

Fifty Years of Geoanalytical Laboratories in a Service of Geology in Slovakia

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The basis for the mineral exploration and technological treatment and evaluation are good analytical results. The history of geoanalyses in Spišská Nová Ves began on 1st December 1953. The Laboratory was established as a part of Geological Survey n. c. Its main aim was to perform the analyses of Fe, Mn and magnezites. At the beginning only classical analytical methods were used for the determinations. Later new and modern instrumental methods like Optical Emission Spectrometry (OES) and Polarography were introduced. At that time the Controlling and Application Department was established in the laboratory. The main goal was the application of new analytical methods as for example silicate analyses including trace elements analyses.

The capacity and the extent of laboratory works were growing until the first half of sixty years. At that time the laboratory had an important role among all other laboratories in Czechoslovakia. A great development of analytical works, which were focused on the analyses of trace elements, took place. Docimastic method for the determination of Au and Ag and Atomic Absorption Spectrometry – one of the most progressive analytical method of raw materials were introduced. The later mentioned method was applied as the first in the resort of geology in Czechoslovakia. The other methodologies as for example sinter and pressure sample decomposition and ionselective electrodes for the determination of F and Cl were established. Semiquantitative methods OES were extended of quantitative determination of Sn, Mo and W. The analyses of all types of water started in 1965.

The cooperation with the laboratories of Central Geological Institute (ÚÚG), Geological Survey in Prague and in Brno and the Institute of Raw Materials (ÚNS) in Kutná Hora was very important for the developing and validation of analytical methods for various types of geological materials – silicate rocks, limestone, magnezite, dolomite, glass sand, ceramic raw materials, Au-Ag ores, polymetallic ores, Cu-Pb-Zn ores and Hg ores. Together with the development of methodologies, in house reference materials for analytical quality control were also prepared. The laboratories of ÚNS in Kutná Hora, ÚÚG in Prague, Geological Survey in Spišská Nová Ves participated in analytical works.

The development of geological research at the end of 70th until the first half of 80th required the extending of laboratory works according to the amount and development of new analytical methods. A new and modern in-

strumental technique – Atomic Emission Spectrometry with Inductively Coupled Plasma (AES-ICP) – ARL 33 000 was bought in 1976. It was the first analyser of this kind in the resort of geology in Czechoslovakia. This technique replaced classical analytical methods and it widened the range of determined trace elements with a low limit of detection. The modernization of instrumental park continued in the following years. Elementary analyser Carlo – Erba for the determination of elementary C, H and N in coal and organic materials as well as Atomic Absorption Spectrometer AAS-875 Varian were added into the laboratory facilities. Simultaneously AES-ICP-ARL 34 000 for the analysis of silicate and trace elements – Cu, Pb, Zn, Ni, Co and Cd and X-Ray diffractometer DRON were bought in 1981. Next year Adiabatic Calorimeters IKA C-400 for the determination of gross calorific value, Gas Chromatograph – CHROM-4a and Derivatograph – Q 1500 D were purchased too.

In the second half of 80th the laboratory reached its largest qualitative and quantitative improvement. The range of analytical and special geological works covered all needs for geological, hydro-geological, geological engineering and geochemical works. Due to the high level of laboratory works and new analytical instruments the laboratory was included among the best laboratories not only in Czechoslovakia but also in the countries of Council of Mutual Economic Assistance (Comecon). All the following analyses were performed in the laboratory:

- analyses of Fe – ores,
- analyses of coloured metals ores,
- analyses of glass sand for special purposes,
- analyses of white salt,
- analyses of silicate and carbonate rocks,
- analyses of soils,
- analyses of surface and underground water,
- determination of Ag, Al, As, Au, B, Ba, Be, Bi, Ca, C, Cd, Ce, Cl, Co, Cr, Cs, Cu, Eu, F, Fe, Ga, Ge, H, Hg, In, K, La, Li, Mg, Mn, Mo, N, Na, Nb, Ni, P, Pb, Pr, Rb, Sb, Sc, Se, Si, Sm, Sn, Sr, Ta, Te, Ti, V, W, Y, Zn, Zr,
- qualitative spectral analyses
- semiquantitative spectral analyses in SPD scale,
- determination of free SiO₂ and analysis of mining air – CO₂, O₂, CH₄, Ar, H₂, H₂S, He, N₂, Ne,
- geotechnical tests of soils,



- technological tests of brick raw materials, clay, kaoline, bentonite for ceramic purposes,
 - technological tests of stone, sand, gravel for building, founding and glasswork purposes,
 - technical tests of solid fuels including the determinations of C, H and N,
 - technological tests of non-metallic materials,
 - chemical analyses of atypical samples,
- Gradually other modern equipment was introduced into the laboratory:
- Wavelength-dispersive X-ray fluorescence spectrometer PHILIPS PW 1404 for the analysis of geological materials with non-destructive technique from pressed pellets and glass bead,
 - a new model of Atomic absorption spectrometer Varian AA - 40 with electrothermal atomization - GTA 95 and hydride generator - VGA - 76 in 1986,
 - Isotachophoretical analyser for the determination of anions in water in 1987,
 - TMA analyzer for the determination of Hg, X-Ray diffractometer - URD-6 in 1989.

Special geological, mineralogical, petrographical and geochemical works were performed in the laboratory. These works were oriented on genesis of material, setting up of the basis for prognosis of raw materials perspectives and for the study of their structure and technological properties. Laboratory works were focused on the identification of minerals using X-Ray diffractometry and DTA, optical spectrometry and Rtg microanalyses, preparation of mineralogical polished specimen with automatic technique Logitech.

In 1991 the laboratory was again modernized. Sequential AES-ICP spectrometer Liberty 200 with ultrasonic nebuliser and Atomic absorption spectrometer Varian AA-20 for the determination of major and trace elements with low limits of detection and Gas chromatographs GC 3400 with Flame Ionization Detector and Electron Capture Detector for the determination of organic pollutants were added.

In 1992 the range of analysing organic compounds was extended of polycyclic aromatic hydrocarbons and the identification of unknown organic compounds with gas chromatograph Saturn II with Mass Detector. The laboratory was equipped with the most modern technology for the isolation of organic compounds from soils and stream sediments with Supercritical Fluid Extraction and the technique of Purge and Trap with concentrator Tekmar for the isolation of volatile halogen hydrocarbons and volatile aromatic hydrocarbons.

Infrared spectrometer Mattson for the determination of drinking and other types of water pollution as well as for the determination of soil pollution by oil contamination was bought in 1983. AMA automatic analyser for the determination of Hg in water and solid materials with a very low limit of detection was introduced too.

The department of sample preparation was very good equipped with jaw crushers, mills, vibratory ring pulveriser, pulveriser with crinding barrels from agate, automatic rotary divider all made in Retsch and Fritsch company.

The largest extend of laboratory works was achieved in 1991. After then the decrease and permanent reduction of state investment in the area of geological and research works caused the change of analytical works from the analysis of geological materials to the analysis of environmental samples.

In 1994 the laboratory started to prepare for the accreditation according to the requirement of EN 45001. After the fulfilling of accreditation criteria set by the European Technical Norm EN 45001 in 1996 the Geoanalytical Laboratories became accredited by the Slovak National Accreditation Service.

Since 1996 The Geoanalytical Laboratories are a part of Geological Survey of Slovak Republic (GSSR). The GSSR was established by the Decree of Minister of the Environment as a contributory organisation of the Ministry of the Environment. The GSSR's headquarters are in Bratislava, but its activities cover the whole Slovakia.

Geoanalytical Laboratories of GSSR were established by the Decree of Minister of Environment on 25th March 1997 as a Reference Laboratory of the Ministry of Environment of SR (RL) for geology and analysis of geological materials and rock environment. The activities of RL are aiming mainly at:

- reliability of analysis and test results for state administration,
- monitoring and control measuring,
- continual improving and holding high level of quality of environmental measuring,
- harmonise conditions for input of Slovakia into market of EU.

Since 1996 the Geoanalytical Laboratories as a RL were an active partner in the project of bilateral cooperation between the Ministry of Environment of SR and Danish Environmental Agency entitled *Implementation of Laboratory Structure to Provide Adequate Analyses for the Environmental Management of the SR*.

The GAL co-operated internationally with laboratories of geological surveys of Europe in testing and developing analytical methods in geochemical and environmental monitoring, in sample collection techniques and in putting up systems to support and to control quality of analytical data. GAL provided homogenisation services for *Global Geochemical Baselines Project and for the European Geochemical Atlas*.

The most significant event in the year 2002 was the re-accreditation of the laboratory according to the European Technical Norm EN ISO/IEC 17025. Geoanalytical Laboratories successfully finished the re-accreditation process and received certificate on accreditation for analyses of geological materials, minerals, ores and non-metallic raw materials, solid fuels and products of combustion, soils, sediments, plant materials, food, waste, determination of physico-chemical, hydrobiological, microbiological and ecotoxicological indicators in waters and taking off samples of coal, soils and sediments.

In 2003 the laboratory was again modernized. Sequential AES-ICP spectrometer VISTA -MPX with CCD was bought, which allows to determine trace elements with a very low limits of detection.

Geoanalytical Laboratories as a Reference Laboratory of the Ministry of Environment organise the inter-laboratory proficiency tests for Slovak laboratories. This programme is a part of Quality Control Programme of the Ministry of Environment for Slovak environmental laboratories. Proficiency test is an external control of analytical system, performance of laboratories, and enable the participants to detect unsuspected source of errors and subsequently made remedial actions.

The Laboratories regularly take part in international interlaboratory tests and certification programmes for geological and environmental samples. These programmes are useful source of reference samples which can be put to good use internally. In the last four years Geo-

analytical Laboratories prepared through interlaboratory exchange programmes two certified reference materials of chemical composition Zeolite1 and Bentonite1. The both reference materials were certified by the Slovak Metrological Institute.

The Geoanalytical Laboratories of Geological Survey of Slovak Republic have 50 – year experience in the performing of the rock and metallic ore analyses. Nowadays they provide comprehensive laboratory analytical service for geological, geochemical, hydrogeological exploration and research, environmental monitoring according to the requirements of the Ministry of Environment and other state and private organisations.