

The General Laboratories of the Geological and Mining Institute of Spain, CSIC / Technical Vice-Directorate

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Abstract

The General Laboratories of the Geological and Mining Institute of Spain, CSIC, is one of the CSIC laboratory centers that make up the Materials Science and Technology Area of the Higher Council for Scientific Research (CSIC). Its activity is based in giving support to research studies related to Earth Sciences materials as well as resources and environment.

The laboratory is assigned to the Technical Vice-Directorate of the IGME, CSIC and it is organized in four Laboratories (Water, Mineralurgy, Geochemistry and Technological Tests and Natural Stone) and an Applied Microscopy/Artificial Vision Tests unit.

This document summarizes the main lines of activity developed and the equipment available.

The IGME General Laboratories

The IGME General Laboratories are a unit that provides specialized technical services. It provides experimental technical data in support of the research projects of the Organism itself and other Organisms and Entities, both public and private, testing a wide range of samples related to earth sciences, such as rocks, drilling cores, soils, sediments, water and leachate. The capacity to carry out analysis and testing covers the entire process, from the initial preparations of the different materials to their analysis or testing by different high precision and accuracy techniques required to identify and quantify basic elements in the evaluation of resources.

Other services provided include the management of laboratory equipment and instruments, technical advice on instruments, equipment and tested materials, support for the training of laboratory technical personnel through the provision of practices and waste management in accordance with current regulations.

Its headquarters are placed in Tres Cantos (Madrid). According to the organization chart, it is divided into four laboratories (Water, Geochemistry, Minerallurgy, and Technological Tests and Natural Stone) and an Applied Microscopy/Artificial Vision Unit, which are independent but complementary to each other and act in a coordinated manner.

The Laboratories have established a Quality Management System based on the requirements of the UNE-EN ISO/IEC 17025 Standard, managed through an application (SILAB) developed by the Area of Geoscientific Information Systems and Institutional Databases of the IGME itself, which promotes the organization and management of work in a computerized way. IGME General Laboratories are part of the Network of Laboratories of the Community of Madrid (RedLab 192) and have an ENAC accreditation to carry out physicochemical analysis of natural mineral waters, spring waters, mineral-medicinal waters, mineral-industrial and thermal waters.

To guarantee the quality of the analysis and test results, the General Laboratories participate in interlaboratory intercomparison tests, such as the AQUACHECK program on water samples, organized by LGC Standards, or those organized by the International Association of Geoanalysts (IAG) on solid samples.

The laboratories have provided, since their creation, infrastructural support to the activity carried out by the IGME in geological, hydrogeological, geotechnical and mining research studies and investigations. The experience acquired and the knowledge and handling of the different available technical equipment gives them a particular degree of distinction in carrying out analysis and testing materials related to Earth Sciences.

Water Analysis Laboratory

Natural waters, both surface and groundwater, have certain characteristics. Its progressive contamination, due, among other things, to anthropogenic factors such as leaks or discharges, makes it necessary to establish an increasingly demanding level of control systems. Water quality control includes the establishment of physical control parameters (organoleptic characteristics, color, smell, suspended elements, temperature, conductivity, radioactivity), chemical (pH, organic matter, nitrogen and derived compounds, COD, phosphates, hydrocarbons, chlorine and chlorides, fluorides, sulfates and sulfides, phenols, cyanides, metals, pesticides, etc.) and dissolved gases.

In the Water Analysis Laboratory, the analysis are carried out in accordance with:

- RD 1798/2010, of December 30, which regulates the exploitation and commercialization of natural mineral waters and bottled spring waters for human consumption, because of the powers that the IGME has by the Mining Law regarding its statement;

- RD 140/2003, which establishes the health criteria for the quality of water for human consumption.

It has the necessary equipment and techniques to determine the parameters required by law. The most used techniques are gravimetric, volumetric, absorption and emission spectroscopy; electrometry techniques, low background and liquid scintillation counters, chromatographic techniques and combustion and infrared detection.

The Water Laboratory has been accredited by ENAC since 1994 according to the UNE-EN ISO/IEC 17025 standard for carrying out physicochemical tests on inland water and water for human consumption in the environmental sector, with accreditation No. 62/LE-169. Table 1 shows the main equipment available in the Water laboratory.

Table 1 Main equipment of the Water Laboratory

LABORATORY EQUIPMENT	COMMERCIAL BRAND	TEST
Atomic Absorption Spectrophotometer (AAS)	AGILENT TECHNOLOGIES	Determination of major and trace elements
Atomic Absorption by Graphite Chamber (GF-AAS)	SpectraAA 220 FS (VARIAN) +Agilent 240 SpectraAA	Metal determination
Organic Carbon Analyzer (TOC)	Shimadzu	Total Organic Carbon, Total Nitrogen
Molecular absorption in continuous flow	ALLIANCE-FUTURA	Anions, cations, cyanides and phenols
liquid scintillation counter	QUANTULUS TM	Tritium
Low background α/β proportional counter	BERTHOLD LB750L	Total alpha and beta activity
Gas Chromatograph GC/MS	VARIAN 4000	Volatile Organic Compounds (VOC)

LABORATORY EQUIPMENT	COMMERCIAL BRAND	TEST
Gas-Mass Chromatograph and triple quadrupole mass detector	Agilent 7000C	Pesticides and polycyclic aromatic hydrocarbons
Ion Chromatograph	881 Compact IC Pro Metrohm	Anions (Bromides, bromates, nitrites, nitrates, sulfates, fluorides, chlorides and phosphates)
UV/Visible Spectrophotometer	HANNA IRIS HI801-02	Color, turbidity, detergents

Mineralurgy Laboratory

The mineralurgy laboratory performs a dual function. On the one hand, it is responsible for preparing the solid samples, by crushing and grinding, for their subsequent chemical analysis or for concentration processes.

On the other hand, in this laboratory mineral processing tests are carried out to support the investigation of new mineral treatment processes, both at laboratory and pilot plant scale, as well as the treatment of effluents for subsequent discharge and/or use. Basically, separation and concentration test, ore leaching, designed to obtain concentrates and/or marketable products from the raw material.

Sorting and concentration techniques are applied, using specific differences in physical or chemical properties between the valuable mineral and tailings. The tests are based on gravimetric methods (particle size classification, dense liquids, shaking tables), physicochemical methods (flotation), and magnetic, electrical and hydrometallurgical methods (leaching, solvent extraction). The main equipment of the Mineralurgy laboratory is showed in table 2.

Table 2 Main equipment of the Mineralurgy laboratory:

LABORATORY EQUIPMENT	COMMERCIAL BRAND	TEST
Jaw and mixed crushers	Humboldt-Wedag	Primary crushing by impact and crushing of the material
Roll mill	Humboldt-Wedag	
Tilting ball mill	--	
Ball mill (porcelain and steel)	Molyba	
Micromills (steel, widia and agate)	Siebtechnik	Grind to analysis size (<0.64 microns)
Elutriator	CYCLOSIZER	Wet sorting of <0.74 micron fraction with fraction collection.
Particle size analyzer	SEDIGRAPH III Plus	Particle size distribution
Helium pycnometer	ACCUPYC 1330	Determination of true and bulk density
Shaking tables	Wilfley	Gravimetric wet separation
UV/Visible Spectrophotometer	PERKIN ELMER	Color, turbidity, detergents
Magnetic separator	FRANTZ ISODYNAMIC	High and low intensity magnetic separation
High voltage electrostatic separator	CARPCO	Separation by electrical conductivity of the material
Flotation cells	DENVER	Concentration of ores by flotation
Pressure reactor	Autoclve Engineers Bolted Closure	High temperature and pressure leaching

Geochemistry Laboratory

In this laboratory, analysis of major, trace and ultra-trace elements are carried out in solid, geological and non-geological materials, using spectroscopic techniques such as XRF, AAS, ICP-AES and ICP-MS. It assists the Water Laboratory in the determination of trace elements in natural waters. The laboratory also determines the chemical parameters of the soil and analyzes for the control of wastewater and mining tailings. It also has a unit for the characterization of mineral species by XRD, another for dating by the U-Pb method using isotopic dilution (ID) and TIMS mass spectrometry. It has a positive pressure clean room as a controlled environment work area for sample preparation with a water purification system and a sub-boiling distiller to obtain highly pure acids.

This laboratory regularly participates in intercomparison exercises of the International Association of Geoanalysts (IAG). Thanks to the good results obtained, it has been selected by the Central Geological Laboratory of Mongolia to participate in the Certification of Certified Reference Materials (CMR). The main equipment of the Geochemistry laboratory is shown in table 3.

Table 3 Main equipment of the Geochemistry laboratory

LABORATORY EQUIPMENT	COMMERCIAL BRAND	TEST
Thermoionization Mass Spectrometer (TIMS)	Thermo Triton	Geochronology (U-Th-Pb Method) and isotope analysis
X-Ray Diffractometer (XRD)	Panalytical Xpert pro MPD	Mineral characterization
Ray Fluorescence Spectrometer (XRF)	Panalytical Zetium	Determination of major and trace elements
Mass Spectrometer with Plasma source (ICP-MS)	Agilent 7500 ce	Determination of trace elements
ICP-AES Spectrometer	Varian Vista-MPX	Determination of major and trace elements
Atomic Absorption Spectrophotometer (AAS)	Varian SpectrAA 220 FS	Determination of major and trace elements
Atomic Absorption by Graphite Chamber (AAS-GF)	SpectraAA 220 FS (VARIAN) +Agilent 240 SpectrAA	Metal determination
Elemental analyzer	Leco CS-800	Determination of C and S

Technological Tests and Natural Stone Laboratory

In this laboratory, geotechnical tests are carried out to characterize soils and rocks and it has a unit specialized in the characterization of non-destructive analysis of drilling cores.

Petrographic, hydric, mechanical, alteration and finished product characterization tests are carried out on natural stone.

In soils, geotechnical tests are carried out for its identification, for evaluation of its resistance and deformability characteristics, and its aggressiveness, with application to the calculation of foundations and retaining structures for buildings.

The Core testing Unit allows to obtain, in a quickly and non-destructively way, high-resolution geophysical and geochemical data in sediment and rock drilling cores, within the geological investigation. The facies classification obtained with these techniques allows better planning for the selection of areas of interest that require destructive analysis.

It has multiparametric geophysical testing techniques (determination of bulk density, porosity, magnetic susceptibility, and p-wave propagation velocity), semi-quantitative and continuous analysis of major and trace elements by XRF, and acquisition of linear mode images through the use of high quality and resolution scanning. The main equipment of the Technological Test and Natural Stone laboratory is shown in table 4.

Table 4 Main equipment of the Technological Test and Natural Stone laboratory

LABORATORY EQUIPMENT	COMMERCIAL BRAND	TEST
MSCL Geotek multiparameter tester	GEOTEK MD-81	Continuous geophysical testification of drill cores (sediments and rocks). Non destructive.
X-ray fluorescence scanner (MSCL-XRF)	GEOTEK XRF Core Scanner	Semi-quantitative and continuous determination of major and trace elements in drilling cores.
GEOTEK Spectrophotometer	CM 700D KONICA-MINOLTA	Measurement of the color and chromatic coordinates of the materials, in the field or in cores (coupled in XRF MSCL Geotek)
Geoscan IV High Resolution Scanner	GEOTEK	Capture of high resolution images and rgb coordinates in rock or sediment cores, whole or sectioned.
Adsorption isotherm analyzer	ASAP 2020 Micromeritics	Determination of the specific surface (BET area), volume and pore size distribution of solid material.
Mercury intrusion porosimetry	Autopore IV 9500	Calculation of the area, distribution of pore sizes, percentage of porosity of the material and real and apparent densities.
Presses	Multiple brands	Flexural and compressive strength for natural stone
Climatic chambers (frost, thermal shock, saline mist)	Dycometal, LCE	Simulation of accelerated aging processes in natural stone

Laboratory of Applied Microscopy/Artificial Vision/Autoclave Tests

The activity of this Laboratory is focused on basic and applied research for the numerical characterization and interpretation of petrographic characteristics (mineralogy, porous system, texture, anisotropy, etc.) of rocks and mineral ores. Rocks and mineral ores obtained in geological-geophysical prospecting for natural resources (oil-natural gas), rock masses, radioactive waste repositories and in advanced studies of materials. Also in the identification of geological formations that allow safe storage of CO₂, the search for unconventional energy deposits and the prospecting of new sources of heavy metals.

It brings together in the same group all the services, from the preparation of thin sections and polished specimens, as well as the equipment for their study and description: scanning electron microscope, digital image analysis system, petrographic microscope, high-resolution scanner for reflected light, and binocular loupe among others. The main equipment of the Applied Microscopy/Artificial Vision/Autoclave Tests laboratory is shown in table 5.

Table 5 Main equipment of the Applied Microscopy/Artificial Vision/Autoclave Tests laboratory

LABORATORY EQUIPMENT	COMMERCIAL BRAND	TEST
Variable pressure Scanning Electron Microscope (SEM)	JEOL JSM -6010 LA PLUS	High-resolution images of the surface of a sample using electron-matter interactions.
Carbon coater	Cressington 108carbon/A	Sample preparation for SEM
Stereomicroscope	Nikon SMZ-745T	Visual characterization
Hyperbaric reactor	HEL. 6 litros: P. max 120 bar. T. Max 100 grados	Characterization of geological stores of CO ₂ Evolution of the porous system of rocks exposed to SC CO ₂

The infrastructure of the laboratories has recently been increased with the incorporation of new equipment purchased with European funds, within the framework of the EU's Recovery, Transformation and Resilience Plan. They are part of the investment made to face the challenges related to the environment, climate change and renewable energy. This technical equipment will contribute to the development of an exclusive infrastructure in the field of Earth Sciences. They are the following:

- For the Water laboratory:
 - A total organic carbon analyzer (TOC), a combustion catalytic oxidation with a non-dispersive infrared detection (NDIR) method. It allows to determine water quality by measuring all carbon forms: TC (Total Carbon); IC (Inorganic Carbon); TOC (Total organic carbon by difference of TC and IC); NPOC (non purgeable organic carbon).
- For the Geochemistry laboratory:
 - A laser ablation system and an ICP-MS inductively coupled plasma mass spectrometry unit for the characterization and spatial distribution of trace elements and mineral deposits and for supporting dating in geochronological studies, in combination with precision dating by ID-TIMS, carried out in the geochronology laboratory of the IGME, CSIC.
 - An inductively coupled plasma atomic emission spectroscopy (ICP-AES) spectrometer for chemical characterization (major and trace elements) in water and solid samples. When elements cannot be determined directly by ICP-MS based on their content, the ICP-AES values are used.
 - An atomic absorption spectrophotometer for the chemical characterization of materials with a high content of total dissolved solids, samples dissolved in organic medium, acid mine drainage, determinations of exchange cations from extractions with ammonium acetate or determinations of polluting elements (mercury, arsenic).
 - An elemental analyzer of carbon, nitrogen and sulfur, which allows obtaining the C/N ratio of great interest in paleoclimatic or paleoceanographic investigations, as well as obtaining information on the sources of sedimentary organic matter and information on ecology, climate and oceanic circulation.
 - A lyophilizer, for continuous rocks and sediments cores samples preparation in a way to facilitate their handling and to increase the performance of subsequent analyses.
- For the core testing unit, two non-destructive techniques in analysis of test cores:

- An X-Ray Computerized Axial Tomograph, to obtain and analyze the internal image of soil, sediment and rock cores, capable of revealing their two-dimensional (2D) and three-dimensional (3D) internal structure through computerized images. Its operation is based on a fast, non-destructive and high-resolution X-ray transmission system, which collects images in a digital system. The advantage of this equipment is that it is designed for loose or fractured cores that cannot be rotated. The source and detector rotate around the core and the three-dimensional structures of the samples are recorded, whatever their state. It has CT reconstruction software that allows multiple sequential volumes to be joined to form a complete image.

- An hyperspectral camera, for the generation of continuous data on the surface of samples, cores or drilling debris, which will allow the identification, quantification and mapping of specific minerals, their chemical composition, alteration zones or the presence of hydrocarbons and contaminants;

For the Applied Microscopy and Artificial Vision laboratory:

- An Automated precision cutting and roughing equipment, to optimize the process of preparing thin sections and polished specimens.

- A vacuum impregnation unit. By embedding geological materials in resin using a vacuum, the process of filling pores and fractures is optimized, facilitating their subsequent study under a microscope.

- An adhesion system for thin sections, which allows continuous pressure to be applied on the sample, optimizing the process of gluing the sample blocks to the slides.

Laboratories activity

In the last five years, 17.903 samples have been received and processed at the laboratories, 7.866 water samples and 10.037 solid samples and drilling cores. In turn, each one of these samples generated multiple subsamples for subsequent preparation, analysis and test in the different laboratories.

These samples managed, correspond to samples sent by other OPIs or external applicants, samples to research projects, agreements or infrastructural actions of the IGME, CSIC.

Technical services catalog

Until their affiliation to the CSIC in March of 2021, the Laboratories had public prices, regulated by the Resolution of May 26, 2009 (BOE nº 144, of June 15, 2009). These prices are actually in the process of being reviewed in order to be included in the CSIC's catalog of technical services. This process is expected to be completed before the end of 2022.