Highly diastereoselective synthesis of 2-monosubstituted 1*R*,5*S*(1*S*,5*R*)-glycoluriles on the basis of *S*- and *R*-*N*-carbamoyl-α-amino acids

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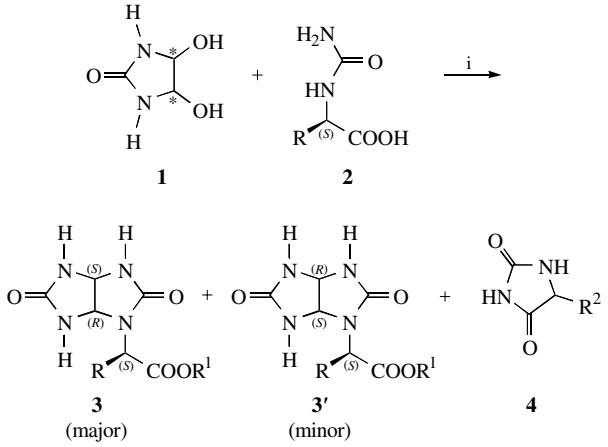
The reactions of 4,5-dihydroxyimidazolidin-2-one with chiral *S*- and *R*-*N*-carbamoyl-α-amino acids occur diastereoselectively with the formation of corresponding 1*R*,5*S*(1*S*,5*R*)-glycoluriles as predominant diastereomers; the absolute configuration is determined for three stereoisomers by X-ray diffraction analysis.

The synthesis and stereochemistry of glycoluriles and their sulfo analogues is of considerable interest.[1–6] Recently, we synthesised racemic 2-monosubstituted glycoluriles using the reactions of 4,5-dihydroxyimidazolidin-2-one **1** with 1-monosubstituted ureas.[6] The formation of two diastereomers would be expected with the use of monosubstituted ureas with substituents having a specified configuration in the reaction of bicycle formation.

In this work, we studied in detail the reactions of compound **1** with chiral *S*- (Scheme 1) and *R*-*N*-carbamoyl-α-amino acids **2a**–**d**, which were prepared by the interaction of KCNO with *S*-norvaline, *S*-α-aminobutyric acid, *S*-methionine, *S*-glutamine and *R*-methionine, respectively. Depending on the solubility of the parent compounds, cyclocondensation was performed in aqueous or aqueous isopropanol solutions at pH 1–2. In all cases, target glycoluriles **3** and **3′** were obtained in preparative yields.



1 **2**

3 (major) **3′** (minor) **4**

2 **a** R = Pr
b R = Et
c R = CH_2CH_2SMe
d R = $CH_2CH_2CONH_2$

3, **3′** **a** R = Pr, R^1 = H
b R = Et, R^1 = H
c R = CH_2CH_2SMe, R^1 = H
d R = CH_2CH_2COOMe, R^1 = Me

4 **a** R^2 = H
b R^2 = Pr
c R^2 = Et
d R = CH_2CH_2SMe

Scheme 1 *Reagents and conditions*: i, H_2O (H_2O/Pr^iOH), HCl, 90 °C, 1 h.

The diastereomeric composition of reaction products was determined by ^{1}H NMR spectroscopy. We found that isolated glycoluriles were formed as two diastereomers **3a**–**d** and **3′a**–**d**, which appeared as the doubling of signals from all groups of protons. An analysis of the most informative region of the spectra of these compounds (the region 3.7–4.5 ppm for the signals of CH protons from the amino acid fragment) indicated that the ratio of diastereomers for glycoluriles **3a** and **3′a**, **3b** and **3′b** is ≈ 3:1 or 5:1, respectively, whereas the ratio for compounds **3c**

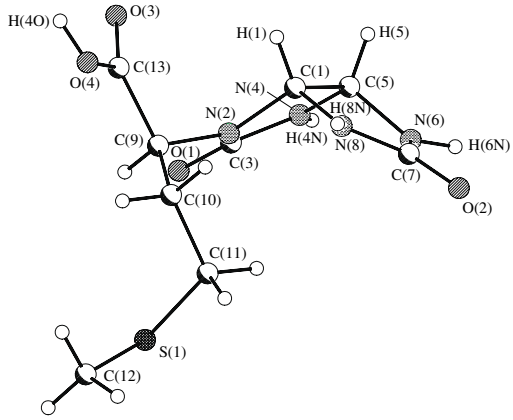


Figure 1 The general view of **3c**. The selected bond lengths (Å): N(2)–C(1) 1.462(2), N(2)–C(3) 1.366(2), N(2)–C(9) 1.455(2), N(8)–C(1) 1.438(2), N(8)–C(7) 1.350(2), N(4)–C(3) 1.353(2), N(4)–C(5) 1.436(2), N(6)–C(5) 1.452(2), N(6)–C(7) 1.358(2), O(1)–C(3) 1.234(2), O(2)–C(7) 1.238(2); bond angles (°): N(8)–C(1)–N(2) 114.4(1), N(8)–C(1)–C(5) 103.1(1), N(2)–C(1)–C(5) 102.2(1), C(3)–N(2)–C(9) 122.2(1), C(3)–N(2)–C(1) 111.3(1), C(9)–N(2)–C(1) 123.7(1), O(1)–C(3)–N(4) 125.3(1), O(1)–C(3)–N(2) 125.7(1), N(4)–C(3)–N(2) 108.9(1), C(3)–N(4)–C(5) 112.7(1), N(4)–C(5)–N(6) 115.0(1), N(4)–C(5)–C(1) 103.0(1), N(6)–C(5)–C(1) 101.9(1), C(7)–N(6)–C(5) 111.6(1), O(2)–C(7)–N(8) 124.0(1), O(2)–C(7)–N(6) 127.0(1), N(8)–C(7)–N(6) 109.0(1), C(7)–N(8)–C(1) 112.3(1). Conformation: C(1)N(2)C(3)N(4)C(5) – envelope [deviation of N(2) 0.16 Å]; C(1)N(1)C(3)N(2)C(2) – envelope [deviation of C(5) 0.22 Å].

and **3′c** is 15:1, as well as for **3d** and **3′d**. This fact is indicative of the high diastereoselectivity of the reactions of **1** with **2a**–**d**. The signals of the CH protons of major diastereomers are downfield shifted with respect to the corresponding signals of minor diastereomers. The signals of the other protons manifest themselves as multiplets. To determine accurately the chemical shifts and spin–spin coupling constants of C(1)H and C(5)H protons in test compounds **3** (all signals were broadened), calculations were performed by the NUMMRIT method with the use of the Xsim (Linux) program.

We failed to separate the mixtures of diastereomers **3a** and **3′a**, **3b** and **3′b** because their physico-chemical properties are similar. Individual diastereomers **3c** and **3′c** were obtained by recrystallization of the resulting mixture from water. For stereoisomers **3c** (major) and **3′c** (minor), the angles of optical rotation were found to be +18.50° and –77.78° (*c* 2, 1 N NaOH), respectively. In the course of separation of a predominant isomer from the product of reaction of **1** and **2d**, which was recrystallised from methanol in the presence of trace hydrochloric acid, the amide group underwent hydrolysis to the carboxyl group followed by esterification of both of the carboxyl groups to methyl esters with the formation of **3d**.†

The interaction of compound **1** with *R*-*N*-carbamoylmethionine also occurred with high diastereoselectivity (the diastereomer ratio 15:1), and predominant diastereomer **3′′c** was separated

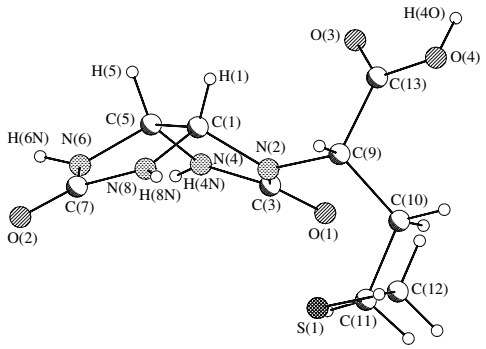


Figure 2 The general view of **3′c**. The selected bond lengths (Å): N(2)–C(1) 1.450(3), N(2)–C(3) 1.382(3), N(2)–C(9) 1.463(3), N(4)–C(3) 1.351(4), N(4)–C(5) 1.435(4), N(6)–C(5) 1.439(4), N(6)–C(7) 1.341(4), N(8)–C(1) 1.445(3), N(8)–C(7) 1.346(4), O(1)–C(3) 1.223(4), O(2)–C(7) 1.231(4); bond angles (°): N(8)–C(1)–N(2) 115.0(2), N(8)–C(1)–C(5) 102.4(2), N(2)–C(1)–C(5) 103.9(2), C(3)–N(2)–C(1) 111.0(2), C(3)–N(2)–C(9) 121.9(2), C(1)–N(2)–C(9) 118.5(2), O(1)–C(3)–N(4) 125.6(2), O(1)–C(3)–N(2) 126.2(3), N(4)–C(3)–N(2) 108.2(2), C(3)–N(4)–C(5) 113.2(2), N(4)–C(5)–N(6) 114.7(3), N(4)–C(5)–C(1) 103.2(2), N(6)–C(5)–C(1) 103.5(2), C(7)–N(6)–C(5) 112.4(3), O(2)–C(7)–N(6) 125.2(3), O(2)–C(7)–N(8) 126.1(3), N(6)–C(7)–N(8) 108.7(3), C(7)–N(8)–C(1) 112.7(3). Conformation: C(1)N(2)C(3)N(4)C(5) cycle – envelope [deviation of C(3) 0.09 Å], C(1)C(5)N(6)C(7)N(8) cycle – envelope [deviation of C(7) 0.06 Å].

† All new compounds exhibited satisfactory elemental analyses, and their structures were confirmed by IR, ^{1}H and ^{13}C NMR spectroscopy. ^{1}H and ^{13}C NMR spectra were recorded on Bruker WM-250 (250 MHz) and Bruker AM-300 (75.5 MHz) spectrometers, respectively. Chemical shifts were measured with reference to the residual protons of a [$^2H_6$]DMSO solvent (δ 2.50 ppm).

Initial 4,5-dihydroxyimidazolidin-2-one **1** was synthesised according to a known method from urea and glyoxal;[9] *N*-carbamoyl-α-amino acids **2a–d** were synthesised analogously to the published methods from α-amino acids and KOCN.[10,11]

*2-[(1*R,5S*)+(1*S,5R*)-(3,7-Dioxo-2,4,6,8-tetraazabicyclo[3.3.0]oct-2-yl)]-2(*S*)-pentanoic acid* **3a + 3′a**: yield 33%. ^{1}H NMR ([$^2H_6$]DMSO) δ: 0.81–0.95 (m, 3H, Me), 1.15–1.51 (m, 2H, CH_2), 1.63–2.06 (m, 2H, CH_2), 3.85 (dd, 1H, CH, ^3J 4.89 Hz, ^3J 10.37 Hz) for **3′a**, 4.23 (dd, 1H, CH, ^3J 6.11 Hz, ^3J 9.27 Hz) for **3a**, 5.11–5.43 (m, 2H, CH–CH), 7.11, 7.27 7.36. 7.36 7.43, 7.61 (5 br. s, 3H, 3 NH), 12.61 (br. s, 1H, COOH).

*2-[(1*R,5S*)+(1*S,5R*)-(3,7-Dioxo-2,4,6,8-tetraazabicyclo[3.3.0]oct-2-yl)]-2(*S*)-butanoic acid* **3b + 3′b**: yield 35%. ^{1}H NMR ([$^2H_6$]DMSO) δ: 0.81–1.02 (m, 3H, Me), 1.67–2.04 (m, 2H, CH_2), 3.78 (dd, 1H, CH, ^3J 6.10 Hz, ^3J 9.76 Hz) for **3′b**, 4.15 (dd, 1H, CH, ^3J 4.89 Hz, ^3J 10.38 Hz) for **3b**, 5.18–5.50 (m, 2H, CH–CH), 7.11, 7.28, 7.33, 7.41, 7.61 (5 br. s, 3H, NH), 12.65 (br. s, 1H, COOH).

*(+)-2-[(1*R,5S*)-(3,7-Dioxo-2,4,6,8-tetraazabicyclo[3.3.0]oct-2-yl)]-2(*S*)-4-methylthiobutanoic acid* **3c**: yield 37%, mp 256–258 °C (decomp.), $[\alpha]_D^{20}$ +18.50° (*c* 2; 1 N NaOH). ^{1}H NMR ([$^2H_6$]DMSO) δ: 2.05–2.17 (m, 5H, Me + CH_2), 2.36–2.64 (m, 2H, CH_2), 4.47 (dd, 1H, CH, ^3J 9.4 Hz, ^3J 6.3 Hz), 5.29 [dt, 1H, C(1)H, ^3J 8.44±0.04 Hz, ^3J 1.7 Hz], 5.41 [dd, 1H, C(5)H, ^3J 8.44±0.04 Hz, ^3J 2.4 Hz], 7.25 (br. s, 2H, 2 NH), 7.48 (br. s, 1H, NH), 12.81 (br. s, 1H, OH). ^{13}C NMR ([$^2H_6$]DMSO) δ: 14.64 (Me), 28.79 (CH_2), 30.28 (CH_2), 53.18 (CH), 62.99 (CH), 67.48 (CH), 160.02 (CO), 161.53 (CO), 173.01 (COOH).

*(–)-2-[(1*S,5R*)-(3,7-Dioxo-2,4,6,8-tetraazabicyclo[3.3.0]oct-2-yl)]-2(*S*)-4-methylthiobutanoic acid* **3′c**: yield 6%, mp 233–235 °C (decomp.), $[\alpha]_D^{20}$ –77.78° (*c* 2; 1 N NaOH). ^{1}H NMR ([$^2H_6$]DMSO) δ: 2.10–2.19 (m, 5H, Me + CH_2), 2.51–2.61 (m, 2H, CH_2), 4.07 (dd, 1H, CH, ^3J 4.89 Hz, ^3J 10.36 Hz), 5.29 [br. d, 1H, C(1)H, ^3J 8.16±0.03 Hz], 5.34 [br. d, 1H, C(5)H, ^3J 8.16±0.03 Hz], 7.28 (s, 1H, NH), 7.38 (s, 1H, NH), 7.51 (s, 1H, NH), 12.75 (br. s, 1H, OH).

*(–)-2-[(1*S,5R*)-(3,7-Dioxo-2,4,6,8-tetraazabicyclo[3.3.0]oct-2-yl)]-2(*R*)-4-methylthiobutanoic acid* **3′′c**: yield 37%, mp 256–258 °C (decomp.), $[\alpha]_D^{20}$ –18.50° (*c* 2; 1 N NaOH). For ^{1}H NMR and ^{13}C NMR ([$^2H_6$]DMSO) see **3c**.

*Dimethyl ester of 2-[(1*R,5S*)-(3,7-Dioxo-2,4,6,8-tetraazabicyclo[3.3.0]-oct-2-yl)]-2(*S*)-pentanedioic acid* **3d**: yield 34%, mp 248–250 °C. ^{1}H NMR ([$^2H_6$]DMSO) δ: 2.00–2.21 (m, 2H, CH_2), 2.28–2.46 (m, 2H, CH_2), 3.59 (s, 3H, OMe), 3.65 (s, 3H, OMe), 4.39 (dd, 1H, CH, ^3J 5.15 Hz, ^3J 11.03 Hz), 5.24 [br. d, 1H, C(1)H, ^3J 8.32±0.02 Hz], 5.48 [br. d, 1H, C(5)H, ^3J 8.32±0.02 Hz], 7.36 (br. s, 2H, 2 NH), 7.64 (s, 1H, NH). ^{13}C NMR ([$^2H_6$]DMSO) δ: 24.35 (CH_2), 29.85 (CH_2), 51.31 (Me), 52.14 (Me), 53.47 (CH), 62.82 (CH), 67.22 (CH), 159.53 (CO), 161.12 (CO), 171.27 (COOMe), 172.58 (COOMe).

by crystallization from water; the angle of optical rotation was –18.52°. Note that stereoisomers **3c** and **3′c** exhibited opposite rotation angles, equal melting temperatures, and identical NMR spectra; that is, they are enantiomers.

In the reactions of compounds **1** with *S*- and *R*-*N*-carbamoyl-α-amino acids, the products of two side processes were also found: the rearrangement of 4,5-dihydroxyimidazolidin-2-one **1** into hydantoin **4a** and the self-cyclization of *N*-carbamoyl-α-amino acids **2a–c** into hydantoins **4b–d** (Scheme 1). Yields of **4a–d** were in the 38–40% region. These processes are well known, and the resulting hydantoins were described in the literature.[7,8]

To determine the configurations of asymmetric C(1) and C(5) atoms in the glycoluriles synthesised, we performed X-ray diffraction analysis of single crystals of the predominant isomers of compounds **3c**,**d** and minor stereoisomer **3′c**.‡

The main geometry parameters in **3c**, **3′c** and **3d** are similar.‡ The torsion angles H(1)C(1)C(5)H(5) are 2° and 12° in **3d** and **3c**, respectively. The conformation of five-membered rings is a flattened envelope. In the ring with the substituted nitrogen atom, the C(3) atom projects out of the plane, whereas the C(5) or C(7) atom projects out of the plane in the other ring (Figures 1–3). Nitrogen atoms are planar with an insignificant pyramidalization of the substituted N(2) atom [the sum of angles at N(2) varied from 351.7(2)° to 357.2(2)°].

The presence of a great number of proton donors and proton acceptors in the molecules resulted in a complex supramolecular organization in crystals. In this case, despite considerable differences, the systems of hydrogen bonds in the three test compounds were somewhat similar. Thus, the H(6N) atoms in all of the structures participate in intermolecular H-bonds with O(1) atoms [N(6)···O(1) varied from 2.876(2) to 3.001(2) Å]. Moreover, in **3c** and **3′c**, the H-bond systems for the H(4N) atom [N(4)–H(4N)···O(3)] and the hydroxyl group [O(4)–H(4O)···O(2)] are coincident, whereas the H(8N) atom partici-

‡ *Crystallographic data for compounds* **3c**, **3′c** and **3d**.

The crystal of **3c** ($C_9H_{14}N_4O_4S$) is orthorhombic at 110 K, space group $P2_12_12_1$, a = 8.9981(9) Å, b = 10.808(1) Å, c = 12.589(1) Å, V = = 1224.2(2) Å^3, Z = 4 (Z' = 1), M = 274.30, d_{calc} = 1.488 g cm^{-3}, μ(MoKα) = 2.79 cm^{-1}, F(000) = 576.

The crystal of **3′c** ($C_9H_{14}N_4O_4S$) is orthorhombic at 298 K, space group $P2_12_12_1$, a = 6.084(5) Å, b = 9.744(7) Å, c = 20.607(17) Å, V = = 1221.7(16) Å^3, Z = 4 (Z' = 1), M = 274.30, d_{calc} = 1.491 g cm^{-3}, μ(MoKα) = 2.79 cm^{-1}, F(000) = 576.

The crystal of **3d** ($C_{11}H_{16}N_4O_6$) is trigonal at 298 K, space group $P3_221$, a = 10.492(2) Å, c = 22.149(4) Å, V = 2111.5(6) Å^3, Z = 6 (Z' = 1), M = 300.28, d_{calc} = 1.417 g cm^{-3}, μ(MoKα) = 1.16 cm^{-1}, F(000) = 948.

Intensities of 10347 (**3c**), 2075 (**3′c**) and 3032 (**3d**) reflections were measured with a Smart 1000 CCD diffractometer [λ(MoKα) = 0.71072 Å, ω-scan with a 10 s exposure, $2\theta < 60°$] at 100 K (**3c**) and with Siemens P3/PC [λ(MoKα) = 0.71072 Å, $\theta/2\theta$ scan, $2\theta < 60°$ and 52°] at 298 K (**3′c** and **3e**), 3582 (**3c**), 2075 (**3′c**) and 2739 (**3d**) independent reflections were used in the further refinement. The structures were solved by a direct method and refined by the full-matrix least-squares technique against F^2 in the anisotropic-isotropic approximation. The analysis of the Fourier synthesis has revealed that ester groups in **3d** are disordered. The absolute configurations of **3c** and **3′c** were determined on the basis of the Flack parameter. The refinement converged to wR_2 = 0.0827 and GOF = 1.043 for all independent reflections [R_1 = 0.0364 was calculated against F for 3245 observed reflections with $I > 2\sigma(I)$] for **3c**; to wR_2 = = 0.1419 and GOF = 1.025 for all independent reflections [R_1 = 0.0492 was calculated against F for 1621 observed reflections with $I > 2\sigma(I)$] for **3′c** and to wR_2 = 0.1550 and GOF = 0.954 for all independent reflections [R_1 = 0.0552 was calculated against F for 1828 observed reflections with $I > 2\sigma(I)$] for **3d**. All calculations were performed using SHELXTL PLUS 5.0.

Atomic coordinates, bond lengths, bond angles and thermal parameters have been deposited at the Cambridge Crystallographic Data Centre (CCDC). These data can be obtained free of charge *via* www.ccdc.cam.uk/conts/retrieving.html (or from the CCDC, 12 Union Road, Cambridge CB2 1EZ, UK; fax: +44 1223 336 033; or deposit@ccdc.cam.ac.uk). Any request to the CCDC for data should quote the full literature citation and CCDC reference number 227399–227401. For details, see 'Notice to Authors', *Mendeleev Commun.*, Issue 1, 2003.

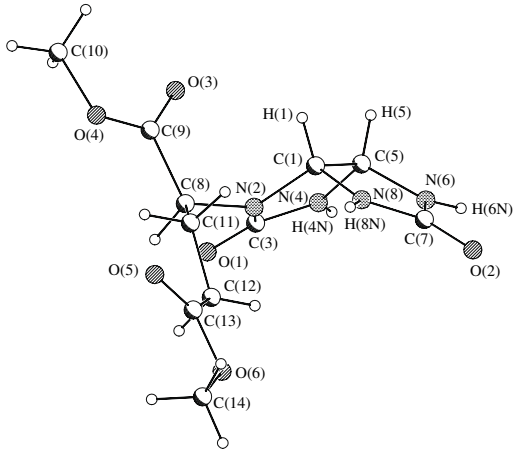


Figure 3 The general view of **3d**. The disordered ester groups are omitted for clarity. The selected bond lengths (Å): N(2)–C(1) 1.451(4), N(2)–C(3) 1.373(4), N(2)–C(8) 1.459(3), N(4)–C(3) 1.332(4), N(4)–C(5) 1.431(4), N(6)–C(5) 1.443(4), N(6)–C(7) 1.354(3), N(8)–C(7) 1.343(3), N(8)–C(1) 1.427(4), O(1)–C(3) 1.229(4), O(2)–C(7) 1.232(3); bond angles (°): N(8)–C(1)–N(2) 115.0(2), N(8)–C(1)–C(5) 102.9(2), N(2)–C(1)–C(5) 102.9(2), C(3)–N(2)–C(1) 111.4(2), C(3)–N(2)–C(8) 119.1(3), C(1)–N(2)–C(8) 123.4(2), O(1)–C(3)–N(4) 126.5(3), O(1)–C(3)–N(2) 124.8(3), N(4)–C(3)–N(2) 108.6(3), C(3)–N(4)–C(5) 114.0(2), N(4)–C(5)–N(6) 114.7(3), N(4)–C(5)–C(1) 102.6(2), N(6)–C(5)–C(1) 102.4(2), C(7)–N(6)–C(5) 112.6(2), O(2)–C(7)–N(8) 125.8(2), O(2)–C(7)–N(6) 125.6(2), N(8)–C(7)–N(6) 108.7(2), C(7)–N(8)–C(1) 113.4(2). Conformation: C(1)N(2)C(3)N(4)C(5) cycle – envelope [deviation of C(3) 0.09 Å], C(1)C(5)N(6)C(7)N(8) cycle – envelope [deviation of C(5) 0.03 Å].

pates in different H-bonds with the O(1) atom in **3c** or S(1) in **3′c**. It is likely that these differences in the H-bond systems of **3c** and **3′c** result in a change in the conformation of the substituent at the N(2) atom [the torsion angle C(12)S(1)C(11)C(10) is equal to –79° to –64.5°, respectively].

In all cases, a three-dimensional H-bound framework is the supramolecular structure; in this case, because of the trigonal symmetry of a **3d** crystal, N–H···O bound bicyclic molecules form honeycombs, within which ester substituents are arranged (Figure 4).

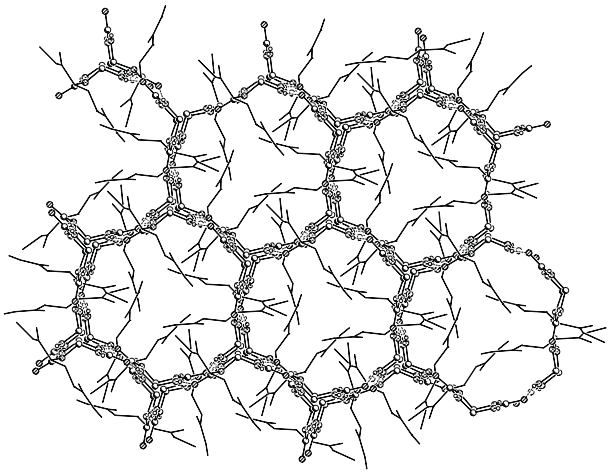


Figure 4 The scheme illustrating formation of the N–H…O bonded honeycombs in the crystal structure of **3d**. The ester substituent is shown without ball representation of atoms.

So, it was found that C(1) and C(5) carbon atoms in **3c** and **3d** exhibit an (1*R*,5*S*) configuration (Figures 1 and 3), whereas an opposite (1*S*,5*R*) configuration was found in stereoisomer **3′c** (Figure 2). A comparison between X-ray diffraction data and the spectroscopic characteristics of the prepared compounds demonstrates that in all cases predominant isomers in the reactions of **1** with *S*-*N*-carbamoyl-α-amino acids or *R*-*N*-carbamoyl-α-amino acids exhibit 1*R*,5*S*- or 1*S*,5*R*-configuration, respectively.

Thus, as a result of a study of the interaction of 4,5-dihydroxyimidazolidin-2-one **1** with *S*- and *R*-*N*-carbamoyl-α-amino acids, we found that these reactions occur with high diastereoselectivity. This fact provides an opportunity to synthesise glycoluriles with specified configurations of bridging carbon atoms. The use of *N*-carbamoyl-α-amino acids with asymmetric centres of opposite configurations is a simple method for the preparation of individual glycolurile enantiomers.

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