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Determination of the ionization energy of NpO_2 and comparative ionization energies of actinide oxides

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Abstract

The ionization energy of NpO_2 was determined by the electron-transfer bracketing technique employing Fourier transform ion cyclotron resonance mass spectrometry. Under thermoneutral conditions the NpO_2^+ ion was found to abstract an electron from *N*,*N*,*N*′,*N*′-tetramethyl-1,4-phenylenediamine (IE = 6.20 ± 0.05 eV) but not from *N*,*N*-dimethyl-1,4-phenylenediamine (IE = 6.46 ± 0.02 eV). Accordingly it is concluded that IE[NpO_2] = 6.33 ± 0.18 eV. This result contrasts with a previously reported value for IE[NpO_2] of ~5.0 eV. The new value for the ionization energy of NpO_2 from the present study is compared with those of UO_2, PuO_2 and AmO_2. Literature values for the ionization energies of actinide monoxides and dioxides are tabulated, and systematic trends are considered. The recently reported second ionization energies for UO_2, NpO_2, PuO_2 and AmO_2 are also assessed. Comparisons of the various actinide oxide ionization energies are made with ionization energies of actinide atoms and with values for other relevant molecules.

1. Introduction

The ionization energy of NpO_2 has been reported as ~5.0 eV by Ackermann et al. [1]. Somewhat surprisingly, this value is substantially lower than IE[NpO] ≈ 5.5 eV that was reported by the same authors [1]. These neptunium monoxide and dioxide ionization energies were determined by electron ionization (EI) appearance threshold measurements of high-temperature vapors. Recent spectroscopic determinations of IE[UO] and IE[UO_2] by Heaven and co-workers [2] demonstrated that the actual IEs are significantly larger than those determined by the EI technique for these uranium oxide molecules. Gagliardi et al. [3] had previously performed ab intio calculations that indicated the EI values for IE[UO_2] were too low and ‘suggested that the possible reason for this difference is that a low value for the first ionization energy has been measured in electron impact mass spectrometric experiments, which involve population of neutral excited states under the high-temperature

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evaporation conditions used to prepare UO_2 in the gas phase' [3].

In view of the doubts associated with molecular ionization energies determined by EI of high-temperature molecular vapors, a re-determination of IE[NpO_2] was carried out in the present study using the more definitive technique of electron-transfer bracketing, as described by Zimmerman et al. [4]. A specific motivation for more accurately determining this ionization energy was that we have previously found discrepancies between the reported IE[NpO_2] [1] and other thermodynamic data determined by us [5,6]. Along with NpO_2^+, electron-transfer experiments were also performed with PuO_2^+, which had previously been examined in detail by this technique [5]. Available first and second ionization energies for actinide monoxides and dioxides, AnO and AnO_2, are tabulated and evaluated in the context of the nature of the bonding in these molecules. The ionization energies of actinide monoxides are also assessed in the context of the corresponding values that are available for lanthanide monoxides.

2. Experimental

The experimental procedures have been described in detail elsewhere [5,6] and only a brief summary is included here. The $^{237}Np^+$ and $^{242}Pu^+$ ions were produced by laser desorption ionization (LDI) of dilute alloys of the respective actinides (~5 at.%) in a platinum matrix. Oxidation of the bare Np^+ and Pu^+ ions to NpO_2^+ and PuO_2^+ was accomplished by pulsing O_2 into the ICR cell of a Fourier transform ion cyclotron resonance mass spectrometer (FTICR-MS). The monopositive dioxide ions were isolated by ejecting all other ions and were then thermalized by collisional cooling with Ar gas maintained in the cell at a static pressure of $(1–5) \times 10^{-6}$ Torr. Effective thermalization was confirmed by the linearity of the reactant ion signals versus time, and by reproducibility of reaction kinetics for different collisional periods or collision gas pressures. The electron-transfer reagents were commercial products of >99% purity and were used as received. Static reagent pressures of $\sim 10^{-7}$ Torr were maintained in the ICR cell; the base pressure in the ICR cell was $\sim 10^{-8}$ Torr.

3. Results and discussion

3.1. Determination of the ionization energy of NpO_2

The electron-transfer reagents and their corresponding ionization energies [7] were as follows: *N*,*N*,*N*′,*N*′-tetramethyl-1,4-phenylenediamine (TMPD)/IE = 6.20 ± 0.05 eV; *N*,*N*-dimethyl-1,4-phenylenediamine (DMPD)/IE = 6.46 ± 0.02 eV; ferrocene/IE = 6.71 ± 0.08 eV; and 1,4-phenylenediamine (PD)/IE = 6.87 ± 0.05 eV. In the case of NpO_2^+, electron transfer from TMPD was observed, but electron transfer from DMPD, ferrocene and PD was not observed. Accordingly, IE[NpO_2] is bracketed between IE[TMPD] = 6.20 ± 0.05 eV and IE[DMPD] = 6.46 ± 0.02 eV. The probable value for IE[NpO_2] is taken as the average between 6.20 eV and 6.46 eV – i.e., 6.33 eV – and the uncertainty is taken as the maximum possible limit – i.e., {6.33 eV – 6.15 eV} = 0.18 eV. The conclusion is that IE[NpO_2] = 6.33 ± 0.18 eV, where this assigned uncertainty is considered a conservative upper limit. This revised value is substantially greater than the previous value of ~5.0 eV [1] and is consistent with systematic trends, as discussed below. The new value for IE[NpO_2] is also consistent with bond energy data obtained previously by us [5,6].

For comparison with earlier results [5], the electron-transfer reactions of PuO_2^+ with TMPD and DMPD were also examined. In accord with the recently reported IE[PuO_2] = 7.03 ± 0.12 eV [5], electron transfer to PuO_2^+ was seen with both TMPD and DMPD. The comparative results for PuO_2^+ confirm that IE[PuO_2] > IE[NpO_2], in accord with the determined value for IE[NpO_2].

In the case of TMPD with both NpO_2^+ and PuO_2^+, the only reactions other than electron transfer that were observed were consecutive formation of the mono-adduct and the bis-adduct, $[AnO_2 \cdot TMPD]^+$ and $[AnO_2 \cdot 2(TMPD)]^+$. It is apparent that neither of these AnO_2^+ efficiently activate bonds in TMPD. In accord with the greater exothermicity for electron transfer in the case of PuO_2^+ compared with NpO_2^+, this pathway was dominant (70%) for PuO_2^+ whereas adduct formation was dominant for NpO_2^+ (85%) and electron transfer to NpO_2^+ was a relatively minor (15%) though definitive channel.

3.2. First ionization energies of actinide oxides

Reported actinide oxide ionization energies are given in Table 1, including the new value for IE[NpO_2] obtained in this work. The IE[AnO] (An = Th, U, Np, Pu) and IE[AnO_2] (An = U, Np, Pu) in Table 1 are revisions to the previously accepted values that were based on electron impact ionization threshold measurements of high-temperature vapors [11]. The values for IE[NpO], IE[PuO] and IE[AmO] given in Table 1 are based on an earlier study [6] that employed a technique developed for lanthanide monoxides by Schwarz and co-workers [12]. From spectroscopic studies of low-temperature UO molecules, Heaven and co-workers [2] have recently reported a reliable value of IE[UO] = 6.0313 ± 0.0006 eV, which is 0.4 eV greater than the earlier EI value of 5.6 eV [13]. In view of this revision to IE[UO], the previously derived IE[NpO], IE[PuO] and

Table 1
First and second ionization energies of actinide oxides (eV)

An	IE[AnO]	IE[AnO$_2$]	IE[AnO$^+$]	IE[AnO$_2^+$]
Th	6.60[a]	(8.7)[d]	$\leqslant$12.8[g]	–
U	6.03[b]	6.13[b]	12.7 ± 0.8[g]	14.6 ± 0.4[g]
Np	6.1 ± 0.2[c]	6.33 ± 0.18[e]	13.8 ± 0.6[g]	15.1 ± 0.4[g]
Pu	6.1 ± 0.2[c]	7.02 ± 0.12[f]	13.7 ± 0.8[g]	15.1 ± 0.4[g]
Am	6.2 ± 0.2[c]	7.23 ± 0.15[c]	13.7 ± 0.6[g]	15.7 ± 0.9[g]

[a] From Ref. [8].

[b] From Ref. [2]. The precise values are IE[UO] = 6.0313 ± 0.0006 eV and IE[UO$_2$] = 6.128 ± 0.003 eV [2].

[c] From Ref. [6]; the values of IE[AnO] have been adjusted for the revised IE[UO] from Han et al. [2].

[d] From Ref. [9]. The uncertainty associated with this electron ionization value is unknown.

[e] This work.

[f] From Ref. [5].

[g] From Ref. [10].

IE[AmO] [6] have been accordingly revised upward – these three new IE[AnO] values are included in Table 1.

The electron-transfer bracketing technique was used in an earlier FTICR-MS study to determine IE[PuO$_2$] = 7.02 ± 0.12 eV [5], this value being in sharp contrast to the recent EI value of 10.1 ± 0.1 eV [13]. The IE[NpO$_2$] = 6.33 ± 0.18 eV from the present study contrasts with the earlier EI value of ~5.0 eV [1]. The discrepancy for NpO$_2$ is consistent with EI of excited state, high-temperature NpO$_2$ molecules [3], which would result in a lower EI ionization appearance threshold than for ground-state NpO$_2$. It should be remarked that the very large discrepancy for IE[PuO$_2$] is not readily rationalized by the same considerations because the EI value reported for PuO$_2$ vapor molecules is ~3 eV larger, not smaller, than the revised value. The values in Table 1 indicate a monotonic increase in IE[AnO$_2$] across the series from UO$_2$ to AmO$_2$. The largest increase, ~0.7 eV, occurs between IE[NpO$_2$] and IE [PuO$_2$]. This is consistent with the decreasing stability of oxidation states above An(IV) beyond Np [14]. Whereas IE[UO$_2$] and IE[NpO$_2$] are only ~0.1–0.2 eV greater than the corresponding IE[AnO], both IE[PuO$_2$] and IE[AmO$_2$] are ~1 eV greater than the corresponding IE[AnO].

A value of IE[UO$_3$] = 10.6 ± 0.1 eV has been reported by Rauh and Ackermann [9] based on EI appearance threshold measurements. Capone et al. [13] recently reported a similar value of IE[UO$_3$] = 10.8 eV, also using the EI technique. As noted above, the possibility of excited state UO$_3$ molecules from the high-temperature vapor source sheds some doubt on the accuracy of these values. However, a key point is that the previously reported IE[PuO$_2$] = 10.1 eV [13] is only ~0.7 eV lower than the IE[UO$_3$] obtained by the same experimental technique [13]. Uranium does not normally exhibit oxidation states above U(VI), the oxidation state in UO$_3$, because hexavalent uranium has no free non-bonding valence electrons outside of the closed Rn core. The Pu(IV) oxidation state is particularly stable [14] but oxidation states up to Pu(VI) – and perhaps Pu(VII) – are known [14], as evidenced by the recent report of the PuO$_3$ molecule [15]. In view of the valence saturation of UO$_3$ it would be expected that its ionization energy should be substantially greater than that of PuO$_2$, for which several valence electrons not involved in bonding remain at the Pu metal center, and oxidation states above Pu(IV) are stable. The ionization energy of ThO$_2$ has been reported as 8.7 eV [9] based on EI threshold measurements. As expected in view of the valence saturation of Th in ThO$_2$, this value is substantially greater than that of AmO$_2$ (i.e., IE[ThO$_2$] $\gg$ 7 eV). It would be expected that the ionization energy of ThO$_2$ should be similar to that HfO$_2$ as Th exhibits characteristics of a group 4 d-block transition metal element [16]. Rauh and Ackermann [9] also reported IE[HfO$_2$] ≈ 9.4 eV based on EI measurements and their value of 8.7 eV for IE[ThO$_2$] is indeed reasonably close to this latter value.

It is desirable to compare the ionization energies of the actinide monoxides with those of the lanthanide monoxides, LnO. To the best of our knowledge, the only determinations of the IE[LnO] are based on EI threshold measurements of high-temperature vapors [7]. Ackermann et al. [17] have reported the first ionization energies for all of the lanthanide monoxides (except PmO) based on EI measurements. With the exception of LuO, it was found that all of the IE[LnO] are within 1 eV of the corresponding IE[Ln]. For the early lanthanides (La, Ce, Pr, Nd) and Gd, the IE[LnO] were ~0.5 eV lower than the corresponding IE[Ln]. Ackermann et al. [17] attributed these low IE[LnO] to removal of an anti-bonding electron in the ionization process. However, in view of the demonstrated uncertainties associated with EI determinations of metal oxide ionization energies [2] it is appropriate to evaluate the EI results of Ackermann et al. by comparing their results for IE[TaO] [17] with those from a subsequent photoelectron study by Dyke et al. [18]. In accord with the tendency for EI measurements of molecular ionization energies to result in somewhat low values (perhaps due to excited-state neutral molecules [3]), the EI value for IE[TaO] was 7.92 eV [17], nearly identical to IE[Ta] = 7.89 eV [7], whereas the photoelectron value was 0.7 eV greater: IE[TaO] = 8.61 ± 0.02 eV [18]. It is concluded that the available IE[LnO] [7,17] determined by the EI method are insufficiently accurate to enable detailed comparisons. It should be a priority to re-determine lanthanide monoxide ionization energies using more reliable techniques.

To the extent that the reported IE[LnO] can be considered qualitatively reasonable, it would appear that IE[LnO] are rather similar to the corresponding IE[Ln]

Table 2
First and second ionization energies of actinide atoms (eV)

An	IE[An]	IE[An$^+$][d]
Th	6.307[a]	11.9 ± 0.1
U	6.194[b]	11.9 ± 0.5
Np	6.266[a]	11.7 ± 0.3
Pu	6.026[c]	11.7 ± 0.2
Am	5.974[a]	12.0 ± 0.2

[a] From Ref. [19].
[b] From Ref. [20].
[c] From Ref. [21].
[d] From J. Sugar, as cited in Ref. [11].

– e.g., typically within ~1 eV. For comparison, the IE[AnO] in Table 1 can be compared with the IE[An] that are included in Table 2. For each of the five AnO, the monoxide ionization energies are within ~0.3 eV of the atomic ionization energies. The IE[AnO] are similar to the IE[An], to within the reported uncertainties, for NpO, PuO and AmO. Whereas IE[ThO] is ~0.3 eV greater than IE[Th], IE[UO] is ~0.2 eV less than IE[U]. That the addition of an oxo-ligand to uranium reduces the ionization energy may be rationalized according to the proposal by Ackermann et al. [17] for the lanthanide monoxides that ionization of UO occurs by removal of an electron from a molecular orbital that possesses some anti-bonding character. In the context of this model, an electron with some bonding character is presumably removed during ionization of ThO.

It is noteworthy that IE[UO_2] is only ~0.1 eV greater than IE[UO] and is slightly smaller than IE[U]. The ionization energy of UO_2 can be compared with those of uranium tetrahalides, in which uranium is in the same formal valence state. Dyke et al. [22] determined IE[UF_4] = 9.51 eV and IE[UCl_4] = 9.18 eV by photoelectron spectroscopy. The ionization energies of these tetravalent uranium halide molecules are $\geqslant$3 eV larger than IE[U]. A possible explanation for the large discrepancy between the ionization energies of uranium(IV) dioxide and uranium(IV) tetrahalides is the comparative degrees of ionic character. The relatively large ionization energies of the tetrahalides are consistent with a significant degree of ionic character, such that the effective positive charge at the uranium metal center is substantial, where it is presumed that ionization of these molecules occurs by removal of an electron occupying a molecular orbital of predominantly uranium character. Referring to the second atomic ionization energies in Table 2, it is evident that the uranium tetrahalide ionization energies are intermediate between IE[U] and IE[U$^+$], suggesting an effective charge δ+ ($0 < \delta < 1$) at the metal center and a significant degree of ionicity in the tetrahalides. The contrasting observation that IE[UO_2] ≈ IE[U] suggests that the bonding in the dioxide is essentially covalent. Dyke et al. [22] also determined IE[ThF_4] = 12.75 eV and IE[$ThCl_4$] = 11.16 eV; these values are similar to IE[Th$^+$] = 11.9 eV (Table 2). In the case of tetravalent thorium compounds the large ionization energies can be taken to reflect the fact that no free valence electrons remain at the metal center outside of the closed Rn core. The relatively large EI value for IE[ThO_2] included in Table 1 is consistent with tetravalent thorium, regardless of the degree of ionic character for this dioxide molecule.

3.3. Second ionization energies of actinide oxides

The second ionization energies of some AnO and AnO_2 were recently determined using FTICR-MS techniques [10] – the values from these studies are included in Table 1. The IE[AnO$^+$] have rather large associated uncertainties and only an upper limit is available for IE[ThO$^+$]. However, it is apparent the IE[AnO$^+$] (An = U, Np, Pu, Am) are rather similar to – perhaps ~1–2 eV greater than – the corresponding IE[An$^+$] (see Tables 1 and 2). Based on the upper limit, it would appear that IE[ThO$^+$] is within 1 eV of IE[Th$^+$]. These observations indicate that just as for the AnO, the bonding in these AnO$^+$ is largely covalent with the result that the ionization energies of the bare and oxo-ligated An$^+$ are similar. Whereas the neutral tetrahalide ionization energies were employed as a basis to evaluate IE[AnO_2] (An = U, Th) no such basis for comparison is available for the monopositive AnO$^+$ and AnO_2^+ ions. That the IE[AnO$^+$] are evidently ~1–2 eV greater than the IE[An$^+$] (An = U, Np, Pu, Am) might initially be taken to suggest that the oxo-ligand withdraws some additional charge from the metal center so that the effective net charge there is slightly greater than +1. However, it should be noted that the addition of the oxo-ligand to An$^+$ might involve the highest-energy valence electrons at the metal ion center in covalent bonding, thereby resulting in the necessity for removal of a lower-energy electron for ionization and a higher ionization energy. It is noted that the third ionization energies of these actinide atoms, IE[An^{2+}], are in the range of 20–22 eV [20], so it can be inferred that the degree of charge withdrawal by the oxo-ligand is minor – i.e., that the effective charge at the metal centers in the AnO$^+$ are close to +1 and much less than +2.

The recently determined [10] approximate values for IE[UO_2^+], IE[NpO_2^+] and IE[PuO_2^+], as well as an estimate for IE[AmO_2^+] [10], are included in Table 1. These IE[AnO_2^+] are ~2.5–3.5 eV greater than the corresponding IE[An$^+$]. In contrast, IE[UO_2] and IE[NpO_2] are within ~0.1 eV of IE[U] and IE[Np], respectively, whereas IE[PuO_2] and IE[AmO_2] are only ~1 eV greater than IE[Pu] and IE[Am], respectively (see Tables 1 and 2). These comparisons might tentatively be taken to suggest that the net effective charges on the metal center for these four AnO_2^+ are somewhat greater than +1. However, in analogy with the AnO^+, the addition of a second oxygen ligand might employ high-energy metal-based electrons in the additional covalent bonding, resulting in an elevated ionization energy absent any significant increase in net charge at the metal center. Ab initio theory on the electronic structures of the actinide oxide neutrals and ions is needed to understand the observed trends in the experimental ionization energies.

4. Summary

The ionization energy of neptunium dioxide has been determined by the electron-transfer bracketing method employing FTICR-MS, with the result: $IE[NpO_2] = 6.33 \pm 0.18$ eV. This value is substantially greater than the previous experimental value of ~5.0 eV [1] but is in accord with the ionization energy trends for the early actinide dioxides, including the revised values recently reported for UO_2 [2] and PuO_2 [5], as well as with other thermodynamic quantities for the neptunium–oxygen system [5,6].

The ionization energies of ThO, UO, NpO, PuO, AmO, UO_2 and NpO_2 are all close – within ~0.3 eV – to those of the corresponding bare actinide atoms. This is taken to indicate that ionization of these oxides occurs by removal of an electron with little bonding character and that the bonding is primarily covalent so that the net effective charges at the actinide metal centers are close to zero. The ionization energies of PuO_2 and AmO_2 are ~1 eV greater than IE[Pu] and IE[Am], respectively, which is taken to indicate that ionization occurs by removal of an electron with some bonding character and/or that there is a slight effective positive charge at the metal center. Similarities between the second ionization energies of actinide monoxides and those of the corresponding bare metal ions suggest a minor degree of electron withdrawal as a result of addition of a single oxygen ligand to an An^+ ion. In contrast, the ionization energies of UO_2^+, NpO_2^+ and PuO_2^+, as well as the estimate for $IE[AmO_2^+]$, are ~3 eV greater than those of the corresponding An^+. Theoretical studies of the neutral, monopositive and dipositive actinide oxide molecules are needed to understand the nature of the ionization processes.

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