Geoscientific Laboratory

November 2011

Quality with Confidence – Safety with Quality

The Geoscientific Laboratory carries out geochemical and geotechnical studies related to the safe disposal of environmentally hazardous waste. Equipped with specialist expertise, valid methods and precision instrumentation, the laboratory looks into the issues of radioactive and toxic wastes as well as the issue of CO_2 – which is currently the topic of public debate - on the basis of the latest scientific and technical standards.

The Geoscientific Laboratory complies with the European International Standard EN ISO/IEC 17025:2005 and therefore also operates in accordance with EN ISO 9001:2008. The accreditation by the regulatory body for recognising the competence of the laboratory is aimed at for 2012. Furthermore, we aim at accreditation according to the European International Standard EN ISO/IEC 17020:2011 with the objective of promoting confidence in a body performing inspections in specific issues.

You ask – we answer. The focus of our work is on the three questions:

- Which components are in a substance?
- How do substances behave under certain conditions?
- How can properties of substances be changed?

Reliable results: By means of a large variety of instruments, the laboratory analyses substances and elements qualitatively and quantitatively to reliably capture even single ions. Moreover, the experimental results are cross-linked with theoretical and modelling work. A high validity of the results is therefore guaranteed.

Fields of application: The analysis results of the laboratory are primarily utilised in the field of research on and safety analyses of final disposal, geothermal energy generation, and chemical safety. They provide a basis for hypotheses, models, and simulations used to predict and to prove the behaviour of waste, host rock and the biosphere.

Concretely: Amongst other things, we investigate where and how the radioactive and toxic wastes can be disposed of in the long term and in a way that no hazardous substances will reach the biosphere. To find answers to these questions, it is necessary

to characterise the host rock, to test and to improve the backfill and sealing materials, and to carry out geotechnical studies of the construction elements. Furthermore, this work focuses on mutual interactions of the hazardous substances and on their behaviour during the possible transport through the geosphere.

Services

The instrumentation of the geoscience laboratory allows determining geochemical and geotechnical parameters for various substances relevant for final disposal and under various boundary conditions. To work on wide-ranging requests, a number of methods and procedures are applied which are newly combined for each contract.

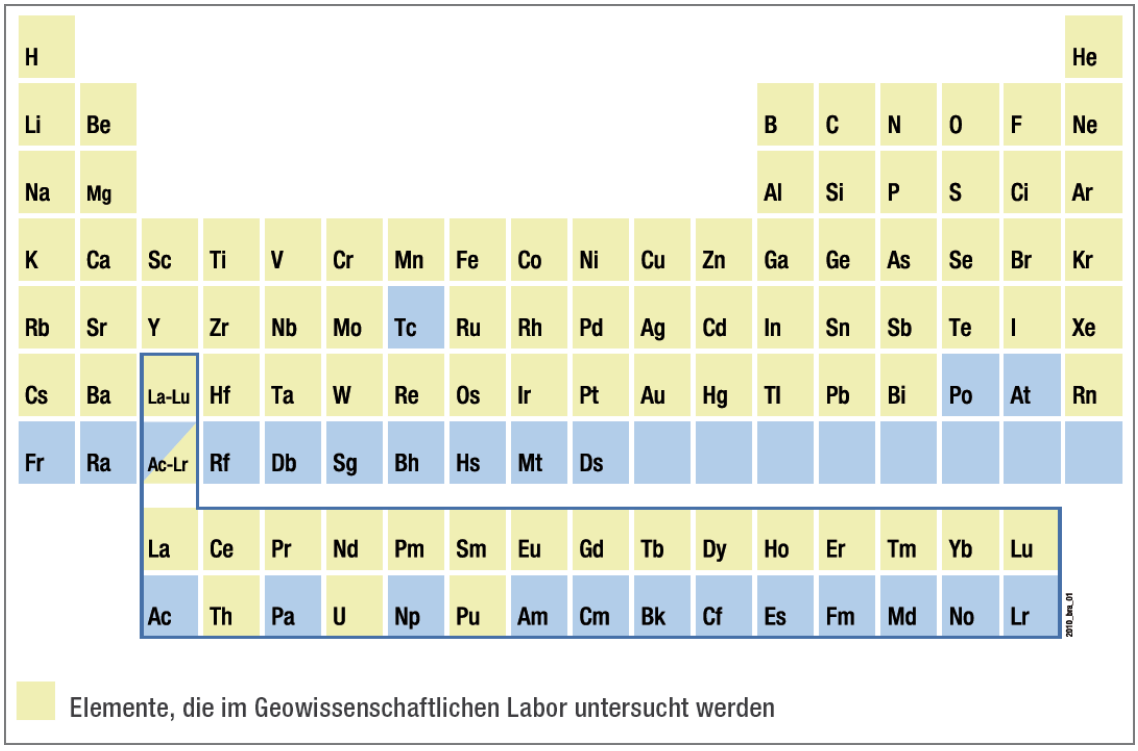
1. Substances and Elements that can be analysed

Due to its specialist expertise and technical equipment, the Geoscientific Laboratory is capable of analysing almost all solid, liquid, and gaseous environmental substances.

Substances that can be analysed

Solids	Liquids	Gases
➢ Wastes ➢ Building materials (e.g., backfill and sealing materials) ➢ Rocks and soils (e.g., salt, clay, granite) Minerals	➢ Leachates (experimentally obtained solutions) ➢ Groundwater	➢ Natural gases ➢ Reaction gases (e.g., release from waste)

Elements that can be analysed



2. Methods and Procedures of the Geoscientific Laboratory

The Geoscientific Laboratory carries out geochemical as well as geotechnical investigations. A further focus is on in situ sampling (e. g., rocks, groundwater) under natural conditions and subsequent preparation of the samples for specific questions and investigations.

Geochemical investigations

Geochemical investigations consider material composition, distribution, stability, and the cycle of chemical materials and elements as well as their interaction under specific boundary conditions in minerals, rocks, soils, water, the atmosphere, and the biosphere. The Geoscientific laboratory performs geochemical investigations of radioactive and chemical-toxic materials. These investigations provide necessary hints to be considered for the establishment of disposal concepts.

Geotechnical investigations

Geotechnical investigations are to look into the interaction of engineered structures and their foundations. The Geoscientific Laboratory has been specialised for geotechnical research relating to final repositories. The researchers are concerned with questions like "what happens in a repository in case of an intrusion of water?" or "how does heat generating high-level waste affect the stability and permeability of host rocks?" The objective of the geotechnical studies is to observe and understand the geo-mechanical behaviour of host rocks and of underground engineered constructions under the impact of heat, pressure, stress, and water.

Concept and design of test assemblies

By its competences the Geoscientific Laboratory has the ability to design, construct and calibrate specific geochemical and geotechnical measuring devices/systems. Thermostats, air-conditioned cabinets as well as a glove box are available for the arrangement of special experiments.

In-situ investigations in Underground Research Laboratories (URL)

Besides the investigations in the above-ground laboratory on a pilot plant scale the Geoscientific Laboratory also performs geotechnical and geochemical experiments in natural and/or repository-relevant environments.

Examples of URLs the staff of the Geoscientific Laboratory is working in are:

- Hard Rock Laboratory Äspö in Sweden (crystalline rock)
- Asse in Germany (rock salt)
- Bure in France (argillaceous rock)
- Grimsel, Switzerland (crystalline rock)
- Mont-Terri, Switzerland (argillaceous rock)
- Teutschenthal, Germany (rock salt)

Here, the laboratory work comprises the drilling of test boreholes, instrumentation, execution and evaluation of geo-electric, geo-mechanic, hydraulic, and geo-chemical experiments.

Sampling and preparation

The quality of a specimen strongly influences the overall result of an analysis. Therefore, it is an essential task of GRS' Geoscientific Laboratory to take samples intended for geochemical or geotechnical investigations, to transport them as well as to prepare them in a suitable manner.

Sampling: The staff of the Geoscientific Laboratory has long-standing experience in the sampling of solid matters, solutions and gases. This experience applies to the test specimen itself, but also to in situ-sampling at a specific site, e.g. a well, a shaft, or a mine. With it, specific methods for sampling of high-saline solutions have been developed, in particular for smallest amounts via capillaries.

Preparation: The preparation of solid matters, solutions and gases follows DIN-procedures (German Institute for Standardization) or specific procedures which have been developed in the Geotechnical Laboratory itself. Problematic solid and fluid samples that are difficult to analyse undergo special digestion techniques like fusion, microwave digestion or chelation for separation and enrichment.

Making of specimens: The staff of the Geoscientific Laboratory are experienced in making specimens of different solid matters with regard to an underground repository, e.g.

- Rock specimens (salt-rock, clay-stone, crystalline rocks)
- Construction materials (backfill and sealing materials, cement, concrete)

Using this experience, the specimens are either drilled directly from the massive rock at the site or turned into necessary sizes at the lab.

3. Safety and Quality

Safety and quality - both terms are inextricably linked at GRS. The main objective of many research projects, performed by the Geoscientific Laboratory on behalf of public contractors, is to guarantee safety concerning the disposal of radioactive as well as hazardous chemical waste. Based on this responsibility, GRS claims to produce high-quality-assured results only. Therefore, all research activities are performed in due consideration of the highest standards with regard to competence, technical safety and environmental protection as well as precise documentation.

Competence and diversity

GRS' Geoscientific Laboratory has long-standing experience in the scientific processing of problem definitions requiring the expertise of manifold specialist disciplines. Its staff members therefore are skilled in various disciplines like chemistry, mining, mechanical, electrical, and fine engineering. Beside the work on research projects, the outstanding professional competence is encouraged by self-education and training measures as well as by specific exchanges with scientists and institutions at home and abroad.

Contact in all matters of the Geoscientific Laboratory is Mrs. Tina Scharge (manager) resp. Mr. Karsten Hellwald (assistant manager) and Karin Schwarzl (assistant manager).

Safety and environmental protection

Claiming to work according to the state of the art in science and technology implies the steady maintenance and screening of all instruments and test materials used in the geoscientific lab. All relevant quality assurance measures are documented. Recurrent internal as well as external inspections ensure that safety standards are up-to-date and applied.

Beside a high level of technical safety, environmental protection according to all relevant applicable regulations is self-evident for GRS.

Documentation and archiving

GRS' approach towards quality, based on the constraints of certification according to DIN EN ISO 9001, requires gapless documentation of all crude data and their archiving as data files as well as printed hard-copies for at least 10 years. A laboratory

information and management system (LIMS) ensures documentation and traceability of all data gained according to national and international standards.

4. References and Co-operation

Clients

Ordering customers of research projects are generally various federal ministries and federal authorities, such as the

- Federal Ministry for the Environment, Nature Conservation and Nuclear Safety (BMU)
- Federal Ministry of Economics and Energy (BMWi)
- Federal Ministry of Education and Research (BMBF)
- Federal Office for Radiation Protection

as well as the Commission of the European Union (EU).

The results of these research projects are documented in GRS' publications – the GRS reports – on the topics of waste disposal and repository safety. All GRS reports can be downloaded from the GRS website (www.grs.de).

Apart from serving governmental customers, the Geoscientific Laboratory of GRS is also available as a contractual partner to private enterprises and industry.

Co-operation partners

Research projects, especially if interdisciplinary, often require a broad repertoire of methods, instruments, knowledge, and expertise as well as a close exchange of experiences with national and international partners. By co-operation the competences of the different partners are added and new possibilities for investigating material behaviour are established.

Among others, co-operation partners of the Geoscientific Laboratory are the

- Technical University of Clausthal
- Technical University of Braunschweig
- Research Centre Jülich

- Nuclear Research Institute (NRI) of the Czech Republic
- Bundesanstalt für Geowissenschaften und Rohstoffe (BGR - Federal Institute for Geosciences and Natural Resources)
- Helmholtz-Zentrum Dresden-Rossendorf e.V., Radio-Chemistry Institute
- Institut für Gebirgsmechanik GmbH (IfG – Institute for Rock Mechanics)
- Agence nationale pour la gestion des déchets radioactifs (ANDRA), France
- Mont Terri Project, Switzerland
- Karlsruher Institut für Technologie (KIT), Nuclear Disposal Institute

Numerous German and foreign universities add to this list.

5. Investigation Methods

Absorbable halogenated hydrocarbons (AOX)

Instrument	Task	Field of Application	GRS Equipment
liquiTOC II	Determination of sorbable halogenated hydrocarbons	Liquids	IR detector with two sensitivity domains

Atomic absorption spectrometry

Instrument	Task	Field of Application	GRS Equipment
AAS spectrometer ZEEnit 60	Quantitative determination of elements	Solids and liquids	➢ Graphite tube technique ➢ Zeemann underground compensation ➢ High-resolution double monochronomator ➢ CCD detector ➢ Coupling with hydride system

Determination of chemical-physical parameters

Instrument	Task	Field of Application	GRS Equipment
One-bar measuring chains (after Ross)	Quantitative determination of hydrogen ion activity (ph-value)	Liquids	➢ Measurement in saline solutions ➢ Special electrode specification
Falling-sphere viscometer	Quantitative determination of viscosity	Liquids	➢ Peltier tempering
Analytical balances and calibrated volume containers	Density determination	Solids and liquids	➢ High-resolution analytical balance
Density swinger	Density determination	Solids and Liquids	➢ High-sensoric swinger ➢ Peltier tempering (1/1000 °C stability) ➢ Temperature range: 0 to 90°C ➢ Compatible with GMP/GLP and FDA determination (e.g. 21 CFR Part 11)

Uniaxial strength determination

Instrument	Task	Field of Application	GRS Equipment
Uniaxial testing machine	Uniaxial load and deformation testing	Solids	➢ Maximum axial force: 2000 kN (approx. 50 MPa) ➢ Maximum testing temperature: ambient temperature ➢ Sample size: 300 mm diameter, 600 mm height (cylindrical)

Measurement of thermal parameters

Instrument	Task	Field of Application	GRS Equipment
Hot Disk Thermal Constants Analyser	Measurement of thermal conductivity, specific heat, and heat capacity	Solids, liquids, and gases	➢ Maximum testing temperature: 100 °C ➢ Maximum relative humidity: 100 %

Photometry

Instrument	Task	Field of Application	GRS Equipment
UV-2450 (Shimadzu)	Quantitative and qualitative determination of colour complexes	Liquids	➢ Spectrometer with 2-ray optics ➢ Wavelength range: 190–1100 nm ➢ Holographic grid ➢ Arrangement for maintaining temperature ➢ Compensation of colloids

Gas chromatography

Instrument	Task	Field of Application	GRS Equipment
Micro-gas chromatograph CP-9400 (Varian) and Trace GC ultra (Thermo)	Qualitative and quantitative analysis of mixtures	Gases	➢ WLD and PDD sensors ➢ Measurement range: 10 ppb – 1000 ppm

Geo-electric tomography

Instrument	Task	Field of Application	GRS Equipment
Geo-electric measuring equipment RESECS	Measurement of spatial-specific electric resistance	In-situ and laboratory measurement for water content distribution in rocks and soils	➢ Two devices with up to 112 and 240 electrodes

Ion chromatography

Instrument	Task	Field of Application	GRS Equipment
Advanced Compact IC (Metrohm)	Qualitative ion determination	Liquids	➢ Conductivity detection with chemical suppression ➢ Cation and anion determination ➢ sample changer

Karl-Fischer-titration

Instrument	Task	Field of Application	GRS Equipment
Titrando 841 (Metrohm)	Quantitative determination of water	Solids and Liquids	➢ Dosing system for volumetric water content determination ➢ High-end device for concentration ranges from ppm to 100% ➢ Distinction between different binding forms of water in solid samples ➢ Differentiation of free and bound water

Carbon analysis (TC)

Instrument	Task	Field of Application	GRS Equipment
Thermo TOC 1200	Quantitative carbon determination	Solis and liquids	➢ IR detector with two sensitivity ranges

Inductively coupled plasma mass spectrometry (ICP-MS)

Instrument	Task	Field of Application	GRS Equipment
ICP-MS spectrometer Thermo X Series + New Wave Research Laser ablation UP-213	Quantitative and qualitative determination of element traces and ultra-traces	Solids and liquids	➢ Integrated 1600 W "solid state" quartz-controlled 27 MHz ICP generator ➢ Plasma screen plus technique ➢ "off-axis" high performance quadrupole with abundance sensitivity and peak form mass filter ➢ Mass range: 2 – 255 AMU ➢ CCT technique ➢ Coupled to a laser excitation unit (ICP-MS-LA)

Inductively coupled plasma optical emission spectrometry (ICP-OES)

Instrument	Task	Field of Application	GRS Equipment
ICP-OES spectrometer IRIS Intrepid II XUV	Quantitative and qualitative determination of principal elements and element traces	Solids and liquids	➢ Low-noise CID detector ➢ New designed Echelle spectrometer ➢ Radial or plasma-duo view ➢ Full framing of pictures for records or re-analysis ➢ Enhanced UV-radial view for halogen analysis ➢ 2000 W HF generator at 27.12 MHz ➢ Wavelength range: 130–1050 nm

Permeability measurement

Instrument	Task	Field of Application	GRS Equipment
Autoclave	Measurement of hydraulic conductivity and gas permeability	Solids	➢ Sample sizes: 50 – 150 mm diameter ➢ Maximum confining stress: 25 MPa

Polarography

Instrument	Task	Field of Application	GRS Equipment
797 VA Computrace Metrohm	Quantitative determination of ions and their speciations	Liquids	➢ Sample changer ➢ Dosing system for automatic addition of auxiliary solutions

Pore pressure and permeability *in-situ* measurements

Instrument	Task	Field of Application	GRS Equipment
GRS mini piezometer	In situ measurement of pore water pressure, gas entry pressure and permeability against gases and pore fluids	Different rock formations, e. g., argillaceous rocks, salt rocks, crystalline rocks	➢ Minipacker system for small borehole diameter (20 mm) for minimisation of rock damage and for near-surface measurements

Pore space properties

Instrument	Task	Field of Application	GRS Equipment
Ultrapycnometer 1000	Determination of pore volume and density	Solids (e. g., powder, granulates, and solids)	➢ Standard equipment

Potentiometric titration

Instrument	Task	Field of Application	GRS Equipment
Titration device Titrando 857	Quantitative determination of ions in solutions	Liquids	➢ Sample exchanger ➢ Control and data unit ➢ Automatic electrode identification ➢ Ion selective electrodes ➢ Compatible with GMP/GLP and FDA determinations, as for instance 21 CFR Part 11

Radon scintigraphy

Instrument	Task	Field of Application	GRS Equipment
EQF 3220 (Sarad)	Determination of radon activity and radon decay products	Gases and aerosols	➢ Radon decay product detector head

X-ray diffractometry (RDA)

Instrument	Task	Field of Application	GRS Equipment
X' Pert MPD/MRD (Phillips)	Qualitative determination of mineral phases	Minerals	➢ X-ray tube with molybdenum or copper anode ➢ Primary ray with characteristic K Mo 0.71 (A°); Cu 1.54 (A°) ➢ Vernier adjustment of mono-crystal in primary ray ➢ Detector records position and intensity of each reflex ➢ Closed system with integrated sample change

Triaxial strength investigation

Instrument	Task	Field of Application	GRS Equipment
Triaxial testing machine	Triaxial loading and deformation testing	Solids	➢ Permeability measurement ➢ Ultrasonic measurement ➢ Investigation of scaling effects ➢ Maximum axial force: 1600 kN (approx. 50 MPa) ➢ Maximum confining stress: 70 MPa ➢ Maximum testing temperature: 100 °C ➢ Sample size: 280 mm diameter, 600 mm height

Equipment-specific overview of elements analysed