

Synthetic Communications: An International Journal for Rapid Communication of Synthetic Organic Chemistry

Publication details, including instructions for authors and subscription information:

<http://www.tandfonline.com/loi/lsyc20>

Facile Synthesis of 3-Alkyl-5-methyloxazolidine-2,4-diones and N-Lactoyl-N,N'-dialkylureas

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Published online: 03 Mar 2011.

To cite this article: Francesco Saliu, Guido Galliani, Marco Orlandi, Bruno Rindone, Eeva-Liisa Tolppa & Ricardo Suarez-Bertoa (2011) Facile Synthesis of 3-Alkyl-5-methyloxazolidine-2,4-diones and N-Lactoyl-N,N'-dialkylureas, Synthetic Communications: An International Journal for Rapid Communication of Synthetic Organic Chemistry, 41:7, 956-962, DOI: [10.1080/00397911003707154](https://doi.org/10.1080/00397911003707154)

To link to this article: <http://dx.doi.org/10.1080/00397911003707154>

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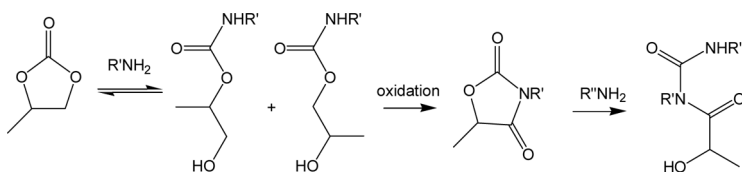
FACILE SYNTHESIS OF 3-ALKYL-5-METHYLOXAZOLIDINE-2,4-DIONES AND *N*-LACTOYL-*N,N'*-DIALKYLUREAS

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GRAPHICAL ABSTRACT



Abstract The reaction between aliphatic amines and propylene carbonate can be performed in solventless conditions under microwave irradiation, becoming nearly complete within 15 min of irradiation. Oxidation of the formed mixture of 2-hydroxyethylcarbamates gives 3,5-methylalkyl-oxazolidine-2,4-diones. These compounds can react further with aliphatic primary amines to give *N*-lactoylureas.

Keywords 3,5-Dialkyl-oxazolidine-2,4-diones; *N*-lactoyl-*N,N'*-dialkylureas; solventless reaction

INTRODUCTION

N-Substituted oxazolidine-2,4-diones are important therapeutic agents and scaffolds for the preparation of biologically active compounds.^[1,2] For example, 3-methyloxazolidine-2,4-diones have analgesic and anticonvulsant properties, with little or no hypnotic action.^[3] Oxazolidine-2,4-diones can be prepared from α -halogeno acids or α -halogenoureaides, by hydrolysis of 2-imino-4-oxazolidinones or dialuric acids, by oxidation of 2-thiooxo-4-oxazolidones, by condensation of esters of α -hydroxy acids with urea, by condensation of amides of α -hydroxy acids with alkyl carbonates or chloroformates, or by cyclization of urethans of α -hydroxy acids.^[4] However, the most efficient method for building 3-substituted oxazolidine-2,4-diones

Received December 9, 2009.

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involves the cyclization of *N*-substituted chloroacylcarbamates.^[5] As a “greener” alternative, in our previous work we reported the possibility of proceeding via the intramolecular cyclization of a carbamate salt, obtained by a 1,8-diazabicyclo[5.4.0]undec-7-ene (DBU)-mediated carbon dioxide fixation.^[6]

Here, we describe a very efficient and easy procedure involving a microwave-assisted solventless reaction between cyclic carbonates and primary amines, followed by an oxidation, to give the corresponding 3,5-substituted oxazolidine-2,4-diones in good yields. In addition, by reacting 3,5-substituted oxazolidine-2,4-diones under the same conditions and with an equimolecular amount of primary amines, *N*-lactoyl-*N,N'*-dialkylureas are formed in very good yields. These compounds represent useful intermediates to prepare new molecules, particularly in pharmaceutical development, with a special interest for peptidomimetic drugs.^[7]

RESULTS AND DISCUSSION

We reacted 4-methyl-1,3-dioxolan-2-one (**1**) with an equimolecular amount of primary amines in the absence of solvent and without any catalyst. The reaction temperature was fixed at 85 °C, to minimize overreaction of the products with unreacted amines. The mixture of 2-hydroxyethylcarbamates **2** + **3** was obtained with a ratio of primary to secondary alcohols ranging from 1.16 to 1.52 (Scheme 1).

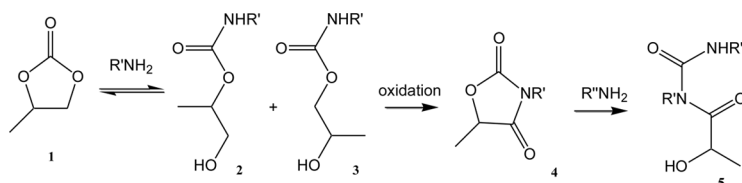
The transformation can be sped up by reacting the solventless mixture under microwave irradiation. The reaction was nearly complete within 15 min of irradiation and the ratio of primary to secondary alcohols ranged from 1.07 to 1.28 (Table 1).

The structures of 2-hydroxyethylcarbamates **2** + **3** were determined by ¹H, ¹³C, and correlation spectroscopy (COSY) NMR, and the results are in good agreement with literature data.^[8]

The ratio of primary alcohol (**2**) to secondary alcohol (**3**) was evaluated by ¹H NMR analysis of the crude reaction mixture by integration of the doublet peaks at 1.06 ppm and 1.13 ppm, corresponding at methyl protons in structures **2** and **3** respectively, and by integration of the nitrogen proton of carbamate groups.

The oxidation of the mixture of 2-hydroxyethylcarbamates with the CrO₃–pyridine complex^[9] gave the corresponding 3,5-dialkyl-oxazolidine-2,4-diones (**4**) in 83–85% yield (Table 2).

Compound **4** is probably formed by oxidation of the primary alcohol (**2**) to the corresponding aldehyde, which is further oxidized and cyclized to **4**, and an equilibrium between the primary and secondary alcohol must be involved, because the yields in compound **4** are larger than the molar fraction of the primary alcohol (**2**)



Scheme 1. Reaction of propylene carbonate with primary amines.

Table 1. Reaction of 4-methyl-1,3-dioxolan-2-one with amines

Amine	Time (h)	Conversion (%)	Primary alcohol (2) (%)	Secondary alcohol (3) (%)	Ratio primary/secondary
Thermal					
n-Pentylamine	6	98	59 (2a)	39 (3a)	1.51
Cyclohexylamine	12	95	57 (2b)	38 (3b)	1.50
Benzylamine	7	96	51 (2c)	44 (3c)	1.16
n-Hexylamine	6	96	58 (2d)	38 (3d)	1.52
Microwave assisted ^a					
n-Pentylamine	0.25	91	(2a) 47	(3a) 44	1.07
Cyclohexylamine	0.25	98	(2b) 54	(3b) 42	1.28
n-Hexylamine	0.25	91	(2d) 49	(3d) 42	1.17

^aIn a microwave oven at 200 W, 2450 MHz for 15 min.

Table 2. Chromic oxidation of alcohols **2** + **3** to form 3-alkyl-5-methyloxazolidindiones (**4**)

Amine in the mixture 2 + 3	Isolated yield in 3-alkyl-5-methyl-oxazolidine-2,4-dione (4) (%)
n-Pentylamine	(4a) 83
Cyclohexylamine	(4b) 85
Benzylamine	(4c) 83

present in the crude starting mixture. The oxidation should proceed faster with the primary alcohol (**2**) than with the secondary alcohol (**3**), thus giving the corresponding aldehyde and subsequently **4** as the main product, and not a ketone, which has been recognized in only trace amounts. Investigations are in progress to verify this mechanism, and results will be presented in a future paper.

The ring of 3,5-dialkyloxazolidine-2,4-diones (**4**) can be further opened with equimolecular amounts of a primary amine in solventless conditions and under microwave heating to give *N*-lactoyl-*N,N'*-dialkylureas (**5**) (Scheme 1) in satisfactory yields, as shown in Table 3.

Table 3. Reaction of 3-alkyl-5-methyloxazolidin-2,4-diones (**4**) with amines to give *N*-lactoyl-*N,N'*-dialkylureas (**5**) in solventless conditions

3-Alkyl-5-methyloxazolidine-2,4-diones (4)	Reacting amine	Isolated yield in <i>N</i> -lactoyl- <i>N,N'</i> -dialkylurea (5) (%)
(4a) R' = <i>n</i> -pentyl	n-Pentylamine	R' = <i>n</i> -pentyl; R'' = <i>n</i> -pentyl (5a): 85
(4a) R' = <i>n</i> -pentyl	Benzylamine	R' = <i>n</i> -pentyl; R'' = benzyl (5b): 83
(4a) R' = <i>n</i> -pentyl	Cyclohexylamine	R' = <i>n</i> -pentyl; R'' = cyclohexyl (5c): 59
(4b) R' = cyclohexyl	n-Pentylamine	R' = cyclohexyl; R'' = <i>n</i> -pentyl (5d): 73
(4b) R' = cyclohexyl	Benzylamine	R' = cyclohexyl; R'' = benzyl (5e): 59
(4b) R' = cyclohexyl	Cyclohexylamine	R' = cyclohexyl; R'' = cyclohexyl (5f): 55
(4c) R' = benzyl	n-Pentylamine	R' = benzyl; R'' = <i>n</i> -pentyl (5g): 78
(4c) R' = benzyl	Benzylamine	R' = benzyl; R'' = benzyl (5h): 62

CONCLUSIONS

In conclusion, the reaction of 4-methyl-1,3-dioxolan-2-one and 3-alkyl-5-methyl-oxazolidine-2,4-diones with amines, carried out in solventless conditions under microwave irradiation, gave respectively 2-hydroxyethylcarbamates and lactoylureas in satisfactory yields.

The oxidation of 2-hydroxyethylcarbamates affords 3-alkyloxazolidine-2,4-diones in good yields.

EXPERIMENTAL

All chemicals were purchased from Aldrich or Fluka chemical companies. ^1H and ^{13}C NMR were recorded on a Varian Mercury 400 instrument in CDCl_3 or dimethylsulfoxide ($\text{DMSO}-d_6$). Chemical shifts were reported in parts per million (δ), relative to the internal standard of tetramethylsilane (TMS). Gas chromatography–mass spectrometry (GC–MS) analyses were performed with a Hewlett Packard 5890 instrument equipped with a 5971A mass selective detector. Melting points were determined in open glass capillaries using a Electrothermal melting-point apparatus and are uncorrected. Infrared (IR) spectra in solutions were recorded on a Nicolet Avatar 360 Fourier transform (FT)–IR spectrometer, using calcium fluoride cells previously purged with N_2 . Elemental analyses were carried out on Perkin-Elmer series II 2400 instrument. All reactions were monitored by thin-layer chromatography (TLC). The reactions under microwave irradiation were carried out with a CEM Discover Labmate instrument. Flash chromatography was performed on silica gel (100–200 mesh). All products were purified through chromatographic column before characterization.

General Procedure for the Preparation of 2-Hydroxyethylcarbamates **2 + 3**

Cyclic carbonates (**1**) (0.1 mol) were mixed with 0.1 mol of amine, and the mixture was stirred at 85°C for 10 h. The oily mixtures of hydroxyethylcarbamates obtained were directly submitted to analysis. Alternatively, the reaction was performed in the same conditions under microwave irradiation at 200 W, 2450 MHz for 15 min, $T < 85^\circ\text{C}$.

General Procedure for the Synthesis of 3-Alkyl-5-methyl-oxazolidine-2,4-diones (**4a–c**)

Pyridine (4.8 mL) was dissolved in dichloromethane (100 mL). The solution was cooled at 5°C , and then CrO_3 (2.88 g) was added. The mixture was stirred 15 min at 5°C and allowed to reach room temperature. After 30 min, the mixture of adducts **2** and **3** (4.8 mmol), dissolved in dichloromethane (10 mL), was added. The mixture was stirred for 15 min at room temperature; black tars were separated and extracted with additional dichloromethane (5×40 mL), stirring for 10 min each time.

The pooled organic solutions were evaporated under reduced pressure, and the oily residue was dissolved in toluene (50 mL) and again evaporated under reduced

pressure. The oily residue was filtered on silica gel (5 g) to remove metal traces, eluting with dichloromethane (150 mL). The eluate was then evaporated under reduced pressure.

5-Methyl-3-pentyl-oxazolidine-2,4-dione (4a). IR (neat): 1810, 1730. ^1H NMR (400 MHz, DMSO-d_6) δ : 0.95 (t, 3H), 1.20–1.40 (m, 4H), 1.40 (m, 2H), 1.50 (d, 3H), 3.00 (m, 2H), 4.80 (q, 1H, $J=8$ Hz). ^{13}C NMR (100 MHz, DMSO-d_6) δ : 13.8, 15.8, 21.6, 28.3, 28.9, 40.1, 75.5, 155.1, 202.9. Mass (m/z): 185 (M^+). Anal. calcd. for $\text{C}_9\text{H}_{15}\text{NO}_3$: C, 58.36; H, 8.16; N, 7.56; O, 25.91. Found: C, 58.54; H, 8.31; N, 7.82; O, 26.02.

3-Cyclohexyl-5-methyl-oxazolidine-2,4-dione (4b). IR (neat): 1810, 1730. ^1H NMR (400 MHz, DMSO-d_6) δ : 1.25–1.60 (m, 11H aliphatic protons), 1.50 (d, 3H, CH_3), 3.15 (m, 2H), 4.80 (q, 1H, $J=8$ Hz). ^{13}C NMR (100 MHz, DMSO-d_6) δ : 15.8, 24.5, 25.0, 32.5, 49.3, 74.6, 154.2, 202.9. Mass (m/z): 197 (M^+). Anal. calcd. for $\text{C}_{10}\text{H}_{15}\text{NO}_3$: C, 60.90; H, 7.67; N, 7.10; O, 24.34. Found: C 60.80; H, 7.90; N, 7.20; O, 24.12.

3-Benxyl-5-methyl-oxazolidine-2,4-dione (4c). Mp 74–75. IR (neat): 1810, 1730. ^1H NMR (400 MHz, DMSO-d_6) δ : 1.50 (d, 3H), 4.58 (s, 2H), 4.80 (q, 1H, $J=8$ Hz), 7.19–7.40 (m, 5H aromatic protons); ^{13}C NMR (100 MHz, DMSO-d_6) δ : 15.8, 43.6, 74.6, 126.4, 126.6, 126.8, 127.3, 127.8, 128.2, 155.4, 202.8. Mass (m/z): 205 (M^+). Anal. calcd. for $\text{C}_{11}\text{H}_{11}\text{NO}_3$: C, 64.38; H, 5.40; N, 6.83; O, 23.39. Found: C, 64.20; H, 5.50; N, 6.72; O, 23.12.

General Procedure for the Synthesis of *N*-Lactoyl-*N,N'*-dialkylureas (5a–h)

3-Alkyl-5-methyloxazolidine-2,4-dione (**4**) (20 mmol) were mixed with amine (20 mmol) under nitrogen, and the resulting mixture was stirred at 100 °C for 5 h, cooled, and dissolved in dichloromethane (50 mL). The resulting solution was filtered on silica gel (200 g), eluting with dichloromethane–ethyl acetate (3:1). Microwave irradiation requires shorter times (20 min).

Compound 5a. Mp 62–63. IR (neat): 3312, 1721, 1684, 1650. ^1H NMR (400 MHz, DMSO-d_6) δ : 0.83 (m, 6H), 1.24–1.37 (m, 12H), 1.40 (d, 3H), 2.92 (m, 2H), 3.03 (m, 2H), 4.83 (qd, 1H), 7.78 (NH); ^{13}C NMR (100 MHz, DMSO-d_6) δ : 14.2, 18.3, 22.1, 29.0, 38.7, 40.4, 69.3, 154.5, 170.2. Mass (m/z): 272, 186, 158, 144, 130, 114, 98, 85. Anal. calcd. for $\text{C}_{14}\text{H}_{28}\text{N}_2\text{O}_3$: C, 61.73; H, 10.36; N, 10.28; O, 17.62. Found: C, 61.60; H, 10.43; N, 10.35; O, 17.51.

Compound 5b. Mp 76–77. IR (neat): 3310, 1720, 1682, 1651. ^1H NMR (400 MHz, DMSO-d_6) δ : 0.83 (m, 3H), 1.25–1.37 (m, 4H), 1.39 (d, 3H), 1.41 (dd, 2H), 2.92 (m, 2H), 4.30 (d, 2H), 4.85 (qd, 1H), 7.19–7.38 (m, 5H), 8.42 (NH); ^{13}C NMR (100 MHz DMSO-d_6) δ : 14.2, 18.3, 22.1, 29.0, 38.7, 42.4, 69.3, 126.5, 128.2, 139.3, 155.1, 170.2. Mass (m/z): 292 (M^+) 206, 179, 161, 133, 106, 91. Anal. calcd. for $\text{C}_{16}\text{H}_{24}\text{N}_2\text{O}_3$: C, 65.73; H, 8.27; N, 9.58; O, 16.42. Found: C, 65.89; H, 8.11; N, 9.43; O, 16.55.

Compound 5c. IR (neat): 3311, 1720, 1682, 1652. ^1H NMR (400 MHz, DMSO-d_6) δ : 0.83 (td, 3H), 1.04–1.68 (m, 16H), 1.40 (d, 3H), 2.92 (m, 2H), 3.60 (m, 1H), 4.83 (qd, 1H), 7.80 (NH); ^{13}C NMR (100 MHz, DMSO-d_6) δ : 14.2, 18.3, 22.2, 24.1, 25.9, 29.0, 31.0, 40.4, 49.0, 69.3, 155.0 (C=O), 168.2. Mass (m/z): 284 (M^+ , 1%), 203 (16%), 186 (42%), 171 (6%), 159 (54%), 130 (100%), 114 (39%), 102 (18%). Anal. calcd. for $\text{C}_{15}\text{H}_{28}\text{N}_2\text{O}_3$: C, 63.35; H, 9.92; N, 9.85; O, 16.88. Found: C, 63.37; H, 9.76; N, 9.95; O, 16.64.

Compound 5d. IR (neat): 3310, 1722, 1684, 1650. ^1H NMR (400 MHz, DMSO-d_6) δ : 0.83 (td, 3H), 1.04–1.65 (m, 16H), 1.39 (d, 3H), 2.93 (m, 2H), 3.29 (m, 1H), 4.83 (qd, 1H), 7.80 (NH); ^{13}C NMR (100 MHz, DMSO-d_6) δ : 14.2, 18.3, 22.8, 23.9, 25.5, 31.1, 40.4, 47.7, 69.3, 156.0, 170.1. Mass (m/z): 284 (M^+ , 1%), 198 (15%), 171 (34%), 159 (22%), 142 (100%), 126 (23%), 114 (36%), 98 (12%), 83 (42%). Anal. calcd. for $\text{C}_{15}\text{H}_{28}\text{N}_2\text{O}_3$: C, 63.35; H, 9.92; N, 9.85; O, 16.88. Found: C, 63.43; H, 9.81; N, 9.92; O, 16.78.

Compound 5e. Mp 67–68. IR (neat): 3312, 1721, 1682, 1650. ^1H NMR (400 MHz, DMSO-d_6) δ : 1.39 (d, 3H), 1.04–1.70 (m, 10H), 3.29 (m, 1H), 4.30 (d, 2H), 4.85 (qd, 1H), 7.18–7.37 (m, 5H), 8.41 (t, 1H); ^{13}C NMR (100 MHz, DMSO-d_6) δ : 18.3, 24.0, 25.5, 31.0, 42.3, 47.7, 69.3, 126.5, 128.2, 139.3, 155.0, 170.0. Mass (m/z): 305 (M^+ , 1%), 179 (15%), 161 (100%), 142 (34%), 133 (42%), 106 (44%), 91 (85%). Anal. calcd. for $\text{C}_{16}\text{H}_{22}\text{N}_2\text{O}_3$: C, 66.18; H, 7.64; N, 9.65; O, 16.53. Found: C, 66.23; H, 7.55; N, 9.81; O, 16.39.

Compound 5f. Mp 64–65. IR (neat): 3310, 1720, 1683, 1651; ^1H NMR (400 MHz, DMSO-d_6) δ : 1.39 (d, 3H), 1.13–1.80 (m, 20H), 3.29 (m, 2H), 4.83 (qd, 1H), 8.4 (t, 1H); ^{13}C NMR (100 MHz, DMSO-d_6) δ : 18.3, 22.8, 23.9, 25.5, 31.1, 47.7, 49.0, 69.3, 156.0, 170.1. Mass (m/z): 296 (M^+ , 1%), 215 (4%), 207 (5%), 198 (11%), 171 (38%), 153 (4%), 142 (50%), 126 (22%), 97 (57%), 83 (100%). Anal. calcd. for $\text{C}_{16}\text{H}_{28}\text{N}_2\text{O}_3$: C, 64.83; H, 9.52; N, 9.45; O, 16.19. Found: C, 64.69; H, 9.56; N, 9.41; O, 16.26.

Compound 5g. Mp 68–69. IR (neat): 3311, 1720, 1683, 1650. ^1H NMR (400 MHz, DMSO-d_6) δ : 0.83 (t, 3H), 1.25–1.37 (m, 6H), 1.40 (d, 3H), 2.93 (m, 2H), 4.27 (s, 2H), 4.85 (qd, 1H), 7.20–7.39 (m, 5H), 7.80 (NH); ^{13}C NMR (100 MHz, DMSO-d_6) δ : 14.2, 18.3, 22.2, 29.0, 40.1, 40.4, 69.3, 126.5, 128.2, 139.3, 155.9, 170.1. Mass (m/z): 292 (M^+ , 1%), 179 (10%), 161 (38%), 150 (9%), 133 (43%), 118 (4%), 106 (26%), 91 (100%). Anal. calcd. for $\text{C}_{16}\text{H}_{24}\text{N}_2\text{O}_3$: C, 65.73; H, 8.27; N, 9.58; O, 16.42. Found: C, 65.78; H, 8.31; N, 9.54; O, 16.40.

Compound 5h. Mp 93–94. IR (neat): 3310, 1721, 1682, 1650. ^1H NMR (400 MHz, DMSO-d_6) δ : 1.40 (d, 3H), 4.27 (s, 2H), 4.30 (d, 2H), 4.86 (qd, 1H), 7.19–7.40 (m, 10H), 8.42 (NH); ^{13}C NMR (100 MHz, DMSO-d_6) δ : 18.3, 40.1, 42.3, 69.4, 126.6, 128.2, 139.3, 155.1, 170.2. Mass (m/z): 206 (M^+ , 7%), 179 (14%), 159 (7%), 150 (47%), 133 (44%), 114 (22%), 105 (26%), 91 (100%). Anal. calcd. for $\text{C}_{16}\text{H}_{18}\text{N}_2\text{O}_3$: C, 67.12; H, 6.34; N, 9.78; O, 16.76. Found: C, 67.24; H, 6.19; N, 9.84; O, 16.59.

ACKNOWLEDGMENTS

We thank CEM Srl. (Innovators in Microwave Technology) for providing us with the instrumentation for microwave irradiation and Mr. Umberto Vallone, Mr. Andrea Rota, and our student Luca Gabrielli for help in performing the experiments.

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