IOCBSERVICE DAYS 200 / 2

Analytical Laboratories

Stanislava Matějková



Analytical Laboratories

Elemental Analysis + OR + DSC Location A 2.87, 2.82 (Stanislava Matějková)

Molecular Spectroscopy (IR, Raman, CD) Location A 2.63, 2.60 (Lucie Bednárová)

Solubility Determination Location A 01.21 (Karel Kudláček)





MS and **NMR** are **not almighty**. A wide range of **impurities** is **invisible** for them. Consequences of such impurities (TFA, Pd...) can be **severe**: your test animals can end up like this...





...or it may lead to misinterpretation of your spectra





Elemental Analysis

Available methods:

- Automatic CHN Analysis
- Fluorine Determination by ISE
- Non-destructive ED-XRF (element range Na U)
- Sensitive ICP-OES method for most elements
- ETV-ICP-OES for the direct solid-state determination in a small amount of e.g. biological material
- Good old-fashioned titration analysis



The confirmed composition of the substance is a criterion of its purity!



Pa

Lr

No

Differential scanning calorimetry

Properties and behavior (e.g. in organism) of polymorphic substances may vary based on their state

DSC can:

- study phase transitions and changes of the sample
- determine the temperature and the enthalpy of phase transitions
- check the quality and purity of substances
- identify and specify the possible polymorphic character affecting the solubility
 T out



Optical rotation measurement

- Is your substance **optically active**?
- Do you need to identify individual enantiomers and determine their specific rotation?
- We offer measurement of optical rotation in various solvents at six wavelengths according to your needs and wishes
 and we hope that soon on a new more modern device :-)
 - ... and we hope that soon on a new, more modern device :-)

Available wavelenghts:

365 nm 405 nm 436 nm 546 nm 589 nm 633 nm



Molecular Spectroscopy

- Vibrational spectroscopy
 - Infrared spectroscopy (IR)
 - Raman spectrometry (RS)
- Chiroptical spectroscopy
 - Electronic (ECD) and Vibrational circular dichroism (VCD)
 - *Raman optical activity* (ROA)
- Spectroscopic identification of natural and/or synthetic compounds



Vibrational Spectroscopy - IR, Raman

- Chemical composition
- Presence of discrete functional groups
- Identification of impurities
- Chemical identification of unknown matter

What can be studied?

- Solution, solid state, gas phase (> 1mg)
- Kinetics, melting temperature
- Possible coupling with electrochemistry (in situ)
- Mapping, polarization measurements (Raman)
- Data interpretation (comercial and own libraries)



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Circular Dichroism Spectroscopy (ECD, VCD)

- Information on chirality stereochemistry assignment
- Structural assignment of biopolymers (secondary and tertiary structure of proteins/peptides; conformation of DNA, RNA)

What can be studied

- Solution, layers, solid state (~ 1mg for ECD, ~ 5mg VCD)
- Linear dichroism (monolayer only) (only UV/VIS)
- Kinetics, melting temperature (only UV/VIS)
- Coupling with electrochemistry (*in situ*) (only UV/VIS)
- Magnetic circular dichroism (only UV/VIS)
- Fluorescence





Solubility Determination

What is the concentration of saturated solution?

How can you get answer?

Methods

- Kinetic (fast, but results are indicative)
- Thermodynamic (slow, true solubility value)

Equipment and procedure

- HPLC + UV/VIS + CAD detection (lack of chromophore) = true solubility value
- PAL-RTC (robotic arm) precise calibration
- Method development for every sample
- Stability testing (sample stress experiment)
- Purity testing (we see impurities)

Sample weight of 1-2 mg Chemical individuals



Precise Weightning

All presented analytical methods require **accurate dosing** of **small amounts of sample around 1 mg**, therefore

- We are equipped with 4 microbalances with a readability of 0.001 mg or 0.005 mg
- We can **weigh your samples** for further processing, typical weights range from 0.1 mg upwards
- Solid, liquid and sticky samples can be weighted





Detailed information about our staff, methods and sample requirements is available on the intraweb



Our service is priceless, but free for you ^(C)



Thank you for your kind attention!

And the management of IOCB AS CR for permanent support of the development on new analytical techniques and instrumentation