Mass Analysis

Equipment, Precautions & Q&A

World Top 10 University by 2030

Jul 13, 2016

UNIST Central Research Facilities

ULSAN NATIONAL INSTITUTE OF SCIENCE AND TECHNOLOGY
2009

1. Equipment

(is currently working)

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UMCL_MALDI-TOF/TOF & GC/MS/MS

❖ MALDI-TOF/TOF

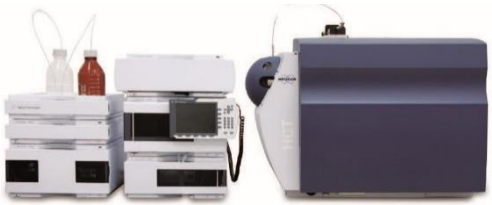
- **UltraflexIII (Bruker, Germany)**
- MALDI: Soft ionization, molecular weight
- Mass accuracy of ≤ 10 ppm (for peptide mass range in reflectron mode)
- Mass accuracy of ≤ 100 ppm (for protein mass range in linear mode)
- High mass resolution at every mass from 700 ~ 6,000 Da
- Analysis of large organic molecules and biomolecules
- Preference for volatile solvents such as THF, chloroform, and etc.
- Surface uniformity → Accuracy & reproducibility
- Choice of matrix
- **Self-user**

❖ GC/MS/MS

- **450-GC & 350-MS (Bruker, Germany)**
- Electron impact ionization: Strong ionization, Fragmentation pattern
- Mass range at GC/MS mode: 10 ~ 450 amu
- Mass range at DIP(EI) MS mode: 10 ~ 1,000 amu
- Library : NIST library
- Nominal mass accuracy
- Confirmation of organic compounds structure
- Confirmation of molecular weight
- Volatile solvents use (**Water is restricted**)
- Solid sample is applicable in DIP mode.

UMCL_LC/MS/MS & GPC/MALS

❖ LC/MS/MS



- **HCT Basic System (Bruker, Germany)**
- Ion source: ESI, APCI
- Mass range: 50 ~ 3,000 (m/z)
- Resolution: 0.6 unit
- Mass accuracy: ±0.15 unit
- Scan speed: 26,000 amu/sec
- Analysis of organic composition
- Identification of molecular weight
- Preference for polar solvents such as MeOH, ACN, and etc.
- Phosphate buffer is restricted.

❖ GPC/MALS

- **Agilent 1200 series (Agilent, USA), miniDwan (Wyatt, USA)**
- GPC - pressure: 0 ~ 400 bar
  - RI detecter
  - Non-polar column (THF)
  - Wavelength range: 190 ~ 600 nm
- MALS - Light source: 60 mW GaAs
  - Laser wavelength : 658 nm
  - Detectors: 3 angles
  - Molecular weight range: 10^3 ~ 10^6 g/mole
  - Molecular size range: 10 ~ 50 nm
  - Sample > 50 mg
- Analysis of synthetic polymer and biopolymer

UMCL_EA

❖ EA_LECO

- **TrueSpec Micro CHNS (LECO Corporation, USA)**
- Measuring elements: C, H, N
- Measuring range: 0.01%~ 100%
- Sample size: 0.01 ~ 2 mg
- Accuracy: 0.1 ~ 0.3% absolute
- Detector: TCD, IR cell
- Furnace temperature: 1,100°C
- Analysis time: 3 min

❖ EA_Thermo

- **Flash 2000 (Thermo Scientific, Netherlands)**
- Measuring elements: C, H, N, S, O
- Detector: TCD
- Furnace temperature : 1,100°C
- Analysis time - C, H, N, S: 10 min
  - oxygen: 5 min

UEAL_GC/HRMS & GCxGC/TOFMS

❖ GC/HRMS

- **Autospec Premier (Waters, USA)**

- Sensitivity - 5 × 10^{-7} C/ug for methyl stearate at m/z 298.3, EI, 1000 resolution

  - 5 × 10^{-8} C/ug for methyl stearate at m/z 298.3, EI, 10000 resolution

- Mass range: 2 - 1,200 Da or more at full sensitivity
- Resolution: Continuously variable above 60,000 (10% valley) or more. Ducts
- Trace analysis of persistent organic pollutants (Dioxins. PCBs, OCPs etc.)
- Quantitative analysis of various pollutants in environmental samples.

❖ GCxGC/TOFMS

- **Pegasus 4D (LECO, USA)**

- LN_2 Modulator Maximum Temperature: 400 °C
- LN_2 Modulator Maximum Heating Rate: 40 °C/min
- Modulation Periods : 1 to 65 seconds
- Ionization Source : Electron Impact
- Mass Range : 5 ~ 1,000 amu
- Spectral Generation Rate : 1 to 500 spectra/sec

UEAL_LC/MS/MS & ICP-MS

❖ LC/MS/MS

- **Xevo TQ-S (Waters, USA)**

- Ion source: ESI, APCI
- Mass range: 2 ~ 2048 m/z
- Collision cell: Two-wave collision cell
- Detector: Dynolite photomultiplier detector
- Sensitivity: ESI (S/N ration = 300 : 1)
- ACSI (S/N ration = 100 : 1)
- Scan speed : 10,000 amu/sec
- Mass stability : within ± 0.05 Da
- Analysis of environmental samples (water, waste, soil, PPCPs)
- Analysis of organic composition

❖ ICP-MS

- **ELAN DRC-Ⅱ (Perkin Elmer, USA)**

- Dual turbo-molecular pumps with ceramic bearings
- Vacuum levels : 1 x 10^{-6} Torr
- 27.12 MHz ~ 40 MHz RF Generator
- Dynamic Reaction Cell
- Thermal stabilization of the quadrupole power supply (QPS) at > 2.5 MHz mass spectrometer
- Quantitative analysis of various atoms
- Limitation: Light elements (~ Na), Si, S, 17 group, 18 group

MALDI-TOF/TOF: Self-users training

1. 대상 장비 : MALDI-TOF/TOF

2. 연간 교육 횟수 (2016년) : 총 6회

3. 1회 교육 수강 인원 : 10명

4. 교육 방식 : 이론 및 실습 교육 – 개별실습 – 테스트

5. 자율사용자 현황 : 14명_2016년 7월 11일 기준

6. 자율사용자 자격 : 3개월 이상 미사용 시, 자격 박탈

7. 2016년 교육 진행 현황

장비명	교육일	신청인원	수강인원	테스트인원
MALDI-TOF/TOF	**2/16**	10	10	5
	3/17	10	9	2
	5/9	4	4	2
	8/4	-	-	-
	9/1	-	-	-
	11/3	-	-	-

2. Equipment
(will be installed in 2017)

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High Resolution Mass Spectrometer

❖ **UNIST 연구지원본부가 기존에 보유하고 있는 첨단 질량분석장비들과 함께 재료/바이오 분야 연구의 시너지효과 창출**

기존 보유장비

(HRMS, GC/MS, LC/MS, MALDI-TOF/MS 등)

+

도입 예정 장비 (HRMS)

- Resolution: 60,000 (m/z 84, CH_2Cl_2 $CDCl_2$)
- Sensitivity (EI): S/N 400 more (R = 1,000)
- Optional Ion source: EI, CI, FD, FAB, DIP
- Mass Range: Max. 24,000 dalton (1 kV)
- Magnetic Analyzer, Cylindrical Electric Analyzer
- Easy exchange for Ion Source
- Full automation control by PC
- High Sensitivity

3. Q & A

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Survey – Q & A

1. 주제: 질량분석 장비 사용의 개선 및 발전을 위한 설문 조사

2. 대상: UNIST 소속 전임교수, 비전임교수

3. 기간: 2016 . 6. 21-30. (10일)

4. 문항: 1) 질량분석 장비를 이용한 결과의 비중
   2) UCRF 질량분석 장비 사용에 대한 불/만족사항, 개선사항
   3) UCRF에 있는 질량분석 장비이나, 외부 의뢰하는 이유
   4) 질량분석 장비 지원 업무와 관련한 요청/요구 사항
   5) 추가 의견

5. 답변: 10명 회신

6. Q & A: 1) High resolution mass spectrometry
   2) Self-user option
   3) Fee
   4) Seminar / Education

THANK YOU

FIRST IN
CHANGE