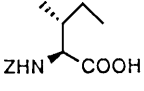
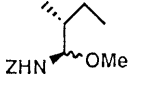
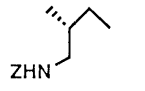
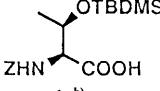
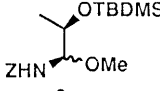
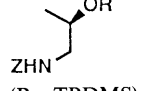
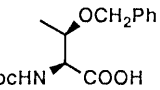
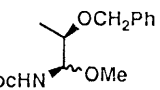
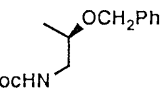
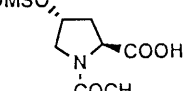
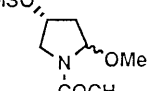
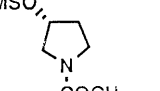
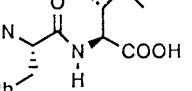
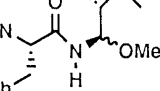
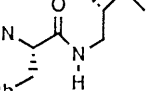




TABLE I. Anodic Oxidation of **1b–f** and Et<sub>3</sub>SiH Reduction of **2b–f**

<i>N</i> -Acyl- $\alpha$ -amino acid	<i>N</i> -Acyl- $\alpha$ -methoxyalkylamine	Yield (%)	<i>N</i> -Acylamine	Method <sup>a)</sup>	Yield (%)
 <b>1b<sup>b)</sup></b>	 <b>2b</b>	98	 <b>3b</b>	A C	84 75
 <b>1c<sup>b)</sup></b>	 <b>2c</b>	96	 <b>3c</b> (R = TBDMS) <b>3c'</b> (R = H)	B C	88 42 ( <b>3c</b> ) 51 ( <b>3c'</b> )
 <b>1d<sup>b)</sup></b>	 <b>2d</b>	98	 <b>3d</b>	B	98
 <b>1e</b>	 <b>2e</b>	97	 <b>3e</b>	B	96
 <b>1f</b>	 <b>2f</b>	86	 <b>3f</b>	A C	89 87

a) Method A, Et<sub>3</sub>SiH-BF<sub>3</sub>·OEt<sub>2</sub> at 5 °C; method B, Et<sub>3</sub>SiH-BF<sub>3</sub>·OEt<sub>2</sub> at -40 °C; method C, Et<sub>3</sub>SiH-TFA at room temperature. b) Z = benzyloxycarbonyl; Boc = *tert*-butoxycarbonyl; TBDMS = *tert*-butyldimethylsilyl.

(30 ml) containing NaOMe (1 mmol) was electrolyzed at 5–10 °C using a 6.4 cm<sup>2</sup> of graphite anode–graphite cathode system in a non-divided cell. The electrolysis current was maintained at 0.6 A during the electrolysis. After the theoretical amount of electricity had passed, the electrolyzed solution was evaporated to dryness *in vacuo*. The residue was dissolved in EtOAc. The solution was washed with saturated aqueous NaHCO<sub>3</sub> solution and brine, dried (MgSO<sub>4</sub>) and evaporated to dryness *in vacuo* to afford compound **2a**.

**(2*R*)-*N*-Acetyl-1-methoxy-2-methylbutylamine (2a)** mp 36–37 °C. IR (Nujol): 3300 (NH), 1660 (CO) cm<sup>-1</sup>. <sup>1</sup>H-NMR (CDCl<sub>3</sub>)  $\delta$ : 0.91 (t, 3H, *J* = 6.0 Hz, C<sub>4</sub>-H), 0.93 (d, 3H, *J* = 5.5 Hz, CH<sub>3</sub>), 1.03–1.29 (m, 1H, C<sub>3</sub>-H), 1.41–1.73 (m, 2H, C<sub>2</sub>-H, C<sub>3</sub>-H), 2.10 (s, 3H, COCH<sub>3</sub>), 3.32 (s, 3H, OCH<sub>3</sub>), 4.90–5.00 (m, 1H, C<sub>1</sub>-H), 6.05 (br 1H, NH). MS *m/z*: 128 (M<sup>+</sup> - CH<sub>3</sub>O). Anal. Calcd for C<sub>8</sub>H<sub>17</sub>NO<sub>2</sub>: C, 60.34; H, 10.76; N, 8.80. Found: C, 60.46; H, 11.10; N, 8.69.

**(2*R*)-*N*-Benzyloxycarbonyl-1-methoxy-2-methylbutylamine (2b)** Colorless syrup. IR (film): 3320 (NH), 1705 (CO), 1520 cm<sup>-1</sup>. <sup>1</sup>H-NMR (CDCl<sub>3</sub>)  $\delta$ : 0.88 (d, 3H, *J* = 6.8 Hz, CH<sub>3</sub>), 0.90 (t, 3H, *J* = 6.6 Hz, C<sub>4</sub>-H), 1.00–1.30 (m, 1H, C<sub>3</sub>-H), 1.40–1.75 (m, 2H, C<sub>3</sub>-H, C<sub>2</sub>-H), 3.34 (s, 3H, OCH<sub>3</sub>), 4.65–4.82 (m, 1H, C<sub>1</sub>-H), 4.99 (br, 1H, NH), 5.13 (s, 2H, PhCH<sub>2</sub>O), 7.34 (s, 5H, Ar-H). MS *m/z*: 219 (M<sup>+</sup> - CH<sub>3</sub>OH). Anal. Calcd for C<sub>14</sub>H<sub>21</sub>NO<sub>3</sub>: C, 66.91; H, 8.42; N, 5.57. Found: C 66.72; H, 8.51; N, 5.45.

**(2*R*)-*N*-Benzyloxycarbonyl-1-methoxy-2-(*tert*-butyldimethylsilyloxy)-propylamine (2c)** Colorless syrup. IR (film): 3460 (NH), 3350 (NH), 1730 (CO), 1720 (CO), 1500 cm<sup>-1</sup>. <sup>1</sup>H-NMR (CDCl<sub>3</sub>)  $\delta$ : -0.06–0.05 (m, 6H, SiCH<sub>3</sub>), 0.81–0.84 (m, 9H, *tert*-Bu), 1.04, 1.11 (each d, 3H, *J* = 6.4 Hz, C<sub>3</sub>-H), 3.29, 3.33 (each s, 3H, OCH<sub>3</sub>), 3.80–3.95 (m, 1H, C<sub>2</sub>-H), 4.61, 4.71 (each dd, 1H, *J* = 1.8, 9.9 Hz, C<sub>1</sub>-H), 5.09 (s, 2H, PhCH<sub>2</sub>O), 5.42 (d, 1H, *J* = 9.9 Hz, NH), 7.25–7.36 (m, 5H, Ar-H). MS *m/z*: 321 (M<sup>+</sup> - CH<sub>3</sub>OH). Anal. Calcd for C<sub>18</sub>H<sub>31</sub>NO<sub>4</sub>Si: C, 61.15; H, 8.84; N, 3.96; Si, 7.94. Found: C, 60.98; H, 9.27; N, 3.96; Si, 8.05.

**(2*R*)-*N*-*tert*-Butyloxycarbonyl-1-methoxy-2-benzyloxypropylamine (2d)** Colorless syrup. IR (film): 3450 (NH), 3320, 1725 (CO), 1500 cm<sup>-1</sup>. <sup>1</sup>H-NMR (CDCl<sub>3</sub>)  $\delta$ : 1.14, 1.23 (each d, 3H, *J* = 6.5 Hz, C<sub>3</sub>-H), 1.44, 1.46 (each s, 9H, *tert*-Bu), 3.37, 3.38 (each s, 3H, OCH<sub>3</sub>), 3.49–3.62, 3.65–3.82

(each m, 1H, C<sub>2</sub>-H), 4.40–4.85 (m, 3H, C<sub>1</sub>-H, OCH<sub>2</sub>), 5.20, 5.35 (each br, 1H, NH), 7.25–7.35 (m, 5H, Ar-H). MS *m/z*: 263 (M<sup>+</sup> - CH<sub>3</sub>OH). Anal. Calcd for C<sub>16</sub>H<sub>25</sub>NO<sub>4</sub>: C, 65.06; H, 8.53; N, 4.74. Found: C, 64.81; H, 8.35; N, 4.41.

**(4*R*)-1-Acetyl-2-methoxy-4-(*tert*-butyldimethylsilyloxy)-pyrrolidine (2e)** Colorless syrup. IR (film): 3450 (NH), 1665 (CO), 1415 cm<sup>-1</sup>. <sup>1</sup>H-NMR (CDCl<sub>3</sub>)  $\delta$ : 0.07 (s, 6H, SiCH<sub>3</sub>), 0.88 (s, 9H, *tert*-Bu), 1.75–2.10, 2.15–2.39 (each m, 2H, C<sub>3</sub>-H), 2.07, 2.13, 2.14 (each s, 3H, COCH<sub>3</sub>), 3.27, 3.29, 3.37, 3.40 (each s, 3H, OCH<sub>3</sub>), 3.10–3.55, 3.65–3.95 (each m, 2H, C<sub>5</sub>-H), 4.30–4.50, 4.50–4.75 (each m, 1H, C<sub>4</sub>-H), 5.00–5.45 (m, 1H, C<sub>2</sub>-H). MS *m/z*: 242 (M<sup>+</sup> - CH<sub>3</sub>O). Anal. Calcd for C<sub>13</sub>H<sub>27</sub>NO<sub>3</sub>Si: C, 57.10; H, 9.95; N, 5.12; Si, 10.27. Found: C, 56.84; H, 9.75; N, 4.78; Si, 9.82.

***N*-Benzyloxycarbonyl-L-phenylalanine *N*-(1-Methoxy-2(*R*)-methyl)butylamide (2f)** mp 138–139 °C (MeOH). IR (Nujol): 3300 (NH), 1690 (CO), 1660 (CO), 1540 cm<sup>-1</sup>. <sup>1</sup>H-NMR (CDCl<sub>3</sub>)  $\delta$ : 0.66 (d, 3H, *J* = 6.8 Hz, CH<sub>3</sub>), 0.80–0.90 (m, 3H, CH<sub>2</sub>CH<sub>3</sub>), 0.85–1.60 (m, 3H, CHCH<sub>2</sub>), 3.11 (s, 3H, OCH<sub>3</sub>), 2.90–3.10 (m, 2H, CH<sub>2</sub>Ph), 4.45 (q, 1H, *J* = 7.0 Hz, NHCHCO), 4.80–5.00 (m, 1H, NHCHO), 5.05, 5.13 (ABq, 2H, *J* = 12.3 Hz, PhCH<sub>2</sub>O), 5.43 (m, 1H, NH), 5.90–6.10 (m, 1H, NH), 7.00–7.35 (m, 5H, Ar-H), 7.33 (s, 5H, Ar-H). MS *m/z*: 366 (M<sup>+</sup> - CH<sub>3</sub>OH). Anal. Calcd for C<sub>23</sub>H<sub>30</sub>N<sub>2</sub>O<sub>4</sub>: C, 69.32; H, 7.59; N, 7.03. Found: C, 69.42; H, 7.70; N, 7.01.

**Typical Procedures for Et<sub>3</sub>SiH Reduction** Method A: A solution of **2a** (2 mmol) and Et<sub>3</sub>SiH (2.4 mmol) in dry CH<sub>2</sub>Cl<sub>2</sub> (3 ml) was treated with BF<sub>3</sub>·OEt<sub>2</sub> (2.4 mmol) at 5 °C. After being stirred at 5 °C for 2 h, the reaction mixture was diluted with CHCl<sub>3</sub>. The solution was washed with saturated aqueous NaHCO<sub>3</sub> solution and brine. The organic layer was dried (MgSO<sub>4</sub>) and concentrated to dryness *in vacuo*, and the resulting syrup was subjected to silica gel chromatography (CHCl<sub>3</sub>–acetone, 5 : 1) to afford compound **3a**.

Method B: A solution of **2c** (2 mmol) and Et<sub>3</sub>SiH (4 mmol) in dry CH<sub>2</sub>Cl<sub>2</sub> (3 ml) was treated with BF<sub>3</sub>·OEt<sub>2</sub> (4 mmol) at -40 °C. The temperature of the solution was maintained at -40 °C until the starting material disappeared on thin layer chromatography (TLC) (1–2 h). The reaction mixture was diluted with CHCl<sub>3</sub>. The solution was washed with saturated aqueous NaHCO<sub>3</sub> solution and brine. The organic layer was dried (MgSO<sub>4</sub>) and evaporated to dryness *in vacuo*. Purification of the

residue by silica gel chromatography (*n*-hexane–EtOAc, 10:1) gave compound **3c**.

**Method C:** A solution of **2b** (2 mmol) and Et<sub>3</sub>SiH (2.4 mmol) in dry CH<sub>2</sub>Cl<sub>2</sub> (3 ml) was treated with TFA (6 mmol) at 5°C. The reaction mixture was stirred at room temperature for 2 h, then quenched by addition of saturated aqueous NaHCO<sub>3</sub> solution. The mixture was extracted with CHCl<sub>3</sub>. The organic layer was separated, dried (MgSO<sub>4</sub>) and then evaporated to dryness *in vacuo* to give a syrup, which was purified by silica gel chromatography (*n*-hexane–EtOAc, 10:1) to afford compound **3b**.

**(2R)-N-Acetyl-2-methylbutylamine (3a)** This compound was prepared by method A. Colorless syrup,  $[\alpha]_D^{25.5} + 5.23^\circ$  ( $c = 1.11$ , CHCl<sub>3</sub>). IR (film): 3300 (NH), 1655 (CO) cm<sup>-1</sup>. <sup>1</sup>H-NMR (CDCl<sub>3</sub>)  $\delta$ : 0.87–0.94 (m, 6H, C<sub>4</sub>-H, CH<sub>3</sub>), 1.04–1.26 (m, 1H, C<sub>3</sub>-H), 1.30–1.67 (m, 2H, C<sub>2</sub>-H, C<sub>3</sub>-H), 1.99 (s, 3H, COCH<sub>3</sub>), 2.99–3.26 (m, 2H, C<sub>1</sub>-H), 5.61 (br, 1H, NH). MS  $m/z$ : 129 (M<sup>+</sup>). Anal. Calcd for C<sub>7</sub>H<sub>15</sub>NO: C, 65.07; H, 11.70; N, 10.84. Found: C, 64.92; H, 12.08; N, 10.61.

**(2R)-N-Benzoyloxycarbonyl-2-methylbutylamine (3b)** This compound was obtained by both method A and method C. Colorless syrup,  $[\alpha]_D^{25.5} + 4.07^\circ$  ( $c = 1.13$ , CHCl<sub>3</sub>). IR (film): 3330 (NH), 1710 (CO), 1540 cm<sup>-1</sup>. <sup>1</sup>H-NMR (CDCl<sub>3</sub>)  $\delta$ : 0.89 (d, 3H,  $J = 6.6$  Hz, CH<sub>3</sub>), 0.90 (t, 3H,  $J = 7.2$  Hz, C<sub>4</sub>-H), 1.00–1.63 (m, 3H, C<sub>2</sub>-H, C<sub>3</sub>-H), 2.90–3.23 (m, 2H, C<sub>1</sub>-H), 4.77 (br, 1H, NH), 5.10 (s, 2H, PhCH<sub>2</sub>O), 7.33 (s, 5H, Ar-H). MS  $m/z$ : 221 (M<sup>+</sup>). Anal. Calcd for C<sub>13</sub>H<sub>19</sub>NO<sub>2</sub>: C, 70.56; H, 8.65; N, 6.33. Found: C, 70.41; H, 9.07; N, 6.36.

**(2R)-N-Benzoyloxycarbonyl-2-(tert-butyltrimethylsilyloxy)propylamine (3c)** This compound was prepared by both method B and method C. Colorless syrup,  $[\alpha]_D^{25.5} - 22.1^\circ$  ( $c = 1.26$ , CHCl<sub>3</sub>). IR (film): 3470 (NH), 3350 (NH), 1730 (CO), 1710 (CO) cm<sup>-1</sup>. <sup>1</sup>H-NMR (CDCl<sub>3</sub>)  $\delta$ : 0.01 (s, 6H, SiCH<sub>3</sub>), 0.85 (s, 9H, *tert*-Bu), 1.07 (d, 3H,  $J = 6.2$  Hz, C<sub>3</sub>-H), 2.91–3.05 (m, 1H, C<sub>1</sub>-H), 3.18–3.31 (m, 1H, C<sub>1</sub>-H), 3.83–3.92 (m, 1H, C<sub>2</sub>-H), 4.96 (br, 1H, NH), 5.06 (s, 2H, PhCH<sub>2</sub>O), 7.21–7.34 (m, 5H, Ar-H). MS  $m/z$ : 323 (M<sup>+</sup>). Anal. Calcd for C<sub>17</sub>H<sub>29</sub>NO<sub>3</sub>Si: C, 63.12; H, 9.04; N, 4.33; Si, 8.68. Found: C, 62.88; H, 8.90; N, 4.10; Si, 8.32.

**(2R)-N-Benzoyloxycarbonyl-2-hydroxypropylamine (3c')** This compound was formed as a by-product in the reduction of **3c** using method C. Colorless syrup,  $[\alpha]_D^{25.5} - 20.3^\circ$  ( $c = 1.19$ , CHCl<sub>3</sub>). IR (film): 3350 (NH), 2910 (OH), 1700 (CO) cm<sup>-1</sup>. <sup>1</sup>H-NMR (CDCl<sub>3</sub>)  $\delta$ : 1.18 (d, 3H,  $J = 6.3$  Hz, CH<sub>3</sub>), 2.04 (br, 1H, OH), 2.98–3.12 (m, 1H, C<sub>1</sub>-H), 3.28–3.40 (m, 1H, C<sub>1</sub>-H), 3.91 (m, 1H, C<sub>2</sub>-H), 5.11 (s, 2H, PhCH<sub>2</sub>O), 5.21 (br, 1H, NH), 7.26–7.37 (m, 5H, Ar-H). MS  $m/z$ : 209 (M<sup>+</sup>). Anal. Calcd for C<sub>11</sub>H<sub>15</sub>NO<sub>3</sub>: C, 63.14; H, 7.23; N, 6.69. Found: C, 62.83; H, 7.25; N, 6.49.

**(2R)-N-tert-Butyloxycarbonyl-2-benzoyloxypropylamine (3d)** This compound was prepared by method B. Colorless syrup,  $[\alpha]_D^{25} - 37.9^\circ$  ( $c = 1.22$ , CHCl<sub>3</sub>). IR (film): 3450 (NH), 3350, 1720 (CO), 1500 cm<sup>-1</sup>. <sup>1</sup>H-NMR (CDCl<sub>3</sub>)  $\delta$ : 1.19 (d, 3H,  $J = 6.2$  Hz, C<sub>3</sub>-H), 1.44 (s, 9H, *tert*-Bu), 3.00–3.13 (m, 1H, C<sub>1</sub>-H), 3.20–3.40 (m, 1H, C<sub>1</sub>-H), 3.58–3.66 (m, 1H, C<sub>2</sub>-H), 4.45, 4.61 (ABq, 2H,  $J = 11.7$  Hz, PhCH<sub>2</sub>O), 4.86 (br, 1H, NH), 7.26–7.36 (m, 5H, Ar-H). MS  $m/z$ : 264 (M<sup>+</sup>). Anal. Calcd for C<sub>15</sub>H<sub>23</sub>NO<sub>3</sub>: C, 67.90; H, 8.74; N, 5.28. Found: C, 67.71; H, 8.65; N, 5.06.

**(3R)-N-Acetyl-3-(tert-butyltrimethylsilyloxy)propylamine (3e)** This compound was prepared by method B. Colorless syrup,  $[\alpha]_D^{24.5} - 23.4^\circ$  ( $c = 1.22$ , CHCl<sub>3</sub>). IR (film): 3350 (NH), 1650 (CO) cm<sup>-1</sup>. <sup>1</sup>H-NMR (CDCl<sub>3</sub>)  $\delta$ : 0.01 (s, 6H, SiCH<sub>3</sub>), 0.80 (s, 9H, *tert*-Bu), 1.60–2.10 (m, 2H, C<sub>4</sub>-H), 1.95, 1.98 (each s, 3H, CH<sub>3</sub>), 3.10–3.80 (m, 4H, C<sub>2</sub>-H, C<sub>5</sub>-H), 4.25–4.58 (m, 1H, C<sub>3</sub>-H). MS  $m/z$ : 228 (M<sup>+</sup> – CH<sub>3</sub>). Anal. Calcd for C<sub>12</sub>H<sub>25</sub>NO<sub>2</sub>Si: C, 59.21; H, 10.35; N, 5.75; Si, 11.54. Found: C, 58.85; H, 10.38; N, 5.80; Si, 11.18.

**N-Benzoyloxycarbonyl-L-phenylalanine N-(2(R)-Methylbutyl)amide (3f)** Both method A and method C were applied to obtain this compound. mp 133–134°C (MeOH),  $[\alpha]_D^{25.5} + 5.49^\circ$  ( $c = 1.02$ , CHCl<sub>3</sub>). IR (Nujol): 3300 (NH), 1690 (CO), 1655 (CO), 1530 cm<sup>-1</sup>. <sup>1</sup>H-NMR (CDCl<sub>3</sub>)  $\delta$ : 0.72 (d, 3H,  $J = 6.7$  Hz, CHCH<sub>3</sub>), 0.81 (3H, t,  $J = 7.3$  Hz, CH<sub>2</sub>CH<sub>3</sub>), 0.85–1.50 (m, 3H, CHCH<sub>2</sub>CH<sub>3</sub>), 2.85–3.18 (m, 4H, CH<sub>2</sub>Ph, NHCH<sub>2</sub>), 4.29–4.41 (m, 1H, NHCHCO), 5.08 (s, 2H, PhCH<sub>2</sub>O), 5.45 (br, 1H, NH), 5.70 (br, 1H, NH), 7.17–7.37 (m, 10H, Ar-H). MS  $m/z$ : 366 (M<sup>+</sup> – H<sub>2</sub>). Anal. Calcd for C<sub>22</sub>H<sub>28</sub>N<sub>2</sub>O<sub>3</sub>: C, 71.71; H, 7.66; N, 7.60. Found: C, 71.46; H, 7.75; N, 7.48.

**Conversion of 3c to 2(R)-(-)-Hydroxypropylamine Hydrochloride** Compound **3c** (835 mg, 2.68 mmol) was dissolved in MeOH (30 ml) and the solution was subjected to hydrogenolysis over 10% Pd-C (0.1 g) at atmospheric pressure. After a theoretical amount of hydrogen had been absorbed, the catalyst was filtered off and the filtrate was evaporated to dryness *in vacuo*. The resulting syrup was dissolved in 22% HCl in MeOH (1 ml) and the solvent was removed under reduced pressure. The resulting

crystals were triturated with ether to give colorless needles (190 mg, 63%), mp 111–113°C (lit.<sup>6</sup>) 113°C),  $[\alpha]_D^{25.5} - 32.5^\circ$  ( $c = 2.74$ , H<sub>2</sub>O) (lit.<sup>6</sup>)  $[\alpha]_D^{20} - 32.4^\circ$  ( $c = 1.46$ , H<sub>2</sub>O)). IR (film): 3350 (NH), 2850, 1960, 1600 cm<sup>-1</sup>. <sup>1</sup>H-NMR (D<sub>2</sub>O)  $\delta$ : 1.24 (d, 3H,  $J = 6.4$  Hz, CH<sub>3</sub>), 2.88 (dd, 1H,  $J = 9.0$ , 13.1 Hz, C<sub>1</sub>-H), 3.10 (dd, 1H,  $J = 3.4$ , 13.1 Hz, C<sub>1</sub>-H), 3.95–4.12 (m, 1H, C<sub>2</sub>-H). MS  $m/z$ : 76 (M<sup>+</sup>).

**Conversion of 3e to (3R)-(-)-Hydroxypyrrolidine Hydrochloride** A mixture of compound **3e** (1.26 g, 5.18 mmol) and 6 N HCl (5 ml) was refluxed for 6 h. After cooling, the reaction mixture was diluted with H<sub>2</sub>O (5 ml), and the solution was washed with EtOAc. The aqueous layer was concentrated to dryness *in vacuo*. The crystalline residue was triturated with ether to give pale brown needles (550 mg, 85%). An analytical sample was prepared by recrystallization from EtOH–ether (1:1), mp 107–108°C (lit.<sup>6</sup>) 109°C),  $[\alpha]_D^{26} - 9.20^\circ$  ( $c = 1.74$ , MeOH) (lit.<sup>6</sup>)  $[\alpha]_D^{20} - 7.60^\circ$  ( $c = 3.45$ , MeOH)). IR (Nujol): 3400 (NH), 1620, 1450 cm<sup>-1</sup>. <sup>1</sup>H-NMR (D<sub>2</sub>O)  $\delta$ : 2.02–2.25 (m, 2H, C<sub>4</sub>-H), 3.26–3.55 (m, 4H, C<sub>2</sub>-H, C<sub>5</sub>-H), 4.61–4.68 (m, 1H, C<sub>3</sub>-H). MS  $m/z$ : 87 (M<sup>+</sup>).

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