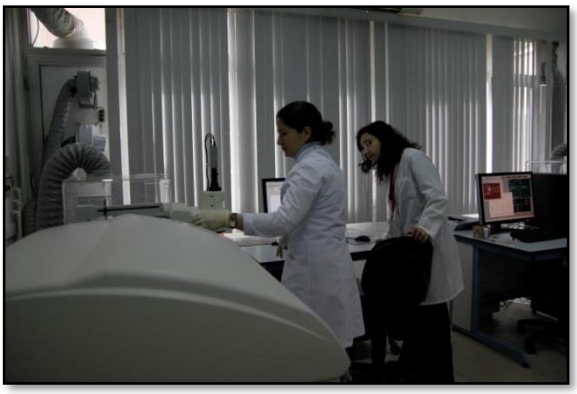
CENTRAL LABORATORY

Advanced Material Characterization R&D Center

METU, Ankara

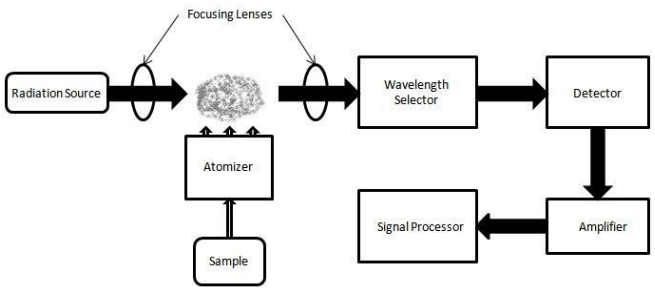
Chemical Analysis Laboratory (KAL)



Determination of the chemical structure of a material is important for both industry and research activities. For this purpose, Atomic Absorption Spectrometer (AAS), Inductively Coupled Plasma Mass Spectrometer (ICPMS), Inductively Coupled Plasma Optical Emission Spectrometer (ICPOES), X-Ray Fluorescence Spectrometer (XRF), UV VIS spectrophotometer (UV-Vis) and a Microwave Sample Preparation Unit are present at the Chemical Analysis Laboratory.

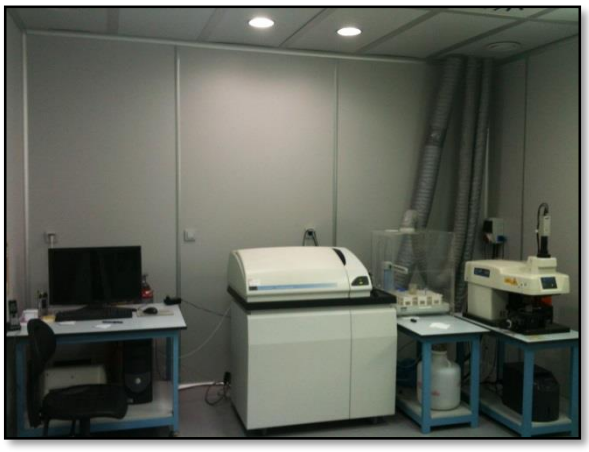
BASIC PRINCIPLES

Atomic Absorption Spectrometer (AAS): Atomic absorption spectrometry (AAS) is a single element technique that measures the concentrations of elements. Air/acetylene or nitrous-oxide/acetylene flame is used to generate ground state atoms. The light emitted from a hollow cathode lamp, specific to the measured element, is passed through the flame and measured by a segmented solid state detector. The sample of interest is aspirated into the flame, if that metal is present in the sample, it will absorb some of the light, thus reducing its intensity. The change in intensity is directly related to the concentration of that element in the sample. The working principle of the instrument is summarized below:



Inductively Coupled Plasma Optical Emission Spectrometer (ICP-OES): Inductively Coupled Plasma (ICP) is an analytical technique used for the detection of trace metals. Sample is introduced to an argon plasma whose temperature is 6000-10000 K. In the plasma, molecular bonds are broken, atoms and ions are formed. These atomic and ionic species are excited and are rapidly relaxed to emit at their characteristic wavelengths. Emission signals are analysed using an Echelle polychromator using a S-CCD (Segmented Charge Couple Device) array detector system. Detection limits are in the range of µg/L.

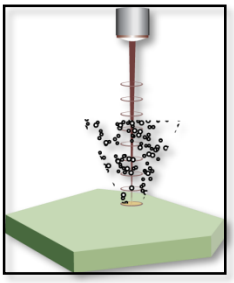
Inductively Coupled Plasma-Mass Spectrometer (ICPMS): Inductively coupled plasma-mass spectrometry is an analytical technique which requires the sample to be introduced to a high temperature plasma, commonly argon, which dissociates molecules and ionizes atoms. The ions are passed into vacuum via a sample and skimmer cone interface, where a lens stack focuses the ion beam into a quadrupole mass spectrometer. Here, the ions are sorted by mass and detected using a scanning electron multiplier. Sample introduction is normally performed via nebulization of solution. As an alternative sampling technique, the use of laser ablation allows direct analysis of solids. ICP-MS can be considered a multi-element technique since scanning over different masses is very fast. detection limits are in the range of ng/L.



ICP-MS system consists of an argon plasma to produce atomic ions and a quadrupole mass spectrometer for analysis.

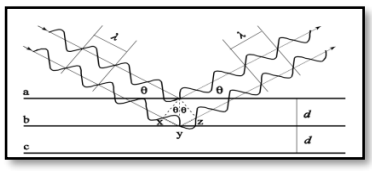
Mass range is 5-270 amu and detection limits are in the range of ng/L. Dynamic Reaction Cell can be used if needed

to eliminate isobaric interferences caused by the presence of gaseous molecules.
As an alternative sampling technique, the use of laser ablation allows direct analysis of solids. ICP-MS can be considered a multi-element technique since scanning over different masses is very fast. In addition, a large variety of solid samples can be handled either following a dissolution step or using LA-ICP-MS. Perhaps one of the most advantageous applications of LA-ICP-MS is semi-quantitative analysis which allows a compositional fingerprint of unknown materials to be obtained.



UV-VIS Spectrophotometer (UV-Vis): Ultraviolet and visible (UV-Vis) absorption spectroscopy is the measurement of the attenuation of a beam of light after it passes through a sample or after reflection from a sample surface. The absorbance of a solution increases as attenuation of the beam increases. The concentration of an analyte in solution can be determined by measuring the absorbance at a specific wavelength. UV-Vis spectroscopy is usually applied to molecules or inorganic complexes in solution. Many molecules absorb ultraviolet or visible light and different molecules absorb radiation of different wavelengths. With this instrument the absorbance, transmittance and reflectance measurements can be performed both in solid and liquid samples.

X-Ray Fluorescence Spectrometry (XRF): If an atom excited with a higher energetic radiation, the higher energy input results an electron to transfer to a higher energetic shell. While the excited electrons transfers back to their original state, they emit X-rays which has an energy of 0,1-50 Å. This secondary X-ray beam is called fluorescence. The radiation wavelengths of each element are different and unique. In another words, these radiations are the fingerprints of each element. By the identification of the wavelength of the radiation, the element type (qualitative) and by the measuring the intensity of each radiation, the concentration (quantitative) of the material can be determined.



Sample Preparation Units

Aqueous samples such as drinking water, wastewater and other samples relevant to fields of environmental studies, petrochemistry, food, hydrogeology and others are analyzed in KAL. Solid samples are dried if necessary, weighed and by digested with microwave digestion system using nitric acid, hydrochloric acid and hydrogen peroxide.

Sample Preparation

Microwave Digestion System

There are several certified reference materials available for XRF instrument suitable for rock, sediment and ore samples analysis. Sample preparation varies according to the structure of the sample and the quality and quantity of elements to be analyzed. Solid samples can be either analyzed directly or be brought into appropriate particle size and pressed using binders or glass discs are prepared using fusion.

Fusion Unit

Grinding Mill

Press

CONTACT INFORMATION:
Laboratuvar Sorumlusu: Serap Kaya (Tel: 210 6455)
Laboratuvar Deney Sorumluları: Leyla Molu, L.Sezen Yıldırım
Laboratory e-mail: mlabkal@metu.edu.tr
Laboratory phone: 312 2107421-6430