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Exotic negative molecules in AMS

Robin Golser [a,*], Hubert Gnaser [b], Walter Kutschera [a], Alfred Priller [a],
Peter Steier [a], Anton Wallner [a]

[a] *VERA Laboratory, Fakultät für Physik, Universität Wien, Währinger Straße 17, A–1090 Wien, Austria*
[b] *Fachbereich Physik, Technische Universität Kaiserslautern, Erwin-Schrödinger-Straße, D–67663 Kaiserslautern, Germany*

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Abstract

"The techniques and equipment developed for AMS studies are well suited for identifying exotic negative ions". With this sentence begins a pioneering paper by Roy Middleton and Jeff Klein (M&K) on small doubly-charged negative carbon clusters [Nucl. Instr. and Meth. B 123 (1997) 532]. M&K were the first to utilize Accelerator Mass Spectrometry to prove the existence of these clusters and a number of other exotic molecules. We review M&K's efforts and show how their work is being continued at other laboratories. The latest developments are: (1) the discovery of long-lived molecular hydrogen anions H_2^-, D_2^- and (2) the unambiguous identification of the smallest doubly-charged negative molecule $(LiF_3)^{2-}$. In particular we show new experimental data for D_3^-, and for $(LiF_3)^{2-}$, and we try to answer the question why M&K's search for this di-anion was unsuccessful.

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1. Introduction

The legacy of Roy Middleton as father of the "Middleton-type" sputter source, and the work on AMS of long-lived radionuclides should not let us forget his important contributions to the physics of negative ions. Together with Jeff Klein, Middleton published a series of four papers [1–4] on doubly-negatively charged molecules (di-anions) and other exotic negative species, work often referred to in the literature of theoretical chemistry. In the following we give a short review of M&K's papers and then show the latest results in this field. Based on our experience with $(LiF_3)^{2-}$, the smallest di-anion predicted to exist, we discuss possible reasons why M&K did not succeed to observe it.

The techniques and equipment developed for AMS studies that are so well suited for identifying exotic negative ions are in particular: (1) the sputter source for the efficient production of negative ions, (2) the low-energy mass spectrometer, (3) the tandem accelerator with a gas stripper, (4) the high-energy mass spectrometer and (5) the energy detector (ideally of the $E-\Delta E$-type). The Vienna Environmental Research Accelerator (VERA) may serve as a typical incarnation of this scheme, see e.g. [5,6]. VERA is similar to the system used by M&K, with one notable exception: at the low-energy side VERA is equipped with both an electrostatic and a magnetic analyzer, whereas the former device was not available to M&K (the importance of this will become clear below).

2. Middleton's work on exotic negative molecules

When M&K published their paper on "Carbon anions and dianion clusters" in 1997 [1], small doubly-charged negative carbon clusters were already known for some years from secondary ion mass spectrometry (SIMS) [7,8]. The trick to distinguishing a comparatively small

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[*] Corresponding author.
E-mail address: robin.golser@univie.ac.at (R. Golser).

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number of di-anions with mass M from the anions of mass $M/2$, which are orders of magnitude more abundant, was to concentrate on di-anions with an odd mass number, e.g. $(^{12}C_9{}^{13}C)^{2-}$. Thus, the doubly-charged negative molecules appear at a half-integer mass. M&K's idea was to inject these mass-selected negative molecules into a tandem accelerator and to utilize AMS to literally "analyze" the molecules further by breaking them up into their atomic constituents during the stripping process. After the accelerator, the positive atomic ions are again filtered according to their mass-to-charge ratio and their energy is measured absolutely in a detector. All the virtues that set AMS apart from conventional mass spectrometry and allow the measurement of long-lived radionuclides with extreme sensitivity, are thus available to search for exotic negative molecules. The multiple occurrence of a particular isotopic species in the molecule, e.g. nine ^{12}C in $(^{12}C_9{}^{13}C)^{2-}$, leads to multiple coincidence peaks in the energy spectrum (and optionally in the ΔE-spectrum as well). A detailed discussion of the properties of such coincidence peaks may be found, e.g. in [1,9]. Examples are shown and discussed below in Figs. 1 and 2, where the three D^+ peaks from D_3^- and the three $^{19}F^{2+}$ peaks from $(LiF_3)^{2-}$ are plotted, respectively. A further technique utilized in [1] may be considered a basic form of Coulomb Explosion Imaging [10] in the terminal of the tandem accelerator: a Lexan foil was installed a fraction of a millimeter behind a very thin carbon foil and was used for track-etch analysis. Whereas the number of atoms in the incident molecules could be clearly resolved, the hope to identify the geometrical structure unambiguously was not fulfilled. In the abstract and in the conclusion of [1], M&K gave hints for the successful detection of other exotic molecules, like N_2^-, N_3^-, and the di-anions $(BeF_4)^{2-}$, $(MgF_4)^{2-}$. Dedicated papers were submitted October 1996, and January 1997 [3,4], respectively, but not published until the end of 1999. Historically next was the publication "Observation of $(BeC_n)^{2-}$, a particularly abundant gaseous dianion" [2]. It led to a number of theoretical papers to explain the existence of $(BeC_n)^{2-}$, e.g. [11,12], and to a further study by AMS [13].

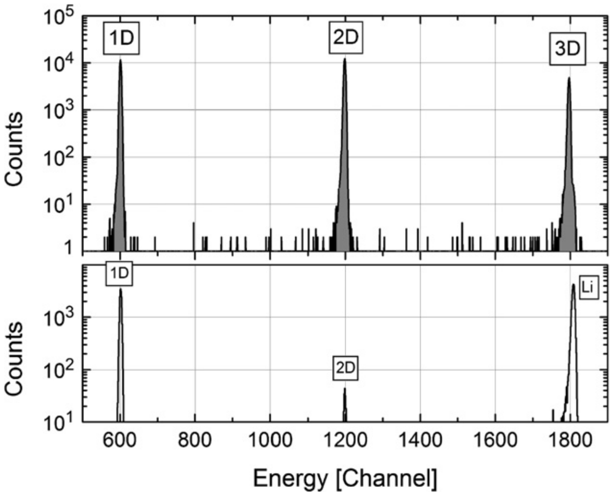

Fig. 1. Energy spectrum measured in a surface barrier detector when negative ions with $M/Q = 6$, e.g. D_3^-, sputtered from enriched TiD_2 are injected into the accelerator and the high-energy side is set to detect positive ions with $M/Q = 2$. Upper panel: the low-energy analyzers are optimized for D_3^- at $M/Q = 6.042$, and the high-energy analyzers for D^+. Lower panel: the injector is slightly de-tuned towards $^6Li^-$ at $M/Q = 6.015$, the high-energy filters remain un-changed.

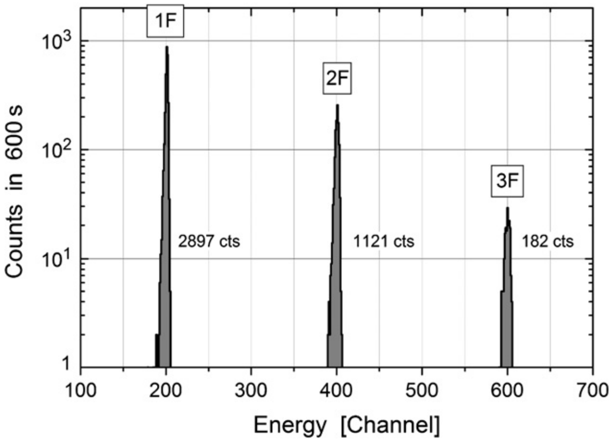

Fig. 2. Energy spectrum measured in a surface barrier detector when negative ions with $M/Q = 31.5$, e.g. $(^6Li^{19}F_3)^{2-}$, sputtered from enriched ^6LiF are injected into the accelerator and the high-energy side is set to detect $^{19}F^{2+}$.

From an atomic theory point of view, the announcements in [1] had a deep impact, as long-lived states of N_2^- were unknown at that time (for a recent review, see [14]). Although the *simultaneous* detection of *all* atomic constituents, i.e. two N^{3+} from N_2^-, in both E and ΔE left hardly any room for misinterpretation, long-lived N_2^- was probably too surprising and meticulous further studies deemed necessary. So experimental results gained independently by SIMS [15] and theoretical analysis, e.g. [16,17], were already published, when M&K's pioneering paper "Production of metastable negative ions in a cesium sputter source: Verification of the existence of N_2^-, and CO^- "[3], finally appeared in print together with "Experimental verification of the existence of the gas-phase di-anions $(BeF_4)^{2-}$, $(MgF_4)^{2-}$" [4].

3. Ongoing AMS work on exotic negative molecules

Let us compare AMS of N_2^- and N_3^- to AMS of the simplest negative molecules conceivable, i.e. to H_2^- and H_3^- and their isotopic isomers. H_2^- was previously believed to exist only for 10^{-15} s [18], but recently discovered to live on the order of 10^{-6} s when produced in a sputter source [19]. In order to be detected with the correct values of all analyzing elements, the molecules have to pass the low-energy mass spectrometer and reach the stripper of the tandem accelerator, i.e. they have to live for a few microseconds at least. As protons and deuterons can only have charge state $Q = 1+$ (or become neutral) and as they are the lightest nuclides, E/Q- and M/Q-ambiguities are only possible in the low-energy mass spectrometer, e.g. between H_2^-, and D^-, but not at the high-energy side. Any decay products or charge-changing products along the way are either eliminated by the low-energy or by the high-energy analysis and will not reach the detector. A significant change of situation occurs at mass $M = 6$ (and beyond), where D_3^- may interfere with $^6Li^-$ at the low energy-side, and D^+ with $^6Li^{3+}$ at the high-energy side.

Fig. 1 shows energy spectra measured in a surface barrier detector when negative ions with $M/Q = 6$ are injected into the accelerator running at 1.5 MV. The high-energy side is set to detect positive ions with $M/Q = 2$. The ions were sputtered with 5 keV Cs^+ from a TiD_2 cathode, depleted in ^1H to less than 2%. In the upper panel we present the result after tuning the low-energy mass spectrometer for negative ions at $M/Q = 6.042$, i.e. for D_3^- and optimizing the high-energy mass spectrometer for positive ions at $M/Q = 2.014$, i.e. for D^+ . Three peaks clearly stand out, which are due to the simultaneous impact of either one D^+, or two D^+, or all three D^+ from the break-up of one D_3^-. A remarkable feature is the high probability of detecting more than one D^+. This is largely due to the fact that the stripping yield for charge state 1+ can reach 100% for hydrogen ions , and it is only due to losses on the way to the detector that we do not always measure all three D^+ simultaneously. SIMS allowed us to quantify the amount of D_3^- from a sputter source: $D_3^-/D^- = 7 \times 10^{-9}$. For completeness, a remark from Roy Middleton's "Negative-Ion Cookbook" [20] should be mentioned: "H_2^- and H_3^- are reported to be stable and we have searched for these using a cathode loaded with deuterium ..., a weak peak was observed corresponding to D_2^-, but none was observed corresponding to D_3^-. The ratio of D_2^- to D^- is about 2.3×10^{-6}, and an upper limit on the D_3^- to D^- is about 10^{-6}". It is unclear on which "reports" the stability of the molecular anions was based on, as the existence of both long-lived H_2^- and H_3^- was under debate till very recently, cf. [19,21].

Careful inspection of the $3 \times D^+$-peak on a finer scale indicates a small, but unusual shoulder at the high-energy edge. The reason for this comes out in the lower panel of Fig. 1. It shows the effect when the injector is slightly detuned towards $^6Li^-$ at $M/Q = 6.015$: the signals from D_3^- decrease and $^6Li^{3+}$ comes up. Before we had enriched TiD_2 available, we tried to produce D_3^- from TiH_2 with a natural isotopic ratio $D/H \sim 1.15 \times 10^{-4}$, and to our surprise we got a much higher count-rate than expected from the previous measurement of H_3^- [19]. However, from the energy spectrum it was immediately clear that the signal did not come from D_3^-, and that our sputter source happened to be "contaminated" from operation with LiF (see below). We wish to point out that the advantage of using AMS is not only the ability to recognize ambiguities by the E- and/or the ΔE-analysis, but also the ability to eliminate them in many cases, e.g. by using a different charge state, or by blocking higher-Z interferences in a foil. To separate D_3^- from $^6Li^-$ ($M/\Delta M \approx 220$) by E/Q- and M/Q-analysis, only a modest resolution is required, but with increasing mass the situation becomes less favorable (or if the ions of interest are extremely rare). For N_2^- and CO^-, there are strong interferences with Si ($^{14}N_2$ – ^{28}Si: $M/\Delta M \approx 960$, and $^{12}C^{16}O$ – ^{28}Si: $M/\Delta M \approx 1600$). We discuss below why the coincidence peaks in both the E- and the ΔE-detector remain a unique signature as long as interferences do not cause extensive pile-up.

In [4] M&K confirmed theoretical predictions [22–24] about the existence of the di-anions $(BeF_4)^{2-}$ and $(MgF_4)^{2-}$ and also shed some light on their structure. Thus, the building principle and the very sophisticated computer codes to calculate the stability of di-anions gained great confidence. For completeness it is worth noting that $(CaF_4)^{2-}$ could not be detected by SIMS [25], but was proved to exist by AMS at VERA [9]. On the other hand, [4] is not only cited often because of the success in detecting $(BeF_4)^{2-}$ and $(MgF_4)^{2-}$, but also because of the failure to observe $(LiF_3)^{2-}$ and $(Li_2F_4)^{2-}$, also predicted to be stable [22,26]. An upper limit was placed on the formation rates (10^{-20} and 10^{-17} times that of $^{19}F^-$, respectively), and M&K speculated whether $(LiF_3)^{2-}$ is either not formed in a sputter source or has a lifetime less than 10 μs because of the high vibrational excitations induced by the sputtering process. The search for $(LiF_3)^{2-}$ by SIMS was also unsuccessful [25]. From our observation of H_2^-, we can confirm the presence of high vibrational and rotational excitations, as they are the very reason to increase the lifetime from fs to μs [19]. But we can also exclude that these excitations destroy all $(LiF_3)^{2-}$, simply because this di-anion has been found recently by AMS at IsoTrace [27], and we have also observed it at VERA, cf. Fig. 2.

Fig. 2 shows the energy spectrum measured in a surface barrier detector when negative ions with $M/Q = 31.5$, e.g. $(^6Li^{19}F_3)^{2-}$, are injected into the accelerator running at 2.7 MV and the high-energy side is set to detect $^{19}F^{2+}$. The ions were sputtered with 5 keV Cs^+ from a LiF cathode, enriched in ^6Li to more than 90%. What sets Fig. 2 apart from the corresponding figure in [27] is the much higher count-rate, indicated next to the peaks by the number of counts acquired in 10 minutes. This is most likely due to the higher stripping yield for $^{19}F^{2+}$ at terminal voltage 2.7 MV (as compared to 2.0 MV in [27]), and the higher output from our Multi-Cathode Source for Negative Ions by Cesium Sputtering, manufactured by National Electrostatics Corporation, USA. Similar to the findings in [27], the relative peak intensities do not follow a binomial distribution. One reason is the random influx of $^{19}F^{2+}$ into the $1 \times F$-peak from neighboring Li_2F^- at $M/Q = 31.0$, and $M/Q = 32.0$, respectively (see discussion below). The other reason is that the necessary assumption – the three F from $(LiF_3)^{2-}$ are statistically equivalent – is most likely not fulfilled in our gas-stripper when a molecule dissociates in time-space-sequence: the probability for liberation, the probability for conversion to 2+, and the probability for detection may all vary among the three F.

The reasonably high count-rate at VERA enabled us to investigate the influence of the electrostatic analyzer (ESA) at the low-energy side in greater detail. As mentioned in the introduction, such a device was absent in the system of M&K, whereas IsoTrace and VERA do have one. Even when the low-energy side of VERA is carefully tuned for mass 44.5, an unambiguous detection of $(Li_2F_4)^{2-}$ is impossible. The cause is ^{19}F-influx due to currents on the order of 10 nA from $^6Li^{19}F_2^-$ at mass 44.0 and of 100 nA from $^7Li^{19}F_2^-$ at mass 45.0, respectively, injected from a LiF sample with natural isotopic ratio. This is surprising as we may consider the challenge comparable to detecting ^{12}C from $(^{12}C_9{}^{13}C)^{2-}$ at apparent mass 60.5 in the vicinity of the strong current from $^{12}C_5^-$ at mass 60.0. Nevertheless, neither M&K nor we had any problems [1,5]. We noticed, however, that setting the slits after the low-energy ESA is more cumbersome for LiF than for graphite. Such difficulties have been experienced earlier for $(^{43}Ca^{19}F_4)^{2-}$ sputtered from CaF_2, where $^{40}Ca^{19}F^-$ gives a strong signal [9]. A possible explanation might be that graphite is conductive whereas LiF and CaF_2 are insulating. Although we mixed the fluorides with Ag-powder, small grains may charge-up locally and the energy distribution may eventually vary in space and in time. But of course, the most important reason for the difficulties is the very low yield of the respective di-anions.

Around mass 31.5 the ^{19}F-background from neighboring masses is comparatively low, if the ESA slits are carefully set, and the unambiguous detection of ^{19}F from $(^6Li^{19}F_3)^{2-}$ is possible (see above). In Fig. 3 we show the count-rate in the energy detector when the injected mass is scanned around 31.5. Mass scans in a narrow range can be comfortably accomplished with VERA by scanning the bias voltage on the magnet chamber, the so called multi-beam-switcher (MBS) usually used for fast sequential injection of, e.g., $^{12}C^-$, $^{13}C^-$, $^{14}C^-$. The dark bars are for optimum slit settings, leading to a well defined maximum at mass 31.5, with almost zero count-rate on both sides of the peak and then a strong increase near the integer masses 31.0, and 32.0, respectively, due to Li_2F^-. When the slits are set only about 10% wider than optimal, the ^{19}F-contribution from the integer masses increases considerably, as is indicated by the shaded area. To avoid excessive count-rate in the detector the scanning range had to be restricted from about 31.1 to about 31.9. But even under circumstances where the di-anion peak in the total count-rate is entirely obscured, we can identify $(LiF_3)^{2-}$ unambiguously by the triple $^{19}F^{2+}$-coincidence in the energy spectrum. This holds true as long as we can distinguish the coincidence peaks (which are due to the simultaneous impact of $^{19}F^{2+}$ out of a single molecule) from pile-up, i.e. from random coincidences of $^{19}F^{2+}$ out of different molecules. As different molecules are uncorrelated, their energy signals frequently overlap only in part and give rise to counts *between* the peaks, from where the importance of pile-up can be assessed, c.f. Fig. 2 in [28].

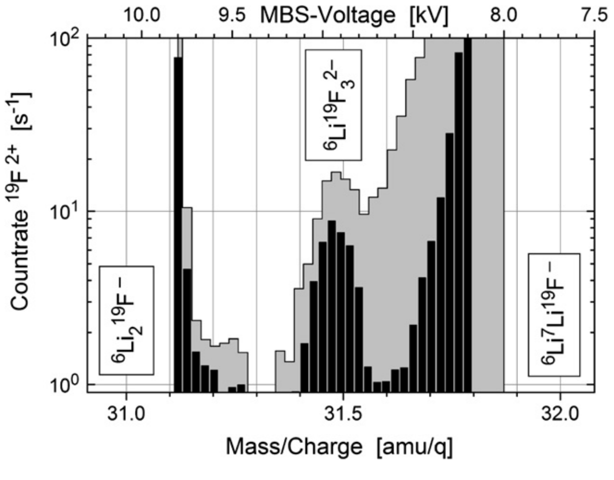

Fig. 3. Count rate of $^{19}F^{2+}$ as function of MBS-Voltage for two settings of the analyzer slits (see text). The dark bars are for optimal slit settings, the shaded area shows the effect of a setting about 10% wider.

It must be noted that the influx from neighboring masses does not only depend on the low-energy analysis, but also on settings at the high-energy side, since the mass differences between the molecules lead to energy differences between the respective break-up ions. For example, if $^{19}F^{2+}$ from the break-up of $(^6Li^{19}F_3)^{2-}$ at $M/Q = 31.5$ leaves the accelerator with 7.070 MeV, then $^{19}F^{2+}$ from the break-up of $(^6Li_2{}^{19}F)^-$ at $M/Q = 31.0$ has 7.097 MeV. This 0.38% energy difference can be exploited for discrimination, see e.g. [27]. However, physical effects like energy loss straggling, angular straggling, Coulomb explosion, etc., and technical effects like terminal voltage ripple perturb the ion energy and the direction of flight [29]. From our experience with extremely rare di-anions, e.g. $(CaF_4)^{2-}$ [9], we consider it advantageous to discriminate as good as possible before injection, but to be tolerant at the high-energy side. Eventually, pile-up from neighboring masses then becomes more intense, but it enables one to detect as many of the most significant all-n coincidences as possible, e.g. $4 \times F$ from $(CaF_4)^{2-}$.

If pile-up dominates the energy spectrum, the beam has to be attenuated appropriately and this is what M&K reportedly [4] did for $(Li_2F_4)^{2-}$. For $(^6Li^{19}F_3)^{2-}$ attenuation is not explicitly mentioned, but their surprisingly low count-rate of about 1 cpm for $^{19}F^{3+}$ is otherwise hard to explain. In a few favorable cases the excessive count-rate comes mainly from the very abundant di-anions themselves, e.g. $(^{12}C_9{}^{13}C)^{2-}$, and the usefulness of attenuation is immediately evident. But for $(LiF_3)^{2-}$ and $(Li_2F_4)^{2-}$ it is due to the influx from neighboring masses. Even though attenuation then reduces the signal from the di-anions as well, it may be useful to a certain limit, since the coincidence peaks depend linearly on count-rate, but pile-up depends quadratically. However, it is our experience with $(Li_2F_4)^{2-}$ that attenuation does not help even when a low-energy ESA is present. And from measuring $(LiF_3)^{2-}$ with the ESA slits only a little bit too wide, it appears practically impossible to measure these di-anions without an

ESA at all. We therefore suppose that due to excessive contributions of ^{19}F from neighboring masses, M&K had hardly a chance to find $(LiF_3)^{2-}$, and $(Li_2F_4)^{2-}$ with their system.

After the first observation of $(LiF_3)^{2-}$, A.E. Litherland suggested another adverse mechanism that may have further prevented M&K from detecting $(LiF_3)^{2-}$ [30]: the very sensitive di-anions may be destroyed due to collision in the cratered cathodes used by M&K. The fairly high yield of $(LiF_3)^{2-}$ at VERA makes a detailed study of this effect possible, "... as $(LiF_3)^{2-}$ is probably the most sensitive ion that would allow one to quickly test the idea whether a fragile anion can be destroyed by reactions soon after its formation" [30].

4. Conclusion and outlook

We briefly reviewed how Middleton and Klein paved the way for AMS into the field of exotic negative ions. From the ongoing work mentioned above we may conclude they also paved the way for negative ion research into AMS. Some of the discoveries could not have been made without AMS, or at least not without the versatile, highly-efficient sputter sources used for AMS. And the sources may well have more surprises in store, e.g. other negative molecules produced under "extreme" conditions, or small triply-negatively charged molecules.

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