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P-Dodecylbenzenesulfonic Acid: A Highly Efficient Catalyst for One-Pot Synthesis of α-Aminophosphonates in Aqueous Media

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ABSTRACT: *A highly efficient one-pot three-component reaction of aldehydes or ketones, amines, and trimethyl or triethyl phosphite catalyzed by p-dodecylbenzensulfonic acid is developed for the synthesis of α-aminophosphonates at room temperature in water.* © 2013 Wiley Periodicals, Inc. Heteroatom Chem. 24:174–178, 2013; View this article online at wileyonlinelibrary.com. DOI 10.1002/hc.21079

INTRODUCTION

Multicomponent reactions constitute an especially attractive synthetic strategy in which three or more different starting materials react to a final product in a one-pot procedure. Moreover, they are powerful tools in the modern drug discovery process and allow the rapid, automated, and high-throughput generation of organic compounds, by constructing several new bonds in one pot [1].

The organic chemistry of phosphorus is a wide and exciting field, with great opportunities for research or applications development. The α-aminophosphonates, amino acid analogues, have attracted considerable attention during the past two decades because of their wide range of biological and medicinal properties [2]. The potential of α-amino phosphonates as anti-HIV [3], antibiotic [4], anticancer [5], antitumor [6], and antiviral agents [7] is well documented. In addition, in agrochemistry, a number of α-aminophosphonates are used as fungicidal [8], insecticidal [9], and herbicidal agents [10].

The usual synthesis of α-aminophosphonates, the Kabachnik–Fields reaction, involves the nucleophilic addition of phosphites to imines catalyzed by Brønsted [11] or Lewis acids such as $MgBr_2$ [12a], $ZnCl_2$ [12a], $BF_3.OEt_2$ [12b,c], and $SnCl_4$ [12b], etc. or by bases [13]. However, these methods have limitations, as many imines are moisture sensitive and not stable enough for isolation. Recently, one-pot three-component preparation has been reported in organic solvents using $BiCl_3$ [14], $SbCl_3/Al_2O_3$ [15], $Cu(OTf)_2$ [16], $InCl_3$ [17], $ZrCl_4$ [18], GaI_3 [19], $In(OTf)_3/MgSO_4$ [20], silica sulfuric acid [21], Amberlyst-15 [22], and H-beta zeolite [23]. More recently, methods employing ionic liquids [24], water [25], neat conditions [26], as well as microwave irradiation [27], and ultrasonic irradiation [28] have been introduced. However, various drawbacks, such as synthetic inconvenience, a long reaction time, use of expensive or toxic reagents, and a costly and moisture-sensitive catalyst, etc. encountered in the reported methodologies necessitate the development of a more efficient and convenient method.

The organic reactions in aqueous media have attracted considerable attention in recent years, since they offer a powerful tool for minimizing waste production and harmful organic solvent disposal [29]. However, water is usually avoided as a medium for organic reactions, because of the insolubility, and occasionally decomposition or deactivation of many reactive substrates, reagents, and catalysts by this solvent [30]. Surfactants are

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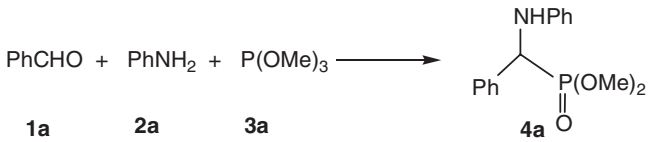
TABLE 1 Optimization of Reaction Conditions



1a **2a** **3a** **4a**

Entry	*Solvent*	*Catalyst (mol%)*	*Time (h)*	*Yield*[a] *(%)*
1	—	5	2	80
2	CH_3CN	5	6	82
3	EtOH	5	3	86
4	$CHCl_3$	5	3	78
5	H_2O	5	0.5	85
6	H_2O	3	1	90
7	H_2O	2	1	87
8	H_2O	1	1	79
9	H_2O	7	1	64
10	H_2O	10	1	73
11	H_2O	15	1	67

[a]Isolated yields.

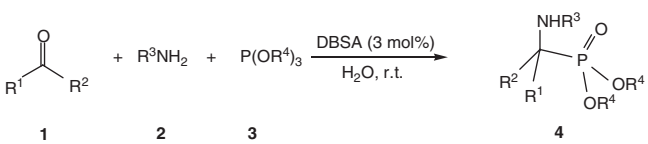
stable in water and can make organic materials soluble or form colloidal dispersions [31], so they can overcome the above drawbacks of the reactions and can be used as good catalysts in water. There are several reports in which a catalytic amount of a Lewis acid-surfactant-combined catalyst, such as scandium *tris*(dodecyl sulfate) [32a–d] or a Brønsted acid-surfactant-combined catalyst, such as dodecylbenzenesulfunic acid (DBSA) [32e–h], was used.

RESULTS AND DISCUSSION

Our continued interest in the development of ecofriendly and green processes using water as a solvent [33] prompted us to develop a highly simple, alternative procedure for the synthesis of α-aminophosphonates by a one-pot three-component reaction of an aldehyde/ketone, amine, and triethyl (trimethyl) phosphite utilizing DBSA in water (Scheme 1).

To optimize the reaction conditions, the reaction of benzaldehyde (1.0 mmol), aniline (1.0 mmol), and trimethyl phosphite (1.2 mmol) was studied in various conditions at room temperature (Table 1). The best reaction condition resulted when DBSA (3 mol%) in water was used at room temperature.

On the basis of the above findings, we further desired to study the general reactivities of various aldehydes (aromatic, heteroatomatic, aliphatic) or ketones (dialkyl, cyclic, aryl alkyl), amines (aromatic, aralkyl, cycloalkyl), and trimethyl- and triethyl phosphite (Table 2). Although complete conversions and high isolated yields were generally obtained for the reactions between a variety of aromatic/heteroaromatic/aliphatic aldehydes with aromatic amines, the reaction of an aromatic aldehyde (benzaldehyde) with cyclohexylamine was slow with poor yield of the corresponding product (Table 2, entry 11). The same observation was found for cyclohexanone; while this ketone reacted smoothly with aniline, affording the corresponding α-aminophosphonate in high yield (Table 2, entry 26), its reaction with benzylamine took longer time with very low yield (Table 2, entry 25). The reaction of acetophenone with aniline resulted in poor yield of the corresponding product (Table 2, entry 27) even after 14 h. Unfortunately, the reaction of the conjugated aldehyde (cinamaldehyde) with aniline and trimethyl phosphite (Table 2, entry 16) gave a mixture of products, which was difficult to separate.



1 **2** **3** **4**

SCHEME 1 Kabachnik–Fields reaction using DBSA as a catalyst in water.

From the mechanistic point of view, we believe that the role of DBSA is to promote the formation of an imine and to convert the same by protonation of the more electrophilic iminium ion intermediate, to facilitate the attack of the phosphite nucleophile.

CONCLUSIONS

In conclusion, the new procedure described here appears to be highly competitive with other methods reported in the literature and in some cases better results are obtained, especially in terms of reaction time and yields. It should be noted that these high yields are obtained in water at room temperature and high reaction rates. This process represents a suitable option to existing methods.

EXPERIMENTAL

General

^1H and ^{13}C NMR spectra were recorded with a Bruker Avance AQS-300 spectrometer (Germany), with $CDCl_3$ as a solvent and the chemical shifts are determined with reference to residual $CHCl_3$ in $CDCl_3$. DBSA, aldehydes, ketones, amines, and dialkyl phosphites were purchased from Merck (Germany) and Acros (France) companies and used without further purification. Melting points were recorded on a Büchi B-540 (Switzerland) apparatus and are uncorrected. IR spectra were obtained using an ABB FTLA 2000 instrument. MS was carried out on a Hewlett–Packard 5973 (United States)

TABLE 2 DBSA-Catalyzed Synthesis of α-Aminophosphonates from Various Aldehydes/Ketones, Amines, and Phosphites in Water

Entry	*Carbonyl Compound*	*R^3*	*R^4*	*Product*	*Time (min)*	*Yield[a,b] (%)*
1	Benzaldehyde	Ph	Me	**4a**	30	90 [26][c]
2	2-Chlorobenzaldehyde	Ph	Me	**4b**	20	95 [21]
3	2,4-Dichlorobenzaldehyde	Ph	Me	**4c**	7	92 [21]
4	*p*-Anisaldehyde	4-$NO_2C_6H_4$	Me	**4d**	5	83 [26][c]
5	Furfural	Ph	Me	**4e**	45	C [26][c]
6	2-Naphthaldehyde	Ph	Me	**4f**	10	96 [34]
7	Thiophene-2-carbaldehyde	Ph	Me	**4g**	45	90
8	3-Nitrobenzaldehyde	Ph	Me	**4h**	20	80 [35]
9	*p*-Anisaldehyde	Ph	Me	**4i**	180	86 [26][c]
10	Isobutyraldehyde	Ph	Me	**4j**	30	86 [26][c]
11	Benzaldehyde	c-C_6H_{11}	Me	**4k**	720	38 [27][c]
12	Cyclohexane carbaldehyde	Ph	Et	**4l**	60	92 [14]
13	*p*-Anisaldehyde	Ph	Et	**4m**	120	82 [26][c]
14	Salicylaldehyde	Ph	Et	**4n**	240	95 [14]
15	Thiophene-2-carbaldehyde	Ph	Et	**4o**	30	83 [26][c]
16	Cinnamaldehyde	Ph	Me	**4p**	40	C [26][c]
17	2-Pyridinecarbaldehyde	Ph	Me	**4q**	150	64 [26][c]
18	4-Hydroxybenzaldehyde	Ph	Me	**4r**	50	98 [26][c]
19	Benzaldehyde	4-ClC_6H_4	Me	**4s**	8	96 [27][c]
20	*p*-Tolualdehyde	Ph	Me	**4t**	45	90 [35]
21	Benzaldehyde	4-MeC_6H_4	Me	**4u**	120	87 [27][c]
22	*N*-(4-Formylphenyl) acetamide	Ph	Me	**4v**	40	93
23	*p*-Anisaldehyde	Ph	Me	**4w**	60	78 [26][c]
24	Benzaldehyde	Ph	Et	**4x**	90	93 [26][c]
25	Cyclohexanone	$PhCH_2$	Me	**4y**	720	20 [26][c]
26	Cyclohexanone	Ph	Me	**4z**	120	88 [26][c]
27	Acetophenone	Ph	Me	**4aa**	840	21 [26][c]

[a] Isolated yields.
[b] References are given for known compounds.
[c] A mixture of products was obtained.

spectrometer (EI, 70 eV). All compounds were characterized by comparing their physical data with those in the literature.

General Procedure for Preparation of DBSA-Catalyzed Synthesis of α-Aminophosphonates

To a solution of DBSA (0.09 mmol, 3 mol%) in H_2O (15 mL), an amine (3 mmol), an aldehyde or ketone (3 mmol), and dialkyl phosphite (3.6 mmol) were added successively at 25°C. After stirring at room temperature for the period of time listed in Table 2, a saturated aqueous $NaHCO_3$ solution (20 mL) and brine (20 mL) were added, the mixture was extracted with EtOAc (25 mL), and the organic layer was dried over anhydrous Na_2SO_4. The solvent was evaporated under reduced pressure, and the crude product was purified either by recrystallization or by preparative TLC (silica gel). Spectral data for selected products is presented below.

*Dimethyl (2-naphthyl)(N-phenylamino) methylphosphonate (**4f**) [26c].* White crystals (no further purification needed), mp 144°C; IR (KBr): $\bar{\nu}$ 3307, 3024, 2947, 1607, 1504, 1237, 1062, 1027 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$): δ 3.17 (d, J = 10.5 Hz, 3H), 3.84 (d, J = 10.7 Hz, 3H), 5.19 (br s, 1H), 5.72 (d, $^1J_{P\text{-}H}$ = 24.1 Hz, 1H), 6.59 (d, J = 7.8 Hz, 2H), 6.68 (t, J = 7.3 Hz, 1H), 7.06 (t, J = 7.4 Hz, 2H), 7.43–7.93 (m, 6H), 8.27 (d, J = 8.5 Hz, 1H); ^{13}C NMR (proton decoupled, 75 MHz, $CDCl_3$): δ 51.1 (d, $^1J_{P\text{-}C}$ = 152.0 Hz, CH), 53.74 (d, $^2J_{P\text{-}C}$ = 6.8 Hz, OCH_3), 53.76 (d, $^2J_{P\text{-}C}$ = 6.7 Hz, OCH_3), 113.64, 118.46, 122.64, 125.57, 125.65, 125.68, 125.76 (d, $^3J_{P\text{-}C}$ = 5.4 Hz, CH), 126.56, 128.70 (d, $^3J_{P\text{-}C}$ = 3.5 Hz, CH), 129.15, 129.26, 131.45, 133.88, 146.12 (d, $^2J_{P\text{-}C}$ = 14.2 Hz, C).

*Dimethyl (3-nitrophenyl)(N-phenylamino) methylphosphonate (**4h**) [35].* Yellow crystals (EtOAc/*n*–hexane), mp 131°C; IR (KBr): $\bar{\nu}$ 3301, 3106, 2940, 1602, 1533, 1024 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$): δ 3.62 (d, J = 10.7 Hz, 3H), 3.81 (d, J = 10.7 Hz, 3H), 4.96 (d, $^1J_{P\text{-}H}$ = 25.0 Hz, 1H), 5.35 (br s, 1H), 6.59 (d, J = 7.8 Hz, 2H), 6.71(t, J = 7.3 Hz, 1H), 7.10 (t, J = 8.4 Hz, 2H), 7.49 (t, J = 7.9 Hz, 1H), 7.85 (d, J = 7.4 Hz, 1H), 8.11 (d, J = 8.1 Hz, 1H),

8.38 (d, $J = 1.9$ Hz, 1H); ^{13}C NMR (proton decoupled, 75 MHz, $CDCl_3$): δ 53.77 (d, $^2J_{P\text{-}C} = 6.8$ Hz, OCH_3), 54.26 (d, $^2J_{P\text{-}C} = 6.9$ Hz, OCH_3), 55.06 (d, $^1J_{P\text{-}C} = 150.3$ Hz, CH), 113.81, 118.99, 122.75 (d, $^3J_{P\text{-}C} = 5.3$ Hz, CH),123.05, 129.33, 129.66, 133.94, 138.67, 145.61 (d, $^2J_{P\text{-}C} = 14.1$ Hz, C), 148.51 (d, $^3J_{P\text{-}C} = 2.8$ Hz, CH).

*Dimethyl 2-methyl-1-(N-phenylamino) propylphosphonate (***4j***) [26c].* Fade white solid (prep. TLC, *n*-hexane–EtOAc, 4:1), mp 97°C; IR (KBr): $\overline{\nu}$ 3344, 2962, 1602, 1497, 1327, 1286, 1239, 1023 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$): δ 1.04 (d, $J = 7.1$ Hz, 3H), 1.07 (d, $J = 7.1$ Hz, 3H), 2.17–2.29 (m, 1H), 3.60–3.73 (m, 7H), 3.88 (m, CH, 1H), 6.64 (d, $J = 8.0$ Hz, 2H), 6.70 (t, $J = 7.2$ Hz, 1H), 7.16 (t, $J = 8.4$ Hz, 2H); ^{13}C NMR (proton decoupled, 75 MHz, $CDCl_3$): δ 18.05 (d, $^3J_{P\text{-}C} = 4.7$ Hz, CH_3), 20.55 (d, $^2J_{P\text{-}C} = 12.2$ Hz, CH), 29.89 (d, $^3J_{P\text{-}C} = 5.8$ Hz, CH_3), 52.34 (d, $^2J_{P\text{-}C} = 7.5$ Hz, OCH_3), 53.34 (d, $^2J_{P\text{-}C} = 7.0$ Hz, OCH_3), 56.01 (d, $^1J_{P\text{-}C} = 150.7$ Hz, CH), 113.19, 117.99,129.34, 147.44.

*Diethyl cyclohexyl(N-phenylamino) methylphosphonate (***4l***) [14].* Light yellow crystals (no further purification needed), mp 87°C; IR (KBr): $\overline{\nu}$ 3317, 2991, 2931, 2857, 1601, 1506, 1445, 1221, 1026 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$): δ 1.07–1.38 (m, 11 H), 1.61–2.03 (m, 6H), 3.57–3.68 (m, 1H), 3.85–4.14 (m, 5H), 6.63 (d, $J = 8.2$ Hz, 2H), 6.69 (t, $J = 7.3$ Hz, 1H), 7.15 (t, $J = 7.7$ Hz, 2H); ^{13}C NMR (proton decoupled, 75 MHz, $CDCl_3$): δ 16.38 (d, $^3J_{P\text{-}C} = 5.6$ Hz, CH_3), 16.46 (d, $^3J_{P\text{-}C} = 5.3$ Hz, CH_3), 26.00, 26.17, 26.29, 28.27 (d, $^3J_{P\text{-}C} = 4.5$ Hz, CH_3), 30.96 (d, $^2J_{P\text{-}C} = 11.4$ Hz), 39.85 (d, $^3J_{P\text{-}C} = 5.6$ Hz), 56.01 (d, $^1J_{P\text{-}C} = 150.1$ Hz), 61.81(d, $^2J_{P\text{-}C} = 7.4$ Hz, OCH_2), 62.63(d, $^2J_{P\text{-}C} = 7.1$ Hz, OCH_2), 113.16, 117.74, 129.26, 147.75 (d, $^3J_{P\text{-}C} = 5.5$ Hz).

*Diethyl (4-methoxyphenyl)(N-phenylamino) methylphosphonate (***4m***) [26c].* White solid (prep. TLC, *n*-hexane–EtOAc, 4:1), mp 106°C; IR (KBr): $\overline{\nu}$ 3294, 2984, 1601,1509, 1310, 1233, 1021 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$): δ 1.14 (t, $J = 7.04$ Hz, 3H), 1.28 (t, $J = 7.05$ Hz, 3H), 3.66–3.74 (m, 1H), 3.76 (s, 3H), 3.91–3.99 (m, 1H), 4.04–4.18 (m, 2H), 4.67–4.82 (m, 2H), 6.59 (d, $J = 8.0$ Hz, 2H), 6.68 (t, $J = 7.3$ Hz, 1H), 6.86 (d, $J = 8.5$ Hz, 2H), 7.10 (t, $J = 7.8$ Hz, 2H), 7.39 (d, $J = 8.6$ Hz, 2H); ^{13}C NMR (proton decoupled, 75 MHz, $CDCl_3$): $\delta = 16.28$ (d, $^3J_{P\text{-}C} = 5.7$ Hz, CH_3), 16.46 (d, $^3J_{P\text{-}C} = 5.8$ Hz, CH_3), 55.21, 55.33 (d, $^1J_{P\text{-}C} = 151.3$ Hz), 63.18 (d, $^2J_{P\text{-}C} = 6.8$ Hz, OCH_2), 63.23 (d, $^2J_{P\text{-}C} = 6.9$ Hz, OCH_2), 113.87, 114.04 (d, $J_{P\text{-}C} = 2.5$ Hz),118.32, 127.66 (d, $J_{P\text{-}C} = 2.9$ Hz), 128.96 (d, $J_{P\text{-}C} = 5.5$ Hz), 129.14, 146.36 (d, $J_{P\text{-}C} = 148$ Hz), 159.28 (d, $J_{P\text{-}C} = 3.0$ Hz).

*Dimethyl (4-methylphenyl)(N-phenylamino) methylphosphonate (***4t***) [24e].* Greenish-white solid (EtOAc–*n*-hexane), mp 130°C; IR (KBr): $\overline{\nu}$ 3299, 2951, 2860, 1521, 1456, 1269, 1028 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$): δ 2.31 (s, 3H), 3.49 (d, $J = 10.5$ Hz, 3H), 3.76 (d, $J = 10.6$ Hz, 3H), 4.81 (d, $^1J_{P\text{-}H} = 24.2$ Hz, 1H), 4.86 (br s, 1H, NH), 6.62–6.72 (m, 3H), 7.08–7.16 (m, 4H), 7.39 (d, $J = 6.8$ Hz, 2H); ^{13}C NMR (proton decoupled, 75 MHz, $CDCl_3$): δ 21.15, 53.72, 53.81, 55.35 (d, $^1J_{P\text{-}C} = 151.1$ Hz, CH), 113.92, 118.43, 127.76 (d, $J_{P\text{-}C} = 5.6$ Hz), 129.18, 129.46 (d, $J_{P\text{-}C} = 2.6$ Hz) 132.56 (d, $J_{P\text{-}C} = 2.5$ Hz), 137.73 (d, $J_{P\text{-}C} = 3.3$ Hz), 146.28 (d, $J_{P\text{-}C} = 14.7$ Hz).

*Dimethyl (4-acetamidophenyl)(N-phenylamino) methylphosphonate (***4v***).* Greenish-white crystals (EtOH–H_2O), mp 181°C; IR (KBr): $\overline{\nu}$ 3332, 3276, 3024, 2957, 1674, 1602, 1525, 1217, 1052, 1021, 749 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$): δ 2.14 (s, 3H), 3.51 (d, $J = 10.6$ Hz, 3H), 3.76 (d, $J = 10.6$ Hz, 3H), 4.70–4.83 (m, 2H), 6.58 (d, $J = 7.9$ Hz, 2H), 6.70 (t, $J = 7.3$ Hz, 1H), 7.10 (t, $J = 8.0$ Hz, 2H), 7.35 (d, $J = 8.4$ Hz, 2H), 7.48 (d, $J = 8.4$ Hz, 2H), 8.21 (s, 1H); ^{13}C NMR (proton decoupled, 75 MHz, $CDCl_3$): δ 24.34, 53.87 (d, $^2J_{P\text{-}C} = 5.0$ Hz, OCH_3), 53.94 (d, $^2J_{P\text{-}C} = 5.1$ Hz, OCH_3), 54.07 (d, $^1J_{P\text{-}C} = 151.9$ Hz, CH), 113.95, 118.69, 120.09, 128.28 (d, $J_{P\text{-}C} = 5.6$ Hz), 129.21, 130.48, 138.40, 145.95 (d, $J_{P\text{-}C} = 14.5$ Hz), 168.98 ppm; Major mass peaks: *m/z* (%) = 348 (7.4), 279 (14.5), 239 (base peak, 100), 197 (47.7), 167 (43.8), 149 (91), 120 (15.6), 104 (28.9), 77 (32.8), 57 (27.7), 43 (44); Anal. calcd. For $C_{17}H_{21}N_2O_4P$ (348.34): C 58.62; H 6.08; N 8.04; found: C 58.87; H 6.06; N 7.62.

*Dimethyl (1-phenylaminocyclohexyl) phosphonate (***4z***) [26c].* Brownish white solid (prep. TLC: *n*–hexane–EtOAc), mp 80°C; IR (KBr): $\overline{\nu}$ 3337, 2934, 2855, 1600, 1498, 1449, 1230, 1028 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$): δ 1.21–1.85 (m, 10H), 3.67 (d, $J = 10.2$ Hz, 6H), 6.81 (t, $J = 7.2$ Hz, 1H), 7.02 (d, $J = 8.2$ Hz, 2H), 7.17 (t, $J = 7.3$ Hz, 2H); ^{13}C NMR (proton decoupled, 75 MHz, $CDCl_3$): δ 19.94 (d, $^2J_{P\text{-}C} = 11.1$ Hz), 25.28, 30.27, 53.08 (d, $^2J_{P\text{-}C} = 7.5$ Hz, OCH_3), 57.46 (d, $^1J_{P\text{-}C} = 159.7$ Hz), 118.40, 119.52, 128.83, 145.65.

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