

Polyfluoroalkyl Derivatives of Nitrogen. Part XL.¹ Reaction of Trifluoronitrosomethane with *NN*-Bistrifluoromethylamino-substituted Allenes²

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The allenes $(\text{CF}_3)_2\text{N}\cdot\text{CH}:\text{C}:\text{CH}_2$ and $(\text{CF}_3)_2\text{N}\cdot\text{CH}:\text{C}:\text{CH}\cdot\text{N}(\text{CF}_3)_2$ react with trifluoronitrosomethane under mild conditions to give the oxazetidines $(\text{CF}_3)_2\text{N}\cdot\text{CH}\cdot\text{O}\cdot\text{N}(\text{CF}_3)\cdot\text{C}:\text{CH}_2$ and $(\text{CF}_3)_2\text{N}\cdot\text{CH}\cdot\text{O}\cdot\text{N}(\text{CF}_3)\cdot\text{C}:\text{CH}\cdot\text{N}(\text{CF}_3)_2$ (mixture of two isomers), respectively, as the major products. The oxazetidines decompose quantitatively on flow pyrolysis at 200–250° to give *NN*-bistrifluoromethylformamide and the iminoethylene $\text{CH}_2:\text{C}:\text{N}\cdot\text{CF}_3$ or $(\text{CF}_3)_2\text{N}\cdot\text{CH}:\text{C}:\text{N}\cdot\text{CF}_3$. Reaction of the allene $(\text{CF}_3)_2\text{N}\cdot\text{CH}:\text{C}:\text{C}[\text{N}(\text{CF}_3)_2]_2$ with trifluoronitrosomethane is more complex and

a mixture of four 1 : 1 adducts is formed which contains the oxazetidine $(\text{CF}_3)_2\text{N}\cdot\text{CH}\cdot\text{O}\cdot\text{N}(\text{CF}_3)\cdot\text{C}:\text{C}[\text{N}(\text{CF}_3)_2]_2$, as shown by flow pyrolysis of the mixture to afford *NN*-bistrifluoromethylformamide and the iminoethylene $[(\text{CF}_3)_2\text{N}]_2\text{C}:\text{C}:\text{N}\cdot\text{CF}_3$ among the products. The allene $[(\text{CF}_3)_2\text{N}]_2\text{C}:\text{C}:\text{CH}_2$ does not react with trifluoronitrosomethane under comparable conditions.

TRIFLUORONITROSOMETHANE reacts with a variety of fluoro-olefins^{3,4} to give either the corresponding oxazetidine or a 1 : 1 copolymer as the major product, depending on the conditions used. The oxazetidines are the predominant products in reactions carried out at 70—

100°, whereas the copolymers predominate in reaction at *ca.* 0°.

The only allene which has been reported⁵ to react

¹ Part XXXIX, D. H. Coy, R. N. Haszeldine, M. J. Newlands, and A. E. Tipping, *J.C.S. Perkin I*, 1973, 1066.

² Preliminary communication, D. H. Coy, R. N. Haszeldine, M. J. Newlands, and A. E. Tipping, *Chem. Comm.*, 1970, 456.

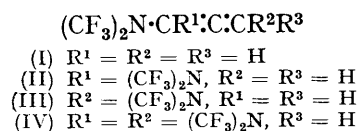
³ D. A. Barr and R. N. Haszeldine, *J. Chem. Soc.*, 1955, 1881; 1956, 3416.

⁴ D. A. Barr, R. N. Haszeldine, and C. J. Willis, *J. Chem. Soc.*, 1961, 1351.

⁵ R. E. Banks, R. N. Haszeldine, and D. R. Taylor, *J. Chem. Soc.*, 1965, 5602.

with trifluoronitrosomethane is tetrafluoroallene. At *ca.* 20° a 1 : 1 copolymer was the major product, but at *ca.* 60° in the vapour phase the cyclic oxazetidine $\text{CF}_3\text{N}\cdot\text{O}\cdot\text{CF}_2\text{C}:\text{CF}_2$ was formed, together with 2 : 1 and 2 : 2 adducts.

In the present work the reaction of trifluoronitrosomethane with bistrifluoromethylamino-substituted allenes (I)—(IV) has been investigated and the products have been subjected to pyrolysis.



Reactions of Allenes with Trifluoronitrosomethane.—The reaction conditions and the products are shown in Table 1. The allenes (I) and (III) reacted to give

TABLE 1
Reaction of allenes with trifluoronitrosomethane

Allene	Conditions	Products
(I)	70°, 7 h	$(\text{CF}_3)_2\text{N}\cdot\text{CH}\cdot\text{O}\cdot\text{N}(\text{CF}_3)_2\text{C}:\text{CH}_2$ (V) (47%) + 1 : 1 copolymer (33%) + breakdown products (10%)
(II)	80°, 48 h	No reaction
(III)	25°, 24 h	<i>cis</i> - and <i>trans</i> - $(\text{CF}_3)_2\text{N}\cdot\text{CH}\cdot\text{O}\cdot\text{N}(\text{CF}_3)_2\text{C}:\text{CH}\cdot\text{N}(\text{CF}_3)_2$ (VI) (89%) in the ratio 32 : 57
(IV)	70°, 24 h	Four 1 : 1 adducts (98%)

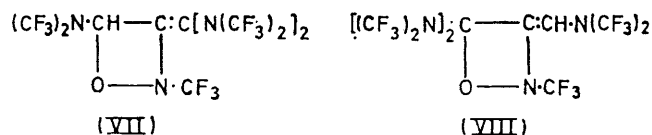
exclusively oxazetidines in which the oxygen atom of the reactant nitroso-compound was bonded to the terminal olefinic carbon atom. A similar orientation of addition has been observed in the reaction of trifluoronitrosomethane with tetrafluoroallene.⁵ With the allene (I) the nature of the product indicates that the $(\text{CF}_3)_2\text{N}\cdot\text{CH}:\text{C}$ double bond is more reactive towards addition than the $\text{CH}_2:\text{C}$ double bond.

The reaction of the allene (I) also gave a 1 : 1 copolymer [i.r. bands at 5.81 and 5.93 μm (C:C str.)] and decomposition products of the oxazetidine later identified as *NN*-bistrifluoromethylformamide and *N*-trifluoromethyliminoethylene.

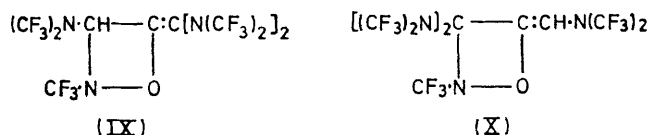
The assignments of the *cis*- and *trans*-configurations (where *cis* and *trans* refer to the configuration of the vinyl proton relative to the ring $\text{N}\cdot\text{CF}_3$ group) to the oxazetidines (VIa and b) are based on n.m.r. spectral evidence (see later).

The allene (IV) reacted with trifluoronitrosomethane to give a mixture (98%) of four compounds in the ratio 7 : 29 : 34 : 30. Elemental analysis of the mixture indicated that all four products were 1 : 1 adducts. If the orientation of addition were as observed with the allenes (I) and (III) then the oxazetidines (VII) and (VIII) would be expected to be formed; the latter could exist in *cis*- and *trans*-configurations comparable to (VIa and b). This would explain the formation of three

oxazetidine isomers; to explain the presence of a fourth isomer either a second mode of addition of trifluoronitrosomethane, to give the oxazetidine (IX) or (X),



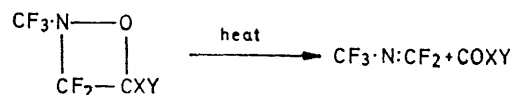
must be invoked, or the two possible ring isomers of the oxazetidine (VII) are present and nitrogen inversion is not taking place (the CF_3 group can be *cis* or *trans* to the



ring proton). As later shown by pyrolysis, isomer (VII) is present in the product mixture to the extent of at least 32%.

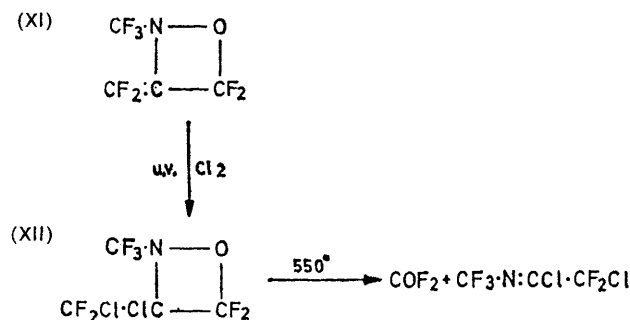
Surprisingly, the allene (II) did not react with trifluoronitrosomethane even on heating at 80° for 48 h. The reason for this is not apparent since the nature of the products obtained from reaction of the allene (IV) implies that reaction can take place with a $[(\text{CF}_3)_2\text{N}]_2\text{C}:\text{C}$ double bond.

Pyrolysis of the Oxazetidines.—On flow pyrolysis at *ca.* 550° the oxazetidines obtained by reaction of trifluoronitrosomethane with fluoro-olefins undergo ring cleavage in which the N-O and C-C bonds are broken^{3,4} (e.g. Scheme 1). The trifluoronitrosomethane-tetra-



SCHEME 1

fluoroallene adduct⁵ (XI) was not pyrolysed directly, but was first chlorinated; the chlorinated oxazetidine (XII) was then pyrolysed to give the expected products (Scheme 2).



SCHEME 2

The oxazetidines prepared in the present work were subjected to flow pyrolysis through a silica tube at *ca.* 1.5 mmHg; the temperatures used and the products

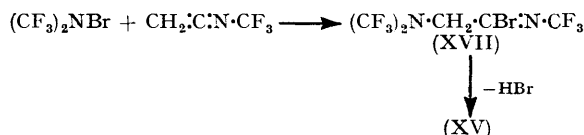
are shown in Table 2. At far lower temperatures than those reported for the successful pyrolysis of oxazetidines formed from fluoro-olefins and trifluoronitrosomethane,

TABLE 2
Pyrolysis of the oxazetidines

Compound	Reaction temp. (°C)	% Reacted	Products
(V)	200	64	(CF ₃) ₂ N·CHO (XIII) (100%) + CH ₂ :C:N·CF ₃ (XIV) (100%)
(VI)	250	87	(XIII) (98%) + (CF ₃) ₂ N·CH·C:N·CF ₃ (XV) (98%)
	550	100	Complex mixture containing CO, C ₂ F ₆ , and (CF ₃) ₂ NH
Mixture of allene (IV) adducts	300	96	(XIII) (32%) + [(CF ₃) ₂ N] ₂ C·C:N·CF ₃ (XVI) (31%) + complex mixture

the oxazetidines (V) and (VI) gave almost quantitative yields of *NN*-bistrifluoromethylformamide (XIII) and the corresponding *N*-trifluoromethyliminoethylene (XIV) or (XV). These products provide unequivocal proof as to the structures of the oxazetidines.

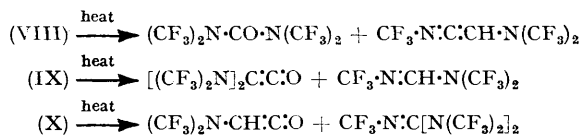
The iminoethylene (XV) could not be separated from (XIII) either by distillation or by g.l.c. on a variety of columns. An attempt was made therefore to synthesise it by the illustrated route (Scheme 3). A mixture of



SCHEME 3

the iminoethylene (XIV) and excess of the *N*-bromamine reacted in the vapour phase in light to give the adduct 3-bromo-1,1,1,6,6,6-hexafluoro-5-trifluoromethyl-2,5-diazahex-2-ene (XVII) in 96% yield. However, attempts to dehydrobrominate (XVII) over powdered potassium hydroxide *in vacuo* resulted in complete decomposition of the adduct.

Pyrolysis of the mixture of oxazetidines prepared from the allene (IV) to give the formamide (XIII) and the iminoethylene (XVI) shows that the oxazetidine mixture contained isomer (VII) to the extent of at least 32%. The pyrolysis products which would be expected from the other isomeric oxazetidines (VIII), (IX), or (X), possibly present in the mixture, are as shown (Scheme 4),

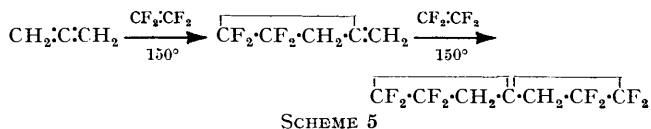


SCHEME 4

but no firm evidence was obtained for the presence of the carbonyl compounds; after several minutes *in vacuo* at room temperature the pyrolysate partially solidified, suggesting further reaction of the initial pyrolysis products.

Attempted Reactions of Allenes with Tetrafluoro-

ethylene.—It has been reported⁶ that tetrafluoroethylene reacts with allene to give either a 1 : 1 or a 2 : 1 adduct depending on the molar ratio of reactants used (Scheme 5). Attempted reactions of the Allenes (I) and



(III) with excess of tetrafluoroethylene at 140 and 100°, respectively, were unsuccessful in that with the allene (I) only unchanged reactants were obtained and with the allene (III) dimerisation (60%) occurred.

EXPERIMENTAL

The Allenes were prepared as reported previously.¹ Experimental techniques were as described in the preceding Part.¹

Reaction of Trifluoronitrosomethane with Allenes.—(a) *NN*-Bistrifluoromethylpropadienylamine (I). The allene (2.01 g, 10.52 mmol) and trifluoronitrosomethane (1.04 g, 10.52 mmol), sealed in a Pyrex ampoule (*ca.* 300 ml) and heated at 70° (7 h), gave (i) a mixture (0.36 g, 2.14 mmol; *M*, 168) of *NN*-bistrifluoromethylformamide and *N*-trifluoromethyliminoethylene; (ii) 4-bistrifluoromethylamino-3-methylene-2-trifluoromethyl-1,2-oxazetidine (V) (1.43 g, 4.96 mmol, 47%) (Found: C, 25.0; H, 1.2; N, 9.4%; *M*, 289. C₆H₃F₉N₂O requires C, 24.8; H, 1.0; N, 9.6%; *M*, 290), b.p. (Siwoloboff) 107 °C at 732 mmHg; λ_{max}, 5.83m μm (C:C str.); *m/e* 290 (14%, *M*⁺), 191 [22%, (CF₃)₂N·C₃H₂⁺], 153 [15%, (CF₃)₂NH⁺], 121 (18%, CF₃·N·C₃H₂⁺), and 69 (100%, CF₃⁺); τ 3.32 (1H, s, CH) and 5.00 (2H, s, :CH₂); ¹⁹F δ -21.6 [6F, s, (CF₃)₂N] and -1.5 p.p.m. (3F, s, N·CF₃); and (iii) a non-volatile, viscous 1 : 1 copolymer (1.01 g) (Found: C, 24.4; H, 1.1; N, 8.9%).

(b) 1,3-Di(bistrifluoromethylamino)propadiene (III). The allene (2.44 g, 7.13 mmol) and trifluoronitrosomethane (0.71 g, 7.13 mmol), sealed in a Pyrex ampoule (*ca.* 300 ml) and shaken at room temperature (24 h), gave (i) a mixture (2.80 g, 6.35 mmol, 89%) which was separated by g.l.c. (8 m Kel-F oil at 150°) into its two components, identified as *cis*-4-bistrifluoromethylamino-3-bistrifluoromethylamino-methylene-2-trifluoromethyl-1,2-oxazetidine (VI) (1.01 g, 2.29 mmol, 32%) (Found: C, 21.8; H, 0.7; N, 9.6. C₆H₃F₁₅N₂O requires C, 21.8; H, 0.5; N, 9.5%), b.p. 118 °C at 745 mmHg (Siwoloboff); λ_{max}, 5.63w and 5.71w μm (C:C str.); *m/e* 441 (12%, *M*⁺), 342 [7%, (*M* - CF₃·NO)⁺], 273 (17%, C₆H₃F₉N₂⁺), 260 (34%, C₅HF₉N₂⁺), 191 (21%, C₄HF₆N⁺), 96 (13%, CF₃·N·CH⁺), and 69 (100%, CF₃⁺), τ 3.34 (1H, s, >CH·O), and 4.33 (1H, s, -CH); ¹⁹F δ -21.2 [6F, s, (CF₃)₂N·CH⁺], -17.5 [6F, s, (CF₃)₂N·CH·O], and -3.7 p.p.m. (3F, s, N·CF₃); its *trans*-isomer (1.79 g, 4.06 mmol, 57%) (Found: C, 21.9; H, 0.8; N, 9.5%), b.p. 135 °C at 745 mmHg (Siwoloboff); λ_{max}, 5.73w μm (C:C str.); *m/e* 441 (10%, *M*⁺), 342 [8%, (*M* - CF₃·NO)⁺], 273 (17%, C₆H₃F₉N₂⁺), 260 (32%, C₅HF₉N₂⁺), 191 (22%, C₄HF₆N⁺), 96 (15%, CF₃·N·CH⁺), and 69 (100%, CF₃⁺); τ 3.27 (1H, s, >CH·O) and 4.19 (1H, s, -CH); ¹⁹F δ -20.7 [6F, s, (CF₃)₂N·CH⁺], -17.6 [6F, s, (CF₃)₂N·CH·O], and -1.9 p.p.m. (3F, s, N·CF₃); and (ii) a viscous, non-volatile residue (0.32 g).

(c) 1,1-Di(bistrifluoromethylamino)propadiene (II). The

⁶ D. D. Coffman, P. L. Barrick, R. D. Cramer, and R. S. Raasch, *J. Amer. Chem. Soc.*, 1949, **71**, 490.

allene (1.48 g, 4.32 mmol) and trifluoronitrosomethane (0.43 g, 4.32 mmol), sealed in a Pyrex ampoule (*ca.* 300 ml) and heated at 85° (48 h), gave only unchanged reactants [allene (99%) and trifluoronitrosomethane (100%)].

(d) 1,1,3-Tri(bistrifluoromethylamino)propadiene (IV). The allene (2.20 g, 4.46 mmol) and trifluoronitrosomethane (0.44 g, 4.46 mmol), sealed in a Pyrex ampoule (*ca.* 300 ml) and heated at 70° (24 h), gave a 1 : 1 adduct mixture (2.60 g, 4.40 mmol, 98%) (Found: C, 20.2; H, 0.3; N, 9.4. Calc. for $C_{10}HF_{21}N_4O$: C, 20.3; H, 0.2; N, 9.5%) which was shown by g.l.c. (4 m Kel-F oil at 100°) to consist of four adducts in the ratio 7 : 29 : 34 : 30.

Pyrolysis of the Oxazetidines.—(a) 4-Bistrifluoromethylamino-3-methylene-2-trifluoromethyl-1,2-oxazetidine (V). The oxazetidine (1.47 g, 5.06 mmol) was passed slowly (45 min) *in vacuo* at 1–2 mmHg through a silica tube (80 cm, 1.5 cm i.d., heated length 40 cm) at 200° and the products were collected in traps cooled to –196°. This afforded (i) unchanged oxazetidine (0.54 g, 1.86 mmol, 36% recovered), (ii) NN-bistrifluoromethylformamide (0.58 g, 3.20 mmol, 100%) (Found: C, 20.2; H, 0.7; N, 7.5%; *M*, 179. C_3HF_6NO requires C, 19.9; H, 0.5; N, 7.7%; *M*, 181, b.p. 51 °C (Siwoloboff); λ_{max} 5.56 and 5.67s μm (C:O str.); *m/e* 181 (19%, M^+), 153 [100%, $(CF_3)_2NH^+$], 134 (30%, $C_2F_5NH^+$), 114 (29%, $C_2F_4N^+$), 85 (18%, $C_3F_5NO^+$), and 69 (99%, CF_3^+); τ 1.32 (qmult, *J* 1.6 Hz); ^{19}F δ –19.55br (s, *cis*- CF_3) and –19.5 p.p.m. (d, *trans*- CF_3); and (iii) N-trifluoromethyliminoethylene (0.35 g, 3.22 mmol, 100%) (Found: C, 33.3; H, 2.0; N, 12.9%; *M*, 109. $C_3H_2F_3N$ requires C, 33.0; H, 1.8; N, 12.8%; *M*, 109, b.p. 6.7 °C (isoteniscope); λ_{max} 4.80m and 4.85s μm (C:C:N str.); *m/e* 109 (39%, M^+), 90 [18%, $(M - F)^+$], 69 (100%, CF_3^+), and 40 (13%, CH_2CN^+); τ 6.09 (q, *J* 2.2 Hz); ^{19}F δ –17.0br p.p.m. (s).

A sample of the iminoethylene polymerised to an orange viscous material during 2 weeks *in vacuo*.

(b) *cis*- and *trans*-4-Bistrifluoromethylamino-3-bistrifluoromethylaminomethylene-2-trifluoromethyl-1,2-oxazetidine (VI). A mixture (2.26 g, 10.25 mmol) of the oxazetidine isomers, pyrolysed as in (a) at 250° (45 min), gave unchanged oxazetidine (0.32 g, 0.72 mmol, 12% recovered) and a mixture (2.21 g, 10.04 mmol, 98%) (Found: *M*, 219. Calc. for a 1 : 1 mixture of C_3HF_6NO and $C_5HF_9N_2$: *M*, 220.5) of NN-bistrifluoromethylformamide (0.91 g, 5.02 mmol, 98%) and 1-bistrifluoromethylamino-2-trifluoromethyliminoethylene (1.30 g, 5.02 mmol, 98%), λ_{max} 4.90s (C:C:N str.) and 10.22vs μm [C–N str. in $(CF_3)_2N$]; *m/e* 260 (M^+), 241 [$M - F^+$], 191 [$(M - CF_3)^+$], and 175 ($C_4HF_5N_2^+$); τ 4.29 (sept, *J* 1.2 Hz); ^{19}F δ –17.3 (3F, s, N- CF_3) and –16.8 p.p.m. [6F, d, $(CF_3)_2N$].

The mixture could not be separated by g.l.c. on a variety of columns, or by distillation.

In a second experiment pyrolysis of the oxazetidine isomers at 550° gave a complex mixture of breakdown products which included hexafluoroethane, NN-bistrifluoromethylamine, and carbon monoxide.

(c) The mixture of 1,1,3-tri(bistrifluoromethylamino)propadiene-trifluoronitrosomethane 1 : 1 adducts. The mixture of adducts (2.22 g, 3.74 mmol), pyrolysed as in (a) at 300° (30 min), gave (i) a complex mixture (1.44 g, 2.43 mmol, 65%) of products containing a small amount of unchanged starting material, which on warming to room temperature reacted further and deposited crystalline material in the fractionation trap, (ii) NN-bistrifluoromethylformamide

(0.23 g, 1.30 mmol, 31%) (Found: *M*, 183. Calc. for C_3HF_6NO : *M*, 181), and (iii) 1,1-di(bistrifluoromethylamino)-2-trifluoromethyliminoethylene (0.54 g, 1.31 mmol, 32%) (Found: *M*, 409. Calc. for $C_7F_{15}N_3$: *M*, 411), λ_{max} 4.85m and 4.90s (C:C:N str.), 10.00m and 10.15s (C–N str.), and 13.61s μm (CF_3 def.); ^{19}F δ –20.1 (3F, s, N- CF_3) and –19.7 [12F, s, 2 × $(CF_3)_2N$].

The iminoethylene rapidly polymerised to a pale yellow oil *in vacuo* at room temperature.

Attempted Preparation of 1,1-Bistrifluoromethylamino-2-trifluoromethyliminoethylene.—N-Trifluoromethyliminoethylene (0.50 g, 4.58 mmol) and N-bromobistrifluoromethylamine (1.39 g, 6.00 mmol), rapidly vapourised into a Pyrex bulb (5 l) *in vacuo* and exposed to daylight (2 h), gave unchanged N-bromoamine (0.35 g, 1.50 mmol, 25% recovered) and 3-bromo-1,1,1,6,6,6-hexafluoro-5-trifluoromethyl-2,5-diazahept-2-ene (1.50 g, 4.40 mmol, 96%) (Found: C, 17.8; H, 0.7; N, 8.2%; *M*, 338. $C_5H_2BrF_9N_2$ requires C, 17.6; H, 0.6; N, 8.2%; *M*, 341, b.p. 111 °C (Siwoloboff); λ_{max} 5.71m, 5.81vs, and 5.93s (C:N str.) and 9.90vs μm (C–N str.); *m/e* 323 and 321 [trace, $(M - F)^+$], 261 [21%, $(M - Br)^+$], 166 [69%, $(CF_3)_2N^+CH_2^+$], 78 (48%, $CF_2N^+CH_2^+$), and 69 (100%, CF_3^+); τ 5.76 (sept, *J* 1.1 Hz); ^{19}F δ –19.7 [6F, t, $(CF_3)_2N$] and –16.4 p.p.m. (3F, s, N- CF_3).

Treatment of the 1 : 1 adduct with an excess of dry, powdered potassium hydroxide *in vacuo* did not give any volatile product.

N.m.r. Spectra.—The spectra were recorded with trifluoroacetic acid as external reference for ^{19}F (negative values to low field) and tetramethylsilane as internal reference for 1H .

The two trifluoromethyl groups in the amide $(CF_3)_2N\cdot CHO$ are non-equivalent and show separate ^{19}F absorptions. This can be attributed to partial double bond character in the nitrogen-carbonyl carbon bond. A *trans*- CF_3, H coupling (1.6 Hz) is observed and there is possibly a *cis*- CF_3, H coupling (<1 Hz) which is hidden by the broadness of the bands. A similar n.m.r. spectrum has been reported for NN-dimethylformamide.⁷ The *cis*- and *trans*-isomers of the oxazetidine (VI) were assigned on the basis that it was expected that a proton *trans* to the $CF_3\cdot N$ group would absorb at lower field than a proton *cis* to this group (*cf.* H *cis* and *trans* to a bromine atom).

The spectrum of the iminoethylene $CF_3\cdot N:C:CH_2$ shows an unusually large CF_3, CH_2 coupling constant (2.2 Hz) through five bonds. In contrast the ketenimine $(CF_3)_2N\cdot CH:C:N\cdot CF_3$ did not exhibit CF_3, CH coupling, but it did show a geminal $(CF_3)_2N, H$ coupling (1.2 Hz).

Mass Spectra.—The base peaks in the spectra of all the compounds, except that of bistrifluoromethylformamide, were at *m/e* 69 (CF_3^+). The amide spectrum contained a strong peak at *m/e* 69 (99%), but, surprisingly, the base peak was at *m/e* 153, assigned to the rearrangement ion $(CF_3)_2NH^+$ formed by loss of CO from the parent. Reasonably strong parent peaks were observed in the spectra of all the compounds except that of the imine $(CF_3)_2N\cdot CH_2\cdot CBr\cdot N\cdot CF_3$ which did, however, show a strong peak at *m/e* 166 [$(CF_3)_2N\cdot CH_2^+$] as expected. In all cases the spectra were in agreement with the proposed structures.

[3/143 Received, 22nd January, 1973]

⁷ J. R. Dyer, 'Applications of Absorption Spectroscopy of Organic Compounds,' Prentice-Hall, New Jersey, 1965, p. 114.