Heteroatom Chemistry
Volume 10, Number 4, 1999

The Reaction of Wittig and Wittig–Horner Reagents with Dicyanomethylene Derivatives of Fluorenone, Xanthone, and Thiaxanthone. A Novel Synthesis of Phosphoranylidenecyclobutylidene Derivatives

Leila S. Boulos and Hoda A. Abd-El-Malek

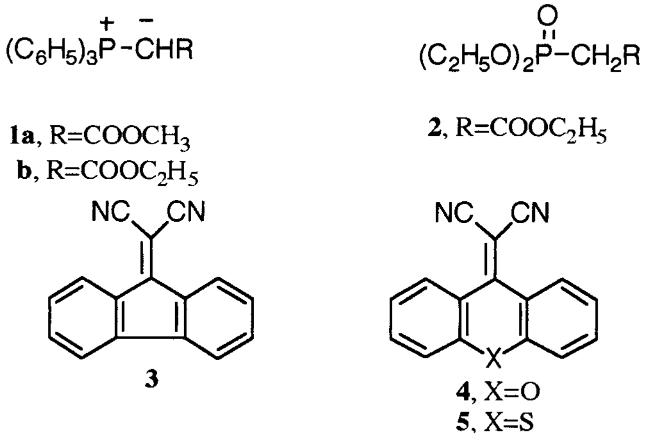
National Research Centre, Dokki, Cairo, Egypt

Received 5 August 1998; revised 27 November 1998

ABSTRACT: *Wittig reagents* **1a,b** *react with dicyanomethylene derivatives of fluorenone* (**3**) *and xanthone* (**4**) *to give the corresponding phosphoranylidenecyclobutylidene adducts* **6a**, **6b**, **9a**, *and* **9b**. *On the other hand, the reaction of Wittig–Horner reagents* (**2**) *with the same nitriles* **3** *and* **4** *afforded the respective phosphonate adduct* **8** *and the alkylated product* **10**. *A mechanism that accounts for the formation of the new products is presented.* © 1999 John Wiley & Sons, Inc. Heteroatom Chem 10: 263–269, 1999

INTRODUCTION

In recent years, considerable interest has been focused on the reaction of phosphorus ylides and phosphonate anions with α,β-unsaturated nitriles [1–5]. However, little information [4] is available about the reaction of these reagents with dicyanomethylene derivatives of fluorenone, xanthone, and thiaxanthone. In continuation of our work on the behavior of Wittig (**1**) and Wittig–Horner (**2**) reagents toward unsaturated centers [6–19], the action of the same reagents on fluorene (**3**)-, xanthene (**4**)-, and thiaxanthene (**5**)-9-ylidenemalononitrile has been investigated (Scheme 1).



SCHEME 1

RESULTS AND DISCUSSION

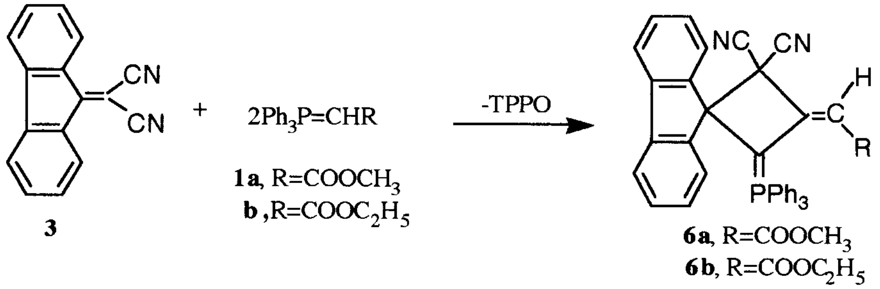
We have found that fluorene-9-ylidenemalononitrile (**3**) reacts with two mole equivalents of methoxycarbonylmethylene (**1a**)- and/or ethoxycarbonylmethylene (**1b**)-triphenylphosphoranes, in refluxing toluene for 25 hours, to give orange crystals formulated as **6a** and **6b**, respectively. Triphenylphosphine oxide was also isolated from both reaction media (Scheme 2).

Compounds **6a** and **6b** are chromatographically pure and possess sharp melting points. Elemental

---

Correspondence to: Leila S. Boulos

CCC 1042-7163/99/040263-07



SCHEME 2

and mass spectral analyses for compound **6a** corresponded to an empirical formula of $C_{39}H_{27}N_2O_2P$. The structure assigned for compound **6a** was based on the ^{31}P-NMR shifts (+18.8 ppm, vs. 85% H_3PO_4), which corresponds to an ylide-phosphorane structure [20]. The IR spectrum of **6a**, in KBr, revealed the presence of strong absorption bands at 1720 cm^{-1} (C=O, ester), 2198 (CN), 1680, 1510 cm^{-1} (C=P), and at 1430, 990 cm^{-1} (P-C, phenyl) [21]. The ^{1}H-NMR spectrum of **6a** in $CDCl_3$ indicated the presence of signals at $\delta = 3.75$ (s, 3H, COOCH_3) and at 6.8–8.0 (m, 23H) corresponding to the aromatic protons. Moreover, its PMR spectrum showed a doublet (1H) at $\delta = 6.17$ with a coupling constant $^4J_{HP} = 5$ Hz. This doublet was ascribed to the methine proton in **6a**. The chemical shift recorded for the methine proton suggested the *Z* form rather than the *E* one, which would require a downfield chemical shift [22]. Moreover, by use of the Alchemy III Computer Energy Minimization Program [23], the *trans* (*E*) isomer of compound **6a** was shown to have a higher energy than the *cis* (*Z*) isomer. So, compound **6a** most probably exists in the *Z* form. The ^{13}C-NMR spectrum (200 MHz) provided strong evidence in support of the ylide linkage incorporated in structure **6a**. The ^{13}C-NMR spectrum of **6a** in $CDCl_3$ shows doublets at $\delta = 140$ ($\rangle P{=}C\langle$, J = 122 Hz) [24,25]. Moreover, the ^{13}C-NMR spectrum of **6a** exhibited signals at 76 [$C(CN)_2$], 116.22 (CN), 141.2 (d, $C{=}CH$, $^2J_{cp} = 29.45$ Hz), 124.3, 126.5, 126.5, 119, 143.2, 143.7 (δc fluorene), 126.2, 131, 128.9, 132 (δc Ph), 23.7 (COOCH_3), and at 167 ppm (C=O, ester). The mass spectrum of **6a** contains a prominent peak for M$^+$ at m/e 586 (100%), which supports the phosphoranylidenecyclobutylidene structure **6a**.

Similarly, carbethoxymethylenetriphenylphosphorane **1b** reacts with fluorene-9-ylidene-malononitrile **3** (**1b** : **3** 2:1) to give adduct **6b** in 75% yield. Triphenylphosphine oxide was also isolated from the reaction mixture. Structure **6b** was deduced from correct microanalysis, IR, ^{1}H-NMR, ^{13}C-NMR, and mass spectral data.

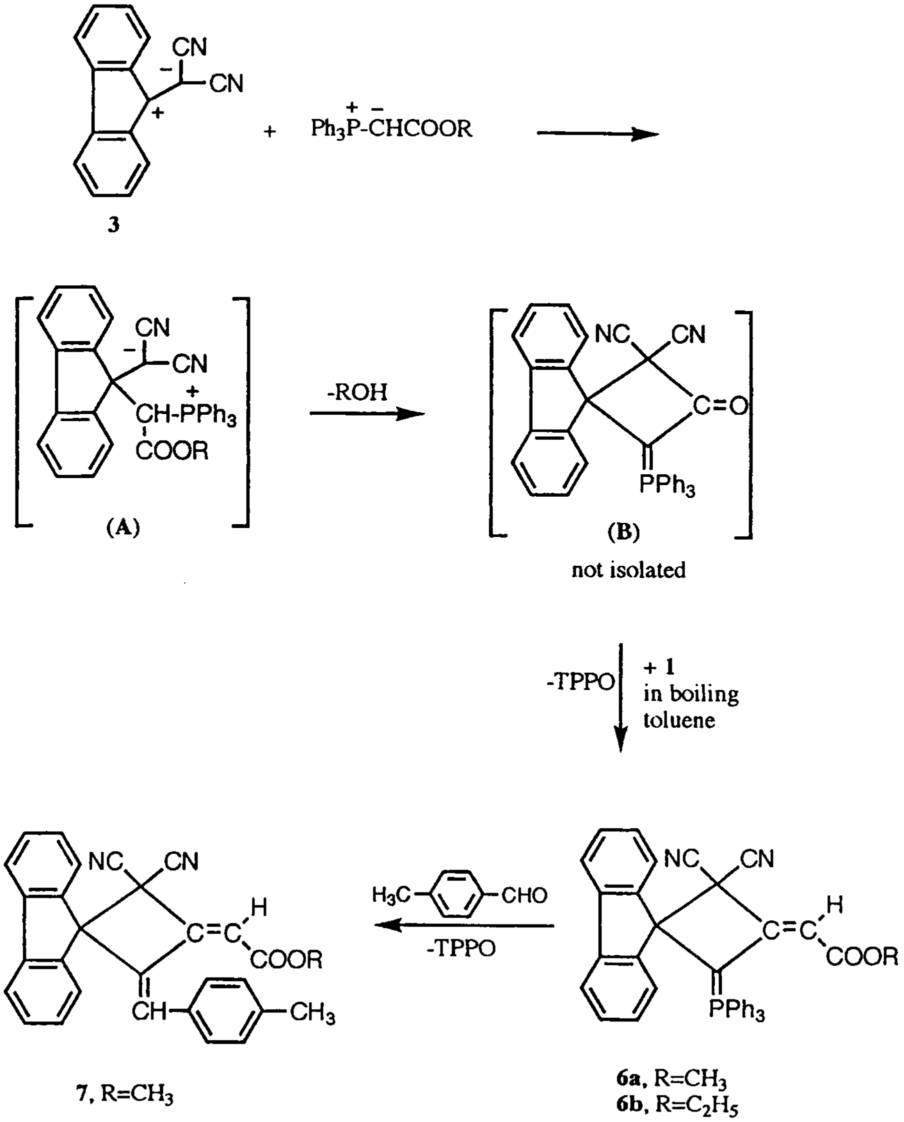
It is worth mentioning that, when fluorene-9-ylidenemalononitrile (**3**) was allowed to react with ylides **1a** and/or **1b** in boiling toluene for 25 hours, adducts **6a** and/or **6b** were obtained despite the fact that previous work reported [4] no reaction between the nitrile (**3**) and the same reagents in boiling tetrahydrofuran, toluene, or ethyl acetate. This is most probably due to the differences in the reaction times and temperatures.

Adducts **6a** and **6b** are obtained in similar yields, irrespective of whether 1 or 2 mole equivalents of the Wittig reagents **1a,b** were used with respect to 1 equivalent of fluorene-9-ylidenemalononitrile (**3**).

Treatment of the phosphoranylidenecyclobutylidene adduct **6a** with 4-methylbenzaldehyde in refluxing toluene for 48 hours afforded the p-methylbenzylidene derivative **7a** and triphenylphosphine oxide (Scheme 3). The structure of compound **7a** was verified through elemental analysis and spectroscopic results.

A possible explanation of the course of the reaction of phosphonium ylides **1** with the nitrile **3** is shown in Scheme 3. Adducts **6a** and **6b** can be obtained via 1:1 addition of the ylides **1a** or **1b** on the more electrophilic and less sterically hindered methide carbon to give betaine (**A**). It may be considered that the phosphoranylidenecyclobutane intermediates (**B**) are formed via the expulsion of a suitable moiety (i.e., RH, R=OCH_3, or OC_2H_5) followed by addition of a second molecule of ylide **1** to yield the phosphoranylidenecyclobutylidene adducts **6a** and **6b** along with triphenylphosphine oxide [26].

The reaction of fluorene-9-ylidenemalononitrile (**3**) with triethylphosphonoacetate **2** was also investigated. We have found that the reaction of nitrile (**3**) with 2 mole equivalents of triethylphosphonoacetate (**2**), in the presence of alcoholic sodium ethoxide solution, proceeds at reflux temperature for 5 hours to give a pure adduct formulated as **8** (Scheme 4). Structure elucidation of adduct **8** was based on the following evidence: (a) Correct elemental analyses and a molecular weight determination by mass spec-

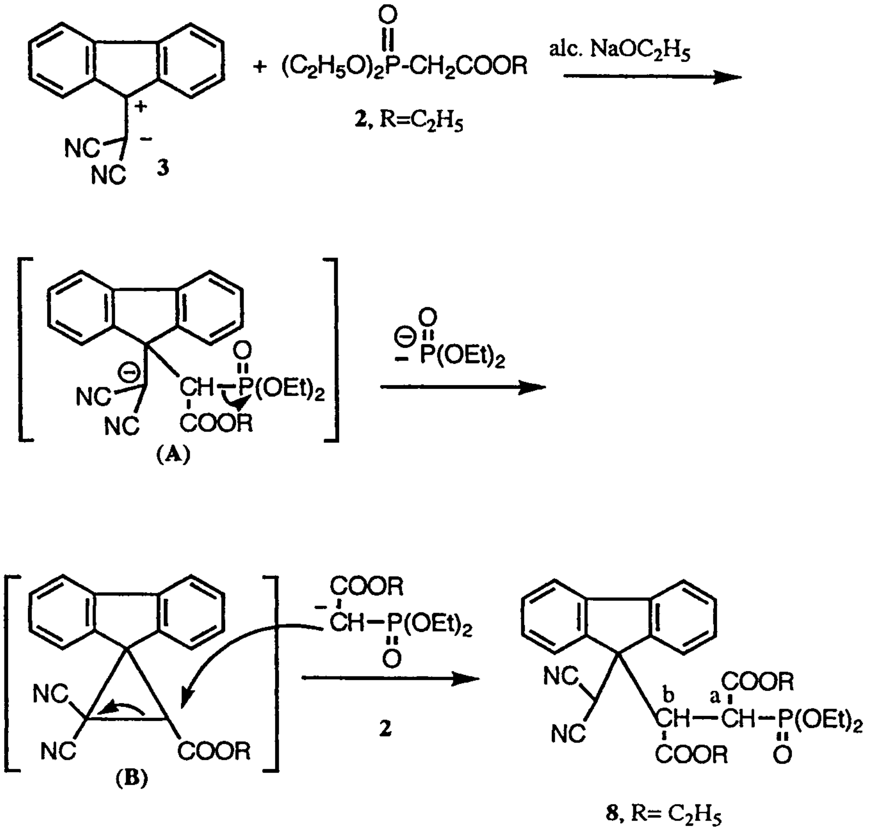


SCHEME 3

troscopy (MS) were obtained, and (b) its IR spectrum, in KBr, exhibits strong absorption bands at 2215 cm^{-1} (CN), 1725, 1730 (C=O, ester), 1248 cm^{-1} (P=O, bonded), and at 1098 cm^{-1} (P-O-C_2H_5). Adduct 8 possesses the phosphonate structure because it exhibits a positive shift in its ^{31}P-NMR spectrum (δ = + 19.7 vs. 85% H_3PO_4) and absorbs in the region characteristic for this class of compounds. The ^{1}H-NMR spectrum of compound 8 revealed the presence of multiplets centered at δ = 0.85 (6H, CH_3, m) and at 4.2 (4H, CH_2, m), corresponding to the two carbethoxy groups. The two ethoxy groups attached to the phosphorus atom appeared as a triplet centered at δ = 1.25 (6H, CH_3, t) and a quintet centered at δ = 4.25 (4H, CH_2, q). Moreover, the ^{1}H-NMR spectrum of compound 8 showed signals centered at 2.9 (dd with J_{HP} = 12 Hz, J_{HH} = 7.5 Hz) and 3.9 ppm (dd, with J_{HP} = 10 Hz, J_{HH} = 7.5 Hz) corresponding to the two methine protons *a* and *b*, respectively [27] (Scheme 4). The vicinal coupling value ($^3J_{HH}$ = 7.5 Hz) for the two methine protons *a* and *b* indicates a free type of rotation (6–8 Hz) [27]. Also, its PMR spectrum showed a singlet (1H) at δ = 5.0, which was ascribed to the methine proton. Meanwhile, the aromatic protons (8H) appeared as a multiplet in the δ 7.33–8.17 region.

The mass spectrum of compound 8 showed the molecular ion peak at m/z 538. The mechanism proposed to account for the formation of the phosphonate adduct 8 is shown in Scheme 4.

Fluorene-9-ylidenemalononitrile (3) reacts with 1 mole of alkylphosphonoacetate 2 to give the re-



SCHEME 4

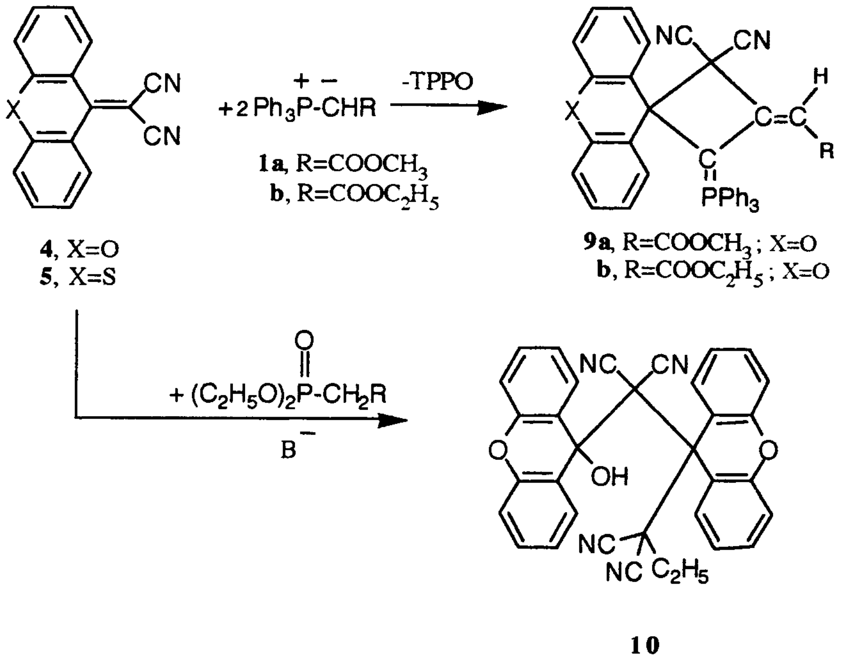
spective intermediate (**A**). Under the influence of the base present in the reaction medium, phosphonate (**A**) is converted to the expected cyclopropane intermediate (**B**) that could then be attacked by another molecule of the phosphonate anion **2**, to give, after a suitable proton transfer, the final product **8** as depicted in Scheme 4. The dialkyl phosphite was detected in the water layer by the development of a violet color on addition of 3,5-dinitrobenzoic acid [28]. Such an observation has been made for the reaction of 1,3-dioxo-Δ^2,α-indanmalononitrile with Wittig–Horner reagents [5].

Although fluorene-9-ylidenemalononitrile **3** reacts with Wittig reagents **1a,b** to give the corresponding phosphoranylidenecyclobutylidene adducts **6a** and **6b**, a different behavior is observed in the reaction of the Wittig–Horner reagents **2** with the same nitrile **3**.

Since compounds **4** and **5** have a structure analogous to that of fluorene-9-ylidene-malononitrile **3**, it was of interest to examine their behavior toward the same reagents to establish the preferential site of attack by these reagents and for the production of new phosphonium ylides.

The reaction of xanthene-9-ylidenemalononitrile (**4**) with ylides **1a,b** was preformed in refluxing toluene in 1:2 molar ratio to give adducts **9a** and **9b**, respectively. Triphenylphosphine oxide was also isolated from both reaction mixtures (Scheme 5). Adducts **9a** and **9b** were assigned analogous structures on the basis of spectroscopic arguments. Structure **9** is confirmed from microanalysis, IR, ^{1}H, ^{31}P, ^{13}C-NMR, and mass spectra.

The reaction of xanthene-9-ylidenemalononitrile (**3**) with triethyl-phosphonoacetate (**2**) was also investigated. When **3** was allowed to react with 1 equivalent of **2** in the presence of alcoholic sodium ethoxide solution at reflux temperature for 5 hours, product **10** was isolated in 75% yield. Structure elucidation of compound **10** was based on the following evidence: (a) Correct elemental analyses and a molecular-weight determination by mass spectroscopy (MS) were obtained, and (b) its IR spectrum in KBr exhibits strong absorption bands at 3500 cm^{-1} (OH) and 2200 cm^{-1} (CN). The IR spectrum of compound **10** revealed the absence of any C$=$O absorption bands in the region 1690–1750 cm^{-1}. The ^{1}H-NMR spectrum of **10** disclosed the absence of any signal at δ = 4–6 corresponding to any methine protons. Moreover, the ^{1}H-NMR spectrum of compound **10**



SCHEME 5

showed signals centered at δ = 1.20 (triplet, 3H, CH_3) and 3.45 (qu, 2H, CH_2) corresponding to the ethyl group attached to $C(CN)_2$. The exchangeable (D_2O) proton (OH) appears as a singlet at δ 7.9. The spectrum also revealed a complex pattern due to the aromatic protons (16H, m) in the region δ = 7.20–8.90. The mass spectrum of compound **10** showed the molecular ion peak at m/z 534.

The alkylated dimeric compound **10** presumably was formed via addition of 2 moles of the xanthene-9-yildenemalononitrile followed by alkylation. A similar alkylation was also observed in the reactions of Wittig–Horner reagents with pyrroles [29].

No reaction was observed when thiaxanthene-9-ylidenemalononitrile (**5**) was allowed to react with ylides of types **1a** and **1b** in various solvents or when **5** was allowed to react with Wittig–Horner reagent **2** for several hours.

CONCLUSIONS

In view of all the facts previously cited, it can be concluded that the reaction of fluorene-9-ylidenemalononitrile (**3**) with Wittig (**1**) and Wittig-Horner reagents (**2**) leads to different products, depending on the nature of the phosphorus reagent used as well as on the stability of the addition products [30]. Although xanthene-9-ylidenemalononitrile (**4**) reacts with Wittig reagents to give the cyclobutane adducts **9a** and **9b**, a different behavior is observed in the reaction of Wittig–Horner reagent with the same nitrile **4**.

The significance of these findings is not only the discovery of a new pattern of the Wittig and Wittig–Horner reaction but also the establishment of a novel method for the synthesis of both the phosphoranylidene-cyclobutylidene and the phosphonate adducts.

EXPERIMENTAL

All melting points are uncorrected. Carbomethoxymethylene and carbethoxymethylenetriphenylphosphoranes (**1a**,**b**) were prepared according to established procedures [31]. Triethyl-phosphonoacetate (**2**) was prepared by means of the Michaelis–Arbuzov reaction [32]. The IR spectra were measured in KBr pellets on a Perkin–Elmer Infracord Spectrophotometer Model 157 (Grating). The ^{1}H-NMR spectra were recorded in $CDCl_3$ on a JNM-GX-400Fa Spectrometer (Tokyo). The ^{31}P-NMR spectra were measured in $CDCl_3$ (vs. H_3PO_4 as external standard) on a JNM-PS-100 Fa Spectrometer. ^{13}C-NMR data were taken in $CDCl_3$ on a Varian Spectrometer 200 MHz. The mass spectra were run at 70 eV on a Kratos MS instrument and a Varian Mat 311 A Spectrometer.

Reaction of Carbomethoxymethylenetriphenylphosphorane **1a** *with Fluorene-9-ylidene-malononitrile* **3**

A mixture of the nitrile (**3**) (0.22 g, 0.001 mole) and ylide **1a** (0.68 g, 0.002 mole) in dry toluene (30 mL) was refluxed for 25 hours. The volatile materials were evaporated under reduced pressure, and the residual substance was chromatographed on a silica-gel column using acetone/petroleum ether (40:60 v:v) as eluent to give the phosphoranylidenecyclobutylidene adduct (**6a**) as orange crystals, m.p. 224–226°C (yield 67%). Anal. calcd for $C_{39}H_{27}N_2O_2P$ (586.63): C, 79.85; H, 4.63; N, 4.77; P, 5.27. Found: C, 79.78; H, 4.66; N, 4.65; P, 5.30. IR (KBr): 1720, 2198, 1680, 1510, 1430, 990 cm^{-1}. ^{1}H-NMR ($CDCl_3$): δ 3.75 (s, 3H, CH_3), 6.17 (d, 1H, $^4J_{HP} = 5$ Hz), 6.8–8.0 (m, 23H, Ar.). ^{31}P-NMR: $\delta = +18.8$. ^{13}C-NMR ($CDCl_3$): $\delta = 140$ (d, P=C, $J = 122$ Hz), 116.22, 116.02 (2CN), 141.2 (d, C=CH, $^2J_{cp} = 24.45$), 167 (C=O), 23.7 (COO*C*H3). MS: m/z(%) 586 (100) [M^+].

Similarly, the reaction of the nitrile **3** (0.001 mole) with carbethoxymethylenetriphenylphosphorane (**1b**) (0.66 g, 0.002 mole) was performed in refluxing toluene for 25 hours to give **6b** as orange crystals, m.p. 257–258°C (yield 65%). Anal. calcd for $C_{40}H_{29}N_2O_2P$ (600.66): C, 79.98; H, 4.86; N, 4.66; P, 5.15. Found: C, 79.80; H, 4.91; N, 4.57; P, 5.21. IR (KBr): 1718, 2190, 1680, 1515, 1430, 992 cm^{-1}. ^{1}H-NMR ($CDCl_3$): δ 1.05 (t, 3H, $J_{HH} = 6$ Hz, CH_3,), 4.15 (q, 2H, $J_{HH} = 6$ Hz, CH_2), 6.1 (d, 1H, $^4J_{HP} = 5$ Hz, =*C*H), 6.85–7.95 (m, 23H, Ar.). ^{31}P-NMR: $\delta = +18.5$. ^{13}C-NMR ($CDCl_3$): δ 143 (d, $J_{CP} = 126$ Hz), 166.96 (C=O), 13.89 (CH_3), 60 (CH_2), 77 [$C(CN)_2$], 116.65 (CN), 141.7 (d, C=CH), 124, 126, 127, 119, 142, 143 (°c fluorene), 126, 131, 128, 132 (δc Ph). MS: m/z(%) 600 (95) [M^+].

Action of Tolualdehyde on **6a**

To a solution of ylide **6a** (0.58 g, 0.001 mol) in toluene (17 cm^3), p-methylbenzaldehyde (0.12 g, 0.001 mole) was added, and the reaction mixture was heated under reflux for 48 hours. After the evaporation of the solvent, the residue was triturated with dichloromethane to yield adduct **7** as yellow crystals (from dichloromethane), m.p. 180–181°C. Anal. calcd for $C_{29}H_{20}N_2O_2$ (428.49): C, 81.29; H, 4.70; N, 6.53. Found: C, 81.26; H, 4.67; N, 6.50. IR (KBr): 1720, 2198, 1600 cm^{-1}. ^{1}H-NMR ($CDCl_3$): δ 6.17 (s, 1H), 6.8 (s, 1H), 2.3 (s, 3H, CH_3), 3.75 (s, 3H, COOC*H*$_3$), and 6.88–7.95 (m, 12H). MS: m/z (%) 428 (95) [M^+].

Reaction of Triethylphosphonoacetate **2** *with Fluorene-9-ylidene-malononitrile* **3**

A solution of 2 moles of sodium ethoxide in absolute ethanol was treated with an equimolar amount of triethylphosphonoacetate (**2**), and then the dicyanocompound (**3**) (1 mole) was added. The resulting reaction mixture was allowed to reflux for 5 hours. Then the reaction mixture was poured into a small amount of water, extracted with diethyl ether, and the extract was dried over anhydrous sodium sulfate and evaporated under reduced pressure. The residual material was placed on a column of silica gel and eluted with ethyl acetate/*n*-hexane (40:60, v:v) to give the new product **8** in 70% yield as colorless crystals, m.p. 235–236°C. Anal. calcd for $C_{28}H_{31}N_2O_7P$ (538.25): C, 62.42; H, 5.80; N, 5.20; P, 5.75. Found: C, 62.40; H, 5.86; N, 5.25, P, 5.78. IR (KBr): 2215, 1725, 1730, 1248, 1098 cm^{-1}. ^{31}P-NMR: $\delta = +19.7$. ^{1}H-NMR ($CDCl_3$): $\delta = 0.85$ (m, 6H, CH_3), 4.2 (m, 4H, CH_2), 1.25 (t, 6H, $2CH_3$), 4.25 (q, 4H, $2CH_2$), 2.9 (dd, $J_{HP} = 12$ Hz, $J_{HH} = 7.5$ Hz, CHa), 3.9 (dd, $J_{HP} = 10$ Hz, $J_{HH} = 7.5$ Hz, CHb), 7.33–8.17 (m, 8H, Ar.). MS: m/z (%) 538 (95) [M^+].

Reaction of Carbomethoxymethylenetriphenylphosphorane **1a** *with Xanthene-9-ylidene-malononitrile* **4**

A mixture of the nitrile (**4**) (0.24 g, 0.001 mol) and ylide **1a** (0.68 g, 0.002 mol) in dry toluene (30 mL) was refluxed for 30 hours. The volatile materials were evaporated under reduced pressure, and the residual substance was chromatographed on a silica-gel column using acetone–petroleum ether (40:60, v:v) as eluent to give the phosphoranylidenecyclobutylidene adduct **9a** as orange crystals, m.p. 209–210°C (yield 65%). Triphenylphosphine oxide was also isolated and identified. Anal. calcd for $C_{39}H_{27}N_2O_3P$ (602.63): C, 77.73; H, 4.51; N, 4.65; P, 5.14. Found: C, 77.68; H, 4.53; N, 4.61; P, 5.16%. IR (KBr): 1720 (C=O, ester), 2195 (CN), 1685, 1520 (C=P), 1435, 990 (P-C-phenyl) cm^{-1}. ^{1}H-NMR ($CDCl_3$): δ 3.66 (s, 3H, $COOCH_3$), 5.6 (d, 1H, $CHCOOCH_3$, $^4J_{HP} = 5$ Hz), 6.9–7.9 (m, 23H, Ar.). ^{31}P-NMR: + 18.82. MS: m/z (%): 602 (30) [M^+], 530 (85) [M-$CHCOOCH_3$].

Similarly, the reaction of the nitrile (**4**) with ylide **1b** was performed in boiling toluene for 30 hours in 1:2 molar ratio to give **9b** as orange crystals, m.p. 254–255°C (yield 60%). Anal. calcd for $C_{40}H_{29}N_2O_3P$: C, 77.91; H, 4.76; N, 4.54; P, 5.02. Found: C, 77.96; H, 4.67; N, 4.6; P, 5.10. IR: 1720, 2200, 1680, 1520, 1430, 990 cm^{-1}. ^{1}H-NMR ($CDCl_3$): $\delta = 1.0$ (t, 3H, $COOCH_2CH_3$), 4.2 (q, 2H, COOC*H*$_2$CH$_3$), 5.6 (d, 1H, CH-$COOC_2H_5$, $^4J_{HP} = 5.2$ Hz), 6.8–7.9 (m, 23H). ^{31}P-NMR: + 18.7 ppm. MS: m/z (%) 616 (25) [M^+], 530 (95) [M-86].

Reaction of Triethylphosphonoacetate **2** *with Xanthene-9-ylidene-malononitrile* **4**

A solution of 1 mole of sodium ethoxide in absolute ethanol was treated with an equimolar amount of triethylphosphonoacetate (**2**), and then the dicyano compound **4** (1 mole) was added. The resulting reaction mixture was allowed to reflux for 5 hours. Then the reaction mixture was poured into a small amount of water, extracted with diethyl ether, and the extract was dried over anhydrous sodium sulfate and evaporated in a vacuum. The residual material was placed on a column of silica gel and eluted with ethyl acetate/*n*-hexane (40:60, v:v) to give adduct **10** as colorless crystals, m.p. 210–211°C (yield 75%). Anal. calcd for $C_{34}H_{22}N_4O_3$ (534.57): C, 76.39; H, 4.15; N, 10.48. Found: C, 76.40; H, 4.20; N, 10.50. IR (KBr): 3500, 2200 cm^{-1}. ^{1}H-NMR ($CDCl_3$): $\delta = 1.20$ (t, 3H, CH_3), 3.45 (q, 2H, CH_2), 7.9 (s, 1H, OH), 7.20–8.90 (m, 16H, Ar.). MS: *m/z* (%) 534 (90) [M^+].

REFERENCES

[1] Soliman, F. M.; Said, M. M. Phosphorus Sulfur Silicon 1991, 61, 335.
[2] Mahran, M. R.; Abdou, W. M.; Abd-El-Rahman, N. M.; Khidre, M. D. Heteroatom Chemistry 1992, 3, 93.
[3] Abdou, W. M.; Ganoub, N. A. F. Heteroatom Chemistry 1992, 3, 133.
[4] Ganoub, N. A. F. Phosphorus Sulfur Silicon 1993, 81, 125.
[5] Boulos, L. S.; Yakout El-Sayed, M. A.; Heteroatom Chemistry 1997, 8, 253.
[6] Boulos, L. S.; Arsanious, M. H. N. Phosphorus Sulfur Silicon 1988, 36, 23.
[7] Boulos, L. S.; Arsanious, M. H. N. Phosphorus Sulfur Silicon 1989, 42, 47.
[8] Boulos, L. S.; El-Khoshnieh, Y. O.; Arsanious, M. H. N. Phosphorus Sulfur Silicon 1990, 47, 199.
[9] Boulos, L. S.; Shabana, R. Phosphorus Sulfur Silicon 1990, 53, 81.
[10] Boulos, L. S.; Hanna, G., Atef, ; Hennawy, I. T.; Arsanious, M. H. N. Phosphorus Sulfur Silicon 1991, 57, 181.
[11] Boulos, L. S.; Hennawy, I. T. Phosphorus Sulfur Silicon 1991, 56, 65.
[12] Boulos, L. S.; Khir El Din, N.; Zayed, M. F. Phosphorus Sulfur Silicon 1991, 61, 131.
[13] Boulos, L. S.; Zayed, M. F.; El-Khoshnieh, Y. O. Phosphorus Sulfur Silicon 1991, 62, 231.
[14] Boulos, L. S.; Khir El Din, N. Tetrahedron 1993, 49, 3871.
[15] Boulos, L. S.; Abd El-Rahman, N. M. Phosphorus Sulfur Silicon 1991, 62, 163.
[16] Boulos, L. S.; Abd El-Rahman, N. M. Phosphorus Sulfur Silicon 1992, 68, 241.
[17] Boulos, L. S.; Hennawy, I. T.; Arsanious, M. H. N. Heteroatom Chemistry 1994, 5, 447.
[18] Boulos, L. S.; Arsanious, M. H. N. Phosphorus Sulfur Silicon 1994, 89, 185.
[19] Boulos, L. S.; Arsanious, M. H. N. Tetrahedron 1997, 53, 3649.
[20] Crutchfield, M. M.; Dungan, C. H.; Letcher, J. H.; Mark, V.; van Wazer, J. R. ^{31}P-Nuclear Magnetic Resonance; Wiley–Interscience: New York, 1967.
[21] Hesse, M.; Meier, H.; Zech, B. Spektroskopische Methoden in der Organischen Chemie; G. Thieme Verlag: Stuttgart, 1979.
[22] Bestmann, H. J.; Lang, H. J. Tetrahedron Lett 1969, 25, 2102.
[23] 3D-Molecular Modeling Software Alchemy III, Tripos Associate, Inc., a subdivision of Evans Suthereland, 1992.
[24] Levy, G. C.; Lichter, R. L.; Nelson, G. I. Eds. Carbon 13 Nuclear Magnetic Resonance Spectroscopy; Wiley: New York, 1980; Chapter 5, p 135.
[25] Kalinowski, H. O.; Berger, S.; Braun, S. ^{13}C-NMR Spectroskopie; G. Thieme Verlag: Stuttgart, 1984.
[26] Nicolaides, D. N.; Adamopoulos, S. G.; Lefkaditis, D. A. J Chem Soc Perkin Trans 1 1992, 283.
[27] Silverstein, R. N .; Bassler, G. C. Spectroscopic Identification of Organic Compounds; 2nd ed.; Wiley: New York, 1967; p 145.
[28] Saunders, B. C.; Stark, B. P. Tetrahedron 1964, 4, 187.
[29] Mercey, M.; Toube, P. J Chem Res Synop 1987, 62, 13 Chem. Abst. 1988, 107, 236149x.
[30] Boulos, L. S.; Hennawy, I. T. Phosphorus Sulfur Silicon 1993, 84, 173.
[31] Bestmann, H. J.; Kratzer, O. K. Chem Ber 1962, 95, 1894.
[32] Kosalapoff, G. M. Organophosphorus Compounds; Wiley: New York, 1950; Chapter 7.