INL/CRADA-02-08

# CRADA FINAL REPORT

# An Ultra-Sensitive Detector for On-Line Measurement of Volatile Molecules Produced by Diseased Potato Tubers

## Idaho National Laboratory

## and

## Insightek, Inc.

**Completed: March 25, 2008**

Prepared by
Idaho National Laboratory
Idaho Falls, Idaho 83415
http://www.inl.gov
Under DOE Idaho Operations Office
Contract No. DE-AC07-05ID14517

**Defer Release Until March 25, 2013**



Idaho National Laboratory

Confidential

# Interim Technical Report

An Ultra-Sensitive Detector for On-Line Measurement of Volatile Molecules Produced by Diseased Potato Tubers

Agreement No.: 2005-33610-16514

## Introduction

The overall objective of the proposed Phase II project is to demonstrate the feasibility of a commercially practicable method of detecting the identified molecules with sufficient sensitivity and specificity so as to provide economic improvements in storage health management practices that exceed the cost of implementing the method. Accordingly, the specific technical objectives to be accomplished in this Phase II project are:

1. Construct a prototype IMS detector that will achieve the expected performance and cost improvements defined in Phase I.

2. Experimentally determine the molecular species from those identified in Phase I that will provide optimum sensitivity to IMS detection in the prototype.

3. Design membrane filters that will pass the selected molecular species and attenuate potential interference molecules in the IMS detection platform.

4. Demonstrate the capability of the prototype MF-IMS system to provide health management information.

This report presents the progress in achieving the foregoing technical objectives.

## Prototype Construction

Figure 1 shows a block diagram of the system components. Ion mobility spectrometry (IMS) is a technique to detect and characterize organic vapors in air. It involves the ionization of molecules and their subsequent temporal drift through an electric field. Analysis and characterization of molecules are based on analytic separations resulting from their different ion mobility. The IMS process can be partitioned into the three functional elements of ion generation, ion separation and ion detection. Figure 1 shows the mechanical implementation of these three elements. These three elements are designed as block modules so that any one can be changed without affecting the others. For example alternative ion generator configurations can be tested using a given separation chamber and detector element. Similarly, alternative separation chamber configurations can be evaluated with a given ion generator. Each of these elements is discussed in the following paragraphs.

Ion generation is conventionally accomplished using a radioactive source and an ion shutter to introduce ion packets into the separation chamber. The radioactive source method was deemed too expensive and environmentally risky for agricultural applications. Consequently this research focused on use of pulsed corona discharge as the method of generating ion packets.

There are four distinct types of electrical breakdown of air, namely, Glow Discharges, Corona, Sparks, and Arcs. When the voltage across two conductors in air is very high, the air becomes **ionized** and begins to conduct electricity, or "breaks down." In fact, any gas that conducts electricity is called plasma.

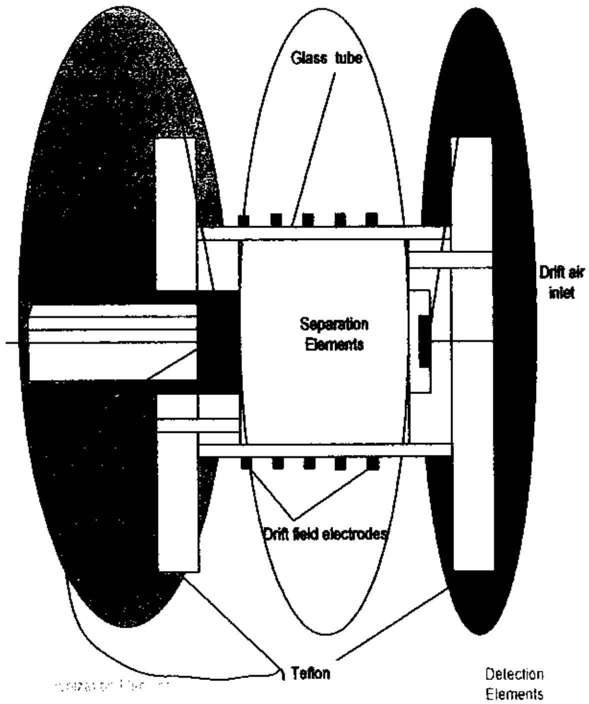



Figure 1 IMS Device Element Assembly

Recent studies of IMS ionization sources in air shows that the reactant ion chemistry in the positive-mode pulsed corona discharge is the same as that in a conventional radioactive ion source. The feasibility of replacement of a conventional radioactive ionization source by pulsed corona discharge has been confirmed. In this study, the electrical pulse used to initiate the corona discharge is obtained by imposing a pulse voltage (from hundreds of volts to several kilovolts) on the two electrodes that are constantly biased by a do voltage (from zero to several kilovolts).

Two conductor configurations for corona ionization that have been tested are the parallel wire configuration shown in figure 2a and the point-to-plane configuration shown in Figure 2b.

There are four distinct types of electrical breakdown of air, namely, Glow Discharges, Corona, Sparks, and Arcs. When the voltage across to conductors in air is very high, the air becomes ionized and begins to conduct electricity, or "breaks down." Coronas have two important voltage parameters: The Disruptive Critical Voltage, $e_0$, and the Visual Critical Voltage, $e_v$. The visual critical voltage is always higher than the disruptive critical voltage. Once $e_v$ is reached, the corona can be seen and remains visible until the voltage reaches $e_0$. Calculations can be made on these voltage parameters as shown below (for parallel wires). In some cases, if the voltage is too high and the distance between the conductors is too small, a spark over will occur. If the voltage is increasing, $e_v$ will be reached. When this occurs, the air surrounding the conductor will ionize and begin to conduct. At this point, the wire's effective radius has been increased since the air is acting like a conductor. The equations below indicate that if this radius changes, the voltage gradient, $g_v$ around the wire changes. If the $g_v$ decreases when the effective radius increases, then corona forms. If, however, $g_v$ increases when the effective radius increases, then spark over will occur.

$$e_0 = m_0\, g_0\, \text{delta}\, r\, (1 + 0.301 / \text{sqrt}(r)) \text{ in kv.}$$

$$e_v = m_v\, g_0\, \text{delta}\, r\, (1 + 0.301 / \text{sqrt}(r)) \ln(S/r) = m_v\, g_v \ln(S/r) \text{ in kv.}$$

Where

- $g_v = g_0$ delta r ( 1 + 0.301 / sqrt(r) ) = Visual Critical Voltage Gradient = de / dr in kv. / cm.
- $g_0$ = 29.8 kv. / cm. (for parallel wires) = Disruptive Critical Voltage Gradient
- $m_v = m_0$ = The wire roughness factor
  = 1 for polished wires
  = 0.98 to 0.93 for roughened, dirty or weathered wires
  = 0.87 to 0.83 for cables
- delta = air density factor = 1 for barometric pressure of 76 cm at 25 degrees C
  = 3.92 * (barometric pressure in cm) / (273 + temperature in C)
- S = distance between surfaces in cm.

r = radius of wire in cm.

In this research we used 18 gage copper wire (r=0.051181 cm) and a separation distance S=0.2 cm. These values yielded a Disruptive Critical Voltage of 3.55kv and a Visual Critical Voltage of 4.83 kv. Experimental results were consistent with these values.

As shown in Figure 2b, a point-to-plane electrode configuration was evaluated for the discharge, since such geometry ensures the smooth passage of the electric field from the ionization region to the drift region, and facilitates ions' movement to the drift region. The discharge takes place inside a sealed tube (inner diameter of 20 mm). The point electrode, which was made of copper wire, was fixed on a Teflon cylinder inside the tube. The measurements were made with the diameter of the point electrode of 0.04 mm.

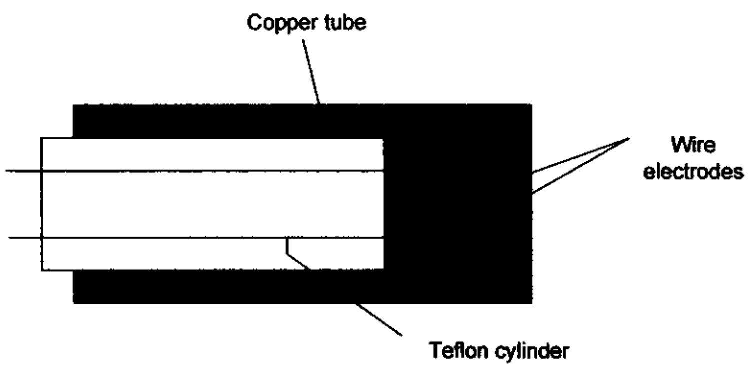



Figure 3a Parallel wire corona generator

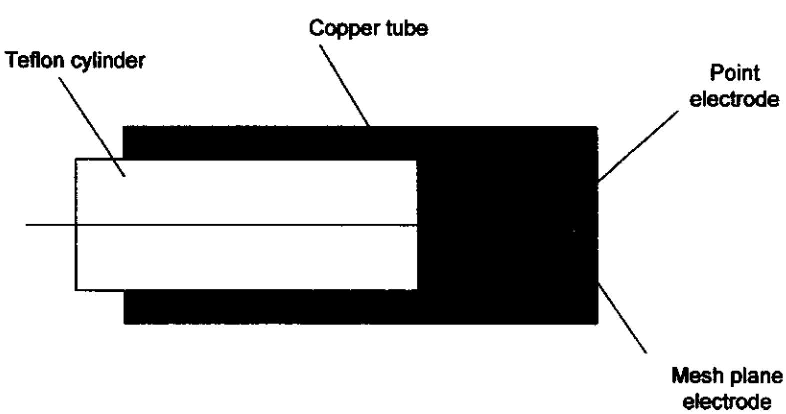



Figure 3b Point and plane corona generator

The high voltage pulse was provided by a pulse generator. The operational principle is to store electrical energy in an electrostatic field, and subsequently create a fast discharge of the stored energy into the load. The schematic of the circuit is shown in Figure 4. A dc power supply (output adjustable from 50 to 150 V) charges the capacitor C1 (100 nF) through a resistor R1 (500 ohms). The high voltage pulse is obtained by discharging C1 into the primary of a pulse transformer (step-up ratio 1:80). A semiconductor switch

IGBT (IRG4BC30U) was used to accomplish the fast discharge. The pulsed current passing through the primary windings will induce a high voltage pulse on the output of the transformer.

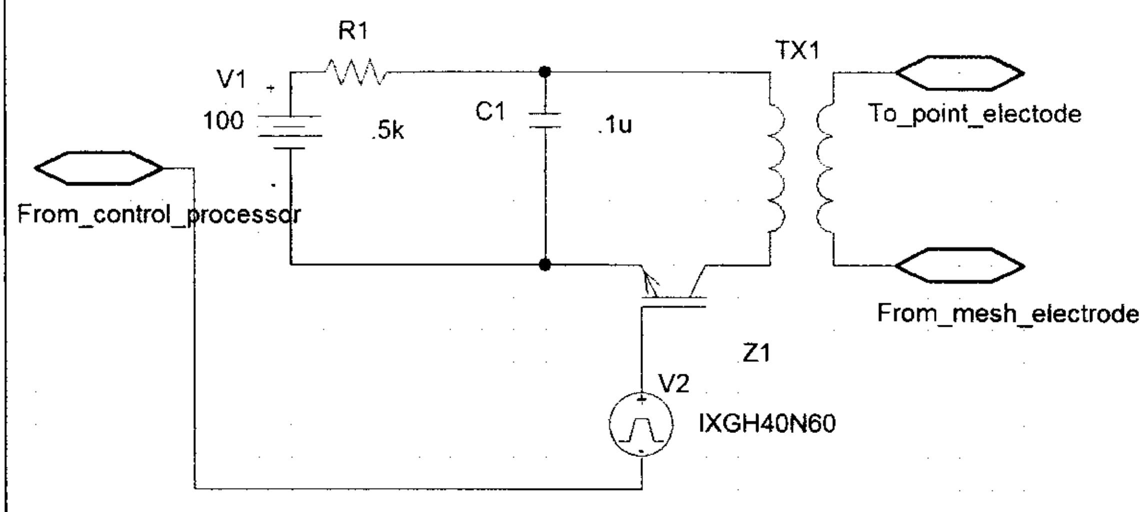



Figure 4 Pulse Generator Schematic

An experiment was carried out in order to investigate the electrical characteristics of the pulsed corona discharge. The experimental setup used the ionization element shown in Figure 1. A round stainless steel mesh 20 mm diameter was used as the plane electrode. This mesh electrode was connected to ground potential by a resistance of 50 ohms. By measuring the drop of voltage across this resistance the discharge current can be obtained. The interelectrode distance d was set from varied by moving the point electrode inside the copper tube. The pulse voltage amplitude was adjusted by changing the dc voltage V1 shown in Figure 3.

Once the pulsed voltage exceeds the Disruptive Critical Voltage, the corona discharge appears. Figure 4 shows the typical wave form of current in a positive discharge. The discharge current during the short pulse is due to the motion of the electrons toward the point electrode. The ions move very slightly in this short time because their motilities are about 100 times smaller than that of electrons. The fast leading edge of the pulse current is due to the electron avalanche at the onset of the discharge. As long as the ionization wave moves towards the mesh electrode, the electronic currents drop due to attachment of electrons with oxygen molecules.

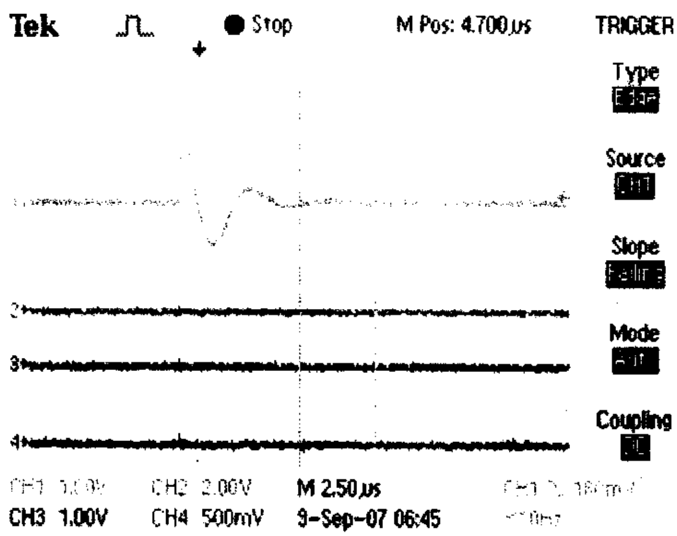
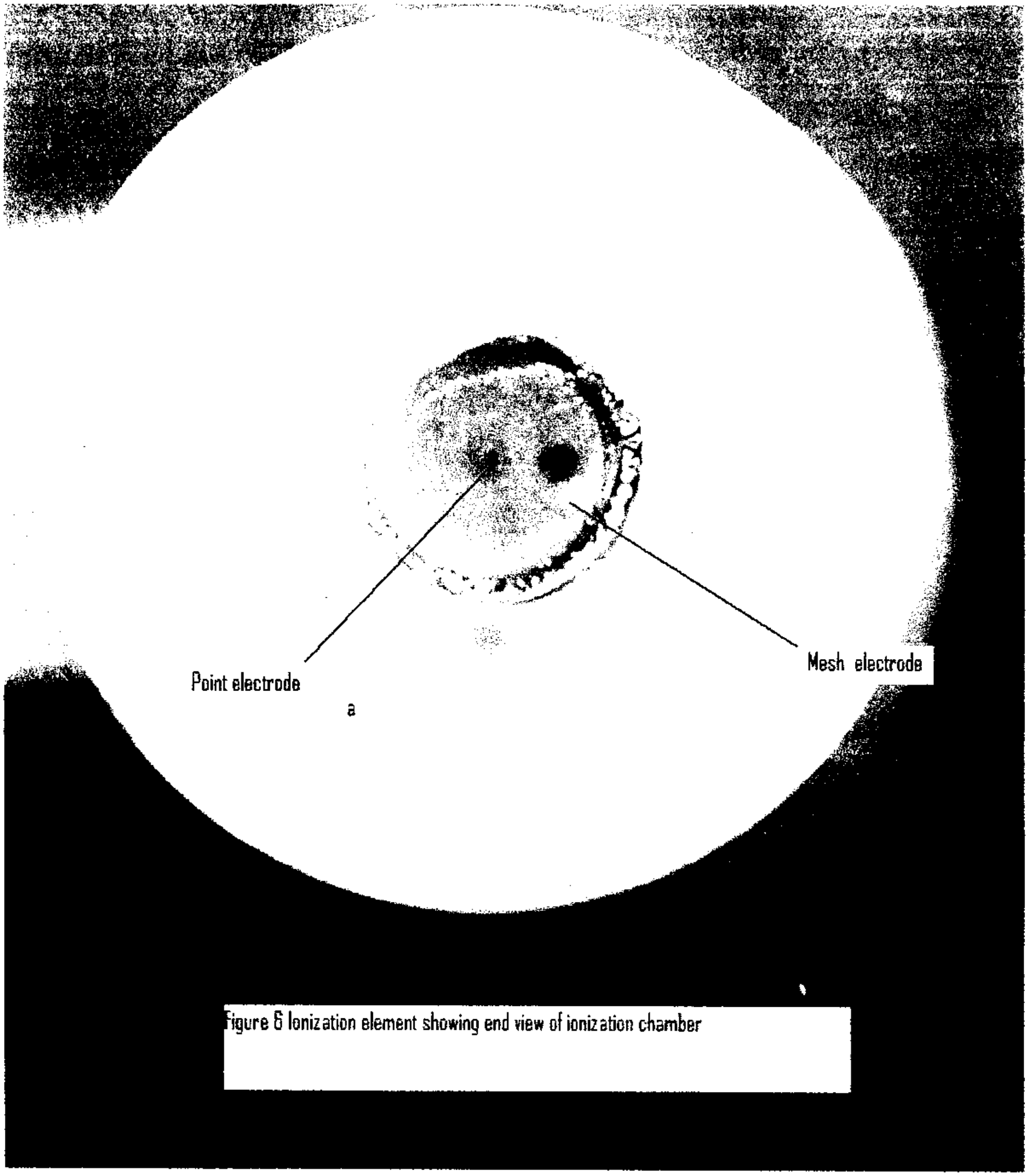



Figure 4 Corona Discharge Current

The positive ions formed by a single pulsed corona discharge will be lost by recombination and diffusion to the wall of the tube. However if an electric field is established axially along the tube outside the ionization region, part of the ions will be extracted from the ionization region. To analyze the behavior of these positive ions, the experimental arrangement shown in Figure 1 was modified so as to place the ion collector 1mm away from the mesh electrode. The stainless steel mesh electrode (30 wire/cm with transparency 64%) which was located at the end of the ionization region to allow positive ions to move out of this region entering into the drift region. The whole ionization region is shielded by a solid copper case inside the Teflon assembly to reduce the effect of the noise generated by the pulse corona discharge on the ion current measurements. It also prevents the discharge field from penetrating into the drift region. The copper case was kept at the same potential as the mesh electrode. The ion collector plate was made of copper (diameter 16 mm) and shielded by a metallic mesh to minimize the noise interference. The electric field between the mesh electrode and the collector plate allows positive ions to be collected. The ion current was amplified by an electrometer with a gain of 10^9 V/A before its input to the oscilloscope. Figure 6 shows an end view of the ionization element. The point electrode is 4 mm behind the mesh electrode.




igure 6 Ionization element showing end view of ionization chamber

Figure 7 shows the temporal distribution of positive ions resulting from the foregoing experiment. Channel 1 shows the amplified ion current converted to a voltage and channel 2 shows the gating pulse for the IGBT that produces the high voltage corona pulse.

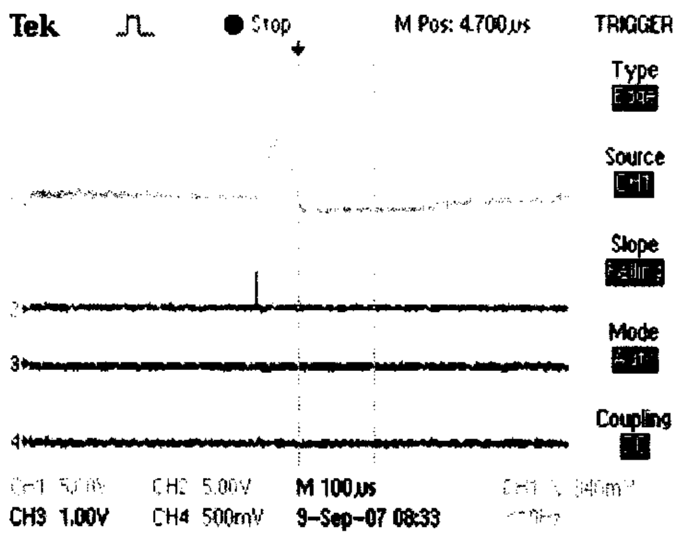



Figure 7 Temporal Distribution of Positive Ions

Figure 8 shows an end view of the ion collector element. The aperture is a copper mesh of the same wire density as the mesh electrode used in the ionization element and is placed 1 mm in front of the ion collector.

The separation element or drift chamber is a glass tube 45 mm in diameter although alternative diameters can be used by simply adjusting the diameter of the Teflon end structure. The drift field electrodes are printed on a plastic filament. An electric field gradient is created by printing a resistor across the electrodes so as to produce the desired gradient (volts/cm). This is achieved by applying a high voltage at the ionization end of the tube and the voltage dropped across each electrode element will produce the desired gradient. Another alternative to use of the printed electrodes is to use a resistive glass tube. The advantage of the resistive glass configuration is that it produces a uniform gradient. However such tubes are more expensive to produce than the printed configuration and the performance advantage may not justify the added cost. Additional work will be done to evaluate this alternative.

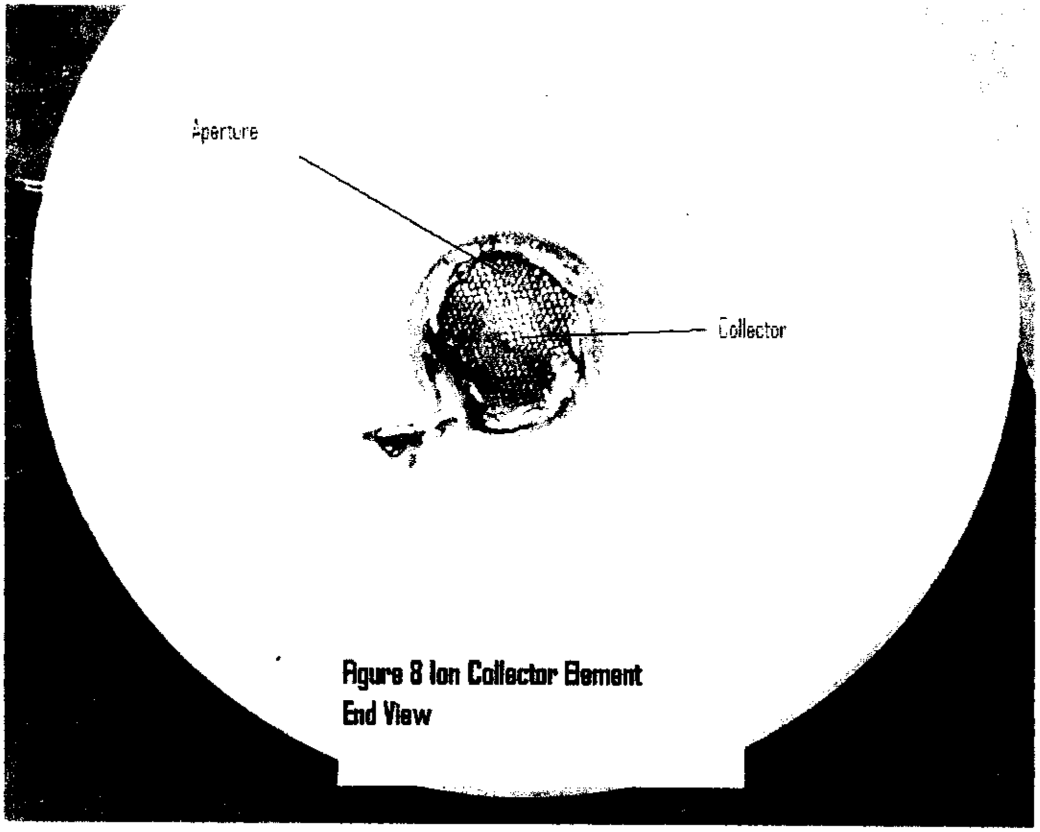



**Figure 8 Ion Collector Element End View**

The foregoing paragraphs describe the work that has been accomplished on the IMS device hardware. The complete prototype system consists of seven elements shown in Figure 9. These elements are:

1. The IMS device (described above).
2. The corona pulse generator (Figure 4)
3. The control processor.
4. The ion signal amplifier.
5. The drift field generator.
6. The analyte membrane filter.
7. The communication module.

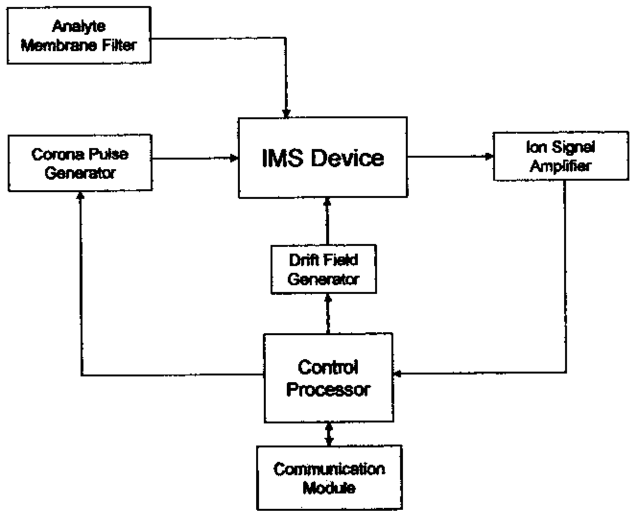


Figure 9 IMS System Diagram

# Project Status

The control processor hardware has been developed and integrated with the corona pulse generator and the ion signal amplifier. The drift field generator has been developed and is now being integrated and tested. The analyte membrane filter has yet to be designed. The communication module has been designed but the operating software is still in the development stage.