

Fluorinating Agents in Organic Chemistry

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Fluorine, the most electronegative of all the elements, forms very strong covalent (see table 1) or ionic bonds to most other elements. The strength of the carbon-fluorine bond and the small size of the fluorine atom (van der Waals radius: 1.35 Å; hydrogen: 1.20 Å) give rise to a range of valuable chemical, physical and biological properties in organic molecules with one or more fluorine atoms attached to carbon. A rapid growth of interest in fluoro-organics has occurred in many areas of application, including polymers and materials, specialty solvents, performance fluids, medicinal agents, agrochemicals and in numerous reagents and intermediates for chemical synthesis.

Table 1: Typical covalent bond energies¹

BOND	BOND ENERGY	
	kcal mol ⁻¹	kJ mol ⁻¹
F-F	38	159
Cl-Cl	58	242
H-F	136	566
H-Cl	103	431
C-H	98	411
C-F	116	484
C-Cl	81	338
Si-F	139	582
Si-Cl	91	381
P-F	117	490
P-Cl	76	319

Because of the reactivity and hazards of elemental fluorine and hydrogen fluoride, the task of introducing fluorine into organic molecules has presented a particular challenge to synthetic chemists and has led to the development of specialized fluorination technologies and reagents. This article gives a brief outline of fluorination methods, highlighting specific reagents available from Alfa Aesar. Further details and literature references can be found under specific products in the main section of the Catalogue and in numerous texts and reviews on fluorination and organofluorine chemistry.²⁻¹⁴

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Electrophilic fluorination

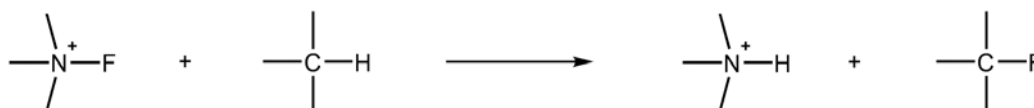
Elemental fluorine is one of the most chemically reactive substances known, due to the relative weakness of the F-F bond and the great strength of its bonds to most other elements, including hydrogen, carbon and silicon, illustrated in Table 1.

Fluorine can behave both as a fluorinating agent and a powerful oxidant. It reacts readily with almost every other element and attacks many common materials, often with near-explosive violence. In organic molecules, C-H bonds tend to be attacked indiscriminately by both free-radical and ionic mechanisms. Elemental fluorine has been successfully harnessed, notably by Chambers' group,¹⁴ using strongly acidic, polar media to promote selective heterolytic fluorination and suppress the non-selective free-radical mode of reaction. Other well-established techniques for moderating the reactivity of fluorine involve the use of inert diluents and very low temperatures. However, many alternative electrophilic fluorination reagents have been introduced with the objective of providing selectivity and ease of handling.

N-Fluoro reagents

A variety of N-fluorinated amines, quaternary salts, amides and sulfonamides have been proposed as reagents for selective electrophilic fluorination under mild conditions (scheme 1). These are usually stable, easily-handled solids, and provide a range of fluorinating power from mild to moderate, depending on the structure of the reagent and the nature of the substrate. Another advantage is that, since free hydrogen fluoride is not a major by-product in fluorination reactions with this type of reagent, conventional glass equipment is often suitable.

Scheme 1



The chemistry of N-F fluorinating agents has been reviewed.¹⁵

L17003 1-Chloromethyl-4-fluoro-1, 4-diazoniabicyclo [2.2.2]octanebis(tetrafluoroborate) [F-TEDA-BF₄]

L13955 N-Fluorobenzenesulfonimide [Accufluor® NFSi]

L17628 1-Fluoro-4-hydroxy-1,4- diazoniabicyclo [2.2.2] octane bis(tetrafluoroborate) [Accufluor® NFTh]

L13231 N-Fluoropyridinium pyridine heptafluoro-diborate [Accufluor® NFPy]

L14324 N-Fluoropyridinium trifluoromethane sulfonate

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Other electrophilic reagents

Xenon fluorides, especially the difluoride, can be used in the selective fluorination of substrates including arenes, alkenes and active methylenes, and also in the fluoro-decarboxylation of carboxylic acids (scheme 2), providing a useful alternative to the Hunsdiecker reaction.¹⁶

Scheme 2



The use of cobalt(III) fluoride [or cobalt(II) fluoride/ fluorine] is a well-established method for the fluorination of hydrocarbons, ethers, etc., but requires high temperatures and specialised equipment. Silver(II) fluoride has found some limited use in the fluorination of aromatics. Several highly reactive species can be generated from elemental fluorine, including trifluoromethyl hypofluorite, acetyl hypofluorite (explosive), and cesium fluoroxy-sulfate (unstable, shock-sensitive), prepared from cesium sulfate (product A16767) and fluorine.

13074 Cobalt(II) fluoride
11490 Cobalt(III) fluoride

11610 Silver(II) fluoride
39739 Xenon difluoride

Electrochemical fluorination

This technique was introduced by J. H. Simons in the 1940's, using anhydrous HF as solvent, with or without an ionic fluoride as supporting electrolyte, and is now an established industrial route to perfluorinated molecules. More recent studies have investigated the selective fluorination of a variety of aliphatic and aromatic substrates, for which acetonitrile has become the preferred solvent, with the addition of a supporting electrolyte such as HF-pyridine or triethylamine trihydrofluoride.¹⁷

L17117 Hydrogen fluoride pyridine complex
A13031 Potassium hydrogen fluoride

L14417 Triethylamine trihydrofluoride
A10211 Tetraethylammonium tetrafluoroborate

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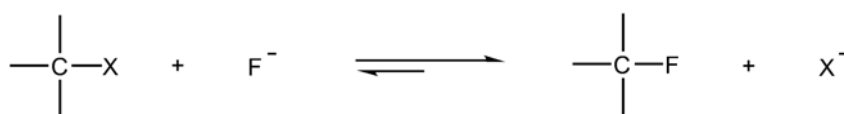
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Nucleophilic fluorination

Inorganic and other ionic fluorides

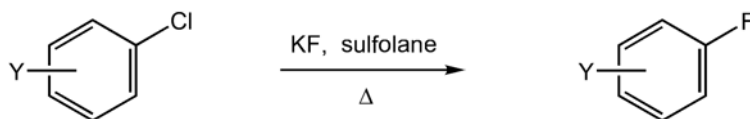
Fluoride ion is normally the least nucleophilic of the halides. Nevertheless, displacement of other halogens in alkyl halides can be effected, since the high stability of alkyl fluorides and the poor leaving group ability of F⁻ can cause the equilibria to be shifted (Scheme 3).

Scheme 3



Dipolar aprotic solvents, such as DMF or acetonitrile, tend to give the best results, and in view of the low solubility of metal fluorides, addition of a crown ether can be beneficial; alternatively, the much more soluble tetraalkylammonium fluorides can be employed. In aromatic systems, displacement of chloride (halogen fluorination) can be achieved in high-boiling polar aprotic solvents including DMSO or sulfolane (Scheme 4).

Scheme 4



The commonest fluoride source is potassium fluoride, though other fluorides are sometimes used, and improved results can often be obtained if the fluoride ion is solubilized by means of a thermally stable phase-transfer catalyst such as tetraphenylphosphonium chloride. The aromatic nucleophilic substitution reactions of fluoride have been reviewed by Vlasov.¹⁸

Inorganic fluorides also have a variety of other uses in synthesis,¹⁹ for example as mild bases in condensation reactions. Inorganic and tetraalkylammonium fluorides are widely used in the selective cleavage of silyl derivative.²⁰

A10176 Calcium fluoride

12885 Cesium fluoride

A11632 Lithium fluoride

14130 Potassium fluoride

11609 Silver(I) fluoride

A13019 Sodium fluoride

A10588 Tetra-n-butylammonium fluoride, 1M soln. in THF

L13303 Tetra-n-butylammonium fluoride trihydrate

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Antimony fluorides

The use of antimony fluorides to displace other halogens has played a major role in the development of the fluorocarbon industry. The reagents can be used either in stoichiometric amounts or catalytically in the presence of HF. The latter method was widely employed in the synthesis of chlorofluorocarbons (CFCs) and hydrochlorofluorocarbons (HCFCs). Further information can be found in relevant sections of general works on fluorination.^{2,21}

A14068 Antimony(III) fluoride

33484 Antimony(V) fluoride

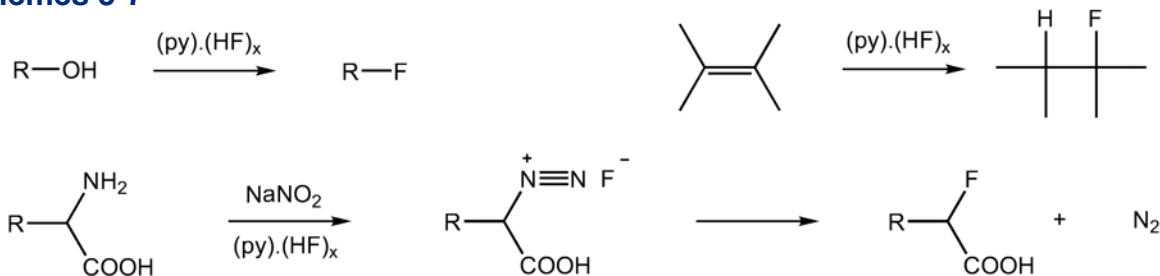
Hydrogen fluoride equivalents

Hydrogen fluoride is a relatively weak acid ($pK_a = 3.45$ at 25°C), providing only low concentrations of fluoride ions in solution. Combined with the comparatively low nucleophilicity of fluoride, this tends to make HF a rather ineffective fluorinating agent, for example in the conversion of C-OH bonds to C-F. Furthermore, anhydrous HF is very volatile (b.p. 20°C), highly toxic, extremely corrosive to skin and other tissues including bone, and readily attacks glass. Many other reagents, formally equivalent to HF are available for the introduction of fluorine in specific molecular environments.

HF-amine complexes

These are either liquids which are less volatile and easier to handle than HF itself, or crystalline solids. They tend to be more nucleophilic than HF, making them valuable reagents for various types of fluorination. (See also use in electrochemical fluorination, above). The most frequently used of these reagents is HF-pyridine (Olah's Reagent²²), applications of which include: preparation of alkyl fluorides from alcohols (schemes 5) or alkenes (scheme 6); acyl fluorides from acyl chlorides or anhydrides; and deaminative fluorination of amino acids and arylamines (Scheme 7).

Schemes 5-7



The use of HF-amine complexes as fluorinating agents has been reviewed by Yoneda.²³

L16789 Hydrogen fluoride 2,4,6-collidine complex

L17117 Hydrogen fluoride pyridine complex

L14417 Triethylamine trihydrofluoride

L17891 Tetra-n-butylammonium dihydrogen-

trifluoride, 50-55% w/w in 1,2-dichloroethane

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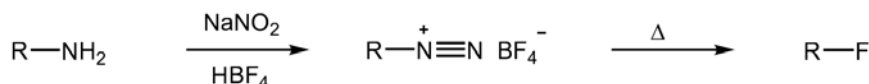
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Fluoroborates and analogues

The classical Balz-Schiemann (or Schiemann) conversion of arylamines to aryl fluorides (Scheme 8) involves the formation and isolation of the diazonium tetrafluoroborate, followed by thermolysis either neat or in an inert solvent.^{24,25}

Scheme 8



Direct formation of aryldiazonium tetrafluoroborates from arylamines and nitrosonium tetrafluoroborate in organic solvents gives good yields of aryl fluorides. Improved results may also be obtained by the use of the diazonium hexafluorophosphate, or a one-pot direct diazotization in anhydrous HF or HF-pyridine (see preceding section).

L14037 Tetrafluoroboric acid

L15728 Hexafluorophosphoric acid

L17117 Hydrogen fluoride pyridine complex

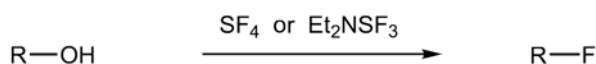
A15806 Nitrosonium tetrafluoroborate

A10826 Silver tetrafluoroborate

Sulfur fluorides

Sulfur tetrafluoride is a powerful fluorinating agent which has the disadvantage of being a highly toxic, corrosive gas. It converts alcohols to alkyl fluorides (Scheme 9) and carboxyl groups to trifluoromethyl (Scheme 10).^{26,27}

Scheme 9



Scheme 10



Diethylaminosulfur trifluoride (DAST), a reagent derived from SF₄, is more convenient to handle and more selective, and is particularly useful for conversion of alcohols to alkyl fluorides, carboxylic acids to acyl fluorides (Scheme 11) and carbonyl compounds to gem-difluorides (Scheme 12). DAST has limited thermal stability at elevated temperatures, and other reagents have been developed, including Morpholinosulfur trifluoride ("Morpho-DAST") with improved thermal characteristics.

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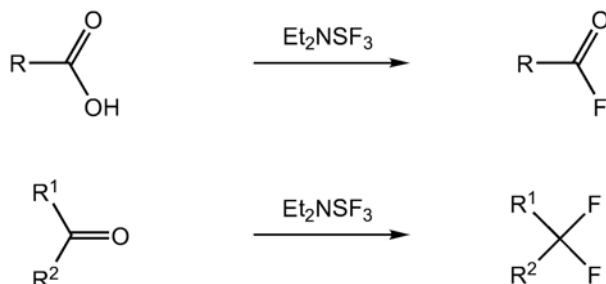
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Schemes 11 and 12



Fluorination with DAST and related reagents has been reviewed by Hudlicky.²⁸

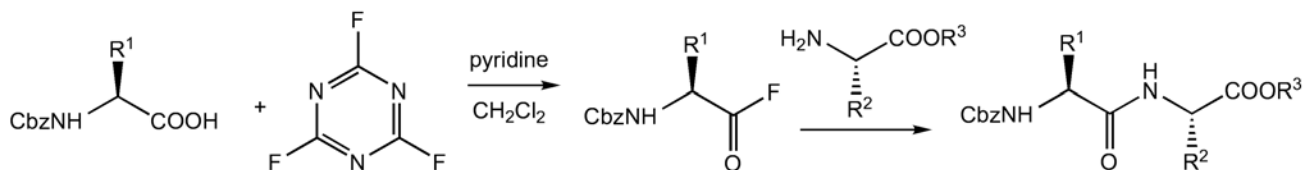
A11992 Diethylaminosulfur trifluoride

L19751 Morpholinosulfur trifluoride

Other HF equivalents

For the conversion of alcohols to alkyl fluorides and carboxylic acids to acyl fluorides, Ishikawa's reagent (N,N-diethyl-1,1,2,3,3,3-hexafluoropropylamine/ N,N-diethyl-1,2,3,3,3-pentafluoropropenamine) has been found effective. Cyanuric fluoride is a particularly valuable reagent, introduced by Olah, which converts carboxylic acids to acyl fluorides under mild conditions.

Scheme 13



Its application to N-protected amino acids and use of the resulting acyl fluorides in peptide synthesis (see Scheme 13 and Appendix 6) has been developed by Carpino.²⁹

A15666 Cyanuric fluoride

L16738 Ishikawa's Reagent

L17330 N,N-Diethyl(2-chloro-1,1,2-trifluoroethyl)-amine (Yarovenko's Reagent)

L20205 Tetrabutylammonium difluorotriphenylstannate (Gingras' Reagent)

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