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Preparation and properties of $CF_3As[ON(H)CF_3]_2$ and $(CF_3)_2AsON(H)CF_3$

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Abstract

Trifluoronitrosomethane reacts with trifluoromethylarsine and bis(trifluoromethyl)arsine to afford $CF_3As[OBN(H)CF_3]_2$ (**A**) and $(CF_3)_2AsON(H)CF_3$ (**B**), respectively. Compound **B** has been found to decompose at 60 °C to give $(CF_3)_2As(O)H$, $(CF_3)_2AsOH$ and $(CF_3)_2AsF$. The reactions of both **A** and **B** with anhydrous hydrogen chloride result in the cleavage of the As–O bond to afford quantitative yields of the respective arsenic chloride. Reactions of **B** with bis(trifluoromethyl)nitroxyl gives $(CF_3)_2AsON(CF_3)_2$, $(CF_3)_2NOH$, $(CF_3)_2NH$, CF_3NO_2, CF_3NO and a non-volatile white solid.

Keywords: Reactions; Trifluoronitrosomethane; Trifluoromethylarsine; Bis(trifluoromethyl)arsine; Thermal decomposition; IR spectroscopy

1. Introduction

There have been a number of reports on the reactions of trifluoronitrosomethane with hydrides of the Group 15 elements. It has been found to undergo condensation reactions with NH_3 to afford $(CF_3)_2NOH$ [1]. Its reactions with $(CF_3)_2PH$ affords the derivative $CF_3N(OH)P(O)(CF_3)_2$ [2]. With the acid phosphites $RR'P(O)H$ ($R=R'=OEt$; $R=Me$, $R'=OCHMe_2$), the reactions follow a similar course to give insertion and oxidative products $CF_3N(OH)P(O)RR'$ [3]. Recently, trifluoronitrosomethane was found to undergo a condensation reaction with arsine to give $CF_3N{=}AsH$, which is unstable even at low temperatures and decomposes to give CF_3H, N_2 and arsenic metal [4]. We now report further investigations on the reactions of trifluoronitrosomethane with the trifluoromethyl-substituted arsines, namely, CF_3AsH_2 and $(CF_3)_2AsH$, and the properties of the derived products.

2. Results and discussion

When a mixture of trifluoronitrosomethane and trifluoromethylarsine was left to stand at room temperature in an evacuated glass ampoule, the blue colour of trifluoronitrosomethane was slowly discharged over a period of 2 d. Fractionation of the products gave $CF_3As[ON(H)CF_3]_2$ (**A**) in 74% yield, together with a small amount of silicon tetrafluoride.

Reaction of trifluoronitrosomethane with bis(trifluoromethyl)arsine follows a similar course to afford $(CF_3)_2AsON(H)CF_3$ (**B**) in 57% yield. The other products isolated were bis(trifluoromethyl)fluoroarsine and silicon tetrafluoride.

The above reactions of trifluoronitrosomethane with the trifluoromethyl substituted arsines could be represented by the equation:

$$nCF_3NO + (CF_3)_{3-n}AsH_n \longrightarrow (CF_3)_{3-n}As[ON(H)CF_3]_n$$

$(n=1, 2)$

These reactions are different from those of CF_3NO with trifluoromethyl-substituted phosphines [2]. With $(CF_3)_2PH$, the phosphoryl-substituted hydroxylamine $CF_3N(OH)$-$P(O)(CF_3)$ is obtained; and with $(CF_3)_2PX$ ($X=CF_3$, Cl), the CF_3 group has been found to migrate to nitrogen, resulting in the formation of $(CF_3)_2NP(O)XCF_3$. In these reactions, the phosphorus shows a tendency to form the P=O bond.

2.1. Spectroscopic properties

The IR spectra of the arsenic derivatives **A** and **B** are summarised in Table 1. The strong C–F vibrations are located

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Table 1
IR data (cm^{-1}) for $(CF_3)_2AsON(H)CF_3$ (**B**) and $CF_3As[ON(H)CF_3]_2$ (**A**)

$(CF_3)_2AsON(H)CF_3$	$CF_3As[ON(H)CF_3]_2$	Tentative assignment
3331 (w)	3333 (m)	ν(N–H)
1436 (m)	1434 (s)	δ(N–H)
1299 (s)	1299 (vs)	ν(C–F)
1224 (s)	1223 (vs)	
1205 (vs)	1195 (vs)	
1162 (vs)	1142 (vs)	
1122 (vs)		
1030 (m)	1030 (s)	ν(N–O)
913 (m)	913 (s)	ν(C–N)
832 (w)	833 (w)	
733 (w)	705 (m)	δ(C–F)
709 (m)		
634 (w)	629 (w)	
571 (w)	571 (w)	ν(As–O)

in the region 1120 to 1299 cm^{-1}. The N–H stretching and deformation modes are found at 3330 and 1430 cm^{-1}, respectively; they are lower than those reported for $(CF_3)_2NH$ (3460 and 1500 cm^{-1}) [5]. The O–H stretching vibration was absent. The spectra confirm that the insertion reactions result in the formation of the As–O and N–H bonds.

^1H and ^{19}F NMR data for the arsenic derivatives are given in Table 2. The ^{19}F NMR spectrum of **A** shows two signals with a relative intensity of 2:1. The doublet at 4.37 ppm is thus assigned to the CF_3 group attached to nitrogen and the singlet at 11.44 ppm to the CF_3 groups attached to arsenic. Similarly, the spectrum of **B** also consists of two signals. The CF_3–N signal appears as a doublet at 3.99 ppm and the CF_3–As signal as a singlet at 19.46 ppm. There is a considerable downfield shift of about 8 ppm for the CF_3–As signal as compared to that of **A**. The ^1H NMR spectra of **A** and **B** give a quartet each at 6.4–6.5 ppm, whose splitting is due to the CF_3 group attached to nitrogen.

2.2. *Thermal decomposition*

Heating compound **B** at 60 °C overnight gives predominantly a volatile liquid **C** in 75% yield, together with $(CF_3)_2AsF$ (19%), SiF_4 and an intractable heavy liquid. Experimental data suggested the empirical formula of C_2F_6AsOH for **C**. Its EI-MS spectrum gives the parent ion at *m*/*z* 230. Fragmentation ions indicative of the CF_3–As as well as the As–O moieties are also detected. Hydrolysis with 20% NaOH at elevated temperature gives a quantitative yield of fluoroform. Its IR spectrum shows strong C–F stretching vibrations from 1211 to 1131 cm^{-1}, which is consistent with CF_3 groups attached to arsenic. An O–H peak is located at 3614 cm^{-1}, and As–H stretching and deformation modes are found at 2147 and 801 cm^{-1}. In addition, there is also a peak at 820 cm^{-1}, which could be attributed to the presence of an As=O group [6]. A CF_3 deformation mode is found at 733 cm^{-1}. Its ^1H NMR spectrum reveals two proton signals, a singlet at 5.29 ppm and a broad signal at 2.85 ppm, which could be assigned to the As–H and O–H signal respectively. Similarly, its ^{19}F NMR spectrum also consists of two fluorine signals at 17.67 and 16.16 ppm. Therefore liquid **C** most probably consists of an isomeric mixture of $(CF_3)_2As(O)H$ and $(CF_3)_2AsOH$, hitherto unreported.

2.3. *Reactions with anhydrous hydrogen chloride*

The reactions of **A** and **B** with anhydrous hydrogen chloride occurred with complete cleavage of the As–O bond to afford quantitative yields of CF_3AsCl_2 and $(CF_3)_2AsCl$, respectively. The other products from the reactions are SiF_4 and a non-volatile hygroscopic white solid containing fluorine. The reactions can be represented by the equation below:

$$(CF_3)_{3-n}As[ON(H)CF_3]_n + nHCl \longrightarrow (CF_3)_{3-n}AsCl_n + nCF_3N(H)OH$$

$(n = 1, 2)$

N-Trifluoromethylhydroxylamine has not been isolated. It has been reported by Banks and Dickinson [7] to be unstable, undergoing dehydrofluorination to give $CF_2{=}NOH$ which on standing undergoes complete degradation.

2.4. *Reactions with bis(trifluoromethyl)nitroxyl*

When an evacuated glass ampoule containing a 1:2 molar ratio of $(CF_3)_2AsON(H)CF_3$ and bis(trifluoromethyl)-nitroxyl was allowed to warm up slowly to room temperature, the purple colour of the nitroxyl radical was replaced by a light blue colour. Repeated vacuum fractionation afforded a number of products, namely, $(CF_3)_2AsON(CF_3)_2$, $(CF_3)_2NOH$, $(CF_3)_2NH$, CF_3NO_2, CF_3NO and a non-volatile white solid. The IR spectrum of the white solid shows strong C–F stretching vibrations at 1190 to 1109 cm^{-1}, which is indicative of CF_3 groups attached to arsenic. The As=O stretching vibrations are found at 820 and 796 cm^{-1}. Its NMR data in CH_3OD show that it only contains one type of fluorine ($\delta = 13.06$ ppm). From its microanalytical data and the above

Table 2
^1H and ^{19}F NMR data [a] for $(CF_3)_2AsON(H)CF_3$ (**B**) and $CF_3As[ON(H)CF_3]_2$ (**A**)

	^1H NMR δ (ppm)	^{19}F NMR δ (ppm)
$(CF_3)_2AsON(H)CF_3$	6.49 (q, $J_{HF} = 8.8$ Hz)	3.99 (d, 1F, $J_{FH} = 9.8$ Hz); 19.461 (s, 2F)
$CF_3As[ON(H)CF_3]_2$	6.43 (q, $J_{HF} = 9.3$ Hz)	4.37 (d, 2F, $J_{FH} = 9.8$ Hz); 11.44 (s, 1F)

[a] Spectra were obtained in $CDCl_3$.

spectra, the white solid is formulated as $[(CF_3)_2As(O)]_2$.

The formation of $(CF_3)_2AsON(CF_3)_2$, $(CF_3)_2NOH$ and CF_3NO could probably be accounted for by the following radical sequence:

$$(CF_3)_2AsON(H)CF_3 + (CF_3)_2NO \longrightarrow (CF_3)_2AsON(H)CF_3[ON(CF_3)_2]$$

$$(CF_3)_2AsON(H)CF_3[ON(CF_3)_2] \longrightarrow (CF_3)_2AsON(CF_3)_2 + CF_3N(H)O\cdot$$

$$CF_3N(H)O\cdot \longrightarrow CF_3NO + H\cdot$$

$$(CF_3)_2NO + H\cdot \longrightarrow (CF_3)_2NOH$$

while the presence of CF_3NO_2 and $(CF_3)_2NH$ could be explained by the following equations:

$$(CF_3)_2AsON(H)CF_3 + 2(CF_3)_2NO \longrightarrow$$

$$(CF_3)_2AsON(H)CF_3[ON(CF_3)_2]_2 \longrightarrow$$

$$(CF_3)_2AsON(CF_3)_2 + (CF_3)_2NON(O)CF_3(H)$$

$$(CF_3)_2NON(O)CF_3(H) \longrightarrow (CF_3)_2NH + CF_3NO_2$$

The formation of the white solid and $(CF_3)_2NH$ can be rationalised in terms of the oxidation of **B** by $(CF_3)_2NO$, as shown below:

$$(CF_3)_2AsON(H)CF_3 + (CF_3)_2NO \longrightarrow (CF_3)_2As(O)ON(H)CF_3 + (CF_3)_2N\cdot$$

$$(CF_3)_2As(O)ON(H)CF_3 \longrightarrow (CF_3)_2As(O) + CF_3N(H)O\cdot$$

$$2(CF_3)_2As(O) \longrightarrow [(CF_3)_2As(O)]_2$$

$$CF_3N(H)O\cdot \longrightarrow CF_3NO + H\cdot$$

$$(CF_3)_2N\cdot + H\cdot \longrightarrow (CF_3)_2NH$$

3. Experimental details

All volatile substances were manipulated in a Pyrex glass vacuum line with PTFE O-ring taps. Infrared spectra were recorded on a Perkin-Elmer 983G infrared spectrophotometer. NMR spectra were recorded on a JEOL FX 900 Fourier-Transformation NMR spectrometer. ^{1}H NMR was measured w.r.t TMS and ^{19}F NMR w.r.t TFA. Mass spectra (70 eV) were measured on a Micromass spectrometer. Molecular weights of volatile compounds were determined by Regnault's method.

Trifluoronitrosomethane was prepared from the photolysis of trifluoroiodomethane with nitric oxide in the presence of mercury at atmospheric pressure [8]. Bis(trifluoromethyl)arsine and trifluoromethylarsine were obtained from the reactions of arsine with bis(trifluoromethyl)iodoarsine and trifluoromethyldiiodoarsine, respectively [9]. Anhydrous hydrogen chloride was prepared from the action of concentrated sulphuric acid on concentrated hydrochloric acid [10]. Bis(trifluoromethyl)nitroxyl was prepared from the oxidation of bis(trifluoromethyl)hydroxylamine with silver(II) oxide [11].

3.1. *Reaction of trifluoronitrosomethane with trifluoromethylarsine*

Trifluoronitrosomethane (0.136 g, 1.38 mmol) and trifluoromethylarsine (0.201 g, 1.38 mmol) were allowed to react in an evacuated glass ampoule at room temperature. The blue colour was discharged gradually over a period of 2 d and a colourless liquid was formed. Repeated vacuum fractionation gave the following fractions.

(i) Pure $CF_3As[ON(H)CF_3]_2$ (0.176 g, 0.51 mmol) was isolated in a −15 °C bath (passed 0 °C). The yield was 74%, based on the amount of trifluoronitrosomethane used. The IR and NMR spectra for this compound are given in Table 1 and Table 2, respectively.

(ii) A colourless liquid (0.024 g) was trapped at −30 °C (passed −15 °C). The IR spectrum of this fraction showed all the peaks of $CF_3As[ON(H)CF_3]_2$. In addition, peaks were also observed at 3629, 2115 and 831 cm^{-1}.

(iii) A mixture of unreacted CF_3AsH_2 and SiF_4 (combined weight 0.123 g) was isolated at the −196 °C trap (passed −60 °C). The IR peaks of SiF_4 were found at 1031 and 338 cm^{-1}.

(iv) A trace amount of an intractable orange solid remained in the reaction vessel.

3.2. *Reaction of trifluoronitrosomethane with bis(trifluoromethyl)arsine*

Trifluoronitrosomethane (0.210 g, 2.12 mmol) and bis(trifluoromethyl)arsine (0.421 g, 1.97 mmol) were sealed under vacuum in a Pyrex glass ampoule. After standing at room temperature for 2 d, the blue colour of trifluoronitrosomethane was discharged completely and a colourless liquid was formed. The following fractions were obtained on fractionation of the product mixture.

(i) $(CF_3)_2AsON(H)CF_3$ (0.348 g, 1.11 mmol) was trapped at −55 °C (passed −30 °C) as a colourless liquid in 56.5% yield. The compound was identified from its spectroscopic data listed in Table 1 and Table 2, and measurement of its molecular weight gave a value of 313 g mol^{-1} (C_3F_9HAsNO requires 313 g mol^{-1}).

(ii) A white crystalline solid which melted to a colourless liquid was trapped at −65 °C (passed −55 °C). Besides the absorptions of $(CF_3)_2AsON(H)CF_3$, additional peaks were found in its IR spectrum at 3614, 2147 and 821 cm^{-1}. It was thus concluded to be a mixture of $(CF_3)_2AsON(H)CF_3$ and its decomposition products. The yield of this fraction was 0.104 g.

(iii) $(CF_3)_2AsF$ (0.019 g, 0.47 mmol) was isolated as a white solid at −86 °C (passed −65 °C). Its IR spectrum (cm^{-1}) with the following peaks was identical to that of an authentic sample [12]: 1211 (s); 1163 (s); 1133 (s); 1124 (sh); 735 (w); 692 (m).

(iv) A mixture of unreacted bis(trifluoromethyl)arsine and silicon tetrafluoride (combined weight 0.100 g) was isolated at the −196 °C trap.

(v) A trace of an intractable colourless heavy liquid was left in the reaction ampoule.

3.3. Thermal decomposition of $(CF_3)_2AsON(H)CF_3$

$(CF_3)_2AsON(H)CF_3$ (0.397 g, 1.27 mmol) was heated in an evacuated glass ampoule overnight at 60 °C. Vacuum fractionation of the product mixture gave the following.

(i) A colourless liquid trapped at −60 °C (passed −30 °C). It was identified as a mixture of $(CF_3)_2As(O)H$ and $(CF_3)_2AsOH$ (0.220 g, 0.96 mmol, 75% yield) from its IR, NMR and mass spectra. In addition to the parent ion with m/z 230, the following fragmentation ions were also observed in its mass spectrum: 213 $[(C_2F_6)As]^{+\cdot}$; 194 $[(C_2F_5)As]^{+\cdot}$; 191 $[(C_2F_4)AsO]^{+\cdot}$; 175 $[(C_2F_4)As]^{+\cdot}$; 163 $[CF_4As]^{+\cdot}$; 161 $[CF_3AsOH]^{+\cdot}$; 144 $[CF_3As]^{+\cdot}$; 141 $[CF_2AsO]^{+\cdot}$; 125 $[CF_2As]^{+\cdot}$; 113 (base peak) $[AsF_2]^{+\cdot}$; 111 $[AsFOH]^{+\cdot}$; 94 $[AsF]^{+\cdot}$; 92 $[AsOH]^{+\cdot}$; 91 $[AsO]^{+\cdot}$; 75 $[As]^{+\cdot}$; 69 $[CF_3]^{+\cdot}$; 50 $[CF_2]^{+\cdot}$. When 0.120 g (0.52 mmol) of this compound was heated with 20% NaOH overnight at 120 °C, a quantitative amount of CF_3H (0.073 g, 1.04 mmol) was recovered.

(ii) $(CF_3)_2AsF$ (0.07 g, 0.28 mmol), identified from its IR spectrum, was trapped at −96 °C (passed −60 °C).

(iii) SiF_4 (0.040 g, 0.39 mmol), trapped at −196 °C, was identified from its IR spectrum.

(iv) An intractable heavy colourless liquid remained in the ampoule. This liquid was hygroscopic and fumed in moist air. It gave the following IR peaks (cm^{-1}): 3207 (br); 1791 (s); 1710 (s); 1629 (w); 1489 (m); 1253 (br); 1173 (s); 1084 (m); 859 (m); 743 (m); 726 (m); 617 (m); 483 (m).

3.4. Reaction of $CF_3As[ON(H)CF_3]_2$ with anhydrous hydrogen chloride

A gas trap containing 1:2 molar ratio of $CF_3As[ON(H)CF_3]_2$ (0.176 g, 0.51 mmol) and anhydrous HCl (0.040 g, 1.08 mmol) was left for 1 d at room temperature. Fractionation of the resulting mixture gave a quantitative yield of CF_3AsCl_2 (0.111 g, 5.16 mmol), SiF_4 (0.025 g) and an intractable white solid.

The IR spectrum of trifluoromethyldichloroarsine showed the following peaks (cm^{-1}): 1191 (s); 1133 (vs); 733 (w); 410 (m). The identity of the chloroarsine was also confirmed by molecular weight determination (found: 213 g mol^{-1}, $CFAsCl_2$ requires 215 g mol^{-1}) and analysis results (Found: CF_3, 32.1; Cl, 33.07%. CF_3AsCl_2 requires: CF_3, 32.1; Cl, 33.02%). The white solid was non-volatile and hygroscopic. Besides the absorptions for water, its IR spectrum, taken on KBr pellet, showed the following peaks (cm^{-1}): 1793 (s); 1708 (s); 1602 (w); 1478 (s); 1257 (s); 1188 (vs); 1085 (vs); 861 (m); 728 (vs); 606 (m); 533 (m); 482 (m). It was insoluble in all common solvents and was found to contain 64.2% fluorine by the sodium fusion test.

3.5. Reaction of $(CF_3)_2AsON(H)CF_3$ with anhydrous hydrogen chloride

$(CF_3)_2AsON(H)CF_3$ (0.255 g, 0.81 mmol) and anhydrous hydrogen chloride (0.032 g, 0.68 mmol) were allowed to react in a gas trap at room temperature. The reaction was completed over a period of 2 d.

The products obtained were $(CF_3)_2AsCl$ (0.202 g, 0.81 mmol), trapped at −86 °C (passed −40 °C), SiF_4 (0.023 g) and an intractable white solid. The chloroarsine was identified by comparing its IR spectrum with that reported [12]. Bands (cm^{-1}) were located at: 1204 (vs); 1162 (sh); 1135 (vs); 1109 (s); 734 (m); 426 (m). Molecular weight determination (Found: 248.4 g mol^{-1}. C_2F_6AsCl requires: 248.5 g mol^{-1}) and analysis results (Found: CF_3, 55.8; Cl, 14.04%. C_2F_6AsCl requires: CF_3, 55.5; Cl, 14.29%) also confirmed its identity. The white solid had the same properties as that obtained for the solid from the reaction of $CF_3As[ON(H)CF_3]_2$ with anhydrous HCl.

3.6. Reaction of $(CF_3)_2AsON(H)CF_3$ with bis(trifluoromethyl)nitroxyl

$(CF_3)_2AsON(H)CF_3$ (0.348 g, 1.11 mol) and bis(trifluoromethyl)nitroxyl (0.383 g, 2.28 mmol) were sealed in a 1:2 molar ratio in an evacuated glass ampoule and allowed to warm up slowly from −86 °C. On attaining room temperature, the ampoule was found to be charged with a light blue gas. A colourless liquid and an amorphous white solid were also formed at the bottom of the ampoule. The product mixture was fractionated to give the following.

(i) A colourless liquid trapped at −65 °C (passed −40 °C) was identified to be $(CF_3)_2AsON(CF_3)_2$ (0.350 g, 0.92 mmol). Its IR spectrum with peaks (cm^{-1}) located at 1310 (vs); 1261 (vs); 1235 (vs); 1208 (sh); 1203 (vs); 1166 (vs); 1152 (s); 1132 (vs); 1114 (vs); 1032 (s); 971 (ms); 802 (m); 750 (sh); 711 (m); 583 (w) agreed with that reported in the literature [13]. Molecular weight determination gave 380 g mol^{-1} ($C_4F_{12}AsNO$ requires 381 g mol^{-1}).

(ii) A crystalline white solid which melted to a colourless liquid on attaining room temperature was trapped at −86 °C (passed −65 °C) (0.163 g). Besides the absorptions of $(CF_3)_2NOH$, peaks which were attributed to $(CF_3)_2AsON(CF_3)_2$ were also found in the IR spectrum, but in much lower intensities. This fraction was thus a mixture of $(CF_3)_2NOH$ and $(CF_3)_2AsON(CF_3)_2$.

(iii) A colourless liquid (0.111 g) was trapped at −126 °C (passed −86 °C). Its IR spectrum indicated that it was a mixture of bis(trifluoromethyl)amine and trifluoronitromethane [14]. The amine was identified from the N–H vibrations located at 3462 and 1497 cm^{-1} [5].

(iv) Pure trifluoronitromethane (0.037 g, 0.32 mmol) was isolated by allowing the −126 °C bath, which contained the mixture of $(CF_3)_2NH$ and CF_3NO_2, to warm up slightly. The IR bands (cm^{-1}) of CF_3NO_2 were located at 2971 (vw); 2709 (vw); 1625 (s); 1615 (sh); 1360 (sh); 1310 (vs); 1286 (vs); 1270 (sh); 1155 (br); 860 (w); 754 (m).

(v) Trifluoronitrosomethane (0.044 g, 0.45 mmol), identified from its IR spectrum, was obtained in the −196 °C trap as a dark blue liquid.

(vi) A non-volatile white solid was left in the reaction vessel. The solid was formulated as $[(CF_3)_2As(O)]_2$ from its IR, NMR and microanalysis data. Elemental analysis gave 10.19% C ($[(CF_3)_2As(O)]_2$ requires 10.48% C); H and N were absent and chemical analysis showed the presence of As.

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