Analytical Laboratory - Development of Analytical Services for Scientific Teams of IOCB: From Classical Chemical Methods to Sophisticated Instrumental Techniques

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About us ...

The Analytical Laboratory, formerly named Central Analytical Laboratory, is a part of the IOCB from its foundation in early fifties of the 20-th century. The Laboratory of Organic Elemental Analysis was a part of the Mass Spectrometry group since 1996. In the year 2007 the laboratory was established as an independent service department.

In the fifties the laboratory was a key source of information about either newly synthesized compounds or the isolated ones from natural materials. Howewer, the laboratory lost partially its original prominent position as a result of development and nowadays practically routine use of new physico-chemical methods of structural analysis, that means, for example, MS, NMR and FTIR spectrometries. Nevertheless, for complete characterization of synthetized and/or isolated substances the detailed and reliable elemental analysis is still an indispensable tool (optimally in combination with advanced methods of structural analysis). However, new methodologies and instrumental techniques are introduced to supplement and/or to replace the classical chemical methods of analysis, e.g. titrimetry. The implementation of instrumental methods is aimed at improvement of detection limit, sensititvity and other performance characteristics of analytical methods. Furthermore, the amount of sample required for analysis should be reduced and non-destructive techniques if possible should be applied. The range of elements determined has been expanded. The first carbon, hydrogen and nitrogen **automatic analyzer Perkin Elmer 240 A** was purchased in 1969, a new model **2450 C** in 1983 and the current PE instrument in 1999. Next step of laboratory innovation was started in the year 2006 focused on the extension of number of elements determined (by instrumental techniques). A **CyberScan Ion 510 Meter with fluoride ion-selective** electrode was purchased in 2006.

On the IOCB meeting in Třešť 2008 we have presented our vision of the future:

Perspectives & Developments - vision 2008

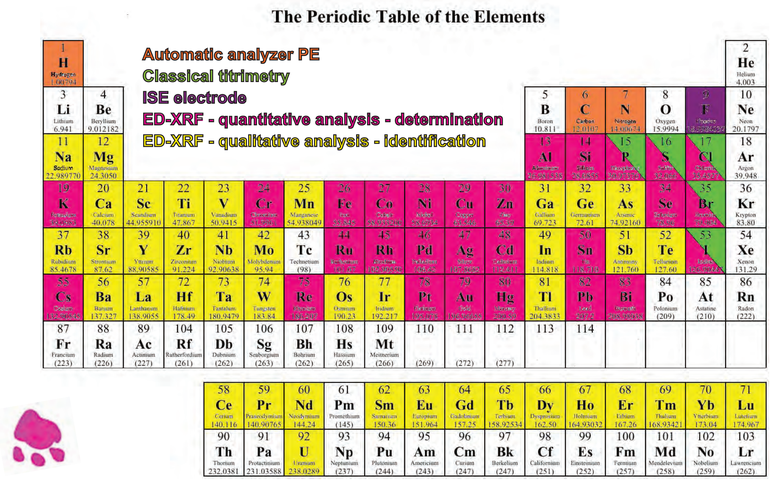
*The aim of the further development of the analytical laboratory is to **decrease demands on required amounts of samples** for analyses and to increase both the **precision and accuracy of analytical results** using more reliable and robust instrumental methods. To fulfill the aim we would like to upgrade laboratory and instrumental equipment of the analytical laboratory.*

*Firstly, we plan to buy an **X-ray fluorescence spectrometer** primary dedicated for determination of "light" elements (from sodium). The instrument allows simultaneous identification and quantification of a lot of elements. X-ray fluorescence is an accepted technique for analyzing the elemental composition of samples in the concentration range between 10^{-4} %…100%. The calculation of the sample composition is based on the intensity of the main elemental lines. The tested SPECTRO iQ spectrometer is equipped with an air cooled 50 W end window X-ray tube. The primary tube spectrum is monochromatized and polarized by a doubly bent HOPG crystal. Solids, pressed pellets, liquids or loose powder can be used in different methods. The excellent analytical performance of the SPECTRO iQ was demonstrated in various application reports. The energy-dispersive X-ray fluorescence spectrometer could replace the classical titrimetric determinations of S, P, Cl, Br and I, which require quite high amounts of samples (8 – 15 mg per one determination of a single element). Furthermore, we expect that various elements (e.g. metals in organic samples - not determined yet) can be determined in future. The method is sufficiently sensitive and non-destructive. We suppose, that the required amount of a sample for determination of a set of elements decrease bellow 5 mg. Considering the minimal requirements on sample preparation for X-ray fluorescence analysis and the non-destructivity of the technique, the major part of the sample will be given back to the client.*

Various sets of samples prepared in our laboratory were tested preliminary in collaboration with Ing. Kolečkář (Spectro CS) on X-ray fluorescence spectrometers in Kleve, Germany and in glassworks Kavalier in Sázava. Both the sample preparation procedure and sample storage conditions have been developed. Satisfactory results have been obtained even at low concentration range. Hence, a successful implementation of the X-ray fluorescence spectrometry is expected for simultaneous determination of a set of elements in organic samples.

...and the real progress 2008 - 2012

In August the 2008, a simultaneous ED-XRF spectrometer SPECTRO iQ was purchased and on the last IOCB meeting in Frymburk (2010) we have presented a succesfull employment of this instrumentation in sometimes almost detective work on detection of composition of various samples and materials. Compared to the situation presented in 2010 (details see the posters on our webbsite) the scale of quantitatively determined elements is nowadays significantly enlarged and new aplications and calibrations models have been developed, e.g. determination of elements in pressed pellets. After the detector replacement with a new one with Ta-colimator the determination of Pd is also possible, which was wery helpful for some groups of our institute. Actually quantitative-derminated and qualitative-identified elements are shown in following periodic table:

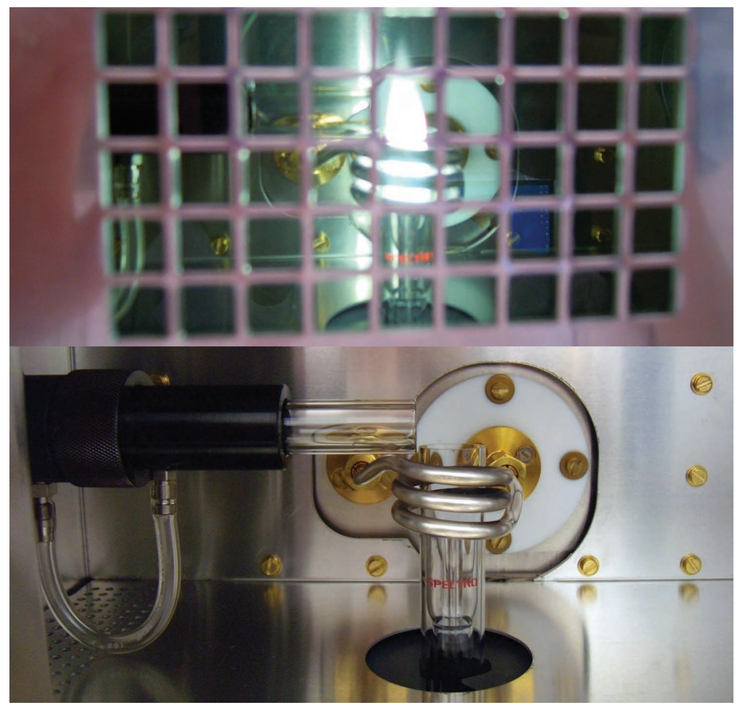


But still several demands of our clients have shown that there are several issues which cannot be solved with our existing equipment, for example the determination of lightest elements such as Li, B,... analysis in cases of strong interferences of chararacteristic lines of determined elements in complex samples and trace analysis e.g. in biochemical and/or biological samples. We have performed many tests and preliminary measurements and finally, we decide to purchase ICP-OES spectrometer.

HOT TOPIC - ICP-OES

ICP-OES spectrometer SPECTRO ARCOS

Sample introduction system



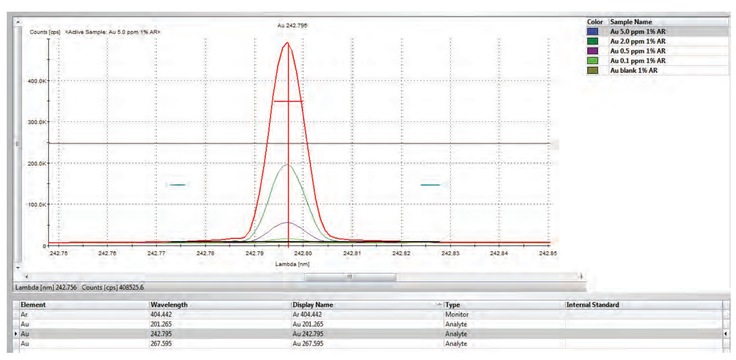
Plasma torch and SOP - optical interface

How does the ICP-OES work?

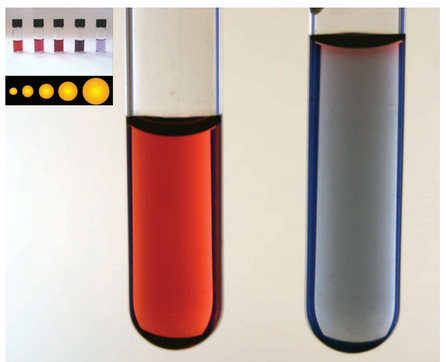
Inductively coupled plasma/optical emission spectrometry (ICP-OES) is a powerful tool for the determination of elements in a variety of different sample matrices. With this technique, usually liquid samples are injected into a radiofrequency-induced argon plasma using one of a variety of nebulizers or sample introduction techniques. The sample mist reaching the plasma is quickly dried, vaporized, and energized through collisional excitation at high temperature (8 000 - 10 000 K). The atomic emission emanating from the plasma is viewed in either a radial or axial configuration, collected with a lens or mirror, and imaged onto the entrance slit of a spectrograph. Simultaneous multielement determinations are performed for up to 70 elements with the combination of a polychromator and an array detector. The LODs´ occur usually in ppb - ppt range. The analytical performance of such systems is competitive with most other inorganic analysis techniques, especially with regards to sample throughput and sensitivity.

In February 2012 the SPECTRO ARCOS optical emission spectrometer (SPECTRO Analytical Instruments, Kleve, Germany) with radial plasma observation was installed. The SPECTRO ARCOS features a Paschen-Runge spectrometer mount, employing the Optimized Rowland Circle Alignment (ORCA) technique. Consisting of two hollow section cast shells, optimized small volume and 32 linear CCD detectors, the wavelength range between 130 and 770 nm can be simultaneously analyzed, allowing complete spectrum capture within 2s. Due to the unique reprocessing capabilities of the system, a new measurement is not required even if additional elements or lines are to be determined at a later date. The optic is hermetically sealed and filled with argon, continuously circulated through a filter, which absorbs oxygen, water vapor and other species. High optical transmission in the VUV is achieved, allowing the determination of non-metals as well as the use of prominent and interference free lines. An air-cooled ICP-generator, based on a free-running 27.12 MHz system, ensures excellent stability of the forward power even in the case of rapidly changing sample loads. All relevant ICP operating parameters are software controlled, allowing easy selection of the optimum operating conditions. The instrument is equipped with several sample introduction systems to perform optimal analyses of a wide variety of samples with their different matrices. The instrument is very robust and stable and allows the element determination also in heavy matrices for example high salted solutions or organic matrices. Currently we have first succesfull experience with determination of several elements in strange matrices using ICP-OES, for example Au in nanoparticlulate systems, Gd in bumblebees cells extracts, P and Au and/or S and Hg simultaneously in complex organic samples (inaccessible by XRF due to overlap of characteristic lines).

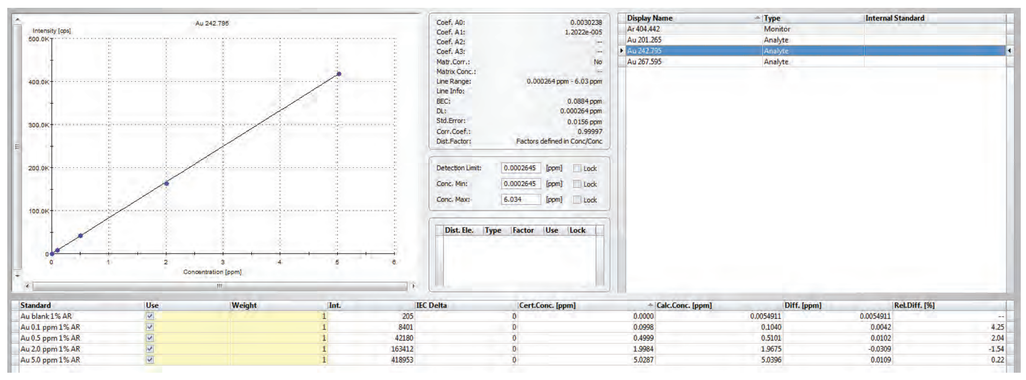
For further development we would like (1) to purchase additional sample introduction systems and an autosampler to minimize the amount of sample required for individual analysis, to reduce consumption of argon and time of analysis, (2) to upgrade our equipment for sample treatment and digestion techniques using heaters with heating controll, and (3) to introduce an equipment for microwave-assisted sample digestion or autoclave digestion.



Example of single line - Au 242.795 nm



Gold nanoparticles



Example of calibration - determination of Au

Other our Services

Furthermore, the standard analytical services provided to the scientific staff of the IOCB include optical rotation measurement of organic compounds using the **polarimeter Autopol IV (Rudolph Research Analytical, USA)** and some other analytical determinations using volumetric analysis or ion selective electrode. Precisely weighting of low amounts of samples for some experiments of our scientific colleagues is also performed. The Laboratory is equipped with **analytical balance AX** and **microbalance MX5 (Mettler Toledo);** they are used for precise weighing of samples. After estabilishing of the project for archivation of all relevant samples synthetized on the Institute, which is partially realised by the staff of our laboratory, a new **microbalance ME5 (Sartorius, Germany)** has been purchased recently.

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