Catalytic Synthesis of the Esters of Phosphorus Acids from White Phosphorus and Aliphatic or Aromatic Alcohols

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Abstract

The various esters of the phosphoric and phosphorous acids have been obtained directly from white phosphorus and aliphatic (or aromatic) alcohols under aerobic atmosphere in the presence of the CuX2 or FeX3 (X = Cl, NO3, C3H7CO2) salts. Irrespective of the variable nature of the used alcohols and catalysts, trialkyl(aryl) phosphates and dialkyl phosphites are a major products, whereas trialkyl(aryl) phosphites and dialkyl phosphates are a minor products of the phosphorylation process. Thanks to the presence of catalysts, the possible side reaction route of the radical chain oxidation of white phosphorus by oxygen to phosphorus oxides has been precluded. A comparison between the catalytic properties of CuX2 and FeX3 has been done. Although both of them have been found an efficient catalysts for the syntheses, the Cu(II) salts are active at 50-65°C, whereas the Fe(III) based catalytic systems become competitive in terms of catalytic efficiency when reaction is carried out at 70-90°C. Aromatic alcohols are characterised by less reactivity in this catalytic reaction as compared with an aliphatic ones. The same coordinative redox mechanism of the oxidative P-O coupling of P4 to ROH in the presence of both Cu(II) and Fe(III) catalysts has been proposed. Relevant steps of the catalytic cycle including the complexation of both white phosphorus and alcohol molecules to metal ion, the reduction of catalyst by white phosphorus, and the oxidation of reduced form of catalyst by oxygen have been also considered.

Introduction

White phosphorus, P4, is a precursor of many useful organophosphorus compounds including the esters of phosphorus acids that are produced in multitons amount and are widely employed in different technological areas as extractors of rare and radioactive elements, flame-retardants for plastic materials, additives for combustible and lubricating materials, bioactive substances for further elaboration in pharmaceutical and agricultural industries, etc. The traditional way for the synthesis of the esters is based on the preliminary oxidative chlorination of P4 followed by the substitutive P-O coupling of phosphorus chlorides with alcohols or phenols [1]. This productive cycle demands consumption of toxic Cl2 and causes the evolution of HCl, which has a negative influence on the product yield because may split the resulting organophosphorus compounds to undesired by-products. In consequence of neutralization, HCl is converted to inorganic chlorides, whose removal from waste may still cause problems in large-scale production plants. Moreover, the present methods of air purification do not secure a cheap and complete removal of both Cl2 and HCl impurities from the technological workshops air. For the reasons, the search for an alternative technology, which does not use chlorine for the preparation of organophosphorus compounds, is attracting considerable industrial and environmental interest.

We have first proposed an original “chlorine-free” method of the oxidative alko(aro)xylation of white phosphorus under mild reaction conditions in the presence of CuX2 or FeX3 salts (X = Cl, Br, NO3, CH3CO2, C3H7CO2, C17H35CO2) which uses dioxygen as a cheap and efficient oxidant [2-5]. The new catalytic protocol allows to remove both Cl2 and HCl from the productive cycle and to incorporate into the catalytic cycle all the advantages of a homogeneous catalytic reac-
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As both P₄ and catalyst are taken in the reaction in the dissolved forms. Depending on the reactions conditions, four types of organophosphorus derivatives bearing P-O bonds are formed and identified by chromatography and NMR: trialkyl(aryl) phosphate PO(OR)₃, dialkyl phosphite P(O)H(OR)₂, dialkyl phosphate P(O)(OH)(RO)₂, and trialkyl(aryl)phosphite P(OR)₃ (see Scheme 1).

\[
\begin{align*}
\text{P} & + 12\text{ROH} + 5\text{O}_2 \rightarrow \text{Catalyst} \rightarrow \text{PO(OR)}_3 + 6\text{H}_2\text{O} \\
\text{P} & + 8\text{ROH} + 3\text{O}_2 \rightarrow \text{Catalyst} \rightarrow \text{P(O)H(OR)}_2 + 2\text{H}_2\text{O} \\
\text{P} & + 8\text{ROH} + 5\text{O}_2 \rightarrow \text{Catalyst} \rightarrow \text{P(O)(OH)(RO)}_2 + 2\text{H}_2\text{O} \\
\text{P} & + 12\text{ROH} + 3\text{O}_2 \rightarrow \text{Catalyst} \rightarrow \text{PO(OR)}_3 + 6\text{H}_2\text{O}
\end{align*}
\]

Scheme 1.

It has been shown by the volumetry method [2-5] that the experimental \( \text{O}_2/\text{P}_4 \) ratio between amounts of oxygen and phosphorus consumed is around 3-5, \textit{i.e.} close to the stoichiometry of the above reactions. Thus, it appeared interesting to conduct the reaction at a preparative scale, to get more precise information about the type of organophosphorus compounds formed and the product composition. We report here several experiments concerning the reaction of white phosphorus with aliphatic and aromatic alcohols in the presence of air and different Cu(II) and Fe(III) salts which were carried out at a laboratory scale (grams) in order to isolate the individual products and characterise them by NMR.

**Experimental**

**Starting materials**

The most attention has been given to drying of reagents because H₂O stimulates the side reaction of the oxidative hydroxylation of P₄ to phosphoric acid. Air was dried over CaCl₂. The alcohols and arenes were purified and dried by the usual methods. The catalysts were used after drying by heating when possible. Since iron(III) and copper(II) nitrates are not stable under heating, these catalysts were used as received. The solid P₄ was weighed in a beaker under water, dipped into two successive beakers containing ethanol and arene and then dissolved in a dried arene at 45°C. The concentration of P₄ in the solution was determined by iodometry.

**Caution**

White phosphorus is flammable and explosive in air and must be handled as soon and carefully as possible. It is known that bubbling oxygen through solutions of P₄ may result in violent explosions. However, no explosions occurred during these experiments,
thanks to the presence of catalysts. Nevertheless, it is strongly recommended to conduct these reactions behind shields.

**Products analysis**

Nuclear magnetic resonance (NMR) spectra of the organophosphorus esters were recorded on Bruker WM-250 and AC-200 spectrometers at 25°C, and on AC-80 at 35°C. Chemical shifts are expressed in ppm upfield from Me₄Si (H and ³¹C) and 85% H₃PO₄ (³¹P). Coupling constants (J) are given in hertz (Hz). In order to eliminate paramagnetic CuCl₂ from the crude reaction solutions, Na₂SO₃ (3 to 10 g) was added, and the resulting suspension was stirred overnight. Such procedure did not removed paramagnetism when FeCl₃ was used; thus the crude reaction products were not analyzed by ³¹P NMR in these cases. Elemental analysis was obtained on Perkin-Elmer Model 2400.

Gas chromatography analysis were recorded on Chrompack 9002 chromatograph equipped with flameionization detector and the capillary columns CP SIL 19CB (25m × 0.25mm) and CP SIL 5CB (10m × 0.25mm).

**Typical procedure**

A round three-neck flask (with volume about 250 cm³) fitted with a refluxing condenser and a gas-inlet tube for air barbotage was used for catalytic synthesis of organophosphorus products. The constant reaction temperature (65-90°C) was supported with an oil bath, under vigorous magnetic stirring. An arene solution of P₄ (30-100 mL) was portionally added to the alcohol solution (10-180 mL) containing the catalyst by syringe through a rubber plug during 5-30 hours. The gradual addition of P₄ was aimed to prevent the formation of white smoke of P₂O₅ and P₄O₁₀.

The rate of P₄ conversion was slowly decreased from portion to portion because the catalytic solution was gradually diluted by solvent (arene). The flow rate of air barbotage was 80-120 mL/min. The reactions were monitored by GC and ³¹P NMR (when applicable), which indicates in most cases the formation of several products in variable amounts. After completing the synthesis, the catalyst was precipitated by K₂CO₃ and filtered from the solution. High vacuum distillation (BUCHI GKR-51) of dark oil remaining after stripping off the excess of alcohol and arene (the rotation evaporator ER-1M2) yields the individual organophosphorus products as colourless oils.

### Physical and spectroscopic data

1a. Tributyl phosphate: bp. 130-132°C (2 mm Hg); ³¹P{¹H}NMR (CDCl₃): δ -0.51 ppm. ³¹P NMR (CDCl₃): δ -0.50 (sep., 3J_P_O = 6.8 Hz) ppm. ¹H NMR (CDCl₃): δ 3.92 (m, 6H, CH₂ α), 1.56 (m, 6H, CH₂ β), 1.30 (m, 6H, CH₃ γ), 0.83 (m, 9H, CH₃) ppm. ¹³C{¹H}NMR (CDCl₃): δ 67.24 (d, 3J_CP = 5.9 Hz, CH₂ α), 32.12 (d, 3J_CP = 6.0 Hz, CH₂ β), 18.53 (s, CH₃ γ), 13.42 (s, CH₃) ppm. Anal. Calcd for C₅H₁₀O₃P: C, 54.07; H, 10.10. Found: C, 54.70; H, 10.10.

2a. Dibutyl phosphite: bp. 110°C (5 mm Hg); ³¹P{¹H}NMR (CDCl₃): δ 7.94 ppm. ³¹P NMR (CDCl₃): δ 7.9 (dt, 3J_P_H = 692 Hz, 3J_P_O-CH₂ = 8.5 Hz) ppm. ¹H NMR (CDCl₃): δ 6.7 (d, 3J_H_H = 692 Hz, 1H, H-β), 3.95 (m, 4H, CH₂ α), 1.55 (m, 4H, CH₂ β), 1.31 (m, 4H, CH₂ γ), 0.84 (t, 3J_H_H = 7.2 Hz, 6H, CH₃) ppm. ¹³C{¹H}NMR (CDCl₃): δ 65.39 (d, 3J_CP = 5.8 Hz, CH₂ α), 32.21 (d, 3J_CP = 5.9 Hz, CH₂ β), 18.57 (s, CH₂ γ), 13.37 (s, CH₃) ppm. Anal. Calcd for C₅H₁₀O₃P: C, 49.5; H, 9.8. Found: C, 49.20; H 9.79.

3a. Dibutyl phosphate: bp. 100°C (10¹¹ mm Hg); ³¹P{¹H}NMR (CDCl₃): δ 0.88 ppm. ¹H NMR (CDCl₃): δ 11.8 (br s, 1H, OH), 3.97 (m, 4H, CH₂ α), 1.60 (m, 4H, CH₂ β), 1.38 (m, 4H, CH₂ γ), 0.89 (t, 3J_H_H = 7 Hz, 6H, CH₃) ppm. ¹³C{¹H} NMR (CDCl₃): δ 67.18 (d, 3J_CP = 6.0 Hz, CH₂ α), 32.06 (d, 3J_CP = 7.8 Hz, CH₂ β), 18.56 (s, CH₂ γ), 13.48 (s, CH₃) ppm. Anal. Calcd for C₅H₁₀O₃P: C, 45.7; H, 9.0. Found: C, 46.33; H 9.29.

1b. Tri-isopropyl phosphate: bp. 83-84°C (5 mm Hg); ³¹P{¹H}NMR (THF-d₈): δ 0.97 ppm. ¹H NMR (THF-d₈): δ 4.55 (m, 3H, CH α), 1.27 (d, 3J_H_H = 6.2 Hz, 18H, CH₃) ppm. ¹³C{¹H}NMR (THF-d₈): δ 72.28 (d, 3J_CP = 5.8 Hz, CH α), 24.38 (d, 3J_CP = 4.0 Hz, CH₃) ppm.

2b. Di-isopropyl phosphate: bp. 91-92°C (20 mm Hg); ³¹P{¹H} NMR (CDCl₃): δ 4.6 ppm. ³¹P{¹H} NMR (CDCl₃): δ 4.6 (d, 3J_H_H = 690 Hz) ppm. ¹H NMR (CDCl₃): δ 6.72 (d, 3J_H_H = 687 Hz, 1H, H-P), 4.6 (m, 2H, CH), 1.24 (d, 3J_H_H = 6.2 Hz, 12H, CH₃) ppm. ¹³C{¹H} NMR (CDCl₃): δ 70.5 (d, 3J_CP = 6 Hz, CH), 23.6 (br d, 3J_CP = 5 Hz, CH₃) ppm.

3b. Di-isopropyl phosphate: bp. 94°C (10¹⁴ mm Hg); ³¹P{¹H}NMR (THF-d₈): δ 2.98 ppm. ³¹P{¹H}NMR (THF-d₈): δ 8.8 (s, 1H, HO-P), 4.54 (m, 2H, CH α), 1.28 (d, 3J_H_H = 6.1 Hz, 12H, CH₃) ppm. ¹³C{¹H}NMR (THF-d₈): δ 72.43 (d, 3J_CP = 5.9 Hz, CH α), 24.25 (d, 3J_CP = 4.5 Hz, CH₃) ppm.

1d. Triphenyl phosphate: bp. 244°C (10 mm Hg);
Results and discussion

We have carried out reactions between white phosphorus and alcohols under aerobic atmosphere in the presence of two types of catalysts, either CuX$_2$ or FeX$_3$. Table 1 summarises the conditions used for all the experiments and the results. It should be emphasised that no organophosphorus products but only phosphorus oxides such as P$_4$O$_6$ and P$_4$O$_{10}$ are yielded in the absence of the catalysts. In order to fasten the phosphorylation reactions, the metal salt is used in a large amount, between 0.7 and 4.0 equivalent of metal salt for each P$_4$. Such a large amount of catalyst is needed for productivity and safety reasons. Indeed, P$_4$ is introduced in the reaction under aerobic conditions, and the only way to preclude its radical chain reaction with O$_2$, which affords various phosphorus oxides, is to use also the catalyst as electron receptor.

**Catalyst CuX$_2$**

At using the CuX$_2$ catalysts, the reaction solution is characterised by a versatile colour in the course of the experiment. The initial transparent green alcohol solution of CuCl$_2$ is immediately converted in a turbid brown at adding the arene solution of P$_4$ at 60°C.

In the course of air barbbling, the reaction solution is gradually clearing up to a colourless solution including white residue of CuCl. Finally, the residue is gradually disappeared, afterwards the catalytic solution is again turned in a transparent green one, as at the beginning of the reaction. The catalytic solution colour is determined by the correlative rates of the reduction of Cu(II) by P$_4$ and the oxidation of Cu, Cu(I) by oxygen. White smoke of phosphorus oxides above the catalytic solution is not observed. This means that the branched-chain route of the P$_4$ oxidation in the gas phase is precluded.

### Table 1

| Run | Catalytic solution | Phosphorus solution | Temp. °C | Time hr | Compounds isolated g (mmol, %) |
|-----|--------------------|---------------------|----------|---------|---------------------------------|
| 1   | BuOH 140           | CuCl$_2$ 3.5 (26.0) | Benzene 94 | 60      | 1a 13.6 (51.0; 87.9), 2a (traces) |
| 2   | BuOH 20            | CuCl$_2$ 3.0 (22.3) | Toluene 50 | 65      | 1a 4.3 (16.1; 50.3), 2a 1.0 (5.1; 15.9), 3a 0.6 (2.8; 8.7) |
| 3   | 95% EtOH 150       | CuCl$_2$.2H$_2$O 3.0 (17.6) | Toluene (95) | 50      | 1a 3.7 (20.3; 52.8), 2a (traces) |
| 4   | i-PrOH 20          | CuCl$_2$ 2.0 (14.9) | Toluene (30) | 65      | 1b 1.9 (8.5; 37.9), 2b 0.5 (3.0; 13.4), 3b 0.2 (1.1; 4.9) |
| 5   | tert-BuOH 20       | CuCl$_2$ 3.0 (22.3) | Toluene (30) | 55      | 1c, 2c, 4c (5/2/1, not separated) |
| 6   | i-AmOH 150         | Cu(NO$_3$)$_2$.3H$_2$O 3.0 (12.4) | Toluene (100) | 65      | 1a 7.4 (24.0; 62.5), 2a 1.1 (4.9; 12.8) |
| 7   | BuOH 150           | CuC$_3$H$_7$CO$_2$.2H$_2$O 5.0 (21.0) | Toluene (100) | 65      | 1a 10.2 (38.3; 84.7), 2a 0.6 (3.0; 6.6) |
| 8   | i-AmOH 150         | FeCl$_3$ 3.0 (11.1) | Benzene (100) | 70      | 1a 8.2 (26.6; 43.4), 2a 2.9 (13.0; 21.2) |
| 9   | BuOH 150           | FeCl$_3$ 4.0 (14.8) | Toluene (60) | 80      | 1a 7.2 (26.9; 59.7), 2a 3.0 (15.6; 34.5) |
| 10  | i-AmOH 180         | FeCl$_3$ 3.0 (11.1) | Benzene (90) | 90      | 1a 12.3 (39.8; 72.6), 2a 2.8 (12.6; 23.0) |
| 11  | i-AmOH 150         | Fe(NO$_3$)$_3$.9H$_2$O 5.0 (12.4) | Toluene (70) | 70      | 1a 0.2 (0.6; 1.5), 2a 1.4 (6.3; 16.4) |
| 12  | PhOH 6.2 g         | FeCl$_3$.I$_2$.0.5 (3.0)-0.24 (0.9) | Toluene (20) | 80      | 1d 1.2 (3.6; 28.1) |
Catalyst FeX₃

The iron-based systems become competitive in terms of catalytic efficiency, when the phosphorylation reactions are carried out at 70-90°C. The reaction temperature dependence of the product composition should also be noted at using the FeX₃ catalysts. At 70°C, the initial light-brown colour of alcohol solution of FeCl₃ is not practically changed neither at the moment of P₄ addition, nor in the course of air babbaging. Some white smoke of phosphorus oxides is observed above the catalytic solution. When the reaction is conducted at 80 or 90°C (runs 9, 10), the reaction solution colour turns into a dark-brown and the white smoke is disappeared. At 90°C, the oxidative alkoxylation of P₄ results in higher yields of 1a and 2a (72.6% and 23.0%; run 10) as compared to reaction carried out at 70°C (43.4% and 21.2%; run 8).

The same products are formed in the presence of iron nitrate although the product yields are lower. The addition of toluene solution of P₄ to alcohol solution of Fe(NO₃)₃ ⋅ 9H₂O leads to appearance of the white smoke of phosphorus oxides above the solution and to formation of an yellow precipitate (presumably, the iron hydroxycomplexes) which are not catalytically active in this reaction. The product yields are therefore low, only 1.5% for 1a and 16.4% for 2a (run 11). Nevertheless, the identity of esters of phosphorus acids obtained from P₄ and alcohols in the presence of both chloride-including and chloride-free catalysts (CuCl₂, Cu(NO₃)₂, Cu(C₃H₇CO₂)₂, FeCl₃, Fe(NO₃)₃) allow us to exclude a probable reaction route via formation of PCl₃ in situ followed by its alcoholysis and to suppose a coordinative mechanism of the reaction.

Finally, we tried to extend the phosphorylation reactions to an aromatic alcohol, PhOH, but we did not succeed in isolating a product when FeCl₃ alone was used as catalyst. This may be explained by less nucleophilicity of the phenoxide anion and its stronger association in organic solutions as compared with alkoxide one [6]. Thus, a promoting additive is needed to carry out the coupling reaction of P₄ with phenol. We used iodine as a reversible co-oxidant whose reduced form (iodide) can be reoxidized by FeCl₃.
adding the toluene solution of \( P_4 \) into a viscous mixture of \( \text{PhOH}, \ FeCl_3, \) and \( I_2 \) at 80°C followed by the air babbling, the initial red-brown solution is not underwent a visible change and the white smoke of a phosphorus oxides is not observed above the solution. Both triphenyl phosphate 1d and triphenyl phosphite 4d are determined by gas chromatography during reaction, however, only 1d (28.1%) is isolated by high vacuum distillation as a yellow fastly thickening oil (run 12).

Thus, one can notice that catalytic reactions of the oxidative alko(aro)xylation of white phosphorus are quite innovative and not characteristic for the known chemistry of elemental phosphorus. The novelty and potentiality of the catalytic functionalisation of white phosphorus is confirmed by the patent [7] illustrating this approach.

Despite of a variable nature of the catalytic systems and alcohols used, the experimental data and products composition indicate to a general features of the oxidative P-O coupling of \( P_4 \), with aliphatic and aromatic alcohols described in Scheme 2. Relevant steps of the process may be considered: (i) the oxidative alko(aro)xylation of \( P_4 \) to \( P(OR)_{3\,4} \), (ii) the oxidation of \( P(OR)_{3\,4} \) to \( PO(OR)_{3\,1} \), (iii) the dealkylation of \( P(OR)_{3\,4} \) to \( P(O)H(OR)_{2\,2} \), and (iv) the oxidation of \( P(O)H(OR)_{2\,2} \) to \( P(O)(OH)(OR)_{3\,3} \).

In their turn, the steps (i), (ii), and (iv) with \( \text{CuX}_2 \) and \( \text{FeX}_3 \) proceed via the repeated stages of the complexation of both ROH and \( P_4 \) (as well as by 4 or 2) to metal ion, the reduction of catalyst by \( P_4 \) (as well as by 4 or 2), and its reoxidation by oxygen. In the presence of the catalytic \( \text{FeCl}_3-I_2 \) system, the stages of the oxidation of \( P_4 \) by \( I_2 \) followed by the reduction of Fe(III) by \( I^- \) and the oxidation of Fe(II) by oxygen are supposed. It should be mentioned that the above stages of the complexation of reagents to Cu(II) and Fe(III) ions followed by the oxidative P-O coupling of white phosphorus to alcohol have been established and studied by the kinetic, potentiometric, thermodynamic, IR, ESR [2-5] and quantum chemical CNDO [8,9] methods. Dealkylation of \( \text{PO}(OR)_3 \) occurs as a result of interaction with water or acid both generated \textit{in situ}. The products composition is determined by the rates of the key steps (i-iv). The predominant formation of 1 with \( \text{CuX}_2 \) and mixed \( \text{FeCl}_3-I_2 \) system points to the relatively higher step (ii) velocity. The step (iv) seems to be accelerated at using of the primary and secondary aliphatic alcohols.

**Conclusion**

Thus, we have shown that the new processes of the oxidative alko(aro)xylation of \( P_4 \) catalysed by the \( \text{CuX}_2 \) or \( \text{FeX}_3 \) based catalytic systems allows to synthesize an organophosphorus derivatives which have a high added value directly from cheap and abundant white phosphorus, aliphatic or aromatic alcohols and oxygen under mild reaction conditions. The variable nature of the used alcohols and transition metal salts does not dramatically change the type of reactions observed. In all cases, compounds bearing P-O bonds are obtained, only the ratio of the various P-O compounds changes. Aromatic alcohols are characterised by less reactivity in this catalytic reaction as compared with an aliphatic ones. Both \( \text{CuX}_2 \) and \( \text{FeX}_3 \) are found an efficient catalysts for the syntheses, however copper(II) salts exhibit the most catalytic activity at 50-65°C, whereas the iron(III) based catalytic
systems become competitive and give better results in terms of catalytic efficiency when reaction is carried out at 70-90°C. On the basis of our present and previously published results, we have proposed that both Cu(II) and Fe(III) catalysts entail the same coordinative redox mechanism comprised of the major stages of the complexation of both P₄ (or its derivative) and ROH to metal ion, the reduction of catalyst by P₄ (or its derivative), liberating organophosphorus products, and the oxidation of reduced form of catalyst by oxygen. Work is in progress to achieve optimisation of the catalytic production of the esters of phosphorus acids through the metal-mediated functionalisation of white phosphorus.

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