Scientific Services Enviromics

Microscopical and Chemical Analyses

Our Microscopical and Chemical Labs are equipped with a variety of state-of-the-art infrastructure. For scientific services, we focus on optical, fluorescence and Raman microscopy, portable FTIR and Raman spectroscopy, SEM-EDX, HPLC, IC and air quality measurements.

Meet the team

— **The Microscopical and Chemical Analyses experts** belong to the A-Sense Lab, one of the research groups of Enviromics and part of the Bioscience Engineering Department (Faculty of Sciences, University of Antwerp). The acronym "A-Sense Lab" stands for "Antwerp Electrochemical and Analytical Sciences Lab". The team has built up **expertise** in the field of microscopical and chemical analysis for more than 10 years and can benefit from an extensive range of analytical and (electro)chemical state-of-the-art equipment.

— Typical activities include **reliable qualitative** and **quantitative** characterization of various types of samples up to the trace level. Highly specific compositional data obtained by means of point measurements can be combined with analytical imaging in case of multilayered or heterogeneous samples.

Regardless the question or challenge, you can count on the deployment of a wide **range of complementary techniques and skills** based on microscopy, spectroscopy and chromatography. The work is always carried out by a fixed team of experienced researchers and laboratory technicians.

— In addition to the techniques listed below, the team has also developed strong expertise in **electrochemical analyses** including electrochemical fingerprinting and sensing, spectro-electrochemistry and bio-electrochemistry. Do you have a specific question or would you like to receive a quote? Our Valorisation Managers will be happy to help you and will look for the best solution to your problem together with our technical experts.

Optical and fluorescence microscopy

Optical microscopy generates magnified images of small objects, allowing the observation of the samples' morphology in detail with a resolution of 0.4 µm. Optical microscopy can be used with visible light, providing images of the samples and their real colors. Furthermore, optical microscopy can be coupled to a monochromatic light source that illuminates the sample in the range of 365 to 770 nm. This provides information on the samples' heterogeneity at the microscopic level and is referred to as fluorescence microscopy.

APPLICATION

The images obtained by optical microscopy provide information on the morphology of the sample. Moreover, fluorescence microscopy provides information on certain heterogeneities that cannot be appreciated with visible light. Optical and fluorescence microscopy are compatible, meaning that both techniques can be applied on the same sample and provide complementary results. This enables us to perform subsequent analyses for a wide range of application areas:

- Morphological studies, including size, shape and layer's thickness or homogeneity (the latter requires a suitable sample preparation)
- Presence and size of particles/defects
- Presence of heterogeneities with different chemical composition
- Porosity
- Detection of degradation processes associated to a color change
- ...

SAMPLES

Optical and fluorescence microscopy can be performed on solid or liquid samples with a maximum size of 10 x 10 x 3 cm³ (w x d x h). However, additional preparation steps are often needed such as cross-sectioning or staining. We can offer these techniques in-house, to guarantee a fast and correct service. Typical examples of samples for optical and fluorescence analyses are:

- Layers or coatings (e.g. paint, ...)
- (Polymeric) surfaces
- (Colored) particles
- Cross sections
- Biological samples (e.g. insects, fungi, ...)
- ...

KEY EQUIPMENT

- Nikon Eclipse LV100N
- Objectives x5, x10, x20 and x50
- Illumination in transmission and reflection
- Dark field and bright field with polarizer
- Light source for fluorescence microscopy: CoolLED pE-4000 (from 365 to 770 nm)
- Specific software for acquiring images of non-flat samples

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Raman microscopy

Raman microscopy is a powerful molecular characterization technique allowing to obtain key information on the chemical composition of unknown samples. A monochromatic laser source is focused down to less than a micron and illuminates the material of interest. The interaction of the material with the laser light causes so-called Raman scattered light which contains information regarding the chemical bounds present.

APPLICATION

Our Raman microscope can efficiently provide Raman spectra for chemical composition and structure analysis, with a sub-micrometer spatial resolution and a spectral resolution below 0.1 cm^{-1}. One of the greatest strengths of our instruments is its flexibility, with four different laser wavelengths allowing us to obtain information in a variety of different sample types. Moreover, our access to comprehensive Raman reference data allows the identification of unknown substances directly. This enables us to perform subsequent analyses for a wide range of application areas:

- Pigment degradation studies
- Identification of polymers
- Band gap determination on semiconductors
- Mineralogical composition
- Crystallography
- ...

SAMPLES

Most organic molecules and inorganic compounds can be easily identified by Raman spectroscopy without the need for elaborate sample preparation, even if knowledge on the sample is limited. To prevent fluorescence, multiple excitation wavelengths are available (405 nm, 532 nm, 785 nm and 1064 nm). Samples of 76 mm x 112 mm and up to 0.5 kg can be completely analyzed. Additional preparation steps could be interesting in order to answer a question, e.g. microtome slicing. We can offer these techniques in-house. Typical examples of samples for Raman analyses are:

- Embedded paint fragments
- (Human) cells
- Electronic components
- (Nano)powders
- Coatings
- ...

KEY EQUIPMENT

Invia Qontor Raman microscope

- Excitation lasers (Raman shift range): 405 nm (100 cm^{-1} - 4000 cm^{-1}), 532 nm (50 cm^{-1} - 4000 cm^{-1}), 785 nm (100 cm^{-1} - 4000 cm^{-1}), 1064 nm (60 cm^{-1} - 4000 cm^{-1})
- Objectives: 5x (0.15 N.A.), 20x (0.50 N.A.), 40x cover slip corrected (L 0.60 N.A.), 50x long working distance (0.55 N.A.), 74x IR (0.65 N.A.) 100x (0.90 N.A.), 100x water immersion (1.25 N.A.)
- Detectors: Centrus CCD (400 – 1100 nm), Andor InGaAs (1070 – 1700 nm)
- 1064 nm laser trap
- Resolution < 1 cm^{-1}

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Portable Fourier transform infrared and Raman spectroscopies

Fourier transform infrared (FTIR) and Raman spectroscopies are powerful molecular characterization techniques allowing to obtain key information on the chemical composition of unknown samples. Both techniques exploit the interaction of a light beam (infrared light for FTIR and a visible laser for Raman) with the sample to understand its molecular and structural properties. The different light sources and working principles of these techniques make it possible to obtain different and complementary information, with a strong synergic effect when used together.

APPLICATION

With our portable FTIR spectrometer, measurements can be performed in transmission mode and in ATR (attenuated total reflectance) mode, yielding great versatility. Our portable Raman can perform contactless analyses, which is particularly relevant for chemically aggressive materials and valuable and/or fragile objects. Since both systems are portable and contain IR/Raman reference databases, unknown substances can be identified directly in the field. This enables us to perform subsequent analyses for a wide range of application areas:

- Illicit drugs screening
- Characterization of pigments and photosensitizers
- Characterization of polymers, binders and coatings
- Characterization of pharmaceuticals
- Characterization of synthesis products
- Characterization of cultural heritage materials (e.g. textiles, dyes, binding media, corrosion products, ...)
- Food analysis
- Soil analysis (organic and inorganic components)
- Wastewater analysis
- ...

SAMPLES

A wide range of solid and liquid materials can be analyzed with our portable FTIR and Raman spectrometers. For FTIR measurements, the transmission mode guarantees an optimal spectral quality but is usually restricted to lab analyses as it requires sample preparation. Direct in situ measurements with a good spectral quality can be performed using the ATR module of the FTIR spectrometer. In this configuration, no sample preparation is required and 1 mg of sample is normally enough to obtain clear results. In principle, any powdered sample and most water-based solutions and organic solvents (apart from chemically aggressive substances such as concentrated acids/bases) can be analyzed by FTIR. Our Raman spectrometer can analyze an even wider range of solids and liquids, including non-invasive in situ analysis of chemically aggressive species (e.g. through a transparent packaging), due to the non-contact nature of the technique. Typical examples of samples for portable FTIR and/or Raman analyses are:

- Aqueous solutions (including concentrated acids and bases)
- Organic solvents and analytes in organic solvents (including DMSO and DMF)
- Powdered samples of unknown composition
- Solid materials (e.g. ores, minerals, jewels, precious stones, ...)
- Cultural heritage objects, in situ and in a non-invasive manner (e.g. paintings, manuscripts, metal objects, windows and glass objects, ...)
- ...

KEY EQUIPMENT

Bruker Alpha II FTIR Spectrometer

- IR source: CenterGlowTM technology for continuously optimized light flux
- Detector: High sensitivity temperature-controlled DLaTGS-detector, high stability against external temperature changes
- Spectral range: 350 – 8,000 cm^{-1}
- Spectral resolution: Better than 2 cm^{-1}, optional better than 0.75 cm^{-1}, free adjustable resolution from 0.75 cm^{-1} to 256 cm^{-1}
- Module for transmission analysis
- ATR module with diamond crystal
- Size: in ATR configuration approx. in transmission configuration approx. 208 x 310 x 140 mm
- Weight: approx. 7 kg
- Multiple libraries for material identification

Bruker BRAVO Handheld Raman Spectrometer

- Spectral range: 3.200 - 300 cm^{-1}
- Spectral resolution: 10 - 12 cm^{-1}
- LASER excitation wavelength: 785 nm and 852 nm (Duo LASERTM)
- LASER power / output: low laser power, < 100 mW for both LASERs
- CCD detector
- Integrated calibration
- Size: 270 mm x 156 mm x 62 mm
- Weight: 1.5 kg
- Operating temperature: 5-35 °C
- Operating relative humidity: 0-95% non-condensing
- Regulatory certification and compliance: CE, FCC
- Instrument qualification : USP 1120, EP 2.2.48, ASTM E2529-06, ASTM E1840-96\

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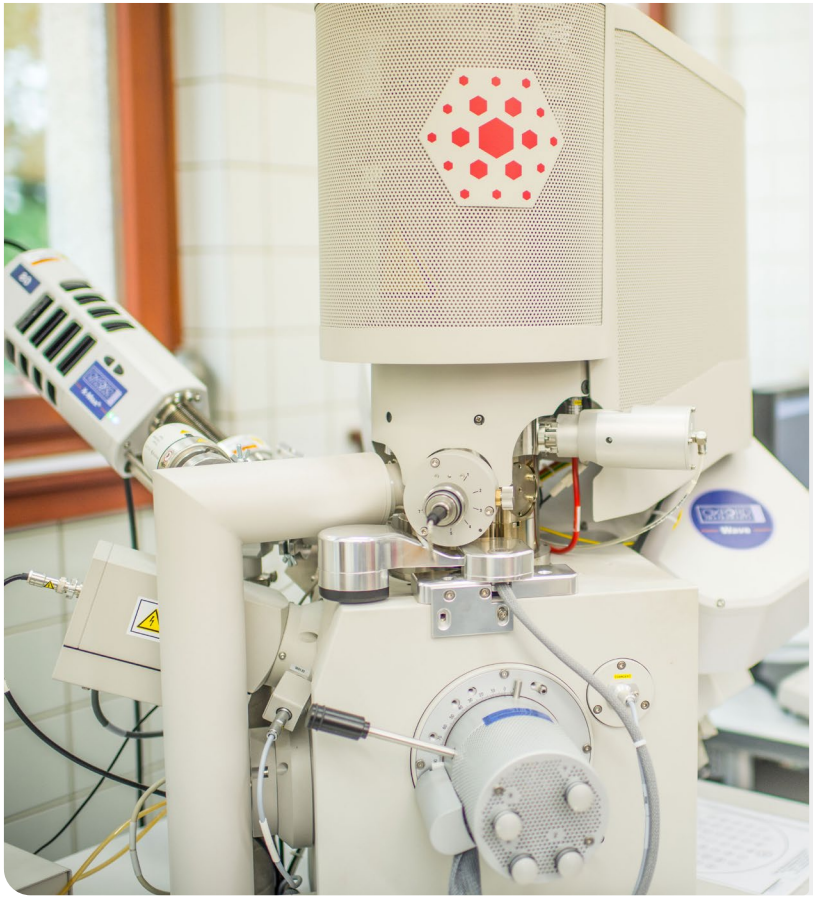
SEM-EDX

With scanning electron microscopy (SEM), an extremely fine electron beam is rapidly moved back and forth across the sample. The interaction of the electron beam with the sample releases various signals. These signals are detected and used by software to form a pixel-by-pixel image of the sample. In addition to the typical detection of electrons, it is also possible to detect characteristic X-rays using an energy dispersive X-ray (EDX) detector.



APPLICATION

SEM is used when the resolution of optical microscopy is insufficient and offers a sub-micrometer resolution combined with a better depth of field. The various signals released during SEM analysis provide different information about the samples: topography/morphology, composition, and very local elemental analysis. This enables us to perform subsequent analyses for a wide range of application areas:

- Microstructural and compositional study on surface and/or cross sections
- Critical dimensions on surface and/or cross sections
- Contamination analysis
- Corrosion analysis
- Particle analysis
- Environmental SEM (ESEM) (for non-conducting samples without any coating and imaging of wet samples and liquids)
- ...

SAMPLES

SEM analyses can be performed on a wide range of materials. The sample, however, must be conductive or coated with a conductive layer, except for ESEM. A sputter coater is available for carbon coating or gold sputtering depending on the requested analysis. Samples of 80 mm x 100 mm and up to 0.5 kg can be completely analyzed. In general, materials need to be vacuum-compatible. However, next to our regular Zeiss EVO10 SEM also an ESEM, Quanta 250, is available. In ESEM mode, when operating under low vacuum or even environmental conditions, also wet (biological) samples and non-conductive samples without the need for sputtering/coating can be analyzed. With our ESEM, samples of 100 mm x 100 mm and up to 2kg can be analyzed. Additional preparation steps are often needed, including embedding samples, cross-sectioning and evaporation/sputtering of conductive material. We can offer these techniques in-house, a must to guarantee a fast and correct service. Typical examples of samples for SEM analyses are:

- Embedded paint fragments
- Cultural heritage objects
- Electronic components
- (Nano)powders
- Coatings
- ...

KEY EQUIPMENT

Zeiss EVO10

- Tungsten filament
- Chamber 310 mm (Ø) x 220 mm
- Acceleration voltage (0.2 kV-30 kV)
- Magnification up to 50,000 X
- Electron beam resolution 3 nm at 30 kV (SE, high vacuum)
- Electron Detectors: Everhart-Thornley SE detector, BS Si detector (BSE)

FEI Quanta 250 ESEM-WDX

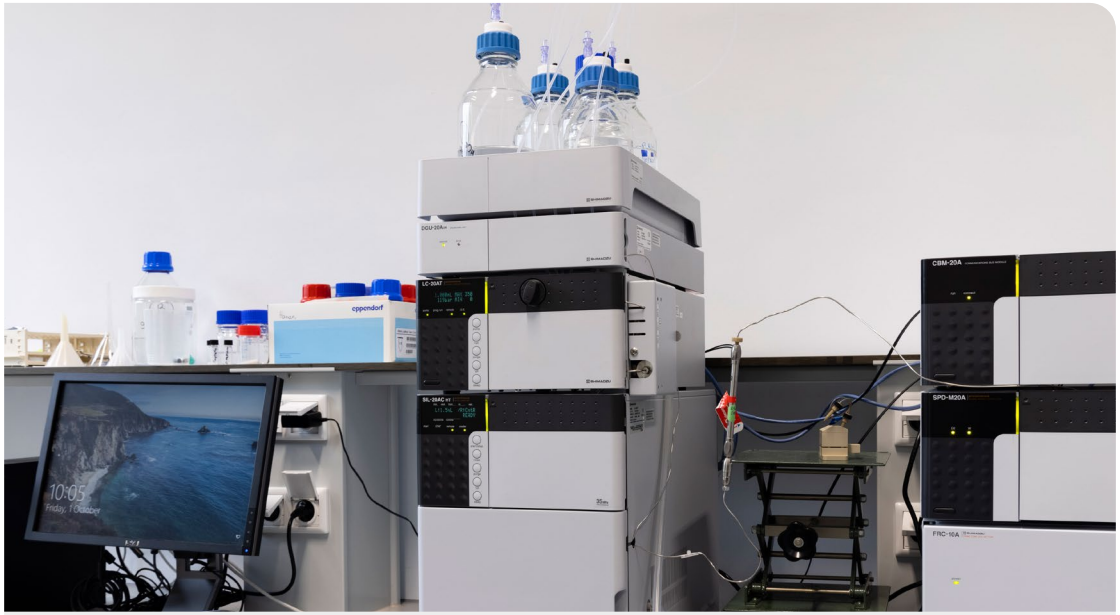
- Schottky FEG
- Acceleration voltage (0.5 -30 kV)
- Magnification up to 100,000 X
- Working chamber pressure (H_2O) 10^{-4} Pa – 4000 Pa
- Peltier stage (-25 °C – +55 °C)
- Electron beam resolution 2.5 nm at 30 kV (BSE, high vacuum)
- Electron beam resolution 1 nm at 30 kV (SE, high vacuum)
- Electron Detectors: Everhardt-Thornley SE detector, BS Si detector (BSE), Large Field Gaseous, SE detector (LFD), Gaseous SE detector (GSED)
- X-ray detectors: Energy Dispersive X-ray (EDX) detector, Wavelength Dispersive X-ray (WDX) detector

LEICA ACE200

- Au sputtering
- Carbon coater
- Thickness control

High-performance liquid chromatography

High Performance Liquid Chromatography (HPLC) is a process of separating components in a liquid mixture. A liquid sample is injected into a stream of solvent (mobile phase) flowing through a column packed with a separation medium (stationary phase). By using different stationary phases (C18-, biphenyl, HILIC) and mobile phases, HPLC can separate and quantify a wide variety of analytes in a diversity of matrices.



APPLICATION

Our HPLC is coupled with a photodiode array detector (DAD) and electrochemical detector. This allows to evaluate UV/vis spectra as well as electrochemical properties of analytes, providing in-depth qualitative and/or quantitative information on the sample. Furthermore, the system is connected to a fraction collector allowing to isolate and purify individual components for further characterization. This enables us to perform subsequent analyses for a wide range of application areas:

- Illicit drugs screening
- Characterization of pharmaceuticals
- Characterization of synthesis products
- Food analysis
- Wastewater analysis
- ...

SAMPLES

A wide range of samples can be analyzed using HPLC. In most cases, filtering (0.45µm) and/or additional sample preparation steps are required to ensure stability of the stationary phase. We can offer these sample preparation steps in-house. Typical examples of samples for HPLC analyses are:

- Illicit drugs
- Legal cannabinoid products with low THC-content (cannabinoid potency testing)
- Samples from pharmaceutical or chemical processes
- Fermentation products (e.g. antibiotics)
- Synthesis products (impurities or degradation products)
- Liquid food samples (phenolic compound or capsaicin quantification)
- ...

KEY EQUIPMENT

Shimadzu 20A HPLC with DAD (190-800nm) and fraction collector.

- Prominence LC20AT with a DGU-20ASR degassing unit, operating pressure up to 35 MPa
- Cooled SIL-20AC HT autosampler; injection volume range 0.1 - 100 µl
- Detector: SPD-20A photodiode array detector (DAD) with temperature-controlled flow cell, wavelength range 190-800 nm, W-halogen- and D2-lamp, 4 channel analogue outlet, includes standard cell, 10 mm path, 10 µl
- FRC-10A fraction collector housing 144 collection vials of 2.5 mL

Electrochemical detector with screen printed electrodes (SPEs)

- In-house developed electrochemical detector employing user-friendly screen-printed electrodes (SPEs); working electrode materials used in the analysis can be easily adapted to the application because of the SPEs
- The electrochemical detector can be used separately or in tandem with the DAD

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Ion chromatography

Ion chromatography (IC) is a process of separating ions in a liquid mixture. A liquid sample is injected into a mobile aqueous stream (mobile phase) flowing through an ion exchanger (stationary phase). By using different ion exchangers, anions or cations can be separated and analyzed.

APPLICATION

IC allows for the analysis of anions and cations in a variety of matrixes. Common ions such as fluoride, chloride, nitrite, bromide, nitrate, phosphate, sulfate, sodium, ammonium, potassium, calcium, magnesium, etc. can be easily quantified. Furthermore, different columns are available to detect other ions. Depending on the amount of sample available, it is possible to detect ions down to ppb levels. This enables us to perform subsequent analyses for a wide range of application areas:

- Environmental analysis
- Waste water analysis
- Quality monitoring
- ...

SAMPLES

A wide range of samples can be analyzed using IC. In most cases, filtering and/or additional sample preparation steps are required to ensure correct analysis. Typical examples of samples for IC analyses are:

- Waste water
- Reactor effluent
- Air filter samplings
- ...

KEY EQUIPMENT

Metrohm 883 Basic IC plus

- Metal free high pressure pump (350 bar)
- Temperature stabilized conductivity meter (0-15,000 µS cm^{-1})
- Anion suppressor
- Metrohm 863 Compact IC Autosampler
- 36 samples
- 10 mL sample tubes (min. 3.0 mL needed)
- Minimizes memory effect

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Air quality

Air quality is important both indoors and outdoors. Poor or suboptimal air quality may limit people's ability to be physically active and is caused by e.g. poorly insulated windows, non-regular maintenance of HVAC, living near busy crossroads, ... Measuring certain air quality parameters gives a general idea of the air quality where it matters.

APPLICATION

Our equipment allows to determine the different particulate matter (PM) fractions and soot concentrations in the air. Additionally, we can examine the collected fractions and try to reveal information on the pollution source. This allows us to perform air quality measurements for different purposes:

- Environmental monitoring
- Workplace health
- Cultural heritage storage conditions
- ...

SAMPLES

Typical examples of air quality measurements that can be performed are:

- Air quality in museums
- Particulate matter at construction sites
- Soot concentrations at busy intersections
- ...

KEY EQUIPMENT

Lighthouse 3016-IAQ

- 6 particle size channels
- 0.3 to 10 µm size range
- Temperature and humidity sensor

Magee scientific aethalometer

- Sensitivity: proportional to flow rate, inversely proportional to time
- Resolution: approximately 0.1 µg/m^{-3} @ 1 minute resolution @ 3 LPM flow rate
- Real-time analysis by measuring the rate of change in absorption of transmitted light due to continuous collection of aerosol deposit on filter. Measurement at 880 nm interpreted as concentration of Black Carbon ('BC')

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