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A convenient one-pot synthesis and bioactivity of *N*-dichloroacetyl-5-aryl-1,3-oxazolidines

Abstract: New *N*-dichloroacetyl-5-aryl-1,3-oxazolidines **4** were synthesized by cycloaddition reaction of an aryl substituted hydroxyalkylamine **1** with aldehyde or ketone **2**, followed by acylation, without isolation of the intermediate product **3**. The structures of compounds **4** were determined by spectral and elemental analyses. The structure of **4b** was determined by an X-ray crystallographic analysis. The bioassay results demonstrate that these compounds could alleviate chlorsulfuron injury to maize.

Keywords: crystal structure; *N*-dichloroacetyl-5-aryl-oxazolidines; one-pot synthesis; safener activity.

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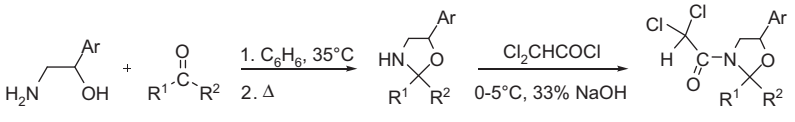
Introduction

1,3-Oxazolidines are prominent heterocyclic compounds that are used not only as intermediates in the synthesis of various organic compounds [1, 2] but also as ligands to catalyze asymmetric synthesis [3, 4]. *N*-Dichloroacetyloxazolidines are also an important class of oxazolidine derivatives that exhibit extensive biological activities [5–7]. It is known in agricultural biochemistry that *N*-dichloroacetyloxazolidines affect herbicide absorption, metabolism, and target enzyme activity [8–10]. In addition, *N*-dichloroacetyloxazolidines act as a herbicide safener by increasing the activities of glutathione-*S*-transferase (GST) to catalyze the conjugation of glutathione (GSH) with some herbicides [11].

Several general methods for the synthesis of various 1,3-oxazolidines have been reported. 1,3-Oxazolidine-2,4-diones have been synthesized by the reaction of readily available α-ketols and isocyanates [12]. Tetrahydro-2*H*-oxazolothiazoles have been prepared by intramolecular 1,3-dipolar cycloaddition reaction [13]. Darabantu et al. synthesized diastereoselectively 1,3-oxazolidines in the presence of α,α,α-trimethylolaminomethane (TRIS) and related aminopolyols [14, 15]. There are limited reports on the preparation of 5-phenyl-1,3-oxazolidines by cyclocondensation of phenylethanolamines with cyclohexanone or another ketone in the presence of K_2CO_3 [16, 17]. In this report, we describe a convenient one-pot synthetic approach to a novel series of *N*-dichloroacetyl-5-aryl-1,3-oxazolidine derivatives. The reaction is carried out without isolation of the intermediate product and in the absence of any catalyst (Scheme 1). Bioassay was carried out for determining the safener activities of the compounds.



4a: Ar = thienyl; $R^1 = R^2$ = Me
4b: Ar = Ph; $R^1 = R^2$ = Me
4c: Ar = *p*-Cl-Ph; $R^1 = R^2$ = Me
4d: Ar = *p*-Me-Ph; $R^1 = R^2$ = Me
4e: Ar = *p*-MeO-Ph; $R^1 = R^2$ = Me
4f: Ar = furyl; $R^1 = R^2$ = Me
4g: Ar = furyl; R^1 = Me; $R^2 = {}^n$Pr
4h: Ar = furyl; $R^1 = R^2$ = Et
4i: Ar = furyl; $R^1 = R^2$ = H
4j: Ar = furyl; R^1 = H; R^2 = Ph
4k: Ar = furyl; R^1 = Me; $R^2 = CH_2CH_2Ph$
4l: Ar = furyl; R^1, $R^2 = (CH_2)_5$

Scheme 1

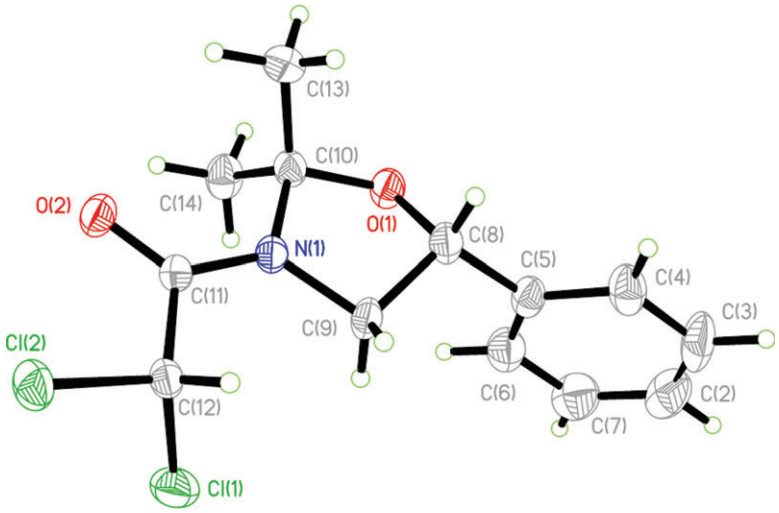


Figure 1 Molecular structure for compound **4b** at 30% probability level.

Results and discussion

Products **4** were obtained in moderate yields by using cycloaddition and acylation reactions [18]. It can be concluded that the presence of a substituent at the *para* position of the aryl group does not significantly affect the yields of the products. The yield of the second acylation step is affected by steric effects of substituents R^1 and R^2 at position 2 of 1,3-oxazolidine.

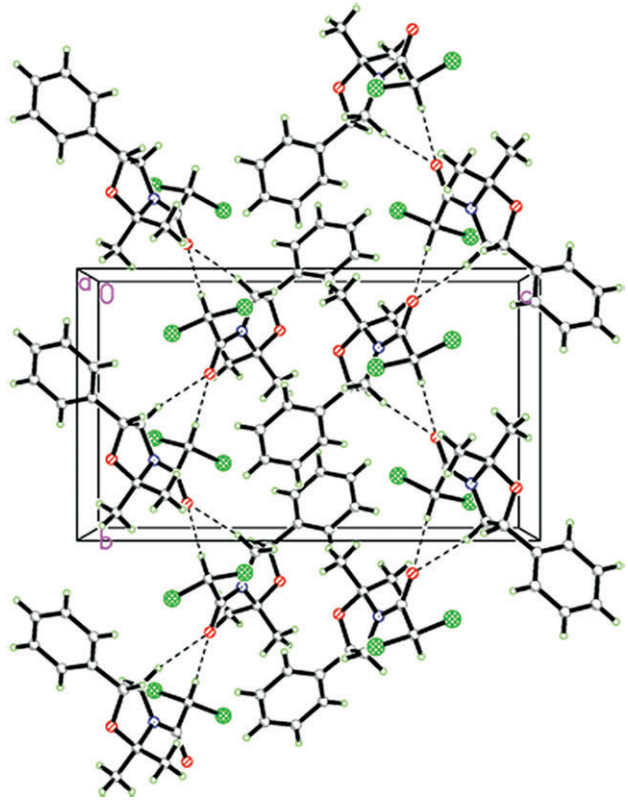


Figure 2 Packing view of the compound **4b**.

The structures of compounds **4a–l** were confirmed by elemental analysis and spectroscopic techniques. In particular, ^{1}H NMR spectra of **4a–l** exhibit the characteristic singlet around δ 6.05 for the Cl_2CH moiety. In the ^{13}C NMR spectra of the synthesized compounds, the signals observed in the region δ 95–100, δ 65–70, and δ 50–55 account for the three carbon atoms of the oxazolidine ring.

The single crystal of **4b** was obtained by slow evaporation of the solution in ethanol and light petroleum. The molecular structure and the packing view of **4b** are shown in Figures 1 and 2, respectively. All bond lengths and angles are in the expected ranges.

Compounds **4a–l** were evaluated for their protection of maize *in vivo* against injury by chlorsulfuron, a sulfonylurea herbicide, at a concentration of 2 μg/kg. Most of the compounds showed notable herbicidal safener activities by increasing GST and acetolactate synthase (ALS) activities. Compound **4c** exhibits a relatively higher effect on GSH and GST than others, whereas the effect of compound **4i** is the most profound.

Conclusion

The current work affords a facile strategy of the synthesis of a series of novel *N*-dichloroacetyl-5-aryl-1,3-oxazolidines. All compounds exhibit safener activities to chlorsulfuron.

Experimental

Infrared (IR) spectra were taken on a KJ-IN-27G infrared spectrophotometer using KBr pellets. ^{1}H NMR spectra (300 MHz) and ^{13}C NMR spectra (75 MHz) were recorded on a Bruker Avance 300 MHz nuclear magnetic resonance spectrometer in $CDCl_3$ (unless stated otherwise). Elemental analysis was performed on a Flash EA1112 elemental analyzer. X-Ray diffraction data were collected on a Bruker AXS II CCD area-detector diffractometer, Mo Kα. Melting points were determined on a Beijng Taike melting point apparatus (X-4) and are uncorrected. All reagents were of analytical grade. Reactions and products were routinely monitored by thin layer chromatography (TLC) on silica gel.

Typical procedure for the synthesis *N*-dichloroacetyl-5-aryl-1,3-oxazolidine 4a–l

Aryl substituted hydroxyalkylamine **1** (0.025 mol) and aldehyde or ketone **2** (0.030 mol) were added to benzene (35 mL) and the mixture was stirred briefly at 33–35°C followed by heating under reflux with azeotropic removal of water with benzene. Then the mixture was cooled to 0°C and treated with sodium hydroxide solution (33%, 0.03 mol). Afterwards, dichloroacetyl chloride (0.030 mol) was added dropwise with stirring and cooling in an ice bath. Stirring was continued for 2 h. The organic phase was washed with water and dried over anhydrous magnesium sulfate. The crude products were crystallized from a mixture of ethyl acetate and light petroleum.

***N*-Dichloroacetyl-5-(2′-thienyl)-2,2-dimethyl-1,3-oxazolidine (4a)** White crystals; yield 65%; mp 107–109°C; IR: ν 3250–2900 (C-H), 1681 (C=O), 1415 (Cl_2HC-CO-), 1217 cm^{-1} (N-C-O); ^{1}H NMR: δ 7.36–7.38 (m, 1H, Ar-H), 7.15 (d, J = 3.5 Hz, 1H, ArH), 7.03 (m, 1H, ArH), 6.05 (s, 1H, Cl_2CH), 5.40 (m, 2H, NCH_2), 3.73 (t, J = 9.7 Hz, 1H, OCH), 1.75 (s, 3H, CH_3), 1.70 (s, 3H, CH_3); ^{13}C NMR: δ 159.5, 139.5, 126.9, 126.4, 126.4, 97.0, 72.7, 67.0, 52.9, 25.4, 23.5. Anal. Calcd for $C_{11}H_{13}Cl_2NO_2S$: C, 45.05; H, 4.47; N, 4.78; S, 10.91. Found: C, 45.12; H, 4.42; N, 4.71; S, 10.85.

***N*-Dichloroacetyl-5-phenyl-2,2-dimethyl-1,3-oxazolidine (4b)** White crystals; yield 85%; mp 110–111°C; IR: ν 3250–2900 (C-H), 1610 (C=O), 1417 (Cl_2HC-CO-), 1244 cm^{-1} (N-C-O); ^{1}H NMR: δ 7.43 (m, 5H, ArH), 6.06 (s, 1H, Cl_2CH), 5.18, 4.26 (m, 2H, NCH_2), 3.54–3.60 (t, J = 9.7 Hz, 1H, OCH), 1.80 (s, 3H, CH_3), 1.72 (s, 3H, CH_3); ^{13}C NMR: δ 159.6, 136.7, 129.0, 128.8, 128.8, 126.4, 126.4, 96.9, 76.5, 67.0, 52.9, 25.4, 23.3. Anal. Calcd for $C_{13}H_{15}Cl_2NO_2$: C, 54.35; H, 5.27; N, 4.88. Found: C, 54.31; H, 5.24; N, 4.92.

***N*-Dichloroacetyl-5-(*p*-chlorophenyl)-2,2-dimethyl-1,3-oxazolidine (4c)** White crystals; yield 84%; mp 87–89°C; IR: ν 3022–3100 (C-H), 1672 (C=O), 1515 (Cl_2HC-CO-), 1218 cm^{-1} (N-C-O); ^{1}H NMR: δ 7.38 (m, 4H, ArH), 6.04 (s, 1H, Cl_2CH), 5.15, 4.26 (m, 2H, NCH_2), 3.46 (t, J = 9.9 Hz, 1H, OCH), 1.77 (s, 3H, CH_3), 1.70 (s, 3H, CH_3); ^{13}C NMR: δ 159.6, 135.3, 134.7, 129.0, 129.0, 127.7, 127.7, 97.0, 75.8, 67.0, 52.7, 25.4, 23.3. Anal. Calcd for $C_{13}H_{14}Cl_3NO_2$: C, 48.60; H, 4.40; N, 4.36. Found: C, 48.69; H, 4.49; N, 4.28.

***N*-Dichloroacetyl-5-(*p*-methylphenyl)-2,2-dimethyl-1,3-oxazolidine (4d)** White crystals; yield 86%; mp 96–98°C; IR: ν 3018–2958 (C-H), 1670 (C=O), 1545 (Cl_2HC-CO-), 1215 cm^{-1} (N-C-O). ^{1}H NMR ($CDCl_3$): δ 7.29 (m, 2H, ArH), 6.91 (m, 2H, ArH), 6.04 (s, 1H, Cl_2CH), 5.18 (m, 2H, NCH_2), 3.80 (s, 3H, CH_3), 3.49–3.56 (t, J = 9.9 Hz, 1H, OCH), 1.75 (s, 3H, CH_3), 1.68 (s, 3H, CH_3); ^{13}C NMR: δ 160.1, 159.6, 128.5, 127.9, 127.9, 114.2, 114.2, 96.7, 76.2, 67.0, 55.4, 52.9, 25.4, 23.3. Anal. Calcd for $C_{14}H_{17}Cl_2NO_2$: C, 55.80; H, 5.69; N, 4.65. Found: C, 55.89; H, 5.73; N, 4.58.

***N*-Dichloroacetyl-5-(*p*-methoxyphenyl)-2,2-dimethyl-1,3-oxazolidine (4e)** White crystals; yield 88%; mp 79–81°C; IR: ν 3022–3000 (C-H), 1672 (C=O), 1514 (Cl_2HC-CO-), 1247 cm^{-1} (N-C-O); ^{1}H NMR: δ 7.27 (m, 4H, ArH), 6.06 (s, 1H, Cl_2CH), 4.23 (m, 2H, NCH_2), 3.52–3.58 (t, J = 9.9 Hz, 1H, OCH), 2.38 (s, 3H, CH_3), 1.78 (s, 3H, CH_3), 1.70 (s, 3H, CH_3); ^{13}C NMR: δ 159.6, 138.9, 133.6, 129.5, 129.5, 126.4, 126.4, 96.8, 76.4, 67.0, 52.9, 25.4, 23.3, 21.3. Anal. Calcd for $C_{14}H_{17}Cl_2NO_3$: C, 52.99; H, 5.40; N, 4.42. Found: C, 53.08; H, 5.46; N, 4.34.

***N*-Dichloroacetyl-5-(2′-furyl)-2,2-dimethyl-1,3-oxazolidine (4f)** White crystals; yield 91%; mp 98–99°C. IR: ν 3250–3000 (C-H), 1674 (C=O), 1423 (Cl_2HC-CO-), 1139 cm^{-1} (N-C-O); ^{1}H NMR: δ 7.44 (m, 1H, ArH), 6.46 (d, J = 3.3 Hz, 1H, ArH), 6.38 (m, 1H, ArH), 6.05 (s, 1H, Cl_2CH-), 5.18 (m, 2H, NCH_2), 3.97 (m, 1H, OCH), 1.69 (s, 3H, CH_3), 1.64 (s, 3H, CH_3); ^{13}C NMR: δ 159.6, 149.0, 143.6, 110.6, 110.0, 96.9, 69.9, 67.0, 48.9, 25.3, 23.5. Anal. Calcd for $C_{11}H_{13}Cl_2NO_3$: C, 47.65; H, 4.73; N, 5.05. Found: C, 47.69; H, 4.71; N, 5.02.

***N*-Dichloroacetyl-5-(2′-furyl)-2-methyl-2-n-propyl-1,3-oxazolidine (4g)** White crystals; yield 84%; mp 149–151°C; IR: ν 3083–2989 (C-H), 1681 (C=O), 1423 (Cl_2HC-CO-), 1143 cm^{-1} (N-C-O); ^{1}H NMR: δ 7.47 (m, 1H, Ar-H), 6.49 (m, 1H, Ar-H), 6.40 (m, 1H, Ar-H), 6.08 (s, 1H, Cl_2CH), 4.20 (m, 2H, N-CH_2), 3.90 (t, J = 9.9 Hz, 1H, O-CH), 1.64 (s, 3H, CH_3), 1.32 (m, 4H, CH_2-CH_2), 0.93 (m, 3H, CH_3); ^{13}C NMR: δ 159.5, 148.8, 143.7, 110.6, 110.1, 98.8, 69.8, 67.1, 49.9, 39.1, 22.9, 16.1, 13.9. Anal. Calcd for $C_{13}H_{17}Cl_2NO_3$: C, 51.14; H, 5.62; N, 4.59. Found: C, 51.21; H, 5.56; N, 4.54.

***N*-Dichloroacetyl-5-(2′-furyl)-2,2-diethyl-1,3-oxazolidine (4h)** White crystals; yield 74%; mp 68–70°C; IR: ν 3050–2979 (C-H), 1660 (C=O), 1417 (Cl_2HC-CO-), 1168 cm^{-1} (N-C-O); ^{1}H NMR: δ 7.47 (m, 1H, Ar-H), 6.48 (m, 1H, Ar-H), 6.40 (m, 1H, Ar-H), 6.11 (s, 1H, Cl_2CH), 4.26 (m, 2H, N-CH_2), 3.97 (t, J = 9.9 Hz, 1H, O-CH), 1.82–2.36 (m, 4H, 2 × CH_2), 0.88–0.99 (m, 6H, 2 × CH_3); ^{13}C NMR: δ 159.5, 149.4, 143.6, 110.6, 109.9, 102.1, 70.7, 67.1, 50.2, 29.3, 28.0, 8.3, 7.0. Anal. Calcd for $C_{13}H_{17}Cl_2NO_3$: C, 51.14; H, 5.62; N, 4.59. Found: C, 51.18; H, 5.68; N, 4.48.

***N*-Dichloroacetyl-5-(2′-furyl)-1,3-oxazolidine (4i)** Yellow oil; yield 64%; IR: ν 3050–3000 (C-H), 1690 (C=O), 1450 (Cl_2HC-CO-), 1200 cm^{-1} (N-C-O); ^{1}H NMR: δ 7.47 (m, 1H, Ar-H), 6.43 (m, 2H, Ar-H), 6.08 (s, 1H, Cl_2CH-), 5.11–5.41 (m, 3H, N-CH_2-O and CH-O), 3.88–4.18 (m, 2H, N-CH_2); ^{13}C NMR: δ 160.4, 149.2, 143.6, 110.6, 109.7, 79.7, 74.1, 66.0, 47.4. Anal. Calcd for $C_9H_9Cl_2NO_3$: C, 43.37; H, 3.64; N, 5.62. Found: C, 43.29; H, 3.74; N, 5.74.

***N*-Dichloroacetyl-5-(2′-furyl)-2-phenyl-1,3-oxazolidine (4j)** White crystals; yield 61%; mp 78–79°C; IR: ν 3250–2900 (C-H), 1681 (C=O), 1421 (Cl_2HC-CO-), 1168 cm^{-1} (N-C-O); ^{1}H NMR: δ 7.45 (m, 8H, ArH), 6.53 (s, 1H, Cl_2CH), 6.13 (s, 1H, H-C), 5.39 (t, J = 6.04 Hz, 1H, O-CH), 4.38, 4.21 (m, 2H, N-CH_2); ^{13}C NMR: δ 160.9, 149.9, 143.5, 137.1, 129.5, 129.3, 129.3, 126.5, 126.5, 110.6, 109.5, 90.1, 72.3, 66.3, 48.5. Anal. Calcd for $C_{15}H_{13}Cl_2NO_3$: C, 55.38; H, 4.03; N, 4.31. Found: C, 55.42; H, 4.01; N, 4.35.

***N*-Dichloroacetyl-5-(2′-furyl)-2-methyl-2-phenethyl-1,3-oxazolidine (4k)** White crystals; yield 77%; mp 114–116°C; IR: ν 3090–3000 (C-H), 1688 (C=O), 1411 (Cl_2HC-CO-), 1232 cm^{-1} (N-C-O); ^1H NMR (DMSO-d_6): δ 7.75 (m, 1H, Ar-H), 7.17 (m, 5H, Ar-H), 7.02 (s, 1H, Cl_2CH), 6.66 (d, J = 3.3 Hz, 1H, Ar-H), 6.51 (m, 1H, Ar-H), 4.23 (m, 2H, N-CH_2), 3.79 (t, J = 9.8 Hz, 1H, CH-O), 2.07–2.69 (m, 4H, 2 × CH_2), 1.57 (s, 3H, CH_3); ^{13}C NMR (DMSO-d_6): δ 160.0, 149.6, 144.6, 141.9, 128.9, 128.9, 128.6, 128.6, 126.3, 111.2, 110.8, 97.4, 69.5, 67.8, 49.8, 38.8, 29.2, 23.1. Anal. Calcd for $C_{18}H_{19}Cl_2NO_3$: C, 58.84; H, 5.22; N, 3.81. Found: C, 58.72; H, 5.28; N, 3.74.

***N*-Dichloroacetyl-2-(2′-furyl)-1-oxa-4-aza-spiro-4,5-noncane (4l)** White crystals; yield 88%; mp 105–108°C; IR: ν 2927–2864 (C-H), 1670 (C=O), 1421 (Cl_2HC-CO-), 1137 cm^{-1} (N-C-O); ^1H NMR: δ 7.47 (m, 1H, Ar-H), 6.48 (m, 1H, Ar-H), 6.40 (m, 1H, Ar-H), 6.05 (s, 1H, Cl_2CH), 4.14 (m, 1H, N-CH_2), 3.93–3.99 (t, J = 9.5 Hz, 1H, O-CH), 2.25–2.62 (m, 10H, $(CH_2)_5$); ^{13}C NMR: δ 159.8, 149.4, 143.5, 110.6, 109.8, 98.6, 69.7, 67.2, 49.0, 33.2, 30.5, 24.5, 23.0, 23.0. Anal. Calcd for $C_{14}H_{17}Cl_2NO_3$: C, 52.99; H, 5.40; N, 4.42. Found: C, 52.87; H, 5.42; N, 4.50.

X-Ray data collection and structure refinement

X-Ray data were collected on a Bruker AXS II CCD area-detector diffractometer using graphite monochromated Mo Kα radiation (λ = 0.071073 nm) at 298(2) K. The structure was solved by direct methods using SHELXS-97, and refined by full matrix least squares on F^2 using full-matrix least-squares procedures [19]. Minimum and maximum, final electron density were -0.616 and 0.401 eÅ^{-3}. Symmetry equivalent reflections were used to optimize crystal shape and size. Crystallographic data have been deposited at the Cambridge Crystallographic Data Centre as supplementary publication number CCDC 808806. Copies of the data can be obtained, free of charge, on application to CCDC, 12 Union Road, Cambridge CB2 1EZ, UK (fax: + 44-1223-336033, e-mail: deposit@ccdc.cam. ac.uk).

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