ISSN 1070-4280, Russian Journal of Organic Chemistry, 2006, Vol. 42, No. 3, pp. 412–422. © Pleiades Publishing, Inc., 2006.
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Stereoselective Synthesis of Functional Derivatives of 2-(2-Carboxyethyl)pyrrolidine-2-carboxylic Acid

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Received June 25, 2005

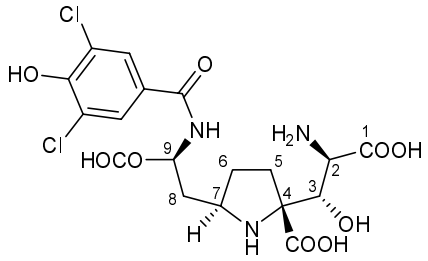
Abstract—Azomethine ylides generated from dimethyl 2-(arylmethylideneamino)pentanedioates by the action of AgOAc and Et_3N reacted with dipolarophiles in regio- and stereoselective fashion to form 5-aryl-2-(2-carboxyethyl)pyrrolidine-2-carboxylic acid derivatives. 1,3-Dipolar cycloaddition of divinyl sulfone to the azomethine ylide generated from the Schiff base derived from methyl (*S*)-2-phthalimido-4-oxobutanoate and dimethyl glutamate gave chiral simplified kaitocephalin analogs.

DOI: 10.1134/S1070428006030134

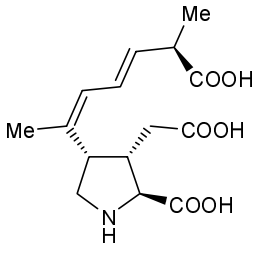
Derivatives of L-proline, which is a genetically coded cyclic α-amino acid, are widely spread in the nature as both individual substances and structural fragments of more complex molecules [1]. Many of these derivatives exhibit pronounced physiological activity toward particular biological targets. For example, kaitocephalin (**I**) [2] is an AMPA-kainate ionotropic glutamate receptor antagonist, while domoic acid (**II**) acts as an agonist selective for the same receptors [3]. The conformationally rigid pyrrolidine fragment in their molecules stabilizes the substituents attached thereto in a certain spatial orientation with respect to each other. As a result, the conformational mobility of the entire molecule is restricted, thus enhancing interaction between the pyrrolidine ligand and active site of a protein target, as compared to an acyclic analog [4]. Structure–property studies in medicinal chemistry are based on the approach implying replacement of the amino acid fragment in a peptide or peptidomimetic with known physiological activity by pyrrolidinecarboxylic acid which is a "superposition" of proline and the acid being replaced, so-called *chimera* [5].

The present study was aimed at synthesizing functional derivatives of 2-(2-carboxyethyl)pyrrolidine-2-carboxylic acid (**III**) which may be regarded as a proline–glutamic acid chimera. We planned to introduce an aryl substituent into the 5-position of the proline fragment in **IV**, taking into account that *cis*-5-phenylproline is considered to be a privileged substructure [6], as well as a group allowing the left-hand part of kaitocephalin to be built up subsequently. The substituents R^2 and R^3 in positions *4* and *3*, respectively, were expected to be either modified (for this purpose, methoxycarbonyl groups were selected) or removed to afford a 2,5-substituted moiety as in kaitocephalin.

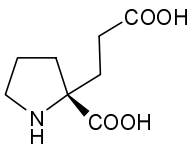
We believed that Scheme 1 was best consistent with our synthetic goals. This scheme implies preparation of racemic pyrrolidines **IV** in two steps starting from dimethyl glutamate **V** and aldehydes. Here, the key step is 1,3-dipolar cycloaddition of olefins activated by electron-acceptor groups to metalated azomethine ylide **A**. The latter is generated from the Schiff base formed by condensation of aldehyde with amino acid



I

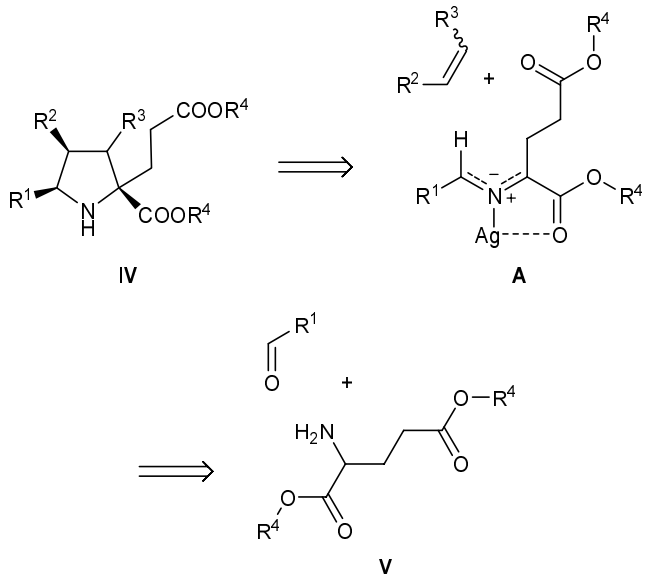


II



III

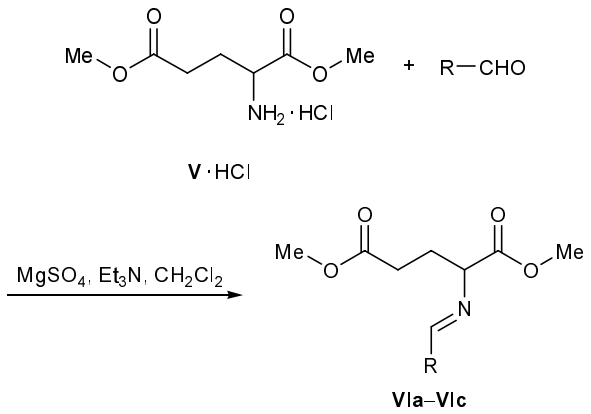
Scheme 1.



derivative **V**. The above scheme for building up pyrrolidine cyclic system was shown to be effective for the synthesis of various substituted prolines [7], but it was not applied previously to preparation of chimeric structures like **IV**. Treatment with soft Lewis acids of Schiff bases derived from aldehydes and amino acid esters stereoselectively gives metal dipole **A** having *syn,syn* configuration, as shown in Scheme 1. The subsequent cycloaddition of $R^2CH{=}CHR^3$ dipolarophile is also characterized by high *endo*-stereoselectivity and regioselectivity, so that the R^1, R^2, and CO_2R^4 substituents in the pyrrolidine ring of **IV** are arranged *cis* with respect to each other [7].

Schiff bases **VIa**–**VIc** were synthesized by reaction of glutamic acid dimethyl ester hydrochloride (**V**·HCl) with benzaldehyde, 3,4-dimethoxybenzaldehyde, and

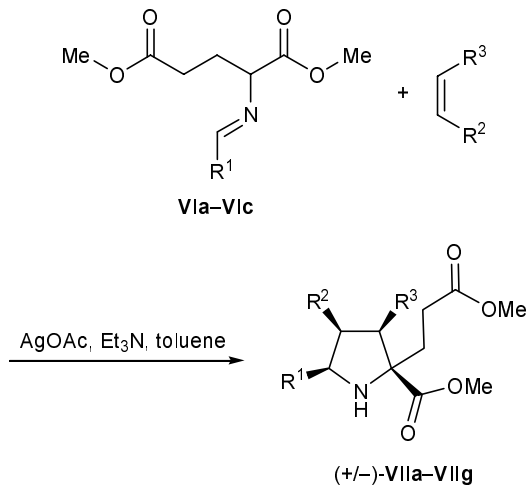
Scheme 2.



R = Ph (**a**), 3,4-$(MeO)_2C_6H_3$ (**b**), 3-Py (**c**).

pyridine-3-carbaldehyde (Scheme 2). *syn*-Dipole **A** was generated (Scheme 1) in toluene in an inert atmosphere by the action of equimolar amounts of silver acetate and triethylamine. As dipolarophiles we used divinyl sulfone, methyl acrylate, and dimethyl maleate. Tetra- and pentasubstituted pyrrolidines **VIIa**–**VIIg** were the only products (according to the TLC data) formed by 1,3-dipolar cycloaddition of the above dipolarophiles to azomethine ylides derived from Schiff bases **VIa**–**VIc** (Scheme 3); they were isolated in good yields by column chromatography on silica gel as spectrally and analytically pure substances. The structure of racemic pyrrolidines **VIIa**–**VIIg** was determined by two-dimensional NMR techniques and by comparison of spectral parameters with those available for analogous compounds [8]. The obtained data showed that the R^1, R^2, and R^3 substituents and the methoxycarbonyl group in position *2* of the pyrrolidine ring in all compounds **VIIa**–**VIIg** are arranged *cis* with respect to each other.

Scheme 3.



VII, R^1 = Ph, R^2 = $CH_2{=}CHSO_2$, R^3 = H (**a**); R^1 = Ph, R^2 = COOMe, R^3 = H (**b**); R^1 = 3,4-$(MeO)_2C_6H_3$, R^2 = COOMe, R^3 = H (**c**); R^1 = 3-Py, R^2 = COOMe, R^3 = H (**d**); R^1 = Ph, R^2 = R^3 = COOMe (**e**); R^1 = 3,4-$(MeO)_2C_6H_3$, R^2 = R^3 = COOMe (**f**); R^1 = 3-Py, R^2 = R^3 = COOMe (**g**).

The only example of using divinyl sulfone in 1,3-dipolar cycloaddition to azomethine ylides obtained from glycine methyl ester and substituted benzaldehydes was reported in [9]; according to the authors, the corresponding pyrrolidines were formed in a poor yield, and the vinylsulfonyl substituent therein was assigned *trans* orientation with respect to the other substituents. The structure of vinylsulfonylpyrrolidine **VIIa** synthesized in the present work was proved by

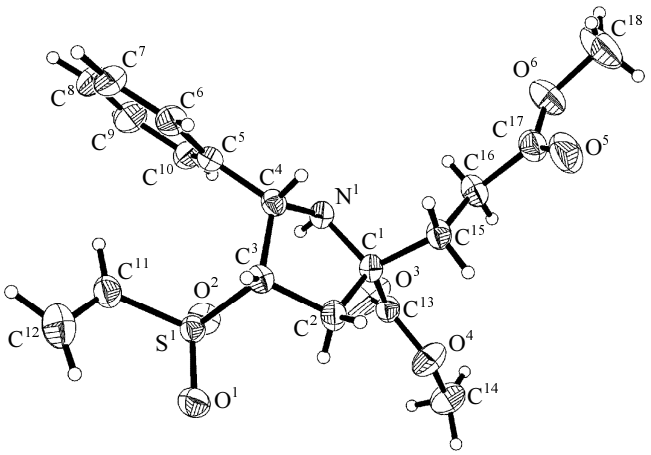


Fig. 1. Structure of the molecule of methyl (2*S**,4*S**,5*S**)-4-ethenylsulfonyl-2-(3-methoxy-3-oxopropyl)-5-phenylpyrrolidine-2-carboxylate (**VIIa**) according to the X-ray diffraction data.

the X-ray diffraction data (Fig. 1, Table 1); we thus confirmed the validity of our assignments based on the spectral data.

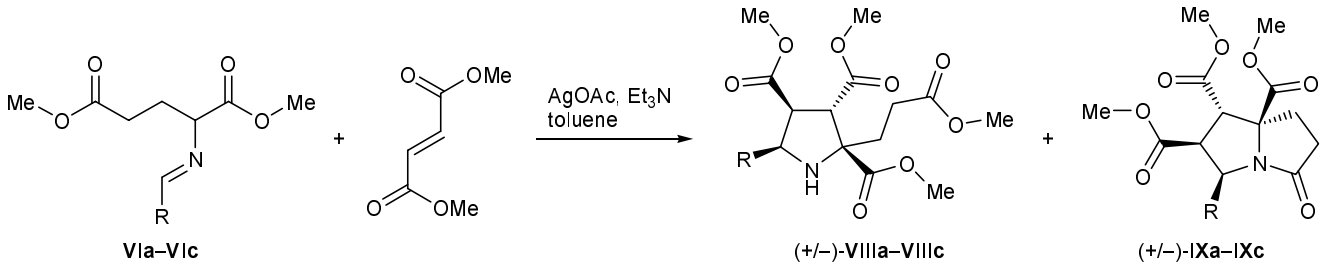
When dimethyl fumarate was used as dipolarophile in 1,3-dipolar cycloaddition to Schiff bases **VIa**–**VIc**, two products were detected in the reaction mixtures (Scheme 4). According to the NMR data, more chromatographically mobile compounds were cycloaddition products **VIIIa**–**VIIIc** whose steric structure is consistent with the available published data on cycloaddition of dimethyl fumarate to azomethine ylides [8]. In the ^{1}H NMR spectra of the minor products, the number of methoxy protons in the ester groups was equal to 9; therefore, these compounds were assigned the structure of perhydropyrrolizines **IXa**–**IXc** formed via lactamization with participation of the 2-(2-methoxycarbonylethyl) substituent. This assignment was proved by the results of X-ray diffraction study of compound **IXa** (Fig. 2, Table 2).

The ability of pyrrolidines **VIIIa**–**VIIIc** to undergo lactamization depends on the substituent R. In the reaction of dimethyl fumarate with Schiff base **VIa** (Scheme 4), pyrrolidine **VIIIa** was isolated in 74% yield by chromatography on silica gel. Likewise, compounds **VIIIb** and **IXb** were isolated in 3 and 71% yield, respectively, while the reaction mixture contained up to 10% of pyrrolizine **IXb** (according to the ^{1}H NMR data). This means that ring closure occurred mainly during chromatographic separation of the products on silica gel. Intramolecular cyclization of pyrrolidinylpropionic acids or their esters is widely used for the preparation of perhydropyrrolizines [10]. Pyrrolidines **VIIIa**–**VIIIc** isolated as individual substances undergo partial lactamization to bicyclic compounds **IXa**–**IXc** on storage. Esters **VIIa**–**VIIg** are stable, and they can be stored for a year at 4°C.

Table 1. Selected bond lengths and bond angles in the molecule of methyl (2*S**,4*S**,5*S**)-4-ethenylsulfonyl-2-(3-methoxy-3-oxopropyl)-5-phenylpyrrolidine-2-carboxylate (**VIIa**)

Bond	*d*, Å	Bond	*d*, Å	Bond	*d*, Å	Bond	*d*, Å
S^1–O^1	1.435(2)	N^1–H^1	0.84(2)	O^6–C^{18}	1.433(5)	C^4–C^5	1.508(3)
S^1–O^2	1.4382(18)	O^3–C^{13}	1.179(3)	C^1–C^{13}	1.519(3)	C^5–C^{10}	1.375(3)
S^1–C^{11}	1.761(3)	O^4–C^{13}	1.314(3)	C^1–C^{15}	1.537(4)	C^5–C^6	1.399(3)
S^1–C^3	1.794(3)	O^4–C^{14}	1.444(3)	C^1–C^2	1.569(4)	C^{11}–C^{12}	1.296(5)
N^1–C^4	1.457(3)	O^5–C^{17}	1.207(3)	C^2–C^3	1.529(3)	C^{15}–C^{16}	1.506(4)
N^1–C^1	1.466(3)	O^6–C^{17}	1.331(4)	C^3–C^4	1.562(3)	C^{16}–C^{17}	1.494(4)
Angle	ω, deg	Angle	ω, deg	Angle	ω, deg	Angle	ω, deg
$O^1S^1O^2$	117.40(11)	$N^1C^1C^{13}$	108.82(18)	$C^4C^3S^1$	115.61(16)	$O^3C^{13}C^1$	124.9(2)
$O^1S^1C^{11}$	108.78(13)	$N^1C^1C^{15}$	110.3(2)	$N^1C^4C^5$	114.80(19)	$O^4C^{13}C^1$	111.71(19)
$O^2S^1C^{11}$	107.29(13)	$C^{13}C^1C^{15}$	110.3(2)	$N^1C^4C^3$	105.59(18)	$C^{16}C^{15}C^1$	113.4(2)
$O^1S^1C^3$	107.59(12)	$N^1C^1C^2$	106.8(2)	$C^5C^4C^3$	117.9(2)	$C^{17}C^{16}C^{15}$	112.9(2)
$O^2S^1C^3$	108.41(12)	$C^{13}C^1C^2$	110.6(2)	$C^{10}C^5C^4$	123.7(2)	$O^5C^{17}O^6$	123.5(3)
$C^{11}S^1C^3$	106.92(12)	$C^{15}C^1C^2$	109.87(19)	$C^6C^5C^4$	117.6(2)	$O^5C^{17}C^{16}$	124.9(3)
$C^4N^1C^1$	105.85(18)	$C^3C^2C^1$	105.5(2)	$C^{12}C^{11}S^1$	119.7(3)	$O^6C^{17}C^{16}$	111.6(2)
$C^{13}O^4C^{14}$	117.8(2)	$C^2C^3C^4$	102.7(2)	$O^3C^{13}O^4$	123.4(2)	$O^6C^{18}H^{18}$	110(4)
$C^{17}O^6C^{18}$	115.9(4)	$C^2C^3S^1$	109.10(19)				

Scheme 4.

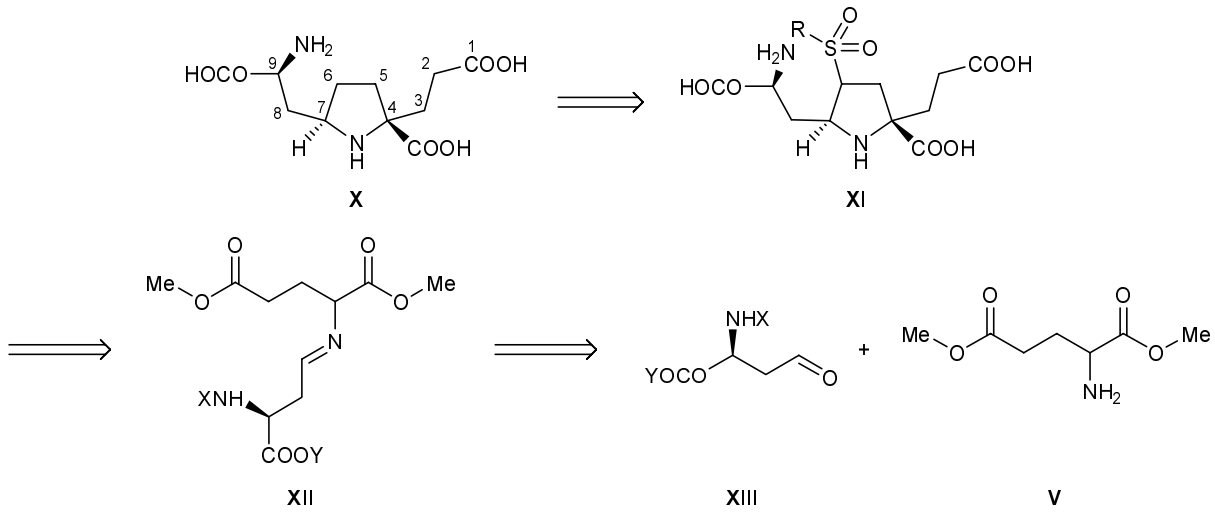


R = Ph (**a**), 3,4-$(MeO)_2C_6H_3$ (**b**), 3-Py (**c**).

Kaitocephalin (**I**) molecule contains five asymmetric centers; in keeping with the latest data, it has 2*R*,3*S*,4*R*,7*R*,9*S* configuration [11] which was confirmed by the total synthesis including 14 steps (overall yield 1.2%) [12]. On the basis of our results on the synthesis of substituted 2-(2-carboxyethyl)pyrrolidine-2-carboxylic acid derivatives **VIIa**–**VIIg** and **VIIIa**–**VIIIc** we believed it possible to obtain a simplified analog of kaitocephalin, amino acid **X**, via 1,3-dipolar cycloaddition of divinyl sulfone to aliphatic Schiff base **XII** according to Scheme 5. Insofar as the chiral center in **XII**, as well as in the azomethine ylide generated therefrom, is not involved in the cycloaddition process, it was expected to be conserved in pyrrolidine **XI**. The transformation of the cycloaddition product into amino acid **X** implies well explored desulfonylation reaction [13]. We planned to use chiral aldehyde **XIII** as carbonyl component for the preparation of Schiff base **XII**. Undoubtedly, successful implementation of the proposed scheme requires thorough selection of protecting groups in intermediate polyfunctional compounds, primarily for the amino and carboxy groups on C^9 (X and Y), as well as for the pyrrolidine nitrogen atom and carboxy groups on C^2 and C^4.*

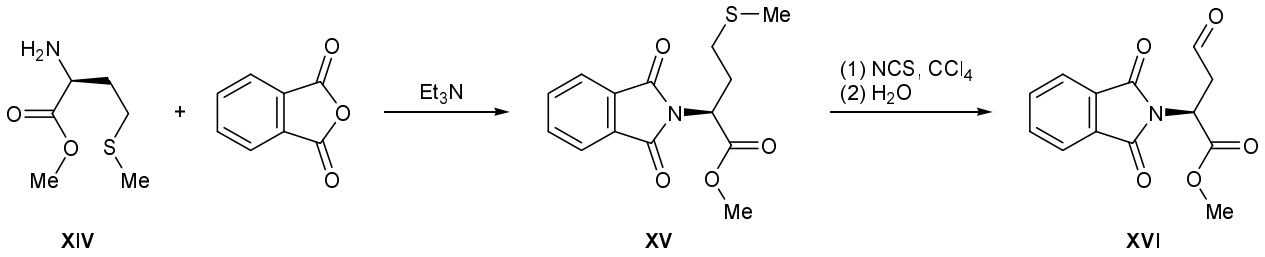
Schiff base **XII** was synthesized from methyl (*S*)-4-oxo-2-phthalimidobutanoate (**XVI**) having the required *S* configuration. Compound **XVI** (optical purity 62.2%) was prepared in two steps according to the known procedure [14] from L-methionine methyl ester (**XIV**) (Scheme 6). The reaction of aldehyde **XVI** with glutamic acid dimethyl ester hydrochloride (**V** · HCl) gave Schiff base **XVII**, and the latter was brought into 1,3-dipolar cycloaddition to divinyl sulfones under the same conditions as in the synthesis of pyrrolidines **VIIa**–**VIIg** and **VIIIa**–**VIIIc** (Scheme 7). 1,2-Prototropic tautomerization and subsequent cycloaddition of aliphatic aldehyde imines having an α-hydrogen atom may be complicated due to imine–enamine tautomerism, which could give rise to side processes [15]. Unlike cycloaddition involving aro-

Scheme 5.



* The atom numbering in amino acid **X** shown in Scheme 5 corresponds to the atom numbering in kaitocephalin [11, 12].

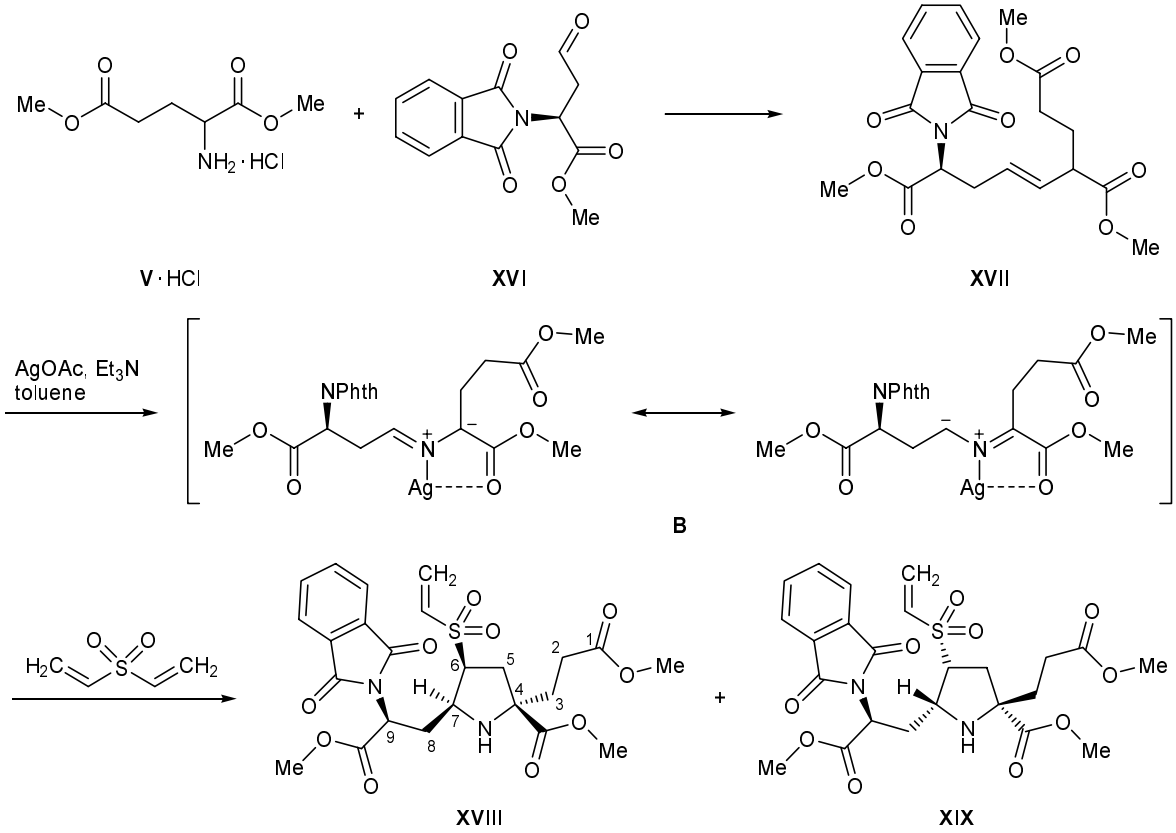
Scheme 6.



matic aldehyde imines **VIa**–**VIc**, the reaction sequence shown in Scheme 7 led to formation of a mixture of products. The product mixture was subjected to chromatographic separation to isolate 19% of a substance which, according to the ^{1}H NMR data, contained two isomers at a ratio of 3.5:1. Each isomer was a product of cycloaddition of divinyl sulfone to azomethine ylide **B**. The isomer mixture was characterized by a specific optical rotation of $[\alpha]_D^{20} = -15.0°$ (c = 1.0, $CHCl_3$), and its elemental composition corresponded to the assumed structure of methyl 4-ethenylsulfonyl-2-(3-methoxy-3-oxopropyl)-5-(3-methoxy-3-oxo-2-phthalimidopropyl)-pyrrolidine-2-carboxylate. The steric structure of the isomers was studied using two-dimensional and correlation NMR techniques, but we were only able to assign signals in the ^{1}H and ^{13}C NMR spectra and determine *cis* orientation of protons on the C^6 and C^7 atoms of the major isomer (Figs. 3, 4).

Taking into account regio- and stereoselectivity of the reaction of divinyl sulfone with Schiff bases **VIa**–**VIc** and assuming *syn*,*syn* configuration of 1,3-dipole **B** and *endo*-transition state for 1,3-dipolar cycloaddition, the obtained products (Scheme 7) were assigned structures **XVIII** and **XIX** which were formed by 1,3-dipolar cycloaddition through *endo-re* and *endo-si* transition states, respectively (Fig. 5). However, our

Scheme 7.



Phth = phthaloyl.

Table 2. Selected bond lengths and bond angles in the molecule of trimethyl (1*S**,2*S**,3*R**,7a*S**)-5-oxo-3-phenyltetrahydropyrrolizine-1,2,7a-tricarboxylate (**IXa**)

Bond	*d*, Å	Bond	*d*, Å	Bond	*d*, Å	Bond	*d*, Å
N^1–C^1	1.3739(19)	O^{13}–C^{14}	1.1919(19)	C^1–C^2	1.507(2)	C^5–C^{16}	1.510(2)
N^1–C^7	1.4668(19)	O^{14}–C^{16}	1.311(2)	C^2–C^3	1.518(2)	C^5–C^6	1.523(2)
N^1–C^4	1.4714(19)	O^{14}–C^{17}	1.464(3)	C^3–C^4	1.546(2)	C^6–C^{18}	1.507(2)
O^{11}–C^1	1.2159(18)	O^{15}–C^{16}	1.192(2)	C^4–C^{14}	1.532(2)	C^6–C^7	1.555(2)
O^{12}–C^{14}	1.3267(19)	O^{16}–C^{18}	1.3252(19)	C^4–C^5	1.547(2)	C^7–C^8	1.509(2)
O^{12}–C^{15}	1.448(2)	O^{17}–C^{18}	1.190(2)				
Angle	ω, deg	Angle	ω, deg	Angle	ω, deg	Angle	ω, deg
$C^1N^1C^7$	121.86(12)	$N^1C^1C^2$	108.14(13)	$C^3C^4C^5$	117.72(12)	$N^1C^7C^6$	100.27(11)
$C^1N^1C^4$	112.54(12)	$C^1C^2C^3$	103.40(12)	$C^{16}C^5C^6$	111.65(13)	$C^8C^7C^6$	114.90(12)
$C^7N^1C^4$	113.44(11)	$C^2C^3C^4$	103.92(13)	$C^{16}C^5C^4$	114.01(13)	$O^{13}C^{14}O^{12}$	124.00(14)
$C^{14}O^{12}C^{15}$	117.13(14)	$N^1C^4C^{14}$	111.50(12)	$C^6C^5C^4$	104.34(12)	$O^{13}C^{14}C^4$	125.08(14)
$C^{16}O^{14}C^{17}$	115.85(19)	$N^1C^4C^3$	102.84(11)	$C^{18}C^6C^5$	112.84(13)	$O^{12}C^{14}C^4$	110.91(13)
$C^{18}O^{16}C^{19}$	117.71(15)	$C^{14}C^4C^3$	109.09(12)	$C^{18}C^6C^7$	115.18(12)	$O^{15}C^{16}O^{14}$	123.40(17)
$O^{11}C^1N^1$	124.11(14)	$N^1C^4C^5$	104.16(11)	$C^5C^6C^7$	105.16(12)	$O^{15}C^{16}C^5$	124.52(16)
$O^{11}C^1C^2$	127.72(14)	$C^{14}C^4C^5$	111.11(12)	$N^1C^7C^8$	115.66(12)	$O^{14}C^{16}C^5$	112.09(16)

experimental data are insufficient to determine which isomer corresponds to the major product; this issue is the subject of our current studies.

EXPERIMENTAL

The ^{1}H and ^{13}C NMR spectra were recorded on Bruker DRX-400 (400 MHz for ^{1}H and 100 MHz for ^{13}C) and Bruker DRX-500 spectrometers (500 MHz for ^{1}H and 125 MHz for ^{13}C). Two-dimensional correlation NMR experiments were performed using modified pulse sequences supplied by the manufacturer. The spectra were recorded at 303 K from solutions in DMSO-d_6; the chemical shifts were referenced to the solvent signals (DMSO-d_5, δ 2.50 ppm; DMSO-d_6, δ_C 39.50 ppm). The IR spectra were obtained in KBr on an IR-200 Fourier spectrometer. The specific rotations were measured on a VNIEKIPRODMASh EPO 1A polarimeter using 10-cm cells. The optical purity was calculated by the standard procedure [16]. X-Ray diffraction study of compounds **VIIa** and **IXa** was performed on an Enraf–Nonius CAD-4 diffractometer at 293 K; the structures were solved by the direct method using SHELXS-97 software [17] and were refined according to [18].

Schiff bases VIa–VIc and XVII. Triethylamine, 0.45 g (4.4 mmol), was added to a suspension of 0.93 g (4.4 mmol) of glutamic acid dimethyl ester hydrochloride and 1.00 g (8.3 mmol) of anhydrous magnesium sulfate in 25 ml of methylene chloride. The mixture was stirred for 1 h at room temperature in an inert atmosphere, 4.0 mmol of the corresponding aldehyde

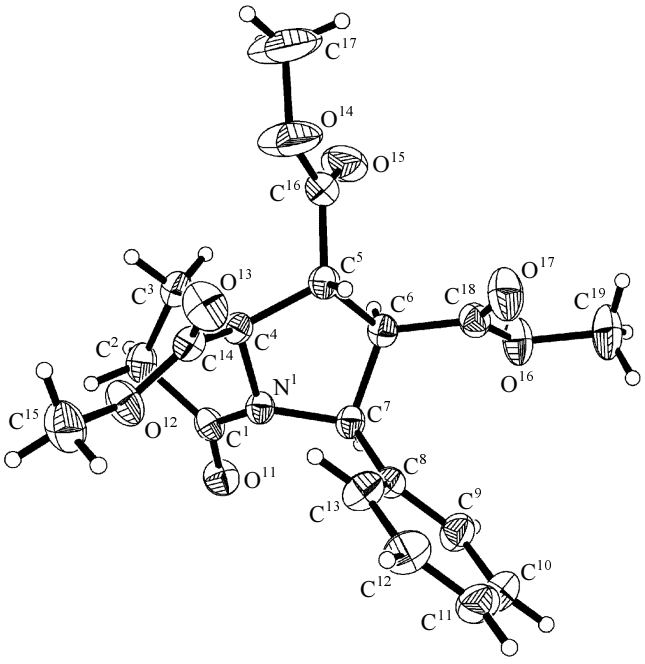


Fig. 2. Structure of the molecule of trimethyl (1*S**,2*S**,3*R**,7a*S**)-5-oxo-3-phenyltetrahydropyrrolizine-1,2,7a-tricarboxylate (**IXa**) according to the X-ray diffraction data.

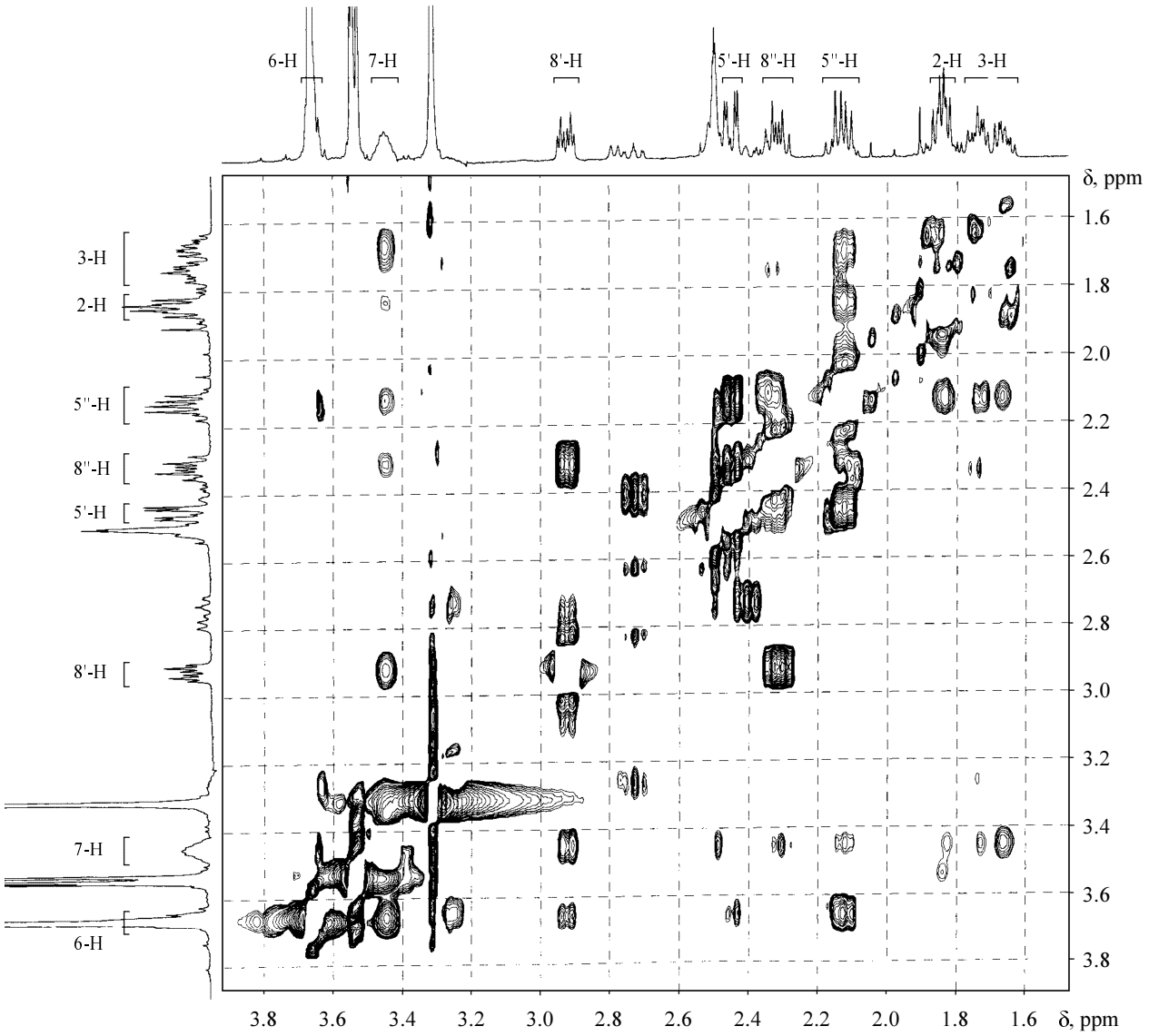


Fig. 3. NOESY spectrum of isomer mixture **XVIII/XIX**.

was added, and the mixture was stirred for 24 h. The precipitate was filtered off, the organic phase was washed with a saturated solution of $NaHCO_3$, water, and a saturated solution of NaCl and dried over Na_2SO_4, and the solvent was removed under reduced pressure. The residue was brought into further syntheses without additional purification.

Dimethyl 2-(benzylideneamino)pentanedioate (VIa) was synthesized from benzaldehyde. Yield 89%, oily substance. ^{1}H NMR spectrum, δ, ppm: 2.01–2.24 m (2H), 2.29–2.37 m (2H), 3.55 s (3H, OCH_3), 3.66 s (3H, OCH_3), 4.13 d.d (1H, H_{arom}, J = 5.5, 7.9 Hz), 7.49 m (3H, H_{arom}), 7.77 m (2H, H_{arom}), 8.39 s (1H).

Dimethyl 2-(3,4-dimethoxybenzylideneamino)-pentanedioate (VIb) was synthesized from 3,4-dimethoxybenzaldehyde. Yield 92%, oily substance. ^{1}H NMR spectrum, δ, ppm: 2.00–2.07 m (1H), 2.11–2.19 m (1H), 2.26–2.36 m (2H), 3.55 s (3H, OCH_3), 3.65 s (3H, OCH_3), 3.79 s (3H, OCH_3), 3.81 s (3H, OCH_3), 4.06 d.d (1H, H_{arom}, J = 5.1, 8.1 Hz), 7.03 d (1H, H_{arom}, J = 8.3 Hz), 7.27 d.d (1H, H_{arom}, J = 1.8, 8.1 Hz), 7.35 d (1H, H_{arom}, J = 1.8 Hz), 8.26 s (1H).

Dimethyl 2-(pyridin-3-ylmethylideneamino)-pentanedioate (VIc) was synthesized from pyridine-3-carbaldehyde. Yield 87%, oily substance. ^{1}H NMR spectrum, δ, ppm: 2.03–2.25 m (2H), 2.31–2.36 m (2H), 3.55 s (3H, OCH_3), 3.67 s (3H, OCH_3), 4.18 d.d (1H, H_{arom}, J = 5.5, 7.9 Hz), 7.51 d.d (1H, H_{arom}, 4.9, 7.9 Hz), 8.16 d (1H, H_{arom}, J = 7.9 Hz), 8.68 d (1H, H_{arom}, J = 4.9 Hz), 8.90 s (1H).

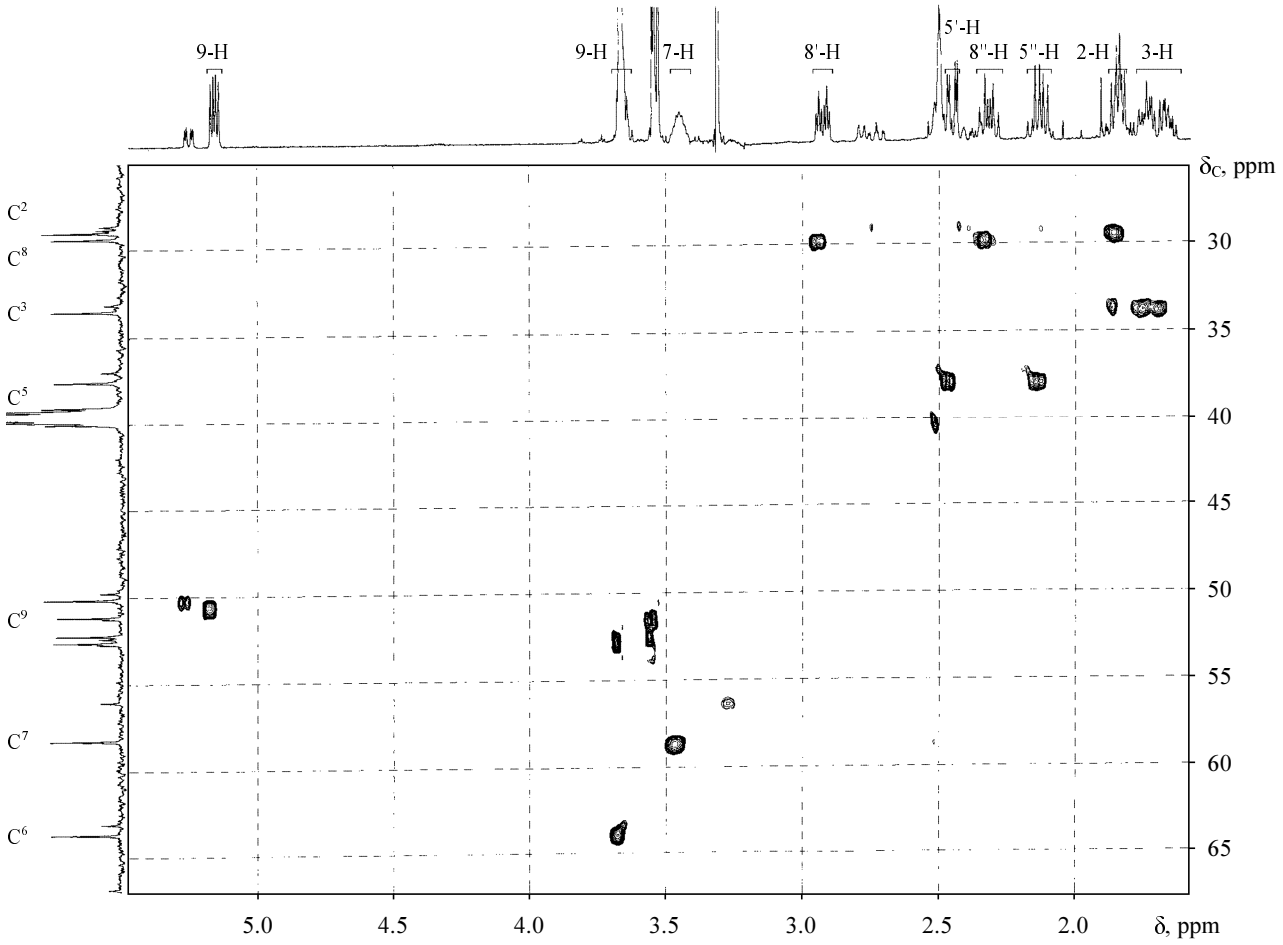


Fig. 4. ^{1}H–^{13}C correlation NMR spectrum of isomer mixture **XVIII**/**XIX**.

Dimethyl 2-[(*S*)-3-(1,3-dioxo-2,3-dihydro-1*H*-isoindol-2-yl)-4-methoxy-4-oxobutylideneamino]pentanedioate (XVII) was synthesized from methyl (*S*)-4-oxo-2-phthalimidobutanoate (**XVI**). Yield 70%, oily substance. ^{1}H NMR spectrum, δ, ppm: 1.57–1.93 m (2H), 2.04 m (2H), 3.39 s (3H, OCH_3), 3.51 s (3H, OCH_3), 3.60 m (2H), 3.65 s (3H, OCH_3), 3.78 m (1H), 5.35 m (1H), 7.74 m (1H), 7.89 s (4H).

1,3-Dipolar cycloaddition of dipolarophiles to Schiff bases VIa–VIc and XVII. Schiff base **VIa**–**VIc** or **XVII**, 2.0 mmol, was dissolved in 5 ml of toluene in an inert atmosphere, and 2.4 mmol of the corresponding dipolarophile and 0.37 g (2.2 mmol) of silver acetate were added in one portions. A solution of 0.24 g (2.4 mmol) of triethylamine in 20 ml of toluene was then added to the resulting suspension under stirring. The mixture was stirred for 24–48 h at room temperature in an inert atmosphere with protection from light. The precipitate was filtered off and washed with 5 ml of toluene, the organic phase was concentrated under reduced pressure, and the residue was purified by chromatography on silica gel.

Methyl (2*S,4*S**,5*S**)-4-ethenylsulfonyl-2-(3-methoxy-3-oxopropyl)-5-phenylpyrrolidine-2-carboxylate (VIIa).** Yield 85%, colorless crystals, mp 148–150°C. IR spectrum, ν, cm^{-1}: 3322, 3103, 2942, 1739, 1604, 1446, 1301, 1198, 1125. ^{1}H NMR

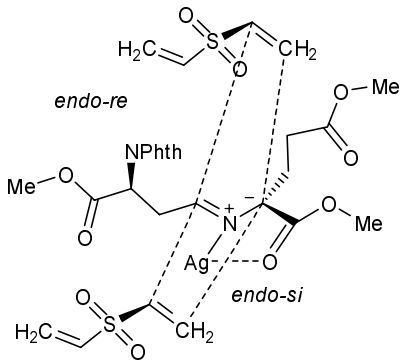


Fig. 5. *endo*-Transition states in the 1,3-dipolar cycloaddition of divinyl sulfone to chiral azomethine ylide **B**.

spectrum, δ, ppm: 2.04 t (2H, CH_2CO, J = 7.5 Hz), 2.21–2.24 m (1H, C**H**$_2$CH$_2$), 2.31–2.34 m (1H, C**H**$_2$CH$_2$), 2.37–2.40 m (1H, 3-H), 2.82 d.d (1H, 3-H, J = 4.0, 15.2 Hz), 3.40 d (1H, 1-H, J = 11.0 Hz), 3.53 s (3H, OCH_3), 3.72 s (3H, OCH_3), 4.10 m (1H, 4-H), 4.64 d.d (1H, 5-H, J = 6.0, 11.0 Hz), 5.61 d (1H, CH=C**H**$_2$, J = 16.5 Hz), 5.65 d (1H, CH=C**H**$_2$, J = 9.8 Hz), 5.94 d.d (1H, C**H**=CH$_2$, J = 9.8, 16.5 Hz), 7.26–7.36 m (5H, H_{arom}). ^{13}C NMR spectrum, δ_C, ppm: 29.56, 33.68, 36.48, 51.22, 52.38, 61.83, 66.03, 66.68, 127.51, 127.62, 127.95, 128.12, 135.70, 136.65, 172.75, 173.94. Found, %: C 56.52; H 5.92; N 4.02. $C_{18}H_{23}NO_6S$. Calculated, %: C 56.68; H 6.08; N 3.67. X-Ray diffraction data: rhombic crystals; a = 19.229(3), b = 29.674(6), c = 13.382(2) Å; α = β = γ = 90°; V = 7636(2) Å^3; space group $Fdd2$; d_{calc} = 1.327 g/cm^3. The complete set of crystallographic parameters was deposited to the Cambridge Crystallographic Data Center (entry no. CCDC-245808).

Dimethyl (2*S,4*S**,5*R**)-2-(3-methoxy-3-oxopropyl)-5-phenylpyrrolidine-2,4-dicarboxylate (VIIb).** Yield 85%, light yellow crystals, mp 60–62°C. IR spectrum, ν, cm^{-1}: 3312, 3058, 2950, 1736, 1603, 1436, 1201, 1174. ^{1}H NMR spectrum, δ, ppm: 1.95–2.03 m (2H, CH_2CO), 2.06–2.12 m (1H, C**H**$_2$CH$_2$), 2.20–2.24 m (1H, C**H**$_2$CH$_2$), 2.33–2.40 m (1H, 3-H), 2.49 d.d (1H, 3-H, J = 5.0, 13.5 Hz), 3.11 s (3H, OCH_3), 3.12 m (1H, 1-H), 3.35 m (1H, 4-H), 3.55 s (3H, OCH_3), 3.73 s (3H, OCH_3), 4.54 t (1H, 5-H, J = 7.8 Hz), 7.21–7.34 m (5H, H_{arom}). Found, %: C 61.92; H 6.34; N 3.85. $C_{18}H_{23}NO_6$. Calculated, %: C 61.88; H 6.64; N 4.01.

Dimethyl (2*S,4*S**,5*R**)-5-(3,4-dimethoxyphenyl)-2-(3-methoxy-3-oxopropyl)pyrrolidine-2,4-dicarboxylate (VIIc).** Yield 62%, oily substance. IR spectrum, ν, cm^{-1}: 3318, 3013, 2842, 1738, 1611, 1590, 1435, 1255, 1197, 1028. ^{1}H NMR spectrum, δ, ppm: 1.95–2.01 m (2H, CH_2CO), 2.03–2.08 m (1H, C**H**$_2$CH$_2$), 2.19–2.22 m (1H, C**H**$_2$CH$_2$), 2.32–2.39 m (1H, 3-H), 2.43–2.46 m (1H, 3-H), 3.04 d (1H, 1-H, J = 9.0 Hz), 3.20 s (3H, OCH_3), 3.32 m (1H, 4-H), 3.56 s (3H, OCH_3), 3.72 s (9H, OCH_3), 4.47 t (1H, 5-H, J = 7.8 Hz), 6.72 d.d (1H, H_{arom}, J = 2.0, 8.5 Hz), 6.82 d (1H, H_{arom}, J = 2.0 Hz), 6.88 d (1H, H_{arom}, J = 8.5 Hz). Found, %: C 58.58; H 6.70; N 3.62. $C_{20}H_{27}NO_8$. Calculated, %: C 58.67; H 6.65; N 3.42.

Dimethyl (2*S,4*S**,5*R**)-2-(3-methoxy-3-oxopropyl)-5-(pyridin-3-yl)pyrrolidine-2,4-dicarboxylate (VIId).** Yield 84%, yellow crystals, mp 60–61°C. IR spectrum, ν, cm^{-1}: 3311, 3029, 2954, 1733, 1589, 1437, 1256, 1207, 1170, 1020. ^{1}H NMR spectrum, δ, ppm: 1.97–2.03 m (2H, CH_2CO), 2.08–2.13 m (1H, C**H**$_2$CH$_2$), 2.22–2.25 m (1H, C**H**$_2$CH$_2$), 2.33–2.39 m (1H, 3-H), 2.48–2.56 m (1H, 3-H), 3.13 s (3H, OCH_3), 3.23 br.s (1H, 1-H), 3.44 d.t (1H, 4-H, J = 6.3, 7.5 Hz), 3.56 s (3H, OCH_3), 3.72 s (3H, OCH_3), 4.64 br.s (1H, 5-H), 7.33 d.d (1H, H_{arom}, J = 4.8, 8.0 Hz), 7.62 d.t (1H, H_{arom}, J = 1.8, 8.0 Hz), 8.44 m (2H, H_{arom}). Found, %: C 58.09; H 6.52; N 7.69. $C_{17}H_{22}N_2O_6$. Calculated, %: C 58.28; H 6.33; N 8.00.

Trimethyl (2*S,3*R**,4*S**,5*R**)-2-(3-methoxy-3-oxopropyl)-5-phenylpyrrolidine-2,3,4-tricarboxylate (VIIe).** Yield 68%, oily substance. IR spectrum, ν, cm^{-1}: 3316, 2953, 1736, 1605, 1437, 1244, 1201, 1174. ^{1}H NMR spectrum, δ, ppm: 2.06–2.27 m (3H), 2.41–2.45 m (1H, CH_2CO), 3.14 s (3H, OCH_3), 3.47–3.53 m (3H, 1-H, 3-H, 4-H), 3.55 s (3H, OCH_3), 3.61 s (3H, OCH_3), 3.65 s (3H, OCH_3), 4.49 d.d (1H, 5-H, J = 4.9, 11.0 Hz), 7.23–7.26 m (5H, H_{arom}). Found, %: C 60.15; H 6.15; N 3.68. $C_{20}H_{25}NO_8$. Calculated, %: C 58.96; H 6.19; N 3.44.

Trimethyl (2*S,3*R**,4*S**,5*R**)-5-(3,4-dimethoxyphenyl)-2-(3-methoxy-3-oxopropyl)pyrrolidine-2,3,4-tricarboxylate (VIIf).** Yield 64%, colorless crystals, mp 105–107°C. IR spectrum, ν, cm^{-1}: 3358, 2956, 2837, 1738, 1610, 1522, 1433, 1260, 1180, 1016. ^{1}H NMR spectrum, δ, ppm: 2.01–2.11 m (1H, C**H**$_2$CH$_2$), 2.16–2.22 m (1H, C**H**$_2$CH$_2$), 2.24–2.29 m (1H, CH_2CO), 2.38–2.45 m (1H, CH_2CO), 3.21 s (3H, OCH_3), 3.36–3.47 m (3H, 1-H, 3-H, 4-H), 3.56 s (3H, OCH_3), 3.62 s (3H, OCH_3), 3.65 s (3H, OCH_3), 3.72 s (6H, OCH_3), 4.42 d.d (1H, 5-H, J = 4.3, 10.4 Hz), 6.77–6.81 m (1H, H_{arom}), 6.89–6.92 m (2H, H_{arom}). Found, %: C 56.72; H 6.37; N 2.95. $C_{22}H_{29}NO_{10}$. Calculated, %: C 56.53; H 6.25; N 3.00.

Trimethyl (2*S,3*R**,4*S**,5*R**)-2-(3-methoxy-3-oxopropyl)-5-(pyridin-3-yl)pyrrolidine-2,3,4-tricarboxylate (VIIg).** Yield 88%, oily substance. IR spectrum, ν, cm^{-1}: 3349, 2953, 1737, 1593, 1437, 1242, 1202, 1174. ^{1}H NMR spectrum, δ, ppm: 2.01–2.22 m (2H, CH_2CO), 2.24–2.26 m (1H, C**H**$_2$CH$_2$), 2.40–2.47 m (1H, C**H**$_2$CH$_2$), 3.15 s (3H, OCH_3), 3.56 s (3H, OCH_3), 3.49–3.60 m (3H, 1-H, 3-H, 4-H), 3.62 s (3H, OCH_3), 3.65 s (3H, OCH_3), 4.58 d.d (1H, 5-H, J = 6.1, 10.4 Hz), 7.36 d.d (1H, C_5H_4N, J = 4.9, 7.8 Hz), 7.71 d (1H, C_5H_4N, J = 8.0 Hz), 8.47 d (1H, C_5H_4N, J = 3.1 Hz), 8.51 d (1H, C_5H_4N, J = 1.8 Hz). Found, %: C 56.00; H 5.95; N 6.97. $C_{19}H_{24}N_2O_8$. Calculated, %: C 55.88; H 5.92; N 6.86.

Trimethyl (2*S,3*S**,4*S**,5*R**)-2-(3-methoxy-3-oxopropyl)-5-phenylpyrrolidine-2,3,4-tricarbox-**

ylate (VIIIa). Yield 74%, oily substance. ^{1}H NMR spectrum, δ, ppm: 1.91–1.95 m (2H, CH_2CO), 2.24–2.32 m (1H, C**H**$_2$CH$_2$), 2.39–2.46 m (1H, C**H**$_2$CH$_2$), 3.09 s (3H, OCH_3), 3.31 br.s (1H, 1-H), 3.57 s (3H, OCH_3), 3.66 s (3H, OCH_3), 3.75 m (1H), 3.79 s (3H, OCH_3), 3.82 m (1H), 4.71 t (1H, 5-H, J = 8.1 Hz), 7.26–7.32 m (5H, H_{arom}). ^{13}C NMR spectrum, δ_C, ppm: 13.98, 20.62, 27.67, 29.02, 51.25, 52.04, 52.70, 54.05, 59.69, 61.85, 69.13, 127.14, 127.93, 140.08, 170.29, 171.02, 172.96, 173.09. Found, %: C 58.88; H 6.17; N 3.64. $C_{20}H_{25}NO_8$. Calculated, %: C 58.96; H 6.19; N 3.44.

Trimethyl (2*S,3*S**,4*S**,5*R**)-5-(3,4-dimethoxyphenyl)-2-(3-methoxy-3-oxopropyl)pyrrolidine-2,3,4-tricarboxylate (VIIIb).** Yield 3%, oily substance. ^{1}H NMR spectrum, δ, ppm: 1.84–1.90 m (2H, CH_2CO), 2.23–2.31 m (1H, C**H**$_2$CH$_2$), 2.37–2.43 m (1H, C**H**$_2$CH$_2$), 3.18 s (3H, OCH_3), 3.28 d (1H, 1-H, J = 7.1 Hz), 3.56 s (3H, OCH_3), 3.64 s (3H, OCH_3), 3.70 m (1H), 3.72 s (3H, OCH_3), 3.73 s (3H, OCH_3), 3.75 m (1H), 3.77 s (3H, OCH_3), 4.63 t (1H, 5-H, J = 7.5 Hz), 6.77 d.d (1H, H_{arom}, J = 2.0, 8.3 Hz), 6.87 d (1H, H_{arom}, J = 8.3 Hz), 6.93 d (1H, H_{arom}, J = 2.0 Hz). Found, %: C 56.74; H 6.24; N 3.15. $C_{22}H_{29}NO_{10}$. Calculated, %: C 56.53; H 6.25; N 3.00.

Trimethyl (2*S,3*S**,4*S**,5*R**)-2-(3-methoxy-3-oxopropyl)-5-(pyridin-3-yl)pyrrolidine-2,3,4-tricarboxylate (VIIIc).** Yield 55%, oily substance. ^{1}H NMR spectrum, δ, ppm: 1.88–1.95 m (2H, CH_2CO), 2.41–2.45 m (1H, C**H**$_2$CH$_2$), 3.08 s (1H, C**H**$_2$CH$_2$), 3.12 s (3H, OCH_3), 3.58 s (3H, OCH_3), 3.65 s (3H, OCH_3), 3.77 s (3H, OCH_3), 3.80 s (1H, 4-H), 3.82 s (1H, 3-H), 4.79 t (1H, 5-H, J = 7.1 Hz), 7.34 d.d (1H, C_5H_4N, J = 4.8, 8.1 Hz), 7.72 d (1H, C_5H_4N, J = 7.8 Hz), 8.45 d (1H, C_5H_4N, J = 4.3 Hz), 8.50 d (1H, C_5H_4N, J = 1.5 Hz). Found, %: C 56.02; H 6.00; N 6.70. $C_{19}H_{24}N_2O_8$. Calculated, %: C 55.88; H 5.92; N 6.86.

Trimethyl (1*S,2*S**,3*R**,7a*S**)-5-oxo-3-phenyltetrahydropyrrolizine-1,2,7a-tricarboxylate (IXa).** Yield 12%, colorless crystals, mp 118–120°C. ^{1}H NMR spectrum, δ, ppm: 2.00–2.09 m (1H, 7-H), 2.26–2.33 m (2H, 6-H), 2.71–2.80 m (1H, 7-H), 3.13 s (3H, OCH_3), 3.72 s (3H, OCH_3), 3.78 s (3H, OCH_3), 4.01 d.d (1H, 1-H, J = 5.3, 8.8 Hz), 4.07 d (1H, 2-H, J = 5.6 Hz), 5.35 d (1H, 3-H, J = 8.8 Hz), 7.24–7.31 m (5H, H_{arom}). ^{13}C NMR spectrum, δ_C, ppm: 30.89, 32.49, 50.64, 51.41, 52.30, 52.99, 53.11, 61.08, 74.06, 126.64, 127.93, 137.45, 170.00, 170.65, 172.85, 176.61. Found, %: C 60.75; H 5.62; N 3.93. $C_{19}H_{21}NO_7$. Calculated, %: C 60.79; H 5.64; N 3.73. X-Ray diffraction data: triclinic crystals; a = 12.068(2), b = 12.499(3), c = 12.966(2) Å; α = 84.47(2), β = 72.02(1), γ = 88.21(2)°; V = 1851.6(6) Å^3; space group P-1; d_{calc} = 1.347 g/cm^3. The complete set of crystallographic parameters is available from the authors.

Trimethyl (1*S,2*S**,3*R**,7a*S**)-3-(3,4-dimethoxyphenyl)-5-oxotetrahydropyrrolizine-1,2,7a-tricarboxylate (IXb).** Yield 71%, colorless crystals, mp 170–172°C. ^{1}H NMR spectrum, δ, ppm: 2.01–2.07 m (1H, 7-H), 2.25–2.31 m (2H, 6-H), 2.71–2.78 m (1H, 7-H), 3.23 s (3H, OCH_3), 3.69 s (3H, OCH_3), 3.70 s (3H, OCH_3), 3.71 s (3H, OCH_3), 3.79 s (3H, OCH_3), 3.95 d.d (1H, 1-H, J = 6.6, 8.8 Hz), 4.11 d (1H, 2-H, J = 6.6 Hz), 5.28 d (1H, 3-H, J = 8.6 Hz), 6.72 d.d (1H, H_{arom}, J = 1.5, 8.1 Hz), 6.80 d (1H, H_{arom}, J = 2.0 Hz), 6.86 d (1H, H_{arom}, J = 8.3 Hz). Found, %: C 57.88; H 5.86; N 3.14. $C_{21}H_{25}NO_9$. Calculated, %: C 57.93; H 5.79; N 3.22.

Trimethyl (1*S,2*S**,3*R**,7a*S**)-5-oxo-3-(pyridin-3-yl)tetrahydropyrrolizine-1,2,7a-tricarboxylate (IXc).** Yield 13%, oily substance. ^{1}H NMR spectrum, δ, ppm: 1.99–2.08 m (1H, 7-H), 2.25–2.34 m (2H, 6-H), 2.71–2.78 m (1H, 7-H), 3.07 s (3H, OCH_3), 3.72 s (3H, OCH_3), 3.78 s (3H, OCH_3), 3.98 d (1H, 1-H, J = 3.5 Hz), 4.06 d.d (1H, 2-H, J = 3.7, 8.8 Hz), 5.37 d (1H, 3-H, J = 9.1 Hz), 7.31 d.d (1H, C_5H_4N, J = 4.8, 7.8 Hz), 7.71 d.t (1H, C_5H_4N, J = 1.8, 8.1 Hz), 8.43 d.d (1H, C_5H_4N, J = 1.4, 4.8 Hz), 8.52 d (1H, C_5H_4N, J = 2.0 Hz). Found, %: C 57.58; H 5.39; N 7.43. $C_{18}H_{20}N_2O_7$. Calculated, %: C 57.44; H 5.36; N 7.44.

Methyl 4-ethenylsulfonyl-5-[2-(1,3-dioxo-2,3-dihydro-1*H*-isoindol-2-yl)-3-methoxy-3-oxopropyl]-2-(3-methoxy-3-oxopropyl)pyrrolidine-2-carboxylate (XVIII/XIX) (mixture of isomers)**.** Yield 19%, colorless crystals, mp 73–75°C, $[\alpha]_D^{20}$ = –15.0° (c = 1.0, $CHCl_3$). ^{1}H NMR spectrum, δ, ppm: major isomer: 1.62–1.78 m (2H), 1.79–1.88 m (2H), 2.09–2.18 m (1H), 2.29–2.37 m (1H), 2.45 m (1H), 2.93 d.t (1H, 8-H, J = 4.8, 14.5 Hz), 3.45 m (1H), 3.54 s (3H, OCH_3), 3.55 s (3H, OCH_3), 3.66 s (3H, OCH_3), 3.67 m (1H), 5.17 d.d (1H, 9-H, J = 4.8, 9.0 Hz), 6.17 d (1H, CH=C**H**$_2$, J = 16.5 Hz), 6.28 d (1H, CH=C**H**$_2$, J = 10.0 Hz), 6.97 d.d (1H, C**H**=CH$_2$, J = 10.0, 16.5 Hz), 7.91 m (4H, H_{arom}). Found, %: C 53.72; H 5.24; N 5.26. $C_{24}H_{28}N_2O_{10}S$. Calculated, %: C 53.72; H 5.26; N 5.22.

Methyl (*S*)-4-methylsulfanyl-2-phthtalimidobutanoate (XV). A mixture of 49.94 g (0.25 mol) of L-methionine methyl ester hydrochloride, 37.03 g (0.25 mol) of phthalic anhydride, 106.65 g (0.72 mol)

of triethylamine, and 1 l of toluene was heated with stirring under reflux in a flask equipped with a Dean–Stark trap until water no longer separated. The mixture was cooled, the precipitate was filtered off, the filtrate was washed with 300 ml of 1 N hydrochloric acid and water (3×300 ml) and dried over $MgSO_4$, and the solvent was removed under reduced pressure. Yield 81%, mp 37–40°C. ^{1}H NMR spectrum, δ, ppm: 2.02 s (3H), 2.42–2.28 m (2H), 2.61–2.47 m (2H), 3.66 s (3H), 5.05 d.d (1H, J = 5.5, 8.9 Hz), 7.92 m (4H).

Methyl (*S*)-4-oxo-2-phthalimidobutanoate (XVI). Compound **XV**, 31.1 g (0.106 mol), was dissolved in 150 ml of carbon tetrachloride, 14.15 g (0.106 mol) of *N*-chlorosuccinimide was added in one portion, and the mixture was stirred for 2 h in an inert atmosphere. It was then filtered under an inert gas, the precipitate was washed with carbon tetrachloride (3×30 ml), the washings were combined with the filtrate, 640 ml of water was added, and argon was bubbled through the mixture under stirring over a period of 20 h, the liberated gases being absorbed by concentrated nitric acid. The aqueous phase was separated and extracted with methylene chloride (3×100 ml). The extracts were combined with the organic phase, washed with a saturated solution of $NaHCO_3$ and with water, dried over Na_2SO_4, and evaporated under reduced pressure. The residue was subjected to chromatography on silica gel (gradient elution with petroleum ether–ethyl acetate, 10:1, 5:1, 1:1). Yield 27%, $[\alpha]_D^{20}$ = –30.0° (c = 1.49, $CHCl_3$), optical purity 62.2%; published data [14]: $[\alpha]_D^{20}$ = –44.4° (c = 1.49, $CHCl_3$), optical purity 92.0%. ^{1}H NMR spectrum, δ, ppm: 3.27 d.d (1H, J = 7.8, 18.3 Hz), 3.55 d.d (1H, J = 6.3, 18.3 Hz), 3.74 s (3H), 5.50 t (1H, J = 7.0 Hz), 7.72–7.95 m (4H), 9.80 s (1H).

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