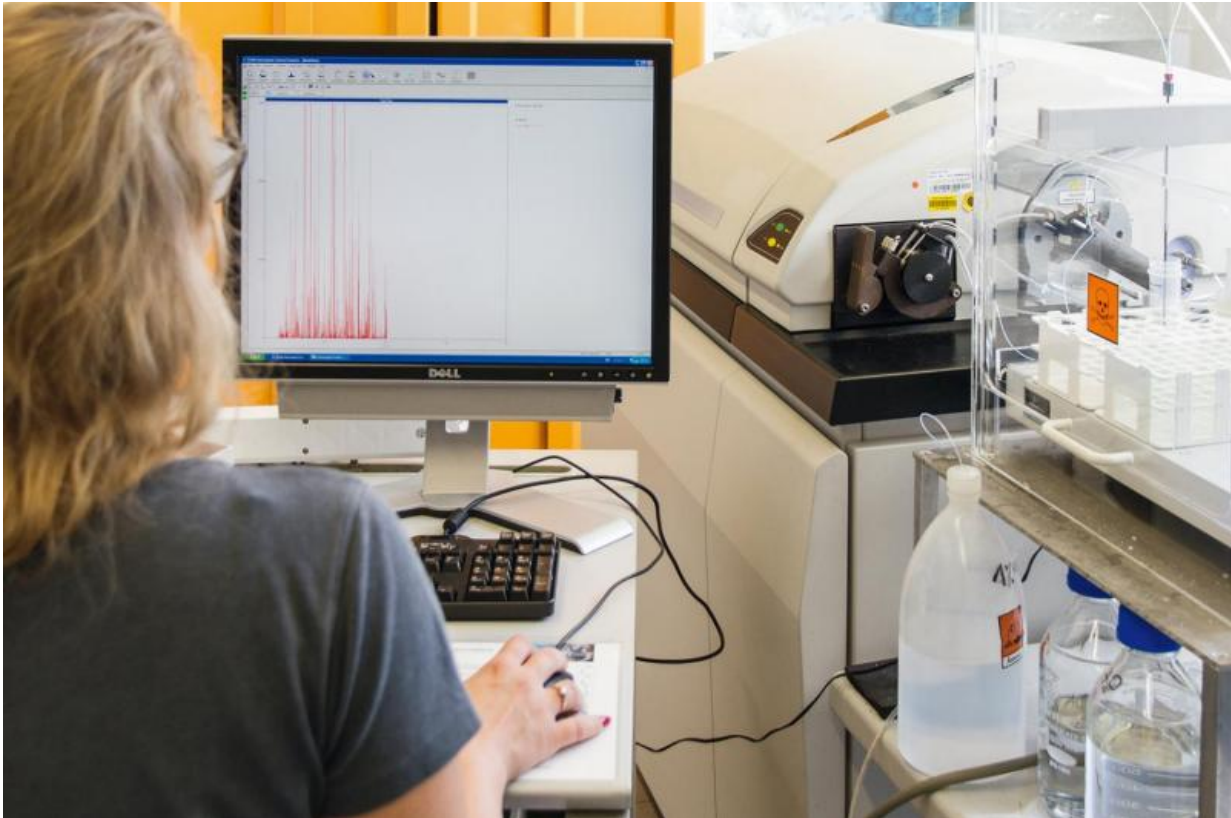


Proving nanoparticles in sunscreen products

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When a nanoparticle enters into plasma, a discontinuous signal occurs. Signal intensity correlates to particle size. Credit: Fraunhofer IGB

Loads of cosmetics like sunscreen lotions contain titanium dioxide. These nanoparticles are contentious. Experts suspect they may have harmful effects on people and the environment. But it is difficult to prove that the particles are in the lotions. Using a method developed by

Fraunhofer researchers, the particles can now be calculated.

Cosmetics increasingly contain [nanoparticles](#). One especially sensitive issue is the use of the miniscule particles in cosmetics, since the consumer comes into direct contact with the products. Sunscreen lotions for example have nanoparticles of titanium oxide. They provide UV protection: like a film made of infinite tiny mirrors, they are applied to the skin and reflect UV rays. But these [tiny particles](#) are controversial. They can penetrate the skin if there is an injury, and trigger an inflammatory reaction. Its use in spray-on sunscreens is also problematic. Scientists fear that the particles could have a detrimental effect on the lungs when inhaled. Even the effect on the environment has not yet been adequately researched. Studies indicate that the titanium oxide which has seeped into public beaches through sunscreens can endanger environmental balance. Therefore, a labeling requirement has been in force since July 2013, based on an EU Directive on cosmetics and body care products. If nano-sized ingredients are used in a product, the manufacturer must make this fact clear by adding "nano-" to the listed ingredient name. Due to requirements imposed by the legislature, the need for analysis methods is huge.

Determining particle sizes down to the tiniest scale

Today's electron microscope imaging processes, such as transmission-electron microscopy or scanning electron microscopy, are based on the properties of light dispersion. They are used to detect all particles present. They do not differentiate between a cell, a nanoparticle – or a piece of lint. These methods are ideally suited for the study of surface properties and shapes.

"The light diffusion process and microscopy are not selective enough for a lot of studies, including toxicological examinations," says Gabriele Beck-Schwadorf, scientist at the Fraunhofer Institute for Interfacial

Engineering and Thin Films IGB in Stuttgart. The group manager and her team have advanced and refined an existing measurement method in a way that allows them to determine titanium nanoparticles within complex media consisting of several different components that are highly sensitive and delicate. Researchers measure individual particles by single particle, inductively coupled plasma mass spectroscopy (or SP-ICP-MS). "With this method, I determine mass. Titanium has an atomic mass of 48 AMUs (atomic mass units). If I set the spectrometer to that, then I can target the measurement of titanium," explains Katrin Sommer, food chemist at IGB.

With particle measurement, a suspension is sprayed into the plasma that contains both large and small particles in non-homogeneous distribution. The suspension has to be thinned out sharply so that one [titanium dioxide](#) particle after another can be detected and analyzed. Ions are formed out of these particles in hot plasma of about 7,000 Kelvins. They get to the spectrometer's detector as an ion cloud, and are counted within the briefest measurement time of about three milliseconds. Signal intensity correlates to particle size. "We convert the intensity into nanometers. At the same time, we count particle signals, from which we calculate particle concentration with up to ten percent accuracy. We can establish exactly how many particles are of a specific size," says Sommers, explaining the procedure.

It was IGB scientists who originally developed the methods for measuring [titanium oxide](#) nanoparticles in wastewater. "But the process is generally suitable for complex media, and can also be applied to sunscreen lotions," the researcher indicates. A unique feature of this approach: the IGB team performs the data analysis and data processing without specialized software. "We have statistically evaluated the raw data using a standard computer program, and thus can work irrespective of the producer. Compared to existing methods, SP-ICP-MS involves a rapid process that uses detection limits that extend down to the ultra-

trace amount scale below ppm." For example, one sample of just a few milliliters can be examined in about six minutes.

Cosmetics makers, nanotechnology businesses, and consumers can benefit from the particle analysis for quality assurance of sun protection and body care products, but also use them for analyzing water, drinking water, and food. The researchers are planning to measure other nanoparticles in the future as well, such as silica dioxide. One can only determine whether a product contains silica dioxide through complex measurements. In order to establish the presence of nanoparticles, one must first determine their size or size distribution. Based on the EU's definition, declaration requirements apply to a nanomaterial if at least 50 percent of the contained particles are of a size measuring between 1 and 100 nanometers (nm). Previous analysis methods are hitting their limits here. These make it possible to establish particle sizes only in pure solutions. They are not suited for analysis of complex media that one finds in modern cosmetics. In addition, nanoparticles with various chemical properties cannot be differentiated from each other this way.

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