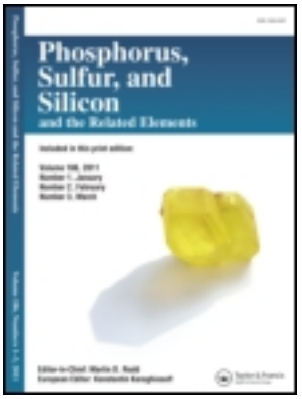

Phosphorus, Sulfur, and Silicon and the Related Elements

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Synthesis and Herbicidal Activity of O,O-Dimethyl-(3-Phenacryloyloxy) Alkyl Phosphonates

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SYNTHESIS AND HERBICIDAL ACTIVITY OF *O,O*-DIMETHYL-(3-PHENACRYLOYLOXY) ALKYL PHOSPHONATES

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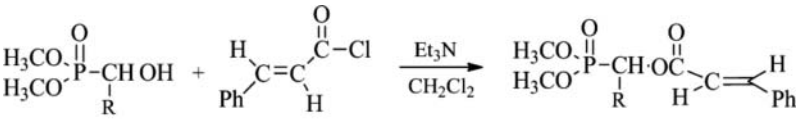
GRAPHICAL ABSTRACT



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Abstract *In an attempt to discover novel compounds with high biological activity and low toxicity, a series of new* O,O*-dimethyl-(3-phenacryloyloxy) alkyl phosphonates* ***4a–m*** *have been designed and synthesized by the reaction of 3-phenacryloyl chloride with α-hydroxyalkyl phosphonate. All new compounds were characterized by elemental analysis, IR, and* ^{1}*H NMR spectroscopy as well as by mass spectrometry. The results of preliminary bioassay indicate that some of the target compounds have excellent inhibitory activities on* Triticum aestivum *(wheat) and* Brassica napus L. *(rape).*

Keywords *O,O*-Dimethyl-(3-phenacryloyloxy) alkyl phosphonates; synthesis; herbicidal activities

INTRODUCTION

Pyruvate dehydrogenase complex (PDHC) is already known to be a site of pesticide action because it plays a pivotal role in cellular metabolism catalyzing the oxidative decarboxylation of pyruvate and the subsequent acetylation of coenzyme A (CoA) to acety-CoA.[1–4] An attempt to design inhibitors of PDHC as herbicides using biochemical reasoning was reported by Baillie et al.[5] Series of acetyl phosphonates have been prepared as mechanism-based inhibitors of PDHC because they are regarded as bioisosters of pyruvate

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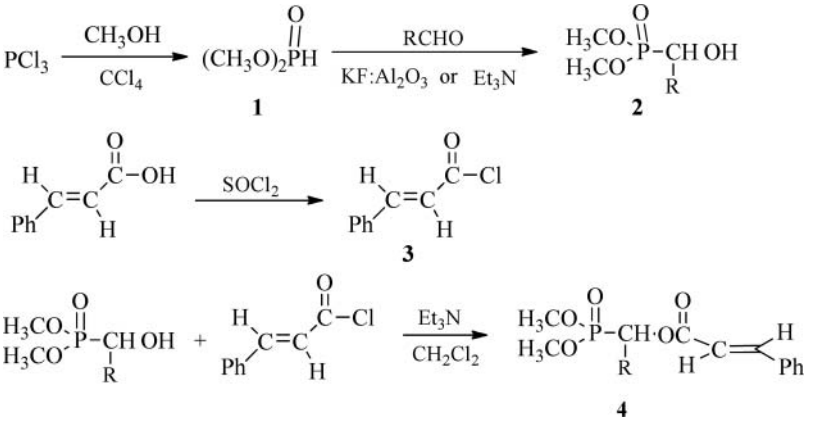
(acetyl formate).[6] Baillie et al. demonstrated that some acetylphosphonates showed modest herbicidal activity due to the inhibition of PDHC. However, their activity was not sufficiently high for full development as herbicides. In the course of our investigations for new phosphonate derivatives with good biological activities, we have shown that certain substituted phenoxy acetoxy alkyl phosphonates possess good herbicide activities.[7–9] To find new phosphonates with better pesticide activity, the 3-phenacryloyloxy structural unit is introduced into phosphonate molecules; thus, *O,O*-dimethyl-(3-phenacryloyloxy) alkyl phosphonates were synthesized by the reaction of 3-phenacryloyl chloride with α-hydroxyalkyl phosphonates under mild conditions. The synthetic route is shown in Scheme 1.



Scheme 1

RESULTS AND DISCUSSION

Synthesis and Structure

The phosphonates **4** were synthesized by the multistep route outlined in Scheme 1. The reaction of dialkyl H-phosphonates with aldehydes is a convenient method used to synthesize α-hydroxyalkyl phosphonates. The addition of a base (triethylamine or KF/Al_2O_3) was essential for the addition reaction. Without the use of triethylamine or KF/Al_2O_3 as a catalyst, the reaction was slow and also the yields were very low. Compounds **4** were prepared from the compound **2** and 3-phenacryloyl chloride **3** in the presence of triethylamine. As the target phosphonate derivatives contain groups sensitive to acid, base, or water, such as carboxylic esters, the reaction required conditions near room temperature and the use of anhydrous solvents. All products **4** were purified by flash column chromatography on silica gel.

The molecular structures of all new compounds prepared were confirmed by ^{1}H NMR, IR spectra, MS, and elemental analyses.

In the ^{1}H NMR spectra of **4**, the proton of the P-CH moiety in **4f**–**m** exhibits a doublet, while the protons in the P-OCH_3 groups display only one signal as a doublet. The IR spectra of all compounds showed normal stretching absorption bands indicating the existence of Ph-H (~2950 cm^{-1}), C=O (~1710 cm^{-1}), C=C (~1650 cm^{-1}), P=O (~1250 cm^{-1}),

P-O-C (∼1050 cm^{-1}), and P-C (∼760 cm^{-1}). The EI mass spectra of compounds **4a–m** gave the anticipated molecular ion peaks. The mass spectrum of **4a** shows a molecular ion peak at m/z = 270 with 11.89% abundance. The mass spectra of compounds **4a–m** show the strongest ion peak at m/z = 93 with 100% abundance. All the fragmentation ions of **4a–m** are consistent with the structure of the corresponding compounds and can be clearly assigned.

Herbicidal Activities

The herbicidal activities of compounds **4a–m** were evaluated at a rate of 100 and 10 ppm in a set of experiments. They were tested for stalk and root inhibitory effect against *Triticum aestivum* (wheat) and *Brassica napus L.* (rape). As is evident from Table 1, compounds **4a**,**b**,**d–f** and **4i** exhibit notable herbicidal activity on the root of rape at 100 ppm, while compounds **4a**,**b**,**e**,**g–j** obviously have a promotive action for plant growth against a stalk of wheat at 10 ppm.

EXPERIMENTAL

General remarks: Melting points are uncorrected. Mass spectra were measured with a Finnigan Trace MS spectrometer. IR spectra were recorded with a PE-983 infrared spectrophotometer as KBr pellets. ^{1}H and ^{31}P NMR spectra were recorded in $CDCl_3$ with a Varian Mercury 400 spectrometer and resonances are given relative to external Tetramethylsilane (TMS) and 85% H_3PO_4, respectively. Elementary analyses were performed with a Vario EL III elementary analysis instrument. The reagents and solvents were commercially available and were purified according to conventional methods before use. Compounds **1**, **2**, and **3** were prepared according to the literature.[10–22]

Table 1 The inhibition percentage of phosphonates **4** to wheat and rape*

		Wheat				Rape			
		Stalk 10 ppm,		Root 10 ppm,		Stalk 10 ppm,		Root 10 ppm,	
4	R	100 ppm		100 ppm		100 ppm		100 ppm	
a	H	−20.98	−12.46	−56.96	−36.96	−2.45	45.28	51.22	91.00
b	Me	−4.59	0.00	−48.30	0.00	−4.75	10.75	24.34	92.33
c	Et	2.95	−15.08	12.05	8.07	22.64	25.53	37.25	64.02
d	Pr	10.49	−0.98	−13.10	−7.37	−0.18	33.40	61.48	89.74
e	*i*Pr	−8.20	−21.64	−32.28	22.81	23.77	49.81	61.27	96.30
f	Ph	−6.56	5.57	−1.17	37.78	12.08	79.25	41.9	96.83
g	4-$CH_3C_6H_4$	4.92	−4.59	−31.11	28.54	3.96	−9.43	78.94	67.20
h	4-ClC_6H_4	−12.79	−18.36	−35.79	28.19	1.89	−14.72	58.52	79.89
i	2-ClC_6H_4	−15.08	−13.77	−39.65	−21.29	24.34	40.57	41.69	91.00
j	2,4-$Cl_2C_6H_3$	12.79	6.56	−30.88	−35.21	2.26	0.00	49.95	0.00
k	4-$MeOC_6H_4$	−5.25	−8.20	−16.14	−43.04	40.94	33.21	−12.70	72.91
l	3-$NO_2C_6H_4$	−7.87	0.00	−3.51	0.00	90.57	78.68	84.13	81.16
m	2-Furyl	−0.33	2.62	9.71	−2.11	−4.53	0.00	75.56	0.00
	2,4-D	30.87	38.56	78.97	92.12	89.54	91.20	98.12	98.50

*Negative inhibition percentage shows promotive action for plant growth.

Synthesis of *O,O*-Dimethyl-(3-Phenacryloyloxy) Alkyl Phosphonates 4: General Procedure

A solution of 3-phenacryloyl chloride **3** (22 mmol) in CH_2Cl_2 (10 mL) was added to a stirred mixture of α-hydroxyalkyl phosphonate **2** (20 mmol) and triethylamine (22 mmol) in CH_2Cl_2 (25 mL) at 20 °C–25 °C. The mixture was stirred at ambient temperature for 4 h and then at 40 °C for 1 h, and was washed with 0.1 M hydrochloric acid, saturated sodium hydrogen carbonate solution, and brine. The resulting mixture was dried and the solvent was evaporated. The residue was chromatographed on silica with acetone/petroleum ether (60 °C–90 °C) (1:4) as eluent to give compounds **4**.

(Dimethoxyphosphoryl)Methyl Cinnamate (4a)

Pale yellow liquid, yield 61%; n_D^{20}: 1.5490. IR (KBr): υ = 3062 (w, Ph-H), 2957, 2854 (m, C-H), 1723 (s, C=O), 1635 (C=C), 1578, 1496, 1450 (s, Ph), 1261 (s, P=O), 1161 (s, C-O-C), 1031, 896 (s, P-O-C), 768 (s, P-C) cm^{-1}. ^1H NMR ($CDCl_3$, 400 MHz): δ = 3.84 (d, J = 10.8 Hz, 6H, OCH_3), 4.54 (d, J = 8.4 Hz, 2H, -OCH_2P), 6.48 (d, J = 16.0 Hz, 1H, PhCH=C<u>H</u>-), 7.40–7.55 (m, 5H, C_6H_5), 7.74 (d, J = 16.0 Hz, 1H, PhC<u>H</u>=CH-). ^{31}P NMR ($CDCl_3$): δ = 22.0 ppm. MS (70 eV), m/z (%): 270 (M^+ 11.89); Anal. calcd. for $C_{12}H_{15}O_5P$: C, 53.34; H, 5.60. Found: C, 53.43; H, 5.72%.

1-(Dimethoxyphosphoryl)Ethyl Cinnamate (4b)

Pale yellow liquid, yield 83%; n_D^{20}: 1.5480. IR (KBr), υ = 3061 (w, Ph-H), 2956, 2854 (m, C-H), 1720 (s, C=O), 1636 (C=C), 1578, 1496, 1450 (s, Ph), 1247 (s, P=O), 1163 (s, C-O-C), 1051, 894 (s, P-O-C), 768 (s, P-C) cm^{-1}; ^1H NMR ($CDCl_3$, 400 MHz): δ = 1.55 (d, J = 9.6 Hz, 3H, -CH_3), 3.84 (d, J = 6.8 Hz, 6H, OCH_3), 5.46 (m, 1H, -OCHP), 6.47 (d, J = 16.0 Hz, 1H, PhCH=C<u>H</u>-), 7.37–7.55 (m, 5H, C_6H_5), 7.73 (d, J = 16.0 Hz, 1H, PhC<u>H</u>=CH-). ^{31}P NMR ($CDCl_3$): δ = 21.8 ppm. MS (70 eV), m/z (%): 284 (M^+ 12.22); Anal. calcd. for $C_{13}H_{17}O_5P$: C, 54.93; H, 6.03. Found: C, 54.77; H, 6.16%.

1-(Dimethoxyphosphoryl)Propyl Cinnamate (4c)

Pale yellow liquid, yield 80%; n_D^{20}: 1.5370. IR (KBr), υ = 3062 (w, Ph-H), 2956, 2854 (m, C-H), 1716 (s, C=O), 1635 (C=C), 1578, 1496, 1450 (s, Ph), 1257 (s, P=O), 1159 (s, C-O-C), 1031, 939 (s, P-O-C), 768 (s, P-C) cm^{-1}; ^1H NMR ($CDCl_3$, 400 MHz): δ = 1.16 (t, J = 9.6 Hz, 3H, -CH_2<u>CH_3</u>), 2.02 (m, 2H, -<u>CH_2</u>CH_3), 3.82 (d, J = 8.4 Hz, 6H, OCH_3), 5.38 (m, 1H, -OCHP), 6.49 (d, J = 16.0 Hz, 1H, PhCH=C<u>H</u>-), 7.40–7.56 (m, 5H, C_6H_5), 7.75 (d, J = 16.0 Hz, 1H, PhC<u>H</u>=CH-). ^{31}P NMR ($CDCl_3$): δ = 21.5 ppm. MS (70 eV), m/z (%): 298 (M^+ 15.86); Anal. calcd. for $C_{14}H_{19}O_5P$: C, 56.37; H, 6.42. Found: C, 56.55; H, 6.50%.

1-(Dimethoxyphosphoryl)Butyl Cinnamate (4d)

Pale yellow liquid, yield 78%; n_D^{20}: 1.5345. IR (KBr), υ = 3062 (w, Ph-H), 2959, 2874 (m, C-H), 1715 (s, C=O), 1635 (C=C), 1578, 1496, 1450 (s, Ph), 1263 (s, P=O), 1160 (s, C-O-C), 1032, 985 (s, P-O-C), 709 (s, P-C) cm^{-1}; ^1H NMR ($CDCl_3$, 400 Hz): δ = 0.96 (t, J = 8.4 Hz, 3H, -CH_2CH_2<u>CH_3</u>), 1.60 (m, 2H, -CH_2<u>CH_2</u>CH_3), 1.93 (m, 2H,

-$\underline{CH_2}CH_2CH_3$), 3.81 (d, J = 6.8 Hz, 6H, OCH_3), 5.48 (m, 1H, -OCHP), 6.48 (d, J = 16.0 Hz, 1H, PhCH=C$\underline{H}$-), 7.39–7.55 (m, 5H, C_6H_5), 7.74 (d, J = 16.0 Hz, 1H, Ph$\underline{CH}$=CH-).^{31}P NMR ($CDCl_3$): δ = 21.1 ppm. MS (70 eV), m/z (%): 312 (M^+ 15.27); Anal. calcd. for $C_{15}H_{21}O_5P$: C, 57.69; H, 6.78. Found: C, 57.75; H, 6.90%.

1-(Dimethoxyphosphoryl)-2-Methylpropyl Cinnamate (4e)

Pale yellow liquid, yield 74%; n_D^{20}: 1.5350. IR (KBr): υ = 3062 (w, Ph-H), 2963, 2854 (m, C-H), 1716 (s, C=O), 1634 (C=C), 1578, 1496, 1451 (s, Ph), 1245 (s, P=O), 1159 (s, C-O-C), 1033, 893 (s, P-O-C), 768 (s, P-C) cm^{-1}; ^{1}H NMR ($CDCl_3$, 400 MHz): δ = 1.08 (d, J = 7.2 Hz, 6H, -CH($\underline{CH_3}$)$_2$), 2.31 (m, 1H, -$\underline{CH}$(CH_3)$_2$), 3.79 (d, J = 5.6 Hz, 6H, OCH_3), 5.29 (m, 1H, -OCHP), 6.50 (d, J = 16.0 Hz, 1H, PhCH=$\underline{CH}$-), 7.40–7.55 (m, 5H, C_6H_5), 7.75 (d, J = 16.0 Hz, 1H, Ph$\underline{CH}$=CH-). ^{31}P NMR ($CDCl_3$): δ = 21.2 ppm. MS (70 eV), m/z (%): 312 (M^+ 11.73); Anal. calcd. for $C_{15}H_{21}O_5P$: C, 57.69; H, 6.78. Found: C, 57.58; H, 6.86%.

(Dimethoxyphosphoryl)(Phenyl)Methyl Cinnamate (4f)

Pale yellow liquid, yield 71%; n_D^{20}: 1.5525. IR (KBr), υ = 3063 (w, Ph-H), 2956, 2854 (m, C-H), 1731 (s, C=O), 1633 (C=C), 1578, 1496, 1450 (s, Ph), 1265 (s, P=O), 1151 (s, C-O-C), 1045, 910 (s, P-O-C), 768 (s, P-C) cm^{-1}; ^{1}H NMR ($CDCl_3$, 400 MHz): δ = 3.68 (d, J = 10.4 Hz, 6H, OCH_3), 6.30 (d, J = 13.2 Hz, 1H, -OCHP), 6.56 (d, J = 16.4 Hz, 1H, PhCH=$\underline{CH}$-), 7.33–7.57 (m, 10H, -C_6H_5), 7.77 (d, J = 16.0 Hz, 1H, Ph$\underline{CH}$=CH-).^{31}P NMR ($CHCl_3$): δ = 19.7 ppm. MS (70 eV) m/z (%): 346 (M^+ 13.10); Anal. calcd. for $C_{18}H_{19}O_5P$: C, 62.43; H, 5.53. Found: C, 62.56; H, 5.61%.

(Dimethoxyphosphoryl)(*p*-Tolyl)Methyl Cinnamate (4g)

Colorless crystals, yield 64%; mp: 67 °C–70 °C. IR (KBr): υ = 3072 (w, Ph-H), 2958, 2857 (m, C-H), 1704 (s, C=O), 1637 (C=C), 1577, 1498, 1448 (s, Ph), 1237 (s, P=O), 1177 (s, C-O-C), 1057, 951 (s, P-O-C), 768 (s, P-C) cm^{-1}; ^{1}H NMR ($CDCl_3$, 400 MHz): δ = 2.35 (s, 3H, CH_3), 3.75 (d, J = 10.4 Hz, 6H, OCH_3), 6.26 (d, J = 13.2 Hz, H, -OCHP), 6.54 (d, J = 16.0 Hz, 1H, PhCH=$\underline{CH}$-), 7.19–7.56 (m, 9H, C_6H_5, C_6H_4), 7.75 (d, J = 16.4 Hz, 1H, Ph$\underline{CH}$=CH-).^{31}P NMR ($CDCl_3$): δ = 19.9 ppm. MS (70 eV), m/z (%): 360 (M^+ 15.82); Anal. calcd. for $C_{19}H_{21}O_5P$: C, 63.33; H, 5.87. Found: C, 63.51; H, 5.94%.

(4-Chlorophenyl)(Dimethoxyphosphoryl)Methyl Cinnamate (4h)

Colorless crystals, yield 69%; mp: 79 °C–82 °C. IR (KBr): υ = 3096 (w, Ph-H), 2928, 2852 (m, C-H), 1729 (s, C=O), 1635(C=C), 1576, 1488, 1449 (s, Ph), 1257 (s, P=O), 1181 (s, C-O-C), 1061, 892 (s, P-O-C), 767 (s, P-C) cm^{-1}; ^{1}H NMR ($CDCl_3$, 400 MHz): δ = 3.73 (d, J = 10.8 Hz, 6H, OCH_3), 6.24 (d, J = 13.6 Hz, 1H, -OCHP), 6.54 (d, J = 16.0 Hz, 1H, PhCH=$\underline{CH}$-), 7.36–7.56 (m, 9H, C_6H_5, C_6H_4), 7.76 (d, J = 16.0 Hz, 1H, Ph$\underline{CH}$=CH-). ^{31}P NMR ($CDCl_3$): δ = 19.7 ppm. MS (70 eV), m/z (%): 380 (M^+ 19.26); Anal. calcd. for $C_{18}H_{18}ClO_5P$: C, 56.78; H, 4.76. Found: C, 56.82; H, 4.86%.

(2-Chlorophenyl)(Dimethoxyphosphoryl)Methyl Cinnamate (4i)

Pale yellow liquid, yield 58%; n_D^{20}: 1.5745. IR (KBr), υ = 3063 (w, Ph-H), 2956, 2854 (m, C-H), 1732 (s, C=O), 1633 (C=C), 1577, 1472, 1449 (s, Ph), 1264 (s, P=O), 1149 (s, C-O-C), 1035, 890 (s, P-O-C), 767 (s, P-C) cm^{-1}; ^1H NMR ($CDCl_3$, 400 MHz): δ = 3.84 (d, J = 10.8 Hz, 6H, OCH_3), 6.54 (d, J = 16.0 Hz, 1H, PhCH=CH-), 6.78 (d, J = 13.6 Hz, 1H, -OCHP), 7.28–7.55 (m, 9H, C_6H_5, C_6H_4), 7.75 (d, J = 16.0 Hz, 1H, PhCH=CH-). ^{31}P NMR ($CDCl_3$): δ = 20.0 ppm. MS (70 eV), m/z (%): 380 (M^+ 17.46); Anal. calcd. for $C_{18}H_{18}ClO_5P$: C, 56.78; H, 4.76. Found: C, 56.87; H, 4.89%.

(2,4-Dichlorophenyl)(Dimethoxyphosphoryl)Methyl Cinnamate (4j)

Pale yellow liquid, yield 75%; n_D^{20}: 1.5635. IR (KBr), υ = 3063 (w, Ph-H), 2956, 2855 (m, C-H), 1728 (s, C=O), 1635 (C=C), 1589, 1473, 1452 (s, Ph), 1268 (s, P=O), 1139 (s, C-O-C), 1037, 846 (s, P-O-C), 767 (s, P-C) cm^{-1}; ^1H NMR ($CDCl_3$, 400 MHz): δ = 3.80 (d, J = 11.2 Hz, 6H, OCH_3), 6.52 (d, J = 16.0 Hz, 1H, PhCH=CH-), 6.68 (d, J = 13.6 Hz, 1H, -OCHP), 7.30–7.67 (m, 8H, C_6H_5, C_6H_3), 7.75 (d, J = 16.0 Hz, 1H, PhCH=CH-). ^{31}P NMR ($CDCl_3$): δ = 19.2 ppm. MS (70 eV), m/z (%): 415 (M^+ 24.16); Anal. calcd. for $C_{18}H_{17}Cl_2O_5P$: C, 52.07; H, 4.13. Found: C, 52.22; H, 4.31%.

(Dimethoxyphosphoryl)(4-Methoxyphenyl)Methyl Cinnamate (4k)

Colorless crystals, yield 57%; mp: 76 °C–80 °C. IR (KBr): υ = 3026 (w, Ph-H), 2956, 2855 (m, C-H), 1705 (s, C=O), 1638 (C=C), 1583, 1498, 1448 (s, Ph), 1254 (s, P=O), 1177 (s, C-O-C), 1030, 882 (s, P-O-C), 767 (s, P-C) cm^{-1}; ^1H NMR ($CDCl_3$, 400 MHz): δ = 3.62 (s, 3H, $C_6H_4O\underline{CH_3}$), 3.79 (d, J = 9.2 Hz, 6H, OCH_3), 6.24 (d, J = 12.8 Hz, 1H, -OCHP), 6.53 (d, J = 16.0 Hz, 1H, PhCH=CH-), 6.91–7.55 (m, 9H, C_6H_5, C_6H_4), 7.75 (d, J = 16.0 Hz, 1H, PhCH=CH-). ^{31}P NMR ($CDCl_3$): δ = 19.6 ppm. MS (70 eV), m/z (%): 376 (M^+ 18.36); Anal. calcd. for $C_{19}H_{21}O_6P$: C, 60.64; H, 5.62. Found: C, 60.73; H, 5.71%.

(Dimethoxyphosphoryl)(3-Nitrophenyl)Methyl Cinnamate (4l)

Pale yellow liquid, yield 59%; n_D^{20}: 1.5850. IR (KBr): υ = 3065 (w, Ph-H), 2957, 2855 (m, C-H), 1732 (s, C=O), 1634(C=C), 1578, 1532, 1450 (s, Ph), 1265 (s, P=O), 1148 (s, C-O-C), 1036, 907 (s, P-O-C), 768 (s, P-C) cm^{-1}; ^1H NMR ($CDCl_3$, 400 MHz): δ = 3.79 (d, J = 11.2 Hz, 6H, OCH_3), 6.37 (d, J = 13.5 Hz, 1H, -OCHP), 6.58 (d, J = 16.0 Hz, 1H, PhCH=CH-), 7.41–8.40 (m, 9H, C_6H_5, C_6H_4), 7.80 (d, J = 16.0 Hz, 1H, PhCH=CH-). ^{31}P NMR ($CDCl_3$): δ = 20.0 ppm. MS (70 eV), m/z (%): 391 (M^+ 31.62); Anal. calcd. for $C_{18}H_{18}NO_7P$: C, 55.25; H, 4.64; N, 3.58. Found: C, 55.38; H, 4.73; N, 3.79%.

(Dimethoxyphosphoryl)(2-(Furan-2-yl)Phenyl)Methyl Cinnamate (4m)

Pale yellow liquid, yield 72%; n_D^{20}: 1.5625. IR (KBr), υ = 3062 (w, Ph-H), 2957, 2855 (m, C-H), 1723 (s, C=O), 1635(C=C), 1578, 1497, 1450 (s, Ph), 1270 (s, P=O), 1151 (s, C-O-C), 1033, 935 (s, P-O-C), 767 (s, P-C) cm^{-1}; ^1H NMR ($CDCl_3$, 400 MHz): δ = 3.80 (d, J = 10.8 Hz, 6H, OCH_3), 6.42 (d, J = 11.6 Hz, 1H, -OCHP), 6.50 (d, J = 16.0 Hz, 1H, PhCH=CH-), 6.41–7.54 (m, 5H, C_4H_3O, C_6H_5), 7.76 (d, J = 16.0 Hz, 1H,

PhCH=CH-). ^{31}P NMR ($CDCl_3$): $\delta = 16.7$ ppm. MS (70 eV), m/z (%): 336 (M^+ 21.62); Anal. calcd. for $C_{16}H_{17}O_6P$: C, 57.15; H, 5.10. Found: C, 57.32; H, 5.30%.

Herbicidal Testing

Herbicidal testing of the new compounds **4** was carried out in a plant (growth room temperature 23 ± 1 °C, RH 60 ± 5%, light intensity 10 Klux, photoperiod 8 h/day). Twenty seeds of each weed species including rape and wheat were chosen for testing. Seedlings were grown in the 9-cm-diameter test plate containing two pieces of filter paper and 9 mL solution of the tested compound (100 and 10 mg/L, respectively). Distilled water and 2,4-dichlorophenoxyl acetic acid (2,4-D), a commercially available herbicide in the market, were used as comparison compounds. The herbicidal activity was assessed as the inhibition rate in comparison with the distilled water. The herbicidal rating score was based on visual observation, ranging from 0% to 100%: 0% means no effect, 100% means complete killing.

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