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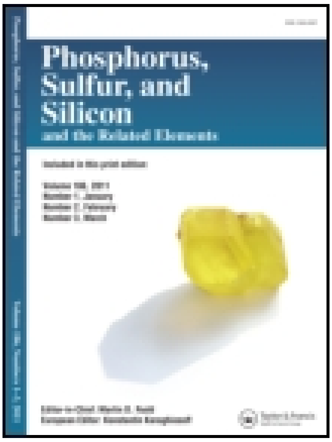

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Synthesis and Herbicidal Activities of Lithium or Potassium Hydrogen 1-(Substituted Phenoxyacetoxy)Alkylphosphonates

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SYNTHESIS AND HERBICIDAL ACTIVITIES OF LITHIUM OR POTASSIUM HYDROGEN 1-(SUBSTITUTED PHENOXYACETOXY)ALKYLPHOSPHONATES

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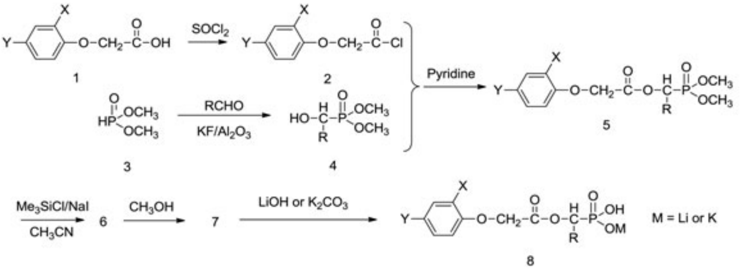
GRAPHICAL ABSTRACT



Abstract *A series of lithium or potassium hydrogen 1-(substituted phenoxyacetoxy)alkylphosphonates were designed and synthesized. All the title compounds were identified by IR, ^{1}H NMR, and ^{31}P NMR, some of them were further analyzed by MS and elemental analyses. The test for herbicidal activity indicated that most of the phosphonates (**8**) possessed excellent postemergence herbicidal activities against broadleaf weeds.*

Supplemental materials are available for this article. Go to the publisher's online edition of Phosphorus, Sulfur, and Silicon and the Related Elements *to view the free supplemental file.*

Keywords Synthesis; herbicidal activity; phosphonate

INTRODUCTION

α-Substituted alkylphosphonate derivatives have received considerable attention in medicine and pesticide chemistry due to their biological activities over the past two decades.[1–5] Especially, α-(substituted phenoxyacetoxy)alkylphosphonates, as potent pyruvate dehydrogenase complex inhibitors,[6,7] possess notable herbicidal activities.[8] Based on

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the bioisosterism, their phosphonic acids were assumed to have better herbicidal activity, because phosphonic acid is more analogous to the pyruvic acid, which acts as the substrate of pyruvate dehydrogenase complex.[9,10] However, as strong acids, these phosphonic acids themselves would lead to the cleavage of carboxylate ester group in the structures. In our previous work, a series of sodium hydrogen 1-(substituted phenoxyacetoxy)alkylphosphonates were synthesized, which are almost neutral salts with higher stability than the corresponding phosphonic acids. Most of these compounds showed excellent postemergence herbicidal activities against broadleaf weeds, controlling more than 90% of rape and amaranth at 150 g/ha. On the other hand, lithium and potassium are often used for the isoelectronic replacement of sodium ions in medical molecules to obtain higher bioactivity and safety,[10,11] which encouraged us to replace the sodium with the lithium or potassium and further study the relationship of structure–herbicidal activity. Herein, we reported the synthesis of 26 new lithium or potassium hydrogen 1-(substituted phenoxyacetoxy)alkylphosphonates and evaluation for their herbicidal activities.

RESULTS AND DISCUSSION

Syntheses

The multistep procedure for the synthesis of the title phosphonates **8** is outlined in Scheme 1. The methods for the synthesis of substituted phenoxyacetyl chloride **2**, dimethyl 1-hydroxyalkylphosphonates **4**, and dimethyl 1-(substituted phenoxyacetoxy)alkylphosphonates **5** were adopted, according to the previous work reported in our group.[12–14] The phosphonates **5** reacted with chlorotrimethylsilane in acetonitrile using sodium iodide as catalyst to provide bis(trimethylsilyl) 1-(substituted phenoxyacetoxy)alkylphosphonates **6**, which was further transformed to 1-(substituted phenoxyacetoxy)alkylphosphonic acids **7** by reaction with methanol. The title phosphonates **8** were then obtained by the reaction with lithium hydroxide or potassium carbonate (Table 1). The structures of **8a–8z** were confirmed by comprehensive IR, ^{1}H NMR, ^{31}P NMR, elemental analysis, and **8a**, **8h**, **8n**, and **8u** were further identified via mass spectrum (MS).

The infra red (IR spectra of compounds **8a–8z** showed normal stretching absorption bands indicating the existence of C=C (~1620, ~1450 cm^{-1}), C—O—C (1080–1200 cm^{-1}), and P—C (740–750 cm^{-1}). A sharp and weak band at 2950–3100 cm^{-1} accounted for the C–H stretching of the benzene ring. The C–H stretching of alkyl appeared at 2860–2950 cm^{-1}. A strong absorption near 1720–1760 cm^{-1} was identified for the absorption C=O. In the ^{1}HNMR spectra of the title compounds **8a–8z**, the chemical shifts of aromatic protons appeared at 6.8–7.8 ppm. As for compounds with aliphatic groups as *R*, the proton signal corresponding to methenyl attached to the P atom displayed multiplets at 5.00–5.23; as for the compounds with aromatic groups as *R*, the proton signal appears at 5.76–6.31 as doublets, due to the coupling with phosphorus.^{31}P NMR chemical shifts of compounds **8a–8z** appeared as a singlet at δ 15.5–16.2 ppm. Their chemical shifts were moved upfield by approximately 7–8 ppm, compared with that of the corresponding *O*,*O*-dimethyl phosphonates at ~23 ppm, whereas the chemical shifts of compounds **8a–8z** moved downfield by approximately 3–4 ppm, compared with the 2-methylpropan-2-aminium phosphonates.[15]

Table 1 Structure and physicochemical data of title compounds **8a–8z**

Compd.	X	Y	M	R	Appearance	mp/°C	Yield/%
8a	Cl	Cl	Li	CH_3	White solid	222–224	84
8b	Cl	Cl	Li	C_2H_5	White solid	226–228	77
8c	Cl	Cl	Li	n-C_3H_7	White solid	219–221	82
8d	Cl	Cl	Li	i-C_3H_7	White solid	207–209	83
8e	Cl	Cl	Li	n-C_4H_9	White solid	188–190	84
8f	Cl	Cl	Li	phenyl	White solid	149–150	85
8g	Cl	Cl	Li	thien-2-yl	White solid	>250	47
8h	CH_3	Cl	Li	CH_3	White solid	175–176	90
8i	CH_3	Cl	Li	C_2H_5	White solid	203–205	87
8j	CH_3	Cl	Li	n-C_3H_7	White solid	236–238	82
8k	CH_3	Cl	Li	i-C_3H_7	White solid	203–205	92
8l	CH_3	Cl	Li	n-C_4H_9	White solid	208–210	89
8m	CH_3	Cl	Li	phenyl	White solid	152–153	85
8n	Cl	Cl	K	CH_3	White solid	138–140	93
8o	Cl	Cl	K	C_2H_5	White solid	99–100	93
8p	Cl	Cl	K	n-C_3H_7	White solid	103–104	84
8q	Cl	Cl	K	i-C_3H_7	White solid	107–108	87
8r	Cl	Cl	K	n-C_4H_9	White solid	139–140	84
8s	Cl	Cl	K	phenyl	White solid	141–142	86
8t	Cl	Cl	K	thien-2-yl	White solid	144–145	65
8u	CH_3	Cl	K	CH_3	White solid	154–155	91
8v	CH_3	Cl	K	C_2H_5	White solid	104–105	85
8w	CH_3	Cl	K	n-C_3H_7	White solid	113–114	81
8x	CH_3	Cl	K	i-C_3H_7	White solid	102–103	95
8y	CH_3	Cl	K	n-C_4H_9	White solid	118–120	90
8z	CH_3	Cl	K	phenyl	White solid	109–110	82

Herbicidal Activities

The preliminary herbicidal activities of title compounds against the roots and stems of *Brassica napus L.*(rape) and *Echinochloa crusgalli Beava* (barnyard grass) were tested at the dosage of 10 and 100 μg/g, respectively. The results showed that some of the synthesized compounds displayed notable herbicidal activities against the tested plants, and they displayed higher inhibitory activities against the growth of the root than that of the stem. For example, **8a–8z** showed 96%–99% inhibitory rate against the root of *B. napus L.* at the dosage of 10 μg/g, but only 75%–92% inhibitory rate against the stem of *B. napus L.* at the same dosage. The inhibitory activities of title compounds against dicotyledon were higher than those against monocotyledon. For example, **8a–8z** showed 75%–92% inhibitory rate against the stem of dicotyledon *B. napus L.* at the dosage of 10 μg/g, but they displayed 28%–76% inhibitory effect against the root of monocotyledon *E. crusgalli* at the same dosage.

Based on the preliminary bioassays, compounds **8a–8g**, **8i**, **8k–8l**, **8n–8t**, **8v**, and **8y** were selected for further bioassay for postemergence herbicidal activity on *Echinochloa crusgalli Beava* (barnyard grass), *Digitaria sanguinalis Scop* (ascendant crabgrass), *Brassica napus L.* (rape), *Amaranthus retroflerus L.* (amaranth), *Setaria viridis* (green bristlegrass), and *Chenopodium serotinum* (small goosefoot). And sodium hydrogen 1-(2,4-dichlorophenoxyacetoxy)ethylphosphonate were selected as a positive control. It was found that the tested compounds displayed higher herbicidal activities against dicotyledonous weeds than monocotyledon, which is agreement with the results in the preliminary bioassays. The compound **8a** showed the best herbicidal activity against the monocotyledon, and with more than 85% inhibitory rate against all the tested dicotyledonous weeds, which is better than the corresponding potassium hydrogen 1-(2,4-dichlorophenoxyacetoxy)ethylphosphonate **8n** and sodium hydrogen 1-(2,4-dichlorophenoxyacetoxy)ethylphosphonate. As a result, lithium salts showed higher herbicidal against the monocotyledons than the corresponding potassium and sodium salts, especially for the compounds with 2,4-Cl_2 as the X and Y groups. However, as for the dicotyledonous plants, the three kinds of salts showed the comparable herbicidal activity, which is different to the results observed against the monocotyledons.

Experimental details and the herbicidal activities (Table S 1 and Table S 2) are presented in the online Supplemental Materials.

CONCLUSIONS

In conclusion, a series of lithium or potassium hydrogen 1-(substituted phenoxyacetoxy)alkylphosphonates were synthesized via the key intermediate 1-(substituted phenoxyacetoxy)alkylphosphonic acids with satisfactory yields. The test for herbicidal activity indicated that most of the title compounds possessed excellent postemergence herbicidal activities against broadleaf weeds, and they displayed higher herbicidal activities against dicotyledonous weeds than monocotyledon. Especially, lithium hydrogen 1-(2,4-dichlorophenoxyacetoxy)ethylphosphonate **8a** showed higher herbicidal activity than the corresponding potassium hydrogen 1-(2,4-dichlorophenoxyacetoxy)ethylphosphonate **8n** and sodium hydrogen 1-(2,4-dichlorophenoxyacetoxy)ethylphosphonate, which provided some indications for further studies on structure modification.

EXPERIMENTAL

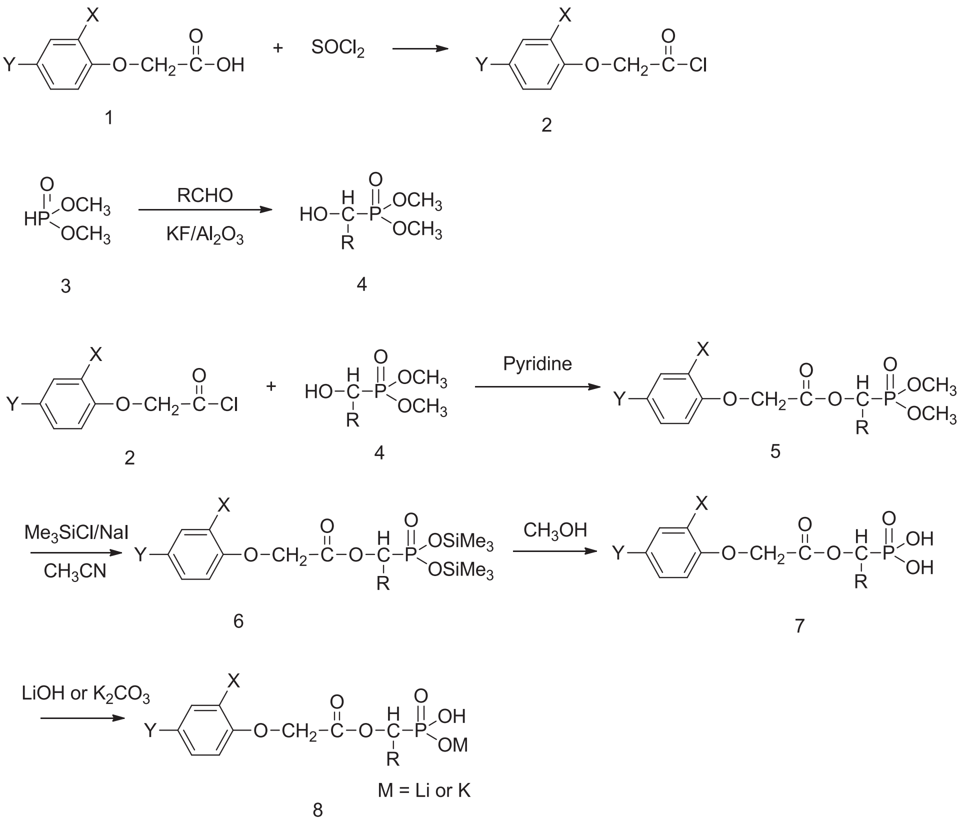
Mass spectra were measured on API2000LC/MS. Infrared spectra were recorded in potassium bromide pellets with a Nicolet Avatar 360 Fourier transform infrared (FTIR) spectrophotometer.^{1}HNMR and ^{31}PNMR spectra were recorded in D_2O solution using sodium 3-(trimethylsilyl)propane-1-sulfonate (DSS) as internal standards with a Varian Mercury-Plus 400 (400 MHz) spectrometer. Elemental analysis was performed with an Elementar Vario EL III elementary analyzer. Melting points (mp) were measured with an electrothermal melting point apparatus and are uncorrected. Selected ^{1}H and ^{31}P NMR spectra are shown in the online Supplemental Materials (Figures S1–S6).

Synthesis

All of the compounds **1–5** were synthesized according to the methods described in the literature.[16–18] (Scheme 1).

General Procedure for 5

To a solution of dimethyl (1-hydroxyalkyl)phosphonates **4** (10 mmol) and pyridine (14 mmol) in CH_2Cl_2 (25 mL), a solution of substituted phenoxyacetyl chloride **2** (10 mmol) was added in CH_2Cl_2 (15 mL) dropwise at below 5°C, and the mixture was stirred for 4 h, which was monitored by thin layer chromatography (TLC). The oily residue was purified



Scheme 1 Synthesis of the title compounds 8.

by flash column chromatography on silica gel with the mixture of acetone/petroleum ether (v:v 1:4) as eluent to furnish **5**.

General Procedure for 6

Under a nitrogen atmosphere and in a dark environment, to a solution of **5** (10 mmol) and sodium iodide (22 mmol) in acetonitrile (25 mL), trimethylchlorosilane (22 mmol) was added at 30°C, then the reaction mixture was stirred for 2 h. The resultant mixture was purified by flash column chromatography on silica gel with a mixture of petroleum ether/*n*-propanol (v:v 3:1) as eluent to furnish **6** as a colorless oil.

General Procedure for 7

To a solution of **6** (10 mmol) in anhydrous methanol (25 mL), the reaction mixture was stirred for 2–3 h at 30°C, which was monitored by TLC. The solvent was removed under reduced pressure followed by column chromatography on silica gel with a mixture of petroleum ether/*n*-propanol (v:v 1:3) as eluent to yield **7**.

General Procedure for Compounds (8)

To a solution of **7** (5.5 mmol) in methanol (20 mL) was added lithium hydroxide or potassium carbonate (5 mmol), and the mixture was stirred at room temperature for 2 h. Removal of the solvent under reduced pressure gave the crude product as a white solid, which was recrystallized from acetonitrile to afford **8**.

Lithium Hydrogen 1-(2,4-Dichlorophenoxyacetoxy)ethylphosphonate (8a). White solid, Yield 84%; ^{1}H NMR (D_2O) δ : 1.41 (dd, 3H, $^3J_{HH} = 7.2$ Hz, $^3J_{PH} = 14.4$ Hz, P—CH—C$\underline{H}_3$), 4.90, 4.93 (q, AB system, 2H, $^2J_{AB} = -16.8$ Hz, O-CH_2-C), 5.00–5.05 (m, 1H, P-CH), 7.00 (d, 1H, $^3J_o = 8.8$ Hz, H^6-Ph), 7.27 (t, 1H, $^3J_o = 8.8$ Hz, H^5-Ph), 7.47 (s, 1H, H^3-Ph); ^{31}P NMR (160 MHz, D_2O) δ: 15.468; IR ν /cm^{-1}: 3424, 2982, 2938, 1751, 1481, 1440, 1392, 1201, 1108, 1078, 992, 869, 800, 720; ESI-MS(m/z): positive: 357(M + 23, 22.27%), 341(M + 7, 35.96%), 334(M$^+$, 3.07%), 301(22.38%), 285(100%), 229(65.74%), 85(26.27%); negative: 331(M-7 + 4, 11.17%), 329(M-7+2, 61.63%), 327(M-7, 100%), 283(7.73%), 255(11.48%), 219(5.82%), 165(21.79%), 161(10.64%), 125(32.82%), 121(20.24%), 107(62.16%), 63(61.59%); Elemental Anal. Calcd. For $C_{10}H_{10}Cl_2LiO_6P$: C 35.85, H 3.01; Found C 35.75, H 2.80.

Lithium Hydrogen 1-(4-Chloro-2-methylphenoxyacetoxy)ethylphosphonate(8h). White solid, Yield 90%; ^{1}H NMR (D_2O) δ : 1.42 (dd, 3H, $^3J_{HH} = 6.8$ Hz, $^3J_{PH} = 14.8$ Hz, P—CH—C$\underline{H}_3$), 2.23 (s, 3H, C_6H_3-C$\underline{H}_3$), 4.86 (s, 2H, O—CH_2-C), 5.13~5.16 (m, 1H, P—CH), 6.84 (d, 1H, $^3J_o = 8.8$ Hz, H^6-Ph), 7.18 (t, 1H, $^3J_o = 8.8$ Hz, H^5-Ph), 7.25 (s, 1H, H^3-Ph); ^{31}P NMR (160 MHz, D_2O) δ: 15.573; IR ν /cm^{-1}: 3410, 2957, 2355, 1763, 1651, 1600, 1493, 1441, 1402, 1381, 1298, 1187, 1138, 1074, 998, 922, 882, 801; ESI-MS(m/z): positive: 337(M+23, 40.30%), 320(M-1+7, 70.40%), 315(M+1, 16.42%), 301(12.19%), 285(45.02%), 254(11.44%), 250(12.44%), 237(12.44%), 229(100%), 221(23.38%), 136(31.84%), 85(29.10%); negative: 321(M + 7, 6.85%), 309(M-7 + 2, 28.90%), 307(M-7, 100%), 201(6.04%), 199(21.09%), 165(2.30%), 143(12.35%), 141(36.38%), 125(28.10%), 107(47.09%), 63(68.86%); Elemental Anal. Calcd. for $C_{11}H_{13}ClLiO_6P$: C 42.00, H 4.17; found C 42.13, H 4.60.

Potassium Hydrogen 1-(2,4-Dichlorophenoxyacetoxy)ethylphosphonate (8n). White solid, Yield 93%; ^{1}H NMR (D_2O) δ : 1.47 (dd, 3H, $^3J_{HH} = 7.0$ Hz, $^3J_{PH} = 15.4$ Hz, P—CH—C$\underline{H}_3$), 4.93 (s, 2H, O—CH_2-C), 5.17–5.23 (m, 1H, P—CH), 6.99 (d, 1H, $^3J_o = 8.8$ Hz, H^6-Ph), 7.28 (t, 1H, $^3J_o = 8.8$ Hz, $^4J_m = 2.0$ Hz, H^5-Ph), 7.49 (d, 1H, $^4J_m = 2.0$ Hz, H^3-Ph); ^{31}P NMR (160 MHz, D_2O) δ: 15.986; IR ν /cm^{-1}: 3423, 2992, 2938, 1754, 1482, 1439, 1391, 1288, 1212, 1084, 924, 870, 801, 720; ESI-MS(m/z): positive: 389(M + 23, 22.74%), 367(M + 1, 19.59%), 317(100%), 301(10.96%), 261(28.93%), 136(22.13%); negative: 331(M-39 + 4, 13.74%), 329(M-39 + 2, 68.27%), 327(M-39, 100%), 283(4.43%), 219(7.33%), 165(19.42%), 161(12.86%), 125(24.46%), 107(44.59%), 63(44.30%); Elemental Anal. Calcd. for $C_{10}H_{10}Cl_2KO_6P$: C 32.71, H 2.75; found C 32.41, H 2.88.

Potassium Hydrogen 1-(4-Chloro-2-methylphenoxyacetoxy)ethylphosphonate(8u). White solid, Yield 91%; ^{1}H NMR (D_2O) δ : 1.42 (dd, 3H, $^3J_{HH} = 6.8$ Hz, $^3J_{PH} = 15.2$ Hz, P—CH—C$\underline{H}_3$), 2.24 (s, 3H, C_6H_3-C$\underline{H}_3$), 4.86 (s, 2H, O—CH_2-C), 5.13~5.16 (m, 1H, P—CH), 6.84 (d, 1H, $^3J_o = 8.8$ Hz, H^6-Ph), 7.18 (t, 1H, $^3J_o = 8.8$ Hz, H^5-Ph), 7.26 (s, 1H, H^3-Ph); ^{31}P NMR (160 MHz, D_2O) δ: 16.002; IR ν /cm^{-1}: 3417, 3218, 2985, 2922, 2275, 1747, 1653, 1493, 1439, 1381, 1338, 1290, 1241, 1191, 1163, 1138, 1077, 1025, 928, 876, 792, 737; ESI-MS(m/z): positive: 369(M + 23, 31.26%), 346(M$^+$, 24.42%), 250(72.34%), 136(17.59%), 60(100%); negative: 309(M-39 + 2, 35.54%), 307(M-7, 95.59%), 199(10.90%), 143(7.48%), 141(17.54%), 125(31.88%), 107(66.38%), 63(100%); Elemental Anal. Calcd. for $C_{11}H_{13}ClKO_6P$: C 38.10, H 3.78; found C 38.41, H 3.75.

The Supplemental Materials contains complete characterization data for all the other reported compounds.

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