Stereochemical Aspects of a Two-Step Staudinger Reaction − Asymmetric Synthesis of Chiral Azetidine-2-ones

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Reaction of *N*-trialkylsilylimines with a variety of glycine-derived ketenes produced 1,3-azadienes, which in some cases have been isolated and characterised. A conrotatory ring closure of these compounds gave rise to the formation of the azetidine-2-ones, thus allowing a formal two-step Staudinger reaction. Exclusive *trans* diastereoselectivity was observed. A less stringent diastereofacial selectivity was obtained. A set of experiments has been performed in order to evaluate the influence of the structural parameters as well as reaction conditions. Ab initio studies at MP2/6–31G* and QCISD(T)/6–311G** levels on model compounds provide a rationalization of the experimental results obtained. From the experimental as well as theoretical data it is clear that the presence of the silyl enol group in the intermediate azadiene is crucial in its stabilisation and plays a fundamental role in the conrotatory ring closure and, therefore, in the formation of the azetidine-2-one ring.

Introduction

An imperative factor in the synthesis of biologically active congeners of natural products is the control of the absolute stereochemistry of the relevant chiral centers. Initially, the exclusive synthesis of *cis*-β-lactams was important for penicillins and cephalosporins,[1] but with the discovery of thienamycin[2] synthetic routes to *trans*-β-lactams have demanded attention. Along with this growth in synthetic activity is the increasing need to control both the relative and absolute configuration.

A traditional pathway to β-lactams is that of the [2+2] cycloaddition of an imine and a ketene (the Staudinger reaction).[3] Although this reaction has been known for over 80 years, its mechanism and the rationale for the stereochemistry are far from clear.[4] The stereochemical outcome of the reaction depends on the structure of the imine, on the ketene precursor, on the sequence of reagent addition, on the solvent, and on the nature of the base used to produce the ketene from an acyl chloride. The requirement that the ketene be generated in situ raises the possibility that species such as the acyl chloride, the tertiary amine, the amine hydrochloride salt, *N*-acylammonium, or *N*-acyliminium may play a role in the reaction. A recent low-temperature FTIR study of the reaction of acyl chlorides with imines in the presence of base to form β-lactams was interpreted as showing that β-lactam formation occurred exclusively through a ketene intermediate.[5] Two chiral centers may be generated in this process, and both the relative and absolute stereochemistries of these two centers are often of critical concern to the use of this reaction in the synthesis of biologically active β-lactams.

In three earlier papers[6] we reported a rather general route to *trans*-β-lactams through a formal two-step Staudinger reaction. By using this protocol we have been able to isolate and identify the neutral azadiene intermediate that resembles the zwitterionic intermediate invoked in the classical Staudinger reaction. With this information in hand, we started more systematic studies of the stereochemical aspects of this novel two-step process. We report the influence of the ketene chiral auxiliary, the alkoxy protecting group of the imine, the steric demand of the parent aldehyde, and the role of solvent on the stereochemical outcome of the reaction. Moreover, computational studies on this subject are also reported. Our final aim was to find a rationale for the stereochemical course of our reaction and to apply the resulting methodology to highly enantio- and diastereoselective syntheses of 3-amido-substituted azetidinones.

Computational Methods

All ab initio calculations were carried out at the MP2/6-31G* level using the GAUSSIAN 94 series of programs.[7] Geometries were fully optimized by standard gradient techniques and the fully optimized structures were checked by frequency analysis. Each transition state showed only one imaginary frequency, the corresponding vibration being associated with the nuclear motion along the reaction coordinate. Zero-point vibration energies (ZPVEs) were computed at the MP2/6−31G* level, scaled by the factor

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  1434−193X/00/0713−2379 $ 17.50+.50/0

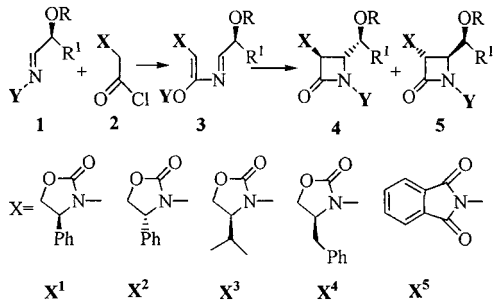
0.9670[8] and used for compounds in Scheme 2. Wiberg bond orders were calculated with the natural bond orbital (NBO) method[9] as implemented in GAUSSIAN 94. With the aim of having full control of the energy profile of the reaction, single-point calculations on MP2/6-31G*-optimized geometries were performed using quadratic configuration interactions [QCISD(T)/6−311G**].

The solvent effect was computed using the isodensity polarization model (IPCM) as implemented by Wiberg.[10] In the present computational study we considered two solvents that were used in our experimental studies: a medium with a dielectric permittivity ε corresponding to diethyl ether (ε = 4.33) and a medium corresponding to a polar solvent like acetonitrile (ε = 36.64). The solvent effect was computed keeping the geometries of the various critical points frozen at the values optimized in the absence of solvent effects. Cartesian coordinates of the optimized structures and the relative energies for all the calculations are reported as Supporting Information.

Results

We prepared and employed ketenes with an oxazolidinone substituent, an auxiliary that has been used extensively for the stereoselective formation of C−C bonds.[11] Moreover, we investigated the effect of the trialkylsilyl-substituted imine nitrogen atom[12] on the stereoselectivity of the Staudinger reaction by preparing several imines with various SiR_3 groups and carried out the same [2+2] addition as above. Scheme 1 and Table 1 report the results obtained. In Table 1, the results are collected in sets depending on the structural parameters that were changed. Entry 1 provides the basis for comparison.

The first set of experiments (Entries 2−5) was concerned with evaluation of the influence of the chiral auxiliary of the ketene on the diastereoselectivity of the reaction. This study originated with the preparation of some chiral (oxazolidinyl)acetic acids, which were converted into their acyl chloride derivatives and then employed in cycloaddition reactions with the *N*-trimethylsilylimine derived from (triisopropylsilyloxy)lactaldehyde. For the second set of experiments (Entries 6−9), we evaluated the influence of the steric demand of the protecting group of the alkoxy function on the imine. The third set (Entries 10−12) of experiments was concerned with the change of the substituent on the silyl atom directly linked to the imine nitrogen atom. The fourth group (Entries 13−16) relates to the experiments in which the side chain of the imine has been changed. Finally, the last set (Entries 17−25) examines the influence of the solvent on the stereochemical course of the final cyclization step.



Y= TMS, TBDMS, TBDPS, TIPS

R= TIPS, TBDMS, TBDPS, TES, *t*-But

R^1= Me, Et, Ph, *i*-Pr, *t*-But

Scheme 1

Discussion

Treatment of the *N*-trimethylsilylimine **1a** (R = TIPS, $R^1 = CH_3$, Y = TMS), obtained from the parent lactaldehyde according to a reported protocol,[12,13] with 1 equiv. of TMSCl followed by the addition of 2 equiv. of TEA and 1.2 equiv. of acyl chloride **2a** (X = X^1) results in the formation of azadiene **3a** (Scheme 1).[14] Heating the azadiene **3a** leads to *trans*-β-lactams **4a** and **5a**.

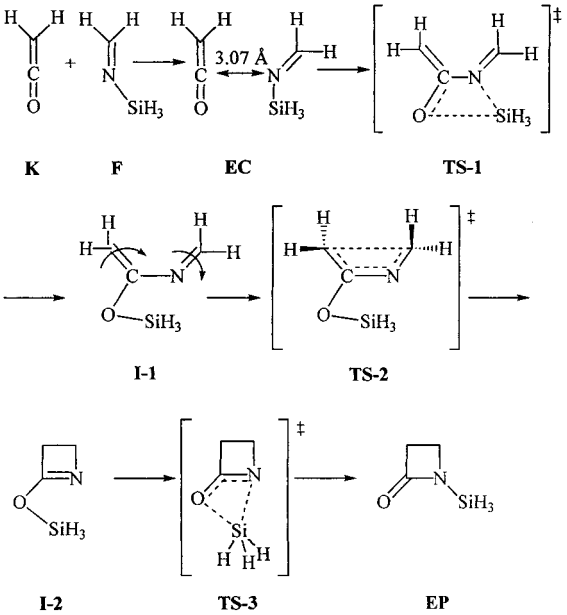
According to the proposed mechanism,[4c][4h,15] ketene formation takes place as the first step, followed by nucleophilic attack of the imine nitrogen atom on the ketene sp carbon atom.[16] To test if the presence of the *N*-silyl group is compatible with this mechanism, an ab initio study was performed, using as model compounds ketene (**K**) (H_2C=C=O) and *N*-silylformaldimine (**F**) (H_2C=N−SiH_3). The results of ab initio calculations are shown in Scheme 2, Table 2 and Figure 1. The simple model reported here, without substituents added to the ketene and silylimine, provides a preliminary insight into the important mechanistic parameters that influence these reactions.

First of all we note that, on going from MP2/6-31G* to QCISD(T)/6-311G**, the relative energy differences affect the nature of the proposed reaction path only in a slight stabilization of **TS-1** in comparison to **TS-2**. The calculations show the preliminary formation of a stabilizing electrostatic complex **EC** followed by the generation of a polar-type transition state **TS-1**. The length and the bond order of the C−N bond being formed (1.792 Å, 0.44 bond order) and the angle between the planes of the two subunits (44°) are in agreement with the transition state already calculated for a (non-silyl)imine.[15] The presence of the silyl atom, however, stabilizes **TS-1** in a *syn* form due to a weak bonding interaction between the oxygen atom and the silicon itself, as shown by the value of the Mulliken overlap population (0.106 e^-). Next, the simultaneous formation of the C−N and Si−O bonds takes place, resulting in the formation of the neutral silyloxyazadiene **I-1**, in which the long-range interaction of the silicon with the nitrogen atom (0.084 e^-) is responsible for the stabilization of a twisted (46°) *s-cis* conformation. This form is the absolute minimum of all the possible conformers. In no case, at this level of calculation, was a minimum found that corresponded to a zwitterionic structure with the silicon bonded to the nitrogen atom. The intermediate **I-1** then undergoes an electrocyclic conrotatory closure to the intermediate **I-2**

Table 1. Yields and diastereomeric ratio of homochiral azetidinones **4** and **5**

Entry	X	R	R^1	Y	Solvent[a]	Temp [°C]	Yield [%]	Products	Ratio
1	**X¹**	TIPS	CH_3	TMS	Toluene (2.38)	110	59	**4a/5a**	80:20
2	**X²**	TIPS	CH_3	TMS	Toluene	110	44	**4b/5b**	35:65
3	**X³**	TIPS	CH_3	TMS	Toluene	110	48	**4c/5c**	72:28
4	**X⁴**	TIPS	CH_3	TMS	Toluene	110	68	**4d/5d**	74:26
5	**X⁵**	TIPS	CH_3	TMS	Toluene	110	63	**4e/5e**	50:50
6	**X¹**	TBDMS	CH_3	TMS	Toluene	110	65	**4f/5f**	82:18
7	**X¹**	TBDPS	CH_3	TMS	Toluene	110	50	**4g/5g**	90:10
8	**X¹**	TES	CH_3	TMS	Toluene	110	11	**4h/5h**	70:30
9	**X¹**	*t*Bu	CH_3	TMS	Toluene	110	51	**4i/5i**	73:27
10	**X¹**	TIPS	CH_3	TBDMS	Toluene	110	54	**4k/5k**	64:36
11	**X¹**	TIPS	CH_3	TBDPS	Toluene	110	64	**4j/5j**	59:41
12	**X¹**	TIPS	CH_3	TIPS	Toluene	110	44	**4l/5l**	55:45
13	**X¹**	TBDMS	Et	TMS	Toluene	110	40	**4m/5m**	60:40
14	**X¹**	TIPS	Ph	TMS	Toluene	110	57	**4n/5n**	80:20
15	**X¹**	TIPS	*i*Pr	TMS	Toluene	110	48	**4o/5o**	73:27
16	**X¹**	TBDMS	*t*Bu	TMS	Toluene	110	84	**4p/5p**	88:12
17	**X¹**	TIPS	CH_3	TMS	CH_3CN (36.6)	82	48	**4a/5a**	85:15
18	**X¹**	TIPS	CH_3	TMS	DMF (38.3)	100	52	**4a/5a**	82:18
19	**X¹**	TIPS	CH_3	TMS	$CHCl_3$ (4.8)	61	60	**4a/5a**	86:14
20	**X¹**	TIPS	CH_3	TMS	CH_2Cl_2 (8.91)	40	8	**4a/5a**	80:20
21	**X¹**	TIPS	CH_3	TMS	$Cl(CH_2)_2Cl$ (10.1)	80	46	**4a/5a**	90:10
22	**X¹**	TIPS	CH_3	TMS	THF (7.52)	67	14	**4a/5a**	73:27
23	**X¹**	TIPS	CH_3	TMS	Ether (4.26)	34	5	**4a/5a**	80:20
24	**X¹**	TIPS	CH_3	TMS	NMP (170.0)	110	40	**4a/5a**	79:21
25	**X¹**	TIPS	CH_3	TBDMS	Neat (microwaves)[6c]	MW	61	**4a/5a**	60:40

[a] In parentheses: dielectric constants.



Scheme 2. Optimized intermediate and transition structures for the ketene-imine reaction

Table 2. Relative energies in kcal mol^{-1} compared to the total energy of the reactants (**K** + **F**) taken as zero; A: MP2/6-31G* + corrected ZPVE; B: QCISD(T)/6-311G**//MP2/6-31G* + corrected ZPVE

	A	B
K + F	0	0
EC	−3.13	−2.90
TS-1	7.97	9.61
I-1	−23.76	−19.72
TS-2	8.49	12.80
I-2	−31.31	−24.33
TS-3	−9.14	−1.07
EP	−41.38	−35.52

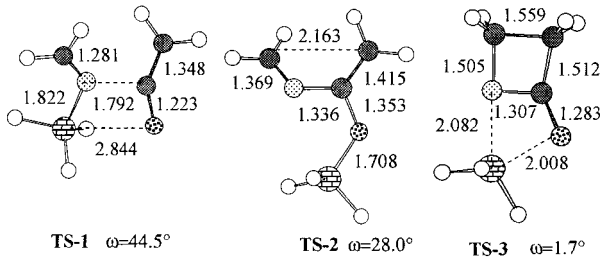


Figure 1. Fully optimized (MP2/6-31G* level) transition structures **TS-1**, **TS-2**, **TS-3**; bond lengths in Å; dihedral angle ω = C=N−C=C

through the transition state **TS-2**. This is characterized by a minor twisting of the skeleton (28°) and by a marked conrotatory twisting (50°) of the two methylene groups out of the plane of the double bonds. The C^1-C^4 distance (2.163 Å) and bond order (0.49) are in agreement with the formation of an incipient C−C bond. The silyl group remains covalently bonded to the oxygen atom, with a long-range interaction of the silicon with the nitrogen atom (0.064 e^-) that further stabilizes the *syn* conformation. This long-range interaction is actually present also in the intermediate **I-2** (0.078 e^-), which is characterized by the presence of a

localized C=N double bond in the ring (bond order 1.64). Transition state **TS-3**, in which the silicon atom is equally bonded to the oxygen (2.008 Å, 0.31 bond order) and to the nitrogen atom (2.082 Å, 0.33 bond order), respectively, in agreement with a silyl tropism from the oxygen to the nitrogen atom, is then reached. Silyl tropism is, ultimately, responsible for the formation of the end product **EP**.

A plot of the relative energy reaction coordinates for our reaction path and for the relative reaction path described by Sordo[15] (dotted line) for the classical Staudinger reaction, reveals some analogies as well as some differences (Figure 2).[19]

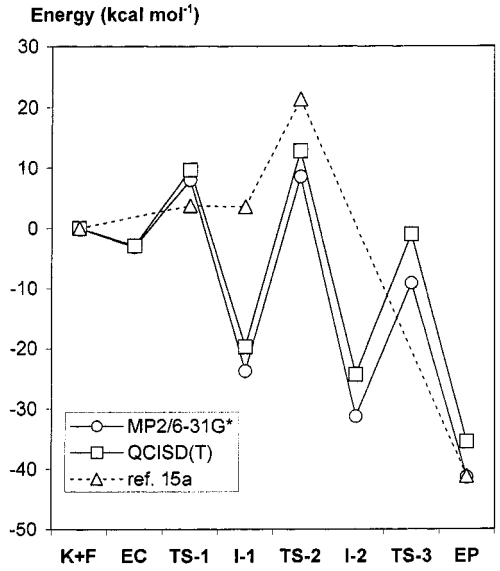


Figure 2. Proposed reaction path profile (full lines) and zwitterionic intermediate reaction path (dotted line)

Analyzing the first part of the diagram describing the formation of the first transition state, the paths, from an energetic point of view, are very close since both transition states are polar apart from the presence or absence of the silyl group. The difference between our path and the Sordo reaction path is evident from the second part of the diagram. In our case, the strong stabilization, due to silyl tropism from the nitrogen to the oxygen atom, stabilizes the azadiene as a neutral intermediate with respect to the zwitterionic structure found by Sordo. This stabilization allows the isolation and characterization of this compound, whereas in the classical Staudinger reaction, no isolation of the zwitterionic intermediate has been reported.

Due to the presence of the trialkylsilyl group, the final ring closure to the β-lactam ring, starting from the neutral azadiene, takes place via prior formation of the intermediate **I-2**, formally a 2-silyloxy-1-azetine (Scheme 2). Structurally this intermediate resembles the analogous 2-methoxyazetine, which has already been prepared and characterized.[21] Nevertheless, the intermediate **I-2** could not be isolated since the lower activation energy necessary for the *retro*-silyl tropism, compared with that necessary for ring closure, forces this intermediate to further evolve, giving rise to the formation of the final *trans*-*N*-silyl-β-lactam, whose greater stability provides the driving force for the whole process.

The well-known effects of solvent in the Staudinger reaction[22] suggest that this factor has to be included in our computational investigation. Calculations were performed on optimized gas-phase geometries as the experimental results show that the formation of a neutral azadiene intermediate occurs and that the diastereoselectivity is not influenced by changing the polarity of the solvent (see Table 1). The introduction of the role of the solvent does not affect the relative energies of the reaction profile except in the case of **TS-1** where a sizeable effect, due to the polar nature of the transition state, is evident. Nevertheless, this effect scarcely influences the entire reaction path. The results are reported in Figure 3 and Table 3.

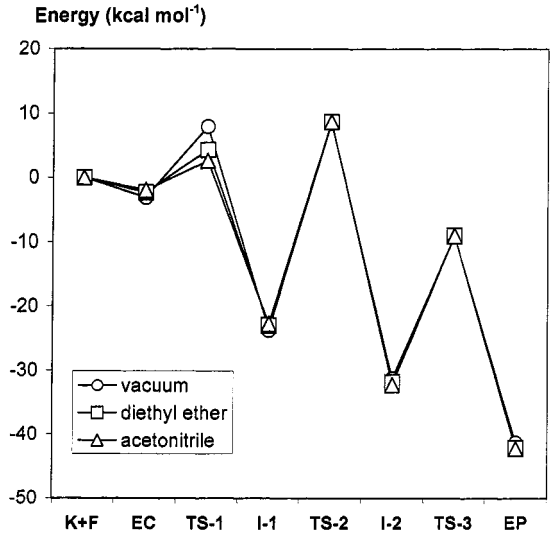
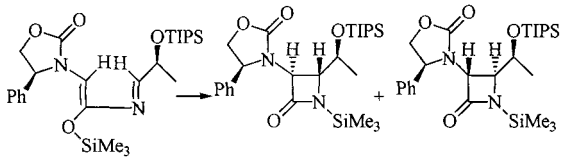


Figure 3. MP2/6-31G* + corrected ZPVE vacuum and solvent energy profiles

Table 3. MP2/6-31G* + corrected ZPVE solvent relative energies in kcal mol^{-1}; A: in vacuo; B: diethyl ether; C: acetonitrile

	A	B	C
K + F	0	0	0
EC	−3.13	−2.35	−1.98
TS-1	7.97	4.31	2.60
I-1	−23.76	−23.00	−22.76
TS-2	8.49	8.67	8.64
I-2	−31.31	−31.89	−32.27
TS-3	−9.14	−9.04	−9.08
EP	−41.38	−42.16	−42.29

At first sight, this mechanism does not consider the stereochemical course of the attack of the imine on a substituted ketene. In the classic Staudinger reaction the stereoselectivity of the ring closure is strongly influenced by the electronic nature of the ketene substituents, the so-called torquoelectronic effect.[15b,22b,22d,23,24] As the transition structure corresponding to the conrotatory closure of the zwitterionic intermediate is the rate-determining step of the ketene−imine cycloaddition reaction, an inward or outward preferential conformation of the substituent in the



Scheme 3

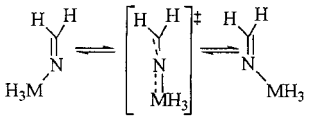
zwitterionic transition state leads to the *cis*- or *trans*-β-lactam rings.[4h,25,26] In our case, due to the high stability of the neutral silyloxyazadiene, the stereoselectivity is already established in the first step of the reaction at the very moment of azadiene formation. In fact we have been able to isolate such an intermediate as a unique compound which has been determined by NOE experiments[6a,27] to have the *syn*-*E*,*Z* configuration. Conrotatory electrocyclic closure of this compound gives rise, necessarily, to exclusive formation of the *trans* derivative (Scheme 3). This part of the reaction represents another example of surpassing the torquoelectronic effect in this well-known cycloaddition.[24b]

A more difficult aspect to explain is how such an azadiene geometry arises, because silylimines are reported to adopt the *anti* configuration,[13] and the attack of an *anti*-imine to the less hindered side of a ketene should lead to a (*Z*,*Z*)-azadiene and consequently to *cis* products.

Two reasons are generally invoked to explain the formation of a *trans*-β-lactam from an *anti*-imine:[4c] (a) reversible nucleophilic attack on the zwitterionic intermediate; (b) the presence of substituents that can stabilize the incipient positive charge on the iminic sp^2-carbon atom, leading to the isomerization of the incipient or just-formed *Z*,*Z* form to the less crowded *E*,*Z* form. If the latter is the case, the silyl group may play a crucial role since the stabilizing effect of the silyl group on a β-carbocation is well established. Nevertheless, it must be stressed that the silicon atom, in order to exert this influence, must remain linked to the imine nitrogen atom. The main drawback of this possibility is that the analysis of the reaction profile shows a "*quasi*" simultaneous migration of the silyl group from the nitrogen to the oxygen atom (see Scheme 2).

If we examine the geometry of **TS-1** we note that the chelating effect of the silicon atom on the ketene oxygen atom constrains the structure in a *syn* form in which the two methylene groups on the ketene and the imine are close together. This fact enhances the steric repulsive effect of the substituents, especially when they are bulky as in our cases, and forces the attack to give the less-crowded *E*,*Z* form. To obtain this result the imine must easily change its configuration. The change of configuration in imines takes place by pyramidal inversion at the nitrogen atom. Alkylimines have a high inversion barrier (27 kcal mol^{-1})[28] but it is known that such a barrier is significantly lowered by higher row atoms belonging to group IVa (germanium) directly linked to the inversion center.[28] In this context we determined the inversion barrier of *N*-silylformaldimine by ab initio calculations. The barrier is taken as the energy difference between the bent form and a linear transition state. The calculated value is 9.3 kcal mol^{-1}. For the sake of comparison these calculations were also performed on *N*-methyl- and *N*-germylformaldimine; the results are reported in Table 4.

Table 4. MP2/6-31G* inversion barrier energies (kcal mol^{-1})



M	calc	exp[28]
C	32.5	27
Si	9.3	--
Ge	14.9	9.2

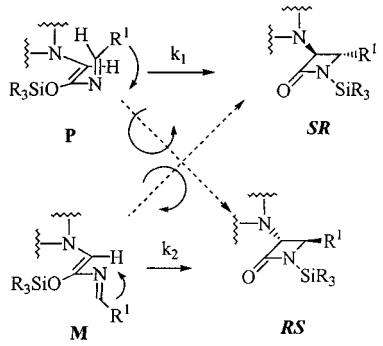
The calculated values compare well with the experimental ones. The value for the *N*-silylimine is predictably lower than that for *N*-alkylimines and allows for the facile inversion at the nitrogen atom as requested from the steric requirements.

To the extent that we changed reaction conditions (including the sequence of addition of reactants, which has been reported to influence the simple diastereoselectivity of the substituents of the final ring)[16,29] sole formation of the *trans*-azetidinone is always observed (Table 1).

From the results collected in Table 1 it is clear that *retro*-silyl tropism must take place during formation of the β-lactam ring since it is possible to isolate the azetidinone protected as the *N*-trialkylsilyl derivative, thus supporting the mechanistic pathway derived from the calculations. Further confirmation of the proposed mechanism comes from the observation that the solvents with different dielectric constants barely influence the stereochemistry of the end product (Table 1, Entries 17−25), supporting the concerted ring-closure mechanism.

NOE studies and ab initio calculations show that the azadiene intermediate prefers two *s-cis* conformations (Scheme 4): a twisted conformation with a C=N−C=C dihedral angle of about 45° (rotamer **P**) and the corresponding counter clockwise conformation (rotamer **M**). Asymmetric induction in the ketene−imine cycloaddition reaction can be explained by inspecting the stability of azadiene rotamers **P** and **M** (Scheme 4).

Conrotatory clockwise ring closure of conformer **P** with the imine moiety on the top face yields the β-lactam ***SR*** and conrotatory counter-clockwise ring closure of the conformer **M**, with the imine moiety on the bottom face, results in the formation of β-lactam ***RS***. Before ring closure can occur, the central C−N bond has to rotate towards an eclipsed arrangement. As pointed out by Hegedus,[4c,26] in



Scheme 4

principle the central bond in intermediate **P** could rotate about 315° to form ***RS*** and the intermediate **M** could also rotate by the same amount to form ***SR***. As the rotation from **P** to ***SR*** and **M** to ***RS*** is only about 45°, the principle of least motion can be invoked to explain the preferential formation of ***SR*** and ***RS*** from **P** and **M**, respectively. Moreover, conrotatory ring closure of the terminal double bonds of intermediate **P** can only occur in a clockwise fashion. Counter-clockwise closure would necessitate the hydrogen atom of the ketene and the hydrogen atom of the imine passing through each other. Of course, for the rotamer **M**, a counter-clockwise rotation must occur to give the ***RS*** β-lactam.

The essentially invariable diastereoselectivity of the reaction, changing temperature and solvent (see Entries 17−25 in Table 1) allows the exclusion of a re-equilibration between the final β-lactam products. In this context, since the ab initio calculations show that the transition states are more reagent- than product-like, it is clear that we must focus our attention on the nature of rotamer **P**, which gives rise to the ***SR*** azetidinone, and of rotamer **M**, which gives rise to the ***RS*** azetidinone. Changing the hydroxyethyl protecting group on the side chain (Table 1, Entries 6−9) scarcely influences the stereochemical outcome of the reaction with a clear prevalence for the ***SR*** azetidinone. Moreover, even the steric demand of the alkyl substituents on the C-4 side chain (Table 1, Entries 13−16) causes only a slight difference in the diastereomeric ratio of the end products. What does have a clear effect on the diastereoselectivity of the ring closure is the nature of the trialkylsilyl group directly linked to the enol oxygen atom in the azadiene and the nature of the substituent on C-3 (Entries 2−5 Table 1). Entries 2−5 take into account the variation of facial diastereoselectivity upon varying these substituents. A phthalimido group has no influence on the diastereoselectivity of the reaction. The azadiene in which Evans' *S* chiral auxiliary and the (*S*)-(triisopropylsilyloxy)ethyl moiety are simultaneously present leads to the best diastereoselectivity.

Because ***RS*** and ***SR*** products are not subject to thermodynamic re-equilibrium (see above), the experimentally observed ratio of the products may depend: (a) on differing thermodynamic stabilities of the precursors or (b) on the ratio between the kinetic constants k_1 and k_2. It has been reported in the Staudinger reaction that the *S* Evans' auxiliary induces preferential *S* chirality on the adjacent carbon atom in the closure of the *cis*-β-lactam. This induction has been explained by Cossío,[30] using semiempirical calculations, as being the result of a destabilization of the transition state, leading to the β-lactam ring, arising from rotamer **M** in comparison with the transition state arising from rotamer **P**. The destabilization is ascribed, at least in part, to a nonbonding interaction between the negatively charged oxygen atom and the phenyl group of Evans' auxiliary. In our case the effect may be caused by the silyloxy group: with relatively small trialkylsilyloxy groups high diastereoselectivity is achieved, whereas sterically more demanding groups decrease the diastereoselectivity as a result of increased perturbation. By analogy to the elegant work by Evans on the asymmetric Diels−Alder cycloaddition reaction with a chiral oxazolidin-2-one auxiliary[31] one can suppose that the sum of weak interactions determines the final facial diastereoselectivity.

Experimental Section

General: Melting points were taken with a Mel-Temp apparatus and are uncorrected. − ^{1}H- and ^{13}C NMR spectra were recorded in $CDCl_3$ with a Varian Gemini-200 spectrometer. Chemical shifts are reported on the δ scale and coupling constants (*J*) are reported in Hz. − Infrared spectra were recorded as nujol mulls with a Nicolet 205 FT-IR spectrophotometer. − Optical rotation measurements were carried out with a Perkin−Elmer 343 Polarimeter and specific rotation $[\alpha]_D^{20}$ values are reported in deg per dm at the specified temperature and with the concentration [*c*] given in g per 100 mL in $CHCl_3$. THF, toluene, and heptane were distilled from benzophenone ketyl.

Materials: *N*-Trialkylsilylimines were prepared according to reported procedures starting from the parent aldehydes.[13,32,33] 2-[(*tert*-Butyldimethylsilyloxy)butyraldehyde was prepared by the Burke procedure.[34] 2-[(*tert*-Butyldimethylsilyloxy)-3,3-dimethylbutyraldehyde was prepared by the Corey procedure.[35] Ethyl 1-bromoacetate was distilled prior to use. (*S*)-(4-Isopropyl-3-propionyl-2-oxazolidinone) was prepared according to the procedure described by Evans.[36] (*S*)-4-Benzyloxazolidinones were prepared from their corresponding amino alcohols using the routes of Evans and Gage.[37] Triethylamine was dried with KOH. Other solvents and reagents were obtained commercially and were used as received. All reactions were performed under nitrogen on a 1.0-mmol scale and the yields refer to the whole process starting from the aldehyde.

Note: Compounds **4a**, **5a**, **4b**, **5b**, **4c**, **5c**, **4d**, **5d**, **4e**, **5e**, **4f**, **5f**, **4g**, **5g**, **4h**, **5h**, **4i**, **5i**, **4m**, **5m**, **4n**, **5n**, **4o**, **5o** and **4p**, **5p** were identified and characterized as NH derivatives due to the facile hydrolysis of the N−$SiMe_3$ bond under workup and purification conditions. For the sake of simplicity, the imines **1** and azadienes **3** have been given the same alphabetical designation as the final azetidinones. For a better understanding see Scheme 1 and Table 1.

(4*S*)-3-{(3*S*,4*R*)-2-Oxo-4-[(*S*)-1-(triisopropylsilyloxy)ethyl]-1-(trimethylsilyl)azetidine-3-yl}-4-phenyloxazolidin-2-one (4a) and (4*S*)-3-{(3*R*,4*S*)-2-Oxo-4-[(*S*)-1-(triisopropylsilyloxy)ethyl]-1-(trimethylsilyl)azetidine-3-yl}-4-phenyloxazolidin-2-one (5a): A solution of (2*S*)-2-(triisopropylsiloxy)-*N*-(trimethylsilyl)propanimine (**1a**) was prepared by dropwise addition of a heptane solution (5 mL) of (2*S*)-2-(triisopropylsiloxy)propionaldehyde (0.23 g, 1.00 mmol) to a cooled (−10 °C) THF solution of lithium bis(trimethylsilyl)amide (LHMDS) (1.10 mL, 1.10 mmol). The reaction mixture was warmed to 0 °C and stirred for 30 min at this temperature. The formation of the imine was confirmed by an infrared spectrum of the reaction mixture [$\tilde{\nu}_{CN}$ = 1685 cm^{-1}]. The imine solution was then warmed to room temperature, trimethylsilyl chloride (0.15 mL, 1.10 mmol) was added in one portion, and this mixture was stirred for 1.5 h. The mixture was cooled to 0 °C and triethylamine (0.30 mL, 2.20 mmol) was added in one portion. After stirring this mixture for 5 min at 0 °C, a toluene solution of oxazolidinone/acetyl chloride **2-X^1** (1.1 equiv.), prepared according Boger's method,[37] was added very slowly (during 5 min) by syringe. Stirring was maintained for 30 min at 0 °C and 30 min at room temp. This yellow-orange mixture was then filtered through Celite. An

aliquot of this mixture, after removal of the solvent, was analyzed by ^{1}H- and ^{13}C-NMR spectroscopy, which showed that it contained essentially pure **3a**.

(5*S*)-1-[(4*S*)-(2-Oxo-4-phenyl-1,3-oxazolidin-3-yl)]-5-(triisopropylsilyloxy)-2-trimethylsilyloxy-3-azahexa-1,3-diene (3a): $[\alpha]_D^{20}$ = +72.2 (*c* = 1.6, $CHCl_3$). − IR ($CHCl_3$): $\tilde{\nu}$ = 1748 cm^{-1}. − ^{1}H NMR (200 MHz, $CDCl_3$): δ = 7.52 (d, 1 H, *J* = 4.6 Hz), 7.35 (m, 5 H), 5.80 (s, 1 H), 5.32 (dd, 1 H, *J* = 3.7, 8.6 Hz), 4.65 (t, 1 H, *J* = 8.6 Hz), 4.30 (m, 2 H), 1.25 (d, 3 H, *J* = 6.4 Hz), 1.02 (m, 3 H), 0.95 (s, 18 H). − ^{13}C NMR (50 MHz, $CDCl_3$): δ = 163.4, 156.5, 146.7, 139.1, 128.9, 128.6, 126.3, 102.0, 70.3, 70.1, 59.4, 21.7, 17.8, 17.0, 12.1, 0.4.

The yellow orange mixture, obtained as reported above, was heated under reflux (110 °C) for 8 h. The crude reaction mixture was cooled to room temp, diluted with ethyl acetate, poured into NH_4Cl solution, and extracted with more ethyl acetate. This organic layer was washed with Na_2CO_3, brine, and dried with Na_2SO_4. After filtration, the solvent was removed under reduced pressure and a ^{1}H-NMR spectrum of the resulting yellow-orange oil was obtained. Diastereomeric ratio = 80:20 (**4a**/**5a**) where **4** will hereafter, except for Entry 2 Table 1, be referred to as the major isomer and **5** as the minor isomer. The crude product was further purified by flash column chromatography (dichloromethane/acetone, 9:1 as eluent). Yields: 0.203 g of **4a** and 0.051 g of **5a** (59% total yield).

Compound 4a (Identified as NH-Azetidinone): M.p. 156−158 °C. − $[\alpha]_D^{20}$ = +40.1 (*c* = 2.0, $CHCl_3$). − IR ($CHCl_3$): $\tilde{\nu}$ = 3420, 1757 cm^{-1}. − ^{1}H NMR (200 MHz, $CDCl_3$): δ = 7.38 (s, 5 H), 5.80 (s, 1 H), 4.95 (dd, 1 H, *J* = 6.6, 8.8 Hz), 4.92 (d, 1 H, *J* = 2.2 Hz), 4.67 (t, 1 H, *J* = 8.8 Hz), 4.20 (dd, 1 H, *J* = 6.6, 8.8 Hz), 3.98 (dq, 1 H, *J* = 2.2, 6.3 Hz), 3.04 (t, 1 H, *J* = 2.2 Hz), 1.12 (d, 3 H, *J* = 6.3 Hz), 0.95 (s, 21 H). − ^{13}C NMR (50 MHz, $CDCl_3$): δ = 165.1, 157.1, 138.3, 129.4, 129.3, 127.3, 70.4, 66.2, 60.1, 59.5, 59.1, 19.7, 17.9, 12.8. − MS; *m*/*z*: 389 [M^+ − 43], 346, 294, 273, 231, 186. − $C_{23}H_{36}N_2O_4Si$ (432.6): calcd. C 63.85, H 8.39, N 6.48; found C 63.51, H 8.42, N 6.52.

Compound 5a (Identified as NH-Azetidinone): M.p. 136−138 °C. − $[\alpha]_D^{20}$ = +92.8 (*c* = 1.8, $CHCl_3$). − IR ($CHCl_3$): $\tilde{\nu}$ = 3425, 1760 cm^{-1}. − ^{1}H NMR (200 MHz, $CDCl_3$): δ = 7.40 (s, 5 H), 5.95 (s, 1 H), 4.93 (dd, 1 H, *J* = 7.1, 8.7 Hz), 4.68 (t, 1 H, *J* = 8.7 Hz), 4.20 (dd, 1 H, *J* = 7.1, 8.75 Hz), 4.08 (d, 1 H, *J* = 2.6 Hz), 4.02 (dd, 1 H, *J* = 2.6, 5.3 Hz), 3.73 (m, 1 H), 0.95 (m, 24 H). − ^{13}C NMR (50 MHz, $CDCl_3$): δ = 165.1, 157.1, 137.2, 129.5, 129.3, 127.4, 70.3, 68.8, 61.7, 61.5, 59.2, 20.2, 18.0, 18.0, 12.5. − MS; *m*/*z*: 389 [M^+ − 43], 346, 294, 273, 186. − $C_{23}H_{36}N_2O_4Si$ (432.6): calcd. C 63.85, H 8.39, N 6.48; found C 64.14, H 8.34, N 6.51.

(4*R*)-3-{(3*S*,4*R*)-2-Oxo-4-[(*S*)-1-(triisopropylsilyloxy)ethyl]-1-(trimethylsilyl)azetidine-3-yl}-4-phenyloxazolidin-2-one (4b) and (4*R*)-3-{(3*R*,4*S*)-2-Oxo-4-[(*S*)-1-(triisopropylsilyloxy)ethyl}-1-(trimethylsilyl)azetidine-3-yl]-4-phenyloxazolidin-2-one (5b): The title compounds were prepared from the acyl chloride **2-X**[2] and the imine **1a** following the procedure reported above. Yields: 0.067 g of **4b** and 0.123 g of **5b** (44% total yield). Diastereomeric ratio 35:65.

Compound 4b (Identified as NH-Azetidinone): M.p. 46−48 °C. − $[\alpha]_D^{20}$ = −71.5 (*c* = 1.0, $CHCl_3$). − IR ($CHCl_3$): $\tilde{\nu}$ = 3423, 1759 cm^{-1}. − ^{1}H NMR (200 MHz, $CDCl_3$): δ = 7.38 (s, 5 H), 5.95 (s, 1 H), 4.90 (dd, 1 H, *J* = 7.2, 8.8 Hz), 4.69 (t, 1 H, *J* = 8.8 Hz), 4.28 (dd, 1 H, *J* = 7.2, 8.8 Hz), 4.26 (d, 1 H, *J* = 2.64 Hz), 4.09 (m, 1 H), 3.88 (dq, 1 H, *J* = 1.8, 6.4 Hz), 0.92 (s, 21 H), 0.72 (d, 3 H, *J* = 6.4 Hz). − ^{13}C NMR (50 MHz, $CDCl_3$): δ = 165.8, 157.0, 137.2, 129.6, 129.3, 127.7, 70.1, 66.2, 61.8, 59.3, 58.4, 19.3, 18.0, 17.9, 12.4. − MS; *m*/*z*: 432 [M^+], 389 [M^+ − 43], 346, 294, 273, 186. − $C_{23}H_{36}N_2O_4Si$ (432.6): calcd. C 63.85, H 8.39, N 6.48; found C 63.48, H 8.36, N 6.45.

Compound 5b (Identified as NH-Azetidinone): M.p. 116−118 °C. − $[\alpha]_D^{20}$ = −37.5 (*c* = 1.1, $CHCl_3$). − IR ($CHCl_3$): $\tilde{\nu}$ = 3431, 1757 cm^{-1}. − ^{1}H NMR (200 MHz, $CDCl_3$): δ = 7.40 (s, 5 H), 5.85 (s, 1 H), 4.95 (dd, 1 H, *J* = 6.8, 8.9 Hz), 4.67 (t, 1 H, *J* = 8.9 Hz), 4.56 (d, 1 H, *J* = 2.6 Hz), 4.20 (dd, 1 H, *J* = 6.8, 8.9 Hz), 3.87 (m, 1 H), 3.00 (dd, 1 H, *J* = 2.6, 6.8 Hz), 1.10 (d, 3 H, *J* = 6.2 Hz), 0.96 (s, 21 H). − ^{13}C NMR (50 MHz, $CDCl_3$): δ = 164.5, 157.3, 138.0, 129.5, 129.4, 127.4, 70.5, 69.9, 62.2, 60.3, 59.2, 19.9, 18.0, 12.5. − MS; *m*/*z*: 432 [M^+], 389 [M^+ − 43], 346, 294, 273, 186. − $C_{23}H_{36}N_2O_4Si$ (432.6): calcd. C 63.85, H 8.39, N 6.48; found C 64.02, H 8.42, N 6.51.

(4*S*)-3-{(3*S*,4*R*)-2-Oxo-4-[(*S*)-1-(triisopropylsilyloxy)ethyl]-1-(trimethylsilyl)azetidine-3-yl}-4-(1-methylethyl)oxazolidin-2-one (4c) and (4*S*)-3-{(3*R*,4*S*)-2-Oxo-4-[(*S*)-1-(triisopropylsilyloxy)ethyl]-1-(trimethylsilyl)azetidine-3-yl}-4-(1-methylethyl)oxazolidin-2-one (5c): The title compounds were prepared from the acyl chloride **2-X**[3] and the imine **1a** following the above procedure. Yields: 0.137 g of **4c** and 0.053 g of **5c** (48% total yield). Diastereomeric ratio 72:28.

Compound 4c (Identified as NH-Azetidinone): M.p. 101 °C. − $[\alpha]_D^{20}$ = +26.5 (*c* = 2.0, $CHCl_3$). − IR ($CHCl_3$): $\tilde{\nu}$ = 3422, 1752 cm^{-1}. − ^{1}H NMR (200 MHz, $CDCl_3$): δ = 6.40 (s, 1 H), 4.72 (d, 1 H, *J* = 2.3 Hz), 4.24 (t, 1 H, *J* = 9.0 Hz), 4.12 (m, 2 H), 3.83 (m, 1 H), 3.78 (t, 1 H, *J* = 2.5 Hz), 2.00 (m, 1 H), 1.21 (d, 3 H, *J* = 6.3 Hz), 1.00 (s, 21 H), 0.95 (d, 3 H, *J* = 6.8 Hz), 0.88 (d, 3 H, *J* = 6.8 Hz). − ^{13}C NMR (50 MHz, $CDCl_3$): δ = 165.6, 157.2, 66.6, 63.3, 60.3, 60.0, 59.9, 29.3, 20.3, 17.9, 17.5, 14.2, 12.4. − MS; *m*/*z*: 398 [M^+], 355, 186. − $C_{20}H_{38}N_2O_4Si$ (398.6): calcd. C 60.26, H 9.61, N 7.03; found C 60.51, H 9.66, N 7.00.

Compound 5c (Identified as NH-Azetidinone): M.p. 115 °C. − $[\alpha]_D^{20}$ = +59.5 (*c* = 1.1, $CHCl_3$). − IR ($CHCl_3$): $\tilde{\nu}$ = 3429, 1770, 1751 cm^{-1}. − ^{1}H NMR (200 MHz, $CDCl_3$): δ = 6.10 (s, 1 H), 4.52 (d, 1 H, *J* = 2.5 Hz), 4.25 (t, 1 H, *J* = 8.9 Hz), 4.10 (m, 2 H), 3.86 (m, 2 H), 2.10 (m, 1 H), 1.22 (d, 3 H, *J* = 6.2 Hz), 1.00 (s, 21 H), 0.88 (m, 6 H). − ^{13}C NMR (50 MHz, $CDCl_3$): δ = 165.4, 157.5, 69.2, 63.0, 61.6, 60.4, 59.3, 28.2, 20.3, 18.1, 17.9, 14.0, 12.6. − MS; *m*/*z*: 398 [M^+], 355, 186. − $C_{20}H_{38}N_2O_4Si$ (398.6): calcd. C 60.26, H 9.61, N 7.03; found C 60.01, H 9.68, N 7.07.

(4*S*)-3-{(3*S*,4*R*)-2-Oxo-4-[(*S*)-1-(triisopropylsilyloxy)ethyl]-1-(trimethylsilyl)azetidine-3-yl}-4-(phenylmethyl)oxazolidin-2-one (4d) and (4*S*)-3-{(3*R*,4*S*)-2-Oxo-4-[(*S*)-1-(triisopropylsilyloxy)ethyl]-1-trimethylsilylazetidine-3-yl}-4-(phenylmethyl)oxazolidin-2-one (5d): The title compounds were prepared from the acyl chloride **2-X**[4] and the imine **1a** following the above procedure. Yields: 0.224 g of **4d** and 0.078 g of **5d** (68% total yield). Diastereomeric ratio 74:26.

Compound 4d (Identified as NH-Azetidinone): Oil. − $[\alpha]_D^{20}$ = +6.4 (*c* = 0.4, $CHCl_3$). − IR ($CHCl_3$): $\tilde{\nu}$ = 3230, 1772, 1745, 1732, 1717 cm^{-1}. − ^{1}H NMR (200 MHz, $CDCl_3$): δ = 7.33 (m, 3 H), 7.20 (d, 2 H, *J* = 6.7 Hz), 5.90 (br. s, 1 H), 4.84 (d, 1 H, *J* = 2.4 Hz), 4.21 (m, 3 H), 4.08 (m, 1 H), 3.86 (t, 1 H, *J* = 2.4 Hz), 3.24 (dd, 1 H, *J* = 3.5, 13.2 Hz), 2.80 (dd, 1 H, *J* = 9.2, 10.6 Hz), 1.28 (d, 3 H, *J* = 6.3 Hz), 1.09 (s, 21 H). − ^{13}C NMR (50 MHz, $CDCl_3$): δ = 165.4, 157.1, 135.0, 129.0, 127.3, 67.3, 66.6, 61.0, 60.2, 58.8, 40.1, 20.3, 18.1, 18.1, 12.5. − MS; *m*/*z*: 403 [M^+ − *i*Pr], 360. − $C_{24}H_{38}N_2O_4Si$ (446.6): calcd. C 64.54, H 8.58, N 6.27; found C 64.22, H 8.53, N 6.31.

Compound 5d (Identified as NH-Azetidinone): Oil. − $[\alpha]_D^{20}$ = +25.35 (*c* = 0.15, $CHCl_3$). − IR ($CHCl_3$): $\tilde{\nu}$ = 3230, 1772, 1743, 1717 cm^{-1}. − ^1H NMR (200 MHz, $CDCl_3$): δ = 7.28 (m, 3 H), 7.18 (d, 2 H, *J* = 6.7 Hz), 6.33 (br. s, 1 H), 4.71 (d, 1 H, *J* = 2.2 Hz), 4.26−4.00 (m, 4 H), 3.92 (dd, 1 H, *J* = 2.4, 5.5 Hz), 3.34 (dd, 1 H, *J* = 3.0, 13.7 Hz), 2.74 (m, 1 H), 1.26 (d, 3 H, *J* = 6.3 Hz), 1.07 (s, 21 H). − ^{13}C NMR (50 MHz, $CDCl_3$): δ = 165.6, 157.2, 135.0, 129.0, 127.2, 69.1, 67.2, 61.4, 59.7, 57.0, 38.5, 20.2, 18.1, 12.6. − MS; *m*/*z*: 403 [M^+ − *i*Pr], 360. − $C_{24}H_{38}N_2O_4Si$ (446.6): calcd. C 64.54, H 8.58, N 6.27; found C 64.60, H 8.61, N 6.32.

(5*S*)-2-(*tert*-Butyldimethylsilyloxy)-1-phthalimido-5-(triisopropylsilyloxy)-3-azahexa-1,3-diene (3e): The title compound was prepared from the acyl chloride **2-X**[5] and the imine **1a** following the above procedure (see preparation of **3a**). − $[\alpha]_D^{20}$ = −26.0 (*c* = 1.0 $CHCl_3$). − IR ($CHCl_3$): $\tilde{\nu}$ = 1735 cm^{-1}. − ^1H NMR (200 MHz, $CDCl_3$): δ = 7.96 (d, 1 H, *J* = 4.6 Hz), 7.82 (m, 2 H), 7.68 (m, 2 H), 5.47 (s, 1 H), 4.48 (m, 1 H), 1.32 (d, 3 H, *J* = 6.5 Hz), 1.02 (m, 21 H), 0.75 (s, 9 H), 0.02 (s, 6 H). − ^{13}C NMR (50 MHz, $CDCl_3$): δ = 168.1, 166.9, 155.7, 133.9, 132.3, 123.2, 90.5, 70.4, 25.5, 21.7, 17.9, 17.6, 17.6, 12.2, −4.1, −4.2.

2-{(3*S*,4*R*)-2-Oxo-4-[(*S*)-1-(triisopropylsilyloxy)ethyl]azetidin-3-yl}isoindole-1,3-dione (4e) and 2-{(3*R*,4*S*)-2-Oxo-4-[(*S*)-1-(triisopropylsilyloxy)ethyl]azetidin-3-yl}isoindole-1,3-dione (5e): The title compounds were obtained following the general procedure, starting from **3e**, as reported for compounds **4a** and **5a** (see above). Yields: 0.131 g of **4e** and 0.131 g of **5e** (63% total yield). Diastereomeric ratio 50:50.

Compound 4e (Identified as NH-Azetidinone): M.p. 182−184 °C. − $[\alpha]_D^{20}$ = −21.4 (*c* = 2.7, $CHCl_3$). − IR ($CHCl_3$): $\tilde{\nu}$ = 3418, 1785, 1773, 1721 cm^{-1}. − ^1H NMR (200 MHz, $CDCl_3$): δ = 7.80 (m, 4 H), 6.20 (s, 1 H), 5.46 (d, 1 H, *J* = 2.6 Hz), 4.22 (dq, 1 H, *J* = 2.6, 6.3 Hz), 3.90 (t, 1 H, *J* = 2.6 Hz), 1.19 (d, 3 H, *J* = 6.3 Hz), 1.06 (s, 21 H). − ^{13}C NMR (50 MHz, $CDCl_3$): δ = 167.0, 165.0, 134.4, 131.8, 123.6, 66.6, 60.2, 54.7, 20.3, 18.1, 18.1, 12.6. − MS; *m*/*z*: 373 [M^+ − 43], 330, 278, 186. − $C_{22}H_{32}N_2O_4Si$ (416.5): calcd. C 63.43, H 7.74, N 6.72; found C 63.71, H 7.71, N 6.80.

Compound 5e (Identified as NH-Azetidinone): M.p. 196−198 °C. − $[\alpha]_D^{20}$ = +21.3 (*c* = 1.2, $CHCl_3$). − IR ($CHCl_3$): $\tilde{\nu}$ = 3420, 1785, 1772, 1723 cm^{-1}. − ^1H NMR (200 MHz, $CDCl_3$): δ = 7.80 (m, 4 H), 6.20 (s, 1 H), 5.10 (d, 1 H, *J* = 2.8 Hz), 4.10 (m, 1 H), 3.93 (dd, 1 H, *J* = 2.8, 7.1 Hz), 1.22 (d, 3 H, *J* = 6.1 Hz), 1.05 (s, 21 H). − ^{13}C NMR (50 MHz, $CDCl_3$): δ = 166.9, 164.3, 134.4, 131.7, 123.7, 70.1, 60.7, 56.5, 19.9, 18.1, 18.0, 12.6. − MS; *m*/*z*: 373 [M^+ − 43], 330, 278, 186. − $C_{22}H_{32}N_2O_4Si$ (416.5): calcd. C 63.43, H 7.74, N 6.72; found C 63.15, H 7.70, N 6.75.

(4*S*)-3-{(3*S*,4*R*)-4-[(*S*)-1-(*tert*-Butyldimethylsilyloxy)ethyl]-2-oxo-1-(trimethylsilyl)azetidine-3-yl}-4-phenyloxazolidin-2-one (4f) and (4*S*)-3-{(3*R*,4*S*)-4-[(*S*)-1-(*tert*-Butyldimethylsilyloxy)ethyl]-2-oxo-1-(trimethylsilyl)azetidine-3-yl}-4-phenyloxazolidin-2-one (5f): The title compounds were prepared from the acyl chloride **2-X**[1] and the imine **1f** following the above procedure. Yields: 0.253 g of **4f** and 0.046 g of **5f** (65% total yield). Diastereomeric ratio 82:18.

Compound 4f (Identified as NH-Azetidinone): M.p. 201 °C. − $[\alpha]_D^{20}$ = +48.8 (*c* = 1.4, $CHCl_3$). − IR ($CHCl_3$): $\tilde{\nu}$ = 3424, 1759 cm^{-1}. − ^1H NMR (200 MHz, $CDCl_3$): δ = 7.38 (s, 5 H), 5.84 (s, 1 H), 4.95 (dd, 1 H, *J* = 6.6, 8.8 Hz), 4.87 (d, 1 H, *J* = 2.4 Hz), 4.67 (t, 1 H, *J* = 8.8 Hz), 4.21 (dd, 1 H, *J* = 6.6, 8.8 Hz), 3.78 (dq, 1 H, *J* = 2.6, 6.4 Hz), 3.00 (t, 1 H, *J* = 2.5 Hz), 1.07 (d, 3 H, *J* = 6.4 Hz), 0.76 (s, 9 H), −0.05 (s, 3 H), −0.10 (s, 3 H). − ^{13}C NMR (50 MHz, $CDCl_3$): δ = 165.2, 157.1, 138.4, 129.5, 129.4, 127.4, 70.4, 66.1, 60.3, 59.2, 59.1, 25.6, 19.9, 17.9, −4.6, −5.0. − MS; *m*/*z*: 375 [M^+ − 15], 333 [M^+ − *t*Bu], 290, 238, 130. − $C_{20}H_{30}N_2O_4Si$ (390.5): calcd. C 61.51, H 7.74, N 7.17; found C 61.79, H 7.71, N 7.15.

Compound 5f (Identified as NH-Azetidinone): M.p. 140−142 °C. − $[\alpha]_D^{20}$ = +90.8 (*c* = 1.0, $CHCl_3$). − IR ($CHCl_3$): $\tilde{\nu}$ = 3425, 1759 cm^{-1}. − ^1H NMR (200 MHz, $CDCl_3$): δ = 7.40 (m, 5 H), 5.85, (s, 1 H), 4.92 (dd, 1 H, *J* = 7.0, 8.7 Hz), 4.68 (t, 1 H, *J* = 8.7 Hz), 4.20 (dd, 1 H, *J* = 7.00, 8.7 Hz), 4.03 (d, 1 H, *J* = 2.5 Hz), 3.98 (dd, 1 H, *J* = 2.5, 4.9 Hz), 3.50 (m, 1 H), 0.90 (d, 3 H, *J* = 6.3 Hz), 0.73 (s, 9 H), −0.08 (s, 3 H), −0.22 (s, 3 H). − ^{13}C NMR (50 MHz, $CDCl_3$): δ = 165.0, 157.8, 137.4, 129.6, 129.5, 127.5, 70.4, 68.2, 61.4, 59.1, 25.6, 20.2, 17.8, −4.21, −5.16. − MS; *m*/*z*: 375 [M^+ − 15], 333 [M^+ − *t*Bu], 290, 238, 130. − $C_{20}H_{30}N_2O_4Si$ (390.5): calcd. C 61.51, H 7.74, N 7.17; found C 61.24, H 7.78, N 7.21.

(4*S*)-3-{(3*S*,4*R*)-4-[(*S*)-1-(*tert*-Butyldiphenylsilyloxy)ethyl]-2-oxo-1-(trimethylsilyl)azetidine-3-yl}-4-phenyloxazolidin-2-one (4g) and (4*S*)-3-{(3*R*,4*S*)-4-[(*S*)1-(*tert*-Butyldiphenylsilyloxy)ethyl]-2-oxo-1-(trimethylsilyl)azetidine-3-yl}-4-phenyloxazolidin-2-one (5g): The title compounds were prepared from the acyl chloride **2-X**[1] and the imine **1g** following the above procedure. Yields: 0.231 g of **4g** and 0.026 g of **5g** (50% total yield). Diastereomeric ratio 90:10.

Compound 4g (Identified as NH-Azetidinone): M.p. 105−110 °C (decomp.). − $[\alpha]_D^{20}$ = +19.0 (*c* = 1.2, $CHCl_3$). − IR ($CHCl_3$): $\tilde{\nu}$ = 3423, 1768 cm^{-1}. − ^1H NMR (200 MHz, $CDCl_3$): δ = 7.58 (m, 4 H), 7.35 (m, 11 H), 5.70 (s, 1 H), 5.06 (d, 1 H, *J* = 2.3 Hz), 4.90 (dd, 1 H, *J* = 6.5, 8.8 Hz), 4.66 (t, 1 H, *J* = 8.8 Hz), 4.18 (dd, 1 H, *J* = 6.5, 8.8 Hz), 3.80 (dq, 1 H, *J* = 2.3, 6.4 Hz), 2.93 (t, 1 H, *J* = 2.3 Hz), 1.05 (3 H, *J* = 6.4 Hz), 0.98 (s, 9 H). − ^{13}C NMR (50 MHz, $CDCl_3$): δ = 165.0, 157.0, 138.1, 135.7, 133.5, 133.1, 129.8, 129.7, 129.3, 129.3, 127.7, 127.5, 127.2, 70.3, 60.0, 59.2, 58.9, 26.7, 19.7, 19.0. − MS; *m*/*z*: 457 [M^+ − *t*Bu], 429, 414, 385, 341, 254. − $C_{30}H_{34}N_2O_4Si$ (514.6): calcd. C 70.01, H 6.66, N 5.44; found C 69.73, H 6.69, N 5.40.

Compound 5g (Identified as NH-Azetidinone): M.p. 165 °C. − $[\alpha]_D^{20}$ = +75.2 (*c* = 1.2, $CHCl_3$). − IR ($CHCl_3$): $\tilde{\nu}$ = 3435, 1772 cm^{-1}. − ^1H NMR (200 MHz, $CDCl_3$): δ = 7.60−7.25 (m, 15 H), 5.72 (s, 1 H), 4.86 (dd, 1 H, *J* = 7.2, 8.6 Hz), 4.66 (t, 1 H, *J* = 8.6 Hz), 4.19 (dd, 1 H, *J* = 7.2, 8.6 Hz), 4.04 (m, 2 H), 3.56 (m, 1 H), 0.90 (s, 9 H), 0.75 (d, 3 H, *J* = 6.3 Hz). − ^{13}C NMR (50 MHz, $CDCl_3$): δ = 164.9, 157.1, 137.1, 135.7, 135.8, 135.5, 133.4, 133.4, 130.0, 123.0, 129.6, 129.4, 127.9, 127.7, 127.5, 70.4. 70.3, 61.8, 61.6, 59.1, 26.8, 19.8, 19.2. − MS; *m*/*z*: 457 [M^+ − *t*Bu], 429, 414, 385, 341, 254. − $C_{30}H_{34}N_2O_4Si$ (514.6): calcd. C 70.01, H 6.66, N 5.44; found C 79.76, H 6.62, N 5.50.

(4*S*)-3-{(3*S*,4*R*)-2-Oxo-4-[(*S*)-1-(triethylsilyloxy)ethyl]-1-(trimethylsilyl)azetidine-3-yl}-4-phenyloxazolidin-2-one (4h) and (4*S*)-3-{(3*R*,4*S*)-2-Oxo-4-[(*S*)-1-(triethylsilyloxy)ethyl]-1-(trimethylsilyl)-azetidine-3-yl}-4-phenyloxazolidin-2-one (5h): The title compounds were prepared from the acyl chloride **2-X**[1] and the imine **1h** following the above procedure. Yields: 0.030 g of **4h** and 0.013 g of **5h** (11% total yield). Diastereomeric ratio 70:30.

Compound 4h (Identified as NH-Azetidinone): M.p. 160 °C. − $[\alpha]_D^{20}$ = +40.2 (*c* = 1.0, $CHCl_3$). − IR ($CHCl_3$): $\tilde{\nu}$ = 3425, 1756 cm^{-1}. − ^1H NMR (200 MHz, $CDCl_3$): δ = 7.38 (s, 5 H), 5.45 (s, 1 H), 4.96 (dd, 1 H, *J* = 6.5, 8.8 Hz), 4.90 (d, 1 H, *J* = 2.5 Hz), 4.68 (t, 1 H, *J* = 8.8 Hz), 4.20 (dd, 1 H, *J* = 6.50, 8.8 Hz), 3.78 (dq, 1 H, *J* = 2.5, 6.3 Hz), 2.98 (t, 1 H, *J* = 2.5 Hz), 1.08 (d, 3 H, *J* = 6.3 Hz), 0.80 (t, 9 H, *J* = 7.1 Hz), 0.46 (q, 6 H, *J* = 7.1 Hz).

– ^{13}C NMR (50 MHz, $CDCl_3$): δ = 165.1, 157.2. 138.5, 125.5, 129.4, 127.4, 70.5, 66.1, 60.5, 59.4, 59.1, 18.8, 6.7, 4.9. – MS; *m*/*z*: 390 [M^+], 361 [M^+ – Et], 332, 318. – $C_{20}H_{30}N_2O_4Si$ (390.5): calcd. C 61.51, H 7.74, N 7.17; found C 61.35, H 7.68, N 6.90.

Compound 5h (Identified as NH-Azetidinone): M.p. 130 °C. – $[\alpha]_D^{20}$ = +60.0 (*c* = 0.8, $CHCl_3$). – IR ($CHCl_3$): $\tilde{\nu}$ = 3421, 1753 cm^{-1}. – ^{1}H NMR (200 MHz, $CDCl_3$): δ = 7.40 (s, 5 H), 5.85 (s, 1 H), 4.92 (dd, 1 H, *J* = 7.1, 8.8 Hz), 4.68 (t, 1 H, *J* = 8.8 Hz), 4.20 (dd, 1 H, *J* = 7.1, 8.8 Hz), 4.04 (d, 1 H, *J* = 2.6 Hz), 3.98 (dd, 1 H, *J* = 2.6, 5.3 Hz), 3.55 (m, 1 H), 0.95 (d, 3 H, *J* = 6.2 Hz), 0.85 (t, 9 H, *J* = 7.1 Hz), 0.42 (q, 6 H, *J* = 7.1 Hz). – MS; *m*/*z*: 390 [M^+], 361 [M^+ – Et], 332, 318.

(4*S*)-3-{(3*S*,4*R*)-4-[(*S*)-1-*tert*-Butoxyethyl]-2-oxo-1-(trimethylsilyl)-azetidine-3-yl}-4-phenyloxazolidin-2-one (4i) and (4*S*)-3-{(3*R*,4*S*)-4-[(*S*)-1-*tert*-Butoxyethyl]-2-oxo-1-(trimethylsilyl)azetidine-3-yl}-4-phenyloxazolidin-2-one (5i): The title compounds were prepared from the acyl chloride **2-X[1]** and the imine **1i** following the above procedure. Yields: 0.123 g of **4i** and 0.045 g of **5i** (63% total yield). Diastereomeric ratio 73:27.

Compound 4i (Identified as NH-Azetidinone): M.p. 180 °C. $[\alpha]_D^{20}$ = +66.1 (*c* = 0.6, $CHCl_3$). – IR ($CHCl_3$): $\tilde{\nu}$ = 3425, 1760 cm^{-1}. – ^{1}H NMR (200 MHz, $CDCl_3$): δ = 7.40 (s, 5 H), 5.66 (s, 1 H), 4.98 (dd, 1 H, *J* = 6.3, 8.8 Hz), 4.84 (d, 1 H, *J* = 2.4 Hz), 4.69 (t, 1 H, *J* = 8.8 Hz), 4.22 (dd, 1 H, *J* = 6.3, 8.8 Hz), 3.60 (dq, 1 H, *J* = 3.2, 6.3 Hz), 3.00 (dd, 1 H, *J* = 3.2, 2.4 Hz), 1.06 (m, 12 H). – ^{13}C NMR (50 MHz, $CDCl_3$): δ = 165.2, 157.2, 138.5, 129.4, 129.4, 127.3, 73.8, 70.5, 65.0, 61.2, 59.0, 58.7, 28.3, 18.9. – MS; *m*/*z*: 275 [M^+ – *t*Bu], 218, 190, 146, 104. – $C_{18}H_{24}N_2O_4$ (332.4): calcd. C 65.04, H 7.28, N 8.43; found C 65.01, H 7.24, N 8.50.

Compound 5i (Identified as NH-Azetidinone): M.p. 162 °C. – $[\alpha]_D^{20}$ = +89.0 (*c* = 0.8, $CHCl_3$). – IR ($CHCl_3$): $\tilde{\nu}$ = 3435, 1760 cm^{-1}. – ^{1}H NMR (200 MHz, $CDCl_3$): δ = 7.38 (s, 5 H), 5.82 (s, 1 H), 4.92 (dd, 1 H, *J* = 7.6, 8.8 Hz), 4.67 (t, 1 H, *J* = 8.8 Hz), 4.20 (dd, 1 H, *J* = 7.6, 8.8 Hz), 4.12 (d, 1 H, *J* = 2.7 Hz), 3.96 (dd, 1 H, *J* = 2.7, 6.4 Hz), 3.40 (m, 1 H), 1.00 (s, 9 H), 0.93 (d, 3 H, *J* = 6.2 Hz). – ^{13}C NMR (50 MHz, $CDCl_3$): δ = 165.1, 157.2, 136.9, 129.7, 129.4, 127.7, 73.9, 70.4, 68.0, 62.0, 61.5, 57.9, 28.6, 19.6. – MS; *m*/*z*: 274 [M^+ – *t*Bu], 218, 190, 146, 104. – $C_{18}H_{24}N_2O_4$ (332.4): calcd. C 65.04, H 7.28, N 8.43; found C 64.76, H 7.24, N 8.39.

(4*S*)-3-{(3*S*,4*R*)-1-(*tert*-Butyldimethylsilyl)-2-oxo-4-[(*S*)-1-(triisopropylsilyloxy)ethyl]azetidine-3-yl}-4-phenyloxazolidin-2-one (4k) and (4*S*)-3-{(3*R*,4*S*)-1-(*tert*-Butyldimethylsilyl)-2-oxo-4-[(*S*)-1-(triisopropylsilyloxy)ethyl]azetidine-3-yl}-4-phenyloxazolidin-2-one (5k): The title compounds were prepared from the acyl chloride **2-X[1]** and the imine **1k** following the above procedure. Yields: 0.188 g of **4k** and 0.105 g of **5k** (54% total yield). Diastereomeric ratio 64:36.

Compound 4k: M.p. 130 °C. – $[\alpha]_D^{20}$ = +58.2 (*c* = 2.1, $CHCl_3$). – IR ($CHCl_3$): $\tilde{\nu}$ =1750, 1741 cm^{-1}. – ^{1}H NMR (200 MHz, $CDCl_3$): δ = 7.35 (s, 5 H), 4.97 (d, 1 H, *J* = 3.1 Hz), 4.93 (dd, 1 H, *J* = 6.7, 8.8 Hz), 4.65 (t, 1 H, *J* = 8.8 Hz), 4.14 (m, 1 H), 4.08 (dd, 1 H, *J* = 6.7, 8.8 Hz), 3.68 (dd, 1 H, *J* = 1.2, 3.1 Hz), 1.18 (d, 3 H, *J* = 6.3 Hz), 0.98 (s, 21 H), 0.84 (s, 9 H), 0.18 (s, 3 H), 0.07 (s, 3 H). – ^{13}C NMR (50 MHz, $CDCl_3$): δ = 170.4, 157.4, 138.5, 129.4, 129.3, 127.1, 70.9, 66.8, 62.3, 59.7, 59.3, 26.1, 20.6, 18.9, 18.3, 13.0, –5.48. – MS; *m*/*z*: 503 [M^+ – *i*Pr], 328, 149. – $C_{29}H_{50}N_2O_4Si_2$ (546.9): calcd. C 63.69, H 9.22, N 5.12; found C 63.40, H 9.17, N 5.14.

Compound 5k: M.p. 89 °C. – $[\alpha]_D^{20}$ = +88.6 (*c* = 2.2, $CHCl_3$). – IR ($CHCl_3$): $\tilde{\nu}$ = 1761, 1748 cm^{-1}. – ^{1}H NMR (200 MHz, $CDCl_3$): δ = 7.40 (s, 5 H), 5.03 (dd, 1 H, *J* = 8.5, 9.4 Hz), 4.58 (t, 1 H, *J* = 8.5 Hz), 4.42 (m, 2 H), 4.18 (dq, 1 H, *J* = 3.1, 6.3 Hz), 3.98 (dd, 1 H, *J* = 8.5, 9.4 Hz), 1.05 (m, 24 H), 0.85 (s, 9 H), 0.18 (s, 3 H), 0.08 (s, 3 H). – ^{13}C NMR (50 MHz, $CDCl_3$): δ = 171.9, 157.5, 136.0, 129.3, 127.4, 70.4, 67.1, 60.10, 59.7, 58.0, 26.0, 18.6, 18.1, 18.0, 16.9, 12.4, –5.5, –5.6. – MS; *m*/*z*: 503 [M^+ – *i*Pr], 149. – $C_{29}H_{50}N_2O_4Si_2$ (546.9): calcd. C 63.69, H 9.22, N 5.12; found C 63.90, H 9.27, N 5.10.

(4*S*)-3-{(3*S*,4*R*)-1-(*tert*-Butyldiphenylsilyl)-2-oxo-4-[(*S*)-1-(triisopropylsilyloxy)ethyl]azetidine-3-yl}-4-phenyloxazolidin-2-one (4j) and (4*S*)-3-{(3*R*,4*S*)-1-(*tert*-Butyldiphenylsilyl)-2-oxo-4-[(*S*)-1-(triisopropylsilyloxy)ethyl]azetidine-3-yl}-4-phenyloxazolidin-2-one (5j): The title compounds were prepared from the acyl chloride **2-X[1]** and the imine **1j** following the above procedure. Yields: 0.253 g of **4j** and 0.176 g of **5j** (64% total yield). Diastereomeric ratio 59:41.

Compound 4j: M.p. 127 °C. – $[\alpha]_D^{20}$ = +74.8 (*c* = 0.9, $CHCl_3$). – IR ($CHCl_3$): $\tilde{\nu}$ = 2971, 1752 cm^{-1}. – ^{1}H NMR (200 MHz, $CDCl_3$): δ = 7.5 (m, 4 H), 7.3 (m, 11 H), 5.15 (d, 1 H, *J* = 3.1 Hz), 4.90 (dd, 1 H, *J* = 6.6, 8.8 Hz), 4.67 (t, 1 H, *J* = 8.7 Hz), 4.10 (dd, 1 H, *J* = 6.6, 8.6 Hz), 3.36 (d, 1 H, *J* = 3.1 Hz), 3.00 (q, 1 H, *J* = 6.5 Hz), 1.12 (s, 9 H), 0.78 (s, 21 H), 0.68 (d, 3 H, *J* = 6.5 Hz). – ^{13}C NMR (50 MHz, $CDCl_3$): δ = 171.4, 157.3, 138.3, 136.1, 135.8, 131.7, 131.2, 130.1, 129.9, 129.3, 129.1, 127.8, 127.7, 127.2, 70.8, 66.3, 62.4, 59.8, 55.3, 27.7, 21.5, 19.7, 18.1, 18.0, 13.2. – MS; *m*/*z*: 628 [M^+ – *i*Pr], 346, 294. – $C_{39}H_{54}N_2O_4Si_2$ (671.0): calcd. C 69.81, H 8.11, N 4.17; found C 70.09, H 8.15, N 4.20.

Compound 5j: M.p. 133 °C. – $[\alpha]_D^{20}$ = +69.7 (*c* = 1.6, $CHCl_3$). – IR ($CHCl_3$): $\tilde{\nu}$ = 2975, 1740 cm^{-1}. – ^{1}H NMR (200 MHz, $CDCl_3$): δ = 7.65 (m, 4 H), 7.38 (m, 11 H), 5.10 (dd, 1 H, *J* = 8.5, 9.0 Hz), 4.59 (t, 1 H, *J* = 8.5 Hz), 4.52 (m, 2 H), 4.00 (dd 1 H, *J* = 8.5, 9.0 Hz), 3.10 (dq, 1 H, *J* = 2.9, 6.3 Hz), 1.05 (s, 9 H), 0.85 (d, 21 H), 0.70 (d, 3 H, *J* = 6.1 Hz). – ^{13}C NMR (50 MHz, $CDCl_3$): δ = 172.4, 157.0, 136.1, 135.9, 135.7, 131.9, 131.3, 130.2, 130.0, 129.3, 128.14, 127.8, 127.3, 70.4, 66.2, 62.0, 59.2, 57.8, 27.7, 19.5, 17.9, 17.9, 16.6, 12.0. – MS; *m*/*z*: 628 [M^+ – *i*Pr], 346, 294. – $C_{39}H_{54}N_2O_4Si_2$ (671.0): calcd. C 69.81, H 8.11, N 4.17; found C 69.54, H 8.13, N 4.21.

(4*S*)-3-{(3*S*,4*R*)-2-Oxo-4-[(*S*)-1-(triisopropylsilyloxy)ethyl]-1-(triisopropylsilyl)azetidine-3-yl}-4-phenyloxazolidin-2-one (4l) and (4*S*)-3-{(3*R*,4*S*)-2-Oxo-4-[(*S*)-1-(triisopropylsilyloxy)ethyl]-1-(triisopropylsilyl)azetidine-3-yl}-4-phenyloxazolidin-2-one (5l): The title compounds were prepared from the acyl chloride **2-X[1]** and the imine **1l** following the above procedure. Yields: 0.142 g of **4l** and 0.116 g of **5l** (44% total yield). Diastereomeric ratio 55:45.

Compound 4l: M.p. 119 °C. – $[\alpha]_D^{20}$ = +56.5 (*c* = 0.9, $CHCl_3$). – IR ($CHCl_3$): $\tilde{\nu}$ = 1748, 1735 cm^{-1}. – ^{1}H NMR (200 MHz, $CDCl_3$): δ = 7.35 (s, 5 H), 5.05 (d, 1 H, *J* = 3.1 Hz), 4.92 (dd, 1 H, *J* = 6.3, 8.5 Hz) 4.66 (t, 1 H, *J* = 8.5 Hz) 4.18 (dd, 1 H, *J* = 1.0, 6.4 Hz), 4.06 (dd, 1 H, *J* = 6.3, 8.5 Hz), 3.54 (dd, 1 H, *J* = 1.0, 3.1 Hz), 1.20 (d, 3 H, *J* = 6.4 Hz), 1.05 (m, 42 H). – ^{13}C NMR (50 MHz, $CDCl_3$): δ = 171.0, 157.5, 138.6, 129.3, 129.2, 126.9, 70.9, 67.4, 62.1, 59.4, 21.2, 18.3, 18.2, 13.3, 12.2. – MS; *m*/*z*: 545 [M^+ – *i*Pr], 346, 294. – $C_{32}H_{56}N_2O_4Si_2$ (588.9): calcd. C 65.26, H 9.58, N 4.76; found C 65.51, H 9.53, N 4.79.

Compound 5l: M.p. 85 °C. $[\alpha]_D^{20}$ = +87.7 (*c* = 1.2, $CHCl_3$). – IR ($CHCl_3$): $\tilde{\nu}$ = 1760, 1740 cm^{-1}. – ^{1}H NMR (200 MHz, $CDCl_3$): δ = 7.40 (s, 5 H), 5.06 (dd, 1 H, *J* = 8.5, 9.4 Hz), 4.63 (t, 1 H, *J* = 3.1 Hz), 4.58 (t, 1 H, *J* = 8.5 Hz), 4.28 (d, 1 H, *J* = 3.3 Hz), 4.20

(dq, 1 H, J = 3.0, 6.2 Hz), 3.97 (dd, 1 H, J = 8.5, 9.4 Hz), 1.08 (m, 45 H). − ^{13}C NMR (50 MHz, $CDCl_3$): δ = 172.6, 157.5, 136.0, 129.3, 129.2, 127.4, 70.4, 67.3, 62.3, 59.1, 58.1, 18.1, 18.0, 17.9, 17.2, 12.4, 12.2. − MS; *m/z*: 545 [M^+ − *i*Pr], 346, 294. − $C_{32}H_{56}N_2O_4Si_2$ (588.9): calcd. C 65.26, H 9.58, N 4.76; found C 64.95, H 9.62, N 4.75.

(4*S*)-3-{(3*S*,4*R*)-4-[(*S*)-1-(*tert*-Butyldimethylsilyloxy)propyl-1-yl]-2-oxo-1-(trimethylsilyl)azetidine-3-yl}-4-phenyloxazolidin-2-one (4m) and (4*S*)-3-{(3*R*,4*S*)-4-[(*S*)-1-(*tert*-Butyldimethylsilyloxy)-2-oxopropyl-1-yl]-1-(trimethylsilyl)azetidine-3-yl}-4-phenyloxazolidin-2-one (5m): The title compounds were prepared from the acyl chloride **2-X[1]** and the imine **1m** following the above procedure. Yields: 0.097 g of **4m** and 0.064 g of **5m** (40% total yield). Diastereomeric ratio 60:40.

Compound 4m (Identified as NH-Azetidinone): M.p. 190 °C. − $[\alpha]_D^{20}$ = +30.2 (c = 0.9, $CHCl_3$). − IR ($CHCl_3$): ṽ = 3435, 1768 cm^{-1}. − ^{1}H NMR (200 MHz, $CDCl_3$): δ = 7.42 (s, 5 H), 5.65 (s, 1 H), 4.96 (m, 2 H), 4.68 (t, 1 H, J = 8.8 Hz), 4.22 (dd, 1 H, J = 6.6, 8.8 Hz), 3.55 (dt, 1 H, J = 2.0, 6.1 Hz), 3.08 (t, 1 H, J = 2.2 Hz), 1.43 (m, 2 H), 0.80 (m, 12 H), −0.02 (s, 3 H), −0.08 (s, 3 H). − ^{13}C NMR (50 MHz, $CDCl_3$): δ = 165.3, 157.1, 138.4, 129.5, 129.4, 127.5, 71.1, 70.4, 60.2, 59.2, 57.5, 27.3, 25.7, 18.0, 9.6, −4.5, −4.7. − MS; *m/z*: 389 [M^+ − 15], 347 [M^+ − *t*Bu], 332, 319, 304, 261, 238. − $C_{21}H_{32}N_2O_4Si$ (404.6): calcd. C 62.34, H 7.97, N 6.92. Found C 62.66, H 8.01, N 6.85.

Compound 5m (Identified as NH-Azetidinone): Oil. − $[\alpha]_D^{20}$ = +16.2 (c = 0.9, $CHCl_3$). − IR ($CHCl_3$): ṽ = 3437, 1768 cm^{-1}. − ^{1}H NMR (200 MHz, $CDCl_3$): δ = 7.42 (s, 5 H), 6.65 (s, 1 H), 4.98 (dd, 1 H, J = 6.7, 8.9 Hz), 4.68 (t, 1 H, J = 8.9 Hz), 4.65 (d, 1 H, J = 2.5 Hz), 4.22 (dd, 1 H, J = 6.7, 8.9 Hz), 3.52 (q, 1 H, J = 6.6 Hz), 3.00 (dd, 1 H, J = 2.5, 6.6 Hz), 1.40 (m, 2 H), 0.78 (m, 12 H), 0.00 (s, 6 H). − ^{13}C NMR (50 MHz, $CDCl_3$): δ = 164.5, 157.3, 138.2, 129.6, 129.4, 127.4, 74.0, 70.5, 62.6, 59.0, 57.7, 26.8, 25.7, 18.0, 8.7, −4.3, −4.6. − MS; *m/z*: 347 [M^+ − *t*Bu], 332, 319, 304, 261, 238. − $C_{21}H_{32}N_2O_4Si$ (404.6): calcd. C 62.34 H, 7.97, N 6.92; found C 62.03, H 7.94, N 6.89.

(4*S*)-3-{(3*S*,4*R*)-2-Oxo-4-[(*S*)-(phenyltriisopropylsilyloxy)methyl]-1-(trimethylsilyl)azetidine-3-yl}-4-phenyloxazolidin-2-one (4n) and (4*S*)-3-{(3*R*,4*S*)-2-Oxo-4-[(*S*)-(phenyltriisopropylsilyloxy)methyl]-1-(trimethylsilyl)azetidine-3-yl}-4-phenyloxazolidin-2-one (5n): The title compounds were prepared from the acyl chloride **2-X[1]** and the imine **1n** following the above procedure. Yields: 0.225 g of **4n** and 0.056 g of **5n** (57% total yield). Diastereomeric ratio 80:20.

Compound 4n (Identified as NH-Azetidinone): M.p. 204−206 °C. − $[\alpha]_D^{20}$ = +72.0 (c = 0.5, $CHCl_3$). − IR ($CHCl_3$): ṽ = 3422, 1760 cm^{-1}. − ^{1}H NMR (200 MHz, $CDCl_3$): δ = 7.40−7.10 (m, 10 H), 6.32 (s, 1 H), 4.90 (d, 1 H, J = 2.5 Hz), 4.85 (d, 1 H, J = 2.2 Hz), 4.50 (m, 2 H), 3.98 (dd, 1 H, J = 4.6, 5.7 Hz), 3.42 (t, 1 H, J = 2.5 Hz), 0.85 (s, 21 H). − ^{13}C NMR (50 MHz, $CDCl_3$): δ = 165.25, 156.9, 140.3, 137.7, 129.4, 129.1, 128.3, 128.0, 127.0, 126.2, 73.0, 70.3, 59.7, 59.5, 59.0, 17.7, 12.1. − MS; *m/z*: 494 [M^+], 451 [M^+ − 43], 273, 248. − $C_{28}H_{38}N_2O_4Si$ (494.7): calcd. C 67.98, H 7.74, N 5.66; found C 67.71, H 7.70, N 5.73.

Compound 5n (Identified as NH-Azetidinone): M.p. 102−104 °C. − $[\alpha]_D^{20}$ = +84.0 (c = 0.5, $CHCl_3$). − IR ($CHCl_3$): ṽ = 3420, 1760 cm^{-1}. − ^{1}H NMR (200 MHz, $CDCl_3$): δ = 7.40−7.10 (m, 10 H), 5.80 (s, 1 H) 4.80 (dd, 1 H, J = 7.6, 8.6 Hz), 4.64 (d, 1 H, J = 5.1 Hz), 4.55 (t, 1 H, J = 8.6 Hz), 4.38 (dd, 1 H, J = 2.6, 5.1 Hz), 4.12 (d, 1 H, J = 2.6 Hz), 4.05 (dd, 1 H, J = 7.6, 8.6 Hz), 0.85 (s, 21 H). − ^{13}C NMR (50 MHz, $CDCl_3$): δ = 165.0, 157.3, 140.4, 136.5, 129.4, 128.6, 128.3, 127.3, 127.2, 126.3, 75.2, 70.3, 61.6, 61.2, 58.8, 17.9, 12.3. − MS; *m/z*: 494 [M^+], 451 [M^+ − 43], 273, 248. − $C_{28}H_{38}N_2O_4Si$ (494.7): calcd. C 67.98, H 7.74 N, 5.66; found C 68.24, H 7.76, N 5.63.

(4*S*)-3-{(3*S*,4*R*)-4-[(1*S*)-2-Methyl-1-(triisopropylsilyloxy)propyl]-2-oxo-1-(trimethylsilyl)azetidine-3-yl}-4-phenyloxazolidin-2-one (4o) and (4*S*)-3-{(3*R*,4*S*)-4-[(1*S*)-2-Methyl-1-(triisopropylsilyloxy)propyl]-2-oxo-1-(trimethylsilyl)azetidine-3-yl}-4-phenyloxazolidin-2-one (5o): The title compounds were prepared from the acyl chloride **2-X[1]** and the imine **1o** following the above procedure. Yields: 0.160 g of **4o** and 0.060 g of **5o** (48% total yield). Diastereomeric ratio 73:27

Compound 4o (Identified as NH-Azetidinone): M.p. 172−174 °C. − $[\alpha]_D^{20}$ = 32.3 (c = 1.2, $CHCl_3$). − IR ($CHCl_3$): ṽ = 3420, 1760 cm^{-1}. − ^{1}H NMR (200 MHz, $CDCl_3$): δ = 7.40 (s, 5 H), 5.85 (s, 1 H), 5.10 (d, 1 H, J = 2.2 Hz), 4.94 (dd, 1 H, J = 7.5, 8.8 Hz), 4.67 (t, 1 H, J = 8.8 Hz), 4.22 (dd, 1 h, J = 7.5, 8.8 Hz), 3.70 (dd, 1 H, J = 0.9, 3.7 Hz), 3.24 (dd, 1 H, J = 0.9, 2.2 Hz), 1.78 (m, 1 H), 0.95 (s, 21 H), 0.85 (d, 3 H, J = 7.0 Hz), 0.70 (d, 3 H, J = 7.1 Hz). − ^{13}C NMR (50 MHz, $CDCl_3$): δ = 165.9, 157.1, 138.0, 129.5, 75.2, 70.2, 60.4, 59.6, 54.6, 33.3, 18.7, 18.03, 16.3, 12.79. − MS; *m/z*: 417 [M^+ − 43], 389, 374, 317, 214. − $C_{25}H_{40}N_2O_4Si$ (460.7): calcd. C 65.18, H 8.75, N 6.08; found C 64.85, H 8; 79, N 6.19.

Compound 5o (Identified as NH-Azetidinone): M.p. 152−154 °C. − $[\alpha]_D^{20}$ = 65.1 (c = 1.0, $CHCl_3$). − IR ($CHCl_3$): ṽ = 3434, 1759 cm^{-1}. − ^{1}H NMR (200 MHz, $CDCl_3$): δ = 7.38 (m, 5 H), 5.70 (s, 1 H), 4.92 (dd, 1 H, J = 6.7, 8.7 Hz), 4.69 (t, 1 H, J = 8.7 Hz), 4.22 (dd, 1 H, J = 6.7, 8.7 Hz), 4.15 (dd, 1 H, J = 2.7, 3.5 Hz), 4.04 (d, 1 H, J = 2.7 Hz), 3.44 (t, 1 H, J = 3.2 Hz), 1.60 (m, 1 H), 0.91 (m, 24 H), 0.82 (d, 3 H, J = 7.1 Hz). − ^{13}C NMR (50 MHz, $CDCl_3$): δ = 165.2, 157.1, 137.5, 129.6, 129.4, 127.5, 76.0, 70.3, 63.0, 61.6, 54.4, 33.3, 18.1, 17.0, 12.9. − MS; *m/z*: 417 [M^+ − 43], 389, 374, 317, 214. − $C_{25}H_{40}N_2O_4Si$ (460.7): calcd. C 65.18, H 8.75, N 6.08; found C 65.40, H 8.71, N 6.07.

(4*S*)-3-{(3*S*,4*R*)-4-[(1*S*)-1-(*tert*-Butyldimethylsilyloxy)-2,2-dimethylpropyl]-2-oxo-1-(trimethylsilyl)azetidine-3-yl}-4-phenyloxazolidin-2-one (4p) and (4*S*)-3-{(3*R*,4*S*)-4-[(1*S*)--1-(*tert*-Butyldimethylsilyloxy)-2,2-dimethylpropyl]-2-oxo-1-(trimethylsilyl)azetidine-3-yl}-4-phenyloxazolidin-2-one (5p): The title compounds were prepared from the acyl chloride **2-X[1]** and the imine **1p** following the above procedure. Yields: 0.362 g of **4p** and 0.043 g of **5p** (84% total yield). Diastereomeric ratio 88:12.

Compound 4p (Identified as NH-Azetidinone): M.p. 92−94 °C. − $[\alpha]_D^{20}$ = +17.4 (c = 1.2, $CHCl_3$). − IR ($CHCl_3$): ṽ = 3419, 1759 cm^{-1}. − ^{1}H NMR (200 MHz, $CDCl_3$): δ = 7.42 (s, 5 H), 5.72 (s, 1 H), 4.98 (d, 1 H, J = 2.2 Hz), 4.92 (dd, 1 H, J = 7.8, 8.9 Hz), 4.67 (t, 1 H, J = 8.9 Hz), 4.23 (dd, 1 H, J = 7.8, 8.9 Hz), 3.50 (d, 1 H, J = 2.2 Hz), 3.29 (s, 1 H), 0.78 (s, 18 H), 0.96 (s, 3 H), 0.92 (s, 3 H). − ^{13}C NMR (50 MHz, $CDCl_3$): δ = 165.9, 157.2, 137.7, 129.6, 129.4, 127.8, 78.3, 70.3, 60.3, 59.9, 54.6, 34.9, 26.4, 26.0, 18.3, −3.5, −4.2. − MS; *m/z*: 432 [M^+], 417, 375, 332, 261. − $C_{23}H_{36}N_2O_4Si$ (432.6): calcd. C 63.85, H 8.39, N 6.48; found C 63.80; H. 8.41; N.6.50.

Compound 5p (Identified as NH-Azetidinone): M.p. 122−124 °C. − $[\alpha]_D^{20}$ = +16.1 (c = 1.7, $CHCl_3$). − IR ($CHCl_3$): ṽ = 3438, 1759 cm^{-1}. − ^{1}H NMR (200 MHz, $CDCl_3$): δ = 7.42 (s, 5 H), 5.50 (s, 1 H), 4.98 (dd, 1 H, J = 7.0, 8.8 Hz), 4.69 (m, 2 H), 4.26 (dd, 1 H, J = 7.0, 8.8 Hz), 3.27 (d, 1 H, J = 6.8 Hz), 2.94 (dd, 1 H, J = 2.4, 6.8 Hz), 0.82 (s, 9 H), 0.70 (s, 9 H), 0.05 (s, 6 H). − ^{13}C NMR (50 MHz, $CDCl_3$): δ = 164.3, 157.4, 138.1, 129.8, 129.4, 127.7,

80.5, 70.3, 65.2, 58.9, 55.1, 35.0, 26.0, 25.9, 18.4, −3.2, −3.8. − MS; *m/z*: 432 [M^+], 417, 375, 332, 261. − $C_{23}H_{36}N_2O_4Si$ (432.6): calcd. C 63.85, H 8.39, N 6.48; found C 64.12, H 8.43, N 6.46.

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