Supporting Information

for

Synthesis of conformationally restricted glutamate and glutamine derivatives from carbonylation of orthopalladated phenylglycine derivatives

Esteban P. Urriolabeitia*, Eduardo Laga, and Carlos Cativiela

Address: Instituto de Síntesis Química y Catálisis Homogénea, CSIC - Universidad de Zaragoza, Pedro Cerbuna 12, 50009 Zaragoza, Spain

Email: Esteban P. Urriolabeitia - esteban@unizar.es

* Corresponding author

General methods and experimental and analytical data of compounds 2b–j
**General Methods.** Elemental analyses were carried out on a Perkin-Elmer 2400-B microanalyzer. Infrared spectra (4000–200 cm\(^{-1}\)) were recorded on a Perkin-Elmer Spectrum One from nujol mulls between polyethylene sheets. The \(^1\)H, \(^{13}\)C\(^{1\text{H}}\) and \(^{19}\)F NMR spectra were recorded in CD\(_2\)Cl\(_2\), CDCl\(_3\) or acetone-\(d_6\) solutions at 25 °C on Bruker Avance-300 and Avance-400 spectrometers (\(\delta\), ppm; \(J\), Hz); \(^1\)H and \(^{13}\)C\(^{1\text{H}}\) spectra were referenced by using the solvent signal as an internal standard, while \(^{19}\)F NMR spectra were referenced to CFCl\(_3\). Electrospray Ionization (ESI)/Atmospheric Pressure Chemical Ionization (APCI) mass spectra were recorded on an Esquire 3000 ion-trap mass spectrometer (Bruker Daltonic GmbH, Bremen, Germany) equipped with a standard ESI/APCI source. Samples were introduced by direct infusion with a syringe pump. Nitrogen served both as the nebulizer gas and the dry gas. Helium served as a cooling gas for the ion trap and collision gas for MSn experiments. Other MS (MALDI-DIT) were recorded from CH\(_2\)Cl\(_2\) solutions on a Bruker MicroFlex spectrometer.

**Synthesis of methyl \(N,N\)-dimethyl-\(\alpha\)-(2-methoxycarbonylphenyl)glycinate (2a)**

Methanol (13 \(\mu\)L, 0.300 mmol) was added to a solution of 1 (100.0 mg, 0.150 mmol) in CH\(_2\)Cl\(_2\) (10 mL), and the resulting mixture was stirred under a CO atmosphere for 16 h. Decomposition to black metallic palladium was observed. The mixture was filtered through a plug of Celite. The light yellow solution was washed with water (3 × 20 mL), dried over MgSO\(_4\), filtered and evaporated to give compound 2a as a yellow oil. Yield: 72.9 mg, 0.290 mmol, 97%.

\(^1\)H NMR (300 MHz, CDCl\(_3\)) \(\delta\) 7.83 (d, \(J = 7.7\) Hz, 1H, C\(_6\)H\(_4\)), 7.68 (d, \(J = 7.7\) Hz, 1H, C\(_6\)H\(_4\)), 7.50 (t, \(J = 7.7\) Hz, 1H, C\(_6\)H\(_4\)), 7.35 (t, \(J = 7.7\) Hz, 1H, C\(_6\)H\(_4\)), 5.12 (s, 1H, CH), 3.89 (s, 3H, OMe), 3.69 (s, 3H, OMe), 2.31 (s, 6H, NMe\(_2\)) ; \(^{13}\)C NMR (75 MHz, CDCl\(_3\)) \(\delta\) 172.08 (s, CO), 168.36 (s, CO), 137.60 (s, C), 131.96 (s, CH), 130.99 (s, C),
130.32 (s, CH), 129.03 (s, CH), 127.88 (s, CH), 68.42 (s, CH), 52.34 (s, OCH₃),
51.88 (s, OCH₃), 42.97 (s, NMe₂); IR (ν, cm⁻¹): 1724 (C=O), 1257 (C-O); ESIMS
(positive mode) (m/z): 251.9 [M + H]⁺; anal. Calcd for C₁₃H₁₇NO₄ (251.12): C, 62.14;
H, 6.82; N, 5.57; found: C, 62.35; H, 6.91; N, 5.36.

Synthesis of methyl N,N-dimethyl-α-(2-ethoxycarbonylphenyl)glycinate (2b)
Compound 2b was prepared following the same experimental procedure to that
described for 2a. Therefore, ethanol (18 μL, 0.300 mmol) was reacted with 1
(100.0 mg, 0.150 mmol) in CH₂Cl₂ (10 mL) under a CO atmosphere for 16 h to give
compound 2b as a yellow oil. Yield: 75.9 mg, 0.286 mmol, 95%.
¹H NMR (300 MHz, CDCl₃) δ 7.82 (dd, J = 7.8, 1.4 Hz, 1H, C₆H₄), 7.68 (dd, J = 8.0,
1.4 Hz, 1H, C₆H₄), 7.49 (td, J = 7.6, 1.5 Hz, 1H, C₆H₄), 7.35 (td, J = 7.5, 1.3 Hz, 1H,
C₆H₄), 5.12 (s, 1H, CH), 4.37 (q, J = 7.1 Hz, 2H, OCH₂), 3.69 (s, 3H, OCH₃), 2.31 (s,
6H, NMe₂), 1.40 (t, J = 7.1 Hz, 3H, CH₃); ¹³C NMR (75 MHz, CDCl₃) δ 172.12 (s,
CO), 168.03 (s, CO), 137.45 (s, C), 131.75 (s, CH), 131.63 (s, C), 130.18 (s, CH),
129.04 (s, CH), 127.86 (s, CH), 68.51 (s, CH), 61.33 (s, OCH₂), 51.83 (s, OCH₃),
43.00 (s, NMe₂), 14.41 (s, CH₃); IR (ν, cm⁻¹): 1739 (C=O), 1713 (C=O), 1257 (C-O);
ESIMS (positive mode) (m/z): 266.1 [M + H]⁺; anal. Calcd for C₁₄H₁₉NO₄ (265.13): C,
63.38; H, 7.22; N, 5.28; found: C, 63.24; H, 7.14; N, 5.36.

Synthesis of methyl N,N-dimethyl-α-(2-isopropoxycarbonylphenyl)glycinate (2c)
Compound 2c was prepared following the same experimental procedure to that
described for 2a. Therefore, 2-propanol (23 μL, 0.300 mmol) was reacted with 1
(100.0 mg, 0.150 mmol) in CH₂Cl₂ (10 mL) under a CO atmosphere for 16 h to give
compound 2c as a yellow oil. Yield: 31.6 mg, 0.113 mmol, 38%.
\(^1\)H NMR (300 MHz, CDCl\(_3\)) \(\delta 7.76 (dd, J = 7.7, 1.6 \text{ Hz}, 1\text{H}, C_6\text{H}_4), 7.67 (dd, J = 7.9, 1.3 \text{ Hz}, 1\text{H}, C_6\text{H}_4), 7.47 (td, J = 7.6, 1.5 \text{ Hz}, 1\text{H}, C_6\text{H}_4), 7.33 (td, J = 7.6, 1.4 \text{ Hz}, 1\text{H}, C_6\text{H}_4), 5.24 (hept, J = 6.3 \text{ Hz}, 1\text{H}, OCH), 5.09 (s, 1\text{H}, CH), 3.68 (s, 3\text{H}, OCH\(_3\)), 2.29 (s, 6\text{H}, NMe\(_2\)), 1.38 (d, J = 6.3 \text{ Hz}, 3\text{H}, CH\(_3\)); \(^13\)C NMR (75 MHz, CDCl\(_3\)) \(\delta 172.15 (s, \text{CO}), 167.58 (s, \text{CO}), 137.15 (s, \text{C}), 132.16 (s, \text{C}), 131.56 (s, \text{CH}), 129.96 (s, \text{CH}), 128.98 (s, \text{CH}), 127.82 (s, \text{CH}), 68.87 (s, \text{CH}), 51.82 (s, OCH\(_3\)), 43.05 (s, NMe\(_2\)), 22.0 (s, CH\(_3\)); IR (\(\nu, \text{cm}^{-1}\)): 1735 (C=O), 1713 (C=O), 1258 (C-O); ESIMS (positive mode) (m/z): 280.1 [M + H]\(^+\); anal. Calcd for C\(_{15}\)H\(_{21}\)NO\(_4\) (279.15): C, 64.50; H, 7.58; N, 5.01; found: C, 64.61; H, 7.55; N, 4.97.

**Synthesis of methyl N,N-dimethyl-\(\alpha\)-[2-(2-hydroxy)ethoxycarbonylphenyl] glycinate (2d)**

Compound 2d was prepared following the same experimental procedure to that described for 2a. Therefore, 1,2-ethanediol (17 \(\mu\)L, 0.300 mmol) was reacted with 1 (100.0 mg, 0.150 mmol) in CH\(_2\)Cl\(_2\) (10 mL) under a CO atmosphere for 16 h to give compound 2d as a yellow oil. Yield: 53.7 mg, 0.191 mmol, 91%.

\(^1\)H NMR (300 MHz, CDCl\(_3\)) \(\delta 7.80 (dd, J = 7.8, 1.4 \text{ Hz}, 1\text{H}, C_6\text{H}_4), 7.71 (dd, J = 7.9, 1.3 \text{ Hz}, 1\text{H}, C_6\text{H}_4), 7.50 (td, J = 7.6, 1.5 \text{ Hz}, 1\text{H}, C_6\text{H}_4), 7.37 (td, J = 7.5, 1.3 \text{ Hz}, 1\text{H}, C_6\text{H}_4), 4.91 (s, 1\text{H}, CH), 4.43 (m, 2\text{H}, OCH\(_2\)), 3.92 (m, 2\text{H}, OCH\(_2\)), 3.70 (s, 3\text{H}, OCH\(_3\)), 3.17 (s br, 1\text{H}, OH), 2.29 (s, 6\text{H}, NMe\(_2\)); \(^13\)C NMR (75 MHz, CDCl\(_3\)) \(\delta 171.99 (s, \text{CO}), 168.27 (s, \text{CO}), 136.44 (s, \text{C}), 131.94 (s, \text{CH}), 131.52 (s, \text{C}), 130.27 (s, \text{CH}), 129.51 (s, \text{CH}), 128.19 (s, \text{CH}), 69.93 (s, \text{CH}), 67.31 (s, OCH\(_2\)), 60.89 (s, OCH\(_2\)), 52.19 (s, OCH\(_3\)), 43.21 (s, NMe\(_2\)); IR (\(\nu, \text{cm}^{-1}\)): 3424 (O-H), 3244 (O-H), 1716 (br, C=O), 1252 (C-O); ESIMS (positive mode) (m/z): 282.1 [M + H]\(^+\); anal. Calcd for C\(_{14}\)H\(_{19}\)NO\(_5\) (281.13): C, 59.78; H, 6.81; N, 4.98; found: C, 58.57; H, 6.71; N, 4.99.
Synthesis of ethane-1,2-diy1 bis(2-(1-(dimethylamino)-2-methoxy-2-oxoethyl)benzoate) (2e)

Compound 2e was prepared following the same experimental procedure to that described for 2a. Therefore, 1,2-ethanediol (8 μL, 0.150 mmol) was reacted with 1 (100.0 mg, 0.150 mmol) in CH₂Cl₂ (10 mL) under a CO atmosphere for 16 h to give compound 2e as a yellow oil. Yield: 20.1 mg, 0.040 mmol, 27%.

¹H NMR (300 MHz, CDCl₃) δ 7.86 (d, J = 7.5 Hz, 1H, C₆H₄), 7.68 (d, J = 7.9 Hz, 1H, C₆H₄), 7.51 (d, J = 7.5 Hz, 1H, C₆H₄), 7.34 (td, J = 7.5, 1H, C₆H₄), 5.13 (s, 1H, CH), 4.64 (s, 2H, OCH₂), 3.67 (s, 3H, OCH₃), 2.28 (s, 6H, NMe₂); ¹³C NMR (75 MHz, CDCl₃) δ 171.96 (s, CO), 167.63 (s, CO), 137.92 (s, C), 132.14 (s, CH), 130.73 (s, C), 130.43 (s, CH), 129.14 (s, CH), 127.93 (s, CH), 68.44 (s, CH), 63.00 (s, OCH₂), 51.82 (s, OCH₃), 42.89 (s, NMe₂); IR (ν, cm⁻¹): 1723 (br, C=O), 1256 (C-O); ESIMS (positive mode) (m/z): 501.2 [M + H]⁺; anal. Calcd for C₂₆H₃₂N₂O₈ (500.22): C, 62.39; H, 6.44; N, 5.60; found: C, 62.24; H, 6.52; N, 5.64.

Synthesis of methyl N,N-dimethyl-α-(2-phenoxy carbonylphenyl)glycinate (2f)

Compound 2f was prepared following the same experimental procedure to that described for 2a. Therefore, phenol (28.2 mg, 0.300 mmol) was reacted with 1 (100.0 mg, 0.150 mmol) in CH₂Cl₂ (10 mL) under a CO atmosphere for 16 h to give compound 2f as a yellow oil. Yield: 40.3 mg, 0.129 mmol, 43%.

¹H NMR (300 MHz, CDCl₃) δ 8.11 (d, J = 7.7 Hz, 1H, C₆H₄), 7.79 (d, J = 7.9 Hz, 1H, C₆H₄), 7.62 (t, J = 7.5 Hz, 1H, C₆H₄), 7.49 (t, J = 7.9 Hz, 2H, C₆H₅), 7.34 (t, J = 7.5 Hz, 1H, C₆H₄), 7.27 (m, 2H, C₆H₅), 6.87 (m, 1H, C₆H₅), 5.31 (s, 1H, CH), 3.76 (s, 3H, OCH₃), 2.41 (s, 6H, NMe₂); ¹³C NMR (75 MHz, CDCl₃) δ 172.03 (s, CO), 166.47 (s, CO), 150.91 (s, C), 138.20 (s, C), 132.44 (s, CH), 130.67 (s, CH), 130.54 (s, C), 129.65 (s, CH), 129.21 (s, CH), 128.07 (s, CH), 126.12 (s, CH), 121.79 (s, CH), 121.79 (s, CH),
68.32 (s, CH), 51.88 (s, OCH₃), 42.88 (s, NMe₂); IR (ν, cm⁻¹): 1735 (br, C=O), 1257 (C-O); ESIMS (positive mode) (m/z) 314.2 [M + H]⁺; anal. Calcd for C₁₉H₁₉NO₄ (313.13): C, 68.99; H, 6.11; N, 4.47; found: C, 69.02; H, 6.03; N, 4.51.

**Synthesis of methyl N,N-dimethyl-α-(2-benzylaminocarbonylphenyl)glycinate (2g)**

Benzyamine (33 μL, 0.300 mmol) was added to a solution of 1 (100.0 mg, 0.150 mmol) in CH₂Cl₂ (10 mL), and the resulting mixture was stirred under a CO atmosphere for 16 h. Decomposition to black metallic palladium was observed. The mixture was filtered through a plug of Celite. The light yellow solution was washed with water (3 × 20 mL), dried over MgSO₄, filtered and evaporated to give compound 2g as a yellow oil. Yield: 57.4 mg, 0.176 mmol, 59%.

¹H NMR (300 MHz, CDCl₃) δ 9.35 (s br, 1H, NH), 7.90 (m, 1H, C₆H₄), 7.44–7.25 (m, 8H, C₆H₄+C₆H₅), 4.71 (dd, J = 14.5, 5.7 Hz, 1H, CH₂), 4.57 (s, 1H, CH), 4.52 (dd, J = 14.5, 4.8 Hz, 1H, CH₂), 3.67 (s, 3H, OCH₃), 2.17 (s, 6H, NMe₂); ¹³C NMR (75 MHz, CDCl₃) δ 171.18 (s, CO), 168.16 (s, CO), 138.58 (s, C), 137.58 (s, C), 132.65 (s, C), 130.83 (s, CH), 130.20 (s, CH), 128.84 (s, 2CH, overlaped signals), 128.79 (s, CH), 128.31 (s, CH), 127.61 (s, CH), 68.07 (s, CH), 51.72 (s, OCH₃), 44.62 (s, NCH₂), 40.31 (s, NMe₂); IR (ν, cm⁻¹): 3285 (br, N-H), 1735 (COO), 1643 (CON); ESIMS (positive mode) (m/z): 327.2 [M + H]⁺; anal. Calcd for C₁₉H₂₂N₂O₃ (326.16): C, 69.92; H, 6.79; N, 8.58; found: C, 69.85; H, 6.68; N, 8.54.

**Synthesis of methyl N,N-dimethyl-α-(2-anilinocarbonylphenyl)glycinate (2h)**

Compound 2h was prepared following the same experimental procedure to that described for 2g. Therefore, aniline (27 μL, 0.300 mmol) was reacted with 1 (100.0 mg, 0.150 mmol) in CH₂Cl₂ (10 mL) under a CO atmosphere for 16 h to give compound 2h as a yellow oil. Yield: 76.0 mg, 0.243 mmol, 81%.
$^1$H NMR (300 MHz, CDCl$_3$) $\delta$ 11.22 (s br, 1H, NH), 7.97 (m, 1H, C$_6$H$_4$), 7.73–7.66 (m, 2H, C$_6$H$_5$), 7.51–7.41 (m, 2H, C$_6$H$_4$), 7.40–7.32 (m, 2H, C$_6$H$_5$), 7.30 (m, 1H, C$_6$H$_4$), 7.12 (m, 1H, C$_6$H$_5$), 4.77 (s, 1H, CH), 3.71 (s, 3H, OCH$_3$), 2.54 (s, 6H, NMe$_2$);

$^{13}$C NMR (75 MHz, CDCl$_3$) $\delta$ 170.91 (s, CO), 166.43 (s, CO), 139.11 (s, C), 138.32 (s, C), 132.09 (s, C), 131.51 (s, CH), 130.54 (s, CH), 129.17 (s, CH), 129.13 (s, CH), 128.92 (s, CH), 128.18 (s, CH), 119.99 (s, CH), 68.25 (s, CH), 51.86 (s, OCH$_3$), 40.94 (s, NMe$_2$).

IR (ʋ, cm$^{-1}$): 3286 (br, N-H), 1736 (COO), 1667 (CON); ESIMS (positive mode) (m/z): 313.2 [M + H]$^+$; anal. Calcd for C$_{18}$H$_{20}$N$_2$O$_3$ (312.15): C, 69.21; H, 6.45; N, 8.97; found: C, 69.35; H, 6.69; N, 9.04.

**Synthesis of methyl N,N-dimethyl-α-[2-(α-methoxycarbonyl)benzylaminocarbonylphenyl]glycinate (2i)**

Compound 2i was prepared following the same experimental procedure to that described for 2g. Therefore, (R)-phenylglycine methyl ester (49.5 mg, 0.300 mmol) was reacted with 1 (100.0 mg, 0.150 mmol) in CH$_2$Cl$_2$ (10 mL) under a CO atmosphere for 16 h to give compound 2i as a yellow oil. Yield: 87.0 mg, 0.226 mmol, 76%. Compound 2i was characterized as a mixture of diastereoisomers (1:0.25) by NMR methods.

**NMR data. Major isomer.** $^1$H NMR (300 MHz, CDCl$_3$) $\delta$ 10.16 (d, $J$ = 7.4 Hz, NH), 7.87 (m, C$_6$H$_4$), 7.48–7.22 (m, C$_6$H$_4$+C$_6$H$_5$), 5.84 (d, $J$ = 7.4 Hz, NHCH), 4.96 (s, CH), 3.79 (s, OCH$_3$), 3.67 (s, OCH$_3$), 2.32 (s, 6H, NMe$_2$); $^{13}$C($^1$H) NMR (75 MHz, CDCl$_3$) $\delta$ 172.04 (s, CO), 171.19 (s, CO), 168.17 (s, CO), 137.16 (s, C), 136.11 (s, C), 132.84 (s, C), 130.94 (s, CH), 130.45 (s, CH), 129.02 (s, CH), 128.92 (s, CH), 128.80 (s, CH), 128.60 (s, CH), 127.63 (s, CH), 67.63 (s, CH), 57.59 (s, NHCH), 52.72 (s, OCH$_3$), 51.68 (s, OCH$_3$), 40.53 (s, NMe$_2$). **Minor isomer.** $^1$H NMR (300 MHz, CDCl$_3$) $\delta$ 10.00 (d, $J$ = 6.2 Hz, NH), 7.88 (m, C$_6$H$_4$), 7.48–7.22 (m, 8H, C$_6$H$_4$+C$_6$H$_5$), 5.69 (d, $J$
\(13C\{^1H\}\) NMR (75 MHz, CDCl\(_3\)) \(\delta\) 172.04 (s, CO), 171.33 (s, CO), 167.93 (s, CO), 136.89 (s, C), 136.55 (s, C), 132.77 (s, C), 131.19 (s, CH), 130.50 (s, CH), 129.20 (s, CH), 128.87 (s, CH), 128.85 (s, CH), 128.64 (s, CH), 127.93 (s, CH), 67.90 (s, CH), 58.05 (s, NHCH), 52.67 (s, OCH\(_3\)), 51.68 (s, OCH\(_3\)), 40.13 (s, NMe\(_2\)).

IR (\(\nu\), cm\(^{-1}\)): 3407 (br, N-H), 1738 (COO), 1651 (CON).

ESIMS (positive mode) \((m/z)\) 385.0 [M + H\(^+\)]; anal. Calcd for C\(_{21}\)H\(_{24}\)N\(_2\)O\(_5\) (384.17): C, 65.61; H, 6.29; N, 7.29; found: C, 65.74; H, 6.32; N, 7.26.

**Synthesis of methyl N,N-dimethyl-\(\alpha\)-(2-N,N-dibutylaminocarbonylphenyl)glycinate (2j)**

Compound 2i was prepared following the same experimental procedure to that described for 2g. Therefore, H\(^9\)Bu\(_2\) (50.4 \(\mu\)L, 0.300 mmol) was reacted with 1 (100.0 mg, 0.150 mmol) in CH\(_2\)Cl\(_2\) (10 mL) under a CO atmosphere for 16 h to give compound 2j as a yellow oil. Yield: 37 mg, 0.106 mmol, 35%. Compound 2j was characterized by NMR methods as a mixture of rotamers in 3:1 molar ratio.

**NMR data. Major isomer:** \(^1H\) NMR (300 MHz, CDCl\(_3\), 258 K) \(\delta\) 7.70 (dd, \(J = 7.7, 1.5\) Hz, C\(_6\)H\(_4\)), 7.37 (td, \(J = 7.6, 1.7\) Hz, C\(_6\)H\(_4\)), 7.31 (td, \(J = 7.5, 1.5\) Hz, C\(_6\)H\(_4\)), 7.16 (dd, \(J = 7.5, 1.5\) Hz, C\(_6\)H\(_4\)), 3.86 (s, CH), 3.81 (m, NCH\(_2\)), 3.66 (s, OCH\(_3\)), 3.16 (m, NCH\(_2\)), 2.94 (m, NCH\(_2\)), 2.20 (s, NMe\(_2\)), 1.70–1.59 (m, NBU), 1.59–1.47 (m, NBU), 1.46–1.30 (m, NBU), 1.18–0.97 (m, NBU), 0.94 (m, CH\(_3\)), 0.75 (m, CH\(_3\)); \(13C\{^1H\}\) NMR (75 MHz, CDCl\(_3\)) \(\delta\) 172.30 (s, CO), 170.15 (s, CO), 138.11 (s, C), 133.27 (s, C), 129.20 (s, CH), 128.62 (s, CH), 128.23 (s, CH), 125.97 (s, CH), 71.11 (s, CH), 52.12 (s, OCH\(_3\)), 48.71 (s, NCH\(_2\)), 44.26 (s, NCH\(_2\)), 43.81 (s, NMe\(_2\)), 30.59 (s, CH\(_2\)), 29.47 (s, CH\(_2\)), 20.50 (s, CH\(_2\)Me), 20.06 (s, CH\(_2\)Me), 14.04 (s, CH\(_3\)), 13.73 (s, CH\(_3\)).

**Minor isomer:** \(^1H\) NMR (300 MHz, CDCl\(_3\), 258 K) \(\delta\) 7.50 (dd, \(J = 7.3, 1.8\) Hz, C\(_6\)H\(_4\)), 7.43–
7.25 (m, C₆H₄), 7.17 (m, C₆H₄), 4.32 (s, CH), 3.80 (m, NCH₂), 3.66 (s, OCH₃), 3.15 (m, NCH₂), 3.03–2.86 (m, NCH₂), 2.23 (s, NMe₂), 1.72–1.29 (m, NBu), 1.18–0.97 (m, NBu), 0.93 (m, CH₃), 0.76 (m, CH₃); IR (ν, cm⁻¹): 1735 (COO), 1623 (CON); ESIMS (positive mode) (m/z): 349.0 [M + H]⁺; anal. Calcd for C₂₀H₃₂N₂O₃ (348.24): C, 68.93; H, 9.26; N, 8.04; found: C, 69.06; H, 9.35; N, 8.10.