Unusual products in the reactions of phosphorus(III) compounds with N=N, C≡C or conjugated double-bonded systems

K C KUMARA SWAMY,* E BALARAMAN, M PHANI PAVAN, N N BHUVAN KUMAR, K PRAVEEN KUMAR and N SATISH KUMAR

School of Chemistry, University of Hyderabad, Hyderabad 500 046
e-mail: kckssc@uohyd.ernet.in

Abstract.   The diversity of products in the reaction of diethyl azodicarboxylate (DEAD)/diisopropyl azodicarboxylate (DIAD) and activated acetylenes with P^{III} compounds bearing oxygen or nitrogen substituents is discussed. New findings that are useful in understanding the nature of intermediates involved in the Mitsunobu reaction are highlighted. X-ray structures of two new compounds (2-*t*-Bu-4-MeC$_6$H$_3$O)P (μ-N-*t*-Bu)$_2$P$^+$[(NH-*t*-Bu){N[(CO$_2$-*i*-Pr)(HNCO$_2$-*i*-Pr)]}](Cl$^-$)(2-*t*-Bu-4-MeC$_6$H$_3$OH) (**23**) and [CH$_2$(6-*t*-Bu-4-Me-C$_6$H$_2$O)$_2$P(O)C(CO$_2$Me)C-(CO$_2$Me)CClNC(O)Cl] (**33**) are also reported. The structure of **23** is close to one of the intermediates proposed in the Mitsunobu reaction.

Keywords.   Mitsunobu reaction intermediates; X-ray crystal structure; phosphonates; pentacoordinate phosphorus.

1.   Introduction

A combination of triphenylphosphine (Ph$_3$P) with a dialkyl azodicarboxylate (RCO$_2$N=NCO$_2$R) or an activated acetylene (R′C≡CCO$_2$R) is a very versatile reagent system for a variety of synthetic organic transformations (scheme 1).[1–3] There are several intermediates (e.g. **1**–**6**) proposed in this reaction but most of them do not have structural proof. Our interest in phosphorus chemistry prompted us to investigate the related basic reactions utilizing other P^{III} systems in an effort to (i) isolate 'intermediates' from the reaction of P^{III} precursors with electron-deficient alkenes/alkynes/azo compounds, and (ii) probe the reaction pathways of known reactions that utilize P^{III} compounds. In this direction we have utilized precursors of the type **7**–**14** (chart 1). Herein we highlight some of the interesting results using these precursors. In addition, we also report the synthesis and structural characterization of two new compounds (**23** and **33**) based on these precursors.

2.   Experimental section

Details of experimental methods and solvents are reported elsewhere.[4–10]

---

*For correspondence
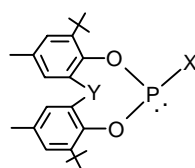
X	Y	
NH-*t*-Bu	S	[**7**, δ(P) 138.2]
NH-*t*-Bu	CH$_2$	[**8**, δ(P) 141.9]
NCO	CH$_2$	[**9**, δ(P) 121.2]
N$_3$	CH$_2$	[**10**, δ(P) 136.5]
OPh	S	[**11**, δ(P) 136.1]
NCS	CH$_2$	[**12**, δ(P) 121.2]
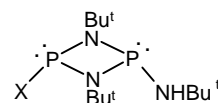
X = Cl [**13**, δ(P) 135.4, 200.3 ^2J(PP) = 44.0 Hz]
= NH-*t*-Bu [**14**, δ(P) 89.4]

Chart 1.

2.1   *Synthesis of (2-t-Bu-4-Me-C$_6$H$_3$O)P (μ-N-t-Bu)$_2$P$^+$[(NH-t-Bu){N[(CO$_2$-i-Pr)(HNCO$_2$-i-Pr)]}](Cl$^-$)(2-t-Bu-4-Me-C$_6$H$_3$OH) (23)*

This compound was prepared in the same way as reported previously under (2-*t*-Bu-6-Me-C$_6$H$_3$O)P(μ-N-*t*-Bu)$_2$P$^+$[(NH-*t*-Bu){N[(CO$_2$-*i*-Pr)(HNCO$_2$-*i*-Pr)]}](Cl$^-$) but with the phenol to compound **18** molarity ratio of ca 3 : 2.[6,7] The crystals (ca 75% yield) were obtained from toluene at 5°C. ^{1}H NMR: δ 1·25–1·55 (many lines, 57 H), 2·23 (*s*, 6 H), 4·78–5·09 (*br*, 2 H), 6·72–7·60 (many lines, *ca* 8 H), 10·4 (*br*, 1 H). ^{13}C NMR: δ 20·8, 21·5, 21·7, 21·8, 22·0, 29·6, 30·5, 30·8, 31·0, 31·1, 31·2, 34·4, 34·9, 56·3, 56·4, 57·3, 70·4, 73·3, 115·8, 116·6, 116·8, 127·0, 127·2, 129·3,

132·8, 135·5, 140·0, 150·5 (d, J = 19·2 Hz), 155·3. ^{31}P NMR: δ 9·4, 114·6 (d each, J = 10·4 Hz).

2.2 *Synthesis of [CH$_2$(6-t-Bu-4-Me-C$_6$H$_2$O)$_2$P(O) C(CO$_2$Me)C(CO$_2$Me)CClNC(O)Cl] (33)*

Compound **30a** (0·55 g, 1·0 mmol) in excess CHCl$_3$ (10 ml) was heated at 70°C for 1 day with continuous stirring. Removal of the solvent afforded **33** as a white solid. This was crystallized using a mixture of dichloromethane (2 ml) and hexane (1 ml). Yield: 0·46 g (70%). M.p.: 142–144°C. IR (KBr): 1750, 1730, 1458, 1375, 1260, 1211, 1100 cm^{-1}. ^{1}H NMR: δ 1·42 (s, 18 H, t-Bu-H), 2·29 (s, 6 H, ArCH_3), 3·51 (d, ^2J(HH) = 13·5 Hz, 1 H, ArCH$_A$H$_X$), 3·96, 3·98 (2 s, 6 H, OCH_3), 4·25 (dd, ^5J(PH) = 2·8 Hz, ^2J(HH) = 13·5 Hz, 1 H, ArCH$_A$H$_X$), 7·06 (br, 4 H, Ar-H). ^{31}P NMR: δ −3·8.

Scheme 1.

2.3 *X-ray structural analysis*

X-ray data were collected on a Bruker AXS SMART diffractometer (for **23**) or an Enraf-Nonius-MACH3 (for **33**) using Mo-K$_\alpha$ (λ = 0·71073 Å) radiation. The structures were solved by direct methods;[8] all non-hydrogen atoms were refined anisotropically. For hydrogen atoms except the NH atoms in **23**, the riding model was used; the methyl carbons of the isopropyl groups in **23** showed disorder and hence for one of the isopropyl groups, refinement was done using a model with three positions for the (expected) two carbons keeping a total occupancy of 2; the remaining parts of the molecule were fine. The next highest residual density was close to the second isopropyl carbons.

2.4 *Crystal data*

Compound **23**: C$_{42}$H$_{74}$ClN$_5$O$_6$P$_2$, M = 842·45, triclinic, space group $P\bar{1}$, a = 10.0975(8), b = 15·8105(13), c = 16·0880(13), α = 82·314(1), β = 79·987(1), γ = 85·4710(1), V = 2502·5(4) Å^3, Z = 2, ρ = 1·118 g cm^{-3}, F(000) = 912, μ = 0·185 mm^{-1}, Data/restraints/parameters: 8782/1/539. S (all data) = 1·068. R indices (I > 2σ(I)): R1 = 0·0583, wR2 (all data) = 0·1792. Max./min. residual electron density (eÅ^{-3}) 0·727/−0·311. Compound **33**: C$_{31}$H$_{36}$Cl$_2$NO$_8$P, M = 652·48, monoclinic, space group $P2_1/n$, a = 9·284(2), b = 17·582(8), c = 20·665(3), β = 102·66(2), V = 3291·2(17) Å^3, Z = 4, ρ = 1·317 g cm^{-3}, F(000) = 1368, μ = 0·295 mm^{-1}, Data/restraints/parameters: 5773/0/398. S (all data) = 1·054, R indices (I > 2σ(I)): R1 = 0·0626, wR2 (all data) = 0·2074. Max/min residual electron density (eÅ^{-3}) 0·325/ −0·481. Further details as CIF files are available from the Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK on request, quoting the deposition numbers CCDC 297195 and 297196.
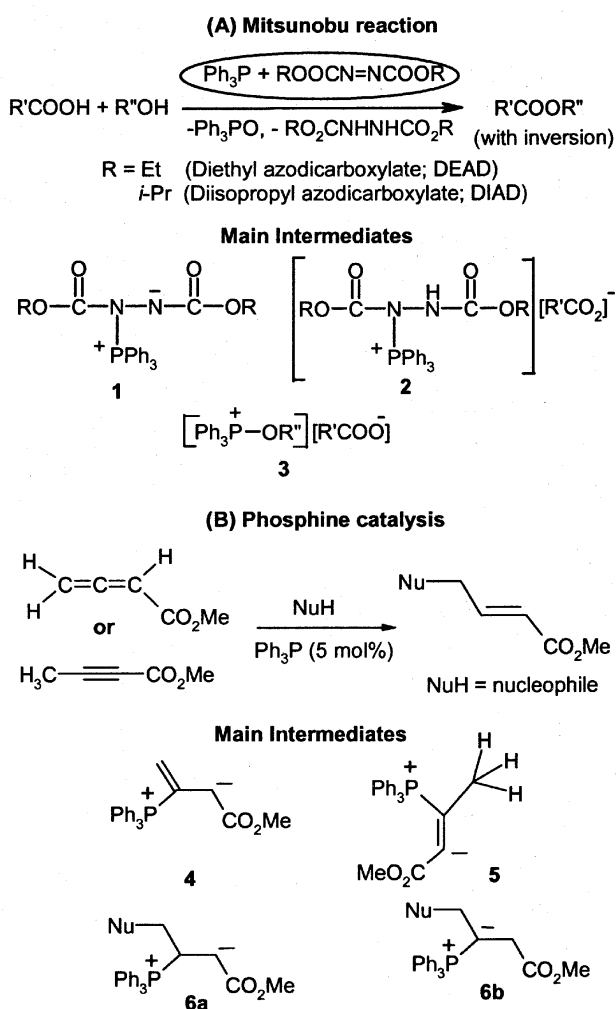
3. Results and discussion

The reaction of **7–8** with DEAD/DIAD led to the imino-phosphorus compounds **15–17**, that have a structure halfway between the classical MBH betaine **1** and protonated betaine in the Mitsunobu reaction (scheme 2).[9] A low temperature ^{31}P NMR study on compounds **16** and **17** revealed an unprecedented solution state behavior wherein *at least four* isomeric phosphoranes (**A**) are present. Thus there is an ap-

parent inconsistency between the solution state and solid-state structures. Interestingly, even the solid-state ^{31}P NMR spectra of **16–17** exhibited a peak at $\delta \sim -50$.[9,10]

In a manner analogous to that for **15–17**, we could readily prepare the compound **18–20** by starting with the diphosphazane precursors **13–14**.[5,9] Interestingly, in the reaction of **18** with the trifluoro-ethanol and 2-*t*-butyl-4-methyl-phenol we obtained products **21** and **23**, respectively, in which the original *t*-BuNH group on phosphorus is *trans* to the alkoxy/phenoxy group, but with 2,6-dichlorophenol we obtained **22** in which the phenoxy group is *cis* to the P-NH-*t*-Bu group (scheme 3).[6,7] However, in **22** and **23** there is an additional phenoxy group in the crystals. The X-ray structure (figure 1) of **23** clearly shows the hydrogen bonding and the disposition of the substituents. The phenoxy group in **23** is *trans* to the *t*-BuNH group on the tricoordinate phosphorus as observed for **21** but in contrast to the *cis* orientation observed for **22**. This difference is rather unexpected and at the moment we do not have a clear-cut explanation for this observation.[11] The geometrical parameters in **23** are close to those observed for **21** and **22**; one noteworthy point is that the P–O distance in the *trans* compounds **21** [1·637(2) Å] and **23** are slightly shorter than that in the *cis* compound **22** [1·670(3) Å]. In all these three structures the tetracoordinate phosphorus [P(2)] has phosphonium character similar to intermediate **2**
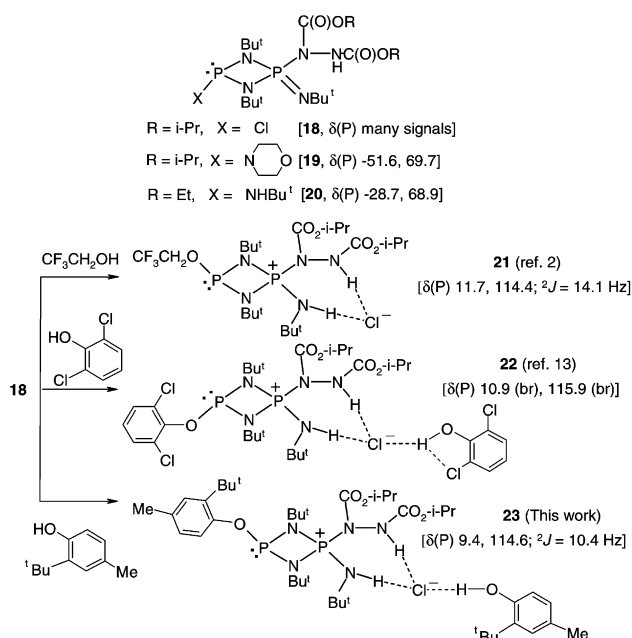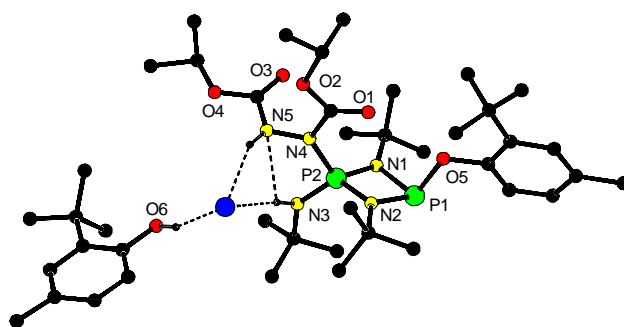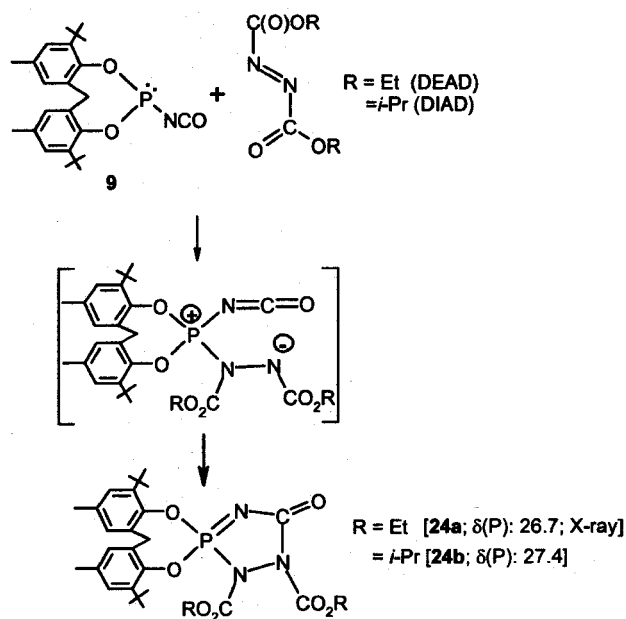
Scheme 3.
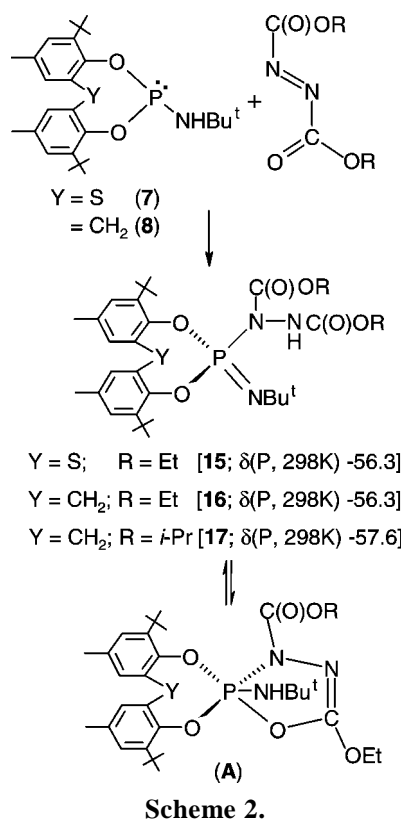
Scheme 2.

Figure 1. A Platon drawing of **23**. Selected bond parameters (Å, °): P(1)–O(5) 1·637(2), P(1)–N(2) 1·734(2), P(1)–N(1) 1·738(2), P(2)–N(3) 1·592(2), P(2)–N(1) 1·631(2), P(2)–N(2) 1·641(2), P(2)–N(4) 1·695(2), N(2)–P(1)–N(1) 80·50(9), N(3)–P(2)–N(1) 118·49(12), N(3)–P(2)–N(2) 119·68(11), N(1)–P(2)–N(2) 86·60(10), N(3)–P(2)–N(4) 100·36(11), N(1)–P(2)–N(4) 118·94(10), N(2)–P(2)–N(4) 113·95(10), P(1)–N(1)–P(2) 96·27(10). Hydrogen bond D–H, H⋯A, D…A and D–H⋯A parameters (symmetry equiv 1 + *x*, *y*, *z*) (Å, Å, Å, °): N(3)–H(3)…N(5) 0·81(3), 2·37(3), 2·742(3), 110(2); O(6)–H(6)…Cl 0·82, 2·28, 3·062(3), 160·6, N(5)–H(5)…Cl′ 0·89(3), 2·24(3), 3·040(3), 149(3); N(3)–H(3)…Cl′ 0·81(3), 2·58(3), 3·358(3), 164(3).

shown in scheme 1; in place of the carboxylate residue in **2**, we have a chloride ion. Also, since the NH hydrogen of the azodicarboxylate residue is involved in hydrogen bonding interactions, it is likely that a similar situation is prevalent in the Mitsunobu reaction also. Further studies are needed to substantiate this assertion, however.

The P(III) isocyanate $CH_2(6\text{-}t\text{-Bu-4-Me-}C_6H_2O)_2$ P-NCO (**9**) reacted with DEAD/DIAD in an entirely different way resulting in the formation of the cyclic products **24a–b**, presumably via betaine in a stepwise fashion (scheme 4).[9] The corresponding isothiocyanate $CH_2(6\text{-}t\text{-Bu-4-Me-}C_6H_2O)_2$P-NCS (**12**) also gives a similar heterocycle along with an unusual tri-phosphorus compound.[12]

We have recorded variable time ^{31}P NMR spectra for **24b**. After 15 min of the addition of DIAD to a solution of $CH_2(6\text{-}t\text{-Bu-4-Me-}C_6H_2O)_2$P-NCO (**9**) in C_6D_6, we observed a peak at $\delta(P)$ –64·9 in the pentacoordinate region along with the peak at $\delta(P)$ 28·6 in the tetracoordinate region. After 25 min the intensity of the down field peak at $\delta(P)$ 28·6 increased at the cost of the up-field peak; after 35 min, the downfield peak at $\delta(P)$ 28·6 was the most predominant one. This corresponds to **24b**; the slight difference in $\delta(P)$ values in $CDCl_3$ [δ 27·4] and C_6D_6 [δ 28·6] is likely to be due to solvent effects[10]. These results suggest that a pentacoordinate intermediate may be involved in the formation of **24b**.
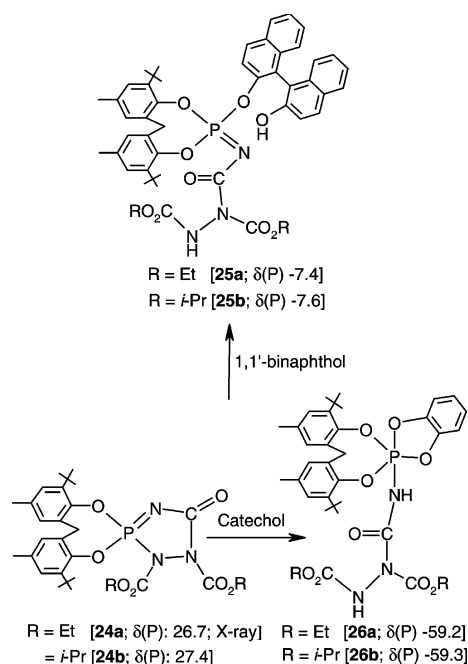
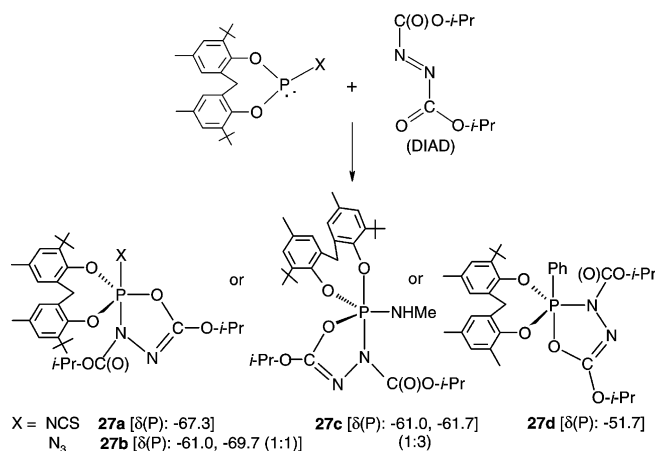Compounds **24a–b** can undergo a two-step addition depending on the diol.[9] First, the P–N single bond is cleaved and then addition across the P=N (double) bond takes place. When 1,1′-bi-2-naphthol is used, the reaction leads to tetra-coordinate compounds **25a–b**. With catechol, addition across the P=N bond also takes place to lead to the pentacoordinate compounds **26a–b** (scheme 5). The structures of **25b** and **26b** have been confirmed by X-ray crystallography. It should be noted that the betaine **1** reacts differently with catechol to lead to the pentacoordinate compound $Ph_3P(1,2\text{-}O_2C_6H_4)$ with the elimination of the hydrazine derivative $EtO_2CNHNHCO_2Et$.[13]

We have shown earlier that a major pathway in the reaction of cyclic phosphites present with

R = Et [**25a**; $\delta(P)$ -7.4]
R = i-Pr [**25b**; $\delta(P)$ -7.6]

1,1′-binaphthol

Catechol

R = Et [**24a**; $\delta(P)$: 26.7; X-ray]   R = Et [**26a**; $\delta(P)$ -59.2]
   = i-Pr [**24b**; $\delta(P)$: 27.4]   R = i-Pr [**26b**; $\delta(P)$ -59.3]

Scheme 5.

R = Et (DEAD)
=i-Pr (DIAD)

9

R = Et [**24a**; $\delta(P)$: 26.7; X-ray]
   = i-Pr [**24b**; $\delta(P)$: 27.4]

Scheme 4.

(DIAD)

X = NCS **27a** [$\delta(P)$: -67.3]   **27c** [$\delta(P)$: -61.0, -61.7]   **27d** [$\delta(P)$: -51.7]
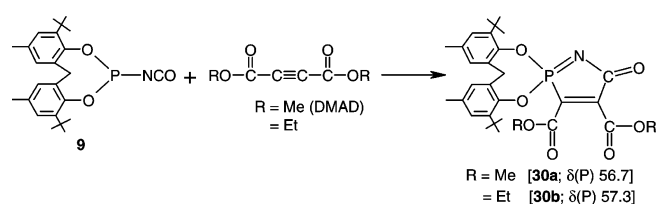 N_3 **27b** [$\delta(P)$: -61.0, -69.7 (1:1)]   (1:3)

Scheme 6.

DIAD/DEAD is the formation of pentacoordinate compounds **27a–d** (scheme 6).[14] It can be noted that three stereochemically different isomers are isolated and in many cases more than one ^{31}P NMR signal is observed in solution. In favorable cases, when there is an additional donor atom is present on the substituents, hexacoordination is also possible (e.g. **28–29**).[9,15] Here also isomerism is possible, as shown by the ^{31}P NMR spectrum of **28**, but we have not been successful in isolating isomers.
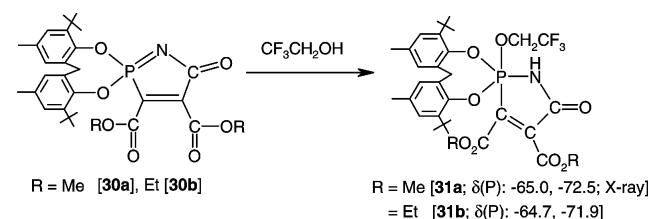

28; δ(P) -71.1, -90.3   **29**; δ(P) -89.8
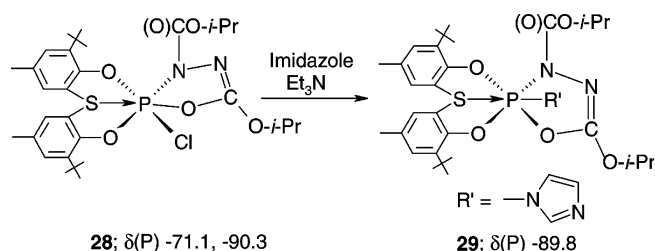
The reaction of CH$_2$(6-t-Bu-4-Me-C$_6$H$_2$O)$_2$P-NCO (**9**) with dipolarophiles like dimethyl acetylenedicarboxylate (DMAD) and diethyl acetylenedicarboxylate (DEACD) in toluene yielded products **30a–b** (scheme 7)[16]. The structure of **30a** was unambiguously proved by X-ray crystallography. This gives a convincing demonstration of the 1,3-(P,C) dipolar nature of P(III) isocyanates. It is also interesting to note that the reaction of **9** with RO$_2$CC≡CCO$_2$R (see above) and RO$_2$CN=NCO$_2$R leads to similar heterocycles.

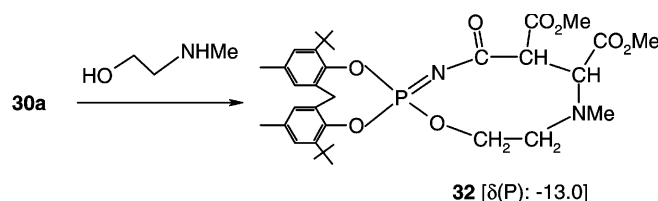Compounds **30a–b** are useful substrates for further reactions. They have a P–N double bond across which alcohols or any acidic components can be added.[16] It may also be noted that there is an α,β-unsaturated ester group in these compounds. The first feature is realized in the reaction of **30a–b** with 2,2,2-trifluoroethanol to lead to the pentacoordinate phosphoranes **31a–b** (scheme 8). The structure of **31a** is unambiguously proved by X-ray crystallography and shows an interesting feature: The carbon (bearing a bulky substituent) and not the nitrogen of the five-membered ring occupies the apical site of trigonal bipyramidal phosphorus. This 'reversed apicophilicity' is against commonly advocated principles using Bent's rule.[4,15,17] The ^{31}P NMR spectrum of **31a–b** at room temperature shows that the pentacoordination is retained in solution. Low temperature spectra recorded for **31a** showed three peaks [δ(P) –71·4, –69·9, –64·3] in toluene-d_8 solution. Although it is difficult to assign the peaks to individual isomers, this feature suggests that the isomerization is frozen at low temperatures. The above results prompted us to check the reactivity of the P=N bond in **31a** with acids. From the reaction with mesitoic acid, a solid that showed δ(P) at –67·8 (>85%) [other peak at –0·62] was obtained, but could not be crystallized.

In contrast to the above, the reaction of 2-methyl-aminoethanol with **30a** leads to a ring expansion to lead to the nine-membered heterocycle **32** (scheme 9)[16]. This type of reaction is unprecedented.

Chloroform also has an acidic proton and therefore it was of interest to see whether a compound of type **33′** could be obtained by heating **30a** with CHCl$_3$. However, the isolated compound **33** had the struc-
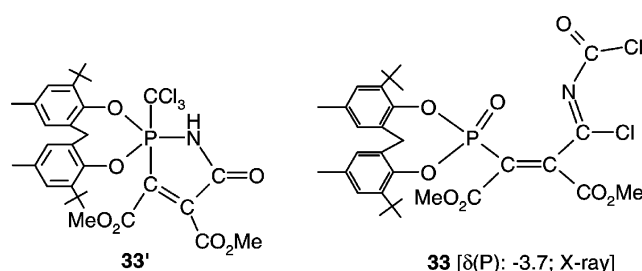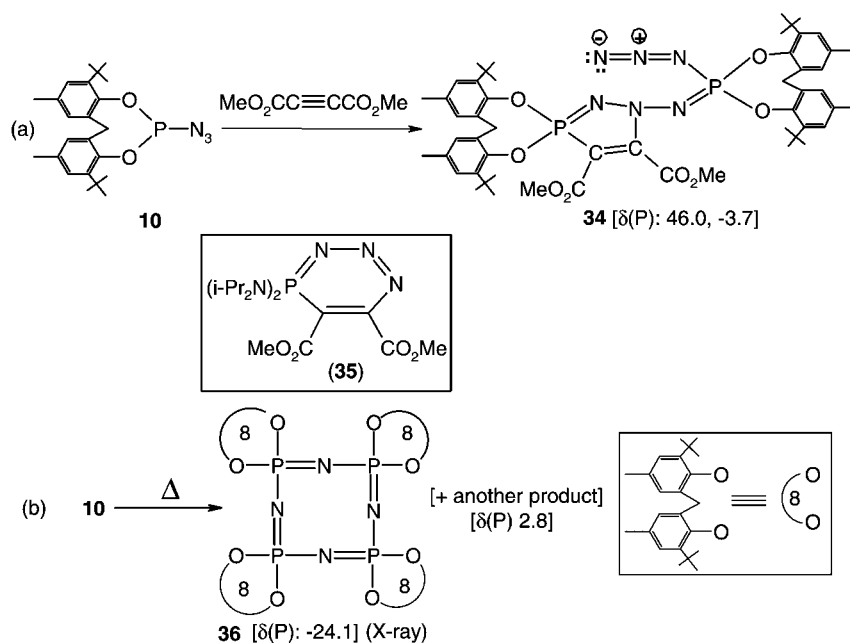

Scheme 7.

32 [δ(P): -13.0]

Scheme 9.

R = Me [**30a**], Et [**30b**]   R = Me [**31a**; δ(P): -65.0, -72.5; X-ray]
= Et [**31b**; δ(P): -64.7, -71.9]

Scheme 8.

33′   **33** [δ(P): -3.7; X-ray]

Table 1. Selected bond lengths [Å] and angles [°] for **33** with esd's in parentheses

P–O(1)	1·578(3)	C(24)–C(27)	1·335(6)
P–O(2)	1·582(3)	C(24)–C(25)	1·510(6)
P–O(3)	1·443(3)	C(27)–C(30)	1·497(6)
P–C(24)	1·794(4)	C(30)–N(1)	1·229(6)
C(30)–Cl(1)	1·727(5)	N(1)–C(31)	1·393(7)
C(31)–O(8)	1·165(7)	C(31)–Cl(2)	1·715(6)
O(1)–P–O(2)	107·60(16)	P–C(24)–C(27)	124·3(3)
O(1)–P–O(3)	116·43(18)	P–C(24)–C(25)	114·8(3)
O(1)–P–C(24)	99·61(17)	C(25)–C(24)–C(27)	120·9(4)
O(3)–P–O(2)	116·26(17)	Cl(2)–C(31)–O(8)	122·3(5)
O(3)–P–C(24)	116·06(19)	N(1)–C(30)–Cl(1)	124·2(4)
O(2)–P–C(24)	98·24(17))		

Scheme 10.
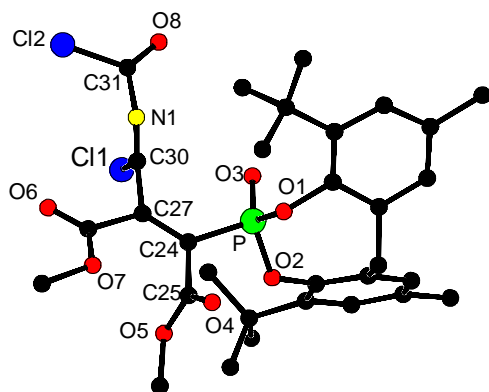
Figure 2. Molecular structure of **33** showing all non-hydrogen atoms (bond parameters in table 1).

ture shown below. Formation of **33** could involve addition of phosgene ($COCl_2$ formed by the air oxidation of $CHCl_3$) to **30a**. At the moment the details are not clear. It is also possible that **33** is formed via **33′**, but again we could not formulate a logical pathway.

The X-ray structure of **33** (figure 2; table 1) clearly shows the presence of two chlorines attached to C(30) and C(31) and the N(1)=C(30) imino bond. The other bond parameters are in the normal range.

The reaction of the P(III) azide **10** with DMAD also leads to a heterocycle, but there are two phosphorus residues per DMAD (scheme 10). This reaction pathway is completely different from that

observed for $[(i\text{-Pr})_2N]_2PN_3$[18] or the organic azide PhN_3.[19] In the reaction using the former, the six-membered heterocycle **35** was formed.[18] An attempted extension of this reaction to less reactive acetylenes was not successful because the precursor azide **10** is thermally unstable and leads to a mixture of cyclophosphazene derivatives from which $[CH_2(6\text{-}t\text{-Bu-4-Me-}C_6H_2O)_2P{=}N]_4$ (**36**) could be isolated.[20]

4. Summary

We have shown that the reactions of many P(III) compounds with dialky azodicarboxylates or dialkyl acetylene dicarboxylates lead to products different from those normally assumed in the first stage of Mitsunobu reaction or those involved in phosphine catalysed reactions of activated acetylenes respectively. We have also characterized X-ray structures of two products thus obtained in these reactions. One of these is similar to the type of intermediate proposed in the second stage of the Mitsunobu reaction.

Acknowledgements

References

1. Selected reviews: (a) Mitsunobu O 1981 *Synthesis* **1**; (b) Hughes D L 1992 *Org. React.* **42** 335; (c) Dembinski R 2004 *Eur. J. Org. Chem.* 2763
2. Selected references: (a) P-Cooper A and Evans Jr S A 1989 *J. Org. Chem.* **54** 2485; (b) Macor J E and Wehner J M 1993 *Heterocycles* **35** 349; (c) Camp D, Hanson G R and Jenkins I D 1995 *J. Org. Chem.* **60** 2977; (d) Hughes D L and Reamer R A 1996 *J. Org. Chem.* **61** 2967; (e) Ahn C, Correla R and deShong P 2002 *J. Org. Chem.* **67** 1751; (f) Schenk S, Weston J and Anders E 2005 *J. Am. Chem. Soc.* **127** 12566
3. Lu X, Zhang C and Xu Z 2001 *Acc. Chem. Res.* **34** 535 (phosphine catalysis).
4. Kommana P, Kumaraswamy S, Vittal J J and Kumara Swamy K C 2002 *Inorg. Chem.* **41** 2356
5. Praveen Kumar K, Chakravarty M and Kumara Swamy K C 2004 *Z. Anorg. Allg. Chem.* **630** 2063
6. Kumara Swamy K C, Praveen Kumar K and Bhuvan Kumar N N 2006 *J. Org. Chem.* **71** 1002 (see 7 also for correction)
7. In ref. 6, 2-*t*-butyl-4-methyl-phenol and not 2-methyl-6-*t*-butyl-phenol was used to prepare compound **16**; in the structure given R should be *para* to the phenolic oxygen
8. Programs used are: (a) Sheldrick G M 1996 *SADABS. Siemens Area Detector Absorption Correction*, University of Göttingen, Germany; (b) Sheldrick G M 1997 *SHELX-97, A package for structure solution and refinement*, University of Göttingen, Göttingen, Germany; (c) Sheldrick G M SHELXLTL+ 1991
9. Satish Kumar N, Praveen Kumar K, Pavan Kumar K V P, Kommana P, Vittal J J and Kumara Swamy K C 2004 *J. Org. Chem.* **69** 1881
10. Satish Kumar N 2004 PhD thesis, University of Hyderabad
11. For *cis-trans* isomerization in cyclodiphosph(III) azanes, see: (a) Reddy V S, Krishnamurthy S S and Nethaji M 1992 *J. Organomet. Chem.* **438** 99; (b) Reddy V S, Krishnamurthy S S and Nethaji M 1994 *J. Chem. Soc. Dalton Trans.* 2661
12. Kumaraswamy S, Senthil Kumar K, Satish Kumar N and Kumara Swamy K C 2005 *Dalton Trans.* 1847
13. Bone S A and Trippett S 1976 *J. Chem. Soc. Perkin I* 156
14. Satish Kumar N, Kommana P, Vittal J J and Kumara Swamy K C 2002 *J. Org. Chem.* **67** 6653
15. Pavan Kumar K V P, Satish Kumar N and Kumara Swamy K C 2006 *New J. Chem.* **30** 717
16. Kumaraswamy S, Kommana P, Satish Kumar N and Kumara Swamy K C 2002 *Chem. Commun.* 40
17. See, for example: (a) Kawashima T, Soda T and Okazaki R 1996 *Angew. Chem. Int. Ed. Engl.* **35** 1096; (b) Timosheva N V, Chandrasekaran A, Prakasha T K, Day R O and Holmes R R 1996 *Inorg. Chem.* **35** 6552; (c) Kojima S, Kajiyama K, Nakamoto M and Akiba K-y 1996 *J. Am. Chem. Soc.* **118** 12866; (d) Kumaraswamy S, Muthiah C and Kumara Swamy K C 2000 *J. Am. Chem. Soc.* **122** 964; (e) Kojima S, Sugino M, Matsukawa S, Nakamoto M and Akiba K-y 2002 *J. Am. Chem. Soc.* **124** 7674 and references cited therein; (f) Kommana P, Satish Kumar N, Vittal J J, Jayasree E G, Jemmis E D and Kumara Swamy K C 2004 *Org. Lett.* **6** 145; (g) Kumara Swamy K C and Satish Kumar N 2006 *Acc Chem. Res.* **39** 324
18. Bieger K, Tejeda J, Rèau R, Dahan F and Bertrand G 1994 *J. Am. Chem. Soc.* **116** 8087
19. Carey F A and Sundberg R J 2001 *Advanced organic chemistry, Part B: Reactions and synthesis* 4th edn (New York: Kluwer Academic/Plenum) pp 359–367
20. Kommana P, Kumaraswamy S and Kumara Swamy K C 2003 *Inorg. Chem. Commun.* **6** 394