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Carbon-cluster mass calibration at SHIPTRAP

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Abstract. A carbon-cluster ion source has been installed and tested at SHIPTRAP, the Penning-trap mass spectrometer for precision mass measurements of heavy elements at GSI. Carbon-cluster ions $^{12}\mathrm{C}_n^+$, $5 \leq n \leq 23$, were produced by laser-induced desorption and ionization from a carbon sample. They were tested for the first time as reference ions in an on-line mass measurement of the radionuclides ^{144}Dy, ^{146}Dy and ^{147}Ho. In addition, carbon clusters of various sizes were used for an investigation of the systematic uncertainty of SHIPTRAP covering a mass range from 84 u to 240 u. The mass-dependent uncertainty was found to be negligible for the case of $(m - m_{ref}) < 100$ u. However, a systematic uncertainty of 4.5×10^{-8} was revealed.

PACS. 07.75.+h Mass spectrometers – 21.10.Dr Binding energies and masses – 36.40.Wa Charged clusters

1 Introduction

In recent years Penning traps have been widely used as a very accurate tool for high-precision mass spectrometry [1–7]. Penning traps are also going to be used in many upcoming projects for mass measurements [8–10]. SHIPTRAP is a Penning-trap mass spectrometer for precision mass measurements of heavy radionuclides at GSI [11,12]. It has been built in particular to enable the investigation of transuranium nuclides and other neutron-deficient nuclides in the medium mass region which are not accessible at ISOL or fragmentation facilities. Most of the masses of transuranium nuclei are only known with an uncertainty of few hundred keV from the extrapolation of systematic trends [13]. The nuclear binding energy, an important parameter for the study of nuclear structure, can be determined from the measured mass. For the future laser spectroscopy, ion chemical reactions and in-trap decay experiments on heavy ions are also planned at SHIPTRAP [11].

For a high-precision mass spectrometer like SHIPTRAP an accurate mass calibration is necessary. Carbon clusters are the mass reference of choice [14] since the unified atomic mass unit is defined as 1/12 of the mass of the ^{12}C atom. The masses of carbon clusters $^{12}\mathrm{C}_n$, $n = 1, 2, 3, \ldots$ are multiples of the unified atomic mass unit since the molecular binding energy of only a few eV per atom can be neglected within the uncertainty level aimed at with SHIPTRAP. Furthermore, carbon clusters can be chosen such that their masses are at most six mass units away from any nuclide. Thus, a possible systematic uncertainty due to the mass difference between the measured mass and the calibrant is reduced. These features give the carbon-cluster ion source a natural advantage over alternative ion sources for mass calibration [15,16].

In addition, cross-reference measurements between cluster ions of different size can reveal systematic uncertainties also over a wide range of masses [17]. They allow to determine the present limit of uncertainty for mass measurements performed at SHIPTRAP. The present work investigates the use of carbon clusters as mass reference and presents the relevant investigations of the measurement uncertainties at SHIPTRAP.

2 SHIPTRAP

2.1 The SHIPTRAP set-up

A schematic drawing of the SHIPTRAP experimental set-up is shown in Figure 1. Radioactive nuclei are produced in fusion-evaporation reactions and separated by the velocity filter SHIP [18]. They are delivered with a typical energy

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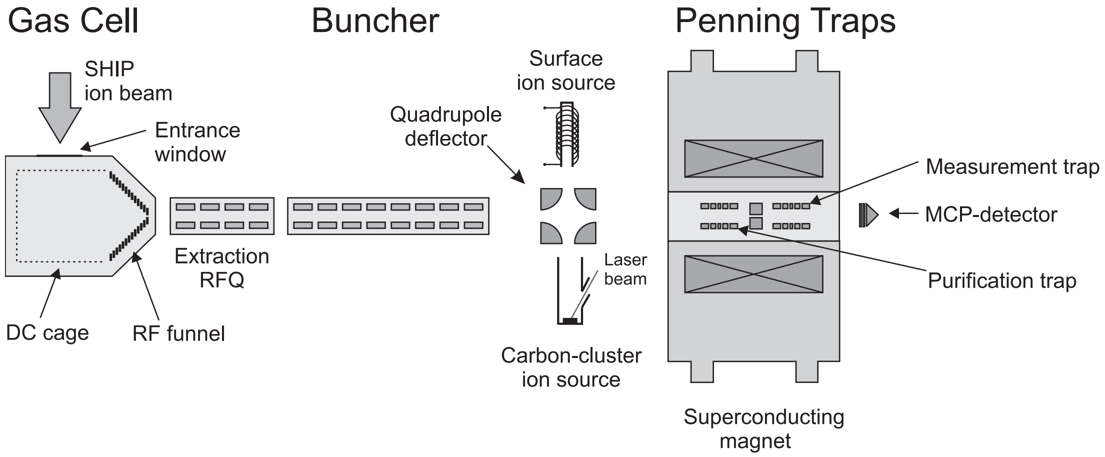

Fig. 1. Schematic overview of the SHIPTRAP facility at GSI.

of a few 100 keV/u, and stopped and thermalized inside a He buffer-gas stopping cell at a pressure of 50 mbar [19]. The ions are extracted from the stopping cell by a combination of gas flow and electrical fields. The subsequent section is an extraction RF quadrupole (RFQ) which serves for differential pumping and also operates as an ion guide. It transports the ions to the next section, a helium-gas filled RFQ cooler and buncher. The ions are cooled, accumulated and then extracted as a low-emittance bunched beam which enhances the efficient injection of ions into the Penning traps. Two cylindrical Penning traps within a 7 T super-conducting magnet are used for the high-precision mass measurement. The inner diameter of the cylindrical traps is 32 mm, the distance between the trap centers is 20 cm. They are separated by a diaphragm for differential pumping to ensure a pressure $<10^{-7}$ mbar at the measurement trap even if the purification trap is filled with helium at a pressure of about 10^{-5} mbar.

The ions are injected into the first Penning trap where the sample is purified by mass-selective buffer gas cooling [20]. The selected ions are transferred into the measurement trap where the cyclotron frequency is measured by a time-of-flight detection method [21,22]. The value of the cyclotron frequency

$$\nu_c = \frac{1}{2\pi}\,\frac{qB}{m_{ion}}, \tag{1}$$

can be used to determine the charge-to-mass ratio q/m_{ion} of the ion if the magnetic field strength B is known. To this end the cyclotron frequency ν_c of the ion of interest is compared to the cyclotron frequency $\nu_{c,ref}$ of a reference ion with well-known mass:

$$\frac{m_{ion}}{m_{ion,ref}} = r\left(\frac{q}{q_{ref}}\right) \tag{2}$$

where

$$r = \frac{\nu_{c,ref}}{\nu_c} \tag{3}$$

is the cyclotron frequency ratio. The primary result of the mass measurement is expressed in terms of the measured frequency ratio r, since the knowledge of the value of the reference mass may change over time. Typically at SHIPTRAP ^{85}Rb$^+$ and ^{133}Cs$^+$ are used as reference ions, which are produced by a surface ion source. In the case where a carbon cluster $^{12}\mathrm{C}_n^+$, $n = 1, 2, 3, \ldots$ is used as reference, the mass determination is absolute as the mass of ^{12}C is equal to 12 u by definition.

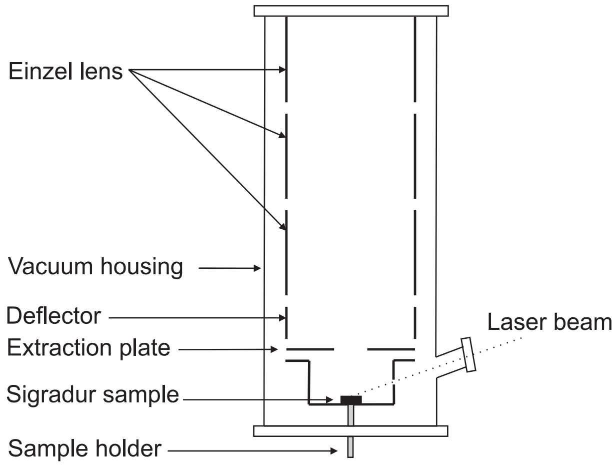

Fig. 2. Schematic diagram of the carbon-cluster laser ion source.

2.2 The carbon-cluster laser ion source

The carbon-cluster laser ion source consists of a sample holder, an extraction electrode, an einzel lens and an Nd:YAG laser (Fig. 2). The main features of this ion source are a high ion-transport efficiency, the possibility of the simultaneous use of different samples, fast exchange of the sample and a rotatable sample holder to change the laser-spot position of the sample.

In the present study, commercially available Sigradur® has been used as target material for the production of carbon clusters by laser-induced desorption, fragmentation and ionization. Sigradur® is pure carbon

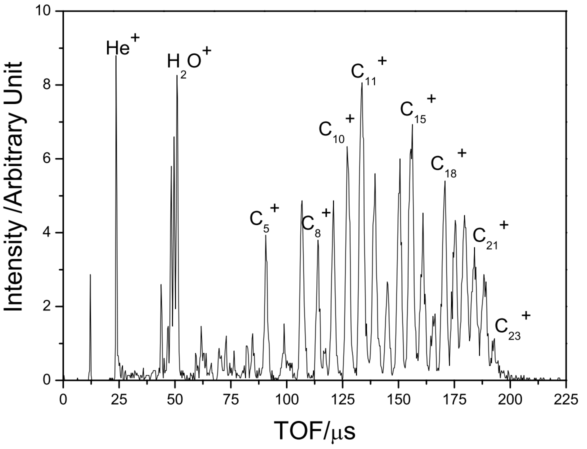

Fig. 3. A typical time-of-flight (TOF) mass spectrum of singly charged carbon-cluster ions and some contaminants detected after trapping and cooling in the purification trap of SHIPTRAP. There was no dipolar or quadrupolar excitation applied in this particular example. The storage time was 380 ms.

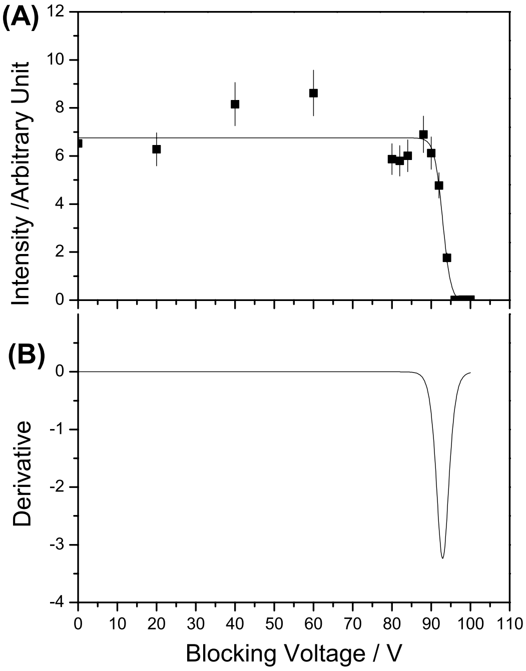

Fig. 4. Number of ions observed per laser pulse as a function of the blocking voltage applied to a copper grid in front of the detector. The solid line (A) is a fit to the data points, (B) the derivative of the fit.

synthesized from phenolic resin, which has fullerene-related micro structures [23]. It has been already used for the production of carbon clusters [24]. A Sigradur® sample (disc-shaped, 10 mm diameter, thickness 2 mm) is placed upon the rotatable sample holder. A frequency-doubled beam of an Nd:YAG laser is focussed off-center on the rotating sample, such that a single sample can be used for a long time. One laser pulse is directed on the sample per measurement cycle i.e. typically every second. The pulse duration is 3–5 ns and the typical pulse energy is 4–12 mJ. The beam is focussed to about 1 mm diameter at the target position by a telescope lens system. The sample is kept at an electrical potential of 60 V with respect to ground, which corresponds to the energy of ions coming from the SHIPTRAP RFQ buncher. The source is installed in front of a quadrupole deflector (Fig. 1) such that the source of ions can be easily selected from the gas cell, the carbon-cluster ion source or the surface ion source. The carbon-cluster beam is bent 90° by the quadrupolar deflector and the ions are injected into the purification Penning trap. They are captured in flight and cooled by collisions with the helium buffer-gas atoms. The large mass acceptance of the capturing process in the purification trap allows to capture several different masses at once. To identify the captured cluster ions, the ion sample is ejected from the first trap towards an MCP detector. A typical time-of-flight (TOF) mass spectrum of singly-charged carbon-cluster ions and some contaminants as recorded on a MCP detector is shown in Figure 3. There was no dipolar or quadrupolar excitation applied to the purification trap in this particular example. The storage time was 380 ms. The spectrum shows mainly carbon clusters $^{12}\mathrm{C}_n$, $5 \leq n \leq 23$ which agrees with an earlier measurement [24]. He^+ and $\mathrm{H_2O}^+$ were identified as main contaminants.

A test set-up, where a copper grid was mounted between the cluster source and an MCP detector, was used to measure the energy spread of the cluster ions after production. For this experiment, the sample was kept at an electrical potential of 90 V with respect to ground. The energy distribution of the ions was obtained by applying a blocking voltage to the copper grid. The solid line in Figure 4A represents a fit of a Boltzmann function to the experimental data points. The derivative of the fit is shown in Figure 4B. The energy spread of the ions extracted from the cluster source as given by the FWHM of the peak in the derivative is 3.4 eV.

As mentioned in Section 2.1, the cooling and centering technique in the purification trap is mass selective [20], hence cluster ions of a specific size can be selected. The number of extracted ions as a function of the quadrupolar excitation frequency, for an excitation time of 200 ms and for the cluster species $^{12}\mathrm{C}_{11}^+$ is shown in Figure 5. The mass resolving power was about 70 000 in this example.

The selected cluster is then transferred to the measurement trap. The time-of-flight technique [21,22] was employed to measure the cyclotron frequency. An example of a typical time-of-flight ion cyclotron resonance is shown in Figure 6. Here the variation of the mean TOF as a function of the applied quadrupolar excitation frequency (duration 900 ms) is plotted. The solid line is a least-square fit of the expected line shape [25] to the data points. The resolving power in this case was about 830 000. The cyclotron frequencies of the measured carbon cluster ions were typically determined with a relative uncertainty of 2×10^{-8}.

All the carbon clusters studied in the present investigation were composed of the most abundant carbon isotope

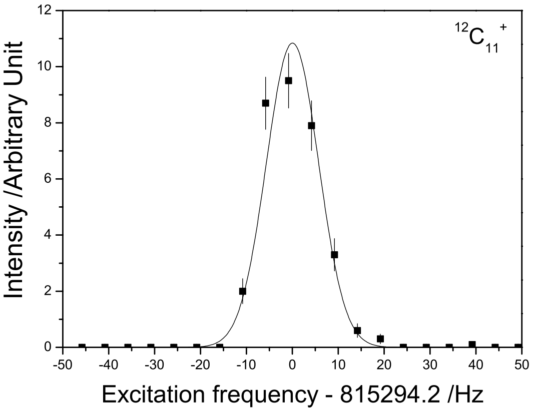

Fig. 5. Number of carbon-cluster ions $^{12}C_{11}^{+}$ recorded at the MCP detector after ejection from the purification trap as a function of the excitation frequency. The solid line depicts a Gaussian fit to the data points. The duration of the cyclotron excitation was 200 ms.

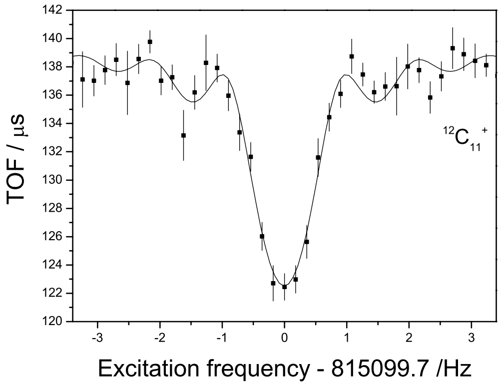

Fig. 6. Time-of-flight of $^{12}C_{11}^{+}$ as a function of the excitation frequency applied in the measurement trap for an excitation time of 900 ms. The solid line represents a fit of the expected resonance curve [25] to the data points.

^{12}C only, so after correction for the missing electron mass their masses are multiples of the unified mass unit. The estimated values of the molecular binding energy/atom of the carbon clusters varies from 3.1 eV in $^{12}C_2$ to 7.0 eV in $^{12}C_{60}$ [26], hence it is negligible at the present uncertainty level for mass measurements at SHIPTRAP.

3 Investigation of the systematic uncertainty of the SHIPTRAP mass spectrometer

3.1 Cross-reference measurements

To investigate the achievable precision of a mass spectrometer and to reveal possible systematic uncertainties, ions with well-known masses covering a large mass range are required. Cluster ions are well suited for such studies since they provide reference ions over the full mass range covered by SHIPTRAP with a maximum spacing of only 12 atomic mass unit. Moreover, the use of carbon-cluster ions eliminates the uncertainty in the mass of the reference ions as long as the binding energies are small compared to the level of precision. Hence, the true value of the frequency ratios is exactly known, if carbon-cluster ions are measured using other carbon-cluster ions as reference.

More than 100 such cross-reference measurements have been performed at SHIPTRAP. For each individual TOF resonance, about 3000 ions were recorded in measurements between 15 minutes and one hour depending on the production rate of the cluster species under investigation. The duration of the quadrupolar excitation in the

Table 1. Matrix of cross-reference measurements. The clusters given in the first column were taken as 'reference ion' and clusters in the first row were taken as 'ion of interest'. A pair of cluster sizes is marked by '*' in case a cross measurement was performed.

	$^{12}C_{10}^{+}$	$^{12}C_{11}^{+}$	$^{12}C_{12}^{+}$	$^{12}C_{15}^{+}$	$^{12}C_{18}^{+}$	$^{12}C_{19}^{+}$	$^{12}C_{20}^{+}$
$^{12}C_{7}^{+}$		*	*				
$^{12}C_{9}^{+}$	*		*	*			
$^{12}C_{10}^{+}$		*	*				*
$^{12}C_{11}^{+}$			*		*	*	*

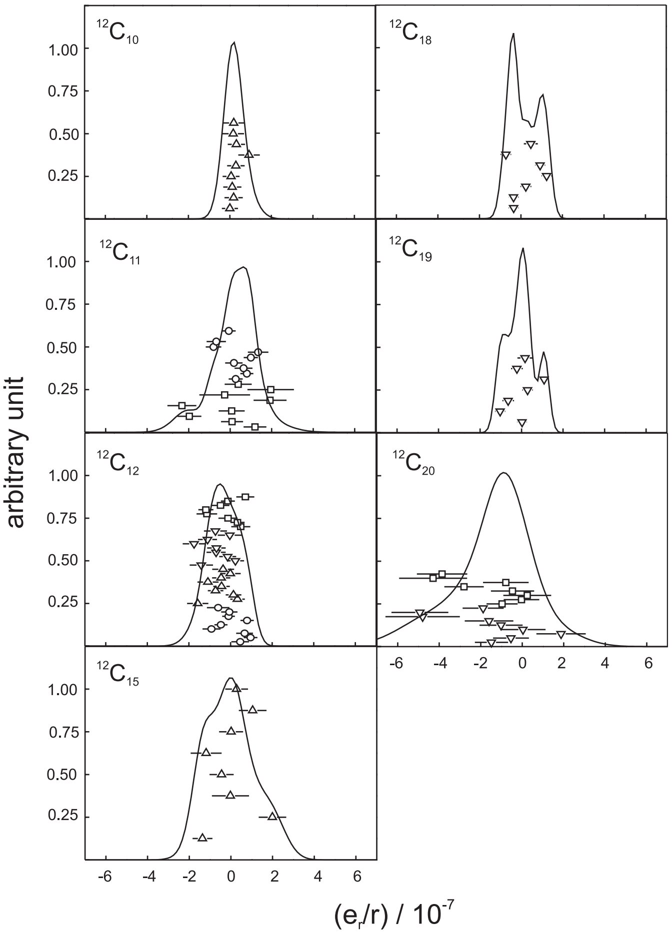

Fig. 7. Ideograms of all carbon-cluster cross-reference measurements. The measurements with different reference ions are represented by symbols: $^{12}C_{7}^{+}$ (circles), $^{12}C_{9}^{+}$ (triangles), $^{12}C_{10}^{+}$ (rectangles) and $^{12}C_{11}^{+}$ (inverted triangles).

measurement trap was 1.2 s and the relative statistical uncertainty obtained was about 2×10^{-8} for each measurement. A matrix of the clusters which have been compared in the cross-reference measurements is shown in Table 1. The results of the entire set of cross-reference measurements are represented in the ideograms [27] shown in Figure 7. Each experimental data point in the plot represents a Gaussian with a central value

$$e_r/r = (r_{measured} - r_{true})/r_{measured}, \tag{4}$$

a width $\delta(e_r/r)$ and an area proportional to $1/\delta(e_r/r)$ where $\delta(e_r/r)$ is the uncertainty of the relative deviation. The ideogram of a cluster species is the sum of all such Gaussians associated with the individual cross-measurement, where that particular cluster was treated as the 'ion of interest'. Different symbols are used in the ideograms to indicate different 'reference ions'. The ideograms show that the distributions of the individual measurements are nearly Gaussian where a sufficient number of measurements was performed.

3.2 Mass-dependent systematic effect

A cyclotron-frequency shift $\delta\nu$ can arise for instance from the imperfection of the electric-quadrupole field or from a misalignment of the axis of the electrostatic trapping field and the magnetic-field axis in a Penning trap. The frequency shift $\delta\nu$ gives rise to an error in the calibration of B with a reference ion. The relative error in the mass determination of an ion is $\delta m/m = (\delta\nu/\nu)((m - m_{ref})/m)$ [3,21]. This mass-dependent effect can be studied using ions of well-known masses and large mass difference between ion of interest and reference ion. The carbon-cluster cross-reference studies have been performed to investigate mass-dependent systematic effects at SHIPTRAP.

The results of the cross-reference measurements are shown in Figure 8. Each data point represents the weighted mean of all individual measurements between two particular cluster species. A relative time-dependent uncertainty of 3×10^{-9} per hour due to the non-linear magnetic field fluctuation [28] was quadratically added to the statistical uncertainty of each frequency ratio. In case of more than one measurement of one cluster species, the uncertainty obtained is,

$$\delta r = \frac{1}{\sqrt{\sum_i 1/\sigma_i^2}} \tag{5}$$

where σ_i is the uncertainty of the individual measurement.

The mass-dependent uncertainty has been found to be in agreement with zero for small mass differences with $(m - m_{ref}) < 100$ u. The $n = 20$ carbon cluster measurements suffer from a large scattering of the measured cyclotron frequencies as can be seen in Figure 7. This might be the reason for the large deviation observed for the $(m - m_{ref}) > 100$ u frequency ratios. The deviation from zero for larger mass differences is presently under further investigation.

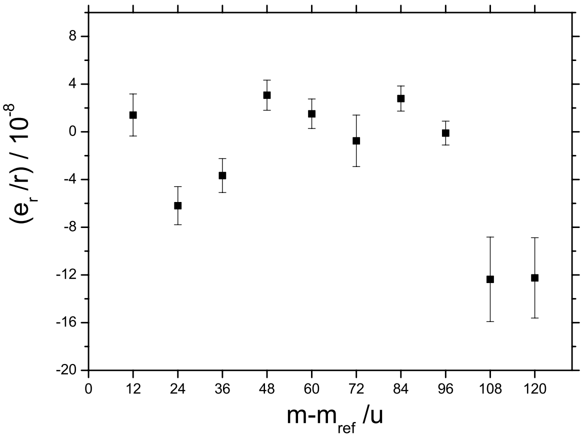

Fig. 8. Deviation of the weighted mean of the frequency ratios from the true values as a function of $(m - m_{ref})$.

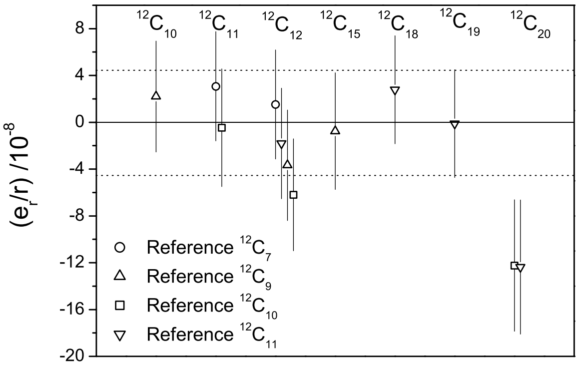

Fig. 9. Deviation of the weighted mean of the frequency ratios of different carbon clusters. The dashed lines indicate the systematic uncertainty, which was added quadratically for each measurement.

3.3 Systematic uncertainty

The reduced χ^2 of the distribution of the mean frequency ratio for the carbon-cluster species used in the cross-reference measurements was calculated to be $\chi^2/N = 5.5$ where

$$\chi^2 = \sum_i \left[\frac{e_r^i}{\delta r^i}\right]^2 \tag{6}$$

and N is the total number of cross measurements. This high value might be due to the presence of a systematic uncertainty δr_{sys}.

The total uncertainty of a frequency ratio can be obtained by

$$\delta r_{total} = \sqrt{(\delta r)^2 + \left(r\frac{\delta r_{sys}}{r}\right)^2}. \tag{7}$$

The one-sigma value of the systematic uncertainty was determined using equations (5), (6) and (7) to be

$$\frac{\delta r_{sys}}{r} = 4.5 \times 10^{-8} \tag{8}$$

Table 2. Summary of the results of the on-line mass measurements of ^{144}Dy, ^{146}Dy and ^{147}Ho. Column one: measured ion; column two: half-life of the radionuclei; column three: reference ion used for the mass calibration; column four: number of recorded scans N_s; column five: total number of ions N_{ion} recorded in all scans; column six: measured frequency ratios $r = \nu_c/\nu_{ref}$ with their statistical (first bracket) and total uncertainty (second bracket); column seven: relative total uncertainty and column eight: resulting mass excess (and uncertainty).

Ion	$T_{1/2}$	Reference	N_s	N_{ion}	Frequency ratio $r(\sigma_{stat})(\sigma_{total})$	$(\delta r/r)/10^{-8}$	Mass excess/keV
^{144}Dy^{2+}	9.1 s	^{12}C$_{11}^{+}$	3	2354	0.545222618(15)(29)	5.3	–56570.5(7.1)
^{144}Dy^{2+}	9.1 s	^{85}Rb^{+}	6	3322	0.847580193(28)(45)	5.3	–56570.1(7.1)
^{146}Dy^{2+}	33.2 s	^{12}C$_{11}^{+}$	5	6076	0.552774142(13)(28)	5.1	–62536.5(6.9)
^{146}Dy^{2+}	33.2 s	^{85}Rb^{+}	13	13372	0.859319365(22)(43)	5.0	–62554.7(6.7)
^{147}Ho^{2+}	5.8 s	^{12}C$_{11}^{+}$	4	14661	0.556589680(7)(26)	4.7	–55738.7(6.4)
^{147}Ho^{2+}	5.8 s	^{85}Rb^{+}	9	11078	0.865250860(24)(44)	5.0	–55753.6(6.9)

such that χ^2/N is approximately one. The frequency-ratio deviations after adding this systematic uncertainty are shown in Figure 9, i.e. the deviations of the weighted means of the frequency ratio from their true values is plotted for different carbon clusters which were taken as the 'ion of interest' in the cross-reference measurements.

4 Carbon-cluster ions for on-line mass calibration

After extensive off-line mass comparisons between ^{133}Cs^{+}, ^{85}Rb^{+} and ^{12}C$_{n}^{+}$, carbon-cluster ions were tested for the first time as reference ions in an on-line mass measurement. The investigated radionuclides were produced in the reaction ^{92}Mo (^{58}Ni, xpyn) at SHIP with primary beam energies between 4.36 and 4.6 MeV/u. The masses of the radionuclides ^{144}Dy, ^{146}Dy and ^{147}Ho were measured using ^{12}C$_{11}$ as reference.

It should be mentioned that the cluster ion source was tuned for the production of ^{12}C$_{11,12}^{+}$ since singly-charged ions were expected from the gas stopping cell. During the beam time it turned out that a large fraction of doubly-charged ions were extracted due to the improved cleanliness of the gas cell. Hence, they were used for the mass measurement. Thus, the carbon-cluster ions did not match the m/q values of the ion of interest. The radionuclides were also measured using ^{85}Rb^{+} as reference ion during the same beam time [29]. The results agree with those where ^{12}C$_{11}^{+}$ was used as reference ion. Table 2 summarizes the results and gives the mass excesses $ME = m - Au$ (where A is the atomic mass numbers and u is the unified mass unit).

The difference between the mass excesses of the investigated radionuclides as measured at SHIPTRAP and the values given in the literature (AME 2003) [13] is plotted in Figure 10. The measured mass excesses of ^{144}Dy and ^{146}Dy agree with [13] which were mainly influenced by the values measured at GSI-ESR [30]. The measured mass excess of ^{147}Ho deviates from the previous value [13,30]. Isomeric contamination in any one of the experiments could cause this deviation but the exact reason is not yet understood [29]. However, the mass values obtained at SHIPTRAP with different reference ions agree with each other.

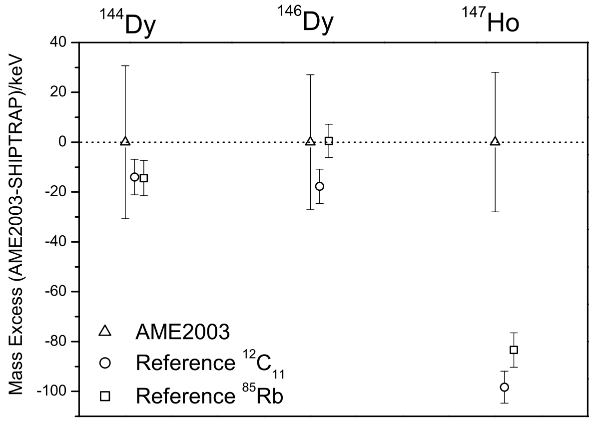

Fig. 10. The mass excess of ^{144}Dy, ^{146}Dy and ^{147}Ho measured with ^{85}Rb reference, measured with ^{12}C$_{11}$ reference, and as given by AME 2003 [13].

5 Summary and outlook

The present results show that carbon clusters are useful as reference ions for on-line mass measurements. Absolute mass measurements of the radionuclides ^{144}Dy, ^{146}Dy and ^{147}Ho were performed using ^{12}C$_{11}^{+}$ as reference ion. A relative mass uncertainty of 5×10^{-8} was obtained. The results agree with measurements performed in the same run using ^{85}Rb^{+} as reference ion.

By cross-reference measurements between carbon-cluster ions from ^{12}C$_{7}^{+}$ to ^{12}C$_{20}^{+}$ the present uncertainty limit of the SHIPTRAP mass spectrometer was investigated. The mass-dependent uncertainty was found to be in agreement with zero for the case of $(m - m_{ref}) < 100$ u. The systematic uncertainty of 4.5×10^{-8} is presently further investigated. In addition, further measurements with carbon clusters and other reference ions are foreseen. Especially the cases of large mass differences between reference ion and ion of interest shall be revisited.

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