SRNS-STI-2010-00340

Extending the Useful Life of Older Mass Spectrometers

J. V. Cordaro, S. R. Johnson, M. K. Holland, and V. D. Jones
Savannah River Nuclear Solutions, Aiken, South, Carolina 29808

Abstract

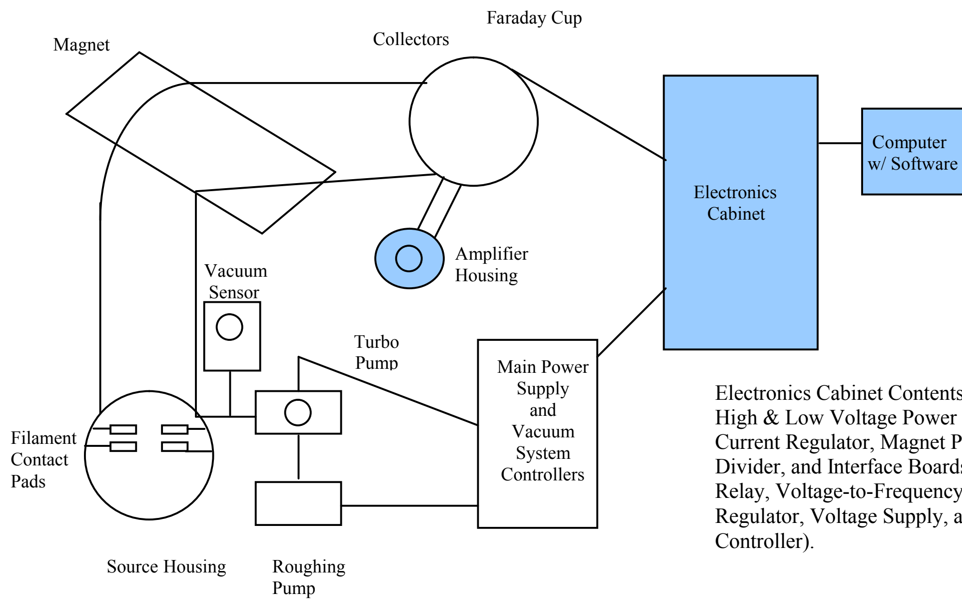
Thermal ionization and gas mass spectrometers are widely used across the Department of Energy (DOE) Complex and contractor laboratories. These instruments support critical missions, where high reliability and low measurement uncertainty are essential. A growing number of these mass spectrometers are significantly older than their original design life. The reality is that manufacturers have declared many of the instrument models obsolete, with direct replacement parts and service no longer available. Some of these obsolete models do not have a next generation, commercially available replacement. Today's budget conscious economy demands for the use of creative funds management. Therefore, the ability to refurbish (or upgrade) these valuable analytical tools and extending their useful life is a cost effective option.

The Savannah River Site (SRS) has the proven expertise to breathe new life into older mass spectrometers, at a significant cost savings compared to the purchase and installation of new instruments. A twenty-seven year old Finnigan MAT-261™ Thermal Ionization Mass Spectrometer (TIMS), located at the SRS F/H Area Production Support Laboratory, has been successfully refurbished. Engineers from the Savannah River National Laboratory (SRNL) fabricated and installed the new electronics. These engineers also provide continued instrument maintenance services. With electronic component drawings being DOE Property, other DOE Complex laboratories have the option to extend the life of their aged Mass Spectrometers.

Introduction

Disabled for a more than a year, the upgrade of the 1980's vintage MAT-261™ was accelerated by making use of several original parts: the complete vacuum system (to include the focus / acceleration lenses, ion flight tube and associated electronics), magnet, Faraday Cup collectors, amplifier housing, and magazine turret. The upgrade consumed 16 total months of effort. Activities consisted of parts procurement, electronic component design, fabrication (10 months), installation and finally instrument acceptance. In addition to a new software package, plug-and-play electronic modules replaced the voltage divider, high/low voltage power supplies, magnet power, and filament current regulator. The next phase of the upgrade will involve enhancements to automate the magazine turret, and further upgrades of the vacuum system electronics.

Figure 1: View of the refurbished Finnigan MAT-261™ Thermal Ionization Mass Spectrometer



Electronics Cabinet Contents:
High & Low Voltage Power Supplies, Filament Current Regulator, Magnet Power, Voltage Divider, and Interface Boards (Mass Selector, Relay, Voltage-to-Frequency, Digital, Field Regulator, Voltage Supply, and Magnet Controller).

Figure 2: Schematic of Thermal Ionization Mass Spectrometer
(Shaded portion denotes upgrade scope.)

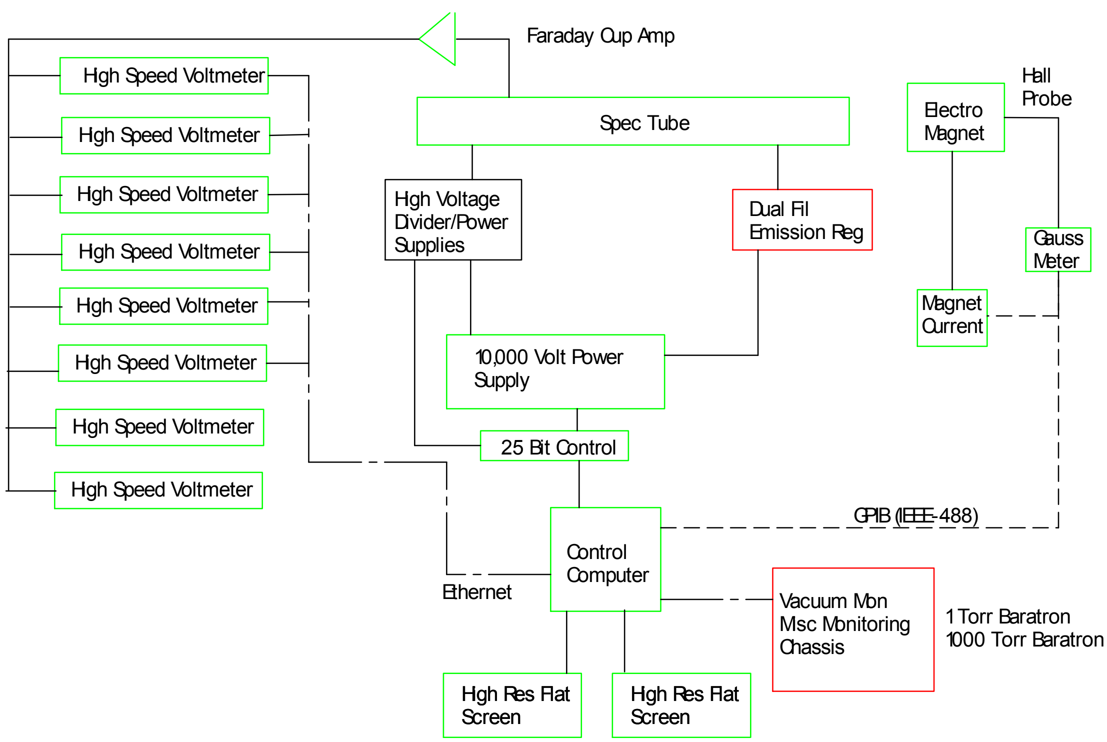


Figure 3: Detail of upgraded electronics, from shaded areas of Figure 2.

Software

National Instruments Lab View software utilizes the Windows XP operating platform. This software has allowed instrument control and data acquisition capabilities beyond that previously possible for the MAT-261™. The mass spectrometer's operation is now autonomous. The user's impact on measurement variance has been minimized by limiting their actions to moving the magazine turret and opening/closing the isolation valve.

Enhanced operational features include:

- Real-time peak scanning;
- Peak Tuning, which allows adjustments in magnet current and operating voltages for peak shape optimization;
- Simultaneous voltage monitoring of all Faraday Cups;
- Diagnostic testing for Faraday Cup noise, high voltage stability, and linearity of the magnetic field;
- Temperature monitoring of the magnet and Faraday Cup Collector housing; and
- Automatic filament current regulation.

The software also features two methods of data acquisition: on the flat peak plateau and integrating the area under the peak. While data acquisition on the flat peak plateau is the more widely used method at the Savannah River Site, integrating the area under the peak is being evaluated. With data acquisition on the flat plateau being unaffected by peak alignment issues, indicators are that the alternate method would respond likewise. Future plans are to incorporate the total evaporation method in the suite of software-controlled data acquisition capabilities.

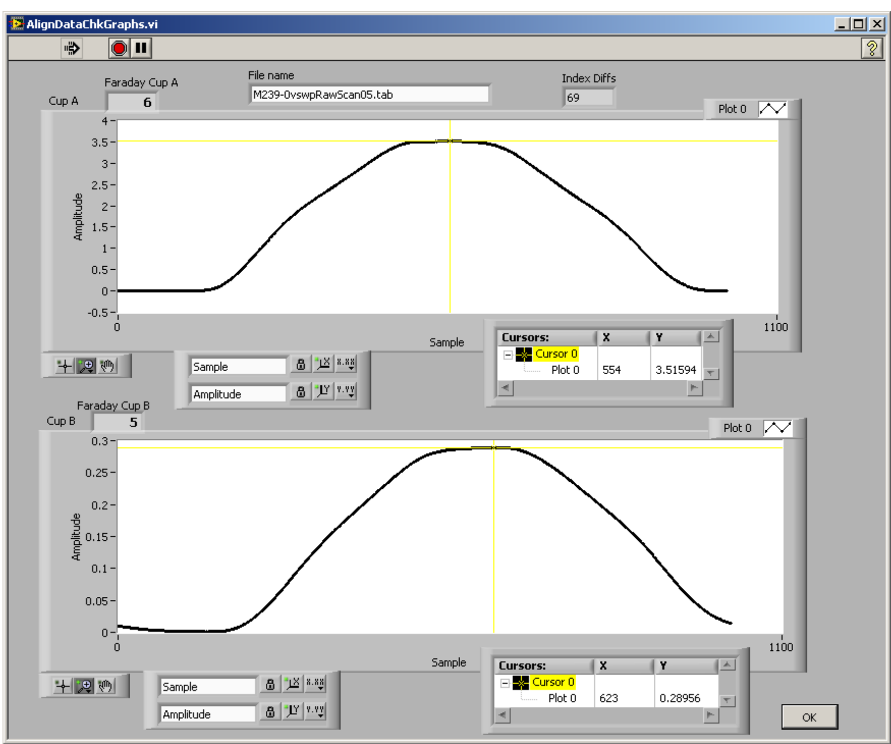


Figure 4: Data acquisition methods are unaffected by peak alignment issues.

Faraday Cup Calibration

The MAT-261™ is a seven Faraday cup TIMS, with one fixed and six manually, movable cups. Once the atomic mass unit (amu) spacing was verified, cup calibration routines were performed. Peak tuning and centering refinements were accomplished by observing the Re-187 peak in each cup.

Adjacent cups were calibrated using multiple forward and backward magnet sweeps. Using plutonium isotopic abundance Certified Reference Standards, the Pu-240/239 ratio was determined on each pair of adjacent cups and compared with the decay-corrected reference value. Interrelating the response of the seven Faraday cups proved to be an accurate and swift method for system calibration. The results are applicable for both uranium and plutonium analyses.

The former MAT-261™ cup configuration placed the U-233 ion beam in cup 8, to accommodate the Isotope Dilution capability. However, cup 8 could not be moved exactly 1amu from the adjacent cup. It appeared that the manufacturer compensated for a design shortcoming, by incorporating a software patch. Output response from this cup was 15% low, unstable, and had poor measurement precision. The most effective and expedient method of resolution was to abandon cup 8. The uranium ion beams were shifted to allow for an alternate cup collection pattern, as illustrated below.

Movable Faraday Cups	8	7	6	5	4	3	2
				Fixed Cup			
Uranium (currently)			**233**	**234**	**235**	**236**	**238**
Pre-upgrade	233	234	235	236		238	
Plutonium		**238**	**239**	**240**	**241**	**242**	**244**

Figure 5: MAT-261™ Faraday Cup Configuration Note: Cups 2/3 have a distance of 2 amu.

Acceptance Testing

Acceptance of the MAT-261™ was based on the full range analysis of uranium isotopic abundance Certified Reference Standards. Performance was gauged against the acceptance criteria, that were based on statistical process control warning limits set at 2-sigma and alarm limits set at 3-sigma. This was the same methodology used eight years earlier, to accept a newly purchased Triton.

The acceptance test data demonstrated that $\geq$ 97% of the test results were within 1-sigma of the reference value. Hence, the instrument's performance proved acceptable for routine operation.

	U-930 Observed at% 235/238 Ratio	Known	MDF
	17.3499220	17.349	-1.771E-05
	17.3376770	17.349	0.0002176
	17.3285879	17.349	0.0003922
	17.3223814	17.349	0.0005114
	17.3429543	17.349	0.0001162
	17.3631146	17.349	-0.0002712
Avg	17.340773	AvgMDF	0.0002
Stdev	0.014724		0.0002
%RSD	0.0849		
% RD	-0.0474		

	U-500 Observed at% 235/238 Ratio	Known	MDF
	1.00263	0.9997	-0.0009756
	1.00125	0.9997	-0.0005157
	0.99930	0.9997	0.00013386
	1.00102	0.9997	-0.0004413
	0.99814	0.9997	0.00051901
Avg	1.000468	AvgMDF	-0.0003
Stdev	0.001757		0.0006
%RSD	0.1756		
% RD	0.0768		

	U-100 Observed at% 235/238 Ratio	Known	MDF
	0.1135770	0.1136	6.763E-05
	0.1136773	0.1136	-0.0002268
	0.1135517	0.1136	0.0001417
	0.1138418	0.1136	-0.0007096
	0.1135541	0.1136	0.0001345
Avg	0.113640	AvgMDF	-0.0002
Stdev	0.000124		0.0004
%RSD	0.1089		
% RD	0.0355		

	U-050 Observed at% 235/238 Ratio	Known	MDF
	0.052888	0.0528	-0.0005569
	0.052864	0.0528	-0.0004049
	0.052799	0.0528	4.0404E-06
	0.052858	0.0528	-0.0003682
	0.052671	0.0528	0.00081237
	0.052891	0.0528	-0.0005715
Avg	0.052829	AvgMDF	-0.0002
Stdev	0.000084		0.0002
%RSD	0.1587		
% RD	0.0543		

	U-350 Observed at% 235/238 Ratio	Known	MDF
	0.54605	0.5465	0.0002726
	0.54622	0.5465	0.0001726
	0.54629	0.5465	0.0001287
	0.54808	0.5465	-0.0009655
	0.54682	0.5465	-0.0001958
Avg	0.546693	AvgMDF	-0.0001
Stdev	0.000829		0.0005
%RSD	0.1516		
% RD	0.0352		

SRS	Working Std, Observed at% 235/238 Ratio	Known	MDF
	0.0103196	0.01033	0.000337
	0.0103335	0.01033	-0.000113
	0.0103416	0.01033	-0.000373
	0.0103558	0.01033	-0.000832
	0.0103395	0.01033	-0.000305
	0.0103386	0.01033	-0.000278
Avg	0.010335	AvgMDF	-0.0003
Stdev	0.000013		0.0004
%RSD	0.1287		
% RD	1.9242		

Figure 6: Acceptance Test Data

Charted are the calculated mass discrimination factors at each isotopic range above, illustrating the instrument's linearity.

	235/238 Reference Ratio	MDF	Stdev
U-930	17.349	0.0002	0.0002
U-500	0.9997	-0.0003	0.0006
U-350	0.5465	-0.0001	0.0005
U-100	0.1136	-0.0002	0.0004
U-050	0.0528	-0.0002	0.0002
SRS-WS	0.01033	-0.0003	0.0004
Overall MDF		-0.0001	0.0002

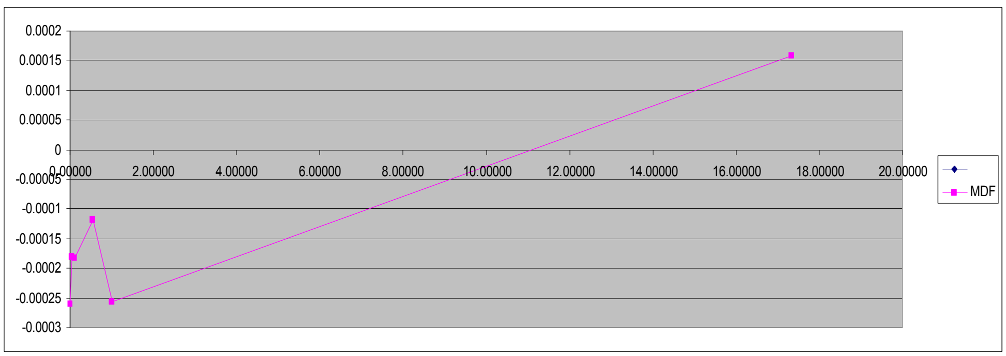


Figure 7: U-235/238 Ratio vs calculated Mass Discrimination Factor

Summary

The Finnigan MAT-261™ Thermal Ionization Mass Spectrometer is one of two instruments required to support the large sample load and demanding turnaround schedule of the SRS F/H Production Support Laboratory. On the average, a 60 hour sample turnaround is achieved. Typically, the annual throughput is 8,000 samples. This encompasses uranium / plutonium isotopic abundance and isotope dilution concentration measurements.

For the upgrade project, all budgetary requirements were met with $600K. This included

- completion of the scope, for software and electronics enhancements;
- limited upgrade of the vacuum electronics; and
- $100K in spare parts.

Functional task areas for distribution of funds were as follows:

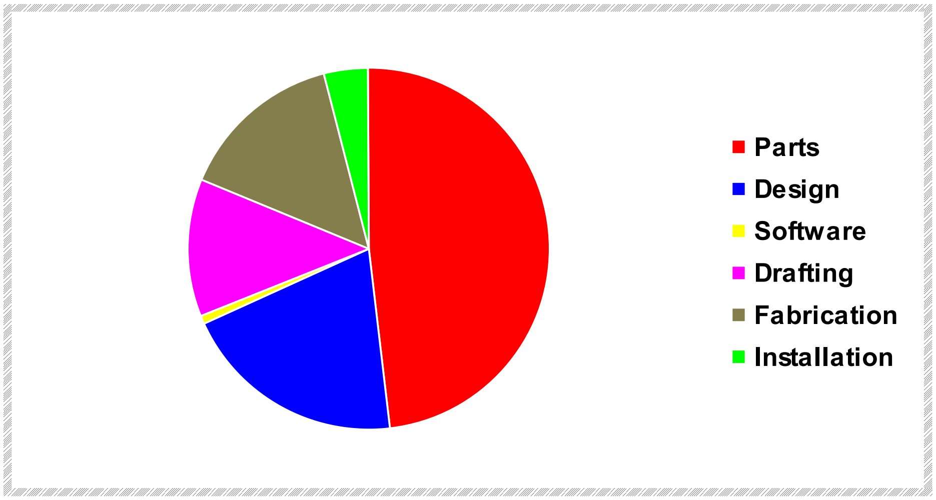


Figure8: Cost distribution

Regaining operability of the MAT-261™ was a major milestone. Once full method capability has been demonstrated for plutonium, the MAT-261™ will be returned to routine service. The use of two instruments will alleviate the vulnerability of a single-point failure, ever present when only one TIMS is in operation.

Lessons Learned

1. Research and development produce unexpected challenges. It is essential to build sufficient time, funding, and mitigation into the schedule.

- The manufacturer's use of a software patch to compensate for Faraday cup 8 design problems was unforeseen. The time-efficient solution was to abandon the cup and shift the beam to an alternate collection array.

- With a twenty-seven year old instrument, conducting the upgrade while making use of the original vacuum electronics was an identified risk. However, mitigation was incorporated into the scope. The best approach would be to include system vulnerabilities in the initial work scope. This will ensure adequate schedule and funding for resolution of challenges.

2. Consider redesigning the source lens stack, as a post upgrade activity. The operational life of any lens stack is limited. Additionally, the parts for the older instruments are difficult to locate. The redesign would prove to be a value-added task, when projecting the life extension of the upgraded spectrometer.

3. A mock-up TIMS unit, assembled in the Engineering fabrication facility, was invaluable for the testing of fabricated modules prior to field installation.

4. Due to the enhanced output from the new electronics, the original procedural parameters for filament loading and heating required adjustment. As a result of the upgrade, less material was required to be loaded onto the filaments. Likewise, the current required to generate the ion beam was reduced.

References

1. "Electronics Upgrade for High Resolution Mass Spectrometers", by J. V. Cordaro and J. B. McIntosh, WSRC-STI-2007-00658. (www.osti.gov/bridge/index.jsp) as of 6/10/2010.