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External-ion accumulation in a Penning trap with quadrupole excitation assisted buffer gas cooling

H.-U. Hasse[a], St. Becker[a], G. Dietrich[b], N. Klisch[a], H.-J. Kluge[a], M. Lindinger[a], K. Lützenkirchen[b], L. Schweikhard[a,*], J. Ziegler[a]

[a]*Institut für Physik, Johannes Gutenberg-Universität, D-55099 Mainz, Germany*
[b]*Institut für Kernchemie, Johannes Gutenberg-Universität, D-55099 Mainz, Germany*

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Abstract

A pulsed ion beam from an external source is injected into a Penning trap and accumulated by repeatedly lowering the electric potential of the entrance end cap. This lowering is performed only partially, i.e. a trapping well is retained during ion capture to prevent the ions already captured from escaping. For the same reason the newly captured ions have to be cooled, which is achieved by buffer gas collisions. To prevent radial ion loss, the ions are exposed to azimuthal quadrupole excitation. By choosing the appropriate frequency (range) this method (selective quadrupole excitation assisted capture and centering (SQUEACE)) allows a mass selection during the capture process and leads to a centering of those ions in the Penning trap. The multiple ion bunch capture results in a significant improvement in signal-to-noise ratio and a decrease in experiment duration.

Key words: ICR; Penning trap; Accumulation; Quadrupole excitation; SQUEACE

Introduction

Ion traps are ideally suited to investigate the properties of charged particles. In recent years many Penning trap systems have been employed for both fundamental studies and analytical applications [1,2]. Most experiments are still being performed with ions created inside (or very close to) the trap region. However, the injection and capture of externally created ions is of increasing importance. These techniques are required when exotic particles are to be examined or the properties of the ion source are incompatible with those of the ion trap. For example, they are mandatory if the ion source includes large sophisticated equipment (e.g. a particle accelerator or an on-line mass separator, see below), requires easy access, or operates at high pressure. Several methods have been developed to guide the ions to the trap, including systems of Einzel lenses, r.f. quadrupole fields and coaxial static ion guides. For a review of these techniques the reader is referred to the literature [3]. Once the ions are inside the trap, further measures have to be taken to keep them from leaving it immediately. Three examples show the diversity of the externally created particles investigated.

(i) Dietz et al. [4] have developed a very versatile cluster ion source which combines laser desorption and expansion of a seeded gas jet into the vacuum. These clusters can be guided to an ICR cell where they are trapped and Fourier transform ion cyclotron resonance (FT-ICR) experiments are performed [5]. Differential pumping has to be

* Corresponding author.

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used to separate the trap region, where UHV is required, from the cluster source and its gas jet. To adjust the energy of the incoming ions to the potential of the ICR cell, the potential of a transfer drift tube is ramped during the passage of the ions.

(ii) For the investigation of radioactive isotopes the ISOLTRAP tandem Penning trap system has been installed at the on-line mass separator ISOLDE at CERN/Geneva [6]. Here the particles of interest are delivered as a continuous ion beam. They are implanted in a rhenium foil which is then aligned with the equipotential surface of the trap electrodes and heated. The atoms evaporate from the foil, are surface-ionized, and trapped inside a Penning trap. They are then cooled [7] and transferred [8] to a second Penning trap where an accurate mass determination [9] is performed under UHV conditions.

(iii) Antiparticles are even more delicate species. They are readily available only as high energy ion beams. In 1986, the first capture and storage of antiprotons in a Penning trap was reported by Gabrielse et al. [10]. Antiprotons from the Low Energy Antiproton Ring (LEAR) at CERN are slowed by a degrader and those with kinetic energy below 3 keV can be captured by pulsing the entrance trap electrode. Cooling is performed by collisions with stored electrons. The trapped low-energy antiprotons allowed an improvement of the proton/antiproton mass ratio by several orders of magnitude [11].

In this paper we report the multiple capture of externally created ions with an apparatus recently constructed at the University of Mainz [12]. Externally created ions are transferred to a Penning trap by an ion optical system of Einzel lenses and deflectors. The Penning trap stores the ions while they are subject to interactions with r.f. excitation fields, electrons, other ions, neutrals or photons. Reaction products can be detected and analyzed either by the FT-ICR method or by axial ejection from the trap and time-of-flight (TOF) mass spectrometry. The latter uses single ion counting and therefore is a very sensitive technique. Currently, the ions investigated are delivered from a cluster ion source developed and built by Weidele et al. [13]. Recent experiments include collision-induced dissociation [14] and chemical reactions [15] of small gold cluster cations. For these preliminary investigations single ion bunches have been captured in flight [8]. However, only a few cluster ions of a given size can be captured and even fewer reaction products are produced in each measurement cycle. Therefore the signals of hundreds of cycles have to be added to produce statistically significant mass spectra. This is a very time consuming procedure. In the following we describe an alternative approach where ions of several bunches are accumulated in the trap. This allows for shorter measurement durations and therefore an increased stability of experimental parameters and for an improvement of the signal-to-noise ratio.

Conceptional considerations

We restrict ourselves to an overview of the main features of Penning traps [16,17]. The ions (charge q, mass m) are confined radially by a static homogeneous magnetic field B and axially by a static electric quadrupolar potential leading to a harmonic axial motion. The frequency of this oscillation is denoted by ω_z. The frequency of the cyclotron motion inside the trap, ω_+, is slightly reduced with respect to the cyclotron frequency $\omega_c = qB/m$ in the absence of any electric field and given by

$$\omega_+ = \frac{\omega_c}{2} + \sqrt{\left(\frac{\omega_c}{2}\right)^2 - \frac{\omega_z^2}{2}} \tag{1}$$

For most applications one finds $\omega_z \ll \omega_c$ and therefore $\omega_+ \approx \omega_c$. Furthermore, there is a second circular motion in the radial direction, centered on the trap axis. This is the magnetron motion with frequency

$$\omega_- = \frac{\omega_c}{2} - \sqrt{\left(\frac{\omega_c}{2}\right)^2 - \frac{\omega_z^2}{2}} \tag{2}$$

Note that

$$\omega_c = \omega_+ + \omega_- \tag{3}$$

Ions produced at small kinetic energies within

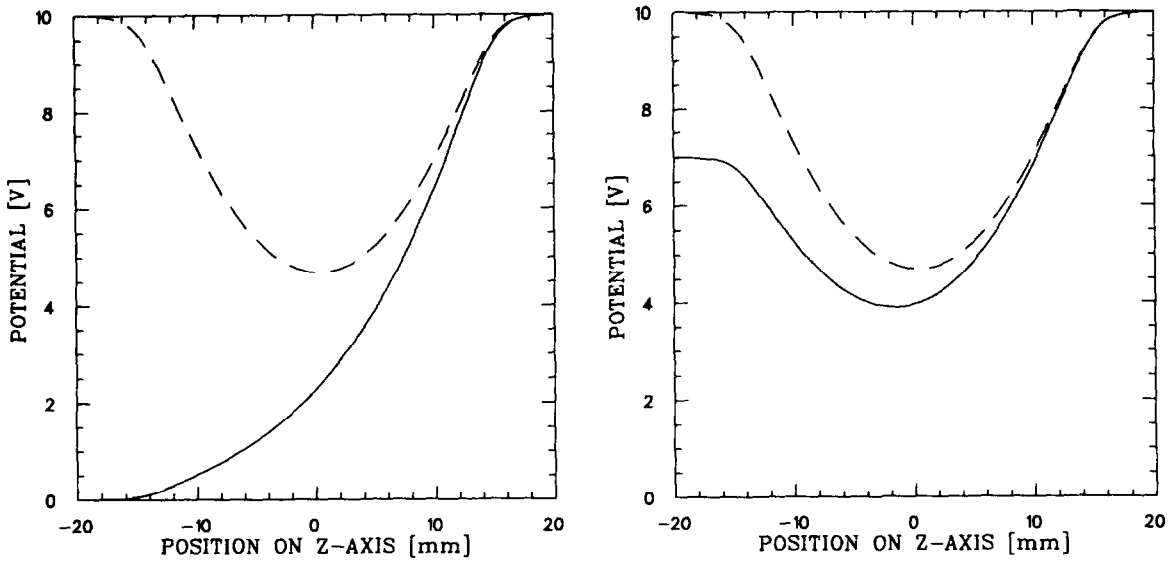

Fig. 1. Electric trap potentials along the trap axis without remaining trapping well (left) and with remaining trapping well (right) as used for multiple ion bunch capture. The latter involves a partial lowering of the potential of the entrance end cap: (– – –), indicates the trap potential during storage periods.

the trap region stay trapped. External ions can be brought into the trap along the magnetic-field axis. In this way their path is not perturbed by the Lorentz force of the magnetic field. As mentioned above, the electric trapping potential is static. An ion entering the trap region while the trap potential is turned on will have enough energy to leave the trap immediately afterwards. In-flight capture can be performed by pulsing the trap potential, e.g. the voltage of the entrance endcap is lowered when the ions are arriving and increased back to the trapping voltage when they are inside the trap. For single-bunch capture this can be efficiently done by pulsing the voltage down to zero with respect to the ring voltage (Fig. 1, left) [14].

If however, a second ion bunch is to be captured this way, some of the previously trapped ions will be able to leave the trap while the arriving ones are slowed down. This problem can be overcome if (i) the trap is not opened all the way but a (smaller) trapping potential is still retained during the capture process (Fig. 1, right) and (ii) a cooling mechanism is applied that reduces the axial energy of the trapped ions and pushes them deeper into the potential well.

This cooling can be achieved by buffer gas collisions (in general long range interaction via polarization of buffer gas particles) which lead to a reduction of the axial (as well as the cyclotron) kinetic energy of the trapped ions. Unfortunately, due to the properties of the Penning trap system, collisional cooling also leads to an increase of the magnetron radius [7]. Hence, the ions are eventually lost by collisions with the ring electrode. As has been shown [7], the increase in magnetron radius may be counteracted or even reversed by a particular excitation mode, the azimuthal quadrupole excitation. This cooling scheme has been developed for the ISOLTRAP system and has recently been implemented in several experiments [18–21] after its first application with an FT-ICR system [22].

The cooling technique is based on the conversion of magnetron motion into cyclotron motion which can be induced by application of an azimuthal quadrupolar r.f. field at the cyclotron frequency, ω_c [17,23]. The cyclotron motion, in turn, is cooled by any kind of frictional force, such as the one from buffer gas collisions. Therefore, the amplitudes of all three ion motions, axial, magnetron and cyclo-

tron, are decreased simultaneously and the ions are axialized and centered in the trap, improving the efficiency of subsequent experiments. Furthermore, since the coupling of magnetron and cyclotron motion takes place at frequency $\omega_c = qB/m$ only, the procedure described is mass selective when a single frequency excitation is applied. We therefore dubbed it selective quadrupole excitation assisted capture and centering (SQUEACE).

Experimental setup

The experimental system used for the reported measurements consists of a cluster ion source, an ion transfer section, a Penning trap and a time-of-flight section. A detailed description of the apparatus is in preparation [12]. In the following we give an overview of the main features.

Cluster ions are produced by laser desorption (frequency doubled Nd:YAG laser, Lumonics HY400, pulse length about 10 ns, pulse energy about 8 mJ) off a gold wire into a helium atmosphere (pressure about 25 mbar). The gas/cluster mixture expands into the vacuum chamber (pressure about 10^{-3} mbar) which is separated from the transfer section by two skimmers [13].

The transfer section includes three Einzel lenses and three deflector pairs to steer the ions into the trap. The skimmers and two additional apertures are used in combination with three diffusion pumps (650, 650, and 300 l s^{-1}) and a turbo-molecular pump (450 l s^{-1}) for differential pumping between ion source and Penning trap. No additional transfer potential (with respect to ground potential) is required if the source is kept at a potential of about +100 V. The ions all leave the source at approximately the same velocity, hence the kinetic energy depends on cluster size [24]. By adjusting the offset of the trap potential the cluster size of interest can be selected.

The trap is shown in Fig. 2. It consists of two end caps (each with an inner and outer part) and a multi-segmented ring electrode allowing for several combinations of excitation and detection schemes [25–27]. The inner diameter of the ring electrode is 40 mm and the distance between the endcaps is $40/\sqrt{2}$ mm for asymptotically symmetric trapping potentials [28]. The trapping well depth used for the experiments reported is 5 V. The magnetic field is provided by a 5 T superconducting magnet (Oxford Instruments, model 5T/183). The r.f. signal for quadrupolar excitation is applied to ring segments I and V (Fig. 2) at typical peak-to-peak amplitudes of 2 V. Note that the standard quadrupolar configuration consists of two equal pairs of opposite electrodes, which are rotated 90° with respect to each other and to which opposite phases of the r.f. are applied. However, a single pair of electrodes will work as well, provided the excitation voltage is small with respect to the trapping voltage. In this case the effective trapping voltage is slightly modulated by the r.f. signal and the quadrupolar excitation strength is reduced by a factor of two.

A pulsed valve is mounted in the gap between the ring electrode and one of the endcaps. This valve is based on a construction by Gerlich [29]; using a sandwiched piezo crystal that can be operated in high magnetic fields. Typical gas pulses have a length of a few milliseconds (Fig. 3) and increase the pressure in the trapping region to about 3×10^{-4} mbar.

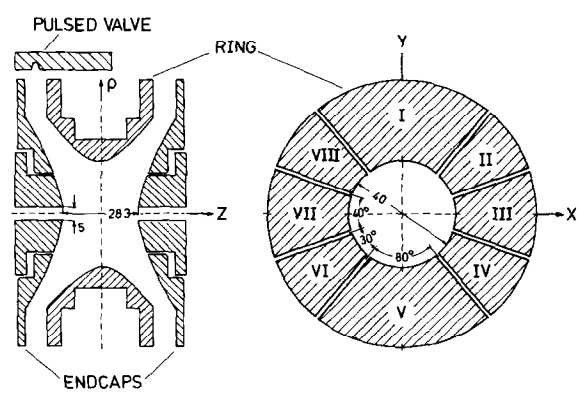

Fig. 2. Cuts through the Penning trap electrodes. Left, cut along the z–ρ-plane; right, cut through the ring electrode along the x–y-plane at $z = 0$. The position of the pulsed valve is indicated at the gap between the ring and one of the end cap electrodes. The segmentation of the ring electrode allows for various excitation and detection modes.

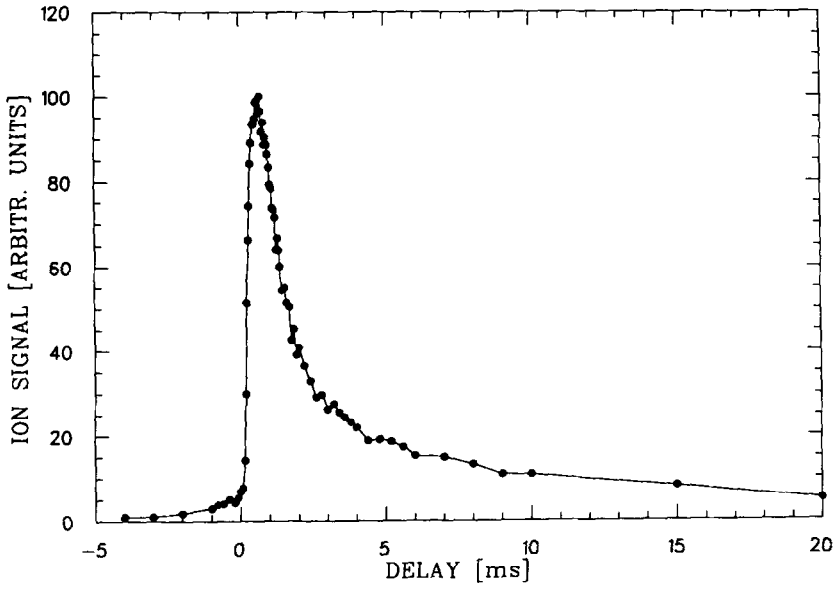

Fig. 3. Gas pulse profile of the piezo valve as monitored by delayed pulsed electron ionization of the gas in the trap. The number of ions is shown as a function of the delay period between pulsed gas inlet and ionization.

The trap region can be used as the first section of a two-stage acceleration for time-of-flight mass spectrometry [30]. The ions are ejected and drift to a micro channel plate detector about 2 m from the trap along the magnetic-field axis. A VME-bus computer and CAMAC electronics serve for the control of the experimental sequence, data acquisition and on-line evaluation.

Results and discussion

In a first experiment we determined the time scale for the loss of ions due to the increase of the magnetron radius caused by collisions with buffer gas. Figure 4(a) shows the experimental sequence: a single ion bunch is produced by laser desorption, transferred and captured by partially opening the trap potential well (Fig. 1 (right)). Simultaneously a gas pulse is applied. Then, at a rate of 10 Hz the trap is opened and the gas is injected without additional laser shots, i.e. without any newly arriving ions. Subsequently, the stored ions are ejected to the TOF drift section and mass selectively registered by a transient recorder. In Fig. 5 the number of detected Au_8^+ cluster ions is shown as a function of the storage period (including potential pulsing and gas injections). The ion numbers are normalized to those for one single opening of the trap potential. If no further precautions are taken the number of ions in the trap decays exponentially ($\tau \approx 2.1$ s) with storage duration (Fig. 5, filled circles).

The open circles in Fig. 5 correspond to a second series of measurements where a quadrupolar r.f. signal of 2 V (peak-to-peak) and 48.72 kHz (the cyclotron frequency of Au_8^+) has been applied continuously. Instead of a decay, a constant ion signal is observed for several seconds, indicating that no ions are lost. The quadrupolar excitation prevents them from leaving the trap. For short storage durations the number of detected ions even increases due to the axialization effect of SQUEACE: Ions that have large cyclotron or magnetron radii after capture may not leave the trap through the exit hole for the TOF detection, but instead collide with the endcap and hence are lost. (Alternatively, even if they can leave the trap they may not be focused onto the MCP detector unless they have been well centered inside the trap.)

The mass selectivity of the method was verified

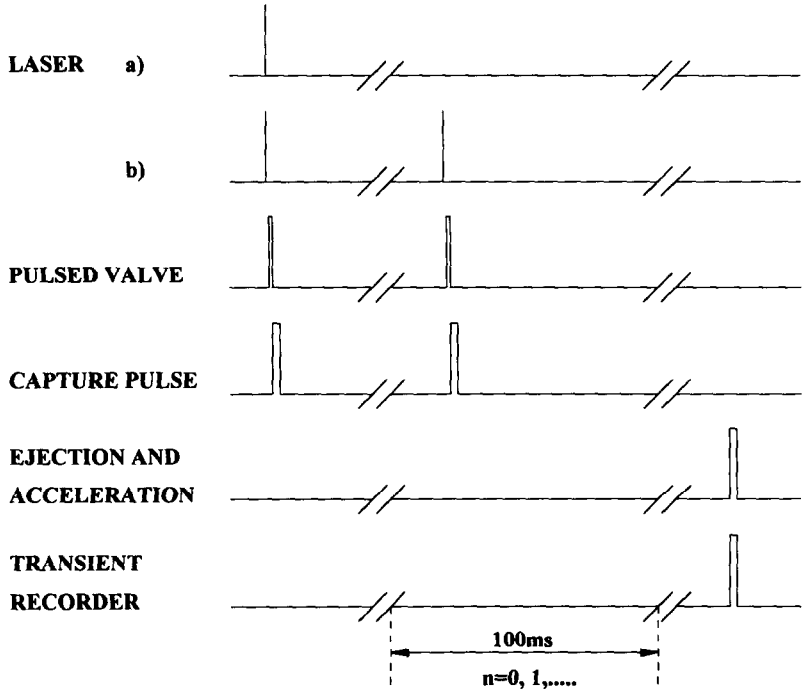

Fig. 4. (a) Experimental sequence for the measurement of the storage efficiency: a single ion bunch is produced and captured. At a rate of 10 Hz the trap is then opened and the gas injected without additional ion capture until the stored ions are ejected and mass selectively registered by a transient recorder. (b) Experimental sequence for accumulation of ion bunches: as in (a), but with multiple shots of the desorption laser, i.e. multiple capture of ion bunches.

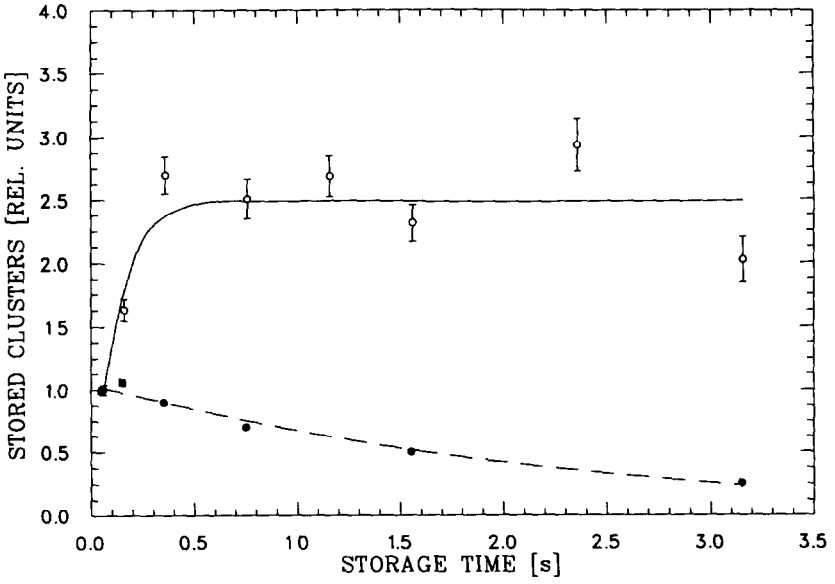

Fig. 5. Number of time-of-flight detected Au_8^+ cluster ions as a function of storage time for the experimental sequence shown in Fig. 4(a). The ion numbers are normalized to those for one single opening of the trap potential: (●), without resonant quadrupole excitation; (○), with quadrupole excitation; (– – –), is an exponential fit to the data points with $\tau = 2.1$ s; (——), to guide the eye.

by varying the (fixed) frequency of the quadrupolar excitation. The number of time-of-flight detected ions, in this case Au_6^+, is plotted in Fig. 6 as a function of the r.f. frequency. Again the experimental sequence of Fig. 4(a) is used, now with a constant storage period of 1.55 s. The number of detected Au_6^+ ions increases by an order of magnitude at the resonance frequency with a FWHM of (46 ± 4) Hz. This corresponds to a mass resolving power of about 1400 at a mass $m \approx 1200$ u.

We now turn to the actual accumulation of externally created ions. Figure 4(b) shows the experimental sequence for accumulation of ion bunches. The scheme is almost the same as described above. However, multiple shots of the desorption laser, i.e. multiple transfer and capture of ion bunches is now performed. In Fig. 7 the number of time-of-flight detected ions is shown as a function of fill duration (here given as the number of fill cycles at a fill rate of 10 Hz). The number of detected ions is normalized with respect to the number for a single fill cycle. If quadrupolar excitation is applied (open circles) the signal increases linearly with the number of fill cycles. If not (filled circles), the number of detected ions also increases, but soon levels off as the number of ions captured during one fill cycle approaches the losses since the last cycle.

For the TOF measurements, single-ion counting has been used. This limits the number of ions of a given mass that can be detected during each measurement cycle, without any significant dead-time losses, to about 20. For the measurements described above the number of ions captured has been intentionally limited (by adjusting the ion source parameters) to lower values. Larger ion ensembles have been detected by FT-ICR. Figure 8 shows the accumulation of gold trimer cations, Au_3^+. The amplitude of the FT-ICR signal is plotted as a function of fill cycles. A linear increase of the amplitude is observed for up to 56 fill cycles (presently the maximum number for FT-ICR due to technical reasons). By comparison of FT-ICR signal height with single ion counting measurements at lower signal intensity we estimate that about 5000 ions contribute to the maximum FT-ICR signal, still far below the space charge limit of the trap's ion storage capacity [31].

An additional advantage of SQUEACE as compared to working with single ion bunches is

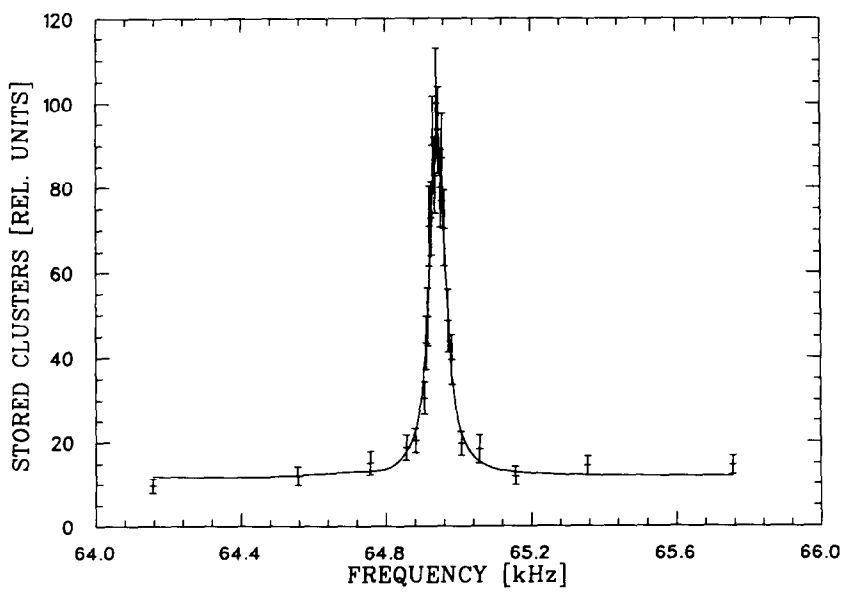

Fig. 6. Number of time-of-flight detected Au_6^+ cluster ions as a function of the r.f. frequency of the quadrupole excitation. Experimental sequence as in Fig. 4(a); storage times, 1.55 s. A Lorentz curve has been fitted to the data points.

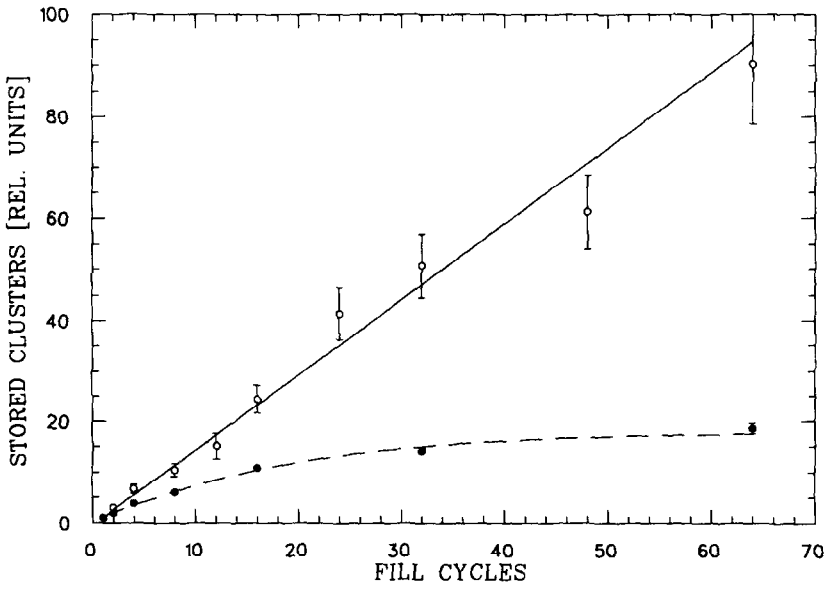

Fig. 7. Accumulation of Au_6^+ cluster ions. Number of detected ions versus the number of fill cycles (normalized to a single fill cycle). Experimental sequence as shown in Fig. 4(b). The ion numbers are normalized to those for one single opening of the trap potential: (●), without resonant quadrupole excitation; (○), with quadrupole excitation. The lines are exponential (– – –) and linear (——) fits to the data points.

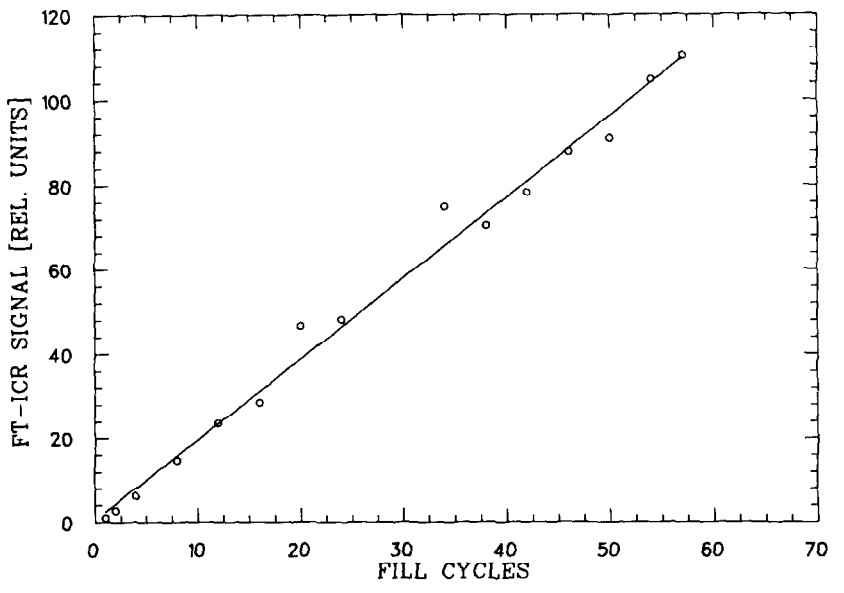

Fig. 8. Accumulation of Au_3^+ cluster ions with resonant quadrupole excitation. Amplitude of FT-ICR signal as a function of the number of fill cycles. A straight line has been fitted to the data points.

improved signal-to-noise (S/N) ratio. Figure 9 demonstrates this for FT-ICR spectra of Au^+ ions. The top three spectra are produced by adding of single ion bunch transients (Fig. 9(a), one bunch; Fig. 9(b) 10 bunches; Fig. 9(c) 100 bunches) and it is clearly seen how the S/N ratio increases with the number of accumulated ion bunches. The bottom spectrum (d) is produced from a single transient after accumulation of ten ion bunches. Its S/N ratio is approximately the same as that from the one hundred added single ion bunch transients.

Summary and conclusion

This work has shown how ion bunches from an external source can be accumulated and centered in a Penning trap. The trap potential is partially lowered for each arriving ion bunch. After capture, the ions are cooled by a buffer gas. The radial loss due to the magnetron instability is counteracted by a resonant azimuthal r.f. excitation which leads to axialization. Since the buffer gas collisions also contract the ion cloud in the axial direction, the ions are effectively centered in the trap. By partial lowering of the end cap potential, further ion bunches can be injected and captured in the trap, allowing an efficient ion accumulation. In addition to the cooling and centering, there are several advantages to experiments using ion accumulation as compared to the use of only one ion bunch at a time.

(i) Since the ion signal is increased, so is the S/N ratio. It has been shown (Figs. 5 and 8) that the ion signal increases linearly with the number of fill cycles n. For a given noise level, the S/N ratio goes up by the same factor n as compared to $\sqrt{n}$ if the same total number of ions is employed in adding n signals from single ion bunch trapping.

(ii) For many experiments the significant parameter is not the number of ions, but rather the duration. From the last section, it is obvious that to achieve the same S/N ratio an accumulation experiment is $\sqrt{n}$ times shorter than the corresponding experiment where single ion bunches are trapped.

(iii) The $\sqrt{n}$-improvement in efficiency as discussed in (ii) applies to the case where the duration of ion production and capture is larger than the time required for the investigation of the ions in the trap (e.g. reactions with neutral atoms and molecules) plus the time for mass analysis. In general, the decrease in experiment duration will be larger than just $\sqrt{n}$, since the latter periods have to be applied only once for ion accumulation, but n times for the single ion bunch analogue.

(iv) Finally, the efficiency will be even further improved, if ion/ion reactions are investigated, where two ion species have to be injected and accumulated. Here the reaction rate will be proportional to the product of both particle numbers and will increase quadratically with the number of fill cycles. Therefore, SQUEACE should allow new experiments with very low reaction cross sections, as e.g. cluster fusion investigations [32].

SQUEACE is a mass selective procedure since it relies on the magnetron-to-cyclotron conversion at $\omega_c = qB/m$. In this paper we have made use of single-frequency excitation only. However, multiple frequency schemes are applicable as well [22].

It should also be noted that the cooling medium need not be a neutral gas. The trap could also be (partially) filled with other charged particles, i.e. ions, electrons or positrons. The incoming ions would then lose their energy to those ions already in the trap. These "cooling ions" can then evaporate from the trap and take away the excess energy [33]. Charged cooling particles are desired for the capture of highly-charged ions where the transfer of electrons, as from neutral species, has to be avoided. Positrons and protons are therefore best-suited for this purpose. This should allow for effective ion capture of highly charged ions, as for ICR studies of these species [35].

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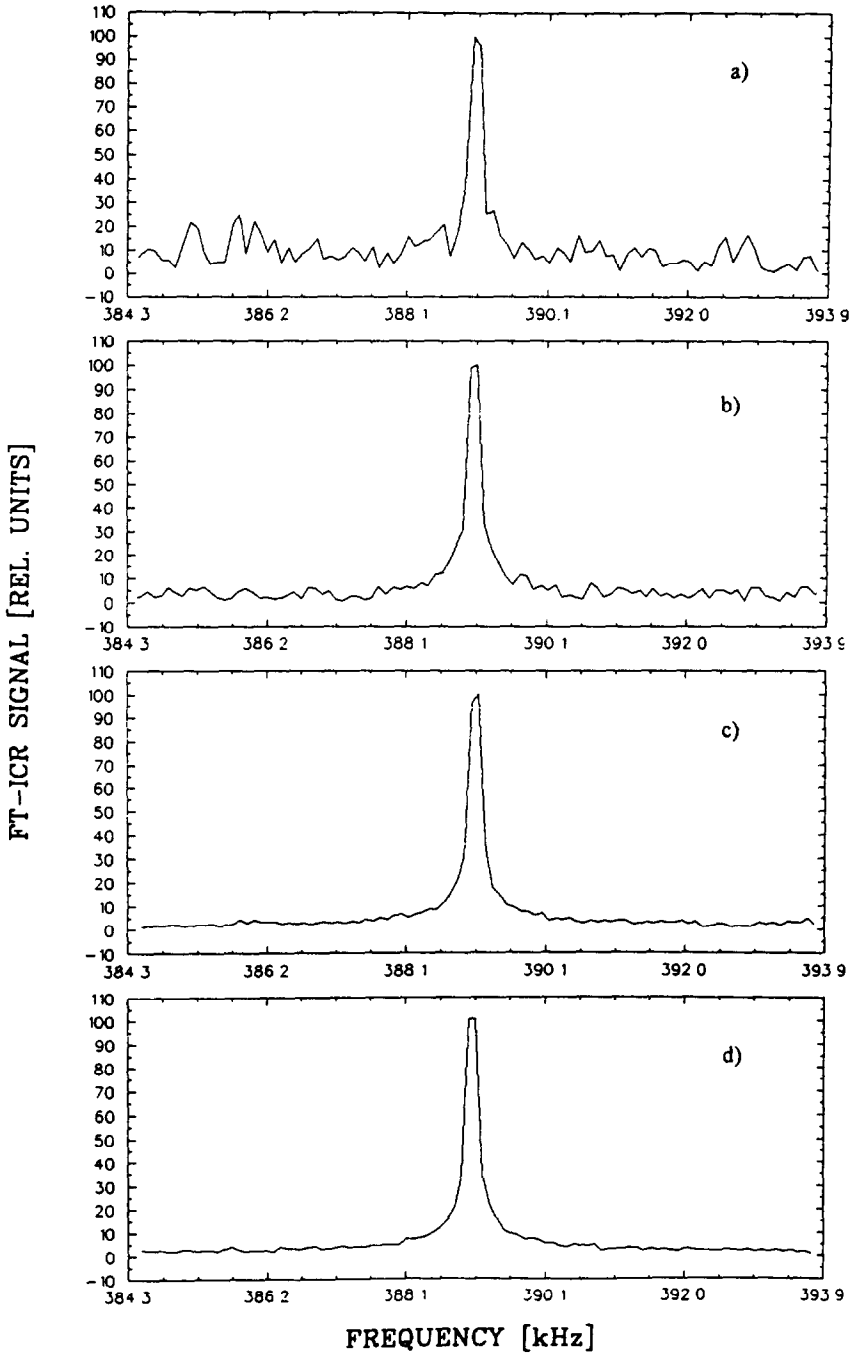

Fig. 9. FT-ICR signals for adding of single ion bunch transients for (a) one bunch, (b) 10 bunches, (c) 100 bunches, and (d) of a single transient after accumulation of 10 ion bunches for the demonstration of the improvement in S/N ratio (see text).

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