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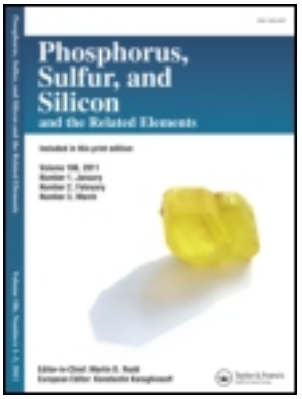

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Reaction of Red Phosphorus with Allylbenzene in Superbasic System KOH-DMSO

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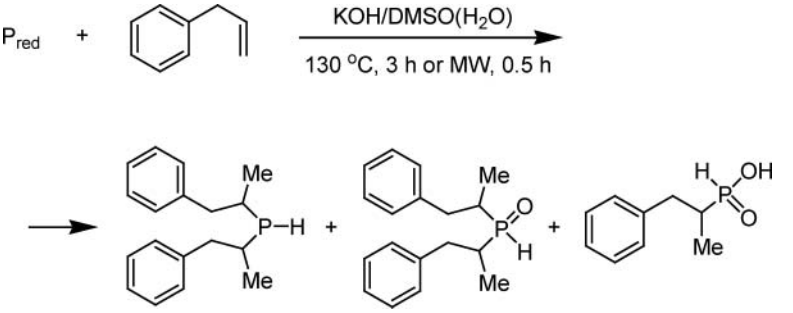
GRAPHICAL ABSTRACT



Abstract *Red phosphorus reacts with allylbenzene in the superbase system KOH-DMSO (130° C, 3 h, Ar) to give a mixture of bis(1-methyl-2-phenylethyl)phosphane (**1**), bis(1-methyl-2-phenylethyl)phosphane oxide (**2**), and 1-methyl-2-phenylethylphosphinic acid (**3**). Secondary phosphane oxide **2** and phosphinic acid **3** have been isolated from this mixture in 35% and 32% yield, respectively. Microwave activation of the reaction (200 W, 30 min) affords secondary phosphane **1** as the main product in 48% yield.*

Keywords Allylbenzene; microwave irradiation; phosphinic acid; red phosphorus; secondary phosphane; secondary phosphane oxide; superbase system

INTRODUCTION

In recent years, the cleavage of the P–P bond of elemental phosphorus under the action of nucleophiles has been studied.[1] Fundamentally, these investigations essentially supplement the existing paradigms in phosphorus chemistry. From the viewpoint of synthetic

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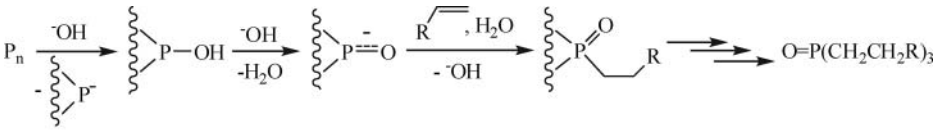
methodology, they open the shortest and environmentally benign ways to the practically important organophosphorus compounds.[1] The possibility of preparative use of phosphorus-centered nucleophiles produced by the cleavage of P–P bonds of white phosphorus under the action of hydroxide anions was reported for the first time in 1963.[2] Successful attempts to utilize the less reactive but nontoxic red phosphorus in organophosphorus synthesis were made by Bogolyubov and Petrov[3] and Bornancini et al.[4] who accomplished the cleavage of P–P bond of three-dimensional macromolecule of red phosphorus in alkali metal–liquid ammonia systems. A direct synthesis of tristyrylphosphane[5] from phenylacetylene and red phosphorus in the KOH-HMPA superbasic system laid the foundation of the systematic application of phosphorus-centered nucleophiles for the preparation of unavailable or unknown organophosphorus compounds. For example, a facile one-pot synthesis of tertiary phosphane oxides by nucleophilic addition of phosphinite anions to vinylarenes (styrenes,[6] 2-vinylnaphthalene[7]) or vinylhetarenes[8] has been developed (Scheme 1).



Scheme 1

At the same time, the literature lacks data on phosphorylation of allylarenes with elemental phosphorus, though it is known that allylbromide reacts with red phosphorus in the system KOH-H_2O-dioxane to give a mixture of tri(propen-2-yl)- and tri(propen-1-yl)phosphane oxides.[9]

In this article, we report for the first time on the reaction of red phosphorus with allylbenzene in superbasic system KOH-DMSO.

RESULTS AND DISCUSSION

We found that heating (130 °C, 3 h, argon blanket) red phosphorus and allylbenzene in the KOH-DMSO suspension (including ~ 0.1 mass% of hydroquinone as a radical processes inhibitor) afforded a mixture of bis(1-methyl-2-phenylethyl)phosphane (**1**), bis(1-methyl-2-phenylethyl)phosphane oxide (**2**), and potassium salt of 1-methyl-2-phenylethylphosphinic acid in a ratio 2:1:3 (^{31}P NMR). Free 1-methyl-2-phenylethylphosphinic acid (**3**) was isolated from the acidified aqueous layer in 32% yield (Scheme 2).

The reaction mixture obtained was diluted with water and extracted with chloroform. After removal of chloroform, a mixture of secondary phosphane **1** and phosphane oxide **2** was exposed in air (24 h, r.t.) to give secondary phosphane oxide **2** in 35% yield.

Microwave-assisted reaction of red phosphorus with allylbenzene not only shortened the process duration, but it affected noticeably its chemoselectivity. So, a microwave irradiation (200 W, 30 min) promoted version of the reaction (with the same reactants ratio) gave mainly secondary phosphane **1** in 48% yield. In addition, the reaction delivered phosphinic acid **3** (after acidic treatment) in 9% yield (Scheme 3).

In the absence of KOH, no reaction occurred. This fact confirms the nucleophilic mechanism of the addition, which can be presented as follows (Scheme 4): In the first

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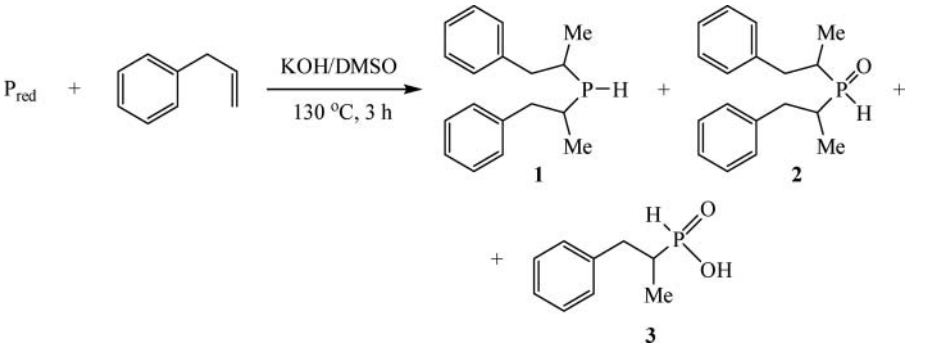
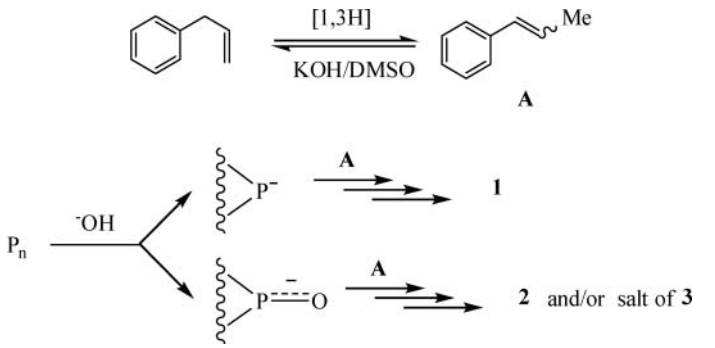


Scheme 2

Scheme 3



Scheme 4

stage, due to the [1,3H] isomerization of allylbenzene under the action of the superbase, 1-phenylprop-1-ene (**A**) is formed. The latter reacts with phosphorus-centered nucleophiles generated by cleavage of the P–P bond in the presence of hydroxide anions. Nucleophilic addition of phosphide- and phosphinite anions to the double bond of **A** results in phosphane **1** and phosphane oxide **2** (or acid salt **3**), respectively.

The isolation of intermediate *E*-1-phenylprop-1-ene (**A**) from the reaction mixture supports the above mechanism.

According to the NMR data (^{1}H, ^{13}C, and ^{31}P), secondary phosphane **1** and phosphane oxide **2** have been formed as a mixture of diastereomers. So, in the ^{31}P NMR spectra of phosphine **1**, three signals at –28.3, –22.8, and –17.4 ppm, in a ratio of 1:4:1 (d, $^{1}J_{PH}$ 207.3, 180.4, and 199.2 Hz); in the ^{31}P NMR spectra of phosphine oxide **2**, three signals at 53.7, 49.7, and 47.3 ppm, in a ratio of 1:3:1 (d, $^{1}J_{PH}$ 438.7, 439.5, and 434.0 Hz) are

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observed. The most intense signal (–22.8 and 49.7 for **1** and **2**, respectively) corresponds to the resonance of the major diastereomer where the configuration of the chiral carbon atoms is identical (S,S or R,R). The second pair of diastereomers (RS, SR), in which the configuration of the chiral carbon in the substituents is different, is presented by two weak signals at –17.4 and –28.3 (for **1**), and at 53.7 and 49.7 (for **2**). The NMR spectra of minor and major diastereomers of secondary phosphane **1** and phosphane oxide **2** are given in the Experimental section.

It is worth noting that secondary phosphane **1** did not react with elemental sulfur or selenium, though its structural congener, bis(2-phenylpropyl)phosphane, under the same conditions readily formed secondary phosphane chalcogenides.[10] The inactivity of secondary phosphane **1** is likely due to the presence of sterically hindered secondary alkyl groups at the phosphorus atom.

In summary, the reaction of red phosphorus with allylbenzene in the system KOH-DMSO has been accomplished for the first time. When applied to other allylarenes, this reaction represents a simple and efficient pathway to novel chiral secondary phosphanes, phosphane oxides, and phosphinic acids with sterically hindered moieties, possible ligands for metal complex catalysts,[11] and reactive building blocks for the synthesis of important tertiary phosphines and corresponding phosphine chalcogenides,[12] as well as intermediates and coordinating solvents for the preparation of conductive nanomaterials.[13]

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EXPERIMENTAL

IR spectra were measured with a Bruker IFS 25 instrument in microlayer or KBr (cm^{-1}). ^{1}H, ^{13}C, and ^{31}P NMR spectra were recorded on a Bruker DPX-400 spectrometer (400.13, 100.61, and 161.98 MHz, respectively) in C_6D_6 solution and referenced to internal HMDS (^{1}H), C_6D_6 (^{13}C), and external 85% H_3PO_4 (^{31}P). 2D homo- and heteronuclear NMR correlation experiments (NOESY, HSQC) were used to assign the signals in ^{1}H and ^{13}C NMR spectra. Microwave mirradiation was performed in a Microwave oven, Samsung M181DNR (max power level 850 W) equipped with reflux condenser. The brand KSAN "SIA" red phosphorus was employed. Brand DMSO (1% water content) was used. All reactions were conducted under argon atmosphere.

Synthesis of Secondary Phosphane Oxide 2 and Phosphinic Acid 3

A mixture of red phosphorus (3.10 g, 100 mmol), allylbenzene (4.71 g, 40 mmol), $KOH{\cdot}0.5H_2O$ (7.00 g, 108 mmol), DMSO (30 mL), and hydroquinone (0.14 g) was stirred for 2 h at 130°C, cooled, and analyzed by ^{31}P NMR. The mixture contained secondary phosphanes **1**, phosphane oxide **2**, and potassium salt of 1-methyl-2-phenylethylphosphinic acid (**3**) in a 2:1:3 ratio, respectively. The mixture was diluted with water (30 mL) and extracted with $CHCl_3$ (3 × 20 mL). The chloroform extract was washed with a 20% aq solution of KCl (3 × 15 mL) and dried over K_2CO_3. The solvent was removed under reduced pressure. The residue was analyzed by ^{31}P NMR. The mixture contained secondary phosphanes **1** and phosphane oxide **2** in a 2:1 ratio. The mixture was exposed to air for 24 h, washed with hexane, and dried in vacuo to give phosphane oxide **2**. The aqueous layer of the reaction mixture was acidified with a 35% aq HCl up to pH 4–5 and extracted with chloroform (3 × 20 mL). The chloroform extract was washed with water (3 × 15 mL)

and dried over $CaCl_2$. The solvent was removed, and the residue was reprecipitated from chloroform with hexane and dried in vacuo to give phosphinic acid **3**.

Bis(1-methyl-2-phenylethyl)phosphane Oxide (2). Colorless viscous oil, 2.00 g (35%) yield. Major diastereomer: ^{1}H NMR, δ (ppm), J (Hz): 0.91 (dd, 6H, $^3J_{HH}$ = 7.1, $^3J_{PH}$ = 15.4, Me), 1.65 (m, 2H, CHMe), 2.44–3.06 (m, 1H, CH_2Ph), 2.44–2.80 (m, 1H, CH_2Ph), 6.11 (ddd, 1H, $^1J_{PH}$ = 439.5, $^3J_{HH}$ = 4.7 and 1.5, PH), 6.89–7.16 (m, 10H, Ph). ^{13}C NMR, δ (ppm), J (Hz): 12.4 (Me), 32.8 (d, $^1J_{PC}$ = 62.6, CHP), 35.5 and 37.2 (CH_2Ph), 128.8 and 128.7 (C-*o*, C-*m*, Ph), 126.7 (C-*p*, Ph), 139.3 (d, $^3J_{PC}$ = 11.8, C-*i*, Ph). ^{31}P NMR, δ (ppm): 49.7 (d, $^1J_{PH}$ = 439.5 Hz). Minor diastereomer 1: ^{1}H NMR, δ (ppm), J (Hz): 0.56 (dd, 6H, $^3J_{HH}$ = 7.2, $^3J_{PH}$ = 19.0, Me), 1.80 (m, 2H, CHMe), 2.30–3.16 (m, 2H, CH_2Ph), 6.08 (dt, 1H, $^1J_{PH}$ = 436.7, $^3J_{HH}$ = 3.6, PH), 6.89-7.16 (m, 10H, Ph). ^{13}C NMR, δ (ppm), J (Hz): 12.0 (Me), 32.2 (d, $^1J_{PC}$ = 63.4, CHP), 35.7 (CH_2Ph), 129.6 and 129.6 (C-*o*, C-*m*, Ph), 126.3 (C-*p*, Ph), 139.3 (d, $^3J_{PC}$ = 10.7, C-*i*, Ph). ^{31}P NMR, δ (ppm): 47.3 (d, $^1J_{PH}$ = 434.0 Hz). Minor diastereomer 2: ^{1}H NMR, δ (ppm), J (Hz): 0.71 (dd, 6H, $^3J_{HH}$ = 7.1, $^3J_{PH}$ = 18.6, Me), 1.68 (m, 2H, CHMe), 2.30-2.70 (m, 2H, CH_2Ph), 6.16 (dt, 1H, $^1J_{PH}$ = 440.5, $^3J_{HH}$ = 2.6, PH), 6.89-7.16 (m, 10H, Ph). ^{13}C NMR, δ (ppm), J (Hz): 13.2 (Me), 32.6 (d, $^1J_{PC}$ = 63.4, CHP), 36.9 (CH_2Ph), 129.3 and 129.4 (C-*o*, C-*m*, Ph), 126.3 (C-*p*, Ph), 139.0 (d, $^3J_{PC}$ = 10.7, C-*i*, Ph). ^{31}P NMR, δ (ppm): 53.7 (d, $^1J_{PH}$ = 438.7 Hz). IR ν (cm^{-1}): 3374, 3293, 3107, 3085, 3062, 3027, 3002, 2966, 2929, 2874, 2853, 1603, 1495, 1454, 1379, 1242, 1198, 1155, 956, 913, 862, 753, 728, 700, 600, 586, 507, 481. Anal. Calcd. for $C_{18}H_{23}OP$: C, 75.50; H, 8.10; P, 10.82. Found: C, 75.34; H, 8.15; P, 10.74.

1-Methyl-2-phenylethylphosphinic acid (3). Colorless viscous oil, 2.35 g (32%) yield. ^{1}H NMR, δ (ppm), J (Hz): 0.93 and 0.95 (dd, 3H, $^3J_{HH}$ = 7.1, $^3J_{PH}$ = 19.2, Me), 1.87–1.95 (m, 1H, CHP), 2.34–2.43 (m, 1H, CH_2Ph), 3.06–3.14 (m, 1H, CH_2Ph), 6.90 (d, 1H, $^1J_{PH}$ = 535.0, PH), 6.92-7.08 (m, 5H, Ph), 13.56 (br. s, 1H, OH). ^{13}C NMR, δ (ppm), J (Hz): 10.9 (Me), 34.9 (CH_2Ph), 35.0 (d, $^1J_{PC}$ = 95.6, CHP), 126.6 (C-*p*, Ph), 128.7 (C-*o*, Ph), 129.4 (C-*m*, Ph), 139.0 (d, $^3J_{PC}$ = 14.1, C-*i*, Ph). ^{31}P NMR, δ (ppm): 40.0 ($^1J_{PH}$ = 535.0 Hz). IR ν (cm^{-1}): 3620, 3086, 3062, 3028, 2968, 2933, 2876, 2632, 2374, 2356, 2343, 1667, 1603, 1496, 1455, 1380, 1243, 1202, 1175, 1080, 1058, 1028, 968, 903, 862, 820, 756, 729, 710, 700, 668, 657, 641, 621, 595, 584, 571, 521, 472. Anal. Calcd. for $C_9H_{13}O_2P$: C, 58.69; H, 7.11; P, 16.82. Found: C, 58.49; H, 7.18; 16.71.

Synthesis of Secondary Phosphane 1

A mixture of red phosphorus (3.10 g, 100 mmol), allylbenzene (9.00 g, 76 mmol), hydroquinone (0.20 g), $KOH{\cdot}0.5H_2O$ (7.00 g, 108 mmol), and DMSO (30 mL) was irradiated in a microwave oven (200 W) for 30 min. The reaction mixture was cooled and diluted with water (30 mL) and extracted with chloroform (3 × 30 mL). The extract was washed with water (3 × 30 mL) and dried over K_2CO_3. Chloroform was distilled, and the residue was fractionized in vacuo to give *E*-1-phenylprop-1-ene and secondary phosphane **1**.

Bis(1-methyl-2-phenylethyl)phosphane (1). Colorless oil, bp 169–170°C (1 Torr), 4.95 g (48%) yield. Major diastereomer: ^{1}H NMR, δ (ppm), J (Hz): 1.03 (dd, 6H, $^3J_{HH}$ = 7.0, $^3J_{PH}$ = 13.2, Me), 2.07 (m, 2H, CH), 2.51 and 2.85 (ddd, 4H, $^2J_{HH}$ = 10.0, $^3J_{PH}$ = 9.9, $^3J_{PH}$ = 8.0, $^3J_{HH}$ = 10.0, $^3J_{HH}$ = 5.9, CH_2Ph), 2.78–2.87 (m, 2H, CH_2Ph), 3.03 (dm, 1H, PH), 7.10–7.26 (m, 10H, Ph). ^{13}C NMR, δ (ppm), J (Hz): 19.3 (d, $^2J_{PC}$ = 15.8, Me), 27.3 (d, $^1J_{PC}$ = 10.6, CH), 42.8 (d, $^2J_{PC}$ 13.9, CH_2Ph), 126.0 (C-*p*, Ph), 128.2 and 129.0 (C-*o,m*, Ph), 140.7 (d, $^3J_{PC}$ = 8.1, C-*i*, Ph). ^{31}P NMR, δ (ppm): −22.8 (d,

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$^1J_{PH}$ = 180.4 Hz). Minor diastereomer: ^{1}H NMR, δ (ppm), J (Hz): 1.06 (dd, 6H, $^3J_{HH}$ = 7.0, $^3J_{PH}$ = 13.2, Me), 2.07, 2,47 and 2.81 (ddd, three-spin system ABM, 3H,, $^2J_{AB}$ = 10.0, $^3J_{PHA}$ = 8.0, $^3J_{PHB}$ = 9.8 $^3J_{AM}$ = 5.9, $^3J_{BM}$ = 10.0, CH and CH_2), 3.03 (dm, 1H, PH), 7.10-7.26 (m, 10H, Ph). ^{13}C NMR, δ (ppm), J (Hz): 19.2 (d, $^2J_{PC}$ = 16.5, Me), 27.3 (d, $^1J_{PC}$ = 10.6, CH), 42.6 (d, $^2J_{PC}$ = 13.2, CH_2), 126.0 (C-*p*, Ph), 128.2 and 129.0 (C-*o,m*, Ph), 140.6 (d, $^3J_{PC}$ = 7.3, C-*i*, Ph). ^{31}P NMR, δ (ppm): −28.3 and −17.4 ppm (d, $^1J_{PH}$ = 207.3 and 199.2 Hz). IR ν (cm^{-1}): 3080, 3060, 3024, 2968, 2917, 2865, 2850, 2294, 1601, 1598, 1491, 1455, 1304, 1276, 1040, 1040, 968, 914, 736, 695. Anal. Calc. for $C_{18}H_{23}P$: C, 79.97; H, 8.58; P, 11.46. Found: C, 79.83; H, 8.44; P, 11.21.

E-1-Phenylprop-1-ene. Colorless liquid, bp 35–40°C (1 Torr), 1.10 g (12%) yield. ^{1}H NMR ($CDCl_3$) δ: 2.03 (d, 3H, $^3J_{HH}$ = 6.1, Me), 6.37 (dd, 1H, $^3J_{HH}$ = 15.4, $^3J_{HH}$ = 6.1, = CHMe), 6.58 (d, 1H, $^3J_{HH}$ = 15.4, = CHPh), 7.28–7.43 (m, 5H, Ph). Anal. Calc. for C_9H_{10}: C, 91.47; H, 8.53. Found: C, 91.40; H, 8.41.

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