

## FLUORINE COMPOUNDS, INORGANIC, OXYGEN

### 1. Oxygen Difluoride

Oxygen difluoride [7783-41-7], OF<sub>2</sub>, is the most stable binary compound of oxygen and fluorine. Under ambient conditions, it is a colorless gas that condenses to a pale yellow liquid at  $-145^{\circ}\text{C}$  (1) and freezes at  $-224^{\circ}\text{C}$  (2). Oxygen difluoride is a powerful oxidizer that has attracted considerable attention as an ingredient of high energy rocket propellant systems (see Explosives and propellants). Several comprehensive reviews of the physical and chemical properties of OF<sub>2</sub> (3–5) and its handling (6) are available.

#### 1.1. Physical Properties

An extensive tabulation of the physical properties of OF<sub>2</sub> is available (4). Selected data are mp  $-224^{\circ}\text{C}$  (2); bp,  $-145^{\circ}\text{C}$  (1); critical temperature  $-58^{\circ}\text{C}$  (7); density of liquid, in g/mL from  $-145$  to  $-153^{\circ}\text{C}$ ,  $t$  in K,  $d = 2.190 - 0.00523 t$  (8); heat of formation 31.8 kJ/mol (7.6 kcal/mol) (9); and heat of vaporization 11.1 kJ/mol (2.65 kcal/mol) (10).

Spectroscopic investigations have shown that OF<sub>2</sub> is bent and has equivalent O—F bonds. The O—F distance is 0.139–0.141 nm and the FOF angle is  $103\text{--}104^{\circ}$  (11–13). Measurements of the dipole moment have yielded values of  $0.6 - 1.3 \times 10^{-30}$  C·m (0.18–0.40 D) (12, 14, 15). The ir (16–18), uv (19), mass (20), and nmr (21) spectra of OF<sub>2</sub> have been reported.

#### 1.2. Chemical Properties

The kinetics of decomposition of OF<sub>2</sub> by pyrolysis in a shock tube are different, as a result of surface effects, from those obtained by conventional decomposition studies. Dry OF<sub>2</sub> is stable up to  $250^{\circ}\text{C}$  (22).

##### 1.2.1. Reactions with Metals

Many common metals react with OF<sub>2</sub>, but the reaction stops after a passive metal fluoride coating is formed (3, 4).

##### 1.2.2. Reactions with Nonmetallic Elements and Inorganic Compounds

Mixtures of OF<sub>2</sub> with carbon, CO, CH<sub>4</sub>, H<sub>2</sub>, or H<sub>2</sub>O vapor explode when ignited with an electrical shock. Elemental B, Si, P, As, Sb, S, Se, and Te react vigorously on slight warming to produce fluorides and oxyfluorides. Oxides such as CrO<sub>3</sub>, WO<sub>3</sub>, As<sub>2</sub>O<sub>3</sub>, and CaO react with OF<sub>2</sub> to form fluorides. The corresponding chlorides react with OF<sub>2</sub> to form the respective fluorides and liberate free chlorine in the process (3, 4).

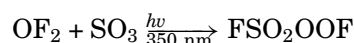
In aqueous solution, OF<sub>2</sub> oxidizes HCl, HBr, and HI (and their salts), liberating the free halogens. Oxygen difluoride reacts slowly with water and a dilute aqueous base to form oxygen and fluorine. The rate of this hydrolysis reaction has been determined (23).

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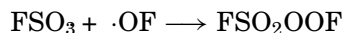
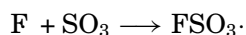
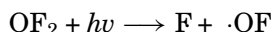
Nitric oxide and OF<sub>2</sub> inflame on contact; emission and absorption spectra of the flame have been studied (24). Oxygen difluoride oxidizes SO<sub>2</sub> to SO<sub>3</sub>, but under the influence of uv irradiation it forms sulfonyl fluoride [2699-79-8], SO<sub>2</sub>F<sub>2</sub>, and pyrosulfonyl fluoride [37240-33-8], S<sub>2</sub>O<sub>5</sub>F<sub>2</sub> (25). Photolysis of SO<sub>3</sub>-OF<sub>2</sub> mixtures yields the peroxy compound FSO<sub>2</sub>OOF [13997-94-9] (25, 26).

### 1.2.3. Oxygen Difluoride as a Source of the OF Radical

The existence of the ·OF radical [12061-70-0] was first reported in 1934 (27). This work was later refuted (28). The ·OF radical was produced by photolysis of OF<sub>2</sub> in a nitrogen or argon matrix at 4 K. The existence of the ·OF species was deduced from a study of the kinetics of decomposition of OF<sub>2</sub> and the kinetics of the photochemical reaction (25, 26):



The existence of the ·OF radical was further established by use of <sup>17</sup>O-labeled compounds and <sup>17</sup>O nmr studies to verify the mechanism (29):



The ·OF radical has also been detected by CO<sub>2</sub> laser magnetic resonance (30). The O—F bond length is 0.135789 nm.

Carbonyl fluoride, COF<sub>2</sub>, and oxygen difluoride react in the presence of cesium fluoride catalyst to give bis(trifluoromethyl)trioxide [1718-18-9], CF<sub>3</sub>OOOCF<sub>3</sub> (31). CF<sub>3</sub>OOF has been isolated from the reaction in the presence of excess OF<sub>2</sub> (32).

### 1.2.4. Reactions with Organic Compounds

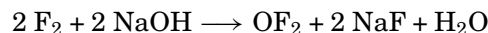
Tetrafluoroethylene and OF<sub>2</sub> react spontaneously to form C<sub>2</sub>F<sub>6</sub> and COF<sub>2</sub>. Ethylene and OF<sub>2</sub> may react explosively, but under controlled conditions monofluoroethane and 1,2-difluoroethane can be recovered (33). Benzene is oxidized to quinone and hydroquinone by OF<sub>2</sub>. Methanol and ethanol are oxidized at room temperature (4). Organic amines are extensively degraded by OF<sub>2</sub> at room temperature, but primary aliphatic amines in a fluorocarbon solvent at -42°C are smoothly oxidized to the corresponding nitroso compounds (34).

The reaction of OF<sub>2</sub> and various unsaturated fluorocarbons has been examined (35, 36) and it is claimed that OF<sub>2</sub> can be used to chain-extend fluoropolyenes, convert functional perfluorovinyl groups to acyl fluorides and/or epoxide groups, and act as a monomer for an addition-type copolymerization with diolefins.

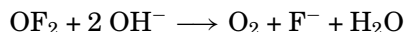
## 1.3. Preparation

The synthesis of OF<sub>2</sub> was first achieved by the electrolysis of molten KHF<sub>2</sub> in the presence of water (37). The electrolysis of aqueous HF in the presence of O<sub>2</sub> and O<sub>3</sub> was also found to produce OF<sub>2</sub> (38-40).

The most satisfactory method of OF<sub>2</sub> generation is probably the fluorination of aqueous NaOH (3, 22, 41–45):



Yields of greater than 60% are obtained (46). This method has been used for the commercial production of OF<sub>2</sub> (8). The NaOH concentration, however, must be kept low to avoid the loss of product by a secondary reaction:



An economic study of the preparation of OF<sub>2</sub> is available (47).

#### 1.4. Analytical Procedures

Oxygen difluoride may be determined conveniently by quantitative application of ir, nmr, and mass spectroscopy. Purity may also be assessed by vapor pressure measurements. Wet-chemical analyses can be conducted either by digestion with excess NaOH, followed by measurement of the excess base (2) and the fluoride ion (48, 49), or by reaction with acidified KI solution, followed by measurement of the liberated I<sub>2</sub> (4).

#### 1.5. Handling and Safety Factors

Oxygen difluoride can be handled easily and safely in glass and in common metals such as stainless steel, copper, aluminum, Monel, and nickel, from cryogenic temperatures to 200°C (4). At higher temperatures only nickel and Monel are recommended. The compatibility of OF<sub>2</sub> with process equipment depends largely on the cleanliness of the equipment; contaminants such as dirt, moisture, oil, grease, scale slag, and pipe dope must be avoided. Equipment should be passivated with elemental fluorine before contact with OF<sub>2</sub>.

Oxygen difluoride must be regarded as a highly poisonous gas, somewhat more toxic than fluorine. It has a foul odor with a limit of detectability of 0.1–0.5 ppm. Repeated exposure of rats to 0.5 ppm OF<sub>2</sub> produced death; repeated exposure to 0.1 ppm, however, caused no discernible effects.

## 2. Dioxygen Difluoride

Dioxygen difluoride [7783-44-0], O<sub>2</sub>F<sub>2</sub>, prepared by passing a 1:1 mixture of O<sub>2</sub> and F<sub>2</sub> through a high voltage electric discharge tube cooled by liquid nitrogen, has also been prepared by uv irradiation of O<sub>2</sub> and F<sub>2</sub> (50, 51) and by radiolysis of liquid mixtures of O<sub>2</sub> and F<sub>2</sub> at 77 K using 3 MeV bremsstrahlung (52). Heating an O<sub>2</sub>/F<sub>2</sub> mixture to 700°C in stainless steel tubes followed by rapid cooling produces O<sub>2</sub>F<sub>2</sub> (53). This compound is also obtained in high yield by subjecting a flowing gas mixture of F<sub>2</sub> to microwave, then downstream and outside of the region of discharge, introducing molecular oxygen (54).

#### 2.1. Physical Properties

Because O<sub>2</sub>F<sub>2</sub> is unstable, it is difficult to purify. Consequently, some of the reported physical properties are open to question. Selected data are density, in g/mL, from –87 to –156°C,  $d = 2.074 - 0.00291 t$  (50); heat of formation 19.8 kJ/mol (4.73 kcal/mol) (55); and heat of vaporization 19.2 kJ/mol (4.58 kcal/mol) at –57°C (55).

The structure of O<sub>2</sub>F<sub>2</sub> is that of a nonlinear FOOF chain, having the following molecular constants (56, 57): O—O distance, 0.122 nm; OOF angle, 109°30'; dihedral angle, 87°30'; dipole moment,  $4.8 \times 10^{-30}$  C·m (1.44 D). Additional physical and spectral data are summarized in References 4 and 58.

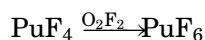
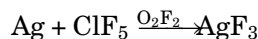
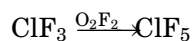
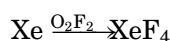
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### 2.2. Chemical Properties

The bond distance of O—O is relatively short ( $121.7 \pm 0.3$  pm) and that of O—F is relatively long ( $157.5 \pm 0.3$  pm) (56). The weakest bond in  $O_2F_2$  is thus the O—F bond and the mechanisms of reaction of  $O_2F_2$  can probably be explained by the formation of  $F\cdot$  and  $\cdot OOF$  and not two  $\cdot OF$  radicals. The  $\cdot OOF$  radical [15499-23-7] is a feasible intermediate as it has been shown to exist at low temperatures (56, 59–61). If  $O_2F_2$  is allowed to react quickly with other compounds, simple fluorination usually results. The controlled reactions of  $O_2F_2$ , however, yield products that appear to be formed via an  $\cdot OOF$  intermediate.

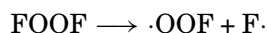
### 2.3. Simple Fluorination Reactions

Some examples (62) of  $O_2F_2$  acting mainly as a fluorinating agent are

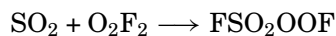


### 2.4. Reactions Involving an $\cdot OOF$ Intermediate

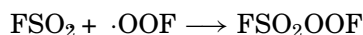
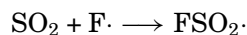
In controlled reactions of  $O_2F_2$  and various compounds,  $^{17}O$  tracer studies and other techniques have shown that the first step in the reaction appears to be



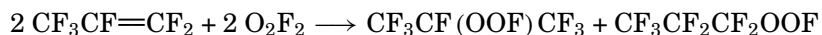
For example:



where the proposed mechanism (63) is

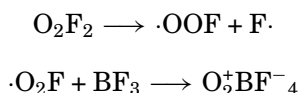


Also:



in which the proposed mechanism (64) involves the transfer of an OOF group.

The formation of a new class of compounds, dioxygenyls, containing  $O_2^+$ , is also thought to take place via an  $\cdot OOF$  intermediate (65).



A number of fluorides have been shown to form  $O_2^+$  compounds upon reaction with  $O_2F_2$ .

### 3. Uses

Oxygen difluoride is mainly a laboratory chemical. It has been suggested as an oxidizer for rocket applications and has been used for small tests in this area.

Dioxygen difluoride has found some application in the conversion of uranium oxides to  $UF_6$  (66), in fluorination of actinide fluorides and oxyfluorides to  $AcF_6$  (67), and in the recovery of actinides from nuclear wastes (68) (see Actinides and transactinides; Nuclear reaction, waste management).

### 4. Higher Oxygen Fluorides

Several higher oxygen fluorides,  $O_3F_2$  [16829-28-0] (50, 69),  $O_4F_2$  [12020-93-8] (70),  $O_5F_2$  [12191-79-6] (71), and  $O_6F_2$  [12191-80-9] (71), and radicals such as  $\cdot O_3F$  (72, 73) have been reported. Only  $\cdot OF$ ,  $OF_2$ ,  $O_2F_2$ ,  $\cdot OOF$ , and  $O_4F_2$ , however, have been satisfactorily characterized. From cryogenic mass spectroscopy, it appears that  $O_3F_2$  consists of loosely bonded  $\cdot O_2F$  and  $\cdot OF$  radicals (74). The  $^{19}F$  nmr spectrum of  $O_3F_2$  suggests an  $O_3F_2$  model consisting of  $O_2F_2$  and interstitial oxygen (75). However,  $^{19}F$  and  $^{17}O$  nmr (7, 76), and other studies have shown that  $O_3F_2$ , as reported in the literature, is actually a mixture of  $O_4F_2$  and  $O_2F_2$ .

Little is known about  $O_4F_2$ . It has been reported to behave similarly to  $O_2F_2$  in that it can act as a fluorinating agent or a source of the  $\cdot OOF$  radical. In fact, it appears to be a better source of the  $\cdot OOF$  radical than  $O_2F_2$  in its reactions with  $SO_2$  and  $BF_3$ .

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I. J. SOLOMON  
IIT Research Institute  
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