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Diazide Chemistry

An Unconventional Reaction of 2,2-Diazido Acylacetates with Amines

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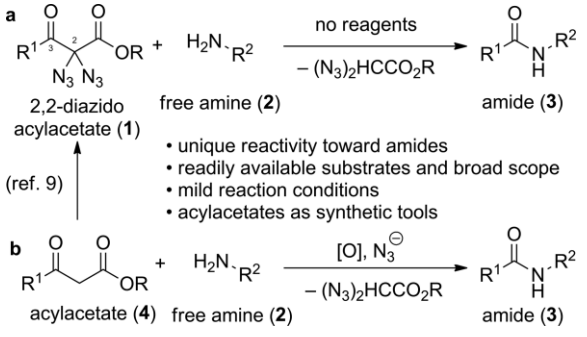
Abstract: We have discovered that 2,2-diazido acylacetates, a class of compounds with essentially unknown reactivity, can be coupled to amines through a new strategy that does not involve any reagents. 2,2-Diazido acetate is the unconventional leaving group under carbon–carbon bond cleavage. This reaction leads to the construction of amide bonds, tolerates various functionalities and is performed equally well in numerous solvents under experimentally simple conditions. We also demonstrate that the isolation of the 2,2-diazido acylacetate compounds can be circumvented: Acylacetates were easily fragmented when treated with $(Bu_4N)N_3$ and iodine in the presence of an amine at room temperature. By using this method, a broad range of acylacetates with various structural motifs were directly transformed into amides.

Introduction

Geminal diazides have received very little attention in chemical sciences:[1,2] Only a small number of synthesis methods have been described since their first appearance in 1908,[3,4] and applications remain rare.[5] The thermal[6] and photochemical[7] degradation of this functionality was studied, and the omnipresent cycloaddition with alkynes was also attempted.[8,4c–4e] Based on our recent studies with geminal diazides,[4c,4d,8b,49] we now report the general fragmentation shown in Scheme 1a. The method uses 2,2-diazido acylacetates **1** as unconventional acyl donors and leads to the direct formation of amides **3** through reaction with amines **2**. The unprecedented activating influence of azido groups on adjacent carbonyl moieties leads to cleavage of the carbon–carbon bond between C2 and C3 in the course of the amide bond formation, and 2,2-diazido acetate[3,10] becomes a unique leaving group. This reactivity was not described before; it loosely resembles a retro-Claisen reaction wherein typically harsh reaction conditions with strong Lewis-acid additives and high temperatures are required.[11] Our acyl transfer, however, occurs at room temperature under experimentally simple conditions in a great range of solvents and, in most cases, without the need for additional reagents. Preliminary studies suggest an excellent chemoselectivity, and numerous coexisting functional groups are tolerated, thus expanding the potential utility of this transformation tremendously. We also report a convenient variant that facilitates the direct reaction of acylacetates **4** with primary amines (Scheme 1b). This straightforward formation of amide bonds through the oxidative in situ diazidation of acylacetates under metal-free reaction conditions does not require the isolation of organic diazide compounds and maintains its compatibility with a diverse array of dicarbonyl substrates.



Scheme 1. Concept: fragmentation of 2,2-diazido acylacetates **1** and acylacetates **4** with amines **2**.

The ability to create amide bonds is an area of ongoing importance in synthetic chemistry and chemical biology with applications that span proteins, pharmaceuticals and functional materials.[12,13] The standard way to approach amide bond formation in the laboratory is the condensation of an amine with a carboxylic acid via an active ester.[14] Despite the omnipresence of this strategy, chemists have, in the context of pursuing new amide bond-forming methods, compiled a great reaction compendium of amide bond-forming methods.[15] Unlike the many existing reactions, we now add acylacetates (or rather their diazidated congeners) to the broad group of reliable precursors for the construction of amides, a strategy that may find considerable use as a tool for the fragmentation of small molecules.[16,17] Our new bond fission methodology is a somewhat rare but powerful synthetic option to afford the controlled breakdown of acylacetates into acylamides through aminolysis and carbon–carbon bond cleavage.[18,19]

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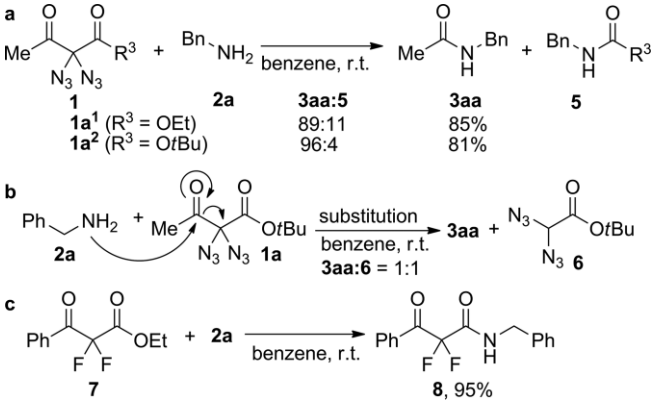
Results and Discussion

The Fragmentation of 2,2-Diazido Acylacetates with Amines

Our studies began with the observation that treatment of ethyl 2,2-diazidoacetoacetate (**1a**;[1] R^3 = OEt) with benzylamine (**2a**) in benzene at 23 °C provided both *N*-acetyl benzylamine (**3aa**) and ethyl *N*-benzyl carbamate in a 89:11 ratio, with **3aa** being the major product, as indicated by the ^{1}H NMR spectrum of the crude mixture (Scheme 2a). By simply switching to sterically more demanding *tert*-butyl 2,2-diazidoacetoacetate (**1a**), the ratio between amide and carbamate formation was improved to

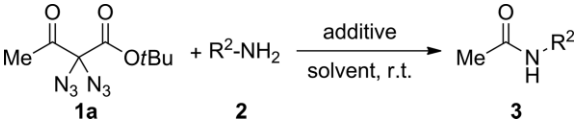


Scheme 2. Initial experiments on the reaction of 2,2-diazido 1,3-dicarbonyl compounds with primary amines: (a) Influence of the substituents. Ratio **3aa**/**5** determined by ^{1}H NMR spectroscopic analysis of the crude mixture; yields of the isolated compounds after column chromatography. (b) Proposed mechanism. (c) Reaction of ethyl 2,2-difluorobenzoylacetate with benzylamine.

96:4, and the isolated chemical yield of **3aa** was high. The acylation of amines with diazido acylacetates appears to be a substitution in which attack of the substituting amine at the carbonyl carbon is followed by elimination of the diazido acetate. In the case of the formation of **3aa** from **1a**, we unequivocally observed the stoichiometric evolution of *tert*-butyl 2,2-diazidoacetate (**6**) based on ^{1}H NMR spectroscopic analysis, and this compound was also isolated in 70 % yield in pure form (Scheme 2b).[3,10] Given that azido groups are known for their electron-withdrawing character,[20] we also assessed whether the acylation could be rendered possible by the choice of other electronegative substituents. Through the use of ethyl 2,2-difluorobenzoylacetate (**7**), for example, the direct benzoylation of benzylamine was not observed (Scheme 2c). Instead of C–C bond cleavage, C–O bond cleavage was favoured, providing amide **8**, as reported before,[21] which underlines the unique reactivity of 2,2-diazido acylacetates with amines.

Encouraged by the ability of 2,2-diazido acetoacetates to transfer the acetyl group onto amines, we studied this reaction using various solvents, and the reaction of **1a** was selected as a model (Table 1). Almost equimolar amounts of diazide **1a** and benzylamine (**2a**) produced the desired product **3aa** in good yields in an attractive collection of standard solvents including benzene, tetrahydrofuran (THF), isopropanol (*i*PrOH), dimethyl sulfoxide (DMSO) and *N*-methyl-2-pyrrolidinone (NMP) (entries 1–6). Branched amines gave similar reactivity profiles with some reduction in yield. For example, reaction of **1a** with 1-cyclohexylethylamine (**2b**; 1.1 equiv.) in benzene gave amide **3ab** in a yield of 56 % (entry 7). We were pleased to find, however, that coupling of substrate **1a** with **2b** (2 equiv.) gave a high yield (entry 8), and further tests showed that even in THF, DMA, DMSO, *N*,*N*-dimethylformamide (DMF), MeCN, and EtOAc, prod-

Table 1. Reaction optimization and robustness toward solvent effects.[a]

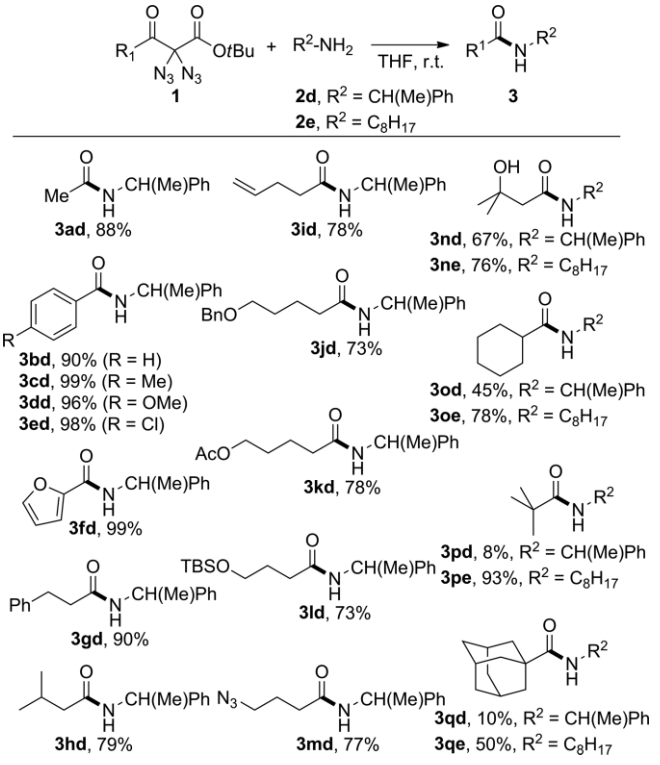


No.	Substrate **2** #	R^2	Equiv.	Solvent	Additive	Product **3**	Yield [%][b]
1	**2a**	CH_2Ph	1.1	THF		**3aa**	71
2			1.1	*i*PrOH			74
3			1.1	$CHCl_3$			64
4			1.1	DMSO			71
5			1.1	DMF			58
6			1.1	NMP			71
7	**2b**	CH(Me)(Cy)	1.1	benzene		**3ab**	56
8			2.0	benzene			84
9			2.0	THF			87
10			2.0	DMSO			71
11			2.0	DMF			79
12			2.0	MeCN			76
13			2.0	EtOAc			86
14			2.0	DMA			81
15	**2c**	Cy	1.1	THF		**3ac**	54
16			1.1	THF	Cs_2CO_3		75
17[c]			1.1	THF	DBU		82

[a] Reaction conditions: **1a**, **2**, room temp., solvent (0.5 M), 4–12 h. [b] Isolated yield after column chromatography. [c] Reaction at 50 °C.

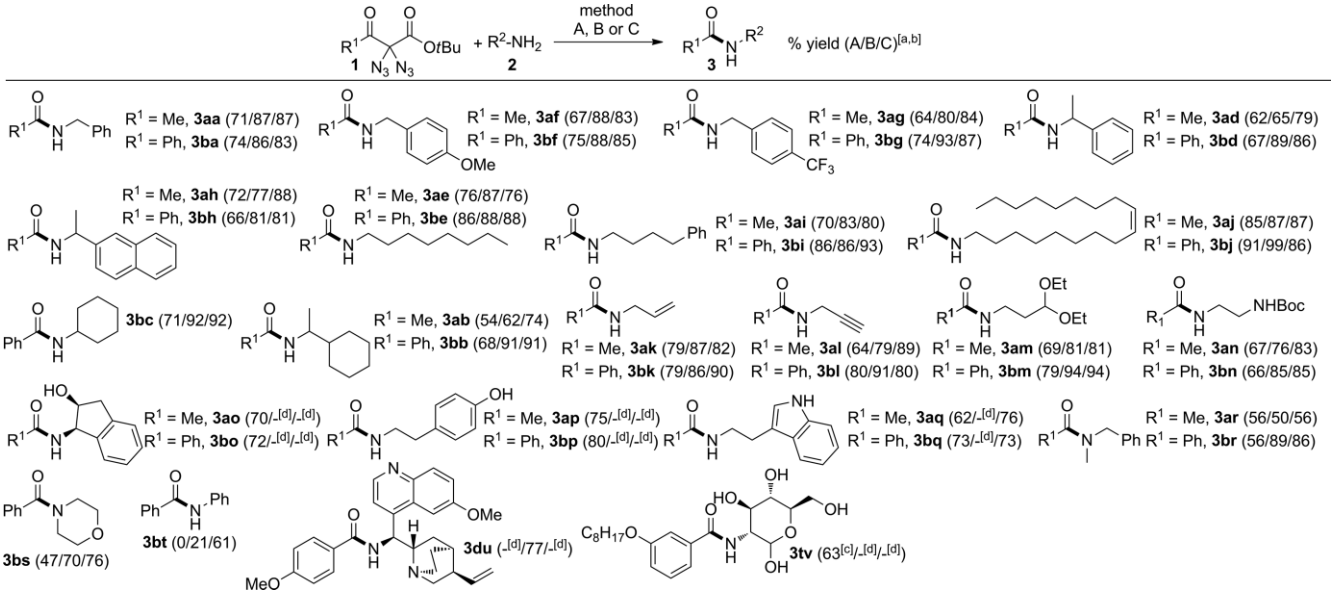
uct formation occurred smoothly (entries 9–14). Our tests also showed that, with no more than 1.1 equiv. of amine, the desired amide was more cleanly formed by using base additives. As exemplified for the acetylation of cyclohexylamine (**2c**), the yield of the reaction in THF was markedly improved when the reaction was conducted in the presence of Cs_2CO_3 or 1,8-diazabicyclo[5.4.0]undec-7-ene (DBU) (entries 16 and 17). We conclude that the acylation of amines with 2,2-diazido acylacetates is rather robust toward variations in solvent polarity, and the addition of bases has further potential to create high yields, even when using the more sterically demanding amines.

Having determined the possible reaction conditions, we began to examine the scope of the transformation with respect to the diazide substrate (Scheme 3). For this study, we chose to utilise 2.0 equiv. of 1-phenylethylamine (**2d**) as the nucleophile without further additives; this amine was one of the lowest yielding under the reaction conditions, and we felt it could serve as a convenient benchmark to demonstrate the capability of the amide bond-forming reaction. In addition to the acetyl donor **1a**, a range of benzoyl donor substrates containing the electron-donating methoxy or methyl substituents or the chloride substituent gave excellent yields. Beyond the use of phenyl substrates, furan compound **1f** readily coupled with **2d**. Furthermore, the reaction tolerated the presence of olefins (**3id**), ethers (**3jd**), esters (**3kd**), silylethers (**3ld**), alkyl azides (**3md**), and unprotected alcohols (**3nd** and **3ne**). We found that the hindered amine **2d** was a poor partner for diazides with bulky alkyl groups, thus resulting in rather low yields for the amides **3od**, **3pd** and **3qd**. In contrast, incorporation of *n*-octylamine (**2e**) was no problem under the standard conditions, converting the challenging diazides into their amides **3oe**–**qe** in good yields.



Scheme 3. The scope of the reaction leading to amide formation was evaluated with respect to the diazide substrate. Reagents and conditions: **1**, **2d** or **2e** (2.0 equiv.), room temp., THF (0.5 M), 12 h.

A variety of amines were also surveyed, as shown in Scheme 4. To this end, we examined the reaction conditions in THF whereby only 1.1 equiv. of amine **2** was used, without any additives (method A), or with Cs_2CO_3 (method B) or DBU (method C). Synthetically useful yields of product were typically obtained with each of the three methods. In most cases, the benzoylation of amines with diazide **1b** gave somewhat higher



Scheme 4. The scope of the amide formation was evaluated with respect to the amine substrate.[a] Reagents and conditions: (Method A) **1**, **2** (1.1 equiv.), room temp., THF (0.5 M); (Method B) **1**, **2** (1.1 equiv.), Cs_2CO_3 (2.0 equiv.), room temp., THF (0.5 M); (Method C) **1**, **2** (1.1 equiv.), DBU (1.0 equiv.), 50 °C, THF (0.5 M). [b] Isolated yield after column chromatography. [c] Reaction was performed in DMF. [d] Experiment was not performed. [e] Compound **2** (3 equiv.) was used.

yields than the acetylation with diazide **1a**, and we could not identify any major limitations. Primary amines, such as benzylic amines (**3aa**–**bh**) and aliphatic amines (**3ae**–**bb**) coupled smoothly to produce the corresponding amides. Compatibility of the reaction with olefins, alkynes, acetals, and carbamate-protected amines was demonstrated by the reactions of amines **2k**–**n**. 1-Amino-2-indanol could be used with no need to block the free hydroxyl (**3ao** and **3bo**). We found that phenol was tolerated in the methodology (**3ap** and **3bp**), in addition to an indole-containing moiety (**3aq** and **3bq**). The amide products were also formed with more hindered secondary amines (**3ar**–**bs**), albeit with markedly reduced yields when using method A (i.e., no base additives). As expected, aniline (and various derivatives thereof) did not undergo the amide formation in the absence of bases (method A), and yields were low even in the presence of bases (**3bt**), a feature that allows for selective amide bond formation with aliphatic amines in the presence of aromatic amines. As demonstrated for amides **3du** and **3tv**, a complex cinchona alkaloid-derived primary amine **2u** with two nucleophilic nitrogen atoms and glucosamine (**2v**), respectively, also underwent the desired amide bond formation in good yields without protecting group chemistry; in the latter case, DMF was the solvent of choice.

The Oxidative Carbon–Carbon Bond Cleavage of Acylacetates with Amines

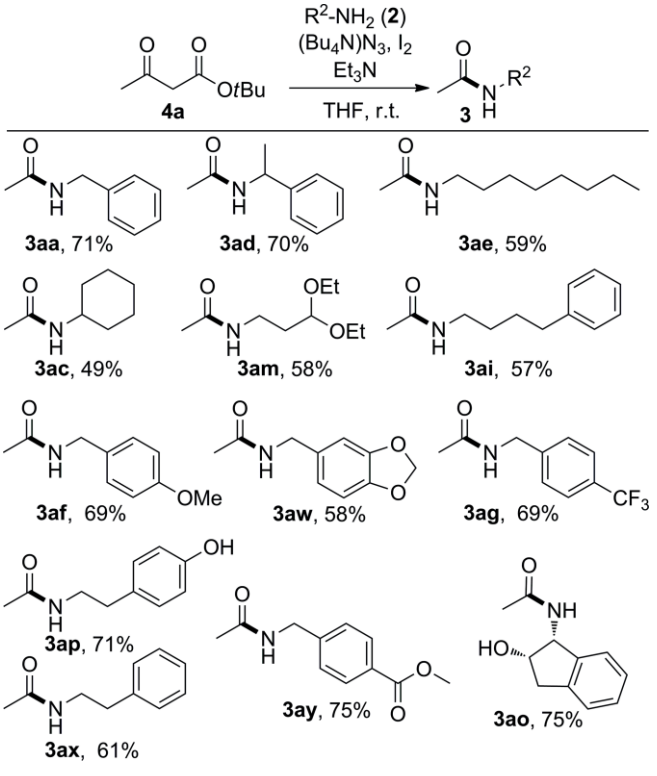
All 2,2-diazido acylacetates **1** were generated through the experimentally simple diazidation of the acylacetates with iodine and sodium azide in aqueous DMSO, as previously reported by us.[9] We point out that diazide compounds of type **1** were shown to be reasonably stable at room temperature (up to 150 °C in most cases), and gram-scale quantities were readily accessible. Nevertheless, *diazides should be considered potentially hazardous and handled accordingly due to their high nitrogen content*.[22,23]

Given the safety issues, we felt that this new amide-forming method will appear less attractive to synthetic chemists. One might hesitate to isolate and employ the diazides **1** although they represent a powerful alternative to the existing acylation agents, as discussed above. With these considerations in mind, we aimed to develop an advantageous protocol that allows for the direct conversion of acylacetates **4** into amides **3**, without the need to isolate the diazide intermediates (Scheme 1b).

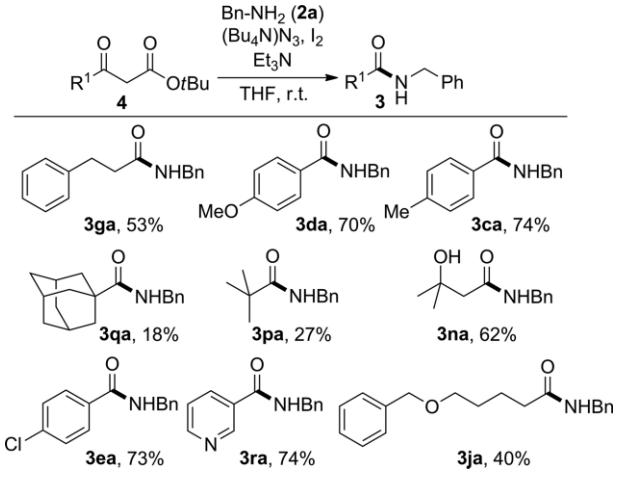
The controlled degradation of the acylacetate core providing synthetically useful building blocks is surprisingly underdeveloped. The retro-Claisen reaction is the textbook fragmentation of acylacetates, typically involving alcohol nucleophiles under strongly basic conditions to trigger the desired carbon–carbon bond cleavage, and several synthetic applications can be found.[24] Alternative methods were reported that make use of Lewis acid catalysts and high temperatures.[10,25] However, only a small number of examples describe retro-Claisen variants in which amine nucleophiles were employed, most of which rely on arguably harsh reaction conditions.[26]

To develop a mild method for the direct fragmentation of acylacetates with primary amines, we started our study with tests on how our protocols[4c,9] for diazidation can be combined with the acylation of amines. To this end, acylacetate **4a** was oxidised first in the presence of IBX-SO_3K,[27] sodium iodide, sodium azide, and benzylamine (**2a**). However, amide formation was not detected under various reaction conditions, and further experiments were performed with iodine as the oxidative agent,[9] instead of the IBX-SO_3K/NaI couple. We then realised that the reaction required water-free conditions because all attempts using aqueous DMSO failed. Gratifyingly, THF was found to be the solvent of choice, and it became mandatory to employ tetrabutylammonium azide as the azide source instead of sodium azide, which did not dissolve in pure THF and remained unreactive. It was also possible to run the reaction in DMSO, benzene, or dichloromethane. Amide **3aa** was formed in 71 % yield by using the optimal conditions: acetylacetate **1a** (1.0 equiv.), (nBu$_4$N)N_3 (4.0 equiv.), Et_3N (4.0 equiv.), I_2 (2.2 equiv.), benzylamine (2.0 equiv.), room temp., THF (0.15 M).

With the optimised conditions in hand, we briefly examined the scope of the direct acylation of amines with acylacetates. First, the reaction of *tert*-butyl acetylacetate (**4a**) with several amines was tested, as summarised in Scheme 5. A range of primary amines gave the corresponding acetyl amides **3**, and the reaction was compatible with various functional groups. Next, the methodology was evaluated with respect to the scope of the acylacetate partner. Scheme 6 shows that aliphatic and aromatic compounds could be used as substrates to generate the expected amides with benzylamine in 18–74 % yield through acylacetate fragmentation. Also in these cases, the functional group tolerance was promising because acylacetates having, for example, pyridine units and tertiary hydroxyls were smoothly converted.

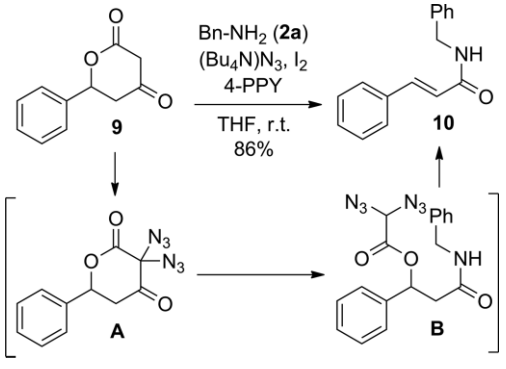


Scheme 5. Fragmentation of *tert*-butyl acetylacetate with amines.



Scheme 6. Fragmentation of *tert*-butyl acylacetates with benzylamine.

When the reaction conditions were applied to cyclic acylacetates, new possibilities opened up. For example, dihydro-2*H*-pyran-2,4(3*H*)dione **9** was smoothly transformed into acrylamide **10** through treatment with benzylamine in the presence of iodine and azide anions (Scheme 7). This high-yielding degradation of 4-oxovalerolactones is unique; we are not aware of related one-step reactions for synthesis on a laboratory scale. We assume that the initial diazidation (→**A**) results in the carbon–carbon bond cleavage (→**B**) and subsequent elimination. Further ring-opening applications are under investigation and will be reported in due course.



Scheme 7. Conversion of 4-oxovalerolactone **5**. 4-PPY = 4-pyrrolidinopyridine.

Conclusions

We have discovered a new reactivity between 2,2-diazido acylacetates and primary amines that results in a nonconventional amide synthesis. The reagent-free conditions are operationally simple (i.e., open flask, room temperature) and have been shown to accommodate a range of diazides and amines. This use of 2,2-diazido acylacetates shows potential for future exploration and may ultimately find new applications for azide-containing small molecules, apart from the ubiquitous cycloaddition with alkynes.

We also described a powerful synthetic application of this carbon–carbon bond cleavage reaction. A mild version of the retro-Claisen reaction became possible through the combination of the aminolysis of 2,2-diazido acylacetates with the oxidative diazidation of acylacetates, in a straightforward one-step manner. Of paramount importance, the controlled disintegration of the acylacetate system with primary amines does not require the isolation of any diazido compounds. We expect to expand this concept to additional reactions in which the generation of diazido compounds in situ is used to trigger a nucleophilic bond cleavage, in the near future. Studies on the mechanism of fragmentation are ongoing.

Experimental Section

General Remarks: *Caution*: We underline that geminal diazides **1** are potentially hazardous chemicals that should be handled with care. Diazido acetates (e.g., **6**), with their high nitrogen content, although not studied in detail, are also potentially hazardous compounds, especially when accumulated during the work-up of large-scale experiments. To this end, we carefully examined a range of work-up conditions with the goal to completely remove the diazido-containing side products from the organic phases to be concentrated and purified. It was found that upon simple stirring of the reaction mixture with a saturated solution of $Na_2S_2O_3$ in water at room temperature for 30 min, no trace of the 2,2-diazidoacetate **6** could be detected in the organic phase. These practical work-up conditions were applied to all the experiments with more than 0.25 mmol of diazido acylacetates. In the case of the smaller scale reactions, the crude mixtures were, upon complete conversion of diazides **1**, directly submitted to purification through column chromatography, and the fractions containing **6** were combined, brought to pH 14 with aqueous NaOH (1 N) and discarded without concentration.

General Procedures

Synthesis of 2,2-Diazido Acylacetates 1: The synthesis of 2,2-diazido acylacetates **1a**–**s** was published recently.[9]

General Procedure A for the Synthesis of Amides 3 from 2,2-Diazido Acylacetates 1: A solution of the corresponding amine **2** (1.10 equiv.) in THF (0.50 M) was added to the 2,2-diazido acylacetate **1** and the mixture was stirred overnight at room temperature. The mixture was purified by chromatography over silica.

General Procedure B for the Synthesis of Amides 3 from 2,2-Diazido Acylacetates 1: Cs_2CO_3 (2.00 equiv.), followed immediately by a solution of the corresponding amine **2** (1.10 equiv.) in THF (0.50 M), were added to the 2,2-diazido acylacetate **1** and the mixture was stirred overnight at room temperature. The mixture was purified by chromatography over silica.

General Procedure C for the Synthesis of Amides 3 from 2,2-Diazido Acylacetates 1: A solution of the corresponding amine **2** (1.10 equiv.) and DBU (1.00 equiv.) in THF (0.50 M) was added at 50 °C to the 2,2-diazido acylacetate **1**, and the mixture was stirred at 50 °C overnight. The mixture was purified by chromatography over silica.

General Procedure D for the Synthesis of Amides 3 from 2,2-Diazido Acylacetates 1: A solution of the corresponding amine **2** (2.00 equiv.) in THF (0.50 M) was added to the 2,2-diazido acylacetate **1** and the mixture was stirred overnight at room temperature. The mixture was purified by chromatography over silica.

General Procedure E for the Synthesis of Amides 3 from Acylacetates 4: Acylacetate (**4**; 1.00 equiv.), tetrabutylammonium azide

(4.00 equiv.), triethylamine (4.00 equiv.) and amine (**2**; 4.00 equiv.) were dissolved in THF (0.15 M), and iodine (2.20 equiv.) was added at room temperature. The reaction mixture was stirred overnight at room temperature. Flash-chromatography furnished the corresponding amides **3**.

Experimental Details

***tert*-Butyl 2,2-Diazido-3-[3-(octyloxy)phenyl]-3-oxopropanoate (1t):** To *tert*-butyl 3-[3-(octyloxy)phenyl]-3-oxopropanoate (0.959 g, 2.75 mmol, 1.00 equiv.) in DMSO (18.3 mL, 0.15 M) and H_2O (9.2 mL, 0.30 M), NaN_3 (1.073 g, 16.51 mmol, 6.00 equiv.) and I_2 (1.537 g, 6.05 mmol, 2.20 equiv.) were added. The solution was stirred for 3.5 h. Further NaN_3 (1.073 g, 16.51 mmol, 6.00 equiv.) was added and the solution was stirred for 3 h. A saturated aqueous solution of $Na_2S_2O_3$ was added and the solution was diluted with H_2O. The mixture was extracted with EtOAc. The organic phase was dried with $MgSO_4$ and evaporated in vacuo. The residue was purified by chromatography over silica (PE → PE/EtOAc, 95:5) to give a mixture of starting material and **1t**. The mixture was once again dissolved in DMSO (18.3 mL, 0.15 M) and H_2O (9.2 mL, 0.30 M) and NaN_3 (1.073 g, 16.51 mmol, 6.00 equiv.), I_2 (1.537 g, 6.05 mmol, 2.20 equiv.) and TBAI (0.051 g, 0.14 mmol, 0.05 equiv.) were added. The solution was stirred for 9 h. A saturated aqueous solution of $Na_2S_2O_3$ was added and the solution was diluted with H_2O. The mixture was extracted with EtOAc. The organic phase was dried with $MgSO_4$ and evaporated in vacuo. The residue was purified by chromatography over silica (PE → PE/EtOAc, 95:5) to give *tert*-butyl 2,2-diazido-3-[3-(octyloxy)phenyl]-3-oxopropanoate (**1t**; 0.712 g, 60 %) as a colourless liquid. R_f (PE/EtOAc, 95:5) = 0.45 [$KMnO_4$]. IR: $\tilde{\nu}$ = 3077, 2928, 2857, 2124, 1751, 1705, 1597, 1580, 1487, 1470, 1458, 1438, 1396, 1372, 1290, 1257, 1237, 1201, 1147, 1049, 998, 945, 899, 830, 773, 751, 723, 682, 618, 547, 481, 468, 443 cm^{-1}. ^1H NMR (400 MHz, $CDCl_3$): δ = 7.59 (ddd, J = 7.7, 1.7, 0.9 Hz, 1 H), 7.54–7.52 (m, 1 H), 7.38–7.32 (m, 1 H), 7.14 (ddd, J = 8.3, 2.6, 1.0 Hz, 1 H), 3.99 (t, J = 6.5 Hz, 2 H), 1.84–1.74 (m, 2 H), 1.51–1.41 (m, 2 H), 1.38 (s, 9 H), 1.37–1.24 (m, 8 H), 0.89 (t, J = 6.9 Hz, 3 H) ppm. ^{13}C NMR (101 MHz, $CDCl_3$): δ = 186.8, 163.8, 159.5, 133.5, 129.8, 122.2, 121.8, 114.5, 86.7, 83.2, 68.5, 32.0, 29.5, 29.4, 29.3, 27.7, 26.1, 22.8, 14.2 ppm. LRMS (ESI): m/z = 397 (80) [M – tBu + Na^+], 289 (85) [M – CO_2tBu-N_3 + H^+], 266 (100). HRMS (ESI) m/z calcd. for $C_{21}H_{30}N_6O_4Na^+$: 453.2221; found 453.2222.

***tert*-Butyl 2,2-Diazidoacetate (6):** To *tert*-butyl 2,2-diazido-3-oxobutanoate (**1a**; 0.500 g, 2.08 mmol) in benzene (5.4 mL, 0.20 M), a solution of benzylamine (**2a**; 0.270 g, 2.50 mmol, 1.20 equiv.) in benzene (5.0 mL) was added. The resulting suspension was stirred for 23 h. Further benzylamine (**2a**; 0.270 g, 2.50 mmol, 1.20 equiv.) was added and the suspension was stirred for 3 h. The suspension was evaporated in vacuo and the residue was purified by chromatography over silica (PE → EtOAc/*i*PrOH, 8:2) to give *tert*-butyl 2,2-diazidoacetate (**6**; 0.290 g, 70 %) as a colourless liquid and *N*-benzylacetamide (**3aa**; vide infra, 0.278 g, 90 %) as a white solid. R_f (PE/EtOAc, 9:1) = 0.60 [$KMnO_4$]. IR: $\tilde{\nu}$ = 2983, 2932, 2873, 2857, 2103, 1746, 1478, 1458, 1396, 1371, 1351, 1293, 1211, 1147, 978, 918, 838, 807, 763, 736, 557, 468, 435 cm^{-1}. ^1H NMR (400 MHz, $CDCl_3$): δ = 4.71 (s, 1 H), 1.54 (s, 9 H) ppm. ^{13}C NMR (101 MHz, $CDCl_3$): δ = 164.0, 85.1, 73.9, 27.9 ppm. LRMS (EI): m/z = 155 (1) [$(M – HN_3)^+$], 97 (20) [CHN_6^+], 57 (100) [tBu$^+$].

As direct HRMS was not possible, *tert*-butyl 2,2-diazidoacetate (**6**) was converted into its bistriazole derivative **6′**: To *tert*-butyl 2,2-diazidoacetate (**6**; 0.030 g, 0.15 mmol) in a 2:1 mixture of *t*BuOH (0.6 mL, 0.25 M) and H_2O (0.3 mL, 0.5 M), (+)-sodium L-ascorbate (0.012 g, 0.06 mmol, 0.40 equiv.), tris[(1-benzyl-1*H*-1,2,3-triazol-4-yl)methyl]amine (0.001 g, 1.5 μmol, 0.01 equiv.), $CuSO_4·5H_2O$ (0.008 g, 0.03 mmol, 0.20 equiv.) and phenylacetylene (0.04 mL, 0.035 g, 0.33 mmol, 2.20 equiv.) were added. The suspension was stirred for 5 h. Water was added, and the mixture was extracted with MTBE. The organic phase was dried with $MgSO_4$ and evaporated in vacuo. The residue was purified by chromatography over silica (PE/EtOAc, 95:5 → 1:1) to give *tert*-butyl 2,2-bis(4-phenyl-1*H*-1,2,3-triazol-1-yl)acetate (**6′**; 0.053 g, 87 %) as a white solid. R_f (PE/EtOAc, 8:2) = 0.19 [UV]. IR: $\tilde{\nu}$ = 3128, 3097, 3063, 3051, 3035, 2975, 2953, 2928, 2872, 2853, 1751, 1650, 1612, 1557, 1484, 1454, 1428, 1393, 1369, 1355, 1324, 1304, 1289, 1260, 1244, 1235, 1213, 1194, 1178, 1151, 1077, 1069, 1038, 1023, 973, 957, 920, 911, 863, 847, 819, 794, 765, 725, 707, 693, 598, 522, 506, 468, 434, 421 cm^{-1}. ^1H NMR (400 MHz, $CDCl_3$): δ = 8.27 (s, 2 H), 7.88–7.82 (m, 4 H), 7.65 (s, 1 H), 7.46–7.39 (m, 4 H), 7.38–7.32 (m, 2 H), 1.53 (s, 9 H) ppm. ^{13}C NMR (101 MHz, $CDCl_3$): δ = 160.9, 148.8, 129.7, 129.1, 128.9, 126.1, 120.5, 87.1, 71.4, 27.9 ppm. LRMS (ESI): m/z = 425 (20) [M + Na^+], 403 (100) [M + H^+]. HRMS (ESI): m/z calcd. for $C_{22}H_{23}N_6O_2^+$: 403.1877; found 403.1872.

***N*-Benzyl-2,2-Difluoro-3-oxo-3-phenylpropanamide (8):** To ethyl 2,2-difluoro-3-oxo-3-phenylpropanoate (**7**; 0.030 g, 0.13 mmol, 1.00 equiv.), a solution of benzylamine (**2a**; 0.028 g, 0.26 mmol, 2.00 equiv.) in THF (0.26 mL, 0.50 M) was added. The solution was stirred for 3 d. The solution was purified by chromatography over silica (PE/EtOAc, 9:1 → 1:1) to give *N*-benzyl-2,2-difluoro-3-oxo-3-phenylpropanamide (**8**; 0.036 g, 95 %) as a white solid. R_f (PE/EtOAc, 8:2) = 0.39 [$KMnO_4$]. IR: $\tilde{\nu}$ = 3313, 3068, 3036, 2951, 2927, 2856, 1708, 1682, 1596, 1580, 1543, 1495, 1451, 1435, 1395, 1357, 1324, 1310, 1273, 1239, 1204, 1155, 1133, 1102, 1041, 1029, 1011, 975, 935, 922, 902, 822, 805, 753, 729, 705, 683, 667, 606, 580, 526, 489, 449, 423 cm^{-1}. ^1H NMR (400 MHz, $CDCl_3$): δ = 8.17–8.09 (m, 2 H), 7.69–7.62 (m, 1 H), 7.54–7.46 (m, 2 H), 7.39–7.23 (m, 6 H), 6.86 (s, 1 H), 4.53 (d, J = 6.1 Hz, 2 H) ppm. ^{13}C NMR: δ = 187.4 (t, J = 27.2 Hz), 161.6 (t, J = 27.5 Hz), 136.5, 135.0, 131.8 (t, J = 1.6 Hz), 130.5 (t, J = 2.9 Hz), 129.0, 128.9, 128.2, 128.0, 111.0 (t, J = 265.7 Hz), 44.0 ppm. LRMS (EI): m/z = 289 (2) [M^+], 105 (100) [$PhCO^+$], 91 (54) [$C_7H_7^+$], 77 (85) [Ph^+]. HRMS (ESI): m/z calcd. for $C_{16}H_{13}F_2NO_2Na^+$: 312.0807; found 312.0803.

***N*-Benzylacetamide (3aa):** By following general procedure A (0.022 g, 71 %), B (0.027 g, 87 %), C (0.027 g, 87 %) or E (0.033 g, 71 % starting from 0.050 g of *tert*-butyl acetoacetate **4a**), *N*-benzylacetamide (**3aa**) was obtained as a white solid after chromatography (PE/EtOAc, 1:1 → EtOAc/*i*PrOH, 8:2). R_f (DCM/MeOH, 95:5) = 0.31 [$KMnO_4$]. ^1H NMR (400 MHz, $CDCl_3$): δ = 7.39–7.16 (m, 5 H), 6.20 (s, 1 H), 4.37 (d, J = 5.7 Hz, 2 H), 1.97 (s, 3 H) ppm. ^{13}C NMR (101 MHz, $CDCl_3$): δ = 170.1, 138.4, 128.7, 127.9, 127.5, 43.7, 23.2 ppm. The analytical data are in agreement with previously reported data.[28]

***N*-(1-Cyclohexylethyl)acetamide (3ab):** By following general procedure A (0.019 g, 54 %), B (0.022 g, 62 %) or C (0.026 g, 74 %), *N*-(1-cyclohexylethyl)acetamide (**3ab**) was obtained as a white solid after chromatography (PE/EtOAc, 1:1 → EtOAc/*i*PrOH, 8:2). R_f (DCM/MeOH, 95:5) = 0.43 [$KMnO_4$]. ^1H NMR (400 MHz, $CDCl_3$): δ = 5.40 (s, 1 H), 3.83 (dp, J = 9.1, 6.7 Hz, 1 H), 1.95 (s, 3 H), 1.78–1.58 (m, 5 H), 1.34–1.07 (m, 4 H), 1.05 (d, J = 6.8 Hz, 3 H), 1.04–0.87 (m, 2 H) ppm. ^{13}C NMR (101 MHz, $CDCl_3$): δ = 169.4, 49.5, 43.2, 29.2, 29.1, 26.5, 26.3, 26.3, 23.7, 18.0 ppm. The analytical data are in agreement with previously reported data.[29]

***N*-Cyclohexylacetamide (3ac):** By following general procedure A (0.016 g, 54 %), B (0.022 g, 75 %), C (0.024 g, 82 %) or E (0.022 g, 49 % using 0.050 g of *tert*-butyl acetoacetate **4a**), *N*-cyclohexylacetamide (**3ac**) was obtained as a white solid after chromatography (PE/EtOAc, 1:1 → EtOAc/*i*PrOH, 8:2). R_f (DCM/MeOH, 95:5) = 0.27

[$KMnO_4$]. ^1H NMR (400 MHz, $CDCl_3$): δ = 5.40 (s, 1 H), 3.81–3.67 (m, 1 H), 1.94 (s, 3 H), 1.93–1.86 (m, 2 H), 1.74–1.65 (m, 2 H), 1.65–1.55 (m, 1 H), 1.43–1.28 (m, 2 H), 1.21–1.03 (m, 3 H) ppm. ^{13}C NMR (101 MHz, $CDCl_3$): δ = 169.2, 48.4, 33.4, 25.7, 25.0, 23.7 ppm. The analytical data are in agreement with previously reported data.[30]

***N*-(1-Phenylethyl)acetamide (3ad):** By following general procedure A (0.021 g, 62 %), B (0.022 g, 65 %), C (0.027 g, 79 %), D (0.030 g, 88 %) or E (0.036 g, 70 % using 0.050 g of *tert*-butyl acetoacetate **4a**), *N*-(1-phenylethyl)acetamide (**3ad**) was obtained as a white solid after chromatography (PE/EtOAc, 1:1 → EtOAc/*i*PrOH, 8:2). R_f (DCM/MeOH, 95:5) = 0.42 [$KMnO_4$]. ^1H NMR (400 MHz, $CDCl_3$): δ = 7.37–7.28 (m, 4 H), 7.28–7.22 (m, 1 H), 5.95 (s, 1 H), 5.16–5.06 (m, 1 H), 1.96 (s, 3 H), 1.47 (d, J = 6.9 Hz, 3 H) ppm. ^{13}C NMR (101 MHz, $CDCl_3$): δ = 169.2, 143.3, 128.8, 127.4, 126.3, 48.9, 23.5, 21.8 ppm. The analytical data are in agreement with previously reported data.[29]

***N*-Octylacetamide (3ae):** By following general procedure A (0.027 g, 76 %), B (0.031 g, 87 %), C (0.027 g, 76 %) or E (0.033 g, 59 % using 0.050 g of *tert*-butyl acetoacetate **4a**), *N*-octylacetamide (**3ae**) was obtained as a colourless liquid after chromatography (PE/EtOAc, 1:1 → EtOAc/*i*PrOH, 8:2). R_f (DCM/MeOH, 95:5) = 0.32 [$KMnO_4$]. ^1H NMR (400 MHz, $CDCl_3$): δ = 5.75 (s, 1 H), 3.24–3.16 (m, 2 H), 1.94 (s, 3 H), 1.53–1.40 (m, 2 H), 1.33–1.17 (m, 10 H), 0.85 (t, J = 6.9 Hz, 3 H) ppm. ^{13}C NMR (101 MHz, $CDCl_3$): δ = 170.1, 39.8, 31.9, 29.7, 29.4, 29.3, 27.0, 23.4, 22.7, 14.1 ppm. The analytical data are in agreement with previously reported data.[31]

***N*-(4-Methoxybenzyl)acetamide (3af):** By following general procedure A (0.025 g, 67 %), B (0.033 g, 88 %), C (0.031 g, 83 %) or E (0.039 g, 69 % using 0.050 g of *tert*-butyl acetoacetate **4a**), *N*-(4-methoxybenzyl)acetamide (**3af**) was obtained as a white solid after chromatography (PE/EtOAc, 1:1 → EtOAc/*i*PrOH, 8:2). R_f (DCM/MeOH, 95:5) = 0.31 [$KMnO_4$]. ^1H NMR (400 MHz, $CDCl_3$): δ = 7.23–7.15 (m, 2 H), 6.88–6.81 (m, 2 H), 5.93 (s, 1 H), 4.32 (d, J = 5.6 Hz, 2 H), 3.78 (s, 3 H), 1.97 (s, 3 H) ppm. ^{13}C NMR (101 MHz, $CDCl_3$): δ = 169.9, 159.1, 130.5, 129.3, 114.2, 55.4, 43.3, 23.3 ppm. The analytical data are in agreement with previously reported data.[31]

***N*-[4-(Trifluoromethyl)benzyl]acetamide (3ag):** By following general procedure A (0.029 g, 64 %), B (0.036 g, 80 %), C (0.038 g, 84 %) or E (0.047 g, 69 % using 0.050 g of *tert*-butyl acetoacetate **4a**), *N*-[4-(trifluoromethyl)benzyl]acetamide (**3ag**) was obtained as a white solid after chromatography (PE/EtOAc, 1:1 → EtOAc/*i*PrOH, 8:2). R_f (DCM/MeOH, 95:5) = 0.40 [$KMnO_4$]. IR: $\tilde{\nu}$ = 3283, 3079, 2937, 2831, 1649, 1620, 1587, 1548, 1454, 1420, 1377, 1324, 1300, 1282, 1229, 1188, 1155, 1109, 1065, 1021, 958, 922, 840, 834, 820, 739, 727, 646, 637, 614, 594, 514, 475, 406 cm^{-1}. ^1H NMR (400 MHz, $CDCl_3$): δ = 7.56 (d, J = 8.0 Hz, 2 H), 7.39–7.33 (m, 2 H), 6.20 (s, 1 H), 4.44 (d, J = 6.0 Hz, 2 H), 2.01 (s, 3 H) ppm. ^{13}C NMR (101 MHz, $CDCl_3$): δ = 170.3, 142.6 (d, J = 1.3 Hz), 129.9 (q, J = 32.5 Hz), 128.0, 125.7 (q, J = 3.8 Hz), 124.2 (q, J = 272.0 Hz), 43.2, 23.2 ppm. LRMS (EI): m/z = 217 (33) [M^+], 174 (38) [$(M - Ac)^+$], 159 (22) [$(M - AcNH)^+$], 109 (19), 106 (100). HRMS (ESI): m/z calcd. for $C_{10}H_{11}F_3NO^+$: 218.0787; found 218.0788.

***N*-[1-(Naphthalen-2-yl)ethyl]acetamide (3ah):** By following general procedure A (0.032 g, 72 %), B (0.034 g, 77 %) or C (0.039 g, 88 %), *N*-[1-(naphthalen-2-yl)ethyl]acetamide (**3ah**) was obtained as a white solid after chromatography (PE/EtOAc, 1:1 → EtOAc/*i*PrOH, 8:2). R_f (DCM/MeOH, 95:5) = 0.36 [$KMnO_4$]. ^1H NMR (400 MHz, $CDCl_3$): δ = 7.84–7.77 (m, 3 H), 7.76–7.71 (m, 1 H), 7.52–7.38 (m, 3 H), 6.07 (d, J = 8.0 Hz, 1 H), 5.33–5.22 (m, 1 H), 1.98 (s, 3 H), 1.55 (d, J = 6.9 Hz, 3 H) ppm. ^{13}C NMR (101 MHz, $CDCl_3$): δ = 169.3, 140.7, 133.4, 132.8, 128.6, 128.0, 127.7, 126.3, 126.0, 124.9, 124.6, 48.9, 23.5, 21.7 ppm. The analytical data are in agreement with previously reported data.[32]

***N*-(4-Phenylbutyl)acetamide (3ai):** By following general procedure A (0.028 g, 70 %), B (0.033 g, 83 %), C (0.032 g, 80 %) or E (0.034 g, 57 % using 0.050 g of *tert*-butyl acetoacetate **4a**), *N*-(4-phenylbutyl)acetamide (**3ai**) was obtained as a white solid after chromatography (PE/EtOAc, 1:1 → EtOAc/*i*PrOH, 8:2). R_f (DCM/MeOH, 95:5) = 0.38 [$KMnO_4$]. IR: $\tilde{\nu}$ = 3291, 3084, 3064, 3026, 2932, 2859, 1637, 1552, 1495, 1453, 1437, 1368, 1292, 1176, 1104, 1030, 1001, 909, 745, 697, 602, 570, 479, 437 cm^{-1}. ^1H NMR (400 MHz, $CDCl_3$): δ = 7.31–7.23 (m, 2 H), 7.21–7.13 (m, 3 H), 5.70 (s, 1 H), 3.24 (td, J = 7.2, 5.8 Hz, 2 H), 2.62 (t, J = 7.5 Hz, 2 H), 1.94 (s, 3 H), 1.69–1.59 (m, 2 H), 1.57–1.46 (m, 2 H) ppm. ^{13}C NMR (101 MHz, $CDCl_3$): δ = 170.2, 142.2, 128.5, 128.4, 125.9, 39.6, 35.6, 29.3, 28.8, 23.4 ppm. LRMS (EI): m/z = 191 (15) [M^+], 100 (41) [$(M - Bn)^+$], 91 (100) [Bn^+], 87 (77), 73 (50). HRMS (ESI): m/z calcd. for $C_{12}H_{18}NO^+$: 192.1383; found 192.1378.

***(Z)*-*N*-(Octadec-9-en-1-yl)acetamide (3aj):** By following general procedure A (0.055 g, 85 %), B (0.056 g, 87 %) or C (0.056 g, 87 %), (*Z*)-*N*-(octadec-9-en-1-yl)acetamide (**3aj**) was obtained as a white solid after chromatography (PE/EtOAc, 1:1 → EtOAc/*i*PrOH, 8:2). R_f (DCM/MeOH, 95:5) = 0.46 [$KMnO_4$]. IR: $\tilde{\nu}$ = 3292, 3089, 3003, 2922, 2853, 1650, 1556, 1463, 1438, 1369, 1293, 1107, 1039, 967, 722, 603, 486 cm^{-1}. ^1H NMR (400 MHz, $CDCl_3$): δ = 5.78 (s, 1 H), 5.39–5.26 (m, 2 H), 3.19 (dd, J = 13.2, 7.0 Hz, 2 H), 2.03–1.91 (m, 4 H), 1.94 (s, 3 H), 1.45 (dd, J = 14.2, 7.0 Hz, 2 H), 1.37–1.17 (m, 22 H), 0.85 (t, J = 6.9 Hz, 3 H) ppm. ^{13}C NMR (101 MHz, $CDCl_3$): δ = 170.1, 130.0, 129.8, 39.8, 32.0, 29.8, 29.8, 29.7, 29.7, 29.6, 29.5, 29.4, 29.4, 29.3, 27.3, 27.3, 27.0, 23.3, 22.8, 14.2 ppm. LRMS (EI): m/z = 309 (15) [M^+], 86 (56), 73 (97), 72 (80), 60 (77), 55 (100). HRMS (ESI): m/z calcd. for $C_{20}H_{39}NONa^+$: 332.2924; found 332.2926.

***N*-Allylacetamide (3ak):** By following general procedure A (0.016 g, 79 %), B (0.018 g, 87 %) or C (0.017 g, 82 %), *N*-allylacetamide (**3ak**) was obtained as a colourless liquid after chromatography (PE/EtOAc, 1:1 → EtOAc/*i*PrOH, 8:2). R_f (DCM/MeOH, 95:5) = 0.43 [$KMnO_4$]. ^1H NMR (400 MHz, $CDCl_3$): δ = 5.81 (ddt, J = 17.1, 10.2, 5.7 Hz, 1 H), 5.16 (dq, J = 17.2, 1.7 Hz, 1 H), 5.10 (dq, J = 10.2, 1.4 Hz, 1 H), 3.84 (tt, J = 5.7, 1.5 Hz, 2 H), 1.98 (s, 3 H) ppm. ^{13}C NMR (101 MHz, $CDCl_3$): δ = 170.1, 134.3, 116.4, 42.1, 23.3 ppm. The analytical data are in agreement with previously reported data.[33]

***N*-(Prop-2-yn-1-yl)acetamide (3al):** By following general procedure A (0.013 g, 64 %), B (0.016 g, 79 %) or C (0.018 g, 89 %), *N*-(prop-2-yn-1-yl)acetamide (**3al**) was obtained as a white solid after chromatography (PE/EtOAc, 1:1 → EtOAc/*i*PrOH, 8:2). R_f (DCM/MeOH, 95:5) = 0.38 [$KMnO_4$]. ^1H NMR (400 MHz, $CDCl_3$): δ = 6.12 (s, 1 H), 4.02 (d, J = 2.6 Hz, 1 H), 4.01 (d, J = 2.6 Hz, 1 H), 2.21 (t, J = 2.6 Hz, 1 H), 1.99 (s, 3 H) ppm. ^{13}C NMR (101 MHz, $CDCl_3$): δ = 170.0, 79.7, 71.5, 29.3, 23.0 ppm. The analytical data are in agreement with previously reported data.[34]

***N*-(3,3-Diethoxypropyl)acetamide (3am):** By following general procedure A (0.027 g, 69 %), B (0.032 g, 81 %), C (0.032 g, 81 %) or E (0.035 g, 58 % using 0.050 g of *tert*-butyl acetoacetate **4a**), *N*-(3,3-diethoxypropyl)acetamide (**3am**) was obtained as a colourless liquid after chromatography (PE/EtOAc, 1:1 → EtOAc/*i*PrOH, 8:2). R_f (DCM/MeOH, 95:5) = 0.38 [$KMnO_4$]. ^1H NMR (400 MHz, $CDCl_3$): δ = 6.19 (s, 1 H), 4.53 (t, J = 5.2 Hz, 1 H), 3.66 (q, J = 7.1 Hz, 1 H), 3.64 (q, J = 7.1 Hz, 1 H), 3.49 (q, J = 7.0 Hz, 1 H), 3.46 (q, J = 7.0 Hz, 1 H), 3.32 (dd, J = 12.6, 5.5 Hz, 2 H), 1.91 (s, J = 2.5 Hz, 3 H), 1.79 (ddd, J = 7.4, 6.3, 5.2 Hz, 2 H), 1.19 (t, J = 7.1 Hz, 6 H) ppm. ^{13}C NMR (101 MHz, $CDCl_3$): δ = 169.9, 102.6, 62.1, 35.7, 33.0, 23.4, 15.4 ppm. The analytical data are in agreement with previously reported data.[35]

***tert*-Butyl (2-Acetamidoethyl)carbamate (3an):** By following general procedure A (0.028 g, 67 %), B (0.032 g, 76 %) or C (0.035 g, 83 %), *tert*-butyl (2-acetamidoethyl)carbamate (**3an**) was obtained as a white solid after chromatography (PE/EtOAc, 1:1 → EtOAc/*i*PrOH, 8:2). R_f (DCM/MeOH, 95:5) = 0.22 [$KMnO_4$]. ^1H NMR (400 MHz, $CDCl_3$): δ = 6.47 (s, 1 H), 5.14 (s, 1 H), 3.36–3.28 (m, 2 H), 3.28–3.14 (m, 2 H), 1.95 (s, 3 H), 1.41 (s, 9 H) ppm. ^{13}C NMR (101 MHz, $CDCl_3$): δ = 171.0, 157.1, 79.7, 40.8, 40.4, 28.5, 23.3 ppm. The analytical data are in agreement with previously reported data.[36]

***N*-[(1*R*,2*S*)-2-Hydroxy-2,3-dihydro-1*H*-inden-1-yl]acetamide (3ao):** By following general procedure A (0.028 g, 70 %) or E (0.045 g, 75 % using 50 mg, 0.32 mmol *tert*-butyl acetoacetate **4a**), *N*-[(1*R*,2*S*)-2-hydroxy-2,3-dihydro-1*H*-inden-1-yl]acetamide (**3ao**) was obtained as a white solid after chromatography (PE/EtOAc, 1:1 → EtOAc/*i*PrOH, 8:2). R_f (DCM/MeOH, 95:5) = 0.16 [$KMnO_4$]. IR: $\tilde{\nu}$ = 3443, 3295, 3074, 3045, 3020, 2972, 2932, 2837, 1636, 1616, 1538, 1478, 1457, 1426, 1372, 1324, 1281, 1247, 1182, 1156, 1136, 1083, 1050, 1020, 999, 977, 885, 862, 817, 753, 733, 648, 591, 565, 534, 505, 476, 421 cm^{-1}. ^1H NMR (600 MHz, $CDCl_3$): δ = 7.28–7.20 (m, 4 H), 6.20 (d, *J* = 7.9 Hz, 1 H), 5.35 (ddd, *J* = 8.3, 5.2, 1.1 Hz, 1 H), 4.61 (td, *J* = 5.2, 2.4 Hz, 1 H), 3.16 (dd, *J* = 16.4, 5.3 Hz, 1 H), 2.93 (dd, *J* = 16.5, 2.4 Hz, 1 H), 2.41 (s, 1 H), 2.09 (s, 3 H) ppm. ^{13}C NMR (151 MHz, $CDCl_3$): δ = 171.0, 140.7, 140.1, 128.4, 127.4, 125.5, 124.7, 73.8, 57.8, 39.9, 23.5 ppm. LRMS (EI): *m*/*z* = 173 (35) [(M – H_2O)$^+$], 131 (100) [(M – AcOH)$^+$], 103 (45), 77 (41). HRMS (ESI): *m*/*z* calcd. for $C_{11}H_{13}NO_2Na^+$: 214.0838; found 214.0838.

***N*-(4-Hydroxyphenethyl)acetamide (3ap):** By following general procedure A (0.028 g, 75 %) or E (0.040 g, 71 % using 0.050 g of *tert*-butyl acetoacetate **4a**), *N*-(4-hydroxyphenethyl)acetamide (**3ap**) was obtained as a white solid after chromatography (PE/EtOAc, 1:1 → EtOAc/*i*PrOH, 8:2). R_f (DCM/MeOH, 95:5) = 0.14 [$KMnO_4$]. IR: $\tilde{\nu}$ = 3330, 3099, 3043, 3012, 2971, 2934, 2855, 2805, 2754, 2693, 2622, 2511, 1628, 1593, 1565, 1514, 1459, 1434, 1377, 1360, 1307, 1245, 1172, 1104, 1040, 1016, 994, 963, 932, 910, 855, 838, 810, 726, 711, 647, 637, 600, 552, 493, 460, 422 cm^{-1}. ^1H NMR (400 MHz, [D_6]DMSO): δ = 9.15 (s, 1 H), 7.90–7.77 (m, 1 H), 7.02–6.92 (m, 2 H), 6.72–6.61 (m, 2 H), 3.23–3.12 (m, 2 H), 2.61–2.53 (m, 2 H), 1.78 (s, 3 H) ppm. ^{13}C NMR (101 MHz, [D_6]DMSO): δ = 169.0, 155.6, 129.5, 129.4, 115.1, 40.6, 34.4, 22.6 ppm. LRMS (EI): *m*/*z* = 179 (2) [M$^+$], 120 (100) [(M – $AcNH_2$)$^+$], 107 (44), 91 (9), 77 (28). HRMS (ESI): *m*/*z* calcd. for $C_{10}H_{13}NO_2Na^+$: 202.0838; found 202.0835.

***N*-[2-(1*H*-Indol-3-yl)ethyl]acetamide (3aq):** By following general procedure A (0.026 g, 62 %) or C (0.032 g, 76 %), *N*-[2-(1*H*-indol-3-yl)ethyl]acetamide (**3aq**) was obtained as a yellow solid after chromatography (PE/EtOAc, 1:1 → EtOAc/*i*PrOH, 8:2). R_f (DCM/MeOH, 95:5) = 0.27 [$KMnO_4$]. ^1H NMR (400 MHz, $CDCl_3$): δ = 8.50 (s, 1 H), 7.63–7.55 (m, 1 H), 7.36 (dt, *J* = 8.1, 0.9 Hz, 1 H), 7.23–7.17 (m, 1 H), 7.12 (ddd, *J* = 8.0, 7.0, 1.0 Hz, 1 H), 6.99 (d, *J* = 2.3 Hz, 1 H), 5.70 (s, 1 H), 3.58 (td, *J* = 6.8, 5.7 Hz, 2 H), 2.96 (td, *J* = 6.8, 0.9 Hz, 2 H), 1.91 (s, 3 H) ppm. ^{13}C NMR (101 MHz, $CDCl_3$): δ = 170.4, 136.6, 127.5, 122.2, 122.2, 119.5, 118.7, 112.9, 111.5, 40.0, 25.4, 23.4 ppm. The analytical data are in agreement with previously reported data.[37]

***N*-Benzyl-*N*-methylacetamide (3ar):** By following general procedure A (0.019 g, 56 %), B (0.017 g, 50 %) or C (0.019 g, 56 %), *N*-benzyl-*N*-methylacetamide (**3ar**) was obtained as a colourless liquid after chromatography (PE/EtOAc, 1:1 → EtOAc/*i*PrOH, 8:2). R_f (DCM/MeOH, 95:5) = 0.51 [$KMnO_4$]. ^1H NMR (400 MHz, $CDCl_3$, two rotamers): δ = 7.40–7.21 (m, 8 H), 7.21–7.13 (m, 2 H), 4.58 (s, 2 H), 4.52 (s, 2 H), 2.94 (s, 3 H), 2.91 (s, 3 H), 2.18–2.12 (m, 6 H) ppm. ^{13}C NMR (101 MHz, $CDCl_3$, two rotamers): δ = 171.1, 170.8, 137.5, 136.7, 129.1, 128.7, 128.1, 127.7, 127.4, 126.4, 54.4, 50.7, 35.6, 33.8, 21.9, 21.6 ppm. The analytical data are in agreement with previously reported data.[38]

***N*-Benzylbenzamide (3ba):** By following general procedure A (0.026 g, 74 %), B (0.030 g, 86 %) or C (0.029 g, 83 %), *N*-benzylbenzamide (**3ba**) was obtained as a white solid after chromatography (PE/EtOAc, 9:1 → 1:1). R_f (PE/EtOAc, 1:1) = 0.63 [$KMnO_4$]. ^1H NMR (400 MHz, $CDCl_3$): δ = 7.82–7.77 (m, 2 H), 7.52–7.46 (m, 1 H), 7.44–7.38 (m, 2 H), 7.36–7.34 (m, 4 H), 7.34–7.27 (m, 1 H), 6.56 (s, 1 H), 4.63 (d, *J* = 5.7 Hz, 2 H) ppm. ^{13}C NMR (101 MHz, $CDCl_3$): δ = 167.5, 138.4, 134.5, 131.6, 128.9, 128.7, 128.0, 127.7, 127.1, 44.2 ppm. The analytical data are in agreement with previously reported data.[15b]

***N*-(1-Cyclohexylethyl)benzamide (3bb):** By following general procedure A (0.026 g, 68 %), B (0.035 g, 91 %) or C (0.035 g, 91 %), *N*-(1-cyclohexylethyl)benzamide (**3bb**) was obtained as a white solid after chromatography (PE/EtOAc, 9:1 → 1:1). R_f (PE/EtOAc, 1:1) = 0.71 [$KMnO_4$]. ^1H NMR (400 MHz, $CDCl_3$): δ = 7.79–7.72 (m, 2 H), 7.51–7.45 (m, 1 H), 7.45–7.37 (m, 2 H), 6.02 (d, *J* = 8.5 Hz, 1 H), 4.13–4.01 (m, 1 H), 1.86–1.61 (m, 5 H), 1.48–1.37 (m, 1 H), 1.29–1.20 (m, 2 H), 1.18 (d, *J* = 6.8 Hz, 3 H), 1.16–0.96 (m, 3 H) ppm. ^{13}C NMR (101 MHz, $CDCl_3$): δ = 167.0, 135.3, 131.3, 128.6, 126.9, 50.0, 43.4, 29.3, 29.3, 26.5, 26.3, 26.3, 18.1 ppm. The analytical data are in agreement with previously reported data.[39]

***N*-Cyclohexylbenzamide (3bc):** By following general procedure A (0.024 g, 71 %), B (0.031 g, 92 %) or C (0.031 g, 92 %), *N*-cyclohexylbenzamide (**3bc**) was obtained as a white solid after chromatography (PE/EtOAc, 9:1 → 1:1). R_f (PE/EtOAc, 1:1) = 0.66 [$KMnO_4$]. ^1H NMR (400 MHz, $CDCl_3$): δ = 7.79–7.71 (m, 2 H), 7.51–7.43 (m, 1 H), 7.43–7.36 (m, 2 H), 6.06 (s, 1 H), 4.04–3.90 (m, 1 H), 2.07–1.97 (m, 2 H), 1.80–1.69 (m, 2 H), 1.69–1.59 (m, 1 H), 1.48–1.34 (m, 2 H), 1.30–1.13 (m, 3 H) ppm. ^{13}C NMR (101 MHz, $CDCl_3$): δ = 166.8, 135.2, 131.3, 128.6, 127.0, 48.8, 33.3, 25.7, 25.0 ppm. The analytical data are in agreement with previously reported data.[39]

***N*-(1-Phenylethyl)benzamide (3bd):** By following general procedure A (0.025 g, 67 %), B (0.033 g, 89 %), C (0.032 g, 86 %) or D (0.034 g, 90 %), *N*-(1-phenylethyl)benzamide (**3bd**) was obtained as a white solid after chromatography (PE/EtOAc, 9:1 → 1:1). R_f (PE/EtOAc, 1:1) = 0.69 [$KMnO_4$]. ^1H NMR (400 MHz, $CDCl_3$): δ = 7.80–7.75 (m, 2 H), 7.51–7.45 (m, 1 H), 7.44–7.32 (m, 6 H), 7.30–7.25 (m, 1 H), 6.47 (d, *J* = 6.8 Hz, 1 H), 5.34 (p, *J* = 7.0 Hz, 1 H), 1.60 (d, *J* = 6.9 Hz, 3 H) ppm. ^{13}C NMR (101 MHz, $CDCl_3$): δ = 166.7, 143.3, 134.7, 131.5, 128.8, 128.6, 127.5, 127.1, 126.4, 49.3, 21.8 ppm. The analytical data are in agreement with previously reported data.[40]

***N*-Octylbenzamide (3be):** By following general procedure A (0.033 g, 86 %), B (0.034 g, 88 %) or C (0.034 g, 88 %), *N*-octylbenzamide (**3be**) was obtained as a white solid after chromatography (PE/EtOAc, 9:1 → 1:1). R_f (PE/EtOAc, 1:1) = 0.68 [$KMnO_4$]. ^1H NMR (400 MHz, $CDCl_3$): δ = 7.79–7.72 (m, 2 H), 7.50–7.43 (m, 1 H), 7.43–7.36 (m, 2 H), 6.33 (s, 1 H), 3.46–3.38 (m, 2 H), 1.65–1.55 (m, 2 H), 1.40–1.21 (m, 10 H), 0.87 (t, *J* = 6.9 Hz, 3 H) ppm. ^{13}C NMR (101 MHz, $CDCl_3$): δ = 167.7, 135.0, 131.4, 128.6, 127.0, 40.3, 31.9, 29.8, 29.4, 29.3, 27.1, 22.7, 14.2 ppm. The analytical data are in agreement with previously reported data.[41]

***N*-(4-Methoxybenzyl)benzamide (3bf):** By following general procedure A (0.030 g, 75 %), B (0.035 g, 88 %) or C (0.034 g, 85 %), *N*-(4-methoxybenzyl)benzamide (**3bf**) was obtained as a yellowish solid after chromatography (PE/EtOAc, 9:1 → 1:1). R_f (PE/EtOAc, 1:1) = 0.51 [$KMnO_4$]. ^1H NMR (400 MHz, $CDCl_3$): δ = 7.81–7.74 (m, 2 H), 7.51–7.44 (m, 1 H), 7.44–7.36 (m, 2 H), 7.30–7.23 (m, 2 H), 6.90–6.83 (m, 2 H), 6.55 (s, 1 H), 4.55 (d, *J* = 5.6 Hz, 2 H), 3.79 (s, 3 H) ppm. ^{13}C NMR (101 MHz, $CDCl_3$): δ = 167.4, 159.2, 134.6, 131.6, 130.4,

129.4, 128.6, 127.1, 114.3, 55.4, 43.7 ppm. The analytical data are in agreement with previously reported data.[42]

***N*-[4-(Trifluoromethyl)benzyl]benzamide (3bg):** By following general procedure A (0.034 g, 74 %), B (0.043 g, 93 %) or C (0.040 g, 87 %), *N*-[4-(trifluoromethyl)benzyl]benzamide (**3bg**) was obtained as a white solid after chromatography (PE/EtOAc, 9:1 → 1:1). R_f (PE/EtOAc, 1:1) = 0.58 [$KMnO_4$]. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.84–7.75 (m, 2 H), 7.56 (d, J = 8.1 Hz, 2 H), 7.54–7.47 (m, 1 H), 7.45–7.37 (m, 4 H), 6.84 (s, 1 H), 4.65 (d, J = 6.0 Hz, 2 H) ppm. ^{13}C NMR (101 MHz, $CDCl_3$): δ = 167.6, 142.6 (d, J = 1.4 Hz), 134.0, 131.7, 129.9 (q, J = 32.5 Hz), 128.8, 128.0, 127.1, 125.8 (q, J = 3.8 Hz), 124.2 (q, J = 272.0 Hz), 43.4 ppm. The analytical data are in agreement with previously reported data.[43]

***N*-[1-(Naphthalen-2-yl)ethyl]benzamide (3bh):** By following general procedure A (0.030 g, 66 %), B (0.037 g, 81 %) or C (0.037 g, 81 %), *N*-[1-(naphthalen-2-yl)ethyl]benzamide (**3bh**) was obtained as a white solid after chromatography (PE/EtOAc, 9:1 → 1:1). R_f (PE/EtOAc, 1:1) = 0.67 [$KMnO_4$]. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.85–7.77 (m, 6 H), 7.53–7.44 (m, 4 H), 7.44–7.37 (m, 2 H), 6.59 (d, J = 7.9 Hz, 1 H), 5.50 (p, J = 7.0 Hz, 1 H), 1.68 (d, J = 6.9 Hz, 3 H) ppm. ^{13}C NMR (101 MHz, $CDCl_3$): δ = 166.8, 140.6, 134.7, 133.5, 132.9, 131.6, 128.7, 128.6, 128.0, 127.7, 127.1, 126.3, 126.0, 124.9, 124.7, 49.4, 21.7 ppm. The analytical data are in agreement with previously reported data.[44]

***N*-(4-Phenylbutyl)benzamide (3bi):** By following general procedure A (0.036 g, 86 %), B (0.036 g, 86 %) or C (0.039 g, 93 %), *N*-(4-phenylbutyl)benzamide (**3bi**) was obtained as a white solid after chromatography (PE/EtOAc, 9:1 → 1:1). R_f (PE/EtOAc, 1:1) = 0.60 [$KMnO_4$]. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.79–7.71 (m, 2 H), 7.52–7.44 (m, 1 H), 7.45–7.36 (m, 2 H), 7.32–7.23 (m, 2 H), 7.23–7.13 (m, 3 H), 6.32 (s, 1 H), 3.46 (q, J = 6.7 Hz, 2 H), 2.66 (t, J = 7.3 Hz, 2 H), 1.77–1.59 (m, 4 H) ppm. ^{13}C NMR (101 MHz, $CDCl_3$): δ = 167.7, 142.2, 134.9, 131.4, 128.6, 128.5, 128.4, 127.0, 125.9, 40.0, 35.6, 29.4, 28.8 ppm. The analytical data are in agreement with previously reported data.[45]

***(Z)*-*N*-(Octadec-9-en-1-yl)benzamide (3bj):** By following general procedure A (0.056 g, 91 %), B (0.062 g, 100 %) or C (0.053 g, 86 %), (*Z*)-*N*-(octadec-9-en-1-yl)benzamide (**3bj**) was obtained as a white solid after chromatography (PE/EtOAc, 9:1 → 1:1). R_f (PE/EtOAc, 1:1) = 0.83 [$KMnO_4$]. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.78–7.73 (m, 2 H), 7.49–7.43 (m, 1 H), 7.42–7.36 (m, 2 H), 6.33 (s, 1 H), 5.40–5.29 (m, 2 H), 3.42 (dd, J = 13.3, 6.9 Hz, 2 H), 2.08–1.90 (m, 4 H), 1.64–1.55 (m, 2 H), 1.41–1.21 (m, 22 H), 0.87 (t, J = 6.9 Hz, 3 H) ppm. ^{13}C NMR (101 MHz, $CDCl_3$): δ = 167.6, 135.0, 131.3, 130.1, 129.9, 128.6, 127.0, 40.2, 32.7, 32.0, 29.9, 29.8, 29.8, 29.8, 29.7, 29.6, 29.6, 29.4, 29.4, 29.3, 27.3, 27.3, 27.1, 22.8, 14.2 ppm. The analytical data are in agreement with previously reported data.[46]

***N*-Allylbenzamide (3bk):** By following general procedure A (0.021 g, 79 %), B (0.023 g, 86 %) or C (0.024 g, 90 %), *N*-allylbenzamide (**3bk**) was obtained as a colourless liquid after chromatography (PE/EtOAc, 9:1 → 1:1). R_f (PE/EtOAc, 1:1) = 0.56 [$KMnO_4$]. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.81–7.75 (m, 2 H), 7.52–7.45 (m, 1 H), 7.45–7.37 (m, 2 H), 6.40 (s, 1 H), 5.93 (ddt, J = 17.1, 10.2, 5.7 Hz, 1 H), 5.25 (ddd, J = 17.1, 3.1, 1.6 Hz, 1 H), 5.17 (dq, J = 10.2, 1.4 Hz, 1 H), 4.07 (tt, J = 5.7, 1.6 Hz, 2 H) ppm. ^{13}C NMR (101 MHz, $CDCl_3$): δ = 167.5, 134.6, 134.3, 131.6, 128.7, 127.1, 116.7, 42.5 ppm. The analytical data are in agreement with previously reported data.[33]

***N*-(Prop-2-yn-1-yl)benzamide (3bl):** By following general procedure A (0.021 g, 80 %), B (0.024 g, 91 %) or C (0.021 g, 80 %), *N*-(prop-2-yn-1-yl)benzamide (**3bl**) was obtained as a white solid after chromatography (PE/EtOAc, 9:1 → 1:1). R_f (PE/EtOAc, 1:1) = 0.61 [$KMnO_4$]. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.81–7.75 (m, 2 H), 7.53–7.47 (m, 1 H), 7.46–7.39 (m, 2 H), 6.48 (s, 1 H), 4.25 (dd, J = 5.2, 2.6 Hz, 2 H), 2.27 (t, J = 2.6 Hz, 1 H) ppm. ^{13}C NMR (101 MHz, $CDCl_3$): δ = 167.3, 133.9, 131.9, 128.7, 127.2, 79.6, 72.0, 29.9 ppm. The analytical data are in agreement with previously reported data.[47]

***N*-(3,3-Diethoxypropyl)benzamide (3bm):** By following general procedure A (0.033 g, 79 %), B (0.039 g, 94 %) or C (0.039 g, 94 %), *N*-(3,3-diethoxypropyl)benzamide (**3bm**) was obtained as a white solid after chromatography (PE/EtOAc, 9:1 → 1:1). R_f (PE/EtOAc, 1:1) = 0.36 [$KMnO_4$]. IR: $\tilde{\nu}$ = 3325, 3065, 3032, 2974, 2930, 2878, 1637, 1603, 1578, 1536, 1489, 1445, 1373, 1344, 1307, 1294, 1222, 1122, 1055, 973, 890, 846, 803, 693, 669, 615, 508 cm^{-1}. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.78–7.72 (m, 2 H), 7.48–7.42 (m, 1 H), 7.42–7.35 (m, 2 H), 7.07 (s, 1 H), 4.63 (t, J = 5.0 Hz, 1 H), 3.71 (q, J = 7.1 Hz, 1 H), 3.69 (q, J = 7.1 Hz, 1 H), 3.56 (ddd, J = 7.6, 6.0, 5.3 Hz, 2 H), 3.53 (q, J = 7.1 Hz, 1 H), 3.50 (q, J = 7.0 Hz, 1 H), 1.92 (ddd, J = 7.7, 6.1, 4.9 Hz, 2 H), 1.21 (t, J = 7.0 Hz, 6 H) ppm. ^{13}C NMR (101 MHz, $CDCl_3$): δ = 167.2, 134.9, 131.3, 128.5, 126.9, 103.1, 62.3, 36.1, 32.9, 15.5 ppm. LRMS (EI): m/z = 222 (2) [(M – Et)$^+$], 205 (5) [(M – EtOH)$^+$], 176 (8) [(M – EtOH – Et)$^+$], 160 (10) [(M – EtOH – EtO)$^+$], 105 (100) [PhCO$^+$], 77 (49) [Ph$^+$]. HRMS (ESI): m/z calcd. for $C_{14}H_{21}NO_3Na^+$: 274.1414; found 274.1419.

***tert*-Butyl (2-Benzamidoethyl)carbamate (3bn):** By following general procedure A (0.029 g, 66 %), B (0.037 g, 85 %) or C (0.037 g, 85 %), *tert*-butyl (2-benzamidoethyl)carbamate (**3bn**) was obtained as a white solid after chromatography (PE/EtOAc, 9:1 → 1:1). R_f (PE/EtOAc, 1:1) = 0.24 [$KMnO_4$]. IR: $\tilde{\nu}$ = 3354, 3322, 3083, 3066, 2990, 2970, 2935, 2872, 1685, 1636, 1603, 1579, 1525, 1490, 1447, 1390, 1367, 1327, 1276, 1250, 1234, 1152, 1075, 1035, 974, 922, 878, 855, 800, 783, 764, 691, 642, 583, 519, 467 cm^{-1}. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.81 (d, J = 7.2 Hz, 2 H), 7.50–7.43 (m, 1 H), 7.43–7.36 (m, 2 H), 7.26 (s, 1 H), 5.14 (s, 1 H), 3.54 (dd, J = 11.0, 5.2 Hz, 2 H), 3.38 (dd, J = 11.1, 5.7 Hz, 2 H), 1.41 (s, 9 H) ppm. ^{13}C NMR (101 MHz, $CDCl_3$): δ = 168.0, 157.6, 134.3, 131.5, 128.6, 127.1, 80.0, 42.1, 40.1, 28.5 ppm. LRMS (EI): m/z = 208 (3) [(M – tBu + H)$^+$], 134 (47) [PhCONHCH$_2^+$], 105 (100) [PhCO$^+$], 77 (50) [Ph$^+$], 57 (51) [tBu$^+$]. HRMS (ESI): m/z calcd. for $C_{14}H_{20}N_2O_3Na^+$: 287.1366; found 287.1365.

***N*-[(1*R*,2*S*)-2-Hydroxy-2,3-dihydro-1*H*-inden-1-yl]benzamide (3bo):** By following general procedure A, *N*-[(1*R*,2*S*)-2-hydroxy-2,3-dihydro-1*H*-inden-1-yl]benzamide (**3bo**; 0.030 g, 72 %) was obtained as a white solid after chromatography (PE/EtOAc, 9:1 → EtOAc). R_f (PE/EtOAc, 1:1) = 0.38 [$KMnO_4$]. IR: $\tilde{\nu}$ = 3295, 3065, 3052, 3021, 2935, 2918, 1637, 1602, 1577, 1525, 1480, 1456, 1345, 1279, 1207, 1184, 1172, 1155, 1110, 1080, 1056, 1023, 1005, 953, 925, 905, 847, 835, 797, 748, 695, 665, 621, 582, 559, 514, 445, 429 cm^{-1}. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.86–7.80 (m, 2 H), 7.55–7.47 (m, 1 H), 7.46–7.39 (m, 2 H), 7.37–7.31 (m, 1 H), 7.30–7.18 (m, 4 H), 6.88 (d, J = 8.3 Hz, 1 H), 5.58 (ddd, J = 8.4, 5.1, 1.1 Hz, 1 H), 4.72 (td, J = 5.2, 2.2 Hz, 1 H), 3.23 (dd, J = 16.3, 5.0 Hz, 1 H), 2.99 (dd, J = 16.6, 2.0 Hz, 1 H), 2.43 (s, 1 H) ppm. ^{13}C NMR (151 MHz, $CDCl_3$): δ = 168.2, 140.9, 140.1, 134.3, 131.8, 128.7, 128.5, 127.4, 127.3, 125.5, 124.8, 73.9, 58.1, 40.1 ppm. LRMS (EI): m/z = 235 (12) [(M – H_2O)$^+$], 105 (100) [PhCO$^+$], 77 (54) [Ph$^+$]. HRMS (ESI): m/z calcd. for $C_{16}H_{15}NO_2Na^+$: 276.0995; found 276.0994.

***N*-(4-Hydroxyphenethyl)benzamide (3bp):** By following general procedure A, *N*-(4-hydroxyphenethyl)benzamide (**3bp**; 0.032 g, 80 %) was obtained as a white solid after chromatography (PE/EtOAc, 9:1 → 3:7). R_f (PE/EtOAc, 1:1) = 0.29 [$KMnO_4$]. ^{1}H NMR (400 MHz, [D$_6$]DMSO): δ = 9.17 (s, 1 H), 8.50 (t, J = 5.6 Hz, 1 H), 7.87–7.77 (m, 2 H), 7.55–7.40 (m, 3 H), 7.08–6.97 (m, 2 H), 6.73–6.64 (m, 2 H), 3.47–3.39 (m, 2 H), 2.77–2.69 (m, 2 H) ppm. ^{13}C NMR

(101 MHz, [D$_6$]DMSO): δ = 166.1, 155.6, 134.7, 131.0, 129.6, 129.5, 128.2, 127.1, 115.1, 41.2, 34.3 ppm. The analytical data are in agreement with previously reported data.[48]

***N*-[2-(1*H*-Indol-3-yl)ethyl]benzamide (3bq):** By following general procedure A (0.032 g, 73 %) or C (0.032 g, 73 %), *N*-[2-(1*H*-indol-3-yl)ethyl]benzamide (**3bq**) was obtained as a brown solid after chromatography (PE/EtOAc, 8:2 → EtOAc). R_f (DCM/MeOH, 95:5) = 0.33 [KMnO$_4$]. ^{1}H NMR (600 MHz, CDCl$_3$): δ = 8.43 (s, 1 H), 7.76–7.60 (m, 3 H), 7.50–7.43 (m, 1 H), 7.43–7.31 (m, 3 H), 7.24–7.17 (m, 1 H), 7.16–7.09 (m, 1 H), 7.02 (d, *J* = 2.4 Hz, 1 H), 6.37 (s, 1 H), 3.86–3.73 (m, 2 H), 3.17–3.02 (m, 2 H) ppm. ^{13}C NMR (151 MHz, CDCl$_3$): δ = 167.7, 136.6, 134.8, 131.5, 128.6, 127.4, 127.0, 122.3, 122.3, 119.6, 118.8, 112.9, 111.5, 40.5, 25.4 ppm. The analytical data are in agreement with previously reported data.[46]

***N*-Benzyl-*N*-methylbenzamide (3br):** By following general procedure A (0.017 g, 46 %), B (0.033 g, 89 %) or C (0.032 g, 86 %), *N*-benzyl-*N*-methylbenzamide (**3br**) was obtained as a white solid after chromatography (PE/EtOAc, 9:1 → 1:1). R_f (PE/EtOAc, 1:1) = 0.53 [KMnO$_4$]. ^{1}H NMR (400 MHz, CDCl$_3$, two rotamers): δ = 7.49–7.43 (m, 4 H), 7.43–7.24 (m, 14 H), 7.17 (s, 2 H), 4.76 (s, 2 H), 4.51 (s, 2 H), 3.03 (s, 3 H), 2.86 (s, 3 H) ppm. ^{13}C NMR (101 MHz, CDCl$_3$, two rotamers): δ = 172.3, 171.6, 137.1, 136.7, 136.3, 129.7, 128.9, 128.8, 128.5, 128.3, 127.6, 127.4, 127.0, 126.9, 122.9, 120.0, 55.2, 50.9, 37.1, 33.2 ppm. The analytical data are in agreement with previously reported data.[49]

Morpholino(phenyl)methanone (3bs): By following general procedure A (0.015 g, 47 %), B (0.022 g, 70 %) or C (0.024 g, 76 %), morpholino(phenyl)methanone (**3bs**) was obtained as a colourless liquid after chromatography (PE/EtOAc, 9:1 → EtOAc). R_f (PE/EtOAc, 1:1) = 0.23 [KMnO$_4$]. ^{1}H NMR (400 MHz, CDCl$_3$): δ = 7.43–7.37 (m, 5 H), 3.90–3.24 (m, 8 H) ppm. ^{13}C NMR (101 MHz, CDCl$_3$): δ = 170.5, 135.4, 130.0, 128.7, 127.2, 67.0, 48.3, 42.6 ppm. The analytical data are in agreement with previously reported data.[50]

***N*-Phenylbenzamide (3bt):** By following general procedure B (0.007 g, 21 %) or C (0.020 g, 61 %), *N*-phenylbenzamide (**3bt**) was obtained as a white solid after chromatography (PE/EtOAc, 9:1 → 1:1). R_f (PE/EtOAc, 1:1) = 0.66 [KMnO$_4$]. ^{1}H NMR (400 MHz, CDCl$_3$): δ = 7.96–7.83 (m, 3 H), 7.68–7.61 (m, 2 H), 7.58–7.51 (m, 1 H), 7.51–7.44 (m, 2 H), 7.40–7.33 (m, 2 H), 7.20–7.12 (m, 1 H) ppm. ^{13}C NMR (101 MHz, CDCl$_3$): δ = 165.9, 138.1, 135.2, 132.0, 129.2, 128.9, 127.2, 124.7, 120.4 ppm. The analytical data are in agreement with previously reported data.[51]

***N*-Benzyl-4-methylbenzamide (3ca):** By following general procedure E using *tert*-butyl 3-oxo-3-(*p*-tolyl)propanoate (**4d**; 50 mg, 0.21 mmol), *N*-benzyl-4-methylbenzamide (**3ca**, 0.036 g, 74 %) was obtained as a yellow solid after chromatography (EA/PE, 1:9 → 6:4). R_f = 0.93 (EA/PE, 6:4) [Cl$_2$]. ^{1}H NMR (400 MHz, CDCl$_3$): δ = 7.79–7.66 (m, 2 H), 7.40–7.25 (m, 5 H), 7.26–7.21 (m, 2 H), 6.52 (br. s., 1 H), 4.62 (d, *J* = 5.6 Hz, 2 H), 2.39 (s, 3 H) ppm. ^{13}C NMR (101 MHz, CDCl$_3$): δ = 167.4, 142.0, 138.5, 131.7, 129.3, 128.9, 128.0, 127.7, 127.1, 44.2, 21.5 ppm. The analytical data are in agreement with previously reported data.[52]

4-Methyl-*N*-(1-phenylethyl)benzamide (3cd): By following general procedure D, 4-methyl-*N*-(1-phenylethyl)benzamide (**3cd**; 0.038 g, 99 %) was obtained as a white solid after chromatography (PE/EtOAc, 3:7 → EtOAc). ^{1}H NMR (400 MHz, CDCl$_3$): δ = 7.68 (d, *J* = 8.2 Hz, 2 H), 7.42–7.31 (m, 4 H), 7.30–7.24 (m, 1 H), 7.23–7.17 (m, 2 H), 6.43 (d, *J* = 7.9 Hz, 1 H), 5.33 (p, *J* = 7.1 Hz, 1 H), 2.38 (s, 3 H), 1.59 (d, *J* = 6.9 Hz, 3 H) ppm. ^{13}C NMR (101 MHz, CDCl$_3$): δ = 166.6, 143.4, 141.9, 131.9, 129.3, 128.8, 127.5, 127.1, 126.4, 49.2, 21.9, 21.5 ppm. The analytical data are in agreement with previously reported data.[53]

***N*-Benzyl-4-methoxybenzamide (3da):** By following general procedure E using *tert*-butyl 5-methyl-3-oxohexanoate (**4c**; 50 mg, 0.20 mmol), *N*-benzyl-4-methoxybenzamide (**3da**; 0.034 g, 70 %) was obtained as a yellow solid after chromatography (EA/PE, 2:8 → 6:4). R_f = 0.68 (EA/PE, 6:4) [Cl$_2$]. ^{1}H NMR (600 MHz, CDCl$_3$): δ = 7.80–7.70 (m, 2 H), 7.40–7.20 (m, 5 H), 6.96–6.83 (m, 2 H), 6.45 (br. s., 1 H), 4.62 (d, *J* = 5.6 Hz, 2 H), 3.83 (s, 3 H) ppm. ^{13}C NMR (151 MHz, CDCl$_3$): δ = 167.0, 162.4, 138.6, 128.9, 128.9, 128.0, 127.6, 126.9, 113.9, 55.5, 44.2 ppm. The analytical data are in agreement with previously reported data.[54]

4-Methoxy-*N*-(1-phenylethyl)benzamide (3dd): By following general procedure D, 4-methoxy-*N*-(1-phenylethyl)benzamide (**3dd**; 0.037 g, 96 %) was obtained as a white solid after chromatography (PE/EtOAc, 3:7 → EtOAc). ^{1}H NMR (400 MHz, CDCl$_3$): δ = 7.79–7.71 (m, 2 H), 7.41–7.30 (m, 4 H), 7.30–7.23 (m, 1 H), 6.93–6.85 (m, 2 H), 6.40 (d, *J* = 7.2 Hz, 1 H), 5.37–5.27 (m, 1 H), 3.82 (s, 3 H), 1.59 (d, *J* = 6.9 Hz, 3 H) ppm. ^{13}C NMR (101 MHz, CDCl$_3$): δ = 166.2, 162.3, 143.5, 128.9, 128.8, 127.4, 127.0, 126.4, 113.8, 55.5, 49.2, 21.9 ppm. The analytical data are in agreement with previously reported data.[55]

4-Methoxy-*N*-{(*S*)-(6-methoxyquinolin-4-yl)[(1*S*,2*S*,4*S*,5*R*)-5-vinylquinuclidin-2-yl]methyl}benzamide (3du): (1*S*,2*S*,4*S*,5*R*)-2-[(*S*)-Ammonio(6-methoxyquinolin-1-ium-4-yl)methyl]-5-vinylquinuclidin-1-ium chloride (0.060 g) was dissolved in saturated aqueous NaHCO$_3$ and the solution was extracted with DCM. The organic phase was dried with MgSO$_4$ and evaporated to obtain (*S*)-(6-methoxyquinolin-4-yl)[(1*S*,2*S*,4*S*,5*R*)-5-vinylquinuclidin-2-yl]methanamine (0.043 g, 0.13 mmol, 1.00 equiv.), which was dissolved in THF (0.27 mL, 0.50 M). *tert*-Butyl 2,2-diazido-3-(4-methoxyphenyl)-3-oxopropanoate (**1d**; 0.044 g, 0.13 mmol, 1.00 equiv.) and cesium carbonate (0.044 g, 0.13 mmol, 1.00 equiv.) were added to the solution. The resulting suspension was stirred for 20 h. The suspension was purified by chromatography over silica (PE/EtOAc/NEt$_3$, 50:45:5 → EtOAc/*i*PrOH/MeOH/NEt$_3$, 70:15:10:5) to give 4-methoxy-*N*-{(*S*)-(6-methoxyquinolin-4-yl)[(1*S*,2*S*,4*S*,5*R*)-5-vinylquinuclidin-2-yl]-methyl}benzamide (**3du**; 0.047 g, 77 %). R_f (DCM/MeOH, 9:1) = 0.32 [KMnO$_4$]. IR: $\tilde{\nu}$ = 3294, 3075, 2931, 2862, 2838, 1621, 1606, 1574, 1541, 1504, 1474, 1434, 1362, 1323, 1305, 1251, 1228, 1175, 1108, 1083, 1028, 991, 908, 844, 827, 790, 766, 727, 686, 668, 644, 611, 592, 522, 494, 444 cm^{-1}. ^{1}H NMR (400 MHz, [D$_6$]DMSO): δ = 8.75 (d, *J* = 4.5 Hz, 1 H), 8.57 (d, *J* = 7.6 Hz, 1 H), 7.94 (d, *J* = 9.2 Hz, 1 H), 7.89 (d, *J* = 2.8 Hz, 1 H), 7.85–7.78 (m, 2 H), 7.64 (d, *J* = 4.6 Hz, 1 H), 7.42 (dd, *J* = 9.0, 2.6 Hz, 1 H), 6.99–6.93 (m, 2 H), 5.92 (ddd, *J* = 17.5, 10.3, 7.5 Hz, 1 H), 5.81 (s, 1 H), 5.04 (ddd, *J* = 17.2, 1.9, 1.3 Hz, 1 H), 4.99 (ddd, *J* = 10.3, 2.0, 1.1 Hz, 1 H), 3.95 (s, 3 H), 3.77 (s, 3 H), 3.59–3.46 (m, 1 H), 3.35–3.22 (m, 1 H), 3.16 (dd, *J* = 13.6, 10.0 Hz, 1 H), 2.79–2.69 (m, 1 H), 2.68–2.55 (m, 1 H), 2.31–2.19 (m, 1 H), 1.62–1.40 (m, 4 H), 0.72 (m, 1 H) ppm. ^{13}C NMR (101 MHz, [D$_6$]DMSO): δ = 165.1, 161.6, 157.3, 147.6, 145.2, 144.0, 142.1, 131.2, 129.2, 128.3, 126.4, 121.2, 120.3, 114.2, 113.4, 102.6, 57.8, 55.6, 55.3, 49.6, 40.7, 39.1, 27.3, 27.2, 26.3 ppm. LRMS (ESI): *m/z* = 458 (100) [M + H$^+$]. HRMS (ESI): *m/z* calcd. for $C_{28}H_{32}N_3O_3^+$: 458.2438; found 458.2439.

***N*-Benzyl-4-chlorobenzamide (3ea):** By following general procedure E using *tert*-butyl 3-(4-chlorophenyl)-3-oxopropanoate (**4h**; 50 mg, 0.23 mmol), *N*-benzyl-4-chlorobenzamide (**3ea**; 0.035 g, 73 %) was obtained as a yellow solid after chromatography (EA/PE, 3:7 → EA). R_f = 0.95 (EA/PE, 6:4) [Cl$_2$]. ^{1}H NMR (400 MHz, CDCl$_3$): δ = 7.78–7.66 (m, 2 H), 7.44–7.27 (m, 7 H), 6.58 (br. s., 1 H), 4.61 (d, *J* = 5.8 Hz, 2 H) ppm. ^{13}C NMR (101 MHz, CDCl$_3$): δ = 166.5, 138.1, 137.9, 132.9, 128.9, 128.6, 128.0, 127.8, 44.3 ppm. The analytical data are in agreement with previously reported data.[54]

4-Chloro-*N*-(1-phenylethyl)benzamide (3ed): By following general procedure D, 4-chloro-*N*-(1-phenylethyl)benzamide (**3ed**; 0.038 g, 98 %) was obtained as a white solid after chromatography (PE/EtOAc, 3:7 → EtOAc). ^{1}H NMR (600 MHz, $CDCl_3$): δ = 7.71–7.66 (m, 2 H), 7.39–7.32 (m, 6 H), 7.30–7.25 (m, 1 H), 6.55 (d, J = 6.8 Hz, 1 H), 5.29 (p, J = 7.0 Hz, 1 H), 1.58 (d, J = 6.9 Hz, 3 H) ppm. ^{13}C NMR (151 MHz, $CDCl_3$): δ = 165.7, 143.1, 137.8, 133.1, 128.9, 128.8, 128.5, 127.6, 126.3, 49.5, 21.8 ppm. The analytical data are in agreement with previously reported data.[55]

***N*-(1-Phenylethyl)furan-2-carboxamide (3fd):** By following general procedure D, *N*-(1-phenylethyl)furan-2-carboxamide (**3fd**; 0.039 g, 99 %) was obtained as a white solid after chromatography (PE/EtOAc, 3:7 → EtOAc). ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.43–7.31 (m, 5 H), 7.30–7.23 (m, 1 H), 7.10 (dd, J = 3.5, 0.7 Hz, 1 H), 6.67–6.54 (m, 1 H), 6.47 (dd, J = 3.5, 1.8 Hz, 1 H), 5.36–5.25 (m, 1 H), 1.59 (d, J = 6.9 Hz, 3 H) ppm. ^{13}C NMR (101 MHz, $CDCl_3$): δ = 157.6, 148.1, 143.8, 143.0, 128.8, 127.6, 126.4, 114.4, 112.2, 48.5, 21.9 ppm. The analytical data are in agreement with previously reported data.[56]

***N*-Benzyl-3-phenylpropanamide (3ga):** By following general procedure E using *tert*-butyl 3-oxo-5-phenylpentanoate (**4b**; 50 mg, 0.20 mmol), *N*-benzyl-3-phenylpropanamide (**3ga**; 0.026 g, 53 %) was obtained as a yellow solid after chromatography (EA/PE, 6:4). R_f = 0.53 (EA/PE, 6:4) [Cl_2]. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.34–7.12 (m, 10 H), 5.71 (br. s., 1 H), 4.39 (d, J = 5.8 Hz, 2 H), 2.99 (t, J = 7.7 Hz, 2 H), 2.51 (t, J = 7.6 Hz, 2 H) ppm. ^{13}C NMR (101 MHz, $CDCl_3$): δ = 172.0, 140.9, 138.3, 128.8, 128.7, 128.5, 127.9, 127.6, 126.4, 43.7, 38.6, 31.8 ppm. The analytical data are in agreement with previously reported data.[52]

3-Phenyl-*N*-(1-phenylethyl)propanamide (3gd): By following general procedure D, 3-phenyl-*N*-(1-phenylethyl)propanamide (**3gd**; 0.035 g, 90 %) was obtained as a white solid after chromatography (PE/EtOAc, 1:1 → 3:7). R_f (PE/EtOAc, 3:7) = 0.85 [Cl_2]. ^{1}H NMR (600 MHz, $CDCl_3$): δ = 7.32–7.22 (m, 5 H), 7.22–7.15 (m, 5 H), 5.66 (d, J = 7.6 Hz, 1 H), 5.10 (p, J = 7.1 Hz, 1 H), 2.96 (t, J = 7.6 Hz, 2 H), 2.48 (td, J = 7.5, 2.2 Hz, 2 H), 1.41 (d, J = 7.0 Hz, 3 H) ppm. ^{13}C NMR (151 MHz, $CDCl_3$): δ = 171.2, 143.2, 140.9, 128.7, 128.6, 128.5, 127.4, 126.3, 126.3, 48.7, 38.7, 31.8, 21.7 ppm. The analytical data are in agreement with previously reported data.[57]

3-Methyl-*N*-(1-phenylethyl)butanamide (3hd): By following general procedure D (2.50 equiv. amine **2d**), 3-methyl-*N*-(1-phenylethyl)butanamide (**3hd**; 0.029 g, 79 %) was obtained as a yellowish solid after chromatography (PE/EtOAc, 4:6 → 1:9). R_f (PE/EtOAc, 3:7) = 0.73 [Cl_2]. IR: $\tilde{\nu}$ = 3279, 3064, 3031, 2957, 2930, 2870, 1637, 1539, 1450, 1368, 1215, 1135, 910, 732, 697, 540 cm^{-1}. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.36–7.29 (m, 4 H), 7.28–7.22 (m, 1 H), 5.77 (d, J = 7.6 Hz, 1 H), 5.21–5.09 (m, 1 H), 2.17–2.06 (m, 1 H), 2.05–2.01 (m, 2 H), 1.48 (d, J = 6.9 Hz, 3 H), 0.94 (d, J = 6.5 Hz, 3 H), 0.92 (d, J = 6.5 Hz, 3 H) ppm. ^{13}C NMR (101 MHz, $CDCl_3$): δ = 171.6, 143.4, 128.7, 127.4, 126.3, 48.6, 46.3, 26.3, 22.6, 22.5, 21.8 ppm. LRMS (ESI): m/z = 206 (100) [M + H$^+$]. HRMS (ESI): m/z calcd. for $C_{13}H_{19}NONa^+$: 228.1359; found 228.1359.

***N*-(1-Phenylethyl)pent-4-enamide (3id):** By following general procedure D, *N*-(1-phenylethyl)pent-4-enamide (**3id**; 0.028 g, 78 %) was obtained as a yellowish liquid after chromatography (PE/EtOAc, 3:7 → EtOAc). R_f (PE/EtOAc, 3:7) = 0.71 [Cl_2]. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.36–7.28 (m, 4 H), 7.28–7.23 (m, 1 H), 5.81 (ddt, J = 16.8, 10.2, 6.5 Hz, 2 H), 5.18–5.09 (m, 1 H), 5.05 (dq, J = 17.1, 1.6 Hz, 1 H), 5.00 (ddd, J = 10.2, 2.2, 0.8 Hz, 1 H), 2.43–2.35 (m, 2 H), 2.29–2.23 (m, 2 H), 1.48 (d, J = 6.9 Hz, 3 H) ppm. ^{13}C NMR (101 MHz, $CDCl_3$): δ = 171.4, 143.3, 137.2, 128.8, 127.5, 126.3, 115.7, 48.8, 36.1, 29.8, 21.9 ppm. The analytical data are in agreement with previously reported data.[58]

***N*-Benzyl-5-(benzyloxy)pentanamide (3ja):** By following general procedure E using *tert*-butyl 7-(benzyloxy)-3-oxoheptanoate (**4j**; 50 mg, 0.16 mmol), *N*-benzyl-5-(benzyloxy)pentanamide (**3ja**; 0.019 g, 40 %) was obtained as a yellow oil after chromatography (EA/PE, 3:7 → EA). R_f = 0.41 (EA/PE, 6:4) [Cl_2]. IR: $\tilde{\nu}$ = 3293, 3086, 3064, 3030, 2929, 2858, 1643, 1541, 1454, 1098, 909, 729, 695 cm^{-1}. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.44–7.18 (m, 10 H), 5.90 (br. s., 1 H), 4.47 (s, 2 H), 4.40 (d, J = 5.8 Hz, 2 H), 3.49 (t, J = 6.2 Hz, 2 H), 2.24 (t, J = 7.5 Hz, 2 H), 1.86–1.70 (m, 2 H), 1.70–1.58 (m, 2 H) ppm. ^{13}C NMR (101 MHz, $CDCl_3$): δ = 172.8, 138.6, 138.5, 128.8, 128.5, 128.0, 127.8, 127.7, 127.6, 73.1, 70.2, 43.7, 36.5, 29.2, 22.9 ppm. LRMS (ESI): m/z = 190.1 (23) [(M – C_7H_7O)$^+$], 298.2 (100) [M + H$^+$], 299.2 (18) [M + 2H$^+$], 300.2 (1) [M + 2H$^+$], 320.2 (2) [M + Na$^+$]. HRMS (ESI): m/z calcd. for $C_{19}H_{23}NO_2Na^+$: 320.1621; found 320.1618.

5-(Benzyloxy)-*N*-(1-phenylethyl)pentanamide (3jd): By following general procedure D, 5-(benzyloxy)-*N*-(1-phenylethyl)pentanamide (**3jd**; 0.029 g, 73 %) was obtained as a colourless liquid after chromatography (PE/EtOAc, 4:6). R_f (PE/EtOAc, 3:7) = 0.54 [Cl_2]. IR: $\tilde{\nu}$ = 3285, 3063, 3030, 2930, 2862, 1639, 1538, 1452, 1101, 734, 696, 540 cm^{-1}. ^{1}H NMR (600 MHz, $CDCl_3$): δ = 7.36–7.30 (m, 6 H), 7.30–7.22 (m, 4 H), 5.81 (d, J = 8.0 Hz, 1 H), 5.12 (p, J = 7.1 Hz, 1 H), 4.48 (s, 2 H), 3.49 (td, J = 6.2, 1.1 Hz, 2 H), 2.21 (t, J = 7.4 Hz, 2 H), 1.77–1.71 (m, 2 H), 1.68–1.61 (m, 2 H), 1.44 (d, J = 7.0 Hz, 3 H) ppm. ^{13}C NMR (151 MHz, $CDCl_3$): δ = 172.0, 143.5, 138.6, 128.7, 128.5, 127.9, 127.7, 127.4, 126.3, 73.2, 70.3, 48.7, 36.6, 29.1, 23.0, 21.9 ppm. LRMS (ESI): m/z = 312 (100) [M + H$^+$], 204 (16) [(M – BnO)$^+$]. HRMS (ESI): m/z calcd. for $C_{20}H_{25}NO_2Na^+$: 334.1778; found 334.1763.

5-Oxo-5-[(1-phenylethyl)amino]pentyl Acetate (3kd): By following general procedure D, 5-oxo-5-[(1-phenylethyl)amino]pentyl acetate (**3kd**; 0.030 g, 78 %) was obtained as a yellowish liquid after chromatography (PE/EtOAc, 4:6). R_f (PE/EtOAc, 3:7) = 0.54 [Cl_2]. IR: $\tilde{\nu}$ = 3294, 3064, 3031, 2960, 2930, 2871, 1721, 1642, 1536, 1451, 1366, 1237, 1041, 912, 731, 699 cm^{-1}. ^{1}H NMR (600 MHz, $CDCl_3$): δ = 7.37–7.27 (m, 4 H), 7.27–7.22 (m, 1 H), 5.81 (d, J = 8.4 Hz, 1 H), 5.12 (p, J = 7.0 Hz, 1 H), 4.05 (t, J = 6.4 Hz, 2 H), 2.22–2.16 (m, 2 H), 2.02 (s, 3 H), 1.74–1.67 (m, 2 H), 1.67–1.60 (m, 2 H), 1.48 (d, J = 6.9 Hz, 3 H) ppm. ^{13}C NMR (151 MHz, $CDCl_3$): δ = 171.6, 171.3, 143.4, 128.8, 127.5, 126.3, 64.1, 48.8, 36.2, 28.3, 22.2, 21.8, 21.1 ppm. LRMS (ESI): m/z = 264 (100) [M + H$^+$], 204 (93) [(M – AcO)$^+$]. HRMS (ESI): m/z calcd. for $C_{15}H_{21}NO_3Na^+$: 286.1414; 286.1408.

4-[(*tert*-Butyldimethylsilyl)oxy]-*N*-(1-phenylethyl)butanamide (3ld): By following general procedure D, 4-[(*tert*-butyldimethylsilyl)oxy]-*N*-(1-phenylethyl)butanamide (**3ld**; 0.030 g, 73 %) was obtained as a yellowish solid after chromatography (PE/EtOAc, 1:1 → EtOAc). R_f (PE/EtOAc, 1:1) = 0.81 [Cl_2]. IR: $\tilde{\nu}$ = 3283, 3064, 3031, 2954, 2928, 2856, 1640, 1542, 1450, 1254, 1100, 834, 775, 697 cm^{-1}. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.36–7.28 (m, 4 H), 7.28–7.22 (m, 1 H), 5.88 (d, J = 8.0 Hz, 1 H), 5.20–5.08 (m, 1 H), 3.70–3.56 (m, 2 H), 2.27 (td, J = 7.1, 0.9 Hz, 2 H), 1.89–1.78 (m, 2 H), 1.48 (d, J = 6.9 Hz, 3 H), 0.88 (s, 9 H), 0.03 (s, 3 H), 0.02 (s, 3 H) ppm. ^{13}C NMR (101 MHz, $CDCl_3$): δ = 172.1, 143.4, 128.8, 127.4, 126.3, 62.2, 48.7, 33.3, 28.7, 26.1, 21.9, 18.4, –5.2 ppm. LRMS (ESI): m/z = 322 (100) [M + H$^+$], 190 (17) [(M – TBSO)$^+$]. HRMS (ESI): m/z calcd. for $C_{18}H_{31}NO_2SiNa^+$: 344.2016; found 344.2010.

4-Azido-*N*-(1-phenylethyl)butanamide (3md): By following general procedure D, 4-azido-*N*-(1-phenylethyl)butanamide (**3md**; 0.029 g, 77 %) was obtained as a colourless liquid after chromatography (PE/EtOAc, 3:7 → EtOAc). R_f (PE/EtOAc, 3:7) = 0.71 [Cl_2]. IR: $\tilde{\nu}$ = 3292, 3064, 3031, 2971, 2929, 2872, 2093, 1639, 1539, 1449, 1249, 909, 732, 698, 540 cm^{-1}. ^{1}H NMR (600 MHz, $CDCl_3$): δ = 7.37–7.28 (m, 4 H), 7.28–7.23 (m, 1 H), 5.83 (d, J = 7.8 Hz, 1 H), 5.12 (p, J = 7.1 Hz, 1 H), 3.38–3.27 (m, 2 H), 2.31–2.20 (m, 2 H), 1.96–1.86

(m, 2 H), 1.49 (d, J = 7.0 Hz, 3 H) ppm. ^{13}C NMR (151 MHz, $CDCl_3$): δ = 170.8, 143.3, 128.8, 127.5, 126.3, 50.9, 49.0, 33.4, 24.9, 21.9 ppm. LRMS (ESI): m/z = 233 (100) [M + H$^+$]. HRMS (ESI): m/z calcd. for $C_{12}H_{16}N_4ONa^+$: 255.1216; found 255.1212.

***N*-Benzyl-3-hydroxy-3-methylbutanamide (3na):** By following general procedure E using *tert*-butyl 5-hydroxy-5-methyl-3-oxohexanoate (**4g**; 50 mg, 0.23 mmol), *N*-benzyl-3-hydroxy-3-methylbutanamide (**3na**; 0.030 g, 62 %) was obtained as a brown liquid after chromatography (EA/PE, 3:7 → EA). R_f = 0.41 (EA/PE, 6:4) [Cl_2]. IR: $\tilde{\nu}$ = 3294, 3088, 3066, 3032, 2971, 2927, 2855, 1634, 1542, 1364, 1236, 909, 687, 608, 481, 458 cm^{-1}. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.41–7.22 (m, 5 H), 6.40 (br. s., 1 H), 4.44 (d, J = 5.6 Hz, 2 H), 4.26 (br. s., 1 H), 2.35 (s, 2 H), 1.26 (s, 6 H) ppm. ^{13}C NMR (101 MHz, $CDCl_3$): δ = [ppm] = 172.4, 138.1, 128.9, 127.9, 127.7, 69.7, 47.8, 43.5, 29.5 ppm. LRMS (ESI): m/z = 190.1 (100) [(M – HO)$^+$], 208.1 (64) [M + H$^+$], 209.1 (7) [M + 2H$^+$], 230.1 (8) [M + Na$^+$]. HRMS (ESI): m/z calcd. for $C_{12}H_{17}NO_2Na^+$: 230.1151; found 230.1150.

3-Hydroxy-3-methyl-*N*-(1-phenylethyl)butanamide (3nd): By following general procedure D, 3-hydroxy-3-methyl-*N*-(1-phenylethyl)butanamide (**3nd**; 0.025 g, 67 %) was obtained as a white solid after chromatography (PE/EtOAc, 8:2 → EtOAc/*i*PrOH/MeOH, 7:2:1). R_f (DCM/MeOH, 95:5) = 0.20 [$KMnO_4$]. IR: $\tilde{\nu}$ = 3440, 3276, 3087, 3033, 2980, 2967, 2930, 2876, 1616, 1558, 1493, 1449, 1420, 1398, 1377, 1362, 1344, 1312, 1274, 1246, 1211, 1182, 1154, 1136, 1104, 1076, 1023, 1003, 988, 960, 928, 911, 877, 849, 803, 777, 756, 736, 701, 637, 619, 590, 555, 529, 469, 456 cm^{-1}. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.36–7.22 (m, 5 H), 6.43 (d, J = 7.9 Hz, 1 H), 5.13 (p, J = 7.1 Hz, 1 H), 4.33 (s, 1 H), 2.31 (s, 2 H), 1.48 (d, J = 6.9 Hz, 3 H), 1.24 (s, 3 H), 1.23 (s, 3 H) ppm. ^{13}C NMR (101 MHz, $CDCl_3$): δ = 171.6, 143.1, 128.8, 127.5, 126.2, 69.7, 48.8, 47.9, 29.5, 29.4, 21.9 ppm. LRMS (EI): m/z = 221 (3) [M$^+$], 203 (5) [(M – H_2O)$^+$], 120 (55) [PhCHNHCH$_3^+$], 105 (100) [PhCHCH$_3^+$], 77 (45) [Ph$^+$]. HRMS (ESI): m/z calcd. for $C_{13}H_{19}NO_2Na^+$: 244.1308; found 244.1307.

3-Hydroxy-3-methyl-*N*-octylbutanamide (3ne): By following general procedure D, 3-hydroxy-3-methyl-*N*-octylbutanamide (**3ne**; 0.029 g, 76 %) was obtained as a colourless liquid after chromatography (PE/EtOAc, 9:1 → 3:7). IR: $\tilde{\nu}$ = 3305, 3098, 2958, 2925, 2855, 1630, 1550, 1465, 1365, 1239, 1152, 908, 603 cm^{-1}. ^{1}H NMR (600 MHz, $CDCl_3$): δ = 5.83 (s, 1 H), 4.36 (s, 1 H), 3.30–3.22 (m, 2 H), 2.30 (s, 2 H), 1.54–1.46 (m, 2 H), 1.32–1.23 (m, 16 H), 0.91–0.83 (m, 3 H) ppm. ^{13}C NMR (151 MHz, $CDCl_3$): δ = 172.5, 69.6, 47.8, 39.5, 31.9, 29.7, 29.5, 29.3, 29.3, 27.0, 22.7, 14.2 ppm. LRMS (ESI): m/z = 230 (100) [M + H$^+$], 212 (83) [(M – OH)$^+$]. HRMS (ESI): m/z calcd. for $C_{13}H_{27}NO_2Na^+$: 252.1934; found 252.1935.

***N*-(1-Phenylethyl)cyclohexanecarboxamide (3od):** By following general procedure D, *N*-(1-phenylethyl)cyclohexanecarboxamide (**3od**; 0.016 g, 43 %) was obtained as a white solid after chromatography (PE/EtOAc, 9:1 → 1:1). R_f (PE/EtOAc, 1:1) = 0.70 [$KMnO_4$]. ^{1}H NMR (600 MHz, $CDCl_3$): δ = 7.36–7.27 (m, 4 H), 7.27–7.22 (m, 1 H), 5.68 (d, J = 7.5 Hz, 1 H), 5.16–5.09 (m, 1 H), 2.07 (tt, J = 11.8, 3.5 Hz, 1 H), 1.90–1.81 (m, 2 H), 1.81–1.74 (m, 2 H), 1.69–1.63 (m, 1 H), 1.47 (d, J = 6.9 Hz, 3 H), 1.46–1.38 (m, 2 H), 1.30–1.20 (m, 3 H) ppm. ^{13}C NMR (151 MHz, $CDCl_3$): δ = 175.2, 143.6, 128.8, 127.4, 126.2, 48.4, 45.7, 29.9, 29.8, 25.9, 25.9, 21.9 ppm. The analytical data are in agreement with previously reported data.[59]

***N*-Octylcyclohexanecarboxamide (3oe):** By following general procedure D, *N*-octylcyclohexanecarboxamide (**3oe**; 0.030 g, 78 %) was obtained as a colourless liquid after chromatography (PE/EtOAc, 9:1 → 3:7). ^{1}H NMR (600 MHz, $CDCl_3$): δ = 5.55 (s, 1 H), 3.24–3.15 (m, 2 H), 2.04 (tt, J = 11.8, 3.5 Hz, 1 H), 1.87–1.79 (m, 2 H), 1.79–1.72 (m, 2 H), 1.68–1.61 (m, 1 H), 1.50–1.34 (m, 4 H), 1.31–1.18 (m, 13 H), 0.85 (t, J = 7.0 Hz, 3 H) ppm. ^{13}C NMR (151 MHz, $CDCl_3$): δ = 176.1, 45.8, 39.5, 31.9, 29.9, 29.8, 29.4, 29.3, 27.0, 25.9, 22.7, 14.2 ppm. The analytical data are in agreement with previously reported data.[60]

***N*-Benzylpivalamide (3pa):** By following general procedure E using *tert*-butyl 4,4-dimethyl-3-oxopentanoate (**4f**; 25 mg, 0.12 mmol), *N*-benzylpivalamide (**3pa**; 0.007 g, 27 %) was obtained as a yellow oil after chromatography (EA/PE, 1:9 → 6:4). R_f = 0.73 (EA/PE, 1:1) [Cl_2]. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.38–7.23 (m, 5 H), 5.89 (br. s., 1 H), 4.44 (d, J = 5.8 Hz, 2 H), 1.23 (s, 9 H) ppm. ^{13}C NMR (101 MHz, $CDCl_3$): δ = 178.4, 138.8, 128.9, 127.8, 127.6, 43.8, 38.9, 27.8 ppm. The analytical data are in agreement with previously reported data.[61]

***N*-(1-Phenylethyl)pivalamide (3pd):** By following general procedure D, *N*-(1-phenylethyl)pivalamide (**3pd**; 0.003 g, 8 %) was obtained as a white solid after chromatography (PE/EtOAc, 9:1 → 1:1). R_f (PE/EtOAc, 1:1) = 0.71 [$KMnO_4$]. ^{1}H NMR (600 MHz, $CDCl_3$): δ = 7.37–7.23 (m, 5 H), 5.79 (s, 1 H), 5.11 (p, J = 7.1 Hz, 1 H), 1.48 (d, J = 6.9 Hz, 3 H), 1.20 (s, 9 H) ppm. ^{13}C NMR (151 MHz, $CDCl_3$): δ = 177.6, 143.7, 128.8, 127.4, 126.2, 48.6, 38.8, 27.7, 21.9 ppm. The analytical data are in agreement with previously reported data.[62]

***N*-Octylpivalamide (3pe):** By following general procedure D, *N*-octylpivalamide (**3pe**; 0.035 g, 93 %) was obtained as a colourless liquid after chromatography (PE/EtOAc, 9:1 → 3:7). ^{1}H NMR (600 MHz, $CDCl_3$): δ = 5.63 (s, 1 H), 3.23–3.17 (m, 2 H), 1.50–1.43 (m, 2 H), 1.31–1.21 (m, 10 H), 1.17 (s, 9 H), 0.86 (t, J = 7.0 Hz, 3 H) ppm. ^{13}C NMR (151 MHz, $CDCl_3$): δ = 178.4, 39.7, 38.7, 31.9, 29.7, 29.4, 29.3, 27.7, 27.0, 22.7, 14.2 ppm. The analytical data are in agreement with previously reported data.[60]

***N*-Benzyladamantane-1-carboxamide (3qa):** By following general procedure E using **4e** (27 mg, 0.10 mmol), *N*-benzyladamantane-1-carboxamide (**3qa**, 0.005 g, 18 %) was obtained as a colourless solid after chromatography (EA/PE, 1:9 → 6:4). R_f = 0.98 (EA/PE, 6:4) [Cl_2]. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.38–7.24 (m, 4 H), 7.25–7.23 (m, 1 H), 5.85 (br. s., 1 H), 4.44 (d, J = 5.6 Hz, 2 H), 2.11–1.97 (m, 4 H), 1.89 (d, J = 2.5 Hz, 6 H), 1.79–1.64 (m, 8 H) ppm. ^{13}C NMR (101 MHz, $CDCl_3$): δ = 178.0, 138.8, 128.9, 127.8, 127.6, 43.5, 40.8, 39.5, 36.7, 28.3 ppm. The analytical data are in agreement with previously reported data.[63]

***N*-(1-Phenylethyl)adamantane-1-carboxamide (3qd):** By following general procedure D, *N*-(1-phenylethyl)adamantane-1-carboxamide (**3qd**; 0.004 g, 10 %) was obtained as a white solid after chromatography (PE/EtOAc, 9:1 → 1:1). R_f (PE/EtOAc, 1:1) = 0.74 [$KMnO_4$]. ^{1}H NMR (600 MHz, $CDCl_3$): δ = 7.36–7.31 (m, 2 H), 7.31–7.27 (m, 2 H), 7.25–7.23 (m, 1 H), 5.76 (d, J = 7.3 Hz, 1 H), 5.12 (p, J = 7.1 Hz, 1 H), 2.06–2.03 (m, 3 H), 1.96–1.94 (m, 2 H), 1.86–1.85 (m, 4 H), 1.75–1.72 (m, 2 H), 1.71–1.69 (m, 2 H), 1.67–1.65 (m, 2 H), 1.47 (d, J = 6.9 Hz, 3 H) ppm. ^{13}C NMR (151 MHz, $CDCl_3$): δ = 176.9, 143.6, 128.6, 127.2, 126.0, 48.2, 42.6, 39.3, 36.5, 36.5, 29.6, 28.2, 21.8 ppm. LRMS (EI): m/z = 283 (14) [M$^+$], 135 (100) [adamantyl$^+$], 104 (43), 79 (50).

***N*-Octyladamantane-1-carboxamide (3qe):** By following general procedure D, *N*-octyladamantane-1-carboxamide (**3qe**; 0.020 g, 50 %) was obtained as a colourless liquid after chromatography (PE/EtOAc, 9:1 → 3:7). R_f (PE/EtOAc, 8:2) = 0.47 [UV]. IR: $\tilde{\nu}$ = 3341, 2904, 2851, 1633, 1530, 1452, 1281 cm^{-1}. ^{1}H NMR (600 MHz, $CDCl_3$): δ = 5.56 (s, 1 H), 3.24–3.19 (m, 2 H), 2.03 (s, 3 H), 1.86–1.82 (m, 6 H), 1.76–1.66 (m, 6 H), 1.50–1.43 (m, 2 H), 1.32–1.23 (m, 10 H), 0.87 (t, J = 7.0 Hz, 3 H) ppm. ^{13}C NMR (101 MHz, $CDCl_3$): δ = 177.9, 40.7, 39.5, 39.5, 36.7, 31.9, 29.8, 29.4, 29.3, 28.3, 27.0, 22.8, 14.2 ppm. LRMS (ESI): m/z = 292 (100) [M + H$^+$]. HRMS (ESI): m/z calcd. for $C_{19}H_{34}NO^+$: 292.2635; found 292.2625.

***N*-Benzylnicotinamide (3ra):** By following general procedure E using *tert*-butyl 3-oxo-3-(pyridin-3-yl)propanoate (**4i**; 50 mg, 0.23 mmol), *N*-benzylnicotinamide (**3ra**; 0.036 g, 74 %) was obtained as a yellow oil after chromatography (EA/PE, 3:7 → EA). R_f = 0.22 (EA/PE, 6:4) [Cl_2]. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.93 (dd, *J* = 2.3, 0.8 Hz, 1 H), 8.65 (dd, *J* = 4.8, 1.8 Hz, 1 H), 8.20–8.02 (m, 1 H), 7.37–7.26 (m, 6 H), 6.87 (br. s., 1 H), 4.62 (d, *J* = 5.8 Hz, 2 H) ppm. ^{13}C NMR (101 MHz, $CDCl_3$): δ = 165.6, 152.3, 148.0, 137.9, 135.3, 130.3, 129.0, 128.1, 127.9, 123.6, 44.3 ppm. The analytical data are in agreement with previously reported data.[46]

3-(Octyloxy)-*N*-[(3*R*,4*R*,5*S*,6*R*)-2,4,5-trihydroxy-6-(hydroxymethyl)tetrahydro-2*H*-pyran-3-yl]benzamide (3tv): D-(+)-Glucosamine hydrochloride (0.038 g, 0.17 mmol, 1.50 equiv.) was suspended in DMF (0.7 mL, 0.10 M) and NaH (60 % dispersion in mineral oil, 0.007 g, 0.16 mmol, 1.40 equiv.) was added. The suspension was stirred for 30 min. *tert*-Butyl 2,2-diazido-3-[3-(octyloxy)phenyl]-3-oxopropanoate (**1t**; 0.050 g, 0.12 mmol, 1.00 equiv.) in DMF (0.5 mL) was added and the suspension was stirred for 2.5 d. The suspension was purified by chromatography over silica (PE/EtOAc, 1:1 → EtOAc/*i*PrOH/MeOH, 50:40:10) to give a 68:32 mixture of diastereomers (NMR) of 3-(octyloxy)-*N*-[(3*R*,4*R*,5*S*,6*R*)-2,4,5-trihydroxy-6-(hydroxymethyl)tetrahydro-2*H*-pyran-3-yl]benzamide (**3tv**; 0.030 g, 63 %) as a white solid. R_f (DCM/MeOH, 9:1) = 0.37 [CAM]. IR: $\tilde{\nu}$ = 3287, 2953, 2921, 2849, 1640, 1608, 1579, 1538, 1485, 1466, 1440, 1387, 1367, 1324, 1296, 1263, 1229, 1153, 1129, 1108, 1088, 1055, 1017, 953, 921, 886, 864, 795, 776, 754, 705, 686, 667, 647, 626, 583, 564, 510, 489, 448, 409 cm^{-1}. ^{1}H NMR (400 MHz, [D_6]DMSO, two diastereomers): δ = 8.14 (d, *J* = 8.8 Hz, 1 H), 8.00 (d, *J* = 7.0 Hz, 1 H), 7.49–7.38 (m, 4 H), 7.38–7.30 (m, 2 H), 7.07 (dd, *J* = 2.6, 1.0 Hz, 1 H), 7.05 (dd, *J* = 2.4, 1.1 Hz, 1 H), 6.52 (d, *J* = 6.3 Hz, 1 H), 6.42 (d, *J* = 4.3 Hz, 1 H), 5.11–4.86 (m, 4 H), 4.71 (s, 1 H), 4.62 (dd, *J* = 8.2, 6.1 Hz, 1 H), 4.52 (t, *J* = 5.8 Hz, 1 H), 4.43 (t, *J* = 5.8 Hz, 1 H), 4.05–3.96 (m, 4 H), 3.81–3.58 (m, 7 H), 3.56–3.39 (m, 3 H), 3.24–3.15 (m, 1 H), 3.12–3.09 (m, 1 H), 1.78–1.66 (m, 4 H), 1.46–1.39 (m, 4 H), 1.35–1.23 (m, 16 H), 0.86 (t, *J* = 6.9 Hz, 6 H) ppm. ^{13}C NMR (101 MHz, [D_6]DMSO, two diastereomers): δ = 166.1, 166.1, 158.5, 158.4, 136.5, 135.9, 129.2, 129.1, 119.6, 119.5, 117.3, 116.9, 113.2, 113.2, 95.4, 90.4, 76.9, 74.1, 72.1, 71.0, 70.9, 70.0, 67.6, 61.3, 61.2, 57.5, 55.4, 31.2, 28.7, 28.7, 28.7, 25.5, 22.1, 13.9 ppm. LRMS (ESI): *m*/*z* = 412 (100) [M + H$^+$]. HRMS (ESI): *m*/*z* calcd. for $C_{21}H_{33}NO_7Na^+$: 434.2149; found 434.2148.

***N*-(Benzo[*d*][1,3]dioxol-5-ylmethyl)acetamide (3aw):** By following general procedure E using *tert*-butyl acetoacetate (**4a**; 50 mg, 0.32 mmol), *N*-(benzo[*d*][1,3]dioxol-5-ylmethyl)acetamide (**3aw**; 0.036 g, 58 %) was obtained as a yellow solid after chromatography (EA). R_f = 0.30 (EA/PE, 8:2) [Cl_2]. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 6.78–6.67 (m, 3 H), 6.06 (br. s., 1 H), 5.91 (s, 2 H), 4.28 (d, *J* = 5.8 Hz, 2 H), 1.97 (s, 3 H) ppm. ^{13}C NMR (101 MHz, $CDCl_3$): δ = 170.0, 148.0, 147.0, 132.3, 121.2, 108.5, 108.4, 101.1, 43.6, 23.3 ppm. The analytical data are in agreement with previously reported data.[61]

***N*-Phenethylacetamide (3ax):** By following general procedure E using *tert*-butyl acetoacetate (**4a**; 50 mg, 0.32 mmol), *N*-phenethylacetamide (**3ax**; 0.031 g, 61 %) was obtained as a yellow solid after chromatography (EA). R_f = 0.24 (EA/PE, 8:2) [Cl_2]. ^{1}H NMR (600 MHz, $CDCl_3$): δ = 7.34–7.15 (m, 5 H), 5.63 (br. s., 1 H), 3.60–3.41 (m, 2 H), 2.81 (t, *J* = 7.0 Hz, 2 H), 1.93 (s, 3 H) ppm. ^{13}C NMR (151 MHz, $CDCl_3$): δ = 170.1, 139.0, 128.8, 128.7, 126.6, 40.8, 35.8, 23.4 ppm. The analytical data are in agreement with previously reported data.[64]

Methyl 4-(Acetamidomethyl)benzoate (3ay): By following general procedure E using *tert*-butyl acetoacetate (**4a**; 50 mg, 0.32 mmol), methyl 4-(acetamidomethyl)benzoate (**3ay**; 0.049 g, 75 %) was obtained as a colourless solid after chromatography (EA/PE, 9:1 → EA). R_f = 0.32 (EA/PE, 9:1) [Cl_2]. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.00–7.86 (m, 2 H), 7.31–7.26 (m, 2 H), 6.33 (br. s., 1 H), 4.42 (d, *J* = 5.8 Hz, 2 H), 3.87 (s, 3 H), 2.00 (s, 3 H) ppm. ^{13}C NMR (101 MHz, $CDCl_3$): δ = 170.3, 166.9, 143.7, 130.0, 129.3, 127.6, 52.2, 43.3, 23.2 ppm. The analytical data are in agreement with previously reported data.[65]

***N*-Benzylcinnamamide (10):** 6-Phenyldihydro-2*H*-pyran-2,4(3*H*)-dione (**9**; 20 mg, 0.11 mmol, 1.00 equiv.), tetrabutylammonium azide (120 mg, 0.42 mmol, 4.00 equiv.), 4-PPY (62 mg, 0.42 mmol, 4.00 equiv.) and benzylamine (**2a**; 23 mg, 0.11 mmol, 2.00 equiv.) were dissolved in THF (1.7 mL; 0.15 M), and iodine (59 mg, 0.23 mmol, 2.20 equiv.) was added. The reaction mixture was stirred overnight at room temperature. Flash chromatography (EA/PE, 2:8 → 3:7) furnished *N*-benzylcinnamamide (**10**; 0.022 mg, 86 %) as a yellow oil. R_f = 0.73 (EA/PE, 1:1) [Cl_2]. ^{1}H NMR (600 MHz, $CDCl_3$): δ = 7.67 (d, *J* = 15.8 Hz, 1 H), 7.53–7.46 (m, 2 H), 7.41–7.27 (m, 8 H), 6.42 (d, *J* = 15.8 Hz, 1 H), 6.00 (br. s., 1 H), 4.57 (d, *J* = 5.6 Hz, 2 H) ppm. ^{13}C NMR (151 MHz, $CDCl_3$): δ = 165.9, 141.6, 138.3, 134.9, 129.9, 129.0, 128.9, 128.1, 128.0, 127.7, 120.6, 44.0 ppm. The analytical data are in agreement with previously reported data.[66]

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