The chemistry of the noble gases

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The conclusive establishment, in 1962, of the existence of compounds of the noble gases has overthrown a long-established chemical belief. This article reviews the preparation and chemistry of these compounds. The molecular structures of the simple xenon compounds are seen to correspond to those of iodine compounds having similar stoichiometries and electronic configurations. The available experimental evidence indicates that the nature of the bonding is similar to that found in other, less exotic, compounds. The article concludes with an assessment of the range of compounds the various noble gases can be expected to form.

The first true compounds of the noble gases were prepared in 1962 [1–3], more than sixty years after the discovery of the gases themselves by Sir William Ramsay. Although many claims to have made compounds in which noble gas atoms were chemically bonded to other atoms had appeared earlier, none had been conclusively established. Clathrate compounds of the noble gases had, however, been known for a number of years; indeed, P. Villard prepared the first, a hydrate of argon, shortly after the discovery of the gas itself [4]. However, there is in such compounds no chemical bonding of the noble gas, the molecules of which are simply caged by the host lattice.

Apparently Moissan, a friend of Ramsay, tried without success to bring about a reaction between argon and fluorine [5]. In his later years, Ramsay was evidently convinced of the chemical inertness of the gases, because he rejected a suggestion by G. Oddo [6] that krypton and xenon ought to form halides. Similar predictions were made by A. von Antropoff [7], who went so far as to predict a possible octavalence for the heavier gases; he and his co-workers attempted to make compounds, but they had no unequivocal success. In 1933, L. Pauling [8] saw that the heavier gases were the more likely to form compounds, and that fluorine and oxygen were the elements most likely to enter into combination with them. He suggested that hexafluorides of krypton and xenon, and an octafluoride of the latter, might be made. In the same prophetic communication he also predicted the existence of an acid H_4XeO_6 and salts of octavalent xenon, such as Ag_4XeO_6. In the same year, unsuccessful attempts were made to synthesize fluorides of argon and krypton by O. Ruff and W. Menzel [9] and of xenon by D. M. Yost and A. L. Kaye [10]. The latter workers, experimenting with electrical discharges through mixtures of xenon and fluorine, came closest to success. Unfortunately, they conducted their experiments in quartz apparatus. They concluded that the etching of their apparatus might have been due to the action of a xenon fluoride, and that the experiments did not rule out the existence of such a compound. Had their apparatus been efficiently cooled, or constructed of fluorite or alumina instead of quartz, the xenon fluorides might well have been discovered thirty years ago. In the event, the failure to confirm the existence of any of the predicted compounds, and the success of the early electronic theories of valence, which stressed the stability of the inert-gas configuration, combined to entrench the conclusion that the noble gases were incapable of chemical reaction.

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Was born in 1930 and educated at King's College, Durham. After a year as senior chemistry master at the Duke's School, Alnwick, Northumberland, he became a lecturer in chemistry at the University of British Columbia, where he is now an associate professor. His prime research interests are in inorganic fluorine chemistry, particularly the structural aspects of the subject, and in correlating molecular structure of new compounds with their electronic configuration.

Preparation of the first true noble-gas compounds

A clear indication that it should be possible to oxidize at least the heavier noble gases to produce salts arose from the discovery of the remarkable oxidizing properties of platinum hexafluoride [11]. N. Bartlett and D. H. Lohmann had established that this gas combined with molecular oxygen in a 1 : 1 molar ratio, forming a red solid. As prepared at low temperatures, this compound proved to be isomorphous with potassium hexafluoroplatinate (v), $K[PtF_6]$. (The symbol (v) refers to the valency of, in this case, the platinum). When formed at a higher temperature, it is isomorphous with potassium hexafluoroantimonate (v), $KSbF_6$. This, together with the magnetic and chemical properties of the oxygen compound, left no doubt that it was dioxygenyl hexafluoroplatinate (v), $O_2^+[PtF_6]^-$. Since the first ionization potential of molecular oxygen (12·2 eV) is almost the same as that of xenon, this element also appeared to be within the oxidizing range of platinum hexafluoride. The lattice energy of a salt is generally inversely proportional to the sum of the ionic radii, but the lattice energy of the hexafluoroplatinates is rather insensitive to cationic radii changes, because the anion is large. A computation made for the hypothetical oxidized xenon compound $Xe^+[PtF_6]^-$ showed that the lattice energy would be little more than 10 kcal less than that of the dioxygenyl salt. The predicted interaction of xenon and platinum hexafluoride was confirmed in a simple and visually dramatic experiment [1]. When deep red platinum hexafluoride vapour was mixed with an excess of xenon gas at room temperature, a yellow solid was immediately precipitated (figure 1). The quantity of xenon consumed was commensurate with a combining ratio of 1 : 1, and accordingly the compound was formulated as xenon hexafluoroplatinate (v), $Xe^+[PtF_6]^-$.

This work aroused much interest at the Argonne National Laboratory, because platinum hexafluoride and the other powerfully oxidizing hexafluorides RuF_6 and RhF_6 were first made there [12]. The Argonne workers confirmed the reaction between xenon and platinum hexafluoride, and then investigated the reaction of xenon with other hexafluorides. They observed that ruthenium hexafluoride reacted with xenon, but that more than two moles of the hexafluoride reacted with each mole of xenon [3]. Further, some reduction of the ruthenium seemed to occur. This suggested that the hexafluoride was, at least in part, acting as a fluorinating agent, and that a fluoride, or some fluorides, of xenon existed. This supposition was very quickly proved correct.

Preparation and chemistry of xenon fluorides

By heating five volumes of fluorine with one volume of xenon to 400°C in a nickel can, followed by rapid cooling in cold water, H. H. Claassen, H. Selig, and J. G. Malm [2] succeeded in making the first fluoride of xenon, xenon tetrafluoride (figure 2). Within a few weeks, they and their collaborators at Argonne

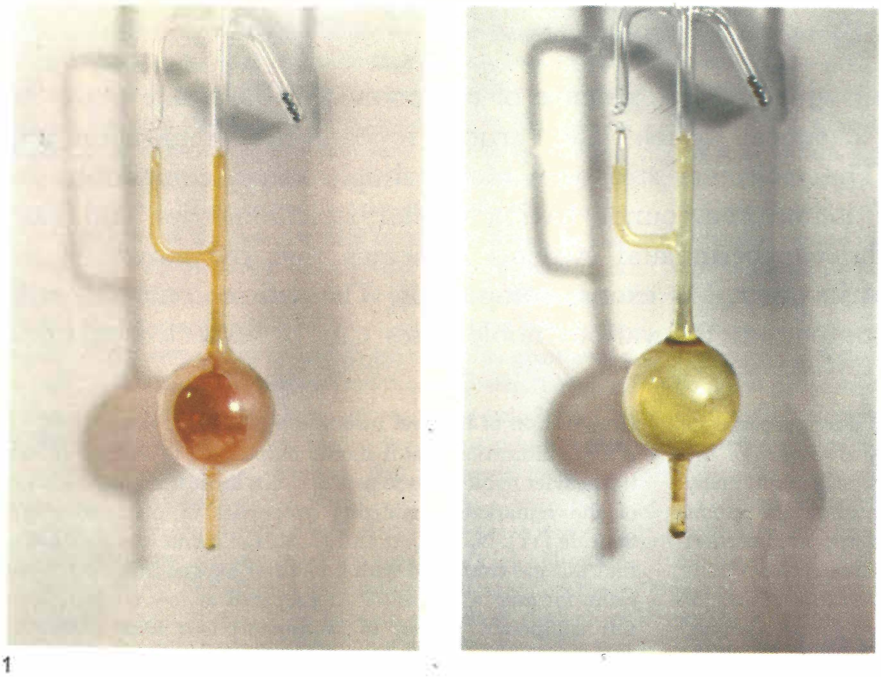

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Figure 1 The xenon–platinum hexafluoride reaction. Left: Platinum hexafluoride vapour separated from xenon by a glass diaphragm. Right: The product ($XePtF_6$) of the xenon, platinum hexafluoride reaction immediately after the breakage of the glass diaphragm.

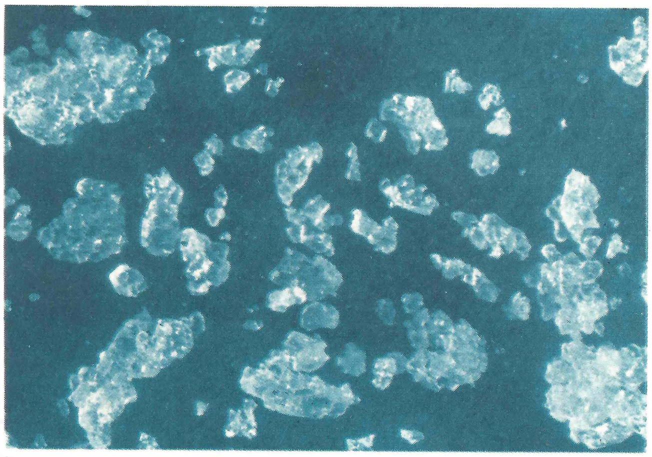

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Figure 2 Crystals of xenon tetrafluoride, seen against a blue background.

National Laboratory had also prepared the difluoride and the hexafluoride. The existence of a difluoride was first indicated by mass-spectrometric studies [3] on impure tetrafluoride samples. It was soon produced quantitatively by irradiating a xenon/fluorine mixture with light from a high-pressure mercury arc, the product being condensed in a cooled limb of the apparatus as soon as it was formed [13]. The hexafluoride was prepared by heating xenon with a large excess of fluorine at 300°C. That independent reports of the preparation of the difluoride [14] and of the hexafluoride [15] appeared simultaneously within three months of the announcement of xenon tetrafluoride testifies to the great interest aroused by that discovery.

At room temperature, all three xenon fluorides are white crystalline solids. The hexafluoride is the most volatile, having a vapour pressure of about 30 mm Hg at 25°C. It melts at 46°C, and the vapour is, surprisingly, pale yellow. The difluoride and tetrafluoride are less volatile, each having a vapour pressure of about 3 mm Hg at 25°C. The xenon fluorides are soluble in liquid hydrogen fluoride, but only the hexafluoride solution is appreciably ionized, presumably as a result of the reaction [16]

$$HF + XeF_6 \rightarrow XeF_5^+ + HF_2^-$$

The tetrafluoride solution fluorinates bright platinum to platinum tetrafluoride. All the fluorides ought to be good fluorinating agents, and it is possible that they may prove to be useful as such, because the reduction product, being xenon, is unlikely to contaminate the fluorinated material. The hexafluoride is thermally less stable than the tetrafluoride, and much more reactive. It appears that the tetrafluoride may be stored indefinitely in dry silica vessels, whereas the hexafluoride reacts rapidly to produce xenon oxide tetrafluoride:

$$2XeF_6 + SiO_2 \rightarrow SiF_4 + 2XeOF_4$$

Compounds of radon and krypton

Efforts were also made at this time to prepare compounds of other noble gases. Radon, in particular, would be expected to form compounds even more readily than xenon. Despite considerable experimental difficulties, P. R. Fields, L. Stein, and M. H. Zirin [17] have established the existence of radon fluoride. Although radon fluoride is apparently much less volatile than any of the xenon fluorides, the very small scale of the preparation does not exclude the possibility of the entire sample being either adsorbed on the nickel reactor surface, or else chemically combined with the protective coating of nickel fluoride. Large-scale preparations, which are technically difficult because of the radiation problem, are in hand and should solve this problem.

A. V. Grosse and his co-workers [18] were able to isolate krypton tetrafluoride by passing an electric discharge through krypton/fluorine mixtures at liquid-nitrogen temperature. The compound is much less stable than its xenon analogue, rapidly decomposing into the elements at 60°C. It is a transparent colourless solid, having a vapour pressure of 115 mm at 25°C.

J. J. Turner and G. C. Pimentel [16] photolysed a solid mixture of fluorine and krypton in an argon matrix at 20°K and obtained a chemical species which infra-red evidence showed to be krypton difluoride. This method, known as the matrix isolation technique, gave no evidence for the formation of an argon fluoride when a mixture of solid argon and fluorine was irradiated alone.

The molecular structures of the fluorides

X-ray and neutron-diffraction studies of the solid xenon difluoride and tetrafluoride have been made [19], and X-ray analysis has revealed a 1 : 1 adduct of the di- and tetra-fluorides [20]. In the case of xenon hexafluoride, neither the X-ray nor the neutron-diffraction studies are complete. Infra-red and

Raman spectra of the two lower fluorides have been unambiguously interpreted [21], but the hexafluoride spectra, although superficially similar to the spectra of molecules with O_h symmetry, exhibit irregularities suggestive of lower molecular symmetry [16]. In both the solid and gaseous state, the difluoride is a linear molecule. In the solid, the molecules lie parallel to one another in a body-centred tetragonal lattice array; the Xe–F distance is $2{\cdot}00 \pm 0{\cdot}01$ Å. Xenon tetrafluoride molecules show square planar coordination of the xenon by the fluorine atoms, in both the solid and gaseous states. The angle between adjacent fluorine ligands is 90°, and the bond length is $1{\cdot}953 \pm 0{\cdot}002$ Å. Infra-red evidence [16] shows that krypton difluoride has a molecular structure similar to its xenon analogue, and it is probable that the tetrafluoride will prove to have the same structure as xenon tetrafluoride.

Oxyfluorides and oxides

Xenon oxide tetrafluoride, which results from the reaction of the hexafluoride with glass, is best made [24] by simply mixing the hexafluoride with an equimolar quantity of water:

$$XeF_6 + H_2O \rightarrow XeOF_4 + 2HF$$

It is a clear, colourless, mobile liquid (m.p. −28°C). The infra-red and Raman spectra have been interpreted [16] on the basis of a model having C_{4v} symmetry, the oxygen atom being at the apex of the square pyramid and the xenon atom being almost coplanar with the four fluorine atoms.

With the fluorides well established, attempts were promptly made to prepare the oxides. Although synthesis from the elements proved to be fruitless, and pointed to the oxides being endothermic, there was an indication that a hydroxide or oxide was produced in the hydrolysis of the tetrafluoride [2]. An oxide was in fact obtained by the hydrolysis of this fluoride, and proved to be the trioxide [22]. When the first samples were obtained there was some uncertainty about the nature of the product. However, it was proved to be the trioxide by X-ray analysis [23], which showed that the material closely resembled iodic acid. Like IO_3^-, the molecule is trigonal pyramidal; the Xe—O bonds are short (1·76 Å) and so must have appreciable double-bond character. The stoichiometry of the reaction is represented approximately by the equation

$$6XeF_4 + 12H_2O \rightarrow 4Xe + 2XeO_3 + 3O_2 + 24HF$$

Although the mechanism is not understood, the xenon trioxide clearly arises by disproportionation of solvated Xe (IV) species. The disproportionation resembles those of the halogen oxy-anions, for example:

$$3ClO_2^- \rightarrow Cl^- + 2ClO_3^-$$

The trioxide is best made by the slow and complete hydrolysis of the hexafluoride [24]:

$$XeF_6 + 3H_2O \rightarrow XeO_3 + 6HF$$

It is non-volatile and dangerously explosive, being comparable in the latter respect to T.N.T. The explosive nature of the compound is compatible with its being highly endothermic ($\Delta H_f{}^\circ{}_{\text{cryst.}} = +96$ kcal mole^{-1}).

Some thermodynamic considerations

The thermal cycle

$$\begin{tikzcd}
N_{(g)} & + & \tfrac{1}{2}yX_{2(g)} \arrow[r, "\Delta H_{f(g)}{}^\circ"] \arrow[d, "y\Delta H_{f(g)}{}^\circ"] & NX_{y(g)} \\
N_{(g)} \arrow[from=u] & + & yX_{(g)} \arrow[ur, "y \times \text{average bond energy}"'] &
\end{tikzcd}$$

shows that the formation of a compound from atoms of a noble gas (N) and of a combining element (X), will be exothermic if the average bond energy of the compound exceeds the enthalpy of formation of the gaseous atom X. For thermodynamically stable compounds, under ordinary conditions of temperature and pressure, the average bond energies should therefore exceed, for fluorides, 18·3 kcal; for oxides, 59·2 kcal (double bond); and for chlorides, 29·01 kcal. From the thermodynamic data obtained for the tetrafluoride, the hexafluoride, and the trioxide (Table 1), the average bond energies for the two fluorides are seen to have the same value of 32 kcal, within experimental error. The average energy of the oxygen–xenon bond, which we can presume to be double, is less than this. If the average bond energies in the oxyfluorides are given these values, then xenon oxide tetrafluoride should be an exothermic compound with $\Delta H_f{}^\circ{}_{(g)}$

$$= -\{[(4 \times 32) + 28] - [4 \times 18{\cdot}3(\Delta H_f{}^\circ{}_{(g)}(F)) + 59{\cdot}2(\Delta H_f{}^\circ{}_{(g)}(O))]\}$$

$$= -24 \text{ kcal mole}^{-1}$$

Table 1

Thermodynamic data for XeF_4, XeF_6, and XeO_3

Compound	$\Delta H_f{}^\circ{}_{\text{cryst.}}$	$\Delta H_{\text{sub.}}$	$\Delta H_f{}^\circ{}_{\text{gaseous}}$ atoms	Average bond energy
XeF_4	−70[1]; −75[2]	15[3]	128; 133	32–33 kcal
XeF_6	−88[2]	14·9[4]	183	30·5
XeO_3	+95[4]	—	<84	<28

[1] L. Stein and P. Plurien [16].
[2] S. R. Gunn and S. M. Williamson [29].
[3] J. Jortner, E. O. Wilson, and S. A. Rice [16].
[4] C. L. Chernick [16].
[5] S. R. Gunn [16].

However, xenon dioxide difluoride should be endothermic, since

$$\Delta H_f{}^\circ{}_{(g)} = -\{[(2 \times 32) + (2 \times 28)] - [(2 \times 18{\cdot}3) + (2 \times 59{\cdot}2)]\}$$
$$= +35 \text{ kcal mole}^{-1}$$

This oxyfluoride, if it can be prepared, should prove to be a thermally unstable compound.

It is interesting that a plot of average bond energy against atomic number for the series of fluorides SbF_3, TeF_4, IF_5, XeF_6 shows a smooth relationship[1], as represented in figure 3. The values for the highest fluorides of antimony, tellurium, and iodine lie on a similar smooth curve. Extrapolation shows that the average bond energy of an octafluoride molecule ought to be approximately 24 kcal, which would mean a standard heat of formation of the gaseous molecule of −45 kcal mole^{-1}. It is possible that J. Slivnik and his co-workers [16], who have described a yellow product which could be pumped from a bath at −78°C but was retained at −196°C and had a fluorine to xenon ratio of 7·9 : 1, have already obtained this compound. The volatility is, however, greater than one would expect for an octafluoride.

The difficulty of formation and the thermal instability of krypton tetrafluoride are consistent with an average bond energy value of 18–19 kcal. This is obtained by extrapolation from values calculated for the fluorides of arsenic, selenium, and

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[1] The decrease in average bond energy, with increasing coordination number, does not mean that the intrinsic bond energy is decreasing; in fact, the reverse may be true. For the derivation of intrinsic bond energies, the valence-state promotional energies (which would be expected to increase with coordination number) for the reacting gaseous species would need to be known.

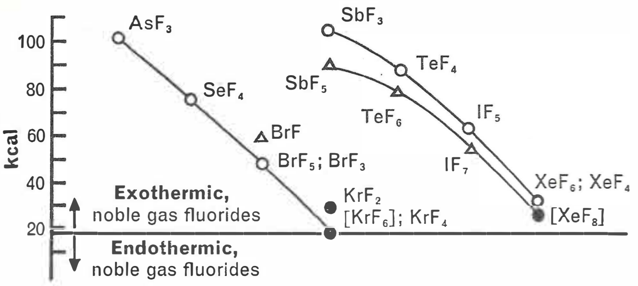

Figure 3 Average bond energy (kcal) for some fluorides. Square brackets indicate unknown compounds, open circles, experimental values; closed circles, hypothetical values.

bromine (figure 3). In view of the greater bond energy of bromine monofluoride (59·4 kcal) compared with the trifluoride and pentafluoride (48·3 and 47.7 kcal respectively), it is possible that the average bond energy for krypton difluoride may be appreciably greater than 19 kcal, making this a thermally much more stable compound than the tetrafluoride. It is of interest that Turner and Pimentel [16], by their matrix isolation technique, obtained evidence for the existence of only the difluoride.

For oxides and chlorides of the noble gases to be formed exothermally, the average single-bond energy needs to be greater than ~30 kcal because $\Delta H_{f(g)}^{\circ}$ (O) $= 59{\cdot}2$ kcal/mole and $\Delta H_{f(g)}^{\circ}$ (Cl) $= 29{\cdot}01$ kcal/mole. Consideration of the average bond energies, estimated from the available thermodynamic data for the oxides and chlorides of antimony, tellurium, and iodine (figure 4), shows, however, that the bond energies for xenon oxides and chlorides should be less than this. Consequently, the xenon oxides and chlorides should be endothermic compounds. This has been confirmed for the trioxide, which exists because of kinetic stability. It is therefore not surprising that attempts to make chlorides by synthesis from the gases failed, and an attempt [25] to prepare the chloride by interaction of boron trichloride with xenon tetrafluoride at −78° also failed:

$$3XeF_4+4BCl_3 \rightarrow 3Xe_{(g)}+6Cl_2+4BF_{3(g)}$$

The chlorides may not be kinetically stable, and hence it may not be possible to prepare them under ordinary conditions of temperature and pressure.

The nature of the bonding and of the molecular shape

There is no doubt that the chemical bonding in the noble gas compounds is similar to that found in other compounds. In particular, the bonding must be similar to that found in the inter-halogen compounds, the halogen oxides, and other halogen compounds. For example, the bond lengths, bond strengths, and molecular shapes of the fluorides of xenon are comparable to those of iodine fluorides. Since the molecular shape of xenon tetrafluoride is the same as that of the iso- or pseudo-isoelectronic ions $[ClF_4]^-$, $[BrF_4]^-$, and $[ICl_4]^-$ and the shape of the difluoride the same as $[ICl_2]^-$, it is to be expected that XeF_6 will be similar to the $[IF_6]^-$ ion. Unfortunately, the structure of the latter is unknown. There is, however, evidence that the non-bonding electron pair is sterically active, because the hexafluoroiodates (v) are not isomorphous with those hexafluorometallates that do not contain non-bonding valence electron pairs, for example $KSbF_6$. Simple valence electron-pair repulsion theory predicts molecular shapes for the noble gas compounds that are consistent with the experimental findings (figure 5) [16]. Thus the difluoride is predicted to be a linear molecule having three non-bonding electron pairs at the points of an equilateral triangle, situated in the equatorial plane of the molecule. The tetrafluoride is predicted to be square planar, having one non-bonding electron pair above and one below the plane, both on the fourfold axis of the molecule. The hexafluoride should be a slightly distorted octahedron, having a non-bonding electron pair at the centre of a face or at the mid-point of an edge. In the xenon oxide tetrafluoride molecule, the doubly bonded oxygen is predicted to lie on one side of the plane containing the xenon atom (which is square coordinated by four fluorine atoms) and the non-bonding electron pair on the other. The fluorine atoms and the xenon are predicted to be almost coplanar, because of the near equality in repulsive power of a double bond and an electron pair. The trigonal pyramid shape of the XeO_3 molecule is seen to be consistent with the presence of a non-bonding electron pair at the apex of the pyramid, this pair repelling the bonding electrons (four in each Xe=O bond) to produce the pyramid.

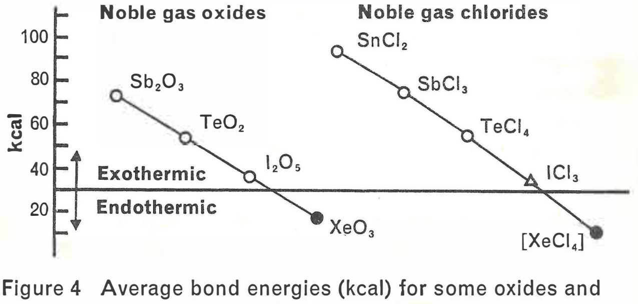

Figure 4 Average bond energies (kcal) for some oxides and chlorides. Square brackets indicate unknown compounds; open circles, experimental values; closed circles, theoretical values.

In this treatment, which is not capable of being made quantitative, all the valence electrons are treated equally, and it is implicit in the theory that *d*-orbitals are involved in the bonding. R. E. Rundle [26] and J. Hinze and K. S. Pitzer [16], however, have argued that *d*-orbital involvement should be small. Indeed, the most popular theoretical model has been the simple molecular orbital scheme; this uses *p*-atomic orbitals and was first used by Pimentel [27] to discuss inert gas

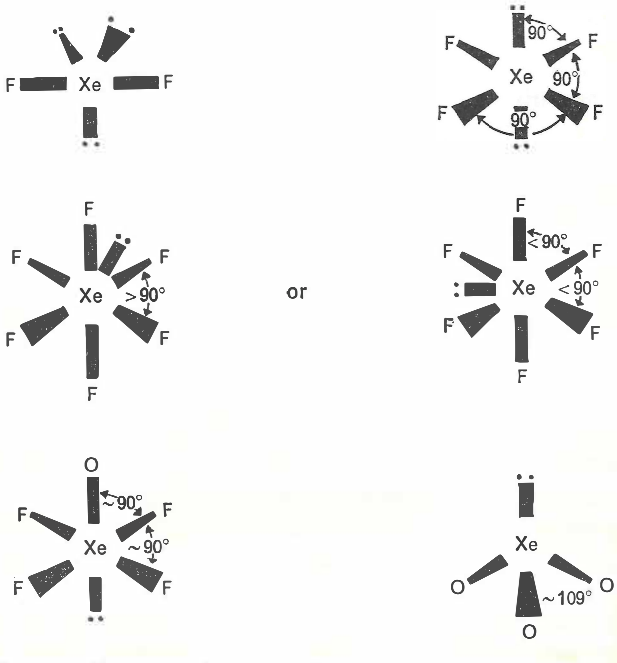

Figure 5 Molecular shapes predicted by simple valence electron-pair repulsion theory [30].

compounds qualitatively. For the xenon fluorides, Rundle has proposed linear, three-centre, four-electron bonds (F—Xe—F) in which two electrons occupy a bonding molecular orbital, ψ_μ, and two occupy a non-bonding molecular orbital, ψ_δ. The latter places the electrons mainly on the fluorine atoms, and consequently the bond must be considered to have some ionic character. Since this situation allows only one bonding pair of electrons for the two F—Xe bonds, the bond length is predicted to be greater than for a conventional electron-pair bond. This theory allows for the existence of the three xenon fluorides, XeF_2, XeF_4, and XeF_6, and correctly predicts the molecular shape of the di- and tetrafluorides, Since the linear F—Xe—F bonds should be approximately at right angles, it predicts an octahedral XeF_6.

As has been pointed out by L. C. Allen [16], the controversial theoretical situation should become clearer when the structure of xenon hexafluoride is known. Since there is an accumulating body of evidence to show that this compound is not octahedral, the case for *d*-orbital involvement in the bonding grows stronger. The preparation of an octafluoride would also prove this involvement.

Octavalent xenon

Although the octafluoride is not yet known, the anticipated octavalence of xenon [8] has been attained with the isolation of the perxenates. The reaction of xenon hexafluoride with sodium hydroxide was shown by J. G. Malm and his colleagues [16] to give white crystalline solids which contain xenon in the +8 oxidation state. Several hydrates were stated to exist, but the one that crystallizes first has recently been shown by W. C. Hamilton, J. A. Ibers, and D. R. Mackenzie [28], by X-ray structural analysis, to be $Na_4XeO_6.8H_2O$. The perxenate ion $(XeO_6)^{4-}$ is nearly regularly octahedral, having a mean Xe—O bond length of 1·875 Å. This bond is 0·12 Å longer than the average Xe—O distance in XeO_3 [23], which compares well with the increase of 0·11 Å in the I—O distances in the isoelectronic iodine-containing ions IO_3^- (1·81 Å) and IO_6^{5-} (1·93 Å). Presumably, all the electrons of the xenon outer octet are involved in the $[XeO_6]^{4-}$ bonding.

Conclusion

Approximately one hundred scientific papers have been published since the report of the first xenon compound in 1962. Never before has the picture of the chemistry of an element and the structural properties of its compounds been so rapidly developed. The limitations of the chemistry of the noble gases can now be clearly seen. It is evident that compounds of helium, neon, and argon are unlikely to be produced other than as highly unstable species; even the synthesis of a fluoride of argon appears unlikely. Although more compounds of xenon and radon may be prepared, those of xenon will probably be confined to substituted fluoride molecules (for example, CF_3XeF_5 and $ClXeF_5$); to adducts of the fluorides; to Xe (I) complexes related to $Xe^+[PtF_6]^-$; and to oxygen-containing salts. Radon may well form a chloride and even a more stable oxide than does xenon.

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