**Workshop – Cal“U”Later　生命探測**

**Introduction**

1. **Inductively Coupled Plasma Mass Spectrometry (ICP-MS)**

It is an analytical technique used for detecting elements. It is a type of mass spectroscopy that is capable of the determination of a range of metals and several non-metals at trace concentration levels.

This technology combines a high-temperature ICP (Inductively Coupled Plasma) source with a mass spectrometer. The liquid samples are introduced by a peristaltic pump, to the nebulizer where the sample aerosol is formed. The sample aerosol is instantaneously decomposed in the argon plasma, which is under high temperature (6000 - 10,000K), to form analyte atoms. The atoms are sent to the mass spectrometer region which is held at high vacuum. The atoms are ionized and are then differentiated based on their *mass-to-charge* ratio and a detector receives an ion signal proportional to the concentration.

The water beverage products consist of numerous minerals, including sodium (Na+), calcium (Ca2+), zinc (Zn2+), and other trace amount of mineral salts. In this experiment, the Calcium and Zinc content in an unknown water sample or beverage sample will be investigated by ICP-MS.

1. **FTIR-ATR (Fourier-Transformed Infrared Spectroscopy-Attenuated Total Reflection)**

Infrared spectroscopy (IR) is based on the light absorptions of sample molecules in the infrared region of the electromagnetic spectrum. FT-IR machines can convert these light absorptions by samples into signals that we can understand and manipulate. The absorptions by samples correspond to the specific chemical bonds present in the molecules and are usually presented in wavelengths (ranging from 4000 cm-1 – 600 cm-1). After determination, a result in the form of a spectrum is produced. The spectrum is unique and can be used, much like a human fingerprint, to identify organic samples.

Simply put, [ATR stands for](https://www.bruker.com/fileadmin/user_upload/8-PDF-Docs/OpticalSpectrospcopy/FT-IR/ALPHA/AN/AN79_ATR-Basics_EN.pdf) attenuated total reflection and is a little device incorporated into an FTIR machine. ATR has become the standard technique for IR spectroscopy since it minimizes sample preparation works and shortens the determination time. Meanwhile, the infrared light passes through a crystal of a particular material (diamond, ZnSe or germanium) and interacts with the sample, which is pressed onto the crystal. In this way, an FTIR spectrum is achieved without destroying the sample.

[ATR-FTIR](https://commons.wikimedia.org/wiki/File:Thermo_Nicolet_iS10_FT-IR_spectrophotometer.jpg) / Nick Birse / [CC BY-SA 4.0](https://creativecommons.org/licenses/by-sa/4.0/deed.en)

**Objectives:**

1. ICP-MS part:
2. To determine the amount of Metal Contents in Distilled Water or Beverage Sample by Inductively Coupled Plasma Mass Spectrometry (ICP-MS)
3. To prepare the sample with a particular dilution factor
4. FTIR-ATR part:
5. Testing the textile by FTIR-ATR technique
6. Identifying the textile materials from the daily life environment

**1. ICP-MS: (Demonstration and hands-on experiment)**

1. **Preparation of Nitric Acid Solution (1%)** *(Note: this step will be completed by the technician)*
2. Prepare 1% HNO3 solution from concentrated nitric acid (use trace metal analysis grade) in the fume hood. Add 500 mL de-ionized water to the 1L volumetric flask.
3. Wear nitrile gloves or PVC gloves. Use a measuring cylinder to transfer 14.50 mL of the concentrated acid to a 1L volumetric flask and make it up to the mark with DI water. Shake the cylinder upside down to mix the solution well.
4. **Preparation of Calibration Curve (3 points)** *(Note: this step will be completed by the technician)*
5. Prepare 3 working solutions in 25 mL volumetric flasks according to the following table.

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
| **Working Solution No.** | **Blank** | **1** | **2** | **3** |
| **Concentration of Zinc (Zn) and Sodium (Na) standard solution (ppm)** | 0 | 1 | 3 | 5 |
| **Volume of 100 ppm mixed standard solution added (mL)** | 0 | 0.25 | 0.75 | 1.00 |
| **(In case of 1 mL equivalent to 1000µl)** | 0 | ?? | ?? | ?? |

1. Make it up to the mark with 1% HNO3 solution and mix well.
2. Transfer the working solutions to 15 mL centrifuge tubes. *(Note: this step will be completed by technician)*
3. **Sample Preparation (REDOXON® Tablet or Distilled Water) (Student hands-on experiment)**
4. Break the REDOXON® tablet into four pieces and weigh the tablet (1/4) using a 250 mL beaker.
5. Add about 70 mL nitric acid solution (1%) to dissolve the REDOXON® pellet until no gas bubbles are observed.
6. Transfer the orange solution into a 100 mL volumetric flask, fill it up to mark with nitric acid solution (1%), and mix well.
7. Transfer 1 mL of the orange solution to a 10 mL volumetric flask and fill it up to the mark with 1% HNO3 solution.
8. Perform the same procedures for the distilled water sample. Transfer 2 mL of the distilled water solution to a 10 mL volumetric flask and fill it up to the mark with 1% HNO3 solution. (2 mL distilled water: 8 mL 1% HNO3)
9. Fill the centrifuge tube (15 mL) with 10 mL diluted sample solution for heavy metal analysis by ICP-MS.
10. **Workflow chart: ICP-MS**

|  |
| --- |
| **Flow Chart**  **For sample: REDOXON® Tablet**  (Break into 4 pieces)  ⇩  Weighting the tablet (1/4)  (use 250 mL beaker)  ⇩  Add 1% HNO3 solution **(~70 mL)** to dissolve it  ⇩  Transfer the **orange solution** into a  volumetric flask (100 mL)  ⇩  Make it up to the mark with 1% HNO3 solution  ⇩  **Dilution:**  Transfer **1 mL** of the orange solution to a  10 mL volumetric flask (Make up to mark by 1% HNO3)  **Or**  **For Distilled Water sample:**  Transfer 2 mL of the distilled water solution to a 10 mL volumetric flask and make it up to the mark with 1% HNO3 solution.  (2 mL distilled water: 8 mL 1% HNO3)  ⇩  🖙**To conduct ICP-MS analysis (Demonstration)** |

**2. FTIR-ATR: (Demonstration and hands-on experiment)**

1. **Demonstration and video sharing:**
2. Video clips: Introduction of FTIR-ATR
3. Demonstration of FTIR-ATR by Staff
4. **Preparing your sample:**
5. This instrument will collect the IR spectra of solid and liquid samples. Most samples will be tested successfully; provided that they are not air-sensitive or highly corrosive – consult HKMU staff if you are confused.
6. Samples (Textile/clothing) are loaded directly onto the crystal plate and no specific preparation is required.
7. **Using the instrument:**
8. Check that there is no visible debris on the crystal plate or beneath the ATR arm. If there is debris, please do the clean-up first.
9. BEFORE loading your sample, label the sample with the name in the appropriate field and click **“start background measurement.”** It will collect a background spectrum (of air) that will be subtracted from your sample spectrum. This is BLANK.
10. Load the instrument by placing a very small amount (a few milligrams at most) of your sample on the crystal plate in the center of the instrument and lower the anvil by pressing down on the ATR arm until it clicks into place. A **red dot** should now be visible on the front of the arm – if not, consult HKMU staff.
11. Click **“start sample measurement.”** Wait a few moments until the collection is completed and the spectrum is obtained.
12. **Preparing your Spectrum:**
13. Once your spectrum has been collected, a number of tools are available. If your spectrum is poorly centered on the screen, the “spectrum adjustment” tool will allow you to correct it.
14. Try to identify your sample using the IR spectrum chart and the built-in library software.
15. Once you are satisfied with your spectrum, you can save your sample spectrum.

1. **Cleaning Up:**
2. Lift the ATR arm and clean the crystal plate as well as the bottom of the arm thoroughly using a **Kimwipe** and *iso*propanol. Allow the instrument to air-dry for a few minutes before the next reading.