Retraction

Retracted: One-Pot Synthesis of Pyrrolidine-2-ones from Erythruronolactone and Amine

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This article has been retracted as it was submitted for publication without the prior knowledge or approval of Dr. Aloysius Siriwardena, who has contributed to the article. Additionally, it has been submitted without prior approval of the Centre National de la Recherche Scientifique (CNRS) and the laboratory in which the intellectual ideas behind the syntheses were established [1].

References

- [1] C. Benhaoua, "One-pot synthesis of pyrrolidine-2-ones from erythruronolactone and amine," *Organic Chemistry International*, vol. 2012, Article ID 482952, 6 pages, 2012.

Research Article

One-Pot Synthesis of Pyrrolidine-2-ones from Erythruronolactone and Amine

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Concise and accessible approaches to pyrrolidine-2-ones were studied. The synthesis involves the conversion of erythruronolactone to pyrrolidine-2-ones in good yields.

1. Introduction

Many pyranoses and furanoses with the ring oxygen replaced by an amino group, known as imino sugars or azasugars, have been found to inhibit specific enzymes such as glycosidases [1]. Inhibition of these enzymes which catalyze the hydrolysis of the glycosidic bond to residues of oligosaccharides or saccharides is a potentially useful target of a wide range of important biological processes, such as intestinal digestion and posttranslational processing of glycoproteins [2], cancer [3], and diabetes [4] and other metabolic disorders [5]. Most inhibitors of glycosidases described to date are mimics of the supposed transition state oxocarbenium [6]. The azasugars or iminosugars [7–9] are structures that are the most widely used.

Members of this class include 2,5-dihydroxymethyl-3,4-dihydropyrrolidine (DMDP) (**1**) that was first isolated from leaves of *derris elliptica* (Fabaceae) [10] and 1,4-dideoxy-1,4-imino-D-arabinitol (DAB) (**2**) that was first found in the fruits of *angylocalyx boutiqueanus* (Fabaceae) [11]. They are selective inhibitors of glycosidase [12] (Figure 1).

The 1,4-dideoxy-1,4-imino-L-arabinitol (LAB, **3**) (Figure 2) was first found in the fruits of *angylocalyx boutiqueanus* (Fabaceae) [11], which is a powerful inhibitor of sucrase and α -glucosidases [13].

Recently, cis-5-benzyl-4-hydroxy-2-pyrrolidinone **4**, named as streptopyrrolidine, was isolated from the fermentation of the broth of marine *Streptomyces* [14] (Figure 2). This

compound exhibited significant antiangiogenesis activity and is expected to be a unique small-molecule bioprobe for studying angiogenesis.

To combine the importance of the compounds **2**, **3**, and **4** we have decided to conduct a modification of compound **3** at the hydroxyl groups and alkylation of the nitrogen atom. Our aim is to study the influence of the N-alkyl chain on the inhibition of glycosidases for treating antiviral infection such as hepatitis C.

Their synthesis is of great importance. Many methodologies for the synthesis of pyrrolidine iminosugars were developed. The nitrogen introduction often involves azide substitution [15] or oximation [16]. Heavy-metal catalysis was also widely used to install ring systems or hydroxyl groups [17].

Most of these methods are restricted requiring harsh reaction conditions, with low moderate yields and relatively long time, and especially several steps are necessary to obtain the expected products

Thus, the development of a short and practicable synthetic pathway of five-member iminosugar derivatives containing a pyrrolidinone core from potentially abundant carbohydrate material is desirable.

2. Results and Discussion

As illustrated in Scheme 1, a protected pyrrolidinone iminosugar (**7**) was obtained by condensation of an appropriate

FIGURE 1: Inhibitors of glycosidase.

FIGURE 2: LAB 3 and streptopyrrolidine 4.

amine (**6**) with the 2,3-O-isopropylidene-D-erythruronolactone (**5**).

From the data presented in Table 1, it was noticed that the compounds (**7**) are prepared at room temperature in yields ranging from 60 to 87%.

The ^1H and ^{13}C NMR spectra determine the formation of the pyrrolidine ring.

In general, we have obtained an inseparable mixture of epimers except of three products when R is Ph, 4-methylmorpholin-2-yl or 2-ethyn-2-yl. Assignment of the majority of protons is ready from the ^1H , ^1H COSY 2D spectrum. For the products **7a-p**, the signal for the H_5 proton of the pyrrolidine cycle is around 4.10 ppm to 4.75 ppm and with coupling constant values ($J_{5,4} = 4.40\text{--}6.08$ Hz), while the compounds **7a'-p'** showed the chemical shift pattern for single proton H_5 of the pyrrolidine cycle around 4.16 ppm to 4.69 ppm and coupling constant values ($J_{5,4}$ trans = 0 Hz). These results are confirmed by the results of Zhou et al. [17] in determining the stereochemistry of γ -lactams derivatives.

In the ^{13}C NMR of the compounds **7a-p**, characteristic ($\text{C}=\text{O}$) appeared at around 168.76 to 170.20 and characteristic ($\text{C}=\text{O}$) products (**7a'-p'**) were recorded at around 169.38–171.31 ppm. The ^{13}C NMR spectrum of all the compounds showed the characteristic of signal for the ($\text{C}(\text{Me})_2$) of isopropylidene at around 112.93 to 112.32 ppm.

3. Conclusion

A series of novel pyrrolidine iminosugars are synthesized in only one step from 2,3-O-isopropylidene-D-erythruronolactone (**5**). None of the above processes involved any azide or heavy-metal catalysis. In this work, we have used ordinary reagents and developed a rapid access to give these important iminosugar derivatives with reasonable yields.

Works are underway to study the biological activities of these compounds and to apply this simple methodology to the synthesis of tricyclic compounds.

4. Experimental

Chemicals were purchased from Aldrich, Acros, and Fluka and used without further purification. Solvents were distilled with appropriate drying agents. All reactions were performed under anhydrous conditions employing routine drying techniques unless otherwise indicated. Reactions were monitored by thin-layer chromatography (TLC) performed on E. Merck glass plate silica gel sheets (Silica Gel F₂₅₄) and stained with vanillin acid aqueous H_2SO_4 solution. Column chromatography was carried out on silica gel (E. Merck 230–400 mesh). Nuclear magnetic resonance (NMR) data (^1H or ^{13}C) were obtained on a AC-Brucker 300 machine, and chemical shifts are reported in parts per million relative to tetramethylsilane in deuterated solvents. Assignments of ^1H and ^{13}C were assisted by 2D ^1H COSY and 2D ^1H – ^{13}C CORR experiments.

Optical rotations were determined with a Jasco Dip 370 electronic micropolarimeter (10 cm cell). High-resolution electrospray mass spectra (ESI-HRMS) in the positive ion mode were obtained on a Q-TOF *Ultima Global* hybrid quadrupole time-of-flight instrument (Waters-Micro mass), equipped with a pneumatically assisted electrospray (Z-spray) ionization source and an additional sprayer (Lock Spray) for the reference compound. Compound (**5**) is synthesized as described in the literature [18].

4.1. General Procedure for Synthesis of (7). To a mixture of 2,3-O-isopropylidene-D-erythruronolactone (0.86 mmol of sugar) and a primary amine (excess) 5 mL of CH_2Cl_2 was added. The mixture was stirred for 12 h at RT. After concentration under reduced pressure, the resulting was purified by chromatography ($\text{CH}_2\text{Cl}_2/\text{MeOH}$, 95/5). The characterization of each compound was obtained by means of NMR and mass spectrometry as reported below.

4.1.1. N-1-(pyridin-2-yl-methyl)-3,4-O-isopropylidendioxy-5(pyridine-2-yl-methyl-amino)-pyrrolidin-2-one (7a, 7a'). These compounds were obtained as a mixture of epimers nonseparable by chromatography, yellow gum. For the epimer (**7a**), NMR ^1H (CDCl_3) δ : 8.56 (m, phenyl protons); 7.70 (m, phenyl protons); 7.43 (d, phenyl protons); 7.22 (m, phenyl protons); 4.93 (d, 1H, $-\text{CH}_2-$, $J = 15.59$ Hz), 4.77 (m, 2H, H-3, H-4); 4.75 (m, 2H, H-5, $-\text{CH}_2-$); 4.10–4.15 (d, 2H, $-\text{CH}_2-$, $J = 14.78$ Hz); 4.02–4.07 (d, 2H, $-\text{CH}_2-$, $J = 14.91$ Hz); 3.85 (NH); 1.51 (s, 3H, CH_3), 1.40 (s, 3H, CH_3). NMR ^{13}C (CDCl_3) δ : 170.20 ($\text{C}=\text{O}$); 148.07–148.85 (C-aromatic); 137.11–137.68 (C-aromatic); 122.43–122.51; (C-aromatic); 112.93; 77.42; 73.59; 72.32; 51.52–44.58; 27.09–26.00. For the epimer (**7a'**), NMR ^1H (CDCl_3) δ : 8.52 (m, phenyl protons); 7.71 (d, phenyl protons); 7.30 (m, phenyl protons); 4.92 (d, 1H, H-4, $J = 5.69$ Hz); 4.81 (d, 1H, CH_2 , $J = 11.52$ Hz); 4.74 (d, 1H, H-3, $J = 5.75$ Hz); 4.68 (s, 1H, H-5, $J = 0$ Hz); 4.58 (d, 1H,

SCHEME 1: Synthesis of polyhydroxylated pyrrolidinone.

TABLE 1: Result of the preparation of protected polyhydroxylated pyrrolidinone iminosugar (7).

Products ^a	R	Yields	7a-p/7a'-p'
(5a', 5a)	Pyridin-2-yl	87	40/60
(5b', 5b)	2-Methylpyridin-2-yl	77	45/55
(5c', 5c)	4-Methylpyridin-2-yl	56	33/67
(5d', 5d)	2-Ethen-2-yl	77	36/63
(5e', 5e)	2,2-Diethoxyeth-2-yl	55	50/50
(5f', 5f)	2,2-Dimethoxymeth-2-yl	56	45/55
(5g', 5g)	2-Ethyn-2-yl	77	53/47
(5h', 5h)	Ethyl	60	50/50
(5i', 5i)	Propyl	50	67/33
(5j', 5j)	Butyl	66	50/50
(5k', 5k)	Pentyl	50	50/50
(5l', 5l)	Hexyl	50	50/50
(5m', 5m)	Heptyl	65	67/33
(5n', 5n)	Octyl	50	50/50
(5o', 5o)	Ph	72	21/79
(5p', 5p)	4-Methylmorpholin-2-yl	60	22/78

^a Mixture of epimers.

–CH₂–, *J* = 15.25 Hz); 4.04 (d, 2H, –CH₂–, *J* = 14.12 Hz), 3.14 (NH); 1.40 (s, 3H, CH₃); 1.31 (s, 3H, CH₃).

NMR ¹³C(CDCl₃) δ: 171.31(C=O); 157.05–122.43 (C-aromatic); 112.95; 77.73; 76.88; 76.33; 48.71; 46.66; 27.00; 25.70. HRMS (*m/z*) [M+H]⁺ calcd for C₁₉H₂₃N₄O₃ 355.1770, found 355.1763.

4.1.2. *N*-1-(pyridin-2-yl-ethyl)-3,4-*O*-isopropylidendioxy-5 (pyridine-2-yl-ethyl-amino)-pyrrolidin-2-one (**7b**, **7b'**). These compounds were obtained as a mixture of epimers non-separable by chromatography, orange gum. For the epimer (**7b**), NMR ¹H (CDCl₃) δ: 8.3 (m, phenyl protons); 7.39 (m, phenyl protons); 6.98 (m, phenyl protons); 4.48 (m, 1H, H-4, *J* = 4.79 Hz); 4.35 (d, 1H, H-3, *J* = 6.12 Hz); 4.10 (d, 1H, H-5, *J* = 4.67 Hz); 3.47 (m, 1H, –CH₂–); 3.25 (m, 1H, –CH₂–); 2.77 (m, 2H, –CH₂–); 2.69 (m, 3H, –CH₂–); 2.56 (m, 1H, –CH₂–); 1.16 (s, 6H, CH₃). NMR ¹³C (CDCl₃) δ: 170.92 (C=O); 149.64–123.42 (C-aromatic); 112.32; 77.32; 73.24; 72.87; 46.34; 39.54; 39.16; 35.57; 28.89; 25.87. For the epimer (**7b'**): NMR ¹H (CDCl₃) δ: 8.33 (m, phenyl protons); 7.39 (m, phenyl protons); 6.98 (m, phenyl protons); 4.51 (d, 1H, H-4, *J* = 5.93 Hz); 4.24 (d, 1H, H-3, *J* = 5.95 Hz); 4.16

(s, 1H, H-5, *J* = 0 Hz); 3.72 (m, 1H, –CH₂–); 3.58 (m, 1H, –CH₂–); 3.01 (m, 1H, –CH₂–); 2.77 (m, 3H, –CH₂–); 2.69 (m, 2H, –CH₂–); 1.14 (s, 6H, CH₃). NMR ¹³C (CDCl₃) δ: 169.67 (C=O); 149.64–123.42 (C-aromatic); 112.37; 78.01; 76.98; 76.01; 46.53; 39.25; 37.36; 35.18; 26.89; 25.50. HRMS (*m/z*) [M+H]⁺ calcd for C₂₁H₂₇N₄O₃ 383.2083, found 383.2089.

4.1.3. *N*-1-(pyridin-4-yl-ethyl)-3,4-*O*-isopropylidendioxy-5 (pyridine-4-yl-ethyl-amino)-pyrrolidin-2-one (**7c**, **7c'**). These compounds were obtained as a mixture of epimers non-separable by chromatography, colorless syrup. For the epimer (**7c**), NMR ¹H (CDCl₃) δ: 8.36 (m, phenyl protons); 7.02 (m, phenyl protons); 4.52 (d, 1H, H-4, *J* = 11.52 Hz); 4.44 (d, 1H, H-3, *J* = 6.11 Hz); 4.00 (d, 1H, H-5, *J* = 4.49 Hz); 3.67 (m, 1H, –CH₂–); 3.39 (m, 1H, –CH₂–); 2.96 (m, 1H, –CH₂–); 2.68 (m, 5H, –CH₂–); 1.24 (s, 6H, CH₃). NMR ¹³C (CDCl₃) δ: 169.68 (C=O); 149.64–124.18 (C-aromatic); 112.64; 77.12; 73.17; 72.99; 46.62; 40.17; 36.29; 33.08; 26.83; 25.83. For the epimer (**7c'**), NMR ¹H (CDCl₃) δ: 8.55 (m, phenyl protons); 7.23 (m, phenyl protons); 4.67 (d, 1H, H-4, *J* = 5.94 Hz); 4.38 (d, 1H, H-3, *J* = 5.99 Hz); 4.23 (s, 1H, H-5,

$J = 0$ Hz); 3.84 (m, 1H, $-\text{CH}_2-$); 3.19 (m, 1H, $-\text{CH}_2-$); 2.75 (m, 6H, $-\text{CH}_2-$); 1.36 (s, 6H, CH_3). NMR ^{13}C (CDCl_3) δ : 170.73 (C=O); 149.57–124.26 (C-aromatic); 112.96; 77.97; 76.82; 76.12; 43.72; 40.00; 35.87; 32.60; 26.48; 25.47. HRMS (m/z) $[\text{M}+\text{H}]^+$ calcd for $\text{C}_{21}\text{H}_{27}\text{N}_4\text{O}_3$ 382.2083, found: 383.2082.

4.1.4. *N*-1-(allyl)-3, 4-*O*-isopropylidendioxy-5-(allylamino)-pyrrolidin-2-one (7d, 7d'). These compounds were obtained as a mixture of epimers nonseparable epimer by chromatography, yellow gum. For the epimer (7d), NMR ^1H (CDCl_3) δ : 5.76 (m, 2H, $=\text{CH}$); 5.17 (m, 4H, $=\text{CH}_2$); 4.75 (d, 1H, H-4, $J = 4.73$ Hz); 4.73 (d, 1H, H-3, $J = 6.12$ Hz); 4.30 (d, 1H, H-5, $J = 4.73$ Hz); 4.16–4.25 (m, 1H, $-\text{CH}_2-$); 3.80–3.88 (dd, 1H, $-\text{CH}_2-$, $J = 16.07$, 6.89 Hz); 3.39 (m, 2H, $-\text{CH}_2-$); 1.94 (NH); 1.45 (s, 3H, CH_3); 1.41 (s, 3H, CH_3). NMR ^{13}C (CDCl_3) δ : 169.34 (C=O); 136.91–132.26; 116.29–118.08; 112.85; 77.49; 73.43; 71.49; 49.58; 41.89; 27.08; 26.12. For the epimer (7d'), NMR ^1H (CDCl_3) δ : 5.74 (m, 2H, $=\text{CH}$); 5.23 (m, 4H, $=\text{CH}_2$); 4.75 (d, 1H, H-4, $J = 5.71$ Hz); 4.53 (d, 1H, H-3, $J = 3.83$ Hz); 4.48 (s, 1H, H-5, $J = 0$ Hz); 4.16–4.35 (m, 1H, $-\text{CH}_2-$); 3.60–3.68 (dd, 1H, $-\text{CH}_2-$, $J = 15.5$, 6.87 Hz); 3.11–3.28 (m, 2H, $-\text{CH}_2-$); 2.77 (NH); 1.44 (s, 3H, CH_3); 1.39 (s, 3H, CH_3). NMR ^{13}C (CDCl_3) δ : 170.79 (C=O); 135.13–131.39; 118.46–117.13; 112.78; 76.14; 77.06; 74.86; 49; 57; 42.41; 27.07; 25.70. HRMS (m/z) $[\text{M}-\text{H}]^+$ calcd for $\text{C}_{13}\text{H}_{21}\text{N}_2\text{O}_3$ 253.1552, found: 253.1554.

4.1.5. *N*-1-(3,3-diethoxypropyl)-3,4-*O*-isopropylidendioxy-5-(3,3-diethoxypropylamino)pyrrolidin-2-one (7e, 7e'). These compounds were obtained as a mixture of epimers nonseparable by chromatography, colorless syrup. For the epimer (7e), NMR ^1H (CDCl_3) δ : 4.65–4.86 (m, 1H, H-4, $J = 4.73$ Hz); 4.56–4.64 (m, 2H, $J = 6.03$ Hz, H-3, $-\text{CH}-\text{OEt}$); 4.42–4.48 (m, 1H, $\text{CH}-\text{OEt}$); 4.27–4.29 (d, 1H, H-5, $J = 4.52$ Hz); 3.41–3.59 (m, 8H, $-\text{OCH}_2-$); 1.99 (NH); 1.73–3.29 (m, 8H, $-\text{CH}_2-$); 1.31–1.35 (2s, 6H, CH_3); 1.10–1.18 (m, 12H, CH_3). NMR ^{13}C (CDCl_3) δ : 169.54 (C=O); 112.36–112.56; 100.82–102.06; 77.58; 73.34; 72.98; 61.12–61.52; 31.23–42.80; 25.63–27.87; 15.28. For the epimer (7e'), NMR ^1H (CDCl_3) δ : 4.58–4.64 (m, 1H, $\text{CH}-\text{OEt}$); 4.51–4.53 (d, 1H, H-4, $J = 5.71$ Hz); 4.42–4.48 (m, 1H, $\text{CH}-\text{OEt}$); 4.40–4.41 (d, 1H, H-3, $J = 5.79$ Hz); 4.36 (s, 1H, H-5, $J = 0$ Hz); 3.41–3.59 (m, 8H, OCH_2); 1.99 (NH); 1.73–3.29 (m, 8H, $-\text{CH}_2-$); 1.31–1.35 (2s, 6H, CH_3); 1.10–1.18 (m, 12H, CH_3). NMR ^{13}C (CDCl_3) δ : 170.69 (C=O); 112.36–112.56; 100.82–102.06; 78.16; 77.09; 76.01; 61.12–61.52; 31.23–42.80; 25.63–27.87; 15.28. HRMS (m/z) $[\text{M}+\text{H}]^+$ calcd for $\text{C}_{21}\text{H}_{41}\text{N}_2\text{O}_7$ 433.2914, found 433.2911.

4.1.6. *N*-1-(2,2-dimethoxy-ethyl)-3,4-*O*-isopropylidendioxy-5-(dimethoxy-ethylamino)-pyrrolidin-2-one (7f, 7f'). These compounds were obtained as a mixture of epimers nonseparable by chromatography, colorless syrup. For the epimer (7f), NMR ^1H (CDCl_3) δ : 4.70–4.74 (m, 1H, H-4, $J = 6.14$ Hz); 4.62–4.64 (d, 1H, H-3, $J = 6.14$ Hz); 4.51–4.56 (m, 1H, $-\text{CH}-\text{O}$); 4.47–4.49 (d, 1H, H-5, $J = 4.6$ Hz); 4.40–4.43 (m, 1H, $-\text{CH}-\text{O}$); 3.36–3.40 (m, 12H, OCH_3); 3.55–3.68 (m, 1H, $-\text{CH}_2-$, $J = 9.65$, 4.60 Hz); 3.44–3.46

(d, 1H, $-\text{CH}_2-$, $J = 4.74$ Hz); 2.91–2.97 (dd, 1H, $-\text{CH}_2-$, $J = 12.9$, 6.36 Hz); 2.81–2.86 (dd, 1H, $-\text{CH}_2-$, $J = 9.20$, 4.39 Hz); 2.02 (NH); 1.38–1.42 (2s, 6H, CH_3). NMR ^{13}C (CDCl_3) δ : 169.73 (C=O); 112.42; 101.86–104.47; 77.19; 73.71; 73.42; 55.16–55.37 (4OCH_3); 40.03–48.39; 25.84–27.03. For the epimer (7f'): NMR ^1H (CDCl_3) δ : 4.70–4.74 (m, 1H, H-4, $J = 5.7$ Hz); 4.57 (s, 1H, H-5, $J = 0$ Hz); 4.51–4.56 (m, 1H, $-\text{CH}-\text{O}$); 4.44–4.46 (d, 1H, H-3, $J = 5.9$ Hz); 4.40–4.43 (m, 1H, $-\text{CH}-\text{O}$); 3.36–3.40 (m, 12H, OCH_3); 3.55–3.68 (m, 1H, $-\text{CH}_2-$, $J = 9.65$, 4.60 Hz); 3.20–3.27 (dd, 1H, $-\text{CH}_2-$, $J = 14.21$, 6.14 Hz); 2.61–2.63 (dd, 1H, $-\text{CH}_2-$, $J = 11.8$, 4.62 Hz); 2.56–2.58 (dd, 1H, $-\text{CH}_2-$, $J = 11.08$, 5.05 Hz); 2.02 (NH); 1.38–1.42 (2s, 6H, CH_3). NMR ^{13}C (CDCl_3) δ : 171.27 (C=O); 112.42; 102.46–103.66; 78.3; 77.11; 76.87; 55.16–55.37 (4OCH_3); 42.51–45.27; 25.84–27.03. HRMS (m/z) $[\text{M}+\text{H}]^+$ calcd for $\text{C}_{15}\text{H}_{29}\text{N}_2\text{O}_7$ 349.1975, found: 349.1979.

4.1.7. *N*-1-(2-propynyl)-3,4-*O*-isopropylidendioxy-5-(2-propynylamino)-pyrrolidin-2-one (7g, 7g'). This compound was obtained as a mixture of epimers separable by chromatography, colorless syrup. For the epimer (7g), $[\alpha]_D^{20} = -2.43$ (C = 0.55, MeOH); NMR ^1H (CDCl_3) δ : 4.91 (m, 1H, H-4, $J = 4.49$ Hz); 4.75 (m, 2H, H-5, H-3, $J = 6.08$ Hz); 4.42 (dd, 1H, $-\text{CH}_2-$, $J = 14.63$, 2.51 Hz); 3.89 (dd, 1H, $-\text{CH}_2-$, $J = 14.64$, 2.5 Hz); 3.33 (m, 2H, $-\text{CH}_2-$); 2.24–2.26 (m, 2H, $-\text{CH}-$); 1.37 (s, 6H, CH_3). NMR ^{13}C (CDCl_3) δ : 168.76 (C=O); 112.92; 82.12; 77.64; 73.39; 72.01; 71.21–71.78; 36.15; 28.79; 26.06–26.97. For the epimer (7g'), $[\alpha]_D^{20} = 6.31$ (C = 0.58, MeOH). NMR ^1H (CDCl_3) δ : 4.71 (d, 1H, H-4, $J = 5.88$ Hz); 4.67 (s, 1H, H-5); 4.60 (d, 1H, H-3, $J = 5.88$ Hz); 4.39 (dd, 1H, $-\text{CH}_2-$, $J = 14.96$, 2.5 Hz); 3.88 (dd, 1H, $-\text{CH}_2-$, $J = 14.19$, 2.5 Hz); 3.44 (m, 2H, $-\text{CH}_2-$); 2.8 (m, 2H, $-\text{CH}-$); 1.36 (s, 6H, CH_3). NMR ^{13}C (CDCl_3) δ : 170.43 (C=O); 112.92; 81.19; 78.06; 75.88; 75.41; 72.90–73.40; 33.38; 30.19; 25.95–27.12. HRMS (m/z) $[\text{M}+\text{Na}]^+$ calcd for $\text{C}_{13}\text{H}_{16}\text{N}_2\text{O}_3\text{Na}$ 271.1059, found: 271.1065.

4.1.8. *N*-1-(propyl)-3,4-*O*-isopropylidendioxy-5-(propylamino)-pyrrolidin-2-one (7h, 7h'). These compounds were obtained as a mixture of epimers nonseparable by chromatography, colorless syrup. For the epimer (7h), NMR ^1H (CDCl_3) δ : 4.73 (m, 2H, H-4, H-3, $J = 4.75$ Hz); 4.31 (d, 1H, H-5, $J = 4.64$ Hz); 3.25 (m, 1H, CH_2); 2.98 (m, 1H, CH_2); 2.83 (m, 1H, CH_2); 2.80 (m, 1H, CH_2); 1.43 (m, 4H, 2CH_2); 0.93 (m, 6H, 2CH_3). NMR ^{13}C (CDCl_3) δ : 170 (C=O); 112.58; 77.54; 73.44; 72.90; 48.97; 41.36; 26.17–27.11; 20.65–23.99; 11.16–11.69. For the epimer (7h'): NMR ^1H (CDCl_3) δ : 4.57 (d, 1H, H-4, $J = 6.08$ Hz); 4.52 (d, 1H, H-3, $J = 5.81$ Hz); 4.46 (s, 1H, H-5, $J = 0$ Hz); 3.59 (m, 1H, CH_2); 3.38 (m, 1H, CH_2); 2.5 (m, 1H, CH_2); 2.47 (m, 1H, CH_2); 1.43 (m, 4H, 2CH_2); 1.38 (m, 6H, 2CH_3); 0.93 (m, 6H, 2CH_3). NMR ^{13}C (CDCl_3) δ : 170 (C=O); 112.58; 78.21; 72.30; 75.53; 45.21; 41.53; 25.75–27.11; 20.07–23.28; 11.16–11.69. HRMS (m/z) $[\text{M}+\text{H}]^+$ calcd for $\text{C}_{13}\text{H}_{25}\text{N}_2\text{O}_3$: 257.1865, found: 257.1863.

4.1.9. *N*-1-(butyl)-3,4-*O*-isopropylidendioxy-5-(butyl-amino)-pyrrolidin-2-one (7i, 7i'). These compounds were obtained as a mixture of epimers nonseparable by chromatography,

colorless syrup. For the epimer (**7i**), NMR ^1H (CDCl_3): δ 4.6 (m, 2H, H-4, H-3, J = 5.6 Hz); 4.27 (d, 1H, H-5, J = 4.62 Hz); 3.58 (m, 1H, CH_2); 3.51 (m, 1H, CH_2), 2.74 (m, 1H, CH_2); 2.64 (m, 1H, CH_2); NH (1.48); 1.31 (m, 4H, 2 CH_2), 1.25 (m, 6H, 2 CH_3); 0.88 (m, 6H, 2 CH_3). NMR ^{13}C (CDCl_3): δ : 169.45 (C=O); 112.53; 77.51; 73.36; 72.79; 46.67; 39.45; 32.89; 29.65; 28.08; 26.13; 19.90–20.30; 13.69–13.86. For the epimer (**7i'**): NMR ^1H (CDCl_3): δ 4.56 (d, 1H, H-4, J = 6.1 Hz); 4.44 (d, 1H, H-3, J = 5.54 Hz); 4.39 (s, 1H, H-5, J = 0 Hz); 3.19 (m, 1H, CH_2); 2.90 (m, 1H, CH_2); 2.38 (m, 1H, CH_2); 2.36 (m, 1H, CH_2); NH (1.48); 1.31 (m, 4H, 2 CH_2); 1.25 (m, 6H, 2 CH_3); 0.88 (m, 6H, 2 CH_3). NMR ^{13}C (CDCl_3): δ 170.78 (C=O); 112.42; 78.27; 77.30; 75.58; 43.02; 39.35; 32.26; 28.80; 25.74–27.09; 19.90–20.30; 13.69–13.86. HRMS (m/z) [$\text{M}+\text{H}$] $^+$ calcd for $\text{C}_{15}\text{H}_{29}\text{N}_2\text{O}_3$: 285.2178, found: 285.2191.

4.1.10. *N*-1-(pentyl)-3,4-isopropylidendioxy-5-(pentylamino)-pyrrolidin-2-one (**7j**, **7j'**). These compounds were obtained as a mixture of epimers nonseparable by chromatography, colorless syrup. For the epimer (**7j**), NMR ^1H (CDCl_3): δ 4.70 (m, 2H, H-4, H-3, J = 4.60 Hz); 4.30 (d, 1H, H-5, J = 4.63 Hz); 3.22 (m, 1H, CH_2), 3.93 (m, 1H, CH_2), 2.80 (m, 1H, CH_2), 2.62 (m, 1H, CH_2), 1.79 (NH), 1.29–1.42 (m, 12H, 6 CH_2), 0.90 (m, 6H, 2 CH_3).

NMR ^{13}C (CDCl_3): δ 169.43 (C=O); 112.46; 77.54; 73.38; 72.80; 46.99; 39.71; 26.99; 26.15; 22.32–30.48; 13.96. For the epimer (**7j'**): NMR ^1H (CDCl_3): δ 4.56 (d, 1H, H-4, J = 6.09 Hz); 4.46 (d, 1H, H-3, J = 5.85 Hz); 4.42 (s, 1H, H-5, J = 0 Hz); 3.59 (m, 1H, CH_2); 3.39 (m, 1H, CH_2); 2.49 (m, 1H, CH_2); 2.38 (m, 1H, CH_2); 1.79 (NH); 1.29–1.42 (m, 12H, 6 CH_2); 0.90 (m, 6H, 2 CH_3). NMR ^{13}C (CDCl_3): δ 170.80 (C=O); 112.42; 78.28; 77.31; 75.52; 43.31; 39.59; 27.09; 25.75; 22.32–30.48; 13.96. HRMS (m/z) [$\text{M}+\text{H}$] $^+$ calcd for $\text{C}_{17}\text{H}_{33}\text{N}_2\text{O}_3$: 313.2491, found: 313.2499.

4.1.11. *N*-1-(hexyl)-3,4-isopropylidendioxy-5-(hexylamino)-pyrrolidin-2-one (**7k**, **7k'**). These compounds were obtained as a mixture of epimers nonseparable by chromatography, colorless syrup. For the epimer (**7k**), NMR ^1H (CDCl_3): δ 4.80 (m, 2H, H-4, H-3, J = 4.57 Hz); 4.28 (d, 1H, H-5, J = 4.62 Hz); 3.23 (m, 1H, CH_2); 3.93 (m, 1H, CH_2); 2.80 (m, 1H, CH_2); 2.62 (m, 1H, CH_2); 1.77 (NH); 1.24–1.6 (m, 18H, 9 CH_2); 0.71–0.85 (m, 6H, 2 CH_3). NMR ^{13}C (CDCl_3): δ 169.43 (C=O); 112.44–112.54; 74.54; 73.38; 72.81; 46.98; 39.75; 27.08; 26.14; 26.36–31.61; 22.53; 13.99. For the epimer (**7k'**): NMR ^1H (CDCl_3): δ 4.63 (d, 1H, H-4, J = 6.08 Hz); 4.48 (d, 1H, H-3, J = 5.84 Hz); 4.45 (s, 1H, H-5, J = 0 Hz); 3.57 (m, 1H, CH_2); 3.38 (m, 1H, CH_2); 2.48 (m, 1H, CH_2); 2.37 (m, 1H, CH_2); 1.77 (NH); 1.24–1.6 (m, 18H, 9 CH_2); 0.71–0.85 (m, 6H, 2 CH_3). NMR ^{13}C (CDCl_3): δ 170.79 (C=O); 112.44–112.55; 78.29; 77.31; 75.53; 43.32; 39.63; 27.08; 25.74; 26.36–31.61; 22.53; 13.99. HRMS (m/z) [$\text{M}+\text{Na}$] $^+$ calcd for $\text{C}_{19}\text{H}_{36}\text{N}_2\text{O}_3$ Na: 363.2624, found: 363.2615.

4.1.12. *N*-1-(heptyl)-3,4-isopropylidendioxy-5-(heptylamino)-pyrrolidin-2-one (**7l**, **7l'**). These compounds were obtained as a mixture of epimers nonseparable by chroma-

tography, colorless syrup. For the epimer (**7l**), NMR ^1H (CDCl_3): δ 4.67 (m, 2H, H-4, H-3, J = 4.75 Hz); 4.26 (d, 1H, H-5, J = 4.60 Hz); 3.18 (m, 1H, CH_2); 2.91 (m, 1H, CH_2); 2.77 (m, 1H, CH_2); 2.58 (m, 1H, CH_2); 1.77 (NH); 1.24–1.6 (m, 20H, 10 CH_2); 0.82–0.86 (m, 6H, 2 CH_3). NMR ^{13}C (CDCl_3): δ 169.40 (C=O); 112.40–112.51; 75.53; 73.37; 72.79; 46.92; 39.73; 27.09; 26.13; 26.36–31.61; 22.53; 13.99. For the epimer (**7l'**): NMR ^1H (CDCl_3): δ 4.63 (d, 1H, H-4, J = 6.08 Hz); 4.48 (d, 1H, H-3, J = 5.84 Hz); 4.45 (s, 1H, H-5, J = 0 Hz); 3.57 (m, 1H, CH_2); 3.38 (m, 1H, CH_2); 2.48 (m, 1H, CH_2); 2.37 (m, 1H, CH_2); 1.77 (NH); 1.24–1.6 (m, 20H, 10 CH_2), 0.71–0.85 (m, 6H, 2 CH_3). NMR ^{13}C (CDCl_3): 170.76 (C=O); 112.40–112.51; 78.27; 77.31; 75.55; 43.34; 39.62; 27.09; 25.73; 22.55–31.83; 14.02. HRMS (m/z) [$\text{M}+\text{H}$] $^+$ calcd for $\text{C}_{21}\text{H}_{41}\text{N}_2\text{O}_3$: 369.3117, found: 369.3112.

4.1.13. *N*-1-(octyl)-3,4-isopropylidendioxy-5-(ortylamino)-pyrrolidin-2-one (**7m**, **7m'**). These compounds were obtained as a mixture of epimers nonseparable by chromatography, colorless syrup. For the epimer (**7m**), NMR ^1H (CDCl_3): δ 4.64–4.66 (m, 2H, H-4, H-3, J = 4.74 Hz); 4.38 (d, 1H, H-5, J = 4.40 Hz); 3.13 (m, 1H, CH_2), 2.91 (m, 1H, CH_2), 2.76 (m, 1H, CH_2), 2.49 (m, 1H, CH_2), 1.72 (NH), 1.24–1.6 (m, 24H, 12 CH_2), 0.85 (m, 6H, 2 CH_3). NMR ^{13}C (CDCl_3): δ 169.38 (C=O); 112.39–112.50; 77.53; 73.31; 72.78; 46.95; 39.72; 27.09; 26.12; 22.60–31.77; 14.04. For the epimer (**7m'**), NMR ^1H (CDCl_3): δ 4.55 (d, 1H, H-4, J = 6.01 Hz); 4.43 (d, 1H, H-3, J = 5.75 Hz); 4.42 (s, 1H, H-5, J = 0 Hz); 3.54 (m, 1H, CH_2); 3.52 (m, 1H, CH_2); 2.37 (m, 1H, CH_2); 2.35 (m, 1H, CH_2); 1.72 (NH); 1.24–1.6 (m, 24H, 12 CH_2); 0.85 (m, 6H, 2 CH_3). NMR ^{13}C (CDCl_3): δ 170.73 (C=O); 112.39–112.50; 78.22; 77.31; 75.54; 43.32; 39.61; 27.09; 25.72; 22.60–31.77; 14.04. HRMS (m/z) [$\text{M}+\text{H}$] $^+$ calcd for $\text{C}_{23}\text{H}_{45}\text{N}_2\text{O}_3$: 397.3430, found: 397.3418.

4.1.14. *N*-1-(nonyl)-3,4-isopropylidendioxy-5-(nonylamino)-pyrrolidin-2-one (**7n**, **7n'**). These compounds were obtained as a mixture of epimers nonseparable by chromatography, colorless syrup. For the epimer (**7n**), NMR ^1H (CDCl_3): δ 4.69–4.72 (m, 2H, H-4, H-3, J = 4.6 Hz); 4.28–4.30 (d, 1H, H-5, J = 4.60 Hz); 3.21 (m, 1H, CH_2); 2.95 (m, 1H, CH_2); 2.79 (m, 1H, CH_2); 2.64 (m, 1H, CH_2), 2.03 (NH); 1.25–1.58 (m, 28H, 14 CH_2); 0.85–0.87 (m, 6H, 2 CH_3). NMR ^{13}C (CDCl_3): δ 169.38 (C=O); 112.39–112.50; 77.53; 73.31; 72.78; 46.95; 39.72; 27.09; 26.12; 22.60–31.77; 14.04. For the epimer (**7n'**): NMR ^1H (CDCl_3): δ 4.58–4.60 (d, 1H, H-4, J = 5.09 Hz); 4.44–4.46 (d, 1H, H-3, J = 5.7 Hz); 4.41 (s, 1H, H-5, J = 0 Hz); 3.55 (m, 1H, CH_2); 3.39 (m, 1H, CH_2); 2.46 (m, 1H, CH_2); 2.39 (m, 1H, CH_2); 2.03 (NH); 1.2–1.58 (m, 28H, 14 CH_2); 0.85–0.87 (m, 6H, 2 CH_3). NMR ^{13}C (CDCl_3): δ 169.38 (C=O); 112.43–112.53; 78.31; 77.33; 75.55; 43.21; 39.65; 26.89; 25.76; 22.63–31.84; 14.09. HRMS (m/z) [$\text{M}+\text{H}$] $^+$ calcd for $\text{C}_{25}\text{H}_{49}\text{N}_2\text{O}_3$: 425.3743, found: 425.3749.

4.1.15. *N*-1-(benzyl)-3,4-O-isopropylidendioxy-5-(benzyl amino)-pyrrolidin-2-one (**7o**, **7o'**). These compounds were obtained as a mixture of epimers separable by chromatography,

white gum. For the epimer (**7o**), $[\alpha]_D^{20} = -3.93$ ($C = 0.63$, MeOH). NMR ^1H (CDCl_3) δ : 7.29 (m, 5H, phenyl protons); 4.89 (d, 1H, $-\text{CH}_2-$, $J = 14.24$ Hz); 4.65 (m, 2H, H-3, H-4); 4.32 (d, 1H, $-\text{CH}_2-$, $J = 14.21$ Hz); 4.22 (d, 1H, H-5, $J = 5.57$ Hz); 3.88–3.84 (d, 1H, $-\text{CH}_2-$, $J = 13.38$ Hz); 3.73–3.80 (d, 1H, $-\text{CH}_2-$, $J = 13.38$ Hz); 2.3 (NH); 1.46 (s, 3H, CH_3); 1.40 (s, 3H, CH_3). NMR ^{13}C (CDCl_3) δ : 168.91 (C=O); 139.81–127.34 (C-aromatic); 112.88; 77.56; 73.03; 71.05; 50.56; 43.19; 27.11; 26.07. For the epimer (**7o'**), $[\alpha]_D^{20} = -1.8$ ($C = 0.4$, MeOH). NMR ^1H (CDCl_3) δ : 7.40 (m, 5H, phenyl protons); 4.76 (d, 1H, $-\text{CH}_2-$, $J = 10.53$ Hz); 4.60 (d, 1H, H-4, $J = 5.84$ Hz); 4.52 (d, 1H, H-3, $J = 6.39$ Hz); 4.32 (s, 1H, H-5, $J = 0$ Hz); 4.24 (d, 2H, $-\text{CH}_2-$, $J = 10.52$ Hz); 3.74–3.69 (d, 1H, $-\text{CH}_2-$, $J = 13.01$ Hz); 3.57–3.62 (d, 1H, $-\text{CH}_2-$, $J = 13.05$ Hz); 2.12 (NH); 1.42 (s, 6H, CH_3). NMR ^{13}C (CDCl_3) δ : 170.91 (C=O); 139.08–127.45 (C-aromatic); 112.64; 78.25; 77.25; 74.99; 47.99; 43.75; 27.80; 25.77. HRMS (m/z) $[\text{M}+\text{H}]^+$ calcd for $\text{C}_{21}\text{H}_{25}\text{N}_2\text{O}_3$ 353.1865, found: 353.1862.

4.1.16. *N*-1-(morpholin-2-yl-ethyl)-3,4-*O*-isopropylidendioxy-5-(morphonyl-2-ylethylamino)pyrrolidin-2-one (**7p**, **7p'**). These compounds were obtained as a mixture of epimers separable by chromatography, yellow gum. For the epimer (**7p**), $[\alpha]_D^{20} = +1.16$ ($C = 0.4$, MeOH), NMR ^1H (CDCl_3) δ : 4.76 (m, 1H, H-4, $J = 4.68$ Hz); 4.67 (d, 1H, H-3, $J = 6.22$ Hz); 4.47 (d, 1H, H-5, $J = 4.61$ Hz); 3.75–3.67 (m, 8H, $-\text{CH}_2-\text{O}$); 3.59 (m, 1H, $-\text{CH}_2-$); 3.42 (m, 1H, $-\text{CH}_2-$); 2.95 (m, 1H, $-\text{CH}_2-$); 2.36–2.67 (m, 13H, $-\text{CH}_2-$); 1.43 (s, 3H, CH_3); 1.39 (s, 3H, CH_3). NMR ^{13}C (CDCl_3) δ : 169.63 (C=O); 112.33; 77.50; 73.16; 66.87; 58.58; 56.19; 53.57; 42.64; 36.10; 27.31; 26.20. For the epimer (**7p'**), $[\alpha]_D^{20} = +15.28$ ($C = 0.31$, MeOH). NMR ^1H (CDCl_3) δ : 4.63 (d, 1H, H-4, $J = 4.75$ Hz); 4.48 (d, 1H, H-3, $J = 6.09$ Hz); 4.31 (s, 1H, H-5); 3.40 (m, 8H, $-\text{CH}_2-\text{O}$); 3.38 (m, 1H, $-\text{CH}_2-$); 3.22 (m, 1H, $-\text{CH}_2-$); 2.79 (m, 1H, $-\text{CH}_2-$); 2.24–2.52 (m, 13H, $-\text{CH}_2-$); 1.29 (s, 3H, CH_3); 1.24 (s, 3H, CH_3). NMR ^{13}C (CDCl_3) δ : 170.82 (C=O); 112.33; 77.37; 73.03; 66.84; 58.16; 56.16; 53.63; 42.48; 36.11; 27.03; 26.13. HRMS (m/z) $[\text{M}+\text{H}]^+$ Calcd for $\text{C}_{19}\text{H}_{35}\text{N}_4\text{O}_5$ 399.2607, found: 399.2616.

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