PHYSICAL REVIEW A **98**, 052701 (2018)

Highly selective mechanisms for the production of N_2 and O_2 dications by electron impact

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(Received 9 August 2018; published 7 November 2018)

Different mechanisms for the dication production of molecular nitrogen and oxygen are reported here. It is shown that the N_2 dication is produced by direct double ionization, or TS-2, while the O_2 dication results primarily from an inner valence shell single ionization followed by a postcollisional Auger decay. In order to unveil these different characteristics of both production pathways, absolute cross-sectional measurements for the homoisotopic ${N_2}^{2+}$ and ${O_2}^{2+}$ by electron impact with energy ranging between 200 and 900 eV were measured. The separation of the doubly ionized parent molecules from the singly ionized fragments N^+ and O^+ was possible by employing the delayed extraction time-of-flight technique. The reasons for the two pure different pathways are discussed in the light of the present data, showing that while the single-ionization dication production process is impossible for nitrogen due to the energy levels limitations of its molecular orbitals, the direct double-ionization channel is strongly inhibited for oxygen due to geometrical constraints of its molecular orbitals orientation.

DOI: 10.1103/PhysRevA.98.052701

I. INTRODUCTION

Molecular dications present in different media which are constantly energized—such as planetary atmospheres, cometary tails, plasma devices, and even the human body, when submitted to radiotherapy treatments—are the subject of many different studies [1–10]. The physical chemistry resulting from such species, due to their intrinsic tendency to fragment into two charged moieties, places them in a very important role in the resulting system [11], since the addition of Coulombic repulsion to the momentum conservation after breakup produces fragments with high kinetic energy, usually leading to kinetic energy releases (KER) of the order of tens of eV [12–14]. Phenomena like subsequent secondary reactions and molecular fragmentations, as well as temperature increases and, as a consequence, planetary escape, can be highly influenced by the presence of dications [1,15–18]. Thus, knowledge of the dynamics that govern the formation of dications of molecules such as nitrogen and oxygen, the most abundant components of the Earth's atmosphere, is paramount to provide a complete description of such environments.

The primary mechanisms leading to the dication production for each specific species are still scarcely known [19,20], and therefore a few important open questions arise, with the present data showing that there is no trivial answer to any of them:

(i) Is the metastable dication probable, in comparison to double ionization leading to fragmentation?

The answer to this question seems to be easy to achieve—one should look at the molecular dication production cross section and compare it to the other cross-section values for different ionization and fragmentation products of that particular molecule. However, for symmetric molecules, this is not simple at all. This is due to the intrinsic impossibility of separating, in a time-of-flight (TOF) spectrum obtained in a standard mass spectrometry measurement, the doubly charged parent molecule peak from the singly charged fragment corresponding to the molecule breaking up in two fragments with equal mass; for example, nitrogen or oxygen molecules have, respectively, N^+ and ${N_2}^{2+}$ or O^+ and ${O_2}^{2+}$ with the same mass-to-charge ratio (m/q). For this reason, besides a single measurement of ${N_2}^{2+}$ production by electron-nitrogen molecule collisions using a special cryogenic detector at 70-eV impact energy [21], only a few absolute cross-section data were reported for the production of N_2 and O_2 dications, all of them using heteroisotopic nuclei (${}^{14}N^{15}N^{++}$ and ${}^{16}O^{17}O^{++}$, respectively) [22–24]. This difficulty was solved with the development of the delayed extraction time-of-flight (DETOF) technique, which is able to separate fragments with the same m/q in a TOF mass spectrometry experiment [25]. With the use of the DETOF technique, homoisotopic nitrogen [26] and oxygen [27] dication productions were measured by electron impact from threshold to 400 eV.

(ii) What is the predominant pathway to produce the metastable dication?

Nevertheless, the answer to this second question was not provided by the previously reported data [26,27], due to the upper limit of the impact energy range. For all double-ionization processes, different channels could lead to the removal of the second electron. Simply putting, the doubly ionized species can be attained by (a) removing two electrons in the same collisional process—called here direct double ionization, which for electrons come mainly via the TS-2 process—in which the projectile itself transfers energy to two different electrons of the target, removing them; or (b)

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removing a single electron from inner valence shell or from the core of the target, which afterward due to a rearrangement of its molecular orbitals in the new ionized state discards a second electron, by a postcollisional—Auger, Auger-like (which transiently follows a direct inner valence shell vacancy production [28]), shake-off, etc.—process [29]. This latter production dynamics, coming from single ionization, is a process that has the same dependency with the impact energy as the production of singly ionized species; therefore, its cross section does not decrease with the increase of the impact energy when comparing it with the singly charged parent molecule. But, in order to evaluate the impact energy dependence of the doubly and singly charged species, one has to investigate up to higher values of the projectile energy.

(iii) What electrons must be removed for the metastable dication to be the end result of a collision?

The possible final states resulting in thermochemically stable, metastable, or unstable dicationic states can be known [30,31], including the predominant quantum contributions leading to these metastable final states [32]. The structure of ${N_2}^{2+}$ and ${O_2}^{2+}$ present the nitrogen dication missing two electrons coming from a bonding σ orbital while the oxygen dication is stable without its two outermost unpaired electrons from antibonding π orbitals. But in order to actually answer the third question, one has to first understand the dication production pathways so that a correspondence between the ionization site of the electrons and the final state can be inferred. The present data provide conclusive results regarding the different particularities present in the dication production processes of the nitrogen and oxygen molecule by electron impact.

II. EXPERIMENTAL SETUP

An upgrade on the insulation of the electron gun now allows collisions with higher impact energies to be accurately measured, as was the case for ethylene [29,33] and water [34,35]. The experimental setup employed here has been described in detail before [36]. An electron beam is produced by an electron gun operating here in the 200- to 900-eV energy range, and which is coupled to a gas cell with a monitored pressure of $\sim 3 \times 10^{-4}$ torr, in order to keep the system in a single-collisional regime, and a Faraday cup. Both the electron beam and an 21-V/mm electrostatic extraction field used to guide the produced positively charged ions into a TOF drift tube are synchronously pulsed with a frequency of 20 kHz and pulse durations of 50 and 100 ns, respectively. The recoil ions are afterward collected by a microchannel plate (MCP) detector controlled by a standard acquisition electronics and are time resolved by a time-to-digital converter analyzer [37]. Therefore, absolute ionization and fragmentation cross sections are obtained by measuring the number of ions collected for each process and normalizing it by the number of scattering centers of the target gas, the number of projectiles, the length of the interaction region, and the absolute efficiency of the spectrometer, which is determined by a procedure described in detail elsewhere [36].

The DETOF technique consists, in short, of varying the time delay between the electron beam and the extraction field pulses [25–27,38]. If the time delay between both pulses was zero, i.e., if the extraction field pulse was turned on immediately after the trailing edge of the electron beam pulse, then all ions formed in the collisional process would be guided by the extraction field through the collimator and into the TOF drift tube. On the other hand, for longer time delays the faster ions produced have enough free flight time to leave the extraction region before the turning on of the electrostatic field. Therefore, a gradual and systematic increase of this delay time beyond the minimum delay time t_0 after the passing of the electrons works as a velocity selector, in which only the progressively slower fragments are collected. In the particular case of the molecular dication, it retains its original Maxwell-Boltzmann velocity distribution, since the momenta carried by the ejected electrons are negligible in comparison with the molecule's. The fragment with the same mass-to-charge ratio (N^+ or O^+, for ${N_2}^{2+}$ or ${O_2}^{2+}$, respectively), on the contrary, acquires kinetic energy via momentum conservation in the molecular breakup process. Therefore, all higher kinetic energy distributions present must belong to the fragmented moieties. Both nitrogen and oxygen dications have metastable lifetimes of a few seconds, since their ground states lie above their dissociation limit [30,39–41], which allow their unequivocal disentanglement from their respective fragments with the same m/q. This metastable lifetime of a few seconds is orders of magnitude higher than the timescale of a DETOF measurement, since the time frame of the experiment is in the order of tens of microseconds [26,27,36], even at our highest extraction time delay (9 μs). This guarantees that a only negligible amount of metastable dications produced in the collisional process would be lost due to fragmentation during the acquisition time of the experiment.

For all measured impact energies, the adjustment curve of the experimental ratio, being the sum of the energy distributions employed, kept the coefficient of determination, R-squared (R^2) [42], above 0.99, and their uncertainties were determined using the criteria of keeping $R^2 > 0.97$.

III. RESULTS

New data for impact energies of 200 to 900 eV were obtained and compared to the upper impact energy cross sections of our previously reported data [26,27], with good agreement. For each impact energy, approximately 52 TOF spectra for different delay times were obtained, for a total of ~400 measurements, for the complete data. The absolute cross sections for ${N_2}^{2+}$ and ${O_2}^{2+}$ can be seen on Table I.

In Fig. 1, it is shown the ratio between the doubly charged and the singly charged parent molecule, namely ${N_2}^{2+}/{N_2}^{+}$ and ${O_2}^{2+}/{O_2}^{+}$, for both the present data and the previous ones [26,27]. The same ratio for both heteroisotopic molecules, $^{14}N\,^{15}N$ and $^{16}O\,^{17}O$, obtained by Märk in 1975, is also presented here for comparison [24]. Discrepancies between the data reported using this experimental setup and Märk's data for oxygen dication are probably due to the normalization procedure employed by Märk in order to calibrate and separate the dication peak from the charged fragments peaks, as discussed previously in both works on the subject [24,27].

Important information can be extracted comparing this ratio for both nitrogen and oxygen molecules at higher impact

TABLE I. Absolute cross sections for N_2^{2+} and O_2^{2+}, for the measured electron impact energies, in Mb. Uncertainties are estimated to be less than 10% for N_2^{2+} and 8% for O_2^{2+}.

E (eV)	N_2^{2+}	O_2^{2+}
200	3.73	1.58
300	2.73	1.36
400	2.05	1.20
500	1.55	1.06
600	1.17	0.951
700	0.938	0.866
800	0.727	0.832
900	0.600	0.821

energies. At high velocities, first-order perturbation theory gives for single ionization a cross-section dependency with the impact energy of $\sim(\ln E)/E$, while for double ionization it follows a $\sim 1/E^2$ behavior [43,44]. Thus, the dependency of the doubly-to-singly ionized parent molecule ratio with the impact energy either approaches a constant value (if both production processes come from a single ionization event, via a postcollisional autoionization process such as Auger decay) or follows a $\sim 1/E$ curve at high velocities (if the doubly ionized species comes from a direct double-ionization event, TS-2). This impact energy dependency is independent of the orbital where the single- or double-ionization events take place, although the value of the respective cross sections (and thus their ratio) depends on the orbitals involved. The nitrogen dication ratio does not seem to exhibit any tendency to reach a constant value, steadily following an approximate $1/E$ tendency, indicating that the nitrogen dication must be produced mainly via a direct double-ionization process, where two vacancies in the electronic orbitals of the molecule are produced in a single collision. The doubly-to-singly ionized oxygen molecule, on the other hand, has a very distinct behavior: It retains a constant value, which does not seem to change throughout the impact energy range. This leads to the conclusion that direct double-ionization processes play at most a very small role in the formation of the stable oxygen dication and that its primary production mechanism must be a single-ionization collisional process followed by an Auger secondary ionization. This kind of postcollisional relaxation leading to multiple ionized moieties after a single-ionization collisional process is an important and well-known mechanism for both atoms [28,45–48] and molecules [49–51].

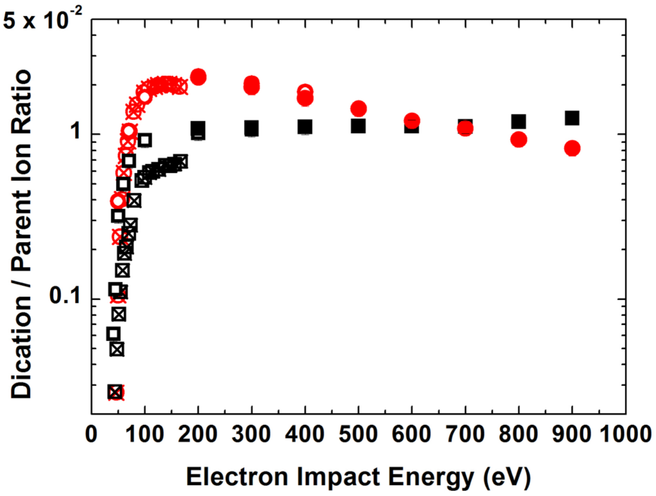

FIG. 1. Ratio between double- and single-ionization cross sections for the unfragmented parent molecule as a function of the electron impact energy. Oxygen molecule, black squares (filled symbols, this work; open symbols, Sigaud *et al.* [27]; crossed symbols, data from Märk [24]); nitrogen molecule, red circles (filled symbols, this work; open symbols, data from Ferreira *et al.* [26]; crossed symbols, data from Märk [24]).

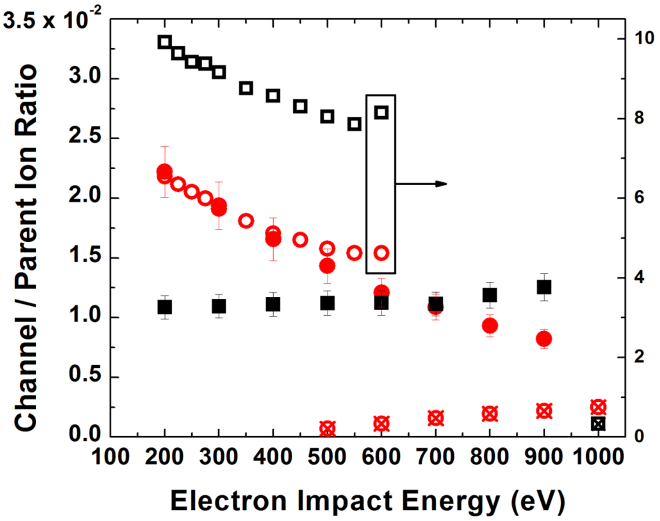

FIG. 2. Ratio between different channels' cross sections—described below—and the single-ionization cross sections for the unfragmented parent molecule as a function of the electron impact energy. Oxygen molecule, black squares (filled symbols, O_2^{2+}, this work; open symbols, $O^+ + O^+$, Tian and Vidal [52]; crossed symbols, O K-shell ionization cross section, Tawara *et al.* [53]). Nitrogen molecule, red circles (filled symbols, N_2^{2+}, this work; open symbols, $N^+ + N^+$, Tian and Vidal [52]; crossed symbols, N K-shell ionization cross section, Tawara *et al.* [53]). Please note that the data from Tian and Vidal [52] are scaled according to the right-hand-side y axis.

Investigation of the different channels that could result in doubly ionized channels for both oxygen and nitrogen can be seen in Fig. 2. A comparison between the ratio between the O_2^{2+} and N_2^{2+} dications and the singly ionized parent molecule cross sections and the channels $O^+ + O^+$ and $N^+ + N^+$ [52], also normalized by their singly ionized parent molecule cross section, can also be seen in Fig. 2, together with the ratios between both O and N K-shell ionization cross sections [53] and their respective singly ionized parent molecule cross section. Two important conclusions can be made by this comparison. First, both K-shell ionization cross sections are small in comparison with both doubly ionized channels presented in Fig. 2, and therefore their contribution must be present but is probably not large enough to significantly alter the ratios presented. These results also seem to be corroborated by theoretical models available for K-shell ionization cross sections [54]. Second, the doubly ionized fragmentation of the oxygen molecule has a larger ratio than the nitrogen one, and both present the $1/E$ impact energy dependence of direct double ionization (when two electrons are removed in the same collisional process). On the other hand, it can be seen that the doubly ionized fragmented nitrogen seems to reach a so-called saturation value at the end of the energy range of the data presented by Tian and Vidal [52]. This indicates that an Auger-like process can in fact be present for the double ionization of the nitrogen molecule. However, the key aspect here is that the energy levels of the molecular orbitals of the nitrogen molecule (or its stable molecular cation) do not allow Auger de-excitation due to energy conservation; in other words, autoionization is energetically forbidden for vacancies in the $2\sigma_g$ inner molecular orbital of N_2, as $I(2\sigma_g)^{-1} - I(3\sigma_g)^{-2} < 0$ (39.43–42.43 eV = −3 eV, according to the vertical ionization energies given by references [55,56]). When fragmentation occurs, the valence molecular orbitals are considerably altered during this transient time, possibly allowing an autoionization process, which is present only for the $N^+ + N^+$ channel. This autoionization process occurring in an inner valence shell during a transient electronic rearrangement has been called "Auger-like" for both atoms [47,57] and molecules [27,29]. These conclusions also clearly show that direct double-ionization contributes to both double-ionization channels for the nitrogen molecule, but only for the doubly ionized fragmentation of oxygen; i.e., if two electrons are removed simultaneously from the oxygen molecule, it leads to its fragmentation and does not contribute significantly to its dication production. Therefore, while for the nitrogen molecule inner valence-shell ionization followed by Auger decay is not energetically allowed, the molecular oxygen dication must come from inner valence shell ionization, since it appears well below the K-shell ionization threshold.

That leads to the answer for the third question proposed at the beginning of this paper. In order to produce an oxygen molecular dication, the final state without its two outermost unpaired electrons must be achieved predominantly by the removal of an inner valence-shell electron, which leads, after a rearrangement of the remaining electrons orbitals, to a post-collisional autoionization process. The most striking feature of this result is that there is no significant contribution from TS-2 direct double ionization to the stable dication formation. This can be due to two reasons. First, direct double ionization of other than the two outermost electrons probably results in a fragmented state. Second, the probability of the two outermost electrons being removed in the same collisional process must be so small as to completely suppress this dication formation pathway. A simpler explanation comes from the fact that the molecular π orbitals of the unpaired electrons absent in the final dicationic state are spatially dislocated from each other in the oxygen molecule. Therefore, the trajectory of the projectile has to be a very particular one in order to access exclusively these two electrons in order to interact with them, as is pictured in Fig. 3. In other words, the geometry of the molecule itself suppresses this possibility.

A further evidence for this can be seen when comparing these results with collisions between electrons and O_2^+ [58]. Although the fragmentation channel still dominates, the stable O_2^{2+} is now produced by direct ionization of the cation, without any geometrical constraint to inhibit the stable dication production. On the other hand, for stable N_2^+ ionization [59], the formation of the stable N_2^{2+} is more probable than its fragmentation, which resembles what was observed in the present work. Moreover, the ratio between dication production and fragmentation into two charged species for both cases reaches a constant value at high impact energies, since both processes arise from a single ionization of the parent cation [58,59].

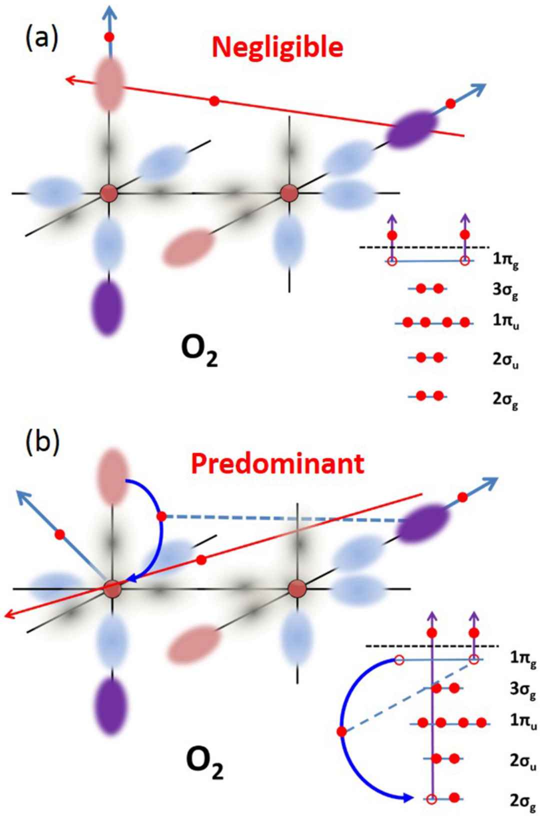

FIG. 3. Pictorial representation of molecular oxygen dication formation, with some O_2 electrons being represented, namely, σ_x (along the internuclear axis), π_y and π_z (closer to the nuclei), and π_y^* and π_z^* (farther from the nuclei), corresponding to the $KK(\sigma_s)^2(\sigma_s^*)^2(\sigma_x)^2(\pi_y)^2(\pi_z)^2(\pi_y^*)^1(\pi_z^*)^1$ O_2 configuration: (a) double direct ionization channel, negligible due to constraints of the molecular orbitals geometry; (b) single inner-valence-shell ionization, followed by Auger decay, representing the predominant dication production pathway.

IV. CONCLUSIONS

A few very important consequences of these results are summed up here: (i) the constant yield of the oxygen dication throughout the measured impact energies indicates that its contribution does not become negligible, not even at high impact energies, as, for example, the nitrogen dication yield, which depends on a second-order process. Therefore, it can be expected that molecular oxygen in the atmosphere (Earth's or any other oxygen-containing atmospheres) submitted to ionizing radiation should yield a significant amount of molecular dications, regardless of the impact energy, in comparison to singly charged species, which does not seem to be the case for nitrogen; (ii) the molecular dication production dynamics is very specific and can greatly vary from one molecule to another, as was shown for the nitrogen and oxygen molecules here, in which one is dominated by direct double ionization while the other comes primarily by inner valence Auger post-collisional decay; (iii) comparison with a few other molecular dications cross sections in the literature [60–62] shows that the understanding or prediction of the mechanisms that govern each dication production for different molecules are still elusive at best. Therefore, more dication production data and the analysis performed here should provide a benchmark to guide theoretical models for such processes.

The dependence of the probability of multiple ionization of molecules with its orientation has been studied via the angular distribution of their produced fragments. For diatomic molecules, in particular, multiple ionization is enhanced if the target molecule is aligned with the incident beam, allowing the projectile to travel through a higher density of electrons, accessing lower impact parameters, with respect to both nuclei [63]. This feature is not clearly shown in the few theoretical studies regarding double ionization of molecules by fast projectile impact, since most of them employ an averaging over all molecular orbitals' magnetic substates [63–66]. On the other hand, when orbital-by-orbital calculations are performed, as done by Oubaziz *et al.* for methane [67], a strong dependence with molecular orientation is unequivocally observed. This result agrees with the interpretation for ${O_2}^{2+}$ production presented above, where the $(\pi_y^*)^1(\pi_z^*)^1$, pictorially represented in Fig. 3 as the ones farther apart from the nuclei, must be hit by the projectile for the stable dication to be produced. In this case, the restriction imposed by this molecule's particular orientation with respect to the incident beam strongly suppresses ionization via the TS-2 mechanism. For nitrogen, the absence of $(\pi_y^*)^1$ and $(\pi_z^*)^1$ electrons impose fewer geometrical restrictions for double ionization, and the stable ${N_2}^{2+}$ can be more easily produced by TS-2. It should be noted that experimental evidence of orientation effects coming from double ionization leading to stable dication production (i.e., without molecular fragmentation) had not been reported to date. This distinct behavior observed for N_2 and O_2 molecules shows that double-ionization calculations must be made, in principle, for each specific molecule and one has to take special care when considering generalizations regarding dication production.

ACKNOWLEDGMENTS

The authors would like to acknowledge the Brazilian funding agencies CNPq, CAPES and FAPERJ, as well as INCT-FNA (Proc. No. 464898/2014-5).

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