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POSTER

RF-GD/FTMS: AN FTMS SYSTEM WITH A LENSLESS EXTERNAL ION SOURCE FOR HIGH RESOLUTION ELEMENTAL MASS SPECTROMETRY (U)

by

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A poster proposed for presentation at
The 40th ASMS Conference on Mass Spectrometry and Allied Topics
Washington, DC
May 31 - June 5, 1992

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R.R. Weller.
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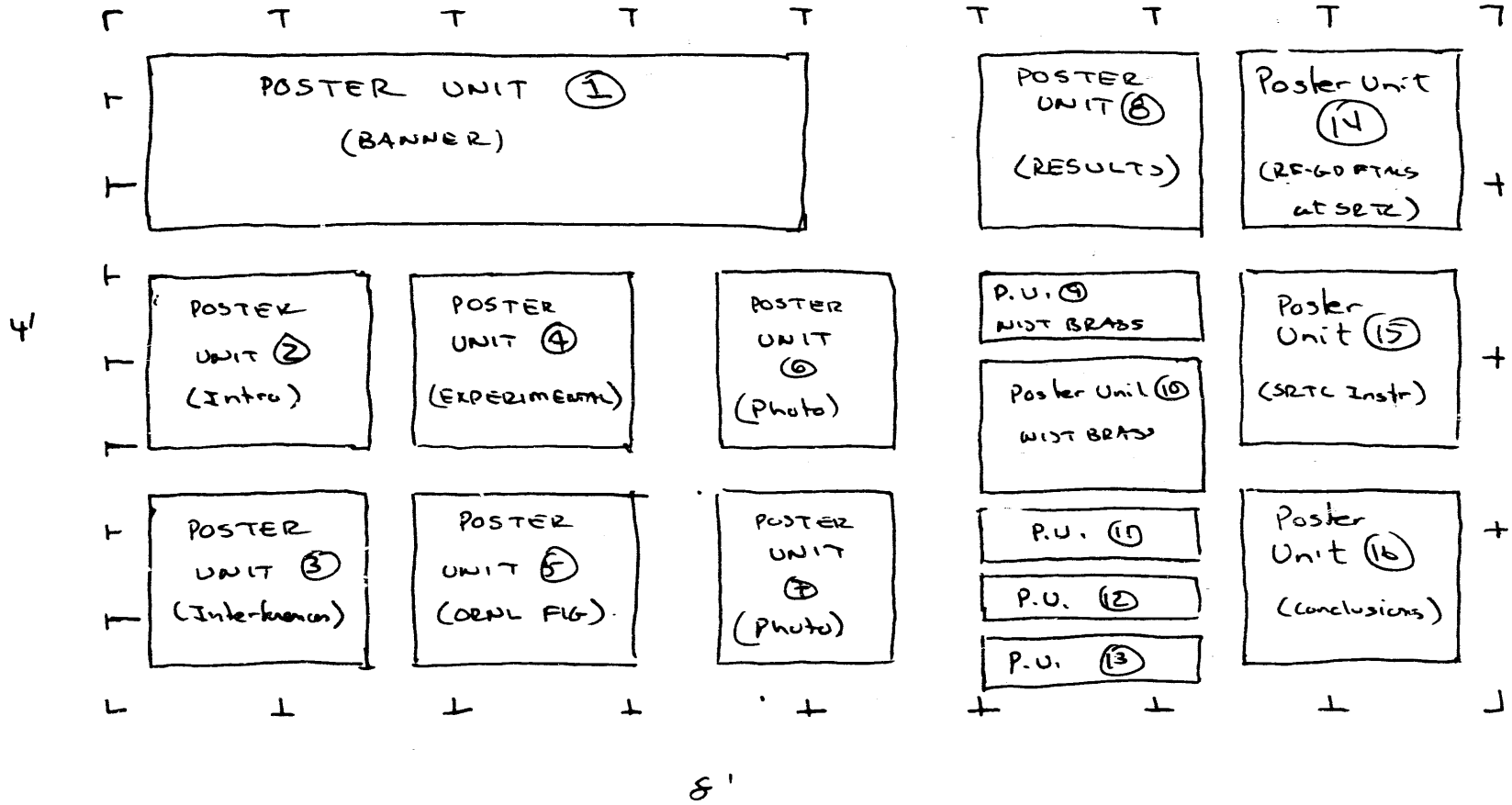

Poster Unit: (1)

RF-GD/FTMS: An FTMS System with a Lensless External Ion Source for High Resolution Elemental Mass Spectrometry

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INTRODUCTION:

Two of the major challenges which face the field of elemental mass spectrometry are the development of versatile atomization/ionization sources for direct solids analysis and methods for reducing the impact of isobaric interferences found on commonly employed quadrupole and sector based instruments. The combination of radio frequency glow discharge (RF-GD) sources with Fourier transform ion cyclotron resonance mass spectrometry (FT-ICR or FTMS) is one technique which addresses both problems.

The need to develop a high resolution elemental mass spectrometer has led to a set of demonstration experiments done in collaboration between Savannah River Technolgy Center (formerly Savannah River Laboratory), Clemson University, and Oak Ridge National Laboratory. The success of these experiments are discussed below along with the resulting design improvements being incorporated into the SRTC instrument currently under construction.

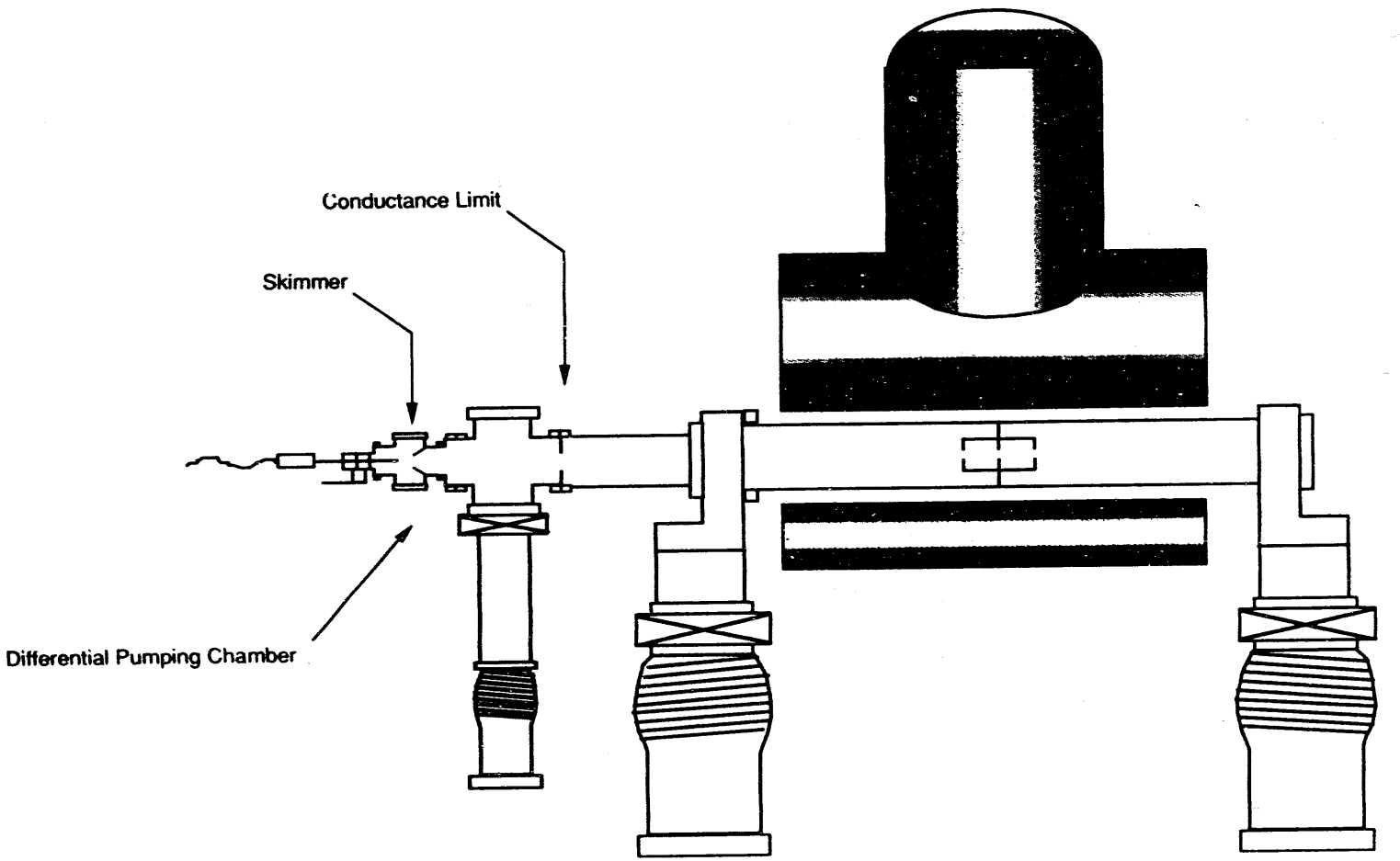
Typical Resolving Powers Necessary in Elemental Mass Spectrometry

Analyte	Interference/ Co-analyte	Required Resolving Power	Analyte	Interference/ Co-analyte	Required Resolving Power
$^{112}Cd^+$	$^{56}Fe_2^+$	3,400	$^{94}Zr^+$	$^{54}Fe^{40}Ar^+$	22,732
$^{108}Cd^+$	$^{54}Fe_2^+$	4,354	$^{88}Sr^+$	$^{52}Cr^{36}Ar^+$	35,889
$^{104}Pd^+$	$^{52}Cr_2^+$	4,351	$^{92}Zr^+$	$^{52}Cr^{40}Ar^+$	54,377
$^{116}Sn^+$	$^{58}Ni_2^+$	3,692	$^{107}Pd^+$	$^{107}Ag^+$	76,000
$^{120}Sn^+$	$^{60}Ni_2^+$	2,960	$^{238}Pu^+$	$^{238}U^+$	180,000
$^{72}Ge^+$	$^{56}Fe^{16}O^+$	8,881	$^{99}Tc^+$	$^{99}Ru^+$	315,000
$^{74}Se^+$	$^{58}Ni^{16}O^+$	9,478	$^{135}Cs^+$	$^{135}Ba^+$	613,000
$^{71}Ga^+$	$^{55}Mn^{16}O^+$	8,734	$^{129}I^+$	$^{29}Xe^+$	625,000
$^{150}Sm^+$	$^{134}Ba^{16}O^+$	8,434			
$^{167}Er^+$	$^{151}Eu^{16}O^+$	9,540			
$^{96}Mo^+$	$^{56}Fe^{40}Ar^+$	13,270			
$^{95}Mo^+$	$^{55}Mn^{40}Ar^+$	17,985			

EXPERIMENTAL:

The RF-GD/FTMS experiments were carried out on a commercial Nicolet FTMS-2000 (EXTREL FTMS, Madison, WI) instrument at ORNL. An external ion source and differential pumping system was designed and built by SRTC utilizing an RF-GD source from Clemson University. The ion transfer system utilized the lensless method developed by Weller, et al (ASMS 1988) for laser desorbed ions. A schematic of the system is illustrated in the Figure. The differential pumping chamber was pumped by a 1200 l/s diffusion pump located (due to space considerations) at the end of about a 10 inch long, 4 inch in diameter conductance. The RG-GD source was separated from the differential pumping chamber by a 0.01 inch conductance. The external source was separated from the main FTMS vacuum system by a 0.375 inch conductance. Pressures during experiments were approximately 140 mtorr in the RF-GD source, 10^{-5} torr in the differential pumping chamber, and low 10^{-7} torr in the FTMS analyzer cell. No special trapping schemes were used, and ions were accumulated for 0.5 to 2 seconds. In later experiments, a potential could be applied to the RF-GD skimmer cone to block the ions from the cell during the non-accumlation events. Standard broadband CHIRP and SWIFT excitations were used. Moderate resolution spectra (< 250,000) were obtained by narrowing the bandwidth and ejecting the major (Ar) ions from the analyzer cell.

RF-GD/FTMS External Source on ORNL Instrument



FTMS - 2000

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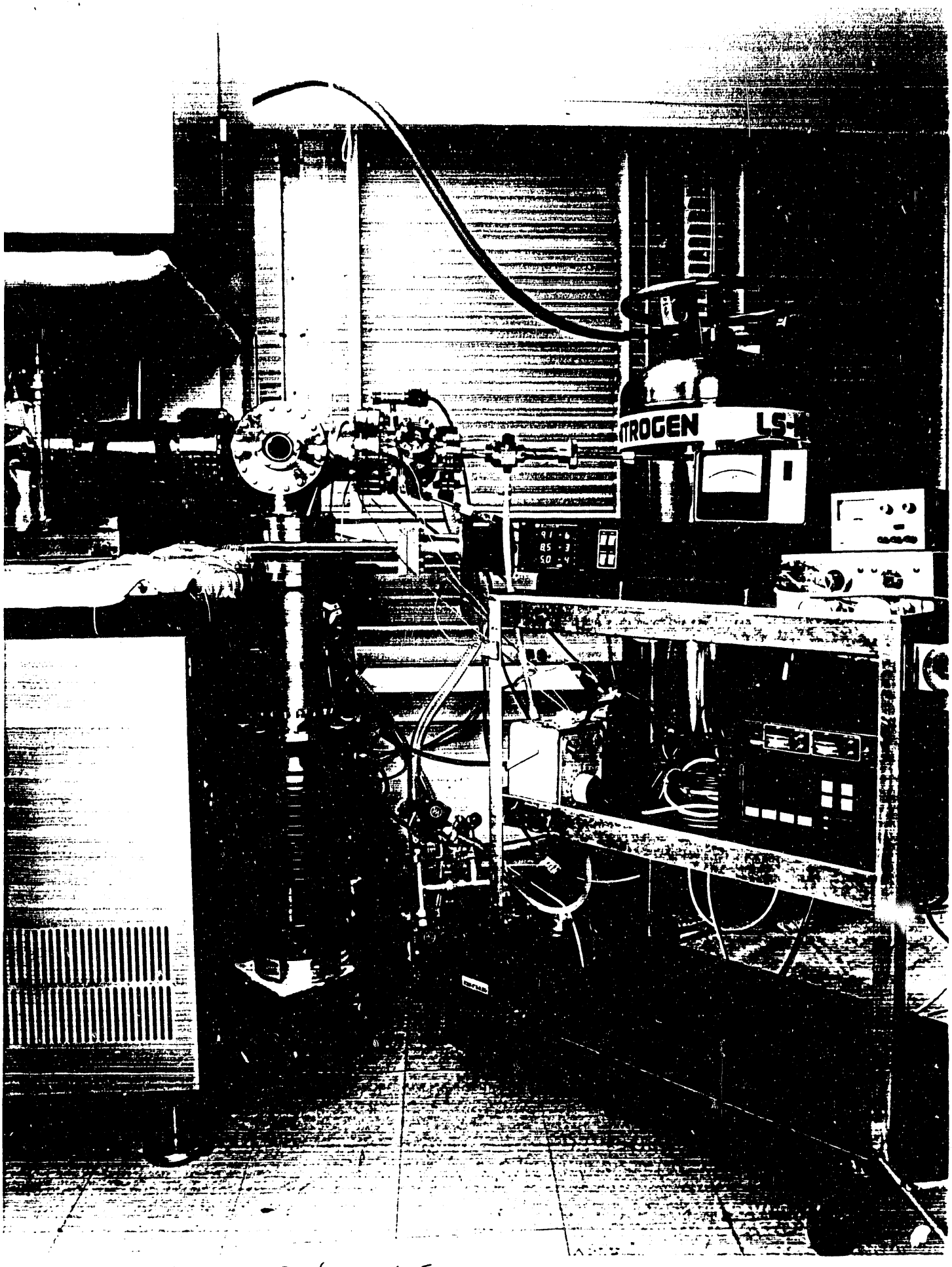
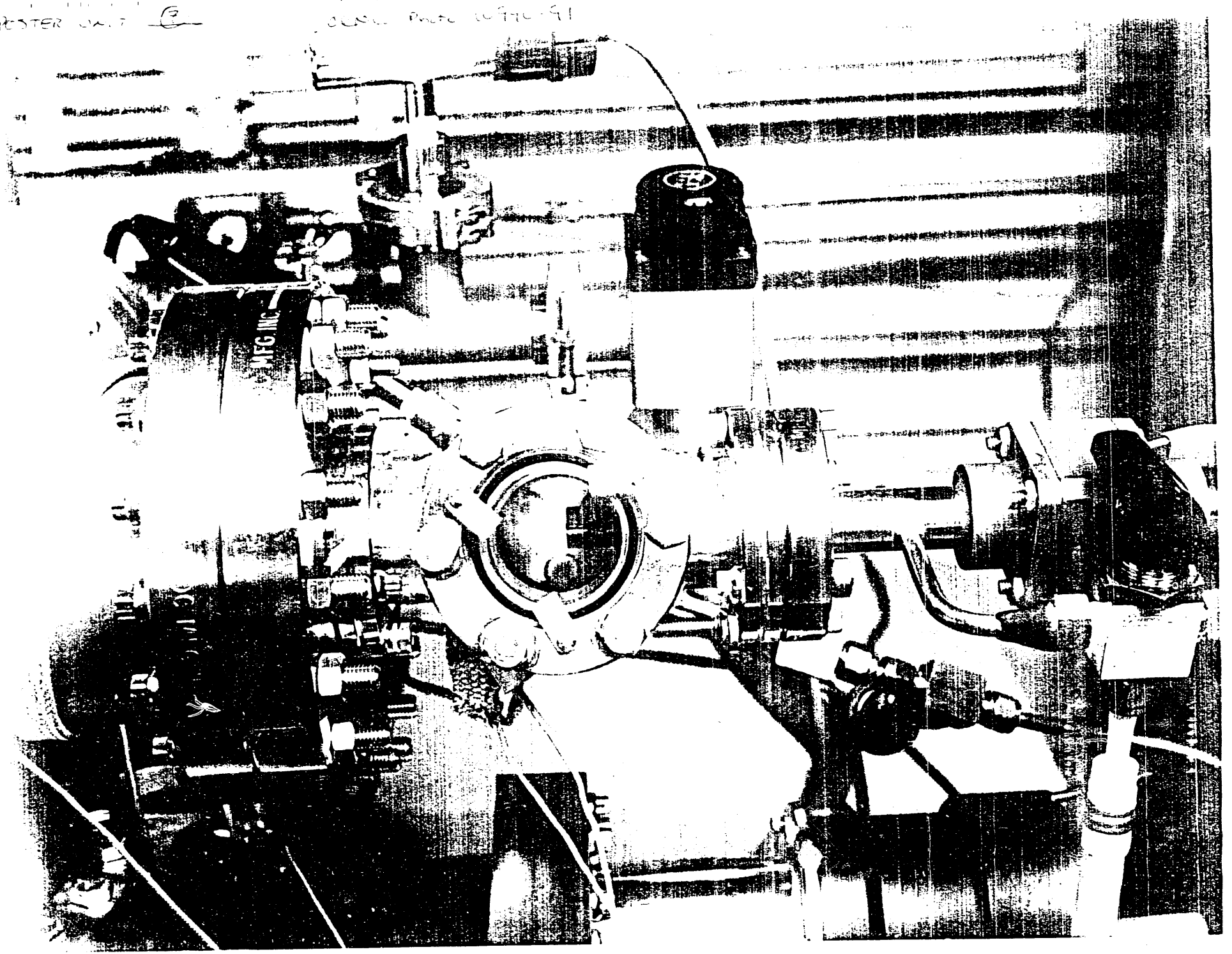
ORNL Photo 10491-91



RF-GD/FTMS External Source



RF-GD Source.

RESULTS:

Spectra have been obtained for NIST 1103 Brass and NIST 1263 Steel and are shown below. All of the expected ions are present, eg. Ar+, ArH+. Argides of the metals are absent. Trace components down to approximately 1% can be observed. Even in the relative high pressure (10^{-7} torr range) in the ICR analyzer cell, relatively high resolution spectra could be obtained, with a resolving power of 74,959 obtained for the steel sample.

The lensless ion transfer system is relatively efficient for transferring the ions from the external source to the ICR analyzer cell. Ion currents measured on the DIP probe were on the order of 100's of nanoamps and are comparable to those observed from this type source when mounted on quadrupole instruments. Beam size is relatively small, only a few millimeters in diameter at the conductance limit between the differential pumping chamber and the main FTMS vacuum system. However, the main problem with this type of external source is the trapping of ions in the ICR cell. Ion accumulation times of 1 to 2 seconds give excellent results, but larger signals are observed up to 6 to 8 seconds. In these experiments, a trapping potential of 6 volts was used, with the conductance limit at 10 volts (maximum with the commercial electronics used) during the ion accumulation event. A simple deceleration grid may be all that is needed to greatly increase the ion trapping efficiency and is planned for future experiments.

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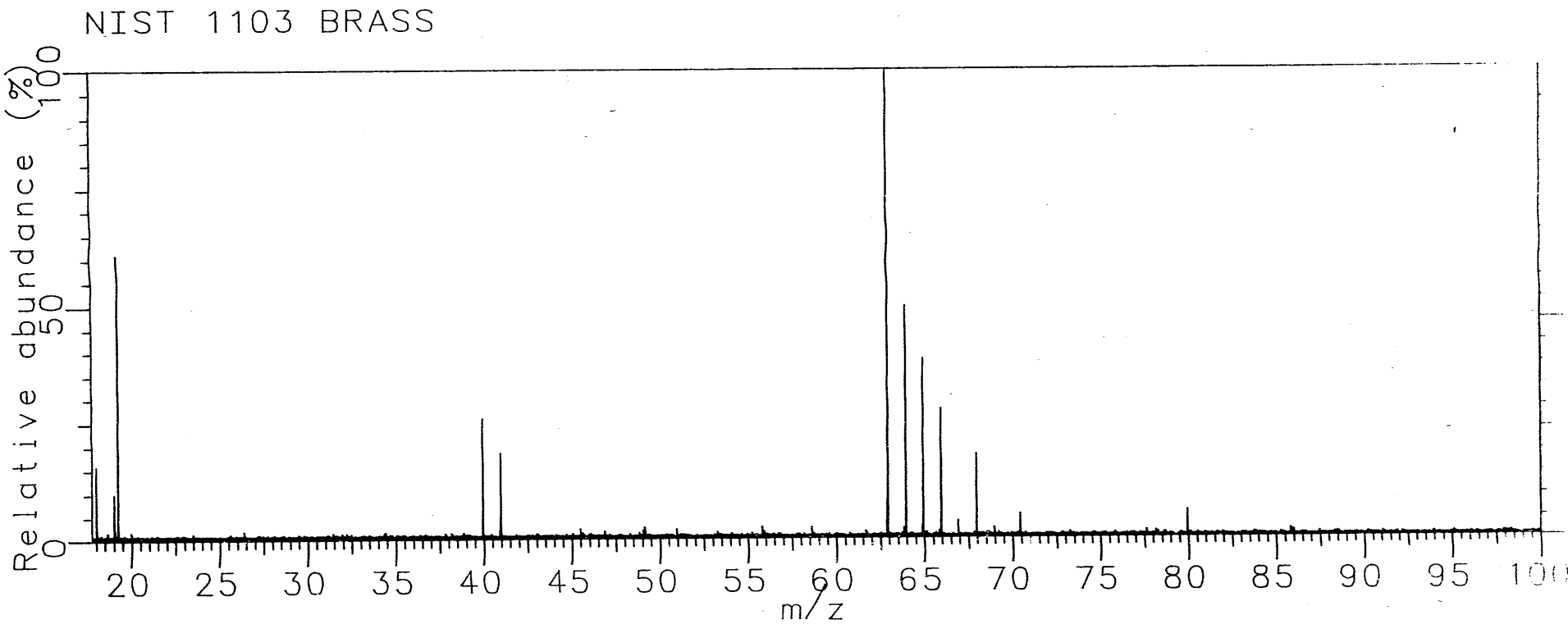

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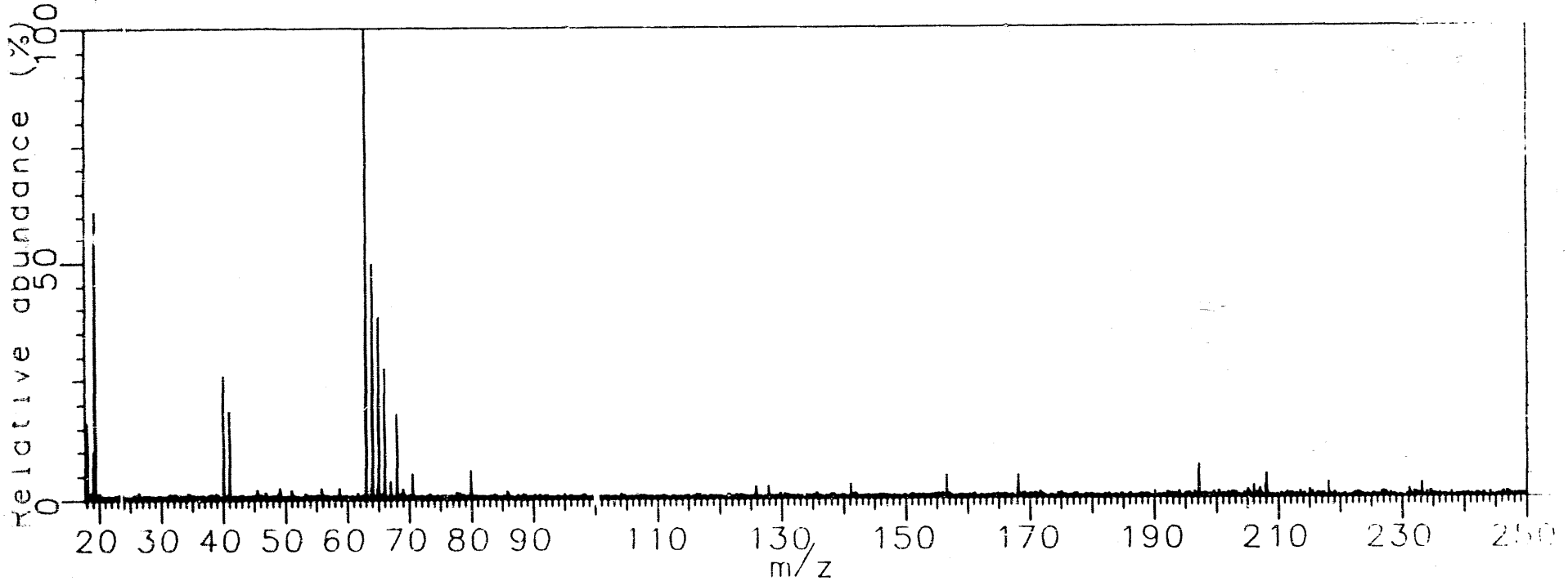
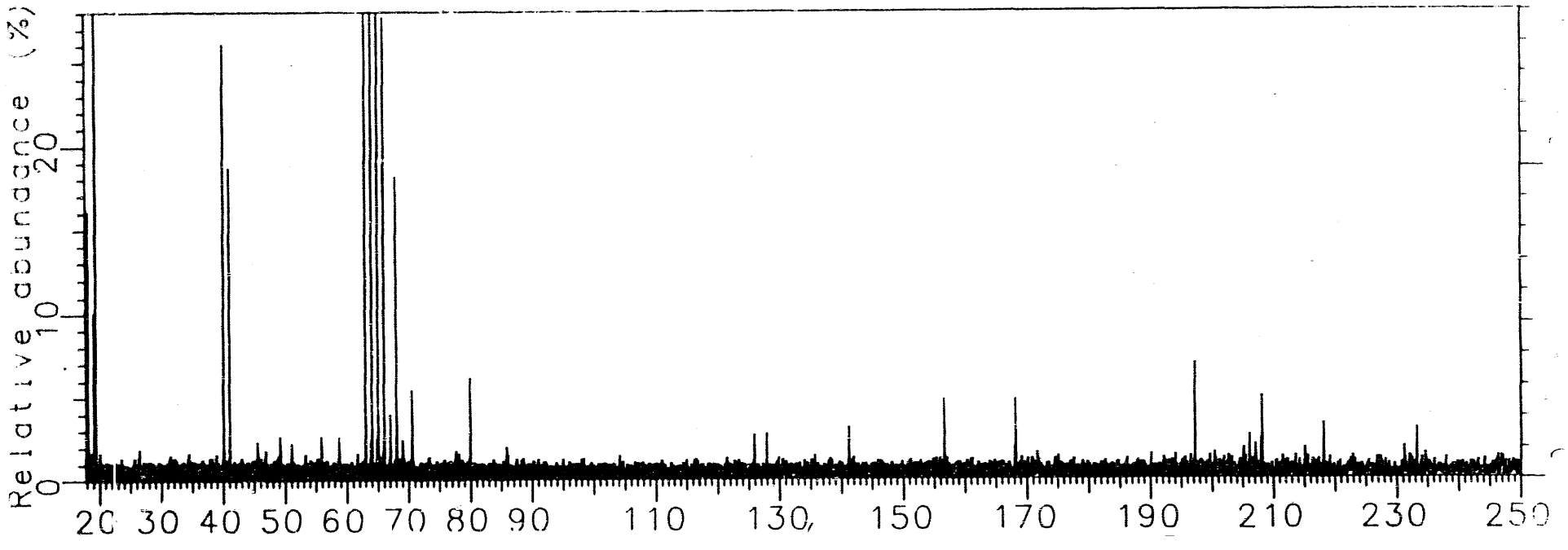
NIST 1103 BRASS



NIST 1103 BRASS



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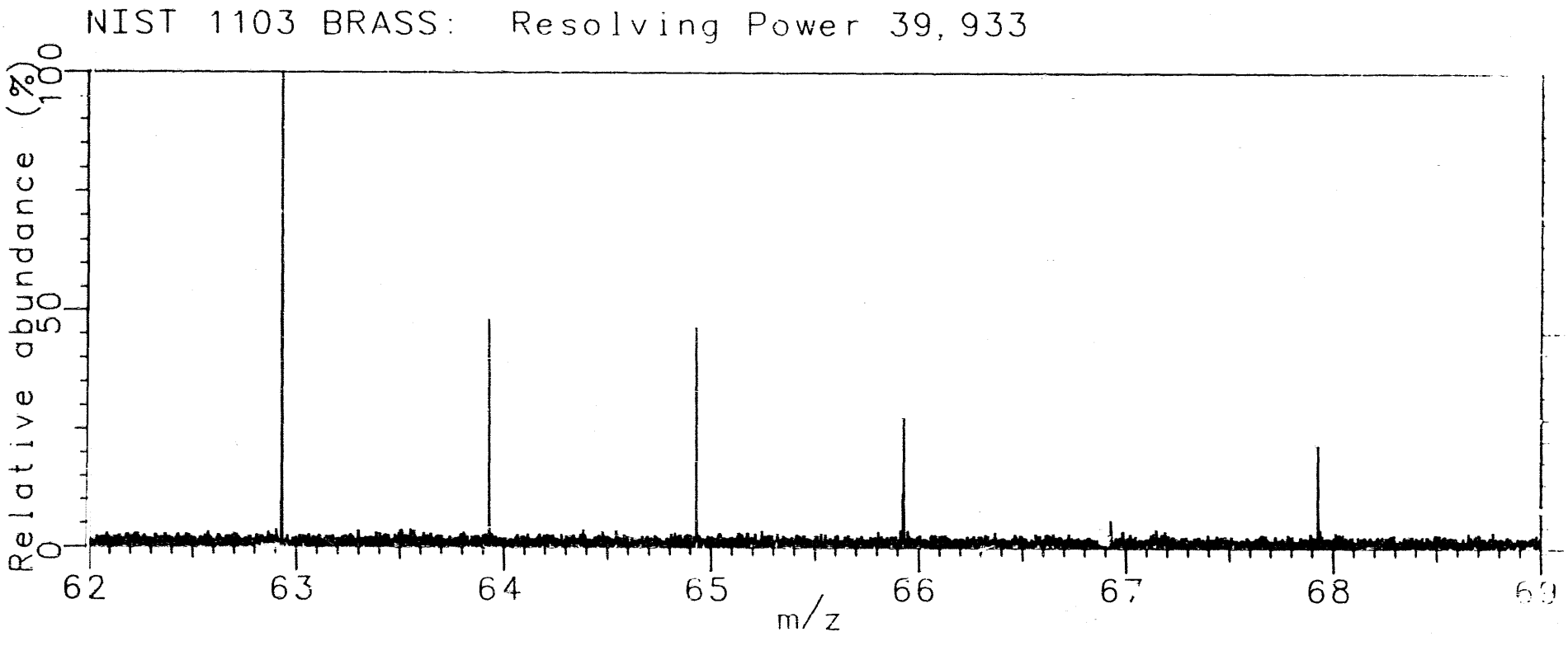

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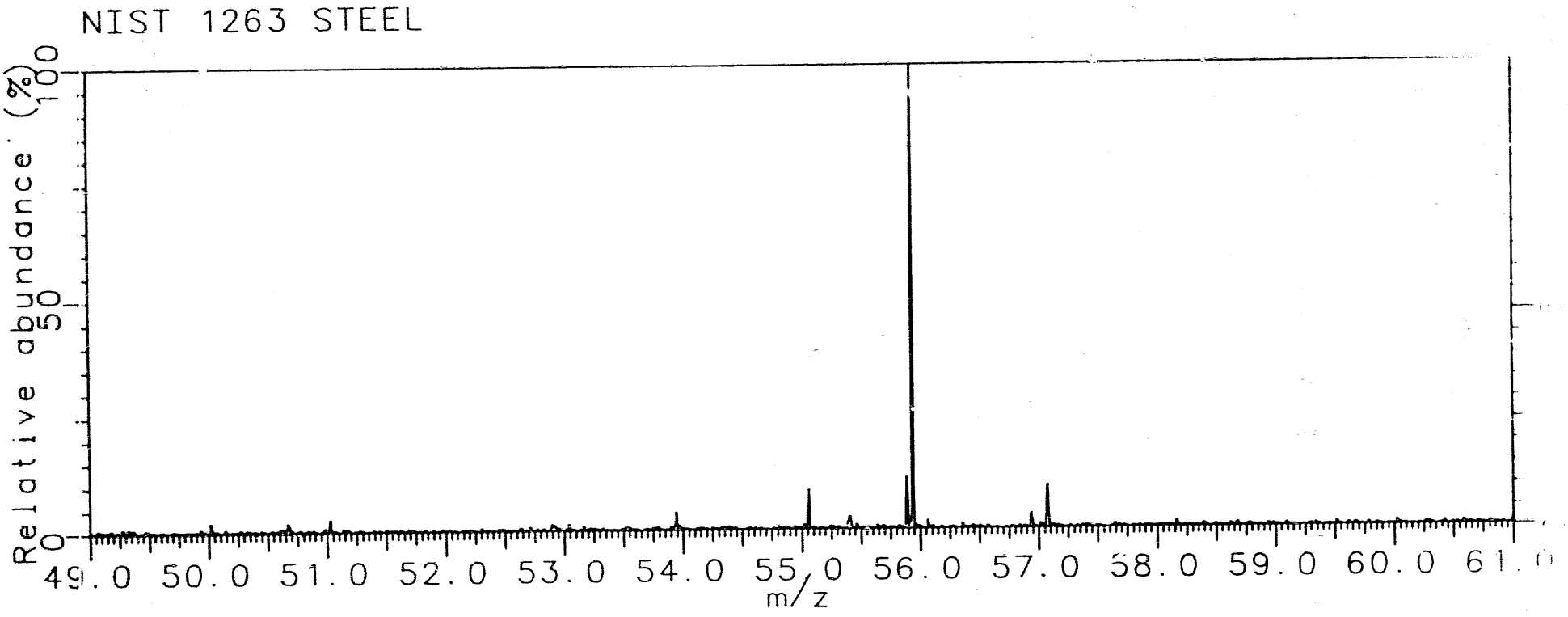

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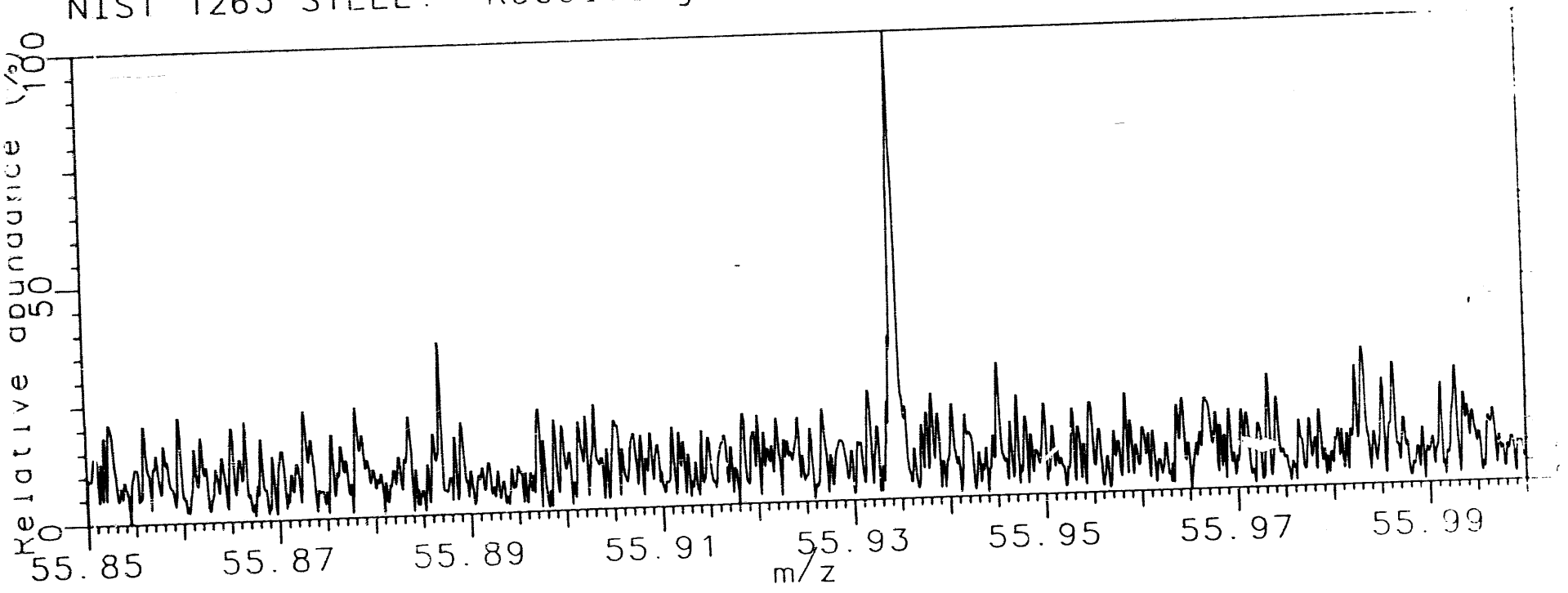


RF-GD/FTMS at the Savannah River Technology Center

The success of these preliminary experiments has led to the design of an RF-GD/FTMS instrument for the Savannah River Technology Center. This instrument is currently under construction and incorporates the following design improvements over the demonstration project instrument.

* Higher differential pumping speed - 2300 l/s mounted directly on the differential pumping chamber. This is compared to 1200 l/s mounted 9 to 12 inches below.

* Smaller conductance limits. The conductance limit between the differential pumping chamber and the main FTMS vacuum system can be reduced by at least 50%. This will greatly reduce the gas load to the analyzer cell vacuum region, enabling higher resolution spectra to be obtained.

* Rigid support frame. The external source needs to be support rigidly to ensure correct alignment relative the ICR dual cell/magnetic field axis. This will improve ion transfer efficiencies and enable the use of the dual analyzer cell (dual cell experiments were not possible in the preliminary experiments due to mis-alignment of the vacuum chamber/cell assembly after the external source was cantilevered off the analyzer flange).

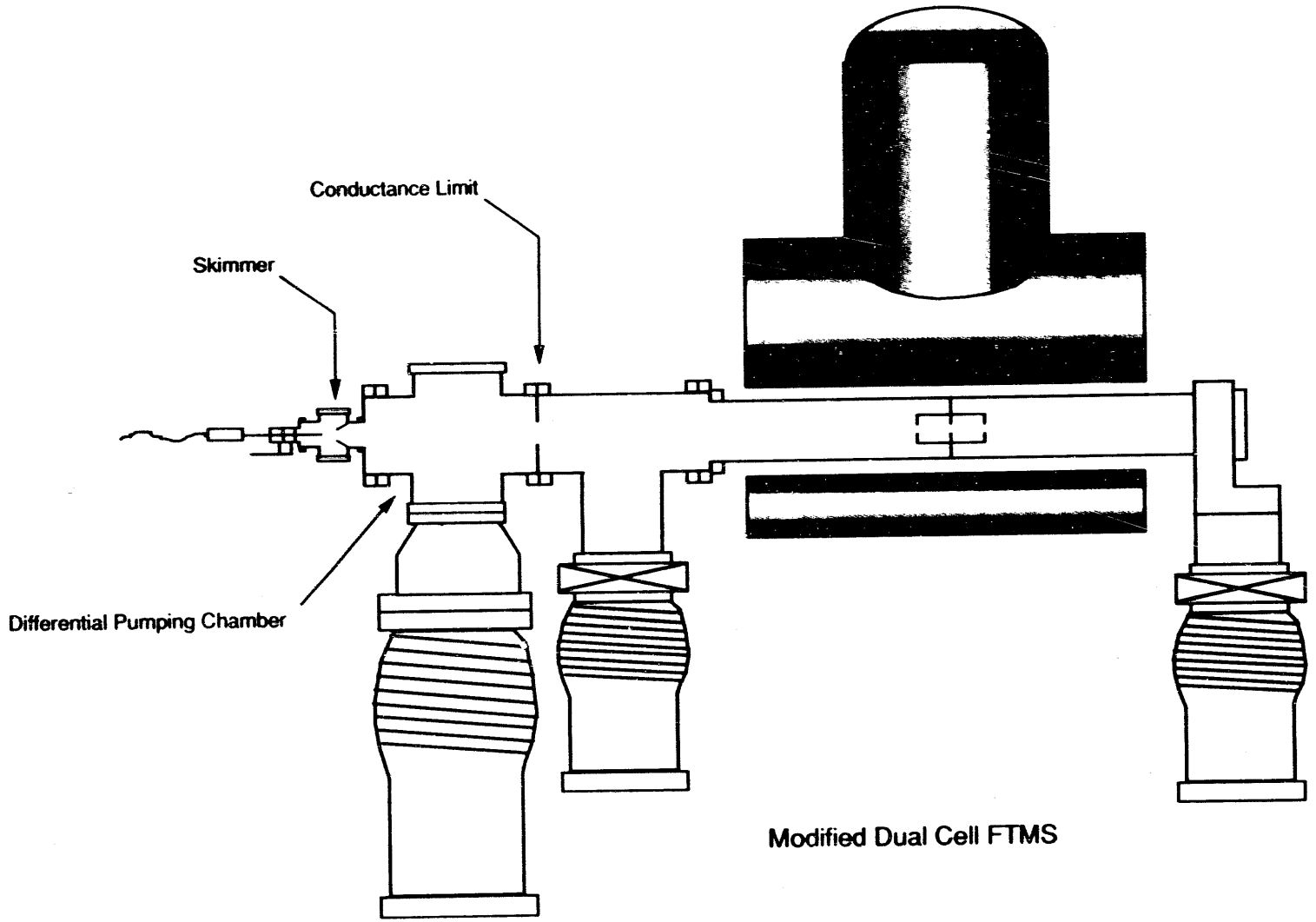
RF-GD/FTMS at the Savannah River Technology Center (Contd.)

* Deceleration grid. A grid that can be used to accelerate or decelerate to find the optimum ion kinetic energy to maximize ion trapping efficiencies. Also, a grid will be employed to block the large number of electrons produced in the glow discharge.

* Dual dissassemblable analyzer cell. The latest generation of commercially available ICR analyzer cells are easily reconfigured between single and dual cell configurations. In the cases where dual cell applications are not required, a single 2 X 2 X 4 inch cell can be used. This will not only increase the sensitivity of the instrument, but all for a larger dynamic range. An additional benefit of this arrangement is that both of the dual cell diffusions pumps (each 900 l/s) will now pumping on the single analyzer cell, increasing the resolution capabilities of the instrument.

* New data system. The latest commercially available data systems allow for larger transients to be digitized and have a higher digitization rate. This allows for higher resolution spectra to be obtained over the entire mass range without the need for heterodyning. Higher digitizing rates allow for the observation of lower mass ions at a given magnetic field, an important feature in elemental mass spectrometry.

SRTC RF-GD/FTMS



Modified Dual Cell FTMS

CONCLUSIONS:

The success of these preliminary RF-GD/FTMS experiments demonstrate that an RG/GD source can easily be coupled with a Fourier transform ion cyclotron resonance mass spectrometer. The ability to transfer ions from an external ion source to the ICR cell does not need complex lens or ion guide systems. The ability to do ultra-high resolution elemental mass spectrometry will allow one to unambiguously identify the species present in a wide variety of samples.

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