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2,3,4-TRIPHENYLCYCLOPENTADIENYLIDES

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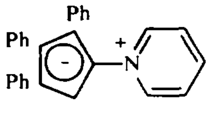
Abstract—The properties of pyridinium, triphenylphosphonium and triphenylarsonium 2,3,4-triphenylcyclopentadienylides are described and discussed, including their UV spectra, basicities and reactivities towards aldehydes and nitrosobenzene. The phosphonium and arsonium ylides are less basic than the corresponding tetraphenylcyclopentadienylides, yet more reactive towards benzaldehyde. The ylides undergo protonation and acylation at the 5-position and their salts condense at this position with *p*-dimethylaminobenzaldehyde.

IN THE previous paper[1] the properties of a number of heteronium tetraphenylcyclopentadienylides were discussed. The present paper deals with the 2,3,4-triphenylcyclopentadienylides (I, II and III). Their preparation has been described in earlier papers.[2, 3]



I

A B

II: X = P; III: X = As

The phosphonium and arsonium ylides (II and III) are stable indefinitely in air, owing their unusual stability for ylides to the stabilization of the dipolar canonical form B by its having a cyclic delocalized sextet of π-electrons. Both are yellow and have UV spectra almost identical with their tetraphenylcyclopentadienylide analogues.

Both are unaffected by alkali and were recovered quantitatively after being heated under reflux in ethanolic sodium hydroxide.

They are soluble in dilute mineral acids and with perchloric acid form yellow crystalline perchlorates. The NMR spectra of the protonated species show clearly that protonation takes place at the unsubstituted 5-position [τ (CF_3CO_2H): (II, perchlorate) 2·0–3·2 m, 5·88s (30:2); (III, perchlorate 2·2–3·3 m, 5·64s (30;2)]. The pK_a values for the resultant triphenyl(triphenylcyclopentadienyl)-phosphonium and -arsonium cations are respectively 4·2 and 5·6. These values are each approximately one pK unit lower than the values for the corresponding tetraphenylcyclopentadienylide salts. This in turn implies that the 2,3,4-triphenylcyclopentadienylides are more thermodynamically stable than the tetraphenylcyclopentadienylides. This extra stabilization may result from steric factors in that there is less crowding in the triphenyl derivatives. It has been shown that fulvalenes with only one substitutent Ph group in each ring in positions next to the interannular bond were sufficiently

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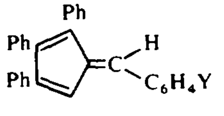
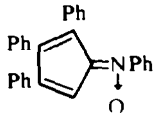
stable to be isolated, whereas fulvalenes having two phenyl groups in each ring adjacent to the interannular bond could not be obtained.[4]

The ylides (II and III) resembled their tetraphenylcyclopentadienylide analogues in that the phosphonium ylide was apparently unreactive towards carbonyl compounds whereas the arsonium ylide reacted with either benzaldehyde or p-nitrobenzaldehyde to give fulvenes (IV).



IV; Y = H or p-NO_2

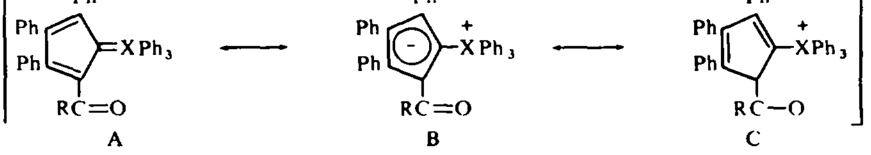


Although the ylide (III) was less basic than its tetraphenylcyclopentadienylide analogue it was somewhat more reactive towards benzaldehyde. This contradiction between the relative basicities and relative reactivities as nucleophiles may be due to lesser steric hindrance to attack by the aldehyde in the case of ylide (III) facilitating reaction in this case. A fulvene is again formed rather than a fulvene oxide from this stabilized arsonium ylide (*cf* ref. 1). Both fulvenes were identical with samples prepared by condensation of 1,2,3-triphenylcyclopentadiene with the appropriate aldehyde; the fulvene (IV, Y = p-NO_2) had not been reported previously.

The arsonium ylide (III) but not the phosphonium ylide (II) reacted with nitrosobenzene to give the anil oxide (V). Whereas in this case only the anil oxide was formed and apparently none of the anil, in the case of the corresponding arsonium tetraphenylcyclopentadienylide both an anil and an anil oxide were produced. Formation of an anil may be more inhibited in the case of the triphenylcyclopentadiene because of destabilization of this product due to conjugation (*cf* ref. 1), since the presence of one less Ph substituent increases the possibility of coplanarity of the molecule and consequent conjugative interaction. The anil oxide was identical with the product obtained by a method used previously to obtain anil oxides,[1,5] namely the reaction of nitrosobenzene with diazo-2,3,4-triphenylcyclopentadiene.

It seemed possible that the anil might in fact have been formed but dimerized before it was isolated since derivatives of 2,3,4-triphenylcyclopentadienone are prone to dimerization. Reduction of the anil oxide (V) with triphenylphosphine readily gave the anil and it proved to be a stable highly crystalline monomer which showed no tendency to dimerize, and which would certainly have been detected and isolated had it accompanied the anil oxide in the reaction mixture obtained from III. This anil was also prepared from 1,2,3-triphenylcyclopentadiene and nitrosobenzene.

Since the 5-membered ring in the ylides (II and III) must have some cyclopentadienide character resulting from the contribution of canonical forms **B** to their overall structures, they would be expected to undergo electrophilic substitution readily at



VI

their vacant 5-positions, especially since this is the favoured site for substitution (*cf* ref. [6–8]). Both ylides were rapidly acetylated when heated in refluxing acetic anydride to give the products (VI, R = Me). The acetylylide (VI, R = Me, X = As) had been obtained previously from the acetic anhydride catalysed condensation reaction between triphenylarsine oxide and triphenylcyclopentadiene.[2,3] No. carbonyl absorption is evident in the IR spectrum of either ylide at > 1600 cm^{-1} and the carbonyl peaks for (VI, R = Me, X = As/P) appeared, respectively, at 1565, 1575 cm^{-1}. This was attributed to the polarity of the CO group due to the contribution from canonical form VIC. The polarity of this group is probably enhanced by interaction with the adjacent heteronium group. The UV and NMR spectra of the two ylides (VI, R = Me, X = P and As) were almost identical.

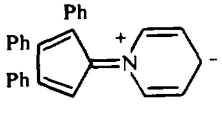
Because of the delocalization of the negative charge onto the CO group as well as over the 5-membered ring the reactivity of these acetylylides to electrophilic attack is diminished and they did not react even with reactive aldehydes such as 2,6-dinitrobenzaldehyde. Both ylides readily formed crystalline perchlorates.

Ylides II and III were formylated by the Vilsmeier method to give 5-formylylides (VI, R = H, X = P or As). Both of these products formed crystalline perchlorates. Although the elemental analyses both of the formylylides and their perchlorates were consistent with the assigned structures their NMR spectra in either deuteriochloroform or trifluoroacetic acid showed no signals in the region where aldehydic protons normally appear. This is in contrast to the pyridinium analogue, described later, and also to formylated derivatives of triphenylphosphonium cyclopentadienylide, which absorb between τ $-0{\cdot}16$ and $+0{\cdot}35$.[7,8] The aldehydic signals in VI (R = H) must presumably be hidden by the Ph signals, primarily due to shielding by adjacent Ph groups, either on the ring or on the heteronium atom, which are forced out of coplanarity with the 5-membered ring in the present examples. The signals due to the Me groups in VI (R = Me) also appear at higher field (τ *ca* 8·2) than in the case of triphenylphosphonium 2-acetylcyclopentadienylide (τ = 7·83).[8] There may also be more contribution from canonical form C in the case of ylides VI than in the case of the pyridinium analogue and triphenylphosphonium 2-formylcyclopentadienylide[7,8] since the latter ylides do show CO absorption at > 1600 cm^{-1}.

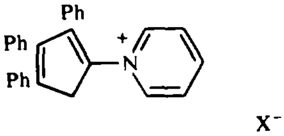
Pyridinium triphenylcyclopentadienylide (I) is a purple solid. Although stable in the solid state, in solution it rapidly decomposes. Its lower stability *vis-a-vis* the phosphonium and arsonium ylides (II and III) presumably reflects the fact that in this case the ylide is not a hydrid of 'dipolar' and 'covalent' forms such as IIA and IIB. Forms such as VII, wherein no charge resides on the 5 membered ring may contribute to the overall structure but their contribution is likely to be small and to contribute little to the stabilization of the ylide. Because of its instability in solution it proved impossible to purify I by recrystallization or chromatography.

The UV spectrum of I closely resembles that of its tetraphenyl analogue[1] and is completely different from the spectra of II and III. The colour of its solutions varied markedly with the nature of the solvent, from orange in methanol to blue in benzene or ether, this difference being due to a bathochromic shift of 52 nm in the longest wavelength absorption band [λ_{max} (EtOH) 255, 311, 530 nm (log ε = 3·34, 3·40, 3·10); λ_{max} (benzene) 292, 320, 582 nm (log ε = 3·50, 3·35, 3·41)]. Similar solvatochromism in the case of pyridinium cyclopentadienylide[9,10] has been correlated with an intramolecular charge-transfer transition.[10] The phosphonium and arsonium ylides (II and III) are not solvatochromic; comparable differences in tetraphenylcyclopentadienylides are discussed in the previous paper.[1]

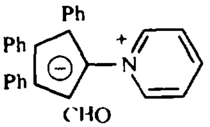
The ylide I dissolved in dilute mineral acids to give yellow solutions; NMR spectra of the protonated species indicated that salts VIII were formed.



VII



VIII



IX

Because of the instability of the ylide I in solution, its formyl derivative IX was prepared from the salt VIII ($X = ClO_4$) by a modified Vilsmeier technique,[11] rather than by direct formylation of the ylide itself. The formylated ylide was a bright orange-red compound which was stable in solution and could be purified by recrystallization or chromatography. This extra stabilization results from the electron-withdrawing effect of the formyl group and consequent further delocalization of the negative charge onto this group. This is shown by the IR spectrum for the CO absorption appears at 1615 cm^{-1}. That this value is not so low as in the case of ylides II and III may be because interaction between the CO group and the heteronium atom is less effective in this case. There was a bathochromic shift of 80 nm in the UV spectra when the solvent changed from methanol to benzene indicating again a highly polar ground state for the molecule. In the NMR spectrum a singlet due to the formyl proton appeared at τ 0·50. This difference from ylides (VI, R = H) can be ascribed to two causes, less crowding between the substituent groups leading to the rings being more nearly coplanar and a consequent absence of shielding of the aldehyde proton, and the lesser polarity of the formyl group in this case.

The salt VIII ($X = ClO_4$), and also the perchlorates of the phosphonium and arsonium ylides (II and III) condensed readily with *p*-dimethylaminobenzaldehyde in acetic anhydride to produce intensely coloured 5-*p*-dimethylaminobenzylidene derivatives which were protonated in acid to give yellow solutions.

EXPERIMENTAL

Light petroleum had b.p. 40–60°.

pK_a *Determinations.* These determinations were carried out as described in the preceding paper,[1] utilising a method previously used for other ylides.[12]

Reaction of triphenylarsonium ylide (III) *with aldehydes*

(*a*) A soln of the ylide (0·537 g, 1 m.mole) and freshly distilled benzaldehyde (0·106 g, 1 m.mole) in CCl_4 (25 ml) was heated under reflux for 18 hr, further benzaldehyde (0·053 g) being added after 1 hr. The solvent was evaporated and the residue was chromatographed on alumina. Elution with light petroleum-benzene (2:1) and removal of the solvent provided IV (Y = H; 0·152 g, 35%), m.p. 175° (from nitromethane), identical (m.p., IR) with an authentic sample.[13] Elution with MeOH, followed by removal of solvent, addition of water, heating under reflux for 5 min and cooling, gave triphenylarsine oxide (0·17 g, 52%).

(*b*) A similar process using ylide (0·537 g) and *p*-nitrobenzaldehyde (0·15 g, 1 m.mole) gave IV (Y = *p*-NO_2; 0·385 g, 90%), m.p. 163–165° (from nitromethane), identical (m.p. IR, UV) with an authentic sample, and triphenylarsine oxide (0·195 g, 60%).

Similar reactions attempted with II led only to quantitative recovery of the ylide.

6-p-Nitrophenyl-1,2,3-triphenylfulvene (IV, Y = p-NO_2). A soln of 1,2,3-triphenylcyclopentadiene (0·588 g, 2 m.moles), p-nitrobenzaldehyde (0·377 g, 2·5 m.moles) and NaOH (2 g) in MeOH (50 ml) was heated under reflux for 6 hr. A brown ppt formed which was filtered off and chromatographed [alumina; light petroleum-benzene (1:1)] to give the *fulvene* as brown needles (0·13 g, 15%), m.p. 163–165° (from nitromethane), λ_{max} ($CHCl_3$) 273, 361 nm (log ε = 3·54, 3·60) (Found: C, 84·3; H, 5·0; N, 3·2. $C_{30}H_{21}NO_2$ requires: C, 84·2; H, 4·9; N, 3·3%).

Reaction of triphenylarsonium ylide (III) *with nitrosobenzene.* A soln of the ylide (0·537 g, 1 m.mole) and nitrosobenzene (0·107 g, 1 m.mole) in benzene (25 ml) was heated under reflux for 6 hr, a further portion of nitrosobenzene (0·054 g) being added after 3 hr. The soln was then evaporated down to *ca* 5 ml and chromatographed on alumina. Elution with light petroleum-benzene (2:1) gave triphenylarsine (0·078 g, 30%). Light petroleum-benzene (1:5) eluted a small quantity of tetraphenylcyclopentadienone and benzene eluted a fraction which, after evaporation and trituration with EtOH (5 ml) gave V (0·068 g, 17%) identical (m.p., IR, UV) with an authentic sample.

N-Phenyl-2,3,4-triphenylcyclopentadienone ketoxime (V). A soln of 2,3,4-triphenyldiazocyclopentadiene[14] (0·64 g, 2 m.mole) and nitrosobenzene (0·214 g, 2 m.mole) in chloroform (20 ml) was heated under reflux for 18 hr, a further portion of nitrosobenzene (0·107 g) being added after 6 hr. Solvent was evaporated and the residue was dissolved in benzene and applied to an alumina column. Elution with light petroleum-benzene (1:1) gave unreacted diazo-compound (0·2 g), benzene eluted a red oil (unidentified) and chloroform eluted a green soln which, on removal of solvent and trituration with EtOH (10 ml) gave the *ketoxime* (0·096 g, 12%), green needles, m.p. 156–158° (from EtOH, λ_{max} ($CHCl_3$) 256, 362 nm (log ε = 3·52, 3·52) (Found: C, 87·2; H, 5·1; N, 3·5. $C_{29}H_{21}NO$ requires: C, 87·1: H, 5·25; N, 3·5%).

Conversion of ketoxime (V) *into 2,3,4-triphenylcyclopentadienone anil.* A soln of the ketoxime (0·236 g, 0·5 m.mole) and triphenylphosphine (0·262 g, 1 m.mole) in benzene (20 ml) was heated under reflux for 4 hr. Solvent was evaporated and the residue was chromatographed on alumina. Light petroleum-benzene (1:1) eluted excess triphenylphosphine and benzene eluted the anil (0·16 g, 70%) identical (m.p. IR, UV) with the product prepared from 1,2,3-triphenylcyclopentadiene and nitrosobenzene.

2,3,4-Triphenylcyclopentadienone anil. NaOEt [1 ml, from Na (0·5 g) in EtOH (10 ml)] was added to a soln of 1,2,3-triphenylcyclopentadiene (0·588 g, 2 m.mole) and nitrosobenzene (0·214 g, 2 m.mole) in benzene (20 ml) and the mixture was heated under reflux for 10 min. Light petroleum was added to the cooled soln and the mixture was kept at room temp. The *anil* (0·23 g, 30%) separated and formed scarlet needles, m.p. 190–192° (from nitromethane), λ_{max} ($CHCl_3$) 261, 321, 408 nm (log ε = 3·65, 3·18, 2·77) (Found: C, 90·2: H, 5·4: N, 4·2. $C_{29}H_{21}N$ requires: C, 90·8: H, 5·5: N, 3·7%).

Triphenylphosphonium 2-acetyl-3,4,5-triphenylcyclopentadienylide (VI, R = Me, X = P) *and its perchlorate.* A soln of triphenylphosphonium 2,3,4-triphenylcyclopentadienylide (0·493 g) in Ac_2O (15 ml) was heated under reflux for 3 hr. When the soln was cooled crystals of the *acetylylide* (0·32 g, 60%) separated and had m.p. 286° (dec), pale yellow plates (from nitromethane), λ_{max} (EtOH) 265, 325, 354 nm. (log ε = 3·45, 3·29, 3·10), τ ($CDCl_3$) 2·1–3·4 m (30 H), 8·21s (3H) (Found: C, 86·4; H; 5·55. $C_{43}H_{33}OP$ requires; C, 86·5; H, 5·5%). Perchloric acid (70%) was added to a soln of this acetylylide (0·268 g) in EtOH (8 ml), followed by ether (15 ml). The *perchlorate*, yellow prisms (0·33 g, 95%) was filtered off and had m.p. 228–230° (dec) (from n-propanol) (Found: C, 73·8: H, 5·0. $C_{43}H_{34}ClO_5P$ requires: C, 74·0; H, 4·9%).

Triphenylarsonium 2-acetyl-3,4,5-triphenylcyclopentadienylide (VI, R = Me, X = As). Prepared as its phosphonium analogue this acetylylide (0·43 g, 75%) had m.p. 249–252° (dec), λ_{max} (EtOH) 268, 325, 354 nm (log ε = 3·82, 3·15, 3·20, τ ($CDCl_3$) 2·2–3·5 m (30 H), 8·18s (3H) and was identical (m.p., IR) with the product previously obtained from 1,2,3-triphenylcyclopentadiene, triphenylarsine oxide and acetic anhydride.[2, 3]

Triphenylphosphonium 2-formyl-3,4,5-triphenylcyclopentadienylide (VI, R = H, X = P) *and its perchlorate.* Phosphorus oxychloride (0·15 ml, *ca* 2 m.mole) was added to a suspension of triphenylphosphonium 2,3,4-triphenylcyclopentadienylide (0·493 g, 1 m.mole) in N,N-dimethylformamide (5 ml) and the mixture was warmed to 60° for 5 min. 2N NaOH (5 ml) was added and the whole was poured into water (50 ml) and extracted with benzene. The benzene extract was washed well with water, dried (Na_2SO_4) and evaporated. Addition of ether (15 ml) to the residue gave the *formylylide* (0·42 g, 80%), yellow prisms, m.p. 252–253° (dec) (from acetonitrile), λ_{max} (EtOH) 285 nm. (log ε = 4·68), τ ($CDCl_3$) 2·1–3·3 m (Found: C, 86·7; H, 5·4. $C_{42}H_{31}OP$ requires; C, 86·5; H, 5·3%). Perchloric acid (70%, 0·1 ml) was added to a solution of this formylylide (0·26 g) in EtOH (5 ml), followed by ether (10 ml). The *perchlorate*, yellow prisms, (0·25 g, 81%) was filtered off and had m.p. 247–249° (dec) (Found: C, 73·8; H, 5·0. $C_{42}H_{32}ClO_5P$ requires: C, 73·9; H, 4·7%).

Triphenylarsonium 2-formyl-3,4,5-triphenylcyclopentadienylide (VI, R = H, X = As) *and its perchlorate.*

Prepared from triphenylarsonium 2,3,4-triphenylcyclopentadienylide (0·538 g, 1 m.mole) by the same method as its phosphonium analogue, this *formylylide* (0·39 g, 63%) formed yellow needles, m.p. 210–212° (dec) (from acetonitrile), λ_{max} (EtOH) 287 nm (log ε = 4·58), τ ($CDCl_3$) 2·2–3·4 m (Found; C, 81·5; H, 5·2. $C_{42}H_{31}AsO$ requires; C, 80·5; H, 5·2%), *perchlorate*, pale yellow prisms (0·34 g, 95%), m.p. 230–232° (dec) (from EtOH) (Found: C, 68·9; H, 4·6. $C_{42}H_{32}AsClO_4$ requires: C, 69·0; H, 4·4%).

Pyridinium 2-*formyl*-3,4,5-*triphenylcyclopentadienylide* (IX). N,N-Dimethylthioformamide (0·89 g, 10 m.mole) was added to a soln of VIII[3] (X = ClO_4) (0·47 g, 1 m.mole) in Ac_2O (5 ml) and the mixture was heated under reflux for 5 min. Addition of ether to the cooled soln caused an oil to separate which crystallized to give red N-(5-dimethylaminomethylene-2,3,4-triphenylcyclopentadienyl)pyridinium perchlorate (0·32 g, 57%). This salt (0·279 g) was dissolved in N,N-dimethylformamide and aqueous sodium hydroxide (2N, 15 ml) was added. The mixture was shaken well for 5 min and then extracted with ether. The extract was dried and the solvent was removed. Addition of EtOH gave orange-red needles of the *formylylide* (0·146 g, 67%), m.p. 250–252° (from benzene-light petroleum), λ_{max} (EtOH) 268, 287, 326, 447 nm (log ε = 3·77, 3·65, 3·49, 2·86), λ_{max} (benzene) 294, 326sh, 528 nm (log ε = 3·66, 3·20, 2·98), λ_{max} [EtOH-$HClO_4$ (70%) (1:1)] 253, 311, 400sh nm (log ε = 3·71, 3·46, 2·81), τ ($CDCl_3$) 0·50s (1H), 1·3–3·3 m (20H) (Found: C, 87·3; H, 5·4. $C_{29}H_{21}NO$ requires: C, 87·2; H, 5·3%).

Reactions of salts of 2,3,4-*triphenylcyclopentadienylides with* p-*dimethylaminobenzaldehyde*. A mixture of the appropriate ylide perchlorate[3] (1 m.mole), *p*-dimethylaminobenzaldehyde (1·2 m.mole) and Ac_2O was heated under reflux for 20 min. In every case the solns became intensely coloured and they were worked up as follows.

(*a*) *From pyridinium ylide*. Ether (15 ml) was added to the cooled soln and the crystals which separated were filtered off and recrystallized from acetonitrile, a few drops of ether being added to initiate crystallization, to afford N-(5-p-*dimethylaminobenzylidene*-2,3,4-*triphenylcyclopentadienyl*)*pyridinium perchlorate* (0·35 g, 58%), deep maroon needles, m.p. 170–173° (dec) (Found: C, 72·2. H, 5·3. $C_{37}H_{31}ClN_2O_4$ requires; C, 73·7; H, 5·15%).

(*b*) *From triphenylphosphonium ylide*. The soln was evaporated to *ca* 5 ml and ether (15 ml) was added. A powder was filtered off and recrystallized from acetonitrile-ether to give (5-p-*dimethylaminobenzylidene*-2,3,4-*triphenylcyclopentadienyl*)*triphenylphosphonium perchlorate* (0·45 g, 65%), dark red-brown needles, m.p. 173° (dec) (Found: C, 76·45; H, 5·2: N, 1·6. $C_{50}H_{41}ClNO_4P$ requires: C, 76·2; H, 5·2; N, 1·8%).

(*c*) *From triphenylarsonium ylide*. Obtained just as its phosphonium analogue (5-p-*dimethylaminobenzylidene*-2,3,4-*triphenylcyclopentadienyl*)*triphenylarsonium perchlorate* formed dark red-brown needles, m.p. 160–163° (dec) (Found: C, 71·0; H, 4·9: N, 2·0. $C_{50}H_{41}AsClNO_4$ requires: C, 72·7; H, 4·95; N, 1·7%).

Although apparently pure, these benzylidene derivatives appeared to give rather unsatisfactory results when analysed.

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