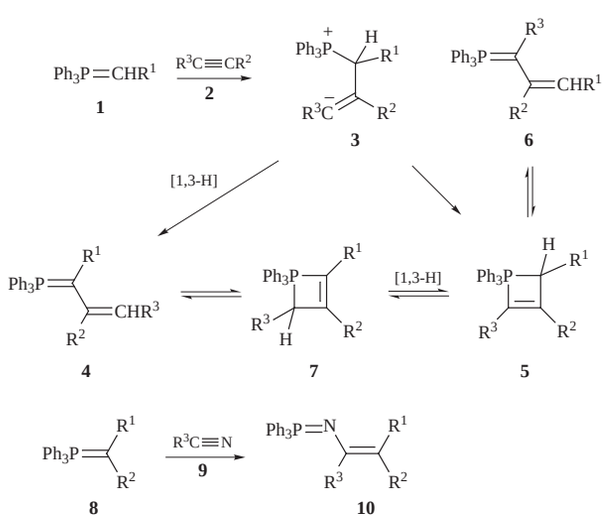
Reaction of stabilised arsonium ylides with acetylenic esters: convenient ring synthesis of a tetrasubstituted benzene

R. Alan Aitken,[a] Alexander J. Blake,[b] Ian Gosney,[b] Robert O. Gould,[b] Douglas Lloyd[a] and Raymond A. Ormiston[b]

[a] *School of Chemistry, University of St. Andrews, North Haugh, St. Andrews, Fife, UK KY16 9ST*
[b] *Department of Chemistry, The University of Edinburgh, West Mains Road, Edinburgh, UK EH9 3JJ*

Reaction of stabilised arsonium ylides such as 24 with methyl and ethyl propiolate in benzene gives the β,γ-unsaturated arsonium ylides 25 and 27 resulting from net insertion of the alkyne fragment into the C=As bond, but the isomeric ylides 26 and 28 corresponding to net insertion into the C–H bond when the reaction is carried out in methanol. The assignment of the structures is confirmed in the case of 25 by an X-ray structure determination. The corresponding phosphonium ylide 29 reacts with methyl propiolate to give 31 in either benzene or methanol in contrast to an earlier report. Further reaction of 25 with DMAD proceeds with net insertion of the alkyne into the C=C double bond and spontaneous intramolecular cyclisation to give the tetrasubstituted benzene derivative 39.

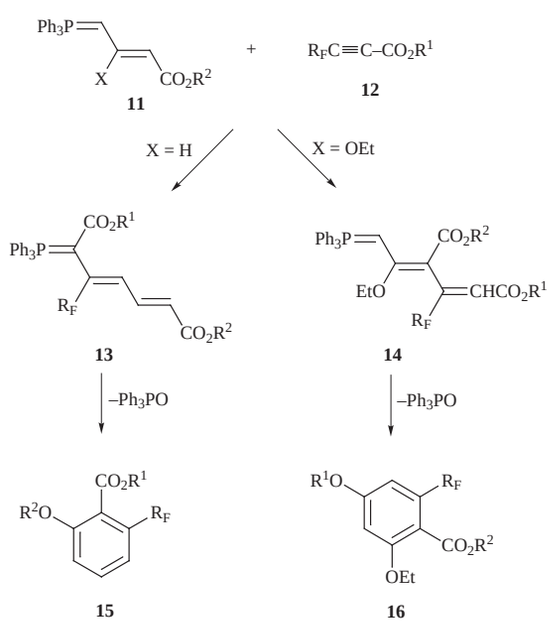
The reaction of phosphonium ylides **1** with activated alkynes **2** can give either of the two isomeric 1 : 1 adducts **4** and **6** depending on the substituents present and the reaction conditions (Scheme 1). The reaction may be considered to proceed by



Scheme 1

initial nucleophilic attack by the ylide carbon to give **3** and this can undergo an intramolecular proton transfer to afford **4** or else ring-close to **5** which can then open in the opposite direction to afford **6**. In 1964, Bestmann and Rothe reported that a variety of simple ylides **1** react readily with DMAD to give exclusively the products of type **6** ($R^2 = R^3 = CO_2Me$).[1] Hendrickson *et al*. reported that reaction of $Ph_3P{=}CHCOPh$ with DMAD gives the product **4** when the reaction is conducted in methanol but **6** in diethyl ether.[2] For the ylides **8** lacking a hydrogen on the ylide carbon, only products of type **6** are possible.[3] More recent studies have shown that the situation is more complex and that in many cases the products **4** and **6** can be interconverted under the reaction conditions. Lower reaction temperatures tend to favour formation of **4** while higher temperatures give **6**,[4–6] and heating a mixture of the two may result in conversion entirely into **6**.[7,8] Regardless of the nature of R^1 and R^2, the corresponding reaction of ylides **8** with activated nitriles **9** gives the product **10** analogous to **6**.[9] The net result of these simple reactions may be viewed as insertion of the alkyne fragment into the C=P bond to give **6** or into the C–H bond to give **4**.

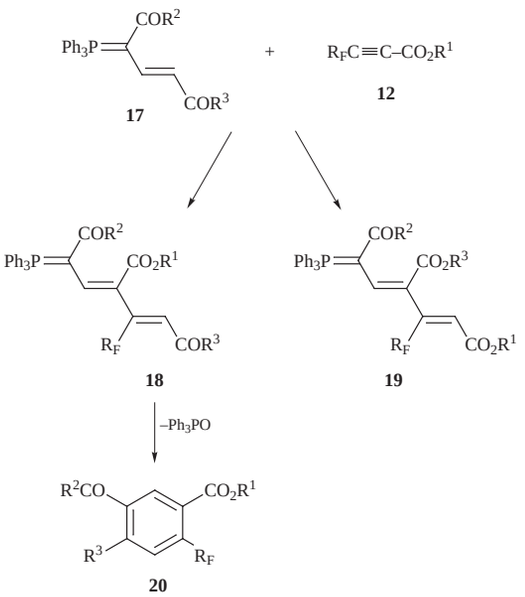
For the homologous β,γ-unsaturated ylides further possibilities arise and these have been extensively investigated by Ding and co-workers. Reaction of ylides **11** with a hydrogen on the ylide carbon with perfluoroalkylacetylenic esters **12** proceeds with net insertion into the C=P bond to give **13** for X = H,[10] but with net insertion into the C–H bond to give **14** for X = OEt (Scheme 2).[11] These reactions are of some synthetic



Scheme 2

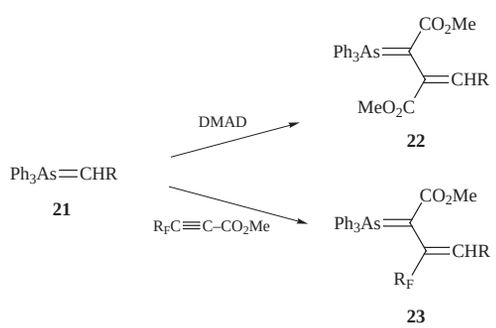
value since solution thermolysis of the products results in an intramolecular Wittig reaction to give the specifically tri- and tetra-substituted benzenes **15** and **16** respectively. For the ylides **17**, reaction with **12** generally proceeds with complete selectivity for insertion into the C=C bond to give **18** which is readily thermolysed to afford the 1,2,4,5-tetrasubstituted benzene

derivatives **20**,[4,5] although in one case a small amount of the isomeric product **19** resulting from net insertion into the γ-C–H bond was observed (Scheme 3).[6]



Scheme 3

It is well established that the arsonium ylides differ markedly from their phosphonium analogues in many aspects of their reactivity,[12,13] and so we were interested to examine the pattern of reactivity of these ylides with activated alkynes. The only two previous reports of reactions of this type involve two examples of ylides **21** (R = CO_2Et and COC_6H_4Br) reacting with DMAD to give **22**,[14] and **21** (R = $C_6H_4NO_2$) reacting with fluorinated acetylenic esters to give **23** (Scheme 4).[8] In



Scheme 4

this paper we describe the reaction of stabilised arsonium ylides with methyl and ethyl propiolate to give isomeric β,γ-unsaturated arsonium ylides depending on the solvent used, and the further reaction of one of the products with DMAD to give a specifically tetrasubstituted benzene.

Results and discussion

We first examined the reaction of benzoylmethylene(triphenyl)-arsorane **24** with methyl and ethyl propiolate (Scheme 5). This proceeded readily to give 1 : 1 adducts in moderate yield but the structure of the products depended on the solvent used. In boiling benzene the products **25** and **27** resulting from net insertion of the alkyne fragment into the C=As bond were obtained, whereas in boiling methanol the products were **26** and **28** resulting from net insertion into C–H. The isomeric ylides were readily distinguished by the fact that the alkene protons in **26** and **28** gave widely separated ^{1}H NMR signals at δ_H 7.7–7.8 and 4.4–4.5 with a coupling constant of 14–15 Hz clearly indicating

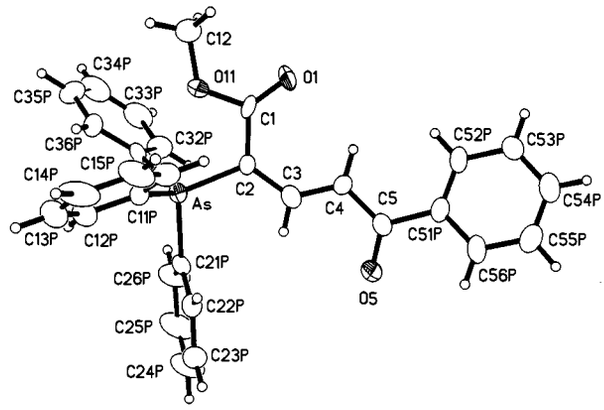
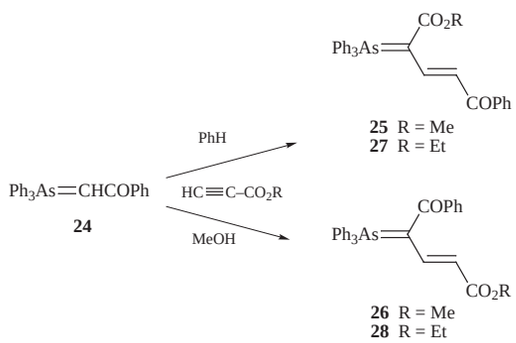
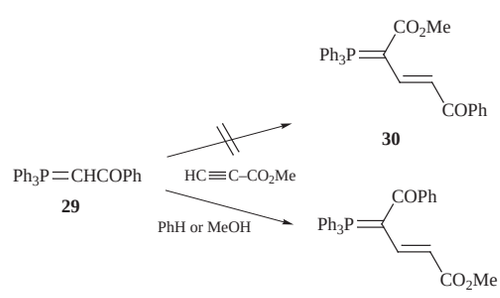


Fig. 1 X-ray structure of the arsonium ylide **25**. Selected bond lengths and angles; As–C(2) 1.877(7), As–C(21) 1.926(6), C(2)–C(1) 1.452(9), C(1)–O(1) 1.205(7), C(2)–C(3) 1.386(8), C(3)–C(4) 1.333(8), C(4)–C(5) 1.442(9), C(5)–C(51P) 1.516(9), C(5)–O(5) 1.234(7) Å; C(1)–C(2)–As 113.8(5), C(1)–C(2)–C(3) 125.0(7), C(3)–C(2)–As 121.2(5), C(2)–C(1)–O(11) 109.2(7), C(2)–C(1)–O(1) 127.1(7), O(1)–C(1)–O(11) 123.7(7), C(11P)–As–C(2) 113.4(3), C(11P)–As–C(21P) 105.3(3)°; dihedral angle As–C(2)–C(1)–O(11) 6.4(7)°; non-bonded distance As–O(11) 2.90 Å.



Scheme 5

the *E* configuration about the double bond, while the corresponding signals for **25** and **27** occurred under the aromatic signals at δ_H 7–8 and could not be readily picked out. The reason for the dramatic shielding of the C*H*CO_2R proton in **26** and **28** is unclear but a similar phenomenon has also been noted for the corresponding phosphonium ylides **30** and **31** (Scheme 6).[4]



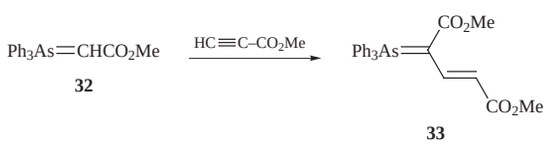
Scheme 6

Since the structure of the isomeric products in this area has previously rested on degradative studies, we thought it wise to confirm this by means of an X-ray structure determination. A suitable single crystal of **25** was obtained and the resulting structure is shown in Fig. 1. The C=As bond length and the shortening of C(1)–C(2), C(2)–C(3) and C(4)–C(5) with lengthening of C(1)–O(1), C(3)–C(4) and C(5)–O(5) indicative of delocalisation, is consistent with the results for previously studied stabilised arsonium ylides.[13] There is also evidence of considerable distortion in order to accommodate a through-

space interaction between arsenic and O(11). This is particularly evident in the reduced angles for As–C(2)–C(1) and C(2)–C(1)–O(11) and also in the anomalous position of the front phenyl group which has moved down to some extent towards a trigonal bipyramid position with a reduced value for C(11P)–As–C(21P) and proportionate widening of C(11P)–As–C(2). This type of interaction between arsenic and a suitably placed oxygen atom has been noted in previous X-ray studies,[15] although in all previous cases it has been with a carbonyl oxygen. The fact that **25** exists entirely with the *E* configuration of the As=C–C=O unit is rather surprising since by doing so it does not benefit from the favourable electrostatic interaction of the $As^{+}–C=C–O^{-}$ contributing form which would be present in the *Z* isomer.

The observed solvent effect on the regioselectivity of the reaction is in good agreement with previous studies of the corresponding phosphonium ylides, particularly the work of Hendrickson mentioned earlier,[2] and may be understood as the more polar solvent facilitating the intramolecular proton transfer in the intermediate corresponding to **3** and thus directing the reaction towards products of type **4** as opposed to **6**. Since Hendrickson's work only used DMAD, we thought it worth making a direct comparison between the arsenic and phosphorus series by reacting the phosphonium ylide **29** with methyl propiolate. This was found to give the product **31** analogous to **26** and **28** when the reaction was carried out either in benzene or methanol. While our work was in progress, Ding *et al.* described the same reaction but they obtained the other isomer **30** as the major product.[4] In their work reaction in CH_2Cl_2 at room temperature gave **30** and **31** in a ratio of 3:2 and this increased to 8.5:1 in DME at 90 °C and to 12:1 using CH_2Cl_2 in a sealed tube at 90 °C. The reason for the apparent discrepancy between these results is not clear.

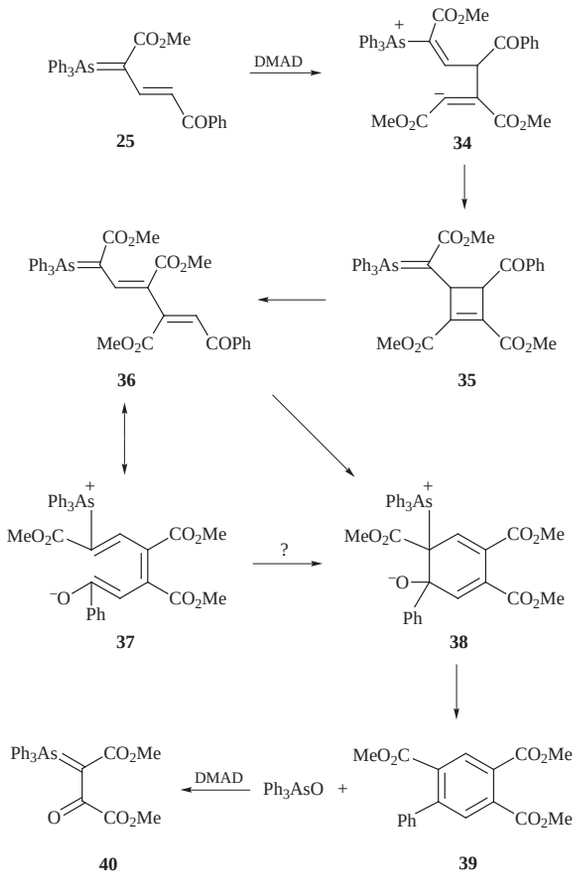
The ester-stabilised arsonium ylide **32** was found to react with methyl propiolate in a similar way (Scheme 7). In this case



Scheme 7

the two alternative mechanisms would give the same product **33** and this was indeed formed from reaction in either benzene or methanol.

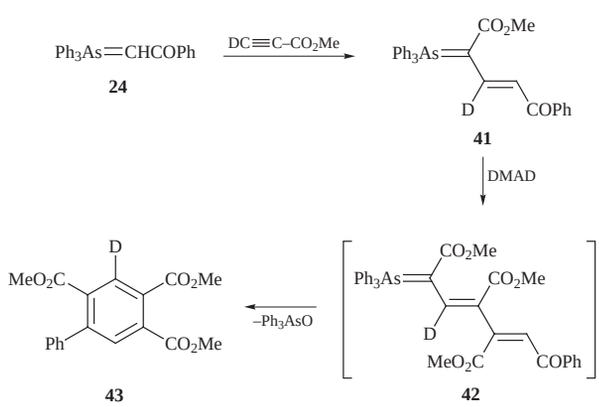
The reaction of **25** with DMAD was now investigated. When equivalent amounts of the two reagents were heated in boiling benzene for 5 h, the products included the ylide **40** formed by interaction of Ph_3AsO with DMAD (Scheme 8).[16] It was thus clear that for optimum yields, a 2:1 ratio of DMAD and **25** should be used. When this was done the products were **40** and the tetrasubstituted benzene **39**, both formed in over 80% yield. The latter gave the expected analytical and spectroscopic data with two singlets at δ_H 8.21 and 7.67 in the ^1H NMR spectrum supporting the 1,2,4,5-substitution pattern. The reaction most likely proceeds by initial nucleophilic attack on DMAD to give **34** which ring-closes to **35** and this then opens in the opposite direction to give **36**. This product is analogous to **18** formed in the corresponding reaction of the phosphonium ylide **17** with **12**. However it is well known that arsonium ylides are more reactive towards Wittig-type reaction than their phosphonium analogues,[12,13] and while the conversion of **18** into the benzene derivatives **20** has typically been carried out by heating in a sealed tube at 150–220 °C,[4,6] the arsonium ylide **36** undergoes spontaneous reaction under the conditions required for its formation to afford **39** and Ph_3AsO which reacts with the second equivalent of DMAD to give **40**. While the reaction could conventionally be regarded as nucleophilic attack of the ylide carbon on the remote carbonyl group to give the inter-



Scheme 8

mediate **38**, it may alternatively be formulated as a 6π electocyclic process of the resonance form **37** as shown.

In order to further confirm the proposed mechanism, we examined the effect of deuterium labelling. When the ylide **41** was prepared as for **25**, from **24** and methyl [3-^{2}H]propiolate, and then reacted with DMAD, the product **43** was that expected from ring-closure of **42**, *i.e.* it was the ^1H NMR signal at δ_H 8.21 corresponding to the ring proton between the two ester groups which was absent (Scheme 9).



Scheme 9

In conclusion, we have demonstrated that the reaction between stabilised arsonium ylides and propiolates may be controlled to give either of two sets of isomeric products with complete selectivity by choosing a solvent of suitable polarity. The reaction of one of the resulting β,γ-unsaturated arsonium ylides with an acetylenic ester has been examined for the first time and is found to result, as for the phosphonium analogues, in formation of a specifically tetrasubstituted benzene, but with the distinction that the more reactive arsonium ylide undergoes

spontaneous ring-closure, thus providing a convenient one-pot procedure.

Experimental

Melting points were recorded on a Kofler hot-stage microscope and are uncorrected. Infrared spectra were recorded as Nujol mulls on a Perkin-Elmer 157G instrument. NMR spectra were obtained for ^{1}H at 100 MHz on a Varian HA 100 instrument and for ^{13}C at 20 MHz and for ^{31}P at 32 MHz using a Varian CFT20 instrument. All spectra were run on solutions in $CDCl_3$ with internal Me_4Si as reference for ^{1}H and ^{13}C and external 85% H_3PO_4 as reference for ^{31}P. Chemical shifts are reported in ppm to high frequency of the reference and coupling constants *J* are in Hz. Mass spectra were obtained on an A. E. I. MS902 spectrometer using electron impact at 70 eV. Column chromatography was carried out using Laporte Industries alumina H. Benzene was dried by distillation from sodium wire under nitrogen.

Benzoylmethylene(triphenyl)arsorane **24**[17] and methoxycarbonylmethylene(triphenyl)arsorane **32**[18] were prepared by the reported methods. Methyl [3-^{2}H]propiolate was prepared by dissolving sodium in D_2O and stirring a mixture of the resulting solution and methyl propiolate at room temperature for 12 h. The organic layer was separated, washed with D_2O and dried over calcium chloride.

Reaction of arsonium ylides with acetylenic esters

Methyl 5-oxo-5-phenyl-2-triphenylarsoranylidenepent-3-enoate 25. A solution of benzoylmethylene(triphenyl)arsorane **24** (1.02 g, 2.4 mmol) and methyl propiolate (0.22 g, 2.6 mmol) in dry benzene (20 cm^3) was heated at 50 °C under nitrogen for 1 h. The solution was evaporated and chromatography of the residue using ethyl acetate gave the product which was recrystallised from dichloromethane–diethyl ether (1:1) to afford the *title compound* (0.57 g, 45%) as yellow prisms, mp 184–185 °C (Found: C, 70.85; H, 4.9. $C_{30}H_{25}AsO_3$ requires C, 70.9; H, 5.0%) (HRMS: M$^+$, 508.1002. $C_{30}H_{25}AsO_3$ requires *M*, 508.1020); ν_{max}/cm^{-1} 1670; δ_H 8.1–7.1 (22 H, m) and 3.57 (3 H, s); δ_C 188.0 (*C*OPh), 167.7 (*C*O$_2$Me), 146.2 (As=C−*C*H), 132.6, 131.7, 129.3, 128.8, 105.8 (PhCO−*C*H) and 50.5 (OMe).

Methyl 5-oxo-5-phenyl-4-triphenylarsoranylidenepent-2-enoate 26. A solution of benzoylmethylene(triphenyl)arsorane **24** (1.02 g, 2.4 mmol) and methyl propiolate (0.22 g, 2.6 mmol) in dry methanol (20 cm^3) was heated under reflux under nitrogen for 3 h. The solution was evaporated and the residue triturated with diethyl ether to give the product which was recrystallised from chloroform–hexane (1:1) to afford the *title compound* (0.67 g, 55%) as pale yellow prisms, mp 181–182 °C (Found: C, 70.8; H, 5.2. $C_{30}H_{25}AsO_3$ requires C, 70.9; H, 5.0%); ν_{max}/cm^{-1} 1686 and 1586; δ_H 7.89 (1 H, d, *J* 15 As=C−C*H*), 7.7–7.3 (20 H, m), 4.42 (1 H, d, *J* 15, PhCO–C*H*) and 3.42 (3 H, s, OMe).

Ethyl 5-oxo-5-phenyl-2-triphenylarsoranylidenepent-3-enoate 27. A solution of benzoylmethylene(triphenyl)arsorane **24** (1.02 g, 2.4 mmol) and ethyl propiolate (0.25 g, 2.6 mmol) in dry benzene (20 cm^3) was heated at 50 °C under nitrogen for 1 h. The solution was evaporated and chromatography of the residue using ethyl acetate gave the product which was recrystallised from chloroform–hexane (1:1) to afford the *title compound* (0.63 g, 50%) as yellow prisms, mp 169–170 °C (Found: C, 71.1; H, 5.2. $C_{31}H_{27}AsO_3$ requires C, 71.3; H, 5.2%); ν_{max}/cm^{-1} 1660; δ_H 7.95–7.15 (22 H, m), 4.03 (2 H, q, *J* 7) and 0.92 (3 H, t, *J* 7).

Ethyl 5-oxo-5-phenyl-4-triphenylarsoranylidenepent-2-enoate 28. A solution of benzoylmethylene(triphenyl)arsorane **24** (1.02 g, 2.4 mmol) and ethyl propiolate (0.25 g, 2.6 mmol) in dry methanol (20 cm^3) was heated under reflux under nitrogen for 3 h. The solution was evaporated and the residue triturated with diethyl ether to give the product which was recrystallised from chloroform–diethyl ether (1:1) to afford the *title compound* (0.89 g, 78%) as pale yellow prisms, mp 168–169 °C (Found: C, 71.3; H, 5.1. $C_{31}H_{27}AsO_3$ requires C, 71.3; H, 5.2%); ν_{max}/cm^{-1} 1680 and 1570; δ_H 7.86 (1 H, d, *J* 14, As=C−C*H*), 7.7–7.2 (20 H, m), 4.48 (1 H, d, *J* 14, PhCO−C*H*), 3.89 (2 H, q, *J* 7) and 1.02 (3 H, t, *J* 7).

Dimethyl 4-triphenylarsoranylidenepent-2-enedioate 33. A solution of methoxycarbonylmethylene(triphenyl)arsorane **32** (0.40 g, 1.06 mmol) and methyl propiolate (0.10 g, 1.2 mmol) in dry benzene (10 cm^3) was heated at 50 °C under nitrogen for 5 h. The solution was evaporated and chromatography of the residue using dichloromethane gave the product which was recrystallised from hexane to afford the *title compound* (0.30 g, 61%) as yellow prisms, mp 152–153 °C (Found: C, 64.8; H, 5.15. $C_{25}H_{23}AsO_4$ requires C, 64.9; H, 5.0%) (HRMS: Found M$^+$, 462.0846. $C_{25}H_{23}AsO_4$ requires *M*, 462.0812); ν_{max}/cm^{-1} 1680 and 1665; δ_H 7.62 (15 H, s), 7.20 (1 H, d, *J* 14), 5.92 (1 H, d, *J* 14) and 3.55 (6 H, s).

Methyl 5-oxo-5-phenyl-4-triphenylphosphoranylidenepent-2-enoate 31

A solution of benzoylmethylene(triphenyl)phosphorane **29** (1.25 g, 3.3 mmol) and methyl propiolate (0.28 g, 3.3 mmol) in dry benzene (15 cm^3) was stirred at room temperature for 24 h. The solution was evaporated and the residue triturated with diethyl ether to give the product which was recrysallised from ethyl acetate to afford the *title compound* (1.07 g, 70%) as yellow needles, mp 214–215 °C (lit.,[4] 219–220 °C) (Found: C, 77.6; H, 5.4. $C_{30}H_{25}O_3P$ requires C, 77.6; H, 5.4%) (HRMS: Found M$^+$, 464.1534. $C_{30}H_{25}O_3P$ requires *M*, 464.1541); ν_{max}/cm^{-1} 1685 and 1580; δ_H 7.9–7.2 (21 H, m), 4.49 (1 H, d, *J* 16) and 3.62 (3 H, s) [lit.,[4] 7.81–7.35 (21 H, m), 4.47 (1 H, d, *J* 15) and 3.46 (3 H, s)].

Reaction as above but using methanol (15 cm^3) in place of benzene gave an identical product (58%).

Reaction of methyl 5-oxo-5-phenyl-2-triphenylarsoranylidenepent-3-enoate 25 with DMAD

A solution of methyl 5-oxo-5-phenyl-2-triphenylarsoranylidenepent-3-enoate **25** (0.32 g, 0.63 mmol) and dimethyl acetylenedicarboxylate (0.18 g, 1.27 mmol) in dry benzene (15 cm^3) was heated under reflux under nitrogen for 5 h. Evaporation followed by trituration of the residue with diethyl ether gave *dimethyl 2-oxo-3-triphenylarsoranylidenesuccinate* **40** (0.24 g, 82%) as a white solid which was recrystallised from ethyl acetate to give colourless crystals, mp 210–211 °C (lit.,[16] 214 °C); δ_H 7.7–7.1 (15 H, m), 3.84 (3 H, s) and 3.30 (3 H, s). Evaporation of the combined ether solutions from the trituration gave an oil which crystallised with time. Recrystallisation from diethyl ether gave *trimethyl biphenyl-2,4,5-tricarboxylate* **39** (0.17 g, 82%) as colourless crystals, mp 131–132 °C (Found: C, 65.9; H, 4.9. $C_{18}H_{16}O_6$ requires C, 65.8; H, 4.9%) (HRMS: Found M$^+$, 328.0940. $C_{18}H_{16}O_6$ requires *M*, 328.0947); ν_{max}/cm^{-1} 1748, 1735 and 1722; δ_H 8.21 (1 H, s), 7.67 (1 H, s), 7.5–7.3 (5 H, m), 3.93, 3.91 and 3.67 (each 3 H, s).

Reaction of methyl 3-deuterio-5-oxo-5-phenyl-2-triphenylarsoranylidenepent-3-enoate 41 with DMAD

Reaction as above but using methyl 3-deuterio-5-oxo-5-phenyl-2-triphenylarsoranylidenepent-3-enoate **41** (0.32 g, 0.63 mmol) {prepared by reaction of benzoylmethylene (triphenyl)arsorane **24** with methyl [3-^{2}H]propiolate} and dimethyl acetylenedicarboxylate (0.18 g, 1.27 mmol) in dry benzene (15 cm^3) gave *trimethyl 3-deuteriobiphenyl-2,4,5-tricarboxylate* **43** (0.18 g, 84%) as colourless crystals, mp 120–122 °C; δ_H 7.67 (1 H, s), 7.5–7.3 (5 H, m), 3.93, 3.91 and 3.67 (each 3 H, s).

X-ray structure determination of 25

A colourless lath suitable for X-ray diffraction was obtained by recrystallisation from dichloromethane–diethyl ether. The following crystal data were obtained.

$C_{30}H_{25}AsO_3$, $M = 508.42$, monoclinic space group $P2_1/n$; $a = 13.3576(13)$, $b = 14.127(2)$, $c = 13.8147(12)$ Å, $\beta = 107.887(8)°$, $V = 2480.9(5)$ Å^3, $Z = 4$, $D_c = 1.361$ g cm^{-3}, $R = 0.0556$, $wR_2 = 0.0788$ for 1404 data with $I > 2\sigma(I)$ and 308 parameters. Data were recorded at 293 K using Mo-Kα radiation and the structure was solved using DIRDIF-96[19] and refined using SHELXL-97.[20]

Atomic coordinates, molecular dimensions and anisotropic displacement parameters have been deposited in CIF format at the Cambridge Crystallographic Database (CCDC). For details of the deposition scheme, see 'Instructions for Authors', *J. Chem. Soc.*, *Perkin Trans 1*, available *via* the RSC Web page (http://www.rsc.org/authors). Any request to the CCDC for this material should quote the full literature citation and the reference number 207/204.

Acknowledgements

Two of us are grateful to our colleague (D. L.) for helpful discussions and encouragement over many years of our work which for him began with J. H. Read 50 years ago this year (see ref. 21).

References

1 H. J. Bestmann and O. Rothe, *Angew. Chem.*, *Int. Ed. Engl.*, 1964, **3**, 512.
2 J. B. Hendrickson, C. Hall, R. Rees and J. F. Templeton, *J. Org. Chem.*, 1965, **30**, 3312.
3 G. W. Brown, *J. Chem. Soc.* (*C*), 1967, 2018; R. A. Aitken, S. V. Raut and G. Ferguson, *Tetrahedron*, 1992, **48**, 8023.
4 W. Y. Ding, W. G. Cao, X. R. Xu, Y. Yao, Y. Shi and Z. Han, *J. Chem. Soc.*, *Perkin Trans. 1*, 1993, 855.
5 W. Y. Ding, W. G. Cao, Y. Yao and Z. M. Zhu, *Chin. J. Chem.*, 1995, **13**, 468.
6 W. G. Cao, W. Y. Ding, W. L. Ding and H. Huang, *J. Fluorine Chem.*, 1997, **83**, 21.
7 W. Y. Ding, J. Q. Pu, C. M. Zhang and W. G. Cao, *J. Chem. Soc.*, *Perkin Trans. 1*, 1991, 1369.
8 W. Y. Ding, P. S. Zhang, J. Q. Pu, C. M. Zhang, S. G. Mao, Q. W. Wang and W. Shen, *Acta Chim. Sin.*, 1991, **49**, 284.
9 E. Ciganek, *J. Org. Chem.*, 1970, **35**, 3631; H. Trabelsi, E. Bollens and A. Cambon, *Synthesis*, 1990, 623.
10 W. Y. Ding, P. S. Zhang and W. G. Cao, *Tetrahedron Lett.*, 1987, **28**, 81.
11 W. G. Cao, W. Y. Ding, T. Yi and Z. M. Zhu, *J. Fluorine Chem.*, 1997, **81**, 153.
12 D. Lloyd, I. Gosney and R. A. Ormiston, *Chem. Soc. Rev.*, 1987, **16**, 45.
13 D. Lloyd and I. Gosney, in *The Chemistry of Organic Arsenic*, *Antimony and Bismuth Compounds*, ed. S. Patai, Wiley, Chichester, 1994, p. 657.
14 S. Trippett and M. A. Walker, *J. Chem. Soc.* (*C*), 1971, 1114.
15 G. Ferguson, D. F. Rendle, D. Lloyd and M. I. C. Singer, *J. Chem. Soc.*, *Chem. Commun.*, 1971, 1647.
16 E. Ciganek, *J. Org. Chem.*, 1970, **35**, 1725.
17 A. W. Johnson and H. Schubert, *J. Org. Chem.*, 1970, **35**, 2678.
18 P. Frøyen, *Acta Chem. Scand.*, 1971, **25**, 2541.
19 P. T. Beurskens, G. Beurskens, R. de Gelder, S. Garcia-Granda, R. O. Gould, R. Israel and J. M. M. Smits, DIRDIF-96 program system, Crystallography Laboratory, University of Nijmegen, Netherlands.
20 G. M. Sheldrick, SHELXL-97, University of Göttingen, Germany.
21 J. H. Read and D. M. G. Lloyd, *Trans. Faraday Soc.*, 1948, **44**, 721.

Paper 8/01205I
Received 11*th February* 1998
Accepted 5*th March* 1998