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# Convenient access to L-3,4,5-trioxygenated phenylalanine compounds from L-tyrosine

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ABSTRACT

A convenient and efficient synthesis of L-3,4,5-trioxygenated phenylalanine derivatives from L-tyrosine was developed. Dibromo phenylalanine is converted easily to bis-phenol via copper-catalyzed hydroxylation. The synthetic potential of this methodology has been demonstrated by efficient synthesis of L-3,4,5-trimethoxyphenylalanine methyl ester and one key intermediate of Trabectedin.



## 1. Introduction

L-3,4,5-trioxygenated phenylalanine derivatives are important sectors in biologically active molecules. Specifically, L-3,4,5- trimethoxygenated phenylalanine **1a** is one of the most important nonproteinogenic amino acid. For example (Fig. 1), as a new class of covalent ClpXP inhibitors, amino acid phenyl ester **2** [1] shows potent inhibition, as well as stimulation of ClpXP-mediated proteolysis. Tripeptide **3** [2] shows high activity in biochemical and cellular assays as a new class of noncovalent proteasome inhibitors. A series of compounds **4** [3] were reported that have high anti-hepatitis B activity in 2018.

Protected L-3,4,5-trioxygenated phenylalanine **1b** [4] is also a star molecule because it is one key segment in the total synthesis of Trabectedin **5** (Fig. 2), which is the first representative of a marine natural product to receive marketing authorization for the treatment of patients with liposarcoma and leiomyosarcoma that cannot be removed by surgery (unresectable) or is advanced (metastatic) [5].

As of now, many synthetic methods have been developed for the preparation of L-3,4,5-trioxygenated phenylalanine derivatives [1,2,6–10]. Resolution, chiral synthesis and natural amino acid derivatization are three typical approaches (Fig. 3). Natural amino acids are commercially available, cheap and optically pure. Therefore, the development methods for natural amino acid derivatization has attracted the attention of organic chemists. Till now two synthetic routes have been established. In 2001 [9], Deboves et al. reported the conversion of L-serine-derived organozinc reagent to L-3,4,5-trimethoxyphenylalanie ester derivative using organometallic chemistry without loss of stereochemical integrity. Chen group published another synthetic route of L-3,4,5-trioxygenated phenylalanine derivatives from L-tyrosine in 2013 [10]. Oxidative hydroxylation of aryboronic acids as a key step was employed in the synthesis. However, in order to avoid the side effection of active NH group, extra synthetic steps were required. Thus, the amino acid was reduced to amino alcohol and transformed to oxazolidine with 2,2-dimethoxypropane. After bishydroxylation, L-3,4,5-trioxygenated phenylalanine derivatives could be obtained by hydrolysis of oxazolidine **11** and oxidation of amino alcohol to amino acid.

Given the significance of L-3,4,5- trimethoxygenated phenylalanine, we started to explore an easier and more convenient method accessing this motif. Herein we report an efficient synthesis of L-3,4,5-trioxygenated phenylalanine compounds through a key copper-catalyzed hydroxylation from dibromo L–tyrosine derivative directly without amino acid reduction.

## 2. Result discussion

In order to identify the proper precursor for bishydroxylation,

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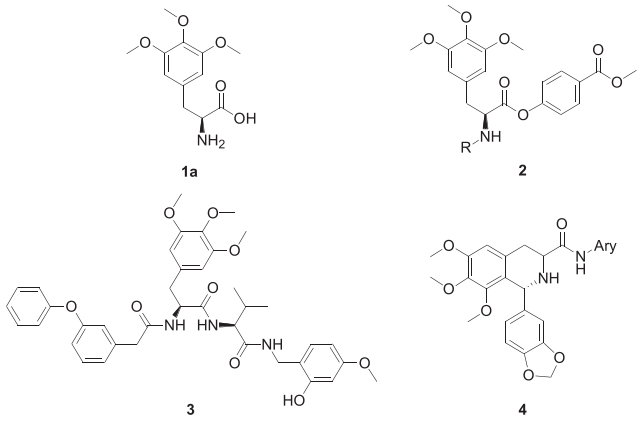



**Fig. 1.** Trimethoxygenated phenylalanine derivatives.

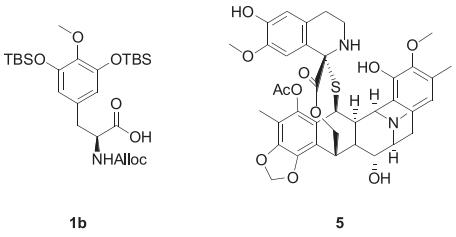



**Fig. 2.** Trabectedin intermediate and Trabectedin.

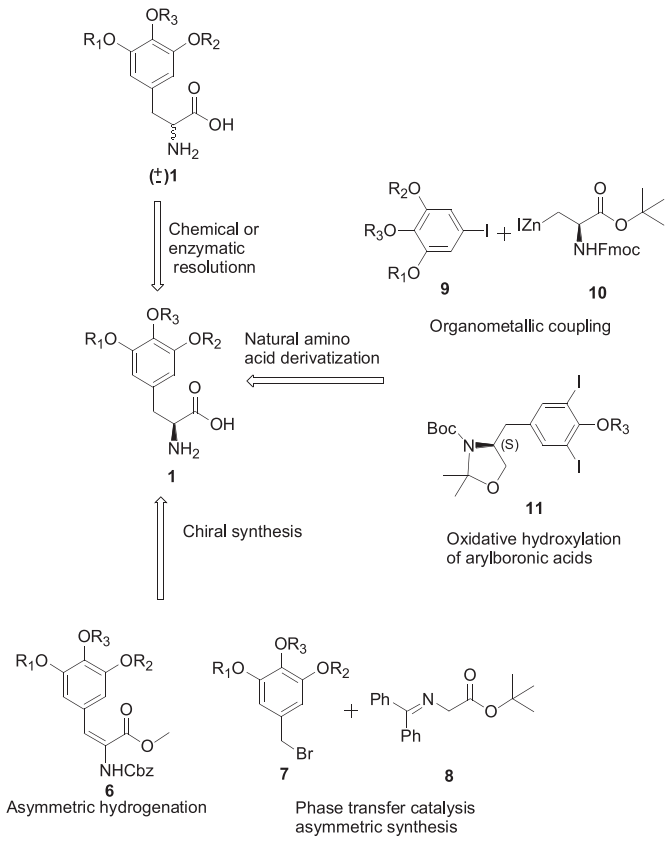



**Fig. 3.** Synthetic methods for L-3,4,5-trioxygenated phenylalanine derivatives.

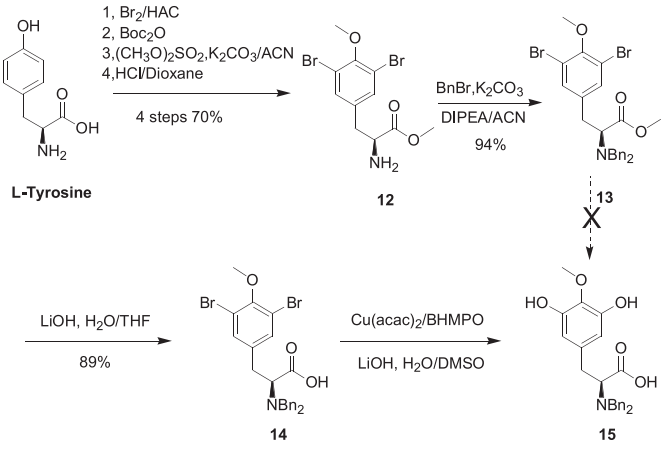
firstly, as outlined in Scheme 1, 3,5-dibromo-O-methyltyrosine ester **12** was prepared from L-tyrosine according to known procedures [10–12]. This 4-step synthesis could be easily scaled up on decagram scale in 70% overall yield.

Dawei Ma's group [13] reported an efficient method on transformation of aryl halides to the phenols via a powerful catalytic system combined by $Cu(acac)_2$ and N,N' -bis(4-hydroxyl-2,6-dimethylphenyl)oxalamide (BHMPO). Considering the condition of hydroxylation by using lithium hydroxide at above 80 °C, dibenzyl group was chosen as amine protection. The protection with benzyl bromide went smoothly in high yield under the condition of $K_2CO_3$ and DIPEA as base in acetonitrile. We then attempted to use this dibenzyl amino ester **13** to run bishydroxylation directly with Ma's method. Unfortunately, the amino ester **13** was only hydroxylized to the amine acid **14** and compound **15** derived from the phenol hydroxylation was not found. Thus, we isolated the amino acid **14** by column chromatography and try the bishydroxylation under the same conditions. To our delight, monohydroxylated product **16** was produced and small amount of bisphenol **15** was detected. Then a systematic optimization of bishydroxylation was investigated. After extensive experiments, increasing the loading of catalyst to 10 mol % and raising the reaction temperature to 110 °C, the conversion of dibromo amino acid **14** to bis-phenol **15** was improved to above 85% and the isolated yield was 77% (Table 1).

With establishment of optimal conditions for the key bishydroxylation of 3,5-dibromo-O-methyltyrosine ester, we started to explore the synthesis of **1a** and **1b**. Firstly, o-methylation of bisphenol **15** and methyl esterification by dimethylsulfate afforded ester **17** in 91% yield. Debenzylation of **17** gave the L-3,4,5- trimethoxyphenylalanine methyl ester **1c** in 96% yield, which is the advanced intermediate one step away from **1a**. On the other hand, protection of bis-phenol **15** with TBSCl gave silyl ether **18**. Debenzylation by hydrogenation of **18** in the presence of a catalytic amount of Pd/C furnished the amino acid **19**. Finally alloc protection to afford the desired amino acid **1b** [14] with high yield (see Scheme 2).

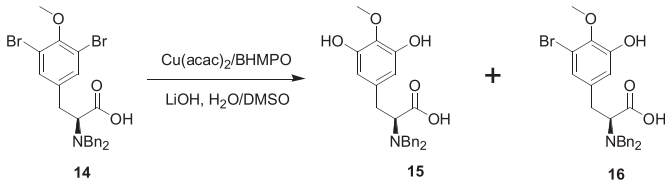
## 3. Conclusions

In summary, a new, convenient and efficient synthesis of protected L-3,4,5-trioxygenated phenylalanine derivatives from commercially available L-tyrosine was developed. The key step is Ma's copper-catalyzed bishydroxylation of dibromide using



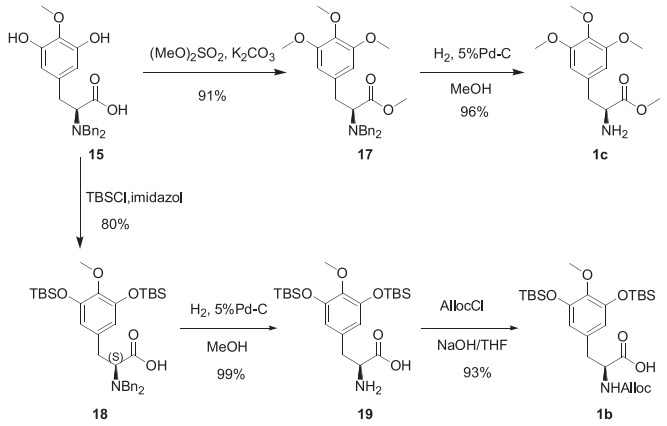

**Scheme 1.** Preparation of bis-phenol **15**.

**Table 1**
Optimization of reaction conditions[a].




| Entry[a] | condition | 14 | 15 | 16 | Other impurities |
|---|---|---|---|---|---|
| 1 | 1%$Cu(acac)_2$, BHMPO, 80 °C, 24 h | 49 | 0.5 | 45 | 5 |
| 2 | 3%$Cu(acac)_2$, BHMPO, 80 °C, 24 h | 17 | 1 | 76 | 6 |
| 3 | 3%$Cu(acac)_2$, BHMPO, 90 °C, 24 h | | 2 | 90 | 8 |
| 4 | 7%$Cu(acac)_2$, BHMPO, 90 °C, 24 h | | 10 | 82 | 8 |
| 5 | 7%$Cu(acac)_2$, BHMPO, 110 °C, 24 h | | 76 | 14 | 10 |
| 6 | 10%$Cu(acac)_2$, BHMPO, 110 °C, 24 h | | 86(77[b]) | 3 | 11 |
| 7 | 20%$Cu(acac)_2$, BHMPO, 110 °C, 24 h | | 86 | 3 | 11 |

[a] Unless specified, all reactions were analyzed by HPLC.
[b] Isolated yield.




**Scheme 2.** Preparation of trimethoxygenated phenylalanine methyl ester **1c** and Trabectedin intermediate **1b**.

BHMPO as the ligand. Two typical L-3,4,5-trioxygenated phenylalanine derivatives **1b** and **1c** have been readily obtained. Notable, the key building block L-trimethoxyphenyl-alanine methyl ester **1c** was prepared in nine steps with 39% overall yield.

## 4. Experimental

All solvents and reagents were obtained from commercial suppliers and used without further purification. Optical rotations were reported as follows: $[a]_D^T$(c g/100 mL, in solvent). $^1H$ and $^{13}C$ NMR spectra were recorded on Varian Mercury plus 400 MHz spectrometer.

### *4.1. (S)-methyl 2-(dibenzylamino)-3-(3,5-dibromo-4-methoxyphenyl)propanoate **13***

To a solution of compound **12** (36.7 g, 100 mmol) in acetonitrile (367 mL) at 25 °C was added benzyl bromide (42.75 g, 250 mmol), DIPEA (12.9 g, 100 mmol) and $K_2CO_3$ (41.4 g, 300 mmol). The reaction mixture was refluxed 24 h. After completion, the reaction mixture was filtered. The filtrate was concentrated and purified by silica gel column chromatography (gradient elution: 1%–5% ethyl acetate in hexane) to give compound **13** (51.4 g, 94%) as an oil.

$[a]_D^{27}$ -27.7 (c 1.31, in MeOH); $^1H$ NMR (400 MHz, $CDCl_3$): $\delta$ (ppm) 7.12–7.29 (m, 12H), 3.90–3.93 (m, 5H), 3.79 (s, 3H), 3.51–3.57 (m, 3H), 2.8–3.0 (m, 2H); $^{13}$CNMR (100 MHz, $CDCl_3$): d (ppm) 172.3, 152.5, 138.7, 137.2, 133.6128.6, 128.4, 127.2, 117.5, 61.7, 60.7, 54.4, 51.4, 34.3.ESIMS 548.0 $[M+H]^+$

### *4.2. (S)- 2-(dibenzylamino)-3-(3,5-dibromo-4-methoxyphenyl) propanoic acid **14***

$LiOH\cdot H_2O$ (8.4 g, 200 mmol) and water (84 mL) was added to the solution of compound **13** (51.0 g, 93 mmol) in THF (168 mL). The solution was allowed to warm to 65–70 °C and stirred overnight. THF was evaporated in vacuo. The pH of the reaction was adjusted to 4–6 by 1 M HCl solution. The mixture was extracted with DCM (400 mL). The organic layer was dried with anhydrous $Na_2SO_4$ and concentrated. The residue was purified by silica gel column chromatography (gradient elution: 5%–30% ethyl acetate in hexane) to afford compound **14** (44.3 g, 89%) as a viscous oil.

$[a]_D^{27}$ -22.9 (c 0.84, in MeOH); $^1H$ NMR (400 MHz, $CDCl_3$): $\delta$ (ppm) 7.14–7.37 (m, 12H), 3.77–3.96 (m, 7H), 3.69 (m, 1H), 3.11 (dd, J = 12, 4 Hz, 1H), 2.98 (dd, J = 12, 4 Hz, 1H); $^{13}$CNMR (100 MHz, $CDCl_3$): d (ppm) 176.7, 152.7, 138.0, 137.0, 133.5128.8, 128.5, 127.5, 117.7, 61.7, 60.7, 54.4, 33.4. ESIMS: 534.0 $[M+H]^+$

### *4.3. (S)-2-(dibenzylamino)-3-(3,5-dihydroxy-4-methoxyphenyl) propanoic acid **15***

To a solution of compound **14** (16.0 g, 30 mmol) in DMSO (80 mL) and water (20 mL) at 25 °C, $LiOH\cdot H_2O$ (6.3 g, 150 mmol), $Cu(acac)_2$ (0.78 g, 3 mmol) and N1,N2-bis(4-hydroxy-2,6-dimethylphenyl)oxalamide (0.98 g, 3 mmol) was added under argon. The reaction mixture was allowed to warm to 110–115 °C and stirred overnight. After the reaction was completed, the mixture was cooled to room temperature and added water (330 mL) and acetic acid (6.0 g, 100 mmol). The crude product (12.5 g) was collected by filtration. Purification by silica gel column chromatography (gradient elution: 15%–50% ethyl acetate in hexane) afforded pure compound **15** (9.4 g, 77%).

$[a]_D^{27}$ -32.8 (c 0.95, in MeOH); $^1H$ NMR (400 MHz, $CDCl_3$): $\delta$ (ppm) 7.21–7.31 (m, 10H), 6.31 (s, 2H), 3.89 (s, 3H), 3.69–3.80 (m, 5H), 3.24 (dd, J = 12, 4 Hz, 1H), 2.87 (dd, J = 12, 4 Hz, 1H); $^{13}$CNMR (100 MHz, $CDCl_3$): d (ppm) 173.2, 149.6, 136.6, 134.0, 133.7, 129.0, 128.6, 127.8, 108.7, 63.7, 60.7, 54.6, 34.0. ESIMS: 408.1 $(M + H)^+$

### 4.4. (S)-3-(3-bromo-5-hydroxy-4-methoxyphenyl)-2-(dibenzylamino)propanoic acid **16**

To a solution of compound **14** (12.8 g, 24 mmol) in DMSO (76.8 mL) and water (19.2 mL) at 25 °C, $LiOH \cdot H_2O$ (4.8 g, 120 mmol), $Cu(acac)_2$ (192 mg, 0.72 mmol) and N1,N2-bis(4-hydroxy-2,6-dimethylphenyl)oxalamide (240 mg, 0.72 mmol) was added under argon. The reaction mixture was allowed to warm to 90–100 °C and stirred overnight. After the reaction was completed, the mixture was cooled to room temperature and added water (288 mL) and acetic acid (4.8 g, 84 mmol). The crude product (9.8 g) was extracted with Ethyl acetate (500 mL). Purification by silica gel column chromatography (gradient elution: 15%–30% ethyl acetate in hexane) afforded pure compound **16** (8.8 g, 78%).

$[a]_D^{27}$ -26.7 (c 0.57, in MeOH); $^1$H NMR (400 MHz, $CDCl_3$): $\delta$ (ppm) 7.22–7.34 (m, 10H), 6.85 (s, 1H), 6.79 (s, 1H), 3.91 (s, 3H), 3.75–3.96 (m, 5H), 3.21 (dd, J = 12, 4 Hz, 1H), 2.96 (dd, J = 12, 4 Hz, 1H); $^{13}$CNMR (100 MHz, $CDCl_3$): d (ppm) 175.0, 149.9, 143.2, 137.0, 136.1, 129.0, 128.6, 127.8, 125.0, 116.4, 115.8, 62.6, 60.1, 54.6, 33.5. ESIMS: 470.1 $(M + H)^+$

### 4.5. (S)-methyl 2-(dibenzylamino)-3-(3,4,5-trimethoxyphenyl) propanoate **17**

Dimethyl sulfate (630 mg, 5.0 mmol) was added to a mixture of **15** (407 mg, 1.0 mmol) and $K_2CO_3$ (700 mg, 5.1 mmol) in acetonitrile (15 mL), and the resulting suspension was allowed to warm to 65–70 °C and stirred for 3 h. Acetonitrile was evaporated in vacuo. The mixture was partitioned with DCM (30 mL) and water (20 mL). The organic layer was dried with anhydrous Na2SO4 and concentrated. The residue was purified by silica gel column chromatography (gradient elution: 5%–20% ethyl acetate in hexane) to afford compound **17** (409 mg, 91%) as an oil.

$[a]_D^{27}$ -54.8 (c 1.12, in MeOH); $^1$H NMR (400 MHz, $CDCl_3$): $\delta$ (ppm) 7.19–7.26 (m, 10H), 6.19 (s, 2H), 3.95 (d, J = 14 Hz, 2H), 3.86 (s, 3H), 3.78 (s, 3H), 3.69 (s, 6H), 3.63 (m, 1H), 3.54 (d, J = 14 Hz, 2H), 3.03 (dd, J = 12, 4 Hz, 1H), 2.88 (dd, J = 12, 4 Hz, 1H); $^{13}$CNMR (100 MHz, $CDCl_3$): d (ppm) 172.8, 152.8, 139.2, 136.2, 134.2, 128.7, 128.1, 127.0, 106.2, 62.5, 60.9, 55.8, 54.4, 51.2, 36.2. ESIMS: 450.2 $(M + H)^+$

### 4.6. (S)-methyl 2-amino-3-(3,4,5-trimethoxyphenyl)propanoate **1c**

A solution of **17** (449 mg, 1.0 mmol) and 10% palladium on activated charcoal (45 mg) in methanol (20 mL) was stirred under 1 atm of hydrogen gas at 45 °C for 6 h. The reaction mixture was gravity filtered, and the filtrate was concentrated to afford amine (260 mg, 96%).

$[a]_D^{27}$ 9.2 (c 1.0, in MeOH); $^1$H NMR (400 MHz, $CDCl_3$): $\delta$ (ppm) 6.39 (s, 2H), 3.80 (s, 6H), 3.78 (s, 3H), 3.70 (s, 3H), 3.77–3.81 (m, 1H), 3.02 (dd, J = 12, 4 Hz, 1H), 2.80 (d, J = 12, 4 Hz, 1H), 2.68 (s, 2H); $^{13}$CNMR (100 MHz, $CDCl_3$): d (ppm) 174.8, 153.2, 136.8, 132.5, 106.1, 60.7, 56.0, 55.6, 54.4, 52.0, 40.8.

### 4.7. (S)-3-(3,5-bis((tert-butyldimethylsilyl)oxy)-4-methoxyphenyl)-2-(dibenzylamino)propanoic acid **18**

To a solution of compound **15** (4.1 g, 10 mmol) in dichloromethane (41 mL) was added TBSCl (6 g, 40 mmol) and imidazole (3.4 g, 50 mmol) and the resulting mixture was stirred at 25 °C for 3 h. The reaction mixture was diluted with dichloromethane (41 mL) and was washed sequentially with saturated aqueous sodium bicarbonate solution (60 mL) and saturated aqueous sodium chloride solution (60 mL). To the organic layer was added trifluoroacetic (2 mL) acid. The reaction mixture was stirred at 25 °C for 0.5 h and then quenched with saturated aqueous sodium bicarbonate solution (60 mL). The organic layer was dried (sodium sulfate) and concentrated, and the residue was purified by flash column chromatography (gradient elution: 5%–30% ethyl acetate in hexane) to afford **18** as an oil (5.1 g, 80%).

$[a]_D^{27}$ -29.0 (c 1.02, in MeOH); $^1$H NMR (400 MHz, $CDCl_3$): $\delta$ (ppm) 7.21–7.33 (m, 10H), 6.38 (s, 2H), 3.73 (s, 3H), 3.60–3.79 (m, 5H), 3.28 (m, 1H), 2.88 (m, 1H), 1.01 (s, 18H), 0.16 (s, 12H); $^{13}$CNMR (100 MHz, $CDCl_3$): d (ppm) 175.7, 149.6, 141.5, 138.0, 133.4, 128.9, 128.5, 127.5, 115.6, 62.9, 60.0, 54.5, 33.8, 25.7, 18.3, −4.6. ESIMS: 636.3 $(M + H)^+$

### 4.8. (S)-2-amino-3-(3,5-bis((tert-butyldimethylsilyl)oxy)-4-methoxyphenyl)propanoic acid **19**

A solution of **18** (636 mg, 1.0 mmol) and 10% palladium on activated charcoal (64 mg) in methanol (20 mL) was stirred under 1 atm of hydrogen gas at 40 °C for 6 h. The reaction mixture was filtered, and the filtrate was concentrated to afford **19** (450 mg, 99%) which could be used directly in the next step without further purification.

$[a]_D^{27}$ -10.0 (c 1.38, in MeOH); $^1$H NMR (400 MHz, $CDCl_3$): $\delta$ (ppm) 6.47 (s, 2H), 3.70 (m, 1H), 3.64 (s, 3H), 3.20 (m, 1H), 2.80 (m, 1H), 0.94 (s, 18H), 0.12 (s, 12H); $^{13}$CNMR (100 MHz, DMSO): d (ppm) 170.2, 149.3, 141.4, 132.9, 115.9, 60.0, 55.6, 36.8, 26.7, 18.4, −4.3. ESIMS: 456.2 $(M + H)^+$

### 4.9. (S)-2-(((allyloxy)carbonyl)amino)-3-(3,5-bis((tert-butyldimethylsilyl)oxy)-4-methoxyphenyl)propanoic acid **1b**

To a solution of **19** (455 mg, 1.0 mmol) in THF (6 mL) at 10 °C was added 1 M NaOH solution (2.5 mL) and allylchloroformate (180 mg, 1.5 mmol), and the reaction was stirred at 25 °C for 50 min. The mixture was quenched with acetic acid (120 mg, 2 mmol) and concentrated. The residue was partitioned between water (20 mL) and dichloromethane (20 mL). The organic layer was dried (sodium sulfate) and concentrated, and the residue was purified by flash column chromatography (15% ethyl acetate in hexane) to give **1b** (501 mg, 93%) as an oil.

$[a]_D^{27}$ 9.7 (c 1.0, in MeOH), 25.4 (c 1.0, in DCM); reference [14] 18.8 (c 1.0, in DCM);

$^1$H NMR (400 MHz, $CDCl_3$): $\delta$ (ppm) 6.32 (s, 2H), 5.87 (m, 1H), 5.28.

(d, J = 16 Hz, 1H), 5.20 (d, J = 12 Hz, 1H), 5.11 (d, J = 8 Hz, 1H), 4.58 (m, 1H), 4.55 (d, J = 4 Hz, 2H), 3.70 (s, 3H), 3.02 (dd, J = 12, 4 Hz, 1H), 2.96 (d, J = 12, 4 Hz, 1H), 0.99 (s, 18H), 0.15 (s, 12H);

## Declaration of competing interest

□ The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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## Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.tet.2020.131243.

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