NanoSchoolBox Descriptions of experiments

- exciting discoveries
- amazing effects
- practical experiments



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1. You use the materials provided at your own risk. NanoBioNet e. V. does not assume any liability for damage or consequential loss sustained through the use of the materials, in particular of the chemicals.

2. During use, the industrial safety regulations in force and routine precautionary measures for handling chemicals are to be observed. You should become acquainted with the relevant safety measures for handling chemicals. Safety specification sheets as well as R and S phrases are to be found in the box and on the CD.

3. Please read through the description of the experiment carefully in each case before conducting the experiment.

4. If you wish to allow children to conduct experiments themselves during the lesson, follow these guiding principles: The experiment box is not suitable for use by children under the age of 14; ensure that the young people conducting the experiments are fitted out with the mandatory safety equipment (long-sleeved laboratory coat and protective goggles, gloves if required). Never leave the children unattended.







5. This manual has been scrupulously checked. Any misprints and errors are excepted.

6. The box should never be stored at a temperature of over 20 °C. It should be protected against frost.



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Preface

he NanoBioNet Association has been developing experiments for schools in the field of nanotechnology since as far back as 2002. In 2004 NanoBioNet introduced the NanoToolBox, the first school experiment box for nanotechnology to be produced in the whole of Europe. In consequence of an accumulation of experience, of careful study of developments in the market and in research and not least thanks to the feedback received from dedicated teachers who were already using nano experiments in class, we succeeded in creating this new box, the NanoSchoolBox.

The Association received financial support for the development of this experiment box from the Ministry of the Economy of the State of Saarland and from the European Regional Development Fund (ERDF) of the European Commission. We would like to express our sincerest thanks to all concerned.

The descriptions of the experiments will direct you through some interesting hours in the nano world. Some of the experiments presented here are suitable as demonstration experiments; others can be integrated into lessons under the guidance of the teacher without too much preparation as 'learning-by-doing' experiments. The NanoSchoolBox experiments are intended first and foremost for chemistry lessons. However, due to the interdisciplinary structure of nanotechnology, some of the experiments are also suitable for physics or biology classes. As school syllabuses vary from state to state, we are not able to recommend precisely when a particular experiment should be used. In this case, we rely on your professional knowledge as a teacher to help you make the appropriate decision. If you would like to use all the NanoSchool-Box experiments together, we suggest organising a nano project day or a nano work group at your school. Group work allows pupils to be proactive, they themselves trying their hand at the experiments.

The experiments were developed with the aim of making nanotechnology easy to explain to schoolchildren. In order to achieve this, they should not, as far as possible, pose any danger and it should be possible to make them understandable with just the relatively simple basics of life sciences. For this reason, some experiments have been included which, strictly speaking, are not related to 'nano' (i.e. the use of nanoscale systems). Nevertheless, these experiments create an excellent opportunity to describe and explain the effects of the nano world.

Please contact us if you have any questions on the experiments. We would appreciate your comments as we aim to continue improving and further developing the experiments in the future. As a non-profit institution working for the benefit of the public, NanoBioNet represents a network of research institutes, companies, and business and technology sponsors. In addition to coordinating a network of over 100 facilities, on the basis of its information service and PR work, NanoBioNet e. V. organises activities related to nano and biotechnology, such as further education for teachers and

practical training for schoolchildren as well as further training for laboratory assistants, technicians, academics and managers.

One of the most important aims of NanoBioNet e. V. is to arouse the interest in young people in one of the most fascinating high technologies in life sciences. If it succeeds in generating enthusiasm in young people for nano and biotechnology, the shortage of young talent in the nano and biotechnology sector will be resolutely relieved and young people will be brought closer to a promising field of activity for the future.

NanoBioNet offers its members on different platforms the opportunity to establish contacts and pass on expertise. We are always happy to welcome new members, irrespective of whether they are institutions or individuals. If you are involved in nano or biotechnology, then you have come to the right place here at NanoBioNet.

NanoBioNet wishes you and your pupils a lot of fun with the experiments!



Martin Monzel, Managing Director NanoBioNet e. V.



Introduction

What is nanotechnology?

ver the last few decades an interdisciplinary science has emerged from the fields of physics, chemistry, biology, medicine and materials sciences: nanotechnology. It aims, through technical means, to make processes or modular units functional on a nanoscale dimension for scientific and industrial use.

Technology had long been striving to miniaturise structures (top-down approach), for example by etching out tiny structures from a block of material. However, nanotechnology uses a completely different technique: Fundamental mechanisms and principles are explored and applied within the scope of today's technology in such a way as to create new functional systems. The objective in this respect is to 'design' minuscule structures on a nanometer scale from the start and, subsequently, to develop larger structures again (bottom-up approach).

It should be noted that this is a process which nature has been following for billions of years. This is how, in the course of evolution, proteins, microorganisms and protozoa, which later developed into multiple cell and higher organisms, were formed from amino acids. However, we should bear in mind that this development stretched over an extremely long period of time, requiring several billion years from primordial soup to Homo sapiens. The prefix 'nano' is derived from the Greek word 'nanos' which means 'dwarf' or 'dwarfish'. It refers to the billionth part, i.e. $10^{.9} = 0,000000001$. One nanometer is equivalent to one billionth of a meter or one thousandth of a micrometer. The functional properties of a particular material are dependent, in the case of tiny extensions, on the dimensions. For example, properties such as hardness, electrical conductivity, colour or chemical reactivity of minuscule particles of any material are directly dependent on the diameter of the particle.

Nanoparticles lie in order of magnitude between isolated atoms and a solid. For example, nanoparticles are no longer able to disperse visible light because they are too small. Nowadays, nanoparticles already play an important role in the pharmaceutical positioning systems of active agents, in the production of emulsion paint and cosmetics or in the optimisation of catalysts.

In fact, nanotechnology is not really new. Particles with small dimensions have long been known to science: for example, nanoscale molecules have long been used in colloid chemistry.

Nature has been producing nanoparticles since the beginning of time, for example through volcanism, in the sea (erosion), in sand storms, in pollen flight and in forest or bush fires. In this connection, particles emerging from natural fire sources may even be smaller than 1,000 nm. Particles present in the sea reach 10,000 nm. Further particle sources are industrial processes, energy generation (especially electrical power stations and district heating plants), traffic, construction and households (heating). Nanoparticles in the order of up to 500 nm are

released in the combustion process of fossil fuels. So, have we always had nanotechnology on earth? Actually, the answer is no – because nanotechnology works in even smaller dimensions. In nanotechnology, specific functionalities are targeted by reduction of size – to less than 100 nm – to comply with the characteristic structural dimensions. Therefore, the term nanotechnology is a collective term for a multitude of technologies. This collective term includes all those fields of research which work within the scope of the isolated atom up to a structural size of 100 nanometers (nm). Establishing the nanoscale at a range of 1–100 nm is appropriate as a variety of completely new size-induced functionalities can be processed in this length scale. This includes isolated atoms or molecules as well as microtechnology.

There is currently a debate as to whether nanolayers should be excluded from the definition of nanotechnology. Nanolayers correspond to the nanoscale structure in only one dimension, namely density. A restriction to a minimum of two nanoscale dimensions (two-dimensional: e.g. nanowires or nanofibres, three-dimensional: e.g. nanoparticles) would rule out the possibility of calling a conventional film on a surface with a density of only a few nanometers a nanotechnological product.

This is not such an easy matter to resolve: If paint contained nanoparticles, would this then again give rise to nanotechnology in the 'third dimension'?

However, the definition of nanotechnology which currently includes developments 'with one dimension' is widely accepted.

As we can see here, it is not easy at present to classify what exactly belongs to nanotechnology and what does not. What constitutes nanotechnology in the final analysis is the goal-oriented, multidisciplinary use of techniques to manufacture nanoscale systems.

The first true visionary by today's standards in the field of nanotechnology in the literal sense is the American physicist and Nobel Prize winner Richard Feynman. Feynman's reflections were visionary insofar as the technical implementation of the 'Feynman machines' seemed to be, at the most, long term and, in some cases, even impossible. While Feynman did not use the term 'nanotechnology' himself, it was introduced as a concrete expression in 1974





by the Japanese Norio Taniguchi. Norio Taniguchi applied the term to technologies which are adapted to monitoring the surface roughness of materials on sub-micrometer length scales.

A milestone in this field was the development of a scanning tunnelling microscope at the end of 1981, which made it possible for the first time to show isolated atoms and not just an entire collective of atoms. Gerd Binnig and Heinrich Rohrer of the IBM research laboratory were awarded the Nobel Prize for physics in 1986 for the development of this device. The importance of this development lies in the fact that the scanning tunnelling microscope became the starting point for an entire series of tools which enable substances to be analysed at a molecular and atomic level and even to be modified. Today, it is no longer possible to imagine nanotechnological research, development and production without the use of scanning tunnelling microscopes as standard instruments.



From the lotus effect to the technical application of nanolayers

an has always modelled new technologies on structures developed by nature in the course of evolution. Due to the dirt-repellent, drip-off effect on the surface of the leaves which contributes to its beauty, the lotus plant has become particularly well known in the field of bionics/nanobiotechnology. This effect, the so-called lotus effect, is by no means a concomitant phenomenon – it has been created in the course of evolution as a means of survival for the plant.

Plants are by nature exposed to different types of contamination. Most are of an inorganic nature (various types of dust, soot) but some are of biological origin (e.g. fungal spores, honeydew, algae). The inorganic substances directly produce several adverse effects on living tissue of the plant, such as overheating under the sun's rays, a higher acidity or the possibility that the stomata (minute pores), which allow the plant to exchange gas, are blocked. Organic particles, such as fungal spores, bacteria and algae, also have a considerable impact on plants. They may cause plant diseases or damage to the surface of the leaves.

The lotus effect offers the plant an elegant way of eliminating these problems. It prevents such substances from settling on the surface in the first place. The rain washes the spores away and, if it does not rain for a long time, the unwelcome visitors do not have enough water to germinate.

How does the 'natural cleaning' system actually work?

In principle, all surfaces of plant leaves possess the same composition: the outer cell layer (epidermis) is covered by a cuticula, which protects the plant tissue from loss of water and increases the mechanical stability of the epidermis. It contains a mixture of different lipids, which are also known as 'waxes'. These waxes make the cuticula impermeable to water and they reduce, to a high degree, permeability to water vapour. Cutin (a biological wax) is often deposited in or on the cuticula. These substances are to be found in crystalline form and can often be identified macroscopically as a whitish coating which can be removed by washing, such as on grapes. Electron-microscopical images show fantastically beautiful micro or nanostructures, which are characteristic to certain plant leaves.

Plant leaves differ considerably with respect to their wettability. Some types of plants have leaves which are not wettable with liquids. Not only the adhesion (=bonding) of water to the surface is reduced but also that of dirt. If a drop of water then rolls over the loosely settled dirt particles, the latter stick to the surface of the drop. The drop carries the particles along with it and removes them from the leaf. Many people believe that the smoother the structure of the surface, the easier it is to clean. However, if you look at a lotus leaf under the scanning electron microscope, you will see uneven wax crystals, which actually make the surface of the leaf rough. You are not able to see these crystals with the naked eye, nor can you feel them.

In principle, water resistance is always related to a micro or nanostructure of the surface. The physical reason for water resistance is the relationship of the interfacial surface tensions of water to air, of water to solid and of solid to air. The respective conformation of the relationships to each other determines the contact angle of the drop to the solid.

According to the physical definition, a surface is hydrophilic (water friendly) if the contact angle to the drop is below 90 degrees. If it lies above 90 degrees, the surface is hydrophobic (water repellent, i.e. water resistant). In the case of full wettability – the contact angle then stands at 0 degrees – the water forms a monomolecular film on the surface of the solid and a drop of water will, at the most, spread over the leaf.

At a contact angle of 180 degrees (which is not present in nature) a drop would theoretically come into contact with the surface of the solid at only one point. The contact angle for conventional water-repellent surfaces (e.g. Teflon) is maximum 120 degrees. Some artificial hydrophobic surfaces can reach a contact angle of 170 degrees. These surfaces are known as superhydrophobic. A drop of water is not able to spread over this microrough water-repellent surface. Due to its own surface tension it draws together into a ball. The adhesion (bonding force), which keeps the drop on the surface of the leaf, is reduced to a minimum. This enables the drop to roll away over the leaf at the slightest inclination of the surface.



Composition of a lotus leaf viewed under a scanning probe microscope



Contact angle of 130°



Drops on a superhydrophobic surface (photo: Nano-X GmbH, Saarbrücken)



In addition to the lotus, there are many other plants, such as the water lily, cabbage, lady's mantle or nasturtium, which possess hydrophobic leaves.

A similar phenomenon is also found in the animal world. For example, the common dung beetle has an easy-toclean surface. This little creature lives in a heap of dung or earth, i.e. in the immediate environment of the excretions of other animals. This is swarming with thousands upon thousands of microorganisms, which are forever trying to do harm to the dung beetle. They would almost succeed, considering its stiff, little legs and its rigid exoskeleton, which make the dung beetle so uncoordinated that it cannot 'clean' itself. There is no way for it to clean its shield.

But evolution was on its side: it provided the dung beetle with the ability, despite its unclean habitat, to remain clean. And this works with the help of nanotechnology. If you examine the surface of its shield through an electron microscope, you will come across a perfect nanostructure, which is very similar to that of the lotus plant. Together with a waxlike secretion, the dung beetle has created an easy-to-clean surface for itself, on which dirt and other microorganisms are not able to adhere. Further examples of the existence of these 'nanosurfaces' in the animal world are to be found on insect wings, such as on dragonflies and butterflies. Researchers are now trying to copy and reproduce the properties of the naturally-occurring surfaces by developing a similar physico-chemical structure and – in order to reinforce the desired effect – to modify and further develop it. This research work has produced coating materials with functional properties:

The coating covering the material is for the most part only a few nanometers thick and basically consists of one organic and one inorganic component.

An inorganic matrix is often composed of silicon dioxide, zirconium dioxide or titanium dioxide, which are integrated into an organic matrix (in most cases an organic solvent). Depending on the requirements, nanoparticles from the same materials can also be introduced into the matrix. These can be used to attain a multitude of functional properties.

At first glance, this system does not seem particularly complex. The key to success is in the correct combination of substances which, depending on the proportion of ingredients, determines which effect can be achieved. This knowledge is guarded in the same way as a treasure by research institutes and companies, who have it patented. Many different effects can be achieved with nanolayers today and such 'coatings' belong to the nanoproducts of the first generation. In the following experiments we are concerned with hydrophobic and hydrophilic layers. Surface of a dung beetle shield magnified several times photo: Sarastro GmbH, Göttelborn



Examples for effects in industrial applications

Composition of nanocoating graphics: Sarastro GmbH, Göttelborn



Experiment 1: Experiments to obtain the lotus effect

Description of experiment	The aim of this experiment is to determine whether certain materials possess a hydrophobic surface. In order to do this you need to collect different sorts of paper (e.g. high- gloss paper from the printer, typewriting paper, coffee-fil- ter paper) and parts of plants, such as the leaves of grass, nasturtiums, kohlrabi, rubber plants, grapes, salad leaves or dandelion. If you can actually come up with a lotus leaf, all the better. But it really does not always have to be the lotus!	Procedure	 Then allow water drops of the same volume (use the pipettes) to drip onto the paper and leaves of different structures. You can also do this with other surfaces, such as glass, wood or plastic. Measure the diameter of the drops or spots of water with the ruler. The more hydrophobic the surface, the smaller the diameter of the drop and, consequently, the greater its curvature. Filter paper completely absorbs the drop of water, which means that it is not a hydrophobic (but hydro-
Materials	 Materials from the box: pipettes Additional materials not provided: different types of paper plant leaves a millimeter-scale ruler fine dust (finely ground garden clay), fine ash (e.g. from cigarettes) or soot tap water 		 philic) surface. The diameter of the drop on the high-gloss paper is very small, but the curvature very high. Find plant leaves which clearly demonstrate a drip-off effect. 4. Now put dust or ash on the leaves showing a drip-off effect. Then add some drops of water with a pipette to the contaminated surface and tilt the leaf so that the drops can roll away. The clay particles are absorbed by the drops of water. The drop leaves a clean track on the dirtied leaf. 5. Now rub the leaf with your thumb (not so hard that the
Safety guidelines	• none		leaf tears!). If, at this stage, you drip water onto the leaf again with the pipette, you will see that the drip-
Procedure	 Collect (if appropriate with your class) different types of paper and plant leaves. 		off effect has clearly diminished or even been comple- tely destroyed.

Observations

- The hydrophilic pulp fibres of some of the leaf surfaces exercise adhesive forces on the water, which increase the greater their surface (or the rougher the surface of the paper). For this reason, the drop of water spreads out over the surface to a greater or lesser degree. In nature, this lotus effect is responsible for the surface of the leaf remaining for the most part clean (even if there are distinct differences in 'cleaning behaviour', which are the result of the different levels of water repellent properties of the surfaces). In this way, the plant preserves its full functional properties for photosynthesis and gas exchange.
- 2. The hydrophilic clay particles are absorbed by the drop because their adhesion to the leaf surface is lower than the adhesion to the water. Not until the drop is saturated with clay is it no longer able to exercise its cleaning effect. This means that the remaining clay particles will be left on the leaf surface.
- 3. The water repellent property has been neutralised on the damaged leaves. The drops stick to the leaf surface although their chemical characteristic has not changed. The leaf surfaces are now easily wetted. The micro or nanostructures which are otherwise responsible for the water-repellent effect of the surface in undamaged leaves have been destroyed.

By the way: In the case of living plants, after a while the lotus effect is re-established, i.e. the plant regenerates and repairs the structure of the surface. This is known as self-structuring.



Cleaning effect of a self-cleaning surface; photo: Nano-X GmbH, Saarbrücken



Materials

Experiment 2: Production of a hydrophobic surface on wood or on a mineral material

Description of experiment

This experiment concerns the development of artificial hydrophobic surfaces on materials such as wood or stone (you will administer a hydrophobic [water-repellent] treatment).

The atoms of a drop of liquid interact with the atoms of a solid surface. The forces of attraction between atoms ('van der Waals forces') cause a reduction of the contact angle and, subsequently, of the interfacial surface tension. The tendency for water to form into the shape of a ball is influenced, amongst other reasons, by gravitational pull, which flattens out the ball.

This results in extremely smooth, water-repellent surfaces and a contact angle against water of maximum 120° develops.

However the hydrophobic property can be destroyed (intentionally or unintentionally) again, specifically if the structures (pores) are sealed off or the coating is mechanically or chemically destroyed. For example, tensides (surfactants) or oils can ruin the effect. In practice, such surfaces are useful because any contamination (e.g. lime) which may occur can be washed away with very little detergent, the process being much less timeconsuming.

A hydrophobic surface is not a self-cleaning surface as in the case of the lotus plant so you cannot avoid also having to clean these surfaces now and again. A positive side effect of using lateral fluorinated chains in the coating material is a reduction in the infestation of algae and germs on such surfaces.

The fields of application of such coatings are chiefly on surfaces which are exposed to water (rain) but are not under constant mechanical strain. In addition to building materials for roofs, this is also suitable for cladding (façade) elements or masonry.

Materials from the box:

spray flask 'wood/stone coating'

Additional material not provided:

 untreated wood or porous mineral substrate (e.g. pebbles, pumice, concrete or similar)

Safety guidelines

- The product itself is not flammable. However, in the case of fire, dangerous gases or vapours (nitrogen oxide, carbon dioxide and hydrogen chloride) may escape into the atmosphere due to thermal decomposition.
- Wear protective goggles, a laboratory coat and protective gloves during the experiment.
- Do not inhale vapours/aerosols. Ensure there is good ventilation during the experiment.
- The coating solution contains polysiloxanes containing aminoalkyl and fluroalkyl groups and hydroformates (Xi, R phrases 36/37/38; S phrases: 26-36) and formic acids (C; R phrases: 36/37/38 S phrases: 26-36). R and S phrases can be found on the safety specification sheets and on the CD.







Procedure

- 1. Find an open-pored or absorbent substrate made of wood or stone, which has not been treated.
- The surface to be treated must be dry and clean; remove any deposits of loose dirt.
- Now spray the substrate with the coating solution until it is saturated.
- Allow it to dry completely. The solution can only really take effect when it has completely dried. The time needed for it to dry completely depends on the temperature (Please do not heat above 40 °C!) – 2 to 6 hours.
- 5. The coated surface can now be tested with different liquids, for example waste water, coffee, lemonade, etc. Particular care is to be taken if façade paints are to be tested as some paints (e.g. those with an acrylic base) may stick to the coating!

Observations

You will see a drip-off effect with a clear formation of drops. Due to its surface tension, the water tries to draw itself into a ball (with minimum surface and maximum volume). Once the coating has been applied and has hardened, the water-repellent molecular chains of the coating will give rise to non-abrasive layers with reduced surface energy. As already explained above, this is due to the van der Waals forces. If the surface has been made hydrophobic, the interactions are restricted to a minimum, which leads to a marked increase in the wetting angle of contact. When the coating has completely dried, it is abrasion-resistant; however, it can be destroyed again by chemical means.











Experiment 3: Production of a hydrophobic surface on textiles

Description of experiment

As in the previous experiment, you will also be developing a hydrophobic effect in this experiment. It is based on the same principle as Experiment 2. However, in this case you are dealing with a special impregnation to obtain a hydrophobic and oleophobic (oil-repellent) effect for textiles and paper.

The solution forms an almost invisible film – which is only a few nanometers thick – on the surface of the fibres. The hydrophobic effect considerably reduces the speed of water penetrating the treated textiles or paper and also the amount of residue from soiling, e.g. with soot, coffee or red wine.

The first textiles (e.g. ties, suits, raincoats) in mass production have already been treated with this type of protection by a variety of manufacturers. Of particular interest is the coating for outdoor fabrics or textiles (awnings, umbrellas, etc.) which are constantly exposed to atmospheric conditions.

Procedure

Safety guidelines

Materials

Mc	aterials	from	the bo	X:
•	spray	flask	'textile	coating

Additional materials not provided:

- textiles which have not already been coated with hydrophobic layers
- Textiles with a polyester base are especially suitable, rather than silk or shorn wool. Any (unwanted) items of clothing from the wardrobe will do.
- Do not use clothes which are being worn at the time to perform the experiment!
- Do not inhale spray mist, vapours or aerosols!
- Wear protective goggles, a laboratory coat and protective gloves.
- Ensure there is good ventilation during the experiment.
- Fluorinated polymer suspension, acetic acid < 2.5 % (C; R:10-35), see safety specification sheets or CD.
- The fabric to be treated must be dry and clean; remove any deposits of loose dirt. Before applying the solution, wash and rinse out the articles to be treated thoroughly in order to remove any textile auxiliary or processing agents (e.g. tensides or lubricants).

- 2. Shake solution before use! The dry fabric is now sprayed with the textile coating. The active component should be applied in such a way that it forms an even, thin, wet film on the surface of the substrate.
- Dry at room temperature (minimum 1 hour or until the material feels dry) or through heat up to 160 °C (e.g. in a circulating air drier or with a hair drier). The solution can only really take effect when it has completely dried.
- 4. The coated surface can now be tested with different liquids.
- Observations Most liquids drip off the surface and are not absorbed into the piece of fabric. Its breathing is not affected as the coating settles on the yarn in the same way as a second skin.







Experiment 4: Invisible ink for glass - production of a hydrophilic surface (anti-fog)

Description of experiment	You are now going to learn about how to develop the op- posite of the hydrophobic effect, namely the hydrophilic (water friendly) effect. What is so special about a surface treated with the so-called 'anti-fog coating' is the fact that it does not absorb water but that watery solutions are 'spread out' (i.e. dispersed). Instead of seeing a drop we see 'nothing'. In this experiment we treat a glass surface with a hydro- philic agent in order to write a secret message on it. A similar process has already been applied in mass pro- duction on car headlights, mirrors and optical apparatus in medicine (e.g. endoscopes, probes, etc.), which must be protected against condensation.	Procedure	 Give the screwtop bottle invisible ink a good shake. Dip the paintbrush provided into the invisible ink and moisten slightly. Use the paintbrush very thinly to write a message or paint something on the glass or mirror. Wait a few minutes to allow the solution to dry. 'Breathe onto' the surface. You can also apply the 'anti-fog' coating to bathroom mir- rors, spectacles or the visor of a motorbike helmet. In this case, we recommend you polish the surface with a soft cloth. It is possible that faint stripes appear on the glass but these can easily be cleaned off again.
Materials	 Materials from the Box: screwcap bottle 'invisible ink' paintbrush Additional materials not provided: untreated glass (window panes also suitable) or a mirror 		
Safety guidelines	The solution contains propanol and is therefore easily flammable. For this reason, please keep away from fire sources and wear protective goggles.		

Observations

Your breath mists up the piece of glass except where something has been written or painted on it.

The image is visible for days or even weeks as long as the glass has not been cleaned (no mechanical strain). The solution forms a thin layer on the surface, which is so hydrophilic that it 'spreads out' the condensation from your breath to the maximum degree. Only an extremely thin film of water molecules can 'adhere' to the glass surface. However, the film is not visible and does not prevent the light shining through. The coated glass surface does not mist up (anti-fog effect).

The formula used here does not compare with industrial coatings. Anti-fog coatings used in industry often prove to have a stronger effect, with increased durability and resistance to abrasion.







Functionality through nanotechnology

o far we have investigated ultra thin coatings with hydrophobic or hydrophilic properties. However, there are further nanolayers which demonstrate other functionalities. In fact, it is often necessary to use nanoparticles in many industrial nanomaterials to achieve an additional functional property.

An essential quality of the particle is that, being so small, it is transparent and cannot be seen with the naked eye.

In this chapter we take a look at 3 particular functional properties, for which the use of nanotechnology can be helpful: increasing scratch resistance, improving electrical conductivity and reducing flammability.

Nanocoating for scratch resistance

Many materials which we use in our daily life become scratched or unsightly, i.e. they are subjected to abrasion caused through mechanical forces. Irrespective of whether it is the car in the car wash, a flour mill, a knife or the parquet floor in the living room, everything we use is subject to wear through abrasion. This is also especially true of industrial machinery – rolling mills, driving mechanisms, conveyor belts or shafts.

Nanolayers or nanoparticles respectively can be used to keep our commodities working longer and to preserve their original appearance.

As a rule, the nano scratch-resistant coating consists of an organic-inorganic nanocomposite material. In this case, the nanoparticles are integrated into a bonding agent.

It is mostly silanes – also called silicic acid esters – with functional groups (epoxide functions, acrylate functions) combined with nanoparticles which are subject in this synthesis to a hydrolysis and condensation reaction. In this process, the water reaction causes reactive Si-OH groups (hydrolysis) to emerge from the ethoxy residue of the silane components, which can react with further OH groups by splitting off the water (condensation). An inorganic, glasslike network develops as a result of the hydrolysis and condensation reaction respectively. The silane functional group, which can be activated with appropriate initiators, takes the process a step further to generate an organic network: hence, the name 'organic-inorganic' nanocomposite.

By developing an organic and inorganic network in the coating, as well as integrating the nanoscale particles, which are also firmly attached to the matrix through chemical bonding, a layer is produced, which contains a combination of glasslike and polymer properties. Such coatings are known as hard-elastic coatings. Their mechanical stability is so high that, even if the layer of the coating is extremely thin, steel wool will no longer scratch the surface, or at least will have difficulty in causing any damage. In order to produce the coating the relevant silanes and nanoparticles are mixed with water to start the hydrolysis and condensation reaction. The reaction mixture, which was two-phase when the water was first added, heats up and becomes single-phase. A reaction initiator is then added to cross-link the silane organic functional group.

Once the coating is produced, it is sprayed onto the wooden pieces of flooring. The pieces are subsequently dried in the air drier at a temperature of approx. 110 °C. This process is often called 'curing'. To be precise, the solvent is removed during the drying phase, i.e. really 'dried'. However, it is also 'cured' as the reaction initiator starts the polymerisation of the organic functional groups and, in addition, the glasslike network is completely condensed. The 'drying' is often referred to in science as 'physical curing', the 'curing' as 'chemical curing'. In an air drier the entire process is completed in 20 minutes and the coating on the wooden pieces has achieved its highest level of scratch resistance. Naturally, this can also be radically accelerated by intelligent curing methods. This is particularly important for furniture production, in which large quantities have to be produced in a short period of time.

Such protective layers are already being used, for example, in mass production on some models in the automobile industry. In our experiment we direct our attention to an example from the household: a piece of wooden flooring.





Integration of nanoparticles in the hydrolysis reaction



Experiment 5: Scratch-resistant coating of wood

Description of experiment	Generally, MDF (middle-density fireboard) panels with a wood pattern are used for the production of interior doors, fronts and backs of furniture. The patterns are printed using various printing techniques, such as inkjet printing, onto the panels. A problem that is often linked to this process is that the surface is highly susceptible to mechanical strain and can, therefore, be easily scratched. In order to protect the patterns on these MDF panels a trans- parent, 10 µm thin protective coating was applied over one half, in which nanoparticles were integrated. This gives the panels greater stability; even if steel wool is scraped over the surface, the latter will not be scratched.	Materials	Materials from the box: • pieces of wooden flooring • steel wool Additional materials not provided: • Edding or other permanent marker, or felt-tip pen
		Safety guidelines	• none
		Procedure	In order to test the mechanical stability the steel wool is scraped over the piece of wood several times – preferably over the border between the coated and untreated area. If you wish, you can draw a line with the marker over the piece of wood before doing the experiment.
		Observations	A scratch can clearly be seen on the untreated part, while the coated side is undamaged. If you drew a line over the piece of wood before the ex- periment, you will see that the colour on the coated side can be rubbed off with the steel wool, while it can no lon- ger be removed from the untreated side.

Nanocomposites for fire protection

In the event of a fire, every second counts. Just a few minutes after a fire has broken out smoke, heat and flames can assume life-threatening proportions. In order to prevent a fire, or at least to delay the progression of a fire, many wood products, plastics or textiles are provided with flame-retardant protection. It extinguishes the fire, encourages carbonisation, and forms damp-proof and insulating layers. Many conventional fire-resisting agents alter, depending on the concentration, the mechanical, electrical and chemical properties respectively of the material added to them. The flame retardants currently available can, at least partially, be replaced by nanoparticles. The nanoparticles ensure that a crust is formed around the burning material and prevent the fire-resistant substances from evaporating too quickly.

Transparent nanocomposites, which are suitable as fillers for insulation materials, are preferred for protection against fire. For some applications (e.g. transparent filling between fire-resisting glass) it is necessary to use gels, which also remain clear for a long period of time without any sign of aging. Further, it is important to ensure that the fire-resisting components retain their exact shape if they are filled in defined spaces of door or wall components made of different materials.







Science has established that it is possible to produce stable, transparent and hard materials, whereby nanoscale particles of inorganic solids are combined with certain organic compounds to prevent signs of aging. They shield the materials subject to fire hazard, at the same time developing for their part sufficiently strong linkage forces, which are necessary to form a mechanically stable body.

In general, fire-protection nanocomposites consist of 3 components:

- Nanoscale particles (between 50–100 nm) from inorganic compounds, in particular from oxides of the elements aluminium, silicon, phosphorous, boron, tin, zinc, titanium, zirconium or tungsten.
- One component which reacts with the nanoparticles through functional groups: this involves a conventional reaction developing covalent, ionic or complex links. The compound classes preferred are polyoles, alkanoamines or polyamines.
- The third component is a solvent, either water or one or more organic solutions (alcohols or carboxylic acids).

If fire protection of up to several thousand (!) degrees is desired, it is therefore possible to use flame retardant systems with inorganic nanoparticles to prevent such a fire. These systems can achieve effective fire protection for glass, walls, textiles or roof beam. They are also already in use in industrial fire protection. In our experiment, which particularly appeals to schoolchildren, we 'only' work with a lighter and paper. The temperatures in this case did not require any nanoparticles so we could select a system that is physiologically considered to be generally safe for use. Therefore, the effect of the solution provided is not based on the presence of nanoparticles but on chemical conversions of ammonium and phosphate compounds with carbohydrates.

Experiment 6: Fire protection

Description of experiment	Paper, cardboard or wood is composed for the most part of cellulose, a carbohydrate. When the solution is poured onto the paper and heated, the phosphate bonds chemi- cally with the carbohydrate and converts to coal, but wit- hout burning in the process. Phosphoric acid esters are formed, which decompose under heat to become phos- ohoric acid and carbon. The nitrogen compounds in this case have the following function: the heat causes nitrogen gas to form (which is in itself incombustible) which displaces the oxygen needed at the source of the fire (no flame without oxygen).
Materials	Materials from the box: • spray flask 'fire protection' Additional materials not provided: • 1 piece of paper or cardboard • a lighter
Safety guidelines	In the experiment we work with fire. Place a fireproof surface underneath for safety and ensure that there are no fire or smoke detectors in the demonstration room.



Procedure

- Take absorbent paper or cardboard and spray it (preferably on both sides) with the 'fire protection' solution from a distance to the object of minimum 10 cm.
- 2. Allow the paper to dry off completely (a hair drier will speed this up).
- 3. Try to light the paper with a lighter.

Especially 'brave' teachers perform the experiment using a slightly modified method:

- 1. Ensure there is a fire-proof surface underneath (sink, chemistry workbench or similar).
- 2. Spray just half of the piece of paper well on both sides until it is dripping wet and mark the side treated.
- 3. Allow the paper to dry off completely.
- Set the untreated half of the paper on fire, making sure you are holding it on the treated side and wait until the flames attack the treated half.
- If you can keep your head and not drop the piece of paper, you will ascertain that the flames extinguish themselves as soon as they spread onto the treated half.

Observations

Inert gases emerge from exposure to the fire and the impregnated material is carbonated. The flames do not spread to the coated material and there is no fire.

Increasing electrical conductivity

Ordinary glass is not conductive, i.e. it is not able to transmit electrical currents. However, the special glass in this box can do this because the glass is covered with an ultra thin, invisible nanolayer, a so called ITO layer.

The term ITO stands for indium tin oxide, a mixed oxide compound of indium oxide and tin oxide. It is semi-conducting and transparent, these properties being the basis for the production of electrically conductive glass. In order to produce this glass, it is covered with an extremely thin layer of indium tin oxide. The glass becomes conductive and remains transparent. The thin nano ITO layers are applied by vapour deposition, sputtering, spin coating or a dipping process. Sputtering, which is sometimes called cathode sputtering, is a physical process, whereby atoms are released from a solid (target) through bombardment with high-energy ions (predominantly noble gas ions) and transferred to the gas phase. Sputtering is generally understood to be only sputter deposition, a high-vacuumbased coating technique belonging to the group of PVD processes.

Indium tin oxide is extremely expensive as very small quantities of this material are found on the earth.





Experiment 7: Increase in electrical conductivity through indium tin oxide (ITO)

Description of experiment

The availability of conductive glass is a prerequisite for the production of solar cells. The conductivity of an ordinary glass panel and of a glass panel coated with ITO is tested by means of a simple multi-circuit switch. A flashing LED verifies the presence of an electric current.

Fields of application:

- photoelectrodes
- solar cells
- transparent contacts in liquid crystal displays
- electroluminescent displays, e.g. organic light emitting diodes
- heated car windows or other reactive glass, which is 'switched on/off' electrically.

Materials

Safety guidelines

- Materials from the box:ITO sheet of glass
- untreated sheet of glass
- 3 alligator cables
- 1 compound battery
- 1 LED

Additional materials not provided:

• none

none

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rocedure	First use the ordinary untreated sheet of glass and set up an electrical circuit with the above-mentioned materials (see diagram). Take extra care to attach the positive termi- nal of the battery to the LONG wire of the LED. (LEDs con- duct electricity in only one direction). Observe. Then replace the sheet of glass with the special ITO sheet of glass. What happens?
Dervations	Ordinary glass is not able to conduct electrical currents so the diode does not light up. In contrast, a sheet of glass coated with ITO is able to do this. The diode flashes to show that the electrical current is flowing.



Use of titanium dioxide in nanotechnology

itanium dioxide is present in nature in three crystallographic phases:

- 1. Brookite forms orthorhombic minerals and is, there fore, of no importance to industry. Below melting point brookite turns into rutile.
- Rutile is a tetragonal mineral of a predominantly prismatic habit. Rutile has a density of 4.26 g/cm³. It is used for the most part as a white pigment in wall paints, print colours and for colouring plastics.
- 3. Anatase forms tetragonal holohedral crystals (holohedral refers to groups having as many planes as required for complete symmetry within a crystal system, in tetragonals therefore 4/m²/m²/m). Anatase gradually (from a temperature of over 700 °C more quickly) turns irreversibly into rutile. The density of anatase is 3.88 g/cm³. Anatase also serves as a white pigment. Titanium dioxide in the anatase phase is the main component of catalysts used for industrial denitration of flue gases in accordance with the SCR (selective catalytic reduction) process. What makes the anatase form so special is that it is much more photocatalytically active than rutile.

As natural titanium dioxide deposits reveal troublesome discolouration, which is caused for example by iron (FeTiO₃), they are refined in a sulphate pumping or chloride (preferable) process.

Titanium dioxide is thermally stable, resistant and has a high refractive index (2.8); consequently, it possesses excellent tinting and covering stability. Chemically, it is extremely inert, light-resistant and economical, and is considered to be non-toxic, making it the most important white pigment.

It was discovered in 1908 in Norway and in the USA and was produced there from 1909 under the name Kronos titanium white. In Germany production began in 1924 under the name Degea titanium white. Until 1938 titanium white was only produced in the anatase phase, but then increasingly in the rutile phase, which proved to be more stable than oleophilic materials and organic dyestuffs. For this reason, it was also called rutile white. Titanium white possesses the highest covering power of all white pigments and an excellent reducing power. It is chemically stable, non-toxic and is to be found under identification E 171 as a food additive, for example, in toothpaste and cough sweets. It is also frequently used in oil painting.

In industry, it is used in paint and coatings (e.g. for wall paint and textiles), in paper, as a UV blocker in sun creams and as a whitening agent in drugs (tablets). Furthermore, the dyestuff solar cell (Grätzel cell) is based on the semiconductive properties of titanium dioxide. Equally, titanium dioxide is used as the main component of the ceramic dielectric in class 1 ceramic capacitators.

The photocatalytic effect of TiO_2 nanoparticles is used in combination with UV activity in the area of tile finishing, which makes the surfaces of the treated tiles dirt-repellent. Such titanium dioxides are also used, for example, in water treatment, added to water or as a stationary phase through irradiation from ultraviolet light to remove bacteria and organic contamination.

We now devote our attention in the following experiment to the photocatalytic property of titanium dioxide in nanoscale form.

Demo object 1: Hemisphere with flip-flop effect

Paints, which 'change' colour when viewed from a diffe-Description of rent angle have the so-called flip-flop effect. This is brought experiment about by the finest titanium dioxide nanoparticles (TiO_2) . Their crystal sizes between 3 and 5 nm, these particles are a hundred times finer than titanium dioxide pigments, which are used as 'white pigments' in emulsion paints. The particles used here show clearly altered physical and chemical properties. Fields of application are the automobile industry but also everyday items made of metal, latex or plastic. Safety guidelines none In contrast to white pigments, titanium dioxide particles in **Observations** flip-flop paint reflect light in the yellow to blue spectrum of sunlight. Therefore, titanium dioxide nanoparticles give paint a yellow, red, green or blue tone, depending on the angle of vision. The flip-flop effect creates fascinating colour compositions as nanoparticles in specific amounts are added to scatter the blue light of the reflected light. The specially produced paints do not only change colour but also protect against discolouration, for example from artificial or natural light.







Materials

Experiment 8: Photocatalysis with titanium dioxide

Description of experiment

In this experiment we use the photocatalytic activity of anatase to remove (discolour) foodstuff colouring (murexide). As already mentioned, titanium oxide is found in two forms: as rutile or as anatase. Although both phases are the same in purely chemical terms, they differ in their electron band structure.

The band-gap energy in anatase is 3.2 eV (this corresponds to a UV wavelength of 388 nm) and in rutile 3.0 eV (this corresponds to a UV wavelength of 413 nm). For this reason, the catalytic effect of anatase is higher than that of rutile.

During photocatalysis a catalyst is stimulated by light, allowing the conversion of a substance. Titanium dioxide can trigger such a process as there is a layer of labile electrons on its surface. If these electrons are stimulated by UV rays (artificial UV lamps with wavelengths of between 320 and 400 nm or the sun) and they absorb sufficient energy, they can leave their position in the valence band and occupy high-energy positions in the conduction band. This leads to positively charged 'gaps', so-called valence band gaps.

The holes are able to react with water to become extremely reactive hydroxyl radicals (OH). The 'holes' as well as the hydroxyl radicals are extremely strong oxidants.

In this experiment, these cause the discolouration of the foodstuffs colouring murexide.

The photocatalytic reactions of TiO_2 can be used to remove germs or organic molecules (e.g. soot) by oxidation so that the surface is cleaned and disinfected.

Materials from the box:

- chemicals provided: titanium dioxide (rutile and anatase phases)
- murexide colouring
- 2 empty crimp-top glass tubes (50 ml)
- spatula
- pipette

Additional materials not provided:

 The experiment requires sun or UV light. If the weather is bad, it is preferable to use a UV lamp.

Safety guidelines Handle Even the

Handle titanium dioxide and murexide with great care. Even the tiniest amount of murexide can cause serious discolouration on clothing and on skin. Therefore, wear gloves, protective goggles and a laboratory coat.

If you are using a UV lamp for the experiment, do not look straight into a UV lamp without special UV glasses to protect you.

Procedure

Observations

- Take 2 empty tubes and label one 'A' for anatase and the other 'R' for rutile.
- Pour a teaspoonful of rutile into one of the tubes and the same amount of anatase into the other. In order to prevent any of one particular substance being carried over to the other, please clean the spoon or spatula after each step.
- 3. Add a drop of water to each tube and use the spatula to mix the water with the dioxide until you have a paste.
- Then add a tiny amount (1/4 of the tip of the spatula provided) of the colouring murexide to each mixture and mix the paste again until it becomes a delicate pink colour.
- Now remove the paste from the tubes and place it on a waterproof surface (e.g. in the two lids of the tubes) and leave in the sun for several hours.
- The reaction time is dependent on the intensity of the UV irradiation and in particular on the amount of murexide used.
- In the experiment only the anatase phase of the titanium dioxide causes the murexide colouring to discolour. On the other hand, rutile shows very little reaction to murexide because it scarcely undergoes photocatalysis.

On examining the paste, you can see that the top layer of the anatase paste in particular has discoloured (the part which is exposed to the sun) whereas the inner part remains pink.







Ferrofluids

errofluids are the only magnetic materials in liquid form. They consist of magnetic particles which are colloidally dispersed in a liquid. The particles contain or consist of ferromagnetic components such as cobalt, nickel or iron. The particles are stabilised on the surface with a polymer surface coating or through ionogenic groups and, as a rule, have a diameter of 10 nm. They are kept apart from each other by long-chain tensides (surfactants) or repel each other due to identical charges. Ferrofluids do not disperse with time and, even in extremely strong magnetic fields, they do not absorb each other or separate from the liquid as another phase.

The most frequent use of ferrofluids is currently in application as a seal, for example in hard disk drives in order to provide protection against dust. A ferrofluid used as a seal can be kept in its position by means of a permanent magnet; it can also resist strong pressure. The ferrofluid seals are not subject to wear and can constantly adapt to altered openings. They are also often used in loudspeakers in order to divert the heat between the voice coil and the magnetic array as well as to damp the movement of the membrane. The paint which makes aeroplanes invisible to radar is made of ferrofluids and non-magnetic substances, whereby the reflection of the radar waves is prevented. Due to their rise in temperature in an alternating magnetic field, ferrofluids can be used in the fight against cancer. For example, it is directly injected into the cancerous cells and then exposed to an alternating magnetic field from the outside. This causes overheating (hyperthermia) which kills off the cancerous cells.

Experiment 9: The magnetic field

Description

of experiment

Materials

- ferrofluid
- empty crimp-top glass tube

Materials from the box:

• tenside solution (surfactant)

the lines of force and try to follow these.

With the aid of ferrofluids, it is very easy to demonstrate

the magnetic field around a magnet, the field pulling from

the north to the south pole of a magnet and forming closed circuits. The ferrofluids align themselves according to

> Diagram showing composition of ferrofluid particles

- magnet
- pipette

Additional materials not provided:

water

Diagram tenside nanocore positive ion nanocore positive ion Ferrofluid 10nm

Safety guidelines

- Ferrofluids must be handled with great care and in a clean environment because they leave permanent stains! The work station must be protected at all times from contamination (paper towels, etc.).
- Wear a laboratory coat, gloves and protective goggles.
- If your skin comes into contact with a ferrofluid, wash the area with soap or any other ordinary hand-cleaning agent.
- Please keep ferrofluids in a sealed container at all times in order to prevent evaporation.
- Use the pipettes to fill the empty crimp-top glass tube with ferrofluid. Do not shake! Measure out slowly and carefully to avoid splashes.
- The ferrofluid and the materials soiled with the sub stance should be disposed of in the same way as motor oil (as hazardous waste or at a collection point) so please do not pour it into the waste water.
- Be careful with magnets in the vicinity of open ferro fluids: a magnet on the border of a bowl filled with ferrofluids will move them out of the bowl!
- Do not place magnets in the direct vicinity of a person with a cardiac pacemaker.

Procedure

- 1. Fill the tube up to the three-quarter level with water and add 2-5 drops of tenside solution (surfactant).
- 2. Then use the pipette to add a few drops of magnetic fluid, which will settle at the bottom.
- 3. Close the tube tight.
- If you now approach the ferrofluids with the magnet, you can clearly observe the porcupine formation and can move the ferrofluid through the liquid with the aid of the magnet.
- 5. Depending on whether the magnet is held parallel or vertically to the surface of the ferrofluid the orientation of the magnetic field changes and, consequently, also of the fluid.
- 6. Shake with care. The ferrofluid disperses in the water. As the ferroparticles do not dissolve, they eventually settle at the bottom. You can accelerate this process with the aid of a magnet, observing beautiful effects. To achieve this, draw the magnet past the side of the tube quickly and away again. In this way, you accele rate the ferrofluids and produce streaks, clouds, etc.

Additional experiment:

Instead of a permanent magnet you can also establish the field of an electromagnet in order to explain the development and the operating principle of coils. Depending on the size and amperage of the coil the magnetic field and, consequently, the orientation of the ferrofluids will change. You can also test the reaction on other magnetic materials such as metals, other magnets or lamps.

Observations

As the particles try to align themselves according to the magnetic field applied, a typical 'porcupine' is formed, the prickles representing the magnetic field lines. Surface tension of the fluids and gravity counteract the magnetic field with the result that ordered structures are created in the liquid as a reaction to the three forces.





Diagram showing magnetic field lines



Experiment 10: Density separation with ferrofluid

Description of experiment	In this experiment a non-magnetic coin, which first sinks to the bottom of the liquid because of its density, 'swims' on the top in the presence of a magnetic field.	Procedure
Materials	Materials from the box: • ferrofluid EFH1 1 • pipette • magnet • petri dish Additional materials not provided: • water • coin (non-magnetic)	
Safety guidelines	See previous experiment	

1. First, place the coin in the Petri dish.

- Now use the pipette to carefully fill with ferrofluid. (Please observe the safety measures).
- The coin remains at the bottom and is completely immersed in the liquid.
- 4. Now hold the magnet under the bowl. The ferrofluid is 'pulled to the bottom' and the coin rises from the bottom and can be 'rolled about' on the ferrofluid.
- 5. The porcupine structure can also be seen here to some extent. Ferrofluid residue on the coin stands upright.

This experiment can also be varied with other non-magnetic materials (aluminium, stones, brass, etc.). You can pour the ferrofluid back into the bottle when the experiment is completed.

Observations

In this density separation, pressure is created by means of a magnetic field gradient with which you can make substances with a higher density 'swim' or separate substances with different densities. This process is used, for example, in the automobile industry for recycling metals or for extracting gold and precious stones.

The magnet creates a force opposing gravity. The magnetite particles of the liquid are pulled to the magnet and, consequently, materials with a fairly low density begin to 'swim' on the ferrofluid.

You can vary this experiment with further non-magnetic materials. And always remember to be careful with magnets in the vicinity of ferrofluid solutions.







Nanoscale gold colloids

he Tyndall effect was first discovered by the Irish physicist John Tyndall (1820–1893) in 1868.

It is caused by the scattering of visible light (400–800 nm wavelengths) on suspended microscopically small particles, the diameter of which is in the order of magnitude of visible light. Such particles have a diameter of 1 to 1,000 nm. Certain phenomena are observed during this scattering of light (polarisation, colouring, etc.).

Systems which demonstrate the Tyndall effect are known as colloids (From the Greek 'colla' meaning glue) and the particles in such a system are colloid or colloidal particles. Colloid solutions contain particles with a diameter of 10⁻⁵ to 10⁻⁷ cm, which are finely spread (dispersed) in a liquid. The nanoscale colloid particles are so small that they can pass through a paper filter and they cannot be detected even under an optical microscope.

For example, in a tenside solution (surfactant) the colloid particles are micelles from the tenside molecules. Micelles (from the Latin mica meaning clusters), also known as association colloids, are aggregates (associates) formed by amphiphilic molecules or surface-active substances, which spontaneously form a cluster in a dispersion medium (usually water). As micelles are relatively large particle associates, tenside solutions behave in the same way as colloid solutions. The Tyndall effect enables a distinction to be made between suspensions (= a heterogeneous mixture of substances from a liquid and the finely dispersed solids in it) and real solutions (= a homogeneous mixture consisting of two or more chemically pure substances); nowadays, it is used in the field of ultramicroscopy, to name but one example. Other colloids are macromolecular solutions (e.g. a protein solution or starch solution) and disperse systems such as emulsions (e.g. milk) or suspensions (e.g. clay in water) or aerosols (e.g. cigarette smoke).

The Tyndall effect is a faster and more reliable way to prove the presence of all these colloid systems.

A simplified method of light scattering is nephelometry, also developed by Tyndall, which determines the intensities of scattered light as a measure of concentration of the colloid particles released or of floating aerosol particles. With the aid of light scattering measurements it is possible to measure the sizes of the colloid particles (e.g. to determine the molar masses of macromolecular molecules released). A perfect, practically scatter-free light is infra red light, which allows us to look through fog. A number of modern infrared techniques are based on this system. As far back as in the Middle Ages, man used nanoscale structures to colour glass, for example church windows, chalices or goblets.

Characteristic of this technique is the deep red colour (ruby red) produced by the small gold particles.

The colour which is produced depends on the metal particles used: while ruby red is achieved with tiny gold particles, silver particles can give glass a bright yellow colour. This effect was discovered as a byproduct of the alchemists' attempts to produce gold. The nanoparticles were produced by mixing metal dust, water and tannic acid from conifers.

The colour effect is caused by surface plasmon resonance. In this process the surface electrons of a metal are stimulated into oscillation by incident light, whereby only certain light wavelengths are absorbed. The wavelength of the light absorbed in this way depends on the size and form of the particles as well as on the density of the particles: the larger and more densely packed the particles are to each other, the greater the absorbed wavelength because the band gap of the particles is reduced as the size of the particles increases. Consequently, the wavelength of the reflected light reduces as the particles increase in size and the wavelength corresponds to the colour of the glass. Colloid gold is a dispersion of gold nanoparticles, generally with a diameter of 12–18 nm. This type of particle is also known as a gold cluster. A cluster is a collection of between 3 to 50,000 atoms. If clusters are spatially distributed in another physical medium, the entire system represents a colloid.

The particles are prevented from clustering by molecules in solvents, long-chain organic molecules or electrical charges on the surface of the particles as these have a repellent effect.

In the following experiment we will produce nanoscale gold clusters, which will easily be identifiable through the manifestation of a typical ruby red colour. One way of producing nanoscale gold is through the citrate method. This involves producing colloid gold or gold clusters respectively in a solution.

Gold particles can be applied in the field of electron microscopy, in which the antibodies tagged with gold are used as markers. The areas of the sample marked by antibodies are highlighted by the electron-concentrated gold particles.









Experiment 11: Identification of colloids through the Tyndall effect

Description of experiment	Using quite simple methods you will see how nanoscale, colloid systems in liquids can be detected through the Tyn- dall effect.	Safety guidelines	Wear personal protective equipment when working with diluted hydrochloric acid (laboratory coat, gloves and pro- tective goggles).
Materials	 Materials from the box: none Additional materials not provided: 2 glass beakers or snap-cap glass containers a concentrated source of light, e.g. laser pointer or small, bright torch common salt, tenside solution (e.g. washing-up liquid) 1 chicken egg diluted hydrochloric acid 	Procedure	 Pour water into a glass beaker. Add some salt and dissolve. In a darkened room, light up the solution with a point source of light. As the salt has completely dissolved, there is no visible effect. Now add a dash of soap or washing-up liquid and mix with the liquid; a path of light rays becomes visi- ble although the solution is still transparent. Additional experiment with biological material: Dissolve the white of an egg in approx. 300 ml of a 1 % salt solution. When viewed from the side, the ray of light emerges as a bright stripe (Tyndall effect). Now add diluted hydrochloric acid to the egg-white solution: the proteins settle and a whitish precipitate forms. On the top level you can no longer see a ray of light.

Observations

If a ray of light is directed sideways on a glass beaker containing a salt solution, the ray of light is not visible. In contrast, if the ray of light is directed through a colloid solution (e.g. tenside solution), it is visible as the light is scattered on the dissolved particles.







Experiment 12: Production of nanoscale gold

Description of experiment	In the following experiment we will produce nanoscale gold clusters, which will easily be identifiable through the mani- festation of a typical ruby red colour. One way of producing nanoscale gold is through the ci- trate method. This involves producing colloid gold or gold clusters respectively in a solution. The experiment is based on a redox reaction of tetrachlo- raurate (also known as tetrachlorauric acid or tetrachlo- rauric (III) acid trihydrate) in which gold ions are reduced to atomic gold clusters. The reducer sodium citrate does not only serve to reduce the gold but also acts as a di- spersion medium to stabilise the gold clusters created. By adding the reducer, the atomic coagulation of the metal ions is halted. The result is a colloid cluster enclosed by a ligand case.		 heat thermometer (up to 100 °C) laser pointer if required (not absolutely necessary) ethanol (e.g. methylated spirits) or propanol if required
		Safety guidelines	Auric chloride is caustic and is harmful to your health if swallowed (hazard classes R 22 and R 34).
		Procedure	 0.5 ml (approx. 15 drops) of the auric chloride solution are added to 28 ml distilled (or bidistilled) water. The solution is then heated on the stirrer or hot plate. At 100°C (If no thermometer is available you know it has reached this temperature when it starts to bubble), add 1.5 ml citrate solution as quickly as possible, at the same time stirring vigorously. The red colouring of the solution intensifies until it becomes a doop and colour.
Materials	 Materials from the box: auric chloride solution: tetrachlorauric (III) acid trihydrate (HAuCl₄) citrate solution: trinatriumcitrate dihydrate (C₆H₅Na₃O₇ x 2 H₂O) Additional materials not provided: distilled water hotplate or immersion heater, something for stirring (spoon, stirrer or similar), ideally a heatable magnetic stirrer 1 fireproof glass beaker (50-100 ml) 		 90 °C) it will take approximately 5 minutes until the colour changes. Sometimes violet colours also emerge. 4. The gold colloids can be detected through the Tyndall effect. In order to do this light is transmitted sideways through the solution with a laser pointer. The light path can be observed in the sol. The solution is extremely stable and no coagulation or sedimentation will be observed, even after several weeks.

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Additional experiment:

- For comparison, repeat the experiment with 0.5 ml Au solution to 50 ml distilled water. Compare the time needed for the change of colour to develop. If you increase the citrate concentration in a further experiment, the colloids will obtain a deep violet colour, which is a result of a different size of the emerging colloids.
- By adding ethanol or isopropanol to the red gold-colloid solution the gold colloids can be precipitated. This solvent contains ions oppositely charged to the gold colloids and so the ions neutralise the repellent force. In so doing, the red colour disappears and the particles begin to settle. However, this may take several days, depending on the conditions.

Observations

A ruby red solution emerges.

If a different concentration is used in the experiment, the solution may turn a violet colour. It is also possible to detect some colloids with the laser pointer in this solution although, in this case, the Tyndall effect is not as powerful as in the ruby red solution.





Chemical equation for the production of gold clusters



Demo object 2: Use of nano gold clusters in a pregnancy test

of experiment	experiment Research scientists use the light-absorbing property of gold particles to detect biomolecules. This allows antibodies to be tagged by coupling them with gold particles. If you then shine a white light through them, the metal particles light up in colour.	
	This is applied in some cases in pregnancy tests, in which nano gold particles are to be found finely distributed on the test strip. The attached antibody recognises the 'lut- einising hormone' in the urine, with which early pregnancy can be detected. A positive test result is indicated by a red stripe in the test window. This ultimed hCG pregnancy test is a monophase sandwich immune test for rapid detection of a pregnancy	Observations
Safety guidelines	• none	

Strictly speaking, the enclosed pregnancy test is intended as a demo object. However, if any of the schoolgirls actually wish to try the test out, there is nothing to stop them. You can obtain further tests in any pharmacy.

Choriongonadotropin (hCG) is a peptide hormone – to be more precise, a gonadotropin – which is formed in the human placenta during pregnancy and is responsible for preserving the pregnancy.

If you wet the tip of the test strip with urine, the colloid gold particles begin to dissolve in the liquid. If the urine contains the hormone hCG, the latter will attach itself to the antibodies tagged with gold particles. Due to their capillary forces, these particles are transported to the control windows. The anti-hCG antibodies with the gold particles conjugate with the lines there. Depending on the concentration of hCG, the intensity of the red colour appearing in the test line region will vary. The anti-hCG antibodies mobilised there absorb the complex made of hCG and colloid gold. The excess gold particles are then absorbed in the area of the control line by the antibodies immobilised there so that a red line is visible in this area. If there is no or only a little hCG in the specimen, the hormone and gold particle complex is not formed or an insufficient amount is produced to generate a visible colouring in the area of the test line.

A further field of application for gold particles would be in the field of electron microscopy, whereby the antibodies tagged with gold are used as markers. The areas marked by antibodies on the specimen are highlighted by the electron concentration of gold particles.









Memory effect

t should be made clear from the start that the memory effect is by no means an achievement of nanotechnology. It does not use nanoparticles or nanolayers to function. Nevertheless, the movement of a memory metal is, in the final analysis, based on a movement at a nanometer level – and that is reason enough to include this amazing experiment, which does not take long to perform.

Shape memory alloys (SMAs) are metallic materials, which, having been deformed by a change in temperature or forces, seem to remember their original shape and can return to it.

The memory effect was discovered as early on as 1951 in a gold cadmium alloy and first ascertained in a copperzinc alloy in 1956. The strongest memory effect has so far been demonstrated by nickel-titanium alloys (NiTi), making them particularly important in the technical field. The structural change of an SMA is dependent on a transformation of the crystal lattice of two different crystallographic structures. A change in temperature or application of an external mechanical strain will result in a diffusion-free phase transformation between the low-temperature phase (martensite) and the high-temperature phase (austenite). If the SMA is deformed in its martensite phase and subsequently heated in a specific temperature range, the components will remember their original shape and return to it.

The structural transformation does not occur by diffusing individual atoms but mainly by relocating atom groups, i.e. a movement into the nanometer dimension. In addition to obtaining these conditions of proximity between the atoms, a further reason for the reversibility of the transformation is found in the fact that only very low elastic stress occurs, which in effect prevents the plastic deformation from being irreversible through the relocation movement.

Memory metals are applied in the following fields:

- Medical technology (e.g. vascular implants, guide wires, needles, etc.)
- Astro- and aeronautics (e.g. in fighter planes as joining elements)
- Dental technology (e.g. tooth brace: this eliminates the need for retainers)
- Automobile industry (e.g. automatic transmission: for this application, the spring has the function of opening a valve at a certain oil temperature. In this case the need for electronic equipment could be eliminated, with cost-saving results.









Experiment 13: Memory metal – atom movements in the nanodimension

Description of experiment	This experiment involves NITINOL wire. NITINOL is the name given to almost all stoichiometrically composed compounds made of nickel and titanium which demon- strate the so-called memory effect. The wire used in this experiment possesses pseudo-ela- stic behaviour.	Procedure	 Iwist the wire provided into any shape you please. Place the wire in a water bath. Then start to heat the water up slowly. Use the thermometer to monitor the temperature. Establish the point at which the wire 'springs back' to its old form. It is important at this point not to heat the water any further and to remove the wire by means of the plastic pincers provided from the bath. The wire temperature should not exceed 70 °C, otherwise the effect will be irreversibly ruined. Apart from water, it is possible to use any other source of heat (e.g. a lighter). However, it is absolutely essential, in this case too, that the wire does not overheat as the memory effect could be ruined. Therefore, never hold the clip too close to the flame.
Materials	 Materials from the box: 'memory' wire in the form of a paper clip plastic pincers Additional materials not provided: heat thermometer water water dish water heater (hot plate, kettle or immersion heater) 		
Safety guidelines	• none		

Observations/ Explanation

Irrespective of the direction in which the wire was previously bent, the wire will always regain its original form if heated to exactly the same temperature as the wire 'remembers' its nominal shape.











From sand to chip

lectronics is playing an ever-increasing role in the field of nanotechnology. There is much talk about integrated nanocircuits or electronic nanostructures on chips. The aim is to make computer components and electronic chips as small as possible to achieve miniaturised components. In some types of microprocessors, memory components, laser diodes, LEDs, LCD displays, CCD chips (digital camera) such electronic nanocomponents have already become reality.

What are nanochips made of? How are they produced? This chapter aims to demonstrate how an extremely complex computer chip can be made from a relatively cheap raw material – namely silica sand. Nowadays, 95 % of all semiconductor components are made of silicon (Si). At a rate of 28 %, Si is the second most abundant element found in the earth's crust. (The most abundant element is oxygen, approx. 46 %.)

The electrical properties of semiconductors are resolutely determined by the number of foreign atoms ('impurities'). Therefore, it is important to have high-purity, monocrystalline Si as the starting material for the production of chips, so that the chips possess a reproducible property.

However, in nature silicon does not exist as a pure element but is often present in compounds as silicate or often as silica sand (quartzite, SiO2). First of all, silicon must be produced on a large scale in a high-purity monocrystalline form.

Mineral quartz is produced by cooling the SiO2-rich melt. Pure quartz is colourless and transparent. If it develops well-formed crystals, it is known as rock crystal or crystalline quartz (see specimen). Raw silicon is obtained from silica sand by reducing carbon in the electric arc at a temperature of 2100 °C according to the following chemical equation:

$SiO_2 + 2C \rightarrow Si + 2CO$

This process consumes a great amount of energy – 14 kilowatt hours (KWh) per kilogram of Si. Raw silicon (also known as technical silicon or metallurgical grade silicon (MGS)) retains a residue of approx. 2 % impurities, above all iron, aluminium, magnesium, calcium and carbon. To purify it further, raw silicon undergoes hydrochlorination treatment (hydrochloric acid is added) at 300 °C and is transformed into in gaseous tri-chloro-silane (SiHCl₃):

Si + $3HCI \rightarrow SiHCI_3 + H_2$

Any impurities are subsequently removed by (fractional) distillation of the trichlorosilane. By reducing the trichlorosilane with hydrogen at a temperature of 1100 °C hyperpure silicon is produced according to the following equations:

$SiHCl_3 + H_2 \rightarrow Si + 3HCl$

$$4$$
SiHCl₃ + H₂ \rightarrow Si + 3SiCl₄ + 2H₂

The Si settles on thin heated Si rods as a thick polysilicon layer.

Photo: Siltronic AG, Munich

Two processes are used to produce monocrystals from the polycrystalline Si:

- The crucible pulling method, whereby the polysilicon is melted in a silicon crucible (melting temperature of Si: 1413 °C), a monocrystalline, so-called seed crystal is dipped into the melt and slowly (1–3 mm/min) pulled out of the melt, resulting in a cylindrically for med monocrystal.
- Zone melting/refining: In the zone melting and refining process a small sector of the poly-Si rod is first heated inductively and melted in the vicinity of the seed crystal. The melt zone is then slowly relocated and the solidifying sector assumes the shape of a monocrystal.







Photo: Nano+Bio Center, Kaiserslautern



Crucible pulling method



In order to produce Si wafers the monocrystalline rod is turned until the diameter required has been reached and then cut with a diamond keyhole saw or separation disk into slices. The edges of the Si wafer are rounded off. Grinding, lapping, polishing, etching and a combination of these processes can be used to create a monocrystalline Si wafer with a perfect surface with parallel planes. Chips are produced from the Si wafers using lithographic processes. The word lithography originates from ancient Greek; it is composed of the words lithos = stone and graphein = write – writing in stone. Writing in stone is certainly not the method used to produce a chip. The first step is to write into a photosensitive layer. This photosensitive layer (also referred to as coating or resist) is applied to the wafer surface in the form of a thin film (typical thickness of the film $\leq 1.5 \mu$ m) (=coating).

The image of the structure to be transmitted into or on the Si wafer is found on a so-called mask. The image on the mask consists of transparent (translucent) and non-transparent sectors. The mask and coated Si-wafer are pressed together and the photosensitive layer exposed onto the transparent parts of the mask using rays of a specified wavelength. (=exposure).

Finally the exposed layer is developed in a developing bath (= developing) so that the image/structure is transmitted into the polymer layer. Step 1–3 are identical to the procedures used in classical photography before the era of the digital camera.

The polymer layer is a soft film which can easily be destroyed. Therefore, the structure in the polymer layer is transmitted into the Si wafer by etching or by coating the Si surface with metal (= structural transmission). The polymer layer is then removed as it is not actually a functional component of the chip but serves solely to transmit the structure.

In order to produce computer chips steps 1–4 are repeated up to 20 times with 20 different masks. Such a set of masks costs several hundred thousand euros.

The shorter the wavelength of the radiation used in the lithographic process, the smaller the structures which can be reproduced and transmitted. At present, wavelengths of between 365 nm (ultraviolet (UV) radiation) and 157 nm (deep UV (DUV) radiation) are being applied for industrial use. Nowadays, DUV radiation can be used to produce structures with as little as approx. 45 nm line width. The smaller the structures produced, the more integrated circuits fit onto a wafer or onto a chip respectively. For example, modern computer chips contain over 100 million individual transistors. This means that several hundred chips can be produced on one Si wafer. Once all the steps of the lithographic process have been completed, the Si wafer is sawed into individual chips. This is carried out by means of a so-called wafer saw with a diamond saw blade. Subsequently, the individual chips are glued into plastic or ceramic chip sockets and connected by means of the small metal legs of the socket through extremely thin (approx. 25 µm diameter) metal wires or gluings (flip-chip technology). Finally, the chip and socket are potted in plastic. And the chip is ready!







Zone melting/refining

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Demo object 3: Silica sand

Demo object 4: Rock crystal

Description	 Silica (quartz) sand refers to a type of sand which is composed for the most part of quartz grains. Silica sand is used as a raw material for chip production. Silica sand is also used in the glass industry to produce flat glass, floating and hollow glass, as well as in the foundry industry and in casting crafts as a moulding medium. White glass can be produced from sand low in iron (less than 0.03 % iron). Predominantly fine silica sand with a grain size of 0.1 to 0.4 mm and a residual moisture content of less than 5 % water (so-called wet sand) is used in melting furnaces. Silica sand is also used in the production of ceramics, in glass fibres, as traction sand, in abrasives and cleaning agents, as a foundry sand, as a shortening material, as a filler and as a blasting sand. Global reserves of silica sand are recorded as being practically unlimited. 	Description
Safety guidelines	• none	
		Safety guidelines

Quartz is the stable phase (transformation) on the earth's surface of crystalline silicon dioxide. Pure quartz is completely transparent and colourless and, if it develops well-defined crystals, is referred to as rock crystal. Due to microscopic inclusions of liquids and gases, quartz is generally milky and cloudy (milky quartz) and appears grey encased in the rock. Due to the integration of colouring ions (in general Fe³⁺ or Fe²⁺), the inclusion of coloured minerals or the effect of ionising radiation, quartz can appear in different colours. Dependent on the colour and the cause of the colour, a distinction is made between the following variations:

Amethyst: violet colouring as a result of the interaction between deposits of iron ions and irradiation with gamma rays Citrine: yellow to amber coloured quartz (also artificially produced through burning)

Ametrine: a rare quartz variety, which shows sectors with amethyst and citrine colouring in one crystal

Prasiolite (green quartz): a transparent, leek-green quartz, which is rarely found in nature and which is also artificially produced by burning amethyst or other yellowish quartz variations

Smoky quartz (morion): coloured greyish brown (smoky colour) to black (morion) by natural or artificial gamma rays Rose quartz: clear, pink quartz, coloured by integrating phosphorous into the crystal lattice

None

Demo object 5: Silicon wafer

Description

Wafers are slices of semi-conductor material on which electronic semi-conductor components, also known as chips, are produced. A chip consists of one or several integrated circuits which are connected with each other. An integrated circuit, on the other hand, is the result of the interconnection of a large number of transistors, condensers, resistance and inductivity components. In order to produce integrated circuits the electrical properties of Si are altered locally to a specific level by various lithographic processes.

Safety guidelines Silicon wafers are extremely fragile! Take great care when handling.







The smaller the particle, the greater the effect

he idea of using nanotechnology in defence systems is by no means new:

A study conducted in 2006 revealed that the special properties of Damascus steel, in particular, go back to carbon nanotubes. In the Middle Ages the Arabs forged swords out of Damascus steel to fight against the Crusaders. At that time, the Persians were using a forging technique which involved a complicated thermomechanical treatment of the steel, ultimately allowing nanostructures to develop, the secret of its exceptional strength and resilience. A cyclic process of heating and cooling resulted in nanotubes made of carbon, which in turn contributed to forming microscopically thin wires of cementite (iron carbide). Of course, the Damascus forges at the time were not yet aware of the existence of nanostructures. It is no wonder then that nanotechnology has captured the attention of defence ministries:

- Nanoparticles can provide higher performance in fuel for rockets or in the batteries of guided missiles.
- Nanoscale tungsten surfaces are ideal for hardening ammunition.
- Ammunition with nano explosive burns with more control and increased firing accuracy.
- Finally, more powerful explosions can be produced with nanofine explosives: the smaller the particle, the greater the reactive surface and, consequently, the greater the effect.

Now it would not be in our interest to allow schoolchildren to produce cannon shot or something similar in order to compare explosives with and without nanoparticles; this would be far too dangerous!

However, our last experiment also works with tiny, reactive particles which are, in this case, quite harmless: lycopodium spores.

Experiment 14: Spitting fire with small particles

Description of experiment

Lycopodium (also known under the common names of wolfpaw, foxtail or club moss) is a plant and representative of the snake moss family. It belongs to the genus of cryptogams.

In the past, lycopodium spores (spores from lycopodium clavatum) were used for medicinal purposes to dry wounds (that is the reason why it also bears the name of witch's flour).

Lycopodium was first used as a medicinal remedy for such ailments as cystitis, gout or rheumatism. In high concentrations lycopodium is poisonous. The spore rods are located in yellow ears and are ripe from July to August. In contrast to the stem and leaves, they are not considered to have any toxic effects. In the past, the spores were used as a type of anti-adhesive for pharmacists to 'roll pills'.

Nowadays lycopodium spores are used in pyrotechnic products and also serve in criminal science to reveal fingerprints (in combination with other substances).





Wrong! The pipette was held the wrong way round and the pressure was too low.



-

Description of experiment	Lycopodium spores have an average diameter of 30 µm (i.e. 30,000 nm). Although this is nowhere near the size of nanoparticles, this 'nothingness' is enough to give a powerful demonstration of how quickly and effectively small particles can react. You can also perform the experiment with flour or cocoa powder. Although these particles are somewhat coarser than lycopodium spores, it is also possible to produce a reaction with these particles (cf. flour dust explosions in mills).	Safety guidelines	There is no completely safe way to spit fire. As it always involves a degree of danger, it is obligatory that the expe- riment is performed by a person of full legal age. You spit fire at your own risk! It is absolutely essential that you use a tube to spit fire. Under no circumstances are you to spit into the source of fire directly from your mouth! Lycopodium spores are non-toxic. Nevertheless, if you get spores in your mouth, wash them out immediately with water as the spores dry out the mucus membrane of the
Materials	 Materials from the box: pipette to be cut up snap-cap glass container of lycopodium spores Additional materials not provided: source of fire (lighter, candle, burner) scissors 		mouth and this would leave you with an unpleasant thirst.

Procedure

- Cut the pipette provided with a pair of scissors at both ends so that you have a tube with approximately the following shape:
- 2. Now carefully allow the lycopodium spores to trickle down the tube and seal them off at the pointed end with your finger so that the spores do not fall out. Approx. 2 cm of spores in the tube are sufficient.
- 3. Now hold the tube horizontally, put the wider end to your lips and blow out the pores with a sharp exhalation (!) in a fine jet through or into a source of fire: lighter (set at high flame), candle with a long wick or a Bunsen burner. Make sure that the flames do not flash back onto your face. In order to avoid this, always give a short, powerful blast.
- Observations

Due to the small particle size lycopodium spores burn with an enormous darting flame.





Right!





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Colour reactions through photo catalysis, practical surfaces and coatings, working with ferrofluids, production of gold clusters and lots more ...

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