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Development and Deployment of Retrofit PolarisQ Ion Trap Mass Spectrometer for Isotope Ratio Measurements

Cyril V. Thompson
William B. Whitten

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OAK RIDGE NATIONAL LABORATORY
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Chemical Sciences Division

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OAK RIDGE NATIONAL LABORATORY
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This report describes Oak Ridge National Laboratory's (ORNL) FY15 progress in support of National Nuclear Security Administration's (NNSA) Portable Mass Spectrometer project. A retrofit PolarisQ ion trap mass spectrometer (RPMS) has been assembled from components of two PolarisQ ion trap mass spectrometers used in previous isotope ratio programs (Figure 1). The retrofit mass spectrometer includes a custom Hastelloy vacuum chamber which is about ¼ the size of the standard aluminum vacuum chamber and reduces the instrument weight from the original by nine pounds. In addition, the new vacuum chamber can be independently heated to reduce impurities such as water, which reacts with UF_6 to produce HF in the vacuum chamber. The analyzer and all components requiring service are mounted on the chamber lid (Figure 2), facilitating quick and easy replacement of consumable components such as the filament and electron multiplier.

Figure 1. RPMS Deployed at ORNL Test Loop

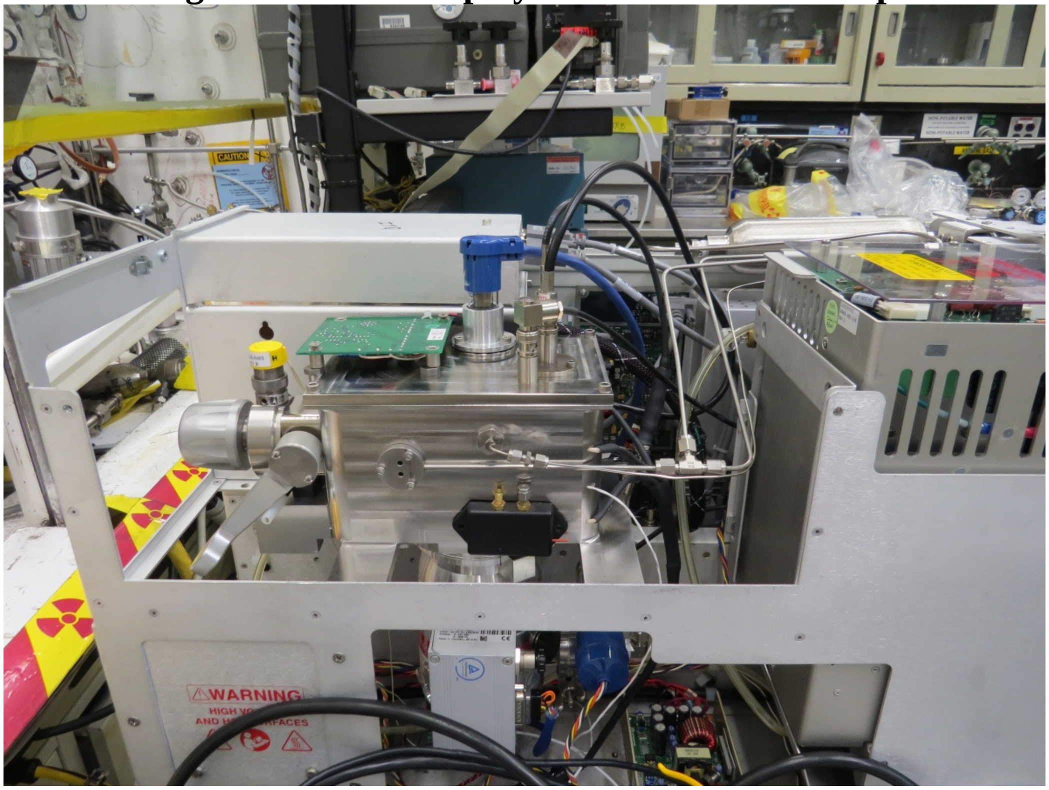
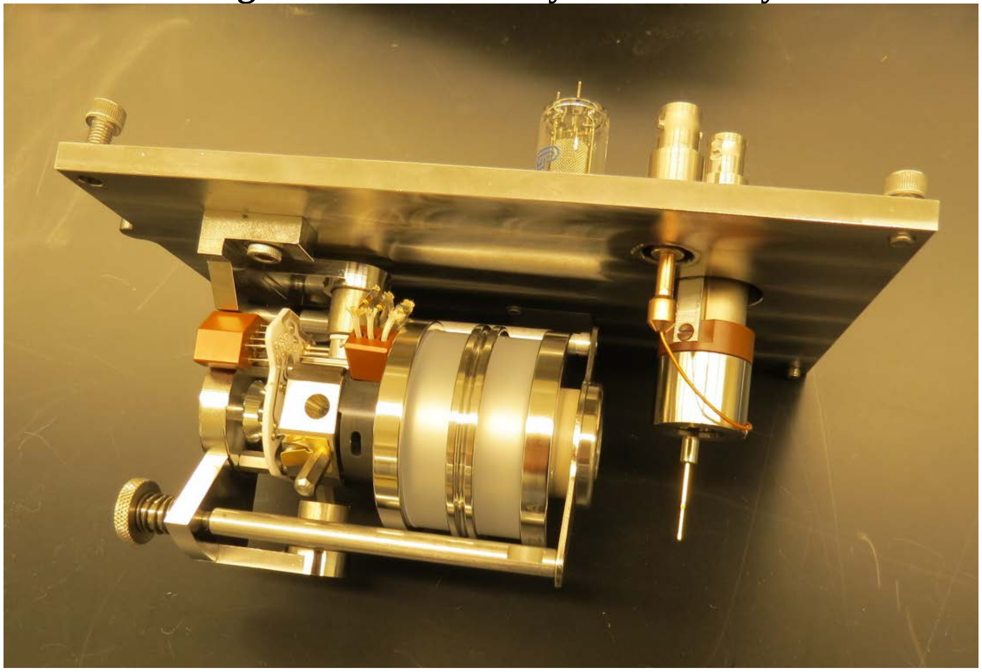


Figure 2. RPMS Analyzer Assembly



The RPMS was assembled and tested with perfluorotributylamine (PFTBA), a standard calibration compound often used with mass spectrometers. In initial tests, the instrument performed well in electron impact (EI) positive ion mode. This is the standard mode of operation used for instrument tuning and calibration.

After confirmation of instrument performance, the RPMS was moved to the ORNL Test Loop and used for analysis of UF_6 to determine its performance in assessment of isotope ratios. Both $^{238}UF_5$ and $^{238}UF_6$ were observed in the tests. However, varying amounts of $^{235}UF_5$ and almost no $^{235}UF_6$ were detected. As can be seen in the mass spectra in Figure 3, the standard PolarisQ spectrum has peaks with leading and trailing shoulders (gentle curvature at the base of the peaks), but the spectrum from the RPMS does not. These shoulders are common phenomena in most ion trap mass spectrometers and their absence is indicative of an instrument malfunction or maladjustment. This phenomenon has also been observed on the standard PolarisQ when the electron multiplier (EM) is reaching the end of its life and is being operated at fairly high voltages (>2000V) (Figure 4). It is believed that in the RPMS, the spacing between the feedthrough for the dynode and the EM assembly was inadequate, and the dynode field (10kV) interacted unfavorably with the trajectory of the ions emitted by the analyzer, causing a non-linear response in the EM. In addition, the cabling for the dynode feedthrough may have experienced an insulation failure which caused fluctuations in the 10 kV dynode potential.

Both of these possible failure modes have been addressed in modifications to the RPMS, which will be incorporated into the ITQ instrument configured for use by International Atomic Energy Agency (IAEA).

Figure 3. Comparison Spectra of UF_6 by RPMS and PolarisQ

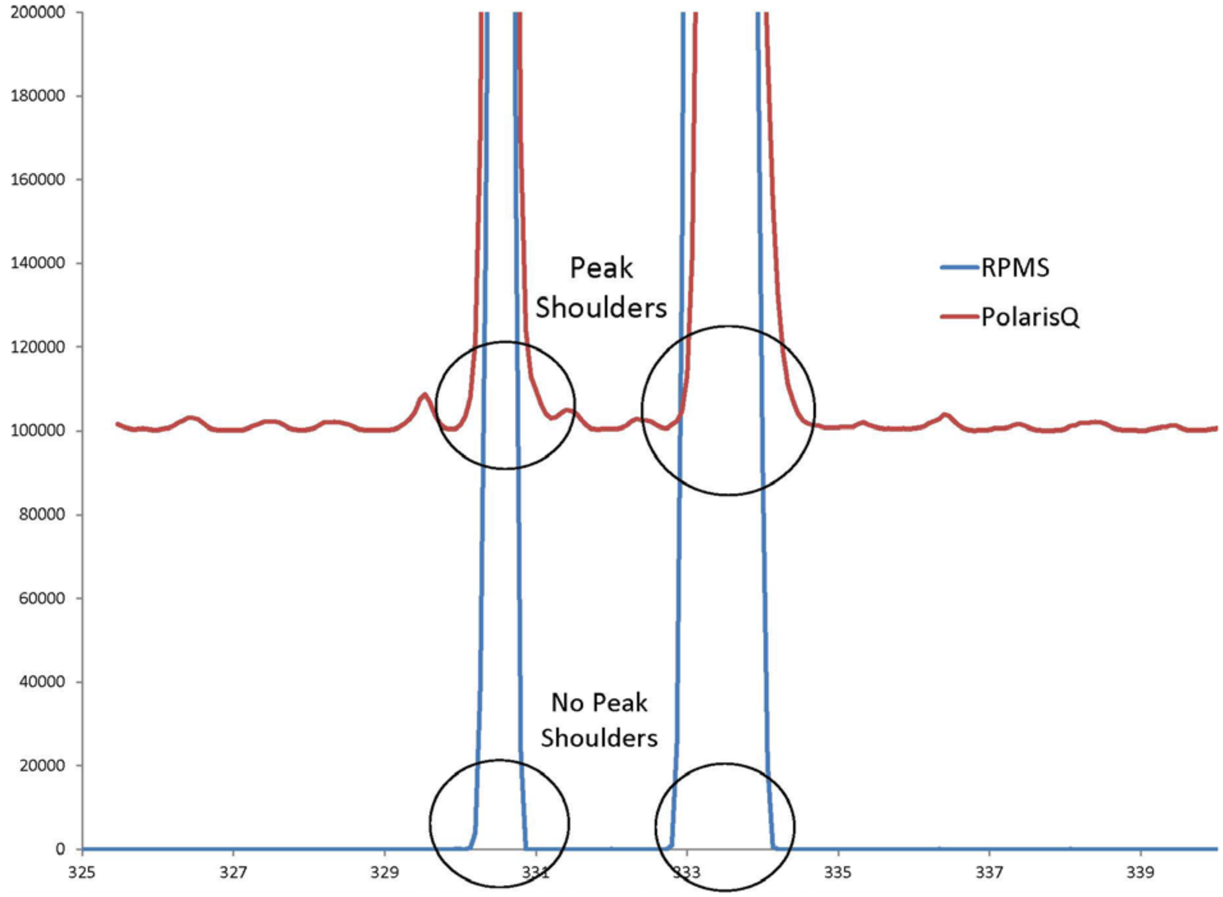
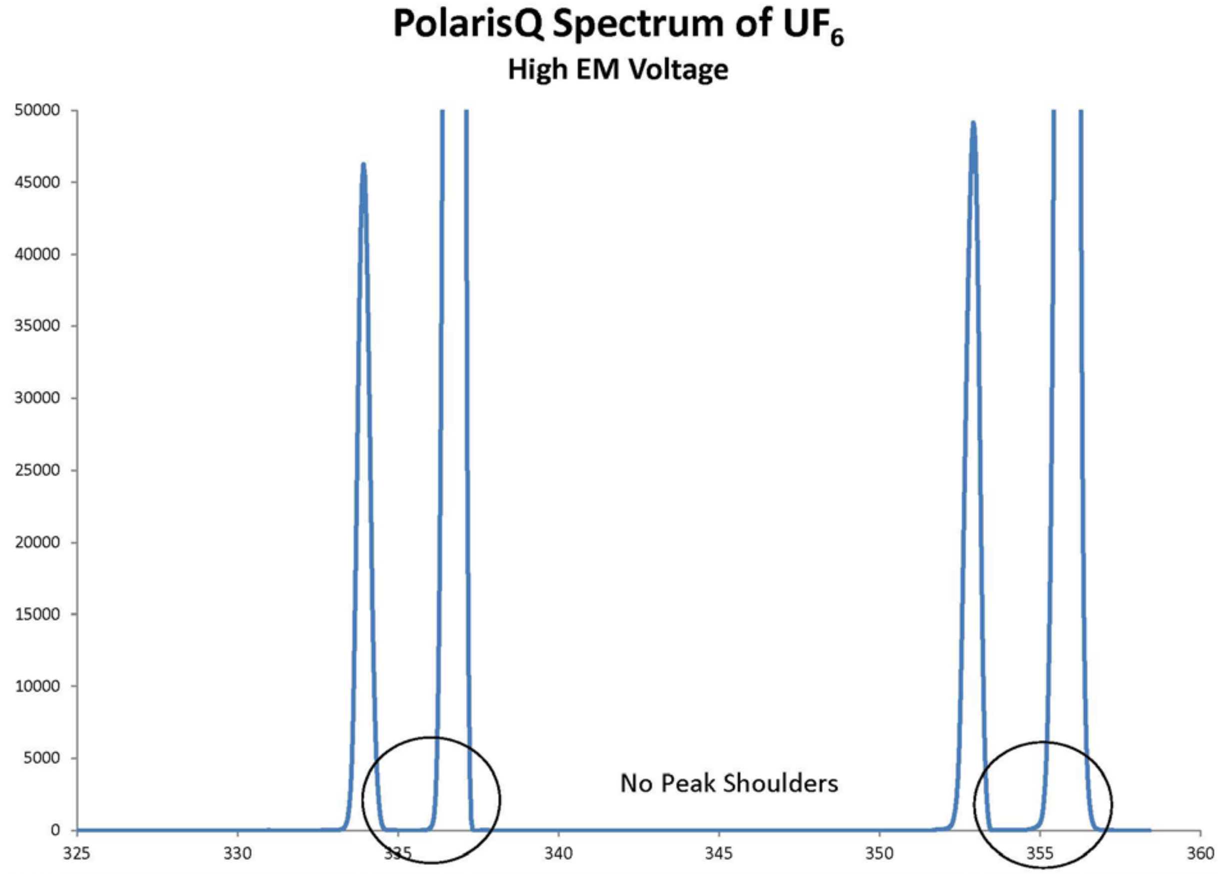
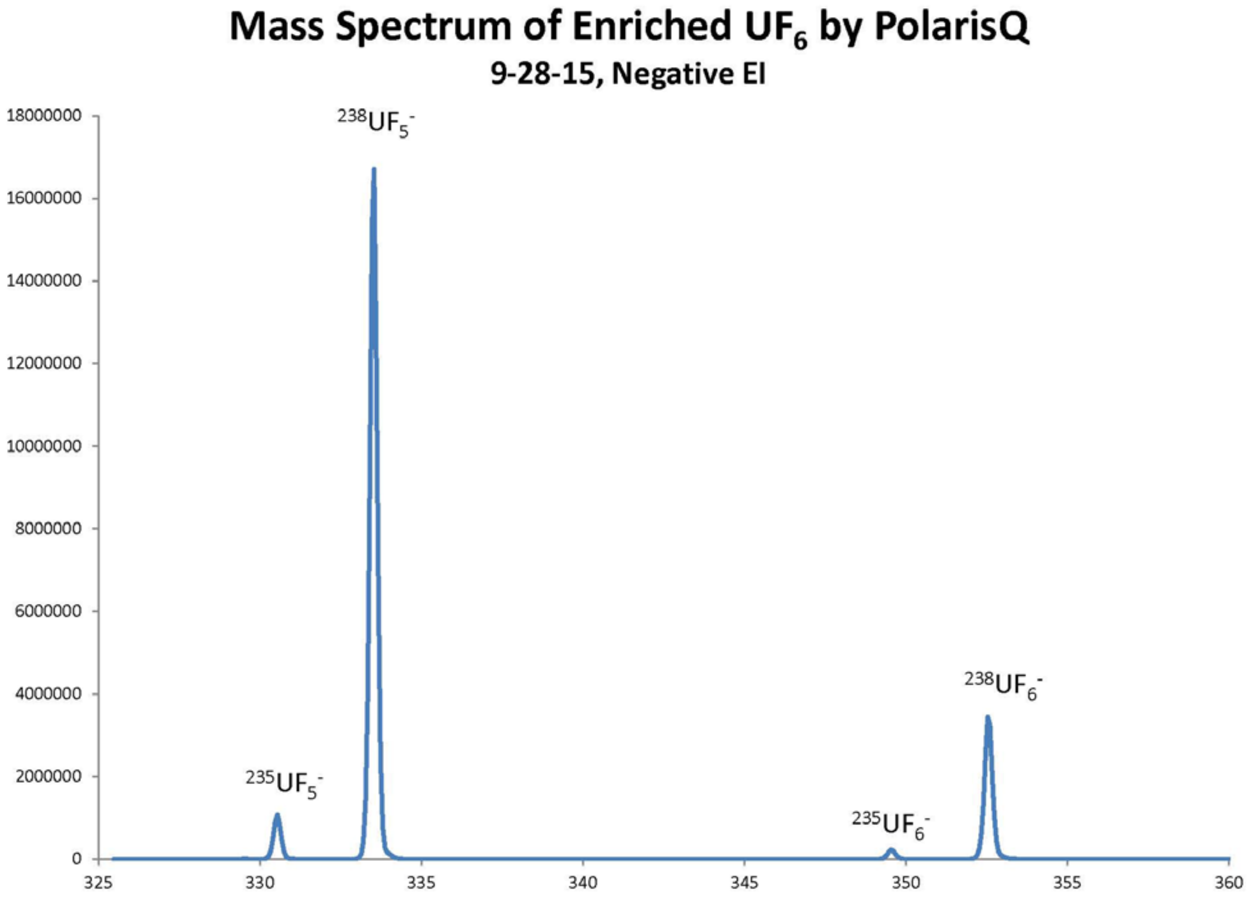


Figure 4



In addition to the testing performed with the RPMS at the ORNL test loop, a number of experiments were also performed with a standard PolarisQ to determine the precision of the mass spectrometer in measuring $^{235}U/^{238}U$ isotope ratios (Figure 5). The goal of IAEA, as communicated by their representatives during the September visit to ORNL, is to have a field deployable mass spectrometer which has a precision variability of less than 1% over one hour of measurement time. By optimizing sampling and mass spectrometric analysis conditions, precisions of 0.84% (UF_5^-) and 1.7% (UF_6^-) were achieved over an hour of sampling. However, some experimental conditions (sample flow rate/pressure, analyzer temperature, exposure of EM to UF_6/HF) could not be controlled as well as was desired. The goal of future experiments is to employ appropriate sampling and analysis equipment and conditions to closely control all the parameters identified in these experiments. With increased control, precision of less than 1% should be achievable. The investigators note that the performance of the standard PolarisQ is expected to be indicative of anticipated performance of the RPMS, as the fundamental instrument parameters are the same. Modifications to convert the ITQ instrument to the RPMS should only enhance instrument performance.

Figure 5



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