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Synthesis of *N*-[5-alkoxy-2(5*H*)-furanonyl] amino acid propargyl esters

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Abstract Using K_2CO_3 as a base and CH_3CN as solvent, different kinds of *N*-[5-alkoxy-2(5*H*)-furanonyl] amino acids were reacted with propargyl bromide via substitution reaction at 40 °C to give 16 *N*-[5-alkoxy-2(5*H*)-furanonyl] amino acid propargyl esters with the yields of 44–85% (mostly over 74%). The structures of all newly synthesized compounds were elucidated and confirmed by FTIR, UV, ^{1}H NMR, ^{13}C NMR, MS, and elemental analysis. The rapid, efficient, and brief synthesis of the series propargyl esters with multiple bioactive units, will afford not only a basis for the activity test of potential drug molecules, but also an important synthetic strategy for 2(5*H*)-furanone derivatives with polyfunctional groups.

Keywords Propargyl ester · Synthesis · 2(5*H*)-Furanone · Amino acid · Substitution reaction · Polyfunctional groups

Introduction

As an important structural subunit, 2(5*H*)-furanone is a frequently found in many natural products. At the same time, many compounds containing 2(5*H*)-furanone moiety have been considered as potential insecticides, bactericides, fungicides, antibiotics, anticancer agents, anti-inflammatories, allergy inhibitors, antisoriasis agents, and cyclooxygenase inhibitors [1–11].

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It is well known that natural amino acids are important substances for life activities, and many non-natural amino acids have been used as building blocks in the fields of organic synthesis, functional material, and medicinal chemistry [12–16]. Therefore, different amino acids as an effective functional group are often applied in the design of drug molecules by many researchers [17–22].

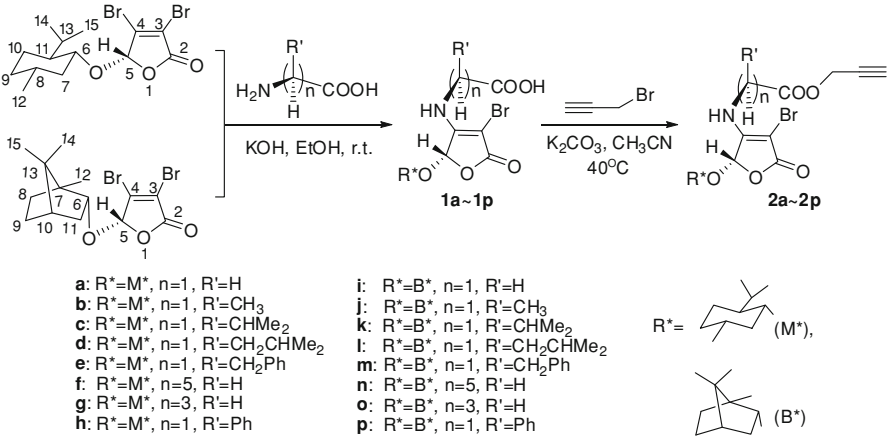
At the same time, many compounds containing terminal alkyne structure are not only important intermediates, especially for the syntheses of different 1,2,3-triazoles [23–27], but also have different bioactivities [28, 29], and even some propargyl ester can be used as a herbicide [30]. However, there is no report on the synthesis of propargyl esters simultaneously containing the structure of 2(5*H*)-furanone and amino acid as the bioactive units.

In this article, in order to design and synthesize novel 2(5*H*)-furanone derivatives with better biological activity, we tried to combine 2(5*H*)-furanone, amino acid and terminal alkyne structure together. Based on our previous studies on the synthesis of *N*-[5-alkoxy-2(5*H*)-furanonyl] amino acids **1a**–**1p** [11, 31], we investigated the reaction of **1** with propargyl bromide, and synthesized a series of *N*-[5-alkoxy-2(5*H*)-furanonyl] amino acid propargyl esters **2a**–**2p** (Scheme 1).

Results and discussion

Condition optimization for substitution reaction

Usually, amino acid propargyl esters were synthesized via condensation using *N*-protected amino acid and propargyl alcohol as materials, and the reported synthetic methods have many drawbacks, such as higher reaction temperature, multi-step reaction processes, more expensive reagents, longer reaction times, especially the tiresome condensing agents for their difficulty in the thorough separation from the products [32–36].



Scheme 1 The synthetic route of the products 2a–2p

There are only a few reports on the synthesis of amino acid propargyl esters using *N*-protected amino acid and propargyl bromide as materials [37]. Referring to the reported method [37], we optimized experimental conditions using the substitution reaction of *N*-[5-menthyloxy-2(5*H*)-furanonyl] 6-aminohexanoic acid **1f** and propargyl bromide as an example (Table 1).

Firstly, the dosages of the base K_2CO_3 were examined (entries 1–3), and the results indicated that 1.5 equiv. K_2CO_3 was the most effective. In this case, when *N*-[5-menthyloxy-2(5*H*)-furanonyl] 6-aminohexanoic acid **1f** (1 mmol) reacted with propargyl bromide (2 mmol) in anhydrous DMF at 50 °C for 24 h, the corresponding yield of amino acid ester **2f** was 70% (entry 2). Once reducing the base dosage, the reaction was slightly incomplete.

While excessive base may cause the hydrolysis of propargyl bromide, which made the yield lowered obviously (entry 3). Even the dosage of the base K_2CO_3 was not increased, the addition of water in the solvent also markedly made the yield lowered (entry 4). This also demonstrated that avoiding the hydrolysis of propargyl bromide was important. However, different anhydrous solvents could give different effects.

THF was not a good solvent for the reaction (entry 5). When the reaction was carried out in DMF or CH_3CN, the result was satisfied. Even in CH_3CN, the reaction time could be shortened from 24 to 8 h (entry 7).

The effect of temperature on the reaction was also significant (entries 7–10). Especially the reaction hardly produced any target product at room temperature after 8 h (entry 8). While the reactant mixture was heated at 40 °C, the product **2f** was obtained in 74% yield (entry 9), and the results showed that at the higher temperature, the yield improved no longer (entries 7 and 10).

Thus, when choosing 1.0 equiv. *N*-[5-menthyloxy-2(5*H*)-furanonyl] 6-aminohexanoic acid **1f** and 2.0 equiv. propargyl bromide as the model substrates, the optimized reaction conditions could be summarized in the following: 40 °C, 8 h 1.5 equiv. K_2CO_3 as the base, and anhydrous CH_3CN as the solvent.

Table 1 Condition optimization for substitution reaction of *N*-[5-menthyloxy-2(5*H*)-furanonyl] 6-aminohexanoic acid **1f** with propargyl bromide

Entry	K_2CO_3 (equiv.)	Solvent	Temp. (°C)	Time (h)	Yield (%)
1	1.0	DMF	50	24	69
2	1.5	DMF	50	24	70
3	2.0	DMF	50	24	60
4	1.5	DMF/H_2O (2/1)	50	24	17
5	1.5	THF	50	24	30
6	1.5	CH_3CN	50	24	73
7	1.5	CH_3CN	50	8	73
8	1.5	CH_3CN	r.t.	8	Trace
9	1.5	CH_3CN	40	8	74
10	1.5	CH_3CN	60	8	73

Influences of different amino acid material on the yield

Under the optimized conditions, the reactions of a series of *N*-[2(5*H*)-furanonyl] amino acids derived from various natural or unnatural amino acids were examined. The results showed that whether the main chain of amino acid was long or short, and whether the side chain of amino acid was alkyl or aryl, the reactions proceeded smoothly.

In most cases, the yield was higher than (or equal to) 74%. However, the yields of the compounds **2h** and **2p** were relatively lower (44–52%). This may be related to the larger steric hindrance of phenyl in *N*-[5-alkoxy-2(5*H*)-furanonyl] (*S*)-phenylglycine (**1h**, **1p**), which caused the normal reaction slower and made the side-reaction more obvious.

Structure characterization of the new compounds

In the IR spectra, the N–H groups show stretching absorption in the region of 3240–3387 cm^{-1}, while unsaturated C–H groups of alkynes show stretching absorption in the region of 3233–3311 cm^{-1}. The weak C≡C stretching band of alkynes occurs in the region of 2127–2133 cm^{-1} and C–H bending vibration of alkynes leads to an absorption in the 639–691 cm^{-1} region.

At the same time, the strong C=O stretching band appears at 1732–1758 cm^{-1}, and the C=C stretching band occurs in 1632–1654 cm^{-1} region. The existence of C=O and C=C groups was also proved by other test method. In the UV spectra, there are strong absorption peaks at 267–274 nm region caused by $\pi \rightarrow \pi^*$ transition of C=C–C=O conjugated system in 2(5*H*)-furanone ring.

In ^{1}H NMR, there is a triplet at 2.54–2.59 ppm, which is the characteristic peak of C≡CH proton. Furthermore, there is a singlet at 5.71–6.80 ppm from 5-H of 2(5*H*)-furanone. Combined with other structural characterizations, it could be seen that the furanone ring was not opened under the above experimental conditions.

Therefore, these characterization not only proved the structure of all newly synthesized compounds was right as expected, also showed that the concise and effective synthetic method for propargyl esters simultaneously containing the structure of 2(5*H*)-furanone and amino acid as the bioactive units had wide applicability and strong tolerance for different sensitive groups.

Conclusions

In summary, a series of *N*-[5-alkoxy-2(5*H*)-furanonyl] amino acid propargyl esters have been efficiently synthesized under very mild reaction conditions. The target compounds possessing multiple bioactive moieties will afford a basis for the activity test of potential drug molecules. Terminal alkynes are widely used in click reaction [23–27], Glaser coupling reaction [38–41], Sonogashira reaction [42–45], dimerization [46], even different kinds of addition reactions to C–O and C–N bonds [47–50]. Thus, the obtained propargyl esters are also important intermediates for more 2(5*H*)-furanone derivatives with polyfunctional groups.

Experimental

General

All the melting points were determined on an X-5 digital melting points apparatus and were uncorrected. Infrared spectra were recorded on a Bruker Vector 33 FT-IR instrument by liquid film method in the absorption range of 4000–400 cm^{-1}. ^{1}H and ^{13}C NMR spectra were obtained in $CDCl_3$ on a Varian DRX-400 MHz spectrometer and tetramethylsilane (TMS) was used as an internal standard. UV absorption peaks were measured by Shimazu UV-2550 ultraviolet absorption detector with dichloromethane as a solvent. Optical rotations were determined with an Autopol IV polarimeter in C_2H_5OH in a 10 cm cell. Elemental analysis was performed on a Thermo FlashEA TM 112 elemental analyzer. The mass spectra (MS) were recorded on Thermo LCQ DECA XP MAX mass spectrometer.

All reagents and solvents were commercially available and were used as received. Using furfural, natural menthol, borneol, and amino acid as starting materials, the intermediate *N*-[5-alkoxy-2(5*H*)-furanonyl] amino acids **1a**–**1p** were prepared according to the literature [11, 31].

Typical procedure for the synthesis of target compounds **2a**–**2p**

A flame-dried 25-mL round-bottomed flask was charged with 1 mmol *N*-[5-alkoxy-2(5*H*)-furanonyl] amino acid and 1.5 mmol anhydrous potassium carbonate in CH_3CN (5 mL). The resulting suspension was stirred for 30 min under an atmosphere of nitrogen. Propargyl bromide (80% in toluene, 2 mmol) was added and the reaction was stirred for 8 h at 40 °C. The resulting mixture was diluted with water (5 mL), and then extracted with ethyl acetate (10 mL × 2). The combined organic layers were dried with magnesium sulfate, and concentrated under vacuum to give a crude product, which was purified by column chromatography on silica gel with gradient eluent of mixtures of petroleum ether and ethyl acetate to afford the sample **2a**–**2p** for analysis.

*N-[3-Bromo-5-(S)-menthoxy-2(5H)-furanonyl] glycine propargyl ester (product **2a**)*

Yellow solid, yield 58%; m.p. 109.8–111.3 °C; $[\alpha]_D^{20} = +59.54°$ (*c* 0.084, CH_3CH_2OH); UV–Vis (CH_2Cl_2) λ_{max}: 267 nm; ^{1}H NMR (400 MHz, $CDCl_3$-TMS) δ: 0.77–0.87 (3H, m, CH_3-12), 0.86–1.02 (7H, m, CH-13, CH_3-14, CH_3-15), 1.01–1.23 (2H, m, CH_2-9), 1.30–1.48 (2H, m, CH-8, CH-11), 1.58–1.74 (2H, m, CH_2-10), 2.08–2.29 (2H, m, CH_2-7), 2.57 (1H, s, CH-21), 3.50–3.65 (1H, m, CH-6), 4.41 (2H, s, CH_2-17), 4.83 (2H, s, CH_2-19), 5.30 (1H, s, NH-16), 5.76 (1H, s, CH-5); ^{13}C NMR (100 MHz, $CDCl_3$-TMS) δ: 15.7, 21.0, 22.1, 22.7, 25.5, 31.5, 33.9, 42.2, 44.5, 48.0, 53.3, 76.0, 76.5, 78.5, 82.2, 98.9, 158.5, 167.7, 168.3; IR (film) *v*: 3377, 3294, 2955, 2927, 2870, 2131, 1758, 1650, 1330, 1189, 1128, 940, 691, 633; ESI-MS *m*/*z* (%): 428 ($[M+H]^+$, 71.5), 450 ($[M+Na]^+$, 90.3). Anal. Calcd for $C_{19}H_{26}BrNO_5$: C 53.28, H 6.12, N 3.27; Found: C 53.38, H 6.40, N 3.49.

*N-[3-Bromo-5-(S)-menthoxy-2(5H)-furanonyl] (S)-alanine propargyl ester (product **2b**)*

Yellow solid, yield 85%, m.p. 106.1–107.4 °C; $[\alpha]_D^{20} = +151.80°$ (*c* 0.057, CH_3CH_2OH); UV–Vis (CH_2Cl_2) λ_{max}: 268 nm; ^{1}H NMR (400 MHz, $CDCl_3$-TMS) δ: 0.86 (3H, d, $J = 6.8$ Hz, CH_3-12), 0.88–1.00 (7H, m, CH-13, CH_3-14, CH_3-15), 1.01–1.18 (2H, m, CH_2-9), 1.33–1.47 (2H, m, CH-8, CH-11), 1.57 (3H, d, $J = 7.2$ Hz, CH_3-22), 1.63–1.74 (2H, m, CH_2-10), 2.08–2.30 (2H, m, CH_2-7), 2.57 (1H, s, CH-21), 3.54–3.65 (1H, ddd, $J_1 = 3.6$ Hz, $J_2 = 4.0$ Hz, $J_3 = 4.0$ Hz, CH-6), 4.80 (2H, d, $J = 2.0$ Hz, CH_2-19), 4.87 (1H, b, NH-16), 5.30–5.41 (1H, m, CH-17), 5.75 (1H, s, CH-5); ^{13}C NMR (100 MHz, $CDCl_3$-TMS) δ: 15.9, 20.5, 20.9, 22.1, 23.0, 26.0, 31.6, 33.9, 42.3, 48.0, 50.7, 53.4, 76.0, 76.5, 82.8, 91.3, 99.2, 156.9, 167.5, 171.3; IR (film) *v*: 3383, 3289, 2955, 2928, 2871, 2129, 1755, 1652, 1328, 1197, 1134, 960, 674, 633; ESI-MS *m/z* (%): 442 ($[M+H]^+$, 62.2), 464 ($[M+Na]^+$, 83.0). Anal. Calcd for $C_{20}H_{28}BrNO_5$: C 54.30, H 6.38, N 3.17; Found: C 54.59, H 6.60, N 3.41.

*N-[3-Bromo-5-(S)-menthoxy-2(5H)-furanonyl] (S)-valine propargyl ester (product **2c**)*

Yellow solid, yield 84%, m.p. 76.2–77.8 °C; $[\alpha]_D^{20} = -160.03°$ (*c* 0.060, CH_3CH_2OH); UV–Vis (CH_2Cl_2) λ_{max}: 269 nm; ^{1}H NMR (400 MHz, $CDCl_3$-TMS) δ: 0.87 (3H, d, $J = 6.8$ Hz, CH_3-12), 0.88–0.98 (7H, m, CH-13, CH_3-14, CH_3-15), 0.99–1.28 (8H, m, CH_2-9, CH_3-23, CH_3-24), 1.32–1.49 (2H, m, CH-8, CH-11), 1.60–1.76 (2H, m, CH_2-10), 2.09–2.38 (3H, m, CH_2-7, CH-22), 2.56 (1H, s, CH-21), 3.53–3.64 (1H, ddd, $J_1 = 3.6$ Hz, $J_2 = 4.0$ Hz, $J_3 = 4.0$ Hz, CH-6), 4.60–4.97 (3H, m, NH-16, CH_2-19), 5.25 (1H, d, $J = 9.2$ Hz, CH-17), 5.73 (1H, s, CH-5); ^{13}C NMR (100 MHz, $CDCl_3$-TMS) δ: 15.7, 17.3, 17.9, 20.9, 22.1, 22.8, 25.9, 31.6, 32.4, 33.9, 42.3, 47.9, 53.0, 59.7, 75.9, 76.6, 82.9, 90.6, 99.5, 157.3, 167.6, 170.0; IR (film) *v*: 3384, 3310, 2957, 2930, 2872, 2129, 1751, 1651, 1329, 1190, 1134, 957, 673, 634; ESI-MS *m/z* (%): 470 ($[M+H]^+$, 74.1), 492 ($[M+Na]^+$, 80.4). Anal. Calcd for $C_{22}H_{32}BrNO_5$: C 56.17, H 6.86, N 2.98; Found: C 56.40, H 7.05, N 3.16.

*N-[3-Bromo-5-(S)-menthoxy-2(5H)-furanonyl] (S)-leucine propargyl ester (product **2d**)*

Yellow solid, yield 79%, m.p. 124.6–126.0 °C; $[\alpha]_D^{20} = -89.62°$ (*c* 0.051, CH_3CH_2OH); UV–Vis (CH_2Cl_2) λ_{max}: 267 nm; ^{1}H NMR (400 MHz, $CDCl_3$-TMS) δ: 0.85 (3H, d, $J = 7.2$ Hz, CH_3-12), 0.86–0.97 (7H, m, CH-13, CH_3-14, CH_3-15), 0.98–1.29 (8H, m, CH_2-9, CH_3-24, CH_3-25), 1.31–1.49 (2H, m, CH-8, CH-11), 1.58–1.86 (5H, m, CH_2-10, CH_2-22, CH-23), 2.06–2.28 (2H, m, CH_2-7), 2.55 (1H, t, $J = 2.4$ Hz, CH-21), 3.53–3.66 (1H, ddd, $J_1 = 3.2$ Hz, $J_2 = 3.6$ Hz, $J_3 = 3.6$ Hz, CH-6), 4.62–4.93 (3H, m, NH-16, CH_2-19), 4.97–5.12 (1H, m, CH-17), 5.74 (1H, s, CH-5); ^{13}C NMR (100 MHz, $CDCl_3$-TMS) δ: 15.9, 20.9, 22.0, 22.1, 22.6, 22.8, 24.7, 25.9, 31.6, 33.9, 42.3, 42.4, 47.9, 53.1, 54.1, 75.9, 76.6, 82.6,

91.3, 99.3, 157.5, 167.5, 171.2; IR (film) *v*: 3311, 2957, 2929, 2871, 2129, 1754, 1651, 1331, 1185, 1130, 962, 671, 508; ESI-MS *m/z* (%): 484 ($[M+H]^+$, 89.3), 506 ($[M+Na]^+$, 90.2). Anal. Calcd for $C_{23}H_{34}BrNO_5$: C 57.03, H 7.07, N 2.89; Found: C 57.30, H 7.36, N 3.15.

*N-[3-Bromo-5-(S)-menthoxy-2(5H)-furanonyl] (S)-phenylalanine propargyl ester (product **2e**)*

Yellow solid, yield 83%, m.p. 99.8–101.5 °C; $[\alpha]_D^{20} = +75.04°$ (*c* 0.061, CH_3CH_2OH); UV–Vis (CH_2Cl_2) λ_{max}: 269 nm; ^1H NMR (400 MHz, $CDCl_3$-TMS) δ: 0.74 (3H, d, $J = 4.0$ Hz, CH_3-12), 0.78–0.94 (7H, m, CH-13, CH_3-14, CH_3-15), 0.95–1.19 (2H, m, CH_2-9), 1.26–1.44 (2H, m, CH-8, CH-11), 1.56–1.71 (2H, m, CH_2-10), 1.86–2.23 (2H, m, CH_2-7), 2.58 (1H, s, CH-21), 3.03–3.31 (2H, m, CH_2-22), 3.37–3.49 (1H, m, CH-6), 4.79 (2H, s, CH_2-19), 4.87–5.46 (3H, m, CH-5, NH-16, CH-17), 7.00–7.20 (2H, m, Ar–H-24, 28), 7.21–7.44 (3H, m, Ar–H-25, 26, 27); ^{13}C NMR (100 MHz, $CDCl_3$-TMS) δ: 15.5, 21.1, 22.1, 22.6, 25.5, 31.6, 33.9, 40.1, 42.3, 47.9, 53.3, 56.1, 76.2, 76.4, 83.0, 99.3, 105.0, 127.8, 128.9, 129.6, 134.1, 157.0, 167.4, 169.6; IR (film) *v*: 3386, 3294, 3062, 3024, 2955, 2925, 2876, 2127, 1758, 1654, 1524, 1454, 1327, 1182, 1116, 955, 744, 702, 683; ESI-MS *m/z* (%): 518 ($[M+H]^+$, 61.6), 540 ($[M+Na]^+$, 100.0). Anal. Calcd for $C_{26}H_{32}BrNO_5$: C 60.23, H 6.22, N 2.70; Found: C 59.96, H 6.44, N 2.99.

*N-[3-Bromo-5-(S)-menthoxy-2(5H)-furanonyl] 6-aminohexanoic acid propargyl ester (product **2f**)*

Yellow solid, yield 74%, m.p. 122.5–124.0 °C; $[\alpha]_D^{20} = +141.20°$ (*c* 0.114, CH_3CH_2OH); UV–Vis (CH_2Cl_2) λ_{max}: 273 nm; ^1H NMR (400 MHz, $CDCl_3$-TMS) δ: 0.81 (3H, d, $J = 6.8$ Hz, CH_3-12), 0.84–0.98 (7H, m, CH-13, CH_3-14, CH_3-15), 0.99–1.19 (2H, m, CH_2-9), 1.30–1.48 (4H, m, CH-8, CH-11, CH_2-19), 1.60–1.78 (6H, m, CH_2-10, CH_2-18, CH_2-20), 2.09–2.29 (2H, m, CH_2-7), 2.40 (2H, t, $J = 7.2$ Hz, CH_2-21), 2.50 (1H, s, CH-25), 3.40–3.53 (2H, m, CH_2-17), 3.54–3.63 (1H, ddd, $J_1 = 3.6$ Hz, $J_2 = 3.6$ Hz, $J_3 = 4.0$ Hz, CH-6), 4.69 (2H, d, $J = 2.0$ Hz, CH_2-23), 4.84–4.99 (1H, m, NH-16), 5.75 (1H, s, CH-5); ^{13}C NMR (100 MHz, $CDCl_3$-TMS) δ: 15.9, 21.1, 22.1, 22.8, 24.2, 25.5, 25.8, 30.3, 31.6, 33.6, 33.9, 42.4, 43.6, 48.1, 51.9, 74.9, 77.6, 81.8, 89.4, 98.5, 159.3, 167.8, 172.5; IR (film) *v*: 3241, 2952, 2917, 2868, 2131, 1733, 1649, 1315, 1154, 1124, 937, 675, 630; ESI-MS *m/z* (%): 484 ($[M+H]^+$, 31.0), 506 ($[M+Na]^+$, 100.0). Anal. Calcd for $C_{23}H_{34}BrNO_5$: C 57.03, H 7.07, N 2.89; Found: C 56.91, H 7.30, N 3.00.

*N-[3-Bromo-5-(S)-menthoxy-2(5H)-furanonyl] 4-aminobutyric acid propargyl ester (product **2g**)*

Yellowish solid, yield 69%, m.p. 92.9–94.5 °C; $[\alpha]_D^{20} = +68.21°$ (*c* 0.029, CH_3CH_2OH); UV–Vis (CH_2Cl_2) λ_{max}: 274 nm; ^1H NMR (400 MHz, $CDCl_3$-TMS) δ: 0.81 (3H, d, $J = 6.8$ Hz, CH_3-12), 0.83–0.99 (7H, m, CH-13, CH_3-14, CH_3-15), 1.00–1.20 (2H, m, CH_2-9), 1.29–1.46 (2H, m, CH-8, CH-11), 1.59–1.75

(2H, m, CH_2-10), 1.92–2.07 (2H, m, CH_2-18), 2.09–2.30 (2H, m, CH_2-7), 2.32–2.64 (3H, m, CH_2-19, CH-23), 3.45–3.73 (3H, m, CH-6, CH_2-17), 4.71 (2H, d, $J = 2.0$ Hz, CH_2-21), 5.14–5.29 (1H, m, NH-16), 5.77 (1H, s, CH-5); ^{13}C NMR (100 MHz, $CDCl_3$-TMS) δ: 15.8, 21.1, 22.1, 22.8, 25.3, 25.5, 30.8, 31.6, 33.9, 42.4, 43.1, 48.1, 52.2, 75.2, 77.3, 81.8, 88.1, 98.5, 159.4, 167.9, 172.2; IR (film) *v*: 3277, 3233, 2953, 2926, 2869, 2129, 1733, 1647, 1316, 1162, 1125, 955, 686, 639; ESI-MS *m/z* (%): 456 ($[M+H]^+$, 74.0), 478 ($[M+Na]^+$, 75.6). Anal. Calcd for $C_{21}H_{30}BrNO_5$: C 55.27, H 6.63, N 3.07; Found: C 55.33, H 6.50, N 3.12.

*N-[3-Bromo-5-(S)-menthoxy-2(5H)-furanonyl] (S)-phenylglycine propargyl ester (product **2h**)*

Yellow liquid, yield 52%; $[\alpha]_D^{20} = 111.5°$ (*c* 0.026, CH_3CH_2OH); UV–Vis (CH_2Cl_2) λ_{max}: 270 nm; ^{1}H NMR (400 MHz, $CDCl_3$-TMS) δ: 0.83–0.88 (3H, m, CH_3-12), 0.89–1.01 (7H, m, CH-13, CH_3-14, CH_3-15), 1.03–1.18 (2H, m, CH_2-9), 1.35–1.47 (2H, m, CH-8, CH-11), 1.62–1.74 (2H, m, CH_2-10), 2.12–2.31 (2H, m, CH_2-7), 2.45–2.58 (1H, m, CH-21), 3.51–3.68 (1H, m, CH-6), 4.62–4.88 (2H, m, CH_2-19), 5.58–5.92 (3H, m, CH-5, NH-16, CH-17), 7.30–7.40 (2H, m, Ar–H-23, 27), 7.39–7.51 (3H, m, Ar–H-24, 25, 26); ^{13}C NMR (100 MHz, $CDCl_3$-TMS) δ: 15.9, 21.0, 22.1, 22.8, 25.8, 31.6, 33.9, 42.1, 48.0, 53.8, 58.5, 72.8, 76.1, 76.2, 83.1, 100.7, 126.9, 129.3, 129.4, 135.4, 155.1, 167.5, 169.4; IR (film) *v*: 3373, 3290, 3069, 2955, 2929, 2867, 2129, 1754, 1654, 1514, 1455, 1318, 1167, 1123, 960, 746, 696, 517; ESI-MS *m/z* (%): 504 ($[M+H]^+$, 56.0), 526 ($[M+Na]^+$, 95.6). Anal. Calcd for $C_{25}H_{30}BrNO_5$: C 59.53, H 5.99, N 2.78; Found: C 59.46, H 6.05, N 2.71.

*N-[3-Bromo-5-(S)-bornyloxy-2(5H)-furanonyl] glycine propargyl ester (product **2i**)*

Yellow solid, yield 63%, m.p. 152.0–152.5 °C; $[\alpha]_D^{20} = +101.25°$ (*c* 0.053, CH_3CH_2OH); UV–Vis (CH_2Cl_2) λ_{max}: 268 nm; ^{1}H NMR (400 MHz, $CDCl_3$-TMS) δ: 0.86 (6H, s, CH_3-14, CH_3-15), 0.91 (3H, s, CH_3-12), 1.18–1.36 (4H, m, CH_2-8, CH_2-9), 1.65–1.75 (2H, m, CH_2-11), 2.22–2.32 (1H, m, CH-10), 2.56 (1H, t, $J = 2.4$ Hz, CH-21), 3.94–4.03 (1H, m, CH-6), 4.36 (2H, s, CH_2-17), 4.82 (2H, t, $J = 2.0$ Hz, CH_2-19), 5.39–5.48 (1H, m, NH-16), 5.76 (1H, s, CH-5); ^{13}C NMR (100 MHz, $CDCl_3$-TMS) δ: 13.9, 18.8, 19.6, 26.5, 27.9, 37.0, 44.6, 44.8, 47.6, 49.4, 53.2, 76.0, 76.6, 77.4, 87.8, 99.4, 158.9, 167.6, 168.5; IR (film) *v*: 3365, 3294, 2953, 2878, 2133, 1758, 1649, 1322, 1185, 1132, 943, 682, 628; ESI-MS *m/z* (%): 426 ($[M+H]^+$, 32.5), 448 ($[M+Na]^+$, 100.0). Anal. Calcd for $C_{19}H_{24}BrNO_5$: C 53.53, H 5.67, N 3.29; Found: C 53.36, H 5.71, N 3.50.

*N-[3-Bromo-5-(S)-bornyloxy-2(5H)-furanonyl] (S)-alanine propargyl ester (product **2j**)*

Yellow solid, yield 74%, m.p. 120.9–122.3 °C; $[\alpha]_D^{20} = 54.50°$ (*c* 0.059, CH_3CH_2OH); UV–Vis (CH_2Cl_2) λ_{max}: 267 nm; ^{1}H NMR (400 MHz, $CDCl_3$-TMS) δ: 0.86 (6H, s, CH_3-14, CH_3-15), 0.92 (3H, s, CH_3-12), 1.21–1.32 (4H, m, CH_2-8, CH_2-9), 1.58 (3H, d, $J = 7.2$ Hz, CH_3-22), 1.63–1.76 (2H, m, CH_2-11),

2.20–2.31 (1H, m, CH-10), 2.56 (1H, t, $J = 2.4$ Hz, CH-21), 3.98 (1H, d, $J = 8.8$ Hz, CH-6), 4.69–4.88 (3H, m, NH-16, CH_2-19), 5.39–5.51 (1H, m, CH-17), 5.76 (1H, s, CH-5); ^{13}C NMR (100 MHz, $CDCl_3$-TMS) δ: 13.9, 18.8, 19.6, 20.1, 26.5, 27.9, 37.1, 44.8, 47.6, 49.4, 51.1, 53.3, 68.8, 76.0, 76.5, 87.7, 99.3, 157.5, 167.3, 171.3; IR (film) *v*: 3289, 2954, 2878, 2131, 1755, 1652, 1330, 1197, 1137, 958, 687, 642; ESI-MS *m/z* (%): 440 ($[M+H]^+$, 74.8), 462 ($[M+Na]^+$, 65.0). Anal. Calcd for $C_{20}H_{26}BrNO_5$: C 54.55, H 5.95, N 3.18; Found: C 54.76, H 5.80, N 3.01.

*N-[3-Bromo-5-(S)-bornyloxy-2(5H)-furanonyl] (S)-valine propargyl ester (product **2k**)*

Yellow solid, yield 74%, m.p. 118.9–120.4 °C; $[\alpha]_D^{20} = -151.2°$ (*c* 0.095, CH_3CH_2OH); UV–Vis (CH_2Cl_2) λ_{max}: 268 nm; ^{1}H NMR (400 MHz, $CDCl_3$-TMS) δ: 0.87 (6H, s, CH_3-14, CH_3-15), 0.94 (3H, s, CH_3-12), 1.03 (6H, d, $J = 6.8$ Hz, CH_3-23, CH_3-24), 1.21–1.32 (4H, m, CH_2-8, CH_2-9), 1.63–1.76 (2H, m, CH_2-11), 1.77–1.89 (1H, m, CH-22), 2.20–2.34 (1H, m, CH-10), 2.55 (1H, s, CH-21), 3.99 (1H, d, $J = 9.6$ Hz, CH-6), 4.65–4.92 (3H, m, NH-16, CH_2-19), 5.30 (1H, d, $J = 10.0$ Hz, CH-17), 5.72 (1H, s, CH-5); ^{13}C NMR (100 MHz, $CDCl_3$-TMS) δ: 13.9, 17.4, 18.2, 18.8, 19.6, 26.5, 27.9, 32.3, 37.0, 44.8, 47.5, 49.4, 52.9, 60.3, 68.8, 75.9, 76.6, 88.0, 99.5, 157.9, 167.2, 170.0; IR (film) *v*: 3388, 3298, 2957, 2878, 2129, 1753, 1654, 1332, 1191, 1135, 957, 675, 581; ESI-MS *m/z* (%): 468 ($[M+H]^+$, 75.7), 490 ($[M+Na]^+$, 82.6). Anal. Calcd for $C_{22}H_{30}BrNO_5$: C 56.41, H 6.46, N 2.99; Found: C 56.20, H 6.61, N 3.28.

*N-[3-Bromo-5-(S)-bornyloxy-2(5H)-furanonyl] (S)-leucine propargyl ester (product **2l**)*

Yellow liquid, yield 82%; $[\alpha]_D^{20} = -102.3°$ (*c* 0.059, CH_3CH_2OH); UV–Vis (CH_2Cl_2) λ_{max}: 269 nm; ^{1}H NMR (400 MHz, $CDCl_3$-TMS) δ: 0.86 (6H, s, CH_3-14, CH_3-15), 0.92 (3H, s, CH_3-12), 0.94–1.08 (6H, m, CH_3-24, CH_3-25), 1.12–1.37 (4H, m, CH_2-8, CH_2-9), 1.64–1.81 (5H, m, CH_2-11, CH_2-22, CH-23), 2.21–2.33 (1H, m, CH-10), 2.55 (1H, t, $J = 2.4$ Hz, CH-21), 3.98 (1H, d, $J = 8.4$ Hz, CH-6), 4.60–4.91 (3H, m, NH-16, CH_2-19), 5.14–5.28 (1H, m, CH-17), 5.72 (1H, s, CH-5); ^{13}C NMR (100 MHz, $CDCl_3$-TMS) δ: 13.9, 18.8, 19.6, 21.9, 22.8, 24.7, 26.4, 27.9, 37.1, 42.4, 44.8, 47.6, 49.4, 53.1, 54.4, 75.9, 76.6, 77.3, 88.1, 99.4, 158.0, 167.2, 171.2; IR (film), *v*: 3293, 2956, 2877, 2133, 1754, 1648, 1334, 1146, 958, 676, 580; ESI-Ms, *m/z* (%): 482 ($[M+H]^+$, 60.4), 504 ($[M+Na]^+$, 82.1). Anal. Calcd for $C_{23}H_{32}BrNO_5$: C 57.26, H 6.69, N 2.90; Found: C 57.49, H 6.91, N 3.04.

*N-[3-Bromo-5-(S)-bornyloxy-2(5H)-furanonyl] (S)-phenylalanine propargyl ester (product **2m**)*

Yellow liquid, yield 80%; $[\alpha]_D^{20} = 83.78°$ (*c* 0.025, CH_3CH_2OH); UV–Vis (CH_2Cl_2) λ_{max}: 271 nm; ^{1}H NMR (400 MHz, $CDCl_3$-TMS) δ: 0.77–0.94 (9H, m, CH_3-12, CH_3-14, CH_3-15), 1.17–1.29 (4H, m, CH_2-8, CH_2-9), 1.61–1.74 (2H, m, CH_2-11), 2.11–2.26 (1H, m, CH-10), 2.58 (1H, t, $J = 2.4$ Hz, CH-21), 3.06–3.35 (2H, m,

CH_2-22), 3.73–3.88 (1H, m, CH-6), 4.74–4.87 (2H, m, CH_2-19), 4.88–5.54 (3H, m, CH-5, NH-16, CH-17), 7.09–7.23 (2H, m, Ar–H-24, 28), 7.26–7.45 (3H, m, Ar–H-25, 26, 27); ^{13}C NMR (100 MHz, $CDCl_3$-TMS) δ: 14.1, 18.7, 19.6, 26.5, 27.9, 36.9, 40.0, 44.8, 47.6, 49.4, 53.4, 56.8, 72.8, 76.1, 76.4, 88.2, 99.4, 127.9, 129.0, 129.5, 134.5, 157.2, 167.2, 169.7; IR (film) *v*: 3372, 3294, 3064, 3029, 2953, 2881, 2130, 1755, 1651, 1523, 1497, 1455, 1336, 1176, 1131, 958, 745, 703, 640, 502; ESI-MS *m/z* (%): 516 ($[M+H]^+$, 68.1), 538 ($[M+Na]^+$, 75.1). Anal. Calcd for $C_{26}H_{30}BrNO_5$: C 60.47, H 5.86, N 2.71; Found: C 60.35, H 5.71, N 2.90.

*N-[3-Bromo-5-(S)-bornyloxy-2(5H)-furanonyl] 6-aminohexanoic acid propargyl ester (product **2n**)*

Yellow solid, yield 81%, m.p. 93.1–94.5 °C; $[\alpha]_D^{20} = 15.27°$ (*c* 0.055, CH_3CH_2OH); UV–Vis (CH_2Cl_2) λ_{max}: 273 nm; ^{1}H NMR (400 MHz, $CDCl_3$-TMS) δ: 0.86 (6H, s, CH_3-14, CH_3-15), 0.90 (3H, s, CH_3-12), 1.18–1.36 (4H, m, CH_2-8, CH_2-9), 1.38–1.48 (2H, m, CH_2-19), 1.60–1.77 (6H, m, CH_2-11, CH_2-18, CH_2-20), 2.21–2.32 (1H, m, CH-10), 2.40 (2H, t, $J = 7.2$ Hz, CH_2-21), 2.50 (1H, t, $J = 2.4$ Hz, CH-25), 3.38–3.58 (2H, m, CH_2-17), 3.99 (1H, d, $J = 9.2$ Hz, CH-6), 4.69 (2H, d, $J = 2.4$ Hz, CH_2-23), 5.11 (1H, s, NH-16), 5.75 (1H, s, CH-5); ^{13}C NMR (100 MHz, $CDCl_3$-TMS) δ: 14.0, 18.8, 19.6, 24.2, 25.8, 26.6, 28.0, 30.3, 33.6, 37.1, 43.9, 44.8, 47.6, 49.4, 51.9, 67.5, 74.9, 77.6, 87.5, 99.1, 160.0, 167.6, 172.5; IR (film) *v*: 3298, 2951, 2878, 2132, 1747, 1644, 1321, 1151, 1133, 954, 679, 639; ESI-MS *m/z* (%): 482 ($[M+H]^+$, 49.2), 504 ($[M+Na]^+$, 84.1). Anal. Calcd for $C_{23}H_{32}BrNO_5$: C 57.26, H 6.69, N 2.90; Found: C 56.97, H 6.76, N 3.00.

*N-[3-Bromo-5-(S)-bornyloxy-2(5H)-furanonyl] 4-aminobutyric acid propargyl ester (product **2o**)*

Yellow solid, yield 76%, m.p. 140.9–142.1 °C; $[\alpha]_D^{20} = 160.25°$ (*c* 0.059, CH_3CH_2OH); UV–Vis (CH_2Cl_2) λ_{max}: 272 nm; ^{1}H NMR (400 MHz, $CDCl_3$-TMS) δ: 0.86 (3H, s, CH_3-14), 0.87 (3H, s, CH_3-15), 0.90 (3H, s, CH_3-12), 1.16–1.35 (4H, m, CH_2-8, CH_2-9), 1.63–1.76 (2H, m, CH_2-11), 1.95–2.07 (2H, m, CH_2-18), 2.20–2.31 (1H, m, CH-10), 2.43–2.59 (3H, m, CH_2-19, CH-23), 3.46–3.62 (2H, m, CH-17), 3.99 (1H, d, $J = 8.8$ Hz, CH-6), 4.71 (2H, d, $J = 2.4$ Hz, CH_2-21), 5.31 (1H, s, NH-16), 5.75 (1H, s, CH-5); ^{13}C NMR (100 MHz, $CDCl_3$-TMS) δ: 14.0, 18.8, 19.6, 25.3, 26.6, 28.0, 30.9, 37.0, 43.3, 44.8, 47.6, 49.4, 52.3, 67.9, 75.2, 77.3, 87.6, 99.2, 159.5, 167.7, 172.2; IR (film) *v*: 3254, 2957, 2876, 2130, 1734, 1633, 1321, 1163, 1131, 953, 635, 604; ESI-MS *m/z* (%): 454 ($[M+H]^+$, 87.0), 476 ($[M+Na]^+$, 100.0). Anal. Calcd for $C_{21}H_{28}BrNO_5$: C 55.51, H 6.21, N 3.08; Found: C 55.80, H 6.42, N 3.30.

*N-[3-Bromo-5-(S)-bornyloxy-2(5H)-furanonyl] (S)-phenylglycine propargyl ester (product **2p**)*

Yellow liquid, yield 44%; $[\alpha]_D^{20} = 157.6°$ (*c* 0.039, CH_3CH_2OH); UV–Vis (CH_2Cl_2) λ_{max}: 268 nm; ^{1}H NMR (400 MHz, $CDCl_3$-TMS) δ: 0.82–1.02 (9H, m, CH_3-12,

CH_3-14, CH_3-15), 1.20–1.38 (4H, m, CH_2-8, CH_2-9), 1.61–1.80 (2H, m, CH_2-11), 2.12–2.30 (1H, m, CH-10), 2.43–2.57 (1H, m, CH-21), 3.70–4.02 (1H, m, CH-6), 4.62–4.87 (2H, m, CH_2-19), 5.29–5.71 (1H, m, NH-16), 5.73–6.09 (1H, m, CH-17), 6.80 (1H, s, CH-5), 7.32–7.53 (5H, m, Ar–H); ^{13}C NMR (100 MHz, $CDCl_3$-TMS) δ: 14.1, 18.8, 19.6, 26.7, 28.0, 36.9, 44.8, 47.6, 49.5, 54.7, 58.5, 72.8, 76.2, 76.3, 88.1, 101.1, 126.6, 129.0, 129.3, 136.4, 155.4, 169.4, 170.4; IR (film) *v*: 3360, 3295, 3070, 3035, 2953, 2877, 2130, 1758, 1653, 1516, 1453, 1333, 1183, 1132, 962, 745, 696, 659, 559; ESI-MS *m*/*z* (%): 502 ($[M+H]^+$, 22.5), 540 ($[M+K]^+$, 74.3). Anal. Calcd for $C_{25}H_{28}BrNO_5$: C 59.77, H 5.62, N 2.79; Found: C 59.80, H 5.57, N 2.82.

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