# **Supplementary Material**

# Photocatalytic Decarboxylative Alkenylation of α-Amino and α-Hydroxy Acid-derived Redox Active Esters by NaI/PPh<sub>3</sub> Catalysis

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## **1. General Information**

## A. Materials:

All reactions were conducted in oven-dried Schlenk tubes under argon atmosphere (purity  $\geq$  99.99%) unless otherwise mentioned. NaI (99.999%) was bought from Acros and used after dried using a heat gun. Other commercial reagents were purchased from Adamas-beta, TCI and Aldrich. Organic solutions were concentrated under reduced pressure on Buchi rotary evaporator. The LED lamps were purchased from Kessil (PR160-390 nm, 427 nm, 440 nm, 456 nm, 467 nm). The Photo Reaction Setup was purchased from Anhui kemi machinery technology Co., Ltd.



Figure S1. The Photo Reaction Setup and Kessil blue LED lamps

## **B.** Analytical Methods:

<sup>1</sup>H-NMR and <sup>13</sup>C-NMR spectra were recorded on a Bruker Avance 400 spectrometer at ambient temperature. Data for <sup>1</sup>H-NMR are reported as follows: chemical shift (ppm, scale), multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet and/or multiplet resonances, br = broad), coupling constant (Hz), and integration. Data for <sup>13</sup>C-NMR are reported in terms of chemical shift (ppm, scale), multiplicity, and coupling constant (Hz). HRMS analysis was performed on Finnigan LCQ advantage Max Series MS System. ESI-mass data were acquired using a Thermo LTQ Orbitrap XL Instrument equipped with an ESI source and controlled by Xcalibur software. Flash column chromatographic purification of products was accomplished using forced-flow chromatography on Silica Gel (200-300 mesh).

## 2. Investigation of the Key Reaction Parameters

	Ph Ph +	Cbz <sup>-N</sup> -CO <sub>2</sub> NPhth -	$\frac{\text{MI (10 mol\%)}}{\text{PPh}_3 (10 \text{ mol\%})} \rightarrow (10 \text{ mol\%})$ solvent (2 mL)	Cbz <sup>-N</sup> Ph
	<b>1</b> (0.2 mmol)	<b>2</b> (0.3 mmol)	r.t., 15 h	3
entry	MI	solvent	light (nm)	<b>3</b> Yield (%)
1	NaI	acetone	456	84
2	NaI	DMF	456	42
3	NaI	DMA	456	64
4	NaI	CH <sub>3</sub> CN	456	16
5	NaI	THF	456	66
6	NaI	dioxane	456	0
7	NaI	PhCF <sub>3</sub>	456	0
8	NaI	EtOAc	456	42
9	NaI	NMP	456	56
10	NaI	$CH_2Cl_2$	456	0
11	<sup>n</sup> Bu <sub>4</sub> NI	acetone	456	70
12	LiI	acetone	456	80
13	KI	acetone	456	84
14	RbI	acetone	456	84
15	CsI	acetone	456	62
16	$ZnI_2$	acetone	456	0
17 <sup>a</sup>	NaI	acetone	456	92
$18^{b}$	NaI	acetone	456	91
19		acetone	456	<5
$20^{c}$	NaI	acetone	456	<5
21 <sup>c</sup>		acetone	456	<5
$22^d$	NaI	acetone		0
23 <sup>a</sup>	NaI	acetone	467	90
24 <sup><i>a</i></sup>	NaI	acetone	440	84
25 <sup><i>a</i></sup>	NaI	acetone	427	85
$26^a$	NaI	acetone	390	89

Table S1: Parameters affecting decarboxylative alkenylation of α-amino acid derived redox active esters

Reaction conditions: **1** (0.2 mmol, 1 equiv), **2** (0.3 mmol), MI (10 mol%), PPh<sub>3</sub> (10 mol%), solvent (2 mL), irradiation by blue LEDs at room temperature for 15 h under argon atmosphere. Yield of isolated product. <sup>*a*</sup>reaction time: 24 h. <sup>*b*</sup>NaI (20 mol%), PPh<sub>3</sub> (20 mol%), 15 h. <sup>*c*</sup>no PPh<sub>3</sub>. <sup>*d*</sup>50 °C.

# Table S2: Different [P] compound in the decarboxylative alkenylation of αamino acid derived redox active esters



Reaction conditions: **1** (0.2 mmol, 1 equiv), **2** (0.3 mmol), NaI (20 mol%), [P] (20 mol%), acetone (2 mL), irradiation by blue LEDs (456 nm) at room temperature for 15 h under argon atmosphere. Yield of isolated product.

## Table S3: Different types of substrates in the decarboxylative alkenylation



## 3. General Procedure for Synthesis of Substrates

General procedure for synthesis of redox active esters<sup>1</sup>



The corresponding alkyl carboxylic acid (10 mmol, 1.0 eq.), *N*-hydroxyphthalimide (11 mmol, 1.1 eq.), and 4-dimethylaminopyridine (1.0 mmol, 10 mol%) were mixed in a flask with a magnetic stirring bar. Dry  $CH_2Cl_2$  (40 mL) was added. Then a solution of N, N'-dicyclohexylcarbodiimide (11.0 mmol, 1.1 eq.) in  $CH_2Cl_2$  (15 mL) was added slowly at room temperature. The reaction mixture was monitored by TLC. After completed, the reaction mixture was filtered and the solution was concentrated on a rotary evaporator. The residue was purified by flash column chromatography to give corresponding redox active esters.

## General procedure for the synthesis of (hetero)aryl acrylic acids<sup>2</sup>



Piperidine (10.0 mol) was added to a solution of the (hetero)aryl aldehyde (10.0 mmol), malonic acid (10.0 mmol) and pyridine (15.0 mmol) under nitrogen atmosphere. The reaction mixture was refluxed under vigorous stirring for 3 hours, after which according to TLC the reaction was completed. Then the solution was poured into 2 N HCl and then on ice. The formed precipitate was collected by filtration and recrystallized from water or from 3:1 water/ethanol mixture. If no precipitate was formed an extraction with  $3 \times 20$  mL CHCl<sub>3</sub> or CH<sub>2</sub>Cl<sub>2</sub> was made and the organic phase was collected, dried over MgSO<sub>4</sub>, and evaporated to dryness affording a residue that was recrystallized from aqueous ethanol.

## 4. Experimental Procedures and Spectral Data

## 4.1 Experimental procedures

#### General Procedure A

Aryl olefin (1.0 eq., 0.2 mmol, if solid), redox-active esters (1.5 eq., 0.3 mmol), NaI (10 mol%), PPh<sub>3</sub> (10 mol%) were placed in a 10 mL transparent Schlenk tube equipped with a stirring bar. The tube was evacuated and filled with argon (three times). To these solids, acetone (2.0 mL) and aryl olefin (1.0 eq., 0.2 mmol) (if liquid) were added via a gastight syringe under argon atmosphere. The reaction mixture was stirred under irradiation with blue LEDs (Kessil, PR160-456 nm), maintained at approximately room temperature ( $28 \pm 2$  °C). After 24 h, the mixture was quenched with 5 mL water, then extracted with ethyl acetate (3 x 10 mL). The organic layers were combined and concentrated on a rotary evaporator. The product was purified *via* flash column chromatography on silica gel (petroleum ether/ethyl acetate = 10:1).

## General Procedure B

Redox-active esters (1.0 eq., 0.2 mmol), (hetero)aryl acrylic acids (1.5 eq., 0.3 mmol), NaI (20 mol%), PPh<sub>3</sub> (20 mol%) were placed in a 10 mL transparent Schlenk tube equipped with a stirring bar. The tube was evacuated and filled with argon (three times). To these solids, acetone (2.0 mL) was added via a gastight syringe under argon atmosphere. The reaction mixture was stirred under irradiation with blue LEDs (Kessil, PR160-440 nm), maintained at approximately room temperature ( $28 \pm 2$  °C). After 24 h, the mixture was quenched with 5 mL water, then extracted with ethyl acetate (3 x 10 mL). The organic layers were combined and concentrated on a rotary evaporator. The product was purified *via* flash column chromatography on silica gel (petroleum ether/ethyl acetate = 10:1).

#### 4.2 Spectral Data

**benzyl (4,4-diphenylbut-3-en-2-yl)carbamate (3)**: Following the general procedure A, using 10.0 mol% NaI, 10.0 mol% PPh<sub>3</sub>, 2.0 mL acetone, obtained in 84% yield as white solid (Eluent: petroleum ether/ethyl acetate = 10/1). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.40 – 7.23 (m, 12H), 7.23 – 7.16 (m, 3H), 5.90 (d, *J* = 9.2 Hz, 1H), 5.11 – 4.99 (m, 2H), 4.80 (s, 1H), 4.33 (s, 1H), 1.23 (d, *J* = 6.7 Hz, 3H). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)  $\delta$  155.2, 142.5, 141.8, 139.2, 136.6, 130.3, 129.6, 128.6, 128.4, 128.2, 128.1, 127.5, 127.5, 127.4, 66.6, 46.8, 22.1. (one carbon signal was overlapped)

HRMS (ESI) calcd. for C<sub>24</sub>H<sub>23</sub>O<sub>2</sub>NNa<sup>+</sup> [M+Na]<sup>+</sup>: 380.1621, found: 380.1621.



**2-(1,4,4-triphenylbut-3-en-2-yl)isoindoline-1,3-dione (4)**: Following the general procedure A, using 20.0 mol% NaI, 20.0 mol% PPh<sub>3</sub>, 2.0 mL acetone, obtained in 81% yield as white solid (Eluent: petroleum ether/ethyl acetate = 10/1).

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.79 – 7.59 (m, 4H), 7.38 – 7.30 (m, 3H), 7.25 – 7.20 (m,5H), 7.19 – 7.07 (m, 3H), 7.07 – 6.90 (m,4H), 6.81 – 6.50 (m, 1H), 5.34 – 4.98 (m, 1H), 3.44 – 3.09 (m, 2H).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 167.8, 145.0, 141.3, 138.6, 137.3, 133.8, 131.8, 129.4, 129.2, 128.3, 128.3, 128.1, 127.7, 127.5, 127.3, 126.5, 124.9, 123.1, 51.9, 39.4.
HRMS (ESI) *calcd.* for C<sub>30</sub>H<sub>23</sub>O<sub>2</sub>NNa<sup>+</sup> [M+Na]<sup>+</sup>: 452.1621, *found*: 452.1621.



**benzyl (3,3-diphenylallyl)carbamate (5):** Following the general procedure A, using 20.0 mol% NaI, 20.0 mol% PPh<sub>3</sub>, 2.0 mL acetone, obtained in 73% yield as viscous liquid (Eluent: petroleum ether/ethyl acetate = 10/1).

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.33 – 7.23 (m, 7H), 7.22 – 7.03 (m, 8H), 6.06 – 5.97 (m, 1H), 5.03 (s, 2H), 4.74 (s, 1H), 3.81 (t, *J* = 6.2 Hz, 2H). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)  $\delta$  156.2, 141.6, 138.9, 136.5, 130.1, 129.7, 128.6, 128.4, 128.3, 128.2, 128.2, 127.62 127.6, 127.5, 124.8, 66.8, 40.4. HRMS (ESI) *calcd.* for C<sub>23</sub>H<sub>21</sub>O<sub>2</sub>NNa<sup>+</sup> [M+Na]<sup>+</sup>: 366.1465, *found*: 366.1465.

*tert*-butyl (4,4-diphenylbut-3-en-2-yl)carbamate (6): Following the general procedure A, using 20.0 mol% NaI, 20.0 mol% PPh<sub>3</sub>, 2.0 mL THF, obtained in 75% yield as viscous liquid (Eluent: petroleum ether/ethyl acetate = 10/1).

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.46 – 7.30 (m, 3H), 7.29 – 7.14 (m, 7H), 5.90 (d, *J* = 9.1 Hz, 1H), 4.54 (s, 1H), 4.28 (d, *J* = 6.7 Hz, 1H), 1.41 (s, 9H), 1.21 (d, *J* = 6.6 Hz, 3H).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 154.8, 142.1, 142.0, 139.2, 131.0, 129.7, 128.3, 128.2, 127.4, 127.4, 79.2, 46.3, 28.4, 22.3. (one carbon signal was overlapped)
HRMS (ESI) *calcd.* for C<sub>21</sub>H<sub>25</sub>O<sub>2</sub>NNa<sup>+</sup> [M+Na]<sup>+</sup>: 346.1778, *found*: 346.1778.



*tert*-butyl 4-(((benzyloxy)carbonyl)amino)-6,6-diphenylhex-5-enoate (7): Following the general procedure A, using 10.0 mol% NaI, 10.0 mol% PPh<sub>3</sub>, 2.0 mL acetone, obtained in 87% yield as viscous liquid (Eluent: petroleum ether/ethyl acetate = 10/1).

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.40 – 7.27 (m, 8H), 7.26 – 7.16 (m, 7H), 5.88 (d, *J* = 9.3 Hz, 1H), 5.07 (s, 2H), 5.02 – 4.90 (m, 1H), 4.24 (s, 1H), 2.26 – 2.11 (m, 2H), 1.97 – 1.75 (m, 2H), 1.34 (s, 9H).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 172.7, 155.40, 143.9, 141.8, 139.0, 136.6, 132.5, 129.7, 128.5, 128.4, 128.2, 128.1, 127.7, 127.4, 125.9, 125.4, 80.5, 66.6, 50.7, 32.0, 30.9, 28.1.

HRMS (ESI) *calcd*. for C<sub>30</sub>H<sub>33</sub>O<sub>4</sub>NNa<sup>+</sup> [M+Na]<sup>+</sup>: 494.2302, *found*: 494.2302.



*tert*-butyl (1-(4-iodophenyl)-4,4-diphenylbut-3-en-2-yl)carbamate (8): Following the general procedure A, using 20.0 mol% NaI, 20.0 mol% PPh<sub>3</sub>, 2.0 mL acetone, obtained in 61% yield as light viscous liquid (Eluent: petroleum ether/ethyl acetate = 10/1).

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.34 – 7.27 (m, 3H), 7.26 – 7.13 (m, 7H), 7.03 – 6.90 (m, 4H), 5.86 (d, *J* = 9.4 Hz, 1H), 4.57 (s, 1H), 4.39 (s, 1H), 2.88 (s, 1H), 2.73 (dd, *J* = 13.3, 7.1 Hz, 1H), 1.39 (s, 9H).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 154.8, 143.9, 141.7, 139.0, 136.2, 132.2, 131.0, 129.4, 128.3, 128.3, 128.2, 127.9, 127.7, 127.4, 127.3, 79.5, 51.6, 41.5, 28.4.

HRMS (ESI) calcd. for C<sub>27</sub>H<sub>28</sub>IO<sub>2</sub>NNa<sup>+</sup> [M+Na]<sup>+</sup>: 548.1075, found: 548.1075.

*tert*-butyl (5-(methylthio)-1,1-diphenylpent-1-en-3-yl)carbamate (9): Following the general procedure A, using 10.0 mol% NaI, 10.0 mol% PPh<sub>3</sub>, 2.0 mL acetone, obtained in 60% yield as viscous liquid (Eluent: petroleum ether/ethyl acetate = 10/1). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.43 – 7.26 (m, 4H), 7.27 – 7.14 (m, 6H), 5.92 (d, *J* = 9.3 Hz, 1H), 4.62 (s, 1H), 4.39 – 4.19 (m, 1H), 2.41 (t, *J* = 7.9 Hz, 2H), 2.01 (s, 3H), 1.91 – 1.64 (m, 2H), 1.41 (s, 9H). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 154.8, 143.7, 141.9, 139.1, 129.6, 128.9, 128.4, 128.2,

127.6, 127.5, 127.4, 79.3, 49.9, 35.8, 30.4, 28.4, 15.5.

HRMS (ESI) *calcd*. for C<sub>23</sub>H<sub>29</sub>O<sub>2</sub>NNa<sup>+</sup>S [M+Na]<sup>+</sup>: 406.1811, *found*: 406.1811.



**benzyl 2-(2,2-diphenylvinyl)pyrrolidine-1-carboxylate (10):** Following the general procedure A, using 10.0 mol% NaI, 10.0 mol% PPh<sub>3</sub>, 2.0 mL acetone, obtained in 86% yield as viscous liquid (Eluent: petroleum ether/ethyl acetate = 20/1). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.45 – 7.07 (m, 14H), 6.94 (s, 1H), 6.00 (d, *J* = 9.1 Hz, 1H), 5.13 (d, *J* = 12.2 Hz, 1H), 4.97 (d, *J* = 10.4 Hz, 1H), 4.36 (s, 1H), 3.52 (t, *J* = 5.9 Hz,2H), 2.17 – 2.01 (m, 1H), 2.00 – 1.88 (m, 1H), 1.88 – 1.67 (m, 2H). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)  $\delta$  155.2, 141.8, 141.2, 139.5, 136.9, 130.5, 129.8, 128.4, 128.3, 128.3, 128.1, 127.8, 127.3, 127.3, 127.1, 66.8, 56.4, 47.2, 34.3, 23.9. HRMS (ESI) *calcd.* for C<sub>26</sub>H<sub>25</sub>O<sub>2</sub>NNa<sup>+</sup> [M+Na]<sup>+</sup>: 406.1778, *found*: 406.1778.



*tert*-butyl 2-(2,2-diphenylvinyl)pyrrolidine-1-carboxylate (11): Following the general procedure A, using 10.0 mol% NaI, 10.0 mol% PPh<sub>3</sub>, 2.0 mL acetone, obtained in 60% yield as viscous liquid (Eluent: petroleum ether/ethyl acetate = 20/1). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.44 – 7.25 (m, 5H), 7.25 – 7.12 (m, 5H), 6.01 (d, *J* = 8.7 Hz, 1H), 4.28 (s, 1H), 3.66 – 3.16 (m, 2H), 2.26 – 1.83 (m, 3H), 1.77 (dd, *J* = 11.4, 6.8 Hz, 1H), 1.39 (s, 9H).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 154.7, 142.4, 140.4, 139.5, 132.2, 130.0, 128.2, 128.1, 127.4, 127.2, 127.1, 79.1, 56.3, 46.8, 34.9, 28.6, 23.8.

HRMS (ESI) *calcd*. for C<sub>23</sub>H<sub>27</sub>O<sub>2</sub>NNa<sup>+</sup> [M+Na]<sup>+</sup>: 372.1934, *found*: 372.1934.



*tert*-butyl 2-(2,2-diphenylvinyl)azetidine-1-carboxylate (12): Following the general procedure A, using 10.0 mol% NaI, 10.0 mol% PPh<sub>3</sub>, 2.0 mL acetone, obtained in 86% yield as viscous liquid (Eluent: petroleum ether/ethyl acetate = 20/1).

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.41 – 7.26 (m, 5H), 7.26 – 7.17 (m, 5H), 6.09 (d, *J* = 9.2 Hz, 1H), 4.40 – 4.09 (m, 1H), 3.41 (s, 1H), 3.20 – 2.91 (m, 1H), 1.80 – 1.58 (m, 2H), 1.37 (s, 9H).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 156.8, 143.4, 141.7, 139.3, 130.4, 129.6, 128.3, 128.2, 127.6, 127.5, 127.5, 79.5, 67.0, 37.9, 37.1, 28.4.
HRMS (ESI) *calcd.* for C<sub>22</sub>H<sub>25</sub>O<sub>2</sub>NNa<sup>+</sup> [M+Na]<sup>+</sup>:358.1778, *found*: 358.1778.

**benzyl** (4-methyl-1,1-diphenylpent-1-en-3-yl)carbamate (13): Following the general procedure A, using 20.0 mol% NaI, 20.0 mol% PPh<sub>3</sub>, 2.0 mL acetone, obtained in 76% yield as viscous liquid (Eluent: petroleum ether/ethyl acetate = 10/1). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.56 – 6.94 (m, 15H), 5.90 (d, *J* = 9.7 Hz, 1H), 5.07 (s, 2H), 4.80 (s, 1H), 4.38 – 3.84 (m, 1H), 2.00 – 1.59 (m, 1H), 0.87 (t, *J* = 7.0 Hz, 6H). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)  $\delta$  155.5, 144.1, 142.3, 139.3, 136.7, 129.8, 128.6, 128.5, 128.4, 128.3, 128.2, 128.1, 127.5, 127.4, 127.3, 66.6, 55.8, 33.5, 18.6. HRMS (ESI) *calcd*. for C<sub>26</sub>H<sub>27</sub>O<sub>2</sub>NNa<sup>+</sup> [M+Na]<sup>+</sup>: 408.1934, *found*: 408.1934.



*tert*-butyl (1,1-diphenylhex-1-en-5-yn-3-yl)carbamate (14): Following the general procedure A, using 10.0 mol% NaI, 10.0 mol% PPh<sub>3</sub>, 2.0 mL acetone, obtained in 61% yield as viscous liquid (Eluent: petroleum ether/ethyl acetate = 10/1).

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.41 – 7.31 (m, 3H), 7.30 – 7.26 (m, 2H), 7.25 – 7.18 (m, 5H), 6.11 (d, *J* = 9.2 Hz, 1H), 4.86 (s, 1H), 4.40 (s, 1H), 2.58 – 2.47 (m, 1H), 2.45 – 2.35 (m, 1H), 2.04 (t, *J* = 2.6 Hz, 1H), 1.41 (s, 9H).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 154.6, 144.1, 141.8, 139.0, 129.6, 128.5, 128.2, 127.7, 127.6, 127.5, 127.4, 80.3, 79.5, 71.0, 48.1, 28.4, 25.8.

HRMS (ESI) calcd. for C<sub>23</sub>H<sub>25</sub>O<sub>2</sub>NNa<sup>+</sup> [M+Na]<sup>+</sup>: 370.1778, found: 370.1778.



*N*-(1,4,4-triphenylbut-3-en-2-yl)pivalamide (15): Following the general procedure A, using 20.0 mol% NaI, 20.0 mol% PPh<sub>3</sub>, 2.0 mL acetone, obtained in 47% yield as viscous liquid (Eluent: petroleum ether/ethyl acetate = 10/1).

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.41 – 7.27 (m, 4H), 7.26 – 7.20 (m, 5H), 7.20 – 7.13 (m, 2H), 7.13 – 7.00 (m, 4H), 5.91 (d, *J* = 9.1 Hz, 1H), 5.59 (d, *J* = 6.9 Hz, 1H), 5.10 – 4.39 (m, 1H), 2.87 (ddd, *J* = 42.2, 13.4, 6.3 Hz, 2H), 1.09 (s, 9H).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 177.3, 143.8, 142.0, 139.1, 137.4, 129.7, 129.5, 128.4, 128.4, 128.3, 128.2, 127.6, 127.4, 127.4, 126.5, 50.1, 41.4, 38.7, 27.5.

HRMS (ESI) calcd. for C<sub>27</sub>H<sub>29</sub>ONNa<sup>+</sup> [M+Na]<sup>+</sup>: 406.2141, found: 406.2141.



tert-butyl(1-((5-(methylthio)-1,1-diphenylpent-1-en-3-yl)amino)-1-oxo-3-

**phenylpropan-2-yl)carbamate (16):** Following the general procedure A, using 10.0 mol% NaI, 10.0 mol% PPh<sub>3</sub>, 2.0 mL acetone, obtained in 76% yield as white solid (Eluent: petroleum ether/ethyl acetate = 5/1, d.r = 54:46).

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.44 – 7.23 (m, 9H), 7.22 – 7.15 (m, 6H), 6.26 – 5.50 (m, 2H), 5.03 (d, *J* = 34.0 Hz, 1H), 4.52 (s, 1H), 4.23 (s, 1H), 3.21 – 2.78 (m, 2H), 2.39 – 2.07 (m, 2H), 1.97 (s, 3H), 1.70 (s, 2H), 1.47 – 1.31 (m, 9H). HRMS (ESI) *calcd.* for C<sub>32</sub>H<sub>38</sub>O<sub>3</sub>N<sub>2</sub>SNa<sup>+</sup> [M+Na]<sup>+</sup>: 553.2495, *found*: 553.2495.



*N*-(2-oxo-2-((1,4,4-triphenylbut-3-en-2-yl)amino)ethyl)benzamide (17): Following the general procedure A, using 20.0 mol% NaI, 20.0 mol% PPh<sub>3</sub>, 2.0 mL acetone, obtained in 51% yield as white solid (Eluent: petroleum ether/ethyl acetate = 2/1). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.87 – 7.73 (m, 2H), 7.57 – 7.46 (m, 1H), 7.48 – 7.36 (m, 2H), 7.35 – 7.26 (m, 3H), 7.26 – 7.18 (m, 3H), 7.18 – 6.94 (m, 9H), 5.96 (d, *J* = 9.3 Hz, 1H), 4.97 – 4.61 (m, 1H), 4.04 (dd, *J* = 16.4, 3.9 Hz, 2H), 2.89 (dd, *J* = 27.1, 6.6 Hz, 2H), 2.39 (s, 1H).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 167.8, 167.7, 144.0, 141.6, 139.0, 137.2, 133.4, 131.9, 129.6, 129.4, 128.6, 128.3, 128.3, 128.2, 127.6, 127.4, 127.4, 127.3, 127.2, 126.5, 50.8, 43.9, 41.7.

HRMS (ESI) *calcd*. for C<sub>31</sub>H<sub>28</sub>O<sub>2</sub>N<sub>2</sub>Na<sup>+</sup> [M+Na]<sup>+</sup>: 483.2043, *found*: 483.2043.



*tert*-butyl (4,4-di-p-tolylbut-3-en-2-yl)carbamate (18): Following the general procedure A, using 10.0 mol% NaI, 10.0 mol% PPh<sub>3</sub>, 2.0 mL acetone, obtained in 69% yield as white solid (Eluent: petroleum ether/ethyl acetate = 10/1).

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.23 – 7.14 (m, 2H), 7.13 – 7.02 (m, 6H), 5.83 (d, *J* = 9.1 Hz, 1H), 4.53 (s, 1H), 4.39 – 4.06 (m, 1H), 2.36 (s, 3H), 2.31 (s, 3H), 1.40 (s, 9H), 1.21 (d, *J* = 6.6 Hz, 3H).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 154.8, 142.1, 139.5, 137.2, 136.9, 136.4, 129.9, 129.6, 129.0, 128.8, 127.3, 79.1, 46.3, 28.4, 22.4, 21.3, 21.1.

HRMS (ESI) calcd. for C<sub>23</sub>H<sub>29</sub>O<sub>2</sub>NNa<sup>+</sup> [M+Na]<sup>+</sup>: 374.2091, found: 374.2091.



*tert*-butyl (4,4-bis(4-methoxyphenyl)but-3-en-2-yl)carbamate (19): Following the general procedure A, using 10.0 mol% NaI, 10.0 mol% PPh<sub>3</sub>, 2.0 mL acetone, obtained in 87% yield as white solid (Eluent: petroleum ether/ethyl acetate = 10/1). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.20 – 7.08 (m, 4H), 6.94 – 6.85 (m, 2H), 6.83 – 6.73 (m, 2H), 6.10 – 5.53 (m, 1H), 4.53 (s, 1H), 4.40 – 4.13 (m, 1H), 3.82 (s, 3H), 3.78 (s,

3H), 1.41 (s, 9H), 1.21 (d, *J* = 6.6 Hz, 3H).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 159.1, 158.8, 154.8, 135.1, 132.3, 131.8, 130.8, 129.0,

128.6, 113.7, 113.5, 79.1, 55.3, 55.2, 46.3, 28.5, 22.5. HRMS (ESI) *calcd.* for C<sub>23</sub>H<sub>29</sub>O<sub>4</sub>NNa<sup>+</sup> [M+Na]<sup>+</sup>: 406.1989, *found*: 406.1989.

**benzyl** (4,4-bis(4-fluorophenyl)but-3-en-2-yl)carbamate (20): Following the general procedure A, using 10.0 mol% NaI, 10.0 mol% PPh<sub>3</sub>, 2.0 mL acetone, obtained in 80% yield as white solid (Eluent: petroleum ether/ethyl acetate = 5/1).

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.43 – 7.25 (m, 5H), 7.23 – 7.02 (m, 5H), 7.01 – 6.64 (m, 3H), 5.83 (d, *J* = 9.2 Hz, 1H), 5.23 – 4.97 (m, 2H), 4.83 (s, 1H), 4.30 (s, 1H), 1.22 (d, *J* = 6.7 Hz, 3H).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 163.7, 161.20 155.2, 140.5, 137.8, 136.5, 134.9, 131.3, 131.2, 130.7, 129.0, 128.9, 128.6, 128.2, 128.1, 115.6, 115.4, 115.2, 115.0, 66.7, 46.7, 22.0.

HRMS (ESI) calcd. for C<sub>24</sub>H<sub>21</sub>O<sub>2</sub>NF<sub>2</sub>Na<sup>+</sup> [M+Na]<sup>+</sup>: 416.1433, found: 416.1433.



**benzyl** (4,4-bis(4-chlorophenyl)but-3-en-2-yl)carbamate (21): Following the general procedure A, using 20.0 mol% NaI, 20.0 mol% PPh<sub>3</sub>, 2.0 mL acetone, obtained in 75% yield as viscous liquid (Eluent: petroleum ether/ethyl acetate = 10/1). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.33 (s, 7H), 7.24 – 7.02 (m, 6H), 6.00 – 5.70 (m, 1H), 5.23 – 4.95 (m, 2H), 4.78 (s, 1H), 4.42 – 4.13 (m, 1H), 1.23 (d, *J* = 6.7 Hz, 3H). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)  $\delta$  155.2, 139.8, 137.1, 136.4, 133.7, 133.6, 131.4, 131.0, 128.8, 128.6, 128.4, 128.2, 128.2, 127.4, 66.7, 46.7, 21.9. (one carbon signal was overlapped)

HRMS (ESI) *calcd*. for C<sub>24</sub>H<sub>21</sub>O<sub>2</sub>NCl<sub>2</sub>Na<sup>+</sup> [M+Na]<sup>+</sup>: 448.0842, *found*: 448.0842.



**benzyl** (4,4-bis(4-bromophenyl)but-3-en-2-yl)carbamate (22): Following the general procedure A, using 20.0 mol% NaI, 20.0 mol% PPh<sub>3</sub>, 2.0 mL acetone, obtained in 76% yield as viscous liquid (Eluent: petroleum ether/ethyl acetate = 10/1). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.55 – 7.29 (m, 8H), 7.18 – 6.89 (m, 5H), 5.88 (d, *J* = 9.2 Hz, 1H), 5.16 – 4.98 (m, 2H), 4.97 – 4.83 (m, 1H), 4.28 (s, 1H), 1.20 (d, *J* = 6.6 Hz, 3H).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 155.2, 140.2, 137.5, 136.4, 134.4, 131.7, 131.4, 131.3, 128.9, 128.6, 128.2, 128.2, 123.5, 121.8, 66.7, 46.7, 21.8. (one carbon signal was overlapped)

HRMS (ESI) calcd. for C<sub>24</sub>H<sub>21</sub>O<sub>2</sub>NBr[81]BrNa<sup>+</sup> [M+Na]<sup>+</sup>: 537.9816, found: 537.9811.



*tert*-butyl(3-(benzyloxy)-1-((4,4-bis(4-methoxyphenyl)but-3-en-2-yl)amino)-1oxobutan-2-yl)carbamate (23): Following the general procedure A, using 10.0 mol% NaI, 10.0 mol% PPh<sub>3</sub>, 2.0 mL acetone, obtained in 90% yield as white solid (Eluent: petroleum ether/ethyl acetate = 2/1, d.r = 58:42).

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.34 – 7.26 (m, 4H), 7.16 – 7.06 (m, 4H), 6.93 – 6.84 (m, 2H), 6.82 – 6.74 (m, 2H), 6.62 – 6.47 (m, 1H), 5.70 (dd, *J* = 9.4, 2.4 Hz, 1H), 5.53 (s, 1H), 4.75 – 4.44 (m, 3H), 4.44 – 3.99 (m, 2H), 3.94 – 3.58 (m, 6H), 1.45 (d, *J* = 5.6 Hz, 9H), 1.28 – 0.92 (m, 6H).

HRMS (ESI) calcd. for C<sub>34</sub>H<sub>42</sub>O<sub>6</sub>N<sub>2</sub>Na<sup>+</sup> [M+Na]<sup>+</sup>: 597.2935, found: 597.2935.



(3-phenoxyprop-1-ene-1,1-diyl)dibenzene (24): Following the general procedure A, using 20.0 mol% NaI, 20.0 mol% PPh<sub>3</sub>, 2.0 mL acetone, obtained in 70% yield as colorless liquid (Eluent: petroleum ether/ethyl acetate = 40/1). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.45 – 7.26 (m, 7H), 7.26 – 7.16 (m, 5H), 6.94 – 6.77

(m, 3H), 6.37 - 6.27 (m, 1H), 4.59 (dd, J = 6.6, 1.6 Hz, 2H).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 158.6, 145.5, 141.7, 139.0, 129.7, 129.5, 128.4, 128.2, 127.8, 127.8, 127.8, 124.0, 120.8, 114.8, 65.9.

HRMS (ESI) calcd. for C<sub>21</sub>H<sub>19</sub>O<sup>+</sup> [M+H]<sup>+</sup>: 287.1430, found: 287.1430.



(3-(3-chlorophenoxy)but-1-ene-1,1-diyl)dibenzene (25): Following the general procedure A, using 20.0 mol% NaI, 20.0 mol% PPh<sub>3</sub>, 2.0 mL acetone, obtained in 86% yield as viscous liquid (Eluent: petroleum ether/ethyl acetate = 40/1).

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.41 – 7.27 (m, 3H), 7.20 – 7.04 (m, 7H), 6.97 (t, *J* = 8.1 Hz, 1H), 6.79 – 6.69 (m, 1H), 6.59 – 6.45 (m, 2H), 5.94 (d, *J* = 9.0 Hz, 1H), 4.68 (dd, *J* = 9.0, 6.3 Hz, 1H), 1.44 (d, *J* = 6.3 Hz, 3H).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 157.4, 143.1, 140.2, 138.0, 133.5, 128.9, 128.6, 128.3, 127.5, 127.2, 126.9, 126.7, 126.4, 119.7, 115.2, 113.7, 70.9, 20.8.

HRMS (ESI) *calcd*. for C<sub>22</sub>H<sub>20</sub>ClO<sup>+</sup> [M+H]<sup>+</sup>: 335.1197, *found*: 335.1197.

# Ph Ph

(3-(2-iodophenoxy)pent-1-ene-1,1-diyl)dibenzene (26): Following the general procedure A, using 20.0 mmol% NaI, 20.0 mol% PPh<sub>3</sub>, 2.0 mL acetone, obtained in 88% yield as viscous liquid (Eluent: petroleum ether/ethyl acetate = 40/1).

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.89 – 7.62 (m, 1H), 7.46 – 7.30 (m, 3H), 7.30 – 7.20 (m, 3H), 7.20 – 7.13 (m, 2H), 7.13 – 7.00 (m, 3H), 6.68 – 6.53 (m, 1H), 6.51 – 6.34 (m, 1H), 6.21 – 6.04 (m, 1H), 4.73 – 4.49 (m, 1H), 2.13 – 1.74 (m, 2H), 1.09 (td, *J* = 7.4, 1.7 Hz, 3H).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 156.9, 144.6, 141.4, 139.3, 139.2, 129.7, 129.0, 128.5,

128.4, 128.2, 127.8, 127.8, 127.5, 122.7, 115.6, 88.5, 78.8, 29.2, 10.1. HRMS (ESI) *calcd.* for C<sub>23</sub>H<sub>22</sub>IO<sup>+</sup> [M+H]<sup>+</sup>: 441.0710, *found*: 441.0710.



2-(4-((4,4-diphenylbut-3-en-2-yl)oxy)phenyl)-4,4,5,5-tetramethyl-1,3,2-

**dioxaborolane (27):** Following the general procedure A, using 20.0 mol% NaI, 20.0 mol% PPh<sub>3</sub>, 2.0 mL acetone, obtained in 80% yield as white solid (Eluent: petroleum ether/ethyl acetate = 10/1).

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.72 – 7.58 (m, 2H), 7.47 – 7.32 (m, 3H), 7.27 – 7.12 (m, 7H), 6.73 – 6.57 (m, 2H), 6.04 (d, *J* = 9.0 Hz, 1H), 4.91 – 4.70 (m, 1H), 1.54 (d, *J* = 6.2 Hz, 3H), 1.31 (s, 12H).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 160.4, 143.8, 141.4, 139.2, 136.3, 129.9, 129.6, 128.5, 128.2, 127.8, 127.7, 127.5, 115.2, 83.5, 71.3, 24.9, 24.8, 21.9.

HRMS (ESI) calcd. for C<sub>28</sub>H<sub>31</sub>O<sub>3</sub>BNa<sup>+</sup> [M+Na]<sup>+</sup>:449.2258, found: 449.2259.

#### O Ph Ph

(3-ethoxyprop-1-ene-1,1-diyl)dibenzene (28): Following the general procedure A, using 20.0 mol% NaI, 20.0 mol% PPh<sub>3</sub>, 2.0 mL acetone, obtained in 66% yield as colorless liquid (Eluent: petroleum ether/ethyl acetate = 50/1).

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.35 – 7.22 (m, 3H), 7.21 – 7.16 (m, 5H), 7.12 – 7.05 (m, 2H), 6.16 (t, *J* = 6.6 Hz, 1H), 3.96 (d, *J* = 6.7 Hz, 2H), 3.39 (q, *J* = 7.0 Hz, 2H), 1.12 (t, *J* = 7.0 Hz, 3H).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 143.3, 140.9, 138.3, 128.7, 127.1, 127.1, 126.6, 126.4, 126.4, 124.8, 67.3, 64.7, 14.3.

HRMS (ESI) *calcd*. for C<sub>17</sub>H<sub>19</sub>O<sup>+</sup> [M+H]<sup>+</sup>: 239.1430, *found*: 239.1430.

# Ph Ph O Ph

(3-(benzyloxy)prop-1-ene-1,1-diyl)dibenzene (29): Following the general procedure A, using 20.0 mol% NaI, 20.0 mol% PPh<sub>3</sub>, 2.0 mL acetone, obtained in 73% yield as

colorless liquid (Eluent: petroleum ether).

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.38 – 7.24 (m, 13H), 7.19 – 7.14 (m, 2H), 6.31 – 6.22 (m, 1H), 4.47 (s, 2H), 4.10 (d, J = 6.7 Hz, 2H). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)  $\delta$  144.9, 141.9, 139.2, 138.3, 129.8, 128.4, 128.2, 127.9, 127.7, 127.6, 127.6, 127.5, 125.5, 72.4, 68.0. (one carbon signal was overlapped) HRMS (ESI) *calcd.* for C<sub>22</sub>H<sub>21</sub>O<sup>+</sup> [M+H]<sup>+</sup>: 301.1587, *found*: 301.1587.



**2-(2,2-diphenylvinyl)-2,3-dihydrobenzo**[*b*][1,4]dioxine (30): Following the general procedure A, using 20.0 mol% NaI, 20.0 mol% PPh<sub>3</sub>, 2.0 mL acetone, obtained in 68% yield as viscous liquid (Eluent: petroleum ether/ethyl acetate = 50/1).

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.37 – 7.24 (m, 3H), 7.23 – 7.18 (m, 6H), 7.17 – 7.13 (m, 1H), 6.93 – 6.64 (m, 4H), 6.01 (d, *J* = 9.2 Hz, 1H), 4.74 – 4.46 (m, 1H), 4.28 – 4.06 (m, 1H), 4.06 – 3.79 (m, 1H).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 147.1, 142.2, 141.9, 140.0, 137.5, 128.5, 127.5, 127.2, 127.2, 127.0, 126.6, 120.9, 120.5, 120.3, 116.4, 116.0, 70.3, 66.5.

HRMS (ESI) *calcd*. for C<sub>22</sub>H<sub>19</sub>O<sub>2</sub><sup>+</sup> [M+H]<sup>+</sup>: 315.1380, *found*: 315.1380.



**2-(2,2-diphenylvinyl)tetrahydrofuran (31):** Following the general procedure B, using 20.0 mol% NaI, 20.0 mol% PPh<sub>3</sub>, 2.0 mL acetone, obtained in 95% yield as viscous liquid (Eluent: petroleum ether/ethyl acetate = 50/1).

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.42 – 7.29 (m, 3H), 7.27 – 7.14 (m, 7H), 6.06 (d, *J* = 9.0 Hz, 1H), 4.39 – 4.20 (m, 1H), 4.01 – 3.86 (m, 1H), 3.79 – 3.66 (m, 1H), 2.09 – 1.93 (m, 2H), 1.92 – 1.79 (m, 1H), 1.79 – 1.67 (m, 1H).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 142.7, 140.9, 138.4, 128.9, 128.7, 127.1, 127.0, 126.6, 126.4, 126.3, 75.6, 67.1, 32.0, 25.4.

HRMS (ESI) calcd. for C<sub>18</sub>H<sub>19</sub>O<sup>+</sup> [M+H]<sup>+</sup>: 251.1430, found: 251.1430.



(3,3-diphenylallyl)(phenyl)sulfane (32): Following the general procedure B, using 20.0 mol% NaI, 20.0 mol% PPh<sub>3</sub>, 2.0 mL acetone, obtained in 42% yield as colorless liquid (Eluent: petroleum ether/ethyl acetate = 50/1).

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.39 – 7.25 (m, 6H), 7.24 – 7.13 (m, 7H), 7.12 – 6.98 (m, 2H), 6.15 (t, *J* = 7.8 Hz, 1H), 3.62 (d, *J* = 7.8 Hz, 2H).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 144.4, 141.9, 138.9, 135.7, 130.3, 129.9, 128.8, 128.3, 128.1, 127.5, 127.5, 127.5, 126.3, 124.2, 33.9.

HRMS (ESI) *calcd*. for C<sub>21</sub>H<sub>19</sub>S<sup>+</sup> [M+H]<sup>+</sup>: 303.1202, *found*: 303.1202.



*tert*-butyl (*E*)-(4-(4-methoxyphenyl)-1-phenylbut-3-en-2-yl)carbamate (33): Following the general procedure B, using 20.0 mol% NaI, 20.0 mol% PPh<sub>3</sub>, 2.0 mL acetone, obtained in 48% yield as colorless liquid (Eluent: petroleum ether/ethyl acetate = 10/1, E/Z = 93/7).

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.42 – 7.14 (m, 7H), 6.92 (dd, *J* = 69.8, 9.4 Hz, 2H), 6.39 (d, *J* = 15.9 Hz, 1H), 6.19 – 5.81 (m, 1H), 4.56 (s, 2H), 3.80 (s, 3H), 2.92 (s, 2H), 1.52 (d, *J* = 82.6 Hz, 9H).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 159.2, 155.2, 137.5, 129.6, 129.6, 128.4, 127.6, 127.5, 126.5, 113.9, 79.5, 55.3, 42.0, 28.4. (two carbon signals were overlapped)
HRMS (ESI) *calcd.* for C<sub>22</sub>H<sub>28</sub>O<sub>3</sub>N<sup>+</sup> [M+H]<sup>+</sup>: 354.2064, *found*: 354.2064.



*tert*-butyl (*E*)-(4-(3,4-dimethoxyphenyl)-1-phenylbut-3-en-2-yl)carbamate (34): Following the general procedure B, using 20.0 mol% NaI, 20.0 mol% PPh<sub>3</sub>, 2.0 mL acetone, obtained in 55% yield as viscous liquid (Eluent: petroleum ether/ethyl acetate = 5/1, E/Z = 98/2).

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.42 – 7.10 (m, 5H), 7.00 – 6.70 (m, 3H), 6.39 (d, *J* = 16.0 Hz, 1H), 6.00 (d, *J* = 15.9 Hz, 1H), 4.58 (s, 2H), 3.87 (s, 6H), 2.92 (s, 2H), 1.41 (s, 9H).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 155.2, 148.9, 148.7, 137.5, 129.9, 129.6, 128.4, 127.9, 126.5, 119.4, 111.1, 108.9, 79.5, 55.9, 55.8, 53.3, 42.0, 28.4. (one carbon signal was overlapped)

HRMS (ESI) *calcd*. for C<sub>23</sub>H<sub>30</sub>O<sub>4</sub>N<sup>+</sup> [M+H]<sup>+</sup>: 384.2169, *found*: 384.2169.



Following the general procedure B, using 20.0 mol% NaI, 20.0 mol% PPh<sub>3</sub>, 2.0 mL DMA, obtained in 59% yield as viscous liquid (Eluent: petroleum ether/ethyl acetate = 10/1, E/Z = 88/12).

*tert*-butyl (*E*)-(4-(2-methoxyphenyl)-1-phenylbut-3-en-2-yl)carbamate (35):

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.38 – 7.32 (m, 1H), 7.32 – 7.27 (m, 1H), 7.27 – 7.10 (m, 5H), 6.94 – 6.82 (m, 2H), 6.82 – 6.73 (m, 1H), 6.30 – 6.02 (m, 1H), 4.79 – 4.34 (m, 2H), 3.81 (s, 3H), 2.93 (s, 2H), 1.41 (s, 9H).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 156.7, 155.2, 137.6, 130.3, 129.7, 128.6, 128.4, 126.9, 126.4, 125.9, 124.9, 120.6, 110.9, 79.4, 55.4, 53.5, 42.0, 28.4.

HRMS (ESI) *calcd*. for C<sub>22</sub>H<sub>28</sub>O<sub>3</sub>N<sup>+</sup> [M+H]<sup>+</sup>: 354.2064, *found*: 354.2064.



*tert*-butyl (*E*)-(4-(4-(dimethylamino)phenyl)-1-phenylbut-3-en-2-yl)carbamate (36): Following the general procedure B, using 20.0 mol% NaI, 20.0 mol% PPh<sub>3</sub>, 2.0 mL DMA, obtained in 76% yield as viscous liquid (Eluent: petroleum ether/ethyl acetate = 10/1).

<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  7.33 – 7.25 (m, 4H), 7.25 – 7.16 (m, 5H), 6.70 (s, 2H), 6.36 (d, J = 15.9 Hz, 1H), 5.93 (d, J = 15.6 Hz, 1H), 4.55 (s, 2H), 2.95 (s, 6H), 1.41 (s,

9H).

<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>) δ 155.2, 150.1, 137.7, 130.2, 129.7, 128.3, 127.3, 126.4, 125.3, 122.3, 112.4, 79.3, 53.4, 42.2, 40.5, 28.4.

HRMS (ESI) calcd. for C<sub>23</sub>H<sub>31</sub>O<sub>2</sub>N<sub>2</sub><sup>+</sup> [M+H]<sup>+</sup>: 367.2380, found: 367.2380.



*tert*-butyl (*E*)-(4-(4-(diphenylamino)phenyl)-1-phenylbut-3-en-2-yl)carbamate (37): Following the general procedure B, using 20.0 mol% NaI, 20.0 mol% PPh<sub>3</sub>, 2.0 mL DMA, obtained in 54% yield as yellow viscous liquid (Eluent: petroleum ether/ethyl acetate = 20/1, E/Z = 92/8).

<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 7.36 – 7.27 (m, 2H), 7.26 – 7.13 (m, 9H), 7.12 – 7.05 (m, 4H), 7.04 – 6.94 (m, 4H), 6.40 (d, *J* = 15.9 Hz, 1H), 6.19 – 5.94 (m, 1H), 4.86 – 4.28 (m, 2H), 2.92 (s, 2H), 1.41 (s, 9H).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 155.2, 147.6, 147.3, 137.5, 131.0, 129.7, 129.6, 129.3, 128.4, 128.1, 127.2, 126.5, 124.4, 123.6, 122.9, 79.5, 53.3, 42.0, 28.4 HRMS (ESI) *calcd.* for C<sub>33</sub>H<sub>35</sub>O<sub>2</sub>N<sub>2</sub><sup>+</sup> [M+H]<sup>+</sup>: 491.2693, *found*: 491.2693.



*tert*-butyl (*E*)-(1-phenyl-4-(thiophen-3-yl)but-3-en-2-yl)carbamate (38): Following the general procedure B, using 20.0 mol% NaI, 20.0 mol% PPh<sub>3</sub>, 2.0 mL acetone, obtained in 70% yield as viscous liquid (Eluent: petroleum ether/ethyl acetate = 20/1, E/Z = 94/6).

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.29 (s, 2H), 7.25 – 7.16 (m, 3H), 7.11 (t, *J* = 7.7 Hz, 1H), 7.01 – 6.79 (m, 2H), 6.57 (d, *J* = 15.8 Hz, 1H), 6.06 – 5.85 (m, 1H), 4.80 – 4.34 (m, 2H), 2.90 (s, 2H), 1.41 (s, 9H).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 155.1, 141.9, 137.2, 129.6, 129.4, 128.4, 127.4, 126.6, 125.7, 124.1, 123.5, 79.6, 53.1, 41.9, 28.4

HRMS (ESI) *calcd*. for C<sub>19</sub>H<sub>24</sub>O<sub>2</sub>NS<sup>+</sup> [M+H]<sup>+</sup>: 330.1522, *found*: 330.1522.



Following the general procedure B, using 20.0 mol% NaI, 20.0 mol% PPh<sub>3</sub>, 2.0 mL DMA, obtained in 51% yield as viscous liquid (Eluent: petroleum ether/ethyl acetate = 20/1, E/Z = 85/15).

*tert*-butyl (*E*)-(4-(benzo[b]thiophen-2-yl)-1-phenylbut-3-en-2-yl)carbamate (39): <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.85 – 7.50 (m, 2H), 7.37 – 7.27 (m, 4H), 7.26 – 7.14 (m, 3H), 7.08 (s, 1H), 6.66 (d, *J* = 15.8 Hz, 1H), 6.07 (dd, *J* = 15.7, 5.5 Hz, 1H), 4.58 (s, 2H), 2.94 (d, *J* = 5.9 Hz, 2H), 1.42 (s, 9H).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 155.1, 142.0, 140.1, 138.8, 137.0, 132.2, 129.6, 128.5, 126.7, 124.7, 124.4, 124.2, 123.4, 122.8, 122.2, 79.7, 53.1, 41.8, 28.4.

HRMS (ESI) *calcd*. for C<sub>23</sub>H<sub>26</sub>O<sub>2</sub>NS<sup>+</sup> [M+H]<sup>+</sup>: 380.1679, *found*: 380.1679.



*tert*-butyl (*E*)-(4-(benzofuran-2-yl)-1-phenylbut-3-en-2-yl)carbamate (40): Following the general procedure B, using 20.0 mol% NaI, 20.0 mol% PPh<sub>3</sub>, 2.0 mL acetone, obtained in 48% yield as yellow viscous liquid (Eluent: petroleum ether/ethyl acetate = 20/1).

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.48 (d, J = 7.6 Hz, 1H), 7.41 (d, J = 8.1 Hz, 1H), 7.30 (dd, J = 14.1, 6.7 Hz, 2H), 7.25 - 7.14 (m, 5H), 6.49 (s, 1H), 6.46 - 6.32 (m, 2H), 4.82 - 4.47 (m, 2H), 2.93 (s, 2H), 1.41 (s, 9H).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 155.2, 154.7, 154.2, 137.1, 131.9, 129.6, 128.9, 128.5, 126.7, 124.5, 122.8, 120.9, 118.8, 110.9, 104.6, 79.7, 53.1, 41.7, 28.4 HRMS (ESI) *calcd.* for C<sub>23</sub>H<sub>26</sub>O<sub>3</sub>N<sup>+</sup> [M+H]<sup>+</sup>: 364.1907, *found*: 364.1907.



Following the general procedure B, using 20.0 mol% NaI, 20.0 mol% PPh<sub>3</sub>, 2.0 mL DMA, obtained in 60% yield as yellow viscous liquid (Eluent: petroleum ether/ethyl acetate = 20/1, E/Z = 76/24).

## *tert*-butyl (*E*)-(1-phenyl-4-(pyridin-3-yl)but-3-en-2-yl)carbamate (41):

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 8.70 – 8.35 (m, 2H), 8.01 – 7.47 (m, 2H), 7.35 – 7.27 (m, 2H), 7.25 – 7.19 (m, 3H), 6.53 – 6.36 (m, 1H), 6.30 – 6.11 (m, 1H), 4.65 (s, 2H), 2.94 (d, *J* = 6.4 Hz, 2H), 1.42 (s, 9H).

<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>) δ 155.1, 147.3, 136.9, 134.3, 133.8, 132.9, 129.5, 128.5, 126.8, 126.3, 123.7, 123.6, 79.8, 53.1, 41.7, 28.4.

HRMS (ESI) calcd. for C<sub>20</sub>H<sub>25</sub>O<sub>2</sub>N<sub>2</sub><sup>+</sup> [M+H]<sup>+</sup>: 325.1911, found: 325.1911.



*tert*-butyl (*E*)-(4-(5-methylfuran-2-yl)-1-phenylbut-3-en-2-yl)carbamate (42): Following the general procedure B, using 20.0 mol% NaI, 20.0 mol% PPh<sub>3</sub>, 2.0 mL acetone, obtained in 61% yield as yellow viscous liquid (Eluent: petroleum ether/ethyl acetate = 20/1).

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.32 – 7.29 (m, 1H), 7.29 – 7.26 (m, 1H), 7.25 – 7.22 (m, 1H), 7.22 – 7.18 (m, 2H), 6.23 – 6.14 (m, 1H), 6.09 – 5.97 (m, 2H), 5.96 – 5.88 (m, 1H), 4.53 (s, 2H), 3.01 – 2.79 (m, 2H), 2.28 (s, 3H), 1.40 (s, 9H).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 155.2, 151.9, 150.8, 137.4, 129.6, 128.4, 126.5, 118.9, 111.6, 109.1, 107.3, 79.5, 53.0, 41.9, 28.4, 13.7.

HRMS (ESI) *calcd*. for C<sub>20</sub>H<sub>26</sub>O<sub>3</sub>N<sup>+</sup> [M+H]<sup>+</sup>: 328.1907, *found*: 328.1907.



**benzyl 4-(2,2-diphenylvinyl)piperidine-1-carboxylate (44)**: Following the general procedure A, using 10.0 mol% NaI, 10.0 mol% PPh<sub>3</sub>, 2.0 mL acetone, obtained in 81% yield as colorless viscous liquid (Eluent: petroleum ether/ethyl acetate = 10/1). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.43 – 7.29 (m, 8H), 7.25 – 7.14 (m, 7H), 5.85 (d, *J* = 9.8 Hz, 1H), 5.12 (s, 2H), 4.13 (d, *J* = 10.3 Hz, 2H), 2.69 (t, *J* = 12.3 Hz, 2H), 2.36 – 2.21 (m, 1H), 1.64 (d, *J* = 12.4 Hz, 2H), 1.41 (dd, *J* = 22.3, 10.6 Hz, 2H). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 155.3, 142.3, 141.3, 140.1, 137.0, 133.2, 129.6, 128.5, 128.4, 128.2, 128.0, 127.9, 127.2, 127.2, 67.0, 43.6, 36.5, 32.1. HRMS (ESI) *calcd.* for C<sub>27</sub>H<sub>28</sub>O<sub>2</sub>N<sup>+</sup> [M+H]<sup>+</sup>: 398.2115, *found*: 398.2121.



(5-(4-methoxyphenyl)pent-1-ene-1,1-diyl)dibenzene (45): Following the general procedure A, using 10.0 mol% NaI, 10.0 mol% PPh<sub>3</sub>, 2.0 mL acetone, obtained in 60% yield as colorless liquid (Eluent: petroleum ether/ethyl acetate = 50/1).

<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  7.30 – 7.25 (m, 2H), 7.24 – 7.20 (m, 1H), 7.20 – 7.12 (m, 5H), 7.10 – 7.07 (m, 2H), 6.96 (d, *J* = 8.6 Hz, 2H), 6.71 (d, *J* = 8.6 Hz, 2H), 6.01 (t, *J* = 7.5 Hz, 1H), 3.69 (s, 3H), 2.49 – 2.43 (m, 2H), 2.08 (q, *J* = 7.4 Hz, 2H), 1.69 – 1.61 (m, 2H).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 156.6, 141.8, 140.8, 139.2, 133.4, 128.9, 128.7, 128.2, 127.1, 127.0, 126.2, 125.8, 125.8, 112.6, 54.2, 33.5, 30.9, 28.3.

HRMS (ESI) *calcd*. for C<sub>24</sub>H<sub>25</sub>O<sup>+</sup> [M+H]<sup>+</sup>: 329.1900, *found*: 329.1906.

## 5. Gram-scale reactions

#### A. Synthesis of benzyl (4,4-diphenylbut-3-en-2-yl)carbamate (3):



1,3-dioxoisoindolin-2-yl 2-(((benzyloxy)carbonyl)amino)propanoate (**2**, 1.5 eq., 7.5 mmol), NaI (10 mol%), PPh<sub>3</sub> (10 mol%) were placed in a 100 mL transparent Schlenk tube equipped with a stirring bar. The tube was evacuated and filled with argon (three times). To these solids, acetone (50.0 mL) and aryl ethene-1,1-diyldibenzene (**1**, 1.0 eq., 5.0 mmol) were added via a gastight syringe under argon atmosphere. The reaction mixture was stirred under irradiation with blue LEDs (Kessil, PR160-456 nm), maintained at approximately room temperature ( $28 \pm 2$  °C). After 24 h, the reaction mixture was transferred to a round bottom flask and concentrated on a rotary evaporator. The residue was purified with silica gel chromatography to give the product (white solid, 1.55 g, yield of 87%. Eluent: petroleum ether/ethyl acetate = 10/1).

### B. Synthesis of 2-(2,2-diphenylvinyl)tetrahydrofuran (31):



1,3-dioxoisoindolin-2-yl tetrahydrofuran-2-carboxylate (**31'**, 1.5 eq., 7.5 mmol), NaI (10 mol%), PPh<sub>3</sub> (10 mol%) were placed in a 100 mL transparent Schlenk tube equipped with a stirring bar. The tube was evacuated and filled with argon (three times). To these solids, acetone (50.0 mL) and aryl ethene-1,1-diyldibenzene (**1**, 1.0 eq., 5.0 mmol) were added via a gastight syringe under argon atmosphere. The reaction mixture was stirred under irradiation with blue LEDs (Kessil, PR160-456 nm), maintained at approximately room temperature ( $28 \pm 2$  °C). After 24 h, the reaction mixture was transferred to a round bottom flask and concentrated on a rotary

evaporator. The residue was purified with silica gel chromatography to give the product (viscous liquid, 0.9 g, yield of 72%. Eluent: petroleum ether/ethyl acetate = 50/1).

## 6. Experimental Studies on Mechanism



**benzyl 3-(3,3-diphenylallyl)pyrrolidine-1-carboxylate (43):** Following the general procedure A, using 20.0 mol% NaI, 20.0 mol% PPh<sub>3</sub>, 2.0 mL acetone, obtained in 57% yield as viscous liquid (Eluent: petroleum ether/ethyl acetate = 10/1). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.34 (s, 8H), 7.28 – 7.21 (m, 4H), 7.21 – 7.15 (m, 2H), 7.14 – 7.05 (m, 1H), 6.20 – 5.93 (m, 1H), 5.77 – 5.47 (m, 1H), 5.14 (s, 2H), 5.02 – 4.84 (m, 2H), 4.13 – 3.78 (m, 2H), 3.43 – 3.14 (m, 2H), 2.30 – 1.92 (m, 2H). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)  $\delta$  155.9, 141.5, 139.0, 136.9, 135.3, 129.7, 128.5, 128.4, 128.2, 128.0, 127.5, 127.3, 125.1, 124.9, 116.7, 67.1, 46.7, 46.0, 45.8, 33.0, 32.5. HRMS (ESI) *calcd.* for C<sub>27</sub>H<sub>27</sub>O<sub>2</sub>NNa<sup>+</sup> [M+Na]<sup>+</sup>: 420.1934, *found*: 420.1934.

## UV-Vis absorption spectroscopic measurements



The sample stock solutions were prepared with the same concentration used in the reaction. The solutions were prepared in the presence of air using acetone as solvent.



**Figure S2:** UV-Vis absorption spectra of the combination between different starting materials recorded in acetone as solvent

## 7. References

1. X. Lu, B. Xiao, L. Liu and Y. Fu, Chem. Eur. J. 2016, 22, 11161.

2. E. Pontiki, D. Hadjipavlou-Litina, K. Litinas, O. Nicolotti and A. Carotti, *Eur. J. Med. Chem.* 2011, **46**, 191.

## 8. NMR spectra

<sup>1</sup>H NMR spectrum of **benzyl (4,4-diphenylbut-3-en-2-yl)carbamate (3)**:





<sup>13</sup>C NMR spectrum of **2-(1,4,4-triphenylbut-3-en-2-yl)isoindoline-1,3-dione (4)**:



210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 f1 (ppm)

<sup>1</sup>H NMR spectrum of **benzyl (3,3-diphenylallyl)carbamate (5):** 



<sup>13</sup>C NMR spectrum of **benzyl (3,3-diphenylallyl)carbamate (5):** 





<sup>1</sup>H NMR spectrum of *tert*-butyl (4,4-diphenylbut-3-en-2-yl)carbamate (6):

<sup>13</sup>C NMR spectrum of *tert*-butyl (4,4-diphenylbut-3-en-2-yl)carbamate (6):



210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 fl (ppm)



<sup>1</sup>H NMR spectrum of *tert*-butyl 4-(((benzyloxy)carbonyl)amino)-6,6-diphenylhex-5-enoate (7):

<sup>13</sup>C NMR spectrum of *tert*-butyl 4-(((benzyloxy)carbonyl)amino)-6,6-diphenylhex-5-enoate (7):







<sup>13</sup>C NMR spectrum of *tert*-butyl (1-(4-iodophenyl)-4,4-diphenylbut-3-en-2-yl)carbamate (8):



<sup>1</sup>H NMR spectrum of *tert*-butyl (5-(methylthio)-1,1-diphenylpent-1-en-3-yl)carbamate (9):



<sup>13</sup>C NMR spectrum of *tert*-butyl (5-(methylthio)-1,1-diphenylpent-1-en-3-yl)carbamate (9):



<sup>1</sup>H NMR spectrum of **benzyl 2-(2,2-diphenylvinyl)pyrrolidine-1-carboxylate (10):** 



<sup>13</sup>C NMR spectrum of benzyl 2-(2,2-diphenylvinyl)pyrrolidine-1-carboxylate (10):



<sup>1</sup>H NMR spectrum of *tert*-butyl 2-(2,2-diphenylvinyl)pyrrolidine-1-carboxylate (11):



<sup>13</sup>C NMR spectrum of *tert*-butyl 2-(2,2-diphenylvinyl)pyrrolidine-1-carboxylate (11):





## <sup>1</sup>H NMR spectrum of *tert*-butyl 2-(2,2-diphenylvinyl)azetidine-1-carboxylate (12):

<sup>13</sup>C NMR spectrum of *tert*-butyl 2-(2,2-diphenylvinyl)azetidine-1-carboxylate (12):



<sup>110 100</sup> fl (ppm) -10 130 120 



## <sup>1</sup>H NMR spectrum of **benzyl (4-methyl-1,1-diphenylpent-1-en-3-yl)carbamate (13):**

<sup>13</sup>C NMR spectrum of **benzyl (4-methyl-1,1-diphenylpent-1-en-3-yl)carbamate (13):** 



210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 fl (ppm)



<sup>1</sup>H NMR spectrum of *tert*-butyl (1,1-diphenylhex-1-en-5-yn-3-yl)carbamate (14):

<sup>13</sup>C NMR spectrum of *tert*-butyl (1,1-diphenylhex-1-en-5-yn-3-yl)carbamate (14):





<sup>1</sup>H NMR spectrum of *N*-(1,4,4-triphenylbut-3-en-2-yl)pivalamide (15):

<sup>13</sup>C NMR spectrum of *N*-(1,4,4-triphenylbut-3-en-2-yl)pivalamide (15):



<sup>1</sup>H NMR spectrum of *tert*-butyl(1-((5-(methylthio)-1,1-diphenylpent-1-en-3-yl)amino)-1-oxo-3-phenylpropan-2-yl)carbamate (16):



<sup>1</sup>H NMR spectrum of *N*-(2-oxo-2-((1,4,4-triphenylbut-3-en-2-yl)amino)ethyl)benzamide (17):





<sup>1</sup>H NMR spectrum of *tert*-butyl (4,4-di-p-tolylbut-3-en-2-yl)carbamate (18):





## <sup>13</sup>C NMR spectrum of *tert*-butyl (4,4-di-p-tolylbut-3-en-2-yl)carbamate (18):







<sup>13</sup>C NMR spectrum of *tert*-butyl (4,4-bis(4-methoxyphenyl)but-3-en-2-yl)carbamate (19):

210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 fl (ppm)







<sup>13</sup>C NMR spectrum of benzyl (4,4-bis(4-fluorophenyl)but-3-en-2-yl)carbamate (20):

210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 fl (ppm)





<sup>13</sup>C NMR spectrum of benzyl (4,4-bis(4-chlorophenyl)but-3-en-2-yl)carbamate (21):



<sup>210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10</sup> fl (ppm)

## <sup>1</sup>H NMR spectrum of **benzyl (4,4-bis(4-bromophenyl)but-3-en-2-yl)carbamate (22):**







140 130 110 100 f1 (ppm) -10 

# <sup>1</sup>H NMR spectrum of *tert*-butyl(3-(benzyloxy)-1-((4,4-bis(4-methoxyphenyl)but-3-en-2-yl)amino)-1-oxobutan-2-yl)carbamate (23):



<sup>1</sup>H NMR spectrum of (3-phenoxyprop-1-ene-1,1-diyl)dibenzene (24):





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<sup>1</sup>H NMR spectrum of (3-(3-chlorophenoxy)but-1-ene-1,1-diyl)dibenzene (25):



<sup>1</sup>H NMR spectrum of (3-(2-iodophenoxy)pent-1-ene-1,1-diyl)dibenzene (26):

<sup>13</sup>C NMR spectrum of (3-(2-iodophenoxy)pent-1-ene-1,1-diyl)dibenzene (26):



<sup>1</sup>H NMR spectrum of **2-(4-((4,4-diphenylbut-3-en-2-yl)oxy)phenyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (27):** 



<sup>13</sup>C NMR spectrum of 2-(4-((4,4-diphenylbut-3-en-2-yl)oxy)phenyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (27):



<sup>110 100</sup> f1 (ppm) 210 200 190 180 170 160 150 140 130 120 90 80 70 60 50 40 30 20 10 0 -10



<sup>1</sup>H NMR spectrum of (3-ethoxyprop-1-ene-1,1-diyl)dibenzene (28):





<sup>1</sup>H NMR spectrum of (3-(benzyloxy)prop-1-ene-1,1-diyl)dibenzene (29):



<sup>13</sup>C NMR spectrum of (3-(benzyloxy)prop-1-ene-1,1-diyl)dibenzene (29):







<sup>13</sup>C NMR spectrum of 2-(2,2-diphenylvinyl)-2,3-dihydrobenzo[*b*][1,4]dioxine (30):





<sup>13</sup>C NMR spectrum of **2-(2,2-diphenylvinyl)tetrahydrofuran (31):** 



<sup>1</sup>H NMR spectrum of (3,3-diphenylallyl)(phenyl)sulfane (32):







<sup>1</sup>H NMR spectrum of *tert*-butyl (*E*)-(4-(4-methoxyphenyl)-1-phenylbut-3-en-2-yl)carbamate (33):



<sup>13</sup>C NMR spectrum of *tert*-butyl (*E*)-(4-(4-methoxyphenyl)-1-phenylbut-3-en-2-yl)carbamate (33):



<sup>210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10</sup> f1 (ppm)

<sup>1</sup>H NMR spectrum of *tert*-butyl (*E*)-(4-(3,4-dimethoxyphenyl)-1-phenylbut-3-en-2-yl)carbamate (34):







210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 f1 (ppm)

<sup>1</sup>H NMR spectrum of *tert*-butyl (*E*)-(4-(2-methoxyphenyl)-1-phenylbut-3-en-2-yl)carbamate (35):



<sup>13</sup>C NMR spectrum of *tert*-butyl (*E*)-(4-(2-methoxyphenyl)-1-phenylbut-3-en-2-yl)carbamate (35):



<sup>210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10</sup> f1 (ppm)

# <sup>1</sup>H NMR spectrum of *tert*-butyl (*E*)-(4-(4-(dimethylamino)phenyl)-1-phenylbut-3-en-2-yl)carbamate (36):



<sup>13</sup>C NMR spectrum of *tert*-butyl (*E*)-(4-(4-(dimethylamino)phenyl)-1-phenylbut-3-en-2-yl)carbamate (36):



<sup>210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10</sup> fl (ppm)

<sup>1</sup>H NMR spectrum of *tert*-butyl (*E*)-(4-(4-(diphenylamino)phenyl)-1-phenylbut-3-en-2-yl) carbamate (37):



<sup>13</sup>C NMR spectrum of *tert*-butyl (*E*)-(4-(4-(diphenylamino)phenyl)-1-phenylbut-3-en-2-yl)carbamate (37):





210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 f1 (ppm)





<sup>13</sup>C NMR spectrum of *tert*-butyl (*E*)-(1-phenyl-4-(thiophen-3-yl)but-3-en-2-yl)carbamate (38):



<sup>1</sup>H NMR spectrum of *tert*-butyl (*E*)-(4-(benzo[b]thiophen-2-yl)-1-phenylbut-3-en-2-yl)carbamate (39):



<sup>13</sup>C NMR spectrum of *tert*-butyl (*E*)-(4-(benzo[b]thiophen-2-yl)-1-phenylbut-3-en-2-yl)carbamate (39):



<sup>210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10</sup> fl (ppm)

<sup>1</sup>H NMR spectrum of *tert*-butyl (*E*)-(4-(benzofuran-2-yl)-1-phenylbut-3-en-2-yl)carbamate (40):



<sup>13</sup>C NMR spectrum of *tert*-butyl (*E*)-(4-(benzofuran-2-yl)-1-phenylbut-3-en-2-yl)carbamate (40):







<sup>13</sup>C NMR spectrum of *tert*-butyl (*E*)-(1-phenyl-4-(pyridin-3-yl)but-3-en-2-yl)carbamate (41):



<sup>1</sup>H NMR spectrum of *tert*-butyl (*E*)-(4-(5-methylfuran-2-yl)-1-phenylbut-3-en-2-yl)carbamate (42):



<sup>13</sup>C NMR spectrum of *tert*-butyl(*E*)-(4-(5-methylfuran-2-yl)-1-phenylbut-3-en-2-yl)carbamate (42):







<sup>13</sup>C NMR spectrum of **benzyl 3-(3,3-diphenylallyl)pyrrolidine-1-carboxylate (43):** 





<sup>13</sup>C NMR spectrum of **benzyl 4-(2,2-diphenylvinyl)piperidine-1-carboxylate (44):** 



210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 fl (ppm)



<sup>1</sup>H NMR spectrum of (5-(4-methoxyphenyl)pent-1-ene-1,1-diyl)dibenzene (45):

<sup>13</sup>C NMR spectrum of (5-(4-methoxyphenyl)pent-1-ene-1,1-diyl)dibenzene (45):

