ISSN 1070-3632, Russian Journal of General Chemistry, 2014, Vol. 84, No. 9, pp. 1701–1705. © Pleiades Publishing, Ltd., 2014.
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Synthesis of Lithium, Sodium, Cesium, and Calcium Salts of (*E*)-4- or (*E*,*S*)-3-Methyl-2-(5-arylisoxazol-3-yl)-methyleneaminobutanoic, (*E*,*S*)-4-Methyl-2- or (*E*,2*S*,3*S*)-3-Methyl-2-(5-arylisoxazol-3-yl)-methyleneaminopentanoic, and (*E*)-6-(5-Arylisoxazol-3-yl)methyleneaminohexanoic Acids

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Received April 24, 2014

Abstract—Preparative method for the synthesis of lithium, sodium, cesium, and calcium salts of (*E*)-4-(5-arylisoxazol-3-yl)methyleneaminobutanoic, (*E*)-6-(5-arylisoxazol-3-yl)methyleneaminohexanoic, (*E*,*S*)-3-methyl-2-(5-arylisoxazol-3-yl)methyleneaminobutanoic, (*E*,*S*)-4-methyl-2-(5-arylisoxazol-3-yl)methyleneaminopentanoic and (*E*,2*S*,3*S*)-3-methyl-2-(5-arylisoxazol-3-yl)methyleneaminopentanoic acids was developed by reacting 5-phenyl(4-tolyl)isoxazole-3-carbaldehydes with amino acids like 4-aminobutyric and 6-aminocaproic acids, L-valine, L-leucine or L-isoleucine in the presence of lithium hydride, sodium methoxide, cesium carbonate or calcium hydride in boiling methanol.

Keywords: salts of 5-arylisoxazol-3-ylmethyleneaminocarboxylic acids, azomethines, synthesis, heterocyclic compounds, amino acids

DOI: 10.1134/S1070363214090102

Derivatives of natural amino acids have high biological activity, which stimulates developing convenient methods of their preparation [1–4]. Synthesis of water-soluble derivatives containing heterocyclic fragments promising for drug designing is of special interest [5–7].

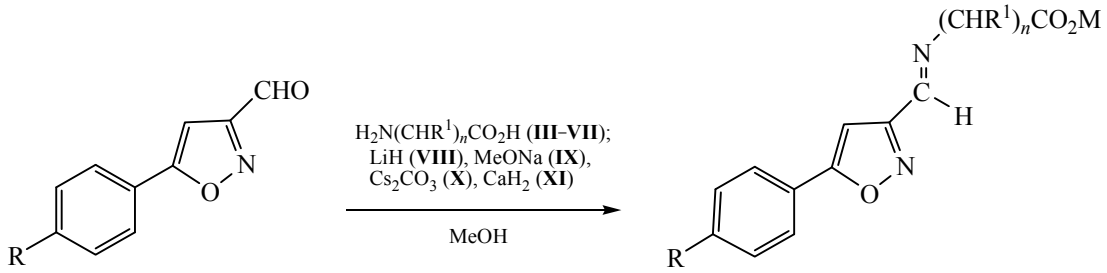
In this work we developed a convenient preparative method for the synthesis of lithium, sodium, cesium, and calcium salts of (*E*)-4-(5-arylisoxazol-3-yl)methyleneaminobutanoic, (*E*)-6-(5-arylisoxazol-3-yl)methyleneaminohexanoic, (*E*,*S*)-3-methyl-2-(5-arylisoxazol-3-yl)methyleneaminobutanoic, (*E*,*S*)-4-methyl-2-(5-arylisoxazol-3-yl)methyleneaminopentanoic, and (*E*,2*S*,3*S*)-3-methyl-2-(5-arylisoxazol-3-yl)methyleneaminopentanoic acids (**XII**–**XLIX**) by reacting 5-(phenyl or 4-tolyl)isoxazole-3-carbaldehyde **I** and **II** with amino acids like as 4-aminobutyric **III** or 6-aminocaproic acids **IV**, L-valine **V**, L-leucine **VI**, or L-isoleucine **VII** in the presence of lithium hydride **VIII**, sodium methoxide **IX**, cesium carbonate **X**, or calcium hydride **XI** in boiling methanol.

After removal of methanol in a vacuum lithium, sodium, cesium, and calcium salts of (*E*)-isoxazolylazomethinecarboxylic acids **XII**–**XLIX** were obtained in quantitative yields (~100%) and with a purity of >98%.

The synthesized salts of (*E*)-isoxazolylazomethinecarboxylic acids **XII**–**XLIX** were colorless crystalline substances soluble in methanol and ethanol (Table 1). In addition, sodium and cesium salts were readily soluble in water. Lithium, sodium, and calcium salts were not hygroscopic, and cesium salts deliquesced by contact with the air moisture, but they can easily be dried by azeotropic distillation with benzene (Scheme 1).

By the example of sodium salt **XXXII** we selected optimal conditions for reduction of imine group to

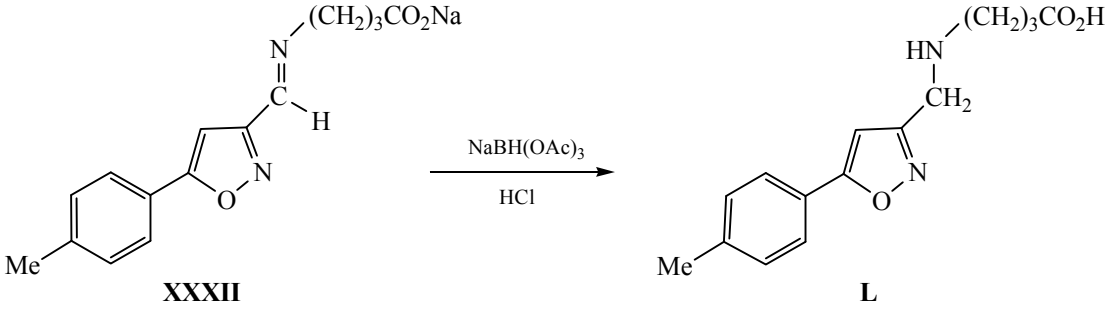
Scheme 1.



I, II **XII–XLIX**

R = H (**I**, **XII–XXX**), Me (**II**, **XXXI–XLIX**); R^1 = H, *n* = 3 (**III**, **XII–XV**, **XXXI–XXXIV**), *n* = 5 (**IV**, **XVI–XIX**, **XXXV–XXXVIII**); *n* = 1, R^1 = $CHMe_2$ (**V**, **XX–XXII**, **XXXIX–XLI**), CH_2CHMe_2 (**VI**, **XXIII–XXVI**, **XLII–XLV**), CHMeEt (**VII**, **XXVII–XXX**, **XLVI–XLIX**); M = Li (**XII**, **XVI**, **XX**, **XXIII**, **XXVII**, **XXXI**, **XXXV**, **XXXIX**, **XLII**, **XLVI**), Na (**XIII**, **XVII**, **XXI**, **XXIV**, **XXVIII**, **XXXII**, **XXXVI**, **XL**, **XLIII**, **XLVII**); Cs (**XIV**, **XVIII**, **XXV**, **XXIX**, **XXXIII**, **XXXVII**, **XLIV**, **XLVIII**); Ca (**XV**, **XIX**, **XXII**, **XXVI**, **XXX**, **XXXIV**, **XXXVIII**, **XLI**, **XLV**, **XLIX**).

Scheme 2.



XXXII **L**

amine with sodium triacetoxyborohydride in anhydrous benzene preventing hydrolysis of the starting azomethine and allowing obtaining the target amine with yield of 65%. The formed sodium salt was converted to 4-[5-(4-tolyl)isoxazol-3-ylmethyl]aminobutanoic acid **L** by treating the formed salt with aqueous HCl (Scheme 2).

The composition and structure of salts of (*E*)-isoxazolylazomethinecarboxylic acids **XII–XLIX** were confirmed by elemental analysis data, IR and ^{1}H NMR spectroscopy. Melting points and elemental analysis data of salts **XII–XLIX** are given in Table 1.

(*E*)-Configuration of **XII–XLIX** was assigned by analogy with related vanillin-derived (*E*)-azomethines [5–7] and according to ^{1}H NMR spectral data [8]. The salts of (*E*)-isoxazolylazomethinecarboxylic acids derived from L-valine **V**, L-leucine **VI**, and L-isoleucine **VII** are chiral compounds and did not undergo racemization during their preparation [5–7].

IR spectra of the salts of (*E*)-isoxazolylazomethinecarboxylic acids **XII–XLIX** contained the following characteristic absorption bands (ν, cm^{-1}): 3100–3005, 860–640 (CH_{Ar}), 2980–2830 (CH_{aliph}), 1610–1580, 1415–1405 (CO_2^-), 1648–1634 (C=N), 1605–1595, 1515–1505 ($C=C_{Ar}$), 1260–1240, 1175–1160, 1030–1015 (C–O), and there was no absorption band at 1700–1680 cm^{-1} ($C=O_{aldehyde}$), characteristic of the original benzaldehydes **I** and **II**.

The IR, ^{1}H and ^{13}C NMR spectra of compounds **XIV**, **XVII**, **XX**, **XXIV**, **XXXII**, **XXXVI**, **XXXVII**, **XL**, **XLI**, **L** contained the signals of all the structural fragments (Table 2).

EXPERIMENTAL

IR spectra were recorded on a Nicolet Protégé-460 FTIR spectrophotometer from KBr pellets. NMR spectra were registered on a Bruker Avance-500 spectrometer relative to the signals of residual protons of the deuterated solvents.

Salts of (*E*)-isoxazolylazomethinecarboxylic acids (XII–XLIX). A mixture of 0.01 mol of aldehyde **I** or

Table 1. Melting points and elemental analysis data of compounds **XII–L**

Comp. no.	mp, °C	Found, %				Formula	Calculated, %				M_{calc}
		C	H	M	N		C	H	M	N	
XII	233–234	63.88	5.10	2.16	10.14	$C_{14}H_{13}LiN_2O_3$	63.64	4.96	2.63	10.60	264.21
XIII	183–184	60.49	4.79	7.65	9.67	$C_{14}H_{13}N_2NaO_3$	60.00	4.68	8.20	10.00	280.25
XIV	62–63	43.30	3.22	34.44	7.09	$C_{14}H_{13}CsN_2O_3$	43.10	3.36	34.06	7.18	390.17
XV	77–78	61.05	4.93	6.99	9.80	$C_{28}H_{26}CaN_4O_6$	60.64	4.73	7.23	10.10	554.61
XVI	204–205	66.23	6.04	1.95	8.97	$C_{16}H_{17}LiN_2O_3$	65.75	5.86	2.37	9.59	292.26
XVII	185–187	62.20	5.35	7.63	9.11	$C_{16}H_{17}N_2NaO_3$	62.33	5.56	7.46	9.09	308.31
XVIII	46–47	46.46	4.31	31.02	6.18	$C_{16}H_{17}CsN_2O_3$	45.95	4.10	31.78	6.70	418.22
XIX	83–84	63.70	5.26	6.54	8.47	$C_{32}H_{34}CaN_4O_6$	62.93	5.61	6.56	9.17	610.71
XX	198–199	64.58	5.32	2.60	10.12	$C_{15}H_{15}LiN_2O_3$	64.75	5.43	2.49	10.07	278.23
XXI	118–119	61.56	5.49	7.21	9.06	$C_{15}H_{15}N_2NaO_3$	61.22	5.14	7.81	9.52	294.28
XXII	163–164	62.23	5.31	6.28	9.17	$C_{30}H_{30}CaN_4O_6$	61.84	5.19	6.88	9.62	582.66
XXIII	185–186	66.11	6.03	2.20	9.08	$C_{16}H_{17}LiN_2O_3$	65.75	5.86	2.37	9.59	292.26
XXIV	74–75	62.83	5.89	7.08	8.56	$C_{16}H_{17}N_2NaO_3$	62.33	5.56	7.46	9.09	308.31
XXV	49–50	46.24	4.28	31.50	6.22	$C_{16}H_{17}CsN_2O_3$	45.95	4.10	31.78	6.70	418.22
XXVI	157–158	63.45	5.29	6.18	8.83	$C_{32}H_{34}CaN_4O_6$	62.93	5.61	6.56	9.17	610.71
XXVII	201–202	65.94	5.70	1.96	9.14	$C_{16}H_{17}LiN_2O_3$	65.75	5.86	2.37	9.59	292.26
XXVIII	108–109	62.73	5.87	7.13	8.72	$C_{16}H_{17}N_2NaO_3$	62.33	5.56	7.46	9.09	308.31
XXIX	42–43	46.13	4.10	31.44	6.35	$C_{16}H_{17}CsN_2O_3$	45.95	4.10	31.78	6.70	418.22
XXX	177–178	63.20	5.78	6.29	8.84	$C_{32}H_{34}CaN_4O_6$	62.93	5.61	6.56	9.17	610.71
XXXI	229–230	65.20	5.31	2.18	9.66	$C_{15}H_{15}LiN_2O_3$	64.75	5.43	2.49	10.07	278.23
XXXII	115–116	61.10	5.07	7.89	9.55	$C_{15}H_{15}N_2NaO_3$	61.22	5.14	7.81	9.52	294.28
XXXIII	59–60	44.82	3.95	32.34	6.48	$C_{15}H_{15}CsN_2O_3$	44.57	3.74	32.88	6.93	404.20
XXXIV	93–94	62.11	5.06	6.62	9.13	$C_{30}H_{30}CaN_4O_6$	61.84	5.19	6.88	9.62	582.66
XXXV	233–234	67.03	6.49	1.96	8.84	$C_{17}H_{19}LiN_2O_3$	66.66	6.25	2.27	9.15	306.29
XXXVI	188–190	63.51	6.05	7.30	8.62	$C_{17}H_{19}N_2NaO_3$	63.34	5.94	7.13	8.69	322.33
XXXVII	147–148	47.58	4.32	30.26	6.18	$C_{17}H_{19}CsN_2O_3$	47.24	4.43	30.75	6.48	432.25
XXXVIII	77–78	64.25	6.29	5.93	8.21	$C_{34}H_{38}CaN_4O_6$	63.93	6.00	6.27	8.77	638.77
XXXIX	223–224	65.90	5.81	2.14	9.18	$C_{16}H_{17}LiN_2O_3$	65.75	5.86	2.37	9.59	292.26
XL	109–110	62.24	5.33	7.68	9.15	$C_{16}H_{17}N_2NaO_3$	62.33	5.56	7.46	9.09	308.31
XLI	162–163	62.55	5.45	6.79	9.02	$C_{32}H_{34}CaN_4O_6$	62.93	5.61	6.56	9.17	610.71
XLII	217–218	66.93	6.51	1.93	8.85	$C_{17}H_{19}LiN_2O_3$	66.66	6.25	2.27	9.15	306.29
XLIII	172–173	63.84	6.08	6.87	8.13	$C_{17}H_{19}N_2NaO_3$	63.34	5.94	7.13	8.69	322.33
XLIV	49–50	47.69	4.19	30.72	6.18	$C_{17}H_{19}CsN_2O_3$	47.24	4.43	30.75	6.48	432.25
XLV	127–128	64.27	6.24	6.01	8.26	$C_{34}H_{38}CaN_4O_6$	63.93	6.00	6.27	8.77	638.77
XLVI	206–207	67.03	6.52	1.90	8.83	$C_{17}H_{19}LiN_2O_3$	66.66	6.25	2.27	9.15	306.29
XLVII	146–147	63.92	6.13	6.82	8.21	$C_{17}H_{19}N_2NaO_3$	63.34	5.94	7.13	8.69	322.33
XLVIII	45–46	47.15	4.64	30.30	6.32	$C_{17}H_{19}CsN_2O_3$	47.24	4.43	30.75	6.48	432.25
XLIX	138–139	64.40	6.25	5.95	8.71	$C_{34}H_{38}CaN_4O_6$	63.93	6.00	6.27	8.77	638.77
L	>250	65.53	6.52	–	10.23	$C_{15}H_{18}N_2O_3$	65.68	6.61	–	10.21	274.32

Table 2. IR and NMR spectral data of compounds **XIV**, **XVII**, **XX**, **XXIV**, **XXXII**, **XXXVI**, **XXXVII**, **XL**, **XLI**, **L**

Comp. no.	IR spectrum, ν, cm^{-1}	^1H and ^{13}C NMR spectra, δ, ppm
XIV	3155 (C=$H_{isoxazole}$); 3061, 3032, 2958, 2923, 2853 (C–H); 1573 (C=O); 1645, 1618, 1589, 1502, 1447, 1393 (C=C, C=N)	1.38–2.58 m (4H, $2CH_2$), 3.58 m (2H, CH_2), 6.79 s (1H, $CH_{isoxazole}$), 7.31 m ($3H_{arom}$), 7.60 m ($2H_{arom}$), 8.09 s (1H, CH=N)
XVII	3120 (C=$H_{isoxazole}$); 3060, 3041, 2935, 2926, 2853 (C–H); 1565 (C=O); 1653, 1612, 1592, 1517, 1497, 1449, 1439, 1416 (C=C, C=N)	1.41 quintet (2H, CH_2, ^3J 7.7 Hz), 1.66 quintet (2H, CH_2, ^3J 7.6 Hz), 1.75 quintet (2H, CH_2, ^3J 7.4 Hz), 2.18 t (2H, CH_2, ^3J 7.6 Hz), 3.69 t (2H, CH_2, ^3J 7 Hz), 7.07 s (1H, $CH_{isoxazole}$), 7.48 m ($3H_{arom}$), 7.84 m ($2H_{arom}$), 8.42 s (1H, CH=N); 27.47 (CH_2), 28.36 (CH_2), 31.41 (CH_2), 39.13 (CH_2), 62.61 (CH_2), 98.10 ($CH_{isoxazole}$), 126.84 ($2CH_{arom}$), 130.26 ($2CH_{arom}$), 131.71 ($1CH_{arom}$), 153.85 (CH=N), 128.25, 163.53, 171.96 ($3C_{quat}$), 182.88 (C=O)
XX	3127 (C=$H_{isoxazole}$); 3066, 3043, 2959, 2929, 2874 (C–H); 1598 (C=O); 1657, 1618, 1574, 1509, 1498, 1451, 1395 (C=C, C=N)	0.91 d (3H, Me, ^3J 6.8 Hz), 0.99 d (3H, Me, ^3J 6.8 Hz), 2.31 m (1H, CH), 3.60 d (1H, CH, ^3J 7.3 Hz), 7.10 s (1H, $CH_{isoxazole}$), 7.51 m ($3H_{arom}$), 7.83 m ($2H_{arom}$), 8.43 s (1H, CH=N)
XXIV	3127 (C=$H_{isoxazole}$); 3063, 3040, 2955, 2928, 2869 (C–H); 1592 (C=O); 1655, 1620, 1574, 1497, 1464, 1450, 1407, 1380, 1366, 1351 (C=C, C=N)	–
XXXII	3125 (C=$H_{isoxazole}$); 3060, 3030, 2947, 2928, 2866 (C–H); 1567 (C=O); 1652, 1614, 1595, 1509, 1479, 1437, 1415 (C=C, C=N)	2.01 quintet (2H, CH_2, ^3J 7.3 Hz), 2.25 t (2H, CH_2, ^3J 7.5 Hz), 2.34 s (3H, Me), 3.70 t (2H, CH_2, ^3J 6.4 Hz), 6.97 s (1H, $CH_{isoxazole}$), 7.26 d ($2H_{arom}$, ^3J 8 Hz), 7.66 d ($2H_{arom}$, ^3J 8 Hz), 8.40 s (1H, CH=N); 21.51 (Me), 28.48 (CH_2), 36.49 (CH_2), 62.44 (CH_2), 97.48 ($CH_{isoxazole}$), 126.75 ($2CH_{arom}$), 130.79 ($2CH_{arom}$), 125.48, 142.21, 163.43, 172.06 ($4C_{quat}$), 182.00 (C=O)
XXXVI	3124 (C=$H_{isoxazole}$); 3059, 3030, 2935, 2926, 2853 (C–H); 1566 (C=O); 1654, 1614, 1594, 1510, 1442, 1416 (C=C, C=N)	1.41 quintet (2H, CH_2, ^3J 7.7 Hz), 1.66 quintet (2H, CH_2, ^3J 7.6 Hz), 1.74 quintet (2H, CH_2, ^3J 7.4 Hz), 2.18 t (2H, CH_2, ^3J 7.6 Hz), 2.37 s (3H, Me), 3.68 t (2H, CH_2, ^3J 7 Hz), 6.98 s (1H, $CH_{isoxazole}$), 7.29 d ($2H_{arom}$, ^3J 8 Hz), 7.70 d ($2H_{arom}$, ^3J 8 Hz), 8.40 s (1H, CH=N); 21.50 (Me), 27.46 (CH_2), 28.36 (CH_2), 31.40 (CH_2), 39.13 (CH_2), 62.61 (CH_2), 97.44 ($CH_{isoxazole}$), 126.80 ($2CH_{arom}$), 130.83 ($2CH_{arom}$), 153.90 (CH=N), 125.53, 142.28, 163.44, 172.15 ($4C_{quat}$), 182.88 (C=O)
XXXVII	3128 (C=$H_{isoxazole}$); 3060, 3011, 2930, 2855 (C–H); 1563 (C=O); 1654, 1615, 1594, 1510, 1447, 1427, 1410 (C=C, C=N)	1.40 quintet (2H, CH_2, ^3J 7.7 Hz), 1.66 quintet (2H, CH_2, ^3J 7.6 Hz), 1.73 quintet (2H, CH_2, ^3J 7.4 Hz), 2.18 t (2H, CH_2, ^3J 7.6 Hz), 2.35 s (3H, Me), 3.66 t (2H, CH_2, ^3J 7 Hz), 6.96 s (1H, $CH_{isoxazole}$), 7.27 d ($2H_{arom}$, ^3J 8.1 Hz), 7.67 d ($2H_{arom}$, ^3J 8.1 Hz), 8.38 s (1H, CH=N); 21.56 (Me), 27.49 (CH_2), 28.38 (CH_2), 31.42 (CH_2), 39.26 (CH_2), 62.61 (CH_2), 97.50 ($CH_{isoxazole}$), 126.80 ($2CH_{arom}$), 130.84 ($2CH_{arom}$), 153.85 (CH=N), 125.50, 142.26, 163.43, 172.10 ($4C_{quat}$), 182.70 (C=O)
XL	3129 (C=$H_{isoxazole}$); 3055, 3033, 2967, 2932, 2871 (C–H); 1597 (C=O); 1651, 1613, 1594, 1509, 1449, 1406 (C=C, C=N)	0.90 d (3H, Me, ^3J 6.8 Hz), 0.99 d (3H, Me, ^3J 6.8 Hz), 2.31 m (1H, CH), 2.35 s (3H, Me), 3.58 d (1H, CH, ^3J 7.3 Hz), 7.18 s (1H, $CH_{isoxazole}$), 7.27 d ($2H_{arom}$, ^3J 8 Hz), 7.69 d ($2H_{arom}$, ^3J 8 Hz), 8.32 s (1H, CH=N); 19.25 (Me), 20.31 (Me), 21.51 (Me), 32.94 (CH), 86.10 (CH), 98.02 ($CH_{isoxazole}$), 126.74 ($2CH_{arom}$), 130.79 ($2CH_{arom}$), 153.07 (CH=N), 125.60, 142.15, 163.57, 171.99 ($4C_{quat}$), 179.03 (C=O)
XLI	3135 (C=$H_{isoxazole}$); 3058, 3031, 2961, 2926, 2871 (C–H); 1596 (C=O); 1651, 1615, 1511, 1449, 1417 (C=C, C=N)	–
L	3224 (NH); 3125 (C=$H_{isoxazole}$); 2500–2900 (COOH); 3049, 3033, 2980, 2952, 2923, 2883 (C–H); 1688 (C=O); 1620, 1599, 1567, 1515, 1494, 1469, 1430, 1417 (C=C, C=N)	1.95 quintet (2H, CH_2, ^3J 7.5 Hz), 2.29 t (2H, CH_2, ^3J 8.1 Hz), 2.35 s (3H, CH_3), 3.64 t (2H, CH_2, ^3J 6.9 Hz), 6.35 s (2H, CH_2), 6.56 s (1H, $CH_{isoxazole}$), 7.33 d ($2H_{arom}$, ^3J 8 Hz), 7.75 d ($2H_{arom}$, ^3J 8 Hz); 18.04 (CH_2), 21.62 (CH_3), 30.66 (CH_2), 38.17 (CH_2), 47.12 (CH_2), 99.60 ($CH_{isoxazole}$), 126.18 ($2CH_{arom}$), 130.40 ($2CH_{arom}$), 124.70, 141.00, 161.59, 170.20 ($4C_{quat}$), 174.87 (C=O)

II, 0.01 mol of amino acid **III–VII**, 0.01 mol of lithium hydride **VIII** (or 0.01 mol of sodium methoxide **IX** prepared by dissolving 0.01 mol of sodium metal in methanol, or 0.005 mol of cesium carbonate **X**, or 0.005 mol of calcium hydride] in 50 mL of anhydrous methanol was refluxed for 3–4 h until complete dissolution of all the reaction components. The solvent was removed in a vacuum at 35–40°C. Yields of the salts **XII–XLIX** were ~100%.

4-[5-(4-Tolyl)isoxazol-3-ylmethyl]aminobutanoic acid (L). To a suspension of 1.2 g (4.08 mmol) of sodium salt **XXXII** in 40 mL of anhydrous benzene was added 0.32 g (8.46 mmol) of sodium borohydride and 1.47 g (24.48 mmol) of glacial acetic acid. The suspension was stirred for 6 h, treated with water and 10% HCl aqueous solution to pH ~ 6.5. The aqueous layer was separated and concentrated under a reduced pressure to a volume of ~5 mL. The resulting suspension was cooled to 5°C, the precipitate was filtered off and dried in a vacuum. Yield 65%.

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