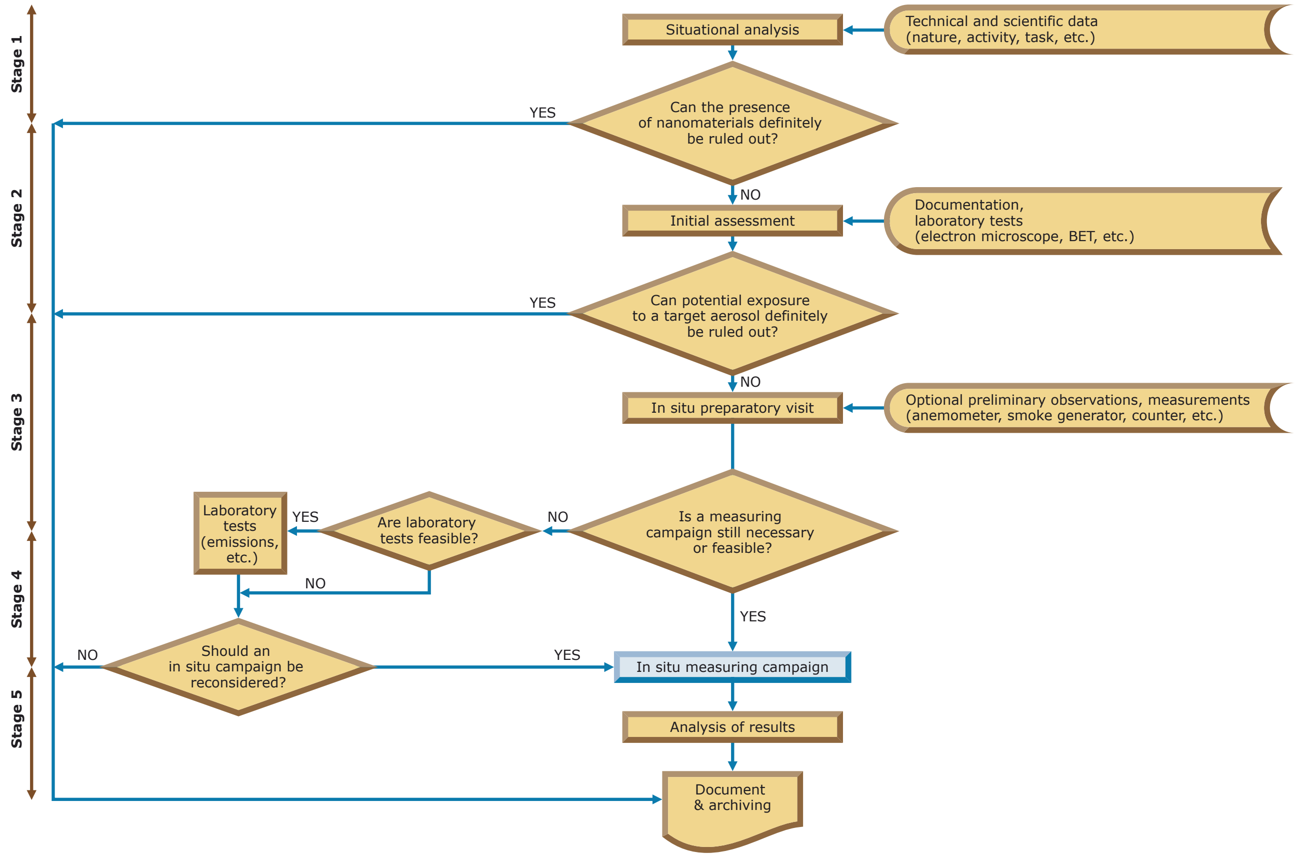
Measuring strategy

In the past few years, several strategies for the assessment of potential emission and occupational exposure during operations involving nanomaterials have been published internationally.

In France, a number of recommendations have been made recently. The overall approach proposed follows a path in five stages (see Figure 14):

- the aim of **the first three stages** is to determine whether the target operation is likely to emit nano-aerosols and to confirm the necessity and feasibility of a measuring campaign;

- the **fourth stage** is the measuring campaign itself. It comprises two levels of intervention: a basic characterisation and/or a specialised characterisation allowing more in-depth investigation. The first level of intervention is intended for people with experience in atmospheric metrology and evaluation of occupational exposure to aerosols, and with a basic understanding of nanomaterial-related risks. The second level is more specialised and is mainly aimed at researchers involved in work related to aerosol metrology and characterisation, and to emissions and/or exposure to substances in particle and nanoparticle form in an occupational health context.
- the **fifth stage** consists in analysing the results.



▲ Figure 14. General flow chart of the strategy developed in France

The aim of the measuring campaign is to identify and characterise the aerosol at the emission sources and at various points distant from them in order to be able to assess potential exposure during the operation in question. There are a number of criteria for choosing between the two levels of intervention:

- skill and experience in measuring nano-aerosols and interpreting results;
- availability of instruments and methods;

- conditions of access to the work station;
- suitability of instruments to the work station environment (ATEX[2] area, etc.);
- existence of previous measuring results at the work station.

If, at the end of the second stage, there is uncertainty about the need to carry out a measuring campaign, or if the campaign is likely to be complicated to carry out (presence of multiple sources, difficult access to the process, specific ATEX[2]-type zoning, etc.), a possible alternative is to carry out specific tests in the laboratory with the aim of estimating potential emissions during the operation in question. Such tests may concern:

- emission of an aerosol from powdered nanomaterials used in the operation in question. Several so-called dustiness approaches are currently undergoing development;
- emission of aerosols from composites or nanoparticle-containing products as a result of various forms of physical stress (sanding, drilling, abrasion, etc) or effects (thermal, UV, etc.) that simulate an operation or ageing.

Measuring methods and instruments

It is generally necessary to implement methods suitable for:

- time-resolved measurement of particle concentrations in the air;
- integrated sampling with a view to perform single particle analysis and/or overall chemical composition of the aerosol sample collected.

The table below provides some examples of methods and instruments that can be used to implement the first level of intervention.

Type of measurement	Parameter	Instrument / Method
Real time	Number concentration (1/cm^3)	Portable condensation nucleus counter (CNC) Portable optical particle counter (OPC)
	Mass concentration (µg/m^3)	Portable optical particle counter (OPC) Portable laser photometer
Integrated	Single particle analysis (morphology, elemental analysis)	Collection* for observation with electron microscope (EM), transmission electron microscope (TEM) or scanning electron microscope (SEM), possibly combined with microanalysis or spectroscopic techniques
	Chemical composition of aerosol	Stationary collection of the alveolar fraction combined with chemical analysis** (e.g. mass spectrometry)

* There are several techniques for particle sampling (by filtration, by electrostatic or thermophoretic precipitation) and various media can be used (membrane, silicon disc, TEM grid, etc.)
** Analytical methods such as those published by INRS (MetroPol database) or NIOSH can be implemented according to the nature of the chemical. Sampling can be carried out using a CATHIA (alveolar) type device.

▲ Figure 15. Examples of methods and instruments that can be used to implement the first level of intervention

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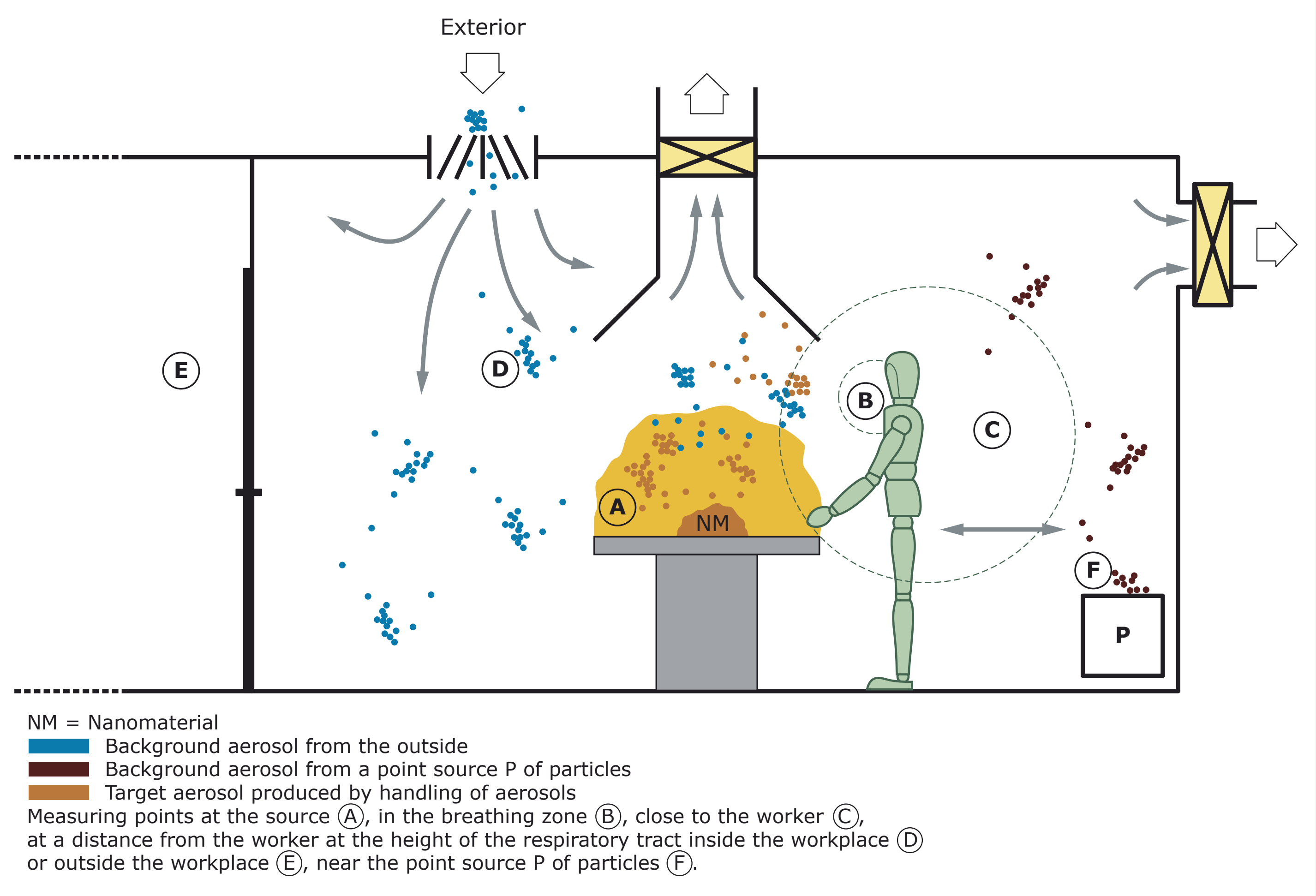
[2] Explosive atmospheres.

With regard to the second, specialised level of intervention, the techniques used are distinguished by improved performance (for instance, regarding the lower detection limit for nanoparticles or the upper number concentration limit for condensation nucleus counters). In addition, it may include:

- real-time techniques for measuring particle number distribution;
- techniques for measuring mass distribution according to size, such as impactors;
- specific techniques for measuring surface area concentrations (µm2 / cm3), knowing that there are different types of surface;
- possibly sampling systems for time-integrated measurements in the breathing zone with the aim of analysing single particles or the overall chemical composition of the aerosol sample collected.

A major problem encountered in real-time measurement comes from the confounding factor represented by the background aerosol, in other words the ambient aerosol already present in the workplace under investigation before the operation in question is carried out. The aerosol is usually omnipresent, and is variable in time and space depending on the various sources from which it may come (combustion or other processes) and on whether it reaches the measuring zone via natural or forced ventilation. It is made up of particles in the nanometre to micrometre size range, and the concentration levels it may attain can easily mask the target aerosol emitted by the operation being monitored. Since real-time measuring instruments (for instance, condensation nucleus counters) are not specific to the nature of the particles they observe, it is not always easy to distinguish this background aerosol. However, making this distinction is essential, since it is important to avoid associating a number concentration (for example) due to the background aerosol with that of the target aerosol, whose real concentration may be several orders of magnitude smaller. The same applies to the particle-size distribution of the target aerosol.

If the situational analysis and the preparatory visit have led to the identification of a possible point of emission, such as transfer of a nanopowder from a container to a beaker, a point as close as possible to the source should be selected for time-resolved and time-integrated measurements. This approach is considered as a worst case scenario for the exposure characterisation, due to the proximity to the emission source. The other measuring points should be carefully chosen nearby and/or further away, taking into account the target operation, the worker, the work station environment, the design of the workplace and building, local and general airflow, etc. (see Figure 16). In general, it is preferable to position integrated measuring points at the height of the respiratory tract.



▲ Figure 16. Possible measuring points in a workplace where a worker is handling a nanomaterial

The recommendations laid out above can be applied to:

- all existing working environments (research laboratories, industrial sites, etc.), at the various stages of production and use of nanomaterials, during cleaning and maintenance of equipment, etc., whether the process and protective equipment is functioning in normal or degraded mode;
- cases of exposure to ultrafine particles emitted during thermal processes (welding, laser cutting, metallisation, etc.), as well as certain mechanical processes on conventional materials (machining, sanding, polishing, drilling).

Developments are naturally to be expected in the field of nanomaterial exposure assessment, especially with regard to instrumentation, measuring criteria and interpretation of results.

In addition, even without suitable instruments and measuring methods, a qualitative assessment of nanomaterial exposure can be carried out (see chapter on ‘Risk assessment’).

Risk assessment

For there to be a risk to a worker's health, it is not only necessary that the nanomaterial be hazardous, i.e. intrinsically toxic, but also that the worker be exposed to it.

Carrying out a quantitative risk assessment at a workstation requires established knowledge of the health hazards of the nanomaterial(s) used as well as of worker's exposure levels. However, currently published data about nanomaterial toxicity remains incomplete, and there is as yet no general agreement as to the measuring strategies and tools to be implemented for the quantification of occupational exposure. It is therefore not usually possible to apply quantitative risk assessment methods in companies where nanomaterials are handled. **Consequently, the use of qualitative risk assessment methods appears to be a possible alternative.**

Identification and characterisation of hazards

Firstly, all the nanomaterials produced or used in the workplace should be detected, identified and listed. This inventory should be exhaustive and regularly updated.

The material safety data sheet (MSDS) provided by the supplier should then be consulted. However, the information currently provided in most MSDSs, and in particular the toxicological data, does not refer to the nanomaterial.

It is therefore usually necessary to undertake a review of the scientific literature concerning the toxicity of each nanomaterial. It is also necessary to search for information about the toxicity of the parent material, in other words, the same material (identical chemical composition and crystal lattice) at micro- and macro-scales. The review should take into account studies carried out on both cells and animals, and even on humans (especially regarding the parent material). When data is available for micrometre-size or larger materials with the same chemical composition, it is accepted that the corresponding nanomaterials have at least the same toxicity, and are indeed likely to be more hazardous.

A review of the physico-chemical properties of each nanomaterial should also be carried out. It has been clearly shown that the physico-chemical characteristics of nanomaterials have a definite effect on their toxicity. It is therefore necessary to examine size, particle-size distribution, specific surface area, morphology, crystallinity, solubility, surface charge, surface treatment, dustiness, and degrees of aggregation and agglomeration. This information may be found in material safety data sheets, technical data sheets, and also in articles and summary documents in the literature.

The material safety data sheet

A material safety data sheet (MSDS) is provided by the chemicals supplier and comes in addition to labelling. It usually provides much fuller information than the label about risks of all kinds from the products and about the prevention measures that should be complied with when using them. An MSDS is therefore a fundamental tool for risk assessment and a key aid to drawing up job descriptions and exposure records. The REACH Regulation lays down procedures for the drawing up and passing on of MSDS, and Annex II provides guidelines for the drawing up of such sheets.

Currently most information provided in safety data sheets (especially toxicological data and prevention measures) does not refer specifically to nanomaterials but rather to the parent material, in other words, the same material (identical chemical composition and crystal lattice) at micro- and macro-scales. In addition, some important physico-chemical characteristics that provide evidence of the nanometric nature of the materials such as the particle-size distribution and the specific surface area are not usually specified in the MSDS. They may sometimes figure in section 9 of the MSDS.

It is therefore important to remain vigilant when reading MSDSs and strongly encourage manufacturers to provide an MSDS that is appropriate for nanomaterials, even though this is not compulsory.

Exposure assessment

Two approaches, a quantitative approach described in the chapter 'Characterisation of occupational exposure' and a qualitative approach described below, may be envisaged.

When no suitable instruments and measuring methods are available, a qualitative assessment of occupational exposure to nanomaterials should be carried out. To do this, it is important to carry out a thorough observation and analysis of each workstation in order to detect and identify operations that may expose workers to nanomaterials in the workplace.

It is then advisable to collect various data relating to exposure of workers during all operations identified as potentially involving exposure:

- processes in which nanomaterials are synthesised or utilised (liquid or gas phase methods, grinding, etc) and operating methods used;
- the state of the nano-objects being handled: in the form of a powder, liquid suspension (see Figure 17), gel, incorporated into a matrix, etc.;
- the tendency of the nanomaterials to become airborne or settle on work surfaces, i.e. to form aerosols or droplets;
- the amounts manufactured or used;
- the duration and frequency of operations;

- exposure routes for workers: inhalation, ingestion and/or skin contact;
- prevention measures (aimed at reducing exposure) that may have been implemented.

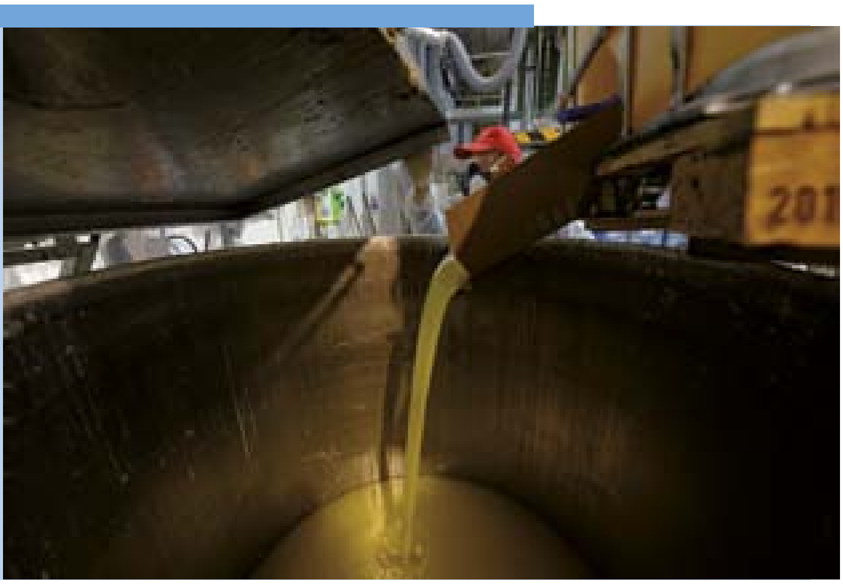

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Figure 17. Transfer of a liquid suspension containing nanoparticles

Qualitative risk assessment

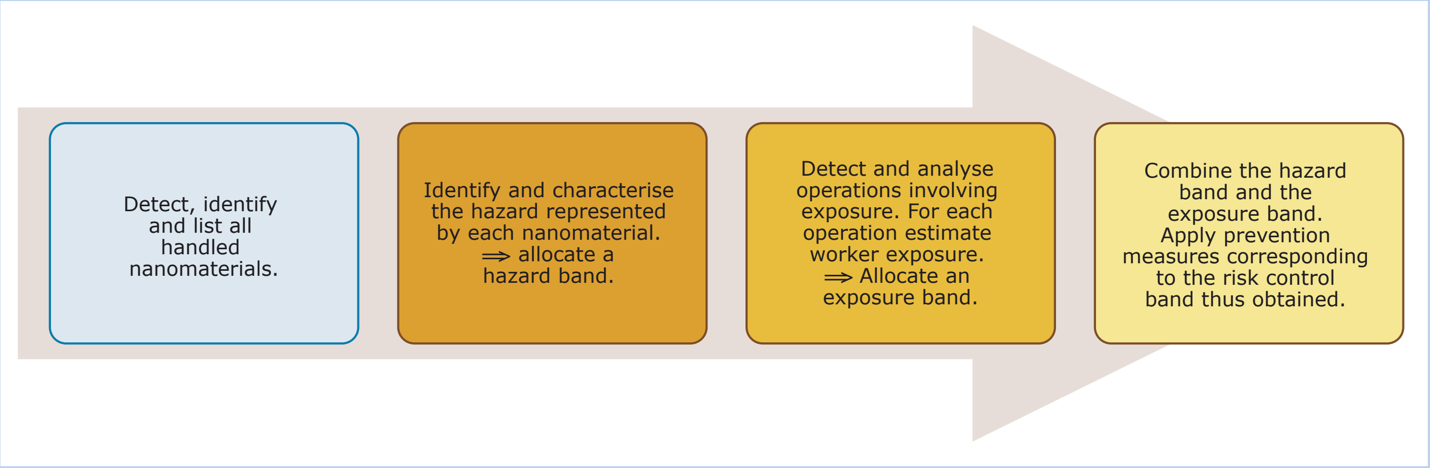
Qualitative risk assessment methods can be used to rank risks and prioritise preventive action. They are based on hazard ratings and exposure factors, defined according to class, and the results are estimated as risk levels.

Among these methods, an approach based on a classification into 'bands' of hazards and exposures (or control banding) can be used (see Figure 18). Control banding is an instrument that combines risk assessment and management. It takes into account existing information and available technical and scientific data, and is based on a certain number of assumptions. It therefore tends to make up for gaps in our knowledge about the toxicity of nanomaterials by taking into account more easily accessible parameters such as the physico-chemical properties (solubility, reactivity, etc) of chemically similar materials (known as parent or analogous materials). Collecting such information enables the nanomaterial in question to be allocated to a hazard band (or level) according to a well-defined procedure that includes several incremental factors. Any operation that potentially causes exposure should be analysed so that it can be allocated to an exposure band (or level), defined for instance according to the physical state of the nanomaterial (solid, liquid, powder, etc), or to the amounts being handled. A risk control band is then obtained by combining the hazard band and the exposure band previously obtained.

The risk control band corresponds to the minimum prevention measures that should be implemented consistent with the estimated risk level. Given that it is necessary to make assumptions about desirable but inaccessible information in order to apply such an approach, it is essential that the user has in-depth expertise in the fields of chemical and nanomaterials risk prevention. Implementing this method without expert knowledge, without a critical eye or without support could lead to inappropriate choices of preventive action for the actual situation.

Knowledge about nanomaterial-related risks is constantly changing. It is therefore crucial to update information regularly and continue to improve the prevention approach on the basis of such new data. As new information becomes available and working methods change, a new assessment should be carried out. The level of uncertainty should eventually fall, and the approach should tend towards an increasingly quantitative risk assessment. It should therefore be seen as an iterative approach that aims to refine risk assessment and determination of necessary prevention measures in the light of further knowledge. **When all the required data becomes available, the use of a qualitative approach should give way to a quantitative risk assessment.**

This information should be entered into the occupational risk assessment document (“document unique”) that should be established and regularly updated. It can also be used to draw up job descriptions.



▲ Figure 18. Principle of the so-called control banding approach to risk assessment and management

Chemical risk prevention regulations

No specific regulations currently govern the handling of nanomaterials in France. However, this does not mean that there is a regulatory vacuum. The general principles relating to the protection of worker's health still apply, as does the legislation applying to the placing on the market of chemicals, pharmaceuticals, cosmetics and foodstuffs.

Nanomaterials are chemical agents. As such, the regulations regarding chemical risk prevention, as laid down in the French Code du Travail, apply to nanomaterials. The chemical risk prevention regulations are based on the general prevention principles defined in article L 4121-2 of the French Code du Travail and are divided into two sections:

- the general chemical risk prevention regulations set forth in articles R. 4412-1 to R. 4412-58 of the Code du Travail;
- the specific chemical risk prevention regulations for activities involving carcinogenic, mutagenic and reprotoxic (CMR) chemical agents (category 1 and 2) defined in articles R. 4412-59 to R. 4412-93 of the Code du Travail.

Consequently, the provisions of labour regulations relating to chemical risk prevention can be used to identify nanomaterial-related risks and to distinguish measures that are specific to carcinogenic, mutagenic and reprotoxic nanomaterials of categories 1 and 2.

The annual declaration of nanomaterials placed on the market in France

Law No 2010-188 of 12 July 2010, known as the 'Grenelle II' law, provides for the implementation of an annual declaration mechanism for 'substances in a nanoparticle state' either as they stand or contained in mixtures without being bonded to them, and for materials likely to release them under normal or reasonably predictable conditions of use. This obligation applies to manufacturers, importers and distributors of such substances placed on the market in France. They must declare the identity, quantities and uses of such substances as well as the identity of professional users who have provided such substances against payment or free of charge. Similarly, they must transmit all available information regarding any hazards relating to these substances and to any exposures they may involve, or which might be useful for the assessment of risks to health and to the environment.

...

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Rules for the implementation of this declaration are laid down in Decrees No 2012-232 and 2012-233 of 17 February 2012. Decree No 2012-232 specifies in particular the definitions ('substance in a nanoparticle state', 'particle', 'aggregate', etc.), and also the minimum threshold above which the annual declaration is compulsory (100 g per year and per substance), as well as the organisation in charge of managing this data (ANSES[3]) and the deadline for providing the information. Provision is also made for interests connected to national defence, respect for industrial and commercial secrecy, and research and development activities. The scheme came into force on 1 January 2013 (declarations to be sent by 1 May 2013). An Order will specify the content and conditions for submission of the declaration. Decree No 2012-233 designates the organisations to which ANSES may transmit the information it holds regarding such declarations.

The scheme aims to improve knowledge of 'substances in a nanoparticle state' and their uses, ensure traceability of the chains of use, improve knowledge of the market and of quantities marketed, and gather information about their toxicological and ecotoxicological properties.

In addition, various regulations have been adopted at European level in order to improve consumer information. They provide that any ingredient present as a nanomaterial in food must be clearly indicated in the list of ingredients (as of December 2014). Similarly, when a nanomaterial is used in the formulation of a cosmetic product it must appear on the label (as of July 2013). Lastly, all biocides containing nanomaterials are subject to a specific authorisation procedure and to labelling indicating the risks related to the nanomaterials used (as of September 2013).

Occupational exposure limits

No occupational exposure limits for nanomaterials are currently defined in French and European legislation. In France, there are occupational exposure limits for various categories of dust: dust considered to be without any specific effect, titanium dioxide, non-fibrous graphite, certain metal oxides and salts, etc. However, these exposure limits are not applicable, as such, to nano-substances.

In 2011, NIOSH[4] established two occupational exposure limits for titanium dioxide: 2.4 mg/m^3 for fine titanium dioxide and 0.3 mg/m^3 for ultrafine titanium dioxide (particles with a diameter below 100 nm). In 2013, NIOSH also proposed an exposure limit for carbon nanotubes of 1 µg/m^3. Other organisations such as BSI[5] and IFA[6] define benchmark exposure levels distinguishing between certain categories of nano-objects: fibres, CMR, insoluble and soluble nano-objects, etc. These two institutes point out that the proposed values aim to reduce exposure of workers according to the state of the art. They also make it clear that the values are not toxicologically justified, and that complying with them is no guarantee against development of a pathology.

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[3] Agence nationale de sécurité sanitaire de l'alimentation, de l'environnement et du travail.

[4] National Institute for Occupational Safety and Health.

[5] British Standards Institution.

[6] Institut für Arbeitsschutz der deutschen gesetzlichen Unfallversicherung: http://www.dguv.de/ifa/Fachinfos/Nanopartikel-am-Arbeitsplatz/Beurteilung-von-Schutzmaßnahmen/index-2.jsp.

Currently there is not enough knowledge about nanomaterial toxicity to enable occupational exposure limits to be established. Whatever the operation carried out, it is therefore advisable to seek the lowest possible exposure level.

The IARC[7] classification of carbon black and titanium dioxide

Due to the recent nature of the manufactured nanomaterials sector, there is currently no published epidemiological study of populations of exposed workers. In older industries such as the titanium dioxide and carbon black industries, several morbidity and mortality studies have been carried out. In February 2006, IARC published the results of reassessments of the potential carcinogenicity of carbon black and titanium dioxide in their nano- and micrometre-forms. For carbon black, it confirmed the categorisation established in 1996, namely that it is possibly carcinogenic to humans (category 2B), while for titanium dioxide it modified the category established in 1989, changing it from category 3 (not classifiable as to carcinogenicity in humans) to category 2B. Neither substance is classified by the European Union.

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▲ Figure 19. Carbon black is incorporated as a filler into tyres

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[7] International Agency for Research on Cancer.

Risk prevention

The prevention approach

Manufactured nanomaterials make up a new family of chemicals that show many differences in terms of chemical composition, physico-chemical properties, toxicological profiles and dimensional characteristics.

The control strategies and best working practices that need to be implemented in companies and laboratories should therefore be developed on a case-by-case basis. They should enable exposure of workers to be reduced to the lowest possible level. Given that knowledge about nanomaterial toxicity is still limited, **risk prevention should principally be based on limiting occupational exposure** (exposure level, duration of exposure, number of exposed workers, etc).

Specifically, safe and appropriate working practices should be defined and implemented according to the results of the risk assessment. These practices are likely to evolve as and when reliable information is published about the hazards of nanomaterials for health and safety. Although such safe practices are not very different from those recommended for any activity involving exposure to hazardous chemicals, they take on special importance given the strong tendency of nanomaterials to persist and propagate in the workplace atmosphere (aerosolisation and dispersion).

Particular attention should be paid to nanomaterials for which there is little toxicological data or for which early research has shown toxic effects, particularly on animals.

The general outline of the prevention approach as laid down by the *Code du Travail* comprises six stages:

- identify the hazards related to the nanomaterial(s);
- avoid risks, where possible by eliminating them;
- assess unavoidable risks to health and safety in the workplace, according to processes applied and working methods (estimate the nature and extent of risks);
- set up measures aimed at preventing or limiting risks (prefer collective protection measures to personal protection measures);
- verify the effectiveness of measures taken;
- provide training and information to workers.

The main steps in the prevention approach are as follows:

- modify the process or activity so that nanomaterials are no longer produced or used;
- replace the nanomaterial(s) by substances that are less harmful to health;

- optimise the process to obtain the lowest possible dust level in order to limit exposure: prefer closed systems, mechanised processes and automated operations;
- capture pollutants at source: install local exhaust ventilation;
- filter air before discharge outside the workplace;
- use personal protective equipment if collective protection measures prove insufficient;
- collect and dispose of waste;
- train and inform exposed workers about potential risks and prevention measures in the present state of knowledge: provide workers with the information they need to carry out their work under optimal safety conditions;
- ensure traceability of worker exposure, i.e. note down and keep any relevant information regarding their exposure: types of nanomaterial handled, quantities involved, operations and tasks carried out, prevention measures implemented, etc;
- analyse and learn from any incidents or accidents that occur.

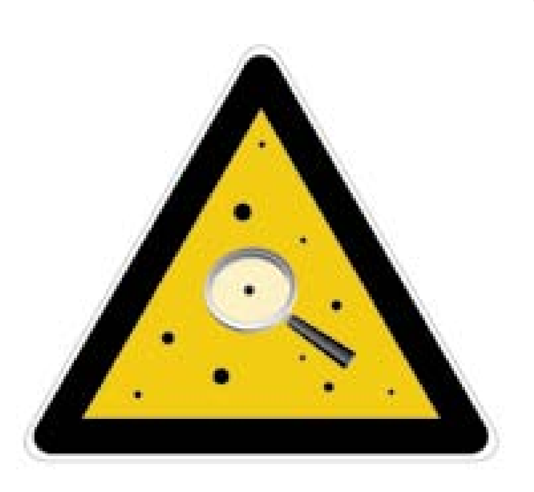
The working area

The working area should be clearly signposted and demarcated. Pictograms, or graphical symbols, can be put up at the entrance to working areas, indicating for example: 'Risk of exposure to nanomaterials'. There is no standardised pictogram in France or Europe. INRS suggests using the symbol shown in Figure 20.

It is advisable to limit the number of workers who are exposed or likely to be so. Access to the working area should be restricted solely to workers directly involved in the production or use of nanomaterials, as well as to cleaning and maintenance staff. These workers should have received appropriate training.

Passageways between areas that may involve exposure to nanomaterials to so-called 'clean' areas should be equipped with the necessary facilities for changing personal protective equipment. The installation of double locker rooms adjoining the activity area should be considered to avoid mixing street clothes and work clothes, and to avoid any risk of contamination outside the working areas.

Floors and work surfaces should preferably be smooth, impervious and non-porous. They should also be easy to clean and resistant to chemicals, mechanical load and shock.



▲ Figure 20. Sample pictogram 'Risk of exposure to nanomaterials'

Substitution / modifying /optimising processes

In the case of nanomaterials, which are generally used because of the unique properties that they impart to the products in which they are incorporated, the substitution approach principally consists in optimising or modifying processes and operating methods:

- handling nanomaterials in the form of a liquid suspension, gel, aggregate or agglomerate state, or pellets, or incorporated into a matrix rather than in powder form so as to limit the formation of nanoaerosols (see Figure 21);
- preferring production methods in the liquid phase to gas phase techniques or mechanical methods;
- modifying facilities for continuous rather than batch production;
- eliminating or restricting certain critical operations such as transfer, weighing, sampling, etc;
- optimising processes so as to use smaller quantities of nanomaterials;
- installing reliable, regularly maintained facilities.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▲ Figure 21. Handling nanoparticles in liquid suspension form

Collective protection

Using a closed circuit process

The production of nanomaterials and particularly of nanopowders (especially in cases where the material is fibrous, carcinogenic, mutagenic or reprotoxic) requires the complete isolation of the process. A closed circuit process combined with mechanisation or automation of the process should be considered and implemented where the context permits, in order to limit the presence and therefore exposure of workers. An enclosed system allows the complete confinement of the production and use of nanomaterials. All contact between workers and nanomaterials is thus avoided. A closed circuit process usually requires mechanisation of the process or even the automation of certain tasks: transfer of substances by mechanical or pneumatic means, mechanised sampling, washing reaction vessels without opening them, etc. Mechanisation makes it possible to eliminate handling between different stages of the process, as well as ensuring unbroken confinement. Automation makes it possible to avoid exposure of workers during certain critical tasks in processes which generally generate aerosols or droplets, such as bagging, transfer, etc.

Ventilation

When a closed circuit process is not required or is not technically feasible (bulky equipment, unsuitable premises, etc), it is advisable to install ventilation, and in first place local exhaust ventilation (see Figure 22). Since transport of nanoaerosols is mainly caused by airflow, ventilation is the best way to keep air clean in the workplace. It helps to keep the concentration of manufactured nanomaterials present in the workplace atmosphere at the lowest possible level.

Local exhaust ventilation should comply with nine simple principles:

- cover the nanomaterials production area to the greatest extent possible;
- capture pollution as close as possible to the emission area;
- place the system in such a way that the worker is not between it and the pollution source;
- make use of the natural motion of pollutants;
- ensure that there is sufficient air velocity;
- ensure uniform distribution of air velocities at the capture area;
- replace air output with equivalent air input;
- avoid draughts and perceived thermal discomfort;
- filter polluted air and discharge it outside away from areas of fresh air input.

Local exhaust ventilation systems should be specifically adapted to the size and type of operations carried out and versatile enough to respond to their diversity.

As well as local exhaust ventilation, it is strongly recommended to set up a general ventilation system operated by mechanical means (see Figure 22).

The aims of general ventilation are to:

- provide fresh air to replace the air extracted by source capture systems;
- provide fresh air to workers;
- ensure the elimination of residual pollutants not directly captured at source, through air exchange.

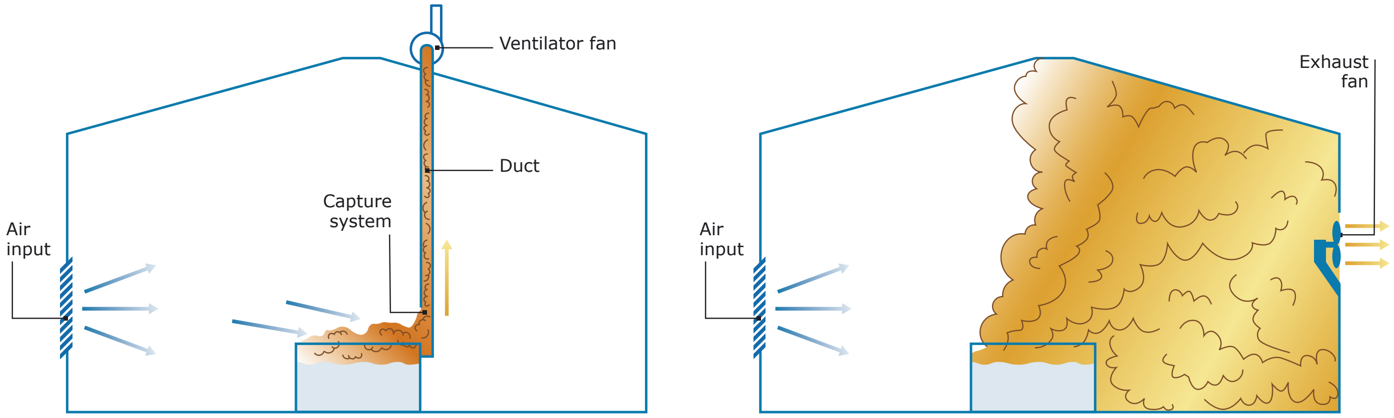
General ventilation alone is not a satisfactory means of prevention because:

- it works by dilution and causes dispersion of the pollutant throughout the workplace, with a risk of it building up in certain poorly ventilated areas;
- it requires the implementation of high flow rates;
- it does not immediately protect workers.

It is important to ensure that general ventilation does not interfere with local exhaust ventilation.

Nanoaerosols have certain specific features that need to be taken into account when designing clean-up systems:

- a significantly higher deposition rate;
- rapidly changing particle size due to agglomeration.



▲ Figure 22. Local ventilation (left) and general ventilation (right)

Agglomeration can occur either through self-agglomeration of nanoparticles or through agglomeration of nanoparticles on larger particles, such as those present in natural atmospheric aerosols or resulting from other processes taking place in the workshop or laboratory.

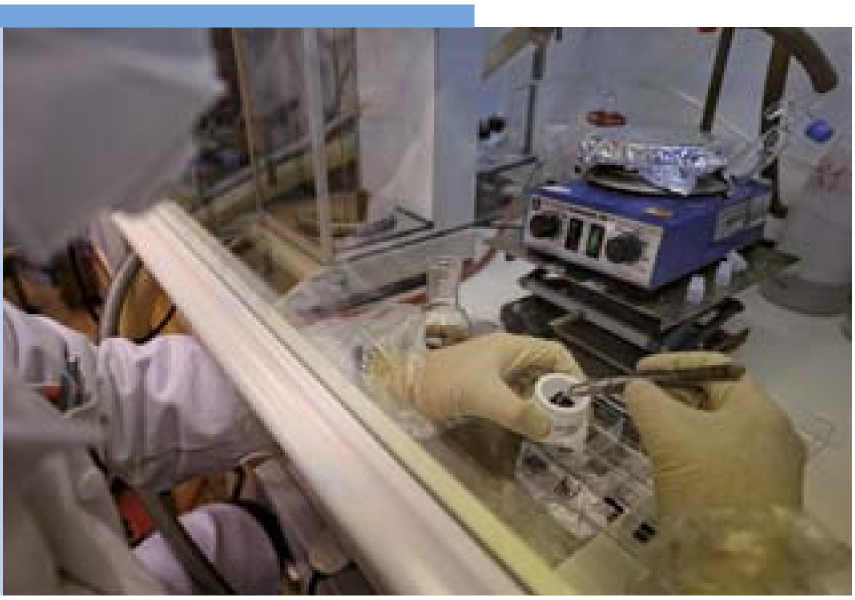
The specific features mentioned above lead to the following recommendations concerning the choice of **local ventilation systems** to install:

- for a given capture efficiency, systems that minimise exposed surfaces should be used;
- in the airflow pathway, it is advisable to insert an appropriate filtering medium as close as possible to the pollution source in order to limit deposition in the exhaust ducts.

Extracted air should be discharged into the outside atmosphere after undergoing prior filtration. Ventilation systems should be connected to a centralised ventilation network with a common ventilator fan and exhaust duct.

1. In laboratories, the preferred local ventilation systems are enclosed systems. Handling of nanomaterials in enclosed, ventilated systems prevents their dispersion throughout the laboratory atmosphere. Several ventilated systems may be used:

- fume hoods: it is recommended to use fume hoods with a face velocity of between 0.4 and 0.6 m/s (see Figure 23). The use of recirculating fume hoods, formerly known as ETRAF (Enclosure for Toxics using Recirculating Air Filtration), should be avoided, since in this case the filtered air is recycled in the laboratory;



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◀ Figure 23. Handling carbon nanotubes in a laboratory fume cupboard

- Class II microbiological safety cabinets (MSC) and laminar flow systems: recently, class II MSC laminar flow systems specifically designed for nanomaterials have become available. If protection of the product is not required, class I microbiological safety cabinets can also be used;
- glove boxes and isolators: due to air stagnation in the glove box and to the tendency of nanomaterials to be deposited on surfaces, major contamination of the glove box interior is to be expected. It is therefore important to remain vigilant if leaks occur and during cleaning and maintenance operations.

2. In workshops, local exhaust ventilation systems should be placed as close as possible to the point of emission and should be adapted for the specific process. Ideally, operations requiring the handling of nanopowders and which therefore particularly lead to exposure (formulation, weighing, depackaging, etc) should be carried out in rooms or booths which are depressurised in relation to the rest of the workplace and equipped with local exhaust ventilation.

Local exhaust ventilation systems which have proved effective for the capture of gas and vapours should prove effective for the capture of nanoaerosols, as long as the input for the capture system is well located and a suitable capture velocity is constantly maintained: between 0.4 and 0.5 m/s at the emission point.

The effectiveness of such local exhaust ventilation systems is closely related to their design and sizing, to the effective replacement of extracted air, and also to their upkeep and to working methods. In addition, they should be checked regularly, especially with regard to air flow.

3. On work sites, the use of hand-held power tools (saws, drills, etc) equipped with integrated pollutant capture systems and with High Efficiency Particulate Air Filters is recommended, for example for machining of nanocomposites.

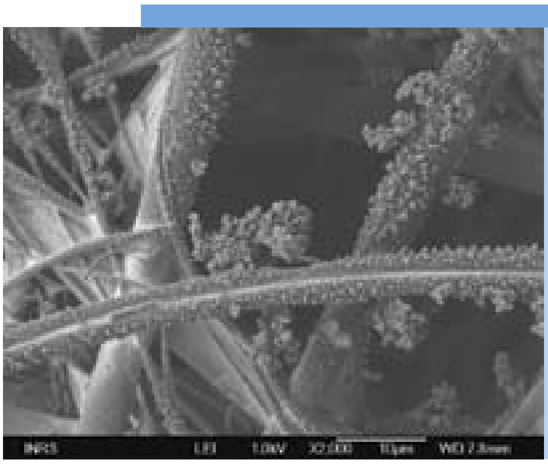
Filtration

The air of workplaces where nanomaterials are manufactured or used must be filtered before being discharged outside. Filtration by means of fibrous media (filters made of synthetic, metallic or natural fibres) is still the most common method because it is efficient, cheap and highly adaptable. Filtration is the result of complex interactions between an aerosol and the fibres in the filter. This complexity is increased due to the fact that the efficiency of the medium changes during the filtering process (filter clogging).

When new, a fibrous filter is characterised by its pressure drop (difference in pressure between upstream and downstream of the filter) and by its so-called initial efficiency (ratio of the difference between upstream and downstream concentrations to the upstream concentration).

In aerosol filtration, a widespread error consists in assuming that capture of particles by a fibrous filter is only due to a sieve effect, in other words, the particles collected are larger than the size of the filter pores. However, it turns out that, in the absence of an electric field, particle capture by a fibrous filter is actually dependent on several physical mechanisms. With regard to nanoparticles, the main collecting mechanism is Brownian diffusion. The smaller the particles, the more significant this mechanism is. Particles with a diameter of less than 100 nm are constantly subject to Brownian motion caused by their interaction with air molecules, which are themselves subject to thermal agitation. This random motion increases the probability of collision of particles with a diameter of less than 100 nm with the fibres in the filter.

A large number of expressions, both empirical and theoretical, can be used to estimate the efficiency of a fibrous filter by Brownian diffusion. They all converge and, in accordance with experimental evidence, show that the efficiency of fibrous filters increases as particle size becomes smaller. This conclusion has been experimentally and theoretically validated for particles down to 1 nm. Below this size, the detection limits of instruments are reached. In addition, for nanoparticles, filtration efficiency decreases with increasing filtration velocity. Fibrous filters are therefore an effective barrier to nanomaterials (whatever their morphology: nanoparticle, nanofibre, etc.) whose size exceeds 1 nm (see Figure 24).



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▲ Figure 24. Copper nanoparticles collected on the fibres of a filter (seen through a scanning electron microscope)

With regard to the protection of people, the workplace and the environment, the use of High Efficiency Particulate Air Filters (HEPA filters) of class H 14 or above according to standard EN 1822-1, is recommended. Premises, source capture systems, etc. should therefore be equipped with High Efficiency Particulate Air Filters (HEPA filters) of class H 14 or above. For moveable filtration devices (industrial vacuum cleaners), standard EN 60335-2-69 applies, and class H systems should be used when handling nanomaterials.

During upkeep and maintenance of filtration facilities, workers must wear personal protective equipment. The use of compressed air guns, brooms and brushes should be strictly prohibited during such work. Contaminated filters should be considered as nanomaterial waste and treated as such.

If gaseous pollutants are also emitted, these should be removed (e.g. by using activated carbon filters) in addition to filtration of nanoparticle pollutants.

Clogging of fibrous filters

Clogging causes increased pressure drop and decreased efficiency of filters over time (except for electrically charged filters). The increase in pressure drop causes increased resistance to air flow, which leads to a lower flow rate for industrial facilities (and breathing difficulties when wearing respiratory protective equipment).

It has been shown that nanomaterials have high clogging capacity. According to feedback from industry, it is also extremely difficult to unclog cartridges when inside dust extractors.

To slow down clogging it is recommended, at high concentrations, to reduce filtration velocity. This is done by increasing the filtering area by a factor of 10 in certain cases. In front of the High Efficiency Particulate Air Filters it is also advisable to place coarser filters, called prefilters, which retain the largest particles.

Personal protection

The choice of personal protective equipment should result from the best possible compromise between the highest attainable level of safety and the need for workers to carry out their task in maximum conditions of comfort. All personal protective equipment should be kept in good condition and, where non-disposable, cleaned after each use.

Respiratory protection

As soon as ventilation of the workplace atmosphere proves insufficient, workers must wear respiratory protective equipment, taking into account that nanometre-scale objects can pass through any gap (the facepiece must fit tightly against the face and there must be no leakage).

The efficiency of the filtering media that equip respiratory protective equipment depends on the nature of the medium, the aerosol and the filtering conditions. According to the classical theory of filtration, the efficiency of anti-aerosol filters is similar to that of filters used in workplace and environmental protection. The effectiveness of anti-aerosol filters thus tends to increase as particle size decreases.

For work involving little exposure (transfer of a liquid suspension, maintenance of a pump, etc) and where ambient air contains enough oxygen (minimum 19 % by volume) it is recommended that respiratory protective equipment with “anti-aerosol” filters be worn. Where operations are of short duration, an half mask or full face mask equipped with a class 3 filter can be used (see Figures 25 and 26) (facepiece equipped with a P3 filter in accordance with standard EN 143 or possibly an FFP3 disposable filtering facepiece in accordance with standard EN 149). Where work is likely to last more than one hour, it is advisable to wear powered air-purifying filtering respiratory protective equipment, and more specifically a powered filtering half mask (TM2P), full face mask (TM3P), or hood (TH3P) in accordance with standards EN 12942 and EN 12941. Standard powered filtering respiratory protective equipment works with an air flow of 120 l/min. It is recommended to use powered filtering equipment providing an air flow of 160 l/min to ensure that positive pressure is maintained inside the equipment.


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▲ Figure 25. Half mask equipped with P3 filters


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▲ Figure 26. Full face mask equipped with P3 filters

For work involving exposure (transfer or depackaging of nanopowders of unknown toxicity for example), it is recommended to wear a breathing apparatus, more specifically a compressed airline mask, hood or suit.

The protective efficiency and correct conditions of use of the equipment used should be checked in actual use and over time (saturation, wear, etc).

Skin protection

There is still little in the current literature about the effectiveness of chemical protective clothing regarding nanomaterials. Nonetheless, on the basis of initial data, it is recommended to wear type 5 chemical protective clothing (protection against chemicals in the form of solid particles) in Tyvek® (see Figure 27). It is therefore advisable to wear disposable clothing, especially disposable coveralls (or coat) with elasticated neck, wrists and ankles, without folds or turn-ups, and with flapped pockets. It may also be advisable to wear Tyvek® gauntlets.

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▲ Figures 27. Type 5 disposable coveralls, TM3 P powered air-purifying filtering full face mask and gloves

Similarly, tight disposable plastic gloves (nitrile, vinyl or neoprene) as well as goggles providing side protection should be worn. According to initial research, nitrile, butyl or vinyl gloves appear to be an effective barrier against nanoaerosols (although data about the effectiveness of gloves regarding suspensions and powders is very incomplete). In the event of extended and repeated skin exposure, it is advisable to wear two pairs of gloves or thicker gloves.

The use of shoe covers is also necessary in order to avoid contamination of areas outside the workplace.

Keeping workplaces and facilities clean

Facilities and workplaces should be free from any build-up of deposited nanomaterials so as to avoid airborne resuspension.

Consequently, facilities, floors and work surfaces should be regularly and thoroughly dusted and cleaned with a damp cloth and a vacuum cleaner equipped with High Efficiency Particulate Air Filters of class H 14 or above (see Figure 28).

The vacuum cleaner should be used exclusively for this purpose and be clearly identified, e.g. on the upper part, by a label such as "Only to be used for nanomaterials". After each period of use, it is important to vacuum the outside of the cleaner and all its accessories and to leave it in operation long enough to empty the tubing. Vacuum cleaner bags and filters containing nanomaterials should be regularly and carefully replaced. For any type of operation that requires the vacuum cleaner to be opened, workers must imperatively wear respiratory protective equipment, coveralls (or coat), gloves and goggles. It is important to ensure that the vacuum cleaner is leak free and in good working order at all times. The vacuum cleaner must also comply with ATEX[8] requirements if it is to be used in an area where there is an explosion risk.

When workplaces and facilities are cleaned, workers should wear personal protective equipment: respiratory protective equipment, coveralls (or coat), gloves and goggles.

Vacuum cleaner bags and filters as well as cleaning cloths should be treated as nanomaterial waste.

The use of compressed air jets (compressed air guns), brushes, brooms and household-type vacuum cleaners must be avoided, whether for regular cleaning of facilities and work premises or in case of accidental spillage.

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▲ Figure 28. Vacuum cleaner equipped with High Efficiency Particulate Air Filters

Personal hygiene

Work clothes (when not disposable) and street clothes should not be mixed. Soiled clothes, especially work clothes, must not be taken home and should be cleaned bearing in mind the risks connected to possible contamination by nanomaterials. The company in charge of cleaning the clothes should be informed.

To avoid the ingestion of nanomaterials, eating and drinking should be banned in the workplace. Areas strictly reserved for such use should be set aside and kept clean.

Washbasins and showers should be systematically used before leaving the work premises for the decontamination of any skin areas (hands, forearms, etc) that may have been exposed to nanomaterials during experimental work.

Storage of products

The storage of nanomaterials has specific features due to the size distribution characteristics and surface reactivity of nanomaterials. The tiny diameter of the materials increases settling time and facilitates resuspension.

Nanomaterials should always preferably be stored in a central facility. Buffer storage should be reduced to the minimum, while unauthorised storage should be systematically prohibited.

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[8] Explosive atmospheres.

The central storage facility should be entirely dedicated to nanomaterials and identified as such. If this is not possible, a storage area exclusively dedicated to nanomaterials should be set up in the central facility and clearly identified, for instance by means of a warning sign “Nanomaterials exposure risk”. The storage facility should be easily accessible, thus allowing rapid evacuation in the event of an incident or accident. It should be closed outside working hours. Access to the facility should be restricted to specially designated and trained persons. A register of stored nanomaterials should be kept up to date, as well as a storage map showing the precise location of products (in addition, incompatible products should be physically separated). A procedure for the disposal of unwanted (or obsolete) nanomaterials should also be drawn up. Spillage and leakage containment facilities should also be provided.

The central storage facility should be equipped with mechanical ventilation. The floor and walls should be smooth (without joints), resistant to the stored products, and impervious. Like the shelving, they should also be easy to clean. Where justified by the risk, they should conduct static electricity in order to avoid build-up of electric charge. An absorbent to be used in the event of leakage and drippage, cleaning cloths, and a vacuum cleaner equipped with High Efficiency Particulate Air Filters should be available in the facility. Personal protective equipment should also be ready-to-hand near the entrance to the facility.

It may be advisable to implement a procedure for storage in a controlled atmosphere (in nitrogen for instance) when handling certain nanopowders (aluminium, magnesium, lithium, carbon nanotubes, etc).

Nanomaterials, whether produced or utilised (once depackaged), should be stored (and transported) in tightly sealed and preferably rigid airtight containers, such as cans, tanks, bottles, vats, flasks, etc. They should bear a label indicating the presence of nanomaterials (see Figure 29) such as "Contains nanomaterials", as well as the chemical composition and regulatory labelling. Where nanomaterials are stored in plastic bags, the use of double packaging is strongly recommended. The plastic bags can then be placed either in an airtight, labelled container, or in another airtight, labelled plastic bag.

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▲ Figure 29. Example of a label indicating the presence of nanomaterials

Transport of nanomaterials outside the workplace

In the absence of specific regulations governing the transport of nanomaterials, it is advisable to apply the rules in force relating to the transport of dangerous goods and refer to the following regulations: ADR[9] (road transport), RID[10] (rail transport), IATA[11] (air transport) and IMDG[12] (maritime transport). Transport of nanomaterials by mail should be avoided.

Waste management

Nanomaterial waste must be **treated as hazardous waste.**

Waste receptacles must be closed (and ventilated if so required by the risk assessment) and clearly identified ("Receptacles reserved for nanomaterials"). They should be placed in close proximity to areas where nanomaterials are handled (if possible, next to each work station so as to limit transport of waste in the workplace).

The following should be treated as nanomaterial waste:

- products that do not meet required production criteria, residues, samples, etc. whether in solid or liquid form (nano-objects in the form of a liquid suspension, powder, gel or incorporated into a matrix);
- empty product containers and packaging (see Figure 30);
- cleaning liquids;
- filters for ventilation facilities;
- vacuum cleaner bags and filters;
- disposable personal respiratory and skin protective equipment (coveralls, coats, filtering facepieces, etc);
- contaminated cleaning cloths and absorbent paper.

Nanomaterial waste should be packaged in closed, airtight packaging. They should bear a label indicating the presence of nanomaterials, for instance "Contains nanomaterials".

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▲ Figure 30. Empty containers should be treated as nanomaterial waste

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[9] European Agreement concerning the International Carriage of Dangerous Goods by Road.
[10] European Agreement concerning the International Carriage of Dangerous Goods by Rail.
[11] International Air Transport Association.
[12] International Maritime Transport for Dangerous Goods.

Solid products, filters, disposable personal protective equipment, etc. should be packaged in airtight labelled plastic bags. It is strongly recommended to doubly wrap such products (this is essential if nanopowders are present). The plastic bag can then be disposed of either in an airtight, labelled container, or in another airtight, labelled plastic bag.

The packaged waste should then be removed to an appropriate storage facility that meets the same criteria as a central storage facility for nanomaterials, before being taken away and processed. The facility should be large enough for the creation of a specific storage area for nanomaterial waste (provided with spillage containment facilities). Only packaged, labelled waste should be stored there.

The packaged waste should than be taken to a suitable waste disposal or treatment facility: either a class 1 storage facility (hazardous waste), an incinerator (up to 1,000 °C), or a cement kiln (up to 1,850 °C).

Waste collection and treatment companies should be informed about the presence of nanomaterials.

Upkeep and maintenance of facilities

Regular upkeep and maintenance of equipment and facilities cut down the risks of unplanned disruptions, malfunctions and accidental release (leakage).

Such operations should be planned and organised so as to avoid co-activity. During such work, access to the workplace should be strictly restricted to upkeep and maintenance staff. Company staff should be informed by means of a notice posted on the door indicating for example “Restricted access: Maintenance work / Decommissioning work in progress”.

The first stage of the operation should consist in careful dust removal and cleaning of the equipment and facilities concerned. This should be done using damp cloths and a vacuum cleaner equipped with High Efficiency Particulate Air Filters. The use of compressed air guns, brooms, brushes and household-type vacuum cleaners must be avoided.

After cleaning, plastic sheeting may be placed on adjacent work surfaces and the surrounding floor area, thus facilitating decontamination of the area when the work is terminated.

Workers taking part in the operation, whether belonging to the establishment or to a subcontractor (in which case a prevention plan should be drawn up), should be informed about the presence of nanomaterials and trained in risks and appropriate prevention measures. They should be equipped with respiratory protective equipment, coveralls, gloves and goggles (see Figure 31).

When the work is terminated, floors and (unprotected) work surfaces should again be dusted using damp cloths and a vacuum cleaner equipped with High Efficiency Particulate Air Filters. Tools that have been in contact with nanomaterials should also be cleaned before being put away. Soiled plastic sheeting should be considered as nanomaterial waste and treated as such.

Figure 31. ▶ Maintenance of a facility in a laboratory producing nanomaterials

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Incident and accident management

Response procedures in the event of accidental release (leakage) and spillage should be drawn up and distributed to workers. Incident and accident scenarios should be defined and regular drills held where necessary. The goals of response procedures are to:

- alert the emergency services (internal and/or external depending on the scale of the event);
- identify the area affected by incidents or accidents of varying severity (all or part of the workplace);
- set up controlled access to the contaminated area;
- ensure availability of appropriate personal protective equipment for any person required to enter the affected area: in particular, workers should wear respiratory protective equipment and skin protective equipment;
- describe cleaning of contaminated equipment and facilities (floors, walls, equipment, etc.) using systems appropriate to the nature and amount of spilt products: depending on the type of incident (liquid or solid spillage), it is advisable to use a vacuum cleaner equipped with High Efficiency Particulate Air Filters and/or damp cloths.

An incident and accident register must be kept up to date. A thorough analysis of every incident and accident must be undertaken to avoid it happening again and if necessary to take prevention measures.

Workplaces must be equipped with an easily accessible first aid kit. In particular, eyewash fountains and safety showers should be installed.

Explosion and fire prevention

To prevent an explosion or fire from taking place, it may prove necessary to develop specific prevention measures and working methods.

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It may be advisable to implement a procedure for synthesis or storage in a controlled atmosphere (in nitrogen for instance).

It is also advisable to:

- restrict certain operations likely to generate an aerosol, such as transfer of nanopowders;
- regularly vacuum clean facilities, floors and work surfaces in order to avoid any deposition or build-up of nanomaterials that might undergo airborne resuspension (avoid sweeping and use of compressed air guns);
- limit the formation of electrostatic charge by earthing the conductive parts of facilities used;
- replace flammable or reactive products;
- enclose energy sources and keep open flames away.

Information and training

Information and training for workers should meet the following objectives:

- provide workers in contact with nanomaterials with the fullest possible understanding of the health and safety risks they are exposed to;
- train them to implement collective prevention measures;
- train them in the use (wearing, removal and upkeep) of personal protective equipment made available to them.

Content should be modular and appropriate to the target audience and to the specific conditions of the company. **At the very least, the following topics should be dealt with:**

- definitions (nanomaterials, hazards, exposure, risk, labelling, Material Safety Data Sheets, etc.);
- the regulatory context;
- health hazards (health effects) and safety hazards (fire and explosion);
- organisational measures;
- collective protective measures implemented, their role, use and maintenance;
- personal protective equipment, its role, use and maintenance;
- best working practices;
- cleaning and waste management procedures;
- hygiene measures;
- measures to be taken in the event of an incident or accident.

Training is the responsibility of the employer, i.e. the director of the establishment. It may be prepared by managers, with the participation of the medical department, preventionists and the *Comité d'Hygiène, de Sécurité et des Conditions de Travail* (CHSCT) (or the staff representatives).

The training can be imparted by managers, the safety officer or any other person with the necessary expertise, in consultation with the medical department.

Traceability of training should also be ensured.

Given the recent nature of nanomaterials and the gaps in our knowledge about their harmfulness and the effectiveness of protective means, it will be necessary to **regularly update and renew information and training**.

Medical surveillance

Given the current medical uncertainty about the health effects of nanomaterials, there is at present no consensus about the content and methods of medical surveillance of workers potentially exposed to nanomaterials.

On the individual level, follow-up should be adapted according to the circumstances of medical consultations. It is essential to lay stress on the risks and on those aspects of technical prevention that enable exposure to be limited.

In the absence of any clear evidence for health effects related to occupational exposure to nanomaterials, prescription of tests such as chest X-rays, pulmonary function tests and electrocardiograms, and the interpretation of the results, remain controversial and limited. However, such tests, which should be decided by the occupational physician, offer the advantage of constituting a baseline checkup for newly hired workers, and an aid to determining their aptitude for jobs that require them to wear heavy-duty personal protective equipment. Such tests can be repeated as part of a longitudinal follow-up of personal health parameters.

Keeping a record and ensuring traceability of all the information collected concerning health events, results of tests and exposures, including accidental ones, is of fundamental importance. The information should be kept in worker's personal medical records.

The modalities of medical follow-up should be adapted in the light of further knowledge, and especially the results of epidemiological studies carried out on potentially exposed workers.

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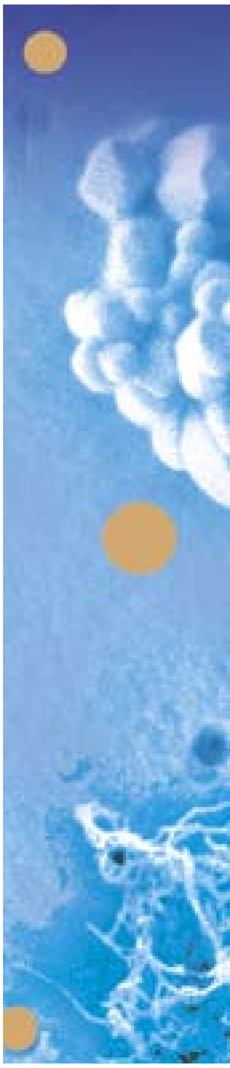
The huge budgets and almost unlimited expectations placed in the production and use of nanoparticles and nanomaterials in a wide range of industry sectors have already resulted in many industrial achievements, demonstrating that occupational exposure to nanoparticles is today a reality. Because of the many uncertainties relating to these novel chemical products, to their potential health effects and to the difficulties encountered in characterising exposure, quantitative risk assessment turns out, in the majority of work environments, to be difficult to implement. It is therefore advisable, in all occupational environments making use of nanomaterials (businesses, research laboratories, universities, etc.) and all the way through the life cycle of products, to adopt a precautionary approach and set up specific risk prevention procedures. Although it is too soon to provide definitive answers, this document sets out to review the characteristics and applications of nanomaterials, current toxicological knowledge, tools for characterising occupational exposure, and lastly, preventive measures.



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