

Twenty-five years of noble gas chemistry

John H. Holloway

This year we celebrate the 25th anniversary of the discovery of noble gas chemistry. The discovery toppled a belief that had grown for almost 70 years—that no member of the family of 'inert gases' could form a thermodynamically stable chemical compound. Now, a quarter of a century on, it is appropriate to look back at the origins, development and more noteworthy recent advances in the field.

In March 1962 Bartlett and Lohmann reported the preparation of $O_2^+[PtF_6]^-$.¹ The production of O_2^+ , by simply mixing oxygen with an equimolar quantity of platinum hexafluoride at room temperature, showed PtF_6 to be 'an oxidizer of unprecedented power',² capable of oxidising molecular oxygen. Because the first ionisation potential of molecular oxygen (12.2 eV) is slightly more than that of xenon (12.13 eV) Bartlett thought that xenon gas might be similarly oxidised. Within a month he had confirmed his suspicion and, in June he published a report of his observation of the spontaneous and rapid oxidation of xenon by the deep red PtF_6 vapour to give a solid, yellow-orange, quinquivalent platinum complex.³

The papers outlining the discoveries appeared in the *Proceedings of the Chemical Society*,^{1,3} the predecessor of

Chemistry in Britain. The second of these short papers, which was less than 250 words in length (Fig. 1), initiated what was to become a flood of activity in an entirely new area of chemistry. Before the end of August 1962, Howard Claassen, Henry Selig and John Malm had prepared the first binary fluoride of a noble gas, XeF_4 ,⁴ and by the end of the year, Rudolf Hoppe and his group had announced the synthesis of XeF_2 ,⁵ Jožef Slivnik and his coworkers the preparation of XeF_6 ,⁶ and Paul Fields, Lawrence Stein and Moshe Zirin the first evidence of the existence of a radon fluoride.⁷ By early 1963* the existence of compounds of krypton, xenon and radon was firmly established.¹¹

Time has confirmed the validity of these early observations. Stable compounds are formed only with the heavier gases, and the only stable compounds

that can be made by direct synthesis are the fluorides. Most compounds that have been obtained consist of noble gas atoms bonded to the most electronegative elements, fluorine and oxygen, but species bonded to chlorine, nitrogen and carbon have also been reported. Xenon chemistry is the most extensive—Xe is known in oxidation states ranging from +2 to +8, while krypton and radon species are known only in oxidation state +2. Stable compounds of helium, neon and argon are *not* known, but experimental estimates of the bond strength in the $[ArF]^+$ cation¹² suggest that continued efforts to lock this into an adduct are worthwhile.

Xenon fluorides^{11,13}

Apart from XeF , which has been obtained as an unstable free radical, there is no evidence for the existence of fluorides in odd-numbered oxidation states. The fluorides, XeF_2 , XeF_4 and XeF_6 can be obtained by reacting xenon directly with fluorine, but early reports of XeF_8 have not been confirmed.

- The difluoride is prepared by heating an excess of xenon with fluorine or by irradiating mixtures of the gases with ultraviolet light or sunlight.
- The tetrafluoride is prepared by heating xenon and fluorine under pressure in a ratio of 1:5 by volume.
- The hexafluoride is prepared by heating xenon and fluorine under pressure in a ratio of 1:20.

All the xenon fluorides are colourless solids except the hexafluoride, which is yellow in the liquid and gaseous states. The di- and tetrafluorides have low volatilities, and linear and square planar structures in the solid and vapour states. Of the 16 known hexafluorides, xenon hexafluoride has the highest boiling point. This suggests that, unlike the other hexafluorides, this compound might be polymeric in the solid state. However, uncertainty about its structure remains, mainly because it is readily hydrolysed to the explosive XeO_3 and its strong fluorinating ability damages most container and window materials, making it difficult to study. Unlike other hexafluorides it is not octahedral in the vapour but is probably a non-rigid (floppy), distorted octahedron.¹⁴ When molten or

Fig. 1. Bartlett's short paper announcing the discovery of $Xe^+[PtF_6]^-$ and carrying his signature. Reprinted from *Proceedings of the Chemical Society*, June 1962, p 218.

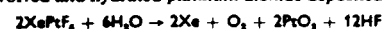
Xenon Hexafluoroplatinate(v) $Xe^+[PtF_6]^-$

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A RECENT Communication¹ described the compound dioxygenyl hexafluoroplatinate(v), $O_2^+[PtF_6]^-$, which is formed when molecular oxygen is oxidised by platinum hexafluoride vapour. Since the first ionisation potential of molecular oxygen,² 12.2 eV, is comparable with that of xenon,² 12.13 eV, it appeared that xenon might also be oxidised by the hexafluoride.

Tensimetric titration of xenon (AIRCO "Reagent Grade") with platinum hexafluoride has proved the existence of a 1:1 compound, $XePtF_6$. This is an orange-yellow solid, which is insoluble in carbon tetrachloride, and has a negligible vapour pressure at room temperature. It sublimes in a vacuum when heated and the sublimate, when treated with water vapour, rapidly hydrolyses, xenon and oxygen being evolved and hydrated platinum dioxide deposited:



¹ Bartlett and Lohmann, *Proc. Chem. Soc.*, 1962, 115.

² Field and Franklin, "Electron Impact Phenomena," Academic Press, Inc., New York, 1957, pp. 114–116.

³ Kapustinskii, *Quart. Rev.*, 1956, 10, 284.

The composition of the evolved gas was established by mass-spectrometric analysis.

Although inert-gas clathrates have been described, this compound is believed to be the first xenon charge-transfer compound which is stable at room temperatures. Lattice-energy calculations for the xenon compound, by means of Kapustinskii's equation,³ give a value ~ 110 kcal. mole⁻¹, which is only 10 kcal. mole⁻¹ smaller than that calculated for the dioxygenyl compound. These values indicate that if the compounds are ionic the electron affinity of the platinum hexafluoride must have a minimum value of 170 kcal. mole⁻¹.

The author thanks Dr. David Frost for mass spectrometric analyses and the National Research Council, Ottawa, and the Research Corporation for financial support. (Received, May 4th, 1962.)

Neil Bartlett

*Early in 1963 it emerged that three other groups,⁸⁻¹⁰ working independently, had all prepared XeF_6 at about the same time.

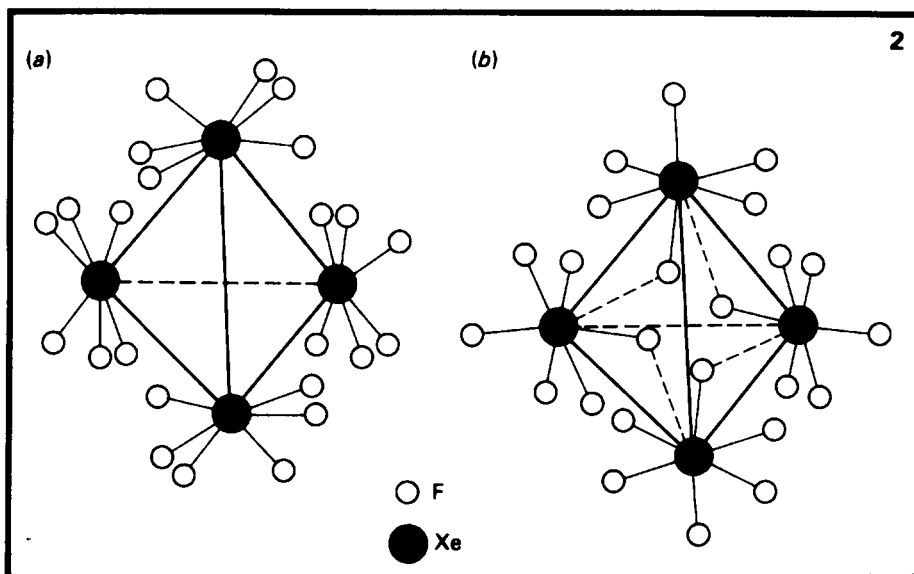


Fig. 2. The structure of liquid XeF_6 .

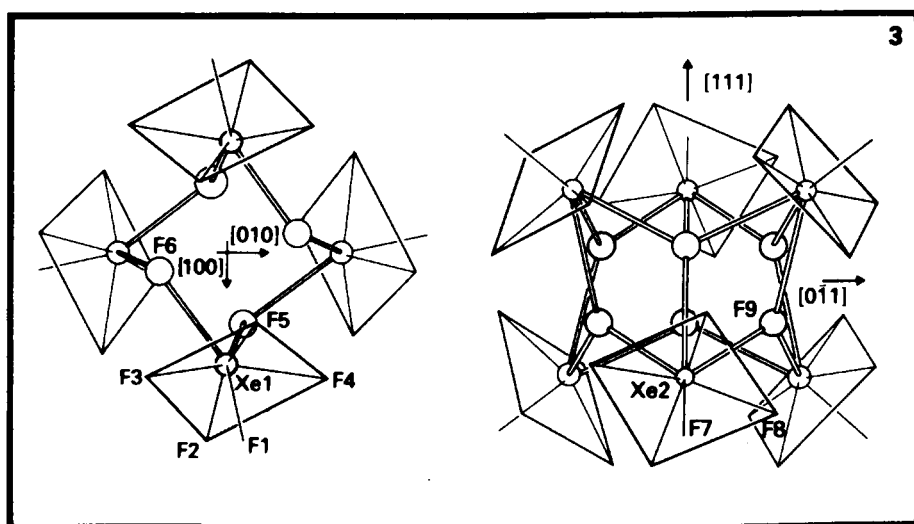


Fig. 3. The crystal structure of the cubic phase of XeF_6 (-80°C). One unit cell contains 144 ' XeF_6 ' units, 24 tetramers and eight hexamers.¹⁸

in solution the monomer is in equilibrium with tetrameric rings consisting of $[\text{XeF}_5]^+$ and F^- ions, but at low temperatures the monomer disappears. The fluorines in the $([\text{XeF}_5]^+\text{F}^-)_4$ tetramers take part in a complex scrambling mechanism in which each of the four units is connected as a cogwheel to its neighbours (Fig. 2).¹⁵ In the solid, although at least four forms exist, only the cubic structure—comprising $[\text{XeF}_5]^+$ and F^- ions grouped as tetramers and hexamers within the unit cell—has been described in detail (Fig. 3).¹⁶ The fact that both hexamers and tetramers occur suggests that the way in which the $[\text{XeF}_5]^+$ ion is bridged is not critical. Furthermore the bridging F^- ions are not close to the four-fold axis of the $[\text{XeF}_5]^+$ groups. This suggests that in keeping with the Gillespie and Nyholm valence shell electron-pair repulsion theory, which has provided the most accurate and simple rationalisation of the shapes of noble gas compounds, a lone pair of electrons may occupy this position.

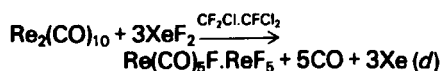
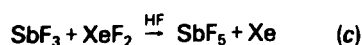
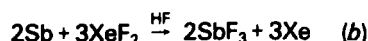
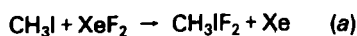
Reactions of the xenon fluorides

With the exception of the reactions of powerful oxidative fluorinating agents such as PtF_6 , the chemistry of xenon has

stemmed from reactions of the binary fluorides.

● *As fluorinating agents.* The difluoride is a versatile mild fluorinating agent.^{17,18} It provides routes to some aromatic compounds which are otherwise difficult to make and reacts with carbon-carbon double and triple bonds, giving addition products. The cleanliness of these reactions is of special significance with some of the products, which are of medicinal and biological importance. One example is the ability of XeF_2 to convert uracil to 5-fluorouracil, one of the first antitumour agents.

Xenon difluoride is also a common inorganic oxidative fluorinating agent, see equations (a)–(d).¹⁷



Recently it was used in the preparation of CrOF_3 .¹⁹ Soluble also in water, the difluoride is stable in neutral or acid

solution and powerfully oxidising, converting for example 2Cl^- to Cl_2 , Cr(III) to Cr(VI) and even $[\text{BrO}_3]^-$ to $[\text{BrO}_4]^-$.¹⁷ The tetra- and hexafluorides are progressively more powerful oxidisers and difficult to control. Their instant hydrolysis by water, however, has provided interesting oxides, oxide fluorides and powerfully oxidising xenate and perxenate salts.

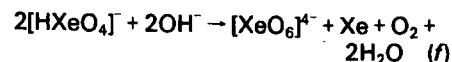
● *Hydrolysis reactions.* The difluoride, XeF_2 , is stable in acid or neutral solution but decomposes immediately in base, liberating xenon, oxygen and HF. The tetrafluoride is instantly hydrolysed and, depending upon conditions, up to a third of the xenon may be retained in solution (rather than the half expected in a simple disproportionation, $2\text{Xe(IV)} \rightarrow \text{Xe(VI)} + \text{Xe(II)}$), and XeF_2 may also be liberated. It has been suggested²⁰ that the initial product may be XeOF_2 , which decomposes to give XeF_2 if the water supply is limited. When larger amounts of water are present XeO_2 and XeO may be formed, which can then give XeO_3 .

The more vigorous reaction of XeF_6 with water can be controlled by passing dry nitrogen over crystalline XeF_6 to sweep the vapour into water where the following reaction occurs:



Thus the hydrolysis of both XeF_4 and XeF_6 give the trigonal pyramidal XeO_3 in solution. Aqueous solutions of XeO_3 (known as 'xenic' acid) are quite stable but solid XeO_3 , which is easily obtained from the solutions, is a sensitive and violent explosive.

● *Reactions giving oxides, oxide fluorides and oxygen-containing salts.* In aqueous solution XeO_3 is a powerful, but kinetically slow, oxidising agent. The addition of alkali yields 'xenate' ions, $[\text{HXeO}_4]^-$, and, although the salts can be isolated, solutions containing xenate disproportionate readily to give the 'perxenate' ion, $[\text{XeO}_6]^{4-}$, and xenon:



The Xe(VIII) solutions are among the most powerful oxidising agents known. For example, the oxidation of Mn(II) to $[\text{MnO}_4]^-$, unlike that by XeO_3 , is immediate and is accompanied by the evolution of oxygen.

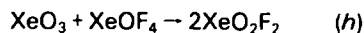
The addition of solid Ba_2XeO_8 to cold concentrated H_2SO_4 produces a second oxide, XeO_4 , which is an unstable and explosive gas. Understandably, it has been studied little, but infrared spectroscopy and electron diffraction have shown it to be a tetrahedral molecule.

Of the known oxide fluorides, XeOF_2 , XeOF_4 , XeO_2F_2 and XeO_3F_2 , the most stable is the liquid, XeOF_4 . Colourless and volatile, with a square pyramidal structure, it derives from the controlled hydrolysis of XeF_6 :



This reaction, equation (g), readily goes further to produce XeO_3 , but the expected intermediate, XeO_2F_2 , has never been isolated from the reaction. But,

XeO₂F₂ can be produced *via* the interaction of xenon trioxide with the oxide tetrafluoride.



The related and possibly even more hazardous reaction with XeO₄ yields XeO₃F₂:



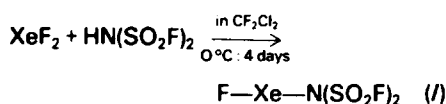
● **Substitution reactions.** Reactions involving the replacement of fluorine in xenon fluorides with other groups began with studies of reactions of XeF₂ with anhydrous oxygen-containing acids:¹⁷



In all cases, the linkage to xenon is *via* oxygen (L = OTeF₅, OSeF₅, OSO₂F, OPOF₂, ONO₂, OClO₃, OIF₄O, OCOCH₃ or OCOCF₃). Only those species containing the highly electronegative —OTeF₅ and —OSeF₅ units are stable under ordinary conditions. The —OSO₂F and —OIF₄O derivatives decompose readily and the remainder are explosive.

The high electronegativity of the —OTeF₅ group means that Xe(OTeF₅)₂ is similar to XeF₂ and an interesting reaction of Xe(OTeF₅)₂ is that with halogenoalkanes such as CF₂=CFCl, CF₂=CCl₂ and CF₂=CFH, which results in the addition of —OTeF₅ groups across the double bond.²¹ The development of substitution reactions involving Xe(IV) and Xe(VI) compounds has required the use of the reagent B(OTeF₅)₃, an obvious relative of BF₃. Reactions involving this molecule have given the only Xe(IV) compound to date, Xe(OTeF₅)₄, while Xe(VI) gives Xe(OTeF₅)₆, OXe(OTeF₅)₄, O₂Xe(OTeF₅)₂ and, under certain conditions in solution, the related partly substituted derivatives. Like many of the Xe(II) compounds, most of these are also unstable.

Because noble gas chemistry is mostly associated with Xe—F and Xe—O bond formation the key to the successful bonding of xenon to other atoms to yield stable compounds lies in producing suitable highly electronegative groups. Such a group is —N(SO₂F)₂ which has provided the route to the first Xe—N bond:²²



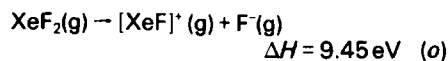
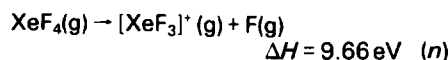
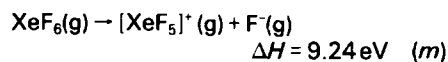
The solid, once formed, is reasonably stable. It contains a linear F—Xe—N group and a planar configuration about the nitrogen.^{22,23} Its reaction with AsF₅ yields the solid, [(FSO₂)₂N—Xe—F—Xe—N(SO₂F)₂]⁺[AsF₆][−], containing a V-shaped cationic relative of [Xe₂F₃]⁺.

Although, in general, the noble gases form bonds with only the most electronegative elements, fluorine and oxygen, the success in making an Xe—N bond in compounds has increased speculation as to whether an Xe—C bond is possible. A plasma-induced reaction of XeF₂ vapour with CF₃ radicals may have provided the answer. The volatile, waxy, white solid

product of the reaction, which decomposes to give XeF₂ and carbon fluorides at room temperature, is thought to be Xe(CF₃)₂.²⁴

● **Reactions with fluoride-ion acceptors and donors.**²⁵ Reactions of fluoride-ion donors and acceptors with the binary fluorides and oxide fluorides of xenon give a variety of cations and anions (Table 1). More unusual entities such as [Xe₂]⁺ and [XeO₃X][−] (X = F, Cl or Br) also occur. The representation of many of the complexes as salts, however, is an oversimplification, especially for the xenon difluoride adducts. Also, some adducts of XeF₂—for example, those with XeOF₄ and the halogen pentafluorides—are not donor-acceptor compounds but simple molecular adducts in which the component molecules preserve their molecular identities.

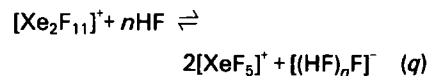
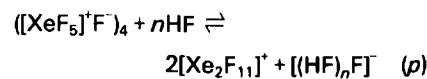
The enthalpies of ionisation of the binary fluorides:



imply that complex formation might be progressively easier in the sequence XeF₄ < XeF₂ < XeF₆. The chemical evidence supports this. Xenon difluoride reacts with AsF₅, SbF₅, BiF₅, and a range of transition metal pentafluorides, to give adducts that are often written [XeF]⁺[M₂F₁₁][−], [XeF]⁺[MF₆][−] and [Xe₂F₃]⁺[MF₆][−] but in which the cation units are linked to the counter ion by fluorine bridges; the V-shaped [Xe₂F₃]⁺ ion can be regarded as two [XeF]⁺ units linked by a fluorine bridge. The most ionic adducts are those derived from the strongest Lewis acids. The related derivatives of MOF₄ (M = W or Mo), which are weaker Lewis acids, have less ionic character.

One of the most interesting reactions of [XeF]⁺ is its reduction with some compounds to give the bright green paramagnetic cation [Xe₂]⁺.²⁶ This cation can also be obtained by oxidising xenon with dioxygenyl salts. Furthermore, Bartlett has shown that the first noble gas compound, Xe[PtF₆][−], has the variable composition, Xe(PtF₆)_x (x = 1–2). Whether this compound contains Xe⁺, [Xe₂]⁺ or [Xe₂F]⁺ or can be described in terms of [XeF]⁺[PtF₆][−] and [XeF]⁺[Pt₂F₁₁][−] is still uncertain.

In accord with the enthalpy of ionisation data, xenon tetrafluoride forms complexes with only the strongest fluoride ion acceptors—SbF₅ and BiF₅—but xenon hexafluoride yields adducts with a number of pentafluorides and also with some tetra- and trifluorides. Solutions of XeF₆ in HF also have a higher conductivity than those of XeF₂ and XeF₄ because both [XeF₅]⁺ and [Xe₂F₁₁]⁺ are readily formed in solution:



These same two ions are found in the adducts. The [XeF₅]⁺ ion has the square pyramidal form found in XeF₆ and [Xe₂F₁₁]⁺ is best described in terms of an ionic model, [XeF₅]⁺[XeF₅]⁺, but with some covalent character in terms of ([XeF₅]⁺XeF₆) to account for the non-linearity of the Xe—F—Xe bond.

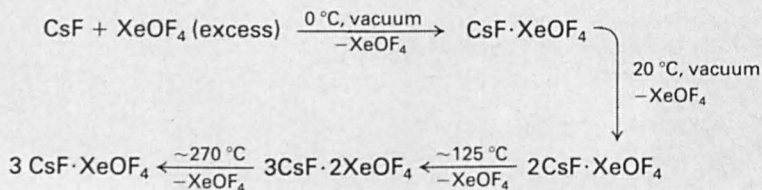
A relatively poor fluoride ion donor, xenon oxide tetrafluoride, forms adducts with only the stronger Lewis acids. Thus, with SbF₅, [XeOF₃]⁺[Sb₂F₁₁][−] and [XeOF₃]⁺[SbF₆][−] are obtained. The related dioxide difluoride, XeO₂F₂, yields [XeO₂F]⁺[Sb₂F₁₁][−] and [XeO₂F]⁺[SbF₆][−], but these are less stable. Reaction of FXeOTeF₅ with AsF₅ gives the complex [XeOTeF₅]⁺[AsF₆][−] and reactions of this complex give rise to a number of as yet uncharacterised xenon cations.

Fluoride ion acceptor behaviour is limited to XeF₆. With alkali metal fluorides it yields M₂XeF₈ (M = Cs, Rb, K and Na) and MXeF₇ (M = Cs, Rb), which lose XeF₆ when heated. The thermal stability of these adducts increases with increasing molecular weight and the low decomposition temperature of the sodium complex, which is below 100°C, provides a convenient way of separating XeF₆ from XeF₂ and XeF₄ with which no reactions occur. Solid state structures have not been determined, but the related complexes derived from NOF and NO₂F have been prepared and [NO]⁺[XeF₈]^{2−} has been shown to contain a slightly distorted square antiprismatic [XeF₈]^{2−}. Valence shell electron pair repulsion theory suggests that a ninth coordination position in which a lone pair can be accommodated should be present in this anion. However, there is no evidence for it, and this implies a rare violation of the theory in noble gas chemistry.

Table 1. The cationic and anionic derivatives of binary fluorides and oxide fluorides of xenon.²⁵

Fluorides	Cationic derivatives	Anionic derivatives
XeF ₂	[XeF] ⁺ ; [Xe ₂ F ₃] ⁺	
XeF ₄	[XeF ₃] ⁺	
XeF ₆	[XeF ₅] ⁺ ; [Xe ₂ F ₁₁] ⁺	[XeF ₇] [−] ; [XeF ₈] ^{2−}
XeOF ₄	[XeOF ₃] ⁺	[XeOF ₅] [−] ; [Xe ₃ O ₃ F ₁₃] [−]
XeO ₂ F ₂	[XeO ₂ F] ⁺	[XeO ₂ F ₃] [−]
XeOF ₂		[XeOF ₃] [−]

Scheme 1. The reaction of XeOF₄ with CsF.



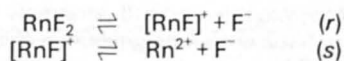
Stable white solids containing the $n\text{M}^+[\text{XeO}_3\text{F}]_n^-$ anion consist of chains of pseudo-octahedral xenon atoms linked by angular bridges. These are deposited when solutions of stoichiometric amounts of XeO₃ and HF are concentrated. The haloxenates, CsXeO₃Cl and CsXeO₃Br have also been obtained.

Complexes of XeOF₄ with CsF, RbF, KF and with NOF have been known for some time. The former are obtained by treating the alkali fluorides with an excess of XeOF₄ and pumping to constant weight under appropriate conditions (see Scheme 1). The structure of CsF·XeOF₄ contains an [XeOF₅]⁻ anion of distorted octahedral geometry with a lone pair of electrons apparently occupying an octahedral face. The CsF·3XeOF₄ adduct contains the [F(XeOF₄)₃]⁻ anion in which the XeOF₄ units, of close to square pyramidal shape, are linked *via* three Xe—F bridging bonds to a common fluorine which is out of the plane of the three xenons. Trigonal bonded fluorine is rare but, where it *does* occur (*ie* (XeF₆)₆ and NaSb₃F₁₀), the arrangements about it are closely similar.

Radon compounds^{7,13,25,27}

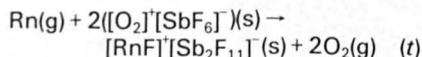
Radon chemistry has assumed increasing importance since it emerged that radon can be oxidised and complexed by certain liquid fluorides and fluoride salts. This offers the possibility of removing radon from mine atmospheres and of developing radon-monitoring devices.²⁷

The high radioactivity of radon has meant that the chemistry of radon has been explored by tracer methods. When microcurie amounts of radon are heated with fluorine a difluoride appears to be formed. The same species is also generated spontaneously when millicurie or larger amounts of radon are mixed with gaseous or liquid fluorine in a small volume; the latter reaction is brought about by the intense α-radiation. Radon dissolves in halogen fluorides as an involatile species. Electromigration studies have shown that in pure solvents the radon is present as a cation and ionisations involving the following equilibria have been suggested:



The existence of the [Rn—F]⁺ cation seems certain in view of the spontaneous reaction of radon with a variety of solids such as [O₂]⁺[SbF₆]⁻ containing oxidising

cations. By analogy with xenon chemistry, the reaction with [O₂]⁺[SbF₆]⁻ is probably:



Krypton compounds^{13,25}

Krypton chemistry is more limited than that of xenon. Although small amounts of the violet free radical, KrF, have been observed in the γ-irradiation of KrF₂, most of the chemistry derives from the difluoride. No oxides or oxide fluorides have been isolated and early reports on the preparation of KrF₄ have not been confirmed.

The volatile, colourless, crystalline difluoride is prepared by subjecting mixtures of krypton and fluorine to an electric discharge at low temperatures or by irradiating liquid krypton-fluorine mixtures with ultraviolet light.²⁸ The linear molecular structure of KrF₂ is similar to that of XeF₂ but, because of its much lower bond energy, it is a very powerful fluorinating agent capable of oxidising iodine to IF₇, xenon to XeF₆, low-valent uranium, neptunium and plutonium compounds to the hexafluorides,²⁹ and AgF₂ to AgF₃.³⁰ KrF₂ is also rapidly decomposed by water.

The adducts of KrF₂ are analogous to those of XeF₂ and are limited to cationic derivatives formed with fluoride ion acceptors. Species such as [Kr₂F₃]⁺[MF₆]⁻ (M = As, Sb, Ta), [KrF]⁺[MF₆]⁻ (M = As, Sb, V, Ta, Pt and Au) and [KrF]⁺[M₂F₁₁]⁻ (M = Sb, Nb and Ta) are known in the solid state and in solution and, more recently, compounds such as [KrF]⁺[MOF₅]⁻ (M = Cr,³¹ Mo and W³²) have been prepared and characterised. The spectra of the ions are related to those of the xenon analogues but additional peaks in the Raman for [Kr₂F₃]⁺ have been interpreted in terms of an unsymmetrical (rather than symmetrical) cation. The oxidising power of [KrF]⁺ and [Kr₂F₃]⁺ is stronger than that of KrF₂, capable of oxidising IF₅ to [IF₆]⁺, O₂ to [O₂]⁺, Xe to [XeF₅]⁺, BrF₅ to [BrF₆]⁺ and Au to [AuF₆]⁺ and [AuF₅].

Noble gas chemistry is now well established. Still inextricably linked with fluorine chemistry, it has provided a means to accomplish novel fluorinations, of introducing us to new classes of compounds and new high oxidation state species. In the next few years it promises to take us into the realms of applied chemistry, with noble gas fluorides being

used as fluorinating agents in industrial processes and radon chemistry being applied in areas of radon monitoring and atmospheric cleansing. Had it not been for Bartlett's astute chemical awareness, however, it is possible that we might still be talking of the 'inert gases'.

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References

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