
# LABORATORY OF INSTRUMENTAL PHARMACEUTICAL ANALYSIS

## DEPARTMENT OF PHARMACY

## UNIVERSITY OF PATRAS

## INSTRUMENTATION LIST

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# SITE: PHARMACEUTICAL ANALYSIS LABORATORY (PHARMACY DPT, UNIVERSITY OF PATRAS)

**Instrument Characteristics**


**Model: Bruker D2 Phaser 2nd Gen**

**X-Ray Tube:** Ceramic. KFLCu-2K. Radiation. Cu-Kα.

**Focus:** line 12mm.

**Generator:** 30kV 10mA – 300W maximum power.
Bragg-Brentano geometry.
2θ:2-150$^0$.
Parallel beam optics.
Knife edge collimators.
Soller slits.
Diffracted beam monochromator.
Standard sample holder.
Appropriate software for instrument control and analysis.
**Detector:** LYNXEYE.

## X-Ray Powder Diffractometer D2 Phaser 2nd Gen

X-Ray Powder Diffraction is a well-known technique for the investigation of the structure of crystalline solids and powders. X-rays wavelength is between the ultraviolet and gamma rays in the electromagnetic spectrum, and its magnitude is of the same order of interatomic distances in crystals. Depending on the x-ray wavelength, crystal orientation and structure, a beam of X-rays falling on a crystal undergoes diffraction. The x-ray beam interacts with the electron density of atoms or ions of the crystal lattice, thus producing a specific electron map for the crystalline material investigated. Each crystal has a unique pattern and X-Ray diffraction gives a fingerprint of the studied material. The data collected from the reflections at various angles are analyzed according to Bragg's law. X-ray Powder Diffraction is widely used both for the qualitative analysis of crystalline materials and for the quantitative measurements of crystalline components in composite materials.

| Services | Sample properties |
|---|---|
| • Phase identification of bulk samples.<br>• Crystallinity percentage.<br>• Identification and Quantitative determination of polymorphism. | Powders, pellets, solids, suspensions, creams.<br>Sample size: 2cm-2.5cm diameter.<br>Weight: 50-500 mg. |

## Instrument Characteristics


**Model: Bruker D2 Phaser 2nd Gen XE-T Edition**

**X-Ray Tube:** Ceramic. KFLCu-2K. Radiation. Cu-Kα.

**Focus:** line 12mm.

**Generator:** 30kV 10mA – 300W maximum power.
Bragg-Brentano geometry.
2θ:2-150$^0$.
Parallel beam optics.
Knife edge collimators.
Soller slits.
Diffracted beam monochromator.
Automatic sample changer with 6 positions.
Appropriate software for instrument control and analysis.
**Detector:** LYNXEYE XE-T.
Energy resolution <380 eV.

# X-Ray Powder Diffractometer D2 Phaser 2nd Gen XE-T Edition

X-Ray Powder Diffraction is a well-known technique for the investigation of the structure of crystalline solids and powders. X-rays wavelength is between the ultraviolet and gamma rays in the electromagnetic spectrum, and its magnitude is of the same order of interatomic distances in crystals. Depending on the x-ray wavelength, crystal orientation and structure, a beam of X-rays falling on a crystal undergoes diffraction. The x-ray beam interacts with the electron density of atoms or ions of the crystal lattice, thus producing a specific electron map for the crystalline material investigated. Each crystal has a unique pattern and X-Ray diffraction gives a fingerprint of the studied material. The data collected from the reflections at various angles are analyzed according to Bragg's law. X-ray Powder Diffraction is widely used both for the qualitative analysis of crystalline materials and for the quantitative measurements of crystalline components in composite materials.

| Services | Sample properties |
|---|---|
| • Phase identification of bulk samples.<br>• Crystallinity percentage.<br>• Identification and Quantitative determination of polymorphism. | Powders, pellets, solids, suspensions, creams.<br>Sample size: 2cm-2.5cm diameter.<br>Weight: 50-1000 mg. |

## Instrument Characteristics


**Model: LabRAM HR Evolution, HORIBA**

**Laser:**
785nm and 532nm
**Raman Filter:**
785nm_Edge, 532nm_Edge, 532nm_ULF
**Grating:**
600(500nm), 300 (600nm) and 1800 (450-850nm)
**Accessory:**
Automated movable sample holder stage with streamline HR capabilities,
Power meter 18LAB250, ULF, Confocal
**Detector:** Syncerity OE, CCD

# Raman Spectrometry

When a photon of a monochromatic laser beam strike/interacts with a molecule then the photon is mainly scattered elastically and we have the so-called elastic Rayleigh scattering. During this interaction there is a low probability of the photon energy to cause vibrational excitation or even rarely vibrational relaxation of the molecule and the photon is thus scattered with respectively lower or higher energy. This process is called Inelastic Raman Scattering effect and these vibrational transitions are characteristic for each molecule. The Raman Scattering is quite weak phenomenon and Raman spectroscopy was considered as an awkward technique especially when it should have to be applied to simple everyday problems that required speed, straightforward results and clarity, although it is an almost no sample preparation technique. However, new Raman instrumentation based on new concepts of monochromator designs, laser technology, CCD cameras, fiber optics application as well as backscattering collection geometry through microscope objectives rendered Raman spectroscopy as one of the most powerful techniques for material characterization. The application of Raman spectroscopy was further expanded by the construction of Raman micro-probes that have been successfully applied to a wide range of analyses to identify contaminants, inhomogeneities and properties of materials and structures encountered in industrial production even in hazardous and not-friendly environments.
In micro-Raman mapping spectroscopy (B) imaging and mapping of samples can also be performed

| Services | Sample properties |
|---|---|
| • Determination of polymorphic forms in extremely low percentages<br>• Identification of substances (Database)<br>• Structure identification of variety of materials in all states of matter<br>• Molecular orientation in polymers<br>• Degree of polymer crystallinity<br>• Quantitative measurements, Thermodynamic functions evaluation<br>• Molten salts, oxide melts, corrosive melts<br>• Ultra-Low-frequency studies<br>• Films<br>• Identification of nanotubes<br>• Biological samples<br>• Particle Size Analysis | Raman spectroscopy is a non-destructive technique, which needs only a small portion of a sample to identify its chemical structure. A variety of samples such as powders, solids, liquids, gases, glasses, fibers can be measured. |

## Instrument Characteristics

**Model: Mastersizer 3000E**

**Accessories:**

Hydro SV:
Max Volume: 10ml
Hydro EV:
Max Volume: 600ml
AERO M:
For dry PSD methods

**Optics:**

Red light source:
Max. 4mW He-Ne, 632.8nm
Lens arrangement:
Reverse Fourier (convergent beam)
Effective focal length:
300mm

**Detector:**

Arrangement:
Log-spaced array
Angular range:
0.015 - 144 degrees
Alignment:
Automatic

**Size:**

Size range:
0.1 - 2100µm
Number of size classes:
100 (user adjustable)
Accuracy:
Better than 1%
Repeatability:
Better than 0.5% variation
Reproducibility:
Better than 1% variation

# Particle Size Distribution (PSD)

The PSD of a material contributes in understanding its physical and chemical properties. It affects the reactivity of solids participating in chemical reactions, and needs to be tightly controlled in many industrial products such as chemical and pharmaceutical products.
For PSD analysis with laser diffraction, a laser beam passes through a dispersed particulate sample and the angular variation in intensity of the scattered light is measured. Large particles scatter light at small angles relative to the laser beam, and small particles scatter light at large angles. The angular scattering intensity data is then analyzed to calculate the size of the particles that created the scattering pattern using the Mie theory of light scattering. The particle size is reported as a volume equivalent sphere diameter.

| Services | Sample properties |
|---|---|
| • Determination of Particle size range from 0.1 – 2100µm<br>• Wet and Aero sample dispersion | Suspensions, emulsions and dry powders |

Hydro SV

Hydro EV

Aero M

**Instrument Characteristics**

**Model: Hydro Sight (Malvern)**

**Measurement Size range:**
9 - 1000μm
**Observable Size range:**
1.4 - 1400μm
**Analysis method:**
Lens-less imaging
**Power consumption:**
< 6W
**Operational temperature range:**
+10°C to +35°C or larger span
**Humidity range:**
80% maximum for temperature up to 31°C, decreasing linearly to 50% at 40°C or larger

## Imaging accessory for laser diffraction particle sizer (HYDRO SIGHT)

The revolutionary Hydro Sight lens-less imaging* accessory provides rapid visualization and assessment of your liquid particle dispersions during a laser diffraction measurement. Real-time observation of the particles in the measurement flow allows improved understanding of how stirring, sonication and addition of surfactants and stabilizers affect the dispersion of a sample. One-click capture of images, videos and dispersion metrics provides quick and effective supporting evidence for method troubleshooting and validation in line with ISO13320 and USP<429> guidance. Hydro Sight is designed to be used with a laser diffraction system which is connected to a recirculating liquid dispersion accessory. It captures images of a dispersed sample flowing through a specially designed cell using lensless dynamic imaging technology. No focusing or calibration by the user is required.

| Services | Sample properties |
|---|---|
| • Particle visualization and analysis<br>• PSD Method validation | Suspensions and emulsions |

## Instrument characteristics

**Model: Light Microscope Leica DM 2500M**

**Mode:**
Transmitted and Reflected light microscopy

**Lenses:**
HCX PL Fluotar objectives, Leica Germany:

i. 100x / 0.90
ii. 5x / 0.15
iii. 10x / 0.30
iv. 20x / 0.50
v. 40x / 0.75 (PH 2)

**Accessory:**
Hot stage plate (Thermoplate Leica TPX-Type D)

**Software:**
LAS v4.11

# Optical Microscope

Optical microscopy is a technique employed to closely view a sample through the magnification of a lens with visible light. This is the traditional form of microscopy, which was first invented before the 18$^{th}$ century and is still in use today.

An optical microscope can generate a micrograph using standard light-sensitive cameras. Technological developments have now enabled digital images to be taken with CMOS and charge-couple device (CCD) cameras for optical microscopes. As a result, the image can be projected onto a computer screen in real time to examine a sample with these digital microscopes. This increases the convenience of use as eyepieces are no longer needed. The power of magnification of a compound optical microscope depends on the ocular and the objective lenses. It is equal to the product of the powers of these lenses (e.g. for a 10x ocular lens and 100x objective lens used together, the final magnification is 1000x.) Light Microscope Leica DM 2500M is a digital microscope that uses a computer to visualize the image without the need for an eyepiece to view the sample. It is connected to Leica DFC420 C, a digital microscope camera with c-mount and with a 5 Mpixel CCD sensor. The camera quickly captures sharp images for darkfield, brightfield and phase contrast microscopy in life science, clinical and industry applications. The DM 2500M can also provide polarized light.

Light Microscope Leica DM 2500M is also connected to a hot stage plate which gives the opportunity for observing the samples thermal behavior from ambient to 60$^{o}$C temperature.

The advance software (LAS v4.11) is updated with image analysis that has with the capability of measuring number and size of particles and can extend in the z-axis producing a better focused image.

| Services | Sample properties |
|---|---|
| • Crystal morphology and particle size<br>• Characterize thermal behavior of crèmes, solids and liquids<br>• Possible determination of melting point of excipients or drugs | Solids, liquids, semi-solids, crèmes and ointments. |

**Instrument characteristics**

**Model: Spectrum 100, Perkin Elmer**

**Range:**
7800-370 $cm^{-1}$ with 0.5 $cm^{-1}$ resolution
**Detector:**
DTGS

**Accessories:**
**A) Crystal ATR:**
ZnSe 9-bounce
**Spectral Band:**
Mid-IR (20000 – 650 $cm^{-1}$)
**Penetration depth:**
1.66μm

**B) Crystal ATR:**
KRS-5 (Thallium-bromoiodide)
**Beam:**
1-bounce
**Spectral Band:**
Mid-IR (20000 – 400 $cm^{-1}$)
**Penetration depth:**
1.73μm

# FT-IR/ATR spectroscopy

The IR spectrum derives from absorption of light exciting molecular vibrations. The positions of absorption bands in the spectrum contain information that can be used to determine the identity of the sample.
In Attenuated Total Reflection (ATR) a beam of infrared light passes through the ATR crystal and is totally reflected at least once. The reflected light that has come in contact with the sample (depending on the crystal and the angle of the beam) contains information about its chemical composition. The examined samples with ATR are the same chemical and biological samples as in the transmission mode, but aqueous solutions can also be examined due to shorter laser path length and smaller attenuation of the IR signal in the highly absorbing water.
The available system (Spectrum 100) can work as both classic FT-IR with KBr pellets and in ATR mode.

| Services | Sample properties |
|---|---|
| • Qualitative and quantitative characterization of materials in forensics, pharmaceutical, biomedical and other departments. | Films, pellets, chunks, fibers, powders, coating, semi-solids, liquids, etc.<br>Reflectance (ATR) is capable of providing depth-profiling analysis in the range of 0.1 to 3 microns. |

**Instrument characteristics**

**Model: Spotlight 200i and Spectrum Two**

**Detector:**
wide-band MCT detector
**Microscope Numerical Aperture (N.A.):**
0.6
**Spectrometer:**
Michelson interferometer
**Spectral Band:**
Mid-IR (8300 – 600 $cm^{-1}$)
**Micro-Attenuated Total Reflection (ATR):**
Integrated Micro-ATR with 100 μ Germanium ATR crystal.
**Sample Area viewing:**
75x50mm
**Stage accuracy movement:**
0.1μm

## Micro-Infrared spectroscopy and micro-ATR spectroscopy

The IR spectrum derives from absorption of light exciting molecular vibrations. The positions of absorption bands in the spectrum contain information that can be used to determine the identity of the sample.
In Attenuated Total Reflection (ATR) a beam of infrared light passes through the ATR crystal and is totally reflected at least once. The reflected light that has come in contact with the sample (depending on the crystal and the angle of the beam) contains information about its chemical composition. The examined samples with ATR are the same chemical and biological samples as in the transmission mode, but aqueous solutions can also be examined due to shorter laser path length and smaller attenuation of the IR signal in the highly absorbing water.
In micro-IR and micro-ATR spectroscopy apart from chemical characterization, imaging and mapping of a sample can also be performed.
The NICODOM IR Polymers and Additives Library has been installed is the system.

| Services | Sample properties |
|---|---|
| • Qualitative and quantitative characterization of materials in forensics, pharmaceutical, biomedical and other departments.<br>• Single point microscopy<br>• Line scans<br>• Automated mapping<br>• Small area imaging<br>• Measurements in Transmission, Reflectance or micro-ATR | Films, pellets, chunks, fibers, powders, coating, semi-solids, liquids, etc. |

## Instrument characteristics

**Model: Chromatographic System Ultimate 3000, Dionex Corporation Sunnyvale, CA, USA**

**Parts**

**ThermoScientific Ultimate 3000 Pump (Pump LPG-3400 A):**
4 vacuum channels (analytical)

**Thermostatted Column Compartment (TCC-3100):**
Wide temperature range from 5 °C to 85 °C

**Switching valves:**
1x two-position six-port

**Photodiode Array Detector (PDA-3000):**
Optical detector capable of measuring the absorbance spectrum from 190-800nm (Deuterium Lamp 190-380nm, Tungsten Lamp 380-800nm).
Up to 5 single wavelengths (2D chromatograms) can be collected

# High-performance liquid chromatography (HPLC)

High performance liquid chromatography (HPLC) is basically a highly improved form of column liquid chromatography. Instead of a solvent being allowed to drip through a column under gravity, it is forced through under high pressures, which makes this technique much faster.

All chromatographic separations, including HPLC operate under the same basic principle; separation of a sample into its constituent parts because of the difference in the relative affinities of different molecules for the mobile phase and the stationary phase used in the separation.

| Services | Sample properties |
|---|---|
| • Separate, identify and quantify each component in a mixture | Semi- and nonvolatile materials in various sample matrices |

Pump LPG-3400 A

PDA-3000

TCC-3100

**Instrument Characteristics**

**Model: AAnalyst 200 (Perkin Elmer)**

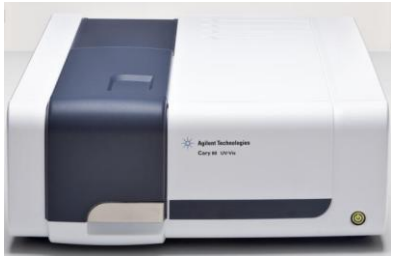
**Accessories:**
Graphite Furnace HGA 900
**Photometer:**
Double beam, time-shared and space-shared optical system. Deuterium background corrector
**Monochromator:**
Wavelenght range:
185-860nm
Diffraction Grating:
1800 lines/nm blazed at 236nm & 597nm
Grating area:
64x72mm
Reciprocal Linear Dispersion:
1.6nm/mm
Focal length:
274nm
Spectral bandwidth:
0.2, 0.7 &2.0nm
**Detector:**
Wide range photomultiplier with UV-transimitting window
**Light source:**
Hollow cathode and electrodeless discharge lamps.
Lamp elements:
Mg, Ca, Mn, Fe, Zn, Cd

## Atomic absorption spectroscopy with Graphite furnace

Atomic Absorption spectroscopy (AAS) is used for identification and quantitative analysis of more than 60 metallic elements. Each element is characterized by its ground and excited energy states. When an element is excited absorbs energy the wavelength corresponding to the excitation energy depends on the ground and excited states of the element. The intensity of the radiation provides quantitative information for the element. The excitation can be achieved either with flame or with the vaporization of the element in a graphite furnace heated electrically. With the graphite furnace the detection limits are in the ppb region.

| Services | Sample properties |
|---|---|
| • Quantitative analysis of metals in ground and spring water, wastewaters and other industrial effluents<br>• Pigment industry<br>• Pharmaceutical industry<br>• Cement industry | Any liquid or solid sample can be analyzed after digestion |

## Instrument Characteristics



**Model: Cary 60 UV-Vis (Agilent Technologies)**

**Spectral range:**
190-1100nm
**Wavelength accuracy:**
±0.06nm
**Spectral bandwidth:**
1.5nm
**Wavelength reproducibility:**
±0.01nm
**Photometric range:**
4.0Abs
**Detector:**
2 silicon diode detectors for simultaneous sample beam and reference
beam measurements
**Light Source:**
Agilent Xenon Flash lamp
**Monochromator:**
Czerny-Turner
**Scan speed**
24000nm/min
**Data collection rate**
80Hz
**Photometric Accuracy:**
absorbance ±0.0004
**Temperature control:**
Peltier & water thermostatted

# UV-Vis- Spectrophotometer

UV-Vis spectroscopy is a widely used technique which probing the electronic transitions of the molecules as they absorb UV and/or visible light. Absorption measurements can be at a single wavelength or over an extended spectral range. UV-Vis spectroscopy is applied mainly in a wide range of molecules or inorganic complexes in solution. The UV-Vis spectra have broad features that are of limited use for sample identification but are very useful for quantitative measurements by using the Beer-Lambert law. It can detect concentrations of molecular species less than $10^{-7}$ M.

| Services | Sample properties |
|---|---|
| • Absorption<br>• Quantitative Analysis of species in solutions<br>• Monitor kinetics of chemical or biological reactions | Liquid<br>Minimum sample volume: 0.5μL |

## Instrument characteristics

**Model: i-Raman PLUS (BW &Tek, USA)**

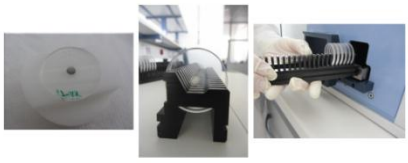
**Laser:**
785nm
**Detector:**
CCD
**Accessories:**
**A)** Microscope with 20x, 50x & 80x objective lens
**B)** Micro-probe attached to 2m optical fiber

# Raman Spectrometry

When a photon of a monochromatic laser beam strike/interacts with a molecule then the photon is mainly scattered elastically and we have the so-called elastic Rayleigh scattering. During this interaction there is a low probability of the photon energy to cause vibrational excitation or even rarely vibrational relaxation of the molecule and the photon is thus scattered with respectively lower or higher energy. This process is called Inelastic Raman Scattering effect and these vibrational transitions are characteristic for each molecule. The Raman Scattering is quite weak phenomenon and Raman spectroscopy was considered as an awkward technique especially when it should have to be applied to simple everyday problems that required speed, straightforward results and clarity, although it is an almost no sample preparation technique. However, new Raman instrumentation based on new concepts of monochromator designs, laser technology, CCD cameras, fiber optics application as well as backscattering collection geometry through microscope objectives rendered Raman spectroscopy as one of the most powerful techniques for material characterization. The application of Raman spectroscopy was further expanded by the construction of Raman micro-probes that have been successfully applied to a wide range of analyses to identify contaminants, inhomogeneities and properties of materials and structures encountered in industrial production even in hazardous and not-friendly environments.

| Services | Sample properties |
|---|---|
| • Determination of polymorphic forms<br>• Identification of substances<br>• Structure identification of variety of materials in all states of matter<br>• Molecular orientation in polymers<br>• Degree of polymer crystallinity<br>• Quantitative measurements, Thermodynamic functions evaluation<br>• Molten salts, oxide melts, corrosive melts<br>• Low-frequency studies<br>• Films<br>• Identification of nanotubes<br>• Biological samples | Raman spectroscopy is a non-destructive technique, which needs only a small portion of a sample to identify its chemical structure. A variety of samples such as powders, solids, liquids, gases, glasses, fibers can be measured. |

## Instrument characteristics

**Model: S2 PICOFOX – with Mo excitation**

**X-ray tube:**
30 W or 40 W metal-ceramic, max. 50 kV, 1 mA air-cooled, Mo target

**X-ray optics:**
Multilayer monochromator

**Detector:**
Peltier-cooled XFlash® Silicon Drift Detector.
30 $mm^2$ active area.
Energy resolution <150 eV at 100 kcps (Mn Ka)

**Element range:**
Al to U (with exception of Nb to Ru)

**Sample changer:**
Automatic version with cassette for up to 25 samples

# Total Reflection X-ray Fluorescence (TXRF)

Total Reflection X-ray Fluorescence (TXRF) analysis combines conventional XRF analysis with monochromatic radiation and total reflection optics. This results in significantly enhanced fluorescence yield, largely reduced background noise and high sensitivity to elements that occur only in traces.

An air-cooled X-ray tube with molybdenum target generates an X-ray beam, which is reduced to a narrow energy range by a multi-layer monochromator. The fine beam impacts on a polished sample carrier at a very small angle (< 0.1°) and is totally reflected. The characteristic fluorescence of the sample is emitted and measured in an energy dispersive X-ray detector. Due to the short distance to the carrier, the fluorescence yield is very high and the absorption by air is very low.

TXRF is suitable for liquids, powders and solid samples. In liquid samples, elements can be detected even in traces down to 0.1ppb, while in powders it ranges from 1-100ppb. TXRF can also be used for element detection in biological tissues, after digestion.

| Services | Sample properties |
|---|---|
| • Trace elements in liquid, powder, suspension and solid samples<br>• Toxicological analysis of blood and urine<br>• Elemental composition of nanoparticles<br>• Fast wipe tests for contamination control<br>• Trace elements in biological tissues, plants and grains, after digestion | Liquid, powder, suspension or solids<br><br>Minimum sample amount: low µg or µl |

## Instrument characteristics


**Model: SkyScan 1174**

**X-ray source:**
20-50kV, 50W, metal ceramic X-ray tube

**X-ray detector:**
1.3MP (130x1024pixels) 14-bit, cooled CCD camera optically coupled to scintillator be motorized zoom lens

**Nominal resolution (pixel size at maximum magnification):**
6μm at maximum magnification

**Reconstructed space (after a single scan):**
Up to 1304x1301x930pixels

**Scanning space:**
Max 30mm diameters, 50mm height

**Radiation safety:**
< 1 μSv/h at any point on the instrument surface

# Micro Computed Tomography (micro-CT)

Micro computed tomography is X-ray imaging in 3D by the same method used in CT or CAT scans, but on smaller scale with greater resolution. It is a 3D microscopy application where very fine internal structure of objects is imaged in a non destructive procedure. In micro-CT scanning a micro-focus X-ray source illuminates the object and a planar X-ray detector collects magnified projection images. Hundreds of angular images are acquired as a pair of second source detectors, or the object, rotates. The computer then collects these images and produces a virtual volume that contains all the internal details of the object. Sample preparation is not needed.

| Services | Sample properties |
|---|---|
| • 3D imaging and microstructure examination non destructively<br>• Images with isotropic detail detectability 6 - 30μm (dependent on magnification) | Solid<br>Maximum height: 50mm<br>Maximum Diameter: 30mm<br>No sample preparation is required |

## Instrument characteristics

**Model: Multiwave ECO, Anton Paar**

**Microwave source:**
850Watt
**Temperature sensor:**
IR
**Rotor:**
16-position rotor
**Sample holders:**
50ml pressure vessel HVT 50
**Maximum pressure:**
20bar
**Maximum Temperature:**
250°C
**Properties:**

A) Built-in computer
B) Integrated cooling system
C) Prevention of thermal overshoots

# Microwave High Pressure Liquid Digester

Microwave digestion optimizes the economic routine analysis of environmental samples, food quality control, plant material and other biological material.
A single magnetron delivers up to 850 W microwave power in an unpulsed mode over the full power range. The software prevents thermal overshoots and the design of the microwave applicator provides utmost field density, allowing efficient heating.

| Services | Sample properties |
|---|---|
| • Short period for digestion of solid samples | Liquid, powder, suspension or solids<br><br>Sample amount: 3-25ml |

## Instrument characteristics

**Model: Viscometer DV3TRV (Brookfield)**

**Viscosity Range:**
100 - 40.000.000 cP
**Speed Range:**
0.01-250 RPM
**Viscosity Accuracy:**
± 1.0%
**Repeatability:**
± 0.2 %
**Temperature Sensing Range:**
100°C to 300°C
**Spring Torque Range:**
7,187.0 dyne•cm to 0.7187 mN•m
**Accessories:**
6 spindles depending on viscosity range
**Software:**
RheocalcT

# Viscometer/Rheometer

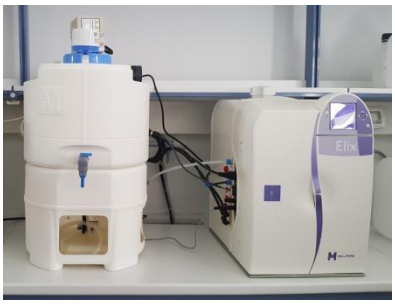
Informally, viscosity is the quantity that describes a fluid's resistance to flow. Fluids resist the relative motion of immersed objects through them as well as to the motion of layers with differing velocities within them. The principle of operation of the DV-III+ is to drive a spindle (which is immersed in the test fluid) through a calibrated spring. The viscous drag of the fluid against the spindle is measured by the spring deflection. Spring deflection is measured with a rotary transducer. The measuring range of a DV-III+ (in centipoise) is determined by the rotational speed of the spindle, the size and shape of the spindle, the container the spindle is rotating in, and the full scale torque of the calibrated spring.
The DV3T Rheometer has a built-in yield stress measurement capability that determines the stress required to initiate flow of slow moving or paste materials. Vane spindles can be immersed into a material without destroying the underlying structures that contribute to yield. The DV3T offers test parameters that create a specific yield test protocol that can be utilized for QC testing or research through the advance software (RheocalcT) that can analyze data, generate multiple plot overlays, print tabular data, run math models and perform other time-saving routines.

| Services | Sample properties |
|---|---|
| • Viscosity (cP or mPa•s)<br>• Shear Rate/Stress<br>• % Torque<br>• Analyze characteristics such as yield stress, flow curves (mixing, pumping, spraying), leveling and recovery | Liquids, semi-solids, creams |

# SUPPORTING INSTRUMENTATION

**Water Purification system (Pure water)**

This system is combined with bacteriocidal UV lamp treatment to produce pure and reliable water quality.



**Millipore, Elix 3**

**Water Purification system (UltraPure water)**

**Millipore, Milli-Q RG**

**Drying/Heating Chamber**

Nominal Temperature: 300°C

**Binder, ED53**

## Centrifuge

**Biofuge stratus, Heraus**

## Pressure pump

Pellet preparation


**Specac**

## Sonicators

**(A)**


**2510E-MT, Branson**

**(B)**


**Ultrasonic bath, XUBA1, Grant**

**(C)**


2 ultrasonic frequencies (37&80kHz)

Extra functions:

- Sweep
- Degassing
- Pulse

**Elmasonic P, Elma**

## Analytical balances

All balances are calibrated using certified weights

**(A)**

MAX=220g
Min=10mg
e=1mg
d=0.1mg

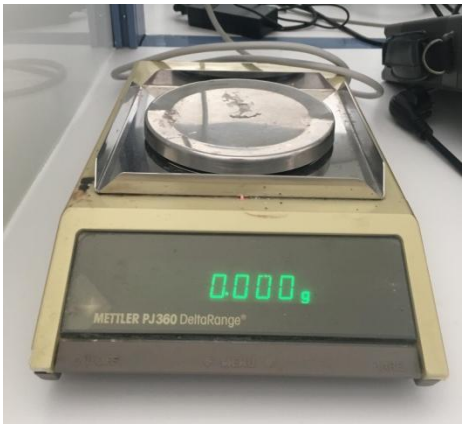
**ABJ 220-4NM, KERN**

**(B)**

MAX=210g
d=0.1mg

**AG204, Mettler Toledo**

**(C)**



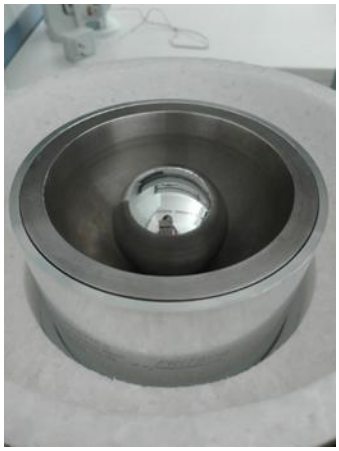

**PJ360, DeltaRange, Mettler**

**Vibratory Micro-Mill & Cryo-box**

Fine comminution of laboratory sample material, powders or suspensions. Homogenizing emulsions or pastes. Grinding in liquid Nitrogen for polymers and biological tissues. The system offers control of grinding duration.

**Grinding duration:** 10 – 30 min (average)
**Amplitude:** 1 – 2 mm (max.)
**Feed quantity:** Max. 10 ml (particle feed size ≤5 mm)





**Model: Pulverisette 0, FRITSCH**

# INSTRUMENTS AT OTHER SITES

**Instrument characteristics**

**Model: Q100, TA Instruments**

*Location: Institute of Chemical Engineering Sciences*

*P.I.C.: I. Parthenios*

**Temperature range:**
-180 to 725 $^{o}$C

**Temperature accuracy:**
± 0.1 $^{o}$C

**Temperature precision:**
± 0.05 $^{o}$C

**Calorimetric reproducibility (indium):**
± 0.1%

**Calorimetric precision (indium):**
± 0.1%

**Dynamic measurement range:**
>± 500mW

**Baseline curvature (Tzero; -50 -300 $^{o}$C):**
10μW

**Baseline reproducibility with Tzero:**
±10μW

**Sensitivity:**
0.2 μW

**Indium Height/width (mW/ $^{o}$C):**
30

## Differential scanning Calorimetry (DSC)

According to the ASTM standard E473, DSC is a technique in which the heat flow rate difference into a substance and a reference is measured as a function of temperature, while the sample is subjected to a controlled temperature program. It uses the temperature difference developed between the sample, and a reference for calculation of the heat flow. An exotherm indicates heat flowing out of the sample, while an endotherm indicates heat flowing in.

If a temperature modulation is overlaid on a linear heating or cooling rate the technique is then called Modulated temperature differential scanning calorimetry (MDSC). An MTDSC experiment can not only generate the total heat flow similar to the heat flow obtained in conventional DSC but also separate the total heat flow into its reversing and nonreversing components. The total heat flow is the sum of the thermal events and is generally equivalent to the heat flow seen in conventional DSC. The reversing heat flow is the heat capacity component of the total heat flow.

| Services | Sample properties |
|---|---|
| • Glass transition temperature detrmination<br>• melting and crystallization temperatures<br>• heat of fusion<br>• heat of reactions<br>• purity determination<br>• heat capacity measurements<br>• characterization of thermosets<br>• chemical reactions kinetics, (cure, thermal and thermoxidative degradation)<br>• polymer crystallization kinetics | Sample quantity: 5-20mg |

## Instrument characteristics

**Model: Zeiss SUPRA 35VP**
*Location: Institute of Chemical Engineering Sciences*
*P.I.C.: V. Drakopoulos*

**Electron Gun:**
FE
**Vacuum modes:**
HV:10-$^{5}$mbar
LV: 133Pa $N_2$
**Resolution:**
1,7nm at 20kV (HV)
2,0nm at 30kV (LV)
**Image Detectors**
ET-SE, VPSE, Inlens-SE, BSE
**Element indentification:**
C-U Standarless
**Quantitative analysis:**
point/line/map
**Standard quantitative analysis:**
point
**Phase imaging**
**Orientation imaging**
**Texture analysis**

# Scanning Electron Microscope (SEM)

Scanning electron microscopy (SEM) is a well-known technique for the morphological characterization of materials. In SEM, a finely focused electron beam with energy of 30kV scans over a sample surface and the secondary, backscattered electron etc. are emitted from the surface. The intensity of the emitted electrons is sensitive to the topographic features of the sample surface. The signals are collected from a detector and amplified. The variation of the resulting signal strength varies the brightness the trace of a CRT that is scanned synchronously with the electron beam. The image of the surface is thus displayed on to the CRT screen.
Elemental chemical analysis can be performed in a SEM microscope by measuring the energy or wavelength distribution of the X-ray signal generated by the electron beam. For this purpose, the SEM microscope is equipped with an EDS analyser allowing for semiquantitative and quantitative chemical analysis. Also phase imaging and orientation imaging can be measured with HKL EBSD system that is added on the microscope.

| Services | Sample properties |
|---|---|
| • Surface morphology<br>• Elemental analysis<br>• Texture analysis<br>• Phase imaging<br>• Orientation imaging | Powders, pellets, solids<br>Max height: 25mm<br>Weight: less than 0.5 Kg |

**Instrument characteristics**

**Model: Invia Reflex, RENISHAW (U.K.)**

*Location: Institute of Chemical Engineering Sciences*

*P.I.C.: I. Parthenios*

**Laser:**

785nm, 632nm and 514nm

**Accessory:**

Automated movable sample holder stage with streamline HR capabilities

## Raman Spectrometry

When a photon of a monochromatic laser beam strike/interacts with a molecule then the photon is mainly scattered elastically and we have the so-called elastic Rayleigh scattering. During this interaction there is a low probability of the photon energy to cause vibrational excitation or even rarely vibrational relaxation of the molecule and the photon is thus scattered with respectively lower or higher energy. This process is called Inelastic Raman Scattering effect and these vibrational transitions are characteristic for each molecule. The Raman Scattering is quite weak phenomenon and Raman spectroscopy was considered as an awkward technique especially when it should have to be applied to simple everyday problems that required speed, straightforward results and clarity, although it is an almost no sample preparation technique. However, new Raman instrumentation based on new concepts of monochromator designs, laser technology, CCD cameras, fiber optics application as well as backscattering collection geometry through microscope objectives rendered Raman spectroscopy as one of the most powerful techniques for material characterization. The application of Raman spectroscopy was further expanded by the construction of Raman micro-probes that have been successfully applied to a wide range of analyses to identify contaminants, inhomogeneities and properties of materials and structures encountered in industrial production even in hazardous and not-friendly environments.

In micro-Raman mapping spectroscopy (B) imaging and mapping of samples can also be performed.

| Services | Sample properties |
|---|---|
| • Determination of polymorphic forms in extremely low percentages<br>• Identification of substances<br>• Structure identification of variety of materials in all states of matter<br>• Molecular orientation in polymers<br>• Degree of polymer crystallinity<br>• Quantitative measurements, Thermodynamic functions evaluation<br>• Molten salts, oxide melts, corrosive melts<br>• Low-frequency studies<br>• Films<br>• Identification of nanotubes<br>• Biological samples | Raman spectroscopy is a non-destructive technique, which needs only a small portion of a sample to identify its chemical structure. A variety of samples such as powders, solids, liquids, gases, glasses, fibers can be measured. |

## Instrument Characteristics

**Model: TA Q50, TA Instruments**

*Location: Department of Chemical Engineering*

*P.I.C.: P. Koutsoukos*

**Software:**
THERMAL ADVANTAGE 3.2.0Q100

**Temperature range:**
ambient to 1000 $^{o}$C

**Temperature accuracy:**
± 1 $^{o}$C

**Isothermal Temperature precision:**
± 0.1 $^{o}$C

**Heating Rate Range:**
0.1 to 100°C/min in 0.01°C/min increments (standard furnace) 0.1 to 50°C/min in 0.01°C/min increments (EGA furnace)

**Furnace Cooling:**
Forced Air 1000°C to 50°C in < 12 min.

**Weighing Capacity:**
1.0 grams

**Sensitivity:**
0.1 μg

**Weighing Precision:**
± 0.01%

# Thermogravimetric Analysis (TGA)

This general purpose thermogravimetric analyser is used to record the mass loss (or gain) of solid samples as a function of temperature. The measured weight changes characterize both the specific material and the processes that take place upon heating. Common applications of TGA include the study of ceramics, catalysts, minerals and composites, pharmaceuticals, food etc.

| Services | Sample properties |
|---|---|
| • Determination of the percentage of volatile substances.<br>• Identification of the hydrate forms of API. | Sample quantity: 5-20mg |

## Surface area and pore size distribution analyzer

The apparatus is suitable for measuring the specific surface area of solid samples as well as their total porosity (0.5-500 Å) and pore size distribution. Measurements are performed in a series of experiments that involve absorption-desorption of nitrogen/helium mixtures at the temperature of liquid nitrogen (absorption) and at room temperature (desorption). It is a valuable instrument for the characterization of catalysts, crystalline and amorphous powders, absorbents, building materials, etc.

| Services | Sample properties |
|---|---|
| • Determination of specific surface area and porosity using BET isotherm | Powders |

### Instrument Characteristics

**Model: GEMINI model 2170, Micromeritics**

*Location: Department of Chemical Engineering*

*P.I.C.: P. Koutsoukos*

**Software:**

Stardriver

**Temperature range:**

Ambient to 1000 $^{o}$C