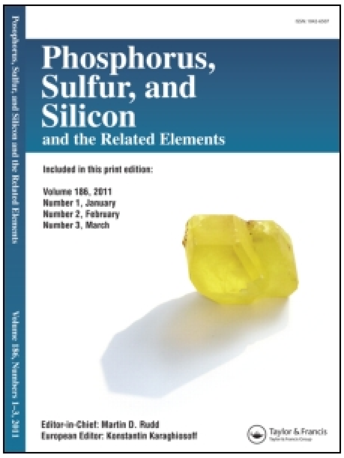

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THE METHYL ESTER OF 3-AZIDOTETRAFLUOROPROPIONIC ACID – A NOVEL REAGENT FOR THE OXIDATIVE FLUORINATION OF GROUP 15 AND 16 ELEMENTS

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The methyl ester of 3-azidotetrafluoropropionic acid, N_3-CF_2CF_2COOMe oxidatively fluorinates P(III), Sb(III) and Te(II) compounds to form the corresponding difluorides in good yield.

Keywords: Methyl 3-azidotetrafluoropropionate; oxidative fluorination; organophosphorus fluorides; organoantimony fluorides; organotellurium fluorides

Fluorinated compounds are of considerable interest in inorganic, organic, polymer, industrial and medicinal chemistry[1–5]. Particularly valuable are mild and selective fluorinating agents and methods for the facile fluorination of organic and organoelement compounds[4–6]. The great majority of fluorinating agents are of nucleophilic character. The reagents which are capable of electrophilic fluorination include F_2 itself [7], XeF_2 [8], $PhIF_2$ [9], acetyl hypofluorite, MeCOOF[10], and N-fluoroamines and amides[11]. Unfortunately, these reagents have various drawbacks and limitations such as hazardous handling (F_2, RCOOF), high cost and lack of selectivity (XeF_2, N-F compounds) or weak fluorinating ability ($PhIF_2$), and finally, they all are usually prepared *via* F_2. Therefore, new electrophilic fluorinat-

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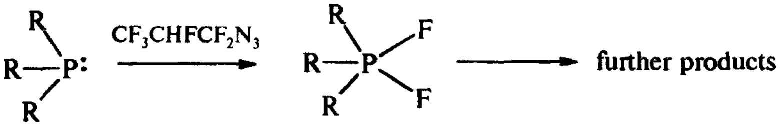
ing agents that allow the facile introduction of fluorine under mild conditions into electron rich substrates are highly desirable and useful.

We have recently shown[12] that the stable 2-hydroperfluoropropyl azide, $CF_3CHFCF_2N_3$, readily fluorinates various organophosphorus(III) compounds, affording difluorophosphoranes in high yield and hence, serving as an oxidative fluorinating agent.



In this paper we describe the reactions of 3-azidotetrafluoropropionic acid methyl ester $MeOCO\text{-}CF_2CF_2\text{-}N_3$, **1**, with some P(III), Sb(III) and Te(II) compounds. This azide (first prepared by C. G. Krespan and his co-workers[13]) has the same $N_3\text{-}CF_2\text{-}$ moiety as $CF_3CHFCF_2N_3$ and we could expect that the fluorinating properties would be similar. Besides, the boiling point of **1** is significantly higher than that of $CF_3CHFCF_2N_3$. This can be convenient in synthetic applications.

REACTIONS OF 1 WITH P(III) COMPOUNDS

We have found that the azide **1** reacts smoothly with trivalent phosphorus compounds, affording the corresponding products of oxidative fluorination. Thus, the trialkyl phosphites **2** and **3** react with the azide **1** at ambient temperature to form the phosphazenes **4** and **5**. These phosphazenes are stable enough at room temperature but decompose readily upon heating in benzene producing the difluorophosphoranes **6** and further reaction products (Eq. 1).

Diethyl fluorophosphate **7** (R = Et) can easily be isolated in a pure state by distillation in 54% yield.

The hexaalkyltriaminophosphines, **9a-b**, also readily react with the azide **1** to form quantitatively stable phosphazides **10a-b** as primary products (no traces of the corresponding phosphazenes could be found in reaction mixtures). The phosphazide **10a** is a crystalline compound which can be isolated in a pure state and analyzed. Its thermal decomposition gives the difluorophosphorane **11a** in a good yield (Eq. 2)

$$\underset{\mathbf{2}}{(CF_3CH_2O)_3P} \xrightarrow{\mathbf{1},\ rt} \underset{\mathbf{4}\quad 90\%}{(CF_3CH_2O)_3P{=}NCF_2CF_2C(O)OMe} \xrightarrow{80\ {}^{\circ}C,\ 2\ h} \underset{\mathbf{6}\quad 70\%}{(CF_3CH_2O)_3PF_2} \qquad (1)$$

$$\underset{\mathbf{3}\ R = Me,\ Et}{(RO)_3P} \xrightarrow{\mathbf{1},\ rt} \underset{\mathbf{5}\ R = Me,\ Et}{(RO)_3P{=}NCF_2CF_2C(O)OMe} \xrightarrow{80\ {}^{\circ}C,\ 2\ h} \underset{\mathbf{7}}{(RO)_2P(O)F} + \underset{\mathbf{6}}{(RO)_3PF_2} + \underset{\mathbf{8}}{(RO)_4{}^{+}PF_6{}^{-}}$$

	7	**6**	**8**
R = Me	65%	10%	25%
R = Et	15%	85% (rt)	
	70%	10%	20% (80 °C/2 h)

$$\underset{\substack{\mathbf{9a\text{-}b}\\ a.\ R = Me\\ b.\ R = Et}}{(R_2N)_3P} \xrightarrow[\substack{C_6H_6\\ 2.\ 80\ {}^{\circ}C,\ 2\ h}]{1.\ \mathbf{1},\ rt} \underset{\substack{100\%\\ \mathbf{10a\text{-}b}}}{(R_2N)_3P{=}N\text{-}N{=}N\text{-}CF_2CF_2C(O)OMe} \qquad (2)$$

$$\mathbf{10a} \xrightarrow[toluene]{110\ {}^{\circ}C,\ 2\ h} \underset{\mathbf{11a}\ 70\%}{(Me_2N)_3PF_2} + \underset{20\%}{XPF_6}$$

X - unknown, phosphorus-containing cation

Diethyl phosphite, **12**, reacts with the azide **1** vigorously in a triethylamine solution to form diethyl fluorophosphate, **7**, in 70% isolated yield (Eq. 3).

$$\underset{\mathbf{12}}{(EtO)_2P(O)H} \xrightarrow[Et_3N,\ 10\ {}^{\circ}C]{\mathbf{1}} \underset{\mathbf{7}\quad 70\%}{(EtO)_2P(O)F} \qquad (3)$$

An interesting Arbuzov-like rearrangement takes place during the reaction of the azide **1** with *iso*-butyl difluorophosphite, **13**. *tert*-Butylphosphonic difluoride, **14**, is formed as the main product (Eq. 4).

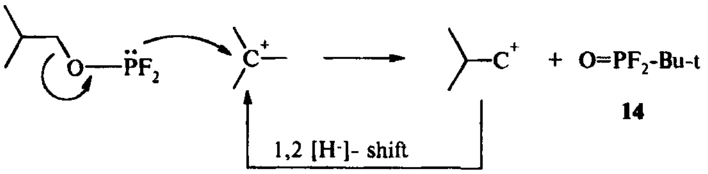
$$\underset{\mathbf{13}}{\text{i-BuOPF}_2} \xrightarrow[\text{50-70 °C, 5-8 h}]{\mathbf{1}} \underset{\mathbf{14}\quad 85-100\%}{\text{t-BuP(O)F}_2} \qquad (4)$$

The reaction proceeds with catalytic amounts of the azide **1** (20% mol.) and is followed by the isomerization of an initial i-Bu substituent into a tertiary one. The mechanism of this transformation is undoubtedly analogous to that described for $CF_3CHFCF_2N_3$ [12] and includes primary oxidative fluorination of **13** to the unstable tetrafluorophosphorane **15** followed by its disproportiontion into the alkylphosphonium salt **16** which is indeed the true catalyst of the rearrangement process (Scheme 1).

$$\underset{\mathbf{13}}{\text{i-BuOPF}_2} \xrightarrow{\mathbf{1}} \underset{\mathbf{15}}{\text{i-BuOPF}_4} \longrightarrow \underset{\mathbf{16}}{\text{t-Bu}^+\text{PF}^-_6} + \text{i-BuOP(O)F}_2$$

SCHEME 1

The disproportionation **15** → **16** + $ROP(O))F_2$ (for R = Me) is described in the literature[14]. The next step is a classical Arbuzov rearrangement accompanied by a catalyst recovery (Scheme 2).



SCHEME 2

On the whole the fluorinating properties of the azide **1** towards P(III) compounds are similar to those described for $CF_3CHFCF_2N_3$ [12] but display at least one peculiarity - the intermediate compounds **4, 5** and **10** are much more stable than in the case of $CF_3CHFCF_2N_3$ and in some cases can even be isolated and analyzed.

FLUORINATION OF Sb(III) AND Te(II) COMPOUNDS

We have found, that the reaction of the azide **1** with Ph_3Sb in toluene leads to a high yield of Ph_3SbF_2, **17** (Eq. 5).

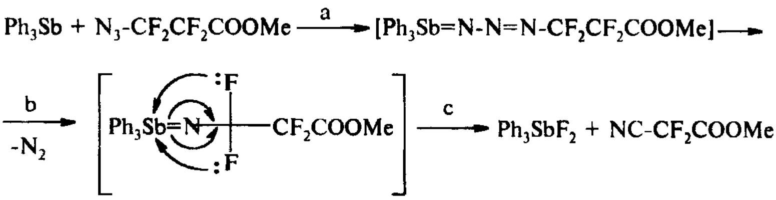
$$\mathrm{Ph_3Sb} + \mathbf{1} \xrightarrow{110\ ^{\circ}\mathrm{C},\ 3\text{-}6\ \mathrm{h}} \underset{\mathbf{17}\ \ 67\text{-}100\%}{\mathrm{Ph_3SbF_2}} \qquad (5)$$

This reaction requires higher temperatures than the analogous reactions with organophosphorus compounds. The difluoride **17** can be isolated in a pure crystalline form.

The reaction of Ph_2Te with the azide **1** proceeds at higher temperatures and also gives the desired product of oxidative fluorination – difluoride Ph_2TeF_2, **18**, in a good yield. In contrast to Ph_3Sb, the reaction appeared to be sensitive to the presence of a dipolar aprotic solvent, namely of hexamethylphosphorotriamide (HMPTA). The addition of small amounts of HMPTA to a reaction mixture increased the yield of **18** significantly (Eq. 6).

$$\mathrm{Ph_2Te} + \mathbf{1} \xrightarrow{160\ ^{\circ}\mathrm{C}} \begin{array}{l}\mathrm{Ph_2TeF_2}\\ \mathbf{18}\\ 72\%\ (9\ \mathrm{h})\\ 20\%\ (3\ \mathrm{h})\\ \mathbf{70\%\ (3\ h\ HMPTA\ added)}\end{array} \qquad (6)$$

The mechanism of the fluorination reaction is undoubtedly similar to that described for $CF_3CHFCF_2N_3$ [12] (Scheme 3), and includes a Staudinger-like reaction – steps (a) and (b), and sigmatropic fluorine transfer with the formation of a stable nitrile and strong element-F bonds (c).



SCHEME 3

Thus, azide **1** can be regarded, to some extent, as a synthon of the following mixture: $MeOCOCF_2CF_2N_3 \Rightarrow [F_2] + N_2 + MeOCOCF_2CN$

EXPERIMENTAL

^{31}P and ^{19}F NMR spectra were recorded on a Bruker CXP-200 spectrometer. Chemical shifts are reported in δ-units downfield from external 85% H_3PO_4 and CF_3COOH, respectively. Melting points are uncorrected. Azide **1** was synthesized according to a literature procedure [13].

Fluorination of Trialkyl phosphites

A. Phosphite **2.** A solution of 0.42 g ($2.09 \cdot 10^{-3}$ mol) of the azide **1** in 2 mL of dry benzene was added dropwise to a stirred solution of 0.595 g ($1.81 \cdot 10^{-3}$ mol) of **2** in 2 mL of dry benzene cooled by cold water, and the resulting mixture was kept for 20 min at rt. The ^{31}P NMR spectrum of the resulting mixture contained the signal of **4** (-6.38, tt, ^3J(P-F) = 18 Hz, ^4J(P-F) = 4.3 Hz; 90%) and 10% of some unidentified impurities. After heating in refluxing benzene during 2 h the ^{31}P NMR of the reaction mixture contained no signal of **4**, but 70% of the difluuorophosphorane $(CF_3CH_2O)_3PF_2$, **6**, (-77.25, t, ^1J(P-F) = 749 Hz; Lit.[15] δP –77.3, t, ^1J(P-F) = 747 Hz).

B. Phosphite **3** (R = Me). The analogous reaction of 0.316 g ($2.55 \cdot 10^{-3}$ mol) of **3** (R = Me) with 0.56 g ($2.79 \cdot 10^{-3}$ mol) of **1** in benzene gave the phosphazene **5** (R = Me) (δP 4.1 (tt, ^3J(P-F) = 15.4 Hz, ^4J(P-F) = 5.3 Hz) and the difluorophosphorane $(MeO)_3PF_2$, **6**, (δP –70, t, ^1J(P-F) = 722 Hz) in 1: 1 ratio. Lit.[16] for **6** (R = Me): ^1J(P-F) = 706 Hz. After heating in refluxing benzene during 2 h the ^{31}P NMR spectrum of the reaction mixture contained the signal of $(MeO)_2P(O)F$, **7** (δP –6.2, d, ^1J(P-F) = 974 Hz; Lit.[17] ^1J(P-F) = 949 Hz), **6**, and $(MeO)_4P^+PF_6^-$ (δP 0, s, –143.3, heptet, ^1J(P-F) = 708 Hz; Lit.[18] (δP 0, s, –147, m, ^1J(P-F) = 700 Hz) in 65: 10: 25 ratio.

C. Phosphite **3** (R = Et). In the analogous reaction of 1.94 g ($1.17 \cdot 10^{-2}$ mol) of $(EtO)_3P$ with 2.8 g ($1.39 \cdot 10^{-3}$ mol) of **1** in benzene the reaction mixture consisted of 85% of the difluorophosphorane $(EtO)_3PF_2$, **6**, (δP –74.1, t, ^1J(P-F) = 725 Hz; Lit.[19] δP –74.78, t, ^1J(P-F) = 722.7 Hz) and

15% of **7** (R = Et) (δP –8.2, d, ^1J(P-F) = 972 Hz; Lit.[17] δP –9.2, d, ^1J(P-F) = 963 Hz). The reaction mixture was refluxed during 2 h and distilled *in vacuo*. 0.98 g (54%) of $(EtO)_2P(O)F$, **7**, were obtained. Bp 60 – 65 °C/15 mm.; Lit.[20] Bp 63 °C/12 mm.

Reaction of 1 with the triaminophosphines 9a and 9b

1.12 g ($5.57 \cdot 10^{-2}$ mol) of **1** in 3 mL of dry benzene were added dropwise to a cold water cooled solution of 0.808 g ($4.96 \cdot 10^{-3}$ mol) of **9a** in 4 mL of dry benzene. The mixture was stirred during 3 h and kept overnight. The ^{31}P NMR spectrum contained only one signal: δP 42.77, S. Heating of the reaction mixture during 2 h at reflux left this signal unchanged. The solvent was evaporated *in vacuo* and the residue was recrystallized from a 1: 3 benzene: heptane mixture. Pure **10a** was obtained as colourless crystals, mp 94 – 95 °C.

^{31}P NMR: δP 42.92, S.

^{19}F NMR: δF –18.7 (t), –42.0 (t) ^3J(F-F) = 6.7 Hz.

^{1}H NMR: δH 2.59 (d, ^3J(H-P) = 9.4 Hz (18H); 3.77, S (3H)).

Anal. Calc. for For $C_{10}H_{21}N_6O_2PF_4$: C, 32.97, H, 5.81, N, 23.07, P 8.50. Found: C, 33.01, H, 5.71, N, 23.05, P, 8.52.

Heating of **10a** in dry toluene during 2 h at reflux afforded a mixture of 70% of **11a** (δP –63.9, t, ^1J(P-F) = 700 Hz; Lit.[21] δP 65.7, t, ^1J(P-F) = 700 Hz; ^{1}J(P-F = 707 Hz[16]), 20% of some PF_6- containing substance (δP 25.6, S; –143.5, heptet, ^1J(P-F) = 710 Hz) and 10% of several minor impurities.

The analogous reaction of 0.54 g ($2.19 \cdot 10^{-3}$ mol) of **9b** and 0.56 g ($2.79 \cdot 10^{-3}$ mol) of **1** in benzene afforded **10b** quantitatively (δP 42.78, s). The substance remained unchanged after 2 h heating in refluxing benzene but our attempts to distill it failed.

Fluorination of diethyl phosphite, 12

A solution of 0.84 g ($4.18 \cdot 10^{-3}$ mol) of **1** in 2 mL of dry Et_3N was added dropwise to a water cooled solution of 0.43 g ($3.1 \cdot 10^{-3}$ mol) of **12** in 2 mL of Et_3N. After the completion of an exothermic reaction the mixture was stirred during 3 h at rt. The ^{31}P NMR spectrum revealed the formation of 70% of $(EtO)_2P(O)F$, **7**, (δP –8.5, d, ^1J(P-F) = 970 Hz) and 30% of several minor compounds.

The reaction of *iso*-butyl difluorophosphite, 13, with 1

0.5 g ($3.52 \cdot 10^{-3}$ mol) of **13** and 0.14 g ($7 \cdot 10^{-4}$ mol) of **1** were mixed in a steel bomb and heated at 50 °C during 5 h. The reaction mixture contained 85% of t-BuP(O)F_2, **14**, (δP 32.1, t of decets, ^1J(P-F) = 1173 Hz; $^3J_{P\text{-}H}$= 19.7 Hz; Lit.[22] δP 31.8, t of decets, ^1J(P-F) = 1195 Hz; ^3J(P-H) = 18.8 Hz), 15% of the starting **13** and traces of i-BuOP(O)F_2 (δP −19.8, tt, ^1J(P-F) = 1004 Hz, ^3J(P-H) = 8 Hz). Heating of the reaction mixture at 70 °C during 8 h afforded practically pure **14.**

Fluorination of Ph_3Sb

A solution of 0.194 g ($5.5 \cdot 10^{-4}$ mol) of Ph_3Sb and 0.35 g ($1.74 \cdot 10^{-3}$ mol) of the azide **1** in 3.5 mL of dry toluene was heated at reflux during 6 h. A known quantity of m-fluorotoluene was added as internal signal for NMR integration. The ^{19}F NMR spectrum revealed the quantitative formation of the difluoride **17.** After removal of the solvent and recrystallization from n-heptane pure **17** was obtained. Mp 112–113 °C, ^{19}F NMR: δF −75.1, S. Lit.[23] δF −153.2 ($CFCl_3$) (s). Mp 120 − 122 °C [24]. Heating of the reaction mixture during 3 h afforded **17** in 67% yield.

Fluorination of Ph_2Te

A. A solution of 0.153 g ($5.43 \cdot 10^{-4}$ mol) of Ph_2Te and 0.35 g ($1.74 \cdot 10^{-4}$ mol) of **1** in 1.5 mL of dry CH_2Cl_2 was heated in a steel bomb at 160 °C during 9 h. ^{19}F NMR revealed the formation of 72% of Ph_2TeF_2, **18** (NMR integration, using a known quantity of m-fluorotoluene as internal standard). After removal of the solvent and recrystallization from heptane the difluoride **18** was obtained in a pure state. Mp 146 - 148 °C, ^{19}F NMR: −50.4, ^1J(Te-F) = 544 Hz; Lit.[25] mp 152 °C.

B. Heating of the previous reaction mixture during 3 h afforded **18** in 20% yield. C.

A solution of 0.157 g ($5.57 \cdot 10^{-4}$ mol) of Ph_2Te, 0.35 g ($1.74 \cdot 10^{-3}$ mol) of **1** and 0.2 mL of dry HMPTA in 1.5 mL of CH_2Cl_2 was heated in a steel bomb at 160 °C during 3 h. The yield of **18** was 70% (NMR integration).

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