

## SUPPORTING INFORMATION

### Zinc catalysed ester hydrolysis. Application to the synthesis of tartronic acid derivatives

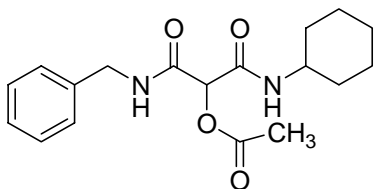
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**General Techniques.** Melting points are uncorrected. IR spectra were recorded as KBr pellets using a Bruker Vector 22 FT-IR spectrometer. Proton and carbon-13 nuclear magnetic resonance (<sup>1</sup>H NMR or <sup>13</sup>C NMR) spectra were obtained on a Bruker 400 MHz spectrometer. Mass spectra (MS) were recorded with a HP 5988-A spectrometer using Electronic Impact (EI, 70 eV) and with a Micromass Autospec spectrometer, using FAB with Xe<sup>0</sup> and 2-methoxyethyl disulfide as matrix. High Resolution Mass Spectra (HRMS) were recorded with a Micromass Autospec spectrometer, either with EI or FAB. GC-MS were run on a Varian Saturn, using a Varian VF-5ms capillary column (30 m x 0.25 mm). Diethyl ether and methanol are PA ACS grade. Isocyanides were purchased from Aldrich. Ethyl glyoxylate and benzyl glyoxylamide were synthesized by oxidation with HIO<sub>4</sub> of, respectively, the diethyl ester and the dibenzylamide of tartaric acid.<sup>1</sup> Zinc 325 mesh dust was purchased from Aldrich. All experiments were carried out in stoppered flasks without using any inert atmosphere. Liquid reagents were measured using Gilson positive-displacement micropipettes with disposable tips and pistons. Ultrasound irradiation was carried out in a 40 KHz sonication bath. Thin layer chromatography was performed on aluminium plates coated with Merck Kieselgel 60 GF-254 silica gel, using 254 nm UV light or a mixture of *p*-anisaldehyde (2.5%), acetic acid (1%) and H<sub>2</sub>SO<sub>4</sub> (3.4%) in 95% ethanol, as developer. Flash column chromatography was performed as described by Still *et al.*<sup>2</sup> employing silica gel Merck 60 (230-400 mesh).

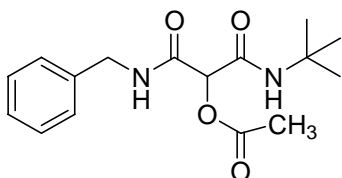
**Synthesis of the Passerini adducts: General procedure.** Benzyl glyoxylamide (**1**) or ethyl glyoxylate (**2**) (5 mmol) was suspended in diethyl ether (5 mL). Acetic acid (**4**, 5 mmol), and the corresponding isocyanide (**3**) (5 mmol) were successively added, and the mixture was stirred 72 h at room temperature. An abundant precipitate was formed, which was filtrated and successively washed with *i*-PrOH (5 mL) and *i*-Pr<sub>2</sub>O (5 mL), yielding a product (**5** or **6**) usually pure enough to be used in the following reaction. For analytical purposes, the adducts may be further purified by recrystallisation from ethanol.



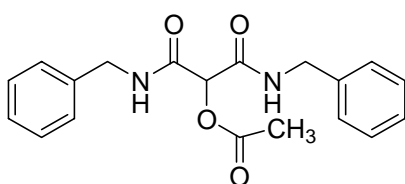
**(Benzylcarbamoyl)(cyclohexylcarbamoyl)methyl acetate (5a).** (68%) obtained as a white solid; mp 175-177 °C; IR (cm<sup>-1</sup>) 3272, 2938, 1747, 1679, 1659, 1543, 1223, 1105; <sup>1</sup>H-NMR (400 MHz, CDCl<sub>3</sub>) δ 7.35-7.20 (m, 6 H), 6.65 (d, 1 H, J = 7.8 Hz), 5.59 (s, 1 H), 4.46 (m, 2 H), 3.75 (m, 1 H), 2.25 (s, 3 H), 2.00-1.10 (m, 10 H); <sup>13</sup>C-NMR (100 MHz, CDCl<sub>3</sub>) δ 168.52 (C), 164.62 (C), 163.51 (C), 137.25 (C), 128.69 (CH), 127.55 (CH), 72.20 (CH), 48.68 (CH), 43.53 (CH<sub>2</sub>), 32.57 (CH<sub>2</sub>), 25.29 (CH<sub>2</sub>), 24.60 (CH<sub>2</sub>), 20.67 (CH<sub>3</sub>); MS (FAB) *m/z* (%) 333 (M<sup>+</sup> + 1, 100), 291 (11); HRMS (FAB) Calcd for C<sub>18</sub>H<sub>25</sub>N<sub>2</sub>O<sub>4</sub>: 333.1813. Found: 333.1814.

<sup>1</sup> P. Xu, W. W. Lin, X. M. Zou, *Synthesis*, 2002, 1017.

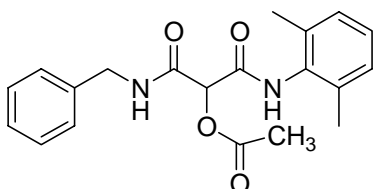
<sup>2</sup> W. C. Still, M. Kahn, A. Mitra, *J. Org. Chem.*, 1978, **43**, 2923.



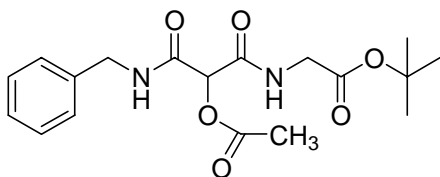
**(*tert*-Butylcarbamoyl)(benzylcarbamoyl)methyl acetate (5b).** (70%) obtained as a white solid; mp 172-174 °C; IR (cm<sup>-1</sup>) 3273, 1765, 1675, 1657, 1540, 1224; <sup>1</sup>H-NMR (400 MHz, CDCl<sub>3</sub>) δ 7.35-7.20 (m, 5 H), 7.18 (br s, 1 H), 6.64 (br s, 1 H), 5.52 (s, 1 H), 4.47 (m, 2 H), 2.25 (s, 3 H), 1.36 (s, 9 H); <sup>13</sup>C-NMR (100 MHz, CDCl<sub>3</sub>) δ 168.42 (C), 164.94 (C), 163.33 (C), 137.25 (C), 128.66 (CH), 127.55 (CH), 127.45 (CH), 72.43 (CH), 51.84 (C), 43.45 (CH<sub>2</sub>), 28.46 (CH<sub>3</sub>), 20.69 (CH<sub>3</sub>); MS (FAB) *m/z* (%) 307 (M<sup>+</sup> + 1, 100), 263 (6), 209 (10); HRMS (FAB) Calcd for C<sub>16</sub>H<sub>23</sub>N<sub>2</sub>O<sub>4</sub> 307.1658, Found 307.1664.



**Bis(benzylcarbamoyl)methyl acetate (5c).** (55%) obtained as a white solid; mp 134-136 °C; IR (cm<sup>-1</sup>) 3256, 3076, 1756, 1685, 1656, 1545, 1213; <sup>1</sup>H-NMR (400 MHz, CDCl<sub>3</sub>) δ 7.35-7.20 (m, 12 H), 5.65 (s, 1 H), 4.45 (m, 4 H), 2.20 (s, 3 H); <sup>13</sup>C-NMR (100 MHz, CDCl<sub>3</sub>) δ 168.53 (C), 164.52 (C), 137.17 (C), 128.70 (CH), 127.59 (CH), 72.33 (CH), 43.53 (CH<sub>2</sub>), 20.56 (CH<sub>3</sub>); MS (EI) *m/z* (%) 341 (M<sup>+</sup> + 1, 1), 280 (12), 176 (15), 165 (12), 146 (14), 106 (51), 91 (100); HRMS Calcd for C<sub>19</sub>H<sub>21</sub>N<sub>2</sub>O<sub>4</sub> 341.1501, Found 341.1500.

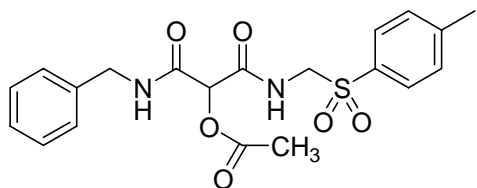


**(2,6-Dimethylphenylcarbamoyl)(benzylcarbamoyl)methyl acetate (5d).** (61%) obtained as a white solid; mp 157-159 °C; IR (cm<sup>-1</sup>) 3215, 3030, 1752, 1691, 1656, 1531, 1215, 1080; <sup>1</sup>H-NMR (400 MHz, CDCl<sub>3</sub>) δ 8.14 (s, 1 H), 7.35-7.25 (m, 5 H), 7.15-7.00 (m, 4 H), 5.79 (s, 1 H), 4.51 (m, 2 H), 2.28 (s, 3 H), 2.16 (s, 6 H); <sup>13</sup>C-NMR (100 MHz, CDCl<sub>3</sub>) δ 168.54 (C), 164.77 (C), 162.71 (C), 137.08 (C), 135.29 (C), 132.50 (C), 128.77 (CH), 128.19 (CH), 127.74 (CH), 127.62 (CH), 72.68 (CH), 43.63 (CH<sub>2</sub>), 20.67 (CH<sub>3</sub>), 18.18 (CH<sub>3</sub>); MS (EI) *m/z* (%) 354 (M<sup>+</sup>, 2), 312 (8), 207 (13), 165 (79), 121 (16), 106 (26), 91 (100); HRMS Calcd for C<sub>20</sub>H<sub>22</sub>N<sub>2</sub>O<sub>4</sub> 354.1580, Found 354.1579.



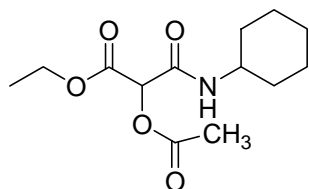
**(2-Acetoxy-2-benzylcarbamoyl-acetyl-amino)-acetic acid *tert*-butyl ester (5e).** (65%) obtained as a white solid; mp 134-137 °C; IR (cm<sup>-1</sup>) 3347, 1731, 1686, 1666, 1533, 1371, 1219, 1162; <sup>1</sup>H-NMR (400 MHz, CDCl<sub>3</sub>) δ 7.35-7.25 (m, 6 H), 7.16 (m, 1 H), 5.68 (s, 1 H), 4.48 (m, 2 H), 4.00 (dd, 1 H J = 18.27 and 5.39 Hz), 3.89 (dd, 1 H J = 18.26 and 4.88 Hz), 2.27 (s, 3 H), 1.48 (s, 9 H); <sup>13</sup>C-NMR (100 MHz, CDCl<sub>3</sub>) δ 168.54 (C), 167.93 (C), 164.78 (C), 164.16 (C), 137.18 (C), 128.71 (CH),

127.68 (CH), 127.63 (CH), 82.64 (C), 72.07 (CH), 43.63 (CH<sub>2</sub>), 42.02 (CH<sub>2</sub>), 27.95 (CH<sub>3</sub>), 20.61 (CH<sub>3</sub>); MS (FAB)  $m/z$  (%) 365 ( $M^+ + 1$ , 16), 331 (4), 309 (100), 267 (26); HRMS Calcd for C<sub>18</sub>H<sub>25</sub>N<sub>2</sub>O<sub>6</sub> 365.1713, Found 365.1713.



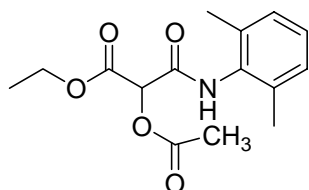
**(Benzylcarbamoyl)(tosylmethylcarbamoyl)methyl acetate (5f).** (87%) obtained as a white solid; mp 175-176 °C; IR (cm<sup>-1</sup>) 3370, 1757, 1715, 1673, 1524, 1281, 1226, 1140;

<sup>1</sup>H-NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.75 (d, 2 H,  $J$  = 8.2 Hz), 7.42 (m, 1 H), 7.38-7.23 (m, 7 H), 6.80 (t, 1 H,  $J$  = 5.5 Hz), 5.54 (s, 1 H), 4.72-4.60 (m, 2 H), 4.52-4.39 (m, 2 H), 2.40 (s, 3 H), 2.23 (s, 3 H); <sup>13</sup>C-NMR (100 MHz, CDCl<sub>3</sub>)  $\delta$  168.10 (C), 164.15 (C), 163.85 (C), 145.47 (C), 136.73 (C), 133.49 (C), 129.97 (CH), 128.85 (CH), 127.88 (CH), 127.66 (CH), 71.73 (CH), 60.00 (CH<sub>2</sub>), 43.62 (CH<sub>2</sub>), 21.67 (CH<sub>3</sub>), 20.63 (CH<sub>3</sub>); MS (FAB)  $m/z$  (%) 419 ( $M^+ + 1$ , 52), 278 (16), 263 (42), 154 (100); HRMS Calcd for C<sub>20</sub>H<sub>23</sub>N<sub>2</sub>O<sub>6</sub>S 419.1277, Found 419.1297.

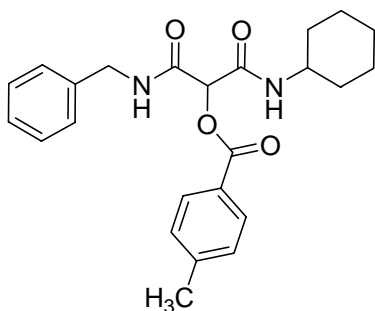


**2-Acetoxy-N-cyclohexyl-malonamic acid ethyl ester (6a).** (45%) obtained as a white solid; mp 67-69 °C; IR (cm<sup>-1</sup>) 3270, 2993, 2860, 1765, 1748, 1660, 1567, 1223, 1189, 1102;

<sup>1</sup>H-NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  6.23 (d, 1 H,  $J$  = 6.4 Hz), 5.37 (s, 1 H), 4.28 (c, 2 H,  $J$  = 7.1 Hz), 3.80 (m, 1 H), 2.24 (s, 3 H), 2.00-1.10 (m, 10 H), 1.32 (t, 3 H,  $J$  = 7.1 Hz); <sup>13</sup>C-NMR (100 MHz, CDCl<sub>3</sub>)  $\delta$  168.97 (C), 165.96 (C), 161.81 (C), 72.94 (CH), 62.47 (CH<sub>2</sub>), 48.55 (CH), 32.63 (CH<sub>2</sub>), 25.32 (CH<sub>2</sub>), 24.63 (CH<sub>2</sub>), 20.52 (CH<sub>3</sub>), 13.92 (CH<sub>3</sub>); MS (CI, MeOH)  $m/z$  (%) 272 ( $M^+ + 1$ , 1), 230 (100); HRMS Calcd for C<sub>13</sub>H<sub>21</sub>NO<sub>5</sub> 271.1420, Found 271.1413.



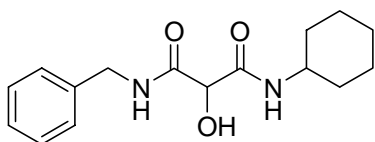
**2-Acetoxy-N-(2,6-dimethyl-phenyl)-malonamic acid ethyl ester (6d).** (61%) obtained as a white solid; mp 119-120 °C; IR (cm<sup>-1</sup>) 3251, 1765, 1750, 1669, 1539, 1232, 1102; <sup>1</sup>H-NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.73 (s, 1 H), 7.15-7.05 (m, 3 H), 5.58 (s, 1 H), 4.33 (c, 2 H,  $J$  = 7.1 Hz), 2.27 (s, 3 H), 2.20 (s, 6 H), 1.33 (t, 3 H,  $J$  = 7.1 Hz); <sup>13</sup>C-NMR (100 MHz, CDCl<sub>3</sub>)  $\delta$  169.08 (C), 165.96 (C), 161.35 (C), 135.33 (C), 132.29 (C), 128.25 (CH), 127.75 (CH), 73.21 (CH), 62.74 (CH<sub>2</sub>), 20.49 (CH<sub>3</sub>), 18.12 (CH<sub>3</sub>), 13.97 (CH<sub>3</sub>); MS (EI)  $m/z$  (%) 293 ( $M^+$ , 11), 251 (10), 178 (9), 148 (73), 121 (52), 104 (100), 76 (34); HRMS Calcd for C<sub>15</sub>H<sub>19</sub>NO<sub>5</sub> 293.1263, Found 293.1266.



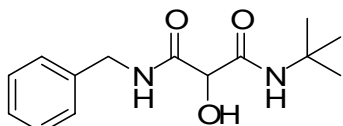
**Synthesis of the (benzylcarbamoyl)(cyclohexylcarbamoyl)methyl 4-methylbenzoate (10).** Benzyl glyoxylamide (**1**, 5 mmol) was suspended in diethyl ether (5 mL). *p*-Toluic acid (**4**, 5 mmol), and cyclohexyl isocyanide (**3a**) (5 mmol) were successively added, and the mixture was stirred 72 h at room temperature. An abundant precipitate was formed, which was filtrated and successively washed with *i*-PrOH and *i*-Pr<sub>2</sub>O, yielding the adduct **10** (31%) as a white solid; mp 176-178 °C; IR (cm<sup>-1</sup>) 3280, 1724, 1663, 1542, 1289, 1115; <sup>1</sup>H-NMR (400 MHz, CDCl<sub>3</sub>) δ 7.99 (d, 2 H, *J* = 8.1 Hz), 7.50-7.15 (m, 8 H), 6.73 (d, 1 H, *J* = 7.6 Hz), 5.84 (s, 1 H), 4.49 (m, 2 H), 3.78 (m, 1 H), 2.42 (s, 3 H), 2.00-1.10 (m, 10 H); <sup>13</sup>C-NMR (100 MHz, CDCl<sub>3</sub>) δ 164.88 (C), 164.57 (C), 163.90 (C), 144.70 (C), 137.36 (C), 130.02 (CH), 129.32 (CH), 128.69 (CH), 127.50 (CH), 125.89 (C), 72.39 (CH), 48.68 (CH), 43.56 (CH<sub>2</sub>), 32.60 (CH<sub>2</sub>), 25.33 (CH<sub>2</sub>), 24.59 (CH<sub>2</sub>), 21.71 (CH<sub>3</sub>); MS (CI, CH<sub>4</sub>) *m/z* (%) 409 (M<sup>+</sup> + 1, 15), 251 (25), 137 (31), 73 (32), 55 (100).

#### General procedure for the hydrolysis.

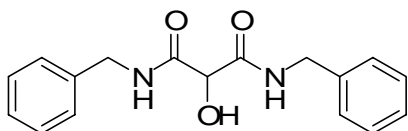
A solution of the starting Passerini adduct (**5 or 6**) (0.3 mmol), and ZnCl<sub>2</sub> (0.03 mmol) in methanol (12 mL) was added to a flask containing Zn dust (3 mmol). The resulting mixture was irradiated in a sonication bath 3-4 h (7 h in the case of adducts **6a,d**), and then decanted from the Zn and concentrated. The product (**8 or 9**) was purified by flash column chromatography (15 cm x 2.5 cm Ø, SiO<sub>2</sub>, hexane-ethyl acetate 7:3 to 1:1).



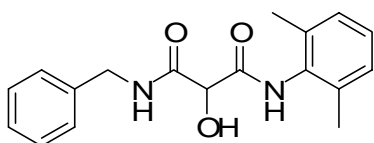
**N<sup>1</sup>-benzyl-N<sup>3</sup>-cyclohexyl-2-hydroxymalonamide (8a).** (99%) obtained as a white solid; mp 142-144 °C; IR (cm<sup>-1</sup>) 3279, 2933, 1644, 1542, 1242, 1132; <sup>1</sup>H-NMR (400 MHz, CDCl<sub>3</sub>) δ 7.61 (br s, 1 H), 7.35-7.10 (m, 5 H), 7.13 (br d, 1 H, *J* = 7.6 Hz), 4.64 (br s, 1 H), 4.47 (m, 3 H), 3.75 (m, 1 H), 2.0-1.10 (m, 10 H); <sup>13</sup>C-NMR (100 MHz, CDCl<sub>3</sub>) δ 168.72 (C), 167.30 (C), 137.21 (C), 128.71 (CH), 127.62 (CH), 127.50 (CH), 70.26 (CH), 48.70 (CH), 43.60 (CH<sub>2</sub>), 32.65 (CH<sub>2</sub>), 25.31 (CH<sub>2</sub>), 24.58 (CH<sub>2</sub>); MS (EI) *m/z* (%) 290 (M<sup>+</sup>, 7), 209 (2), 165 (41), 157 (25), 106 (35), 91 (100), 74 (49); HRMS Calcd for C<sub>16</sub>H<sub>22</sub>N<sub>2</sub>O<sub>3</sub>: 290.1630. Found: 290.1642.



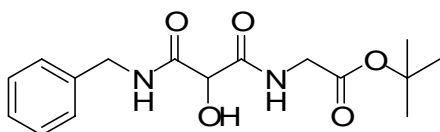
**N<sup>1</sup>-tert-butyl-N<sup>3</sup>-benzyl-2-hydroxymalonamide (8b).** (97%) obtained as a white solid; mp 70-73 °C; IR (cm<sup>-1</sup>) 3361, 1652, 1639, 1555, 1224, 1119; <sup>1</sup>H-NMR (400 MHz, CDCl<sub>3</sub>) δ 7.60 (br s, 1 H), 7.35-7.20 (m, 5 H), 7.11 (br s, 1 H), 4.66 (br s, 1 H), 4.48 (d, 2 H, *J* = 6.0 Hz), 4.41 (s, 1 H), 1.37 (s, 9 H); <sup>13</sup>C-NMR (100 MHz, CDCl<sub>3</sub>) δ 168.99 (C), 167.17 (C), 137.24 (C), 128.69 (CH), 127.60 (CH), 127.41 (CH), 70.44 (CH), 51.65 (C), 43.52 (CH<sub>2</sub>), 28.51 (CH<sub>3</sub>); MS (EI) *m/z* (%) 264 (M<sup>+</sup>, 5), 207 (3), 165 (45), 131 (13), 106 (23), 91 (100), 74 (53); HRMS Calcd for C<sub>14</sub>H<sub>20</sub>N<sub>2</sub>O<sub>3</sub>: 264.1474, Found 264.1464.



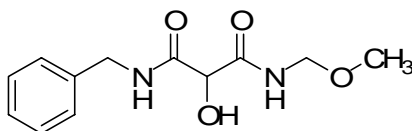
**RCC127: *N*<sup>1</sup>,*N*<sup>3</sup>-dibenzyl-2-hydroxymalonamide (8c).** (77%) obtained as a white solid; mp 136-137 °C; IR (cm<sup>-1</sup>) 3331, 1676, 1538, 1254, 1101; <sup>1</sup>H-NMR (400 MHz, CDCl<sub>3</sub>) δ 7.62 (m, 2 H), 7.40-7.20 (m, 10 H), 4.65 (d, 1 H, J = 2.9 Hz), 4.54 (d, 1 H, J = 2.7 Hz), 4.46 (m, 4 H); <sup>13</sup>C-NMR (100 MHz, CDCl<sub>3</sub>) δ 168.40 (C), 137.13 (C), 128.71 (CH), 127.62 (CH), 127.51 (CH), 70.40 (CH), 43.63 (CH<sub>2</sub>); MS (EI) *m/z* (%) 299 (M<sup>+</sup>, 30), 208 (26), 165 (32), 106 (57), 91 (100), 65 (28); HRMS Calcd for C<sub>17</sub>H<sub>18</sub>N<sub>2</sub>O<sub>3</sub> 298.1317, Found 298.1326.



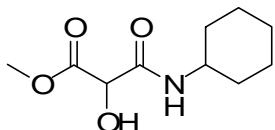
***N*<sup>1</sup>-benzyl-2-hydroxy-*N*<sup>3</sup>-(2,6-dimethylphenyl)malonamide (8d).** (99%) obtained as a white solid; mp 110-112 °C; IR (cm<sup>-1</sup>) 3309, 1672, 1655, 1522, 1133; <sup>1</sup>H-NMR (400 MHz, CDCl<sub>3</sub>) δ 8.63 (br s, 1 H), 7.63 (br s, 1 H), 7.35-7.25 (m, 5 H), 7.15-7.05 (m, 3 H), 4.75 (m, 2 H), 4.52 (m, 2 H), 2.17 (s, 6 H); <sup>13</sup>C-NMR (100 MHz, CDCl<sub>3</sub>) δ 168.57 (C), 166.90 (C), 137.15 (C), 135.00 (C), 132.57 (C), 128.71 (CH), 128.22 (CH), 127.60 (CH), 70.59 (C), 43.66 (CH<sub>2</sub>), 18.21 (CH<sub>3</sub>); MS (EI) *m/z* (%) 312 (M<sup>+</sup>, 9), 221 (7), 165 (40), 148 (14), 120 (13), 91 (100), 74 (35); HRMS Calcd for C<sub>18</sub>H<sub>20</sub>N<sub>2</sub>O<sub>3</sub> 312.1474, Found 312.1464.



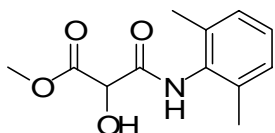
**(2-Benzylcarbamoyl-2-hydroxy-acetyl-amino)-acetic acid *tert*-butyl ester (8e).** (94%) obtained as a white solid; mp 60-62 °C; IR (cm<sup>-1</sup>) 3348, 1742, 1683, 1526, 1369, 1230, 1157; <sup>1</sup>H-NMR (400 MHz, CDCl<sub>3</sub>) δ 7.72 (m, 1 H), 7.54 (m, 1 H), 7.35-7.25 (m, 5 H), 4.59 (m, 1 H), 4.57 (m, 1 H), 4.48 (m, 2 H), 3.95 (m, d, 2 H, J = 5.5 Hz), 1.47 (s, 9 H); <sup>13</sup>C-NMR (100 MHz, CDCl<sub>3</sub>) δ 168.81 (C), 168.14 (C), 167.81 (C), 137.12 (C), 128.71 (CH), 127.62 (CH), 82.46 (C), 70.38 (CH), 43.67 (CH<sub>2</sub>), 42.05 (CH<sub>2</sub>), 27.95 (CH<sub>3</sub>); MS (FAB) *m/z* (%) 323 (M<sup>+</sup> + 1, 13), 289 (7), 267 (100); HRMS Calcd for C<sub>16</sub>H<sub>23</sub>N<sub>2</sub>O<sub>5</sub> 323.1607, Found 323.1618.



***N*<sup>1</sup>-benzyl-2-hydroxy-*N*<sup>3</sup>-(methoxymethyl)malonamide (11).** (77%) obtained as a white solid; mp 74-77 °C; IR (cm<sup>-1</sup>) 3327, 1698, 1676, 1638, 1542, 1523, 1132, 1111; <sup>1</sup>H-NMR (400 MHz, CDCl<sub>3</sub>) δ 7.92 (br s, 1 H), 7.50 (br s, 1 H), 7.35-7.25 (m, 5 H), 4.72 (dd, 2 H, J = 1.0 and 6.9 Hz), 4.60-4.54 (m, 2 H), 4.48 (m, 2 H), 3.31 (s, 3 H); <sup>13</sup>C-NMR (100 MHz, CDCl<sub>3</sub>) δ 169.71 (C), 168.14 (C), 137.05 (C), 128.78 (CH), 127.76 (CH), 127.62 (CH), 71.53 (CH<sub>2</sub>), 70.41 (CH), 56.14 (CH<sub>3</sub>), 43.73 (CH<sub>2</sub>); MS (EI) *m/z* (%) 252 (M<sup>+</sup>, 5), 220 (30), 91 (100), 87 (35), 65 (35); HRMS (FAB) Calcd for C<sub>12</sub>H<sub>17</sub>N<sub>2</sub>O<sub>4</sub> 253.1188, Found 253.1178.



**Methyl 2-(cyclohexylcarbamoyl)-2-hydroxyacetate (9a).** (12%); obtained as a clear oil; IR (cm<sup>-1</sup>) 3431, 2932, 1744, 1655, 1262, 1098, 803; <sup>1</sup>H-NMR (400 MHz, CDCl<sub>3</sub>) δ 6.57 (br s, 1 H), 4.62 (s, 1 H), 3.87 (s, 3 H), 3.83-3.74 (m, 1H), 1.95-0.85 (m, 10 H); <sup>13</sup>C-NMR (100 MHz, CDCl<sub>3</sub>) δ 170.86 (C), 165.12 (C), 71.28, (CH), 53.65 (CH<sub>3</sub>), 48.71 (CH), 32.73 (CH<sub>2</sub>), 32.64 (CH<sub>2</sub>), 25.30 (CH<sub>2</sub>), 24.55 (CH<sub>2</sub>); MS (EI) *m/z* (%) 216 (M<sup>+</sup>, 100), 156 (2), 134 (5), 90 (19), 83 (4).



**Methyl 2-(2,6-dimethylphenylcarbamoyl)-2-hydroxyacetate (9d).** (51%) obtained as a white solid; mp 114-116 °C; IR (cm<sup>-1</sup>) 3507, 3250, 1745, 1670, 1540, 1266, 1132; <sup>1</sup>H-NMR (400 MHz, CDCl<sub>3</sub>) δ 8.01 (br s, 1 H), 7.15-7.07 (m, 3 H), 4.92 (s, 1 H), 3.92 (s, 3 H), 2.18 (s, 6 H); <sup>13</sup>C-NMR (100 MHz, CDCl<sub>3</sub>) δ 170.70 (C), 164.70 (C), 135.07 (C), 132.40 (C), 128.27 (CH), 127.70 (CH), 71.64, (CH), 53.65 (CH<sub>3</sub>), 18.14 (CH<sub>3</sub>); MS (EI) *m/z* (%) 238 (M<sup>+</sup>, 100), 148 (17), 120 (14), 105 (10), 90 (9); HRMS Calcd for C<sub>12</sub>H<sub>15</sub>NO<sub>4</sub> 237.1001, Found 237.0991.

