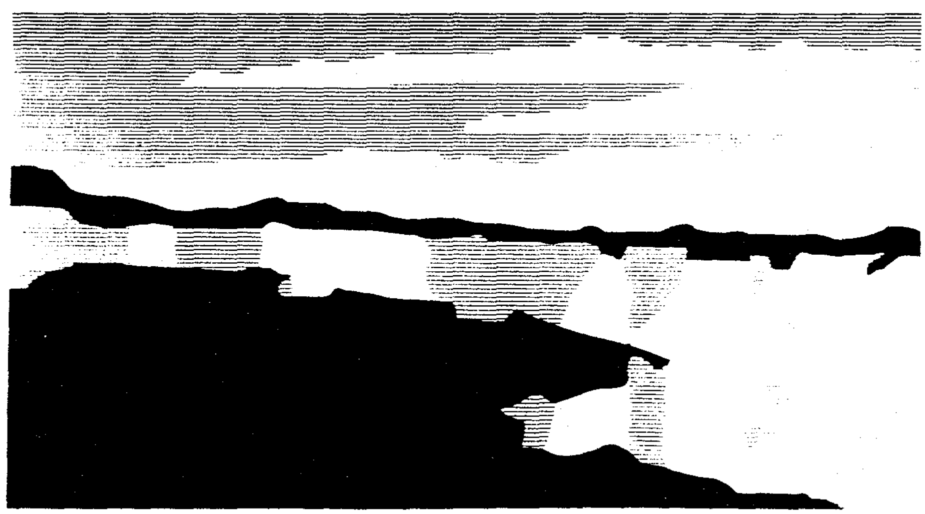
LA-UR- 98-5697

Author(s): Robert E. Steiner, David M. Wayne, and Vahid Majidi

Submitted to: Conference Proceeding for GLOBAL '99, Jackson Hole, WY



Los Alamos
NATIONAL LABORATORY

A Modular Steady State Glow Discharge Quadrupole Mass Spectrometer System for the At-Line Analysis of Plutonium Metal

Robert Steiner and David Wayne, Nuclear Materials Technology Division, NMT-1, Los Alamos National Laboratory, Los Alamos, NM 87545

Vahid Majidi, Chemical Science and Technology Division, CST-9, Los Alamos National Laboratory, Los Alamos, NM 87545

Historically, glow discharge mass and optical spectrometric techniques have been used in industry for the characterization of processed metals, such as steels and other alloys. This technique is especially well suited for this type of product analysis because the glow discharge ionization source accommodates solid conducting samples with minimal or no sample preparation. This characteristic along with minimal matrix effect considerations makes the glow discharge source well suited for these types of applications.

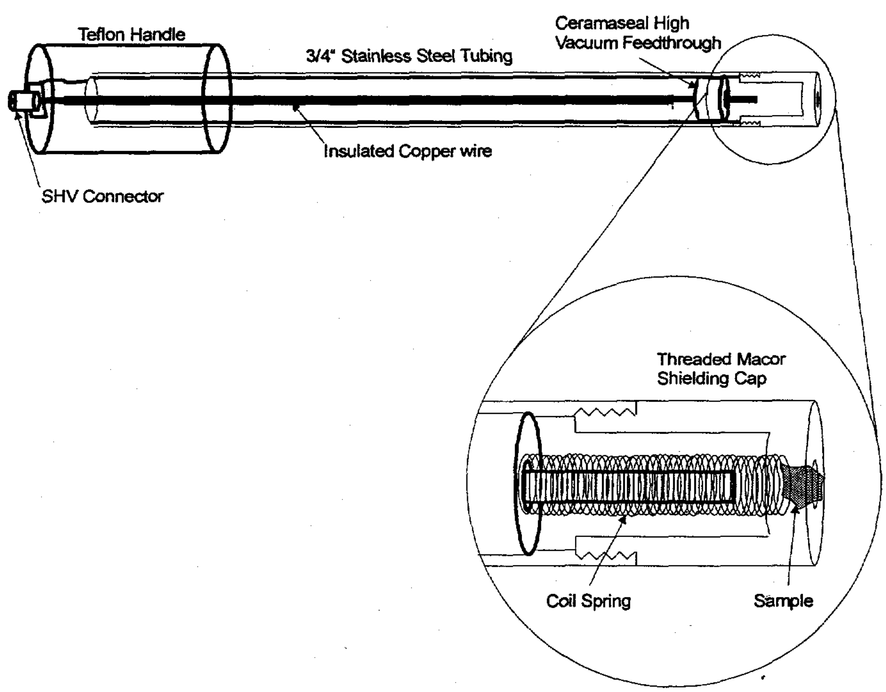


Figure 1: Direct insertion probe for GDMS.

DISCLAIMER

DISCLAIMER

Portions of this document may be illegible in electronic image products. Images are produced from the best available original document.

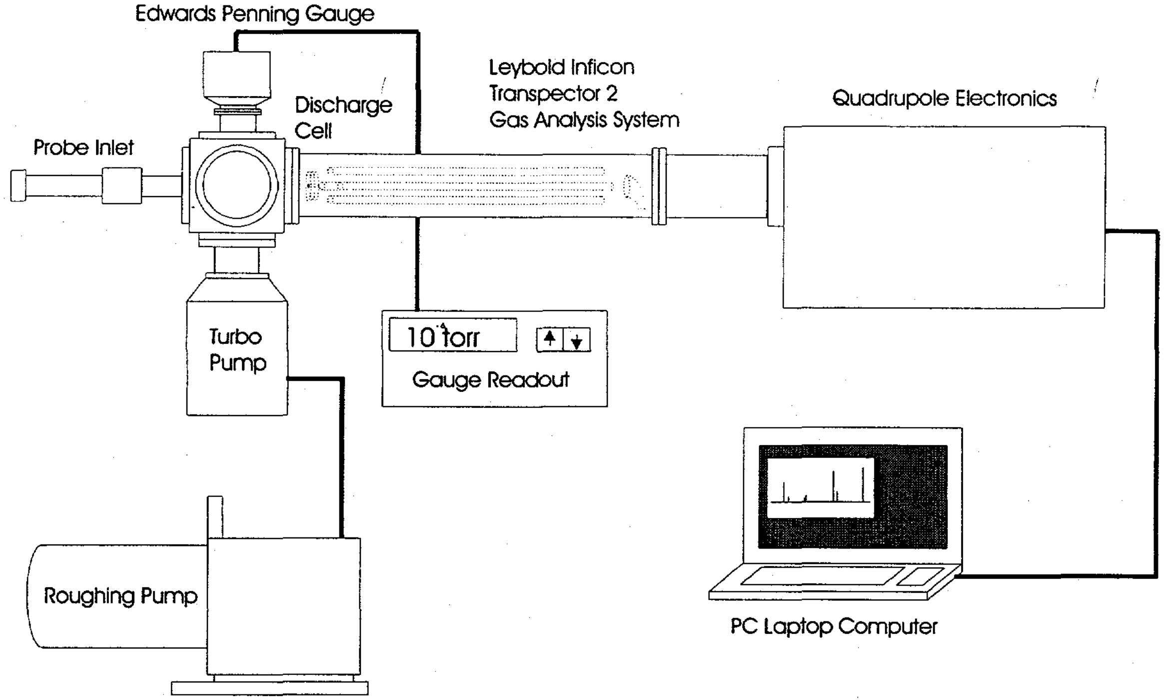


Figure 2: Glow discharge mass spectrometer setup.

The plutonium processing facility at Los Alamos National Laboratory is continuously in search of techniques or hardware that will streamline their process, making processing more reliable and efficient. One such area of interest is online or atline analysis of recently processed plutonium for trace impurities. Unfortunately, this type of application places stringent requirements on both the design and operation of such a system because it must be mounted and operated within a glovebox. One of the major obstacles that must be overcome is the introduction of a non-uniform sample while reproducibly sampling the same surface area with every analysis. To minimize this problem, samples will be fixed to the end of a ¾" diameter direct insertion probe by sandwiching them between a conducting coil spring and a macor orifice with a fixed diameter, Figure 1. This configuration should allow reproducible sampling on irregular samples while keeping the loading and sample introduction processes simple. We are in the process of designing and characterizing a small glow discharge quadrupole based on the Leybold Inficon Transpector 2 Residual Gas Analyzer System, Figure 2. It's small size and relative simplicity make it an ideal system for this type of application.

The glow discharge is a rather simple device to understand. For operation, a potential gradient is established between the anode (discharge cell at ground) and the cathode (the sample fixed to the end of the direct insertion probe at – 2KV). When this gradient is initiated, electrons are accelerated toward the anode. As they accelerate they may interact with the discharge gas (usually Ar) that is introduced into the chamber at approximately 1 torr. Some of these collisions will have sufficient energy to ionize the argon atoms. These argon ions, in turn, are accelerated to the cathode where they impact and sputter sample material from the cathode's surface. The newly formed gas phase anayte atoms then diffuse into the plasma glow region where they may be ionized via a number of different excitation pathways. It is important to note that the atomization and excitation/ionization mechanisms are mutually exclusive processes, minimizing the matrix effects that hinder so many techniques. The analyte ions are then extracted from the plasma and into the quadrupole mass analyzer where there are detected and quantified.