

*A Laboratory at the service of the Academia and the Industry*





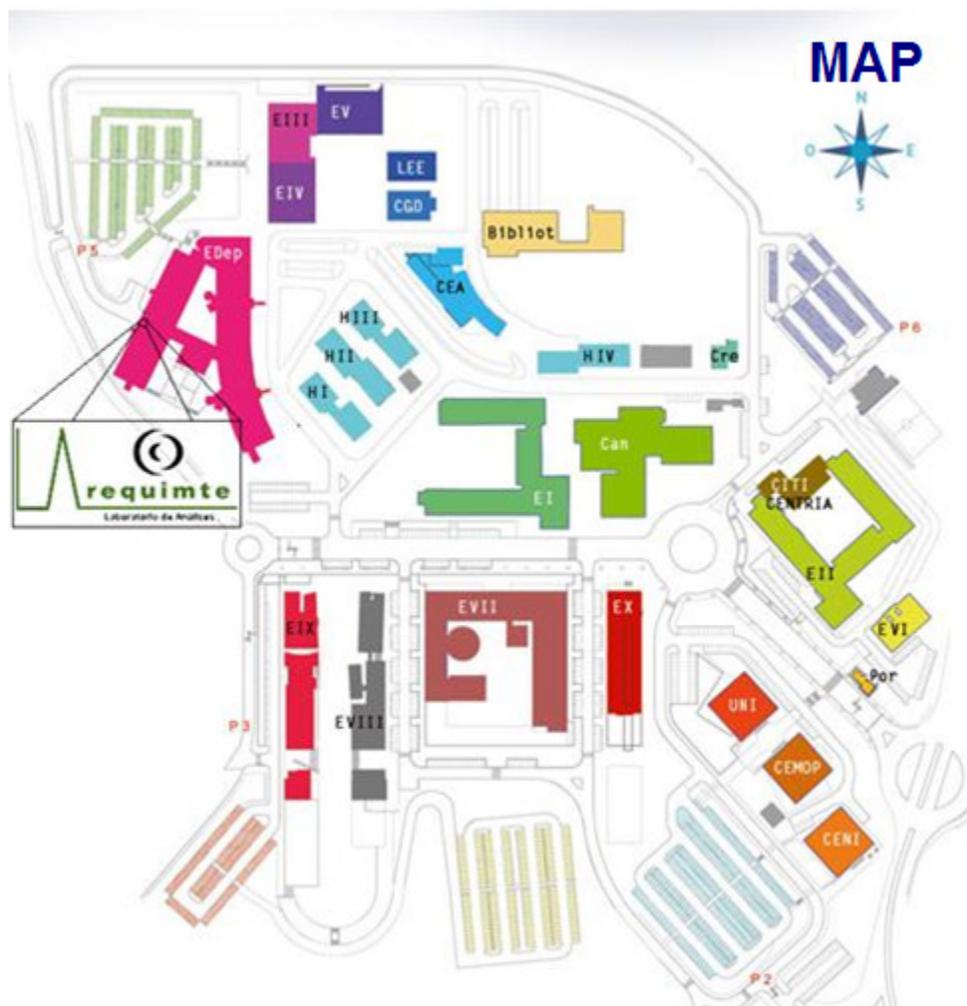
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## THE LABORATORY

The Laboratório de Análises is an analytical services facility located in the Chemistry Department of Faculdade de Ciências e Tecnologia – Universidade Nova de Lisboa, created in 2002 in order to support the research activities of the Associated Laboratory REQUIMTE, and public or private external entities.



## CONTACTS

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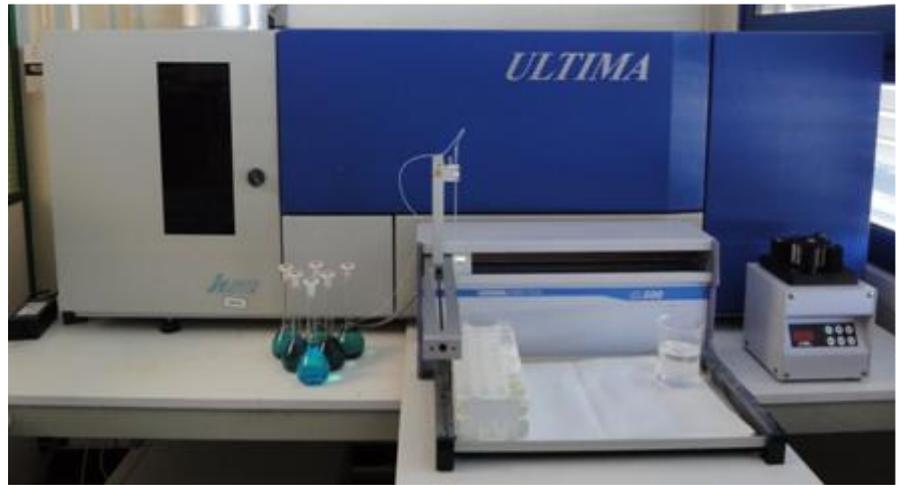


# ELEMENTAL ANALYSIS

There are several techniques to study the elemental composition of samples of a wide range of matrices. In order to achieve the best result is important finding the adequate technique to the sample features.

The elemental analysis is usually required in different research fields, academic or industrial, such as chemistry and processes, pharmaceutical, food, agriculture, environment and geology.

# ICP-AES



ICP-AES – Inductively Coupled Plasma-Atomic Emission Spectrometry technique is suitable for the analysis of elements in different matrices solid, liquid, organic and inorganic (biological materials / proteins, animal and plant cells, milk, fish, water, metal alloys, synthetic products / pharmaceuticals or other), present in any matrix that is soluble in aqueous media (acidic, basic or salt).

The ICP allows analysis of virtually all chemical elements (which exhibit emission bands of adequate sensitivity) in the range of 180-800 nm, with detection limits of concentration to the ppb for most elements and the simultaneous analysis of up to 73 elements in a single sample.

## **Equipment**

ICP (*Inductively Coupled Plasma-Atomic Emission Spectrometer*) **Horiba Jobin-Yvon**, France, **Ultima**, model equipped with a 40,68 MHz RF generator, Czerny-Turner monochromator with 1.00 m (sequential), autosampler AS500 and CMA (Concomitant Metals Analyzer).

# GFAAS



Graphite Furnace Atomic Absorption Spectroscopy is a highly sensitive spectroscopic technique that provides excellent detection limits for measuring concentrations of metals in aqueous and solid samples.

The graphite furnace is an electrothermal atomizer system that can produce temperatures as high as 3000 °C. The heated graphite furnace provides thermal energy to break chemical bonds within the sample and produce free ground-state atoms. Ground-state atoms then are capable of absorbing radiation energy for the transition to an excited state. The amount of radiation energy absorbed increases as the concentration of the selected element increases.

The advantages of GFAAS include:

- Greater sensitivity and detection limits than other methods
- Low spectral interference
- Very small sample size

## **Equipment**

GFAAS (*Graphite Furnace Atomic Absorption Spectrometer*) **Analytik Jena AG**, Germany, **AAS ZEE nit 650** model equipped with Zeeman as well as deuterium background correction, uses a transverse-heated graphite tube atomizer and autosampler MPE 60.

# FAAS



Flame Atomic Absorption Spectroscopy is a fast and easy technique with an extremely high sensitivity (particularly for elements like Pb, Cd, Cu and Cr). The sample is atomized in the flame, through which radiation of a chosen wavelength (using a hollow cathode lamp) is sent. The amount of absorbed radiation is a quantitative measure for the concentration of the analyzed element.

The gas mixture is ignited in a flame at a temperature ranging from 2100 to 2300 °C. During combustion, atoms in the sample are reduced to the atomic state. A light beam from a lamp whose cathode is made of the element being determined is passed through the flame into a monochromator and detector. Free, unexcited ground state atoms of the element absorb radiation at characteristic wavelengths; this reduction of the radiation energy at the analytical wavelength is a measure of the amount of the element in the sample.

## **Equipment**

FAAS (*Flame Atomic Absorption Spectrometer*) **Analytik Jena AG**, Germany, **AAS 5 FL** model equipped with a deuterium lamp, air-acetylene gas mixture and autosampler AS51.

# ELEMENTAL ANALYZER



The analyzer operates on the basis of combustion dynamics of the sample. The sample is weighed into tin capsules and introduced into the combustion reactor using an automatic sampler, together with a certain amount of oxygen.

After combustion, the gases  $N_2$ ,  $CO_2$ ,  $H_2O$  e  $SO_2$  are transported by the helium stream through the copper surface inside the reactor, separated in a GC column and finally detected by thermal conductivity (TCD). The total analysis time is of 12 minutes.

In the end of the analysis it generates a complete report with results of sample composition in CHNS total percentage from 0,01 % (100 ppm) to 100 %.

## **Equipment**

Elemental Analyzer **Thermo Finnigan-CE Instruments**, Italy, model **Flash EA 1112 CHNS series**.

# MASS SPECTROMETRY

Mass Spectrometry is a powerful analytical technique applied to the identification of unknown chemical compounds, with high sensitivity and speed.

The technique consists on the compounds ionization in the gaseous state, and makes the charged particles separation in the analyzer according to their ratio of mass/charge ( $m/z$ ). In practice, what is obtained is a mass spectrum that is a graphical representation of the intensity of each ion on the basis of their ratio  $m/z$  ratio.

Each mass spectrum allows the collection of information on the molecular mass of a compound, and the ionic fragments obtained provide detailed information about the structure of that compound.

# GC-TOF-MS



The GCT mass spectrometer with an analyzer time of flight (TOF) of orthogonal acceleration and high resolution (FWHM ~ 7000) is equipped with four sources of ionization.

When coupled with a gas chromatograph is used to analyze mixtures of volatile and semi-volatile compounds, using three different modes of ionization: electron impact (EI), chemical ionization (CI) and field ionization (FI).

Relatively pure samples may also be introduced directly into the ionization source with a probe. The latter mode allows the direct analysis of samples using the three modes of ionization available.

## **Equipment**

GC-TOF-MS (Gas Chromatography - Time of Flight - Mass Spectrometer) **Micromass**, model **GCT**.



# MALDI-TOF-MS

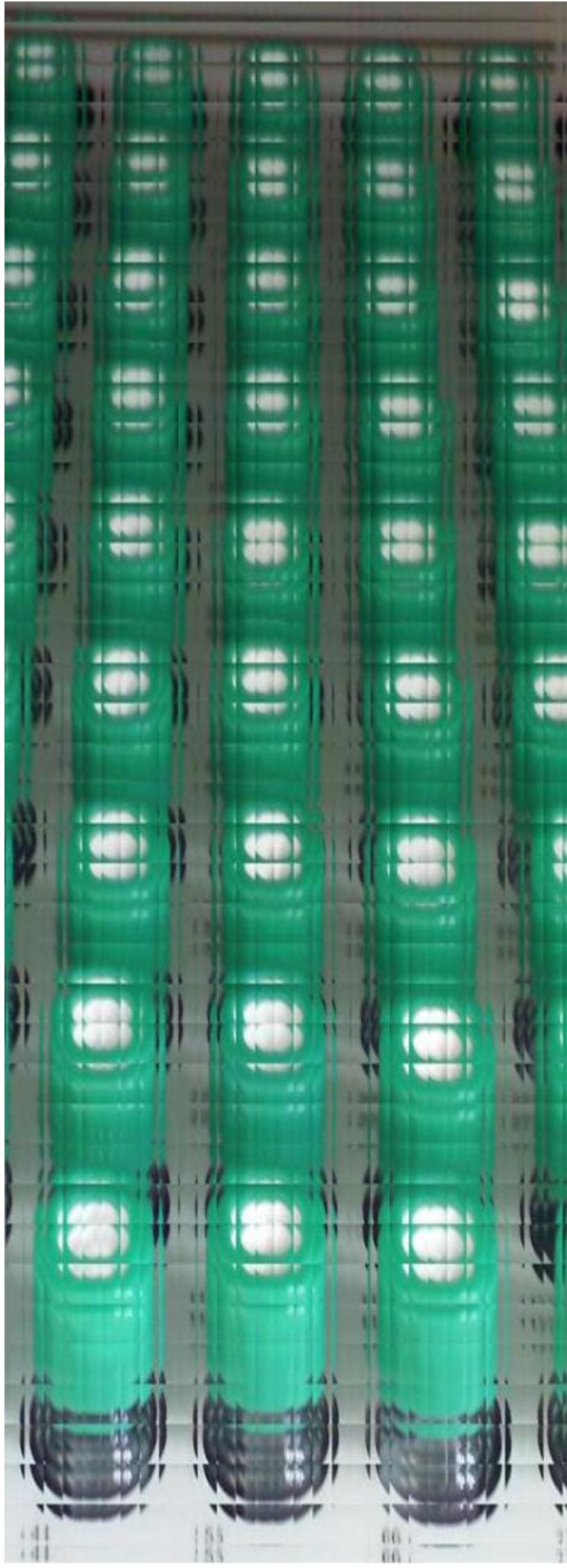
The Voyager-DE™ PRO Biospectrometry Workstation is a mass spectrometer with time of flight analyzer (TOF), and matrix assisted laser desorption ionization (MALDI). The device is equipped with nitrogen laser (337nm) and the TOF analyzer allows work in linear and reflector modes.



The theoretical "unlimited" mass range of TOF and the "soft ionization" characteristic of MALDI allow this technique to be one of the best choices for the analysis of macromolecules with high sensitivity and resolution, including peptides, proteins of high molecular weight, oligonucleotides, polysaccharides and synthetic polymers. In reflective mode, molecules of mass under 10 kDa can be analyzed (peptides, organic molecules, complexes, polymers and proteins of low molecular weight), with a resolution of  $\sim 7,000$  (or  $> 7,000$  for  $m/z < 6$  kDa). The linear mode allows the analysis of high molecular weight molecules, with a resolution greater than 2,000 for small proteins ( $m/z < 20$  kDa) and  $\sim 50$  for large proteins ( $m/z \sim 100$  kDa).

## Equipment

MALDI-TOF-MS (Matrix Assisted Laser Desorption Ionization – Time Of Flight – Mass Spectrometry) **Applied Biosystems**, model **Voyager DE-PRO Biospectrometry Workstation**.



# CHROMATOGRAPHY

Chromatography holds a prominent place among the analytical techniques.

The different variants existing are based on the compounds partition between two distinct phases, a mobile and a stationary phase immiscible with each other. The separation results from the interaction of the compounds among the phases. They are eluted at different speeds, as a result of successive stages of adsorption/desorption of the sample through the stationary phase.

Depending on the method used for separation and detection is possible to analyze many different groups of analytes in a broad variety of matrices.

# ION CHROMATOGRAPHY



The ion chromatography proves to be a versatile analytical technique for most of the ionic species, both organic and inorganic.

Separation can occur by ionic exchange processes between the mobile phase and the active groups linked to the stationary phase, ion exclusion and even when the selectivity is determined by the mobile phase (HPIC - High Performance Ion Chromatography / HPICE Ion Chromatography-High Performance Exclusion / MPIC - Mobile Phase Ion Chromatography).

The working range is extended as a result of the development of separation of species, but also detection. The conductivity detector (CD) allows a rapid analysis of ions enhanced by the introduction of a suppressor which reduces the conductivity of the electrolyte in the eluent prior to detection. The amperometric detector (ED) allows a sensitive determination of carbohydrates, amino acids and others. It also has a photodiode detector and refractive index.

It is used for analysis of inorganic ions in various matrices as waste water or fermentation broths (anions such as chloride, bromide, fluoride, nitrite, nitrate and sulfate; cations such as lithium, calcium, ammonium, magnesium, potassium and sodium). This equipment enables the determination of weak organic and inorganic acids, and organic sulfonic acids, compounds of biological origin as carbohydrates, alcohols, amino acids and other compounds. The technique has advantages over others, in particular for anionic species, such as speed, sensitivity, selectivity, simultaneous detection and stability of the separation columns.

## **Equipment**

Ion chromatography system – **DIONEX**, model **ICS-3000** equipped with the following detectors: *PDA* - Photodiode Array Detector - 190 to 800 nm; *ED* - Electrochemical Detector - Pulsed / Integrated Amperometry; *CD* - Conductivity Detector; *101 Shodex RI* - refractive index detector.

## GC – FID

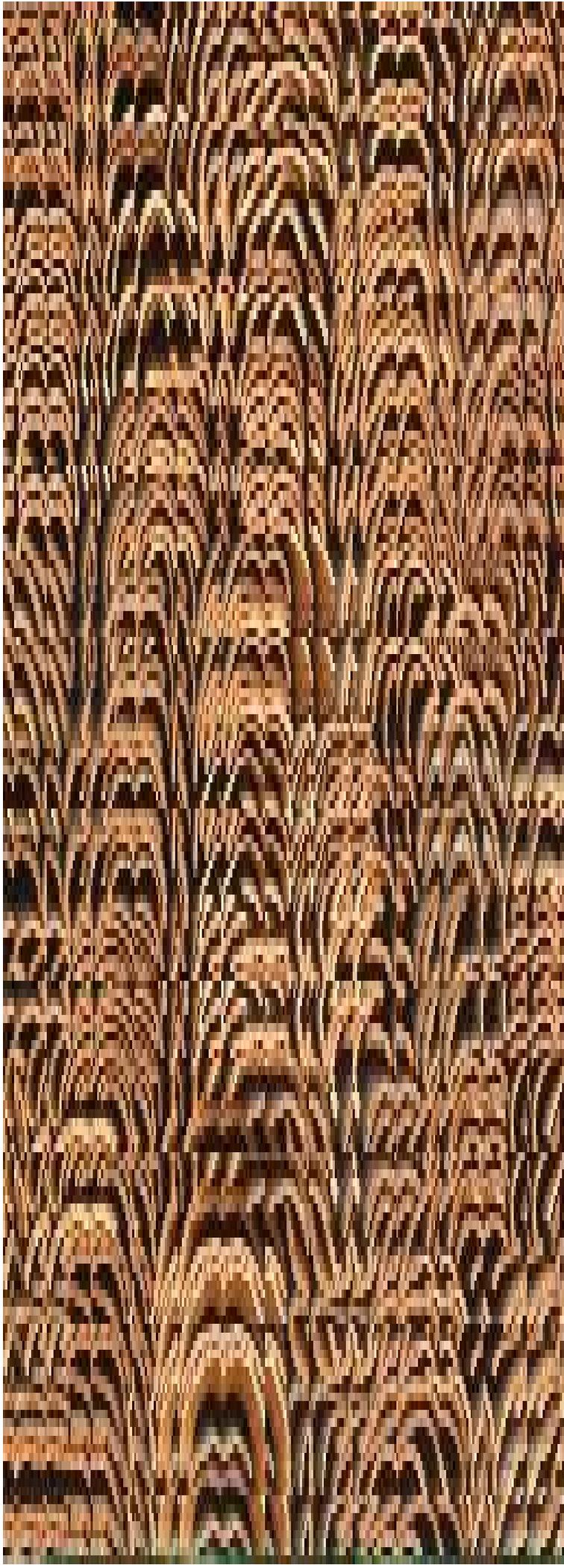


GC-FID – Gas Chromatography with Flame Ionization Detection - the samples (approximately 1 $\mu$ L) are introduced into the injector through a syringe. The sample is drawn by the mobile phase (carrier gas) along the column. By applying a temperature ramp program, the different components of the mixture are separated. These compounds elute from the column dissolved in the carrier gas, pass through FID detector where they will undergo pyrolysis at the flame temperature, producing ions and electrons which conduct electricity through the flame.

The GC-FID can be used for the analysis of volatile organic compounds or semi-volatile compounds such as hydrocarbons, pesticides, PAHs, fatty acids, higher alcohols, essential oils and solvents.

### *Equipment*

GC-FID (Gas Chromatography - Flame Ionization Detector) **Konik**, model **Konik HRGC 4000B**.



# MATERIALS CHARACTERIZATION

The materials characterization is a key procedure in materials science – a transversal area to several research and application fields.

It comprises a series of experimental techniques to the determination of parameters and structural characteristics essential to the comprehension of the application potentialities in new products development.

Among the common parameters there are porosity, thermal behavior, crystallinity and purity.

# MERCURY POROSIMETRY

The mercury porosimetry allows analyzing a set of properties of solid structures by mercury intrusion at controlled pressure. The penetrometer containing the sample passes through a stage of degassing and then it is filled with mercury. Pressure is applied to force the mercury into the sample and is shown a curve intrusion / extrusion. The information obtained leads to the calculation of parameters that characterize the sample. This technique is based on the Washburn equation:

$$D = \frac{-4 \cdot \gamma \cdot \cos \theta}{P}$$

where D is the pore radius which mercury enters,  $\gamma$  is the surface tension of mercury,  $\theta$  the contact angle of mercury with the surface of the solid and P is the applied pressure.

It is possible to obtain data on pore size distribution, mean diameter and total pore volume and density of the sample. Among the common applications are ceramic and building materials, catalysts, pharmaceuticals, filtration and adsorbent materials.

## **Equipment**

Mercury porosimeter – **Micromeritics, Autopore, model IV 9500.**

- Operation maximum pressure – 33.000 Psia (228 MPa)
- Pore size distribution: 360 – 0,005  $\mu\text{m}$
- Low pressure – up to 50 Psia (345 kPa): 360 – 3,6  $\mu\text{m}$
- High pressure – up to 33,000 Psia (228 MPa): 6 – 0,005  $\mu\text{m}$



# DSC



The differential scanning calorimetry (DSC) is a thermal analysis technique that records the flow of heat energy associated with transitions in materials as a function of temperature. This is a method of enthalpy variation, in which the difference in the supply of heat energy between a substance and a reference material is measured as a function of temperature, while both are subjected to the same program of heating or cooling, carefully controlled.

These measurements provide qualitative and quantitative processes in endothermic (absorbing heat) and exothermic (release heat) conditions, allowing to obtain information about changes in physical properties and / or chemical properties such as, Temperatures characteristics (melting, crystallization, glass transition); Degree of crystallinity of polymers; Phase diagram; Enthalpies of phase transition and reaction; Thermal and oxidative stability; Purity; Kinetics of reactions.

This technique can be used to characterize a wide range of materials other than polymers, including other common classes of organic and inorganic materials, crystalline or amorphous ceramics, glass, metals and alloys, fibers and plastics, composites, etc.. Application areas are extended from the scientific to the industrial field, with particular importance for industries of pharmaceuticals and cosmetics, food, and polymers, in research and development stage, in raw materials, and in products quality control.

## **Equipment**

Differential scanning calorimeter - **Setaram**, France, model **DSC 131**.

This equipment works in the temperature range between  $-150\text{ }^{\circ}\text{C}$  and  $+550\text{ }^{\circ}\text{C}$ , the heating and cooling speeds between  $0,001$  and  $50\text{ }^{\circ}\text{C} / \text{min}$ . The sign of heat flux ranges from  $-100$  to  $+100\text{ mW}$  and the resolution is  $\pm 0,2\text{ microW}$ .

# XRD

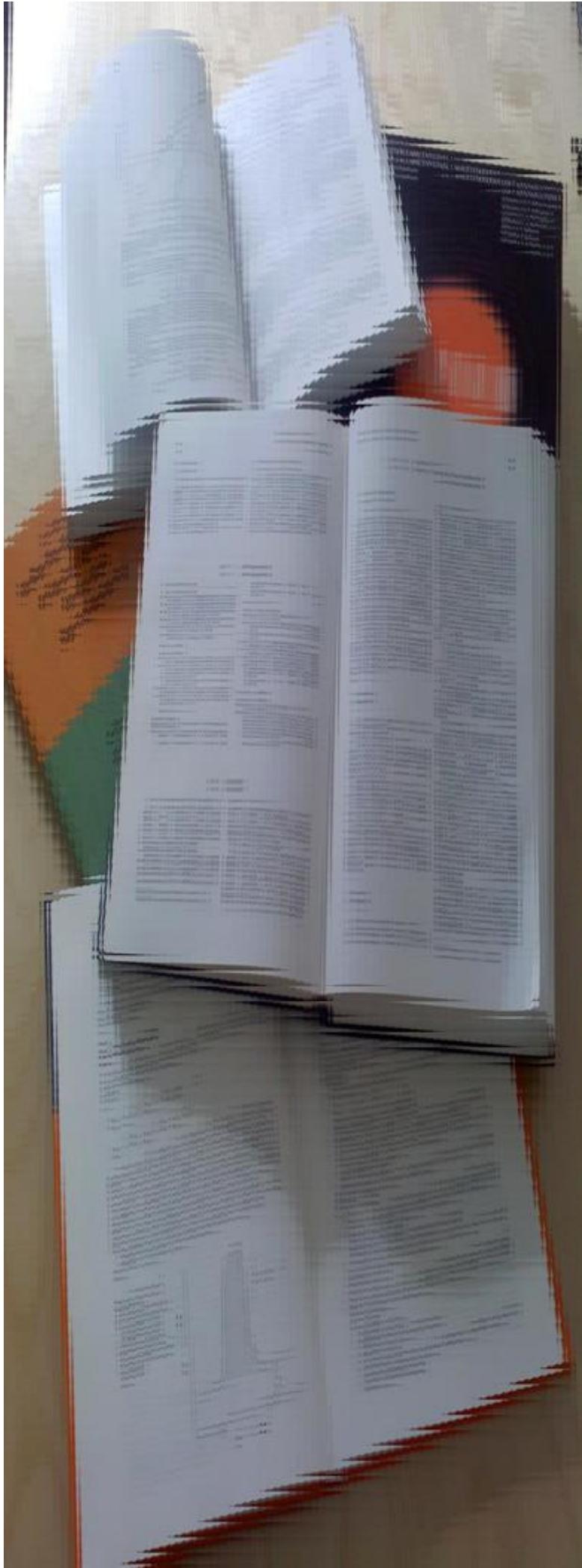


The powder X-ray diffraction (XRD) is one of the techniques used by mineralogists and solid state chemists to examine the physical-chemical make-up of solids. In this technique a diffraction pattern is obtained by registering the intensity of X-rays as a function of a scanning parameter (angle  $2\theta$ ).

The powder diffraction of X-ray (XRD) is applied to various materials such as cement, cosmetics, food, forensic chemistry, geology and mineralogy, paints, pharmaceuticals, petrochemicals, among others.

## ***Equipment***

Desktop X-Ray Diffractometer **RIGAKU**, model **MiniFlex II** (30 kV/15 mA) with X-Ray tube CuK $\alpha$ .



# TRAINING

As part of academic training, the laboratory provides theoretical and practical support on the techniques currently available. It also supports practical training in collaboration with Specanalítica Equipamentos Científicos, Lda



For further information, contact the Laboratório de Análises.

