

Finding trace material in waste materials

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Credit: Swiss Federal Laboratories for Materials Science and Technology

Last year Empa's inorganic analytics lab was granted the status of "Reference Laboratory" within the scope of the ProSUM project, funded by the EU. Fine-grained samples of shredder waste from scrapped cars, e-waste or mine dumps from all over Europe end up here. Empa chemists find out what is in them, what is worth extracting and what could be dangerous for staff at recycling plants.



"Goggles on – and don't touch anything," says Renato Figi, head of Empa's inorganic analytics lab. As soon as we step into his lab, we realize that this somewhat unusual greeting makes perfect sense: There are beakers of green, yellow and tangerine-colored solutions standing in the fume hood. The tangerine beaker is covered with a watch glass, which Figi, wearing protective gloves, removes and carefully rinses with distilled water. "The beaker contains aqua regia, a mixture of concentrated hydrochloric acid and nitric acid." One drop on your shirt and you can kiss it goodbye; if it splashes in your eye, it's an immediate trip to the hospital for you.

However, it is not just laboratory visitors who need protection from the caustic solutions; it goes both ways. After all, we are talking about quantities measured in "parts per billion" (ppb). Or to put it another way: a billionth of a gram of the substance under investigation in one gram of sample substance. One speck of street dust, one flake of dandruff – this would already be fatal for the level of accuracy the Empa chemists are striving for.

For the ProSUM project, Figi and his team analyzed granulated samples from different waste groups: ground-up electrical and electronic devices, vehicle scrap, all manner of chopped-up batteries and mining waste. Decoding a sample begins in the dry – with an X-ray fluorescence analysis (XRF). "Thanks to this device, we can find every element that is heavier than fluorine with the atomic weight 19 up to uranium with the atomic weight 238," explains Figi. Many analytics labs in industry content themselves with this kind of analysis; their accuracy, however, ends in the percentage range.

In order to get down to the ppb level, you need good old wet chemistry: the samples are poured into a Teflon container along with concentrated <u>nitric acid</u>, hydrogen peroxide, aqua regia or even hydrofluoric acid and heated to temperatures of up to 280 degrees Celsius in a special



microwave oven. Figi: "This dissolves most things, bar a couple of fluoride compounds with rare earths." The liquid samples are then atomized and analyzed in 18,000-degree plasma. This is provided by two special devices called ICP-OES (Inductive Coupled Plasma-Optical Emission Spectrometer) and QQQ-ICP-MS (Inductive Coupled Plasma Mass Spectrometer). The resulting spectra – series of figures in a computer table – are evaluated by both Figi and his colleague Claudia Schreiner to avoid missing something. But this is when the real detective work begins.

Leaving traces – reading traces

As Figi knows: many elements that are only present in tiny amounts in the sample might be hiding behind other more dominant components. Iron and nickel, for instance, are close in terms of atomic weight, i.e. they are not always easy to tell apart in the mass spectrometer. However, the chemistry detectives have a trick up their sleeves: "We can remove the unwanted elements from the solution using a chemical reaction," explains Figi. "I precipitate the iron out of the solution as iron oxide, leaving only nickel behind in the mass spectrum – and I can determine the exact amount in the sample." Sometimes the chemist even goes a step further and adds a small amount of an element, which he suspects is in the sample, analyzes it under the spectroscope again and compares the results. This is known as "spiking".

"You end up amazed by all the things you can find in a normal, run-of-the-mill hair-drier," says Figi. Not just neodymium from the magnets for the drier's electric motor – that is to be expected. The Empa team also found traces of praseodymium and samarium in the samples. "Looking for traces isn't just about recycling the waste in the most lucrative way possible," says Figi. "It's also a question of protecting the staff at recycling plants from being poisoned." For instance, if it comes into contact with acids, a high arsenic content in a <u>sample</u> can cause arsine to



form – a notorious poison gas from World War I. "It strongly smells of garlic," says Figi. "One whiff and there's only one thing to do: get the hell out of here!"

Provided by Swiss Federal Laboratories for Materials Science and Technology

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