

FLUORINE COMPOUNDS, INORGANIC, FLUOROSULFURIC ACID

Fluorosulfuric acid [7789-21-1], HSO_3F , is a colorless-to-light yellow liquid that fumes strongly in moist air and has a sharp odor. It may be regarded as a mixed anhydride of sulfuric and hydrofluoric acids. Fluorosulfuric acid was first identified and characterized in 1892 (1). It is a strong acid and is employed as a catalyst and chemical reagent in a number of chemical processes, such as alkylation (qv), acylation, polymerization, sulfonation, isomerization, and production of organic fluorosulfates (see Friedel-Crafts reactions).

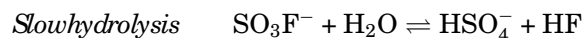
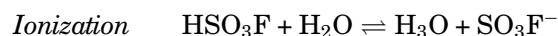
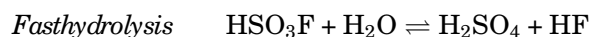
1. Properties

Selected physical properties of fluorosulfuric acid are shown in Table 1. Fluorosulfuric acid is soluble in acetic acid, ethyl acetate, nitrobenzene, and diethyl ether, and insoluble in carbon disulfide, carbon tetrachloride, chloroform, and tetrachloroethane. Many inorganic and organic materials dissolve in fluorosulfuric acid; the physical and chemical properties of such solutions have been extensively investigated (5–8). The structure of fluorosulfuric acid has been determined (9), and the ir, Raman, and nmr spectra have been reported (10). The solution of antimony pentafluoride [7783-70-2] in fluorosulfuric acid results in a superacid possessing protonating power orders of magnitude greater than 100% sulfuric acid. Extensive studies on the properties of superacid compositions containing fluorosulfuric acid have been published (11, 12).

Fluorosulfuric acid is stable to heat up to decomposition at about 900°C (13), where vapor-phase dissociation into hydrogen fluoride and sulfur trioxide probably occurs. Reviews of the chemistry and properties of fluorosulfuric acid have been published (14–16).

1.1. Reactions

The reaction of fluorosulfuric acid and water is violent and exothermic; it proceeds as follows:



The extent of the initial hydrolysis depends on temperature and how the water is added. Hydrolysis is reduced at slower addition rates and lower temperatures. The hydrolysis subsequent to the initial fast reaction is slow,

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Table 1. Physical and Chemical Constants of Fluorosulfuric Acid

Property	Value ^a	References
molecular weight	100.07	
boiling point, °C	162.7	(1, 2)
freezing point, °C	−88.98	3
density, g/mL	1.726	2
viscosity, mPa·s(=cP)	1.56	2
dielectric constant	ca 120	2
specific conductance, (Ω·m) ^{−1}	1.085 × 10 ^{−6}	2
heat of formation, ^b ΔH _f , kJ/mol ^c	792.45	4

^aAll values at 25°C.

^bFrom SO₃ and HF.

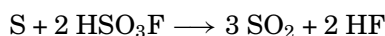
^cTo convert kJ to kcal, divide by 4.184.

Table 2. Reactions of Fluorosulfuric Acid and Inorganic Compounds

Reactant	Product		References
	Name	Formula	
nitrogen oxides	nitrosyl fluorosulfate, nitryl fluorosulfate	FSO ₃ NO, FSO ₃ NO ₂	(26, 27)
H ₃ BO ₃	boron trifluoride	BF ₃	28
KClO ₄	perchloryl fluoride	ClO ₃ F	29
KMnO ₄	manganese(VII) fluoride trioxide	MnO ₃ F	30
As ₂ O ₃	arsenic trifluoride	AsF ₃	31
As ₂ O ₅	arsenic pentafluoride	AsF ₅	31
CrO ₃	difluorodioxochromium (VI)	CrO ₂ F ₂	32
P ₄ O ₁₀	phosphoryl fluoride	POF ₃	31
H ₂ S in ethanol	monothiosulfuric acid	H ₂ S ₂ O ₃	33

presumably because part of the acid is converted to fluorosulfate ions which hydrolyze slowly even at elevated temperatures. The hydrolysis in basic solution has also been studied (17). Under controlled conditions, hydrates of HSO₃F containing one, two, and four molecules of water have been observed (18, 19).

The pure acid does not react in the cold with sulfur, selenium, tellurium, carbon, silver, copper, zinc, iron, chromium, or manganese, but slowly dissolves mercury and tin (20). At higher temperatures, lead, mercury, tin, and sulfur react rapidly, eg:



Precipitated (hydrated) silica reacts vigorously with fluorosulfuric acid to give silicon tetrafluoride [7783-61-1] (21), but glass (qv) is not attacked in the absence of moisture (20). Alkali and alkaline-earth metal chlorides are readily converted to fluorosulfates by treatment with fluorosulfuric acid (7, 13, 22, 23).

Electrolysis of fluorosulfuric acid produces either S₂O₆F₂ [13709-32-5] (24) or SO₂F₂ [13036-75-4] plus OF₂ (25), depending on specific conditions. Various reactions of fluorosulfuric acid with inorganic compounds are shown in Table 2, and with organic compounds in Table 3.

Other studies which have been reported describe unusual chemistry such as HSO₃F–Nb(SO₃F)₅ systems (42). Also the unique properties of fluorosulfuric acid have been found to provide unusual solvent systems, which can vary properties such as acidity, heats of solution, enthalpy, and heats of neutralization (43).

Fluorosulfuric acid may be used to prepare diazonium fluorosulfates, ArN⁺₂SO₃F[−] (44), which decompose on heating to give aryl (Ar) fluorosulfates (36, 45). Aryl fluorosulfates are also obtained from arylsulfonyl

Table 3. Reactions of Fluorosulfuric Acid and Organic Compounds

Reactant	Product		References
	Name	Formula	
benzene ^a	benzenesulfonic acid	C ₆ H ₅ SO ₃ H	(20, 34)
	diphenyl sulfone	(C ₆ H ₅) ₂ SO ₂	34
	benzenesulfonyl fluoride	C ₆ H ₅ SO ₂ F	35
aliphatic amines	amidosulfuric acids	R ₂ NSO ₃ H or RNHSO ₃ H	36
aromatic compound	arylsulfonyl fluorides ^b	ArSO ₂ F	
carboxylic acids	acid fluorides	RCOF	37
alcohol or alkene	alkyl fluorosulfates	ROSO ₂ F	(20, 38, 39)
perhaloolefins or perhaloalkyl iodides	perhaloalkyl fluorosulfates	R _f OSO ₂ F	(40, 41)

^aProduct is dependent on reaction conditions and proportions of reagents.

^bAr represents an aryl group.

chlorides and fluorosulfuric acid (35). Alkyl and other organofluorosulfates form during electrolysis of fluorosulfuric acid in the presence of organic species (46, 47).

1.2. Preparation and Manufacture

Fluorosulfuric acid, first prepared by combining anhydrous HF and cooled, anhydrous SO₃ in a platinum container (1), has also been prepared from ionic fluorides or fluorosulfates and sulfuric acid (20, 48). The reaction of chlorosulfuric acid (qv) with ionic fluorides also gives fluorosulfuric acid (49).

Commercially, fluorosulfuric acid is made by processes utilizing the product as a solvent. Solutions of HF and SO₃ in fluorosulfuric acid are mixed in stoichiometric quantities, or SO₃ and HF are separately introduced into a stream of fluorosulfuric acid to produce essentially pure HSO₃F. Some of the product is then recycled (50, 51).

Fluorosulfuric acid can be very corrosive. A study of the corrosive properties of fluorosulfuric acid during preparation and use showed carbon steel to be acceptable up to 40°C, stainless steel up to 80°C, and aluminum alloys up to 130°C (52).

1.3. Economic Aspects

U.S. manufacturers of fluorosulfuric acid are AlliedSignal and Du Pont. These companies have a combined annual capacity estimated at 20,000 metric tons, most of which is used internally although some merchant sales exist. Fluorosulfuric acid is shipped in tank cars.

1.4. Specifications and Analysis

Commercial fluorosulfuric acid contains approximately 98% HSO₃F and approximately 1% H₂SO₄ and lesser amounts of sulfur trioxide and dioxide. No free HF is present.

The free sulfur trioxide can be titrated with water; the end point is determined conductimetrically. The sulfuric acid content is determined from the specific conductivity of the liquid at the point in the titration where no free SO₃ or excess water is present. If the presence of HF is suspected, a known amount of SO₃ is added to the acid and the excess SO₃ is determined as above. The content of another common impurity, SO₂, may be determined iodometrically in a dilute, aqueous solution.

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1.5. Health and Safety Factors

Fluorosulfuric acid is a strong acid capable of causing severe burns similar to those experienced with sulfuric and hydrofluoric acids. In addition, the fumes of fluorosulfuric acid are extremely irritating, and breathing of the fumes is to be avoided. Precautions and first aid measures generally observed in handling strong sulfuric acid and hydrofluoric acid are applicable to fluorosulfuric acid. Small containers of fluorosulfuric acid should be well cooled before opening and precautions taken to relieve any gas pressure that may have developed. In the laboratory, fluorosulfuric acid may be handled in glass if water is not present; otherwise, containers of inert polymers or platinum should be used. For larger-scale equipment, iron or carbon steel (not stainless steel) may be used. Material safety data sheets and other literature from manufacturers describe additional precautions in handling large quantities of fluorosulfuric acid.

1.6. Uses

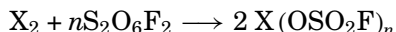
Fluorosulfuric acid serves as catalyst in the alkylation (qv) of branched-chain paraffins (53–58) and aromatic compounds (59), and in the polymerization of monoolefins (60) and rosin (61). Addition of strong Lewis acids, such as SbF_5 , TaF_5 , and NbF_5 , to fluorosulfuric acid markedly increases the system acidity and catalytic activity (62–69). Other examples which show the marked catalytic effect of fluorosulfuric acid alone or in systems including SbF_5 , etc, are the synthesis of methyl *tert*-butyl ether (70), the stereospecific formation of 2-naphthol (71), formation of aromatic aldehydes using CO (72), and polymerization of tetrahydrofuran (73, 74) (see CAtalysis).

As a reagent, fluorosulfuric acid has been employed in the preparation of boron trifluoride (28), silicon tetrafluoride (75, 76), alkyl fluorosulfates (20, 38), arenesulfonyl fluorides (35), acyl fluorides (77), sulfamic acid (78), and diazonium fluorosulfates (44). Among its other uses are the removal of small amounts of organic fluorides from petroleum alkylate made by the hydrogen fluoride process (79), the removal of HF from process exhaust gases (80), the removal of HF from F_2 (81), and as a constituent of baths for electropolishing metals (82, 83) and glass polishing (84).

1.7. Derivatives

The nonmetallic inorganic derivatives of fluorosulfuric acid are generally made indirectly, although complex fluorosulfates of the Group 15 (V) elements and of xenon can be made directly (85, 86), as can the NO^+ and NO^+_2 salts (26, 27).

Peroxydisulfuryl difluoride [13709-32-5], $\text{FSO}_2\text{OOSO}_2\text{F}$, prepared from fluorine and SO_3 (87), is a ready source of fluorosulfate radicals, $\text{FSO}_2\text{O}\cdot$, (88) which react with many substances to form stable fluorosulfates (89, 90). By using the route



where $n = 1$ or 3, compounds of the type FOSO_2F [13536-85-1] (91), BrOSO_2F [13997-93-8], and $\text{I}(\text{OSO}_2\text{F})_3$ [13709-37-0] (92) have been prepared. Fluorosulfates of most metallic elements have been prepared but none have any commercial significance. The physical properties of some fluorosulfates are summarized in Table 4.

Ammonium fluorosulfate is produced from ammonium fluoride by reaction with sulfur trioxide, oleum, or potassium pyrosulfate, $\text{K}_2\text{S}_2\text{O}_7$ (48). Solutions of ammonium fluorosulfate show little evidence of hydrolysis and the salt may be recrystallized from hot water. Ammonium fluorosulfate absorbs anhydrous ammonia to form a series of liquid amines that contain 2.5–6 moles of ammonia per mole of salt (77).

Sodium fluorosulfate may be prepared by the action of fluorosulfuric acid on powdered, ignited sodium chloride (13) or of sulfur trioxide on sodium fluoride (48). In general, the alkali metal fluorosulfates may be

Table 4. Physical Properties of Some Fluorosulfates

Salt	CAS Registry Number	Appearance	Mp, °C	Solubility	
				Water ^a	Other solvents ^a
NH ₄ SO ₃ F	[13446-08-7]	long colorless needles	245	s	sl s ethanol; v s methanol
LiSO ₃ F	[13453-75-3]	white powder	360	v s	v s ethanol, ether, acetone, amyl alcohol, ethyl acetate; i ligroin
LiSO ₃ F·3H ₂ O		long shiny needles	60–61		
NaSO ₃ F	[14483-63-7]	shiny leaflets, hygroscopic		s	s ethanol, acetone; i ether
KSO ₃ F	[13455-22-6]	short white prisms	311	6.9 ^b	sl s methanol
RbSO ₃ F	[15587-05-0]	colorless needles	304	s	sl s methanol
CsSO ₃ F	[13530-70-6]	colorless rhombic	292	2.23 ^b	

^ai = insoluble; sl s = slightly soluble; v s = very soluble.

^bIn g/100 mL water.

prepared from the ammonium salt by evaporating a solution containing that salt and an alkali metal hydroxide (77). The solubilities of some Group 1 and 2 fluorosulfates in fluorosulfuric acid have been determined (93).

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