# Supporting Information 

# $\alpha$-Carbamoylsulfides as N -Carbamoylimine Precursors in the Visible Light Photoredox-Catalyzed Synthesis of $\alpha, \alpha$ Disubstituted Amines 

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## I. General Information:

All reactions were carried out under argon atmosphere in oven dried glassware with magnetic stirring. Reagents were obtained from commercial supplier and used without further purification unless otherwise noted. All solvents used in the reactions were distilled from appropriate drying agents prior to use. Analytical thin layer chromatography (TLC) was purchased from Merck KGaA (silica gel 60 F254). Visualization was accomplished by irradiation with a UV light at 254 nm . Flash column chromatography was carried out using kieselgel 35-70 $\mu \mathrm{m}$ particle sized silica gel (200-400 mesh). Chromatography was performed using silica gel 60 (0.040-0.063 mm) from Merck.
${ }^{1} \mathrm{H}$ NMR and ${ }^{13} \mathrm{C}$ spectra were recorded with Bruker 500 MHz and 300 MHz instruments. Proton chemical shifts are reported in ppm ( $\delta$ ) relative to tetramethylsilane (TMS) with the solvent resonance employed as the internal standard $\left(\mathrm{CDCl}_{3}, \delta 7.26 \mathrm{ppm}\right)$. Data are reported as follows: chemical shift, multiplicity ( $\mathrm{s}=$ singlet, $\mathrm{br} \mathrm{s}=$ broad singlet, $\mathrm{d}=$ doublet, $\mathrm{t}=$ triplet, $\mathrm{q}=$ quartet, $\mathrm{qt}=$ quintuplet, $\mathrm{h}=$ hexuplet, $\mathrm{ht}=$ heptuplet, $\mathrm{m}=$ multiplet), coupling constants $(\mathrm{Hz})$ and integration. ${ }^{13} \mathrm{C}$ chemical shifts are reported in ppm from tetramethylsilane (TMS) with the solvent resonance as the internal standard ( $\mathrm{CDCl}_{3}, \delta 77.2 \mathrm{ppm}$ ).
Infrared spectra were recorded on neat samples, on a Perkin Elmer Spectrum BX FT-IR spectrometer and the characteristic IR absorption frequencies are reported in $\mathrm{cm}^{-1}$.
Melting points were recorded using Reichert melting point apparatus and temperatures were uncorrected.
Optical rotations were performed on a Jasco P-1010 polarimeter ( 589 nm ) using a $700-\mu \mathrm{L}$ cell with a path length of 1 dm .
Mass spectra were determined on an AEI MS-9 using electrospray ionization (ESI).
Visible light irradiations were performed with a Flexled INSPIRE LED lamp (3.6 W; $\lambda=465$ nm ).
$\mathrm{Ru}(\mathrm{bpy}))_{3}\left(\mathrm{PF}_{6}\right)_{2}$ was purchased from Sigma Aldrich. All other commercially available reagents and solvents were used without further purification. The $\alpha$-amidosulfides $\mathbf{1}$ were prepared according to literature procedure. ${ }^{1}$

[^0]
## II. Reaction Optimization:




3a Ru(bpy) $)_{3}\left(\mathrm{PF}_{6}\right)_{2}$
3b $\operatorname{Ir}(p p y)_{2}(\mathrm{dtb}-\mathrm{bpy}) \mathrm{PF}_{6}$
3c Eosin $Y$

| Entry | 1 | 3 | Additive (x eq) | Solvent | Yield (\%) ${ }^{\text {a,b }}$ |
| :---: | :---: | :---: | :---: | :---: | :---: |
| 1 | 1a | 3a | - | MeCN | 60 |
| 2 | 1a | 3a | HFIP (10.0) | MeCN | 77 |
| 3 | 1a | 3a | $t$-BuOH (10.0) | MeCN | 87 |
| 4 | 1a | 3b | $t$-BuOH (10.0) | MeCN | 70 |
| 5 | 1a | 3c | $t$-BuOH (10.0) | MeCN | $63^{c}$ |
| 6 | 1a | 3a | $t$-BuOH (10.0) | THF | 8 |
| 7 | 1a | 3a | $t$-BuOH (10.0) | $\mathrm{CH}_{2} \mathrm{Cl}_{2}$ | 6 |
| 8 | 1a | 3a | - | $t-\mathrm{BuOH}$ | 5 |
| 9 | 1a | 3a | $t$-BuOH (10.0) | MeCN | $60^{\text {d }}$ |
| 10 | 1a | 3a | $t$-BuOH (10.0) | MeCN | $90^{e}$ |
| 11 | 1a | 3a | $t$-BuOH (5.0) | MeCN | 77 |
| 12 | 1a | 3a | $t$-BuOH (20.0) | MeCN | 63 |
| 13 | 1a | 3a | $t$-BuOH (10.0) | MeCN | $67 f$ |
| 14 | 1a | 3a | $t$-BuOH (10.0) | MeCN | 769 |
| 15 | 1b | 3a | $t$-BuOH (10.0) | MeCN | 57 |
| 16 | 1c | 3a | $t$-BuOH (10.0) | MeCN | 63 |
| 17 | 1d | 3a | $t$-BuOH (10.0) | MeCN | 91 |
| 18 | 1 e | 3a | $t$-BuOH (10.0) | MeCN | -h |
| 19 | 1a | 3a | $t$-BuOH (10.0) | MeCN | -h,i |
| 20 | 1a | - | $t$-BuOH (10.0) | MeCN | -h |

${ }^{a}$ General conditions: $\mathbf{1}$ ( 0.10 mmol ), 1,3,5-Trimethoxybenzene $\mathbf{5 a}$ ( 0.15 mmol ), $\mathbf{3}$ ( 0.025 equiv), Additive ( x eq) in MeCN ( 1.0 mL ) irradiated at rt for 24 h . ${ }^{b}$ Yields referred to chromatographically pure product. ${ }^{\text {I Irradiated with }}$ Green LEDs. ${ }^{d} \mathrm{MeCN}(2 \mathrm{~mL})$ was used. ${ }^{e} \mathrm{MeCN}(0.5 \mathrm{~mL})$ was used. $f \mathrm{BrCCl}_{3}(0.025 \mathrm{mmol})$ was used as oxidative quencher. ${ }^{g} N, N^{\prime}$-dimethyl-4,4'-bipyridinium ( 0.015 mmol ) was used as oxidative quencher. ${ }^{h}$ Starting material was recovered. 'Without any irradiation.

## III. Mechanistic Studies:



| Entry | Control Conditions | Product |
| :---: | :---: | :---: |
| 1 | w/o photocatalyst 3 | $0 \%$ |
| 2 | w/o light | $0 \%$ |
| 3 | Standard Conditions, w/all | $90 \%$ |

Fig. S1. Control Experiments.


| Entry | Light On and Off Conditions | Product |
| :---: | :---: | :---: |
| 1 | on 1h | $8 \%$ |
| 2 | on 1h, off 23h | $30 \%$ |
| 3 | on 24h | $90 \%$ |

Fig. S2. Light on and off experiments.


| Entry | Degas Procedure | Ratio SM/Pa |
| :---: | :---: | :---: |
| 1 | Under Ar atmosphere $^{\mathrm{b}}$ | $<20: 1$ |
| 2 | Under Air atmosphere $^{\mathrm{b}}$ | $>1: 20$ |
| 3 | ${\text { Under } \mathrm{O}_{2} \text { atmosphere }^{\mathrm{b}}} \quad>1: 20$ |  |

a Determined by ${ }^{1} \mathrm{H}$ NMR.
b. Freeze-pump-thraw degassed solution (3 cycles) of $\left[\mathrm{Ru}(\mathrm{bpy})_{3}\left(\mathrm{PF}_{6}\right)_{2}\right]$ in $\mathrm{MeCN} / t$ - BuOH was used.

Fig. S3. Oxygen Effects.

## Control experiment:



Phenyl disulfide was observed in the case of $\mathbf{1 b}$ was used as $\alpha$-amidosufide. The NMR data is in accordance with the literature. ${ }^{2}$

1,2-diphenyldisulfane

${ }^{\mathbf{1}} \mathbf{H}$ NMR $\left(300 \mathrm{MHz}, \mathrm{CDCl}_{3}\right) \delta 7.54-7.50\left(\mathrm{~m}, 4 \mathrm{H}, \mathrm{CH}_{\mathrm{ar}}\right), 7.35-7.24(\mathrm{~m}, 6 \mathrm{H}, \mathrm{CH} \mathrm{ar})$ ppm.


[^1]
## IV. Electrochemical studies:

Electrochemical studies were performed using acetonitrile as a solvent, with N tetrabutylammonium hexafluorophosphate (Fluka, puriss.) as the supporting electrolyte. The substrate concentration was ca. 1 mM . A 2 mm platinum electrode was used as the working electrode, along with a $\mathrm{Ag}^{+} / \mathrm{Ag}\left(10^{-2} \mathrm{M}\right)$ reference electrode and a Pt wire counter electrode. The cell was connected to a PAR 273A potentiostat. The reference electrode was checked vs. ferrocene as recommended by IUPAC: the oxidation potential of ferrocene was measured at 0.08 V . As a consequence, one has to substract 0.08 V to the values read on the curves to obtain potentials $\mathrm{Vs} \mathrm{Fc}+/ \mathrm{Fc}$, and to add 0.32 V to the values read on the curves to obtain potentials Vs SCE.


Fig S4: Cyclic voltammogram of compound 1a. Oxidation potential is estimated (irreversible process) at +1.17 V Vs SCE which is in agreement with values published for related compounds. ${ }^{3}$

[^2]

Fig S5: Cyclic voltammograms of compound 1a (13mM) in presence of increasing amounts of $t$-BuOH. Top: whole cyclic voltamogramms, bottom: zoom on the region of interest.

## IV. General Procedure for compounds 6:

A flame-dried test tube, flushed with Argon, was charged with the corresponding $\alpha$ amidosulfide 1 ( $0.1 \mathrm{mmol}, 1.0 \mathrm{eq}$ ) and dissolved in $\mathrm{MeCN}(0.5 \mathrm{~mL})$ and $t$ - $\mathrm{BuOH}(0.1 \mathrm{~mL}, 10$ eq). $\mathrm{Ru}(\mathrm{bpy})_{3}\left(\mathrm{PF}_{6}\right)_{2}$ 3a ( $2.2 \mathrm{mg}, 2.50 \mathrm{~mol} \%$ ) then the corresponding nucleophile 5 ( 0.15 mmol, 1.50 eq ) was then added. The resultant reaction mixture was irradiated with blue LEDs during 24 h . Then, the reaction mixture was directly purified by flash chromatography on silica gel ( $n$-Heptane/EtOAc) to afford the corresponding pure compound $\mathbf{6 a - 6 x}$.

## V. Spectroscopic data for compounds 6:

Data of Compounds $\mathbf{6 a} \mathbf{a}^{\mathbf{4}}, \mathbf{6} \mathbf{c}^{\mathbf{4}}, \mathbf{6} \mathrm{e}^{\mathbf{4}}, \mathbf{6 l}^{\mathbf{4}}, \mathbf{6} \mathbf{m}^{\mathbf{4}}, \mathbf{6} \mathbf{n}^{\mathbf{4}}, \mathbf{6} \mathbf{o}^{\mathbf{5}}, \mathbf{6} \mathbf{v}^{\mathbf{6}}, \mathbf{6} \mathbf{w}^{\mathbf{7}}, \mathbf{6} \mathbf{x}^{\mathbf{4}}$ and $\mathbf{7}^{\mathbf{8}}$ were previously described.

Tert-butyl (3-phenyl-1-(2,4,6-trimethoxyphenyl)propyl)carbamate 6a


Molecular Weight: 401,50

Tert-butyl (1-(2,4,6-trimethoxyphenyl)octyl)carbamate 6b $1.92\left(\mathrm{~m}, 2 \mathrm{H}, \mathrm{CH}_{2}\right), 1.46\left(\mathrm{~s}, 9 \mathrm{H}, 3 \times \mathrm{CH}_{3}\right) \mathrm{ppm}$.

According to the general procedure, 6a was obtained as a colourless oil ( 36 mg , Isolated yield $90 \%$ ); ${ }^{1} \mathbf{H}$ NMR ( $300 \mathrm{MHz}, \mathrm{CDCl}_{3}$ ) $\delta 7.28-$ $7.16\left(\mathrm{~m}, 5 \mathrm{H}, 5 \times \mathrm{CH} \mathrm{Har}_{\text {) }}, 6.15\left(\mathrm{~s}, 2 \mathrm{H}, 2 \times \mathrm{CH}_{\mathrm{ar}}\right), 5.86\right.$ (br. d, $J=10.2 \mathrm{~Hz}$, $1 \mathrm{H}, \mathrm{NH}$ ), 5.43 (dd, 1H, J = 9.9 and $15.3 \mathrm{~Hz}, \mathrm{CH}$ ), $3.84\left(\mathrm{~s}, 3 \mathrm{H}, \mathrm{CH}_{3}\right), 3.83$ (s, $6 \mathrm{H}, 2 \times \mathrm{CH}_{3}$ ), 2.75-2.65 (m, 1H, CH2), 2.53-2.43 (m, 1H, CH $\mathrm{CH}_{2}$ ), 2.19-


Chemical Formula: $\mathrm{C}_{22} \mathrm{H}_{37} \mathrm{NO}_{5}$ Molecular Weight: 395,54

According to the general procedure, 6b was obtained as a colourless oil ( 33.5 mg , Isolated yield $85 \%$ ); ${ }^{1} \mathbf{H}$ NMR ( 300 MHz , $\mathrm{CDCl}_{3}$ ) $\delta 6.11\left(\mathrm{~s}, 2 \mathrm{H}, 2 \times \mathrm{CH}_{\mathrm{ar}}\right.$ ), 5.76 (br. d, $J=10.2 \mathrm{~Hz}, 1 \mathrm{H}, \mathrm{NH}$ ), $5.32-$ $5.24(\mathrm{~m}, 1 \mathrm{H}, \mathrm{CH}), 3.80\left(\mathrm{~s}, 6 \mathrm{H}, 2 \times \mathrm{CH}_{3}\right), 3.79\left(\mathrm{~s}, 3 \mathrm{H}, \mathrm{CH}_{3}\right), 1.82-1.65$ ( $\mathrm{m}, 2 \mathrm{H}, \mathrm{CH}$ ) , $1.42\left(\mathrm{~s}, 9 \mathrm{H}, 3 \times \mathrm{CH}_{3}\right.$ ), 1.30-1.19 (m, 10H, $5 \times \mathrm{CH}_{2}$ ), 0.85 ( $\mathrm{t}, J=6.5 \mathrm{~Hz}, 3 \mathrm{H}, \mathrm{CH}_{3}$ ) ppm; ${ }^{13} \mathrm{C}$ NMR ( $75 \mathrm{MHz}, \mathrm{CDCl}_{3}$ ) $\delta 160.0\left(C_{\mathrm{q}}\right)$, $158.7\left(C_{\mathrm{q}}\right), 155.6\left(C_{\mathrm{q}}\right), 111.9\left(C_{\mathrm{q}}\right), 91.5\left(C_{\mathrm{q}}\right), 91.1\left(3 \times C \mathrm{H}_{\mathrm{ar}}\right), 78.6\left(C_{\mathrm{q}}\right), 55.9\left(2 \times \mathrm{CH}_{3}\right), 55.4\left(\mathrm{CH}_{3}\right)$, $45.9(\mathrm{CH}), 36.0\left(\mathrm{CH}_{2}\right), 32.0\left(\mathrm{CH}_{2}\right), 29.6\left(\mathrm{CH}_{2}\right), 29.4\left(\mathrm{CH}_{2}\right), 28.7\left(3 \times \mathrm{CH}_{3}\right), 26.6\left(\mathrm{CH}_{2}\right), 22.8\left(\mathrm{CH}_{2}\right)$, $14.2\left(\mathrm{CH}_{3}\right)$ ppm; IR: $v\left(\right.$ neat, $\left.\mathrm{cm}^{-1}\right) 3458,2928,2855,1710,1607,1591,1493,1455,1418$, 1364, 1327, 1224, 1204, 1151, 1134, 1061, 1042, 952, 874, 812; ESI-HRMS (positive) [M+Na]+ Calc. for $\mathrm{C}_{22} \mathrm{H}_{37} \mathrm{NO}_{5} \mathrm{Na} 418.2564$, found 418.2553.

Tert-butyl (2-methyl-1-(2,4,6-trimethoxyphenyl)propyl)carbamate 6c


hemical Formula: $\mathrm{C}_{18} \mathrm{H}_{29} \mathrm{NO}_{5}$ Molecular Weight: 339,43

According to the general procedure, $\mathbf{6 c}$ was obtained as a colourless oil ( 17 mg , Isolated yield $50 \%$ ); ${ }^{1} \mathbf{H}$ NMR ( $300 \mathrm{MHz}, \mathrm{CDCl}_{3}$ ) $\delta 6.12$ (s, $2 \mathrm{H}, 2 \times \mathrm{CH}_{\mathrm{ar}}$ ), 5.74 (br. d, $J=10.6 \mathrm{~Hz}, 1 \mathrm{H}, \mathrm{N} H$ ), $4.98(\mathrm{t}, J=10.3 \mathrm{~Hz}, 1 \mathrm{H}$, CH ), $3.80\left(\mathrm{~s}, 3 \mathrm{H}, \mathrm{CH}_{3}\right), 3.79\left(\mathrm{~s}, 6 \mathrm{H}, 2 \times \mathrm{CH}_{3}\right), 2.10-1.95(\mathrm{~m}, 1 \mathrm{H}, \mathrm{CH})$, $\left.1.42\left(\mathrm{~s}, 9 \mathrm{H}, 3 \times \mathrm{CH}_{3}\right), 1.00(\mathrm{~d}, J=6.8 \mathrm{~Hz}, 3 \mathrm{H}, \mathrm{CH})_{3}\right), 0.69(\mathrm{~d}, J=6.8 \mathrm{~Hz}$, $\left.3 \mathrm{H}, \mathrm{CH}_{3}\right) \mathrm{ppm}$.

[^3]


According to the general procedure, 6d was obtained as a colourless oil ( 18.5 mg , Isolated yield $55 \%$ ); ${ }^{1} \mathbf{H}$ NMR ( $300 \mathrm{MHz}, \mathrm{CDCl}_{3}$ ) $\delta 6.14$ ( $\mathrm{s}, 2 \mathrm{H}, 2 \times \mathrm{CH}_{\mathrm{ar}}$ ), $5.91(\mathrm{~d}, J=10.2 \mathrm{~Hz}, 1 \mathrm{H}, \mathrm{NH}$ ), $4.67(\mathrm{t}, J=9.7 \mathrm{~Hz}, 1 \mathrm{H}$, CH ), 3.82 ( $\mathrm{s}, 6 \mathrm{H}, 2 \times \mathrm{CH}_{3}$ ), $3.80\left(\mathrm{~s}, 3 \mathrm{H}, \mathrm{CH}_{3}\right), 1.43\left(\mathrm{~s}, 9 \mathrm{H}, 3 \times \mathrm{CH}_{3}\right)$, 1.36-1.20 (m, 1H, CH), 0.56-0.18 (m, 4H, $2 \times \mathrm{CH}_{2}$ ) ppm; ${ }^{13} \mathrm{C}$ NMR ( 75 $\left.\mathrm{MHz}, \mathrm{CDCl}_{3}\right) \delta 160.2\left(C_{\mathrm{q}}\right), 159.7\left(C_{\mathrm{q}}\right), 158.7\left(C_{\mathrm{q}}\right), 115.8\left(C_{\mathrm{q}}\right), 111.8$ $\left(C_{q}\right), 91.7(\mathrm{CH}), 91.2(\mathrm{CH}), 78.7\left(C_{q}\right), 55.9\left(2 \times \mathrm{CH}_{3}\right), 55.4\left(\mathrm{CH}_{3}\right), 50.1(\mathrm{CH}), 28.7\left(3 \times \mathrm{CH}_{3}\right), 17.2$ $(\mathrm{CH}), 3.8\left(\mathrm{CH}_{2}\right), 3.3\left(\mathrm{CH}_{2}\right) \mathrm{ppm}$; IR: $v\left(\right.$ neat, $\left.\mathrm{cm}^{-1}\right) 3460,3002,2975,2939,2839,1709,1608$, 1592, 1493, 1455, 1418, 1390, 1365, 1329, 1223, 1204, 1150, 1111, 1041, 1017, 957, 928, 880, 813; ESI-HRMS (positive) [M+Na]+ Calc. for $\mathrm{C}_{18} \mathrm{H}_{27} \mathrm{NO}_{5} \mathrm{Na} 360.1781$, found 360.1784 .

Tert-butyl (2-(benzyloxy)-1-(2,4,6-trimethoxyphenyl)ethyl)carbamate $\mathbf{6 e}$


Chemical Formula: $\mathrm{C}_{23} \mathrm{H}_{31} \mathrm{NO}_{6}$ Molecular Weight: 417,50

According to the general procedure, $\mathbf{6 e}$ was obtained as a colourless oil ( 24.0 mg , Isolated yield $58 \%$ ); ${ }^{1} \mathbf{H}$ NMR ( $300 \mathrm{MHz}, \mathrm{CDCl}_{3}$ ) $\delta 7.30-$ $7.23(\mathrm{~m}, 5 \mathrm{H}, 5 \times \mathrm{CH}$ ar) $) 6.12\left(\mathrm{~s}, 2 \mathrm{H}, 2 \times \mathrm{CH}_{\mathrm{ar}}\right), 5.80-5.72(\mathrm{~m}, 2 \mathrm{H}, \mathrm{CH}$, $\mathrm{N} H$ ), 4.64 (br. d, $J=11.8 \mathrm{~Hz}, 1 \mathrm{H}, \mathrm{CH}_{2}$ ), 4.55 (br. d, $J=11.6 \mathrm{~Hz}, 1 \mathrm{H}$, $\mathrm{CH}_{2}$ ), $3.80\left(\mathrm{~s}, 9 \mathrm{H}, 3 \times \mathrm{CH}_{3}\right), 3.73-3.68\left(\mathrm{~m}, 1 \mathrm{H}, \mathrm{CH}_{2}\right), 3.57-3.53(\mathrm{~m}, 1 \mathrm{H}$, $\mathrm{CH}_{2}$ ), $1.44\left(\mathrm{~s}, 9 \mathrm{H}, 3 \times \mathrm{CH}_{3}\right) \mathrm{ppm}$.

Tert-butyl (3-phenyl-1-(2,4,6-trimethoxyphenyl)prop-2-yn-1-yl)carbamate $\mathbf{6 f}$


Chemical Formula: $\mathrm{C}_{23} \mathrm{H}_{27} \mathrm{NO}_{5}$ Molecular Weight: 397,47

According to the general procedure, $\mathbf{6 f}$ was obtained as a colourless oil ( 26 mg , Isolated yield $65 \%$ ); ${ }^{1} \mathbf{H}$ NMR ( $300 \mathrm{MHz}, \mathrm{CDCl}_{3}$ ) $\delta 7.40-$ $7.34(\mathrm{~m}, 2 \mathrm{H}, 2 \times \mathrm{CH} \mathrm{ar}), 7.26-7.22\left(\mathrm{~m}, 3 \mathrm{H}, 3 \times \mathrm{CH}_{\mathrm{ar}}\right), 6.39$ (br. d, $J=$ $10.2 \mathrm{~Hz}, 1 \mathrm{H}, \mathrm{CH}$ ), $6.16\left(\mathrm{~s}, 2 \mathrm{H}, 2 \times \mathrm{CH}_{\mathrm{ar}}\right.$ ), 5.94 (br. d, $J=10.0 \mathrm{~Hz}, 1 \mathrm{H}$, $\mathrm{N} H$ ), $3.89\left(\mathrm{~s}, 6 \mathrm{H}, 2 \times \mathrm{CH}_{3}\right), 3.81\left(\mathrm{~s}, 3 \mathrm{H}, \mathrm{CH}_{3}\right), 1.45\left(\mathrm{~s}, 9 \mathrm{H}, 3 \times \mathrm{CH}_{3}\right)$ ppm; ${ }^{13}$ C NMR ( $75 \mathrm{MHz}, \mathrm{CDCl}_{3}$ ) $\delta 161.1\left(C_{\mathrm{q}}\right), 158.6\left(2 \times C_{\mathrm{q}}\right), 155.0$ $\left(C_{\mathrm{q}}\right), 131.9(2 \times C \mathrm{H}), 128.2(\mathrm{CH}), 127.9(2 \times C \mathrm{H}), 123.8\left(C_{\mathrm{q}}\right), 109.5\left(C_{\mathrm{q}}\right), 91.6(2 \times C \mathrm{H}), 90.0\left(C_{\mathrm{q}}\right)$, $80.3\left(C_{q}\right), 79.6\left(C_{q}\right), 56.3\left(2 \times \mathrm{CH}_{3}\right), 55.5\left(\mathrm{CH}_{3}\right), 37.2(\mathrm{CH}), 28.6\left(3 \times \mathrm{CH}_{3}\right) \mathrm{ppm}$; IR: $v$ (neat, $\left.\mathrm{cm}^{-1}\right)$ 3454, 2973, 2940, 2841, 1709, 1608, 1594, 1488, 1455, 1419, 1392, 1366, 1322, 1276, 1222, 1205, 1152, 1121, 1042, 1017, 951, 912, 873, 814, 758, 732, 692; ESI-HRMS (positive) [M+Na]+ Calc. for $\mathrm{C}_{23} \mathrm{H}_{27} \mathrm{NO}_{5} \mathrm{Na} 420.1781$, found 420.1784 .


Chemical Formula: $\mathrm{C}_{25} \mathrm{H}_{27} \mathrm{NO}_{4}$ Molecular Weight: 405,49

According to the general procedure, $\mathbf{6 g}$ was obtained as a colourless oil ( 30 mg , Isolated yield $74 \%$ ); ${ }^{1} \mathbf{H}$ NMR ( $300 \mathrm{MHz}, \mathrm{CDCl}_{3}$ ) $\delta 7.77-$ 7.74 (m, 2H, $2 \times$ CHar), 7.66 (br. d, $J=9.7 \mathrm{~Hz}, 1 \mathrm{H}, \mathrm{N} H$ ), 7.50-7.39 (m, $3 \mathrm{H}, 3 \times \mathrm{CH}_{\mathrm{ar}}$ ), $7.26-7.11\left(\mathrm{~m}, 5 \mathrm{H}, 5 \times \mathrm{CH}_{\mathrm{ar}}\right.$ ), $6.17\left(\mathrm{~s}, 2 \mathrm{H}, 2 \times \mathrm{CH}_{\mathrm{ar}}\right), 6.03-$ $5.94(\mathrm{~m}, 1 \mathrm{H}, \mathrm{CH}), 3.87\left(\mathrm{~s}, 6 \mathrm{H}, 2 \times \mathrm{CH}_{3}\right), 3.82\left(\mathrm{~s}, 3 \mathrm{H}, \mathrm{CH}_{3}\right), 2.79-2.69$ ( $\mathrm{m}, 1 \mathrm{H}, \mathrm{CH}_{2}$ ), 2.61-2.51 (m, 1H, CH2 $), 2.29-2.03\left(\mathrm{~m}, 2 \mathrm{H}, \mathrm{CH}_{2}\right) \mathrm{ppm} ;$ ${ }^{13}$ C NMR ( $75 \mathrm{MHz}, \mathrm{CDCl}_{3}$ ) $\delta 166.2\left(C_{\mathrm{q}}\right), 160.5\left(2 \times C_{\mathrm{q}}\right), 158.9\left(C_{\mathrm{q}}\right)$, $142.6\left(C_{q}\right), 135.6\left(C_{q}\right), 131.1\left(\mathrm{CH}_{\mathrm{ar}}\right), 128.6\left(2 \times \mathrm{CH}_{\mathrm{ar}}\right), 128.5\left(2 \times \mathrm{CH}_{\mathrm{ar}}\right)$, $128.3\left(2 \times C H_{a r}\right), 127.0\left(2 \times C H_{\mathrm{ar}}\right), 125.7\left(\mathrm{CHar}_{\mathrm{ar}}\right), 110.5\left(\mathrm{C}_{\mathrm{q}}\right), 91.3\left(2 \times \mathrm{CH}_{\mathrm{ar}}\right), 56.0\left(2 \times \mathrm{CH}_{3}\right), 55.5$ $\left(\mathrm{CH}_{3}\right), 45.2(\mathrm{CH}), 37.3\left(\mathrm{CH}_{2}\right), 33.0\left(\mathrm{CH}_{2}\right)$ ppm; IR: $v$ (neat, $\left.\mathrm{cm}^{-1}\right) 3444,2940,2839,1652,1605$, 1520, 1488, 1454, 1418, 1361, 1330, 1250, 1224, 1204, 1148, 1118, 1060, 1031, 950, 918, 843, 813, 766, 730, 699; ESI-HRMS (positive) [M+Na]+ Calc. for $\mathrm{C}_{25} \mathrm{H}_{27} \mathrm{NO}_{4} \mathrm{Na} 428.1832$, found 428.1841 .

4-methoxy-N-(3-phenyl-1-(2,4,6-trimethoxyphenyl)propyl)benzamide $\mathbf{6 h}$

Chemical Formula: $\mathrm{C}_{26} \mathrm{H}_{29} \mathrm{NO}_{5}$ Molecular Weight: 435,52

According to the general procedure, 6h was obtained as a colourless oil ( 35 mg , Isolated yield $80 \%$ ); ${ }^{1} \mathbf{H}$ NMR ( $300 \mathrm{MHz}, \mathrm{CDCl}_{3}$ ) $\delta 7.74-$ $7.70\left(\mathrm{~m}, 2 \mathrm{H}, 2 \times \mathrm{CH}_{\mathrm{ar}}\right.$ ), 7.57 (br. d, $J=9.7 \mathrm{~Hz}, 1 \mathrm{H}, \mathrm{NH}$ ), $7.26-7.10(\mathrm{~m}$, $5 \mathrm{H}, 5 \times \mathrm{CH}_{\mathrm{ar}}$ ), 6.94-6.89 (m, 2H, $2 \times \mathrm{CH}_{\mathrm{ar}}$ ), $6.16\left(\mathrm{~s}, 2 \mathrm{H}, 2 \times \mathrm{CH}_{\mathrm{ar}}\right), 6.01-$ 5.93 (m, 1H, CH), $3.87\left(\mathrm{~s}, 6 \mathrm{H}, 2 \times \mathrm{CH}_{3}\right.$ ), $3.84\left(\mathrm{~s}, 3 \mathrm{H}, \mathrm{CH}_{3}\right), 3.81(\mathrm{~s}, 3 \mathrm{H}$, $\mathrm{CH}_{3}$ ), 2.84-2.68 (m, 1H, CH2), 2.60-2.50 (m, 1H, CH2), 2.28-2.01 (m, $\left.2 \mathrm{H}, \mathrm{CH}_{2}\right) \mathrm{ppm} ;{ }^{13} \mathrm{C}$ NMR ( $75 \mathrm{MHz}, \mathrm{CDCl}_{3}$ ) $\delta 165.7\left(C_{\mathrm{q}}\right), 161.9\left(C_{\mathrm{q}}\right)$, $160.4\left(2 \times C_{q}\right), 158.9\left(C_{q}\right), 142.7\left(C_{q}\right), 128.7\left(2 \times C H_{\text {ar }}\right), 128.4(2 \times$ CHar), $128.3\left(2 \times \mathrm{CH}_{\mathrm{ar}}\right), 127.9\left(\mathrm{C}_{\mathrm{q}}\right), 125.6\left(\mathrm{CH}_{\mathrm{ar}}\right), 113.7\left(2 \times \mathrm{CH}_{\mathrm{ar}}\right)$,
$110.7\left(C_{q}\right), 91.3\left(2 \times C H_{\text {ar }}\right), 56.0\left(2 \times \mathrm{CH}_{3}\right), 55.5\left(2 \times \mathrm{CH}_{3}\right), 45.0(\mathrm{CH}), 37.4\left(\mathrm{CH}_{2}\right), 33.0\left(\mathrm{CH}_{2}\right)$ ppm; IR: $v$ (neat, $\mathrm{cm}^{-1}$ ) 3448, 2938, 2839, 1651, 1605, 1523, 1492, 1454, 1418, 1360, 1330, 1306, 1251, 1224, 1204, 1176, 1148, 1118, 1059, 1031, 950, 910, 844, 813, 766, 731, 699; ESI-HRMS (positive) [M+Na]+ Calc. for $\mathrm{C}_{26} \mathrm{H}_{29} \mathrm{NO}_{5} \mathrm{Na} 458.1938$, found 458.1938.


According to the general procedure, $\mathbf{6 i}$ was obtained as a colourless oil ( 30 mg , Isolated yield $69 \%$ ); ${ }^{1} \mathbf{H}$ NMR ( $300 \mathrm{MHz}, \mathrm{CDCl}_{3}$ ) $\delta 7.39-$ $7.11\left(\mathrm{~m}, 10 \mathrm{H}, 10 \times \mathrm{CH}_{\mathrm{ar}}\right), 6.13(\mathrm{~s}, 2 \mathrm{H}, 2 \times \mathrm{CH} \mathrm{ar}), 6.08(\mathrm{~d}, J=10.2 \mathrm{~Hz}$, $1 \mathrm{H}, \mathrm{N} H), 5.53-5.45(\mathrm{~m}, 1 \mathrm{H}, \mathrm{CH}), 5.16-5.02\left(\mathrm{~m}, 2 \mathrm{H}, \mathrm{CH}_{2}\right), 3.81(\mathrm{~s}, 9 \mathrm{H}, 3$ $\times \mathrm{CH}_{3}$ ), 2.74-2.64 (m, 1H, CH2), 2.54-2.44 (m, 1H, CH2), 2.20-1.94 (m, $\left.2 \mathrm{H}, \mathrm{CH}_{2}\right) \mathrm{ppm} ;{ }^{13} \mathrm{C}$ NMR ( $75 \mathrm{MHz}, \mathrm{CDCl}_{3}$ ) $\delta 160.4\left(2 \times C_{\mathrm{q}}\right), 158.7\left(\mathrm{C}_{\mathrm{q}}\right)$, $156.2\left(C_{\mathrm{q}}\right), 142.6\left(C_{\mathrm{q}}\right), 137.0\left(C_{\mathrm{q}}\right), 128.6\left(2 \times \mathrm{CH}_{\mathrm{ar}}\right), 128.5\left(2 \times C \mathrm{H}_{\mathrm{ar}}\right)$, $128.3\left(2 \times C H_{\mathrm{ar}}\right), 128.2\left(2 \times C \mathrm{H}_{\mathrm{ar}}\right), 125.6\left(2 \times C \mathrm{H}_{\mathrm{ar}}\right), 110.7\left(C_{\mathrm{q}}\right), 91.1\left(2 \times C \mathrm{H}_{\mathrm{ar}}\right), 66.6\left(\mathrm{CH}_{2}\right), 55.9$ $\left(\mathrm{CH}_{3}\right), 55.5\left(2 \times \mathrm{CH}_{3}\right), 46.7(\mathrm{CH}), 37.4\left(\mathrm{CH}_{2}\right), 33.0\left(\mathrm{CH}_{2}\right)$ ppm; IR: $v$ (neat, $\left.\mathrm{cm}^{-1}\right) 3444,2939$, 2839, 1717, 1607, 1592, 1496, 1454, 1419, 1330, 1204, 1149, 1118, 1038, 950, 814, 740, 698; ESI-HRMS (positive) [M+Na]+ Calc. for $\mathrm{C}_{26} \mathrm{H}_{29} \mathrm{NO}_{5} \mathrm{Na} 458.1938$, found 458.1945 .

Tert-butyl (((R)-2,2-dimethyl-1,3-dioxolan-4-yl)(2,4,6-trimethoxyphenyl)methyl)carbamate 6j


According to the general procedure, $\mathbf{6 j}$ was obtained as a colourless oil ( 20 mg , Isolated yield $50 \%$ ); dr>95:5; ${ }^{1} \mathbf{H}$ NMR ( $300 \mathrm{MHz}, \mathrm{CDCl}_{3}$ ) $\delta 6.13$ (s, 1H, CHar), $6.11(\mathrm{~s}, 1 \mathrm{H}, \mathrm{CH}$ ar), 5.83-5.77 (m, 1H, NH), 5.46$5.35(\mathrm{~m}, 1 \mathrm{H}, \mathrm{CH}), 4.38-4.32\left(\mathrm{~m}, 1 \mathrm{H}, \mathrm{CH}_{2}\right), 4.07-3.98\left(\mathrm{~m}, 1 \mathrm{H}, \mathrm{CH}_{2}\right)$, $3.82\left(\mathrm{~s}, 6 \mathrm{H}, 2 \times \mathrm{CH}_{3}\right), 3.79\left(\mathrm{~s}, 3 \mathrm{H}, \mathrm{CH}_{3}\right), 3.75-3.69\left(\mathrm{~m}, 1 \mathrm{H}, \mathrm{CH}_{2}\right), 1.43$ (s, 9H, $3 \times \mathrm{CH}_{3}$ ), 1.60-1.29 (m, 6H, $2 \times \mathrm{CH}_{3}$ ) ppm; ${ }^{13}$ C NMR ( 75 MHz , $\left.\mathrm{CDCl}_{3}\right) \delta 160.8\left(C_{\mathrm{q}}\right), 159.7\left(C_{\mathrm{q}}\right), 159.1\left(C_{\mathrm{q}}\right), 113.2\left(C_{\mathrm{q}}\right), 108.7\left(C_{\mathrm{q}}\right), 91.2\left(2 \times C \mathrm{H}_{\mathrm{ar}}\right), 78.0\left(C_{\mathrm{q}}\right), 69.2$ $\left(C_{q}\right), 66.9\left(\mathrm{CH}_{2}\right), 56.0\left(\mathrm{CH}_{3}\right), 55.8\left(\mathrm{CH}_{3}\right), 55.5\left(\mathrm{CH}_{3}\right), 49.0(\mathrm{CH}) 48.0(\mathrm{CH}), 28.6\left(3 \times \mathrm{CH}_{3}\right), 26.0$ $\left(\mathrm{CH}_{3}\right), 25.8\left(\mathrm{CH}_{3}\right) \mathrm{ppm}$; IR: $v$ (neat, $\left.\mathrm{cm}^{-1}\right) 3457,2980,2937,1710,1607,1592,1495,1455$, 1418, 1367, 1220, 1204, 1152, 1131, 1060, 1043, 952, 913, 814, 732; ESI-HRMS (positive) $[\mathrm{M}+\mathrm{Na}]^{+}$Calc. for $\mathrm{C}_{23} \mathrm{H}_{27} \mathrm{NO}_{5} \mathrm{Na} 420.1993$, found 420.1987.

Benzyl (2R)-2-(((tert-butoxycarbonyl)amino)(2,4,6-trimethoxyphenyl)methyl)pyrrolidine-1carboxylate 6k

According to the general procedure, $\mathbf{6 k}$ was obtained as a colourless oil ( 31 mg , Isolated yield 62\%); dr 3:1; ${ }^{\mathbf{1}} \mathbf{H}$ NMR (300 $\mathrm{MHz}, \mathrm{CDCl}_{3}$ ) (mixture of diastereoisomers and rotamers) $\delta 7.52-$ 7.14 (m, 7H, CHar), 6.28-5.78 (m, 2H, CH, NH), 5.19-4.73 (m, 2H, $\mathrm{CH}_{2}$ ), 4.34-4.15 (m, 1H, CH), 3.79-3.58 (m, 9H, $3 \times \mathrm{CH}_{3}$ ), 3.49-3.11 ( $\mathrm{m}, 2 \mathrm{H}, \mathrm{CH}_{2}$ ), 2.11-1.44 (m, 4H, $2 \times \mathrm{CH}_{2}$ ), 1.39-1.29 (m, $9 \mathrm{H}, 3 \times \mathrm{CH}_{3}$ ) ppm; ${ }^{13}$ C NMR ( $75 \mathrm{MHz}, \mathrm{CDCl}_{3}$ ) (mixture of diastereoisomers and rotamers) $\delta$ 160.8-158.5 $\left(2 \times C_{q}\right)$, 156.4-155.1 $\left(2 \times C_{q}\right)$, 137.6-137.0 $\left(C_{q}\right)$, 129.4-127.5 (5 $\times$ $\left.C \mathrm{H}_{\mathrm{ar}}\right)$, 109.2-108.5 $\left(\mathrm{C}_{\mathrm{q}}\right)$, 91.1-90.7 $\left(2 \times \mathrm{CH}_{\mathrm{ar}}\right)$, 79.1-78.5 $\left(\mathrm{C}_{\mathrm{q}}\right)$, 68.0-65.0 $\left(\mathrm{CH}_{2}\right)$, 60.9-59.8 ( CH$)$, 56.1-54.7 $\left(3 \times \mathrm{CH}_{3}\right), 48.7-47.0(\mathrm{CH}), 46.4-46.0\left(\mathrm{CH}_{2}\right), 28.7-28.6\left(3 \times \mathrm{CH}_{3}\right), 28.5-27.9\left(\mathrm{CH}_{2}\right)$, 23.4-22.3 $\left(\mathrm{CH}_{2}\right) \mathrm{ppm}$; IR: $v$ (neat, $\left.\mathrm{cm}^{-1}\right) 3501,2945,1718,1596,1453,1357,1216,1145$, 1113, 1002, 945, 801, 737, 673; ESI-HRMS (positive) [M+Na]+ Calc. for $\mathrm{C}_{27} \mathrm{H}_{36} \mathrm{~N}_{2} \mathrm{O}_{7} \mathrm{Na}$ 523.2420, found 523.2437.



Chemical Formula: $\mathrm{C}_{21} \mathrm{H}_{27} \mathrm{NO}_{5}$ Molecular Weight: 373,45

According to the general procedure, 61 was obtained as a white foam ( 33 mg , Isolated yield $88 \%$ ); ${ }^{\mathbf{1}} \mathbf{H}$ NMR ( $300 \mathrm{MHz}, \mathrm{CDCl}_{3}$ ) $\delta$ 7.26-7.11 (m, 5H, $5 \times \mathrm{CH}_{\text {ar }}$ ), 6.59 (br. d, $J=10.1 \mathrm{~Hz}, 1 \mathrm{H}, \mathrm{CH}$ ), 6.25 (br. d, $J=10.2$ $\mathrm{Hz}, 1 \mathrm{H}, \mathrm{NH}$ ), $6.15\left(\mathrm{~s}, 2 \mathrm{H}, 2 \times \mathrm{CH}_{\mathrm{ar}}\right.$ ), $3.81\left(\mathrm{~s}, 3 \mathrm{H}, \mathrm{CH}_{3}\right), 3.77(\mathrm{~s}, 6 \mathrm{H}, 2 \times$ $\mathrm{CH}_{3}$ ), 1.47 ( $\mathrm{s}, 9 \mathrm{H}, \mathrm{CH}_{3}$ ) ppm.

Tert-butyl (furan-2-yl(2,4,6-trimethoxyphenyl)methyl)carbamate 6m



Chemical Formula: $\mathrm{C}_{19} \mathrm{H}_{25} \mathrm{NO}_{6}$ Molecular Weight: 363,41

According to the general procedure, $\mathbf{6 m}$ was obtained as a colourless oil ( 21.5 mg , Isolated yield $59 \%$ ); ${ }^{\mathbf{1}} \mathbf{H}$ NMR ( 300 MHz , $\mathrm{CDCl}_{3}$ ) $\delta 7.27$ (s, 1H, CH $\mathrm{ar}^{\text {r }}$, 6.57 (br. d, $J=10.2 \mathrm{~Hz}, 1 \mathrm{H}, \mathrm{CH}$ ), 6.22 (br. $\mathrm{s}, 1 \mathrm{H}, \mathrm{CH}_{\mathrm{ar}}$ ), $6.15\left(\mathrm{~s}, 2 \mathrm{H}, 2 \times \mathrm{CH}_{\mathrm{ar}}\right.$ ), $6.10(\mathrm{br} . \mathrm{s}, 1 \mathrm{H}, \mathrm{NH}$ ), 5.92 (br. s, 1 H , $\mathrm{CH}_{\mathrm{ar}}$ ), 3.81 ( $\mathrm{s}, 3 \mathrm{H}, \mathrm{CH}_{3}$ ), $3.79\left(\mathrm{~s}, 6 \mathrm{H}, 2 \times \mathrm{CH}_{3}\right), 1.45\left(\mathrm{~s}, 9 \mathrm{H}, 3 \times \mathrm{CH}_{3}\right)$ ppm.

Tert-butyl ((4-(trifluoromethyl)phenyl)(2,4,6-trimethoxyphenyl)methyl)carbamate $\mathbf{6 n}$



Chemical Formula: $\mathrm{C}_{22} \mathrm{H}_{26} \mathrm{~F}_{3} \mathrm{NO}_{5}$ Molecular Weight: 441,45

According to the general procedure, $\mathbf{6 n}$ was obtained as a white foam ( 33 mg , Isolated yield $75 \%$ ); ${ }^{\mathbf{1}} \mathbf{H}$ NMR ( $300 \mathrm{MHz}, \mathrm{CDCl}_{3}$ ) $\delta$ 7.48 (d, $J=8.4 \mathrm{~Hz}, 2 \mathrm{H}, 2 \times \mathrm{CH}_{\mathrm{ar}}$ ), $7.35\left(\mathrm{~d}, J=8.4 \mathrm{~Hz}, 2 \mathrm{H}, 2 \times \mathrm{CH}_{\mathrm{ar}}\right.$ ), 6.60 (br. d, $J=9.9 \mathrm{~Hz}, 1 \mathrm{H}, \mathrm{CH}$ ), 6.20 (br. d, $J=9.9 \mathrm{~Hz}, 1 \mathrm{H}, \mathrm{N} H$ ), 6.15 ( $\mathrm{s}, 2 \mathrm{H}, 2 \times \mathrm{CH}_{\mathrm{ar}}$ ), $3.80\left(\mathrm{~s}, 3 \mathrm{H}, \mathrm{CH}_{3}\right), 3.77\left(\mathrm{~s}, 6 \mathrm{H}, 2 \times \mathrm{CH}_{3}\right), 1.47(\mathrm{~s}, 9 \mathrm{H}$, $\left.3 \times \mathrm{CH}_{3}\right) \mathrm{ppm}$.

Tert-butyl ((4-methoxyphenyl)(2,4,6-trimethoxyphenyl)methyl)carbamate 60


Chemical Formula: $\mathrm{C}_{22} \mathrm{H}_{29} \mathrm{NO}_{6}$ Molecular Weight: 403,48

According to the general procedure, $\mathbf{6 0}$ was obtained as a white foam ( 24.5 mg , Isolated yield $60 \%$ ); ${ }^{1} \mathbf{H}$ NMR ( $300 \mathrm{MHz}, \mathrm{CDCl}_{3}$ ) $\delta$ 7.19-7.14 (m, 2H, $2 \times \mathrm{CH}_{\mathrm{ar}}$ ), 6.80-6.75 (m, 2H, $2 \times \mathrm{CH}$ ar), 6.52 (br. d, $J$ $=10.2 \mathrm{~Hz}, 1 \mathrm{H}, \mathrm{CH}$ ), 6.23 (br. d, $J=10.2 \mathrm{~Hz}, 1 \mathrm{H}, \mathrm{NH}$ ), 6.15 (s, 2H, $2 \times$ $\mathrm{CH}_{\mathrm{ar}}$ ), 3.80 ( $\mathrm{s}, 3 \mathrm{H}, \mathrm{CH}_{3}$ ), 3.77 ( $\mathrm{s}, 6 \mathrm{H}, 2 \times \mathrm{CH}_{3}$ ), 3.75 ( $\mathrm{s}, 3 \mathrm{H}, \mathrm{CH}_{3}$ ), 1.46 (s, $9 \mathrm{H}, 3 \times \mathrm{CH}_{3}$ ) ppm.

Chemical Formula: $\mathrm{C}_{19} \mathrm{H}_{25} \mathrm{NO}_{3} \mathrm{~S}$ Molecular Weight: 347,47

According to the general procedure, $\mathbf{6 p}$ was obtained as a white foam ( 16.5 mg , Isolated yield $47 \%$ ); ${ }^{1} \mathbf{H}$ NMR ( $300 \mathrm{MHz}, \mathrm{CDCl}_{3}$ ) $\delta$ 7.22-7.07 (m, 5H, $5 \times \mathrm{CH}_{\mathrm{ar}}$ ), 6.49 ( $\mathrm{s}, 1 \mathrm{H}, \mathrm{CH}_{\mathrm{ar}}$ ), 5.95-5.93 (m, 1H, $\mathrm{CH}_{\mathrm{ar}}$ ), 4.74-4.59 (m, 2H, CH, NH), 3.78 ( $\mathrm{s}, 3 \mathrm{H}, \mathrm{CH}_{3}$ ), 2.65-2.55 (m, 2H, $\mathrm{CH}_{2}$ ), 2.05-1.97 (m, 2H, CH2), 1.37 (s, 9H, $3 \times \mathrm{CH}_{3}$ ) ppm; ${ }^{13} \mathbf{C}$ NMR $\left(75 \mathrm{MHz}, \mathrm{CDCl}_{3}\right) \delta 165.4\left(C_{\mathrm{q}}\right), 155.2\left(C_{\mathrm{q}}\right), 141.5\left(C_{\mathrm{q}}\right), 132.5\left(C_{\mathrm{q}}\right)$, $128.6(2 \times \mathrm{CH}), 128.4(2 \times \mathrm{CH}), 126.1(\mathrm{CH}), 121.8(\mathrm{CH}), 103.1(\mathrm{CH}), 80.1\left(C_{q}\right), 60.4\left(\mathrm{CH}_{3}\right), 50.9$ $(\mathrm{CH}), 38.5\left(\mathrm{CH}_{2}\right), 32.5\left(\mathrm{CH}_{2}\right), 28.5\left(3 \times \mathrm{CH}_{3}\right) \mathrm{ppm}$; IR: $v$ (neat, $\left.\mathrm{cm}^{-1}\right) 3336,1762,1601,1513$, 1473, 1367, 1251, 1140, 1019, 927, 801, 706; ESI-HRMS (positive) [M+Na]+ Calc. for $\mathrm{C}_{19} \mathrm{H}_{25} \mathrm{NO}_{3} \mathrm{SNa} 370.1453$, found 370.1460 .

Tert-butyl ((5-methoxythiophen-2-yl)(phenyl)methyl)carbamate $\mathbf{6 q}$



Chemical Formula: $\mathrm{C}_{17} \mathrm{H}_{21} \mathrm{NO}_{3} \mathrm{~S}$ Molecular Weight: 319,42

According to the general procedure, $\mathbf{6 q}$ was obtained as a white foam ( 16.5 mg , Isolated yield $51 \%$ ); ${ }^{\mathbf{1}} \mathbf{H}$ NMR ( $300 \mathrm{MHz}, \mathrm{CDCl}_{3}$ ) $\delta$ 7.29-7.18 (m, 5H, $5 \times \mathrm{CH}_{\mathrm{ar}}$ ), 6.28 (br. d, $J=3.7 \mathrm{~Hz}, 1 \mathrm{H}, \mathrm{CH} \mathrm{Har}_{\mathrm{ar}}$, 5.90 (br. d, $J=3.7 \mathrm{~Hz}, 1 \mathrm{H}, \mathrm{CH} \mathrm{ar}$ ), 5.86 (br. s, $1 \mathrm{H}, \mathrm{CH}$ ), 5.10 (br. s, $1 \mathrm{H}, \mathrm{NH}$ ), $3.75\left(\mathrm{~s}, 3 \mathrm{H}, \mathrm{CH}_{3}\right), 1.36\left(\mathrm{~s}, 9 \mathrm{H}, 3 \times \mathrm{CH}_{3}\right) \mathrm{ppm} ;{ }^{\mathbf{1 3}} \mathbf{C}$ NMR $(75 \mathrm{MHz}$, $\left.\mathrm{CDCl}_{3}\right) \delta 166.3\left(C_{\mathrm{q}}\right), 154.9\left(C_{\mathrm{q}}\right), 141.5\left(C_{\mathrm{q}}\right), 132.1\left(C_{\mathrm{q}}\right), 128.7\left(2 \times \mathrm{CHar}_{\mathrm{ar}}\right), 127.8\left(\mathrm{CH}_{\mathrm{ar}}\right), 127.0(2 \times$ $\left.C H_{\text {ar }}\right), 123.1\left(\mathrm{CH}_{\mathrm{ar}}\right), 103.2\left(\mathrm{CH}_{\mathrm{ar}}\right), 80.1\left(\mathrm{C}_{\mathrm{q}}\right), 60.3\left(\mathrm{CH}_{3}\right), 55.0(\mathrm{CH}), 28.5\left(3 \times \mathrm{CH}_{3}\right) \mathrm{ppm}$; IR: $v$ (neat, $\mathrm{cm}^{-1}$ ) 3336, 1795, 1628, 1363, 1147, 1009, 921, 807, 705; ESI-HRMS (positive) [M+Na]+ Calc. for $\mathrm{C}_{17} \mathrm{H}_{21} \mathrm{NO}_{3} \mathrm{SNa} 319.1242$, found 319.1238.

## 3,3'-(3-phenylpropane-1,1-diyl)bis( 1 H -indole) 7



Chemical Formula: $\mathrm{C}_{25} \mathrm{H}_{22} \mathrm{~N}_{2}$ Molecular Weight: 350,47

According to the general procedure, 7 was obtained as a colourless oil ( 15 mg , Isolated yield $43 \%$ ); ${ }^{1} \mathrm{H}$ NMR ( $300 \mathrm{MHz}, \mathrm{CDCl}_{3}$ ) $\delta 7.91$ ( $\mathrm{s}, 2 \mathrm{H}$, NH ), $7.55\left(\mathrm{~d}, \mathrm{~J}=8.0 \mathrm{~Hz}, 2 \mathrm{H}, \mathrm{CH}\right.$ ar) $, 7.34-6.99\left(\mathrm{~m}, 13 \mathrm{H}, \mathrm{CH}_{\mathrm{ar}}\right), 4.50(\mathrm{t}, J=$ 7.2 Hz, 1H, CH), 2.75-2.70 (m, 2H, CH2), 2.59-2.51 (m, 2H, CH2) ppm.


According to the general procedure with 5 ( $0.3 \mathrm{mmol}, 3.0 \mathrm{eq}$.$) , \mathbf{6 r}$ was obtained as a colourless oil ( 24 mg , Isolated yield $68 \%$ ); ${ }^{\mathbf{1}} \mathbf{H}$ NMR ( $300 \mathrm{MHz}, \mathrm{CDCl}_{3}$ ) $\delta 8.21$ (br. s, 1H, NH), 7.74-7.70 (m, 3H, $3 \times$ $\mathrm{CH}_{\mathrm{ar}}$ ), 7.49-7.11 (m, 12H, $12 \times \mathrm{CH}_{\mathrm{ar}}$ ), $6.34(\mathrm{~d}, \mathrm{~J}=8.3 \mathrm{~Hz}, 1 \mathrm{H}, \mathrm{NH}), 5.63$ (dt, $J=8.3$ and $7.2 \mathrm{~Hz} 1 \mathrm{H}, \mathrm{CH}$ ), 2.87-2.71 (m, 2H, CH2), 2.45-2.36 (m, $\left.2 \mathrm{H}, \mathrm{CH}_{2}\right) \mathrm{ppm} ;{ }^{13} \mathrm{C}$ NMR ( $75 \mathrm{MHz}, \mathrm{CDCl}_{3}$ ) $\delta 166.9\left(C_{\mathrm{q}}\right), 142.0\left(\mathrm{C}_{\mathrm{q}}\right)$, $136.8\left(C_{\mathrm{q}}\right), 131.5\left(\mathrm{CHar}_{\mathrm{ar}}\right), 128.7\left(2 \times \mathrm{CHar}_{\mathrm{ar}}\right), 128.6\left(4 \times \mathrm{CHar}_{\mathrm{ar}}\right), 127.2\left(C_{\mathrm{q}}\right)$, $127.0\left(2 \times \mathrm{CH}_{\mathrm{ar}}\right), 126.0(2 \times \mathrm{CHar}), 122.8\left(\mathrm{CH}_{\mathrm{ar}}\right), 121.9\left(\mathrm{CH}_{\mathrm{ar}}\right), 120.2$ $\left(C_{q}\right), 119.6\left(\mathrm{CH}_{\mathrm{ar}}\right), 117.0\left(C_{\mathrm{q}}\right), 111.5\left(\mathrm{CH}_{\mathrm{ar}}\right), 47.1(\mathrm{CH}), 36.8\left(\mathrm{CH}_{2}\right), 33.1\left(\mathrm{CH}_{2}\right) \mathrm{ppm}$; IR: $v$ (neat, $\mathrm{cm}^{-1}$ ) 3405, 2977, 1697, 1485, 1424, 1375, 1201, 1065, 1001, 700; ESI-HRMS (positive) $[\mathrm{M}+\mathrm{Na}]^{+}$Calc. for $\mathrm{C}_{24} \mathrm{H}_{22} \mathrm{~N}_{2} \mathrm{ONa}$ 377.1630, found 377.1636.

Tert-butyl (1-(3-methyl-1H-indol-2-yl)-3-phenylpropyl)carbamate 6s



Chemical Formula: $\mathrm{C}_{23} \mathrm{H}_{28} \mathrm{~N}_{2} \mathrm{O}_{2}$ Molecular Weight: 364,49

According to the general procedure, $\mathbf{6 s}$ was obtained as a colourless oil ( 25.5 mg , Isolated yield $70 \%$ ); ${ }^{1} \mathbf{H}$ NMR ( $300 \mathrm{MHz}, \mathrm{CDCl}_{3}$ ) $\delta 8.46$ (s, 1H, NH $H_{\text {indole }}$ ), 7.47 (d, $J=7.7 \mathrm{~Hz}, 1 \mathrm{H}, \mathrm{CH}_{\mathrm{ar}}$ ), $7.26-7.01(\mathrm{~m}, 8 \mathrm{H}$, $\mathrm{CH}_{\mathrm{ar}}$ ), 4.97 (br. s, 1H, NH) 4.71-4.64 (m, 1H, CH), 2.65-2.48 (m, 2H, $\mathrm{CH}_{2}$ ), 2.36-2.12 (m, 2H, CH2), $2.19\left(\mathrm{~s}, 3 \mathrm{H}, \mathrm{CH}_{3}\right), 1.37\left(\mathrm{~s}, 9 \mathrm{H}, 3 \times \mathrm{CH}_{3}\right)$ ppm; ${ }^{13} \mathrm{C}$ NMR $\left(75 \mathrm{MHz}, \mathrm{CDCl}_{3}\right) \delta 156.0\left(C_{\mathrm{q}}\right), 141.2\left(C_{\mathrm{q}}\right), 135.5\left(C_{\mathrm{q}}\right)$, $134.7\left(C_{\mathrm{q}}\right), 129.1\left(C_{\mathrm{q}}\right), 128.6\left(2 \times \mathrm{CH}_{\mathrm{ar}}\right), 128.5\left(2 \times \mathrm{CH}_{\mathrm{ar}}\right), 126.2\left(\mathrm{CH}_{\mathrm{ar}}\right), 121.9\left(\mathrm{CH}_{\mathrm{ar}}\right), 119.2$ $\left(\mathrm{CH}_{\mathrm{ar}}\right), 118.7\left(\mathrm{CH}_{\mathrm{ar}}\right), 110.9\left(\mathrm{CHar}_{\mathrm{ar}}\right), 107.8\left(\mathrm{C}_{\mathrm{q}}\right), 80.2\left(\mathrm{C}_{\mathrm{q}}\right), 48.0(\mathrm{CH}), 32.7\left(\mathrm{CH}_{2}\right), 28.5\left(3 \times \mathrm{CH}_{3}\right)$, $28.1\left(\mathrm{CH}_{2}\right), 8.8\left(\mathrm{CH}_{3}\right) \mathrm{ppm}$ IR: $v$ (neat, $\left.\mathrm{cm}^{-1}\right) 3330,2974,2922,1694,1490,1467,1368,1235$, 1160, 1013, 740, 697; ESI-HRMS (positive) [M+Na] Calc. for $\mathrm{C}_{23} \mathrm{H}_{28} \mathrm{~N}_{2} \mathrm{O}_{2} \mathrm{Na} 387.2048$, found 387.2051 .

Tert-butyl (1-(1H-indazol-1-yl)-3-phenylpropyl)carbamate 6t


Chemical Formula: $\mathrm{C}_{21} \mathrm{H}_{25} \mathrm{~N}_{3} \mathrm{O}_{2}$ Molecular Weight: 351,45

According to the general procedure, 6t was obtained as a colourless oil ( 32 mg , Isolated yield 91\%); ${ }^{\mathbf{1}} \mathbf{H}$ NMR ( $300 \mathrm{MHz}, \mathrm{CDCl}_{3}$ ) $\delta 8.06$ (s, $1 \mathrm{H}, \mathrm{CH}$ ar), 7.71 (d, $J=8.1 \mathrm{~Hz}, 1 \mathrm{H}, \mathrm{CH}$ ar), $7.62-7.56\left(\mathrm{~m}, 1 \mathrm{H}, \mathrm{CH}_{\mathrm{ar}}\right), 7.37$ ( $\mathrm{t}, J=7.4 \mathrm{~Hz}, 1 \mathrm{H}, \mathrm{CH} \mathrm{ar}$ ), 7.27-7.06 (m, $6 \mathrm{H}, 6 \times \mathrm{CH}_{\mathrm{ar}}$ ), 6.28-6.17 (m, 1H, CH), 5.58 (br. d, $J=8.8 \mathrm{~Hz}, 1 \mathrm{H}, \mathrm{NH}$ ), 2.64-2.41 (m, 4H, $2 \times \mathrm{CH}_{2}$ ), 1.36 (s, 9H, $3 \times \mathrm{CH}_{3}$ ) ppm; ${ }^{13} \mathbf{C}$ NMR ( $75 \mathrm{MHz}, \mathrm{CDCl}_{3}$ ) $\delta 154.9\left(C_{\mathrm{q}}\right), 140.5\left(C_{\mathrm{q}}\right), 139.8\left(C_{\mathrm{q}}\right), 134.5$ $\left(C_{\mathrm{ar}}\right), 128.6\left(4 \mathrm{x} \mathrm{CH}_{\mathrm{ar}}\right), 126.7\left(\mathrm{CHar}_{\mathrm{ar}}\right), 126.3\left(\mathrm{CHar}_{\mathrm{ar}}\right), 123.9\left(\mathrm{C}_{\mathrm{q}}\right), 121.1\left(\mathrm{CH}_{\mathrm{ar}}\right), 120.9\left(\mathrm{CH}_{\mathrm{ar}}\right), 110.0$ $\left(\mathrm{CH}_{\mathrm{ar}}\right), 80.3\left(\mathrm{C}_{\mathrm{q}}\right), 63.1(\mathrm{CH}), 36.6\left(\mathrm{CH}_{2}\right), 31.8\left(\mathrm{CH}_{2}\right), 28.4\left(3 \mathrm{x} \mathrm{CH}_{3}\right)$ ppm; IR: $v$ (neat, $\left.\mathrm{cm}^{-1}\right) 3314$, 2969, 1710, 1497, 1337, 1225, 1123, 1037, 1018, 876, 739, 635; ESI-HRMS (positive) [M+Na]+ Calc. for $\mathrm{C}_{26} \mathrm{H}_{29} \mathrm{NO}_{5} \mathrm{Na} 374.1844$, found 374.1836.


According to the general procedure, $\mathbf{6 u}$ was obtained as a colourless oil ( 21 mg , Isolated yield $70 \%$ ); ${ }^{\mathbf{1}} \mathbf{H}$ NMR ( 300 MHz , $\mathrm{CDCl}_{3}$ ) $\delta 7.55$ (br. d, $J=1.9 \mathrm{~Hz}, 1 \mathrm{H}, \mathrm{CH}_{\mathrm{ar}}$ ), 7.50 (br. s, $1 \mathrm{H}, \mathrm{CH}_{\mathrm{ar}}$ ), 7.27$7.07\left(\mathrm{~m}, 5 \mathrm{H}, 5 \times \mathrm{CH}_{\mathrm{ar}}\right), 6.20(\mathrm{t}, J=2.1 \mathrm{~Hz}, 1 \mathrm{H}, \mathrm{CH} \mathrm{ar}), 5.72-5.64(\mathrm{~m}, 1 \mathrm{H}$, CH ), 5.52 (br. d, $J=8.6 \mathrm{~Hz}, 1 \mathrm{H}, \mathrm{NH}$ ), 2.64-2.43 (m, $4 \mathrm{H}, 2 \times \mathrm{CH}_{2}$ ), 1.36 $\left(\mathrm{s}, 9 \mathrm{H}, 3 \times \mathrm{CH}_{3}\right) \mathrm{ppm} ;{ }^{13} \mathbf{C}$ NMR ( $75 \mathrm{MHz}, \mathrm{CDCl}_{3}$ ) $\delta 154.8\left(\mathrm{C}_{\mathrm{q}}\right), 140.3\left(\mathrm{CH}_{\mathrm{ar}}\right), 130.0\left(\mathrm{CH}_{\mathrm{ar}}\right), 129.8$ $\left(C_{\mathrm{q}}\right), 128.7(2 \mathrm{x} \mathrm{CH} \mathrm{ar}), 128.6\left(2 \times \mathrm{CH}_{\mathrm{ar}}\right), 126.4\left(\mathrm{CH}_{\mathrm{ar}}\right), 105.0\left(\mathrm{CH}_{\mathrm{ar}}\right), 80.5\left(\mathrm{C}_{\mathrm{q}}\right), 66.5(\mathrm{CH}), 36.2$ $\left(\mathrm{CH}_{2}\right), 31.6\left(\mathrm{CH}_{2}\right), 26.4\left(3 \times \mathrm{CH}_{3}\right) \mathrm{ppm}$; IR: $v$ (neat, $\left.\mathrm{cm}^{-1}\right) 3303,2973,1700,1504,1496,1374$, 1329, 1242, 1160, 1090, 1054, 1038, 889, 750, 719, 685, 668, 653; ESI-HRMS (positive) $[\mathrm{M}+\mathrm{Na}]^{+}$Calc. for $\mathrm{C}_{17} \mathrm{H}_{23} \mathrm{~N}_{3} \mathrm{O}_{2} \mathrm{Na} 324.1688$, found 324.1681.

Tert-butyl (cyano(phenyl)methyl)carbamate 6v


Chemical Formula: $\mathrm{C}_{13} \mathrm{H}_{16} \mathrm{~N}_{2} \mathrm{O}_{2}$ Molecular Weight: 232,28

According to the general procedure with KCN ( $0.3 \mathrm{mmol}, 3.0 \mathrm{eq}$ ) and a mixture $\mathrm{MeCN}: \mathrm{H}_{2} \mathrm{O}$ ( $0.5 \mathrm{~mL}: 0.5 \mathrm{~mL}$ ), $\mathbf{6 v}$ was obtained as a white foam ( 21.5 mg , Isolated yield 93\%); ${ }^{\mathbf{1}} \mathbf{H}$ NMR ( 300 MHz , $\left.\mathrm{CDCl}_{3}\right) \delta 7.50-7.41\left(\mathrm{~m}, 5 \mathrm{H}, 5 \times \mathrm{CH}_{\mathrm{ar}}\right), 6.80(\mathrm{br} . \mathrm{d}, J=7.7 \mathrm{~Hz}, 1 \mathrm{H}, \mathrm{CH})$, 5.13 (br. s, 1H, NH), 1.48 (s, $9 \mathrm{H}, 3 \times \mathrm{CH}_{3}$ ) ppm.

Tert-butyl (4-acetyl-5-oxo-1-phenylhexan-3-yl)carbamate 6w


Chemical Formula: $\mathrm{C}_{19} \mathrm{H}_{27} \mathrm{NO}_{4}$ Molecular Weight: 333,43

According to the general procedure, $\mathbf{6 w}$ was obtained as a white foam ( 13.5 mg , Isolated yield $40 \%$ ); ${ }^{1} \mathbf{H}$ NMR ( $300 \mathrm{MHz}, \mathrm{CDCl}_{3}$ ) $\delta$ 7.31-7.16 (m, 5H, $5 \times \mathrm{CH}_{\mathrm{ar}}$ ), 5.33 (br. d, $J=10.2 \mathrm{~Hz}, 1 \mathrm{H}, \mathrm{N} H$ ), 4.374.27 (m, 1H, CH), 3.87 (d, $J=5.1 \mathrm{~Hz}, 1 \mathrm{H}, \mathrm{CH}$ ), 2.81-2.55 (m, 2H, CH2), $2.24\left(\mathrm{~s}, 3 \mathrm{H}, \mathrm{CH}_{3}\right), 2.10\left(\mathrm{~s}, 3 \mathrm{H}, \mathrm{CH}_{3}\right), 1.99-1.86\left(\mathrm{~m}, 1 \mathrm{H}, \mathrm{CH}_{2}\right), 1.76-1.63$ (m, 1H, CH2), $1.42\left(\mathrm{~s}, 9 \mathrm{H}, 3 \times \mathrm{CH}_{3}\right) \mathrm{ppm}$.

Tert-butyl (2-acetyl-3-oxo-1-phenylbutyl)carbamate 6x


Chemical Formula: $\mathrm{C}_{17} \mathrm{H}_{23} \mathrm{NO}_{4}$ Molecular Weight: 305,37

According to the general procedure, $\mathbf{6 x}$ was obtained as a white foam ( 15 mg , Isolated yield $49 \%$ ); ${ }^{1} \mathbf{H}$ NMR ( $300 \mathrm{MHz}, \mathrm{CDCl}_{3}$ ) $\delta 7.31-$ 7.21 (m, 5H, $5 \times \mathrm{CH}_{\mathrm{ar}}$ ), 5.80 (br. s, 1H, NH), 5.46 (br. s, 1H, CH), 4.18 (d, J = 6.9 Hz, 1H, CH), $2.15\left(\mathrm{~s}, 3 \mathrm{H}, \mathrm{CH}_{3}\right), 2.08\left(\mathrm{~s}, 3 \mathrm{H}, \mathrm{CH}_{3}\right), 1.36(\mathrm{~s}, 9 \mathrm{H}$, $3 \times \mathrm{CH}_{3}$ ) ppm.

## VI. ${ }^{1} \mathrm{H}$ and ${ }^{13} \mathrm{C}$ NMR data for compounds 6:

Tert-butyl (3-phenyl-1-(2,4,6-trimethoxyphenyl)propyl)carbamate 6a


Tert-butyl (1-(2,4,6-trimethoxyphenyl)octyl)carbamate 6b


Tert-butyl (2-methyl-1-(2,4,6-trimethoxyphenyl)propyl)carbamate 6c


Tert-butyl (cyclopropyl(2,4,6-trimethoxyphenyl)methyl)carbamate 6d


Tert-butyl (2-(benzyloxy)-1-(2,4,6-trimethoxyphenyl)ethyl)carbamate 6e


Tert-butyl (3-phenyl-1-(2,4,6-trimethoxyphenyl)prop-2-yn-1-yl)carbamate $\mathbf{6 f}$


$N$-(3-phenyl-1-(2,4,6-trimethoxyphenyl)propyl)benzamide 6g



4-methoxy- $N$-(3-phenyl-1-(2,4,6-trimethoxyphenyl)propyl)benzamide $\mathbf{6 h}$



Benzyl (3-phenyl-1-(2,4,6-trimethoxyphenyl)propyl)carbamate $\mathbf{6 i}$



Tert-butyl (((R)-2,2-dimethyl-1,3-dioxolan-4-yl)(2,4,6-trimethoxyphenyl)methyl)carbamate 6j



Benzyl (2R)-2-(((tert-butoxycarbonyl)amino)(2,4,6-trimethoxyphenyl)methyl)pyrrolidine-1carboxylate 6k



Tert-butyl (phenyl(2,4,6-trimethoxyphenyl)methyl)carbamate $6 \mathbf{1}$


Tert-butyl (furan-2-yl(2,4,6-trimethoxyphenyl)methyl)carbamate $\mathbf{6 m}$


Tert-butyl ((4-(trifluoromethyl)phenyl)(2,4,6-trimethoxyphenyl)methyl)carbamate $\mathbf{6 n}$


Tert-butyl ((4-methoxyphenyl)(2,4,6-trimethoxyphenyl)methyl)carbamate 60 (14)


Tert-butyl (1-(5-methoxythiophen-2-yl)-3-phenylpropyl)carbamate 6p



Tert-butyl ((5-methoxythiophen-2-yl)(phenyl)methyl)carbamate 6q



3,3'-(3-phenylpropane-1,1-diyl)bis(1H-indole) 7


N -(1-(1H-indol-3-yl)-3-phenylpropyl)benzamide $\mathbf{6 r}$



Tert-butyl (1-(3-methyl-1H-indol-2-yl)-3-phenylpropyl)carbamate 6s



Tert-butyl (1-(1H-indazol-1-yl)-3-phenylpropyl)carbamate 6t



Tert-butyl (3-phenyl-1-(1H-pyrazol-1-yl)propyl)carbamate $\mathbf{6 u}$



Tert-butyl (cyano(phenyl)methyl)carbamate 6v


Tert-butyl (4-acetyl-5-oxo-1-phenylhexan-3-yl)carbamate 6w


Tert-butyl (2-acetyl-3-oxo-1-phenylbutyl)carbamate 6x



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