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RECENT DEVELOPMENTS IN FLUORINATION CHEMISTRY OF DAST WITH SPECIAL REFERENCE TO ALCOHOLS

Meenakshi Jain[1] and Anjali Tiwari[2*]

[1]Deptt. of Chemistry, University of Rajasthan, Jaipur-302 004, India
[2]Deptt. of Chemistry, UDML College of Engineering, Jaipur-302 028, India
**e-mail: at.chem@gmail.com*
Dedicated to late Prof. V. N. Pathak

Abstract

Fluorine containing organic compounds have influenced both medicinal and agrochemical fields. The presence of fluorine or a fluorine containing group causes notable changes in the physical and chemical properties of ordinary organic compounds. The most significant method to introduce fluorine into organic compounds is the nucleophilic replacement of oxygen with fluorine. Diethylaminosulfur trifluoride (DAST), Bis(2-methoxyethyl)aminosulfur trifluoride (BAST) or deoxofluor are the popular fluorinating reagents. By the use of these reagents, organic compounds that contain oxygen in hydroxyl and carbonyl groups are readily converted into their corresponding fluorinated analogues by the introduction of one or two fluorine atoms respectively. Our interest in applying various synthetic methods to incorporate fluorine or a fluorinated group into a large variety of organic compounds encouraged us to summarize the recent chemistry of DAST.

Keywords: Agrochemical; Diethylaminosulfur trifluoride (DAST); Bis(2-methoxyethyl)aminosulfur trifluoride (BAST); Fluorinated analogues

1. INTRODUCTION

Fluorine is the most electronegative element. Therefore, fluorine or a fluorine containing group into organic molecules cause changes in their physical, chemical and biological properties[1] and makes them suitable for diverse applications in agricultural[2] and pharmaceutical fields[3]. The carbon-fluorine bond gives more stability to the organic molecules besides enhancing its lipophilicity[4]. This review highlights the recent progress in fluorination reactions of alcohols using diethylaminosulfur trifluoride (DAST) as key nucleophilic fluorinating reagent.

Several reviews[5-8] in this area in the last few years have been compiled and the most recent of these have been included.

Diethylaminosulfur trifluoride is a useful selective and multipurpose activating reagent for replacing oxygen with fluorine in organic compounds under very mild conditions[9,10]. The ready availability of molecules, low cost and stability makes it very devisable in selective fluorination and cross-coupling reactions. DAST was introduced as a mild and selective reagent

by Middleton *et al.*[11,12] for the conversion of primary, secondary, tertiary alcohols to fluorides; aldehydes and ketones to difluorides; carboxylic acids to acyl fluorides and sulfoxides to α-fluorosulfides. The structure of DAST and its NMR studies have also been reported earlier[13,14].

Keeping all these observations in mind and in continuation to our other work on organo-fluoro compounds[15,16] we have compiled the fluorination reactions of DAST with special reference to alcohols.

2. SYNTHESIS AND PROPERTIES OF DAST

DAST **2** have been prepared by the treatment of sulfur tetrafluoride with diethylaminotrimethylsilane **1** and separated from the volatile trimethylfluorosilane by distillation Fig. **1**.

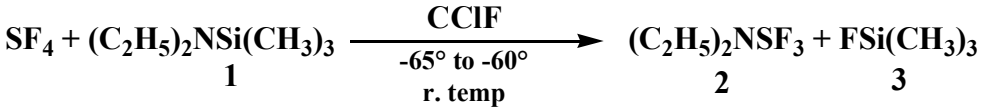


Fig. 1. : Synthesis of DAST

When this reaction is conducted in trichlorofluoromethane (b.p. 25°c) at –70°c, high yield products are obtained with very high purity since the only appreciable by product is fluorotrimethylsilane **3** which is an early separated low boiling (b.p. 17°c) material.

Reaction mechanism of DAST with alcohol is presented in Fig. **2**.

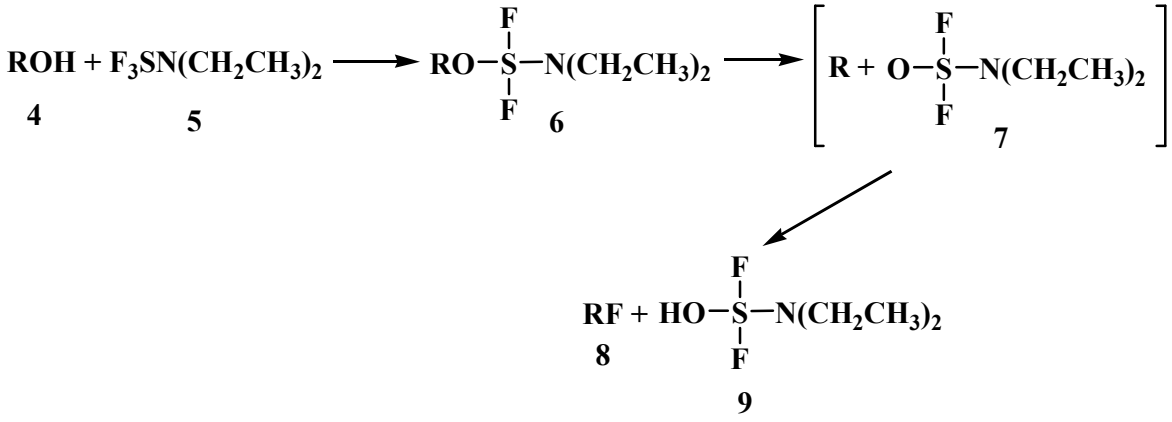


Fig. 2 : Reaction Mechanism of Diethylaminosulfur trifluoride

Several other methods have also been reported for preparation of diethylaminosulfur trifluoride[17,18] Fig. **3**.

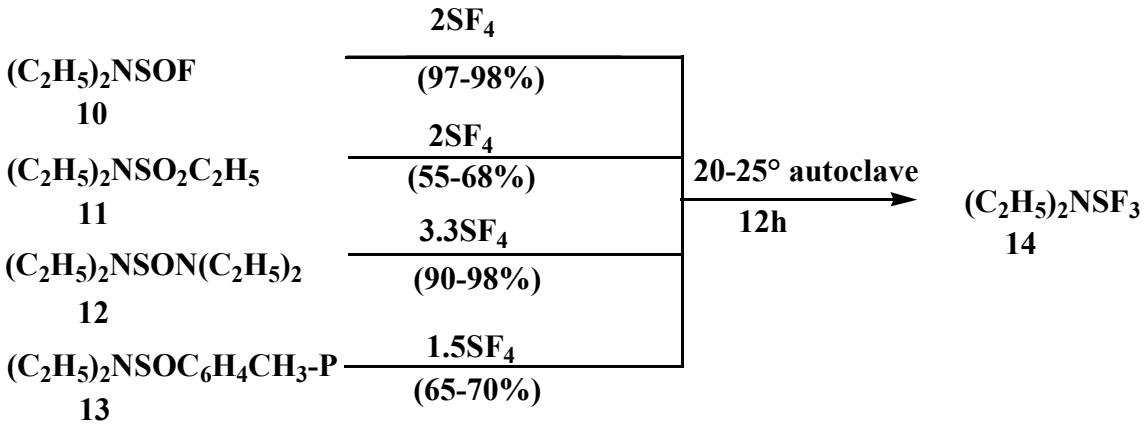


Fig. 3 : Other methods for preparation of DAST

Although several other dialkylaminosulfur trifluorides have been prepared[19] but most of the work has been done with DAST. Some difficulties are associated with DAST as it decomposes at 90°C and can explode if heated at higher temperatures and it is hazardous if not properly handled[20-22]. DAST can be distilled and stored in plastic bottles at room temperature. Most reactions with DAST can be carried out in conventional glass wares at room temperature. DAST requires special handling because it is flammable and reacts violently with water. The boiling point of DAST is 43-44°/12 min and its density is 1.4125.

DAST has found widespread utility in the fluorination of alcohols, aldehydes, ketones, polyfunctional molecules such as cephalosporins, carbohydrates, proteins, steroids, terpenoids, glycosides and peptides.

3. FLUORINATION REACTIONS OF DAST

Reactions of DAST with organic compounds are summarized as follows Fig. **4**.

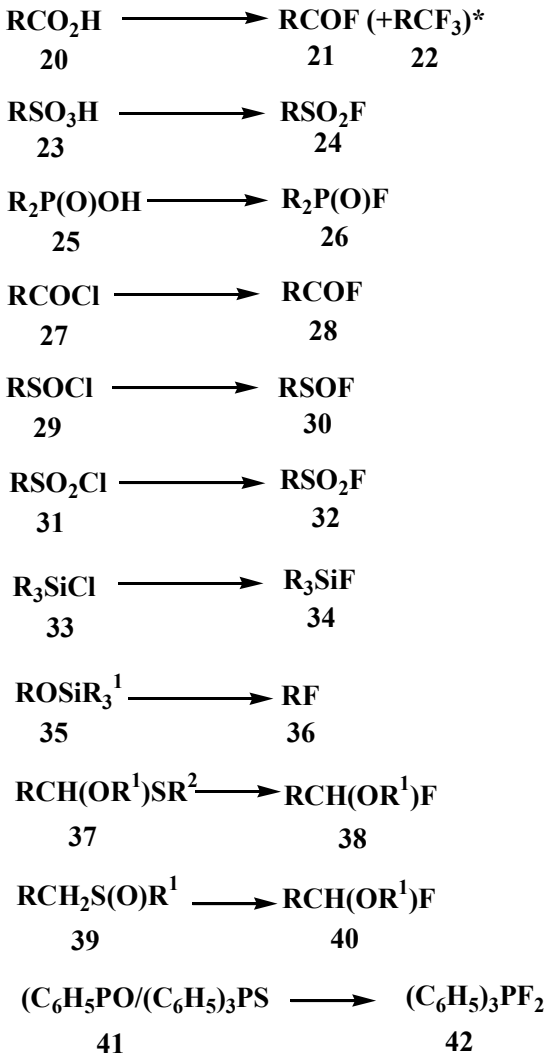


Fig. 4: Reactions of DAST with organic compounds

3.1 FLUORINATION OF ALCOHOLS

DAST **2** was used to convert the hydroxyl group into corresponding monofluorides[23-28] at 25°C but in some cases either higher or lower temperatures were also used. Primary, secondary, tertiary, allylic and benzylic alcohols are converted into corresponding fluorides in high yields. Carbocation rearrangements occur although to a lesser extent than other fluorinating agents.

A rearrangement occurs when non-racemic indolylhydroxylpiperidine-1-carboxylic acid ester **43** and **45** are treated with DAST to afford non-racemic indolylfluoropiperidine-1-carboxylate **44 and 46** with complete regio- and stereoselectivity with 91% ee after debenzoylation[29] Fig. **5**.

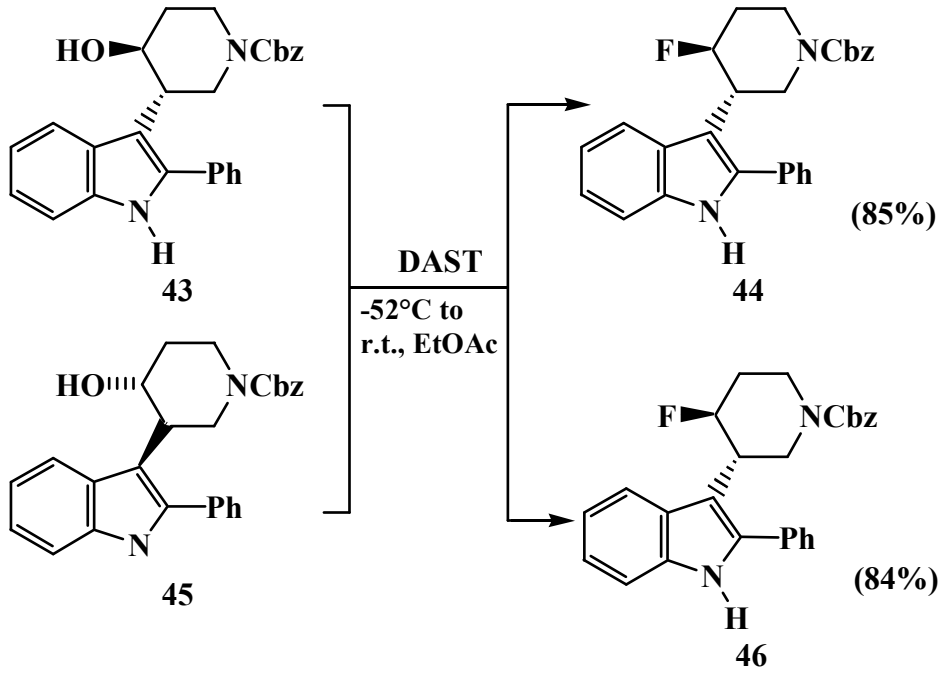


Fig. 5: Fluorination of hydroxyl containing piperidine ring with DAST

The selective fluorination of hydroxyketones into fluoroketones has also been accomplished *viz*. transformation of hydroxyketosteroids into fluoroketosteroids by treatment with DAST at room temperature has also been noticed[30-32].

1-(5-chlorobenzoxazol-2-yl)-1-(4-fluoro-3-trifluoromethylbenzyl)propanol fluorinated with DAST in CH_2Cl_2 at 50° to yield the corresponding 1-(5-chlorobenzoxazol-2-yl)-1-(4-fluoro-3-trifluoromethylbenzyl) propyl fluoride in 88% yield[33].

Treatment of (2S,3S)-methyl-3-benzyloxy-2-hydroxytetradec-6-enoate **47** with DAST afforded (2R,3S)-methyl-3-benzyloxy-2-fluorotetradec-6-enoate **48**[34] Fig. **6**.

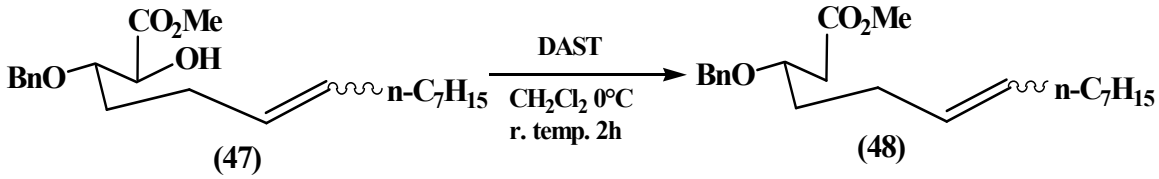


Fig.6:Fluorination of (2S,3S)-Methyl-3-benzyloxy-2-hydroxytetradec-6-enoate

A series of unsaturated ω-fluoroalcohols have been prepared stereoselectively with DAST. These simple compounds are structural analogues of the trail pheromone of termites in the genus reticulitermes[35]. Trans-3-hydroxy-4-(2-oxopyrrolidin-1-yl)benzopyrans have been shown to undergo inversion to cis-3-hydroxy-4-(2-oxopyrrolidin-1-yl)benzopyrans on treatment with DAST[36].

Fluorination of secondary alcohols vicinal to an arene $Cr(CO)_3$ unit with DAST gives the corresponding fluorides[37] with very high exo stereoselectivity.

Fluorination of chiral propargylic alcohols with DAST gives high stereoselective propargylic fluorides Fig. **7**[38,39].

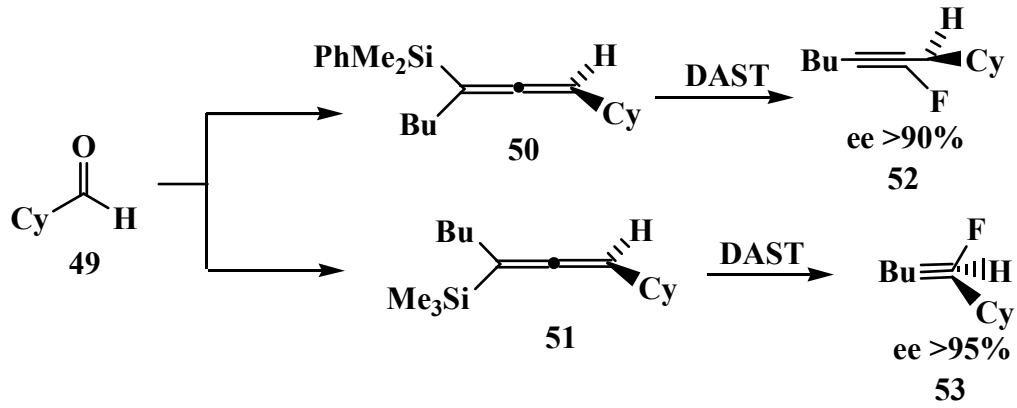


Fig. 7 : Fluorination of chiral propargylic alcohols

Treatment of 1,1-difluoro-1-alken-3-ols with DAST afford (E)-1,1,1-trifluoro-2-alkens with high regio and stereoselectivity[40,41].

Treatment of 1,1-difluoro-1,4-dien-3-ol by DAST yielded (E,E)-8,10-dodecadienol (Codlemone)[42] **54** Fig. **8**.

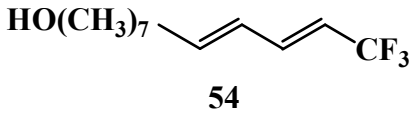


Fig. 8 : Fluorination of 1,1-difluoro-1,4-dien-3-ol

1-Nonylcyclobutanol reacts with DAST to give a 1-fluoro-1-nonylcyclobutane[43]. Blackburn *et al*.[44] have reported the novel synthesis of α- and γ-fluoroalkyl-phosphonates with DAST. The fluorination of DAST with 3-nonyne-2-ol or 1-dodecyne-3-ol, both in racemic and optically active form has been investigated[45]. Treatment of (+)-(S)-2-octanol with DAST affords (-)-R-2-fluorooctane along with octane[46] Fig. **9**.

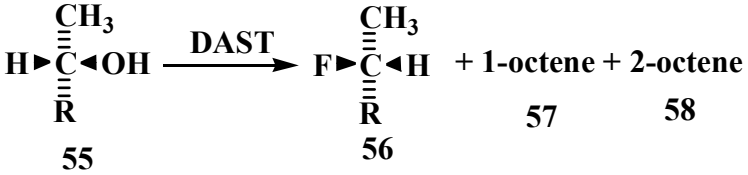


Fig. 9: Fluorination of (+)-(S)-2-octanol

Reaction of steroidal 17-acetylenic alcohols with DAST affords C-17 β-fluoro derivatives[47]. Lakshmipathi *et al*.[48] showed that DAST promotes an unusually easy C-C bond cleavage when epoxy alcohols are used, leading exclusively to monofluoro vinyl ethers Fig. 10.

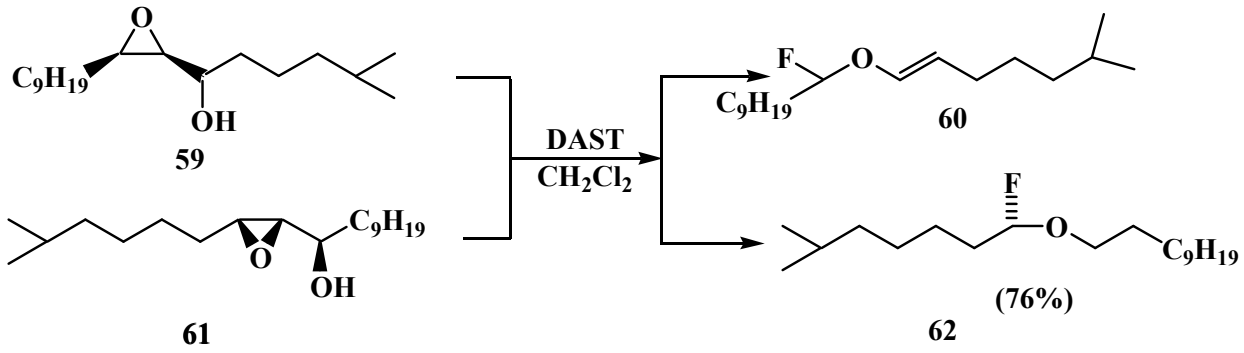


Fig. 10: Fluorination of epoxy alcohols

Induced 1,2-migration occurs via a proposed spiroaziridinium intermediate when 1-phthaloylamino-3-[4-(2-methoxyphenyl)piperazin-1-yl] propanol is treated with DAST to afford N-[2-fluoro-3-(4-(2-methoxyphenyl)piperazin-1-yl)propyl] phthalimide in 13% yield and N-[2-fluoromethyl-2-(4-(2-methoxyphenyl)piperazin-1-yl)ethyl] phthalimide in 73% yield[49].

The (R)-decynol **63** was transformed into the (S)-4-fluoro-1-decyne **64** and the (S)-decynol **65** was converted into the (R)-4-fluoro-1-decyne **66** with DAST[50] Fig. **11**.

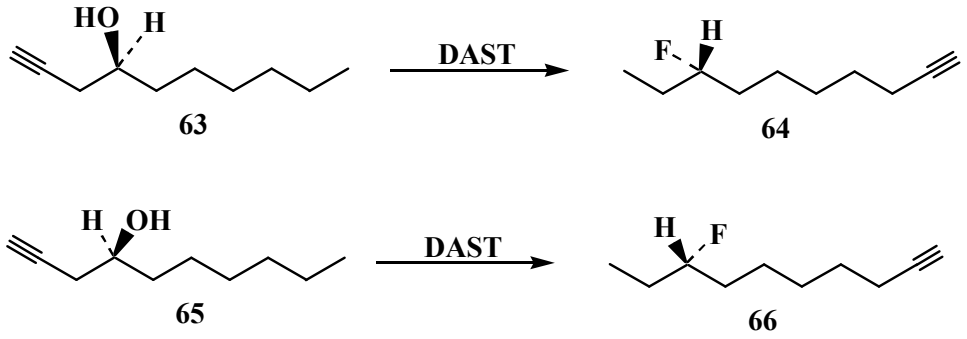


Fig. 11: Fluorination of (R)- and (S)- decynol

DAST reacts with dialcohols to give difluoride sulfite esters or cyclic ethers depending on the number of carbons separating the two alcoholic groups[51].

A series of 1,1-bis(indol-3-yl) and 1-(indol-2-yl)-1-(indol-3-yl)-ω-hydroxyalkanes have been prepared from the corresponding indole derivatives and suitable hydroxyaldehydes via routine coupling reactions with DAST under mild conditions[52].

Bis(spirodienol) derivative **67** reacts with DAST to give fluorinated calixarenes **68, 69** Fig. **12**[53].

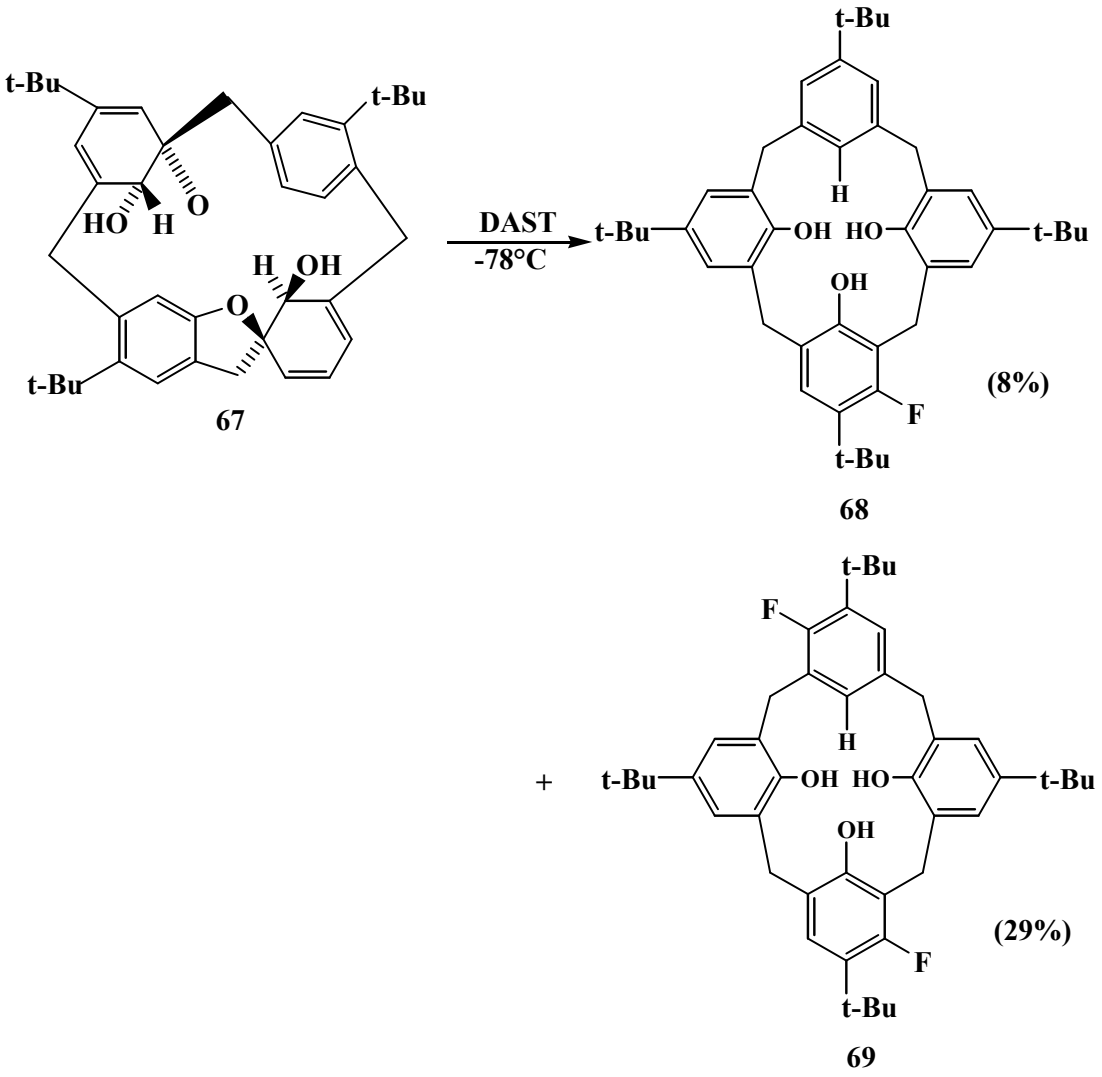


Fig. 12: Fluorination of bis (spirodienol) derivative with DAST

1,1-Bis(trifluoromethyl) substituted olefins are prepared by treatment of 1,1-difluoro-2-trifluoromethyl-1-alken-3-ols with DAST with high regioselectivity[54].

Ionic liquids are reported as recyclable solvents for DAST mediated fluorination of alcohols Fig. **13**[55].

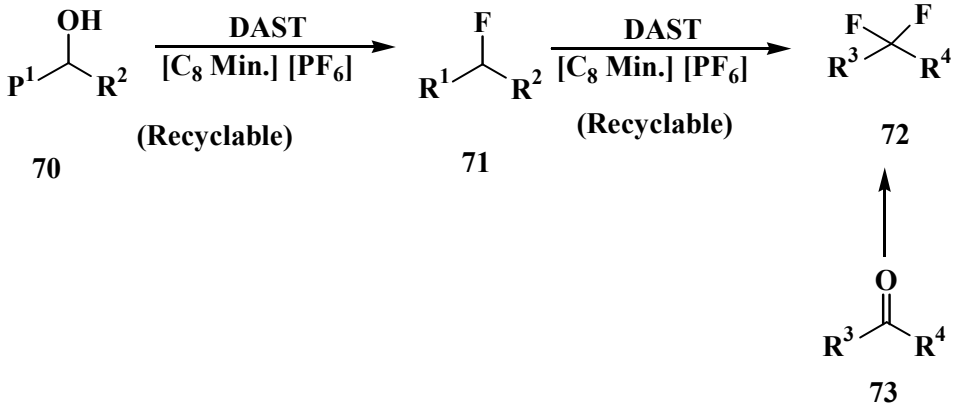


Fig. 13: Fluorination of Ionic liquids

Piyasena *et al.*[56,57] and Dischino *et al.*[58] have prepared 3-fluoro-3-phenyloxindole derivatives **74** on fluorination with DAST in 93% yield which act as modulators of KCNQ potassium channels for treatment of migranes Fig. **14**.

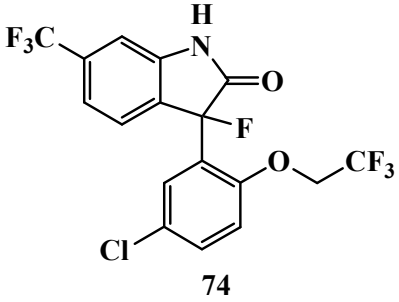


Fig. 14 : Fluorination of tertiary hydroxyl groups in oxindole derivatives

Fluorination of oxazolidine substituent containing alcohols **75a,b** with DAST produced the corresponding monofluoro derivatives **76a,b** with inverted configuration. Cleavage of the oxazolidine ring of the monofluoro derivatives and deprotection of *t*-Boc group with trifluoroacetic acid gave the L-threo-3-fluorosphinganine analogues **77a,b** in good yields Fig. **15**[59].

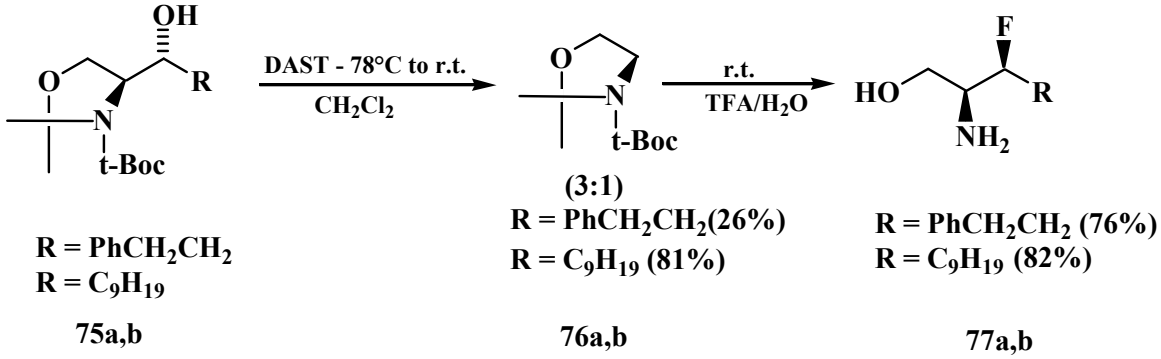
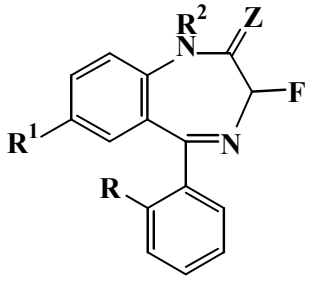


Fig.15: Fluorination of hydroxyl group in substituted oxazolidine rings with DAST

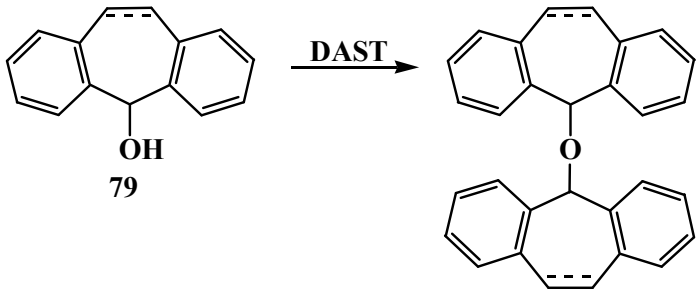
1-Methyl-3-hydroxy-5-phenyl-7-chloro-1,3-dihydro-2H-1,4-benzodiazepin-2-one was treated with DAST in CH_2Cl_2 at ≤50°C to give **78** where R≡H, R^1≡Cl, R^2=Me, Z=0 Fig. **16**[60,61].



78

Fig. 16: Fluorination of 1-methyl-3-hydroxy-5-phenyl-7-chloro-1,3-dihydro-2H-1,4-benzodiazepin-2-one

Treatment of DAST with diarylcarbinols **79** yields bis(diarylmethyl)ethers **80**[62] Fig. **17**.



79 **80**

Fig. 17 : Fluorination of diarylcarbinols

Substituent effects on the regioselectivity in fluorination of allylic alcohols with DAST have also been reported[63]. Reactions of various phosphonate derivatives with DAST were reported[64-66] to give the corresponding monofluoride derivatives which were claimed to be reactive immunization agents towards the hydrolysis of organophosphorus nerve agents Fig. **18**[67].

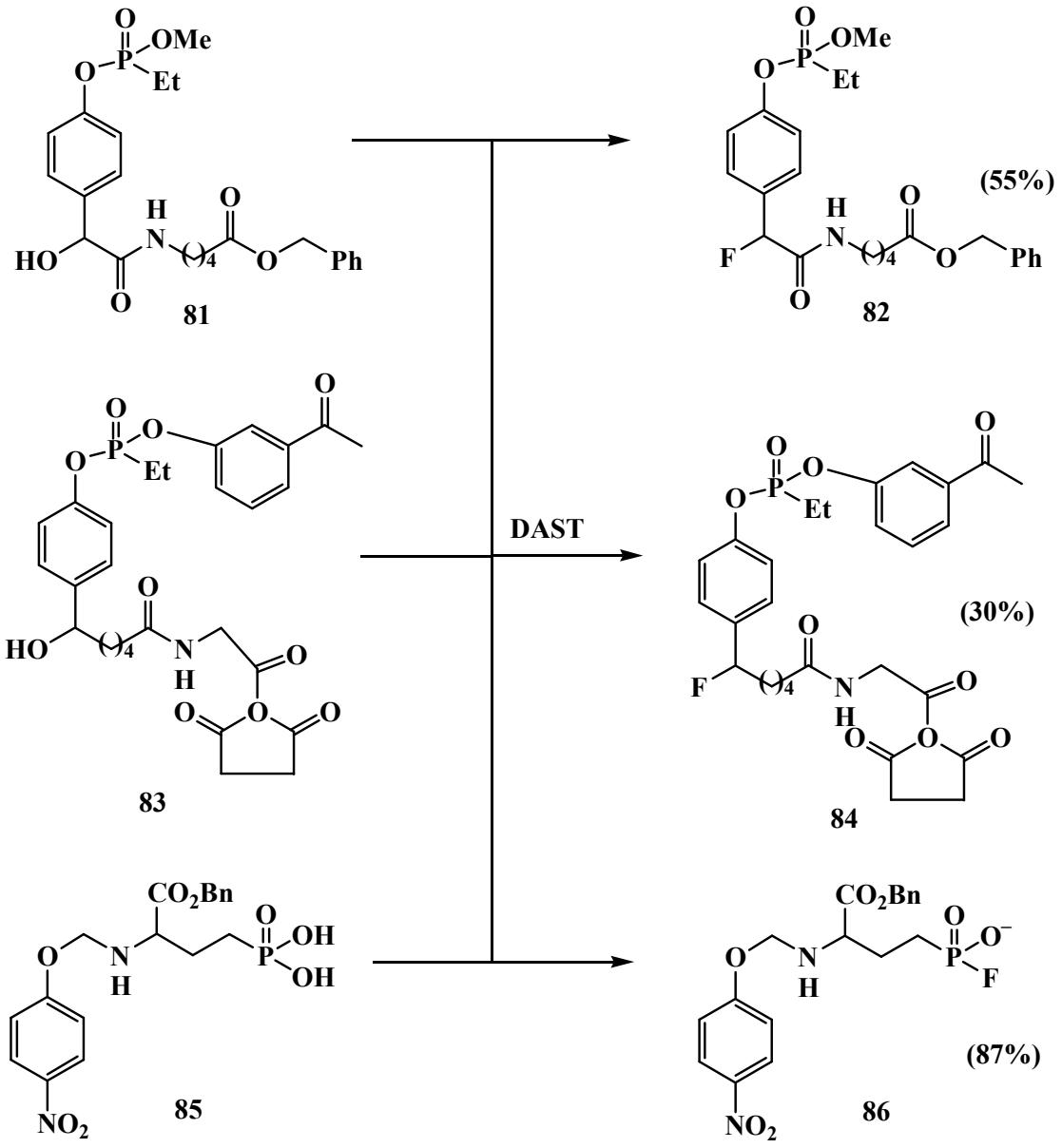


Fig. 18 : Fluorination of hydroxyl groups in various substituted phosphonates with DAST

DAST also reacts with hydroxyl group of benzoylamino benzopyrans[68]. Treatment of α-hydroxyphosphonates with DAST affords α-fluorophosphonates[69]. Fluorination of rhenium complex **87** with DAST yielded 40% allylic fluoride complex[70] **88** Fig. **19**.

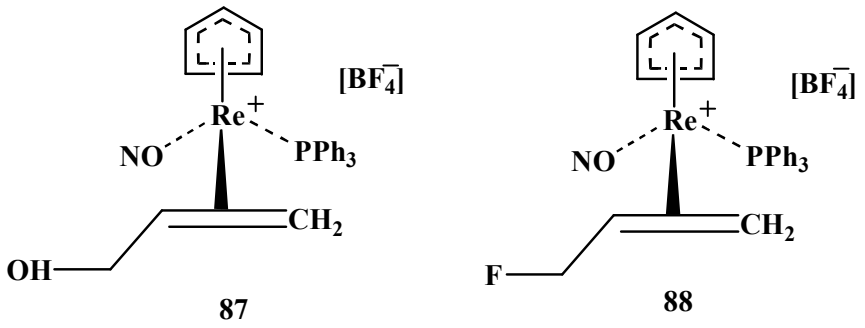


Fig. 19: Fluorination of Rhenium complex

A convergent approach for the synthesis of fluorinated sphingosine analogues with DAST have also been reported[71]. Fluorination of L-glucodiol with DAST followed by treatment with AcOH lead to the D-ido-fluorohydrin[72]. Fluorination of 2-hydroxyalkylazetidines with DAST gives 3-fluoropyrrolidines[73]. Theoretical studies have also been done on fluorination mechanism of 2-hydroxy-3-phenylalkanoate with DAST[74]. DAST-mediated conversions have also been done of a range of alcohols to the corresponding fluorides in microreactors[75] Fig. **20**.

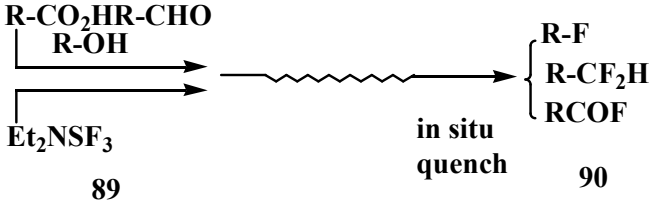


Fig. 20: Fluorination of alcohols

Conversion of α-hydroxyl group into fluorine was achieved by the reactions of α-hydroxybenzylphosphonate with DAST in dichloromethane to give diethyl α-fluorobenzylphosphonate in 53% yield[76].

3.2 GENERAL REACTION MECHANISMS FOR FLUORINATION OF ALCOHOLS USING DAST

DAST involves the nucleophilic displacement of fluorine on sulfur by oxygen of the hydroxyl group through elimination of hydrogen fluoride.

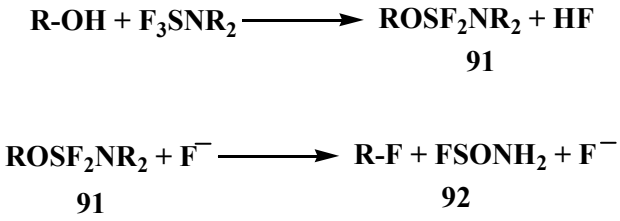


Fig. 21: Reaction mechanism for nucleophilic fluorination reactions of alcohols with DAST

Generally, intermediate **91** is converted into alkyl fluoride **92** by reaction with fluoride

ion Fig. **21**.

β-Diketones exist in keto **93** and enol **94** forms, when fluorination occurs, the enol form **95** react with one molecule of DAST and the hydroxyl group was replaced by fluorine. An α-fluoro derivative **96** was generated during the reaction which also existed in equilibrium with the enol form **97**. Another molecule of DAST fluorinated with **97** to give α,β-difluoro product **99** as a mixture of E and Z isomers in 1:1 ratio[77] Fig. **22**.

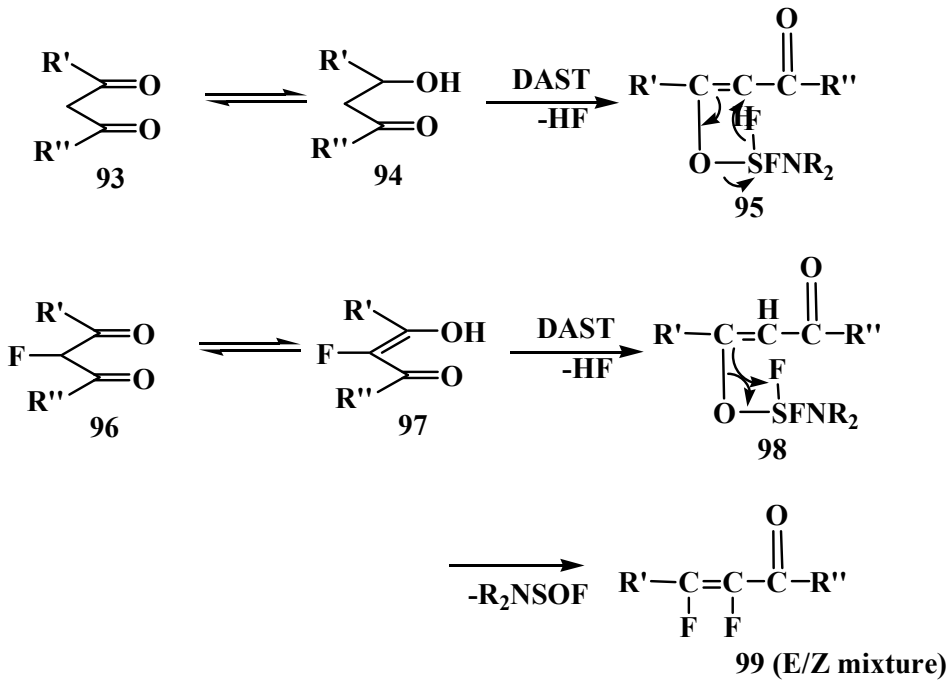


Fig. 22: Reaction mechanism for fluorination of β-diketones with DAST

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