On the Trail of Rocks

Geological Age Determination by Means of Ultratrace Analysis in the Picogram Range

Customer: Institute for Geochemistry at Tubingen University, Germany

Many people bring them back from vacation near home or overseas: rocks and minerals are very popular and fascinating because of their form, color and, above all, the enchanting stories associated with them. At the Institute for Geochemistry at the University of Tubingen, researchers are investigating the structure and chemical composition of the earth's crust through analysis of rocks and minerals. The locations involved range from the Bavarian Forest, Turkey, the Himalayas, to China and various regions in Africa. Elmar Reitter works as a technician at the Institute for Geochemistry. He was interviewed by LABO regarding his work in the area of ultratrace analysis.

Background Information

At the Institute for Geochemistry of the University of Tubingen, researchers are investigating the structure and chemical composition of the earth's crust using rocks and minerals. The locations involved range from the Bavarian Forest to Turkey and the Himalayas, and even as far as China.

ELGA is a global brand in the area of delivering ultrapure water for use in laboratories. ELGA is an integral part of Veolia Water Solutions and Technologies who is a leading design and build company and a specialized provider of technological solutions in water treatment. The company's array of products includes a wide range of solutions for its customers: from building swimming pool technology, power plants and industrial enterprises such as the beverage, food and pharmaceutical industries to laboratories, international aid organizations, ships and municipalities.

Photo 1 – Ultra trace analysis in a cleanroom lab.

Editorial staff: What projects are you currently working on?

Elmar Reitter: Mostly I work in geochemical isotope analysis. It is a small but important building block of projects in which various problems are tackled. It involves geochronological work to reconstruct regional and plate-tectonic processes. In the end, our investigations will help shed light on the complex geological history of various parts of the earth. In addition, we also work with other disciplines, such as archaeometry, where, for example, strontium isotropy in human teeth is studied in order to reconstruct dietary habits and migration patterns during the early history of man.

Editorial staff: *How many rock samples do you analyze each year? And are there any rocks you consider particularly fascinating?*

Elmar Reitter: Luckily we are not a routine laboratory that earns its living from a high throughput of samples, but rather a teaching and research facility.
At least our samples do not deteriorate as easily as most of them are many millions of years old.
What impresses me in particular as a chemical engineer are the diverse optical characteristics of rocks and minerals. On close inspection all are different and each rock has its own history.

Editorial staff: *In geochemistry the chemical composition of rocks and minerals and their main and trace elements are studied. In addition to chemical methods, examinations with extremely low detection limits are carried out. How is this done?*

Elmar Reitter: Procedures can differ greatly depending on what is being investigated. With uranium(U)-lead(Pb) dating, i.e. age determination, of minerals such as zirconium, this mineral is elaborately separated from the whole rock, for example granite. Under cleanroom conditions, the zirconium particles (100 to 200 µm) are picked up one by one by means of a stereo- microscope and weighed on an ultra-microbalance (used for weights up to 0.1 µg), transferred to a small, very clean Teflon disintegration vessel, enclosed and exposed to hydrofluoric acid under pressure and a temperature of 210°C for a week. U and Pb are then separated from the dissolved sample solution by means of ion chromatography, also under cleanroom conditions. The elements obtained in this manner are then analyzed with respect to their isotopic composition using a high-resolution thermal ionization mass spectrometer (TIMS). This is a decisive step as we are able to determine age on the basis of lead isotope ratios and the concentration of uranium isotopes. Depending on the objectives of the research project, this can be of decisive importance.

Editorial staff: *Besides checking for the elements lead and uranium in your geological age determinations, you also test for elements such as rubidium and strontium, as well as rare ones such as neodymium and samarium. What role does ultrapure water play in your investigations in this connection?*

Elmar Reitter: Exactly the same conditions apply in the analysis of “rare elements” as for lead and uranium. The “rare” aspect in this case only relates to their frequency of occurrence in the earth’s crust. For neodymium, for example, the content is

about 0.0024%, which, however, adds up to a considerable amount, considering the large mass of the earth.

Ultrapure water, as a universal solvent, plays a central role in all our investigations, as all work such as cleaning of sample vessels, disintegration of samples, and ion chromatography takes place in a water based environment. Only with high-quality ultrapure water is it possible to complete analyses in the picogram range. In other words, analysis in this weight range only makes sense if contamination levels, such as from lead, lie in the femtogram range. By way of illustration: 1 picogram per gram (ppt) is equivalent to finding a single grain of rye (0.1 g) in approximately 2000 trucks each loaded with 50 tons of wheat; 1 femtogram per gram (ppqu) is equivalent to finding a single human hair among all human hairs on earth. The validity of an analysis stands or falls with this factor.

Editorial staff: *What parameter limits must be satisfied with water used for ultratrace analyzes of rocks and minerals?*

Elmar Reitter: In our special cleanroom we use the ELGA PURELAB Ultra Analytic pure water system. With this system we achieve blank values for lead <100 ppqu, and <3 ppgu for uranium. Besides the blind values in water of elements of interest, the specific resistance of 18.2 MΩ-cm and a low TOC value range of 1 to 2 ppb are of major importance. Thus, with the correct water treatment we are given the basis for carrying out high-quality analyzes.

Editorial staff: *You base your work on the technology of ELGA. What were the most important criteria for making this decision?*

Elmar Reitter: Ultrapure water is indispensable in ultratrace analysis applications. In addition to the good blind values for elements to be evaluated, the continuous real-time TOC-value monitoring and periodic recirculation of pre-treated water (with the PURELAB Option water purification system) gives us the security we need. Besides the high water quality, economic considerations, good counselling and reliable customer service were decisive criteria for us.

Editorial staff: *Could you give us an idea about what future demands on your laboratory might be?*

Elmar Reitter: As the tendency is going in the direction of ever smaller samples, for example, in the dating of single minerals or their fragments, we will attempt to lower detection thresholds also for other elements. Purchase of additional ICP-MS devices is planned. With our, our cleanroom, and the ultrapure water unit installed there, we will be in good shape to meet the challenges of future requirements.

For more information about ELGA LabWater and its PURELAB range of water purification systems please visit www.elgalabwater.com.

This article was printed in the Labor Journal (February, 2010).