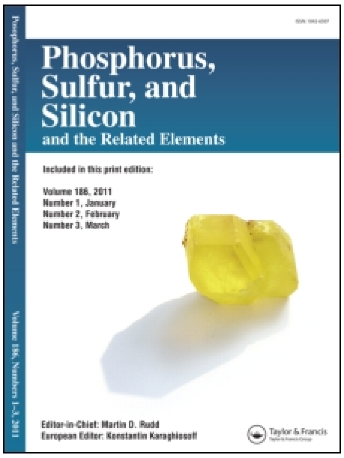

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Synthesis of Heterocyclic Pentavalent Phosphorus Compounds from Phosphite Derivatives and Indane-1,2,3-Trione

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SYNTHESIS OF HETEROCYCLIC PENTAVALENT PHOSPHORUS COMPOUNDS FROM PHOSPHITE DERIVATIVES AND INDANE-1,2,3-TRIONE

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Reaction of trialkylphosphite derivatives with indane-1,2,3-trione proceeds smoothly at room temperature to afford the corresponding heterocyclic pentavalent [P(V)] phosphorus compounds via an intermolecular [4+1] cycloaddition reaction in excellent yields under solvent-free conditions. We also used dimethylphosphite and diethylphosphite instead of trialkylphosphites in this reaction, but the corresponding pentavalent phosphorus compounds were not isolated, and in both cases phosphate derivatives were obtained. The structures of the products were deduced from their IR, ^{1}H NMR, ^{13}C NMR, and ^{31}P NMR spectra, and mass spectrometry.

Keywords Dialkylphosphite; heterocyclic pentavalent phosphorus compound; indane-1,2,3-trione; intermolecular [4+1] cycloaddition reaction; solvent-free conditions; stereochemistry; trialkylphosphite

INTRODUCTION

Phosphorus is present in a multitude of forms in nature and is essential to life.[1–4] Phosphorus-containing materials range from calcium phosphate in bone and teeth via the biochemical energy transfer agent adenosine triphosphate (ATP) to the essence of life itself in the form of the carriers of genetic information, the nucleic acids RNA and DNA.[1–4] In nature, phosphorus is found almost exclusively in the pentavalent oxidation state.[1–4] Early in the last century, interest in the chemistry of phosphorus compounds was based primarily on practical applications. For example, phosphorus compounds were widely used as plasticizers, as extraction agents, as oxidation inhibitors for lubricants, as flotation agents, and as complexing agents for transition metals.[1–4]

Research in phosphorus chemistry has developed rapidly in recent decades.[5] Study of a number of systems show the unique possibilities phosphorus introduces via coordination in different valence states.[4,5] The significance of phosphorus chemistry is reflected in its many industrial applications, for example as fertilizers, pesticides, flame retardants, and antioxidants, and its role in biological systems.[1–4] Interest in phosphorus chemistry is also stimulated by the theoretical problems that arise when attempting to understand the details

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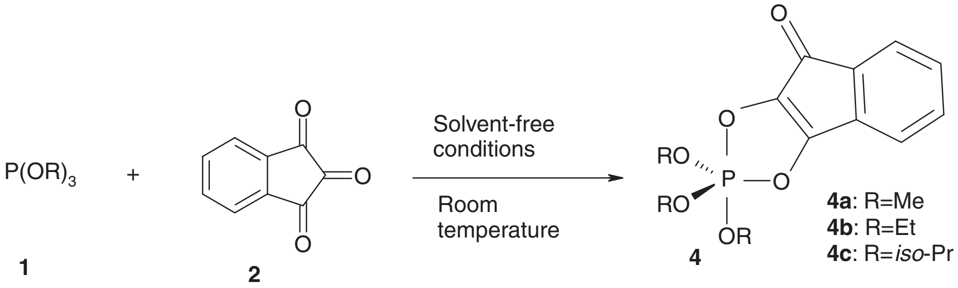
of the reactions and structures of its compounds. Significant contributions have been made both by direct investigations of relevant systems and by studies of model compounds.[4–7] The results of these studies may in turn be helpful in understanding the mechanistic aspects of more complicated reactions. Another important feature of phosphorus compounds is their capability for group transfer. In this respect, mention should be made of the powerful alkylating properties of oxyphosphoranes towards carboxylic acids.[4]

During last few decades, a dramatic increase in research activity of the chemistry of pentavalent [P(V)] phosphorus compounds (phosphorans) has taken place.[8] This has led to a more thorough understanding of the features that determine the stability and the ligand reorganization within such molecules. Nucleophilic attack on one of the alkoxy ligands followed by ring opening leads to the formation of the products. However, ring opening might precede the alkyl transfer, generating the isomeric phosphonium ion.[8] In general, several methods are available for the synthesis of oxyphosphoranes. The reaction of dialkyl peroxides with phosphites in aprotic media at room temperature affords a general synthesis of oxyphosphoranes.[4] One major disadvantage of this method is the fact the products are not obtained in pure form as a consequence of the decomposition into their corresponding phosphates.[4] Phosphorus pentachloride has been used in many condensation reactions with compounds containing an active hydrogen atom, such as phenols and alcohols. Generally in these reactions, different products can be obtained depending on the number of chlorine substituents. One of the earliest and most useful methods of preparing oxyphosphoranes involves the condensations of trivalent phosphorus compounds with *O*-quinolines, α-diketones, or α,β-unsaturated ketones.[4] From both the theoretical and X-ray crystallographic analyses of several P(V) structures, it could be established that the trigonal bipyramid (TBP) is the most stable configuration for acyclic and monocyclic P(V) compounds.[8]

These days, phosphorus chemistry forms an important discipline in element chemistry.[5] Heterocyclic phosphorus chemistry is a dynamically developing field[5] including, among other aspects, the chemistry of P-ligands[2–5] and fragmentation-related phosphorylations.[2–4] During the past 20 years, Keglevich et al. introduced a number of families of P-heterocycles embracing 5- and 6-membered ring compounds and functionalized derivatives.[9–15] The heterocycles with a sterically demanding substituent on the phosphorus atom[9–15] are of especial importance, as a bulky substituent, e.g., a 2,4,6-trialkylphenyl group, affects the reactivity of the parent molecule due to mainly steric effects.[9–15]

For several years, ninhydrin has been attracting the attention of organic chemists.[5] Ninhydrin and its dehydrated form (indane-1,2,3-trione) are reactive systems and take part in many chemical syntheses.[5] In addition, the compounds act as a electrophile in organic reactions.[5] The carbon atom of a carbonyl bears a partial positive charge, so the central carbon of a 1,2,3-tricarbonyl is less stable and more electrophilic than a simple ketone. In most compounds, a carbonyl is more stable than the dihydroxy (hydrate) form. However, ninhydrin is a stable hydrate of the central carbon because this form does not have the destabilizing effect of adjacent carbonyl partial-positive centers. Indane-1,2,3-trione **2** reacts readily with nucleophiles.[5]

In recent years, there has been increasing interest in the applications of indane-1,2,3-trione **2** in multicomponent[5] synthesis. Due to the atom economy, convergent character, and simplicity of one-pot procedures, multicomponent condensation reactions (MCRs) have an advantageous position among other reactions.[16–23] The discovery and development of novel MCRs is receiving growing interest from industrial chemistry research groups and represents a new challenge for organic chemists and to the basic understanding of organic chemistry itself.[5] In recent years, we have established a one-pot method for the synthesis of organophosphorus compounds.[24–30] As part of our ongoing program to develop efficient and robust methods for the preparation of heterocyclic compounds,[31–60] we sought to develop a convenient preparation of heterocyclic pentavalent phosphorus compounds **4** from trialkylphosphite derivatives **1** and indane-1,2,3-trione **2** in fairly good yields under neutral conditions (Scheme 1).

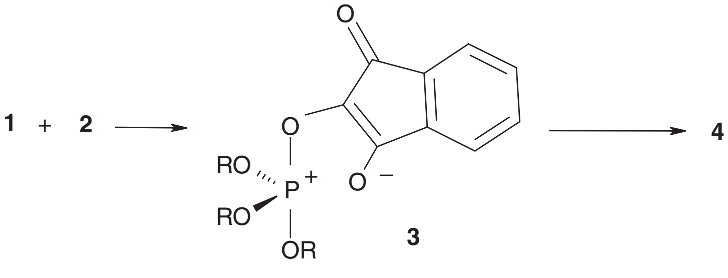


Scheme 1 Preparation of heterocyclic pentavalent phosphorus compounds **4** from trialkylphosphite derivatives **1** and indane-1,2,3-trione **2**.

RESULTS AND DISCUSSION

In the last several years, numerous synthetic methods have been reported for the preparation of heterocyclic pentavalent phosphorus compounds.[8] However, application of indane-1,2,3-trione **2** in the synthesis of heterocyclic pentavalent phosphorus compounds has not been reported. As part of our ongoing program to develop efficient and robust methods for the preparation of heterocyclic compounds,[31–60] we sought to develop a convenient preparation of heterocyclic pentavalent phosphorus compounds **4** from trialkylphosphite derivatives **1** and indane-1,2,3-trione **2** in fairly good yields under neutral conditions (Scheme 1). The trialkylphosphite derivatives **1** and indane-1,2,3-trione **2** in solvent-free conditions react together in a 1:1 ratio at room temperature to produce corresponding heterocyclic pentavalent phosphorus compounds **4** in fairly good yields under neutral conditions (Scheme 1). The reaction proceeds smoothly and cleanly under mild conditions.

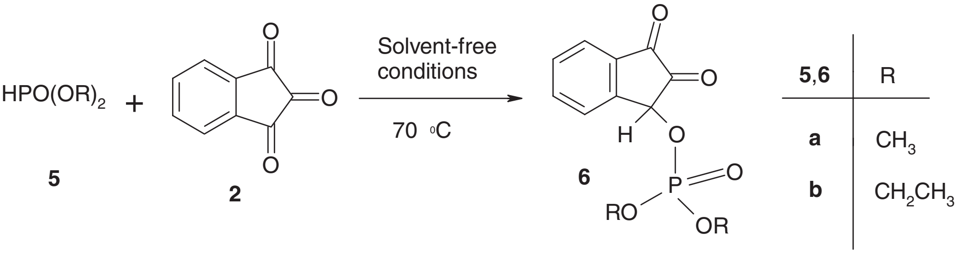
The mechanism of the reaction between the trialkylphosphite derivatives **1** and indane-1,2,3-trione **2** has not been established experimentally. However, a possible explanation is proposed in Scheme 2. On the basis of the well established chemistry of trialkylphosphites,[8]



Scheme 2 Proposed mechanism for the formation of heterocyclic pentavalent phosphorus compounds **4** from trialkylphosphite derivatives **1** and indane-1,2,3-trione **2**.

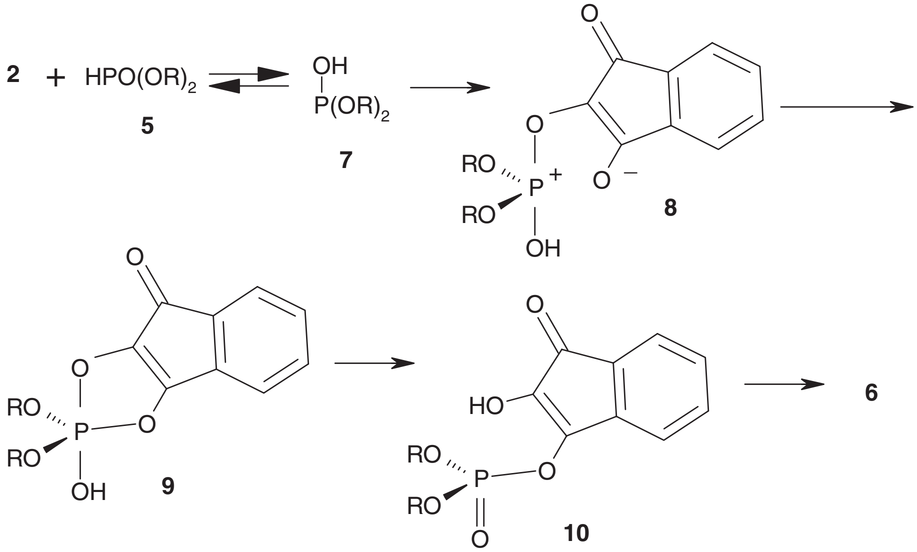
it is reasonable to assume that nucleophilic addition of **1** to indane-1,2,3-trione **2** followed by a cyclization reaction can generate the corresponding heterocyclic pentavalent phosphorus compounds **4** (Scheme 1).[8] The structures of the products **4a–c** were deduced from their IR, ^{1}H NMR, and ^{13}C NMR spectra. The mass spectra of these compounds displayed molecular ion peaks at the appropriate *m/z* values. The IR spectrum of **4a** showed strong absorptions at 1719 (C=O, ketone); 1641 (C=C, aromatic); 1285 and 1028 (C—O) cm^{-1}, indicating the presence of the mentioned functionalities in its formula. ^{1}H NMR spectrum of compound **4a** exhibited three signals readily recognized as arising from two equatorial OMe groups [δ = 3.94 (6H, d, $^3J_{PH}$ = 11.3 Hz, 2OCH$_3$, eq.)]; an axial OMe group [δ = 4.34 (3H, s, OCH$_3$, ax.)], and an aromatic moiety [δ = 7.11–7.36 (4H, m, arom)]. The ^{1}H decoupled ^{13}C NMR spectrum of **4a** showed 11 distinct resonances [δ = 55.50 (2OCH$_3$, d, $^2J_{POC}$ = 6.3 Hz, eq.); 59.73 (OCH$_3$, ax.); 119.08, 121.14, 129.87, and 133.01 (4CH, arom); 121.86 (*C*=C—C=O, d, $^2J_{POC}$ = 8.3 Hz, olefinic C, eq.); 127.62 and 137.00 (2C, arom); 161.30 (C=*C*—C=O, d, $^2J_{POC}$ = 6.0 Hz, olefinic C, ax.) and 187.92 (C=O)] that are in agreement with the formula and structure of **4a**. The ^{31}P NMR spectrum of compound **4a** exhibited a signal [δ = −2.64 (s)] readily recognized as arising from the pentavalent oxyphosphorane group. Partial assignment of these resonances is given in the spectral analysis section (see the Experimental section).

We also used dimethylphosphite and diethylphosphite instead of trialkylphosphites **1** in this reaction, but the corresponding products **4** were not isolated, and in both cases phosphate derivatives **6** were isolated (Scheme 3). The dialkylphosphite derivatives **5** and indane-1,2,3-trione **2** in solvent-free conditions react together in a 1:1 ratio at 70°C to produce the corresponding phosphate derivatives **6** in fairly good yields under neutral conditions (Scheme 3). The reaction proceeds smoothly and cleanly under fairly mild conditions.



Scheme 3 Preparation of phosphate derivatives **6** from dialkylphosphites **5** and indane-1,2,3-trione **2**.

The mechanism of the reaction between the dialkylphosphite derivatives **5** and indane-1,2,3-trione **2** has not been established experimentally. However, a possible explanation is proposed in Scheme 4. On the basis of the well established chemistry of phosphites,[8] it is reasonable to assume that nucleophilic addition of **7** (tautomer of **5**) to indane-1,2,3-trione **2** followed by a cyclization reaction can generate the corresponding heterocyclic pentavalent phosphorus intermediates **9** (Scheme 4).[8] The intermediates **9** are not stable and convert to phosphate derivatives **6** via unstable anti-aromatic enols of **10** (Scheme 4). The structures of the products **6a,b** were deduced from their IR, ^{1}H NMR, and ^{13}C NMR spectra. The IR spectrum of **6a** showed strong absorptions at 1725 (C=O, ketone); 1258 and 1028 (C—O) cm^{-1} indicating the presence of the mentioned functionalities in its formula. The ^{1}H NMR spectrum of compound **6a** exhibited three signals readily recognized as arising from two



Scheme 4 Proposed mechanism for the formation of phosphate derivatives **6** from dialkylphosphites **5** and indane-1,2,3-trione **2**.

OMe groups [$\delta = 3.52$ (6 H)]; a OCH group [$\delta = 7.12$ (1 H, s.)]; and an aromatic moiety [$\delta = 7.85$–8.21 (4 H, m, arom)]. The ^{1}H decoupled ^{13}C NMR spectrum of **6a** showed 10 distinct resonances [$\delta = 49.60$ (2 OCH_3, d, $^2J_{POC} = 6.9$ Hz); 73.38 (OCH, $^2J_{POC} = 13.2$ Hz); 118.00, 118.26, 132.01, and 132.25 (4 CH, arom); 135.78 and 135.99 (2 C, arom); 186.68 and 190.24 (2 C=O)] that are in agreement with the formula and structure of **6**a. ^{31}P NMR spectrum of compound **6a** exhibited a signal [$\delta = -2.75$ (s)] readily recognized as arising from the phosphate group. Partial assignment of these resonances is given in the spectral analysis section (see the Experimental section).

CONCLUSION

In summary, we have found a new method for the preparation of heterocyclic pentavalent phosphorus compounds **4** from trialkylphosphite derivatives **1** and indane-1,2,3-trione **2** in fairly good yields under neutral conditions (Scheme 1). We have also found a new method for the preparation of phosphate derivatives **6** from dialkylphosphite **1** and indane-1,2,3-trione **2** in fairly good yields under neutral conditions (Scheme 3). We believe that the reported method offers a mild and simple route for the preparation of these derivatives. Its easy workup and reaction conditions make it a useful addition to modern synthetic methodologies. Other aspects of this process are under investigation.

EXPERIMENTAL

Starting materials and solvents were obtained from Merck (Germany) and Fluka (Switzerland) and were used without further purification. The methods used to follow the reactions were TLC and NMR; they indicated that there was no side product. IR spectra were measured on a Shimadzu IR-460 spectrometer. ^{1}H and ^{13}C NMR spectra were measured (in

$CDCl_3$ solution) with a Bruker DRX-250 AVANCE spectrometer at 250.0 and 62.5 MHz, respectively. Mass spectra were recorded on a Finnigan-MATT 8430 mass spectrometer operating at an ionization potential of 20 eV.

General Procedure for the Preparation of Compounds 4a–c

Trialkylphosphite derivative **1** (1 mmol) was added to indane-1,2,3-trione **2** (0.16 g, 1 mmol) at −10°C. The reaction mixture was allowed to warm up to room temperature and was magnetically stirred for 24 h at room temperature in solvent-free conditions. The viscous residue was purified by flash column chromatography (silica gel; petroleum ether/ethyl acetate, 1:1). The solvent was removed under reduced pressure, and the product **4** was obtained. The characterization data of the compounds are given below:

4,4,4-Trimethoxy-3,5-dioxa-4λ^5-phospha-tricyclo[6.4.0.0^{2,6}]dodeca-1(8),2(6),9,11-tetraen-7-one (4a). Yellow viscous oil; Yield: 65%. IR (KBr): $v = 1719$ (C=O), 1641, 1285, and 1028 cm^{-1}. ^1H NMR ($CDCl_3$): $\delta = 3.94$ (6H, d, $^3J_{PH} = 11.3$ Hz, $2OCH_3$, eq.); 4.34 (3H, s, OCH_3, ax.) and 7.11–7.36 (4H, m, arom).^{13}C NMR ($CDCl_3$): $\delta = 55.50$ ($2OCH_3$, d, $^2J_{POC} = 6.3$ Hz, eq.); 59.73 (OCH_3, ax.); 119.08, 121.14, 129.87 and 133.01 (4CH, arom); 121.86 (*C*=C—C=O, d, $^2J_{POC} = 8.3$ Hz, olefinic C, eq.); 127.62 and 137.00 (2C, arom); 161.30 (C=*C*—C=O, d, $^2J_{POC} = 6.0$ Hz, olefinic C, ax.) and 187.92 (C=O). ^{31}P NMR (CDCl3): $\delta = -2.64$ (s). MS: *m/z* (%) (EI) 285 (M^+ +1, 19), 284 (M^+, 100), 255 (14), 241 (30), 223 (11), 175 (10), 58 (32), 147 (45), 130 (50), 119 (4), 109 (99), 104 (57), 93 (12), 76 (44), 65 (5), 50 (17) and 41 (3).

4,4,4-Triethoxy-3,5-dioxa-4λ^5-phospha-tricyclo[6.4.0.0^{2,6}]dodeca-1(8),2(6),9,11-tetraen-7-one (4b). Yellow viscous oil; Yield: 52%. IR (KBr): $v = 1723$ (C=O), 1638, 1276, and 1030 cm^{-1}. ^1H NMR ($CDCl_3$): $\delta = 1.28$ (6H, t, $^3J_{HH} = 7.0$ Hz, $2CH_3$, eq); 1.37 (3H, t, $^3J_{HH} = 7.0$ Hz, CH_3 ax.); 4.12–4.29 (4H, m, $2CH_2$, eq.); 4.58–4.66 (2H, m, CH_2, ax.) and 7.03–7.26 (4H, m, arom). ^{13}C NMR ($CDCl_3$): $\delta = 15.21$ (CH_3, ax.); 15.98 ($2CH_3$, d, $^3J_{POCC} = 6.9$ Hz, eq.); 65.04 ($2CH_2$, d, $^2J_{POC} = 6.3$ Hz, eq.); 68.17 (CH_2, ax.); 118.89, 120.82, 129.63 and 132.80 (4CH, arom); 121.73 (*C*=C—C=O, d, $^2J_{POC} = 8.2$ Hz, olefinic C, eq.); 127.70 and 137.26 (2C, arom); 160.37 (C=*C*—C=O, d, $^2J_{POC} = 5.7$ Hz, olefinic C, ax.) and 187.87 (C=O). ^{31}P NMR (CDCl3): $\delta = -5.12$ (s). MS: *m/z* (%) (EI) 325 (78), 162 (100) and 104 (70).

4,4,4-Triisopropoxy-3,5-dioxa-4λ^5-phospha-tricyclo[6.4.0.0^{2,6}]dodeca-1(8),2(6),9,11-tetraen-7-one (4c). Yellow viscous oil, Yield: 57%. IR (KBr): $v = 1723$ (C=O), 1638, 1276, and 1015 cm^{-1}. ^1H NMR ($CDCl_3$): $\delta = 1.31$ (12H; dd, $^3J_{HH} = 6.0$ Hz,$^4J_{PH} = 3.0$ Hz, $4CH_3$, eq); 1.36 (6H, d, $^3J_{HH} = 6.0$ Hz, $2CH_3$, ax); 4.76–4.93 (2H, m, 2CH, eq); 5.33 (1H, sept, $^3J_{HH} = 6$ Hz, CH, ax); 7.06–7.28 (4H, m, arom).^{13}C NMR ($CDCl_3$): $\delta = 22.63$ ($2CH_3$, ax); 23.53 ($2CH_3$, d, $^3J_{POCC} = 5.7$ Hz, eq.); 23.60 ($2CH_3$, d, $^3J_{POCC} = 3.8$ Hz, eq.); 73.88 (2OCH, d, $^2J_{POC} = 6.3$ Hz, eq.); 74.81 (OCH, ax.); 118.81, 120.66, 129.50 and 132.69 (4CH, arom); 121.50 (*C*=C—C=O, d, $^2J_{POC} = 8.2$ Hz, olefinic C, eq.); 127.90 (C, arom); 137.82 (C, $^3J_{POCC} = 1.9$ Hz, arom); 159.19 (C=*C*—C=O, d, $^2J_{POC} = 6.3$ Hz, olefinic C, ax.); 188.07 (C=O). ^{31}P NMR (CDCl3): $\delta = -6.43$ (s). MS: *m/z* (%) (EI) 369 (M^+ +1, 3), 368 (M^+, 5), 326 (7), 284 (10), 242 (100), 225 (10), 186 (5), 162 (71), 149 (7), 133 (8), 104 (13), 88 (5), 76 (9), 43 (25), 41 (11).

General Procedure for the Preparation of Compounds 6a,b

Dialkylphosphite derivative **1** (1 mmol) was added to indane-1,2,3-trione **2** (0.16 g, 1 mmol) at −10°C. The reaction mixture was allowed to warm up to room temperature and then was magnetically stirred for 2 h at 70°C in solvent-free conditions. The yellow residue was washed with MeOH, and pure white solid products **6a** and **6b** were isolated via simple filtration. The characterization data of the compounds are given below:

2,3-Dioxo-2,3-dihydro-1*H*-inden-1-yl dimethyl phosphate (6a). White powder, mp 232.5°C (dec.), yield 65%. IR (KBr): $v = 1725$ (C=O), 1258 (C—O) and 1056 (C—O) cm^{-1}. ^1H NMR (DMSO-d_6): $\delta = 3.52$ (6 H, s, 2 OCH_3); 7.12 (1 H, bs, OCH) and 7.85–8.21 (4 H, m, arom). ^{13}C NMR (DMSO-d_6): $\delta = 49.60$ (2 OCH_3, d, $^2Jpoc = 6.9$ Hz); 73.38 (1CH, d, $^2Jpoc = 13.2$ Hz); 118.00, 118.26, 132.01 and 132.25 (4 CH, arom.); 135.78 and 135.99 (2 C, arom.), 186.68 and 190.24 (2 C=O). ^{31}P NMR (DMSO-d_6): $\delta = -2.75$ (s).

2,3-Dioxo-2,3-dihydro-1*H*-inden-1-yl diethyl phosphate (6b). White powder, mp 235.4°C (dec.), yield 63%. IR (KBr): $v = 1723$ (C=O), 1261 (C—O) and 1030 (C—O) cm^{-1}. ^1H NMR (DMSO-d_6): $\delta = 0.95$–1.06 (6 H, m, 2 CH_3); 3.63–3.76 (4 H, m, 2 OCH_2); 7.00 (1 H, bs, OCH) and 7.95–8.11 (4 H, m, arom). ^{13}C NMR (DMSO-d_6): $\delta = 15.94$ (2 CH_3, d, $^3J_{pocc} = 6.3$ Hz); 64.53 (2 OCH_2, d, $^2J_{poc} = 6.3$ Hz); 78.77 (1 CH, d, $^2J_{poc} = 13.2$ Hz); 123.32, 123.55, 137.21 and 137.48 (4 CH, arom.); 141.24 and 141.43 (2 C, arom.); 192.11 and 195.63 (2 C=O). ^{31}P NMR (DMSO-d_6): $\delta = -5.36$ (s).

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