Drug analysis, laboratory III year, Module Prof. Barbato. aa. 2015-2016

1. Elementary analysis: calcination, HCN analysis of organic molecules of pharmacological interest.

1.A Laboratory practical exercise: Minimum formula of compounds from HCN analysis data, examples.

1.B Laboratory practical exercise: HCN analysis of 8 unknown compounds and minimum formula proposal.

2. Digitalization of signal: ADC conversion, digitazion process, S/N and its effects.

3. IR spectroscopy: vibro-rotational states, the vibrating diatomic molecule model, the diatomic, the vibro-rotation spectrum of carbon monoxide, vibration of polyatomic molecules. IR spectra of organic molecules with characteristic functional groups: aldehyde, ketons, carboxylic acids, esthers, alcohols, primary, secondary and tertiary amines, alkenes, alkines, alkanes, aromatics

Laboratory activities: Each student is lead to acquire a spectra hands on the FT-IR instrument.

3.A Laboratory practical exercise: Recognition of functional groups of 24 different organic compounds

3.B Practical aspects of FT-IR acquisition: The scan concept, sum of scans, scan speed and resolution, interferences, difference spectra. Control software management and essential parameters to acquire an IR spectra. Transfer of acquired spectra for printing, peak picking and threshold setup.

3.C Laboratory experience: Difference spectra of 8 unknown compounds (same as 1.B) and solvent. Identification of functional groups.

4. Nuclear Magnetic Resonance (NMR) spectroscopy: the physics fundamentals, Nuclear spin and applied field, vectorial description of the magnetization, radiofrequency pulse effects; 1D NMR signal detection. Chemical shift; spin-spin interaction, scalar coupling and dipolar coupling. Decoupling. 1H and 13C spectra, DEPT. Integration of proton spectra. NMR spectra interpretation of organic compounds.

Laboratory activities: Each student is lead to acquire a spectra hands on the NMR300 MHz instrument.

4.A Laboratory practical exercise: Integration of 1H spectra and matching with minimum formula; practical analysis of spectra from 24 different compounds (same as 3.A), and molecular structure proposal.

4.B Practical aspects of NMR acquisition: The Lock function, Tuning and Matching, pulse calibration. Control software management and essential acquisition parameters for 1D acquisition.

4.C Laboratory experience: NMR spectra of 8 unknown compounds (same as 1.B).

5. Mass spectrometry: The physics fundamentals, sample introduction techniques, Ionization techniques, Mass analyzers, Resolution; Magnetic and electrostatic sector; Quadrupoles; Time-of-Flight; Ion cyclotron Resonance; Ion trap. Fragmentation and profile use. Mass spectra interpretation of organic compounds.

Laboratory activity:

5.A Laboratory practical exercise: practical analysis of spectra from 24 different compounds (same as 3.A), and molecular ion identification.

5.B Laboratory practical exercise: analysis of the mass spectra of 8 unknown compounds (same as 1.B).

6. Analysis strategies: combined use of different spectroscopic techniques to analyze pharmacologically relevant organic molecules.

Laboratory activity

6.A Combined analysis of HCN, MS, IR and NMR to identify the molecular structure of organic compounds and of molecules of pharmaceutical relevance (specific examples: GABA, Aspirin, Nimensulide).

Books:

The spectrometric identification of organic compounds. Silverstein, Webster, Kiemle. 7th edition (2005) or more recent, Wiley & sons. ISBN-10: 0471393622
Books for reference, not to buy, but where to find different and complementary explanations of what seen at lesson. You can find these books at the University library under the codes indicated.
Fundamentals of molecular spectroscopy. C.N. Banwell, McGraw Hill. [F33-0002] (Chap. #1-3, 5,6,7)***
Biophysical chemistry. Vol. II. Cantor & Schimmell. Freeman & Co. [574-19-CAN] (Chap. #7, 8,9)***
Instrumental Methods of Analysis. Willard Hobart H. 7nth edition. Wadsworth Publishing Co. [543-08-WIL] (Chap #5-7, 11, 15, 16)***