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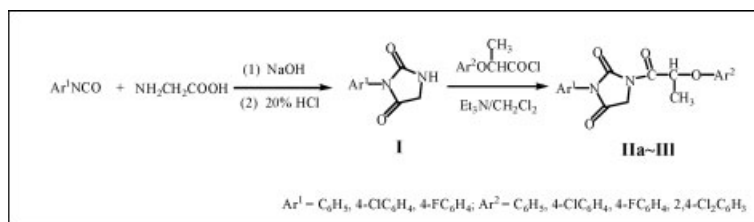
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Received May 21, 2008

DOI 10.1002/jhet.114

Published online 5 May 2009 in Wiley InterScience (www.interscience.wiley.com).



A series of novel 3-aryl-1-[2-(aryloxy)propanoyl]imidazolidine-2,4-diones were synthesized by the condensation of 3-aryl-imidazolidine-2,4-diones with 2-(aryloxy)propanoyl chlorides under mild conditions. Their structures were confirmed by IR, ¹H NMR, mass spectroscopy, and elemental analyses. The preliminary bioassay indicated that the target compounds **II** displayed excellent herbicidal activity against monocotyledonous (barnyard grass) and dicotyledonous (oil rape) plants.

J. Heterocyclic Chem., **46**, 544 (2009).

INTRODUCTION

Recently, imidazolidine-2,4-dione derivatives have attracted the interest of chemists because some of them exhibit wide range of pharmaceutical activity such as anti-HIV [1], microbicidal activity [2], or nonpeptide inhibitors of human heart chymase [3] as well as agrochemical activity such as herbicidal [4] and fungicidal activity [5]. In the area of modern crop protection, amide derivatives are widely used as bactericides [6] and fungicides [7]. Especially, some of the amide derivatives are found to be an inhibitor of photosystem II electron transport and used as a very important class of herbicides in the world agrochemical market, there are about 57 amide herbicides; acetochlor, metolachlor, and butachlor are the best selling amide herbicides in the world [8,9]. As a continuation of our ongoing project aimed at investigating novel biologically nitrogen-containing heterocyclic compounds [10,11], we designed and synthesized a series of novel 3-aryl-1-[2-(aryloxy)propanoyl]imidazolidine-2,4-diones, which have both imidazolidine-2,4-dione and 2-(aryloxy)propanoyl moieties. Herein, we would like to report the synthesis and biological activity of the title compounds **II** (Scheme 1).

RESULTS AND DISCUSSION

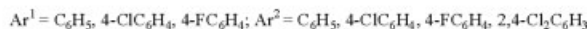
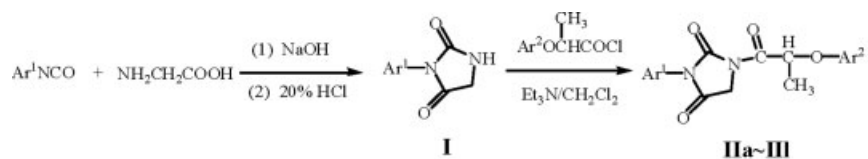
3-Aryl-imidazolidine-2,4-diones (**I**) were prepared from the reaction of aromatic isocyanates with α-amino acids in a basic medium according to the reported method. Compound **I** reacted with various 2-(aryloxy)-

propanoyl chlorides under mild conditions to give the target compounds **II** in good yields (73–90%).

Their structures of the products were confirmed by IR, ¹H NMR, mass spectroscopy, and elemental analyses. The structures of compounds **II** were deduced from their spectroscopic data. In the ¹H NMR spectra of compounds **II**, the two methylene protons display two doublets because of their different magnetic environments with the coupling constant of 18 Hz, whereas the two methylene protons in compounds **I** appear as a singlet at δ 4.1. The IR spectra of compounds **II** showed normal stretching absorption bands indicating the existence of the C=O, Ar group, and C—O—C moiety. The EI mass spectra of compounds **II** revealed the existence of their molecular ion peaks and main fragmentation peaks, which were in accordance with the given structures of products **II**.

Herbicidal activity. The herbicidal activity values of the title compounds **II** against *Brassica campestris* (oil rape) and *Echinochloa crus-galli* (barnyard grass) has been investigated at the dosages of 100 mg/L and 10 mg/L compared with distilled water and the commercially available herbicide, 2,4-dichlorophenoxyacetic acid (2,4-D) according to the method described in the Experimental section. The preliminary results of bioassay showed that compounds **II** possessed excellent herbicidal activity. The activity data were listed in Table 1. For example, compounds **IIh** and **IIj** exhibited as good herbicidal activity as the commercially available herbicide 2,4-D. Further structure-activity relationships are under investigation.

Scheme 1. Synthetic route to compounds II.



In conclusion, a series of novel 3-aryl-1-[2-(aryloxy)propanoyl]imidazolidine-2,4-diones were synthesized by the condensation of 3-aryl-imidazolidine-2,4-diones with 2-[aryloxy]propanoyl chlorides under mild conditions. The preliminary bioassay indicated that the target compounds II displayed excellent herbicidal activity against monocotyledonous (barnyard grass) and dicotyledonous (oil rape) plants.

EXPERIMENTAL

Melting points were determined with a WRS-1B digital melting point apparatus and are uncorrected. ¹H NMR spectra were recorded with a Varian Mercury PLUS400 (400 MHz) spectrometer with TMS as the internal reference and CDCl₃ as the solvent, whereas mass spectra were obtained with a Finnigan TRACEMS2000 spectrometer using the EI method. IR spectra were measured by a Nicolet NEXUS470 spectrometer. Elemental analyses were performed with an Elementar Vario ELIII CHNSO elemental analyzer. All of the solvents and materials were reagent grade and purified as required. 2-(Aryloxy)propanoyl chlorides and 3-aryl-imidazolidine-2,4-diones were prepared according to the methods described in references [12] and [13–16], respectively.

Table 1

The herbicidal activity of compounds II (inhibitory rate %)

Compd.	Relative inhibition (root %/stalk %)			
	Oil rape		Barnyard grass	
	100 mg/L	10 mg/L	100 mg/L	10 mg/L
IIa	99.0/93.0	94.3/74.4	97.5/33.3	75.0/23.0
IIb	98.1/93.0	97.1/83.7	97.5/23.8	90.0/4.8
IIc	99.0/93.0	97.1/86.0	100/88.0	95.0/73.8
IId	100/95.3	99.0/86.0	97.5/83.3	95.0/78.6
IIe	99.0/95.3	98.1/86.0	97.5/64.3	97.5/31.0
IIf	99.3/95.5	97.1/86.0	97.5/65.3	92.5/38.0
IIg	99.0/93.0	98.1/88.4	100/52.4	97.5/52.4
IIh	100/97.7	99.0/81.4	97.5/78.6	97.5/64.3
IIi	100/97.7	98.1/86.0	97.5/59.5	95.0/52.4
IIj	100/95.3	98.1/83.7	100/90.5	97.5/33.3
IIk	99.0/95.3	98.1/83.7	97.5/54.8	97.5/50.0
III	99.0/95.3	98.1/86.0	100/95.0	97.5/61.9
2,4-D	99.0/91.5	98.2/91.2	97.5/33.5	97.5/31.2

General procedure for the preparation of 3-aryl-1-[2-(aryloxy)propanoyl]imidazolidine-2,4-diones II. To the solution of 3-aryl-imidazolidine-2,4-dione (3 mmol) and triethyl amine (6 mmol) in dry CH₂Cl₂ (15 mL) was added dropwise the solution of 2-(aryloxy)propanoyl chloride (3 mmol) in dry CH₂Cl₂ (10 mL) while cooling in an ice-bath, after the addition, the mixture was stirred at room temperature for 3–4 h (monitored by TLC). The workup involved stripping the solvent followed by an addition of water and extraction of the product mixture into CH₂Cl₂. After phase separation, washing by aqueous sodium hydrogen carbonate, drying over anhydrous sodium sulfate, filtration and evaporation, the crude product was purified by flash column chromatography on silica gel using petroleum ether and ethyl acetate (3:1--1:1, v/v) as the eluent to give white crystals (IIa–III).

1-(2-Phenoxypropanoyl)-3-phenylimidazolidine-2,4-dione (IIa). Yield: 75%, mp 56.2–57.8°C; ¹H NMR (CDCl₃): δ 1.70 (d, *J* = 6.8 Hz, 3H, CH₃), 4.57 (dd, *J* = 18.4 Hz, 2H, CH₂N), 5.95 (q, *J* = 6.4 Hz, 1H, CH), 6.90–7.00 (m, 3H, Ar–H), 7.28–7.30 (m, 2H, Ar–H), 7.40–7.55 (m, 5H, Ar–H); IR: C=O 1786, 1738, 1725, Ar 1592, 1484, 1445, Ar–O–C 1040, C–Cl 756 cm⁻¹; ms: *m/z* 324 (M⁺, 16), 247 (23.9), 231 (100), 122 (35.2), 121 (90.2), 105 (15.9), 93 (35.5), 77 (85.6). *Anal.* Calcd. for C₁₈H₁₆N₂O₄: C, 66.66; H, 4.97; N, 8.64. Found: C, 66.42; H, 5.13; N, 8.79.

3-(4-Fluorophenyl)-1-(2-phenoxypropanoyl)imidazolidine-2,4-dione (IIb). Yield: 73%, mp 56.8–58.2°C; ¹H NMR (CDCl₃): δ 1.71 (d, *J* = 6.8 Hz, 3H, CH₃), 4.47 (dd, *J* = 18.8 Hz, 2H, CH₂N), 5.92 (q, *J* = 6.8 Hz, 1H, CH); 6.90 (d, 2H, *J* = 7.6 Hz, Ar–H), 6.97 (q, 1H, *J* = 7.6 Hz, Ar–H), 7.25–7.30 (m, 2H, Ar–H), 7.37–7.42 (m, 2H, Ar–H), 7.48–7.51 (m, 2H, Ar–H); IR: C=O 1784, 1739, 1727, Ar 1587, 1489, 1425, Ar–O–C 1078, C–Cl 760 cm⁻¹; ms: *m/z* 344 (10.1), 342 (M⁺, 32), 267 (40.6), 265 (100), 223 (15.5), 153 (27.2), 122 (78.1), 121 (78.1), 105 (15.9), 93 (24.9), 77 (70.2). *Anal.* Calcd. for C₁₈H₁₅FN₂O₄: C, 63.15; H, 4.42; N, 8.18. Found: C, 63.37; H, 4.60; N, 8.05.

3-(4-Chlorophenyl)-1-(2-phenoxypropanoyl)imidazolidine-2,4-dione (IIc). Yield: 78%, mp 66.8–68.4°C; ¹H NMR (CDCl₃): δ 1.69 (d, *J* = 7.2 Hz, 3H, CH₃), 4.45 (dd, *J* = 18.8 Hz, 2H, CH₂N), 5.93 (q, *J* = 6.4 Hz, 1H, CH); 6.90 (d, 2H, *J* = 7.6 Hz, Ar–H), 6.97 (q, 1H, *J* = 7.6 Hz, Ar–H), 7.17–7.29 (m, 4H, Ar–H), 7.37–7.41 (m, 2H, Ar–H); IR: C=O 1756, 1725, Ar 1578, 1494, 1482, Ar–O–C 1050, C–Cl 759 cm⁻¹; ms: *m/z* 360 (7.5), 358 (M⁺, 22.5), 281 (27.7), 238 (100), 122 (65.0), 121 (88.1), 105 (12.8), 93 (38.7), 77 (65.3). *Anal.* Calcd. for C₁₈H₁₅ClN₂O₄: C, 60.26; H, 4.21; N, 7.81. Found: C, 60.33; H, 4.49; N, 7.92.

1-[2-(2,4-Dichlorophenoxy)propanoyl]-3-phenylimidazolidine-2,4-dione (II d). Yield: 81%, mp 52.6–54.2°C; ¹H NMR

(CDCl₃): δ 1.59 (d, J = 6.8 Hz, 3H, CH₃), 4.63 (dd, J = 18.8 Hz, 2H, CH₂N), 5.86 (q, J = 6.8 Hz, 1H, CH); 6.88–7.33 (m, 8H, Ar—H); IR: C=O 1800, 1746, 1725, Ar 1585, 1488, 1440, Ar—O—C 1022, C—Cl 760 cm⁻¹; ms: m/z 394 (16.2), 393 (5.1), 392 (M⁺, 25.0), 315 (100), 122 (45.4), 121 (62.6), 105 (19.5), 93 (53.8), 77 (25.2). *Anal.* Calcd. for C₁₈H₁₄Cl₂N₂O₄: C, 54.98; H, 3.59; N, 7.12. Found: C, 54.83; H, 3.47; N, 6.95.

1-[2-(2,4-Dichlorophenoxy)propanoyl]-3-(4-fluorophenyl)imidazolidine-2,4-dione (IIe). Yield: 76%, mp 54.2–55.4°C; ¹H NMR (CDCl₃): δ 1.74 (d, J = 6.8 Hz, 3H, CH₃), 4.50 (dd, J = 18.4 Hz, 2H, CH₂N), 5.86 (q, J = 6.8 Hz, 1H, CH); 6.85 (d, 1H, J = 8.8 Hz, Ar—H), 7.14 (d, 1H, J = 8.4 Hz, Ar—H), 7.33–7.40 (m, 3H, Ar—H), 7.48 (d, 2H, J = 8.4 Hz, Ar—H). IR: C=O 1787, 1740, 1724, Ar 1596, 1487, 1436, Ar—O—C 1076, C—Cl 748 cm⁻¹; ms: m/z 412 (5.2), 410 (M⁺, 26.5), 317 (23.8), 300 (100), 111 (58.2), 95 (62.0). *Anal.* Calcd. for C₁₈H₁₃Cl₂FN₂O₄: C, 52.57; H, 3.19; N, 6.81. Found: C, 52.69; H, 3.33; N, 6.57.

3-(4-Chlorophenyl)-1-[2-(2,4-dichlorophenoxy)propanoyl]imidazolidine-2,4-dione (IIf). Yield: 87%, mp 54.2–55.4°C; ¹H NMR (CDCl₃): δ 1.74 (d, J = 6.8 Hz, 3H, CH₃), 4.92 (dd, J = 18.4 Hz, 2H, CH₂N), 5.84 (q, J = 6.4 Hz, 1H, CH); 6.84 (d, 1H, J = 8.0 Hz, Ar—H), 7.13 (q, 1H, J = 7.6 Hz, Ar—H), 7.19 (d, 2H, J = 8.0 Hz, Ar—H), 7.38 (d, 1H, J = 7.6 Hz, Ar—H), 7.40 (d, 2H, J = 8.0 Hz, Ar—H); IR: C=O 1803, 1740, 1728, Ar 1594, 1496, 1485, Ar—O—C 1020, C—Cl 746 cm⁻¹; ms: m/z 430 (2.9), 428 (11.2), 426 (M⁺, 13.3), 268 (29.3), 267 (14.6), 265 (100), 237 (3.2), 223 (8.4), 211 (10.7), 189 (6.2), 161 (4.7), 153 (8.6), 125 (6.5), 109 (2.3), 56 (4.1). *Anal.* Calcd. for C₁₈H₁₃Cl₃N₂O₄: C, 50.55; H, 3.06; N, 6.55. Found: C, 50.41; H, 2.94; N, 6.34.

1-[2-(4-Chlorophenoxy)propanoyl]-3-phenylimidazolidine-2,4-dione (IIg). Yield: 86%, mp 74.7–75.5°C; ¹H NMR (CDCl₃): δ 1.69 (d, J = 6.8 Hz, 3H, CH₃), 4.50 (dd, J = 18.4 Hz, 2H, CH₂N), 5.92 (q, J = 6.8 Hz, 1H, CH); 6.84 (d, 2H, J = 7.6 Hz, Ar—H), 7.23 (d, 2H, J = 7.6 Hz, Ar—H), 7.41–7.54 (m, 5H, Ar—H); IR: C=O 1741, 1726, Ar 1597, 1490, CH₃ 1403, Ar—O—C 1046, C—Cl 762 cm⁻¹; ms: m/z 360 (4.2), 358 (M⁺, 12.1), 267 (12.4), 265 (100), 237 (36.8), 209 (52.3), 105 (9.3), 93 (52.5), 77 (36.0). *Anal.* Calcd. for C₁₈H₁₅ClN₂O₄: C, 60.26; H, 4.21; N, 7.81. Found: C, 60.05; H, 4.14; N, 7.60.

1-[2-(4-Chlorophenoxy)propanoyl]-3-(4-fluorophenyl)imidazolidine-2,4-dione (IIh). Yield: 90%, mp 147.8–149.0°C; ¹H NMR (CDCl₃): δ 1.69 (d, J = 6.8 Hz, 3H, CH₃), 4.47 (dd, J = 18.4 Hz, 2H, CH₂N), 5.86 (q, J = 6.8 Hz, 1H, CH); 6.84 (d, 2H, J = 8.0 Hz, Ar—H), 7.20–7.24 (m, 2H, Ar—H), 7.35–7.38 (m, 2H, Ar—H), 7.47–7.50 (m, 2H, Ar—H); IR: C=O 1741, 1713, Ar 1500, 1488, CH₃ 1386, C—F 1267, Ar—O—C 1090, C—Cl 821 cm⁻¹; ms: m/z 378 (2.5), 376 (M⁺, 16.3), 237 (82.3), 210 (100), 139 (24.6), 112 (68.2), 96 (70.2). *Anal.* Calcd. for C₁₈H₁₄ClFN₂O₄: C, 57.38; H, 3.75; N, 7.44. Found: C, 57.47; H, 3.70; N, 7.61.

1-[2-(4-Chlorophenoxy)propanoyl]-3-(4-chlorophenyl)imidazolidine-2,4-dione (IIi). Yield: 83%, mp 74.7–75.5°C; ¹H NMR (CDCl₃): δ 1.69 (d, J = 6.8 Hz, 3H, CH₃), 4.46 (dd, J = 18.4 Hz, 2H, CH₂N), 5.87 (q, J = 6.8 Hz, 1H, CH); 6.82–6.86 (m, 2H, Ar—H), 7.19–7.25 (m, 4H, Ar—H), 7.37–7.41 (m, 2H, Ar—H); IR: C=O 1801, 1737, 1730, Ar 1585, 1494, 1481, Ar—O—C 1022, C—Cl 760 cm⁻¹; ms: m/z 394 (4.8), 392 (M⁺, 24.1), 267 (25.1), 265 (100), 237 (65.4), 209 (38.6), 183 (4.9),

127 (58.6), 111 (24.2). *Anal.* Calcd. for C₁₈H₁₄Cl₂N₂O₄: C, 54.98; H, 3.59; N, 7.12. Found: C, 54.83; H, 3.71; N, 7.21.

1-[2-(4-Fluorophenoxy)propanoyl]-3-phenylimidazolidine-2,4-dione (IIj). Yield: 85%, mp 64.2–65.5°C; ¹H NMR (CDCl₃): δ 1.67 (d, J = 6.8 Hz, 3H, CH₃), 4.47 (dd, J = 18.8 Hz, 2H, CH₂N), 5.85 (q, J = 6.4 Hz, 1H, CH); 6.85–6.98 (m, 4H, Ar—H), 7.38–7.54 (m, 5H, Ar—H); IR: C=O 1738, 1716, Ar 1599, 1504, 1436, CH₃ 1402, C—F 1262, 1201, Ar—O—C 1095; ms: m/z 358 (M⁺, 3.1), 267 (21.8), 265 (100), 238 (10.5), 210 (45.0), 122 (30.2), 121 (56.9), 105 (9.0), 93 (41.0), 77 (25.2). *Anal.* Calcd. for C₁₈H₁₅FN₂O₄: C, 63.15; H, 4.42; N, 8.18. Found: C, 63.30; H, 4.67; N, 7.95.

1-[2-(4-Fluorophenoxy)propanoyl]-3-(4-fluorophenyl)imidazolidine-2,4-dione (IIk). Yield: 82%, mp 162.2–163.8°C; ¹H NMR (CDCl₃): δ 1.68 (d, J = 6.8 Hz, 3H, CH₃), 4.50 (dd, J = 18.8 Hz, 2H, CH₂N), 5.84 (q, J = 6.8 Hz, 1H, CH); 6.86–6.89 (m, 2H, Ar—H), 6.95–6.99 (m, 2H, Ar—H), 7.38 (d, 2H, J = 8.8 Hz, Ar—H), 7.50 (d, 2H, J = 8.8 Hz, Ar—H); IR: C=O 1787, 1739, 1720, Ar 1576, 1514, 1462, Ar—O—C 1060, C—Cl 765 cm⁻¹; ms: m/z 362 (2.0), 360 (M⁺, 4.3), 267 (41.7), 265 (100), 223 (10.2), 211 (13.6), 153 (8.8), 139 (21.4), 125 (4.9), 111 (9.8), 95 (10.2). *Anal.* Calcd. for C₁₈H₁₄F₂N₂O₄: C, 60.00; H, 3.92; N, 7.77. Found: C, 59.81; H, 3.75; N, 7.64.

3-(4-chlorophenyl)-1-[2-(4-fluorophenoxy)propanoyl]imidazolidine-2,4-dione (III). Yield: 82%, mp 66.8–68.4°C; ¹H NMR (CDCl₃): δ 1.67 (d, J = 6.8 Hz, 3H, CH₃), 4.48 (dd, J = 18.4 Hz, 2H, CH₂N), 5.85 (q, J = 6.8 Hz, 1H, CH); 6.86–6.99 (m, 4H, Ar—H), 7.29–7.51 (m, 4H, Ar—H); IR: C=O 1785, 1740, 1722, Ar 1586, 1494, 1463, Ar—O—C 1074, C—Cl 758 cm⁻¹; ms: m/z 379 (5.9), 378 (5.8), 376 (M⁺, 28.1), 269 (4.4), 267 (40.9), 266 (62.7), 265 (100), 237 (8.2), 223 (13.6), 211 (19.4), 153 (30.4), 140 (44.7), 139 (66.5), 125 (15.7), 111 (37.3), 95 (46.9), 83 (24.3), 56 (53.2). *Anal.* Calcd. for C₁₈H₁₄ClFN₂O₄: C, 57.38; H, 3.75; N, 7.44. Found: C, 57.14; H, 3.83; N, 7.60.

Bioassay method. Herbicidal activity testing. Herbicidal testing of the newly synthesized compounds II was carried out in a greenhouse, with temperature 23 ± 1°C, relative humidity (RH) 60 ± 5%, light intensity 10 Klux, photoperiod 8 h/day. Twenty seeds of each weed species including oil rape and barnyard grass were chosen for testing. Seedlings were grown in the test plate of 9-cm diameter containing two pieces of filter paper and 9 mL solution of the tested compound (100 mg/L and 10 mg/L, respectively). Distilled water and 2,4-D were used as the comparison compounds. The herbicidal activity was assessed as the inhibitory rate in comparison with the distilled water. The herbicidal rating score was based on visual observation. Range from 0 to 100%, 0% means no effect and 100% means complete killing. The test was run three times, and the results were averaged and given as activity in Table 1.

Acknowledgments. This work was supported by the Natural Science Foundation of China (Grant numbers: 20302002, 20872046), and the Scientific Research Foundation for the Returned Overseas Chinese Scholars, Ministry of Education of China (Grant number: [2007] 1108).

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