



Voltage-induced frequency drift correction in fourier transform electrostatic linear ion trap mass spectrometry using mirror-switching



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ABSTRACT

Ion capture from an external nano-electrospray ionization source in a Fourier transform electrostatic linear ion trap has been effected by mirror-switching. This capture method can suffer from time-dependent frequency shifts in the measured ion motion, which compromises mass resolution when using Fourier transform techniques for mass determination. This phenomenon was determined to be a result of the transient voltage recovery of the power supplies used for mirror-switching in response to a pulsed capacitive load, for which several examples are shown. A circuit, based on the AD210AN isolation amplifier, was fabricated to compensate for the voltage perturbation induced by mirror-switching by superimposing the inverted perturbation to the electric field of the opposing reflectron. In doing so, the dependence of the ions path length and frequency on the power supply output was greatly reduced throughout data acquisition. With this circuit enabled, no frequency shifts were observed in the mass spectrum when using mirror-switching, and thus pressure-limited theoretical resolutions were achieved. For example, an absorption mode resolving power of greater than 50,000 $M/\Delta M$ FWHM was observed for iodide (m/z 126.9) at a transient length of 300 milliseconds. The use of mirror-switching led to a much greater m/z range than in-trap potential lift for a single ion injection which is demonstrated via simulation and experimental results. This correction method is simple to implement and does not require user intervention once properly tuned.

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1. Introduction

In the last two decades, routine polyatomic mass spectrometry (MS) has transitioned to make use of the high resolution, accurate mass analysis offered by Fourier transform ion cyclotron resonance (FT-ICR) and OrbitrapTM mass spectrometers. When the acquisition rate is not a primary concern, these techniques are favored over time-of-flight (TOF) MS and have allowed researchers to probe the isotopic fine structure of molecules to elucidate empirical formulas and determine compound classes [1–3], directly identify post-translational modifications of peptides [4], etc. Today, these platforms are in common use in many ‘-omic’ communities due to their ability to resolve and analyze complex samples where thousands of signals may be present, each of which may have multiple

ion forms and isotopologues [5–7]. In order for these Fourier transform (FT) based instruments to perform at such a high level, two criteria must be met: (1) the transient length must be sufficient to obtain the necessary mass resolution for the m/z region of interest, which is a function of the background pressure, packet dephasing [8–12], detection sensitivity, and ion frequency; (2) the experimental ion frequency should not change beyond a specified margin over the course of data acquisition and should not change between runs. With the use of appropriate vacuum techniques, the Orbitrap and FT-ICR can achieve pressures of 10^{-10} and 10^{-12} Torr, respectively, with typical operating pressures being between 10^{-9} – 10^{-10} Torr. Even better vacuum may be achieved through the use of specialized pumps and cryogenic cooling [13–15] and as such is not discussed in detail here. In an effort to minimize frequency drift throughout data acquisition and enable the generation of high resolution mass spectra, numerous technologies have been developed by vendors of OrbitrapTM and FT-ICR instruments. These include, but are not limited to, the use of automatic gain control (AGC) [16–19], the dynamically harmonized cell [20–23], the high-field

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Orbitrap [24,25], ultra-stable magnets and power supplies, low-noise preamplifiers [19,26,27], and various modes of calibration [19,28–30]. All of these serve to minimize and/or compensate for field effects arising from space charge, analyzer geometry/field homogeneity, and electric/magnetic field drift, which, if left uncorrected, would compromise the mass resolution when using Fourier transform techniques for mass determination.

The Fourier transform electrostatic linear ion trap (ELIT) is another type of FT-based mass analyzer in which ions are reflected back and forth between two opposing reflectrons. Like Orbitrap, the frequency of an ion in an ELIT is inversely proportional to the square root of its mass-to-charge ratio:

$$f_{ion} = \frac{k}{\sqrt{m/z}} + b \quad (1)$$

where k and b are experimentally determined constants. The image charge induced on a pickup electrode is digitized and Fourier transformed to obtain the mass spectrum. Under low resolution conditions (i.e., short transient times), the ELIT has been shown to be capable of performing absorption mode data acquisition [31], surface induced dissociation [32], and ion isolation [33], thereby demonstrating that it can act as a stand-alone tandem mass spectrometer.

Two approaches have been described for the capture of ions that have been axially injected into an ELIT (Fig. 1): mirror-switching [14,34,35] and in-trap potential lift [36–39]. In the former (Fig. 1, left), the entrance reflectron plates are initially held at potentials sufficiently low to allow ions with sufficient kinetic energy (KE) to enter the ELIT (A). Once all ions of interest are within the accepted time-of-flight distance (D), the entrance reflectron is pulsed high to axially confine the ions (B), after which a transient is recorded (C). With in-trap potential lift (Fig. 1, right), all plate potentials are continually held at their respective trapping potentials. Ions are injected with sufficient KE to overcome the electrostatic barrier set by the trapping potentials of the entrance reflectron (A). When the ions of interest enter the repulsive lift electrode, the applied voltage is pulsed to ground, thereby reducing the potential energy of the ions (B) such that they are no longer able to overcome the electrostatic barriers of the reflectrons and a transient can be recorded (C). If the ions are not within the lift electrode when it is pulsed down, they will have sufficient energy to overcome the electrostatic barrier of the rear reflectron and exit the ELIT.

As can be deduced from a comparison of capture methods, for two traps of equivalent geometry, the accepted time-of-flight distance (D), or m/z range, of mirror-switching is much greater than that of in-trap potential lift and is therefore the desired capture method for an axially injected ion packet comprised of ions of significantly different m/z values. This figure of merit is especially important in biological analysis where the various charge states and dissociation channels of peptides and proteins can encompass a wide m/z range. Unfortunately, just as others encountered with the early designs of the ICR [21,40–42] and Orbitrap [24,30,43,44], as we sought to increase the performance of our ELIT using mirror-switching, we discovered that the ion frequency shifts throughout the course of data acquisition. This phenomenon is known to originate from the trapping action of mirror-switching and is the main limiting factor in achieving higher mass resolution [36,38].

Under steady-state conditions, the amount of charge stored on the output capacitance of a power supply is given by the following relationship:

$$Q_T = C_{output} \cdot V_S \quad (2)$$

where Q_T is the total charge in coulombs, C_{output} is the output capacitance in Farads, and V_S is the supply voltage. As ions are injected into the ELIT, the switch, cables, and reflectron plate represent a

capacitive load with zero charge stored on them (grounded). When ions are to be trapped, the capacitive load is rapidly connected in series with the output capacitance of the power supply where the stored charge quickly (nanoseconds) establishes a trapping voltage determined by:

$$Q_T = (C_{output} + C_{load}) \cdot V_{trap} \quad (3)$$

where C_{load} is the total capacitance of the switch, cables, and plate in Farads, and V_{trap} is the initial potential realized on the plate used for mirror switching. The relationship between the initial trapping potential and the set steady-state potential is:

$$V_{trap} = \frac{Q_T}{C_{output} + C_{load}} = V_S \frac{C_{output}}{C_{output} + C_{load}} \quad (4)$$

As the capacitive load will always be greater than zero, the initial trapping potential will always be lower in magnitude than the steady-state power supply voltage. At this point, the power supply will undergo transient voltage recovery wherein the voltage feedback loop of the power supply seeks to increase the output voltage back to the nominally set potential. As this recovery is not instantaneous, and can occur over hundreds of milliseconds, the ions experience a time-dependent trapping potential and thereby have a time-dependent path length and frequency. It should be noted that as the output capacitance of our power supplies are typically on the order of hundreds of nanofarads, and the capacitance of the load is expected to be on the order of hundreds of picofarads, the initial trapping potential is expected to differ from the set potential (\sim –2500 V) by a few volts. As the capacitance of the load is minimized, the initial trapping potential approaches the nominally set potential of the power supply, and thus the magnitude of the observed frequency shift will become smaller. Increasing the output capacitance of the power supply might be considered as an approach to minimize the time of the voltage adjustment. However, this is not recommended since the in-rush current just after the switch is closed can be hundreds of amperes. Many switches are not rated for such a current; a lower output capacitance limits the in-rush current due to the lower number of stored charges.

While in-trap potential lift does not suffer from frequency shifts associated with the trapping method [39], it suffers from relatively narrow m/z range, lower ion frequencies (see discussion), and a more complex detection scheme. Herein, we describe the fabrication and experimental evaluation of an active circuit that is capable of minimizing changes in the ion path length throughout the recorded transient when using mirror-switching to capture ions in an ELIT. To accomplish this task, the circuit measures the transient voltage recovery of the power supply, inverts it, and adds it to the voltage applied to the opposing plate. In this manner, the turning point of the ion packet in both reflectrons moves concurrently in the same direction, minimizing the observed frequency drift. This approach enables the acquisition of high resolution mass spectra across the wide m/z range offered by mirror-switching. We report our findings with respect to the design of the circuit, its use in measuring the transient recovery of various power supplies in response to a pulsed capacitive load, and its experimental implementation for mass analysis.

2. Experimental

2.1. Materials

A mixture of carboranes consisting of the major components AgCHB₁₁H₅Cl₆, and CsCH₃CB₁₁I₁₁ (100 μM each) was prepared by dissolving the analytes in 50% H₂O, 50% methanol (v/v). The carboranes were synthesized and provided by Professor C.A. Reed's group at the University of California Riverside, Department of Chemistry. Water and 2-iodopropane were purchased from Sigma-Aldrich

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