Synthesis of substituted fulvenes with inden-2-ylphosphonic acid fragments

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We have developed preparative methods for the synthesis of substituted fulvenes including inden-2-ylphosphonic acid fragments, as well as aromatic and heterocyclic substituents, based on bis(trimethylsilyl)inden-2-ylphosphonate and substituted azomethines in the presence of sodium hydride as an efficient initiator of the reaction. Subsequent treatment of the phosphonates with methanolic solution of sodium methoxide furnished water-soluble disodium salts of the corresponding phosphonic acids.

Key words: organophosphorus fulvenes, bis(trimethylsilyl)inden-2-ylphosphonate, substituted azomethines, sodium hydride, disodium salts.

Fulvenes are an important class of cross-conjugated alkenes with unique electronic, spectroscopic, and chemical properties. Substituted fulvenes are convenient synthons for the preparation of metallocene catalysts^{1,2} and are used in materials science and molecular electronics,³ and as key intermediate products in the synthesis of biologically active compounds.^{4,5} Some fulvenes have been isolated from natural objects and found to possess antibacterial activity, for example, fulvoplumierin, which was later synthesized in several steps.^{6,7}

There are known methods for the synthesis of some fulvenes based on the condensation of aldehydes and ketones with substituted cyclopentadienes in the presence of basic catalysts, however, this was accompanied by the formation of a considerable amount of resin-like polymers.^{8,9} At the same time, stable 6-dimethylaminofulvene was obtained in high yield based on cyclopentadiene and dimethylformamide.¹⁰ There is also known an original method for obtaining fulvenes, which involves radical cyclization of substituted *o*-phenylenediynes in the presence of tributylstannane.¹¹ Recently, we have developed convenient methods for the synthesis of some organophosphorus compounds with different functional groups

and heterocyclic fragments based on trimethylsilyl esters of tervalent phosphorus acids. $^{12-14}$

In the present work, we study the reaction of esters 2 of available inden-2-ylphosphonic acid 1, the synthesis of which has been described earlier, ¹⁵ with substituted azomethines, which leads to new fulvenes 3 bearing aromatic and heterocyclic substituents.¹⁶ The starting esters **2a,b** were obtained in high yields by the reaction of inden-2-ylphosphonic acid 1 with triethylorthoformate or bis(trimethylsilyl)amine, respectively (Scheme 1).

We have found that the stirring the mixture of phosphonate 2b with azomethines in diethyl ether in the presence of sodium hydride as the reaction initiator is accompanied by the evolution of methylamine and leads to the formation of good yields of fulvenes 3a-d (Scheme 2). Note that the reaction of phosphonate 2a under similar conditions leads mainly to polymeric products, that can be apparently explained by the higher stability of trimethylsilyl-containing fulvenes formed in the reaction process. The reaction scheme includes the formation of an intermediate adduct as a result of the base-catalyzed addition of the phosphorus-substituted indene as a CH-acid at the double bond of the azomethine with subsequent elimination of methylamine (see Scheme 2).



Scheme 1

Reaction conditions: 100–125 °C.

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It is obvious that an increased CH-acidity of ester **2b** is related to the influence of the electron-withdrawing phosphorus-containing group.

Scheme 2



Reaction conditions: Et₂O, 25 °C.

The first step of the suggested reaction scheme includes the metallation of CH-acid **2b** with sodium hydride, the addition of sodium intermediate **2c** at the double bond of the azomethine with the formation of intermediate **2d**, which then disintegrates to the target fulvenes **3a**-**d** and sodium *N*-methylamide (Scheme 3).

Scheme 3



Reaction conditions: Et₂O, 25 °C.

Then, sodium *N*-methylamide successfully catalyzed this reaction with the formation of substituted fulvenes 3a-d and elimination of methylamine (Scheme 4).

Scheme 4



Reaction conditions: Et₂O, 25 °C.

Treatment of both phosphonate 2b and phosphonates 3a-d with a dilute solution of sodium methoxide in methanol gave stable water-soluble disodium salts 4 and 5, respectively (Scheme 5), which are hygroscopic white (4) and lemon-yellow (5) crystals.

Scheme 5



Reaction conditions: Et₂O, 10 °C.

In conclusion, based on the readily available starting compounds we have developed original methods for the synthesis of new stable phosphorus-substituted fulvenes including aromatic and heterocyclic fragments together with the inden-2-ylphosphonic acid moieties. The new compounds are convenient synthons for the preparation of various functionalized organophosphorus compounds and are interesting as biologically active compounds and promising polydentate ligands.

Experimental

¹H, ¹³C{¹H}, and ³¹P{¹H} NMR spectra were recorded on a Bruker Avance 400 spectrometer (400, 100, and 162 Hz, respectively), using Me₄Si (¹H, ¹³C{¹H}) and 85% H₃PO₄ in D₂O (³¹P{¹H}) as references. All the reactions were carried out under dry argon in anhydrous solvents. The starting inden-2-ylphosphonic acid was obtained according to the known method.¹³ Disodium salts **4** and **5** undergo decomposition upon heating higher 100 °C and do not have sharp melting points.

0,0-Diethyl 1*H***-inden-2-ylphosphonate (2a).** A mixture of triethylorthoformate (101 g, 0.68 mol) and 1*H*-inden-2-ylphosphonic acid (1) (33.3 g, 0.17 mol) (obtained according to the method described in the literature³) was refluxed for 2 h and then distilled to obtain ester **2a** (34.7 g, 81%), b.p. 144 °C (1 Torr). Found (%): C, 61.74; H, 6.68. C₁₃H₁₇O₃P. Calculated (%): C, 61.90; H, 6.79. ¹H NMR (CDCl₃), δ : 1.2 (t, 6 H, 2 Me, ${}^{3}J_{\rm H,H} =$ 7.2 Hz); 3.50 (s, 2 H, C(1)H₂); 4.00–4.10 (m, 4 H, 2 OCH₂); 7.20–7.40 (m, 4 H, 4 C_{arom}H); 7.50 (d, 1 H, C(3)H, ${}^{3}J_{\rm P,H} =$ 10.4 Hz). ¹³C{¹H} NMR (CDCl₃), δ : 1.2 (t, Me, ${}^{3}J_{\rm P,C} =$ 6.2 Hz); 39.6 (d, C(1), ${}^{2}J_{\rm P,C} =$ 14.0 Hz); 61.3 (d, OCH₂, ${}^{2}J_{\rm P,C} =$ 5.5 Hz); 122.3 (s, C_{arom}); 123.5 (s, C_{arom}); 126.6 (s, C_{arom}); 133.1 (d, C(2), {}^{1}J_{\rm P,C} = 198.9 Hz);

142.1 (d, C(3a), ${}^{3}J_{P,C} = 21.1$ Hz); 144.6 (d, C(3), ${}^{2}J_{P,C} = 13.9$ Hz); 144.8 (d, C(7a), ${}^{3}J_{P,C} = 11.9$ Hz). ${}^{31}P{}^{1}H{}$ NMR (CDCl₃), δ : 17.7 (s).

0,0-Bis(trimethylsilyl) 1*H*-inden-2-ylphosphonate (2b). A mixture of bis(trimethylsilyl)amine (100 g, 0.62 mol) and 1*H*-inden-2-ylphosphonic acid 1 (43 g, 0.22 mol) (obtained according to the method described in the work³) was refluxed until ammonia ceased to evolve, then distilled to obtained ester **2b** (66.4 g, 89%), b.p. 149 °C (1 Torr), m.p. 52 °C. Found (%): C, 52.76; H, 7.26. $C_{15}H_{25}O_3PSi_2$. Calculated (%): C, 52.91; H, 7.40. ¹H NMR (CDCl₃), δ : 0.21 (d, 18 H, 2 Me₃Si, ⁴J_{P,H} = = 4.8 Hz); 3.50 (s, 2 H, C(1)H₂); 7.10-7.40 (m, 5 H, C₉H₇). ¹³C{¹H} NMR (CDCl₃), δ : 0.60 (s, 2 Me₃Si); 39.6 (d, C(1), ²J_{P,C} = 15.2 Hz); 122.2 (s, C_{arom}); 123.6 (s, C_{arom}); 126.3 (s, 2 C_{arom}); 137.3 (d, C(2), ¹J_{P,C} = 208.2 Hz); 141.9 (d, C(3), ²J_{P,C} = 14.7 Hz); 142.5 (d, C(3a), ³J_{P,C} = 21.3 Hz); 144.6 (d, C(7a), ³J_{P,C} = 12.3 Hz). ³¹P{¹H} NMR (CDCl₃), δ : -1.1 (s).

O,O-Bis(trimethylsilyl) (1-benzylidene-1H-inden-2-yl)phos**phonate (3a).** *N*-Benzylidene-*N*-methylamine (1.6 g, 0.013 mol) and sodium hydride (0.1 g, 0.004 mol) were added to a solution of phosphonate **2b** (4.5 g, 0.013 mol) in diethyl ether (10 mL) with stirring. After 2 h, the solvent was removed and the residue was distilled to obtain phosphonate 3a (4.3 g, 75%), b.p. 212 °C (2 Torr), m.p. 74 °C. Found (%): C, 61.56; H, 6.71. $C_{22}H_{29}O_3PSi_2$. Calculated (%): C, 61.65; H, 6.82. ¹H NMR (CDCl₃), δ: 0.43 (s, 18 H, 2 Me₃Si); 6.80-7.60 (m, 9 H, 5 C_{Ph}H); 7.52 (d, 1 H, C(3)H, ${}^{3}J_{P,H} = 6.8$ Hz); 7.97 (s, 1 H, C(8)H). ${}^{13}C{}^{1}H{}$ NMR (CDCl₃), δ : 1.9 (s, 2 Me₃Si); 122.5 (s, C_{arom}); 123.6 (s, C_{arom}); 127.0 (s, C_{arom}); 128.3 (s, C_{Ph}); 128.5 (s, C_{Ph}); 129.1 (s, C_{Ph}); 129.2 (s, C_{Ph}); 136.5 (s, C_{arom}); 135.9 (d, C(2), ${}^{1}J_{P,C} = 208.3 \text{ Hz}$); 136.3 (d, C(1), ${}^{2}J_{P,C} = 14.9 \text{ Hz}$); 136.8 (d, C(8), ${}^{3}J_{P,C} = 6.1 \text{ Hz}$); 139.1 (d, C(3a), ${}^{3}J_{P,C} = 16.0 \text{ Hz}$); 141.9 (d, C(3), ${}^{2}J_{P,C} = 12.3 \text{ Hz}$); 141.7 (d, C(7a), ${}^{3}J_{P,C} = 18.4 \text{ Hz}$). $^{31}P{^{1}H} NMR (CDCl_3), \delta: -2.9 (s).$

Phosphonates **3b**-**d** were obtained similarly.

0,0-Bis(trimethylsily) [1-(4-methoxybenzylidene)-1*H*-inden-2-yl]phosphonate (3b). The yield was 78%, b.p. 210 °C (1.5 Torr), m.p. 89 °C. Found (%): C, 60.06; H, 6.69. C₂₃H₃₁O₄PSi₂. Calculated (%): C, 60.23; H, 6.81. ¹H NMR (CDCl₃), δ : 0.29 (s, 18 H, 2 Me₃Si); 3.84 (s, 3 H, MeO); 6.81–7.60 (m, 8 H, 4 C_{arom}H, 4 C_{C6}H₄H); 7.64 (d, 1 H, C(3)H, ³J_{P,H} = 8.4 Hz); 7.83 (s, 1 H, C(8)H). ¹³C{¹H} NMR (CDCl₃), δ : 0.03 (s, 2 Me₃Si); 55.2 (s, MeO); 60.0 (s, C_{C6}H₄O); 113.8 (s, 2 C_{C6}H₄); 122.3 (s, C_{arom}); 123.4 (s, C_{arom}); 126.7 (s, C_{arom}); 127.8 (s, C_{arom}); 128.7 (s, C_{C6}H₄); 131.0 (s, 2 C_{C6}H₄); 135.3 (d, C(2), ¹J_{P,C} = 206.2 Hz); 136.1 (d, C(1), ²J_{P,C} = 14.8 Hz); 136.5 (s, C_{arom}); 137.1 (s, C(8)); 138.9 (d, C(3a), ³J_{P,C} = 12.1 Hz); 140.9 (d, C(3), ²J_{P,C} = 11.9 Hz); 141.6 (d, C(7a), ³J_{P,C} = 12.1 Hz); 160.0 (s, CO). ³¹P{¹H} NMR (CDCl₃), δ : -2.6 (s).

O,*O*-Bis(trimethylsily) [1-(furan-2-ylmethylene)-1*H*-inden-2-yl]phosphonate (3c). The yield was 74%, b.p. 210 °C (2 Torr), m.p. 61 °C. Found (%): C, 57.23; H, 6.42. $C_{20}H_{27}O_4PSi_2$. Calculated (%): C, 57.39; H, 6.50. ¹H NMR (CDCl₃), δ : 0.42 (s, 18 H, 2 Me₃Si); 6.57–7.60 (m, 7 H, 4 C_{arom}H, 3 C_{Het}H); 7.70 (s, 1 H, C(8)H); 8.70 (d, 1 H, C(3)H, ³J_{P,H} = 7.6 Hz). ¹³C{¹H} NMR (CDCl₃), δ : 0.9 (s, 2 Me₃Si); 112.8 (s, C_{Het}); 118.4 (s, C_{Het}); 120.9 (s, C_{arom}); 122.3 (s, C_{arom}); 124.9 (s, C_{arom}); 127.2 (s, C_{arom}); 127.8 (s, C(8)); 133.8 (d, C(2), ¹J_{P,C} = 205.6 Hz); 134.9 (d, C(3a), ³J_{P,C} = 16.4 Hz); 135.5 (d, C(1), ²J_{P,C} = 15.6 Hz); 140.6 (d, C(3), ²J_{P,C} = 12.3 Hz); 141.4 (d, C(7a), ³J_{P,C} = 21.7 Hz); 144.9 (s, C_{Het}HO); 151.7 (s, C_{Het}O). ³¹P{¹H} NMR (CDCl₃), δ: -2.2 (s). *O*,*O*-Bis(trimethylsily) [1-(pyridin-3-ylmethylene)-1*H*-inden-2-yl]phosphonate (3d). The yield was 72%, b.p. 200 °C (1 Torr), m.p. 65 °C. Found (%): C, 58.62; H, 6.52. C₂₁H₂₈NO₃PSi₂. Calculated (%): C, 58.71; H, 6.57. ¹H NMR (CDCl₃), δ: 0.45 (s, 18 H, 2 Me₃Si); 7.05–7.86 (m, 8 H, 4 C_{arom}H, 4 C_{Py}H); 8.66 (d, 1 H, C(3)H, ${}^{3}J_{P,H} = 9.7$ Hz); 8.78 (s, 1 H, C(8)H). ${}^{13}C{}^{1}H$ } NMR (CDCl₃), δ: 1.1 (s, 2 Me₃Si); 122.2 (s, C_{arom}); 123.7 (s, C_{Py}); 127.4 (s, C_{arom}); 128.6 (s, C_{arom}); 132.1 (s, C_{arom}); 132.7 (s, C_{Py}); 133.3 (d, C(2), ${}^{1}J_{P,C} = 208.2$ Hz); 135.9 (d, C(1), ${}^{2}J_{P,C} = 12.3$ Hz); 136.8 (s, C(8)); 141.3 (d, C(3a), ${}^{3}J_{P,C} = 15.9$ Hz); 141.9 (d, C(7a), ${}^{3}J_{P,C} = 20.9$ Hz); 142.7 (d, C(3), ${}^{2}J_{P,C} = 12.5$ Hz); 149.1 (s, C_{Py}N); 149.5 (s, C_{Py}N). ${}^{31}P{}^{1}H$ NMR (CDCl₃), δ: –3.3 (s).

1*H***-Inden-2-ylphosphonic acid disodium salt (4).** A solution of phosphonate **2b** (3.4 g, 0.01 mol) in diethyl ether (5 mL) was added to a solution of sodium methoxide (1.1 g, 0.02 mol) in methanol (30 mL) with stirring at 10 °C. The reaction mixture was brought to reflux, the solvent was removed, the residue was allowed to stand *in vacuo* (1 Torr) for 1 h. The yield of salt **4b** was 2.3 g (96%). Found (%): C, 44.91; H, 3.03. C₉H₇Na₂O₃P. Calculated (%): C, 45.02; H, 2.94. ¹H NMR (D₂O), δ : 3.65 (s, 2 H, C(1)H₂); 7.20–7.50 (m, 4 H, 4 C_{arom}H); 7.58 (d, 1 H, C(3)H, ³J_{P,H} = 7.6 Hz). ¹³C{¹H} NMR (D₂O), δ : 40.7 (d, C(1), ²J_{P,C} = = 11.9 Hz); 121.8 (s, C_{arom}); 124.1 (s, C_{arom}); 125.4 (s, C_{arom}); 126.6 (s, C_{arom}); 135.9 (d, C(3), ²J_{P,C} = 10.7 Hz); 144.6 (d, C(3a), ³J_{P,C} = 18.9 Hz); 145.6 (d, C(7a), ³J_{P,C} = 10.7 Hz); 147.1 (d, C(2), ¹J_{P,C} = 176.2 Hz). ³¹P{¹H} NMR (D₂O), δ : 10.7 (s). Salt **5a**–**d** were obtained similarly.

1-Benzylidene-1*H***-inden-2-ylphosphonic acid disodium salt** (5a). The yield was 97%. Found (%): C, 58.43; H, 3.41. C₁₆H₁₁Na₂O₃P. Calculated (%): C, 58.55; H, 3.38. ¹H NMR (D₂O), δ : 6.75–7.55 (m, 9 H, 4 C_{arom}H, 5 C_{Ph}H); 7.64 (d, 1 H, C(3)H, ³J_{P,H} = 7.2 Hz); 7.89 (s, 1 H, C(8)H). ¹³C{¹H} NMR (D₂O), δ : 121.9 (s, C_{arom}); 123.7 (s, C_{arom}); 127.1 (s, C_{arom}); 128.1 (s, C_{Ph}); 128.4 (s, C_{Ph}); 129.2 (s, C_{Ph}); 129.4 (s, C_{Ph}); 136.1 (d, C(1), ²J_{P,C} = 8.4 Hz); 136.4 (s, C(8)); 136.7 (d, C(3), ²J_{P,C} = 9.8 Hz); 137.4 (s, C_{arom}); 140.0 (d, C(3a), ³J_{P,C} = 12.1 Hz); 143.2 (d, C(2), ¹J_{P,C} = 174.4 Hz); 143.6 (d, C(7a), ³J_{P,C} = 20.4 Hz). ³¹P{¹H} NMR (D₂O), δ : 7.8 (s).

1-(4-Methoxybenzylidene)-1*H*-inden-2-ylphosphonic acid disodium salt (5b). The yield was 97%. Found (%): C, 56.89; H, 3.74. C₁₇H₁₃Na₂O₄P. Calculated (%): C, 57.00; H, 3.66. ¹H NMR (D₂O), δ : 3.78 (s, 3 H, MeO); 6.80–7.40 (m, 4 H, 4 C_{arom}H); 6.99 (d, 2 H, 2 C_{C6}H₄H, ³J_{H,H} = 8.4 Hz); 7.45 (d, 1 H, C(3)H, ³J_{P,H} = 8.0 Hz); 7.55 (d, 2 H, 2 C_{C6}H₄H, ³J_{H,H} = 8.4 Hz); 7.93 (s, 1 H, C(8)H). ¹³C{¹H} NMR (D₂O), δ : 55.5 (s, MeO); 114.1 (s, 2 C_{C6}H₄); 121.6 (s, C_{arom}); 123.1 (s, C_{arom}); 125.3 (s, C_{arom}); 128.0 (s, C_{arom}); 130.0 (s, C_{C6}H₄); 131.2 (s, 2 C_{C6}H₄); 135.7 (d, C(3), ²J_{P,C} = 9.5 Hz); 136.0 (d, C(1), ²J_{P,C} = 8.3 Hz); 137.1 (s, C(8)); 139.8 (d, C(3a), ³J_{P,C} = 12.8 Hz); 143.3 (d, C(2), ¹J_{P,C} = 172.9 Hz); 143.6 (d, C(7a), ³J_{P,C} = 16.0 Hz); 159.1 (s, C_{C6}H₄O). ³¹P{¹H} NMR (D₂O), δ : 8.1 (s).

1-(Furan-2-ylmethylene)-1*H*-inden-2-ylphosphonic acid disodium salt (5c). The yield was 95%. Found (%): C, 52.64; H, 2.91. $C_{14}H_9Na_2O_4P$. Calculated (%): C, 52.85; H, 2.85. ¹H NMR (D₂O), δ : 6.63–7.67 (m, 7 H, 4 $C_{arom}H$, 3 $C_{Het}H$); 7.75 (s, 1 H, C(8)H); 8.63 (d, 1 H, C(3)H, ³ $J_{P,H}$ = 7.2 Hz). ¹³C{¹H} NMR (D₂O), δ : 113.2 (s, C_{Het}); 118.4 (s, C_{Het}); 121.3 (s, C_{arom}); 121.8 (s, C_{arom}); 124.7 (s, C_{arom}); 126.1 (s, C_{arom}); 128.3 (s, C(8)); 135.5 (d, C(1), ² $J_{P,C}$ = 8.1 Hz); 135.8 (d, C(3), ² $J_{P,C}$ = 11.4 Hz); 136.5 (d, C(3a), ³ $J_{P,C}$ = 12.1 Hz); 143.5

(d, C(7a), ${}^{3}J_{P,C} = 17.4 \text{ Hz}$); 143.9 (d, C(2), ${}^{1}J_{P,C} = 173.3 \text{ Hz}$); 145.5 (s, C_{Het}HO); 152.2 (s, C_{Het}O). ${}^{31}P{}^{1}H$ NMR (D₂O), δ : 8.3 (s).

1-(Pyridine-3-ylmethylidene)-1*H*-inden-2-ylphosphonic acid disodium salt (5d). The yield was 98%. Found (%): C, 54.59; H, 3.04. $C_{15}H_{10}NNa_2O_3P$. Calculated (%): C, 54.73; H, 3.06. ¹H NMR (D₂O), δ : 7.10–7.90 (m, 8 H, 4 $C_{arom}H$, 4 $C_{Py}H$); 8.61 (d, 1 H, C(3)H, ${}^{3}J_{P,H} = 9.2$ Hz); 8.82 (s, 1 H, C(8)H). ¹³C{¹H} NMR (D₂O), δ : 122.1 (s, C_{arom}); 123.8 (s, 2 C_{Py}); 127.3 (s, C_{arom}); 128.9 (s, C_{arom}); 132.0 (s, C_{arom}); 133.0 (s, C_{Py}); 136.0 (d, C(1), ${}^{2}J_{P,C} = 7.2$ Hz); 137.3 (s, C(8)); 137.8 (d, C(3), ${}^{2}J_{P,C} = 11.6$ Hz); 142.9 (d, C(3a), ${}^{3}J_{P,C} = 11.8$ Hz); 143.3 (d, C(2), ${}^{1}J_{P,C} = 175.5$ Hz); 144.1 (d, C(7a), ${}^{3}J_{P,C} = 16.8$ Hz); 149.7 (s, $C_{Py}N$); 150.0 (s, $C_{Py}N$). ${}^{31}P{}^{1}H$ NMR (D₂O), δ : 7.3 (s).

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