FREQUENTLY ASKED QUESTIONS

Nanotechnology and Engineered Nanomaterials

A Primer

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Introduction

In recent years there has been ever increasing activity and excitement within the scientific and engineering communities, driven heavily by government investment, about engineered nanotechnology applications. The U.S. National Science Foundation has estimated that the global nanotechnology market could be worth U.S.$1 trillion by 2015.[1] In parallel, much has been written and presented about the excitement and possible dangers of these materials. The tone of these media articles range from how these wonder materials are going to revolutionize all aspects of our lives to how they might kill us! The purpose of this primer is to provide some basic information about engineered nanomaterials so that you will be better informed, understand the new 'jargon' and appreciate some of the potential new applications of these materials. In addition, understanding the wide range and types of measurements needed to characterize these nanomaterials along with what solutions PerkinElmer has to support customers working in this field are outlined.

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What is nanotechnology?

Nanotechnology is the science and technology of precisely manipulating the structure of matter at the molecular level. The term nanotechnology embraces many different fields and specialties, including engineering, chemistry, electronics, and medicine, among others, but all are concerned with bringing existing technologies down to a very small scale, measured in nanometers.[2] Processes and functionality take place at the nanoscale, exhibiting properties not available in the bulk material. But what is a nanometer? Figure 1 compares the nano-region to things we know, such as a pin, insect and cells and provides a visual perspective.

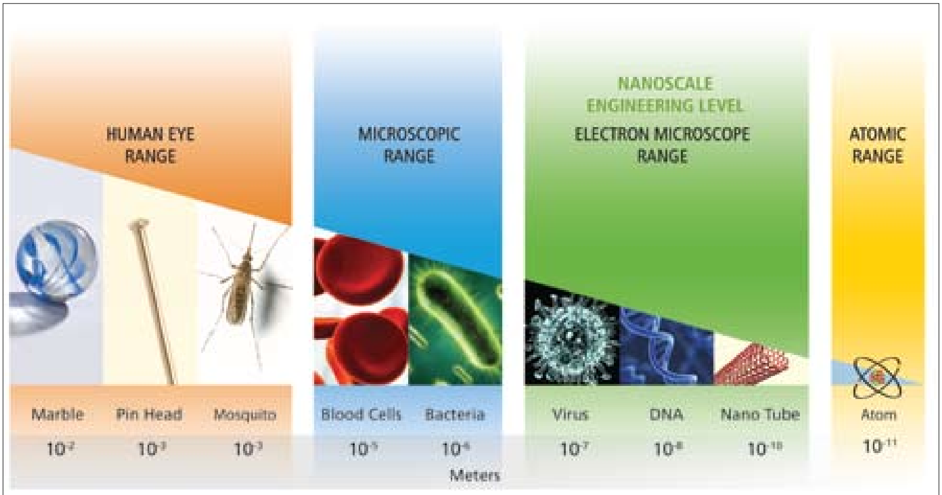

Figure 1. Size relationships from large to small to nano.

A nanometer is a thousandth of a micron and a micron is a thousandth of a millimeter, so a nanometer is a millionth of a millimeter or 10^{-9} meters. To be classified as a nanomaterial (NM), the material must be less than 100 nm in size in at least one direction. According to the International Standards Organization® (ISO) a nano-object is a material with at least one, two or three external dimensions in the nanoscale range of 1 to 100 nm and a nanoparticle is a nano-object with all three external dimensions in the 1 to 100 nm range and showing a property not evident in the bulk material. Hence, a nanofiber, 400 nm long and 12 nm in diameter, and a 20 nm diameter nanoparticle, are both classified as nanomaterials.[3]

Even though ISO does not distinguish between engineered nanoparticles and naturally occurring nanoparticles, you should be aware that there are naturally occurring nanoparticles in the aquatic environment such as biodegraded organic matter and colloidal inorganic species and in soils; clays, organic matter and various metal oxides.[4] Many important functions of living organisms take place at the nano-scale. The human body uses natural nanoscale materials such as proteins and other molecules, to control the body's many systems and processes. A typical protein such as hemoglobin, which carries oxygen through the bloodstream, is 5 nm in diameter.[5] However, this primer concentrates on Engineered Nanomaterials (ENMs).

Q What is the market and potential of nanotechnology?

A

According to the U.S. National Nanotechnology Initiative (NNI), Federal Government funding in the United States, for nanotechnology, has increased from approximately $464 million in 2001 to nearly $1.9 billion for the 2010 fiscal year. Private industry is investing at least as much as the government, according to estimates. The United States is not the only country to recognize the tremendous economic potential of nanotechnology. While it is difficult to measure accurately, estimates from 2005 showed the European Union (EU) and Japan are investing approximately $1.5 billion and $1.8 billion, respectively, in nanotechnology. Behind them were Korea, China and Taiwan with $300 million, $250 million and $110 million respectively, invested in nanotechnology research and development.[6]

Last year the Russian government announced that it was investing $11 billion in an ambitious plan to develop and commercialize nanotechnologies.[7] It is not only governments that are investing heavily in this area, venture capital firms invested $702M in nanotechnology start-ups in 2007 across 61 investments. The Japanese Mitsubishi Institute projected nanotechnology to be worth U.S.$150 billion on the global market by 2010 and Lux Research® estimated a U.S.$2.6 trillion global market by 2014.[1] The U.S. NNI continues to be well funded with a 2010 budget of $1.6B, with total spending since 2001 of nearly $14B. However, to put some of these numbers into perspective, allocation of NNI funds for environmental, health and safety research since 2005 totals $480M.[8] In spite of this it is clear that significant investments are being made in all aspects of nanotechnology and that there is considerable potential.

Q What are engineered nanomaterials?

A There are many new material terminologies associated with this field. This section gives a short overview of some of the different types of nanomaterials.

Fullerenes, graphene and carbon nanotubes

A Fullerene is any molecule in the form of a hollow sphere, ellipsoid or tubular structure composed entirely of carbon. They are commonly referred to as "Buckyballs" – named after Buckminster Fuller who designed geodesic physical structures and buildings based on this geometry. A Buckyball is a carbon based hollow geometric sphere, first found in soot developed from a laboratory experiment.

It resembles a hollow spherical geodesic dome and is comprised of 60 carbon atoms (C_{60}). Discovered in 1985, it is the roundest and most symmetrical large molecule known to man.[9] Fullerenes or Buckyballs are used in nanotechnology. Graphene is a one atom thick planar sheet of carbon atoms densely packed in a honeycomb crystal lattice. Graphene is the basic structural building block of carbon nanotubes and fullerenes. Carbon nanotubes (CNT) also known as 'buckytubes' have a cylindrical nanostructure in the form of a tube and an engineered CNT typically has a nanoscale thick wall, geometrically shaped similar to a Buckyball, with a nanoscale diameter, and a length that may exceed 100 nm.

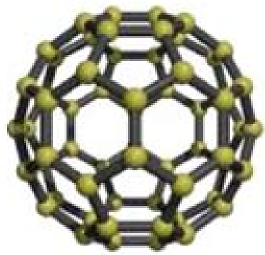

Figure 2. C_{60} buckyball.

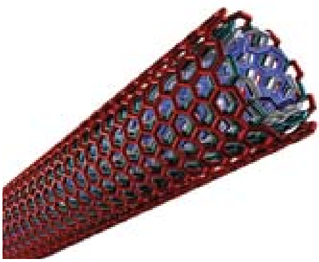

Figure 3. Multiwalled carbon nanotube.

Carbon nanotubes are manufactured as single wall carbon nanotubes (SWCNT) or multiwall carbon nanotubes (MWCNT). An example is shown in Figure 3. They are synthesized in a variety of ways, including arc discharge, laser ablation and chemical vapor deposition. With respect to tensile strength, carbon nanotubes are the strongest and stiffest materials yet discovered, more than 5 times stronger than Kevlar®. Since CNTs have a very low density, their specific strength is 300 times greater than stainless steel, though under compression CNTs appear to be a lot weaker.

Quantum dots

Quantum dots, also known as nanocrystals, are another form of nanomaterial and are a specific type of semiconductor. They are 2-10 nanometers (10-50 atoms) in diameter, and because of their electrical characteristics, they are [electrically] tunable.[10] The electrical conductivity of semiconductors can change due to external stimulus such as voltage or exposure to light, etc. As quantum dots have such a small size they show different properties to bulk material. Hence the 'tunability', for example, sensitivity to different wavelengths of light, can be adjusted by the number of atoms or size of the quantum dot. Quantum dots are typically made from CdSe, ZnS or CdTe compounds, though from a EU Restriction of Hazardous Substances (RoHS) perspective, cadmium-free quantum dots are required.[11] For an excellent explanation of quantum dots and their operation in a cadmium selenide semiconductor see the website associated with reference.[10]

Nanoparticles

Nanoparticles (NP) are synthesized or machined. They range in size from 2 nm to 100 nm. Nanoparticle materials vary depending on their application. Because Nanoparticles are invisible to the naked eye, they are usually supplied suspended in a liquid. This is done for safety and handling reasons. Figure 4 shows gold nanoparticles suspended in liquid. The color is due to the refraction of light the surface area of the particular nanoparticle reflects. Different sized nanoparticles exhibit different colors based on its surface area.[12]

Figure 4. Suspension of gold nanoparticles.

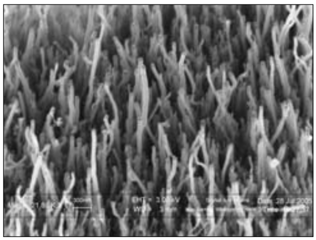

Figure 5. SEM image of aligned nanofibers. *Photo courtesy of Univ. of Wisconsin – Madison, Department of Chemistry.*

Nanofibers and Nanowires

Nanofibers are slightly larger in diameter than the typical nanomaterial definition, though still invisible to the naked-eye. Their size ranges between 50 nm - 300 nm in diameter and are generally produced by electro spinning in the case of inorganic nanofibers or catalytic synthesis for carbon nanotubes. Figure 5 shows an SEM image of aligned nanofibers. Nanofibers can be electrostatically aligned and biochemically aligned.[13,14] Further information about nanofibers fabrication can be found in reference.[15] Similar to nanofibers are nanowires, though nanowires are considerably smaller in diameter, of the order of 4 nm and conduct electricity.

In Table 1, the different size characteristics of the various nanomaterials are summarized.

Table 1. **Nanomaterial types and dimension characteristics.**

Type of Nanomaterial	Number of dimensions and size
Nanoparticle	Three dimensions in the 1 to 100 nanometers (nm) range
Nanotubes/nanowires	Two dimensions in the 1 to 100 nm range
Nanofibers	Length ranges between 50 nm and 300 nm with diameter <50 nm
Nanofilms	One dimension in the 1 to 100 nm range
Nanoplates	Two dimensions in the 1 to 100 nm range

Q Where are nanomaterials being used today and in the future?

A Some of the current applications of many of these nano-related materials and technology are outlined in Table 2 (Page 8). While this table is not intended to be exhaustive, it does show how wide ranging the applications are. It is clear that the nanomaterial science revolution has the potential and magnitude to be an enormous leap forward in technology. However, it should be noted that there are increasing concerns about the impact of these materials in the environment and their possible impact on human health.

Currently the Woodrow Wilson Center for Scholars through their Project on Emerging Nanotechnologies (PEN) lists in their database, 1015 commercially available nanotechnology containing consumer products in over 20 countries[16] up to 2009. This website and searchable database is recommended for those wishing to learn more.

A more comprehensive listing of current and possible future applications of nanomaterials is available on www.PerkinElmer.com/nano

Q How are nanomaterials characterized?

A It is important to understand that the excitement regarding the synthesis and application of nanomaterials is based on the fact that, because of their very small size, the characteristics and behavior are quite different to bulk materials with the same composition. Consequently, the range of parameters that has to be assessed to characterize these materials is large. Fundamentally there are seven key characteristics that contribute to the uniqueness of nanomaterials and these are summarized in Table 3.

In addition to the key seven characteristics, there are two additional qualities that are unique to nanomaterials and important in characterizing them. These are agglomeration, which is the tendency of the particles to clump together and form larger combined particles, and the particle size distribution.

Table 2. **Selection of nanomaterials and usage or application area.**			
Market	**Industry Segment**	**Type of Nanomaterial**	**Use/Application Area**
Environmental	Water	Nano zero valent iron (nZVI)	Being tested for the remediation of ground and surface waters exposed to chlorinated hydrocarbons[17]
		Gold nanoparticles	Various gold nanomaterials are used to enhance imaging properties of a variety of MRI and CT-based contrast agents[18]
		UV absorbing nanomaterials	Improved and sustainable water based surface coatings to protect and preserve wood, concrete and metal surfaces used in construction[19]
Safety and Security	Food	Clay	Nanomaterials are being used in food packaging. The penetration of light, moisture, or gases can alter the sensory characteristics of food products, as well as increase spoilage. Nanomaterials enhance packaging barrier properties[20]
	Energy	Pd and V doped carbon nanotubes	Enhance hydrogen fuel cells by increasing storage capacities and showing faster hydrogen absorption kinetics[21]
	Medical	Various materials	Nanomaterials coated with pharmaceutical compounds are being considered as novel inhalation delivery systems for medications difficult to administer by other means[22]
	Textiles/ Apparel	Silver nanoparticles	Integrated with sports clothing to prevent microbial growth, and odor[23,24]
	Cosmetics/ Personal Care Products	Nano titanium dioxide and nano zinc oxide	Used in some cosmetics. The applications include: eye liners, moisturizers, lipsticks, make-up foundations, soaps, sunscreen, mascara, and nail polish[16]
Industrial	Defense	CNTs	Body armor – multilayer-epoxy composites manufactured with CN sheets, the size of a piece of plywood 4′ x 8′ foot, provide a shield that can stop a 9 mm bullet and weighs no more than a pack of playing cards[25]
	Aerospace	Clay nanoparticles	Incorporated with thermoplastics to create improved fire retardant aircraft interiors[26]
	Automotive	10 nm Cerium oxide nanoparticles	Forms part of the Envirox™ diesel fuel catalyst which improves combustion due to the increased surface area of the cerium oxide nanoparticles[27]
	Recreation/ Manu-facturing	Unknown	Holmenkol® AG supply a chemical nanotechnology coating system under the brand name 'Nanowax®' to replace conven-tional ski and snowboard waxes[28]
	Sports equipment	CNTs/Yarn	High end golf club shafts are made with nano-composites to make the shaft stronger and more flexible. Racing bicycle components[29]

Table 3. **Nanomaterial characteristics, their impact and importance.**	
Nanomaterial Characteristic	**Impact and Importance**
Size	Key defining criteria for a nanomaterial[3] (see Table 1).
Shape	Carbon nanosheets with a flat geodesic (hexagonal) structure show improved performance in epoxy composites versus carbon fibers.[30]
Surface Charge	Surface charge is as important as size or shape. Can impact adhesion to surfaces and agglomeration characteristics. Nanoparticles are often coated or 'capped' with agents such as polymers (PEG) or surfactants to manage the surface charge issues.
Surface Area	This is a critical parameter as the surface area to weight ratio for nanomaterials is huge. For example, one gram of an 8 nm diameter nanoparticle has a surface area of 32 m^2. Nanoparticles may have occlusions and cavities on the surface.
Surface Porosity	Many nanomaterials are created with zeolite-type porous surfaces. These engineered surfaces are designed for maximum absorption of a specific coating or to accommodate other molecules with a specific size
Composition	The chemical composition of nanomaterials is critical to ensure the correct stoichiometry has been achieved. The purity of nanomaterials, impact of different catalysts used in the synthesis and presence of possible contaminants needs to be assessed along with possible coatings that may have been applied.
Structure	Knowledge of the structure at the nano level is important. Many nanomaterials are heterogeneous and information concerning crystal structure and grain boundaries is required.

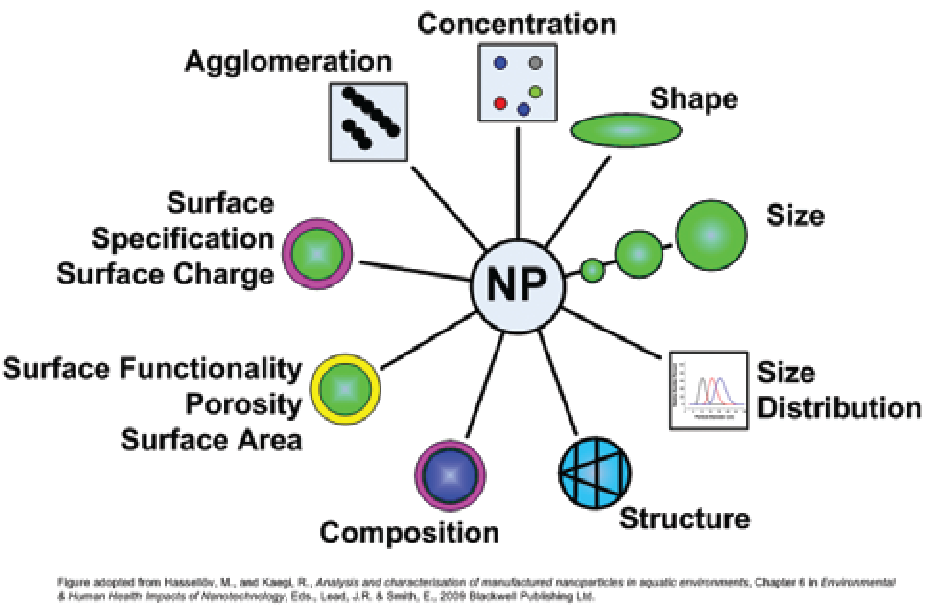

Figure 6. Key parameters to characterize nanomaterials.

Q What analytical techniques are used to characterize nanomaterials?

A As shown in Figure 6 there are seven key characteristics along with agglomeration and particle size distribution that need to be measured to fully describe a nanomaterial. Consequently, at the nanoscale, analytical measurement challenges are considerable and the ability to use, for example, one technique such as inductively coupled plasma-mass spectrometry (ICP-MS) to measure the elemental concentration of gold in a suspension of gold nanoparticles as the only metric to assess the material, does not provide all the information needed. To completely characterize the material it is necessary to know a multitude of chemical and physical parameters including; the size of the particles, their shape, surface characteristics, presence of any surface coating and presence of impurities. This small subset illustrates the magnitude of the measurement challenge facing the nanomaterials industry. Table 4 lists the key characteristics and many of the current analytical technologies that can be applied.

In addition to looking at a variety of analytical techniques and their application to nanomaterials it is also important to understand where measurements need to be made, what type of measurements are required and why. To understand this, an overview of the nanomaterial manufacturing process and value chain is necessary. This includes consideration of aspects such as source and quality of raw materials, control of the synthesis/manufacturing process, validation of the final product and subsequent use or incorporation into another product, e.g., a cosmetic preparation. Along this manufacturing chain are a variety of points at which material and hazardous waste may need to be disposed of and there is potential for environmental exposure. Figure 7 provides a high level view of this process in a very fast changing technology area and outlines which characteristics may need to be assessed at the various measurement points. To understand which analytical technologies may be required to provide this information, Figure 7 and Table 4 can be compared. This chain has been developed from recent market research and customer feedback.

Key nanomaterial characteristics require new measurement technologies. An analytical technique that is becoming more prevalent in the nanomaterial field is that of Field Flow Fractionation (FFF) coupled with Light Scattering (LS) and possibly ICP-MS for elemental nanoparticle characterization. Field Flow Fractionation is a separation technique similar to chromatography whereby colloids, macromolecules and nanoparticles are separated by size and should allow a separation of natural and engineered nanomaterials. Further details can be found in a recent review article on the coupling of FFF with ICP-MS[31] and the websites under references.[32,33]

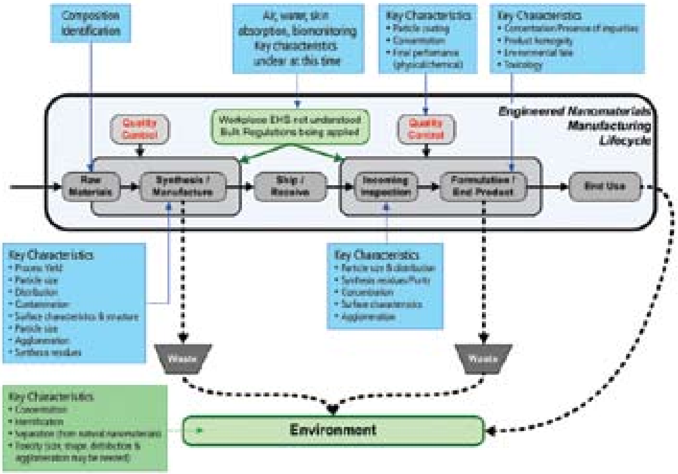

Figure 7. High-level overview of the engineered nanomaterial manufacturing process and key characteristics.

Electron microscopy is also widely used to characterize nanoparticles. The surface area, porosity, particle shape, and agglomeration can be examined with Scanning Electron Microscopes (SEM), Atomic Force Microscopes (AFM), Tunneling Electron Microscopes (TEM), and Confocal Microscopes.

In the production and characterization of carbon nanotubes (CNTs) the use of Thermogravimetric Analysis (TGA) has found considerable application and can be used to show batch to batch reproducibility, detect changes in the process and validate purification protocols.[34] During both the production and formulation process, many nanoparticles are coated or ‘capped’ with a variety of molecules. For assessing the coating, the hyphenated technique of TGA coupled with GC/MS is finding use.[35] A critical application in this area is determining the amount of anti-cancer drug that is coated on nanoparticles. This is needed to characterize the dosage being consumed by the patient.

Q What are the environmental implications of nanotechnology?

A Process waste has always been a manufacturing issue. It is slightly different today when nanoparticles are considered. Nano-waste is different than bulk material waste. It’s been seen in laboratory experiments that nanomaterials can enter the human body by dermal exposure, inhalation, and ingestion.[36] While there are no specific nanomaterials regulations, yet, there is increasing review and concern both within the industry and in the environmental field as to the fate and behavior of these materials in the environment. Many nanomaterial manufacturers are following bulk material

Table 4. **Nanomaterial characteristics and applicable analytical technologies.**

Analytical Technique		**Concentration**	**Particle Size**	**Particle Distrib**
Inductively Coupled Plasma – Mass Spectrometry	ICP-MS	●		
Field-flow Fractionation + ICP-MS	FFF-ICP-MS	●	●	
Liquid Chromatography – Mass Spectrometry	LC-MS	●		
Optical Spectroscopy – UV/Vis	UV/Vis	●	●	
Fluorescence Spectroscopy	FL	●	●	
Turbidity			●	●
Scanning Electron Microscopy	SEM		●	●
Transmission Electron Microscopy (+EDX)	TEM		●	●
Atomic Force Microscopy	AFM		●	●
Confocal Microscopy			●	●
Field Flow Fractionation	FFF		●	●
Dynamic Light Scattering	DLS		●	●
Static Light Scattering	SLS		●	
Molecular Gas Adsorption (BET)	BET			
Dialysis			●	
Electrophoresis and Capillary Electrophoresis			●	●
Ultrafiltration			●	●
Centrifugation			●	●
Filtration			●	●
Nanoparticle Tracking Analysis	NTA		●	●
Size Exclusion Chromatography	SEC		●	●
Selected Area Electron Diffraction	SAED		●	●
Zeta Potential by DLS				
X-ray Diffraction	XRD			
Thermogravimetric Analysis	TGA		●	
Quartz Microbalances			●	
Differential Scanning Calorimetry	DSC			
Dynamic Mechanical Analysis	DMA			
Fourier Transform Infrared Spectroscopy	FT-IR			
FT-IR Imaging				
Raman Spectroscopy			●	
TGA coupled with Gas Chromatography – Mass Spectrometry	TGA-GC/MS			
Laser Induced Plasma Spectroscopy	LIPS		●	
Hydrodynamic Chromatography	HDC		●	●
Laser Induced Breakdown Detection	LIBD		●	●
X-ray Photoelectron Spectroscopy	XPS			
Electron Energy Loss Spectroscopy	EELS (+EDX)			

● Commonly used in the characterization of nanomaterials

● Microscopy techniques

Nanomaterial Characteristics						
e Size ution	Surface Charge	Surface Area	Shape	Agglomeration	Structure	Composition
						●
			●	●		**●**
						●
						●
				●		**●**
●				●		
●			●	●	●	
●		●	●	●	●	●
●	●	●	●	●		
●			**●**	**●**	**●**	
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	●	●				○
						○

○ Not widely applicable

● Available from PerkinElmer

regulations and working with the EPA to establish nanomaterial guidelines for health and safety for the workers and for the end users. The EPA has declared that nano-carbon is a new material and use and requires that it be substantiated as safe.[37] So airborne nanoparticles, nanoparticles in water, and skin exposure to nanomaterials are being addressed by all parties concerned, but there is much research to be done and a key aspect of this work is the need for methods and analytical techniques that can separate, identify and quantitate ENPs in amongst naturally occurring nanoparticles. As consumers, we should to be aware of nanoparticles in the products we use and the food we eat, but currently there are no labeling regulations. There is legislation being implemented in Europe that requires cosmetic manufacturers to list any nanoparticles used in their products.[38,39] This is the first European industry to have required labeling. To date labeling is not required for any other industries anywhere in the world.

Within the United States, the EPA and other government agencies are proactive in regards to nanotechnology. The Federal Government has established the National Nanomaterial Initiative (NNI) where government agencies and private industry meet to discuss and understand nanomaterial implications of the environment and human health. PerkinElmer participates in NNI meetings and is working with the EPA and other agencies to better understand nanomaterials. Figure 8 depicts the life cycle of nanomaterials in the environment and identifies what government agencies are addressing these segments of the life cycle. The source and emission in Figure 8 corresponds to the manufacturing waste in figure 7. The waste interaction with the environment could occur from material taken to a dump, incinerated or washed down the drain. Environmental Health and Safety (EHS) applies to nanomaterial workers as human exposure could occur during the manufacturing process.

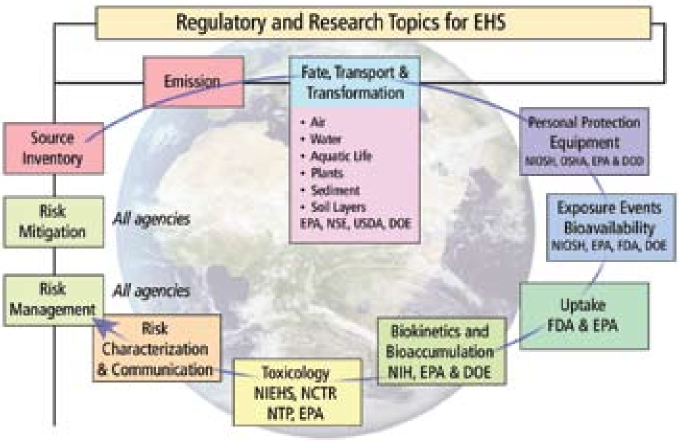

Figure 8. Nanomaterial life cycle in the environment.
Source: DOE Molecular Foundry – Lawrence Berkeley National Laboratory.

Q What solutions are provided by PerkinElmer for nanomaterials characterization?

A Although nanomaterials are small and cannot be seen with the naked eye, it seems likely that their impact on the world will be huge. PerkinElmer is involved in the nanomaterial revolution by participating and working with government agencies, research universities, nanomaterial manufacturers, and end-user industries. While PerkinElmer does not supply all the possible measurement technologies required as listed in Table 4, in certain important areas we have a rich solution offering to enable customers to make critical measurements. As customers discover what measurement parameters and performance criteria are important, we believe that our offerings will deliver more value and come to be recognized as important solutions to challenging problems.

PerkinElmer has the following solutions available for customers who require nanomaterial characterization:

Table 5. **PerkinElmer analytical solutions.**

Analytical Technology	Application
UV/Vis	The LAMBDA™ 850/950 are being used to assess nanomaterial surface coating on glass for the solar energy industry. The LAMBDA 1050 equipped with a 150 mm integrating sphere has been used to measure the band gap (an important semiconductor characteristic) of TiO_2 nanomaterials.[40]
Fluorescence	The LS-55 is being used to measure the fluorescence shift in quantum dots. In addition, quantum dots are being considered as reference materials to calibrate fluorescence spectrometers.[41]
FT-IR	Photocatylitic degradation of dyes and other photosensitive materials. Use of FT-IR imaging to examine gold nanostructures embedded in 50 nm thin polymethylmethacrylate film to develop novel materials.[40]
Raman	Surface characterization of films and other substrate materials that are coated with nanomaterials.
TGA	Pyris™ 1 TGA finds application in characterizing CNTs during the manufacturing process and for incoming inspection.
DSC	DSC 8500 is being used to characterize amorphous pharmaceuticals that employ nanomaterials such as determining the glass transition temperature (Tg) to assess the nano-crystalline structure.[40] StepScan™ and HyperDSC® have been used to study the rigid amorphous fraction in polymethylmethacrylate silicon oxide nano-composites.[42]
DSC-Raman	Morphology characterization of SWCNTs in composites.
DMA	To assess the strength of different composite mixtures of CNT/epoxy.
TGA-GC/MS	Being used by an EPA lab to measure the degree of coating on ENPs under different conditions.
AA	Mainly used to measure bulk concentrations in fabricated materials such as Ag nanoparticle impregnated fabrics [Ag in textile/Germany].
ICP	Assessment of gold and copper concentration in digests of elemental nanomaterial suspensions.[43]
ICP-MS	Rapidly becoming the elemental measurement technique of choice for ENPs, especially Au, Ag, Pt, Ce, W, Ti, etc. in the environment and increasingly being coupled with Field Flow Fractionation. In a recent review article on this hyphenated technique, of the 28 papers referenced, 18 used PerkinElmer® ICP-MS systems.[31] Researchers are now looking to perform single particle analysis with ICP-MS as this gives additional size and distribution information.[44]

PerkinElmer is an active member of the ISO group establishing nanomaterial testing protocols and participates in NNI meetings and a variety of international nanomaterial scientific meetings.

Q Where can I find more information?

A To learn more about PerkinElmer analytical solutions for nanomaterial applications, please visit http://www.perkinelmer.com/nano. This will continue to develop in the future to provide access to key scientific publications, background information, application notes and links to useful websites.

This 'Primer' is intended to provide you with useful background information; it cannot answer every question, but it should stimulate material characterization discussions that hopefully will lead to an analytical solution.

Have questions, need more information? Please contact Andrew Salamon, Patrick Courtney or your local PerkinElmer sales representative. We are happy to answer your nano-related questions.

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