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Instruments

AEC RESEARCH AND DEVELOPMENT REPORT

MASTER

**A PLUG-IN-TYPE MASS SPECTROMETER SURFACE IONIZATION SOURCE
FOR USE WITH A VACUUM LOCK**

W. W. Rice

UNION CARBIDE NUCLEAR COMPANY
DIVISION OF UNION CARBIDE CORPORATION

Operating

- OAK RIDGE GASEOUS DIFFUSION PLANT • OAK RIDGE Y-12 PLANT
- OAK RIDGE NATIONAL LABORATORY • PADUCAH GASEOUS DIFFUSION PLANT

for the Atomic Energy Commission

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Contract W-7405-eng-26
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W. W. Rice

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ABSTRACT

A plug-in-type surface ionization source has been designed and built to be used with a mass spectrometer equipped with a vacuum lock. The sample filament mounts on a male plug which fits into a female receptacle mounted on a case plate.

A firm electrical contact is realized. Sample plugs are changed without loss of vacuum and can be used with any geometrically orientated analyzer tube.

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SUMMARY

A plug-in-type ionization source was designed and built to be used with existing vacuum locks and Nier-type sources of the 6 and 12-inch mass spectrometers. These sources are designed to decrease, or eliminate, the erratic data now received through poor electrical contacts and to decrease the time lost when the present samples are changed. This model is adaptable to both the 5 kv Nier-type sources which are used with the 6-inch, 60-degree sector mass spectrometers and the 10 kv KAPL-type sources which are used with the 12-inch, 90-degree instruments.

In this model the sample filament is mounted on a male-type plug of stainless steel which is inserted into a nichrome, female-type receptacle mounted on the case plate. Electrical contact is made from the case plate receptacle to the plug.

Operational experience has been very satisfactory. Changing sample filaments is convenient and the male plugs can be cleaned without disassembling. Vacuum loss, while introducing a new sample, is held to a minimum and at the same time a positive electrical contact is assured. The source can be used with an analyzer tube which is in either the vertical or horizontal position.

INTRODUCTION

When a surface ionization source is employed for the isotopic mass spectrometric analysis of various elements, accurate current regulation of the sample filament is of prime importance. Loss of vacuum, or the returning of the mass spectrometer analyzer to atmosphere, is necessary when inserting a new filament support since the lead wires are manually fastened in place. In this way the number of samples assayed per unit time is very limited. Consequently, a more rapid procedure was sought; one whereby the vacuum loss would be minimized and at the same time still have assurance of good electrical contact.

The basic design for this ionization source was taken from one designed by Sladky.⁽¹⁾ In Sladky's design the electrical contacts were made through the wedge-shaped sides of the case plate receptacle to the filament support. This design has proved to be very useful in routine mass spectrometer work but can only be used when the analyzer tube of the mass spectrometer is in a vertical position since the electrical contacts are made between the sloping sides of the plug and the case plate receptacle. The electrical contact is entirely dependent upon the gravitational pull of the plug to the receptacle, a method which very frequently proved to be too poor.

Numerous other reports of methods to facilitate filament changing have been made. Blewett⁽²⁾ employed a ground-glass joint through which filament leads were sealed. He still had to return the analyzer to atmosphere between samples. Stevens⁽³⁾ used a probe which was pushed sideways through two vacuum locks before entering the source region.

Bentley⁽⁴⁾ used a valve to isolate the source region from the rest of the analyzer when replacing the thermal filament.

These types of surface ionization sources, although unique, could not be adapted to equipment in this laboratory without elaborate machining, a costly fabrication operation. So, a source was designed which could be used with existing vacuum locks.^(5, 6)

DESCRIPTION OF THE IONIZATION SOURCE

DESIGN CONSIDERATIONS

The following factors were considered in designing a surface ionization source to be used with the existing vacuum locks:

1. Filament support plug should be designed in such a way that no difficulty would be encountered in loading and removing the ionization source from the receptacle.
2. Designed in such a way that the filament would be protected during loading.
3. Electrical contacts must be of the friction type which would position and hold the filament in alignment regardless of analyzer orientation.
4. Maintain assembly which would make for ease of assembly and cleaning.

The complete source assembly is shown in Figure 1.

MECHANICAL FEATURES

Plug Subassembly

The filament support plug assembly, as shown in Figure 1, is fabricated from stainless steel and consists of two parts, A and B, which are electrically insulated from each other by a 1/16-inch-thick piece of Alsimag, C. A 0.093-inch-diameter by 0.875-inch-long male stud, D, is screwed into each of the two parts which have been drilled and tapped. The filament stud rods, E, are 0.500-inch long and threaded on one end. These parts are also fitted to each of the two components. Both assembled parts and their Alsimag insulator, are then joined to each other by two stud rods, F.

The top insulator, G, is positioned and the complete assembly aligned and secured by means of 0.080-inch hex nuts. A threaded hole in the top of the plug fits the extractor rod of the vacuum lock.

Case Plate Receptacle

The case plate receptacle also consists of two individual units, H and I, which are fabricated from nichrome V. Into each unit the female attachment, J, is made. The individual units are then attached to the case plate, K, by means of four stud rods, L, which are insulated with Alsimag sleeving. Also, as shown in Figure 1, each side of the case plate receptacle is insulated from the case plate by 1/32-inch-thick pieces of mica.

Figure 1. SURFACE IONIZATION SOURCE ASSEMBLY.

Case Plate

The case plate, K, is the same, in general, as the case plate for the ordinary surface ionization source. The one shown is used on the Nier-type source. Four additional holes were drilled and tapped for the stud rods, L, for mounting the case plate receptacle. A 1/4-inch-square opening at each end of the slit provides an air gap for electrical insulation around the ends of the male stud when the filament support is in the receptacle.

In fabrication, all metal parts of the complete assembly are given a four microinch finish.

ELECTRICAL FEATURES

Electrical contact is made through the male stud of the filament support to the case plate receptacle. Until very recently the filament voltage was supplied by a filament transformer. A variac on the primary side of the filament transformer was used to adjust the output. The center tap of the secondary was connected to the case voltage of the source. A DC regulated supply is now being used. The output of the DC supply is controlled by a variac and a vernier control in the AC circuit, and also by a regulation control in the DC circuit. This output is center tapped to the filament. Both sides of the case plate receptacle are insulated from the case plate.

OPERATING EXPERIENCE

PROCEDURE

When the filament support assembly is ready for loading into the instrument, it is threaded tightly onto an extractor rod. Note is made of a mark on the knob of the extractor rod with respect to the filament support. This orientation will assure an accurate mating of the two within the instrument. After passing through the vacuum lock, the plug is pushed down into the case plate receptacle. Finally, the extractor rod is removed, returned to the vacuum lock, and the trap door closed. The reverse is practiced in removing the sample.

RESULTS

This plug-in-type surface ionization source has been used for about two years in the Special Samples Mass Spectrometer Laboratory. It has been found that through the use of this design, sample time and vacuum loss have been reduced to a minimum, positive electrical contact is assured, and a steadier and more controllable ion current is realized.

Also, this source is now being used successfully on a 12-inch, 90-degree mass spectrometer which is positioned in a horizontal plane. This orientation of the analyzer tube rendered earlier designs inoperable. Figure 2 shows the ionization source assembly for the 12-inch, 90-degree instrument; Figure 3 shows the assembly for the 6-inch, 60-degree mass spectrometer.

Figure 4 is a view of a 6-inch, 60-degree sector, open-frame, mass spectrometer showing the position of the plug-in-type ionization source. Figure 5 is an overall view of a 12-inch, 90-degree instrument. The instrument on the right has a "ferris wheel", multiple filament loading device. Both instruments are mounted on hydrostatic bearing plates for accurate leveling and vibration-free operation.

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Figure 2. PLUG-IN-TYPE IONIZATION SOURCE. (Used With 12-Inch, 90-Degree Mass Spectrometer)

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Figure 3. PLUG-IN-TYPE SURFACE IONIZATION SOURCE. (Used With 6-Inch, 60-Degree Sector Mass Spectrometer)

Figure 4. SIX-INCH, SIXTY-DEGREE MASS SPECTROMETER.

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Figure 5. TWELVE-INCH, NINETY-DEGREE MASS SPECTROMETER.

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