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Multicollision-induced dissociation of multiply charged gold clusters, Au_n^{2+}, $n = 7–35$, and Au_n^{3+}, $n = 19–35$

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Abstract

Multicollision-induced dissociation (MCID) has been applied to gold clusters, Au_n^{2+} ($n = 7–35$) and Au_n^{3+} ($n = 19–35$) stored in a Penning trap. By application of ion cyclotron resonance excitation and pulses of argon collision gas, fragmentation yields have been measured as a function of the clusters' kinetic energy. The corresponding dissociation energies have been determined by use of the impulsive collision theory and the quantum Rice–Ramsperger–Kassel (RRK) model for the energy transfer to internal cluster modes and for delayed dissociation, respectively. As compared to earlier measurements of singly charged gold clusters the variation of the stability as a function of cluster size is reduced. The doubly charged clusters show an odd–even effect that is reversed with respect to that of singly charged gold clusters. This is similar to findings by electron impact ionization/dissociation and in line with the expectations for simple metal clusters, where the structure and stability is governed by the number of atomic valence electrons. However, no cluster sizes of particular stability (magic numbers) are observed. In general, the dissociation energy of small clusters is smaller than that of the larger ones because of the influence of the Coulomb force. In contrast to the singly charged gold clusters the odd–even effect of Au_n^{2+} disappears at small cluster sizes $n < 11$; explained as a consequence of the dominance of trimer fission in that size region. (Int J Mass Spectrom 202 (2000) 47–54) © 2000 Elsevier Science B.V.

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1. Introduction

The question of how their properties vary as a function of size is the main theme of atomic and molecular cluster research. The variation of their charge state adds a new dimension to the investigations because such measurements can lead to further insight with respect to their structure. Thus, there is

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considerable interest in the field of multiply charged atomic clusters [1]. Among the clusters, metal clusters are a special subset because of their close conceptual relation to atomic nuclei [2,3]. Several concepts that had been developed in nuclear physics (like the liquid drop model or the determination of fission barriers) have been adapted for the description of multiply charged metal clusters, as recently reviewed by Näher et al. [4]. After initial studies of the Konstanz group on multiply charged clusters of different species [5], much of the pioneering work on metal clusters, both with respect to the experimental investigations as well their interpretation, has been performed by Saunders [6,7]. He focused his studies on the case of gold and by use of a triple-quadrupole mass spectrometer he was able to investigate the collision induced dissociation of size-selected cluster ions.

Most investigations on multiply charged metal clusters, however, have concentrated on the abundance spectra that result from the respective ion sources (liquid metal ion source [7], laser vaporization source with integrated field ionization [8], or sputtering techniques [9,10]). Often the initial clusters are neutral and not size selected before ionization (as e.g. by electrons [11,12], photons [13,14], or the interaction with highly charged atomic ions [15]). Thus, clusters of various sizes and charge states are produced simultaneously, which nevertheless yield interesting information on, e.g. the appearance size, the smallest cluster size observed for a given charge state. Only a few experiments have been performed on size-selected multiply charged metal clusters, in particular by Saunders, as already mentioned above [6,7] and by Bréchignac and co-workers (see e.g. [16]) that allow the determination of decay pathways and dissociation energies [17]. For a comprehensive review the interested reader is referred to the above-mentioned review [4].

At the Mainz cluster trap [18,19] electron impact ionization has been adopted for the production of multiply charged metal clusters inside a Penning trap where they are stored for further investigation. Storage provides the possibility of further preparatory steps, like the selection of defined cluster sizes, before the experiments of interest are carried out [20]. Results with respect to the dissociation pathways of multiply charged gold clusters have been presented recently [21]. In addition, the abundance pattern of gold clusters of different charge states as produced by electron induced ionization/dissociation of size-selected gold clusters have been investigated [22]. The present article complements these findings with a closer look at the energetics of the decays and relates the observed decay channels to the stabilities of the corresponding precursor clusters and fragments.

Because the experimental procedure and the data evaluation have been described in detail recently for the case of silver [23,24], their present description (Secs. 2 and 3) is restricted to a brief overview. In Sec. 4 the results for multiply charged gold clusters are given, and then they are discussed in Sec. 5.

2. Experimental setup and procedure

The cluster ions are produced in a Smalley type [25] external source by laser vaporization of a metal wire in a helium gas pulse that is expanded through a nozzle into the vacuum [26]. The (singly charged) cationic species are guided to a Penning trap [27] where they are captured in flight [28]. Several cluster ion pulses are accumulated [29] before the ensemble is irradiated by an electron beam (of an energy of about 200 eV) for the production of multiply charged clusters [30, 31]. The clusters of n/z ratio of interest—where n denotes the number of constituent atoms and z is the charge state—are selected by radial ejection of all other species by excitation of their cyclotron motion. After these preparatory measures the cyclotron radius of the selected cluster ions is increased by single-frequency ion cyclotron resonance (ICR) excitation and argon gas is pulsed into the trap [29]. In a multicollision process kinetic energy is converted into the internal energy of the clusters that leads to their dissociation. Finally, the resulting ensemble of stored ions is axially ejected from the trap into a drift section for time-of-flight (TOF) mass analysis [18]. Single ion detection is performed by use of a conversion electrode detector.

For Fig. 1 the experimental sequence has been

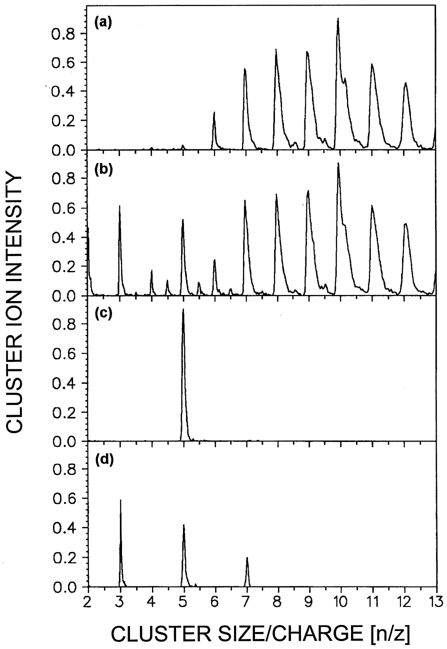

Fig. 1. TOF spectra demonstrating the experimental sequence. From top to bottom: (a) after capture and accumulation of Au_{10}^{+}, (b) after electron impact ionization/dissociation, (c) after selection of clusters with $n/z = 5$, and (d) after collision induced dissociation.

terminated after a given event by ejection of the ions for TOF mass analysis. The resulting spectra demonstrate the sequence: (a) capture and accumulation of Au_{10}^{+}, (b) electron impact ionization/dissociation, (c) selection of Au_{10}^{2+}, and (d) collision-induced dissociation. In this specific example Au_{10}^{2+} decays to $Au_7^+ + Au_3^+$. This "trimer fission" is the dissociation pathway of most small multiply charged gold clusters; the larger ones typically evaporate a neutral monomer [21].

Note that the ejection of the unwanted n/z clusters may not always result in a unique cluster species. In general, the clusters Au_{3n}^{3+}, Au_{2n}^{2+}, and Au_n^+ will contribute to the same signal in the mass spectra. Thus, in the case shown in Fig. 1, the Au_{10}^{2+} signal coincides with that of Au_5^+. However, this does not constitute a severe problem because the respective decay pathways are not the same and the decay pathways of the singly charged species are well known [32]. Even in the example of Fig. 1 where both Au_{10}^{2+} and Au_5^+ decay to Au_3^+, the second decay product of Au_{10}^{2+} is Au_7^+, and thus has a unique origin. (Note that its n/z value is larger than that of the precursor.) As a matter of fact, the Au_{10}^{2+}/Au_5^+ example with both precursors decaying to Au_3^+ is one of the most difficult cases. In general, the distinction is much easier. For example, all doubly charged odd-size clusters have no contamination by singly charged clusters. Furthermore, for the large clusters the ranges of the different charge states are well separated [22,30]. Thus, in most cases the decay of the precursor may be monitored to determine the fragmentation yield (Sec. 4).

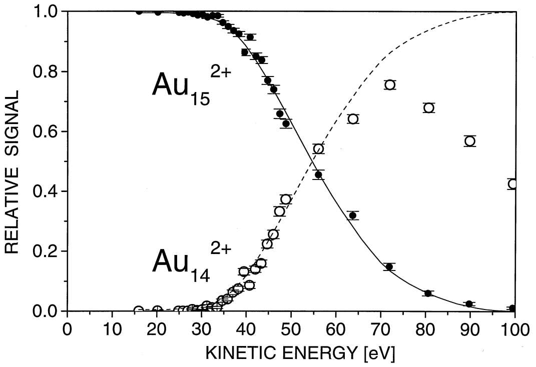

Fig. 2. Experimental values of the relative signal intensities of the precursor Au_{15}^{2+} (full symbols) and the first product Au_{14}^{2+} (open symbol) after MCID as a function of the kinetic energy. The full line indicates the best fit to the Au_{15}^{2+} data. The broken line indicates the corresponding intensity of all dissociation products.

Typically 20 to 40 ions are averaged for each experimental sequence. The sequence is repeated to improve the statistical significance of the data and 25 spectra are added for a given set of experimental parameters. For the investigation of a given cluster species the ICR excitation amplitude of the multicollision induced dissociation (MCID) is varied and the corresponding intensity of the precursor and the fragments is determined for about 20 to 30 excitation values. For the case of Au_{15}^{2+} Fig.2 shows the relative signal intensities of the precursor and the first product, Au_{14}^{2+}, as a function of the clusters' kinetic energy. With increasing excitation the precursor clusters are converted to Au_{14}^{2+} by neutral monomer evaporation. At high excitation amplitudes the internal energy becomes high enough for sequential decays and smaller clusters appear. Thus, as the excitation amplitude is raised, the Au_{14}^{2+} intensity shows a maximum before it decreases again.

3. Data evaluation

The determination of the dissociation energies from the fragmentation yield as a function of ICR excitation amplitude is based on two main ingredients: (1) an evaluation of the fraction of the kinetic energy that is converted into the internal energy of the clusters by the collisions and (2) a description of the decay rate of the cluster of interest at a given internal energy.

For the first step (i.e. the conversion of kinetic into internal energy) the impulsive collision theory [33,34] is employed [23]: Each inelastic collision of the cluster is described as an elastic collision of one of its constituents. It can be shown that the ratio of the energy transferred to internal cluster modes and the loss of the cluster's kinetic energy is a constant independent of any collision parameter. Thus, when the cluster is stopped due to many collisions and has lost all its kinetic energy, this ratio gives the final internal energy due to collisions. To this value the initial energy (at room temperature) is added to calculate the total excitation energy. Because of the random motion of the collision gas atoms there is a Doppler broadening of collision energies [35]. Finally, whereas the collision is modeled under the assumption of a quasifree cluster atom colliding elastically, any real system will have some binding between the cluster atoms (or, after all, the cluster would not be a stable species). The more tightly the atoms are bound the more the energy transfer will be overestimated. As discussed by Jarrold for the case of silicon clusters [36], this effect is accounted for by the introduction of a correction factor. In analogy with the silver cluster investigations [23] this factor (0.422) has been found by comparison of the MCID data for the singly charged gold cluster Au_{15}^{+} with its dissociation energy [3.32 (5) eV] as determined by time resolved photodissociation [37].

For the second step (i.e. the calculation of the expected fragmentation yield within the experimental time window for a given cluster species, excitation amplitude and dissociation energy) the quantum version of the Rice–Ramsperger–Kassel (RRK) model has been employed [38]: The cluster is modeled as a system of coupled harmonic oscillators where the excitation energy is distributed randomly and continuously reshuffled (at the Debye frequency of 3.54×10^{12} Hz [39]). The probability that the energy accumulated in one mode exceeds the dissociation energy determines the expected fragmentation yield.

This two-step procedure is described in detail for the case of singly charged silver clusters [23] and has been applied to doubly charged silver clusters, as well [24,40]. The dissociation energy is treated as a free parameter that is fitted to the experimental data via a comparison between the theoretical fragmentation yield as a function of cyclotron excitation and the observed cluster abundance (full line in Fig. 2 for the case of Au_{15}^{+}). As compared to the case of silver, the data evaluation is facilitated by the fact that gold has only one stable isotope. Thus, all clusters of a given n/z ratio experience the same excitation of their cyclotron motion, whereas for silver clusters the distribution of isotopomers has to be taken into account [23].

In light of recent time-resolved measurements of photo-induced dissociation of stored vanadium clusters [41] it is noted that the present MCID data evaluation does not include a possible radiative cluster cooling. In its presence the MCID excitation would have to be increased to result in the observed fragment yield and thus the dissociation energy would appear larger than it actually is. However, preliminary results on the photodissociation of gold clusters indicate that the radiative cooling plays no significant role [42].

4. Experimental results

4.1. Doubly charged gold clusters

The measurements have been performed for doubly charged gold clusters of size $n = 7–35$. Smaller

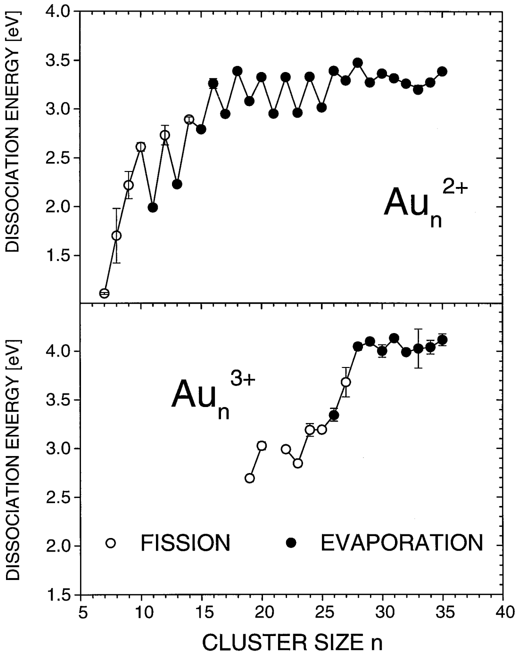

Fig. 3. Dissociation energies of doubly (top) and triply charged gold clusters (bottom) as a function of cluster size. Full symbols: monomer evaporation. Open symbols: fission. The error bars include the statistical uncertainties as well as systematic effects due to the assumption of the inital temperature (300 K ± 50 K) and of the rearrangement frequency in the RRK modeling (varied by a factor of 2).

clusters, Au_5^{2+}, were observed in the Penning trap; the signal intensity was not sufficient for further studies. (By other means, even Au_2^{2+} has been observed [43] and its (meta-)stability investigated theoretically [44,45].) For $n \geq 16$ the ranges of doubly and singly charged clusters were well separated and the decrease of the precursor signal as a function of ICR excitation amplitude was used for the determination of the dissociation energy. Similarly, the small odd-size clusters Au_n^{2+}, $n = 7, 9, 11, 13$, and 15, can be selected unequivocally and have been treated the same way. For the case of fission, i.e. for $n = 8, 10, 12$, and 14, the signal intensity of the heavy fission product, Au_{n-3}^+, was fitted.

The resulting dissociation energies (Fig. 3, top) show an increase as a function of cluster size that is rather steep for small clusters and then levels off to reach a plateau of about 3.2 eV above $n = 16$. From roughly $n = 10$–30 there is an odd–even staggering superimposed on the general behavior: In this size range the dissociation energy of even-size clusters is significantly higher than that of their odd-size neighbors.

4.2. Triply charged gold clusters

In analogy to the dissociation energies of doubly charged gold clusters, the values for triply charged gold clusters have been determined. Again, small clusters (down to Au_{16}^{3+}) were observed, but only in very small quantities. The clusters available for MCID were restricted to the sizes $n = 19, 20$, and 22–35. The case of Au_{21}^{3+} was inaccessible due to contamination with the clusters of equal size over charge ratio, Au_{14}^{2+} and Au_7^+.

The dissociation energies of other "contaminated" clusters ($n = 24, 27, 30$, and 33) have been fitted to the yields of the decay products. Au_{24}^{3+} and Au_{27}^{3+} result (by trimer fission) in Au_{21}^{2+} and Au_{24}^{2+}, respectively; Au_{30}^{3+} and Au_{33}^{3+} decay (by neutral monomer evaporation) to Au_{29}^{3+} and Au_{32}^{3+}, respectively. For all other cluster sizes the fits have been performed on the yields of the decaying precursor clusters.

In the bottom of Fig. 3 the resulting dissociation energies are shown as a function of cluster size. The dissociation energy increases up to about $n = 28$, where it levels off. In contrast to the case of the doubly charged gold clusters there is no apparent odd–even effect.

5. Discussion

In contrast to related investigations on metal clusters there are no cluster sizes that appear particularly stable, i.e. there is no indication of "magic numbers." In early CID measurements on singly charged gold clusters with the Mainz cluster trap [46] the cluster sizes $n = 3, 9$, and 19 showed an increased relative stability in agreement with the expectations for clusters of simple metals [47–49]: The valence electrons of the constituent atoms are expected to be delocalized and to fill the energy levels of the combined Coulomb potentials of the remaining ionic cores.

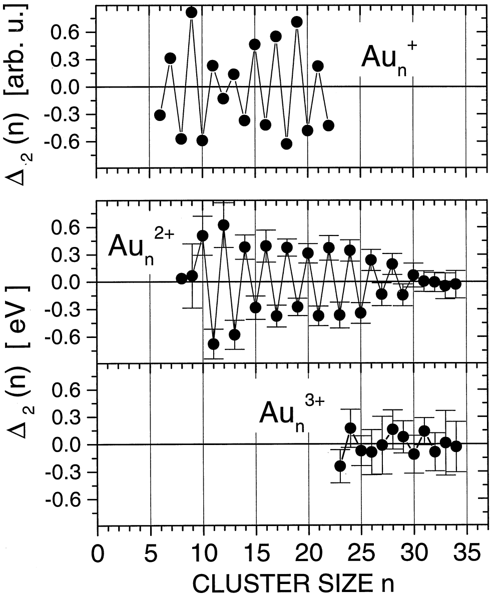

Fig. 4. Odd–even energy $\Delta_2(n)$ (see text) of singly (top [46]), doubly (middle, this work), and triply charged gold clusters (bottom, this work) as a function of cluster size, n. (Due to different experimental conditions the values for Au$_n^+$ cannot be compared quantitatively with the present results and are thus given in arbitrary units.)

According to the Pauli principle this leads to electronic shell structures. Thus, the relevant number is the number of free atomic valence electrons, i.e. the cluster size minus the charge state. A recent comparison of the dissociation energies of singly and doubly charged silver clusters is in full agreement with this picture [24].

Whereas gold, in contrast to its homologue, shows no obvious indication of magic numbers of the doubly charged clusters, there is one prominent effect: an odd–even staggering. In order to better visualize and compare this phenomenon with respect to different charge states, the difference between the dissociation energy at a given cluster size and the average of the values of its next neighbors (upper and lower) is displayed in Fig. 4 (“odd–even energy”). The top part shows the results of the earlier measurements for singly charged clusters [46]. The middle and bottom parts show the present results for doubly and triply charged clusters, respectively.

As mentioned above, there is no odd–even effect in the case of the triply charged clusters. As for the doubly charged ones, even-size clusters are much more stable than odd-size ones. The largest effect is found around $n = 10–13$ and it decreases in steps at $n = 14$, 26 and 30, above which it has disappeared. Below $n = 10$ there are presently available only the values of $n = 8$ and 9, which are very small.

In contrast, the odd–even effect of singly charged gold clusters is very large at $n = 9$, small around $n = 12$, and increases up to $n = 19$, above which it seems to decrease again. Most obvious, however, is the inversion in sign between the odd–even effect of singly and doubly charged gold clusters. This behavior is analogous to the silver measurements mentioned above [24] and is, after all, indicative of electronic structure effects. It has also been observed in the abundance spectra after electron impact ionization/dissociation of size-selected metal clusters for both the elements silver [50] and gold [22].

As pointed out before [24], the disappearance of the odd–even effect at low cluster size is a natural consequence of fission: Whereas during monomer evaporation the cluster changes from an odd-electron system to an even-electron system, or vice versa, the trimer fission leaves it an odd- or an even-electron system just as the precursor was before its decay. Thus, even if there is an odd–even alternation in the total binding energy of the clusters, it is not to show up in the respective dissociation energies at cluster-size ranges where there is fission only. On the other hand, in the range where fission competes with monomer evaporation, it is energetically favored with respect to the stable even-electron systems but not with respect to the less stable odd-electron system. This results in a decay-pathway alternation, as observed for the doubly charged gold clusters Au$_n^{2+}$ between $n = 10$ and 15.

The argument of the fission-related disappearance of the odd–even effect may also be applied to the triply charged clusters Au$_n^{3+}$ with $n < 26$. In addition, note that the doubly charged clusters above $n = 29$ show no odd–even alternation either. If the origin of the decrease of the odd–even effect with increasing cluster size is assumed to be independent of charge state then there are hardly any cluster sizes left where Au$_n^{3+}$ could show an odd–even effect. Thus, its absence is well in line with the other observations.

6. Conclusion and outlook

Collision-induced dissociation has been applied to multiply charged metal clusters. In contrast to the case of doubly charged silver clusters, the respective gold clusters show no magic numbers for the dissociation energy as a function of cluster size. As expected, the odd–even effect reverses sign when compared to singly charged clusters.

With respect to triply charged metal clusters these are, to our knowledge, the first studies on dissociation energies. The variation of dissociation energy of Au_n^{3+} as a function of cluster size are even smaller than those for Au_n^{2+}, i.e. there are no particularly stable cluster sizes, nor any observation of an odd–even alternation.

Future investigations are to include the extension of time-resolved photodissociation studies [37,51,52] from singly to multiply charged clusters. This will allow one to improve the determination of the dissociation energies (for example, for the case of Ag_n^+ compare [23] and [52]) as well as lead to further insight with regard to the possible competition of radiative cooling [41,42].

Recently, dianionic gold clusters have been produced and stored in the Penning trap [53–55]. Thus, in addition to cationic systems, singly as well as multiply charged metal cluster anions may be investigated by either of the methods mentioned above.

In particular, it will be interesting to see whether the pattern and variation of stability as a function of cluster size, which is reduced when the positive charge state is increased, shows a similar reduction for anionic systems.

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