



1. AGENCY USE ONLY (Leave Blank) 2. REPORT DATE 5/27/94 3. REPORT TYPE AND DATES COVERED Technical Rept. 7/93-5/94

4. TITLE AND SUBTITLE High Magnetic Field Glow Discharge Ionization Source
5. FUNDING NUMBERS N0014-87-J-1248

6. AUTHOR(S) K.L. Goodner, C. Dejsupa, C.M. Barshick and J.R. Eyler

7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES) Department of Chemistry
University of Florida
P.O. Box 117200
Gainesville, FL 32611
8. PERFORMING ORGANIZATION REPORT NUMBER 45

9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES) Office of Naval Research
Chemistry Program
800 N. Quincy St.
Arlington, VA 22217-5660
10. SPONSORING/MONITORING AGENCY REPORT NUMBER

DTIC QUALITY INSPECTED 2

DTIC ELECTE JUN 06 1994 S G D

11. SUPPLEMENTARY NOTES To be submitted to the Journal of the American Society for Mass Spectrometry

12a. DISTRIBUTION/AVAILABILITY STATEMENT 12b. DISTRIBUTION CODE

Reproduction in whole or in part is permitted for any purpose of the United States Government. This document has been approved for public release and sale; its distribution is unlimited.

13. ABSTRACT (Maximum 200 words)

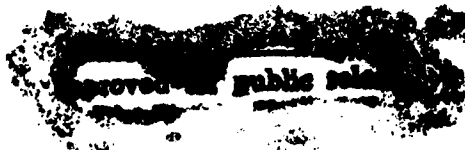
A glow discharge ionization source has been developed which operates inside the high magnetic fields necessary for a Fourier transform mass spectrometer. Diagrams of the source and a sample spectrum are shown.

14. SUBJECT TERMS 15. NUMBER OF PAGES 3

Elemental Mass Spectrometry, Glow Discharge Ionization, High Magnetic Fields

16. PRICE CODE

17. SECURITY CLASSIFICATION OF REPORT	18. SECURITY CLASSIFICATION OF THIS PAGE	19. SECURITY CLASSIFICATION OF ABSTRACT	20. LIMITATION OF ABSTRACT
Unclassified	Unclassified	Unclassified	Unlimited



94 6 3 102

SPO 94-16657



OFFICE OF NAVAL RESEARCH

GRANT # N0014-87-J-1248

R&T Code 4134052

Dr. John Pazik

Technical Report No. 45

High Magnetic Field Glow Discharge Ionization Source

by

K.L. Goodner, C. Dejsupa, C.M. Barshick and J.R. Eyer

To be submitted

to the

Journal of the American Society for Mass Spectrometry

University of Florida
Department of Chemistry
Gainesville, FL

May 27, 1994

Accession For	
NTIS	CRA&I <input checked="" type="checkbox"/>
DTIC	TAB <input type="checkbox"/>
Unannounced <input type="checkbox"/>	
Justification	
By	
Distribution /	
Availability Codes	
Dist	Avail and/or Special
A-1	

Reproduction in whole or in part is permitted for any purpose of the United States Government.

This document has been approved for public release and sale; its distribution is unlimited.

High Magnetic Field Glow Discharge Ionization Source

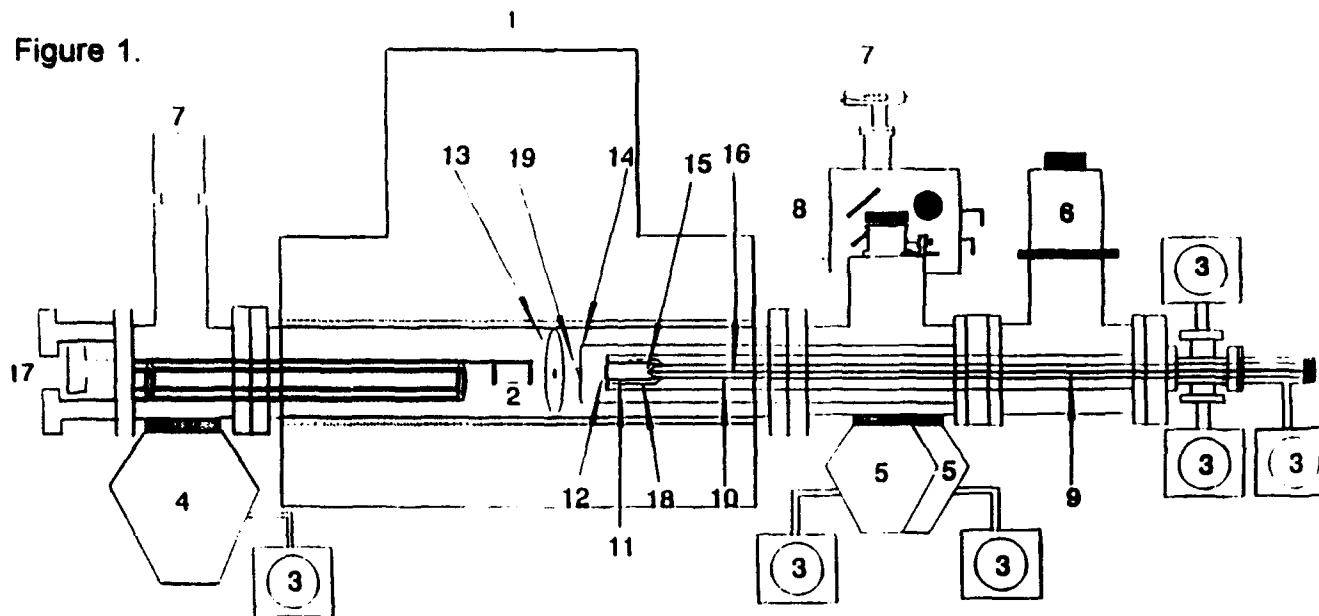
Kevin L. Goodner, Chadin Dejsupa, Christopher M. Barshick, and John R. Eyler
Department of Chemistry, University of Florida, Gainesville, FL 32611-7200

We have modified our "internal" electrospray source, which operates inside the high magnetic field associated with Fourier transform ion cyclotron resonance (fticr) mass spectrometry, to allow it to serve as a glow discharge (GD) ionization source. Figure 1 shows the overall experimental apparatus for this system, and Figure 2 is an expanded view of the modifications which make glow discharge possible.

Quite reasonable ion signals are obtained with this source, in spite of the fact that the glow discharge ionization is taking place at a magnetic field of about 1.8 tesla. This corresponds to a field at least a factor of 25 higher than has ever been used with glow discharge sources before. One example of an analysis of the $^{60}\text{Ni}^+$ signal in a NIST 661 stainless steel signal is shown in Figure 3. Signal-to-noise calculations for the top trace yield a limit of detection of 38 ppm for the peak at m/z 60. The predominant ion is $^{56}\text{Fe}^+$, arising from the high concentration of iron in the steel. In the middle trace, the main isotope of iron at m/z 56 has been ejected from the icr cell, leading to an improved LOD of 28 ppm for the $^{60}\text{Ni}^+$. In the bottom trace, quadrupolar axialization has been applied to the m/z 60 peak, causing it to be retained in the cell while other ions are (partially) lost due to collisions with a buffer gas. The $^{60}\text{Ni}^+$ LOD is lowered to 6 ppm by this approach.

Preliminary experiments also indicate that the sputter rates for samples in the high magnetic field GD source are a factor of almost 10 lower than those in conventional "external" sources, which do not operate at high magnetic fields. This is promising for the analysis of materials where a large amount of sample cannot be sacrificed, and for analysis of thin films or layered materials.

Figure 1.



The high magnetic field GD/FTICR. (1) superconducting magnet (2T), (2) cell, (3) mechanical pumps, (4) 700 L/s diffusion pump, (5) 300 L/s diffusion pumps, (6) 2900 L/s cryo pump, (7) ion gauge, (8) gas/liquid inlet, (9) high voltage lead, (10) 3/4" stainless steel tubing, (11) Stainless steel mesh, (12) 1 mm orifice, (13) 2nd conductance limit, (14) 1st conductance limit, (15) sample cathode, (16) Ar gas in, (17) laser windows, (18) Delrin insulator and (19) shutter.

Figure 2.

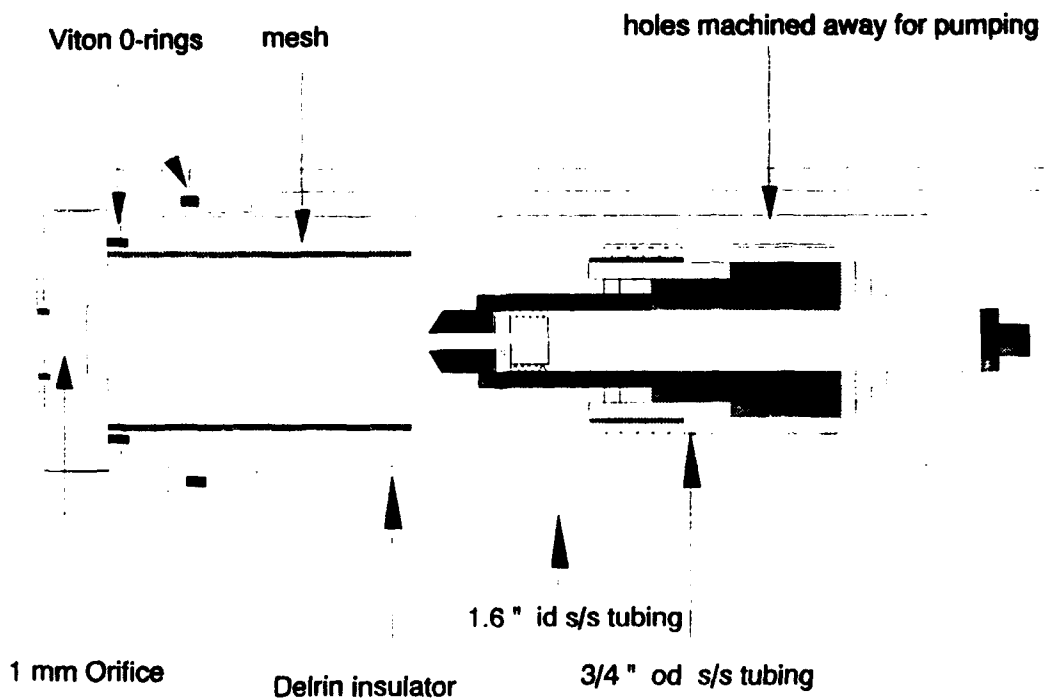


Figure 3.

