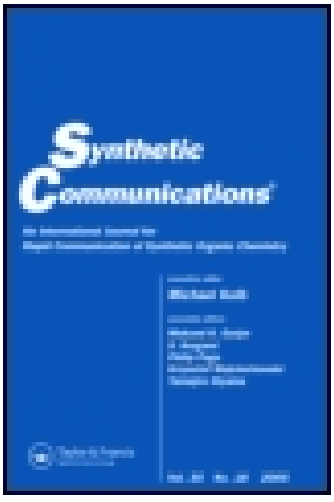

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Formation of N-Sulfonylcarbamates from Epichlorohydrin: Synthesis of Polyfunctional Oxazolidin-2-ones

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Formation of *N*-Sulfonylcarbamates from Epichlorohydrin: Synthesis of Polyfunctional Oxazolidin-2-ones

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Abstract: The addition of 3-methylbut-2-enoic acid-3-bromo-2-hydroxypropyl ester **2** and 1-bromo-3-phenylsulfanyl propan-2-ol **3**, obtained from epichlorohydrin, to aroxy and alkoxysulfonyl isocyanates **1** in anhydrous ether at ambient temperature affords the corresponding *N*-sulfonyl bromocarbamates **4**. These are treated with triethylamine in refluxing acetone to give exclusively the corresponding polyfunctional oxazolidin-2-ones **5** with good yields.

Keywords: aroxy and alkoxysulfonyl isocyanates, *N*-sulfonyl bromocarbamates, oxazolidin-2-one

INTRODUCTION

The addition of hydroxyl compounds, particularly β-bromoalcohols in alkali media, to isocyanates generally led to oxazolidinones. These compounds

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are widely studied because of their biological and pharmacological properties.[1–4]

These heterocyclic compounds may be obtained by the action of threonine on trichloromethyl carbonate,[5] whereas other oxazolidinones were obtained by the condensation reaction of γ-hydroxyesters or γ-hydroxysulfones on tosylisocyanate.[6]

In a previous work related to the synthesis of new *N*-sulfonylcarbamates that may have important biological properties, we have studied the reactivity of aroxy and alkoxysulfonyl isocyanates toward fluorinated and highly fluorinated alcohols,[7,8] *F*-alkylthiols,[8] and polyfluorinated aminoalcohols.[9] Here we extend their reactivity to 3-methylbut-2-enoic acid-3-bromo-2-hydroxypropyl ester and 1-bromo-3-phenylsulfanylpropan-2-ol to synthesize new bromocarbamates. These carbamates undergo ring formation, by action of triethylamine in refluxing acetone, to afford *N*-aroxy and alkoxysulfonyl oxazolididin-2-ones.

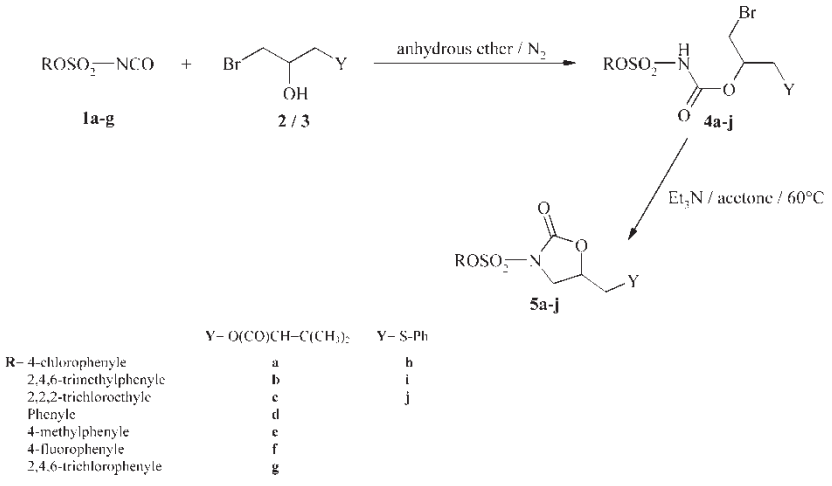
RESULTS AND DISCUSSION

In this work we describe an efficient method to synthesize new polyfunctional oxazolidin-2-ones starting from bromo derivatives **2** and **3**.[10] The latter have been obtained from epichlorohydrin. This allows an easy substitution at the C5 position of the oxazolidinone ring to easily obtain different substituted oxazolidinones.

The 3-methylbut-2-enoic acid-3-bromo-2-hydroxypropyl ester **2** reacts easily at ambient temperature, in anhydrous ether and under a nitrogen atmosphere with *N*-aroxy and alkoxysulfonyl isocyanates **1a–g** to give the corresponding *N*-sulfonyl bromocarbamates **4a–g** (see Table 1). These carbamates, described for the first time, have an additional reactivity because of the presence of the bromine atom. For instance, the action of

Table 1. Yields and times of *N*-sulfonylbromocarbamates **4a–j**

Carbamates	Yield (%)	Time (min)
4a	92	2
4b	86	4
4c	84	2
4d	93	5
4e	92	3
4f	95	1
4g	90	1
4h	94	2
4i	92	5
4j	95	2



Scheme 1.

triethylamine in refluxing acetone led to the intramolecular cyclization reaction yielding oxazolidin-2-ones **5a**–**g** as outlined in Scheme 1.

In a similar manner, the 1-bromo-3-phenylsulfanyl propan-2-ol **3** reacts in anhydrous ether, at room temperature, with *N*-sulfonylisocyanates **1a**–**c** to afford the corresponding bromo carbamates **4h**–**j**. Next, the oxazolidin-2-one ring was formed by the action of the triethylamine to obtain **5h**–**j** in moderate yields (Scheme 1). The different data for the synthesis and characterization of the carbamates **4a**–**j** and oxazolidin-2-ones **5a**–**j** are summarized respectively in Tables 1 and 2.

It is worth noting that the chlorohomologues of **2** and **3** may be obtained in one step, on treatment of epichlorohydrin with acrylic acid and thiophenol

Table 2. Yields and times of polyfunctional oxazolidin-2-ones **5a**–**j**

Oxazolidinones	Yield (%)	Time (h)
5a	80	1.5
5b	88	2
5c	72	2
5d	79	2
5e	76	2
5f	84	1.5
5g	86	1
5h	78	2
5i	87	2
5j	73	2

in acidic media respectively.[11] The addition reaction of these compounds with *N*-sulfonylisocyanates is easily performed to yield the corresponding chlorocarbamates, but given the less nucleofuge character of the chlorine atom, the cyclization reaction leads to much lower yields (less than 15%).

EXPERIMENTAL

NMR spectra were recorded on an Ac 300 Bruker instrument at 300 MHz for ^{1}H in TMS as internal reference. IR spectra ($CHCl_3$) were recorded on a Perkin-Elmer Paragon 1000 PC. Mass spectra were obtained by negative and positive modes of electrospray ionization (ESI NEG, ESI POS). Aroxy and alkoxysulfonyl isocyanates **1a–g** were prepared by the action of corresponding phenols and alcohols on chlorosulfonyl isocyanate.[12] Bromoderivates **2** and **3** were prepared following a method described in the literature.[10]

Synthesis of *N*-Sulfonyl Bromocarbamates 4a–j

To 10 mmol of aroxy(alkoxy)sulfonyl isocyanates 1a–g dissolved in 10 mL of ether, 10 mmol of the bromoderivate **2** or **3** dissolved in 10 mL of the same solvent were added. The mixture was then stirred for 1 to 5 min (depending on the nature of isocyanate used) at ambient temperature and under a nitrogen atmosphere. The solvent was evaporated in vacuum, and the products were isolated by column chromatography as viscous oils (eluant: ethylic ether 30%, petroleum ether 70%).

Data

4a: IR (ν, cm^{-1}): 3360 (NH), 1767 (C═O), 1719 (C═O), 1649 (C═C), 1393–1175 (SO_2); ^{1}H NMR (δ, ppm): 9.00 (1H, NH), 7.20 (4H, arom, $^3J = 8.8$ Hz), 5.70 (1H, CH═C), 5.25 (1H, CH-O), 4.30 (2H, CH_2-O, $^2J_{ab} = 12.4$ Hz, $^3J_{ax} = 6.2$ Hz, $^3J_{bx} = 4.1$ Hz), 3.55 (2H, CH_2-Br, $^2J_{ab} = 12.1$ Hz, $^3J_{ax} = 5.9$ Hz, $^3J_{bx} = 4.8$ Hz), 2.15 (3H, CH_3), 1.90 (3H, CH_3); ^{13}C NMR (δ, ppm): 20.4, 27.6, 34.4, 62.1, 73.9, 114.7, 123.3, 129.9, 133.6, 148.1, 149.5, 159.9, 166.1; HRMS $[M\text{-}H]^-$: calcd. 467.9519, found 467.9526.

4b: IR (ν, cm^{-1}): 3359 (NH), 1766 (C═O), 1718 (C═O), 1648 (C═C), 1390–1172 (SO_2); ^{1}H NMR (δ, ppm): 9.10 (1H, NH), 6.80 (2H, arom), 5.65 (1H, CH═C), 5.20 (1H, CH-O), 4.30 (2H, CH_2-O, $^2J_{ab} = 12.4$ Hz, $^3J_{ax} = 6.3$ Hz, $^3J_{bx} = 4.1$ Hz), 3.50 (2H, CH_2-Br, $^2J_{ab} = 12.3$ Hz, $^3J_{ax} = 5.8$ Hz, $^3J_{bx} = 4.6$ Hz), 2.35 [6H, 2(CH_3)], 2.25 (3H, CH_3), 2.15 (3H,

CH_3), 1.90 (3H, CH_3); ^{13}C NMR (δ, ppm): 17.1, 20.4, 20.6, 27.5, 34.4, 62.2, 73.8, 114.9, 129.9, 131.3, 136.3, 145.9, 149.5, 159.1, 166.1; HRMS $[M-H]^-$: calcd. 476.0378, found 476.0383.

4c: IR (ν, cm^{-1}): 3352 (NH), 1763 (C═O), 1716 (C═O), 1648 (C═C), 1399–1177 (SO_2); ^1H NMR (δ, ppm): 9.15 (1H, NH), 5.70 (1H, CH═C), 5.20 (1H, CH-O), 4.90 (2H, CCl_3-CH_2-O), 4.35 (2H, CH_2-O, $^2J_{ab} = 12.4$ Hz, $^3J_{ax} = 6.2$ Hz, $^3J_{bx} = 4.3$ Hz), 3.55 (2H, CH_2-Br, $^2J_{ab} = 12.1$ Hz, $^3J_{ax} = 5.9$ Hz, $^3J_{bx} = 4.8$ Hz), 2.20 (3H, CH_3), 1.95 (3H, CH_3); ^{13}C NMR (δ, ppm): 20.4, 27.5, 34.5, 62.1, 73.8, 80.8, 92.9, 114.9, 149.8, 159.2, 165.9; HRMS $[M-H]^-$: calcd. 487.8739, found 487.8753.

4d: IR (ν, cm^{-1}): 3362 (NH), 1768 (C═O), 1719 (C═O), 1648 (C═C), 1396–1172 (SO_2); ^1H NMR (δ, ppm): 9.30 (1H, NH), 7.35 (5H, arom), 5.65 (1H, CH═C), 5.20 (1H, CH-O), 4.25 (2H, CH_2-O, $^2J_{ab} = 12.3$ Hz, $^3J_{ax} = 6.3$ Hz, $^3J_{bx} = 4.2$ Hz), 3.50 (2H, CH_2-Br, $^2J_{ab} = 12.1$ Hz, $^3J_{ax} = 5.9$ Hz, $^3J_{bx} = 4.7$ Hz), 2.10 (3H, CH_3), 1.90 (3H, CH_3); ^{13}C NMR (δ, ppm): 20.5, 27.6, 34.3, 62.2, 73.7, 114.8, 121.6, 127.2, 130.1, 149.3, 149.8, 159.5, 166.1; HRMS $[M-H]^-$: calcd. 433.9909, found 433.9917.

4e: IR (ν, cm^{-1}): 3360 (NH), 1767 (C═O), 1719 (C═O), 1649 (C═C), 1393–1175 (SO_2); ^1H NMR (δ, ppm): 9.00 (1H, NH), 7.20 (4H, arom, $^3J = 8.8$ Hz), 5.70 (1H, CH═C), 5.25 (1H, CH-O), 4.30 (2H, CH_2-O, $^2J_{ab} = 12.1$ Hz, $^3J_{ax} = 6.4$ Hz, $^3J_{bx} = 4.3$ Hz), 3.55 (2H, CH_2-Br, $^2J_{ab} = 12.2$ Hz, $^3J_{ax} = 5.8$ Hz, $^3J_{bx} = 4.8$ Hz), 2.30 (3H, CH_3), 2.15 (3H, CH_3), 1.90 (3H, CH_3); ^{13}C NMR (δ, ppm): 20.5, 20.9, 27.5, 34.4, 62.1, 73.7, 114.9, 121.5, 130.5, 138.3, 147.6, 149.2, 159.5, 166.0; HRMS $[M-H]^-$: calcd. 448.0065, found 448.0082.

4f: IR (ν, cm^{-1}): 3358 (NH), 1769 (C═O), 1719 (C═O), 1649 (C═C), 1396–1174 (SO_2); ^1H NMR (δ, ppm): 9.10 (1H, NH), 7.30 (4H, arom), 5.70 (1H, CH═C), 5.30 (1H, CH-O), 4.30 (2H, CH_2-O, $^2J_{ab} = 12.3$ Hz, $^3J_{ax} = 6.4$ Hz, $^3J_{bx} = 4.2$ Hz), 3.55 (2H, CH_2-Br, $^2J_{ab} = 12.1$ Hz, $^3J_{ax} = 5.9$ Hz, $^3J_{bx} = 4.7$ Hz), 2.15 (3H, CH_3), 1.90 (3H, CH_3); ^{13}C NMR (δ, ppm): 20.4, 27.6, 34.4, 62.2, 73.8, 114.8, 116.0, 124.0, 146.5, 149.6, 159.9, 160.8, 166.1; HRMS $[M-H]^-$: calcd. 451.9814, found 451.9814.

4g: IR (ν, cm^{-1}): 3353 (NH), 1768 (C═O), 1719 (C═O), 1649 (C═C), 1408–1177 (SO_2); ^1H NMR (δ, ppm): 9.20 (1H, NH), 7.20 (2H, arom), 5.75 (1H, CH═C), 5.25 (1H, CH-O), 4.25 (2H, CH_2-O, $^2J_{ab} = 12.4$ Hz, $^3J_{ax} = 6.4$ Hz, $^3J_{bx} = 4.1$ Hz), 3.55 (2H, CH_2-Br, $^2J_{ab} = 12.1$ Hz, $^3J_{ax} = 5.7$ Hz, $^3J_{bx} = 4.7$ Hz), 2.15 (3H, CH_3), 1.90 (3H, CH_3); ^{13}C NMR (δ, ppm): 20.4, 27.5, 34.6, 62.3, 73.8, 114.9, 129.1, 130.5, 133.5, 142.9, 149.3, 159.1, 166.1; HRMS $[M-H]^-$: calcd. 535.8739, found 535.8752.

4h: IR (ν, cm^{-1}): 3362 (NH), 1765 (C═O), 1397–1174 (SO_2); ^1H NMR (δ, ppm): 8.65 (1H, NH), 7.30 (5H, arom), 7.25 (4H, arom, $^3J = 8.8$ Hz), 4.15 (1H, CH-O), 3.60 (2H, CH_2-Br, $^2J_{ab} = 12.8$ Hz, $^3J_{ax} = 8.5$ Hz, $^3J_{bx} = 5.1$ Hz), 3.40 (2H, CH_2-S, $^2J_{ab} = 9.1$ Hz, $^3J_{ax} = 4.3$ Hz, $^3J_{bx} = 3.8$ Hz); ^{13}C NMR (δ, ppm): 38.1, 54.5, 68.2, 123.4, 127.4, 129.5, 129.9, 130.3, 133.2, 133.5, 148.1, 149.6; HRMS $[M\text{-}H]^-$: calcd. 477.9185, found 477.9186.

4i: IR (ν, cm^{-1}): 3357 (NH), 1765 (C═O), 1393–1173 (SO_2); ^1H NMR (δ, ppm): 8.55 (1H, NH), 7.30 (5H, arom), 6.80 (2H, arom), 4.20 (1H, CH-O), 3.60 (2H, CH_2-Br, $^2J_{ab} = 12.9$ Hz, $^3J_{ax} = 8.3$ Hz, $^3J_{bx} = 5.0$ Hz), 3.40 (2H, CH_2-S, $^2J_{ab} = 8.9$ Hz, $^3J_{ax} = 4.2$ Hz, $^3J_{bx} = 3.6$ Hz), 2.30 [6H, 2(CH_3)], 2.20 (3H, CH_3); ^{13}C NMR (δ, ppm): 17.1, 20.7, 38.5, 54.6, 68.1, 127.6, 129.4, 129.9, 130.5, 131.4, 133.2, 136.5, 145.8, 149.8; HRMS $[M\text{-}H]^-$: calcd. 486.0044, found 486.0054.

4j: IR (ν, cm^{-1}): 3354 (NH), 1764 (C═O), 1400–1174 (SO_2); ^1H NMR (δ, ppm): 8.90 (1H, NH), 7.30 (5H, arom), 4.90 (2H, CCl_3-CH_2-O), 4.15 (1H, CH-O), 3.60 (2H, CH_2-Br, $^2J_{ab} = 12.8$ Hz, $^3J_{ax} = 8.5$ Hz, $^3J_{bx} = 5.2$ Hz), 3.35 (2H, CH_2-S, $^2J_{ab} = 8.9$ Hz, $^3J_{ax} = 4.3$ Hz, $^3J_{bx} = 3.9$ Hz); ^{13}C NMR (δ, ppm): 38.2, 54.5, 68.3, 80.9, 92.8, 127.5, 129.4, 130.2, 133.1, 149.8; HRMS $[M\text{-}H]^-$: calcd. 497. 8405, found 497.8413.

Synthesis of *N*-Sulfonyl Oxazolidin-2-ones 5a–j

To 10 mmol of *N*-sulfonyl bromocarbamates **4a–j** in acetone solution, 10 mmol of triethylamine were added dropwise with stirring. The mixture was stirred at 60°C for 1 to 2 h. The precipitate of triethylammonium bromide that formed was filtered. The organic solution was evaporated in vacuum, and the residue was purified by column chromatography to give *N*-sulfonyloxazolidin-2-ones **5a–j** as very viscous oils (eluant: ethylic ether 20%, cyclohexane 80%).

Data

5a: IR (ν, cm^{-1}): 1803 (C═O), 1721 (C═O), 1647 (C═C), 1396–1182 (SO_2); ^1H NMR (δ, ppm): 6.85 (4H, arom, $^3J = 8.8$ Hz), 5.55 (1H, CH═C), 4.75 (1H, CH-O), 4.20 (2H, CH_2-O, $^2J_{ab} = 12.5$ Hz, $^3J_{ax} = 4.1$ Hz, $^3J_{bx} = 3.0$ Hz), 3.80 (2H, CH_2-N, $^2J_{ab} = 9.2$ Hz, $^3J_{ax} = 8.8$ Hz, $^3J_{bx} = 6.6$ Hz), 2.10 (3H, CH_3), 1.80 (3H, CH_3); ^{13}C NMR (δ, ppm): 20.5, 27.5, 47.7, 62.3, 72.3, 114.3, 123.1, 129.4, 134.0, 147.7, 150.8, 160.8, 166.1; HRMS $[M + Na]^+$: calcd. 412.0233, found 412.0241.

5b: IR (ν, cm^{-1}): 1804 (C═O), 1718 (C═O), 1646 (C═C), 1392–1176 (SO_2); ^1H NMR (δ, ppm): 6.80 (2H, arom), 5.65 (1H, CH═C),

4.80 (1H, CH-O), 4.25 (2H, CH_2-O, $^2J_{ab} = 12.5$ Hz, $^3J_{ax} = 4.1$ Hz, $^3J_{bx} = 3.1$ Hz), 4.05 (2H, CH_2-N, $^2J_{ab} = 9.2$ Hz, $^3J_{ax} = 8.6$ Hz, $^3J_{bx} = 6.5$ Hz), 2.30 [6H, 2(CH_3)], 2.20 (3H, CH_3), 2.10 (3H, CH_3), 1.80 (3H, CH_3); ^{13}C NMR (δ, ppm): 17.1, 20.3, 20.6, 27.4, 47.5, 62.3, 72.0, 114.7, 129.8, 131.2, 136.2, 145.2, 150.5, 159.8, 165.8; HRMS $[M + Na]^+$: calcd. 420.1092, found 420.1098.

5c: IR (ν, cm^{-1}): 1798 (C═O), 1713 (C═O), 1648 (C═C), 1378–1190 (SO_2); ^{1}H NMR (δ, ppm): 5.70 (1H, CH═C), 5.00 (2H, CCl_3-CH_2-O), 4.95 (1H, CH-O), 4.35 (2H, CH_2-O, $^2J_{ab} = 12.4$ Hz, $^3J_{ax} = 4.2$ Hz, $^3J_{bx} = 3.3$ Hz), 4.10 (2H, CH_2-N, $^2J_{ab} = 9.3$ Hz, $^3J_{ax} = 8.8$ Hz, $^3J_{bx} = 6.5$ Hz), 2.20 (3H, CH_3), 1.90 (3H, CH_3); ^{13}C NMR (δ, ppm): 20.4, 27.5, 47.2, 62.2, 72.6, 81.3, 92.8, 114.4, 150.1, 160.0, 165.8; HRMS $[M + Na]^+$: calcd. 431.9454, found 431.9465.

5d: IR (ν, cm^{-1}): 1803 (C═O), 1719 (C═O), 1648 (C═C), 1380–1187 (SO_2); ^{1}H NMR (δ, ppm): 7.35 (5H, arom), 5.60 (1H, CH═C), 4.80 (1H, CH-O), 4.20 (2H, CH_2-O, $^2J_{ab} = 12.5$ Hz, $^3J_{ax} = 4.1$ Hz, $^3J_{bx} = 3.0$ Hz), 3.85 (2H, CH_2-N, $^2J_{ab} = 9.2$ Hz, $^3J_{ax} = 8.5$ Hz, $^3J_{bx} = 6.5$ Hz), 2.15 (3H, CH_3), 1.85 (3H, CH_3); ^{13}C NMR (δ, ppm): 20.3, 27.5, 47.8, 62.3, 72.3, 114.7, 121.6, 128.3, 130.3, 149.4, 150.6, 159.7, 166.2; HRMS $[M + Na]^+$: calcd. 378.0623, found 378.0625.

5e: IR (ν, cm^{-1}): 1805 (C═O), 1719 (C═O), 1649 (C═C), 1395–1173 (SO_2); ^{1}H NMR (δ, ppm): 7.15 (4H, arom, $^3J = 8.8$ Hz), 5.60 (1H, CH═C), 5.15 (1H, CH-O), 4.15 (2H, CH_2-O, $^2J_{ab} = 12.4$ Hz, $^3J_{ax} = 4.1$ Hz, $^3J_{bx} = 3.3$ Hz), 3.80 (2H, CH_2-N, $^2J_{ab} = 9.2$ Hz, $^3J_{ax} = 8.8$ Hz, $^3J_{bx} = 6.6$ Hz), 2.30 (3H, CH_3), 2.10 (3H, CH_3), 1.85 (3H, CH_3); ^{13}C NMR (δ, ppm): 20.5, 20.8, 27.6, 48.0, 62.2, 73.8, 114.8, 121.5, 130.6, 138.4, 147.5, 150.8, 159.4, 166.1; HRMS $[M + Na]^+$: calcd. 392.0779, found 392.0786.

5f: IR (ν, cm^{-1}): 1804 (C═O), 1722 (C═O), 1647 (C═C), 1396–1182 (SO_2); ^{1}H NMR (δ, ppm): 6.95 (4H, arom), 5.55 (1H, CH═C), 4.75 (1H, CH-O), 4.20 (2H, CH_2-O, $^2J_{ab} = 12.5$ Hz, $^3J_{ax} = 4.2$ Hz, $^3J_{bx} = 3.2$ Hz), 3.80 (2H, CH_2-N, $^2J_{ab} = 9.2$ Hz, $^3J_{ax} = 8.7$ Hz, $^3J_{bx} = 6.4$ Hz), 2.10 (3H, CH_3), 1.80 (3H, CH_3); ^{13}C NMR (δ, ppm): 20.5, 27.5, 47.7, 62.3, 72.3, 114.3, ^{13}C NMR (δ, ppm): 20.4, 27.5, 47.7, 62.3, 72.4, 114.5, 116.0, 124.0, 146.5, 150.8, 160.5, 160.8, 166.1; HRMS $[M + Na]^+$: calcd. 396.0529, found 396.0540.

5g: IR (ν, cm^{-1}): 1811 (C═O), 1719 (C═O), 1648 (C═C), 1388–1173 (SO_2); ^{1}H NMR (δ, ppm): 7.25 (2H, arom), 5.70 (1H, CH═C), 5.00 (1H, CH-O), 4.40 (2H, CH_2-O, $^2J_{ab} = 12.5$ Hz, $^3J_{ax} = 4.1$ Hz, $^3J_{bx} = 3.0$ Hz), 4.20 (2H, CH_2-N, $^2J_{ab} = 9.4$ Hz, $^3J_{ax} = 8.6$ Hz, $^3J_{bx} = 6.6$ Hz), 2.20 (3H,

CH_3), 1.95 (3H, CH_3); ^{13}C NMR (δ, ppm): 20.5, 27.5, 47.3, 62.0, 72.2, 114.4, 129.3, 130.6, 133.9, 142.1, 150.0, 158.9, 165.7; HRMS $[M + Na]^+$: calcd. 479.9454, found 479.9463.

5h: IR (ν, cm^{-1}): 1802 (C═O), 1365–1179 (SO_2); ^1H NMR (δ, ppm): 7.30 (5H, arom), 6.90 (4H, arom, $^3J = 8.8$ Hz), 4.65 (1H, CH-O), 3.90 (2H, CH_2-N, $^2J_{ab} = 10.7$ Hz, $^3J_{ax} = 8.6$ Hz, $^3J_{bx} = 6.4$ Hz), 3.10 (2H, CH_2-S, $^2J_{ab} = 9.0$ Hz, $^3J_{ax} = 4.2$ Hz, $^3J_{bx} = 3.2$ Hz); ^{13}C NMR (δ, ppm): 50.1, 56.8, 73.1, 123.3, 127.5, 129.4, 129.8, 130.1, 133.2, 133.7, 147.9, 151.0; HRMS $[M + Na]^+$: calcd. 421.9899, found 421.9903.

5i: IR (ν, cm^{-1}): 1805 (C═O), 1378–1192 (SO_2); ^1H NMR (δ, ppm): 7.30 (5H, arom), 6.80 (2H, arom), 4.70 (1H, CH-O), 4.05 (2H, CH_2-N, $^2J_{ab} = 10.9$ Hz, $^3J_{ax} = 8.6$ Hz, $^3J_{bx} = 6.5$ Hz), 3.30 (2H, CH_2-S, $^2J_{ab} = 8.9$ Hz, $^3J_{ax} = 4.1$ Hz, $^3J_{bx} = 3.0$ Hz), 2.30 [6H, 2(CH_3)], 2.20 (3H, CH_3); ^{13}C NMR (δ, ppm): 17.2, 20.7, 50.0, 56.8, 74.1, 127.2, 129.3, 129.6, 130.2, 131.5, 133.0, 136.4, 145.7, 150.8; HRMS $[M + Na]^+$: calcd. 430. 0758, found 430.0764.

5j: IR (ν, cm^{-1}): 1797 (C═O), 1395–1184 (SO_2); ^1H NMR (δ, ppm): 7.30 (5H, arom), 4.95 (2H, CCl_3-CH_2-O), 4.75 (1H, CH-O), 4.10 (2H, CH_2-N, $^2J_{ab} = 10.8$ Hz, $^3J_{ax} = 8.5$ Hz, $^3J_{bx} = 6.5$ Hz), 3.25 (2H, CH_2-S, $^2J_{ab} =$ 9.1 Hz, $^3J_{ax} = 4.2$ Hz, $^3J_{bx} = 3.2$ Hz); ^{13}C NMR (δ, ppm): 49.7, 57.0, 74.8, 81.5, 92.7, 127.6, 129.2, 130.3, 133.5, 151.1; HRMS $[M + Na]^+$: calcd. 441.9120, found 441.9127.

CONCLUSION

As shown in Table 2, a series of new compounds with different substitutions on nitrogen and the C5 position of the oxazolidin-2-one ring was prepared and may allow the establishment of structure–activity relationships. This will be done in collaboration with appropriate laboratories.

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