

Study on the Lewis Acid-promoted Aza-Diels – Alder Reaction of Azetidin-2-one-tethered Imines with Siloxydienes in the Asymmetric Synthesis of 2-Aryl(alkyl)-2,3-dihydro-4-pyridones

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RECEIVED AUGUST 8, 2007; REVISED OCTOBER 31, 2007; ACCEPTED NOVEMBER 21, 2007

Keywords

β -lactam
imine
pyridone
aza-Diels – Alder reaction
Lewis acid

trans-3-Amino- β -lactams were evaluated as the chiral building blocks in the aza-Diels – Alder reaction of azetidin-2-one-tethered imines with siloxydienes under Lewis acid catalysis, as a route for the asymmetric synthesis of 2-aryl(alkyl)-2,3-dihydro-4-pyridones.

INTRODUCTION

Diversely substituted monocyclic β -lactams occupy a central place among medicinally important compounds due to their diverse and interesting antibiotic activities. Consequently their synthesis has been of considerable interest to the synthetic community in the past few decades.^{1,2} Because of the recent developments using β -lactams as synthons for several biologically active compounds, research on this topic has gained tremendous attention.^{3–6} Hetero Diels – Alder reactions involving imino-dienes or imino-dienophiles are widely used for the construction of nitrogen-containing compounds.^{7,8} Our interest in the use of *trans*-3-amino- β -lactams,^{9,10} as starting substrates, for the preparation of potentially bioactive products prompted us to evaluate the combination of aza-Diels – Alder reaction of azetidin-2-one-tethered imines **1** with siloxydienes **2** as a route to the asymmetric syn-

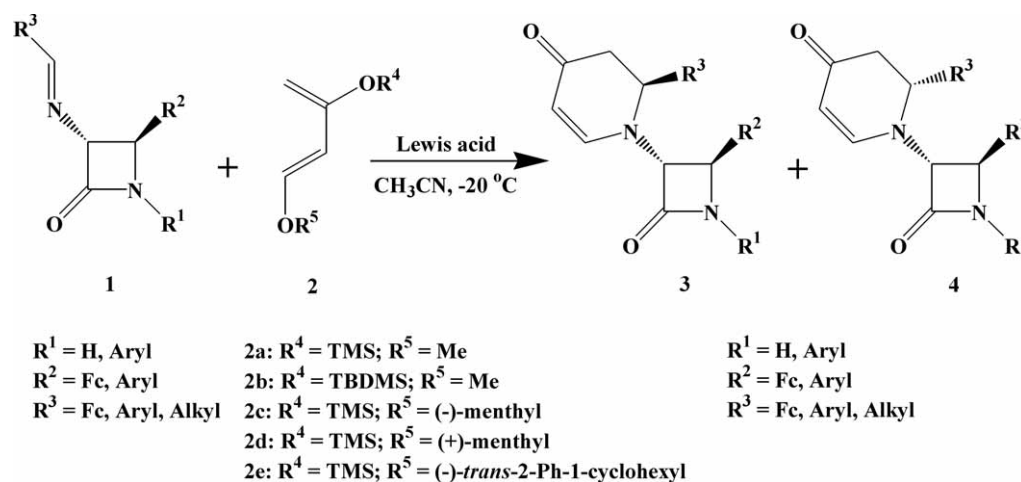
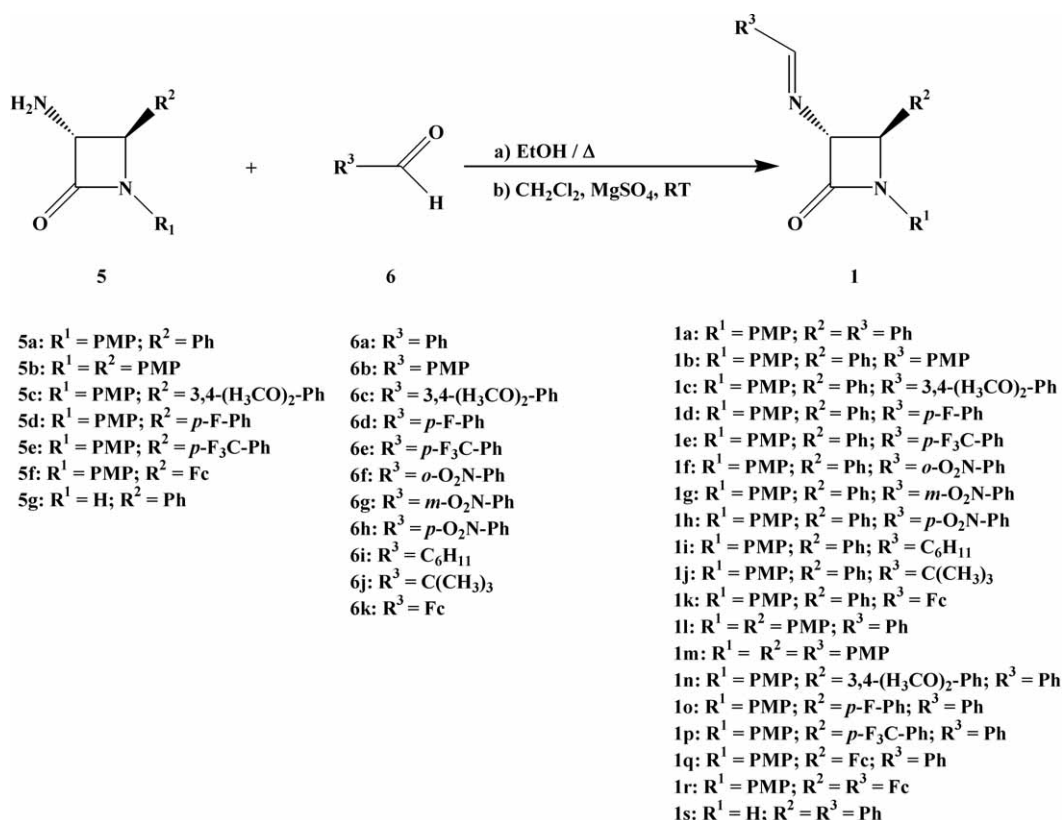
thesis of 2-aryl(alkyl)-2,3-dihydro-4-pyridones **3** and **4**, which are interesting heterocycles and attractive building blocks for alkaloid synthesis^{11–19} (Scheme 1).

RESULTS AND DISCUSSION

We applied lithium chiral ester enolate-imine condensation strategy,^{20–22} synthetic methodology based on the azetid-2-one nucleus: the β -lactam synthon method,^{23,24} to the asymmetric synthesis of *trans*-3-amino- β -lactams^{9,10} **5**. Treatment of *trans*-3-amino- β -lactams **5a–g** with a variety of aldehydes **6a–k** in boiling ethanol followed by stirring in dichloromethane at room temperature in the presence of sodium sulfate provided the corresponding imines **1a–s** (Scheme 2). Aryl and ferrocenyl imines **1a–h** and **1k–s** (isolated yields vary from 59 to 91 %) were purified by recrystallization, while alkyl imines **1i–j** (¹H NMR spectra calculated yields: 65 and 60 %) were generated

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Scheme 1. Preparation of **3** and **4**.Scheme 2. Preparation of imines **1a-s**.

in dichloromethane at room temperature, filtered, evaporated to dryness and as such applied in the next reaction step.

1-Methoxy-3-trimethylsilyloxy-1,3-butadiene¹¹ **2a** (Danishefsky's diene; TMS = trimethylsilyl) and *trans*-3-(*tert*-butyl-dimethylsilyloxy)-1-methoxy-1,3-butadiene¹² **2b** (TBDMS = *tert*-butyl-dimethylsilyl) were used in Diels – Alder reaction with azetidino-2-one-tethered imines **1a-s** as dienophiles. First we studied the

aza-Diels – Alder reaction of imine **1a** with Danishefsky's diene **2a** in the presence of equimolar ($x = 100\%$, mole fraction) and a catalytic ($x = 20\%$) amount of various catalysts. The cycloaddition took place at low temperature ($-20\text{ }^\circ\text{C}$) in acetonitrile under Lewis acid catalysis. Diastereoselectivities were reasonable but the chemical yield of the process depended on the nature of the Lewis acid (Table I). We found that zinc(II) iodide and indium(III) chloride (Table I, entries 1–4) provided the

TABLE I. Lewis acid mediated Diels – Alder cycloaddition between (3*R*,4*R*)-**1a** and **2a**

Entry	Lewis acid	mole fraction	3a/4a Ratio ^(a)	Yield 3a/4a
1	ZnI ₂	20	68 : 32	92
2	ZnI ₂	100	66 : 34	78
3	InCl ₃	20	66 : 34	85
4	InCl ₃	100	66 : 34	85
5	LiClO ₄	20	67 : 33	44
6	LiClO ₄	100	68 : 32	36
7	AlCl ₃	20	62 : 38	57
8	AlCl ₃	100	63 : 37	67
9	TiCl ₄	20	62 : 38	34
10	TiCl ₄	100	60 : 40	34
11	In(OTf) ₃	20	64 : 36	31
12	In(OTf) ₃	100	–	–

^(a)The diastereomeric ratio was determined by integration of well-resolved signals in the ¹H NMR spectra of the crude reaction mixtures before purification and confirmed by RP-HPLC (Figure 1).

best chemical yields combined with the highest diastereoselectivity and decided to continue our study with a variety of imines **1** and dienes **2a-b** with zinc(II) iodide (*x* = 20 %) in acetonitrile at –20 °C. The results are presented in Table II. We studied the influence of various groups on imine (R¹ = H, PMP; R² = aryl, ferrocenyl;

R³ = alkyl, aryl, ferrocenyl) and on diene (TMS, TBDMS) part on diastereomeric ratio and product yield of the reaction. Displacement of PMP group with H at the R¹ position (Table II, entries 1, 20) did not influence on the diastereomeric ratio but yield significantly dropped. Combination of a variety of the substituents (Table II, entries 1-8) on phenyl ring at the R³ position with TBDMS group in **2b** generated increase in product yield and diastereomeric ratio, exhibiting the best diastereomeric ratio (78:22) with R³ = 4-fluoro- and 4-nitro-phenyl (entries 4, 8). Displacement of aryl with alkyl-substituent (R³ = cyclohexyl, *tert*-butyl; entries 9, 10) resulted in significant drop in product yield and diastereomeric ratio especially with less bulky *tert*-butyl group. The change of the substituents on phenyl ring at the R² position (entries 10-16) compared with the same change at the R³ position (entries 1-8) created more significant drop in product yield than in diastereomeric ratio. Introducing ferrocenyl group at the R³ position (entry 11) resulted with the best diastereomeric ratio (85:15) and the lowest yield (11 %) in product formation. The best results in yield (84 % and 79 %) and diastereomeric ratio (80:20) were obtained with ferrocenyl group at the R² position (entries 17 and 18) *vs.* no reaction with ferrocenyl group at both R² and R³ positions (Table II, entry 19). In order to improve diastereomeric ratio of the reaction, we replaced^{26,27} methyl at the C1 position of Danishefsky's diene **2a** with (1*R*,2*S*,5*R*)-(–)-menthyl (diene – **2c**), (1*S*,2*R*,5*S*)-(+)-menthyl (**2d**), and (1*R*,2*S*)-(–)-*trans*-2-phenyl-1-cyclohexyl (**2e**) introducing the new chiral informations

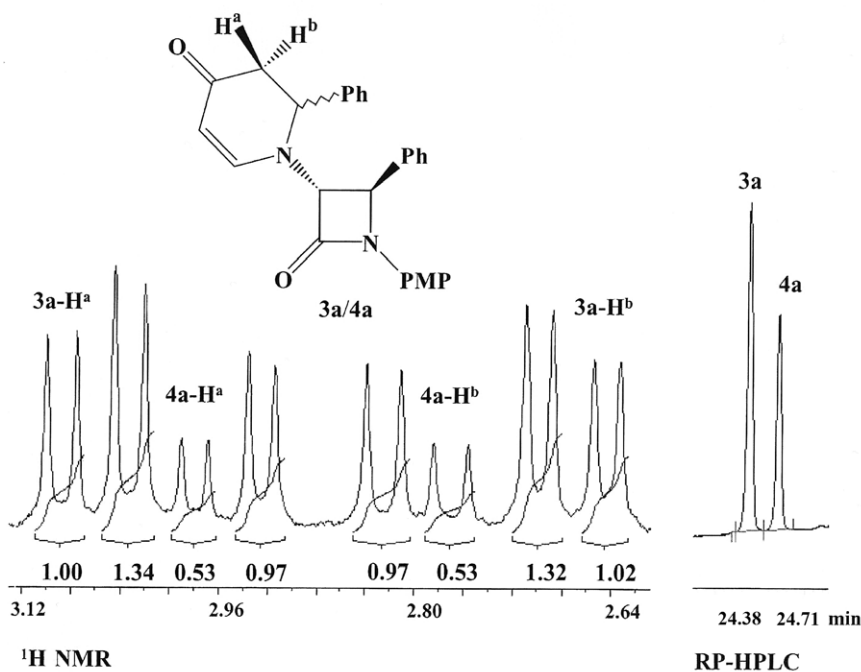


Figure 1. ¹H NMR spectra of C3 H^a and H^b well-resolved proton signals on pyridone ring and RP-HPLC profile of **3a/4a** diastereomeric mixture.

TABLE II. Lewis acid mediated Diels – Alder cycloaddition between imines **1a-s** and dienes **2a-b**

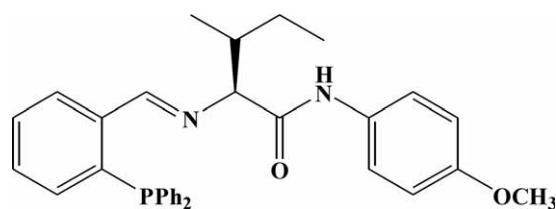
Entry	Imine	Diene	R ¹	R ²	R ³	3/4 Ratio	Yield 3/4		
1	1a	2a	PMP ^(a)	Ph ^(b)	Ph	3a/4a	<i>R,R,R</i> : <i>S,R,R</i>	68 : 32	92
		2b						69 : 31	89
2	1b	2a	PMP	Ph	PMP	3b/4b		66 : 34	77
		2b						72 : 28	87
3	1c	2a	PMP	Ph	3,4-(H ₃ CO) ₂ -Ph	3c/4c		70 : 30	92
		2b						70 : 30	91
4	1d	2a	PMP	Ph	<i>p</i> -F-Ph	3d/4d		70 : 30	81
		2b						78 : 22	98
5	1e	2a	PMP	Ph	<i>p</i> -F ₃ C-Ph	3e/4e		73 : 27	77
6	1f	2b	PMP	Ph	<i>o</i> -O ₂ N-Ph	3f/4f		73 : 27	59
7	1g	2b	PMP	Ph	<i>m</i> -O ₂ N-Ph	3g/4g		73 : 27	85
8	1h	2b	PMP	Ph	<i>p</i> -O ₂ N-Ph	3h/4h		78 : 22	97
9	1i	2a	PMP	Ph	C ₆ H ₁₁	3i/4i		60 : 40	39
		2b						67 : 33	64
10	1j	2a	PMP	Ph	C(CH ₃) ₃	3j/4j		55 : 45	44
11	1k	2a	PMP	Ph	Fc	3k/4k		85 : 15	11
		2b						67 : 33	49
12	1l	2b	PMP	PMP	Ph	3l/4l		68 : 32	58
13	1m	2a	PMP	PMP	PMP	3m/4m		68 : 32	55
14	1n	2a	PMP	3,4-(H ₃ CO) ₂ -Ph	Ph	3n/4n		66 : 34	76
		2b						66 : 34	79
15	1o	2b	PMP	<i>p</i> -F-Ph	Ph	3o/4o		72 : 28	89
16	1p	2b	PMP	<i>p</i> -F ₃ C-Ph	Ph	3p/4p		74 : 26	59
17	1q	2a	PMP	Fc ^(c)	Ph	3q/4q		80 : 20	84
		2b						78 : 22	85
18	1q	2a	PMP	Fc	Ph	3q/4q	<i>S,S,S</i> : <i>R,S,S</i>	80 : 20	79
		2b						–	–
19	1r	2a	PMP	Fc	Fc	3r/4r		–	–
		2b						–	–
20	1s	2a	H ^(d)	Ph	Ph	3s/4s	<i>R,R,R</i> : <i>S,R,R</i>	65 : 35	44
		2b						66 : 34	50

^(a)PMP = 4-Methoxy-phenyl; ^(b)Ph = Phenyl; ^(c)Fc = Ferrocenyl; ^(d)PMP group in (3*R*,4*R*)-**1a** was removed with ceric ammonium nitrate²⁵

on the diene part. The reaction with dienes **2c-d** was performed in the presence of zinc(II) iodide ($x = 20$ %) as catalyst in acetonitrile at -20 °C and atmospheric pressure (Table III, entries 1, 4, and 7). The reaction proceeded slower, in 72 h only 40 % of mixture of **3a/4a** was formed, but diastereomeric ratio significantly increased from 68:32 (Table II, entry 1) to 86:14 (Table III, entries 1 and 4). No difference in diastereomeric ratio was observed with dienes **2c** and **2d** employing (+)-*vs.* (–)-menthol, it seems diastereoselectivity is rather regulated with menthol's steric effect than chiral information. Furthermore, in order to improve the reaction yield, we combined increased pressure (8 kbar) at room temperature and 100 °C (Table III, entries 2, 3; 5, 6 and 8, 9). The reaction yield increased from 63 % (room tem-

perature, 12 h) to almost quantitative (96 %, 100 °C, 12 h), while diastereomeric ratio little dropped to 83:17 at room temperature, and more significantly dropped at 100 °C (75:25) (Table III, entries 2, 3 and 5, 6).

We also investigated in the literature-described^{28–31} chiral ligands (*e.g.* commercially available (*R*)-, (*S*)-BINOL, 2,2'-*iso*-propylidenebis[(4*S*)-4-*tert*-butyl]-2-oxazoline and **7** (Figure 2) prepared³¹ from amino acid L-Ile) in combination with a variety of Lewis acids (*e.g.* indium(III) chloride, titanium(IV) isopropoxide, triphenyl borate, copper(II) trifluoromethanesulfonate, and copper(I) trifluoromethanesulfonate benzene complex) in the Diels – Alder cycloaddition between imine **1a** and diene **2b**. Although under similar conditions these chiral ligands exhibited extremely high asymmetric induction, the

Figure 2. Structure of chiral ligand **7**.

observed diastereoselectivities remained reasonable (**3a/4a** diastereomeric ratios varied from 56:44 to 67:33) in our case.

Crystal Structure Determination of (2*S*,3'*S*,4'*S*)-**3q**

The crystal structure of (2*S*,3'*S*,4'*S*)-**3q** was determined in order to establish unambiguously both absolute and relative configuration at the stereogenic center C21. The *S* absolute configurations of two stereogenic centers at the azetidin-2-one ring, C11 and C12 were determined previously,¹⁰ and in relation to them, the absolute configuration of C21 was also found to be *S*. Ferrocenyl moiety and the pyridone ring (N2-C21-C22-C23-C24-C25) are in *trans*-position with respect to the C11-C12 bond with torsion angle N2-C12-C11-C1 being 118.4(12) ° (Figure 3). The geometrical parameters of the azetidin-2-one ring agree well with the average values¹⁰ extracted from the Cambridge Structural Database.³² Conformation of the pyridone ring is a half-chair with Cremer-Pople³³ parameters $Q = 0.406$ (14) Å, $\Theta = 58(2)$ °, $\varphi = 88(2)$ °. The phenyl ring bound to C12 and the *p*-methoxyphenyl ring bound to N1 are almost perfectly planar (Figure 3).

Since there are no strong proton donors, the crystal packing of compound (2*S*,3'*S*,4'*S*)-**3q** is dominated by weak C-H...O hydrogen bonds forming double layers parallel with [001] plane (Figure 4). C-H... π interactions link the layers into a 3D network. C20 interacts with both phenyl rings *via* H20B and H20C atoms (symmetry

operator is in both cases 1-*x*, 1/2+*y*, 2-*z*). There are no π ... π interactions between ferrocenyl moieties and/or phenyl rings (Figure 4).

Molecular Modelling

Semiempirical PM3 calculation method^{34,35} was used to model Diels – Alder reaction of dienes with azetidin-2-one-tethered imines catalyzed by Lewis acids. FMO analysis has revealed that the preferred regioselectivity is governed by orbital symmetry and the size of coefficients. It was assumed that for Lewis acid catalysis (using zinc(II) iodide and indium(III) triflate as a model compounds), azetidin-2-one-tethered imine acts as a bidentate ligand, coordinating with metal *via* imine nitrogen and carbonyl oxygen. Such a metal coordination determines geometrical relationship between azetidin-2-one and imine unit, while C4 phenyl and N1 *p*-methoxyphenyl substituents on azetidin-2-one ring freely rotate. PM3 modelling has shown the preferential in-plane orientation of *p*-methoxyphenyl group (in respect to azetidin-2-one ring), while phenyl group is oriented almost perpendicularly. Restricted rotation of imine double bond yields two rotamers, while phenyl substituent on the imine bond freely rotates. These geometrical arrangements of the substituents and Lewis acid predetermine possible modes of the diene approach and the consequence of cycloadditions. There is a slight preference to diene approach from the top side yielding *R*-isomer, while diene approach from the bottom side leading to *S*-isomer is less preferred, (Figure 5). These computational predictions are in good agreement with experimental results, where in the most of Lewis acid catalyzed reactions, *R*:*S* ratio \approx 70:30 is found (Table II).

EXPERIMENTAL

Melting points were determined on a Reichert Thermovar 7905 apparatus and were not corrected. The IR spectra were

TABLE III. The diastereomeric ratio and yield dependence on reaction temperature and pressure in Lewis acid mediated Diels – Alder cycloaddition between imine **1a** and dienes **2c-e**

Entry	Diene	<i>t</i> /h	<i>T</i> /°C	p/kbar	3a/4a Ratio	Yield 3a/4a
1		72	-20-RT ^(a)	1	86 : 14	40
2	2c	12	RT	8	83 : 17	63
3		12	100	8	75 : 25	96
4		72	-20-RT	1	86 : 14	39
5	2d	12	RT	8	81 : 19	64
6		12	100	8	71 : 29	95
7		72	-20-RT	1	79 : 21	51
8	2e	12	RT	8	73 : 27	74
9		12	100	8	68 : 32	99

^(a)RT = room temperature

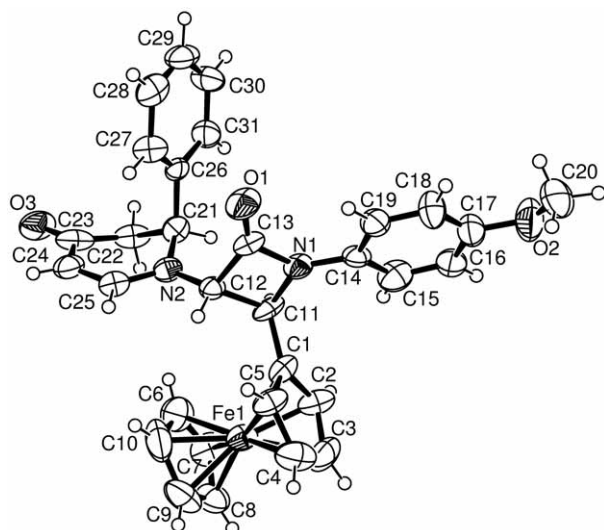


Figure 3. ORTEP drawing of compound (2*S*,3*S*,4*S*)-**3q**. Thermal ellipsoids are drawn at 50 % probability.

recorded on a Perkin Elmer Spectrum RX I FT-IR System spectrometer (KBr pellets technique) (PerkinElmer Instruments, Norwalk, CT, USA). The ^1H and ^{13}C NMR spectra (in CDCl_3 at room temperature) were measured on a Bruker AV 300 and/or AV 600 spectrometer (Bruker BioSpin GmbH., Rheinstetten, Germany), δ in ppm relative to tetramethylsilane as the internal reference. Microanalyses were performed on a PE 2400 Series II CHNS/O Analyzer (PerkinElmer Instruments, Shelton, CT, USA). Optical rotations: Automatic Polarimeter AA-10 in a 1 dm cell; c in g/100 mL (Optical Activity Ltd., Ramsey, England). HPLC analyses were performed on a HPLC System (Dr ing. Herbert Knauer GmbH., Berlin, Germany) supplied with UV/VIS WellChrom Diode Array Detector K-2800 using Waters Nova-Pak 4 μm -Spherical C18 (reversed phase) 3.9×150 mm HPLC Column (Waters Corporation, Milford, MA, USA) operated at a room temperature and a flow rate 1 mL/min; linear gradient of water containing 0.1 % trichloroacetic acid (solvent A) and methanol (solvent B); 100 % A, 5 min; 80 % A + 20 % B, 5 min; 50 % A + 50 % B, 10 min; 20 % A + 80 % B, 10 min; 100 % B, 5 min. Column chromatography on silica gel 60, 70–230 mesh, 60 Å (E. Merck, Darmstadt, Germany) was performed at room temperature. Thin layer chromatography was carried out on TLC aluminium sheets, 20 \times 20 cm, silica gel 60 F₂₅₄ and preparative thin layer chromatography on PLC plates, 20 \times 20 cm, silica gel 60 F₂₅₄, 2 mm (E. Merck). High pressure reaction was performed using the high pressure piston-cylinder apparatus, in teflon cells and petroleum ether as piezo-transmitter liquid. High resolution, positive ion mass spectra were recorded on a FT/MS 2001-DD Fourier transform mass spectrometer (Finnigan, Madison, WI, USA) equipped with a 3 T superconducting magnet and a Nicolet 1280 data station, using PFTBA as standard for external calibration. LC-MS spectra were recorded on a Hewlett Packard HP-1100 Series System (Hewlett Packard, Palo Alto, CA, USA) equipped with a binary solvent pump, an auto-

sampler (volume injection set to 20 μL), and a mass selective detector (HP MSD) with electrospray ionization (ESI) using Macherey-Nagel 5 μm Kromasil C18 4.0×250 mm HPLC column (Macherey-Nagel GmbH & Co. KG, Düren, Germany) operated at room temperature and a flow rate 1 mL/min; linear gradient of water containing 0.1 % formic acid (solvent A) and methanol (solvent B); 60 % A + 40 % B, 0 min; 5 % A + 95 % B, 10 min; 5 % A + 95 % B, 5 min; 60 % A + 40 % B, 2 min. Operating conditions of the ESI interface in positive ion mode were: nebulizer gas (nitrogen) pressure 25 psig, drying gas (nitrogen) flow rate 10 L/min, drying gas temperature 350 $^\circ\text{C}$, capillary voltage 4000 V. Crystal structure measurements were performed on an Enraf Nonius CAD4 diffractometer (Bruker-Nonius, Delft, Netherlands), using a graphite monochromated Cu K_α (1.54179 Å) radiation at room temperature [293(2) K].

Geometrical optimizations were carried out employing PM3 semiempirical method within Gaussian03 suite of programs, implemented on dual core Opteron 240 personal computer under Linux OS. Full optimizations were followed by vibrational analysis to verify that the obtained structures are true minima on the potential surface.

Preparation of Azetidin-2-one-tethered Imines **1a-s**

General Procedure. – A mixture of *trans*-3-amino- β -lactams **5a-g** and aldehydes **6a-h** and **6k** was dissolved in anhydrous ethanol (5.0 mL), heated under reflux for 12 h and cooled; evaporated to dryness, redissolved in dichloromethane (5.0 mL) and stirred at room temperature over anhydrous sodium sulfate (100.0 mg) for additional 30 minutes. Sodium sulfate was filtered off, washed with dichloromethane (3 \times 5 mL) and filtrate evaporated to dryness.

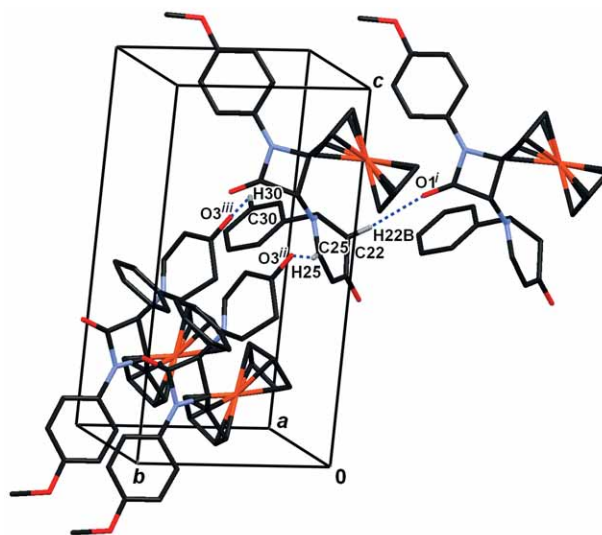


Figure 4. Crystal packing of compound (2*S*,3*S*,4*S*)-**3q**. Hydrogen bonded chains C22-H22B \cdots O1^{*i*} run in [010] direction and the 3D network is completed by C-H \cdots π interactions. Hydrogen atoms not participating in hydrogen bonds have been omitted for clarity. Symmetry codes: *i*) $x, -1+y, z$; *ii*) $-x, 1/2+y, 2-z$; *iii*) $1-x, 1/2+y, 2-z$.

Imines **1a-h**, **1k-q** and **1s** were recrystallized from a proper solvent.

(3*R*,4*R*)-3-Benzylideneamino-1-(4-methoxyphenyl)-4-phenylazetidin-2-one (**1a**). – Obtained from **5a** (150.0 mg, 5.59×10^{-1} mmol) and **6a** (59.3 mg, 5.59×10^{-1} mmol) as white crystals (from dichloromethane-petroleum ether (b.p. 40–70 °C)), 147.7 mg (74 %), $R_f = 0.25$ (dichloromethane), m.p. 181–182 °C; $[\alpha]_D = +275.3$ ($c = 0.46$ g/100 mL dichloromethane); IR (KBr) $\nu_{\max}/\text{cm}^{-1}$: 3468, 2885, 2839, 1740, 1638, 1511, 1246, 695; ^1H NMR (CDCl_3) δ/ppm : 3.74 (s, 3H, OCH_3), 4.66 (m, 1H, C4, β -lactam), 5.18 (d, 1H, $J = 1.84$ Hz, C3, β -lactam), 6.80 (d, 2H, $J = 9.03$ Hz, C3 and C5, PMP), 7.29 (d, 2H, $J = 9.03$ Hz, C2 and C6, PMP), 7.34–7.45 (m, 8H, Ph) 7.77–7.80 (m, 2H, Ph), 8.40 (s, 1H, $\text{CH}=\text{N}$); ^{13}C NMR (CDCl_3) δ/ppm : 55.37 (OCH_3), 63.50 (C4, β -lactam), 83.59 (C3, β -lactam), 114.27 (C3 and C5, PMP), 118.72 (C2 and C6, PMP), 126.25 (C2 and C6, Ph), 128.59 (C4, Ph), 128.62 (C2 and C6, $\text{PhC}=\text{N}$), 129.16 (C3 and C5, Ph), 130.84 (C1, Ph), 131.44 (C3 and C5, $\text{PhC}=\text{N}$), 135.39 (C1, $\text{PhC}=\text{N}$), 136.63 (C1, PMP), 156.20 (C4, PMP), 163.21 (CO), 164.75 ($\text{CH}=\text{N}$). Anal. Calcd. for $\text{C}_{23}\text{H}_{20}\text{N}_2\text{O}_2$ ($M_r = 356.43$): C 77.51, H 5.66, N 7.86 %; found: C 77.80, H 5.40, N 8.10 %.

(3*R*,4*R*)-3-(4-Methoxybenzylideneamino)-1-(4-methoxyphenyl)-4-phenylazetidin-2-one (**1b**). – Obtained from **5a** (111.0 mg, 4.14×10^{-1} mmol) and **6b** (56.4 mg, 4.14×10^{-1} mmol) as yellow crystals (from dichloromethane-petroleum ether (b.p. 40–70 °C)), 135.0 mg (85 %), $R_f = 0.25$ (dichloromethane), m.p. 132–135 °C; $[\alpha]_D = +309.7$ ($c = 0.20$ g/100 mL dichloromethane); IR (KBr) $\nu_{\max}/\text{cm}^{-1}$: 3471, 1743, 1638, 1575, 1511, 1456, 1396, 1249, 1168, 1115, 838; ^1H NMR (CDCl_3) δ/ppm : 3.74 (s, 3H, OCH_3), 3.84 (s, 3H, OCH_3), 4.61 (d, 1H, $J = 0.60$ Hz, C4, β -lactam), 5.15 (s, 1H, C3, β -lactam), 6.79 (d, 2H, $J = 9.00$ Hz, C3 and C5, PMP), 6.92 (d, 2H, $J = 8.64$ Hz, C3 and C5, PMP), 7.28 (d, 2H, $J = 9.00$ Hz, C2 and C6, PMP), 7.30–7.40 (m, 5H, Ph), 7.72 (d, 2H, $J = 8.70$ Hz, C2 and C6, PMP), 8.31 (s, 1H, $\text{CH}=\text{N}$); ^{13}C NMR (CDCl_3) δ/ppm : 55.35 (OCH_3), 55.40 (OCH_3), 63.71 (C4, β -lactam), 83.70 (C3, β -lactam), 114.05 (C3 and C5, PMP), 114.33 (C3 and C5, PMP), 118.75 (C2 and C6, PMP), 126.27 (C2 and C6, PMP), 128.40 (C1, Ph), 128.59 (C4, Ph), 129.13 (C2 and C6, Ph), 130.29 (C3 and C5, Ph), 131.01 (C1, PMP), 136.88 (C1, PMP), 156.24 (C4, PMP), 162.28 (C4, PMP), 163.61 (CO), 163.89 ($\text{CH}=\text{N}$). Anal. Calcd. for $\text{C}_{24}\text{H}_{22}\text{N}_2\text{O}_3$ ($M_r = 386.45$): C 74.59, H 5.74, N 7.25 %; found: C 74.46, H 5.55, N 7.45 %.

(3*R*,4*R*)-3-(3,4-Dimethoxybenzylideneamino)-1-(4-methoxyphenyl)-4-phenylazetidin-2-one (**1c**). – Obtained from **5a** (100.0 mg, 3.73×10^{-1} mmol) and **6c** (61.9 mg, 3.73×10^{-1} mmol) as yellow crystals (from dichloromethane-petroleum ether (b.p. 40–70 °C)), 117.9 mg (76 %), $R_f = 0.25$ (dichloromethane), m.p. 180–182 °C; $[\alpha]_D = +316.0$ ($c = 0.20$ g/100 mL dichloromethane); IR (KBr) $\nu_{\max}/\text{cm}^{-1}$: 3855, 1736, 1509, 1458, 1243, 1155, 1027; ^1H NMR (CDCl_3) δ/ppm : 3.75 (s, 3H, OCH_3), 3.93 (s, 3H, OCH_3), 3.95 (s, 3H, OCH_3), 4.63 (bs, 1H, C4, β -lactam), 5.16 (d, 1H, $J = 1.38$

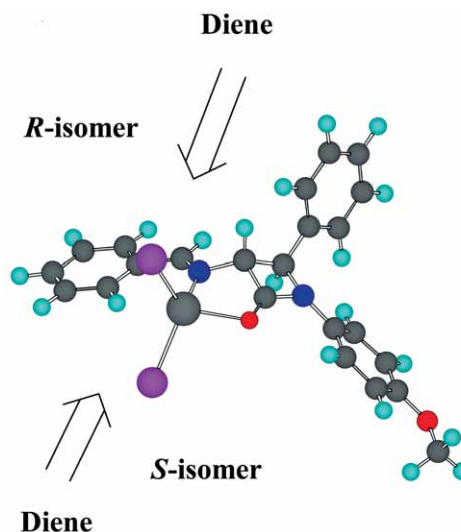


Figure 5. PM3 optimized structure of complex of zinc(II) iodide with azetidin-2-one-tethered imine **1a**.

Hz, C3, β -lactam), 6.80 (d, 2H, $J = 9.00$ Hz, C3 and C5, PMP), 6.88 (d, 1H, $J = 8.22$ Hz, C6, 3,4-(CH_3O) $_2$ -Ph), 7.18 (d, 1H, $J = 8.22$ Hz, C5, 3,4-(CH_3O) $_2$ -Ph), 7.29 (d, 2H, $J = 8.94$ Hz, C2 and C6, PMP), 7.30–7.40 (m, 5H, Ph), 7.49 (d, 1H, $J = 1.38$ Hz, C2, 3,4-(CH_3O) $_2$ -Ph), 8.30 (s, 1H, $\text{CH}=\text{N}$); ^{13}C NMR (CDCl_3) δ/ppm : 55.42 (OCH_3), 55.97 ($2 \times \text{OCH}_3$), 63.68 (C4, β -lactam), 83.60 (C3, β -lactam), 109.08 (C5, 3,4-(CH_3O) $_2$ -Ph), 110.42 (C6, 3,4-(CH_3O) $_2$ -Ph), 114.34 (C3 and C5, PMP), 118.75 (C2 and C6, PMP), 124.00 (C2, 3,4-(CH_3O) $_2$ -Ph), 126.28 (C2 and C6, Ph), 128.63 (C4, Ph), 128.81 (C3, 3,4-(CH_3O) $_2$ -Ph), 129.15 (C3 and C5, Ph), 131.00 (C1, PMP), 136.84 (C1, Ph), 149.38 (C1, 3,4-(CH_3O) $_2$ -Ph), 152.05 (C4, 3,4-(CH_3O) $_2$ -Ph), 156.26 (C4, PMP), 163.65 (CO), 163.99 ($\text{CH}=\text{N}$). Anal. Calcd. for $\text{C}_{25}\text{H}_{24}\text{N}_2\text{O}_4$ ($M_r = 416.48$): C 72.10, H 5.81, N 6.73 %; found: C 72.15, H 5.88, N 6.91 %.

(3*R*,4*R*)-3-(4-Fluorobenzylideneamino)-1-(4-methoxyphenyl)-4-phenylazetidin-2-one (**1d**). – Obtained from **5a** (100.0 mg, 3.73×10^{-1} mmol) and **6d** (46.3 mg, 3.73×10^{-1} mmol) as yellow crystals (from dichloromethane-petroleum ether (b.p. 40–70 °C)), 92.6 mg (66 %), $R_f = 0.25$ (dichloromethane), m.p. 143–145 °C; $[\alpha]_D = +234.7$ ($c = 1.01$ g/100 mL dichloromethane); IR (KBr) $\nu_{\max}/\text{cm}^{-1}$: 3855, 1736, 1638, 1602, 1511, 1394, 1250, 1234, 1151, 839, 698; ^1H NMR (CDCl_3) δ/ppm : 3.74 (s, 3H, OCH_3), 4.64 (bs, 1H, C4, β -lactam), 5.17 (d, 1H, $J = 1.80$ Hz, C3, β -lactam), 6.80 (d, 2H, $J = 9.03$ Hz, C3 and C5, PMP), 7.11 (t, 2H, $J_{1,2} = 8.64$ Hz, C3 and C5, p -F-Ph), 7.29 (d, 2H, $J = 9.03$ Hz, C2 and C6, PMP), 7.39 (m, 5H, Ph), 7.78 (dd, 2H, $J_1 = 8.67$ Hz, $J_2 = 5.52$ Hz, C2 and C6, p -F-Ph), 8.37 (s, 1H, $\text{CH}=\text{N}$); ^{13}C NMR (CDCl_3) δ/ppm : 55.41 (OCH_3), 63.56 (C4, β -lactam), 83.41 (C3, β -lactam), 114.33 (C3 and C5, PMP), 115.81 (d, $J = 87.40$ Hz, C3 and C5, p -F-Ph), 118.76 (C2 and C6, PMP), 126.26 (C2 and C6, Ph), 128.72 (C4, Ph), 129.20 (C3 and C5, Ph), 130.60 (d, $J = 35.21$ Hz, C2 and C6, p -F-Ph), 130.84 (C1, PMP), 131.80 (d, $J = 2.78$ Hz,

C1, *p*-F-Ph), 136.62 (C1, Ph), 156.28 (C4, PMP), 163.07 (C4, *p*-F-Ph), 163.19 (CO), 166.41 (CH=N). Anal. Calcd. for C₂₃H₁₉FN₂O₂ (*M_r* = 374.41): C 73.78, H 5.11, N 7.48 %; found: C 74.01, H 5.26, N 7.73 %.

(3*R*,4*R*)-3-(4-Trifluoromethylbenzylideneamino)-1-(4-methoxyphenyl)-4-phenylazetididin-2-one (**1e**). – Obtained from **5a** (100.0 mg, 3.73 × 10⁻¹ mmol) and **6e** (65.0 mg, 3.73 × 10⁻¹ mmol) as yellow crystals (from dichloromethane-petroleum ether (b.p. 40–70 °C)), 93.9 mg (59 %), *R_f* = 0.25 (dichloromethane), m.p. 150–152 °C; [α]_D = +207.9 (*c* = 1.00 g/100 mL dichloromethane); IR (KBr) *v*_{max}/cm⁻¹: 2361, 1752, 1513, 1324, 1249, 1065; ¹H NMR (CDCl₃) δ/ppm: 3.75 (s, 3H, OCH₃), 4.70 (bs, 1H, C4, β-lactam), 5.20 (bs, 1H, C3, β-lactam), 6.81 (d, 2H, *J* = 8.88 Hz, C3 and C5, PMP), 7.29 (d, 2H, *J* = 8.88 Hz, C2 and C6, PMP), 7.32–7.42 (m, 5H, Ph), 7.68 (d, 2H, *J* = 7.98 Hz, C2 and C6, *p*-CF₃-Ph), 7.90 (d, 2H, *J* = 7.98 Hz, C3 and C5, *p*-CF₃-Ph), 8.46 (s, 1H, CH=N); ¹³C NMR (CDCl₃) δ/ppm: 55.43 (OCH₃), 63.59 (C4, β-lactam), 83.28 (C3, β-lactam), 114.41 (C3 and C5, PMP), 118.81 (C2 and C6, PMP), 125.23 (CF₃), 125.62 (d, *J* = 3.58 Hz, C3 and C5, *p*-CF₃-Ph), 126.29 (C2 and C6, Ph), 128.79 (C3 and C5, Ph), 128.82 (C4, Ph), 129.25 (C2 and C6, *p*-CF₃-Ph), 130.80 (C1, *p*-CF₃-Ph), 132.94 (d, *J* = 33.10 Hz, C4, *p*-CF₃-Ph), 136.52 (C1, PMP), 138.56 (C1, Ph), 156.43 (C4, PMP), 162.75 (CO), 163.04 (CH=N). Anal. Calcd. for C₂₄H₁₉F₃N₂O₂ (*M_r* = 424.40): C 67.92, H 4.51, N 6.60 %; found: C 67.70, H 4.43, N 6.57 %.

(3*R*,4*R*)-1-(4-Methoxyphenyl)-3-(2-nitrobenzylideneamino)-4-phenylazetididin-2-one (**1f**). – Obtained from **5a** (60.0 mg, 2.24 × 10⁻¹ mmol) and **6f** (34.0 mg, 2.24 × 10⁻¹ mmol) as yellow crystals (from dichloromethane-petroleum ether (b.p. 40–70 °C)), 70.8 mg (79 %), *R_f* = 0.11 (dichloromethane), m.p. 148–150 °C; [α]_D = +264.7 (*c* = 0.20 g/100 mL dichloromethane); IR (KBr) *v*_{max}/cm⁻¹: 3483, 1752, 1630, 1514, 1386, 1350, 1252, 1148, 826; ¹H NMR (CDCl₃) δ/ppm: 3.75 (s, 3H, OCH₃), 4.76 (bs, 1H, C4, β-lactam), 5.21 (d, 1H, *J* = 1.80 Hz, C3, β-lactam), 6.81 (d, 2H, *J* = 9.00 Hz, C3 and C5, PMP), 7.29 (d, 2H, *J* = 9.00 Hz, C2 and C6, PMP), 7.36–7.44 (m, 5H, Ph), 7.61 (t, 1H, *J*_{1,2} = 7.80 Hz, C4, Ph-NO₂), 7.70 (t, 1H, *J*_{1,2} = 7.44 Hz, C5, Ph-NO₂), 8.04 (d, 1H, *J* = 7.80 Hz, C6, Ph-NO₂), 8.16 (d, 1H, *J* = 7.80 Hz, C3, Ph-NO₂), 8.86 (s, 1H, CH=N); ¹³C NMR (CDCl₃) δ/ppm: 55.42 (OCH₃), 63.40 (C4, β-lactam), 83.38 (C3, β-lactam), 114.41 (C3 and C5, PMP), 118.81 (C2 and C6, PMP), 124.43 (C3, Ph-NO₂), 126.25 (C2 and C6, Ph), 128.85 (C4, Ph), 129.28 (C3 and C5, Ph), 129.89 (C6, Ph-NO₂), 130.40 (C1, Ph-NO₂), 130.81 (C1, PMP), 131.38 (C4, Ph-NO₂), 133.49 (C5, Ph-NO₂), 136.32 (C1, Ph), 148.50 (C2, Ph-NO₂), 156.43 (C4, PMP), 160.64 (CH=N), 162.38 (CO). Anal. Calcd. for C₂₃H₁₉N₃O₄ (*M_r* = 401.42): C 68.82, H 4.77, N 10.46 %; found: C 68.89, H 4.68, N 10.34 %.

(3*R*,4*R*)-1-(4-Methoxyphenyl)-3-(3-nitrobenzylideneamino)-4-phenylazetididin-2-one (**1g**). – Obtained from **5a** (60.0 mg, 2.24 × 10⁻¹ mmol) and **6g** (34.0 mg, 2.24 × 10⁻¹ mmol) as yellow crystals (from dichloromethane-petroleum ether (b.p. 40–70 °C)), 80.0 mg (89 %), *R_f* = 0.19 (dichloromethane), m.p. 136–137 °C; [α]_D = +306.8 (*c* = 0.20 g/100 mL di-

chloromethane); IR (KBr) *v*_{max}/cm⁻¹: 3463, 2956, 1736, 1639, 1528, 1511, 1459, 1396, 1348, 1248, 835; ¹H NMR (CDCl₃) δ/ppm: 3.75 (s, 3H, OCH₃), 4.23 (bs, 1H, C4, β-lactam), 5.22 (bs, 1H, C3, β-lactam), 6.81 (d, 2H, *J* = 9.00 Hz, C3 and C5, PMP), 7.29 (d, 2H, *J* = 9.00 Hz, C2 and C6, PMP), 7.35–7.45 (m, 5H, Ph), 7.62 (t, 1H, *J*_{1,2} = 7.80 Hz, C5, Ph-NO₂), 8.10 (d, 1H, *J* = 7.80 Hz, C6, Ph-NO₂), 8.30 (d, 1H, *J* = 7.20 Hz, C4, Ph-NO₂), 8.50 (s, 1H, CH=N), 8.66 (s, 1H, C2, Ph-NO₂); ¹³C NMR (CDCl₃) δ/ppm: 55.42 (OCH₃), 63.55 (C4, β-lactam), 82.96 (C3, β-lactam), 114.40 (C3 and C5, PMP), 118.81 (C2 and C6, PMP), 123.12 (C4, Ph-NO₂), 125.70 (C2, Ph-NO₂), 126.28 (C2 and C6, Ph), 128.87 (C4, Ph), 129.27 (C3 and C5, Ph), 129.70 (C5, Ph-NO₂), 130.70 (C1, PMP), 134.09 (C6, Ph-NO₂), 136.39 (C1, Ph), 137.12 (C1, Ph-NO₂), 148.63 (C3, Ph-NO₂), 156.44 (C4, PMP), 161.81 (CH=N), 162.50 (CO). Anal. Calcd. for C₂₃H₁₉N₃O₄ (*M_r* = 401.42): C 68.82, H 4.77, N 10.46 %; found: C 68.79, H 4.55, N 10.24 %.

(3*R*,4*R*)-1-(4-Methoxyphenyl)-3-(4-nitrobenzylideneamino)-4-phenylazetididin-2-one (**1h**). – Obtained from **5a** (60.0 mg, 2.24 × 10⁻¹ mmol) and **6h** (34.0 mg, 2.24 × 10⁻¹ mmol) as yellow crystals (from dichloromethane-petroleum ether (b.p. 40–70 °C)), 73.2 mg (81 %), *R_f* = 0.11 (dichloromethane), m.p. 170–172 °C; [α]_D = +324.7 (*c* = 0.20 g/100 mL dichloromethane); IR (KBr) *v*_{max}/cm⁻¹: 3449, 1736, 1702, 1638, 1509, 1499, 1458, 1347, 1301, 1252, 828; ¹H NMR (CDCl₃) δ/ppm: 3.75 (s, 3H, OCH₃), 4.73 (bs, 1H, C4, β-lactam), 5.22 (d, 1H, *J* = 1.20 Hz, C3, β-lactam), 6.81 (d, 2H, *J* = 9.00 Hz, C3 and C5, PMP), 7.29 (d, 2H, *J* = 9.00 Hz, C2 and C6, PMP), 7.35–7.45 (m, 5H, Ph), 7.96 (d, 2H, *J* = 8.40 Hz, C2 and C6, Ph-NO₂), 8.28 (d, 2H, *J* = 8.40 Hz, C3 and C5, Ph-NO₂), 8.50 (s, 1H, CH=N); ¹³C NMR (CDCl₃) δ/ppm: 55.42 (OCH₃), 63.56 (C4, β-lactam), 83.20 (C3, β-lactam), 114.43 (C3 and C5, PMP), 118.81 (C2 and C6, PMP), 123.87 (C3 and C5, Ph-NO₂), 126.28 (C2 and C6, Ph), 128.90 (C4, Ph), 129.26 (C2 and C6, Ph-NO₂), 129.28 (C3 and C5, Ph), 130.70 (C1, PMP), 136.37 (C1, Ph), 140.79 (C1, Ph-NO₂), 149.49 (C4, Ph-NO₂), 156.49 (C4, PMP), 162.08 (CH=N), 162.37 (CO). Anal. Calcd. for C₂₃H₁₉N₃O₄ (*M_r* = 401.42): C 68.82, H 4.77, N 10.46 %; found: C 68.58, H 4.50, N 10.19 %.

(3*R*,4*R*)-3-Cyclohexylmethylideneamino-1-(4-methoxyphenyl)-4-phenylazetididin-2-one (**1i**). – Obtained from **5a** (40.0 mg, 1.49 × 10⁻¹ mmol) and **6i** (16.7 mg, 1.49 × 10⁻¹ mmol) in dichloromethane stirring at room temperature over anhydrous sodium sulfate (50.0 mg) for 4 h. Sodium sulfate was filtered off, washed with dichloromethane (3 × 5 mL) and filtrate evaporated to dryness. Raw product (50.2 mg, ¹H NMR spectra calculated yield of **1i** – 65 %) was used directly in the Diels-Alder reaction. ¹H NMR (CDCl₃) δ/ppm: 1.20–1.40 (m, 5H, cyclohexyl), 1.60–1.90 (m, 6H, cyclohexyl), 3.74 (s, 3H, OCH₃), 4.39 (bs, 1H, C4, β-lactam), 5.04 (d, 1H, *J* = 1.75 Hz, C3, β-lactam), 6.78 (d, 2H, *J* = 9.00 Hz, C3 and C5, PMP), 7.24 (d, 2H, *J* = 9.00 Hz, C2 and C6, PMP), 7.30–7.40 (m, 5H, Ph), 7.69 (d, 1H, *J* = 5.00 Hz, CH=N); ¹³C NMR (CDCl₃) δ/ppm: 25.36 (C2 and C6, cyclohexyl), 25.88 (C3 and C5, cyclohexyl), 29.40 (C4, cyclohexyl), 43.88 (C1,

cyclohexyl), 55.37 (OCH₃), 63.38 (C4, β-lactam), 83.09 (C3, β-lactam), 114.25 (C3 and C5, PMP), 118.69 (C2 and C6, PMP), 126.23 (C2 and C6, Ph), 128.61 (C4, Ph), 129.12 (C3 and C5, Ph), 130.85 (C1, Ph), 136.73 (C1, PMP), 156.15 (C4, PMP), 163.43 (CO), 173.93 (CH=N). Mass Spectra (MS) for C₂₃H₂₆N₂O₂ (M_r = 362.47): calcd. *m/z* [M+H]⁺ 363.206704, found 363.199878.

(3*R*,4*R*)-1-(4-Methoxyphenyl)-3-

(2,2-dimethylpropylideneamino)-4-phenylazetididin-2-one (**Ij**). – Obtained from **5a** (40.0 mg, 1.49 × 10⁻¹ mmol) and **6j** (12.8 mg, 1.49 × 10⁻¹ mmol) in dichloromethane stirring at room temperature over anhydrous sodium sulfate (50.0 mg) for 4 h. Sodium sulfate was filtered off, washed with dichloromethane (3 × 5 mL) and filtrate evaporated to dryness. Raw product (46.2 mg, ¹H NMR spectra calculated yield of **Ij** – 60 %) was used directly in the Diels-Alder reaction. ¹H NMR (CDCl₃) δ/ppm: 1.10 (s, 9H, 3 × CH₃), 3.74 (s, 3H, OCH₃), 4.39 (dd, 1H, J₁ = 1.80 Hz, J₂ = 1.00 Hz, C3, β-lactam), 5.04 (d, 1H, J = 1.90 Hz, C4, β-lactam), 6.78 (d, 2H, J = 9.00 Hz, C3 and C5, PMP), 7.23 (d, 2H, J = 9.00 Hz, C2 and C6, PMP), 7.30–7.40 (m, 5H, Ph), 7.69 (d, 1H, J = 0.90 Hz, CH=N); ¹³C NMR (CDCl₃) δ/ppm: 26.76 (3 × CH₃), 29.67 (C(CH₃)₃), 55.38 (OCH₃), 63.38 (C4, β-lactam), 83.09 (C3, β-lactam), 114.24 (C3 and C5, PMP), 118.66 (C2 and C6, PMP), 126.24 (C2 and C6, Ph), 128.58 (C4, Ph), 129.11 (C3 and C5, Ph), 130.93 (C1, Ph), 136.82 (C1, PMP), 156.11 (C4, PMP), 163.58 (CO), 176.68 (CH=N). MS for C₂₁H₂₄N₂O₂ (M_r = 336.44): calcd. *m/z* [M+H]⁺ 337,191054, found 337,198083.

(3*R*,4*R*)-3-Ferrocenylmethylideneamino-1-

(4-methoxyphenyl)-4-phenylazetididin-2-one (**Ik**). – Obtained from **5a** (100.0 mg, 3.73 × 10⁻¹ mmol) and **6k** (79.8 mg, 3.73 × 10⁻¹ mmol) as red crystals (from petroleum ether (b.p. 40–70 °C)), 109.7 mg (63 %), R_f = 0.25 (dichloromethane), m.p. 167–168 °C; [α]_D = +314.3 (c = 1.00 g/100 mL dichloromethane); IR (KBr) ν_{max}/cm⁻¹: 2928, 1736, 1702, 1458, 1347, 1253, 828; ¹H NMR (CDCl₃) δ/ppm: 3.74 (s, 3H, OCH₃), 4.23 (s, 5H, Fc), 4.28 (s, 1H, Fc), 4.41–4.45 (m, 2H, Fc), 4.49 (bs, 1H, C4, β-lactam), 4.61 (m, 1H, Fc), 4.77 (m, 1H, Fc), 5.14 (d, 1H, J = 1.41 Hz, C3, β-lactam), 6.79 (d, 2H, J = 9.06 Hz, C3 and C5, PMP), 7.28 (d, 2H, J = 9.06 Hz, C2 and C6, PMP), 7.38–7.40 (m, 5H, Ph), 8.26 (s, 1H, CH=N); ¹³C NMR (CDCl₃) δ/ppm: 55.37 (OCH₃), 63.49 (C4, β-lactam), 68.16, 69.56, 71.09, 71.30 and 73.15 (Fc), 79.08 (C1, Fc), 83.88 (C3, β-lactam), 114.25 (C3 and C5, PMP), 118.68 (C2 and C6, PMP), 126.15 (C2 and C6, Ph), 128.55 (C4, Ph), 129.13 (C3 and C5, Ph), 130.92 (C1, Ph), 136.84 (C1, PMP), 156.11 (C4, PMP), 163.44 (CO), 165.46 (CH=N). Anal. Calcd. for C₂₇H₂₄FeN₂O₂ (M_r = 464.35): C 69.84, H 5.21, N 6.03 %; found: C 69.65, H 5.13, N 6.21 %.

(3*R*,4*R*)-3-Benzylideneamino-1,4-bis(4-

methoxyphenyl)azetididin-2-one (**Il**). – Obtained from **5b** (100.0 mg, 3.35 × 10⁻¹ mmol) and **6a** (35.6 mg, 3.35 × 10⁻¹ mmol) as white crystals (from dichloromethane-petroleum ether (b.p. 40–70 °C)), 83.0 mg (64 %), R_f = 0.36 (dichloromethane), m.p. 163–164 °C; [α]_D = +284.4 (c = 0.20 g/100 mL dichloromethane); IR (KBr) ν_{max}/cm⁻¹: 3855, 1736, 1637, 1510, 1395, 1246, 1168, 836; ¹H NMR (CDCl₃) δ/ppm: 3.74 (s,

3H, OCH₃), 3.80 (s, 3H, OCH₃), 4.63 (m, 1H, C4, β-lactam), 5.13 (d, 1H, J = 1.70 Hz, C3, β-lactam), 6.80 (d, 2H, J = 9.00 Hz, C3 and C5, PMP), 6.91 (d, 2H, J = 8.70 Hz, C3 and C5, PMP), 7.24–7.36 (m, 4H, 2 × C2 and 2 × C6, PMP), 7.38–7.44 (m, 3H, Ph), 7.77 (dd, 2H, J₁ = 7.60 Hz, J₂ = 1.80 Hz, Ph), 8.39 (s, 1H, CH=N); ¹³C NMR (CDCl₃) δ/ppm: 55.30 (OCH₃), 55.39 (OCH₃), 63.21 (C4, β-lactam), 83.74 (C3, β-lactam), 114.27 (C3 and C5, PMP), 114.56 (C3 and C5, PMP), 118.78 (C2 and C6, PMP), 127.59 (C2 and C6, PMP), 128.50 (C1, PMP), 128.58 (C2 and C6, Ph), 128.62 (C3 and C5, Ph), 130.90 (C1, PMP), 131.40 (C4, Ph), 135.45 (C1, Ph), 156.18 (C4, PMP), 159.89 (C4, PMP), 163.36 (CO), 164.61 (CH=N). Anal. Calcd. for C₂₄H₂₂N₂O₃ (M_r = 386.45): C 74.59, H 5.74, N 7.25 %; found: C 74.34, H 5.96, N 7.36 %.

(3*R*,4*R*)-3-(4-Methoxybenzylideneamino)-1,4-bis

(4-methoxyphenyl)azetididin-2-one (**Im**). – Obtained from **5b** (100.0 mg, 3.35 × 10⁻¹ mmol) and **6b** (45.6 mg, 3.35 × 10⁻¹ mmol) as yellow crystals (from dichloromethane-petroleum ether (b.p. 40–70 °C)), 96.0 mg (69 %), R_f = 0.25 (dichloromethane), m.p. 182–184 °C; [α]_D = +364.3 (c = 0.20 g/100 mL dichloromethane); IR (KBr) ν_{max}/cm⁻¹: 3458, 1736, 1607, 1511, 1442, 1306, 1248, 1167, 1150, 843, 831; ¹H NMR (CDCl₃) δ/ppm: 3.74 (s, 3H, OCH₃), 3.80 (s, 3H, OCH₃), 3.84 (s, 3H, OCH₃), 4.59 (d, 1H, J = 0.50 Hz, C4, β-lactam), 5.10 (s, 1H, C3, β-lactam), 6.79 (d, 2H, J = 9.00 Hz, C3 and C5, PMP), 6.91 (d, 2H, J = 8.20 Hz, C3 and C5, PMP), 6.92 (d, 2H, J = 8.50 Hz, C3 and C5, PMP), 7.28 (d, 2H, J = 9.00 Hz, C2 and C6, PMP), 7.32 (d, 2H, J = 8.50 Hz, C2 and C6, PMP), 7.71 (d, 2H, J = 8.60 Hz, C2 and C6, PMP), 8.31 (s, 1H, CH=N); ¹³C NMR (CDCl₃) δ/ppm: 55.31 (OCH₃), 55.36 (OCH₃), 55.42 (OCH₃), 63.41 (C4, β-lactam), 83.83 (C3, β-lactam), 114.06 (C3 and C5, PMP), 114.33 (C3 and C5, PMP), 114.59 (C3 and C5, PMP), 118.81 (C2 and C6, PMP), 127.60 (C2 and C6, PMP), 128.57 (C1, PMP), 128.76 (C1, PMP), 130.28 (C2 and C6, PMP), 131.07 (C1, PMP), 156.23 (C4, PMP), 159.92 (C4, PMP), 160.07 (C4, PMP), 162.27 (CO), 163.76 (CH=N). Anal. Calcd. for C₂₅H₂₄N₂O₄ (M_r = 416.48): C 72.10, H 5.81, N 6.73 %; found: C 71.95, H 5.90, N 6.83 %.

(3*R*,4*R*)-3-(4-Benzylideneamino)-4-(3,4-dimethoxyphenyl)-

1-(4-methoxyphenyl)azetididin-2-one (**In**). – Obtained from **5c** (100.0 mg, 3.05 × 10⁻¹ mmol) and **6a** (32.2 mg, 3.05 × 10⁻¹ mmol) as white crystals (from dichloromethane-petroleum ether (b.p. 40–70 °C)), 93.7 mg (74 %), R_f = 0.47 (dichloromethane), m.p. 172–174 °C; [α]_D = +284.4 (c = 0.20 g/100 mL dichloromethane); IR (KBr) ν_{max}/cm⁻¹: 3855, 1748, 1636, 1512, 1460, 1251, 1139, 1027, 834; ¹H NMR (CDCl₃) δ/ppm: 3.75 (s, 3H, OCH₃), 3.84 (s, 3H, OCH₃), 3.88 (s, 3H, OCH₃), 4.67 (bs, 1H, C4, β-lactam), 5.12 (bs, 1H, C3, β-lactam), 6.80 (d, 2H, J = 8.90 Hz, C3 and C5, PMP), 6.88 (s, 1H, C2, 3,4-(CH₃O)₂-Ph), 6.75–6.97 (m, 1H, C6, 3,4-(CH₃O)₂-Ph), 6.97 (d, 1H, J = 8.20 Hz, C5, 3,4-(CH₃O)₂-Ph), 7.30 (d, 2H, J = 8.90 Hz, C2 and C6, PMP), 7.40–7.45 (m, 3H, Ph), 7.79 (d, 2H, J = 7.60 Hz, C2 and C6, Ph), 8.41 (s, 1H, CH=N); ¹³C NMR (CDCl₃) δ/ppm: 55.38 (OCH₃), 55.91 (OCH₃), 55.98 (OCH₃), 63.55

(C4, β -lactam), 83.66 (C3, β -lactam), 108.83 (C6, 3,4-(CH₃O)₂-Ph), 111.49 (C5, 3,4-(CH₃O)₂-Ph), 114.26 (C3 and C5, PMP), 118.76 (C2 and C6, PMP), 118.86 (C2, 3,4-(CH₃O)₂-Ph), 128.58 (C2 and C6, Ph), 128.63 (C3 and C5, Ph), 128.97 (C1, 3,4-(CH₃O)₂-Ph), 130.92 (C1, PMP), 131.44 (C4, Ph), 136.84 (C1, Ph), 149.30 (C3, 3,4-(CH₃O)₂-Ph), 149.62 (C4, 3,4-(CH₃O)₂-Ph), 156.22 (C4, PMP), 163.39 (CO), 164.71 (CH=N). Anal. Calcd. for C₂₅H₂₄N₂O₄ (*M_r* = 416.48): C 72.10, H 5.81, N 6.73 %; found: C 71.98, H 6.03, N 6.80 %.

(3*R*,4*R*)-3-Benzylideneamino-4-(4-fluorophenyl)-1-(4-methoxyphenyl)azetid-2-one (**1o**). – Obtained from **5d** (100.0 mg, 3.49 × 10⁻¹ mmol) and **6a** (37.0 mg, 3.49 × 10⁻¹ mmol) as white crystals (from ethanol), 115.8 mg (89 %), *R_f* = 0.54 (dichloromethane), m.p. 176–178 °C; [α]_D = +269.7 (*c* = 0.20 g/100 mL dichloromethane); IR (KBr) $\nu_{\max}/\text{cm}^{-1}$: 3855, 3752, 3678, 3651, 3630, 1752, 1637, 1509, 1251, 1231; ¹H NMR (CDCl₃) δ/ppm : 3.75 (s, 3H, OCH₃), 4.63 (bs, 1H, C4, β -lactam), 5.17 (d, 1H, *J* = 1.70 Hz, C3, β -lactam), 6.81 (d, 2H, *J* = 9.00 Hz, C3 and C5, PMP), 7.08 (t, 2H, *J*_{1,2} = 8.60 Hz, C3 and C5, *p*-F-Ph), 7.26 (d, 2H, *J* = 9.00 Hz, C2 and C6, PMP), 7.35–7.50 (m, 5H, Ph), 7.78 (dd, 2H, *J*₁ = 7.60 Hz, *J*₂ = 1.80 Hz, C2 and C6, *p*-F-Ph), 8.41 (s, 1H, CH=N); ¹³C NMR (CDCl₃) δ/ppm : 55.41 (OCH₃), 62.92 (C4, β -lactam), 83.58 (C3, β -lactam), 114.36 (C3 and C5, PMP), 116.22 (d, *J* = 21.71 Hz, C3 and C5, *p*-F-Ph), 118.73 (C2 and C6, PMP), 127.95 (C2 and C6, Ph), 128.06 (C3 and C5, Ph), 128.63 (d, *J* = 3.80 Hz, C2 and C6, *p*-F-Ph), 130.65 (C1, PMP), 131.51 (C4, Ph), 132.44 (C1, *p*-F-Ph), 135.36 (C1, Ph), 156.33 (C4, PMP), 161.18 (CO), 163.77 (d, *J* = 105.50 Hz, C4, *p*-F-Ph), 164.81 (CH=N). Anal. Calcd. for C₂₃H₁₉FN₂O₂ (*M_r* = 374.41): C 73.78, H 5.11, N 7.48 %; found: C 74.01, H 5.36, N 7.73 %.

(3*R*,4*R*)-3-Benzylideneamino-4-(4-trifluoromethylphenyl)-1-(4-methoxyphenyl)azetid-2-one (**1p**). – Obtained from **5e** (100.0 mg, 2.97 × 10⁻¹ mmol) and **6a** (31.5 mg, 2.97 × 10⁻¹ mmol) as white crystals (from ethanol), 97.1 mg (77 %), *R_f* = 0.71 (dichloromethane), m.p. 156–158 °C; [α]_D = +368.9 (*c* = 0.20 g/100 mL dichloromethane); IR (KBr) $\nu_{\max}/\text{cm}^{-1}$: 1741, 1637, 1511, 1326, 1248, 1170, 1126, 1113, 1081, 1066, 830; ¹H NMR (CDCl₃) δ/ppm : 3.76 (s, 3H, OCH₃), 4.65 (bs, 1H, C4, β -lactam), 5.26 (bs, 1H, C3, β -lactam), 6.82 (d, 2H, *J* = 8.90 Hz, C3 and C5, PMP), 7.25 (d, 2H, *J* = 8.80 Hz, C2 and C6, PMP), 7.47–7.41 (m, 3H, Ph), 7.53 (d, 2H, *J* = 8.00 Hz, C3 and C5, *p*-CF₃-Ph), 7.66 (d, 2H, *J* = 8.00 Hz, C2 and C6, *p*-CF₃-Ph), 7.79 (d, 2H, *J* = 7.10 Hz, C2 and C6, Ph), 8.41 (s, 1H, CH=N); ¹³C NMR (CDCl₃) δ/ppm : 55.43 (OCH₃), 62.95 (C4, β -lactam), 83.40 (C3, β -lactam), 114.47 (C3 and C5, PMP), 118.68 (C2 and C6, PMP), 125.23 (CF₃), 126.22 (C2 and C6, *p*-CF₃-Ph), 126.59 (C3 and C5, *p*-CF₃-Ph), 128.65 (C2 and C6, Ph), 128.69 (C3 and C5, Ph), 130.48 (C4, Ph), 130.94 (d, *J* = 32.90 Hz, C4, *p*-CF₃-Ph), 131.63 (C1, PMP), 135.26 (C1, Ph), 140.84 (C1, *p*-CF₃-Ph), 156.47 (C4, PMP), 162.78 (CO), 165.10 (CH=N). Anal. Calcd. for C₂₄H₁₉F₃N₂O₂ (*M_r* = 424.40): C 67.92, H 4.51, N 6.60 %; found: C 67.60, H 4.35, N 6.61 %.

(3*R*,4*R*)-3-Benzylideneamino-4-ferrocenyl-1-(4-methoxyphenyl)azetid-2-one ((3*R*,4*R*)-**1q**). – Obtained from (3*R*,4*R*)-**5f** (90.0 mg, 2.39 × 10⁻¹ mmol) and **6a** (25.4 mg, 2.39 × 10⁻¹ mmol) as brown crystals (from dichloromethane and petroleum ether (b.p. 40–70 °C)), 67.3 mg (61 %), *R_f* = 0.28 (dichloromethane), m.p. 165–167 °C; [α]_D = +342.7 (*c* = 0.20 g/100 mL dichloromethane); IR (KBr) $\nu_{\max}/\text{cm}^{-1}$: 3855, 3752, 3650, 3630, 3449, 1743, 1636, 1511, 1388, 1246; ¹H NMR (CDCl₃) δ/ppm : 3.74 (s, 3H, OCH₃), 4.24 (s, 5H, Fc), 4.27 (m, 2H, Fc), 4.32 (d, 1H, *J* = 1.07 Hz, C4, β -lactam), 4.43 (s, 1H, Fc), 4.99 (d, 1H, *J* = 1.30 Hz, C3, β -lactam), 5.12 (s, 1H, Fc), 6.80 (d, 2H, *J* = 8.85 Hz, C3 and C5, PMP), 7.34 (d, 2H, *J* = 9.06 Hz, C3 and C5, PMP), 7.44 (m, 3H, Ph), 7.84 (m, 2H, Ph), 8.68 (s, CH=N); ¹³C NMR (CDCl₃) δ/ppm : 55.41 (OCH₃), 60.93 (C4, β -lactam), 66.44, 68.22, 68.63, 69.70 and 70.66 (Fc), 80.81 (C3, β -lactam), 83.12 (C1, Fc), 114.24 (C3 and C5, PMP), 119.11 (C2 and C6, PMP), 128.54 (C2 and C6, Ph), 128.67 (C3 and C5, PMP), 130.96 (C1, Ph), 131.39 (C4, Ph), 135.73 (C1, PMP), 156.34 (C4, PMP), 163.77 (CO), 163.89 (CH=N). Anal. Calcd. for C₂₇H₂₄FeN₂O₂ (*M_r* = 464.35): C 69.84, H 5.21, N 6.03 %; found: C 69.65, H 5.02, N 5.90 %.

(3*S*,4*S*)-3-Benzylideneamino-4-ferrocenyl-1-(4-methoxyphenyl)azetid-2-one ((3*S*,4*S*)-**1q**). – Obtained from (3*S*,4*S*)-**5f** (75.0 mg, 1.99 × 10⁻¹ mmol) and **6a** (21.2 mg, 1.99 × 10⁻¹ mmol) as brown crystals (from ethyl acetate and petroleum ether (b.p. 40–70 °C)), 72.6 mg (79 %), *R_f* = 0.28 (dichloromethane), m.p. 166–167 °C; [α]_D = –343.05 (*c* = 0.20 g/100 mL dichloromethane); IR (KBr) $\nu_{\max}/\text{cm}^{-1}$: 3630, 3448, 1743, 1637, 1511, 1246; ¹H NMR (CDCl₃) δ/ppm : 3.74 (s, 3H, OCH₃), 4.24 (s, 5H, Fc), 4.27 (m, 2H, Fc), 4.32 (d, 1H, *J* = 1.07 Hz, C4, β -lactam), 4.43 (s, 1H, Fc), 4.99 (d, 1H, *J* = 1.30 Hz, C3, β -lactam), 5.12 (s, 1H, Fc), 6.80 (d, 2H, *J* = 8.85 Hz, C3 and C5, PMP), 7.34 (d, 2H, *J* = 9.06 Hz, C3 and C5, PMP), 7.44 (m, 3H, Ph), 7.84 (m, 2H, Ph), 8.68 (s, CH=N); ¹³C NMR (CDCl₃) δ/ppm : 55.42 (OCH₃), 60.93 (C4, β -lactam), 66.44, 68.23, 68.64, 69.71 and 70.67 (Fc), 80.82 (C3, β -lactam), 83.13 (C1, Fc), 114.24 (C3 and C5, PMP), 119.11 (C2 and C6, PMP), 128.55 (C2 and C6, Ph), 128.68 (C3 and C5, PMP), 130.98 (C1, Ph), 131.40 (C4, Ph), 135.74 (C1, PMP), 156.33 (C4, PMP), 163.76 (CO), 163.89 (CH=N). Anal. Calcd. for C₂₇H₂₄FeN₂O₂ (*M_r* = 464.35): C 69.84, H 5.21, N 6.03 %; found: C 69.82, H 5.28, N 6.30 %.

(3*S*,4*S*)-4-Ferrocenyl-3-ferrocenylmethylideneamino-1-(4-methoxyphenyl)azetid-2-one (**1r**). – Obtained from **5f** (50.0 mg, 1.33 × 10⁻¹ mmol) and **6k** (28.5 mg, 1.33 × 10⁻¹ mmol). Raw product (69.5 mg, ¹H NMR spectra calculated yield of **1r** – 91 %) was used directly in the Diels-Alder reaction. IR (KBr) $\nu_{\max}/\text{cm}^{-1}$: 3432, 1742, 1629, 1511, 1246; ¹H NMR (CDCl₃) δ/ppm : 3.74 (s, 3H, OCH₃), 4.23 (s, 5H, Fc), 4.25 (s, 5H, Fc), 4.27 (s, 1H, Fc), 4.29 (s, 1H, Fc), 4.41–4.45 (m, 4H, Fc), 4.68 (bs, 1H, C4, β -lactam), 4.80 (s, 1H, Fc), 4.93 (s, 1H, Fc), 4.98 (s, 1H, C3, β -lactam), 6.79 (d, 2H, *J* = 8.54 Hz, C3 and C5, PMP), 7.32 (d, 2H, *J* = 8.54 Hz, C2 and C6, PMP), 8.51 (s, 1H, CH=N); ¹³C NMR (CDCl₃) δ/ppm : 55.42 (OCH₃), 60.80 (C4, β -lactam), 66.41, 68.09, 68.35, 68.62, 69.29, 69.46, 69.64, 70.64,

70.88 and 71.10 (Fc), 79.83 (C1, Fc), 81.48 (C3, β -lactam), 83.43 (C1, Fc), 114.23 (C3 and C5, PMP), 119.03 (C2 and C6, PMP), 131.11 (C1, PMP), 156.23 (C4, PMP), 163.89 (CO), 164.61 (CH=N). MS for $C_{31}H_{28}Fe_2N_2O_2$ ($M_r = 572.28$): calcd. m/z $[M+H]^+$ 571.075906, found 571.077821.

(3R,4R)-3-Benzylideneamino-4-phenylazetididin-2-one (**1s**). – Obtained from **5g** (42.0 mg, 2.59×10^{-1} mmol) and **6a** (27.5 mg, 2.59×10^{-1} mmol) as white crystals (from dichloromethane and petroleum ether (b.p. 40–70 °C)), 56.5 mg (87 %), $R_f = 0.25$ (dichloromethane), m.p. 142–144 °C; $[\alpha]_D = +22.0$ ($c = 1.00$ g/100 mL dichloromethane); IR (KBr) ν_{max}/cm^{-1} : 3448, 1752, 1702, 1686, 1655, 1628, 1560, 1364, 691; 1H NMR ($CDCl_3$) δ/ppm : 4.62 (bs, 1H, C4, β -lactam), 4.93 (d, 1H, $J = 1.50$ Hz, C3, β -lactam), 6.45 (bs, 1H, NH), 7.30–7.50 (m, 8H, Ph), 7.73–7.80 (m, 2H, Ph), 8.37 (s, 1H, CH=N); ^{13}C NMR ($CDCl_3$) δ/ppm : 59.88 (C4, β -lactam), 84.60 (C3, β -lactam), 125.89 (C2 and C6, Ph), 128.50 (C4, Ph), 128.61 (C2 and C6, Ph; C4, Ph), 128.93 (C3 and C5, Ph), 131.42 (C3 and C5, Ph), 135.46 (C1, Ph), 138.59 (C1, Ph), 164.43 (CH=N), 167.17 (CO). Anal. Calcd. for $C_{16}H_{14}N_2O$ ($M_r = 250.30$): C 76.78, H 5.64, N 11.19 %; found: C 76.80, H 5.38, N 11.23 %.

Preparation of 2-Aryl(alkyl)-2,3-dihydro-4-pyridones **3a-s/4a-s**

General Procedure. – To a suspension of Lewis acid (zinc(II) iodide, 0.2 mmol) in anhydrous acetonitrile (0.5 mL), a solution of imines **1a-s** (1.0 mmol) in acetonitrile (1.0 mL) was added dropwise under stirring for 15 minutes at –20 °C. To the reaction mixture dienes **2a-e** (1.1 mmol) were added, and the mixture was left under stirring for 7 h at –20 °C. The reaction mixture was poured onto ethyl acetate (20 mL) containing saturated solution of sodium bicarbonate (10 mL), the product mixture was further extracted with ethyl acetate (2×20 mL), combined extracts (60 mL) were dried over anhydrous sodium sulfate, filtered and filtrate evaporated to dryness. Product mixtures were isolated by a silica gel column chromatography using a mixture of ethyl acetate-hexane in ratio 1 mL : 1 mL containing triethylamine (0.1 mL) (eluens-A), ethyl acetate-hexane in ratio 2 mL : 1 mL, containing triethylamine (0.1 mL) (eluens-B), or a mixture of ethyl acetate-petroleum ether in ratio 3:1 (eluens-C) as eluents.

(2R/2S)-1-[trans-(3'R,4'R)-1'-(4-Methoxyphenyl)-2'-oxo-4'-phenylazetididin-3'-yl]-2-phenyl-1,2,3,4-tetrahydropyridin-4-one (**3a/4a**). – Obtained from (3R,4R)-**1a** (100.0 mg, 2.81×10^{-1} mmol), diene **2a** (62.0 mg, 3.09×10^{-1} mmol) and zinc(II) iodide (17.8 mg, 5.62×10^{-2} mmol) as a diastereomeric mixture **3a/4a**, 109.7 mg (92 %), $R_f = 0.25$ (eluens-A). RP-HPLC analysis of diastereomeric mixture showed ratio of **3a/4a** – 68:32 % (**3a**, 24.38 min and **4a**, 24.71 min). LC-MS for $C_{27}H_{24}N_2O_3$ ($M_r = 424.5019$): calcd. m/z $[M+H]^+$ 425.50, found 425.20 (**3a**, 10.56 min) and 425.20 (**4a**, 10.90 min). Diastereomeric mixture of **3a/4a** was separated by a preparative thin layer chromatography using a mixture of hexane-isopropanol-triethylamine in ratio 45:4:1.

(2R)-1-[trans-(3'R,4'R)-1'-(4-Methoxyphenyl)-2'-oxo-4'-phenylazetididin-3'-yl]-2-phenyl-1,2,3,4-tetrahydropyridin-4-one (**3a**). – 74.6 mg; m.p. 103–105 °C; $[\alpha]_D = -284.0$ ($c = 0.32$ g/100 mL dichloromethane); IR (KBr) ν_{max}/cm^{-1} : 3449, 1752, 1654, 1648, 1594, 1512, 1249, 830, 699; 1H NMR ($CDCl_3$) δ/ppm : 2.67 (dd, 1H, $J_1 = 16.50$ Hz, $J_2 = 6.40$ Hz, C3, THPO), 3.06 (dd, 1H, $J_1 = 16.50$ Hz, $J_2 = 7.30$ Hz, C3, THPO), 3.73 (s, 3H, OCH₃), 4.29 (d, 1H, $J = 1.90$ Hz, C3', β -lactam), 4.61 (t, 1H, $J_{1,2} = 6.60$ Hz, C2, THPO), 4.68 (bs, 1H, C4', β -lactam), 5.25 (d, 1H, $J = 7.90$ Hz, C5, THPO), 6.72–6.76 (m, 2H, C3 and C5, PMP and 1H, C6, THPO), 7.10–7.35 (m, 2H, C2 and C6, PMP and 10H, Ph); ^{13}C NMR ($CDCl_3$) δ/ppm : 43.60 (C3, THPO), 55.42 (OCH₃), 64.21 (C2, THPO), 65.01 (C4', β -lactam), 76.94 (C3', β -lactam), 101.44 (C5, THPO), 114.42 (C3 and C5, PMP), 119.02 (C2 and C6, PMP), 125.78 (C4, Ph), 127.02 (C4, Ph, β -lactam), 128.74 (C2 and C6, Ph, β -lactam), 128.80 (C2 and C6, Ph), 129.01 (C3 and C5, Ph, β -lactam), 129.25 (C3 and C5, Ph), 129.85 (C1, PMP), 134.97 (C1, Ph), 139.40 (C1, Ph, β -lactam), 148.62 (C6, THPO), 156.74 (C4, PMP), 161.82 (CO, β -lactam), 190.46 (C4, THPO).

(2S)-1-[trans-(3'R,4'R)-1'-(4-Methoxyphenyl)-2'-oxo-4'-phenylazetididin-3'-yl]-2-phenyl-1,2,3,4-tetrahydropyridin-4-one (**4a**). – 35.1 mg; IR (KBr) ν_{max}/cm^{-1} : 3449, 1752, 1654, 1648, 1594, 1512, 1249, 830, 699; 1H NMR ($CDCl_3$) δ/ppm : 2.79 (dd, 1H, $J_1 = 16.40$ Hz, $J_2 = 8.40$ Hz, C3, THPO), 2.95 (dd, 1H, $J_1 = 16.40$ Hz, $J_2 = 6.50$ Hz, C3, THPO), 3.73 (s, 3H, OCH₃), 4.29 (d, 1H, $J = 2.20$ Hz, C3', β -lactam), 4.65 (t, 1H, $J_{1,2} = 6.60$ Hz, C2, THPO), 4.69 (d, 1H, $J = 2.10$ Hz, C4', β -lactam), 5.24 (d, 1H, $J = 7.90$ Hz, C5, THPO), 6.72–6.76 (m, 2H, C3 and C5, PMP and 1H, C6, THPO), 7.10–7.35 (m, 2H, C2 and C6, PMP and 10H, Ph); ^{13}C NMR ($CDCl_3$) δ/ppm : 43.59 (C3, THPO), 55.37 (OCH₃), 62.81 (C2, THPO), 64.95 (C4', β -lactam), 76.87 (C3', β -lactam), 102.43 (C5, THPO), 114.40 (C3 and C5, PMP), 118.98 (C2 and C6, PMP), 128.69 (C4, Ph), 128.76 (C4, Ph, β -lactam), 128.96 (C2 and C6, Ph, β -lactam), 129.09 (C2 and C6, Ph), 129.20 (C3 and C5, Ph, β -lactam), 129.34 (C3 and C5, Ph), 129.79 (C1, PMP), 135.41 (C1, Ph), 139.34 (C1, Ph, β -lactam), 149.97 (C6, THPO), 156.68 (C4, PMP), 161.77 (CO, β -lactam), 190.54 (C4, THPO).

(2R/2S)-2-(4-Methoxyphenyl)-1-[trans-(3'R,4'R)-1'-(4-methoxyphenyl)-2'-oxo-4'-phenylazetididin-3'-yl]-1,2,3,4-tetrahydropyridin-4-one (**3b/4b**). – Obtained from (3R,4R)-**1b** (50.0 mg, 1.29×10^{-1} mmol), diene **2a** (27.6 mg, 1.42×10^{-1} mmol) and zinc(II) iodide (8.3 mg, 2.59×10^{-2} mmol) as a diastereomeric mixture **3b/4b**, 45.2 mg (77 %), $R_f = 0.32$ (eluens-A). RP-HPLC analysis of diastereomeric mixture showed ratio of **3b/4b** – 66:34 % (**3b**, 24.34 min and **4b**, 24.74 min). LC-MS for $C_{28}H_{26}N_2O_4$ ($M_r = 454.5283$): calcd. m/z $[M+H]^+$ 455.53, found 455.20 (**3b**, 10.48 min) and 455.20 (**4b**, 10.89 min). IR (KBr) ν_{max}/cm^{-1} : 3467, 2918, 1750, 1591, 1513, 1457, 1300, 1249, 1179, 831; 1H NMR ($CDCl_3$) δ/ppm : 2.68 (dd, 1H, $J_1 = 16.50$ Hz, $J_2 = 7.20$ Hz, C3, THPO, **3b**), 2.80 (dd, 1H, $J_1 = 16.44$ Hz, $J_2 = 9.36$ Hz, C3, THPO, **4b**), 2.87 (dd, 1H, $J_1 = 16.44$ Hz, $J_2 = 6.18$ Hz, C3, THPO, **4b**), 2.98 (dd, 1H, $J_1 = 16.50$ Hz, $J_2 = 7.02$ Hz, C3, THPO, **3b**), 3.72 (s, 3H, OCH₃, PMP, THPO, **3b**), 3.73 (s,

3H, OCH₃, PMP, THPO, **4b**), 3.74 (s, 3H, OCH₃, PMP, **3b**), 3.73 (s, 3H, OCH₃, PMP, **4b**), 4.27 (d, 1H, *J* = 1.92 Hz, C3', β-lactam, **3b**), 4.29 (d, 1H, *J* = 1.86 Hz, C3', β-lactam, **4b**), 4.56–4.62 (m, 2 × 1H, C2, THPO, **3b/4b**), 4.64 (d, 1H, *J* = 1.44 Hz, C4', β-lactam, **3b**), 4.68 (d, 1H, *J* = 1.80 Hz, C4', β-lactam, **4b**), 5.24 (d, 2 × 1H, *J* = 7.92 Hz, C5, THPO, **3b/4b**), 6.64 (d, 2H, *J* = 8.58 Hz, C3 and C5, PMP, THPO, **3b**), 6.71 (d, 2H, *J* = 8.58 Hz, C3 and C5, PMP, THPO, **4b**), 6.74 (d, 2H, *J* = 9.06 Hz, C3 and C5, PMP, β-lactam, **3b**), 6.76 (d, 2H, *J* = 9.18 Hz, C3 and C5, PMP, β-lactam, **4b**), 6.83 (d, 2 × 1H, *J* = 7.14 Hz, C6, THPO, **3b/4b**), 7.07 (d, 2H, *J* = 8.58 Hz, C2 and C6, PMP, THPO, **4b**), 7.11 (d, 2H, *J* = 8.58 Hz, C2 and C6, PMP, β-lactam, **3b/4b**), 7.15 (d, 2 × 2H, *J* = 8.82 Hz, C2 and C6, PMP, β-lactam, **3b/4b**), 7.18–7.35 (m, 2 × 5H, Ph, **3b/4b**); ¹³C NMR (CDCl₃) δ/ppm: 43.68 (C3, THPO, **3b**), 43.78 (C3, THPO, **4b**), 55.21 (2 × OCH₃, **3b/4b**), 55.38 (2 × OCH₃, **3b/4b**), 61.23 (C2, THPO, **4b**), 62.92 (C4', β-lactam, **4b**), 63.60 (C2, THPO, **3b**), 65.09 (C4', β-lactam, **3b**), 76.18 (C3', β-lactam, **4b**), 76.54 (C3', β-lactam, **3b**), 101.25 (C5, THPO, **3b**), 102.42 (C5, THPO, **4b**), 114.35 (C3 and C5, PMP, **3b**), 114.37 (C3 and C5, PMP, **4b**), 114.46 (2 × C3 and 2 × C5, PMP, **3b/4b**), 118.95 (2 × C2 and 2 × C6, PMP, β-lactam, **3b/4b**), 125.78 (C2 and C6, PMP, THPO, **4b**), 125.88 (C2 and C6, PMP, THPO, **3b**), 128.32 (C2 and C6, Ph, **4b**), 128.36 (C2 and C6, Ph, **3b**), 128.77 (C3 and C5, Ph, **4b**), 128.92 (C3 and C5, Ph, **3b**), 129.05 (C4, Ph, **4b**), 129.34 (C4, Ph, **3b**), 129.53 (C1, PMP, THPO, **4b**), 129.81 (C1, PMP, THPO, **3b**), 131.13 (2 × C1, PMP, β-lactam, **3b/4b**), 134.99 (2 × C1, Ph, **3b/4b**), 148.69 (C6, THPO, **3b**), 149.94 (C6, THPO, **4b**), 156.65 (2 × C4, PMP, β-lactam, **3b/4b**), 159.84 (2 × C4, PMP, THPO, **3b/4b**), 162.00 (2 × CO, β-lactam, **3b/4b**), 190.86 (C4, THPO, **3b**), 191.01 (C4, THPO, **4b**).

(2R/2S)-2-(3,4-Dimethoxyphenyl)-1-[trans-(3'R,4'R)-1'-(4-methoxyphenyl)-2'-oxo-4'-phenylazetididin-3'-yl]-1,2,3,4-tetrahydropyridin-4-one (**3c/4c**). – Obtained from (3R,4R)-**1c** (50.0 mg, 1.20 × 10⁻¹ mmol), diene **2a** (25.8 mg, 1.32 × 10⁻¹ mmol) and zinc(II) iodide (6.4 mg, 2.40 × 10⁻² mmol) as a diastereomeric mixture **3c/4c**, 53.4 mg (92 %), *R*_f = 0.37 (eluens-C). RP-HPLC analysis of diastereomeric mixture showed ratio of **3c/4c** – 70:30 % (**3c**, 23.19 min and **4c**, 23.77 min). LC-MS for C₂₉H₂₈N₂O₅ (*M*_r = 484.5547): calcd. *m/z* [M+H]⁺ 485.55, found 485.20 (**3c**, 9.53 min) and 485.20 (**4c**, 10.04 min). Diastereomeric mixture of **3c/4c** was separated by a silica gel column chromatography using eluens-C.

(2R)-2-(3,4-Dimethoxyphenyl)-1-[trans-(3'R,4'R)-1'-(4-methoxyphenyl)-2'-oxo-4'-phenylazetididin-3'-yl]-1,2,3,4-tetrahydropyridin-4-one (**3c**). – 37.4 mg; IR (KBr) *v*_{max}/cm⁻¹: 3448, 2934, 2837, 1750, 1589, 1513, 1249, 1141, 831; ¹H NMR (CDCl₃) δ/ppm: 2.72 (dd, 1H, *J*₁ = 16.54 Hz, *J*₂ = 7.14 Hz, C3, THPO), 3.02 (dd, 1H, *J*₁ = 16.51 Hz, *J*₂ = 7.11 Hz, C3, THPO), 3.68 (s, 3H, OCH₃), 3.72 (s, 3H, OCH₃), 3.82 (s, 3H, OCH₃), 4.33 (d, 1H, *J* = 2.10 Hz, C3', β-lactam), 4.55 (t, 1H, *J*_{1,2} = 7.08 Hz, C2, THPO), 4.62 (d, 1H, *J* = 2.04 Hz, C4', β-lactam), 5.26 (d, 1H, *J* = 7.92 Hz, C5, THPO), 6.57 (d, 1H, *J* = 8.25 Hz, 3,4-(CH₃O)₂-Ph), 6.66 (d, 1H, *J* = 1.95 Hz, C6, THPO), 6.74 (d, 2H, *J* = 9.06 Hz, C3 and C5, PMP), 6.77–6.85 (m, 2H, 3,4-(CH₃O)₂-Ph), 7.14 (d, 2H, *J* = 9.03

Hz, C2 and C6, PMP), 7.20–7.30 (m, 5H, Ph); ¹³C NMR (CDCl₃) δ/ppm: 43.49 (C3, THPO), 55.38 (OCH₃), 55.71, 55.83 (2 × OCH₃, 3,4-(CH₃O)₂-Ph), 64.05 (C2, THPO), 64.99 (C4', β-lactam), 76.50 (C3', β-lactam), 101.19 (C5, THPO), 110.02 (C6, 3,4-(CH₃O)₂-Ph), 111.40 (C5, 3,4-(CH₃O)₂-Ph), 114.34 (C3 and C5, PMP), 118.93 (C2 and C6, PMP), 119.58 (C2, 3,4-(CH₃O)₂-Ph), 125.83 (C4, Ph), 128.85 (C2 and C6, Ph), 128.92 (C3 and C5, Ph), 129.77 (C1, PMP), 131.69 (C1, 3,4-(CH₃O)₂-Ph), 134.94 (C1, Ph), 148.78 (C6, THPO), 149.25 (C4, 3,4-(CH₃O)₂-Ph), 149.33 (C3, 3,4-(CH₃O)₂-Ph), 156.66 (C4, PMP), 162.02 (CO, β-lactam), 191.05 (C4, THPO).

(2S)-2-(3,4-Dimethoxyphenyl)-1-[trans-(3'R,4'R)-1'-(4-methoxyphenyl)-2'-oxo-4'-phenylazetididin-3'-yl]-1,2,3,4-tetrahydropyridin-4-one (**4c**). – 16.0 mg; IR (KBr) *v*_{max}/cm⁻¹: 3448, 2934, 2837, 1750, 1589, 1513, 1249, 1141, 831; ¹H NMR (CDCl₃) δ/ppm: 2.80 (dd, 1H, *J*₁ = 16.39 Hz, *J*₂ = 9.12 Hz, C3, THPO), 2.89 (dd, 1H, *J*₁ = 16.54 Hz, *J*₂ = 6.66 Hz, C3, THPO), 3.70 (s, 3H, OCH₃), 3.73 (s, 3H, OCH₃), 3.82 (s, 3H, OCH₃), 4.30 (d, 1H, *J* = 2.22 Hz, C3', β-lactam), 4.58 (dd, 1H, *J*₁ = 8.97 Hz, *J*₂ = 6.48 Hz, C2, THPO), 4.69 (d, 1H, *J* = 2.13 Hz, C4', β-lactam), 5.25 (d, 1H, *J* = 7.86 Hz, C5, THPO), 6.64–6.72 (m, 3H, 3,4-(CH₃O)₂-Ph and 1H, C6, THPO), 6.77 (d, 2H, *J* = 9.09 Hz, C3 and C5, PMP), 7.16 (d, 2H, *J* = 9.03 Hz, C2 and C6, PMP), 7.20–7.30 (m, 5H, Ph); ¹³C NMR (CDCl₃) δ/ppm: 43.85 (C3, THPO), 55.41 (OCH₃), 55.77, 55.90 (2 × OCH₃, 3,4-(CH₃O)₂-Ph), 61.73 (C2, THPO), 63.05 (C4', β-lactam), 76.04 (C3', β-lactam), 102.48 (C5, THPO), 109.63 (C6, 3,4-(CH₃O)₂-Ph), 111.33 (C5, 3,4-(CH₃O)₂-Ph), 114.40 (C3 and C5, PMP), 118.95 (C2 and C6, PMP), 119.58 (C2, 3,4-(CH₃O)₂-Ph), 125.82 (C4, Ph), 129.09 (C2 and C6, Ph), 129.39 (C3 and C5, Ph), 129.75 (C1, PMP), 130.14 (C1, 3,4-(CH₃O)₂-Ph), 135.56 (C1, Ph), 147.17 (C4, 3,4-(CH₃O)₂-Ph), 149.17 (C3, 3,4-(CH₃O)₂-Ph), 149.87 (C6, THPO), 156.69 (C4, PMP), 161.70 (CO, β-lactam), 191.05 (C4, THPO).

(2R/2S)-2-(4-Fluorophenyl)-1-[trans-(3'R,4'R)-1'-(4-methoxyphenyl)-2'-oxo-4'-phenylazetididin-3'-yl]-1,2,3,4-tetrahydropyridin-4-one (**3d/4d**). – Obtained from (3R,4R)-**1d** (40.0 mg, 1.07 × 10⁻¹ mmol), diene **2a** (22.4 mg, 1.18 × 10⁻¹ mmol) and zinc(II) iodide (7.0 mg, 2.14 × 10⁻² mmol) as a diastereomeric mixture **3d/4d**, 38.5 mg (81 %), *R*_f = 0.30 (eluens-A). RP-HPLC analysis of diastereomeric mixture showed ratio of **3d/4d** – 70:30 % (**3d**, 24.32 min and **4d**, 24.76 min). LC-MS for C₂₇H₂₃FN₂O₃ (*M*_r = 442.4924): calcd. *m/z* [M+H]⁺ 443.49, found 443.20 (**3d**, 10.57 min) and 443.20 (**4d**, 11.02 min). IR (KBr) *v*_{max}/cm⁻¹: 3448, 2927, 1751, 1589, 1513, 1300, 1249, 831; ¹H NMR (CDCl₃) δ/ppm: 2.65 (dd, 1H, *J*₁ = 16.48 Hz, *J*₂ = 6.42 Hz, C3, THPO, **3d**), 2.76 (dd, 1H, *J*₁ = 16.40 Hz, *J*₂ = 8.49 Hz, C3, THPO, **4d**), 2.93 (dd, 1H, *J*₁ = 16.45 Hz, *J*₂ = 6.39 Hz, C3, THPO, **4d**), 3.04 (dd, 1H, *J*₁ = 16.51 Hz, *J*₂ = 7.26 Hz, C3, THPO, **3d**), 3.72 (s, 3H, OCH₃, **3d**), 3.73 (s, 3H, OCH₃, **4d**), 4.26 (d, 1H, *J* = 2.16 Hz, C4', β-lactam, **4d**), 4.29 (d, 1H, *J* = 2.10 Hz, C4', β-lactam, **3d**), 4.61 (bs, 1H, C2, THPO, **4d**), 4.63 (bs, 1H, C2, THPO, **3d**), 4.65 (d, 1H, *J* = 1.92 Hz, C3', β-lactam, **3d**), 4.70 (d, 1H, *J* = 2.16 Hz, C3', β-lactam, **4d**),

5.25 (d, $2 \times 1\text{H}$, $J = 7.92\text{ Hz}$, C5, THPO, **3d/4d**), 6.72-6.96 (m, $2 \times 2\text{H}$, C3 and C5, PMP, $2 \times 1\text{H}$, C6, THPO and $2 \times 2\text{H}$, C3 and C5, 4-F-Ph, **3d/4d**), 7.12-7.38 (m, $2 \times 2\text{H}$, C2 and C6, PMP, $2 \times 2\text{H}$, C2 and C6, 4-F-Ph and $2 \times 5\text{H}$, Ph, **3d/4d**); ^{13}C NMR (CDCl_3) δ/ppm : 43.41 (C3, THPO, **3d**), 43.63 (C3, THPO, **4d**), 55.38 ($2 \times \text{OCH}_3$, **3d/4d**), 60.95 (C2, THPO, **4d**), 62.94 (C4', β -lactam, **4d**), 63.25 (C2, THPO, **3d**), 64.99 (C4', β -lactam, **3d**), 76.32 (C3', β -lactam, **4d**), 76.81 (C3', β -lactam, **3d**), 101.40 (C5, THPO, **3d**), 102.63 (C5, THPO, **4d**), 114.38 ($2 \times \text{C3}$ and $2 \times \text{C5}$, PMP, **3d/4d**), 116.11 (d, $J = 18.58\text{ Hz}$, $2 \times \text{C3}$ and $2 \times \text{C5}$, 4-F-Ph, **3d/4d**), 118.96 ($2 \times \text{C2}$ and $2 \times \text{C6}$, PMP, **3d/4d**), 125.73 ($2 \times \text{C2}$ and $2 \times \text{C6}$, Ph, **3d/4d**), 128.80 (d, $J = 8.00\text{ Hz}$, $2 \times \text{C2}$ and $2 \times \text{C6}$, 4-F-Ph, **3d/4d**), 129.01 ($2 \times \text{C4}$, Ph, **3d/4d**), 129.08 ($2 \times \text{C3}$ and $2 \times \text{C5}$, Ph, **3d/4d**), 129.62 ($2 \times \text{C1}$, PMP, **3d/4d**), 134.81 ($2 \times \text{C1}$, Ph, **3d/4d**), 135.12 (d, $J = 3.27\text{ Hz}$, $2 \times \text{C1}$, 4-F-Ph, **3d/4d**), 148.66 (C6, THPO, **3d**), 149.86 (C6, THPO, **4d**), 156.73 ($2 \times \text{C4}$, PMP, **3d/4d**), 161.39 (CO, β -lactam, **4d**), 161.67 (CO, β -lactam, **3d**), 164.41 ($2 \times \text{C4}$, 4-F-Ph, **3d/4d**), 190.43 (C4, THPO, **3d**), 190.51 (C4, THPO, **4d**).

(2R/2S)-2-(4-Trifluoromethylphenyl)-1-[trans-(3'R,4'R)-1'-(4-methoxyphenyl)-2'-oxo-4'-phenylazetididin-3'-yl]-1,2,3,4-tetrahydropyridin-4-one (**3e/4e**). – Obtained from (3R,4R)-**1e** (30.0 mg, $7.07 \times 10^{-2}\text{ mmol}$), diene **2a** (13.8 mg, $7.78 \times 10^{-2}\text{ mmol}$) and zinc(II) iodide (4.5 mg, $1.41 \times 10^{-2}\text{ mmol}$) as a diastereomeric mixture **3e/4e**, 26.7 mg (77 %), $R_f = 0.26$ (eluens-A). RP-HPLC analysis of diastereomeric mixture showed ratio of **3e/4e** – 73:27 % (**3e**, 25.05 min and **4e**, 25.36 min). LC-MS for $\text{C}_{28}\text{H}_{23}\text{F}_3\text{N}_2\text{O}_3$ ($M_r = 492.5003$): calcd. m/z [$\text{M}+\text{H}$] $^+$ 493.50, found 493.20 (**3e**, 11.34 min) and 493.20 (**4e**, 11.68 min). IR (KBr) $\nu_{\text{max}}/\text{cm}^{-1}$: 3449, 1752, 1654, 1648, 1594, 1512, 1249, 830, 699; ^1H NMR (CDCl_3) δ/ppm : 2.65 (dd, 1H, $J_1 = 16.51\text{ Hz}$, $J_2 = 5.76\text{ Hz}$, C3, THPO, **3e**), 2.75 (dd, 1H, $J_1 = 16.33\text{ Hz}$, $J_2 = 7.47\text{ Hz}$, C3, THPO, **4e**), 3.01 (dd, 1H, $J_1 = 16.39\text{ Hz}$, $J_2 = 6.63\text{ Hz}$, C3, THPO, **4e**), 3.11 (dd, 1H, $J_1 = 16.54\text{ Hz}$, $J_2 = 7.53\text{ Hz}$, C3, THPO, **3e**), 3.72 (s, 3H, OCH_3 , **3e**), 3.73 (s, 3H, OCH_3 , **4e**), 4.28 (d, 1H, $J = 2.25\text{ Hz}$, C4', β -lactam, **4e**), 4.29 (d, 1H, $J = 2.16\text{ Hz}$, C4', β -lactam, **3e**), 4.65 (d, 1H, $J = 2.16\text{ Hz}$, C3', β -lactam, **3e**), 4.66-4.75 (m, $2 \times 1\text{H}$, C2, THPO, **3e/4e** and 1H, C3', β -lactam, **4e**), 5.24 (d, 1H, $J = 7.05\text{ Hz}$, C5, THPO, **3e**), 5.25 (d, 1H, $J = 7.92\text{ Hz}$, C5, THPO, **4e**), 6.73-6.81 (m, $2 \times 2\text{H}$, C3 and C5, PMP and $2 \times 1\text{H}$, C6, THPO, **3e/4e**), 7.12-7.54 (m, $2 \times 2\text{H}$, C2 and C6, PMP, $2 \times 4\text{H}$, C2, C3, C5 and C6, 4- CF_3 -Ph and $2 \times 5\text{H}$, Ph, **3e/4e**); ^{13}C NMR (CDCl_3) δ/ppm : 42.91 (C3, THPO, **3e**), 43.25 (C3, THPO, **4e**), 55.37 ($2 \times \text{OCH}_3$, **3e/4e**), 55.78 (d, $J = 358.59\text{ Hz}$, $2 \times \text{CF}_3$, **3e/4e**), 60.96 (C2, THPO, **4e**), 62.99 (C4', β -lactam, **4e**), 63.42 (C2, THPO, **3e**), 64.97 (C4', β -lactam, **3e**), 76.67 (C3', β -lactam, **4e**), 77.12 (C3', β -lactam, **3e**), 101.56 (C5, THPO, **3e**), 102.78 (C5, THPO, **4e**), 114.37 (C3 and C5, PMP, **3e**), 114.42 (C3 and C5, PMP, **4e**), 118.95 ($2 \times \text{C2}$ and $2 \times \text{C6}$, PMP, **3e/4e**), 125.59 (C2 and C6, Ph, **3e**), 125.68 (C2 and C6, Ph, **4e**), 126.10 (d, $J = 3.83\text{ Hz}$, C3 and C5, 4- CF_3 -Ph, **4e**), 126.23 (d, $J = 3.76\text{ Hz}$, C3 and C5, 4- CF_3 -Ph, **3e**), 127.23 (C3 and C5, Ph, **4e**), 127.37 (C3 and C5, Ph, **3e**), 129.12 ($2 \times \text{C2}$ and $2 \times \text{C6}$, 4- CF_3 -Ph, **3e/4e**), 129.30 (C4, Ph, **4e**), 129.49 (C4, Ph, **3e**), 129.60 ($2 \times \text{C1}$, PMP, **3e/4e**),

134.64 (C1, 4- CF_3 -Ph, **3e**), 135.14 (C1, 4- CF_3 -Ph, **4e**), 143.38 (C4, 4- CF_3 -Ph, **3e**), 143.40 (C4, 4- CF_3 -Ph, **4e**), 148.56 (C6, THPO, **3e**), 149.72 (C6, THPO, **4e**), 156.75 (C4, PMP, **3e**), 156.78 (C4, PMP, **4e**), 161.15 (CO, β -lactam, **4e**), 161.33 (CO, β -lactam, **3e**), 189.76 (C4, THPO, **4e**), 189.79 (C4, THPO, **3e**).

(2R/2S)-1-[trans-(3'R,4'R)-1'-(4-Methoxyphenyl)-2'-oxo-4'-phenylazetididin-3'-yl]-2-(2-nitrophenyl)-1,2,3,4-tetrahydropyridin-4-one (**3f/4f**). – Obtained from (3R,4R)-**1f** (15.0 mg, $3.74 \times 10^{-2}\text{ mmol}$), diene **2b** (10.7 mg, $4.11 \times 10^{-2}\text{ mmol}$) and zinc(II) iodide (2.6 mg, $7.47 \times 10^{-3}\text{ mmol}$) as a diastereomeric mixture **3f/4f**, 10.31 mg (59 %), $R_f = 0.19$ (eluens-C). RP-HPLC analysis of diastereomeric mixture showed ratio of **3f/4f** – 73:27 % (**3f**, 25.60 min and **4f**, 25.95 min). LC-MS for $\text{C}_{27}\text{H}_{23}\text{N}_3\text{O}_5$ ($M_r = 469.4995$): calcd. m/z [$\text{M}+\text{H}$] $^+$ 470.50, found 470.20 (**3f**, 10.52 min) and 470.20 (**4f**, 10.95 min). Diastereomeric mixture of **3f/4f** was separated by a silica gel column chromatography using eluens-C.

(2R)-1-[trans-(3'R,4'R)-1'-(4-Methoxyphenyl)-2'-oxo-4'-phenylazetididin-3'-yl]-2-(2-nitrophenyl)-1,2,3,4-tetrahydropyridin-4-one (**3f**). – 7.53 mg; IR (KBr) $\nu_{\text{max}}/\text{cm}^{-1}$: 3449, 2926, 1752, 1636, 1594, 1513, 1458, 1300, 1249, 830; ^1H NMR (CDCl_3) δ/ppm : 2.67 (dd, 1H, $J_1 = 16.81\text{ Hz}$, $J_2 = 3.30\text{ Hz}$, C3, THPO), 3.32 (dd, 1H, $J_1 = 16.81\text{ Hz}$, $J_2 = 8.70\text{ Hz}$, C3, THPO), 3.73 (s, 3H, OCH_3), 4.41 (d, 1H, $J = 2.10\text{ Hz}$, C3', β -lactam), 4.73 (d, 1H, $J = 2.10\text{ Hz}$, C4', β -lactam), 5.24 (d, 1H, $J = 7.80\text{ Hz}$, C5, THPO), 5.42 (dd, 1H, $J_1 = 8.70\text{ Hz}$, $J_2 = 3.30\text{ Hz}$, C2, THPO), 6.75 (d, 2H, $J = 9.00\text{ Hz}$, C3 and C5, PMP), 6.82 (d, 1H, $J = 6.60\text{ Hz}$, C6, THPO), 7.14 (d, 2H, $J = 8.70\text{ Hz}$, C2 and C6, PMP), 7.20-7.30 (m, 5H, Ph), 7.37 (d, 1H, $J = 7.80\text{ Hz}$, C6, Ph- NO_2), 7.48 (t, 1H, $J_{1,2} = 8.10\text{ Hz}$, C4, Ph- NO_2), 7.60 (t, 1H, $J_{1,2} = 7.80\text{ Hz}$, C5, Ph- NO_2), 7.88 (dd, 1H, $J_1 = 20.11\text{ Hz}$, $J_2 = 7.80\text{ Hz}$, C3, Ph- NO_2); ^{13}C NMR (CDCl_3) δ/ppm : 42.66 (C3, THPO), 55.38 (OCH_3), 62.55 (C2, THPO), 64.70 (C4', β -lactam), 77.16 (C3', β -lactam), 101.90 (C5, THPO), 114.38 (C3 and C5, PMP), 118.92 (C2 and C6, PMP), 124.38 (C3, Ph- NO_2), 125.70 (C2 and C6, Ph), 127.91 (C6, Ph- NO_2), 129.23 (C3 and C5, Ph), 129.48 (C1, PMP), 129.60 (C4, Ph), 131.38 (C4, Ph- NO_2), 133.49 (C5, Ph- NO_2), 134.56 (C1, Ph), 146.09 (C1, Ph- NO_2), 147.89 (C2, Ph- NO_2), 148.75 (C6, THPO), 156.78 (C4, PMP), 161.03 (CO, β -lactam), 189.39 (C4, THPO).

(2S)-1-[trans-(3'R,4'R)-1'-(4-Methoxyphenyl)-2'-oxo-4'-phenylazetididin-3'-yl]-2-(2-nitrophenyl)-1,2,3,4-tetrahydropyridin-4-one (**4f**). – 2.78 mg; IR (KBr) $\nu_{\text{max}}/\text{cm}^{-1}$: 3449, 2926, 1752, 1636, 1594, 1513, 1458, 1300, 1249, 830; ^1H NMR (CDCl_3) δ/ppm : 2.68 (dd, 1H, $J_1 = 16.81\text{ Hz}$, $J_2 = 5.70\text{ Hz}$, C3, THPO), 3.26 (dd, 1H, $J_1 = 16.81\text{ Hz}$, $J_2 = 7.80\text{ Hz}$, C3, THPO), 3.74 (s, 3H, OCH_3), 4.17 (d, 1H, $J = 2.10\text{ Hz}$, C3', β -lactam), 4.83 (d, 1H, $J = 1.80\text{ Hz}$, C4', β -lactam), 5.28 (d, 1H, $J = 8.10\text{ Hz}$, C5, THPO), 5.51 (m, 1H, C2, THPO), 6.75 (d, 2H, $J = 9.00\text{ Hz}$, C3 and C5, PMP), 6.83 (m, 1H, C6, THPO), 7.10-7.30 (m, 2H, C2 and C6, PMP and 5H, Ph), 7.34 (m, 1H, C6, Ph- NO_2), 7.46 (m, 1H, C4, Ph- NO_2), 7.67 (t, 1H, $J_{1,2} = 7.80\text{ Hz}$, C5, Ph- NO_2), 7.88 (dd, 1H, $J_1 = 23.71\text{ Hz}$, $J_2 = 8.10\text{ Hz}$, C3, Ph- NO_2);

^{13}C NMR (CDCl_3) δ/ppm : 43.02 (C3, THPO), 55.38 (OCH_3), 60.64 (C2, THPO), 63.06 (C4', β -lactam), 76.87 (C3', β -lactam), 103.02 (C5, THPO), 114.44 (C3 and C5, PMP), 118.92 (C2 and C6, PMP), 124.38 (C3, Ph- NO_2), 125.63 (C2 and C6, Ph), 127.72 (C6, Ph- NO_2), 129.35 (C3 and C5, Ph), 129.48 (C1, PMP), 129.60 (C4, Ph), 131.38 (C4, Ph- NO_2), 133.49 (C5, Ph- NO_2), 134.99 (C1, Ph), 144.89 (C1, Ph- NO_2), 148.05 (C2, Ph- NO_2), 149.57 (C6, THPO), 156.78 (C4, PMP), 160.95 (CO, β -lactam), 189.36 (C4, THPO).

(2R/2S)-1-[trans-(3'R,4'R)-1'-(4-Methoxyphenyl)-2'-oxo-4'-phenylazetididin-3'-yl]-2-(3-nitrophenyl)-1,2,3,4-tetrahydropyridin-4-one (**3g/4g**). – Obtained from (3R,4R)-**1g** (15.0 mg, 3.74×10^{-2} mmol), diene **2b** (10.7 mg, 4.11×10^{-2} mmol) and zinc(II) iodide (2.6 mg, 7.47×10^{-3} mmol) as a diastereomeric mixture **3g/4g**, 14.89 mg (85 %), $R_f = 0.12$ (eluens-A). RP-HPLC analysis of diastereomeric mixture showed ratio of **3g/4g** – 73:27 % (**3g**, 25.70 min and **4g**, 26.05 min). LC-MS for $\text{C}_{27}\text{H}_{23}\text{N}_3\text{O}_5$ ($M_r = 469.4995$): calcd. m/z [$\text{M}+\text{H}$] $^+$ 470.50, found 470.20 (**3g**, 10.52 min) and 470.20 (**4g**, 10.86 min). IR (KBr) $\nu_{\text{max}}/\text{cm}^{-1}$: 3448, 1751, 1647, 1593, 1530, 1513, 1457, 1349, 1249, 831, 700; ^1H NMR (CDCl_3) δ/ppm : 2.70 (dd, 1H, $J_1 = 16.51$ Hz, $J_2 = 5.70$ Hz, C3, THPO, **3g**), 2.77 (m, 1H, C3, THPO, **4g**), 3.00-3.18 (m, $2 \times$ 1H, C3, THPO, **3g/4g**), 3.72 (s, 3H, OCH_3 , **3g**), 3.73 (s, 3H, OCH_3 , **4g**), 4.27 (d, 1H, $J = 1.80$ Hz, C3', β -lactam, **4g**), 4.32 (d, 1H, $J = 1.50$ Hz, C3', β -lactam, **3g**), 4.72 (d, 1H, $J = 1.80$ Hz, C4', β -lactam, **3g**), 4.75-4.85 (m, 1H, C4', β -lactam, **4g** and $2 \times$ 1H, C2, THPO, **3g/4g**), 5.28 (d, 1H, $J = 7.80$ Hz, C5, THPO, **4g**), 5.29 (d, 1H, $J = 8.10$ Hz, C5, THPO, **3g**), 6.70-6.80 (m, $2 \times$ 2H, C3 and C5, PMP, **3g/4g**), 6.89 (d, $2 \times$ 1H, $J = 7.20$ Hz, C6, THPO, **3g/4g**), 7.10-7.55 (m, $2 \times$ 2H, C2 and C6, PMP and $2 \times$ 5H, Ph, **3g/4g**), 7.60-7.70 (m, $2 \times$ 1H, C5, Ph- NO_2 , **3g/4g**), 8.00-8.15 (m, $2 \times$ 2H, C4 and C6, Ph- NO_2 , **3g/4g**); ^{13}C NMR (CDCl_3) δ/ppm : 43.01 (C3, THPO, **3g**), 43.22 (C3, THPO, **4g**), 55.39 ($2 \times$ OCH_3 , **3g/4g**), 60.70 (C2, THPO, **4g**), 62.71 (C2, THPO, **3g**), 62.97 (C4', β -lactam, **4g**), 64.75 (C4', β -lactam, **3g**), 76.80 (C3', β -lactam, **4g**), 77.17 (C3', β -lactam, **3g**), 102.09 (C5, THPO, **3g**), 103.26 (C5, THPO, **4g**), 114.40 (C3 and C5, PMP, **3g**), 114.45 (C3 and C5, PMP, **4g**), 118.92 (C2 and C6, PMP, **4g**), 118.95 (C2 and C6, PMP, **3g**), 121.89 (C4, Ph- NO_2 , **3g**), 122.03 (C4, Ph- NO_2 , **4g**), 123.65 ($2 \times$ C2, Ph- NO_2 , **3g/4g**), 125.56 ($2 \times$ C2 and $2 \times$ C6, Ph, **3g/4g**), 129.21 (C3 and C5, Ph, **3g**), 129.29 (C3 and C5, Ph, **4g**), 129.43 (C5, Ph- NO_2 , **4g**), 129.54 ($2 \times$ C1, PMP, **3g/4g**), 129.61 (C5, Ph- NO_2 , **3g**), 130.40 ($2 \times$ C4, Ph, **3g/4g**), 132.63 (C6, Ph- NO_2 , **4g**), 133.08 (C6, Ph- NO_2 , **3g**), 134.55 (C1, Ph, **3g**), 134.95 (C1, Ph, **4g**), 139.69 (C1, Ph- NO_2 , **4g**), 141.22 (C1, Ph- NO_2 , **3g**), 148.35 ($2 \times$ C3, Ph- NO_2 , **3g/4g**), 148.68 (C6, THPO, **3g**), 149.54 (C6, THPO, **4g**), 156.80 (C4, PMP, **3g**), 156.82 (C4, PMP, **4g**), 160.94 (CO, β -lactam, **4g**), 161.15 (CO, β -lactam, **3g**), 189.33 (C4, THPO, **4g**), 189.36 (C4, THPO, **3g**).

(2R/2S)-1-[trans-(3'R,4'R)-1'-(4-Methoxyphenyl)-2'-oxo-4'-phenylazetididin-3'-yl]-2-(4-nitrophenyl)-1,2,3,4-tetrahydropyridin-4-one (**3h/4h**). – Obtained from (3R,4R)-**1h** (15.0 mg, 3.74×10^{-2} mmol), diene **2b** (10.7 mg, 4.11×10^{-2}

mmol) and zinc(II) iodide (2.6 mg, 7.47×10^{-3} mmol) as a diastereomeric mixture **3h/4h**, 17.1 mg (97 %), $R_f = 0.11$ (eluens-A). RP-HPLC analysis of diastereomeric mixture showed ratio of **3h/4h** – 78:22 % (**3h**, 25.70 min and **4h**, 25.95 min). LC-MS for $\text{C}_{27}\text{H}_{23}\text{N}_3\text{O}_5$ ($M_r = 469.4995$): calcd. m/z [$\text{M}+\text{H}$] $^+$ 470.50, found 470.20 (**3h**, 10.50 min) and 470.20 (**4h**, 10.85 min). IR (KBr) $\nu_{\text{max}}/\text{cm}^{-1}$: 3449, 2925, 1750, 1647, 1588, 1512, 1346, 1248, 830, 698; ^1H NMR (CDCl_3) δ/ppm : 2.62-2.78 (m, $2 \times$ 1H, C3, THPO, **3h/4h**), 3.02-3.08 (m, 1H, C3, THPO, **4h**), 3.14 (dd, 1H, $J_1 = 16.51$ Hz, $J_2 = 7.50$ Hz, C3, THPO, **3h**), 3.72 (s, 3H, OCH_3 , **3h**), 3.74 (s, 3H, OCH_3 , **4h**), 4.28 (d, 1H, $J = 2.10$ Hz, C3', β -lactam, **4h**), 4.33 (d, 1H, $J = 2.10$ Hz, C3', β -lactam, **3h**), 4.70 (d, 1H, $J = 2.10$ Hz, C4', β -lactam, **3h**), 4.76 (d, 1H, $J = 2.10$ Hz, C4', β -lactam, **4h**), 4.78-4.85 (m, $2 \times$ 1H, C2, THPO, **3h/4h**), 5.27 (d, 1H, $J = 7.80$ Hz, C5, THPO, **4h**), 5.28 (d, 1H, $J = 8.10$ Hz, C5, THPO, **3h**), 6.75 (d, $2 \times$ 2H, $J = 9.00$ Hz, C3 and C5, Ph, **3h/4h**), 6.90 (d, $2 \times$ 1H, $J = 6.90$ Hz, C6, THPO, **3h/4h**), 7.14 (d, $2 \times$ 2H, $J = 9.00$ Hz, C2 and C6, Ph, **3h/4h**), 7.15-7.40 (m, $2 \times$ 5H, Ph, **3h/4h**), 7.43 (d, $2 \times$ 2H, $J = 8.70$ Hz, C2 and C6, Ph- NO_2 , **3h/4h**), 8.02 (d, 2H, $J = 8.70$ Hz, C3 and C5, Ph- NO_2 , **3h**), 8.14 (d, 2H, $J = 8.70$ Hz, C2 and C6, Ph- NO_2 , **4h**); ^{13}C NMR (CDCl_3) δ/ppm : 42.65 (C3, THPO, **3h**), 44.87 (C3, THPO, **4h**), 55.37 ($2 \times$ OCH_3 , **3h/4h**), 60.64 (C2, THPO, **4h**), 62.54 (C2, THPO, **3h**), 63.05 (C4', β -lactam, **4h**), 64.69 (C4', β -lactam, **3h**), 76.86 (C3', β -lactam, **4h**), 77.15 (C3', β -lactam, **3h**), 101.89 (C5, THPO, **3h**), 103.01 (C5, THPO, **4h**), 114.37 (C3 and C5, PMP, **3h**), 114.43 (C3 and C5, PMP, **4h**), 118.91 ($2 \times$ C2 and $2 \times$ C6, PMP, **3h/4h**), 124.37 ($2 \times$ C3 and $2 \times$ C5, Ph- NO_2 , **3h/4h**), 125.62 (C2 and C6, Ph, **4h**), 125.69 (C2 and C6, Ph, **3h**), 127.71 (C2 and C6, Ph- NO_2 , **4h**), 127.90 (C2 and C6, Ph- NO_2 , **3h**), 129.22 (C3 and C5, Ph, **3h**), 129.35 (C3 and C5, Ph, **4h**), 129.47 ($2 \times$ C1, PMP, **3h/4h**), 129.59 ($2 \times$ C4, Ph, **3h/4h**), 134.53 (C1, Ph, **4h**), 134.55 (C1, Ph, **3h**), 144.88 ($2 \times$ C1, Ph- NO_2 , **3h/4h**), 146.08 (C4, Ph- NO_2 , **3h**), 147.88 (C4, Ph- NO_2 , **4h**), 148.74 (C6, THPO, **3h**), 149.54 (C6, THPO, **4h**), 156.77 ($2 \times$ C4, PMP, **3h/4h**), 160.94 (CO, β -lactam, **4h**), 161.02 (CO, β -lactam, **3h**), 189.34 (C4, THPO, **4h**), 189.38 (C4, THPO, **3h**).

(2R/2S)-2-Cyclohexyl-1-[trans-(3'R,4'R)-1'-(4-methoxyphenyl)-2'-oxo-4'-phenylazetididin-3'-yl]-1,2,3,4-tetrahydropyridin-4-one (**3i/4i**). – Obtained from (3R,4R)-**1i** (30.0 mg, 8.28×10^{-2} mmol), diene **2a** (17.1 mg, 9.11×10^{-2} mmol) and zinc(II) iodide (5.3 mg, 1.66×10^{-2} mmol) as a diastereomeric mixture **3i/4i**, 14.02 mg (39 %), $R_f = 0.30$ (eluens-A). RP-HPLC analysis of diastereomeric mixture showed ratio of **3i/4i** – 60:40 % (**3i**, 25.17 min and **4i**, 25.78 min). LC-MS for $\text{C}_{27}\text{H}_{30}\text{N}_2\text{O}_3$ ($M_r = 430.5496$): calcd. m/z [$\text{M}+\text{H}$] $^+$ 431.55, found 431.20 (**3i**, 11.39 min) and 431.20 (**4i**, 12.04 min). IR (KBr) $\nu_{\text{max}}/\text{cm}^{-1}$: 3468, 2926, 2851, 1751, 1645, 1591, 1513, 1386, 1300, 1248, 830; ^1H NMR (CDCl_3) δ/ppm : 1.00-1.30 (m, $2 \times$ 6H, cyclohexyl, **3i/4i**), 1.55-1.80 (m, $2 \times$ 5H, cyclohexyl, **3i/4i**), 2.45-2.55 (m, $2 \times$ 1H, C2, THPO, **3i/4i**), 3.75 (s, 3H, OCH_3 , **3i**), 3.76 (s, 3H, OCH_3 , **4i**), 4.32 (d, 1H, $J = 2.04$ Hz, C4', β -lactam, **3i**), 4.39 (d, 1H, $J = 2.10$ Hz, C4', β -lactam, **4i**), 4.84 (d, 1H, $J = 1.95$ Hz, C3', β -lactam, **4i**), 4.87 (d, 1H, $J = 1.98$ Hz, C3',

β -lactam, **3i**), 5.03 (d, 1H, $J = 7.80$ Hz, C5, THPO, **3i**), 5.10 (d, 1H, $J = 7.59$ Hz, C5, THPO, **4i**), 6.79 (d, 2H, $J = 9.03$ Hz, C3 and C5, PMP, **3i**), 6.78–6.82 (m, 2H, C3 and C5, PMP, **4i**), 7.09 (d, 2 \times 1H, $J = 7.83$ Hz, C6, THPO, **3i/4i**), 7.19 (m, 2H, C2 and C6, PMP, **4i**), 7.24 (d, 2H, $J = 9.09$ Hz, C2 and C6, PMP, **3i**), 7.30–7.44 (m, 2 \times 5H, Ph, **3i/4i**); ^{13}C NMR (CDCl_3) δ/ppm : 26.07 (C2 and C6, cyclohexyl, **4i**), 26.10 (C2 and C6, cyclohexyl, **3i**), 27.85 (C3 and C5, cyclohexyl, **3i**), 28.45 (C3 and C5, cyclohexyl, **4i**), 29.25 (C4, cyclohexyl, **3i**), 29.83 (C4, cyclohexyl, **4i**), 36.68 (C3, THPO, **3i**), 37.70 (C3, THPO, **4i**), 38.95 (C1, cyclohexyl, **4i**), 41.92 (C1, cyclohexyl, **3i**), 55.41 (2 \times OCH_3 , **3i/4i**), 63.67 (C2, THPO, **4i**), 64.58 (C2, THPO, **3i**), 65.32 (C4', β -lactam, **4i**), 65.37 (C4', β -lactam, **3i**), 77.86 (C3', β -lactam, **4i**), 78.40 (C3', β -lactam, **3i**), 100.22 (C5, THPO, **3i**), 101.37 (C5, THPO, **4i**), 114.43 (C3 and C5, PMP, **3i**), 114.46 (C3 and C5, PMP, **4i**), 119.00 (C2 and C6, PMP, **4i**), 119.06 (C2 and C6, PMP, **3i**), 125.57 (C2 and C6, Ph, **4i**), 126.20 (C2 and C6, Ph, **3i**), 129.23 (C4, Ph, **4i**), 129.43 (C3 and C5, Ph, **4i**), 129.48 (C3 and C5, Ph, **3i**), 129.61 (C4, Ph, **3i**), 129.76 (2 \times C1, PMP, **3i/4i**), 135.22 (2 \times C1, Ph, **3i/4i**), 147.91 (C6, THPO, **4i**), 148.02 (C6, THPO, **3i**), 156.73 (2 \times C4, PMP, **3i/4i**), 162.04 (2 \times CO, β -lactam, **3i/4i**), 191.52 (C4, THPO, **3i**), 191.64 (C4, THPO, **4i**).

(2R/2S)-2-tert-Butyl-1-[trans-(3'R,4'R)-1'-(4-methoxyphenyl)-2'-oxo-4'-phenylazetidin-3'-yl]-1,2,3,4-tetrahydropyridin-4-one (**3j/4j**). – Obtained from (3R,4R)-**1j** (18.0 mg, 5.35×10^{-2} mmol), diene **2a** (12.1 mg, 5.89×10^{-2} mmol) and zinc(II) iodide (3.4 mg, 1.07×10^{-2} mmol) as a diastereomeric mixture **3j/4j**, 9.6 mg (44 %), $R_f = 0.37$ (eluens-A). RP-HPLC analysis of diastereomeric mixture showed ratio of **3j/4j** – 55:45 % (**3j**, 25.20 min and **4j**, 25.70 min). LC-MS for $\text{C}_{25}\text{H}_{28}\text{N}_2\text{O}_3$ ($M_r = 404.5115$): calcd. m/z [$\text{M}+\text{H}$] $^+$ 405.51, found 405.20 (**3j**, 10.60 min) and 405.20 (**4j**, 11.25 min). IR (KBr) $\nu_{\text{max}}/\text{cm}^{-1}$: 3468, 2926, 2851, 1751, 1645, 1591, 1513, 1386, 1300, 1248, 830; ^1H NMR (CDCl_3) δ/ppm : 0.83 (s, 9H, $\text{C}(\text{CH}_3)_3$, **3j**), 1.00 (s, 9H, $\text{C}(\text{CH}_3)_3$, **4j**), 2.56–3.28 (m, 2 \times 2H, C3 and 2 \times 1H, C2, THPO, **3j/4j**), 3.74 (s, 3H, OCH_3 , **3j**), 3.75 (s, 3H, OCH_3 , **4j**), 4.40 (d, 2 \times 1H, $J = 1.56$ Hz, C4', β -lactam, **3j/4j**), 4.82 (d, 1H, $J = 1.89$ Hz, C3', β -lactam, **4j**), 5.06 (d, 1H, $J = 2.04$ Hz, C3', β -lactam, **3j**), 5.10 (d, 1H, $J = 8.58$ Hz, C5, THPO, **3j**), 5.13 (d, 1H, $J = 8.64$ Hz, C5, THPO, **4j**), 6.78 (d, 2H, $J = 8.79$ Hz, C3 and C5, PMP, **3j**), 6.81 (d, 2H, $J = 8.64$ Hz, C3 and C5, PMP, **4j**), 7.11 (d, 2 \times 1H, $J = 7.74$ Hz, C6, THPO, **3j/4j**), 7.23 (d, 2H, $J = 8.97$ Hz, C2 and C6, PMP, **4j**), 7.24 (d, 2H, $J = 9.00$ Hz, C2 and C6, PMP, **3j**), 7.25–7.45 (m, 2 \times 5H, Ph, **3j/4j**); ^{13}C NMR (CDCl_3) δ/ppm : 27.06 ($\text{C}(\text{CH}_3)_3$, **4j**), 27.40 ($\text{C}(\text{CH}_3)_3$, **3j**), 36.88 (C3, THPO, **4j**), 36.92 (C3, THPO, **3j**), 37.91 ($\text{C}(\text{CH}_3)_3$, **3j**), 38.75 ($\text{C}(\text{CH}_3)_3$, **4j**), 55.40 (OCH_3 , **3j**), 55.41 (OCH_3 , **4j**), 64.19 (C2, THPO, **3j**), 66.14 (C2, THPO, **4j**), 67.34 (C4', β -lactam, **4j**), 69.29 (C4', β -lactam, **3j**), 79.83 (C3', β -lactam, **4j**), 80.24 (C3', β -lactam, **3j**), 101.66 (C5, THPO, **3j**), 102.44 (C5, THPO, **4j**), 114.41 (C3 and C5, PMP, **3j**), 114.46 (C3 and C5, PMP, **4j**), 119.04 (2 \times C2 and 2 \times C6, PMP, **3j/4j**), 125.49 (C2 and C6, Ph, **4j**), 126.48 (C2 and C6, Ph, **3j**), 129.17 (C4, Ph, **4j**), 129.32 (C4, Ph, **3j**), 129.39 (C3 and C5, Ph, **3j**), 129.64 (C3 and C5, Ph, **4j**),

129.72 (C1, PMP, **3j**), 129.79 (C1, PMP, **4j**), 135.62 (C1, Ph, **3j**), 135.85 (C1, Ph, **4j**), 147.63 (C6, THPO, **3j**), 147.83 (C6, THPO, **4j**), 156.66 (C4, PMP, **3j**), 156.71 (C4, PMP, **4j**), 161.57 (CO, β -lactam, **4j**), 162.94 (CO, β -lactam, **3j**), 191.68 (C4, THPO, **4j**), 192.01 (C4, THPO, **4j**).

(2R/2S)-2-Ferrocenyl-1-[trans-(3'R,4'R)-1'-(4-methoxyphenyl)-2'-oxo-4'-phenylazetidin-3'-yl]-1,2,3,4-tetrahydropyridin-4-one (**3k/4k**). – Obtained from (3R,4R)-**1k** (30.0 mg, 6.46×10^{-2} mmol), diene **2a** (13.8 mg, 7.11×10^{-2} mmol) and zinc(II) iodide (3.8 mg, 1.29×10^{-2} mmol) as a diastereomeric mixture **3k/4k**, 3.6 mg (11 %), $R_f = 0.17$ (eluens-A). RP-HPLC analysis of diastereomeric mixture showed ratio of **3k/4k** – 85:15 % (**3k**, 25.57 min and **4k**, 25.82 min). LC-MS for $\text{C}_{31}\text{H}_{28}\text{FeN}_2\text{O}_3$ ($M_r = 532.4251$): calcd. m/z [$\text{M}+\text{H}$] $^+$ 533.43, found 533.00 (**3k**, 11.84 min) and 533.00 (**4k**, 12.11 min). IR (KBr) $\nu_{\text{max}}/\text{cm}^{-1}$: 3448, 2928, 1750, 1638, 1590, 1513, 1299, 1249, 1224, 830; ^1H NMR (CDCl_3) δ/ppm : 3.03 (dd, 1H, $J_1 = 16.27$ Hz, $J_2 = 5.16$ Hz, C3, THPO, **3k**), 3.19 (dd, 1H, $J_1 = 16.31$ Hz, $J_2 = 6.83$ Hz, C3, THPO, **3k**), 2.98–3.22 (m, 2H, C3, THPO, **4k**), 3.66 (m, 2 \times 1H, Fc, **3k/4k**), 3.71 (s, 3H, OCH_3 , **3k**), 3.75 (s, 3H, OCH_3 , **4k**), 3.89 (m, 2 \times 1H, Fc, **3k/4k**), 4.03 (m, 2 \times 1H, Fc, **3k/4k**), 4.12 (s, 2 \times 5H, Fc, **3k/4k**), 4.15 (m, 2 \times 1H, Fc, **3k/4k**), 4.41 (d, 2 \times 1H, $J = 2.01$ Hz, C4', β -lactam, **3k/4k**), 4.40–4.47 (m, 2 \times 1H, C2, THPO, **3k/4k**), 4.49 (d, 1H, $J = 1.80$ Hz, C3', β -lactam, **3k**), 4.59 (d, 1H, $J = 2.01$ Hz, C3', β -lactam, **4k**), 5.20 (d, 2 \times 1H, $J = 7.77$ Hz, C5, THPO, **3k/4k**), 6.73 (d, 2H, $J = 7.14$ Hz, C3 and C5, PMP, **3k**), 6.78 (d, 2H, $J = 9.12$ Hz, C3 and C5, PMP, **4k**), 6.96 (d, 2 \times 1H, $J = 7.83$ Hz, C6, THPO, **3k/4k**), 7.03–7.10 (m, 2 \times 2H, C3 and C5, Ph, **3k/4k**), 7.15 (d, 2 \times 2H, $J = 8.94$ Hz, C2 and C6, PMP, **3k/4k**), 7.27–7.33 (m, 2 \times 2H, C2 and C6, Ph, **3k/4k**), 7.36–7.40 (m, 2 \times 1H, C4, Ph, **3k/4k**); ^{13}C NMR (CDCl_3) δ/ppm : 42.25 (C3, THPO, **3k**), 42.97 (C3, THPO, **4k**), 55.37 (2 \times OCH_3 , **3k/4k**), 57.16 (C4', β -lactam, **4k**), 59.33 (C4', β -lactam, **3k**), 63.72 (C2, THPO, **4k**), 65.18 (C2, THPO, **3k**), 66.55 (**3k/4k**), 67.64 (**4k**), 68.05 (**3k**), 68.90 (5C, **3k**), 68.99 (5C, **4k**), 69.01 (**3k**), 69.12 (**4k**), 69.25 (**3k**), 69.33 (**4k**) (Fc), 75.39 (C3', β -lactam, **4k**), 76.06 (C3', β -lactam, **3k**), 85.74 (2 \times C1, Fc, **3k/4k**), 101.28 (C5, THPO, **3k**), 101.58 (C5, THPO, **4k**), 114.34 (C3 and C5, PMP, **3k**), 114.39 (C3 and C5, PMP, **4k**), 119.01 (2 \times C2 and 2 \times C6, PMP, **3k/4k**), 125.90 (C2 and C6, Ph, **4k**), 126.78 (C2 and C6, Ph, **3k**), 128.96 (C3 and C5, Ph, **3k**), 129.08 (C4, Ph, **3k**), 129.22 (C4, Ph, **4k**), 129.46 (C3 and C5, Ph, **4k**), 129.76 (2 \times C1, PMP, **3k/4k**), 135.02 (2 \times C1, Ph, **3k/4k**), 147.05 (C6, THPO, **3k**), 149.09 (C6, THPO, **4k**), 156.64 (2 \times C4, PMP, **3k/4k**), 162.11 (2 \times CO, β -lactam, **3k/4k**), 191.68 (C4, THPO, **3k**), 191.76 (C4, THPO, **4k**).

(2R/2S)-1-[trans-(3'R,4'R)-1',4'-bis(4-Methoxyphenyl)-2'-oxoazetidin-3'-yl]-2-phenyl-1,2,3,4-tetrahydropyridin-4-one (**3l/4l**). – Obtained from (3R,4R)-**1l** (20.0 mg, 5.17×10^{-2} mmol), diene **2b** (12.9 mg, 5.69×10^{-2} mmol) and zinc(II) iodide (3.2 mg, 1.04×10^{-2} mmol) as a diastereomeric mixture **3l/4l**, 13.6 mg (58 %), $R_f = 0.19$ (eluens-A). RP-HPLC analysis of diastereomeric mixture showed ratio of **3l/4l** – 68:32 % (**3l**, 24.34 min and **4l**, 24.74 min). LC-MS for

$C_{28}H_{26}N_2O_4$ ($M_r = 454.5283$): calcd. m/z $[M+H]^+$ 455.53, found 455.20 (**3I**, 10.44 min) and 455.20 (**4I**, 10.83 min). IR (KBr) ν_{max}/cm^{-1} : 3467, 2918, 1750, 1591, 1513, 1457, 1300, 1249, 1179, 831; 1H NMR ($CDCl_3$) δ/ppm : 2.66 (dd, 1H, $J_1 = 16.56$ Hz, $J_2 = 6.12$ Hz, C3, THPO, **3I**), 2.79 (dd, 1H, $J_1 = 16.44$ Hz, $J_2 = 8.22$ Hz, C3, THPO, **4I**), 2.94 (dd, 1H, $J_1 = 16.44$ Hz, $J_2 = 8.22$ Hz, C3, THPO, **4I**), 3.07 (dd, 1H, $J_1 = 16.56$ Hz, $J_2 = 7.38$ Hz, C3, THPO, **3I**), 3.72 (s, 3H, OCH₃, **3I**), 3.73 (s, 3H, OCH₃, **4I**), 3.76 (s, 3H, OCH₃, **3I**), 3.80 (s, 3H, OCH₃, **4I**), 4.27 (d, 1H, $J = 1.44$ Hz, C3', β -lactam, **3I**), 4.29 (s, 1H, C3', β -lactam, **4I**), 4.59-4.63 (m, 2 \times 1H, C2, THPO, **3I/4I**), 4.64 (s, 1H, C4', β -lactam, **3I**), 4.69 (s, 1H, C4', β -lactam, **4I**), 5.20-5.25 (m, 2 \times 1H, C5, THPO, **3I/4I**), 6.66-6.72 (m, 2 \times 2H, C3 and C5, PMP, **3I/4I**), 6.72-6.78 (m, 2 \times 2H, C3 and C5, PMP, **3I/4I**), 6.85 (d, 1H, $J = 8.46$ Hz, C6, THPO, **4I**), 7.04 (d, 1H, $J = 8.46$ Hz, C6, THPO, **3I**), 7.14-7.20 (m, 2 \times 4H, C2 and C6, PMP, **3I/4I**), 7.20-7.28 (m, 2 \times 5H, Ph, **3I/4I**), 7.30 (d, 2 \times 2H, $J = 7.98$ Hz, C2 and C6-Ph, **3I/4I**); ^{13}C NMR ($CDCl_3$) δ/ppm : 43.50 (C3, THPO, **3I**), 43.57 (C3, THPO, **4I**), 55.27 (OCH₃, **3I**), 55.34 (OCH₃, **4I**), 55.39 (2 \times OCH₃, **3I/4I**), 61.42 (C2, THPO, **4I**), 62.50 (C4', β -lactam, **4I**), 64.12 (C2, THPO, **3I**), 64.67 (C4', β -lactam, **3I**), 76.52 (C3', β -lactam, **4I**), 76.97 (C3', β -lactam, **3I**), 101.19 (C5, THPO, **3I**), 102.28 (C5, THPO, **4I**), 114.34 (C3 and C5, PMP, **3I**), 114.36 (2 \times C3 and 2 \times C5, PMP, **3I/4I**), 114.74 (C3 and C5, PMP, **4I**), 119.00 (C2 and C6, PMP, **4I**), 119.03 (C2 and C6, PMP, **3I**), 126.76 (2 \times C1, PMP, **3I/4I**), 126.91 (C2 and C6, PMP, **4I**), 126.99 (C2 and C6, PMP, **3I**), 127.07 (C2 and C6, Ph, **3I**), 127.13 (C2 and C6, Ph, **3I**), 128.67 (C4, Ph, **4I**), 128.70 (C4, Ph, **3I**), 129.10 (C3 and C5, Ph, **4I**), 129.22 (C3 and C5, Ph, **3I**), 129.82 (2 \times C1, PMP, **3I/4I**), 137.84 (C1, Ph, **4I**), 139.47 (C1, Ph, **3I**), 148.69 (C6, THPO, **3I**), 150.06 (C6, THPO, **4I**), 156.64 (2 \times C4, PMP, **3I/4I**), 159.87 (2 \times C4, PMP, **3I/4I**), 161.72 (CO, β -lactam, **4I**), 161.82 (CO, β -lactam, **3I**), 190.47 (C4, THPO, **4I**), 190.57 (C4, THPO, **3I**).

(2R/2S)-2-(4-Methoxyphenyl)-1-[trans-(3'R,4'R)-1',4'-bis(4-methoxyphenyl)-2'-oxoazetidin-3'-yl]-1,2,3,4-tetrahydropyridin-4-one (**3m/4m**). – Obtained from (3R,4R)-**1m** (50.0 mg, 1.20×10^{-1} mmol), diene **2a** (25.8 mg, 1.32×10^{-1} mmol) and zinc(II) iodide (6.4 mg, 2.40×10^{-2} mmol) as a diastereomeric mixture **3m/4m**, 32.2 mg (55 %), $R_f = 0.30$ (eluens-A). RP-HPLC analysis of diastereomeric mixture showed ratio of **3m/4m** – 68:32 % (**3m**, 24.32 min and **4m**, 24.71 min). LC-MS for $C_{29}H_{28}N_2O_5$ ($M_r = 484.5547$): calcd m/z $[M+H]^+$ 485.55, found 485.20 (**3m**, 10.40 min) and 485.20 (**4m**, 10.79 min). IR (KBr) ν_{max}/cm^{-1} : 3467, 2918, 1750, 1591, 1513, 1457, 1300, 1249, 1179, 831; 1H NMR ($CDCl_3$) δ/ppm : 2.69 (dd, 1H, $J_1 = 16.56$ Hz, $J_2 = 7.38$ Hz, C3, THPO, **3m**), 2.79 (dd, 1H, $J_1 = 16.44$ Hz, $J_2 = 9.12$ Hz, C3, THPO, **4m**), 2.87 (dd, 1H, $J_1 = 16.38$ Hz, $J_2 = 6.18$ Hz, C3, THPO, **4m**), 2.98 (dd, 1H, $J_1 = 16.56$ Hz, $J_2 = 7.02$ Hz, C3, THPO, **3m**), 3.72 (s, 3H, OCH₃, PMP, THPO, **3m**), 3.73 (s, 3H, OCH₃, PMP, THPO, **4m**), 3.74 (s, 2 \times 3H, OCH₃, PMP, **3m/4m**), 3.75 (bs, 2 \times 1H, C4', β -lactam, **3m/4m**), 3.77 (s, 3H, OCH₃, PMP, **3m**), 3.80 (s, 3H, OCH₃, PMP, **4m**), 4.26 (d, 1H, $J = 1.80$ Hz, C3', β -lactam, **4m**), 4.27 (d, 1H, $J = 1.62$ Hz, C3', β -lactam, **3m**), 4.56-4.58 (m, 2

\times 1H, C2, THPO, **3m/4m**), 4.59 (d, 1H, $J = 1.38$ Hz, C4', β -lactam, **3m**), 4.64 (d, 1H, $J = 1.38$ Hz, C4', β -lactam, **4m**), 5.22 (d, $J = 7.86$ Hz, 2 \times 1H, C5, THPO, **3m/4m**), 6.64-6.82 (m, 2 \times 6H, C3 and C5, PMP, **3m/4m**), 6.83 (d, 2 \times 1H, $J = 8.58$ Hz, C6, THPO, **3m/4m**), 7.04-7.28 (m, 2 \times 6H, C2 and C6, PMP, **3m/4m**); ^{13}C NMR ($CDCl_3$) δ/ppm : 43.61 (C3, THPO, **3m**), 43.76 (C3, THPO, **4m**), 55.15 (OCH₃, **3m**), 55.22 (OCH₃, **3m**), 55.24 (OCH₃, **4m**), 55.32 (OCH₃, **4m**), 55.36 (OCH₃, **3m**), 55.38 (OCH₃, **4m**), 61.14 (C2, THPO, **4m**), 62.59 (C4', β -lactam, **4m**), 63.54 (C2, THPO, **3m**), 64.78 (C4', β -lactam, **3m**), 76.15 (C3', β -lactam, **4m**), 76.52 (C3', β -lactam, **3m**), 101.06 (C5, THPO, **3m**), 102.22 (C5, THPO, **4m**), 114.27 (**4m**), 114.30 (**3m**), 114.33 (**3m**), 114.35 (**4m**), 114.46 (**3m**), 114.70 (**4m**) (C3 and C5, PMP), 118.98 (2 \times C2 and 2 \times C6, PMP, β -lactam, **3m/4m**), 126.81 (2 \times C1, PMP, β -lactam, **3m/4m**), 127.15 (C2 and C6, PMP, C4', β -lactam, **4m**), 127.21 (C2 and C6, PMP, C4', β -lactam, **3m**), 128.30 (C2 and C6, PMP, THPO, **4m**), 128.40 (C2 and C6, PMP, THPO, **3m**), 129.82 (C1, PMP, THPO, **4m**), 129.85 (C1, PMP, THPO, **3m**), 131.12 (C1, PMP, C4', β -lactam, **3m**), 131.15 (C1, PMP, C4', β -lactam, **4m**), 148.77 (C6, THPO, **3m**), 149.99 (C6, THPO, **4m**), 156.58 (C4, PMP, β -lactam, **3m**), 156.60 (C4, PMP, β -lactam, **4m**), 159.80 (2 \times C4, PMP, C4', β -lactam, **3m/4m**), 159.88 (2 \times C4, PMP, THPO, **3m/4m**), 161.79 (CO, β -lactam, **4m**), 162.07 (CO, β -lactam, **3m**), 186.96 (C4, THPO, **4m**), 190.86 (C4, THPO, **3m**).

(2R/2S)-1-[trans-(3'R,4'R)-1'-(4-Methoxyphenyl)-4'-(3,4-dimethoxyphenyl)-2'-oxoazetidin-3'-yl]-2-phenyl-1,2,3,4-tetrahydropyridin-4-one (**3n/4n**). – Obtained from (3R,4R)-**1n** (20.0 mg, 4.80×10^{-2} mmol), diene **2a** (10.3 mg, 5.28×10^{-2} mmol) and zinc(II) iodide (3.2 mg, 9.60×10^{-3} mmol) as a diastereomeric mixture **3n/4n**, 17.6 mg (76 %), $R_f = 0.24$ (eluens-B). RP-HPLC analysis of diastereomeric mixture showed ratio of **3n/4n** – 66:34 % (**3n**, 23.04 min and **4n**, 23.67 min). LC-MS for $C_{29}H_{28}N_2O_5$ ($M_r = 484.5547$): calcd. m/z $[M+H]^+$ 485.55, found 485.20 (**3n**, 9.32 min) and 485.20 (**4n**, 9.86 min). IR (KBr) ν_{max}/cm^{-1} : 3449, 2923, 1750, 1647, 1593, 1512, 1248, 1025, 831; 1H NMR ($CDCl_3$) δ/ppm : 2.66 (dd, 1H, $J_1 = 16.51$ Hz, $J_2 = 6.48$ Hz, C3, THPO, **3n**), 2.80 (dd, 1H, $J_1 = 16.39$ Hz, $J_2 = 8.43$ Hz, C3, THPO, **4n**), 2.88-2.98 (m, 1H, C3, THPO, **4n**), 3.06 (dd, 1H, $J_1 = 16.48$ Hz, $J_2 = 7.20$ Hz, C3, THPO, **3n**), 3.68 (s, 3H, OCH₃, **3n**), 3.73 (s, 3H, OCH₃, **4n**), 3.74 (s, 2 \times 3H, OCH₃, **3n/4n**), 3.84 (s, 3H, OCH₃, **3n**), 3.88 (s, 3H, OCH₃, **4n**), 4.33 (m, 2 \times 1H, C3', β -lactam, **3n/4n**), 4.64 (m, 2 \times 1H, C2, THPO and 2 \times 1H, C4', β -lactam; **3n/4n**), 5.23 (d, 2 \times 1H, $J = 7.89$ Hz, C5, THPO, **3n/4n**), 6.27 (d, 1H, $J = 1.80$ Hz, C5, 3,4-(CH₃O)₂-Ph, **3n**), 6.41 (dd, 2 \times 1H, $J_1 = 8.19$ Hz, $J_2 = 1.86$ Hz, C6, 3,4-(CH₃O)₂-Ph, **3n/4n**), 6.49 (d, 1H, $J = 1.68$ Hz, C5, 3,4-(CH₃O)₂-Ph, **4n**), 6.69 (d, 1H, $J = 8.25$ Hz, C6, THPO, **3n**), 6.76 (d, 2 \times 2H, $J = 9.03$ Hz, C3 and C5, PMP, **3n/4n**), 6.83 (d, 1H, $J = 8.22$ Hz, C6, THPO, **4n**), 7.12-7.33 (m, 2 \times 5H, Ph and 2 \times 2H, C2 and C6, PMP; **3n/4n**); ^{13}C NMR ($CDCl_3$) δ/ppm : 43.46 (C3, THPO, **4n**), 43.54 (C3, THPO, **3n**), 55.37 (2 \times OCH₃, **3n/4n**), 55.88 (2 \times OCH₃, **3n**), 55.94 (**4n**), 55.96 (**4n**), 61.43 (C2, THPO, **4n**), 62.72 (C4', β -lactam, **4n**), 64.06 (C2, THPO, **3n**), 64.93 (C4',

β -lactam, **3n**), 76.29 (C3', β -lactam, **4n**), 76.75 (C3', β -lactam, **3n**), 101.31 (C5, THPO, **3n**), 102.38 (C5, THPO, **4n**), 107.99 (C6, 3,4-(CH₃O)₂-Ph, **4n**), 108.20 (C6, 3,4-(CH₃O)₂-Ph, **3n**), 111.26 (C5, 3,4-(CH₃O)₂-Ph, **3n**), 111.47 (C5, 3,4-(CH₃O)₂-Ph, **4n**), 114.32 (2 × C3 and 2 × C5, PMP, **3n/4n**), 118.63 (C2, 3,4-(CH₃O)₂-Ph, **3n**), 118.82 (C2, 3,4-(CH₃O)₂-Ph, **4n**), 118.93 (C2 and C6, PMP, **4n**), 118.97 (C2 and C6, PMP, **3n**), 126.94 (C2 and C6, Ph, **4n**), 127.00 (C2 and C6, Ph, **3n**), 127.22 (C1, 3,4-(CH₃O)₂-Ph, **3n**), 127.58 (C1, 3,4-(CH₃O)₂-Ph, **4n**), 128.66 (2 × C4, Ph, **3n/4n**), 129.11 (2 × C3 and 2 × C5, Ph, **3n/4n**), 129.84 (2 × C1, PMP, **3n/4n**), 137.79 (C1, Ph, **4n**), 139.16 (C1, Ph, **3n**), 148.65 (C6, THPO, **3n**), 149.40 (C3 and C4, 3,4-(CH₃O)₂-Ph, **3n**), 149.78 (C3 and C4, 3,4-(CH₃O)₂-Ph, **4n**), 150.05 (C6, THPO, **4n**), 156.64 (2 × C4, PMP, **3n/4n**), 161.86 (CO, β -lactam, **4n**), 161.95 (CO, β -lactam, **3n**), 190.45 (C4, THPO, **3n**), 190.60 (C4, THPO, **4n**).

(2R/2S)-1-[trans-(3'R,4'R)-4'-(4-Fluorophenyl)-1'-(4-methoxyphenyl)-2'-oxoazetididin-3'-yl]-2-phenyl-1,2,3,4-tetrahydropyridin-4-one (**3o/4o**). – Obtained from (3R,4R)-**1o** (20.0 mg, 5.34 × 10⁻² mmol), diene **2b** (12.9 mg, 5.87 × 10⁻² mmol) and zinc(II) iodide (3.2 mg, 1.07 × 10⁻² mmol) as a diastereomeric mixture **3o/4o**, 21.0 mg (89 %), R_f = 0.22 (eluens-A). RP-HPLC analysis of diastereomeric mixture showed ratio of **3o/4o** – 72:28 % (**3o**, 24.42 min and **4o**, 24.77 min). LC-MS for C₂₇H₂₃FN₂O₃ (M_r = 442.4924): calcd. m/z [M+H]⁺ 443.49, found 443.20 (**3o**, 10.59 min) and 443.20 (**4o**, 10.95 min). IR (KBr) $\nu_{\max}/\text{cm}^{-1}$: 3448, 2927, 1751, 1589, 1513, 1300, 1249, 831; ¹H NMR (CDCl₃) δ/ppm : 2.66 (dd, 1H, J₁ = 16.50 Hz, J₂ = 6.06 Hz, C3, THPO, **3o**), 2.80 (dd, 1H, J₁ = 16.38 Hz, J₂ = 8.70 Hz, C3, THPO, **4o**), 2.93 (dd, 1H, J₁ = 16.44 Hz, J₂ = 6.24 Hz, C3, THPO, **4o**), 3.07 (dd, 1H, J₁ = 16.56 Hz, J₂ = 7.44 Hz, C3, THPO, **3o**), 3.73 (s, 3H, OCH₃, **3o**), 3.74 (s, 3H, OCH₃, **4o**), 4.26 (bs, 1H, C3', β -lactam, **3o**), 4.27 (bs, 1H, C3', β -lactam, **4o**), 4.59 (t, 1H, J_{1,2} = 6.66 Hz, C2, THPO, **3o**), 4.63 (t, 1H, J_{1,2} = 7.56 Hz, C2, THPO, **4o**), 4.66 (bs, 1H, C4', β -lactam, **3o**), 4.69 (bs, 1H, C4', β -lactam, **4o**), 5.24 (d, J = 7.86 Hz, 2 × 1H, C5, THPO, **3o/4o**), 6.68-6.72 (m, 2 × 1H, C6, THPO, **3o/4o**), 6.74-6.77 (m, 2 × 2H, C3 and C5, PMP, **3o/4o**), 6.87 (t, 2H, J_{1,2} = 8.34 Hz, C3 and C5, 4-F-Ph, **3o**), 7.03 (t, 2H, J_{1,2} = 8.34 Hz, C3 and C5, 4-F-Ph, **4o**), 7.08-7.14 (m, 2 × 2H, C2 and C6, 4-F-Ph, **3o/4o**), 7.16-7.32 (m, 2 × 5H, Ph and 2 × 2H, C2 and C6, PMP, **3o/4o**); ¹³C NMR (CDCl₃) δ/ppm : 43.45 (C3, THPO, **3o**), 43.65 (C3, THPO, **4o**), 55.40 (2 × OCH₃, **3o/4o**), 61.63 (C2, THPO, **4o**), 62.06 (C4', β -lactam, **4o**), 64.28 (C2, THPO, **3o**), 64.37 (C4', β -lactam, **3o**), 76.50 (C3', β -lactam, **4o**), 77.05 (C3', β -lactam, **3o**), 101.38 (C5, THPO, **3o**), 102.61 (C5, THPO, **4o**), 114.41 (C3 and C5, PMP, **3o**), 114.44 (C3 and C5, PMP, **4o**), 116.02 (d, J = 21.85 Hz, C3 and C5, *p*-F-Ph, **3o**), 116.46 (d, J = 21.99 Hz, C3 and C5, *p*-F-Ph, **4o**), 118.95 (C2 and C6, PMP, **4o**), 118.97 (C2 and C6, PMP, **3o**), 126.89 (C2 and C6, Ph, **4o**), 126.98 (C2 and C6, Ph, **3o**), 127.50 (d, J = 8.19 Hz, C2 and C6, *p*-F-Ph, **3o**), 127.57 (d, J = 10.40 Hz, C2 and C6, *p*-F-Ph, **4o**), 128.80 (C4, Ph, **4o**), 128.82 (C4, Ph, **3o**), 129.16 (C3 and C5, Ph, **4o**), 129.31 (C3 and C5, Ph, **3o**), 129.54 (2 × C1, PMP, **3o/4o**), 130.71 (d, J = 2.64 Hz, 2 ×

C1, *p*-F-Ph, **3o/4o**), 137.73 (C1, Ph, **4o**), 139.60 (C1, Ph, **3o**), 148.47 (C6, THPO, **3o**), 149.89 (C6, THPO, **4o**), 156.77 (2 × C4, PMP, **3o/4o**), 161.58 (CO, β -lactam, **3o**), 161.90 (CO, β -lactam, **4o**), 163.54 (2 × C4, *p*-F-Ph, **3o/4o**), 190.40 (C4, THPO, **3o**), 190.58 (C4, THPO, **4o**).

(2R/2S)-1-[trans-(3'R,4'R)-4'-(4-Trifluoromethylphenyl)-1'-(4-methoxyphenyl)-2'-oxoazetididin-3'-yl]-2-phenyl-1,2,3,4-tetrahydropyridin-4-one (**3p/4p**). – Obtained from (3R,4R)-**1p** (20.0 mg, 4.71 × 10⁻² mmol), diene **2b** (12.9 mg, 5.18 × 10⁻² mmol) and zinc(II) iodide (3.2 mg, 9.42 × 10⁻³ mmol) as a diastereomeric mixture **3p/4p**, 13.7 mg (59 %), R_f = 0.38 (eluens-A). RP-HPLC analysis of diastereomeric mixture showed ratio of **3p/4p** – 74:26 % (**3p**, 25.16 min and **4p**, 25.48 min). LC-MS for C₂₈H₂₃F₃N₂O₃ (M_r = 492.5003): calcd. m/z [M+H]⁺ 493.50, found 493.20 (**3p**, 11.34 min) and 493.20 (**4p**, 11.68 min). IR (KBr) $\nu_{\max}/\text{cm}^{-1}$: 3449, 1752, 1654, 1648, 1594, 1512, 1249, 830, 699; ¹H NMR (CDCl₃) δ/ppm : 2.67 (dd, 1H, J₁ = 16.48 Hz, J₂ = 6.18 Hz, C3, THPO, **3p**), 2.80 (m, 1H, C3, THPO, **4p**), 2.93 (dd, 1H, J₁ = 16.39 Hz, J₂ = 6.33 Hz, C3, THPO, **4p**), 3.08 (dd, 1H, J₁ = 16.51 Hz, J₂ = 7.44 Hz, C3, THPO, **3p**), 3.73 (s, 3H, OCH₃, **3p**), 3.74 (s, 3H, OCH₃, **4p**), 4.27 (d, 1H, J = 2.04 Hz, C3', β -lactam, **3p**), 4.28 (d, 1H, J = 2.40 Hz, C3', β -lactam, **3p**), 4.58 (t, 1H, J_{1,2} = 6.75 Hz, C2, THPO, **3p**), 4.64 (t, 1H, J_{1,2} = 8.60 Hz, C2, THPO, **4p**), 4.72 (d, 1H, J = 1.83 Hz, C4', β -lactam, **3p**), 4.74 (d, 1H, J = 1.95 Hz, C4', β -lactam, **4p**), 5.26 (d, 1H, J = 7.86 Hz, C5, THPO, **4p**), 5.27 (d, 1H, J = 7.98 Hz, C5, THPO, **3p**), 6.73-6.82 (m, 2 × 2H, C3 and C5, PMP and 2 × 1H, C6, THPO, **3p/4p**), 7.10-7.30 (m, 2 × 2H, C2 and C6, PMP, 2 × 2H, C2 and C6, 4-CF₃-Ph and 2 × 5H, Ph, **3p/4p**), 7.42 (d, 2H, J = 8.10 Hz, C3 and C5, 4-CF₃-Ph, **3p**), 7.60 (d, 2H, J = 8.04 Hz, C3 and C5, 4-CF₃-Ph, **4p**); ¹³C NMR (CDCl₃) δ/ppm : 43.38 (C3, THPO, **3p**), 43.69 (C3, THPO, **4p**), 55.42 (2 × OCH₃, **3p/4p**), 55.78 (d, J = 358.59 Hz, 2 × CF₃, **3p/4p**), 61.70 (C2, THPO, **4p**), 62.02 (C4', β -lactam, **4p**), 64.37 (C2, THPO, **3p**), 64.43 (C4', β -lactam, **3p**), 76.64 (C3', β -lactam, **4p**), 77.14 (C3', β -lactam, **3p**), 101.65 (C5, THPO, **3p**), 102.94 (C5, THPO, **4p**), 114.52 (C3 and C5, PMP, **3p**), 114.55 (C3 and C5, PMP, **4p**), 118.89 (C2 and C6, PMP, **4p**), 118.92 (C2 and C6, PMP, **3p**), 126.41 (d, J = 3.80 Hz, C3 and C5, 4-CF₃-Ph, **3p**), 126.08 (C2 and C6, Ph, **3p**), 126.16 (C2 and C6, Ph, **4p**), 126.41 (d, J = 4.09 Hz, C3 and C5, 4-CF₃-Ph, **4p**), 126.87 (C2 and C6, 4-CF₃-Ph, **4p**), 127.00 (C2 and C6, 4-CF₃-Ph, **3p**), 128.92 (C4, Ph, **4p**), 128.96 (C4, Ph, **3p**), 129.21 (C3 and C5, Ph, **4p**), 129.32 (C1, PMP, **4p**), 129.35 (C1, PMP, **3p**), 129.40 (C3 and C5, Ph, **3p**), 137.62 (C1, Ph, **4p**), 139.04 (2 × C1, 4-CF₃-Ph, **3p/4p**), 139.61 (C1, Ph, **3p**), 143.38 (C4, 4-CF₃-Ph, **3p**), 143.40 (C4, 4-CF₃-Ph, **4p**), 148.26 (C6, THPO, **3p**), 149.77 (C6, THPO, **4p**), 156.91 (2 × C4, PMP, **3p/4p**), 161.33 (2 × CO, β -lactam, **3p/4p**), 190.37 (2 × C4, THPO, **3p/4p**).

(2R/2S)-1-[trans-(3'R,4'R)-4'-Ferrocenyl-1'-(4-methoxyphenyl)-2'-oxoazetididin-3'-yl]-2-phenyl-1,2,3,4-tetrahydropyridin-4-one ((2R,3'R,4'R)-**3q**/(2S,3'R,4'R)-**4q**). – Obtained from (3R,4R)-**1q** (30.0 mg, 6.46 × 10⁻² mmol), diene **2a** (13.8 mg, 7.11 × 10⁻² mmol) and zinc(II) iodide

(3.8 mg, 1.29×10^{-2} mmol) as a diastereomeric mixture **3q/4q**, 29.0 mg (84 %), $R_f = 0.17$ (eluens-A). RP-HPLC analysis of diastereomeric mixture showed ratio of **3q/4q** – 80:20 % (**3q**, 25.07 min and **4q**, 25.46 min). LC-MS for $C_{31}H_{28}FeN_2O_3$ ($M_r = 532.4251$): calcd. m/z $[M+H]^+$ 533.43, found 533.00 (**3q**, 11.35 min) and 533.00 (**4q**, 11.78 min). IR (KBr) ν_{max}/cm^{-1} : 3394, 1736, 1702, 1686, 1654, 1648, 1637, 1618, 1560, 1458, 699; 1H NMR ($CDCl_3$) δ/ppm : 2.68–2.88 (m, $2 \times 1H$, C3, THPO, **3q/4q**), 2.95–3.12 (m, $2 \times 1H$, C3, THPO, **3q/4q**), 3.79 (s, $2 \times 3H$, OCH_3 , **3q/4q**), 3.96 (s, 5H, Fc, **3q**), 4.01 (s, 5H, Fc, **4q**), 4.06–4.10 (m, $2 \times 3H$, Fc, **3q/4q**), 4.14 (m, $2 \times 1H$, Fc, **3q/4q**), 4.53 (d, 1H, $J = 2.07$ Hz, C4', β -lactam, **3q**), 4.56 (d, $J = 2.01$ Hz, 1H, C4', β -lactam, **4q**), 4.71 (d, 1H, $J = 2.13$ Hz, C3', β -lactam, **4q**), 4.81 (m, 1H, C2, THPO, **4q**), 4.86 (d, $J = 2.01$ Hz, 1H, C3', β -lactam, **3q**), 4.96 (m, 1H, C2, THPO, **3q**), 5.22–5.29 (m, $2 \times 1H$, C5, THPO, **3q/4q**), 6.84–6.90 (m, $2 \times 2H$, C3 and C5, PMP and $2 \times 1H$, C6, THPO, **3q/4q**), 7.20–7.52 (m, $2 \times 2H$, C2 and C6, PMP and $2 \times 5H$, Ph, **3q/4q**); ^{13}C NMR ($CDCl_3$) δ/ppm : 43.67 (C3, THPO, **4q**), 44.80 (C3, THPO, **3q**), 55.42 ($2 \times OCH_3$, **3q/4q**), 58.81 (C2, THPO, **3q**), 59.01 (C2, THPO, **4q**), 61.15 (C4', β -lactam, **4q**), 63.04 (C4', β -lactam, **3q**), 65.86 (**4q**), 65.98 (**3q**), 68.02 (**3q**), 68.05 (**3q**), 68.21 (**4q**), 68.47 (**4q**), 68.54 (5C, **4q**), 68.59 (5C, **3q**), 68.74 (**3q**) and 68.95 (**4q**) (Fc), 74.56 (C3', β -lactam, **3q**), 75.49 (C3', β -lactam, **4q**), 84.21 ($2 \times C1$, Fc, **3q/4q**), 101.59 (C5, THPO, **4q**), 101.92 (C5, THPO, **3q**), 114.31 ($2 \times C3$ and $2 \times C5$, PMP, **3q/4q**), 120.39 ($2 \times C2$ and $2 \times C6$, PMP, **3q/4q**), 126.71 (C2 and C6, Ph, **4q**), 127.40 (C2 and C6, Ph, **3q**), 128.71 (C4, Ph, **4q**), 128.91 (C4, Ph, **3q**), 129.26 (C3 and C5, Ph, **3q**), 129.36 (C3 and C5, Ph, **4q**), 129.39 ($2 \times C1$, PMP, **3q/4q**), 138.30 ($2 \times C1$, Ph, **3q/4q**), 149.53 (C6, THPO, **3q**), 150.55 (C6, THPO, **4q**), 157.05 ($2 \times C4$, PMP, **3q/4q**), 162.59 ($2 \times CO$, β -lactam, **3q/4q**), 190.64 ($2 \times C4$, THPO, **3q/4q**).

(2S/2R)-*1*-[trans-(3'S,4'S)-4'-Ferrocenyl-1'-(4-methoxyphenyl)-2'-oxoazetidin-3'-yl]-2-phenyl-1,2,3,4-tetrahydropyridin-4-one ((2S,3'S,4'S)-**3q**/(2R,3'S,4'S)-**4q**). – Obtained from (3S,4S)-**1q** (20.0 mg, 4.31×10^{-2} mmol), diene **2a** (8.6 mg, 4.74×10^{-2} mmol) and zinc(II) iodide (2.6 mg, 8.61×10^{-3} mmol) as a diastereomeric mixture **3q/4q**, 18.1 mg (79 %), $R_f = 0.17$ (eluens-A). RP-HPLC analysis of diastereomeric mixture showed ratio of **3q/4q** – 80:20 % (**3q**, 25.07 min and **4q**, 25.46 min). LC-MS for $C_{31}H_{28}FeN_2O_3$ ($M_r = 532.4251$): calcd. m/z $[M+H]^+$ 533.43, found 533.00 (**3q**, 11.37 min) and 533.00 (**4q**, 11.79 min). Diastereomeric mixture of **3q/4q** was separated by a preparative thin layer chromatography using eluens-A.

(2S)-*1*-[trans-(3'S,4'S)-4'-Ferrocenyl-1'-(4-methoxyphenyl)-2'-oxoazetidin-3'-yl]-2-phenyl-1,2,3,4-tetrahydropyridin-4-one ((2S,3'S,4'S)-**3q**). – 14.48 mg, m.p. 183–184 °C; $[\alpha]_D = +56.8$ ($c = 0.35$ g/100 mL dichloromethane); IR (KBr) ν_{max}/cm^{-1} : 3448, 1752, 1654, 1593, 1511, 1458, 1249, 1027, 799; 1H NMR ($CDCl_3$) δ/ppm : 2.81 (dd, 1H, $J_1 = 16.51$ Hz, $J_2 = 7.20$ Hz, C3, THPO), 3.06 (dd, 1H, $J_1 = 16.51$ Hz, $J_2 = 6.60$ Hz, C3, THPO), 3.70 (m, 1H, Fc), 3.76 (m, 1H, Fc), 3.79 (s, 3H, OCH_3), 3.96 (s, 5H, Fc), 4.05–4.10 (m, 2H, Fc),

4.53 (bs, 1H, C4', β -lactam), 4.86 (bs, 1H, C3', β -lactam), 4.81 (t, 1H, $J_{1,2} = 7.20$ Hz, C2, THPO), 5.28 (d, 1H, $J = 9.60$ Hz, C5, THPO), 6.87 (d, 2H, $J = 8.10$ Hz, C3 and C5, PMP), 7.24 (d, 1H, $J = 8.82$ Hz, C6, THPO), 7.30 (d, 2H, $J = 8.40$ Hz, C2 and C6, PMP), 7.36–7.52 (m, 5H, Ph); ^{13}C NMR ($CDCl_3$) δ/ppm : 43.77 (C3, THPO), 55.49 (OCH_3), 58.85 (C2, THPO), 66.06 (C4', β -lactam), 68.07, 68.10, 68.61, 68.65 and 68.79 (Fc), 74.64 (C3', β -lactam), 84.35 (C1, Fc), 102.08 (C5, THPO), 114.41 (C3 and C5, PMP), 120.46 (C2 and C6, PMP), 127.50 (C2 and C6, Ph), 128.96 (C4, Ph), 129.31 (C3 and C5, Ph), 129.53 (C1, PMP), 138.43 (C1, Ph), 149.45 (C6, THPO), 157.17 (C4, PMP), 162.66 (CO, β -lactam), 190.62 (C4, THPO).

(2R)-*1*-[trans-(3'S,4'S)-4'-Ferrocenyl-1'-(4-methoxyphenyl)-2'-oxoazetidin-3'-yl]-2-phenyl-1,2,3,4-tetrahydropyridin-4-one ((2R,3'S,4'S)-**4q**). – 3.62 mg; IR (KBr) ν_{max}/cm^{-1} : 3448, 1752, 1654, 1593, 1511, 1458, 1249, 1027, 799; 1H NMR ($CDCl_3$) δ/ppm : 2.75 (dd, 1H, $J_1 = 16.51$ Hz, $J_2 = 6.60$ Hz, C3, THPO), 3.08 (dd, 1H, $J_1 = 16.51$ Hz, $J_2 = 6.90$ Hz, C3, THPO), 3.79 (s, 3H, OCH_3), 3.90 (m, 1H, Fc), 3.96 (m, 1H, Fc), 4.01 (s, 5H, Fc), 4.14 (m, 2H, Fc), 4.56 (d, 1H, $J = 2.10$ Hz, C4', β -lactam), 4.71 (d, 1H, $J = 2.10$ Hz, C3', β -lactam), 4.81 (t, 1H, $J_{1,2} = 6.90$ Hz, C2, THPO), 5.25 (d, 1H, $J = 9.60$ Hz, C5, THPO), 6.86 (d, 2H, $J = 9.00$ Hz, C3 and C5, PMP), 7.22–7.32 (m, 1H, C6, THPO and 2H, C2 and C6, PMP), 7.36–7.50 (m, 5H, Ph); ^{13}C NMR ($CDCl_3$) δ/ppm : 44.99 (C3, THPO), 59.12 (OCH_3), 61.28 (C2, THPO), 65.93 (C4', β -lactam), 68.25, 68.48, 68.65 and 68.98 (Fc), 75.64 (C3', β -lactam), 84.35 (C1, Fc), 101.72 (C5, THPO), 114.41 (C3 and C5, PMP), 120.50 (C2 and C6, PMP), 126.80 (C2 and C6, Ph), 128.75 (C4, Ph), 129.41 (C3 and C5, Ph), 129.53 (C1, PMP), 138.43 (C1, Ph), 150.48 (C6, THPO), 157.17 (C4, PMP), 162.66 (CO, β -lactam), 190.62 (C4, THPO).

(2R/2S)-*1*-[trans-(3'R,4'R)-2'-Oxo-4'-phenylazetidin-3'-yl]-2-phenyl-1,2,3,4-tetrahydropyridin-4-one (**3s/4s**). – Obtained from (3R,4R)-**1s** (15.0 mg, 5.99×10^{-2} mmol), diene **2b** (15.0 mg, 6.59×10^{-2} mmol) and zinc(II) iodide (3.8 mg, 1.20×10^{-2} mmol) as a diastereomeric mixture **3s/4s**, 9.52 mg (50 %), $R_f = 0.20$ (eluens-B). RP-HPLC analysis of diastereomeric mixture showed ratio of **3s/4s** – 66:34 % (**3s**, 23.80 min and **4s**, 24.10 min). LC-MS for $C_{20}H_{18}N_2O_2$ ($M_r = 318.3772$): calcd. m/z $[M+H]^+$ 319.38, found 319.20 (**3s**, 9.25 min) and 319.20 (**4s**, 9.46 min). IR (KBr) ν_{max}/cm^{-1} : 3087, 2918, 1774, 1622, 1566, 1229, 1199, 699; 1H NMR ($CDCl_3$) δ/ppm : 2.65 (dd, 1H, $J_1 = 16.51$ Hz, $J_2 = 6.90$ Hz, C3, THPO, **3s**), 2.76 (dd, 1H, $J_1 = 16.51$ Hz, $J_2 = 8.10$ Hz, C3, THPO, **4s**), 2.95 (m, 1H, C3, THPO, **4s**), 3.00 (dd, 1H, $J_1 = 16.51$ Hz, $J_2 = 6.90$ Hz, C3, THPO, **3s**), 4.21 (m, $2 \times 1H$, C4', β -lactam, **3s/4s**), 4.44 (d, 1H, $J = 1.80$ Hz, C3', β -lactam, **3s**), 4.49 (d, 1H, $J = 1.80$ Hz, C3', β -lactam, **4s**), 4.59 (t, 1H, $J_{1,2} = 6.90$ Hz, C2, THPO, **3s**), 4.62 (m, 1H, C2, THPO, **4s**), 5.24 (m, $2 \times 1H$, C5, THPO, **3s/4s**), 6.42 (bs, $2 \times 1H$, NH, **3s/4s**), 6.81 (d, $2 \times 1H$, $J = 6.60$ Hz, C6, THPO, **3s/4s**), 7.10–7.35 (m, $2 \times 10H$, $2 \times Ph$, **3s/4s**); ^{13}C NMR ($CDCl_3$) δ/ppm : 43.54 (C3, THPO, **3s**), 43.63 (C3, THPO, **4s**), 59.17 (C2, THPO, **4s**), 61.28 (C2, THPO, **3s**), 61.38 (C4', β -lactam, **4s**), 64.07 (C4', β -lactam, **3s**),

77.65 (C3', β -lactam, **4s**), 77.86 (C3', β -lactam, **3s**), 101.37 (C5, THPO, **3s**), 102.38 (C5, THPO, **4s**), 125.42 (C2 and C6, Ph, β -lactam, **4s**), 125.44 (C2 and C6, Ph, β -lactam, **3s**), 126.78 (C2 and C6, Ph, THPO, **4s**), 126.96 (C2 and C6, Ph, THPO, **3s**), 128.65 (C4, Ph, β -lactam, **4s**), 128.71 (C4, Ph, THPO, **3s** and C3 and C5, Ph, β -lactam, **4s**), 128.76 (C3 and C5, Ph, β -lactam, **3s**), 128.96 (C4, Ph, THPO, **4s**), 129.13 (C3 and C5, Ph, THPO, **4s** and C4, Ph, THPO, **3s**), 129.19 (C3 and C5, Ph, THPO, **3s**), 136.76 (C1, Ph, β -lactam, **3s**), 137.17 (C1, Ph, β -lactam, **4s**), 137.68 (C1, Ph, THPO, **4s**), 139.16 (C1, Ph, THPO, **3s**), 148.78 (C6, THPO, **3s**), 150.08 (C6, THPO, **4s**), 165.51 (CO, β -lactam, **4s**), 165.77 (CO, β -lactam, **3s**), 190.56 (C4, THPO, **3s**), 190.59 (C4, THPO, **4s**).

CONCLUSIONS

The study demonstrates the asymmetric approach in the synthesis of pyridin-4-one ring attached to the β -lactam ring *via* C-N bond, using the chirality and functionalisation of the homochiral amino- β -lactam nucleus as the building block and stereocontrolling element.

The study was focused on the aza-Diels – Alder reaction of azetidin-2-one-tethered imines with siloxydienes performed in the presence of Lewis acid. First the effect of the amount of various catalysts, equimolar ($x = 100\%$) and a catalytic ($x = 20\%$), on the conversion rate and as well as on the product ratio was studied. The cycloaddition at low temperature ($-20\text{ }^\circ\text{C}$) in acetonitrile under zinc(II) iodide ($x = 20\%$) catalysis provided the best chemical yield (92%) combined with the highest diastereoselectivity (68:32). Furthermore, the influence of various groups on azetidin-2-one-tethered imines ($R^1 = \text{H, PMP}$; $R^2 = \text{aryl, ferrocenyl}$; $R^3 = \text{alkyl, aryl, ferrocenyl}$) and on diene (TMS, TBDMS, (–)- and (+)-menthyl, (–)-*trans*-2-phenyl-1-cyclohexyl) have been applied in cycloaddition reaction leading to the formation of two stereoisomers of 2-aryl(alkyl)-2,3-dihydro-4-pyridones with diastereomeric ratio varying from 86:14 to 55:45.

The crystal structure of compound (2*S*,3'*S*,4'*S*)-**3q** was determined in order to establish unambiguously both absolute and relative configuration at the stereogenic center C21 of the pyridin-4-one ring and the absolute configuration of C21 found to be *S*.

Semiempirical PM3 calculation method was used to model Diels – Alder reaction of dienes with azetidin-2-one-tethered imines catalyzed by Lewis acids. FMO analysis has revealed that the preferred regioselectivity is governed by orbital symmetry and the size of coefficients. It was assumed that for Lewis acid (zinc(II) iodide), azetidin-2-one-tethered imine acts as a bidentate ligand, coordinating with metal *via* imine nitrogen and carbonyl oxygen. PM3 modelling has shown a slight preference to diene approach from the top side yielding *R*-isomer, while diene approach from the bottom side leading to *S*-isomer is less preferred. These computatio-

nal predictions are in good agreement with experimental results, where in the most of Lewis acid catalyzed reactions, *R*:*S* ratio $\approx 70:30$ is found.

Acknowledgements. – The financial support from the Croatian Ministry of Science, Education and Sports (Programs 098-0982915-2948, 098-1191344-2943 and 0098147) and GlaxoSmithKline Research Centre Zagreb is gratefully acknowledged.

Supplementary Materials. – Crystallographic data for the structure of compound (2*S*,3'*S*,4'*S*)-**3q** reported in this paper have been deposited with the Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK (fax: +44 1223 336033; e-mail: deposit@ccdc.cam.ac.uk) and can be obtained on request, free of charge, by quoting the publication citation and the deposition numbers 652331.

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SAŽETAK

Studija asimetrične sinteze 2-aril(alkil)-2,3-dihidro-4-piridona aza-Diels – Alderovom reakcijom imina, izvedenih iz aminoazetidina-2-ona, i siloksidena kataliziranom Lewisovim kiselinama

Tanja Poljak, Krešimir Molčanov, Davor Margetić i Ivan Habuš

Ispitani su *trans*-3-amino- β -laktami kao kiralne građevne jedinice u asimetričnoj sintezi 2-aril(alkil)-2,3-dihidro-4-piridona aza-Diels – Alderovom reakcijom imina, izvedenih iz aminoazetidina-2-ona, i siloksidena kataliziranom Lewisovim kiselinama.

Crystallographic Data for the Compound (2*S*,3'*S*,4'*S*)-**3q**

SUPPLEMENT

to article: Study on the Lewis Acid-promoted Aza-Diels – Alder Reaction of Azetidin-2-one-tethered Imines with Siloxydienes in the Asymmetric Synthesis of 2-Aryl(alkyl)-2,3-dihydro-4-pyridones

by Tanja Poljak, Krešimir Molčanov, Davor Margetić, and Ivan Habuš

Crystals of compound (2*S*,3'*S*,4'*S*)-**3q** were prepared by liquid-liquid diffusion using dichloromethane solution and hexane (1 mL : 5 mL). Crystal size and quality pro-

hibited use of MoK α radiation, so CuK α (1.54179 Å) had to be used for single crystal measurement, despite high absorption coefficient of iron atoms.

Table I. Crystallographic data and structure refinement data for compound (2*S*,3'*S*,4'*S*)-**3q**

Empirical formula	C ₃₁ H ₂₇ O ₃ N ₂ Fe
Formula wt. / g mol ⁻¹	531.41
Crystal dimensions / mm	0.29 × 0.07 × 0.05
Space group	<i>P</i> 2 ₁
<i>a</i> / Å	11.4773(6)
<i>b</i> / Å	7.4679(7)
<i>c</i> / Å	15.6163(14)
α / °	90
β / °	105.925(6)
γ / °	90
<i>Z</i>	2
<i>V</i> / Å ³	1287.12(18)
<i>D</i> _{calc} / g cm ⁻³	1.371
μ / mm ⁻¹	4.982
Θ range / °	2.94 – 76.25
Range of <i>h</i> , <i>k</i> , <i>l</i>	-14 > <i>h</i> > 0; -9 > <i>k</i> > 0; -18 > <i>l</i> > 19
Reflections collected	3056
Independent reflections	2913
Observed reflections (<i>I</i> ≥ 2 σ)	1272
<i>R</i> _{int}	0.078
<i>R</i> (<i>F</i>)	0.079
<i>R</i> _w (<i>F</i> ²)	0.1972
Goodness of fit	1.002
$\Delta\rho_{\max}$, $\Delta\rho_{\min}$ (eÅ ⁻³)	0.396; -0.458

Table II. Selected torsion angles (in degrees) defining conformation of (2*S*,3'*S*,4'*S*)-**3q**

N2 – C12 – C13 – O1	57(2)
N2 – C12 – C13 – N1	-126.8(11)
N2 – C12 – C11 – C1	118.4(12)
N2 – C12 – C11 – N1	-124.3(11)
C12 – C13 – N1 – C11	3.0(10)
C12 – C13 – N1 – C14	-170.4(13)
O1 – C13 – N1 – C14	7(2)
O1 – C13 – N1 – C11	-179.8(14)
O1 – C13 – C12 – C11	-179.5(18)
N1 – C11 – C12 – C13	2.7(9)
C11 – C12 – C13 – N1	-2.8(9)
C14 – N1 – C11 – C1	-54.0(17)
C14 – N1 – C11 – C12	-170.5(12)
C1 – C11 – N1 – C13	-119.4(11)
C1 – C11 – C12 – C13	120.0(11)
C12 – C11 – N1 – C13	-2.9(10)
C19 – C14 – N1 – C13	10(2)
C19 – C14 – N1 – C11	-161.1(12)
C15 – C14 – N1 – C13	-165.9(13)
C15 – C14 – N1 – C11	23(2)
C21 – N2 – C12 – C13	-52.1(15)
C21 – N2 – C12 – C11	51.5(16)
C25 – N2 – C12 – C13	-114.7(12)
C25 – N2 – C12 – C11	-141.7(11)

Table III. Bond lengths (in Å) and bond angles (in degrees) in the azetidín-2-one ring

N2 – C12	1.433(13)
C11 – C12	1.563(15)
C1 – C11	1.526(15)
C11 – N1	1.477(14)
N1 – C14	1.416(14)
N1 – C13	1.391(14)
C13 – C12	1.547(16)
C13 – O1	1.205(14)
N2 – C12 – C11	121.7(10)
C12 – C11 – C1	114.6(10)
C12 – C11 – N1	88.1(8)
C1 – C11 – N1	115.6(10)
C11 – N1 – C14	132.2(10)
C11 – N1 – C13	94.6(9)
C14 – N1 – C13	132.8(11)
N1 – C13 – O1	129.1(12)
N1 – C13 – C12	91.9(9)
O1 – C13 – C12	138.9(12)
C13 – C12 – N2	119.2(9)
C13 – C12 – C11	85.3(9)

Data collection was performed on an Enraf Nonius CAD4 diffractometer at room temperature [293(2) K]. The WinGX standard procedure was applied for data reduction.¹ Three standard reflections were measured every 120 minutes as intensity control. Absorption correction based on eight Ψ -scan reflexions was performed.² The structure was solved with SHELXS97³ and refined with SHELXL97.⁴ The models were refined using the full matrix least squares refinement. Hydrogen atoms were refined as riding entities. The atomic scattering factors were those included in SHELXL97.⁴ Molecular geometry calculations were performed with PLATON,⁵ and molecular graphics were prepared using ORTEP-3⁶ and CCDC-Mercury.⁷

Absolute configurations of C11 and C12 atoms were known from a previously prepared compound,⁸ and the absolute configuration of the C21 atom was determined in relation to them.

Table IV. Geometric parameters of the hydrogen bonds

	$D-H\cdots A / \text{Å}$	$D-H / \text{Å}$	$H\cdots A / \text{Å}$	$D-H\cdots A / ^\circ$	Symm. operation on A
C22-H22B \cdots O1	3.391(15)	0.97	2.43	171	$x, -1+y, z$
C25-H25 \cdots O3	3.363(16)	0.93	2.45	167	$-x, \frac{1}{2}+y, 2-z$
C30-H30 \cdots O3	3.274(18)	0.93	2.57	132	$1-x, \frac{1}{2}+y, 2-z$

Table V. Geometric parameters of the C-H $\cdots\pi$ interactions

	$H\cdots C_g / \text{Å}$	$\gamma / ^\circ$	$C-H\cdots C_g / ^\circ$	$C\cdots C_g / \text{Å}$	Symm. op. on C_g
C20-H20c \cdots Cg(C14 \rightarrow C19)	2.79	12.85	152	3.670(17)	$1 - x, \frac{1}{2} + y, 2 - z$
C20-H20B \cdots Cg(C26 \rightarrow C31)	3.25	25.13	146	4.078(14)	$1 - x, \frac{1}{2} + y, 2 - z$

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