Lab 1: Extrel Mass Spectrometry

A. Getting to Know Your Mass Spec
B. IQ-2000 Software Control

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A mass spectrometer is a gas analyzer. Gaseous mixtures are introduced to a mass spectrometer, ionized, filtered based on mass to charge ratios, and detected. This lab will focus on identifying and understanding the components of a mass spectrometer, experimental setup, and basic data interpretation.

Introduction

There are four main components of a mass spectrometer.

- Vacuum System
- Sample Inlet
- Ionization Source
- Mass Filter
- Detector
- Electronics
- Data System

The ionization source, mass filter, and detector are all enclosed in a vacuum chamber. The IQ-2000 is shown below:

A sample inlet is responsible for bringing gas into the analyzer. The IQ-2000 should be configured for this lab with a 30µm fused silica line running between analysis sample and vacuum chamber. This acts as a very slight “leak” allowing small amounts of gas to pass into the vacuum chamber for analysis, (see Figure 2 below).

Understanding Vacuum Systems

Mass spectrometers need to operate in vacuum systems for two reasons. First, ion-ion interactions need to be limited. Second, the possibility for arcing between high voltage components needs to be minimized.

The mean free path of a gas particle is defined as the distance a particle will travel before in interacts with another particle. Lower pressures yield lower gas densities, increasing the mean free path distance. Increasing this distance can reduce any ion-ion interactions to the point of negligibility.

Additionally, lowering the gas density avoids electrical gas breakdown. At low enough pressures, high voltages can be placed on a conductor sitting very close to another conductor without breakdown or “arcing.” This is because at low enough pressures, there are not enough gas particles to conduct electricity. This phenomenon is dictated by what is called the “Paschen Law.”

An ionization source is needed to apply a charge to gas particles, creating ions. The ionization source found in the IQ-2000 utilizes electron ionization for this process. Gas enters the ionization source, which features two yttria coated iridium filaments. Current is run through these filaments causing them to become very hot and emit electrons. Electrons are propelled from the filament using a voltage bias (giving the electrons kinetic energy) towards our gas molecules. If the kinetic energy in these electrons is high enough to electrostatically repel and electron from a gas molecule, ionization occurs. An ion is a molecule that has a different number of electrons and protons, leaving it with an overall charge. Gas particles can be given different charges, depending on how many electrons are lost in this process. These charges (and mass of a gas particle) will be used to describe an ion’s “mass-to-charge ratio.” The IQ-2000 ionizer also features several lenses (disc-shaped metal plates with applied voltages) that are used to accelerate and focus ions towards the mass filter. A diagram of our ionizer is shown in Figure 2 below:

The mass filter is responsible for separating ions of different masses (using mass/charge ratios). The IQ-2000 features a quadrupole mass filter. A quadrupole is four parallel conductive rods (9.5mm in diameter,

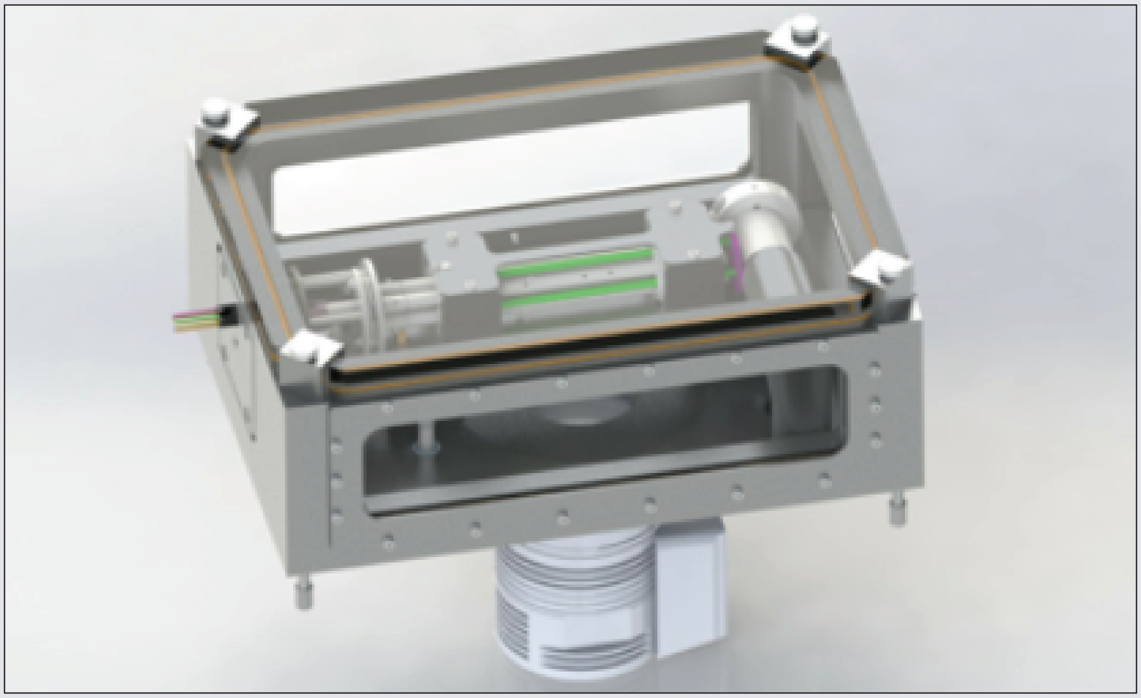

Figure 8.1.1: The capillary inlet configuration for the IQ-2000 vacuum chamber and internal components.

approximately 20cm long) with wiring connecting opposite rods. A quadrupole mass filter uses a combination of RF (radio frequency) and DC (direct current) voltages, ramping in magnitude, to “guide” ions towards a detector. Particular voltage magnitude allows ions of specific mass-to-charge ratios to pass to the detector. Lower voltage magnitudes correspond to lower mass-to-charge ratio ions and higher voltage magnitudes correspond to higher mass-to-charge ratio ions. See diagram below:

A detector measures ion current exiting a mass filter. The IQ-2000 uses a continuous diode electron multiplier detector, featuring a conversion dynode. In normal operating mode (positive ion detection using a conversion dynode), ions are attracted to the high potential (typically ~ -5kV) of the conversion dynode. Upon striking the dynode, electrons are ejected from this abundant source and attracted to the electron multiplier. The electrons will strike the wall of this electron multiplier tube several times, each time emitting more electrons. This creates a cascade effect, and a multitude of electrons are generating ion current from each ion reaching the detector. See Figure 8.1.5

Pre-Lab Questions

In-Depth Study Questions

1. Why is it important to have the sample inlet tubing diameter very small?
2. What characteristic of the chamber may cause arcing between the internal components?
3. Why is it necessary to create “ions” before attempting to filter and detect gases?