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Nitrosyl and dioxygenyl cations and their salts—Similar but further investigation needed

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This paper is dedicated to Henry Selig on the occasion of his winning the 2008 Award for Creative Work in Fluorine Chemistry given by the American Chemical Society.

ABSTRACT

The O_2^+ ion is sufficiently similar in size to NO^+ and so the difference in lattice energy in salts with a common anion can be expected to be negligible. Comparative analysis of O_2F *vs.* NOF and of O_2^+ *vs.* NO^+-salts is given in this paper and some surprising and unexpected differences between corresponding species are discussed.

1. Introduction

Oxygen and nitric oxide are both colorless, odorless, tasteless diatomic gases at ambient temperature. The former is necessary for the majority of the living organisms, the latter for many. Both species are extremely important for the chemical industry. They are both paramagnetic, the O_2 with two and NO with one-electron in antibonding π orbitals. For both species it is possible to oxidatively remove an electron to form the NO^+ and O_2^+ ions with a shorter and stronger bonds than in the neutral diatomics NO or O_2 themselves. The NO^+ is sufficiently similar in size to O_2^+ (as defined by bond length or molecular volume; $V(NO^+) = (0.010 \pm 0.010)$ nm^3; $V(O_2^+) = (0.015 \pm 0.011)$ nm^3 [1]) that the difference in lattice energy in salts with a common anion can be expected to be negligible. However, there are several significant differences between O_2 and NO. The ionization potential [2] of NO 893.900(2) kJ/mol is appreciably lower than for O_2 1164.60(2) kJ/mol. To calibrate our thinking, the nearly 300 kJ/mol difference for NO and O_2 is greater than the ca. 230 kJ/mol difference of the highly reactive metal Li (520.22 kJ/mol) and the rather unreactive Cu (745.494 kJ/mol). This last difference is, in turn, even greater than that of Cu with the noble metal Au (890.155 kJ/mol) that generally requires oxidants such as aqua regia and elemental fluorine to result in formation of $AuCl_4^-$ and AuF_6^- salts with Au(III) and Au(V), respectively [3]. Said differently, both the NO^+ and O_2^+ ions are strong one-electron oxidizers, where the later is considerably stronger than the former [4–6]. The electron affinities of NO and O_2 are in the reverse order, 2.5(5) kJ/mol and 43.2(6) kJ/mol [7] – acknowledging relatively big standard deviations for the electron affinities in comparison to ionization energies – as are the oxidizing powers of the two neutral diatomic molecules.

The oxidizing power of O_2 is well known, although it is rarely the case that this arises from the above-cited puny electron affinity

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at work removing an electron to form the anion O_2^-. The same trends are seen for the related triatomics, NO_2 and O_3 which are much poorer reducing agents, but in fact, quite powerful oxidants. However interesting, we now defer the discussion of nitrogen dioxide and ozone to other studies and limit ourselves in this paper to O_2F and NOF and to discussion of salts containing O_2^+ and NO^+ ions.

2. O_2F and NOF

The O_2F is a radical [8] that exists only at low temperature. It is thermodynamically unstable towards O_2 and F_2. Reactions between the fluorobasic alkali fluoride salts (or suitable fluoro-amphoteric molecules [9]) and O_2^+-fluoride-salts in anhydrous HF at 248 K [10] lead to the formation of free O_2F radicals which readily decompose to O_2 and atomic F. The extraordinary oxidizing capability of solution of solvated O_2F in aHF was shown by oxidation of Au(III) to Au(V) [10]. Solvated O_2F is a more potent oxidizer than F_2 and more akin to atomic F or KrF_2.

NOF has a melting point of 140.65 K and boiling point of 213.25 K. At ambient temperature it is stable monomeric gas as are most nitroso species with electron withdrawing groups attached. Beside NOF, chlorine and bromine analogues, i.e. NOCl and NOBr, are also known. They both decompose reversibly even at room temperature. Nonetheless, they are far more stable and less labile than their O_2 counterparts, by which we mean ClOO and BrOO and not the more classical (and less thermodynamically stable) triatomics with the halogen sitting, perhaps more sensibly and certainly more symmetrically, in the middle.

3. O_2^+ and NO^+-salts

3.1. Syntheses

NOF readily reacts with a variety of binary fluorides to form NO^+-salts. Beside fluoride, other NO^+ non-fluoride-salts as $NOClO_4$ and $NOSbCl_6$ are also known. Briefly harkening back to NO_2^+, we note a recent attempt of synthesizing NO_2ClO_3 that resulted in the synthesis of $NOClO_4$ [11].

In the case of O_2^+ only fluorine containing compounds are known. The first one, which was prepared was O_2PtF_6 [12,13]. Its preparation was one of the most important steps in the history of chemistry. This was the decisive discovery which led to the preparation to the first compound of xenon and to the initiation of noble-gas chemistry. A large number of different O_2^+ salts have been subsequently prepared. They are conveniently prepared by UV-photolysis of oxygen/fluorine mixtures in the presence of respective Lewis acid fluorides [14,15]. O_2AsF_6 and O_2SbF_6 could be prepared using a photochemical-reaction exposure of O_2/F_2 and appropriate pentafluoride mixture to daylight [16]. Other synthetic paths involve reactions between O_2F_2 and corresponding Lewis acid, thermal syntheses by the reaction of some pentafluorides with O_2/F_2 mixture [17] or with OF_2 under pressure [14,18]. No evidence for O_2F^+ or OF^+ salts has been offered [19], although both cations are well-established in the gas phase [19,20] and proposed as mechanistic intermediates [21]. On the basis of quantum chemical calculations, the OF_3^+ cation should be stable as an isolated ion in the gas phase. Thermodynamic estimations on the basis of Born–Haber energy cycles show that the formation of O_2AsF_6 from $OF_2/F_2/AsF_5$ mixture is thermodynamically favorable with respect to OF_3AsF_6 salt [22]. The reaction of RuO_2 with KrF_2 in aHF leads to O_2RuF_6 [23]. We note now that the one published synthetic attempt of synthesizing O_3^+ salts resulted in those of O_2^+, when a product other than broken apparatus (i.e. explosions) was observed [24]. With few exceptions, the known O_2^+ salts generally have $[MF_6]^-$ or $[M_2F_{11}]^-$ (M = metal in oxidation state 5+) as a counteranion [25].

3.2. Crystal structures and O–O and N–O bond lengths in O_2^+ and NO^+-salts

Due to the similar sizes of O_2^+ and NO^+, the corresponding O_2^+ and NO^+-salts are usually isomorphous [26,27,3]. Most of the available crystallographic data are for AMF_6 salts. Due to the orientational disorder of the cations, the accuracy and precision of determined bond lengths in O_2^+ and NO^+ (as is seen from the data that follow) are very often problematic.

The O–O bond distances in O_2^+ cations have been reported for β-O_2AuF_6 (106.8(30) pm) [28], α-O_2AuF_6 (107.9(27) pm at 104 K) [29], $[O_2][Mn_2F_9]$ (110 pm) [30], O_2PtF_6 (121(17) pm) [31], O_2RuF_6 (112.5(17) pm at 146 K and 112(4) pm at 293 K) [32] with threefold model for disordered cation. Because of the orientational disorder of the O_2^+ cations, the O–O bond distance (101.7 pm) in $O_2Ni(AsF_6)_3$ appears short [25]. Anomalously short O–O bond lengths were reported for $[O_2]_2[Ti_7F_{30}]$ (96 pm) [33] and for O_2MF_6 (95, 96, 97 and 100 pm for M = Sb, Pt, Au and Ru, respectively) [34].

The N–O bond length in solid NOF is equal to 108.3(3) pm (at 128 K), meanwhile in the gas phase it has been reported to be 113.15(4) pm [35]. The N–O distances have been also reported for solid $[NO(NOF)_2][IF_8]$ (102.1–107.7(6) pm at T = 110 K) [36], $[NO]_2[BrO_3F_2][F]$ (1.038(9)/1.066(8) pm at 100 K) [37], $[NO][HF_2]\cdot IF_5$ (102.2(5) pm at 120 K) [38].

The paucity of data unencumbered by orientational disorder precludes a simple comparison of NO^+ and O_2^+ salts with the same counteranion. Indeed, there are few cases for which both cations form corresponding salts. Is this because O_2 is generally too poor of an oxidant so preventing the O_2^+ salt forming? This is presumably the case for such species as $NOClO_4$. Alternatively, is it because NO is a sufficiently powerful reductant that it overreduces the anion, e.g. the formation of the tetravalent hexafluorometallate PdF_6^{2-} as opposed to forming the Pd(V) counterpart, as was erroneously suggested to be the case in O_2PdF_6? [39,40]. The ONF_3 was obtained in a good yield by the pyrolysis of $(NO)_2NiF_6$ in fluorine (4.8 bar) at about 350 °C, itself prepared by the reaction of NiF_2 and a mixture of fluorine (~4.1 bar) and an excess of NOF (3.4 bar) in a Nickel vessel held at ~200 °C [41,42].

3.3. Stretching frequencies and ionic nature

The values of O_2^+ and NO^+ stretches are strongly dependent on the nature of the counter anions (Tables S1 and S2 in Supporting Material). The relationship between O_2^+ stretching frequency, the ionic nature of the salt and the influence of Lewis basicity of the anion is often treated [28] in terms of molecular orbital theory. The lengthening of the O–O bond (and decreasing of $\nu(O_2^+)$) in O_2^+ salts is caused by donation of electron density from the HOMO of the anion to the LUMO of the cation. Relations between $\nu(O_2^+)$ and fluoride ion affinities (FIA) for different O_2^+ respectively NO^+-salts are presented in Table 1. AuF_5 is one of the strongest Lewis acids and BF_3 is a weaker Lewis acid than many MF_5 compounds. According to that, the O_2^+ frequency of $[AuF_6]^-$ salt seems to be to low, meanwhile the corresponding value for $[BF_4]^-$ salt appears too high. The same behavior is evident also for NO^+-salts.

The comparison of O_2^+ stretching frequencies in O_2MF_6 (A = P, As, Sb) with much lower value for O_2AuF_6 indicates that the $\nu(O_2^+)$ is not influenced by the competing FIA of O_2^+ and parent pentafluorides (Table 1) [28]. According to Ref. [28], the O_2^+ vibrational frequency is strongly influenced by the valence electron configuration of the central anion and, to a lesser extent, by the electron affinity of the neutral fluoride species. When the central atoms of the anion have closed shell electron configura-

Table 1
Relation between $\nu(O_2^+)/\nu(NO^+)$ and fluoride ion affinities (FIA) for different O_2^+- and NO^+-salts.

O_2^+ salt	$\nu(O_2^+)$ [cm^{-1}]	Ref.	NO^+-salt	$\nu(NO^+)$ [cm^{-1}]	Ref.	FIA [kJ/mol]	Ref.
O_2AuF_6	1837	[28]	$NOAuF_6$	2326	[43]	591.0	[29]
–	–	–	$NOPF_6$	2339	[43]	397.1	[44]
O_2GeF_5	1849	[45]	–	–	–	347.3	[44]
O_2AsF_6	1858	[46]	$NOAsF_6$	2339	[43]	443.1	[44]
O_2BF_4	1860	[25]	$NOBF_4$	2340	[47]	347.7	[44]
O_2SbF_6	1861	[46]	$NOSbF_6$	2342	[46]	503.3	[44]
$O_2Sb_2F_{11}$	1864	[46]	–	–	–	609 (liquid)	[48]
						671 (gas)	[48]

tions, then corresponding O_2^+ salts have the highest $\nu(O_2^+)$ value. In the same paper [28], it was also discussed, how the charge density influence the $\nu(O_2^+)$, since low $\nu(O_2^+)$ values were observed for dianion O_2^+ salts (MF_6^{2-}, M = Ni, Mn, Pd). In Supporting Material the list of NO stretching frequencies, $\nu(NO^+)$, for $(NO)_2MF_6$ (M = Ni [41], Cr, Pd, Ru, Rh, Ir and Pt [42]), which are dianion salts, is given. Similar trend as in the case of dianion O_2^+ salts is observed, i.e. the $\nu(NO^+)$ stretching frequencies of dianion NO^+-salts are lower then those displayed by the monoanion NO^+-salts.

It should also be emphasized that in general, the electron affinity of the neutral species cannot dominate the explanation for the stability of salts. Consider the archetypal MX species, the alkali metal halides. The lattice energies for the fluorides are higher than the other halides and their enthalpies of formation are more negative. However, the electron affinity [7] of atomic fluorine (328.1790(3) kJ/mol) is less than that of chlorine (348.6625(42) kJ/mol), and indeed is only slightly higher than that of bromine (324.5414(42) kJ/mol). It does not suffice to say that fluorides are more ionic than the other halides because the electronegativity of fluorine is higher than the heavier halogens because electronegativity is not a measured quantity. Rather, it may be argued that fluorine is smaller than the other elements and hence the lattice energy is higher by simple application of Coulomb's law and classical electrostatics, "volume based thermodynamics" (VBT) offering quantitative refinements with encouragingly simple equations [49]. Furthermore, the "second electrons" of few doubly charged anions are bound. For example, free $[SO_4]^{2-}$ is unstable relative to $[SO_4]^-$ and a free electron. This does not mean that sulfates contain univalent anions and that there should be $NaSO_4$ and not $MgSO_4$, the latter as a 2:2 salt rather than 1:1. Again, the enhanced Coulombic attraction involving double charged anions is sufficiently greater than that for single charged anions and so sulfate is found in $[SO_4]^{2-}$ in both sodium and magnesium salts. However, should the anion be too small and too polarizing, then the salt (or more correctly, the ion pair) collapses to form a covalent species. After all, as said above, O_2F, O_2Cl, NOF and NOCl are not dioxygenyl and nitrosonium fluoride salts (despite the existence of $[NO]_2[BrO_3F_2][F]$ [37]) but covalent triatomic molecules, however unstable, labile and reactive.

In principle, one should be able to gain understanding through a comparative analysis of lattice energies of diverse NO^+ and O_2^+ salts, obtained in turn from enthalpies of formation of nitrosonium and dioxygenyl salts. For many such species this analysis is facilitated by the high symmetry of the anion. More precisely, we may quite reliably approximate the chemical electrostatics of these ions with tetrahedral or octahedral symmetry as having all of the charge located on the central metal ion much as the solid state salt and gas phase ion complexation energetics of tetrahedral NH_4^+ is well-described by putting all of the charge of the cation on the central nitrogen [50]. Accordingly, the NH_4^+ ion is prone to free rotation in many of its environments and this is why the charge acts as though it were concentrated at the centre of the sphere by a well known theorem of electrostatics. This may be understood in terms of the regular polyhedral shape as found in neutral AB_n, $n = 4$ or 6, that lack nonzero dipole and quadrupole moments and corresponding relatively long-range electrostatic interaction.

There is one direct thermochemical comparison that can be made of corresponding nitrosonium and dioxygenyl salts. This is for the $[SbF_6]^-$ salt. The enthalpies of formation of a collection of $[SbF_6]^-$ salts were discussed in Refs. [48,51] studies that included the alkali metals and O_2^+. Since the lattice energy of NO^+-salts and Cs^+ salts have been equated [52] we can now posit almost thermoneutrality (i.e. $\Delta H(1) \approx 0$ kJ mol^{-1}) for the reaction:

$$NOSbF_6(s) + Cs^+(g) \overset{\Delta H(1)}{\longrightarrow} CsSbF_6(s) + NO^+(g) \tag{1}$$

$$\begin{aligned} \Delta H(1)/\text{kJ mol}^{-1} &\approx U_{POT}(NOSbF_6) - U_{POT}(CsSbF_6) \\ &= 566 - 571 = -5 \end{aligned} \tag{2}$$

and further:

$$\begin{aligned} \Delta_f H^\circ(NOSbF_6, s)/\text{kJ mol}^{-1} &\approx \Delta_f H^\circ(CsSbF_6, s) + \Delta_f H^\circ(NO^+, g) - \Delta_f H^\circ(Cs^+, g) - \Delta H(1) \\ &= (\{-2082 \pm 15\} + 990 - 458 + 5) = -1545 \pm 15 \end{aligned} \tag{3}$$

Similarly, for the reaction:

$$O_2SbF_6(s) + Cs^+(g) \overset{\Delta H(2)}{\longrightarrow} CsSbF_6(s) + O_2{}^+(g) \tag{4}$$

$$\begin{aligned} \Delta H(2)/\text{kJ mol}^{-1} &\approx U_{POT}(O_2SbF_6) - U_{POT}(CsSbF_6) \\ &= 560 - 571 = -11 \end{aligned}$$

so that:

$$\begin{aligned} \Delta_f H^\circ(O_2SbF_6, s)/\text{kJ mol}^{-1} &\approx \Delta_f H^\circ(CsSbF_6, s) + \Delta_f H^\circ(O_2^+, g) - \Delta_f H^\circ(Cs^+, g) - \Delta H(2) \\ &= (\{-2082 + 15\} + 1172 - 458 + 11) = -1357 + 15 \end{aligned} \tag{6}$$

hence the enthalpy change, $\Delta H(3)/\text{kJ mol}^{-1}$ for the reaction:

$$NOSbF_6(s) + O_2{}^+(g) \overset{\Delta H(3)}{\longrightarrow} O_2SbF_6(s) + NO^+(g) \tag{7}$$

is given by

$$\Delta H(3)/\mathrm{kJ\,mol^{-1}} \approx U_{\mathrm{POT}}(\mathrm{NOSbF_6}) - U_{\mathrm{POT}}(\mathrm{O_2SbF_6}) = 566 - 560 = +6 \quad (8)$$

or, equivalently, by

$$\Delta H(3)/\mathrm{kJ\,mol^{-1}} \approx \Delta_f H^\circ(\mathrm{O_2SbF_6,s}) + \Delta_f H^\circ(\mathrm{NO^+,g}) - \Delta_f H^\circ(\mathrm{NOSbF_6,s}) - \Delta_f H^\circ(\mathrm{O_2^+,g}) = \{-1357 + 15\} + 990 - (-1545 \pm 15) - (1172) = +\{6 \pm 21\} \quad (9)$$

In contrast to these VBT calculations, from Ref. [52] we find the enthalpy of formation of O_2SbF_6 is reported to be -1468 ± 40 kJ/mol. This assumption leads to the conclusion that $\Delta H(3)/\mathrm{kJ\,mol^{-1}}$ for reaction (7) should have a rather larger exothermic enthalpy change estimated to be:

$$\Delta H(3)/\mathrm{kJ\,mol^{-1}} \approx \Delta_f H^\circ(\mathrm{O_2SbF_6,s}) + \Delta_f H^\circ(\mathrm{NO^+,g}) - \Delta_f H^\circ(\mathrm{NOSbF_6,s}) - \Delta_f H^\circ(\mathrm{O_2^+,g}) = (\{-1468 \pm 40\} + 990 - (-1545 \pm 15) - 1172) = -\{105 \pm 43\} \quad (10)$$

These latter numbers suggest an exothermicity of ca. 100 $\mathrm{kJ\,mol^{-1}}$—a very different value from the expected result. Bearing in mind the difficult experimental thermochemistry associated with this area and the rather large uncertainties recorded, since $\Delta_f H^\circ(\mathrm{O_2SbF_6,\ s}) = -1468(40)\ \mathrm{kJ\,mol^{-1}}$ [52] and $\Delta_f H^\circ(\mathrm{CsSbF_6,\ s}) = -2082(15)\ \mathrm{kJ\,mol^{-1}}$ [52], these authors suggest it might be timely to redo these experimental measurements. This could then in order to settle the question as to whether NO^+ and O_2^+ are "*thermochemically*" anomalus in their behavior or whether the conformity, as found in other areas of their chemistry, is maintained in their thermodynamics.

4. Conclusions

We conclude that nitrosyl, NO^+, and dioxygenyl, O_2^+, cations and their salts are similar but certainly not the same as unexpected differences between corresponding species as O_2F *vs.* NOF. To determine whether these similarities truly extend to the thermochemistry of these materials re-determination of some standard enthalpies of formation is recommended.

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Appendix A. Supplementary data

Supplementary data associated with this article can be found, in the online version, at doi:10.1016/j.jfluchem.2009.06.012.

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