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ORGANOPHOSPHORUS CHEMISTRY. PART 21 [1]. INSERTION OF OLEFINS INTO P - CF_3 BONDS

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SUMMARY

Tris(trifluoromethyl)phosphine and ethylene reacted efficiently under u.v. irradiation to give 3,3,3-trifluoropropylbis(**trifluoromethyl**) **phosphine** in good yield. With vinyl fluoride, vinylidene fluoride, and propene the reaction was regioselective rather than regiospecific, and the yield of 1:1 adduct was low. In these reactions, and in those with vinyl chloride, but-1-ene, and hexafluoropropene, in which only traces of 1:1-adduct could be detected, the bulk of the olefin and of the phosphine was recovered, and numerous by-products consistent with radical intermediates were identified. With propyne, 1,1,1-trifluoro-3-bis(trifluoromethyl)phosphino-cis-but-2-ene was obtained in moderate yield, but no reaction occurred between the phosphine and either but-2-yne or hexafluorobut-2-yne. Tris(trifluoromethyl)phosphine oxide did not form an adduct with ethylene, tetrafluoroethylene, or propyne.

Bis(trifluoromethyl)phosphine and dimethylphosphine both reacted readily under u.v. irradiation with 3,3,3-trifluoropropene, the phosphinyl radical attacking the terminal carbon in each case.

INTRODUCTION

In an earlier paper in this series [2], the insertion of ethylene into the P - CF_3 bond of ethylbis(trifluoromethyl)phosphine was reported.

$$(CF_3)_2PC_2H_5 + C_2H_4 \xrightarrow{u.v.} \underset{79\%}{CF_3(C_2H_5)PCH_2CH_2CF_3}$$

We now report our further investigations of the scope of this unusual photochemical reaction.

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RESULTS AND DISCUSSION

Ultraviolet irradiation of an equimolar mixture of ethylene and bis(trifluoromethyl)phosphine, with the liquid products shielded from further irradiation, gave 3,3,3-trifluoropropylbis(trifluoromethyl)phosphine (1) in excellent yield, together with recovered tris(trifluoromethyl)-phosphine, tetrakis(trifluoromethyl)diphosphine, and an unidentified high-boiling mixture.

$$(CF_3)_3P + C_2H_4 \xrightarrow{u.v.} \underset{(1)\ 73\%}{(CF_3)_2PCH_2CH_2CF_3} + \underset{1\%}{(CF_3)_2PP(CF_3)_2}$$

The major product (1), which was fully characterised, was identical to that formed when bis(trifluoromethyl)phosphine reacted photochemically with 3,3,3-trifluoropropene (see below).

$$(CF_3)_2PH + CF_3CH{=}CH_2 \xrightarrow{u.v.} \underset{(1)\ 90\%}{(CF_3)_2PCH_2CH_2CF_3}$$

The same product was also formed, but much more slowly, when ethylene and tris(trifluoromethyl)phosphine were kept together in vacuo at 200 °C in the dark. Only 8% of (1) was obtained after 200 h, the bulk of the starting materials being recovered unchanged, compared with 73% in a shorter time under u.v. irradiation.

Irradiation of tris(trifluoromethyl)phosphine with other olefins also gave 1:1 adducts in some cases, but in much lower yields, and in several instances mixtures of by-products also resulted, as shown in the Table. These by-products, and the large difference in yield between the photochemical and thermal reactions with ethylene strongly suggest the involvement of radical intermediates in the reactions.

Vinyl fluoride, vinylidene fluoride, and propene clearly react non-regiospecifically with tris(trifluoromethyl)phosphine. Since $\dot{C}F_3$ and $(CF_3)_2\dot{P}$ are both known to react regiospecifically with these olefins [2,3,4], the mixture of 1:1 adducts can best be accounted for by two radical paths. The other products can be accounted for by side-reactions of the intermediate radicals, e.g. for propene, Scheme 1 (p. 320).

Table

Photochemical Reaction of $(CF_3)_3P$ with Olefins[a]

Olefin	$(CF_3)_3P$	1:1 Adduct(s)[b]	Other Volatile Products[b]
C_2H_4 (<3)	(14)	$(CF_3)_2PCH_2CH_2CF_3$ (86)	$(CF_3)_2PP(CF_3)_2$ (1)
$CH_2 = CHF$ (<2)	(30)	$(CF_3)_2PCHFCH_2CF_3$ (19) $(CF_3)_2PCH_2CHFCF_3$ (4)	$CF_3CH_2CH_2F$ (45)
$CH_2 = CF_2$ (*ca.* 30)	(*ca.* 30)	$(CF_3)_2PCF_2CH_2CF_3$ (6) $(CF_3)_2PCH_2CF_2CF_3$ (?)(2)	$(CF_3)_2PCH_2X$[c], $(CF_3)_2PX$[c]
$CH_2 = CHCl$ (74)	(83)	$(CF_3)_2PCH_2CHClCF_3$ &/or $(CF_3)_2PCHClCH_2CF_3$(tr)[d]	$(CF_3)_2PCH_2CHCl$ or $(CF_3)_2PCHClCH_3$ (12) $(CF_3)_2PCH = CHCl$ or $(CF_3)_2PCCl = CH_2$ (8) $CF_3CH_2CH_2Cl$ or $CF_3CHClCH_3$ (16) $(CF_3)_2PCH = CHCF_3$ (tr), $(CF_3)_2PCH = CH_2$ (tr)
$CH_3CH = CH_2$ (65)	(73)	$(CF_3)_2PCH(CH_3)CH_2CF_3$ (16) $(CF_3)_2PCH_2CH(CF_3)CH_3$ (tr)	$(CF_3)_2PCH_2CH_2CH_3$ (27) $(CF_3)_2PCH = CHCH_3$ (21)
$EtCH = CH_2$ (76)	(86)	$(CF_3)_2PCHEtCH_2CF_3$[e] and/or $(CF_3)_2PCH_2CHEtCF_3$[e]	$(CF_3)_2PCH = CHEt$[e] and/or $(CF_3)_2PCEt = CH_2$[e] $CF_3CH = CHEt$ (16), $(CF_3)_2PBu^n$[e] $(CF_3)_2PCH = CHEt$[e] or $(CF_3)_2PCEt = CH_2$[e] (7)
$CF_3CF = CF_2$[f] (53)	(60)	$(CF_3)_2PCF_2CF(CF_3)_2$ and/or $(CF_3)_2PCF(CF_3)C_2F_5$ (tr)	Not identified (*ca* 70% by weight of consumed reactants)

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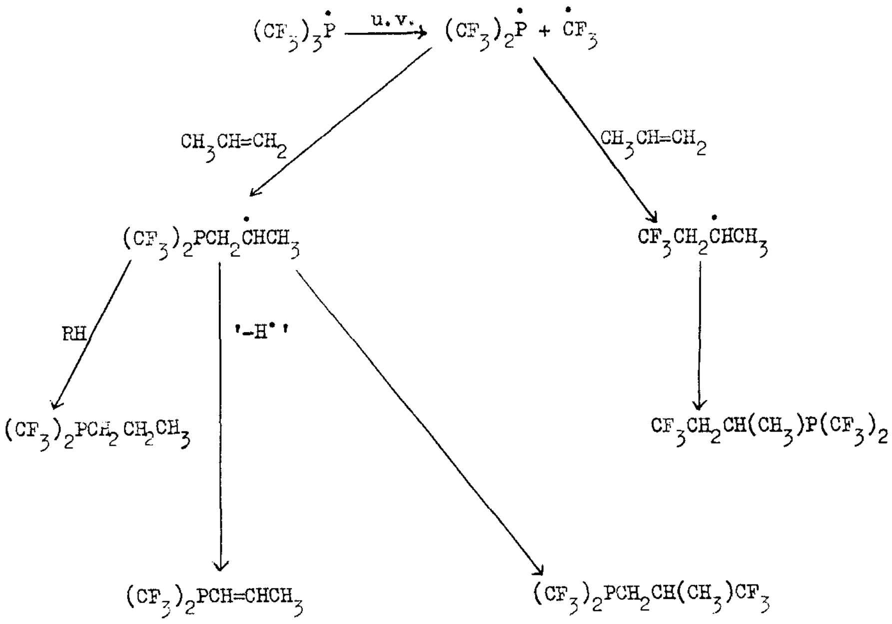
[a] % recovery or % yield in brackets. [b] Based on phosphine consumed. [c] Detected by ^{19}F n.m.r., X not determined. [d] tr=trace [e] Unresolved on g.c., total yield *ca.* 70% on consumed $(CF_3)_3P$.
[f] 300 h reaction time.

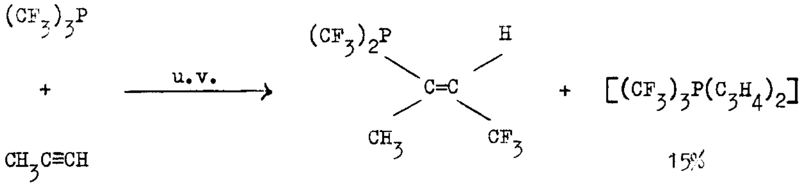


Scheme 1

Although attack by $\dot{C}F_3$ radicals appears to be a major reaction path, attempts to initiate the reaction between tris(trifluoromethyl)phosphine and propene by the incorporation into the reaction mixture of trifluoroiodomethane, which was expected to provide an alternative source of $\dot{C}F_3$, were unsuccessful; the phosphine and olefin were recovered (100% and 91% respectively). Azomethane proved similarly unsuccessful.

The photochemical reaction with vinyl chloride gave only a trace of the expected 1:1 adducts, the major products, identified by g.c./m.s., presumably being derived from the intermediate radicals in reactions similar to those shown above for the reaction with propene. Although the analysis of this reaction mixture did not distinguish between pairs of isomers (e.g. $(CF_3)_2PCH{=}CHCl$ or $(CF_3)_2PCCl{=}CH_2$; $CF_3CH_2CH_2Cl$ or $CF_3CHClCH_3$) radical attack, whether by $(CF_3)_2\dot{P}$ or by $\dot{C}F_3$ would be expected to take place at the $CH_2{=}$ group, so that products with the chlorine attached to the terminal carbon atom are the more likely. With hexafluoropropene, only a trace of the expected 1:1 adducts could be detected in a very complicated product mixture.

When a mixture of tris(trifluoromethyl)phosphine was irradiated with propyne, the 1:1 adduct (2) was obtained in 24% yield (based on consumed phosphine), and a less volatile product (15%) was identified by its mass spectrum as a 2:1 adduct of propyne to the phosphine. Whether this latter was formed by insertion into two P-CF_3 bonds, or by telomerisation could not be determined.



(2) 24%

But-2-yne and hexafluorobut-2-yne, however, failed to react with tris(trifluoromethyl)phosphine, both the phosphine and the alkynes being recovered essentially quantitatively.

U.v irradiation of tris(trifluoromethyl)phosphine oxide, most conveniently prepared by the reaction of dichlorotris(trifluoromethyl) phosphorane with oxalic acid [5], gave tris(trifluoromethyl)phosphine in 16% yield, together with a mixture of carbonyl fluoride, hexafluoroethane, and phosphorus trifluoride. Presumably an α-fluorine migrates from carbon to phosphorus to liberate difluorocarbene, as occurs in the pyrolysis of difluorotris(trifluoromethyl)phosphorane [6]; abstraction of oxygen from the starting material and from phosphorus oxyfluoride by the difluorocarbene would then give the carbonyl fluoride, phosphorus trifluoride and tris(trifluoromethyl)phosphine observed [cf.6] .

$$(CF_3)_3P{=}O \xrightarrow{-\ddot{C}F_2} (CF_3)_2\,P(O)F \xrightarrow{-\ddot{C}F_2} CF_3P(O)F_2 \xrightarrow{-\ddot{C}F_2} F_3PO$$

$$F_3PO + \ddot{C}F_2 \longrightarrow F_3P + COF_2$$

$$(CF_3)_3PO + \ddot{C}F_2 \longrightarrow (CF_3)_3P + COF_2$$

The formation of hexafluoroethane, however, suggested a radical cleavage similar to that found with tris (trifluoromethyl) phosphine, was also occurring.

Although irradiation in the presence of ethylene gave a small amount of volatile material, this did not contain P-CF_3 groups; the involatile product contained CF_3, $CF_3P(O)$, and CH_2CH_2 groups, but was clearly not a 1:1 adduct, and could not be identified. Since 71% of the phosphine oxide was recovered unchanged, the reaction was not investigated further. Irradiation of the phosphine oxide with propyne and with tetrafluoroethylene was also unsuccessful, the only major reaction being complete polymerisation of the tetrafluoroethylene.

The photochemical reactions of bis(trifluoromethyl)phosphine and of dimethylphosphine with 3,3,3-trifluoropropene gave excellent yields of the 1:1 adducts (1) and (3) respectively, with no trace of the possible isomers (4) and (5).

$Me_2PCH_2CH_2CF_3$ (3)

$(CF_3)_2PCHMeCF_3$ (4)

$Me_2PCHMeCF_3$ (5)

Attack by the $R_2\dot{P}$ radical on this olefin is thus exclusively at the terminal position, as predicted on the simple basis of stability of the intermediate radicals

$R_2PCH_2\dot{C}HCF_3$ more stable than $R_2PCH(CF_3)\dot{C}H_2$

without regard to the electronegativity of the substituents attached to the phosphorus [cf. 7]. Dimethylphosphine failed to react with trifluoropropene in the dark at 50 °C. The possibility of nucleophilic attack by the phosphine on this olefin is thus discounted, although it occurs with such highly electron-deficient olefins as hexafluoropropene and 1,1-dichloro-2,2-difluoroethylene [8].

EXPERIMENTAL

Reactions were carried out in sealed silica or Pyrex reaction tubes (ca. 300 cm^3 volume), using standard vacuum-line manipulation techniques. N.m.r. chemical shifts (^1H at 60.0 MHz, internal T.M.S., ^{19}F at 56.46 MHz, external T.F.A.) are given positive values to low field of the reference. Mass spectra were obtained using an A.E.I. MS902 spectrometer.

Photochemical Reactions of Tris(trifluoromethyl)phosphine

These reactions were carried out in silica tubes, placed 20 cm from a Hanovia U.V.S. 500 medium pressure mercury lamp, with the products shielded from further irradiation.

(a) With ethylene

Irradiation (120 h) of ethylene (0.27g, 9.7 mmol) and tris(trifluoromethyl)phosphine (2.30g, 9.7 mmol) gave tetrakis(trifluoromethyl)diphosphine (0.014g, 1%), recovered tris(trifluoromethyl)phosphine (0.30g, 14%), bis(trifluoromethyl)-3,3,3-trifluoropropylphosphine (nc)(1.61g, 73% based on consumed phosphine), b.p. (isoteniscope) 91.3 °C, ^{19}F n.m.r + 8.2 (complex multiplet, $\underline{CF_3}CH_2$) + 21.5 (d, CF_3-P 74.7 Hz) with intensity ratio 1:2, ^{1}H n.m.r. + 2.7(complex AA'BB' with further coupling to CF_3 and to P).

Analysis: Found: C, 22.9; H, 1.7%; $\underline{M}^+$, 266.

$C_5H_4F_9P$ requires C, 22.6; H, 1.5%; $\underline{M}^+$, 266.

(b) With vinyl fluoride

Vinyl fluoride (0.46g, 10 mmol) and the phosphine (2.38g, 10 mmol) were irradiated (120 h) to give a mixture (0.66g, 23%) shown by n.m.r. to consist mainly of $CF_3CH_2CHFP(CF_3)_2$ [^{19}F n.m.r. + 27.6, a doublet of multiplets, resolved at 94.1 MHz into a pair of doublets (76 Hz) of quintets (8 Hz), separated by 39 Hz, assigned to the $(CF_3)_2P$ group adjacent to a chiral carbon, +12.6, (d, 8Hz of t, 10 Hz, CF_3C) and -117.5(multiplet, CFH)] and $CF_3CHFCH_2P(CF_3)_2$ [^{19}F n.m.r. + 23.2(d 77 Hz of multiplets $(CF_3)_2PCH_2$), + 13.6(d 8 Hz of q 8 Hz, CF_3C), and -66.0 (d 48Hz of d 30 Hz of d 12 Hz of q 7 Hz, CFH)] in the ratio 5.8:1, together with another small component. Vinyl fluoride (<2%), tris-(trifluoromethyl)phosphine (0.7g, 30%), and 1,1,1,3-tetrafluoropropane (0.6g, 45%), and a small amount of fluoroform were also identified. A yellow oil (0.46g) was shown to be complex mixture.

(c) With vinylidene fluoride

Vinylidene fluoride (0.64g, 10 mmol) and the phosphine (2.38g, 10 mmol) were irradiated (120 h) to give a mixture (0.20g, 6%) consisting mainly of $CF_3CH_2CF_2P(CF_3)_2$ [^{19}F n.m.r. + 29.2, d 81 Hz, $(\underline{CF}_3)_2PCF_2$; -12.4, d 85 Hz of nonets 8 Hz, CF_2; + 18 mult, overlapped by other signals, CF_3. ^1H, + 3.0 complex multiplet, CH_2 group] together with two other components containing $(CF_3)_2PCH_2$ and one containing $(CF_3)_2P$ group [^{19}F n.m.r., +23, two doublets (78 Hz) of triplets (2 Hz) and a doublet (76 Hz) of multiplets]. Tris(trifluoromethyl)phosphine (0.7g, 30%) and vinylidene fluoride (0.2g, 30%) were recovered.

(d) With vinyl chloride

After irradiation (168 h) of a mixture of tris(trifluoromethyl) phosphine (2.35g, 9.8 mmol) and vinyl chloride (0.61g, 9.8 mmol), the phosphine (1.93g, 83%) and olefin (0.45g, 74%) were recovered. G.c./m.s. allowed the identification of the following components of the volatile product (0.2g): $(CF_3)_2PCH=CHCF_3$ (trace); $CH_2=CHP(CF_3)_2$ (trace); $CF_3CH_2CH_2Cl$ or $CF_3CHClCH_3$ (*ca.* 16%); $CF_3CH_2CH_2P(CF_3)_2$ (trace); $CHCl=CHP(CF_3)_2$ or $CH_2=CClP(CF_3)_2$ (*ca.* 8%); $CF_3CHClCH_2P(CF_3)_2$ or $CF_3CH_2CHClP(CF_3)_2$ (trace); $CH_2=CClP(CF_3)_2$ or $CHCl=CH_2P(CF_3)_2$ (8%); and $CH_2ClCH_2P(CF_3)_2$ or $CH_3CHClP(CF_3)_2$ (ca. 12% based on phosphine consumed).

(e) With propene

Tris(trifluoromethyl)phosphine (1.73g, 74%) and propene (0.27g, 66%) were recovered after the phosphine (2.35g, 9.8 mmol) and propene (0.41g, 9.8 mmol) had been irradiated for 168 h. The volatile product (0.40g) was shown by g.c./m.s. to contain: $CH_3CH{:}CHP(CF_3)_2$ (*ca.* 21%); $CH_3CH_2CH_2P(CF_3)_2$ (27%; ^{19}F n.m.r. shows doublet +22.6δ, with $J(CF_3-P)$ 72.5 Hz, in agreement with that found for $CH_3CH_2CH_2P(CF_3)_2$ prepared from bis(trifluoromethyl)phosphine and propene [4]); $CH_3CH(CH_2CF_3)P(CF_3)_2$ (16% based on phosphine consumed); and $CH_3CH(CF_3)CH_2P(CF_3)_2$ (trace).

(f) With hexafluoropropene

After irradiation (300 h) of a mixture of tris(trifluoromethyl)-phosphine (2.35g, 9.8 mmol) and hexafluoropropene (1.45g, 9.8 mmol), the phosphine (1.40g, 60%) and olefin (0.78g, 53%) were recovered, and the higher-boiling volatile product (1.12g) was shown by g.c./m.s. to be a very complex mixture containing only a trace of $[(CF_3)_3PC_3F_6]$.

(g) With propyne

The phosphine (1.76g, 7.4 mmol) and propyne (0.30g, 7.4 mmol) were irradiated (96 h) to give, after preparative g.c., 1,1,1-trifluoro-3-bis(trifluoromethyl)phosphino-cis-but-2-ene (n.c.)(0.34g, 24% based on phosphine consumed), ^{19}F n.m.r. +25.3(d, $J(CF_3)_2P$ 82 Hz), and +16.8 (d, J CF_3-H 7.3 Hz, d, JCF_3-P 4 Hz, q JCF_3-CH_3 2 Hz), ^{1}H n.m.r. 6.0δ, (d, J H-P 19.3 Hz, q, JH-CF_3 7.3 Hz, q, JH-CH_3 1.6 Hz) and 1.9δ (multiplet) in the ratio 1:2. Tris(trifluoromethyl)phosphine (0.54g, 31%) and propyne (0.03g, 10%) were recovered, and a high-boiling liquid product (0.5g) was shown by g.c./m.s. to be mainly a 2:1 adduct $[(CF_3)_3PC_6H_8]$.

Analysis: Found: C, 26.4; H, 1.8%; $\underline{M}^+$, 278
$C_6H_4F_9P$ requires C, 25.9; H, 1.4%; $\underline{M}$, 278

(h) With propene in the presence of trifluoroiodomethane

Tris(trifluoromethyl)phosphine (1.32g, 5.6 mmol), propene (0.23g, 5.6 mmol) and trifluoroiodomethane (0.10g, 0.5 mmol) were irradiated (268 h) to give recovered phosphine (1.32g, 100%), propene (0.21g, 91%), and small amounts of high-boiling liquid and of iodine.

Thermal Reaction of Tris(trifluoromethyl)phosphine with Ethylene

Tris(trifluoromethyl)phosphine (1.60g, 6.7 mmol) and ethylene (0.19g, 6.7 mmol) were kept at 200 °C (200 h) to give recovered ethylene (0.16g, 87%) and phosphine (1.44g, 90%), together with bis(trifluoromethyl)-3,3,3-trifluoropropylphosphine (0.15g, 8% on initial phosphine, 81% on phosphine consumed), and a tarry residue.

Photochemical Reactions of Tris(trifluoromethyl)phosphine Oxide

Tris(trifluoromethyl)phosphine oxide, conveniently prepared by chlorination of the phosphine at room temperature, followed by reaction of the dichlorophosphorane with oxalic acid [5], was irradiated alone and with olefins as described above for the phosphine.

(a) Alone

Tris(trifluoromethyl)phosphine oxide (0.35g, 1.4 mmol) was irradiated (200 h) to give a small amount of non-condensable material (0.05 mmol), recovered phosphine oxide (0.11g, 31%), tris(trifluoromethyl)phosphine (0.05g, 16%), and a mixture of carbonyl fluoride, hexafluoroethane, and phosphorus trifluoride, identified by their i.r. spectra.

(b) With ethylene

Irradiation of the phosphine oxide (1.96g, 7.7 mmol) and ethylene (0.22g, 7.7 mmol) for 200 h gave recovered phosphine oxide (1.39g, 71%) and ethylene (0.089g, 41%), a small volatile fraction (0.5g) which showed CF_3(but not P-CF_3) signals in its ^{19}F n.m.r. spectrum, and an unidentified liquid product muxture (0.30g) which showed a CF_3-P(V) doublet (+4.3, 98 Hz) and complex signals at +10.4, + 11.4 in its ^{19}F n.m.r. spectrum.

(c) With tetrafluoroethylene

The phosphine oxide (1.72g, 6.8 mmol) and tetrafluoroethylene (0.68g, 6.8 mmol) were irradiated (70 h) to give a mixture (ca. 1 mmol) of carbonyl fluoride, phosphorus trifluoride, and hexafluoroethane, recovered tris(trifluoromethyl)phosphine oxide (1.54g, 90%), and polytetrafluoroethylene (0.65g, 97%).

Photochemical Reaction of Bis(trifluoromethyl)phosphine with 3,3,3-Trifluoropropene

Bis(trifluoromethyl)phosphine (2.01g, 11.8 mmol) and 3,3,3-trifluoropropene (1.14g, 11.8 mmol) were irradiated (120 h) to give hydrogen (0.1 mmol, 2%); a mixture of trifluoromethane (0.7 mmol, 6%), trifluoropropene (0.15g, 13% recovery), 3,3,3-trifluoropropane (ca. 0.1 mmol, 1%), bis(trifluoromethyl)phosphine (0.2g, 10% recovery) and an unidentified component (0.9 mmol); tetrakis(trifluoromethyl)diphosphine (0.08g, 4% on phosphine consumed) and bis(trifluoromethyl)-3,3,3-trifluoropropylphosphine (2.46g, 90% on olefin consumed).

Photochemical Reaction of Dimethylphosphine with 3,3,3-Trifluoropropene

Dimethylphosphine (0.62g, 10.1 mmol) and 3,3,3-trifluoropropene (1.10g, 11.4 mmol) were irradiated (50 h) to give hydrogen (0.3 mmol, 4%), a mixture of dimethylphosphine (0.05g, 7%) and trifluoropropene (0.06g, 6%), trifluoropropane (0.3 mmol), and dimethyl-3,3,3-trifluoropropylphosphine (n.c.) (1.29g, 78% on olefin consumed), b.p. (isoteniscope) 105.5 °C, ^{19}F n.m.r. + 10.3 (t, JCF_3CH_2 10.3 Hz, d, JCF_3-P 2.8 Hz) and ^1H n.m.r. 2.1 (d, JCH_3P 2.7 Hz) 2.6 and 3.3 (complex multiplets).

Analysis: Found: C, 38.3; H, 6.5%; $\underline{M}^+$, 158.

$C_5H_{10}F_3P$ requires C, 38.0; H, 6.3%; $\underline{M}$, 158.

Thermal Reaction of Dimethylphosphine with 3,3,3-Trifluoropropene

Dimethylphosphine (0.75g, 12.1 mmol) and 3,3,3-trifluoropropene (1.16g,12.1 mmol) were kept in the dark at 50 °C (120 h) to give recovered olefin (1.09g, 95%) and phosphine (0.62g, 83%), and an unidentified solid (0.15g).

REFERENCES

1 Part 20. R.N. Haszeldine, D.R. Taylor and E. White, J. Fluorine Chem., (1978) 11, 441.

2 R. Fields, R.N. Haszeldine and N.F. Wood, J.Chem.Soc.(C), (1970) 1370.

3 R.N. Haszeldine, J.Chem.Soc., (1952) 2504; R.N. Haszeldine and B.R. Steele, J.Chem.Soc., (1953) 1199.

4 R. Fields, R.N. Haszeldine and J. Kirman, J.Chem.Soc.(C), (1970) 197.

5 R.C. Paul, J.Chem.Soc., (1955) 574.

6 W. Mahler, J. Amer. Chem.Soc., 90 (1968) 523.

7 R. Fields, R.N. Haszeldine and N.F. Wood, J.Chem.Soc. (C), (1970) 744.

8 P. Cooper, R. Fields and R.N. Haszeldine, J.Chem.Soc.Perkin Trans. I, (1975) 702.