

Mass Spectrometry Unit

Introduction

Although the combination of gas chromatography with mass spectrometry (GC-MS) was first reported in 1958, made available commercially in 1967 and used widely for decades, many polar compounds are impossible to analyze with GC-MS. The need to combine the resolving power of liquid chromatography (LC) with the detection specificity of mass spectrometry (MS) for large biologically important molecules, led to the development of electrospray ionisation (ESI). This powerful technique was invented by Malcom Dole *et al.* in 1970 for mass spectrometry. His pioneering experiments demonstrated the use of electrospray to ionise intact chemical species. Twenty years later John Fenn demonstrated for the first time the use of ESI for the ionisation of high mass biologically important compounds and their subsequent analysis by mass spectrometry. For this work John Fenn was awarded a share of the 2002 Nobel prize for chemistry.

ESI, atmospheric pressure chemical ionisation (APCI) and nanoelectrospray ionisation (NSI) are complementary atmospheric pressure ionisation (API) techniques. Sample components are separated by HPLC and then introduced into the MS where single or multiple charged ions are created and detected. API is a soft ionisation method generating primarily molecular ions $[M + H]^+$, $[M - H]^-$, simple adduct ions $[M + Na]^+$ and ions representing simple losses, e.g. $[M + H - H_2O]^+$. Fragmentation of ions is rare. Multiple charges enable the detection of molecules as large as 150,000 u even though the mass-to-charge range for a typical LC/MS instrument is around 3000 m/z (e.g.: 100,000 u / 10 z = 1,000 m/z). Today, instruments that have more than one analyser, the so-called tandem (MS/MS) mass spectrometers, are used to provide information about the molecular weight, structure and sequence, identity and quantity of specific chemical components. LC-ESI works at atmospheric pressure at a flow rate of solute and solvent between 1 μ L/min and 1 mL/min. APCI works at similar flow rates and is used to detect less polar chemical components. NSI is a low (30-1000 nL/min.) flow rate version of ESI, more suitable for peptide and protein detection. LC-MS instruments are capable of "direct sample injection". Subsequent fragmentation of detected ions by MS/MS is reminiscent of the classical electron impact (EI) technique. Popular mass analysers for API instruments, include the quadruple, ion-trap, Fourier transform-ion cyclotron resonance and time-of-flight analysers, depending on the requirements of an analysis.

Facilities & Infrastructure

The Unit possesses an Agilent 1100 Series LC/MSD Trap System (Figure 1) that consists of the following components:

- (1) ion trap mass spectrometer, MS^n n = 11 (MSD trap, model SL),
- (2) atmospheric pressure interface: (a) electrospray ionisation, (b) chemical ionisation,
- (3) Agilent 1100 HPLC system (a) solvent cabinet, (b) degasser, (c) control module, (d) LC pump, (e) standard autosampler, (f) thermostated column compartment, (g) diode-array visible-UV detector,
- (4) monitor,
- (5) computerized data acquisition and manipulation by Chemstation logistics,
- (6) binary pump,
- (7) nitrogen generator,
- (8) UPS system.



Figure 1: General view of the low resolution Agilent 1100 Series LC/MSD Trap System. Ion trap mass spectrometer (1) with its interface (2), the Agilent 1100 HPLC system (3), monitor (4) and computer (5) are shown, only.

Services

The Unit typically renders services in the following areas:

- Molecular weight determination of novel organic and inorganic materials
- Novel material characterization
- Clinical applications
- Environmental Applications
- Biochemical applications
- Food applications
- Pharmaceutical applications



Staff & Contact Information

A Scientific Committee, made up of staff personnel of the University of Ioannina is responsible for the Mass Spectrometry Unit. Information may be obtained from G. Varvounis.

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Representative publications

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3. Zois Syrgiannis, Z.; Fotios Koutsianopoulos, F.; Kenneth W. Muir, K. W.; Yiannis Elemes, Reaction of a triazolinedione with simple alkenes. Isolation and characterization of hydration products. *Y. Tetrahedron Lett.* **2009**, *50*, 277–280.
4. Kallimanis, A.; Kavakiotis, K.; Perisynakis, A.; Spröer, C.; Pukall, R.; Drainas, C.; Koukkou, A. I. *Arthrobacter phenanthrenivorans* sp. nov., to accommodate the phenanthrene-degrading bacterium *Arthrobacter* sp. strain Sphe3. *Int. J. Syst. Evol. Microbiol.* **2009**, *59*, 275–279.
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6. Goulas, V.; Papoti, V. T.; Exarchou, V.; Tsimidou, M. Z.; Gerothanassis, I. P. Contribution of Flavonoids to the Overall Radical Scavenging Activity of Olive (*Olea europaea* L.) Leaf Polar Extracts. *J. Agric. Food Chem.* **2010**, *58*, 3303–3308.