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Reaction Between 5-Isopropylidene-2,2-dimethyl-1,3-dioxane-4,6-dione and *tert*-Butyl Isocyanide in the Presence of Primary or Secondary Amines

Issa Yavari[1,*], **Azizollah Habibi**[1], **Mohammad R. Hosseini-Tabatabaei**[2], and **Hamid R. Bijanzadeh**[1]

[1] Department of Chemistry, University of Tarbiat Modarres, Tehran, Iran
[2] Department of Chemistry, Islamic Azad University, Zahedan, Iran

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Summary. 5-Isopropylidene-2,2-dimethyl-1,3-dioxane-4,6-dione (the condensation product of *Meldrum*'s acid and acetone) reacts smoothly with *tert*-butyl isocyanide in the presence of primary or secondary amines to produce *N-tert*-butyl-2,2-dimethylbutyramide derivatives and/or 1-*tert*-butyl-4,4-dimethyl-2,5-dioxopyrrolidine-3-carboxamides in good yields.

Keywords. *Meldrum*'s acid; Alkyl isocyanides; Triamides; Amidosuccinimides; *Ugi* reaction.

Introduction

The development of simple synthetic routes for widely used organic compounds from readily available reagents is one of the major tasks in organic synthesis [1]. Due to its high acidity (pK_a 7.5) [2] and tendency to regenerate acetone, *Meldrum*'s acid (2,2-dimethyl-1,3-dioxane-4,6-dione) [3], appears to be an attractive reagent in organic synthesis. However, synthesis applications of this compound have received little attention except as an alternative for cyclic malonic esters [4]. Useful applications of alkylidene derivatives of *Meldrum*'s acid as dienophiles in *Diels-Alder* reactions [5] and *Michael* acceptors have been reported, showing advantages of these systems over their acyclic analogues [6, 7]. Alkylidene *Meldrum*'s acids are readily accessible from *Meldrum*'s acid and carbonyl compounds (ketones and aldehydes) in a relatively large scale [8].

We report herein that *tert*-butyl isocyanide undergoes a smooth addition reaction with 5-isopropylidene-2,2-dimethyl-1,3-dioxane-4,6-dione in the presence of

* Corresponding author. E-mail: isayavar@yahoo.com

primary or secondary amines, yielding *N-tert*-butyl-2,2-dimethylbutyramide derivatives (**4**) and/or 1-*tert*-butyl-4,4-dimethyl-2,5-dioxopyrrolidine-3-carboxamides (**8**).

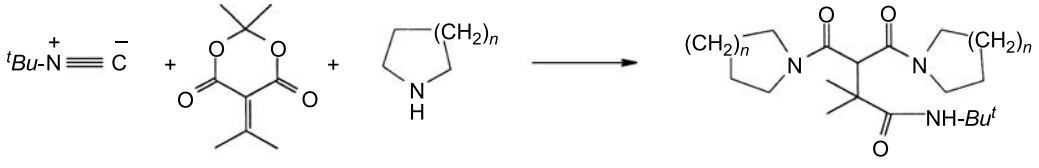
Results and Discussion

Pyrrolidine and Piperidine

The reaction of *tert*-butyl isocyanide with 5-isopropylidene-2,2-dimethyl-1,3-dioxane-4,6-dione in the presence of pyrrolidine or piperidine was carried out in dichloromethane at room temperature. The colorless crystals separated from these reaction mixtures were identified as *N-tert*-butyl-2,2-dimethyl-4-oxo-3-(pyrrolidin-1-ylcarbonyl)-4-(pyrrolidin-1-yl)butyramide (**4a**) and *N-tert*-butyl-2,2-dimethyl-4-oxo-3-(piperidin-1-ylcarbonyl)-4-(piperidin-1-yl)butyramide (**4b**). Any product other than **4** could not be detected by NMR spectroscopy. The structures of compounds **4a** and **4b** were deduced from their elemental analyses and their IR, ^{1}H NMR, and ^{13}C NMR spectra.

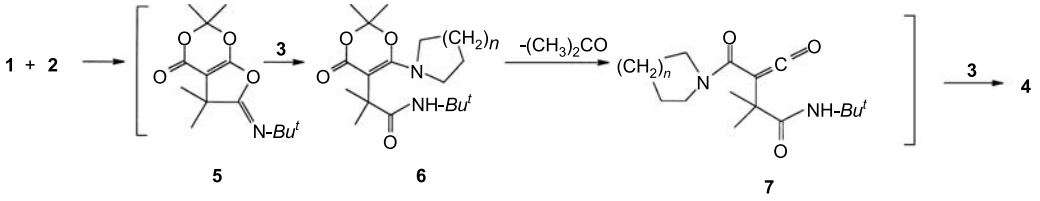
The mass spectra of these compounds displayed molecular ion peaks at appropriate m/z values. Any initial fragmentation involved the loss of amide moieties. The ^{1}H NMR spectrum of **4a** exhibited three single sharp lines readily recognized as arising from *tert*-butyl (1.32 ppm), *gem*-dimethyl (1.38 ppm), and methine (4.16 ppm) protons along with a fairly broad singlet at 6.1 ppm for the NH group. The tetramethylene moiety of the pyrrolidine residue exhibits two multiplets at 1.82–1.95 ppm and 3.38–3.57 ppm. The ^{13}C NMR spectrum of **4a** showed eleven distinct resonances in agreement with the triamide structure. Partial assignment of these resonances is given in the Experimental section. The ^{1}H and ^{13}C NMR spectra of **4b** are similar to those of **4a** except for the piperidine residue, which exhibits characteristic signals with appropriate chemical shifts (see Experimental section). The ^{15}N NMR spectrum of **4b** exhibits two signals at 122.38 ppm (N–C=O) and 129.25 ppm (NH–C=O) in the amide region [16] of the spectrum, in agreement with the **4b** structure (Scheme 1).

The structure assignment of compounds **4a** and **4b** made on the basis of the ^{1}H, ^{13}C, and ^{15}N NMR spectra was supported by their IR spectra, the carbonyl region of which displayed three distinct absorption bands for each compound (see Experimental). The NH absorption bands of **4a** and **4b** appear at about 3270 cm^{-1}.

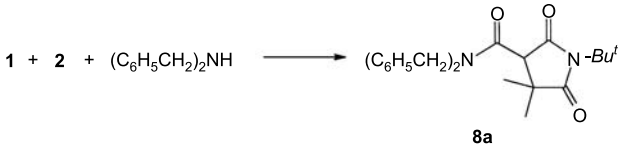


1 **2** **3a** $n = 1$ **3b** $n = 2$ **4a** $n = 1$ **4b** $n = 2$

Scheme 1



Scheme 2



Scheme 3

On the basis of the well established chemistry of isocyanides [9–13] it is reasonable to assume that the triamides **4** result from an initial [4 + 1] cycloaddition reaction of the electron-defficient heterodiene moiety of **2** with *tert*-butyl isocyanide, producing an iminolactone intermediate **5**. Conjugate addition by the amine on the enone moiety of **5**, followed by cleavage of the five-membered ring gives **6** and hence the ketene **7** by well precedented [14] electrocyclic ring opening of *O*-alkylated *Meldrum*'s acids. The ketene **7** can then be trapped by the amine to give the triamides **4** (see Scheme 2).

Dibenzylamine

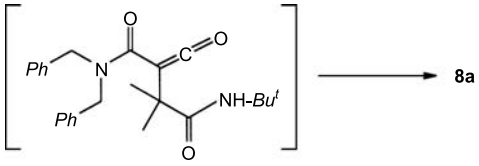
Under the reaction conditions given for pyrrolidine and piperidine, only one product was isolated from the reaction mixture of dibenzylamine, *tert*-butyl isocyanide, and **2** (see Scheme 3). Structure **8a** was assigned to the isolated product on the basis of its elemental analysis and IR, ^{1}H, ^{13}C, and ^{15}N NMR spectra.

The mass spectrum of **8a** exhibited a molecular ion peak at $m/z = 407$ (MH^+). The ^{1}H NMR spectrum of **8a** showed four signals for *tert*-butyl, methyl, and methine protons. The methylene groups of the benzyl moieties exhibited two doublets and an AB quartet. The ^{13}C NMR of this product showed nineteen signals in agreement with the suggested structure for **8a**. The ^{15}N NMR spectrum of **8a** exhibits two signals at 128.92 ppm and 185.29 ppm for the amide and imide nitrogen atoms of **8a**, respectively. The observed ^{15}N shift for the imide moiety of **8a** is in excellent agreement with the previously reported values for *N*-alkylsuccinimides [15].

Compound **8a** may be considered as a product of intramolecular reaction between the amide and ketene moieties of intermediate **9** (Scheme 4), which is presumably formed by similar steps given for **7** in Scheme 2.

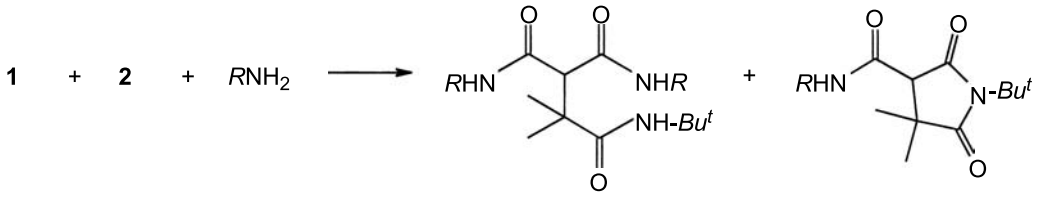
Aniline, p-Toluidine, 1-Naphthylamine, Allylamine, and Benzylamine

The reaction of *tert*-butyl isocyanide with isopropylidene *Meldrum*'s acid in the presence of aniline (**10a**), *p*-toluidine (**10b**), 1-naphthylamine (**10c**), allylamine



9

Scheme 4



	R		R		R
10a:	Phenyl	**4c:**	Phenyl	**8b:**	Phenyl
10b:	*p*-Tolyl	**4d:**	*p*-Tolyl	**8c:**	*p*-Tolyl
10c:	1-Naphthyl	**4e:**	1-Naphthyl	**8d:**	1-Naphthyl
10d:	Allyl	**4f :**	Allyl	**8e:**	Allyl
10e:	Benzyl	**4g:**	Benzyl	**8f :**	Benzyl

Scheme 5

(**10d**), or benzylamine (**10e**) was carried out in dichloromethane at ambient temperature. Structures **4c**–**g** and **8b**–**f** (Scheme 5) were assigned to the isolated products on the basis of their elemental analyses and IR, ^{1}H, ^{13}C, and ^{15}N NMR spectra.

The mass spectra of these compounds displayed molecular ion peaks at appropriate m/z values. The ^{1}H NMR spectra of the amidosuccinimide products **8b**–**8f** showed two signals for the diastereotopic methyl groups, along with *tert*-butyl, methine, and amide protons. The ^{1}H NMR spectra of triamides **4c**–**4g** showed five signals for *tert*-butyl, methyl, and methine protons, together with two signals for the amide protons. The ^{13}C NMR spectra of the amidosuccinimide derivatives **8b**–**8f** and the triamides **4c**–**4g** are consistent with the proposed structures. Partial assignment of these resonances are given in the Experimental section. The ^{15}N NMR spectrum of **8f** exhibited two signals at $\delta = 122.5$ and 185.0 ppm, downfield from external liquid ammonia, which is assigned to the amido and imido nitrogen atom, respectively [15, 16].

Conclusions

We have found a simple and efficient three-component reaction for the synthesis of highly functionalized triamides and amidosuccinimides of potential synthetic interest. The one-pot nature of the present procedure makes it an acceptable method for preparation of triamides and amidosuccinimides with variable functionalities.

Experimental

Melting points were measured on an Electrothermal 9100 apparatus. Elemental analyses for C, H, and N were performed using a Heraeus CHN–O-Rapid analyzer; the results agreed favourably with the calculated values. IR spectra were measured on a Shimadzu IR-460 spectrometer. Mass spectra were recorded on a FINNIGAN-MAT 8430 mass spectrometer operating at an ionization potential of 70 eV. ^{1}H, ^{13}C, and ^{15}N NMR spectra were recorded at 500.1, 125.7, and 50.7 MHz on a Bruker DRX-500 Avance instrument with $CDCl_3$ as solvent and *TMS* as internal standard (for ^{15}N NMR, liquid NH_3 was used as external standard). *Meldrum*'s acid and other reagents were obtained from Fluka (Buchs, Switzerland) and used without further purification. Chromatography columns were prepared from Merck silica gel.

General Procedure for the Syntheses of **4** *and* **8**

To a magnetically stirred solution of 0.368 g of **2** (2 mmol) and 4 mmol of amine in 10 cm^3 of CH_2Cl_2, a mixture of 0.170 g of **1** (2 mmol) in 2 cm^3 of CH_2Cl_2 was added dropwise at room temperature. The reaction mixture was then stirred for 24 h, the solvent was removed under reduced pressure, and the oily residue was dissolved in ethyl acetate. The product was precipitated by addition of *n*-hexane, collected by filtration, and recrystalized from a 2:3 mixture of ethyl acetate:hexane. The solid residue obtained from the reaction of amines **10a**–**10e** with **1** and **2** was separated by silica column chromatography (Merck 230–400 mesh) using *n*-hexane-ethyl acetate as eluent. The first compound was eluted using a 3:1 mixture and identified as **4c**–**4g**. Elution with a 2:1 mixture gave **8b**–**8f**.

N-tert-Butyl-2,2-dimethyl-4-oxo-3-(pyrrolidin-1-ylcarbonyl)-4-(pyrrolidin-1-yl)butyramide (**4a**, $C_{19}H_{33}N_3O_3$)

Colorless crystals, mp 169–173°C; yield: 0.48 g (68%); IR (KBr): $\bar{\nu} = 3270$ (NH), 1657, 1646, and 1606 (C=O) cm^{-1}; MS: m/z (%) = 352 (MH^+, 35), 281 (10), 279 (54), 251 (18), 210 (15), 180 (15), 154 (100), 98 (34), 78 (38), 70 (74), 55 (69), 42 (59); ^{1}H NMR: $\delta = 1.32$ (9H, s, CMe_3), 1.38 (6H, s, CMe_2), 1.82–1.95 (8H, m, $2NCH_2CH_2CH_2$), 3.38–3.57 (8H, m, $4NCH_2$), 4.16 (1H, s, CH), 6.09 (1H, s, NH) ppm; ^{13}C NMR: $\delta = 24.05$ and 24.21 ($4NCH_2CH_2$), 26.36 (CMe_2), 28.49 (CMe_3), 45.87 (CMe_2), 46.27 and 46.77 ($4NCH_2$), 50.67 (CMe_3), 55.20 (CH), 166.74 (2NC=O), 176.56 (NHC=O) ppm.

N-tert-Butyl-2,2-dimethyl-4-oxo-3-(piperidin-1-ylcarbonyl)-4-(piperidin-1-yl)butyramide (**4b**, $C_{21}H_{37}N_3O_3$)

Colorless crystals, mp 122–126°C; yield: 0.55 g (73%); IR (KBr): $\bar{\nu} = 3272$ (NH), 1645 and 1638 (C=O) cm^{-1}; MS: m/z (%) = 380 (MH^+, 13), 307 (47), 295 (32), 279 (10), 239 (15), 222 (12), 194 (32), 168 (100), 152 (15), 112 (24), 84 (82), 57 (46), 41 (62); ^{1}H NMR: $\delta = 1.32$ (9H, s, CMe_3), 1.34 (6H, s, CMe_2), 1.45–1.62 (12H, m, $2NCH_2CH_2CH_2CH_2$), 3.40 (8H, m, $4NCH_2$), 4.49 (1H, s, CH), 5.89 (1H, s, NH) ppm; ^{13}C NMR: $\delta = 24.51$ (CMe_2), 24.59 ($2NCH_2CH_2CH_2$), 25.80 and 25.82 ($4NCH_2CH_2$), 28.50 (CMe_3), 43.4 and 47.4 ($4NCH_2$), 45.64 (CMe_2), 50.77 (CMe_3), 51.14 (CH), 167.05 (2NC=O), 176.98 (NHC=O) ppm; ^{15}N NMR: $\delta = 122.38$ (NC=O), 129.25 (NHC=O) ppm.

1-tert-Butyl-4,4-dimethyl-2,5-dioxopyrrolidine-3-carboxylic acid dibenzylamide (**8a**, $C_{25}H_{30}N_2O_3$)

Colorless crystals, mp 141–144°C; yield: 0.60 g (74%); IR (KBr): $\bar{\nu} = 1694$ and 1623 (C=O) cm^{-1}; MS: m/z (%) = 407 (MH^+, 19), 315 (72), 259 (31), 196 (82), 106 (100), 91 (88), 83 (32), 78 (34), 57

(26), 41 (26); ^{1}H NMR: $\delta = 1.16$ and 1.23 (6H, 2s, CMe_2), 1.62 (9H, s, CMe_3), 3.63 (1H, s, CH), 4.1 (1H, d, $J = 14.5$ Hz, NC*H*H), 4.47 (2H, AB quartet, $J = 17.2$ Hz, NCH_2), 5.14 (1H, d, $J = 14.5$ Hz, NCH*H*), 7.19–7.41 (10H, m, $2C_6H_5$) ppm; ^{13}CNMR: $\delta = 19.68$ and 27.69 (CMe_2), 28.21 (CMe_3), 43.12 (CMe_2), 48.71 and 50.28 ($2NCH_2$), 56.18 (CH), 58.60 (CMe_3), 126.53–136.65 ($2C_6H_5$), 168.54, 174.62, and 183.07 (3C=O) ppm; ^{15}N NMR: $\delta = 128.92$ (NC=O), 185.92 (N imide) ppm.

N^1-tert-Butyl-2,2-dimethyl-N^4-phenyl-3-(phenylcarbamoyl)succinamide (**4c**, $C_{23}H_{29}N_3O_3$)

Colorless crystals, mp 178–180°C; yield: 0.09 g (15%); IR (KBr): $\bar{\nu} = 3245$ (NH), 1680 and 1642 (C=O) cm^{-1}; MS: m/z (%) = 395 (M^+, 25), 247 (20), 176 (43), 154 (27), 149 (23), 127 (23), 97 (30), 94 (42), 93 (90), 82 (100), 69 (40), 54 (86), 52 (50); ^{1}H NMR: $\delta = 1.27$ (9H, s, CMe_3), 1.49 (6H, s, CMe_2), 4.09 (1H, s, CH), 5.87 (1H, s, *t*-BuNH), 7.11–7.61 (10H, m, $2C_6H_5$), 9.80 (2H, s, 2PhNH) ppm; ^{13}C NMR: $\delta = 24.00$ (CMe_2), 28.53 (CMe_3), 46.15 (CMe_2), 51.60 (CH), 60.94 (CMe_3), 120.44, 124.53, 128.83, and 137.74 (C_6H_5), 167.10 (NHC=O), 175.66 (*t*-BuNHC=O) ppm.

1-tert-Butyl-4,4-dimethyl-2,5-dioxopyrrolidine-3-carboxylic acid phenylamide (**8b**, $C_{17}H_{22}N_2O_3$)

Colorless crystals, mp 167–170°C; yield: 0.47 g (85%); IR (KBr): $\bar{\nu} = 3235$ (NH), 1704, 1685, and 1661 (C=O) cm^{-1}; MS: m/z (%) = 302 (M^+, 74), 247 (18), 176 (32), 127 (37), 93 (100), 78 (44), 57 (35), 41 (51); ^{1}H NMR: $\delta = 1.33$ and 1.53 (6H, 2s, CMe_2), 1.64 (9H, s, CMe_3), 3.40 (1H, s, CH), 7.13–7.59 (5H, m, C_6H_5), 9.5 (1H, s, NH) ppm; ^{13}C NMR: $\delta = 22.04$ and 24.22 (CMe_2), 28.41 (CMe_3), 44.26 (CMe_2) 56.21 (CH), 59.09 (CMe_3), 121.36, 124.80, 129.0, and 137.26 (C_6H_5), 163.44 (NHC=O), 176.66 and 182.23 (2NC=O) ppm.

N^1-tert-Butyl-2,2-dimethyl-N^4-p-tolyl-3-(p-tolylcarbamoyl)succinamide (**4d**, $C_{25}H_{33}N_3O_3$)

Colorless crystals, mp 178–180°C; yield: 0.14 g (28%); IR (KBr): $\bar{\nu} = 3240$ (NH), 1682, and 1636 (C=O) cm^{-1}; MS: m/z (%) = 423 (M^+, 53), 316 (100), 260 (10), 190 (18), 107 (13), 83 (40), 57 (32), 41 (44); ^{1}H NMR: $\delta = 1.28$ (9H, s, CMe_3), 1.45 (6H, s, CMe_2), 2.31 (6H, s, 2ArMe), 3.98 (1H, s, CH), 5.18 (1H, s, *t*-BuNH), 7.10–7.45 (8H, 2d, $J = 8.6$ Hz, $2C_6H_4$), 9.54 (2H, s, 2ArNH) ppm; ^{13}C NMR: $\delta = 20.90$ (CMe_3), 24.16 (CMe_2), 28.54 ($ArCH_3$), 46.14 (CMe_2), 51.55 (CMe_3), 60.79 (CH), 120.35, 129.35, 134.13, and 135.11 ($2C_6H_4$), 167.01 (2NHC=O), 175.75 (*t*-BuNHC=O) ppm.

1-tert-Butyl-4,4-dimethyl-2,5-dioxopyrrolidine-3-carboxylic acid p-tolylamide (**8c**, $C_{18}H_{24}N_2O_3$)

Colorless crystals, mp 170–173°C; yield: 0.37 g (72%); IR (KBr): $\bar{\nu} = 3210$ (NH), 1696, 1650, and 1640 (C=O) cm^{-1}; MS: m/z (%) = 316 (M^+, 84), 260 (26), 190 (32), 127 (54), 107 (100), 83 (47), 57 (43), 41 (60); ^{1}H NMR: $\delta = 1.30$ and 1.46 (6H, 2s, CMe_2), 1.60 (9H, s, CMe_3), 2.29 (3H, s, $ArCH_3$), 3.34 (1H, s, CH), 7.10 and 7.41 (4H, 2d, $J = 8.6$ Hz, C_6H_4), 9.34 (1H, s, ArNHCO) ppm; ^{13}C NMR: $\delta = 20.85$ and 21.72 (CMe_2), 24.45 (Ar*Me*), 28.34 (CMe_3), 44.11 (CMe_2), 56.57 (CH), 58.94 (CMe_3), 120.37, 129.45, 134.41, and 134.63 (C_6H_4), 163.47 (NHC=O), 176.39 and 182.26 (NC=O) ppm.

N^1-tert-Butyl-2,2-dimethyl-N^4-(1-naphthyl)-3-((1-naphthyl)carbamoyl)succinamide (**4e**, $C_{31}H_{32}N_3O_3$)

Colorless crystals, mp 156–158°C; yield: 0.15 g (31%); IR (KBr): $\bar{\nu} = 3255$ (NH), 1668 and 1629 (C=O) cm^{-1}; MS: m/z (%) = 495 (M^+, 15), 348 (15), 252 (17), 221 (20), 169 (29), 143 (100), 127 (25), 115 (20), 91 (24), 82 (37), 54 (26); ^{1}H NMR: $\delta = 1.29$ (9H, s, CMe_3), 1.58 (6H, s, CMe_2), 4.41

(1H, s, CH), 5.82 (1H, s, *t*-BuN*H*), 7.47–8.07 (14H, m, $2C_{10}H_7$), 10.14 (2H, s, 2ArN*H*) ppm; ^{13}C NMR: $\delta = 24.90$ (C*Me*$_2$), 28.58 (C*Me*$_3$), 46.06 (*C*Me$_2$), 51.70 (CH), 61.18 (*C*Me$_3$), 119.84, 121.36, 125.60, 125.70, 126.02, 126.47, 126.94, 128.54, 132.48, and 134.11 ($2C_{10}H_7$), 168.21 (ArNHC=O), 176.14 (*t*-BuNHC=O) ppm.

1-tert-Butyl-4,4-dimethyl-2,5-dioxopyrrolidine-3-carboxylic acid naphth-1-ylamide
(**8d**, $C_{21}H_{24}N_2O_3$)

Colorless crystals, mp 238–242°C (*n*-hexane:ethyl acetate = 4:1, first eluted); yield: 0.43 g (69%); IR (KBr): $\bar{\nu} = 3225$ (NH), 1694, 1661, and 1634 (C=O) cm^{-1}; MS: m/z (%) = 352 (M^+, 100), 296 (28), 169 (19), 143 (82), 127 (44), 115 (26), 83 (31), 57 (34), 41 (43); ^{1}H NMR: $\delta = 1.40$ and 1.59 (6H, 2s, CMe$_2$), 1.66 (9H, s, CMe$_3$), 3.52 (1H, s, CH), 7.48–8.16 (7H, m, $C_{10}H_7$), 10.22 (1H, s, NH) ppm; ^{13}C NMR (δ $CDCl_3$:*DMSO*-d_6 = 1:1): $\delta = 19.70$ and 26.90 (C*Me*$_2$), 28.14 (C*Me*$_3$), 43.19 (*C*Me$_2$), 58.02 (CH), 59.27 (*C*Me$_3$), 121.58, 122.06, 125.38, 125.80, 125.96, 125.98, 127.59, 128.26, 132.42, and 133.89 ($C_{10}H_7$), 166.30 (NHC=O), 174.87 and 182.94 (NC=O) ppm.

N^1-tert-Butyl-2,2-dimethyl-N^4-allyl-3-(allylcarbamoyl)succinamide (**4f**, $C_{17}H_{28}N_3O_3$)

Colorless crystals, mp 140–142°C; yield: 0.096 g (30%); IR (KBr): $\bar{\nu} = 3285$ (NH), 1690 and 1652 (C=O) cm^{-1}; MS: m/z (%) = 323 (M^+, 27), 251 (20), 140 (82), 126 (24), 98 (28), 82 (100), 55 (30), 53 (40); ^{1}H NMR: $\delta = 1.28$ (9H, s, CMe$_3$), 1.55 (6H, s, CMe$_2$), 3.69 (1H, s, CH), 3.84 (4H, 2ABX systems, $J = 15.7$, 5.5, 5.3 Hz, $2NCH_2$), 5.10 (2H, d, $J = 10.2$ Hz, 2C*H*H), 5.15 (2H, d, $J = 17.1$ Hz, 2=CH*H*), 5.78 (2H, ddt, $J = 17.1$, 10.2, 5.5 Hz, 2=CH), 5.93 (1H, s, *t*-BuNH), 7.61 (2H, s, $2CH_2NH$) ppm; ^{13}C NMR: $\delta = 24.14$ (C*Me*$_2$), 28.50 (C*Me*$_3$), 41.95 (CH_2NH), C 45.22 (*C*Me$_2$), 51.20 (CH), 59.47 (N*C*Me$_3$), 116.40 (=CH_2), 133.78 (=CH), 168.97 (NHC=O), 175.46 (*t*-BuNHC=O) ppm.

1-tert-Butyl-4,4-dimethyl-2,5-dioxopyrrolidine-3-carboxylic acid allylamide
(**8e**, $C_{14}H_{22}N_2O_3$)

Colorless crystals, mp 129–131°C; yield: 0.33 g (70%); IR (KBr): $\bar{\nu} = 3265$ (NH), 1705, 1664 and 1640 (C=O) cm^{-1}; MS: m/z (%) = 266 (M^+, 19), 211 (26), 194 (29), 140 (16), 83 (60), 56 (100); ^{1}H NMR: $\delta = 1.20$ and 1.45 (6H, 2s, CMe$_2$), 1.58 (9H, s, CMe$_3$), 3.22 (1H, s, CH), 3.93 (2H, ABX system, $J = 15.7$, 5.5, 5.3 Hz, NCH_2), 5.15 (1H, d, $J = 10.2$ Hz, =C*H*H), 5.21 (1H, d, $J = 17.1$ Hz, =CH*H*), 5.85 (1H, ddt, J=17.1, 10.7, 5.5 Hz, =C*H*), 7.49 (1H, s, NH) ppm; ^{13}C NMR: $\delta = 21.78$ and 24.41 (C*Me*$_2$), 28.35 (C*Me*$_3$), 41.64 (NCH_2), 43.86 (*C*Me$_2$), 56.24 (CH), 58.80 (*C*Me$_3$), 116.52 (=CH_2), 133.69 (=CH–), 165.23 (NHC=O), 176.26 and 182.37 (2 NC=O) ppm.

N^4-Benzyl-3-(benzylcarbamoyl)-N^1-tert-butyl-2,2-dimethylsuccinamide (**4g**, $C_{25}H_{33}N_3O_3$)

Colorless crystals, mp 165–168°C; yield: 0.13 g (32%); IR (KBr): $\bar{\nu} = 3280$ (NH), 1670, 1655, and 1645 (C=O) cm^{-1}; MS: m/z (%) = 424 (MH^+, 35), 351 (12), 324 (28), 190 (57), 106 (68), 91 (100), 57 (32), 41 (28); ^{1}H NMR: $\delta = 1.20$ (9H, s, CMe$_3$), 1.25 (6H, s, CMe$_2$), 3.8 (1H, s, CH), 4.37 (4H, 2ABX systems, $J = 14.8$, 5.8, 5.8 Hz), 5.92 (1H, s, *t*-BuNH), 7.2 (10H, m, $2C_6H_5$), 8.1 (2H, t, $J = 5.8$ Hz, 2NH) ppm; ^{13}C NMR: $\delta = 23.9$ (C*Me*$_2$), 28.3 (C*Me*$_3$), 43.4 (*C*Me$_2$), 51.0 (*C*Me$_3$), 59.26 (CH), 127.1, 127.6, 128.4, and 128.6 ($2C_6H_5$), 169 (2NHC=O), 175.4 (NHC=O) ppm.

1-tert-Butyl-4,4-dimethyl-2,5-dioxopyrrolidine-3-carboxylic acid benzylamide
(**8f**, $C_{18}H_{24}N_2O_3$)

Colorless crystals, mp 144–146°C; yield: 0.21 g (68%); IR (KBr): $\bar{\nu} = 3260$ (NH), 1701, 1662, and 1632 (C=O) cm^{-1}; MS: m/z (%) = 316 (M^+, 12), 106 (100), 91 (41), 78 (23), 57 (19), 41 (31);

^{1}H NMR: $\delta = 1.25$ and 1.45 (6H, 2s, CMe_2), 1.57 (9H, s, CMe_3), 3.2 (1H, s, CH), 4.48 (2H, ABX system, $J = 14.8, 5.5, 5.5$ Hz, NCH_2), 7.2–7.4 (5H, m, C_6H_5), 7.7 (1H, s, NH) ppm; ^{13}C NMR: $\delta = 21.5$ and 24.7 (CMe_2), 28.3 (CMe_3), 43.2 (CH_2NH), 43.7 (CMe_2), 56.7 (CH), 58.7 (CMe_3), 127.5, 127.7, 128.7, and 137.8 (C_6H_5), 165.5, 175.9, and 182.4 (3C=O) ppm; ^{15}N NMR: $\delta = 122.5$ (NHC=O), 185.0 (N imide) ppm.

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