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Synthesis of imidazo[5,1-*b*]thiazoles or spiro-β-lactams by reaction of imines with mesoionic compounds or ketenes generated from *N*-acyl-thiazolidine-2-carboxylic acids

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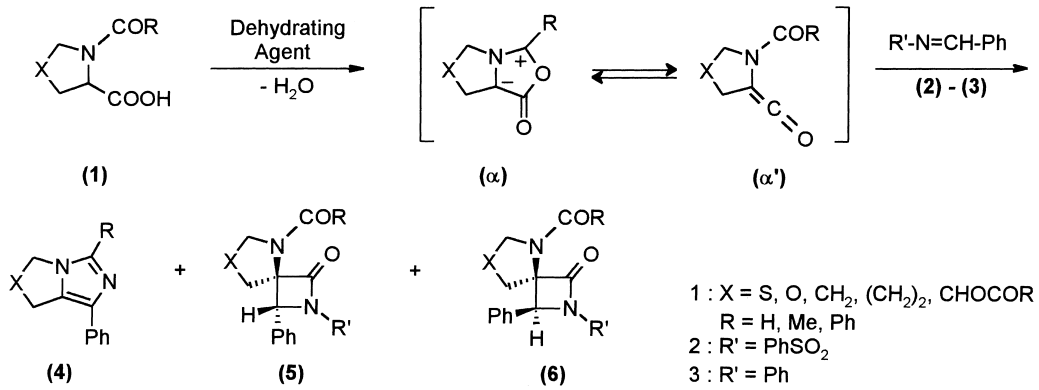
Abstract—New mesoionic compounds (2H, 3H-thiazolo[3,2-*c*]oxazol-7-ones) (β) or ketenes ((3-acyl-1,3-thiazolidin-2-ylidene)methanone) (β′) were generated from *N*-acetyl and *N*-benzoyl-thiazolidine-2-carboxylic acids (**7a**,**b**) using different methods, and their reactivity towards *N*-(phenylmethylene)benzenesulfonamide (**2**) and *N*-(phenylmethylene)aniline (**3**) was tested. When (**7a**,**b**) were treated with (**2**) and acetic anhydride in refluxing toluene solution, only imidazo[5,1-*b*]thiazoles (**8a**,**b**) were obtained from the mesoionic compound intermediates (β). When the ketene intermediates (β′) were generated from (**7a**,**b**) by means of Mukaiyama's reagent, only spiro-β-lactams (**9a**,**b**) were isolated.

1. Introduction

In previous communications, we have reported on the reactivity of bicyclic mesoionic compounds (α) derived from cyclic *N*-acyl-α-amino acids as (*R*)-thiazolidine-4-carboxylic acids, (*S*)-oxazolidine-4-carboxylic acid, (*S*)-prolines, (*R*,*S*)-pipecolinic acids and (2*S*,4*R*)-4-acyloxy-prolines (**1**) (Scheme 1).[1–3] These substrates were cyclo-dehydrated with *N*,*N*′-dicyclohexylcarbodiimide[1] or acetic anhydride[2,3] to the mesoionic compounds (münchnones intermediates) (α) in equilibrium with their ketene valence tautomers (α′). The cycloaddition reactions of (α) and (α′) with *N*-(phenylmethylene)benzenesulfonamide (**2**) and *N*-(phenylmethylene)aniline (**3**) afforded mixtures of imidazole-condensed products (**4**) and diastereoisomeric spiro-β-lactams (**5**)/(**6**), with ratios depending on the nature of R′ ($PhSO_2$ or Ph) and the experimental conditions.

In connection with these results, and as an extension of our studies of the reactivity of new bicyclic mesoionic compounds and their usefulness in the synthesis of condensed heterocycles,[4] we now report on the reactivity



Scheme 1.

Keywords: Mesoionic compounds; Ketenes; Imines; β-Lactams; Mukaiyama's reagent.
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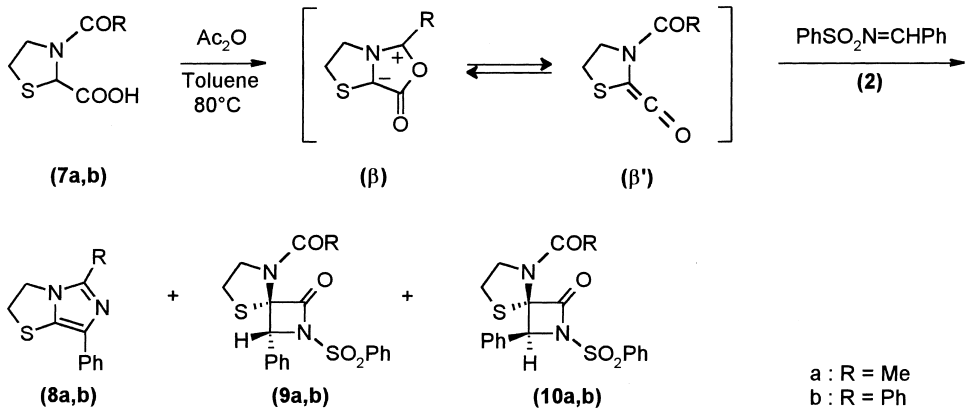


Scheme 2.

of the mesoionic compounds (β) and ketenes (β′) derived from *N*-acetyl and *N*-benzoyl-thiazolidine-2-carboxylic acids (**7a**,**b**) to imines (**2**) and (**3**).

Our interest in compounds (**7a**,**b**) is based on the position of the sulphur atom in relation to the carboxylic group, which is different from that in the previously studied substrates (**1**, X=S): this could modify the reactivity of the corresponding mesoionic or ketene intermediates (β) or (β′) as a result of either electronic or steric factors.

To the best of our knowledge, compounds (**7a**,**b**) have been used as precursors of mesoionic compounds in only one previous study, in which they were reacted with dimethyl-acetylenedicarboxylate in acetic anhydride to afford 2,3-dihydropyrrolo[2,1-*b*]thiazole derivatives.[5]

The 1,3-dipolar cycloaddition reactions between imine (**2**) and the bicyclic mesoionic compounds (β) could produce new condensed bicyclic heterocycles (imidazo[5,1-*b*]thiazoles) that have never previously been prepared by means of 1,3-dipolar cycloaddition reactions.

Another important aim of the study was to obtain only spiro-β-lactams by finding the best experimental conditions for generating only ketenes (β′) from compounds (**7a**,**b**). In our previous experiments using *N*,*N*′-dicyclohexylcarbodiimide or acetic anhydride (specific means of generating mesoionic compounds), spiro-β-lactams were always obtained mixed with (**4**) or, in the cases in which (**4**) did not form, in poor yields. In that case, the formation of spiro-β-lactams such as (**5**)/(**6**) depended on the tautomeric equilibrium between the mesoionic and ketene compounds, and therefore on the stability of the bicyclic mesoionic compounds.

Interest in the synthesis of β-lactams has been renewed by recent discoveries of their activity as inhibitors of cholesterol absorption[6] (in particular those with a spiranic structure[6d]) or thrombin.[7] These heterocycles have also been found to be useful synthons in the synthesis of new polyfunctionalised compounds, as we have demonstrated with our spiro-β-lactams.[8]

2. Results and discussion

The reaction of (**7a**,**b**) with imine (**2**) was run in toluene solution at 80 °C for 28 h with acetic anhydride as the dehydrating agent. This led to a mixture of the imidazo[5,1-*b*]thiazoles (**8a**,**b**) and the diastereoisomeric spiro-β-lactams (**9a**,**b**) and (**10a**,**b**) (Scheme 2), which were separated by means of column chromatography; their structures were confirmed on the basis of analytical and spectroscopic data.

The regiochemistry of compound (**8a**) was confirmed by means of direct comparison with the other theoretical regioisomer called 2,3-dihydro-5-methyl-6-phenyl-imidazo[2,1-*b*]thiazole, which was prepared as previously described.[9]

The relative configuration was assigned to spiro-β-lactams (**9**) and (**10**) on the basis of ^{1}H NMR experiments. In the case of the benzylic proton, the 300 MHz ^{1}H NMR spectra of products (**9a**,**b**) showed a signal with a chemical shift that was 0.5 ppm lower than that of the corresponding diastereoisomers (**10a**,**b**) because of the deshielding effect of the *N*-acyl carbonyl group.

Bicyclic compounds (**8a**,**b**) derive from a typical regio-selective 1,3-dipolar cycloaddition of the mesoionic intermediate (β) to the C=N double bond of imine (**2**) with the subsequent loss of carbon dioxide and benzenesulfinic acid, whereas spiro-β-lactams (**9a**–**b**–**10a**–**b**) are the result of a Staudinger reaction between the imine (**2**) and the ketene intermediate (β′).[10]

As shown in Table 1, the total yields of compounds (**8a**–**b**–**10a**–**b**) were always better than those of the corresponding **4**–**6** (X=S, R′=$PhSO_2$); furthermore, when R=Me, the reaction also affords the bicyclic compound (**8a**).

These results can be explained as being due to the increased stability of mesoionic compounds (β) with respect to (α) as a consequence of the different position of the sulphur atom

Table 1.

Products yield (%) (Ac_2O, toluene, 80 °C)

	(**8**)	(**9**)	(**10**)	(**4**)	(**5**)	(**6**)	(X=S, R′=$PhSO_2$)
R=Me	16	16	4	0	10	6	
R=Ph	29	24	4	22	12	5	

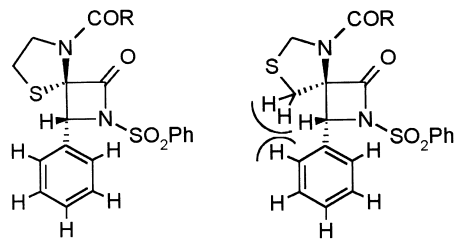


Figure 1.

which, in (β), is adjacent to the negative charge. It is well known that the presence of a sulphur atom increases the acidity of the adjacent CH bonds, an effect that has been attributed to the stabilisation of the carbanion by the sulphur atom.[11] This stabilisation could make up for the cyclisation difficulty of these bicyclic mesoionic compounds.[2]

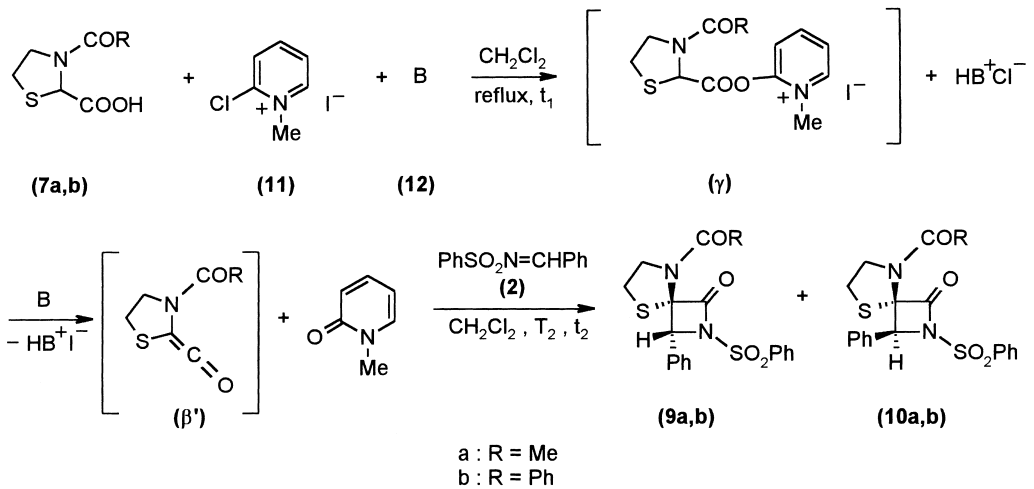
Another difference is the ratio of the diastereoisomeric spiro-β-lactams, which is about 2.5 times in favour of the thermodynamically more stable β-lactam (**9a**,**b**) (3-C phenyl and *N*-acyl groups *trans* to each other). This major diastereoselectivity could also be ascribed to the presence of sulphur, which could favour the formation of products (**9a**,**b**) probably as a result of steric factors: the molecular models show less steric encumbrance between the 3-C phenyl group and the 8-S atom in compounds (**9a**,**b**) in comparison with the same 3-C phenyl group and the 8-CH_2 group in compounds (**5**) (X=S, R′=$PhSO_2$) (Fig. 1).

In order to obtain only imidazo[5,1-*b*]thiazoles (**8a**,**b**), the reactions of (**7a**,**b**) and (**2**) were carried out in boiling toluene for 24 h in the presence of Ac_2O: under these conditions, compounds (**8a**) and (**8b**) were obtained in, respectively, 30 and 50% yields.

On the contrary, it was more difficult to find good conditions for obtaining exclusively spiro-β-lactams. Various methods were tried in order to generate the ketenes (β′) selectively: for example, $SOCl_2$/TEA/toluene, *N*,*N*′-diisopropylcarbodiimide/TEA/THF and 1,1′-carbonyldiimidazole/DBU/THF were all unsuccessful.

With *N*,*N*-dimethylchlorosulfitemethaniminium chloride ($SOCl_2$–DMF), a dehydrating reagent that is also used to prepare β-lactams,[12] the reactions of (**7a**,**b**) with (**2**) led to poor yields (about 15%) of spiro-β-lactams (**9a**,**b**) when run in THF solution at 5–10 °C in the presence of TEA. Better results were obtained using the Mukaiyama reagent (2-chloro-1-methylpyridinium iodide)[13] (**11**) as an acid-activating agent in the presence of a base (**12**) in dichloromethane solution (Scheme 3).

We investigated the effects of different reagent ratios, bases, temperatures and times on reaction yields and stereoselectivity (Table 2). The reactions were run in two steps: first, the aminoacids were refluxed with the Mukaiyama reagent in the presence of 1 equiv. of the base for time t_1, and then the imine and 2 equiv. of the base were added and the reactions continued at temperature T_2 for time t_2.



Scheme 3.

Table 2.

Entry	(**7**)	Ratio (**7**):(**11**):(**12**):(**2**)	Base	t_1 (h)	T_2	t_2 (h)	Total yields (%)	(**9**):(**10**)
1	**7a**	1.0:1.0:2.0:1.0	TPA	1	Reflux	10	17	100:0
2	**7a**	1.0:2.4:6.0:1.0	TPA	1	Reflux	24	34	100:0
3	**7a**	1.5:1.8:4.5:1.0	TPA	1	Reflux	24	52	100:0
4	**7a**	1.8:2.1:4.5:1.0	TPA	1	Reflux	15	55	95:5
5	**7a**	1.8:2.1:5.4:1.0	TPA	10	Reflux	10	70	97:3
6	**7a**	1.8:2.1:5.4:1.0	TEA	10	rt	48	58	100:0
7	**7a**	1.8:2.1:5.4:1.0	TEA	10	Reflux	10	62	95:5
8	**7b**	1.8:2.1:5.4:1.0	TEA	10	rt	20	46	92:8
9	**7b**	1.8:2.1:5.4:1.0	TEA	10	Reflux	10	78	95:5
10	**7b**	1.8:2.1:5.4:1.0	TPA	10	Reflux	10	80	97:3

The best results were obtained using an acid/Mukaiyama reagent/base/imine reagent ratio of 1.8:2.1:5.4:1, with tripropylamine (TPA) as a base, and reflux temperature for 10 h before and after the addition of the imine (entries 5 and 10, Table 2).

In comparison with the preceding method (Table 1) the diastereoselectivity was greatly increased favouring the formation of the more stable spiro-β-lactams (**9a**,**b**). The yield and diastereoselectivity were affected by the reaction time and temperature: for example, at reflux temperature (entry 7, Table 2), spiro-β-lactams (**9a**) and (**10a**) were obtained with a 62% total yield and 95:5 ratio; at room temperature, the yield decreased to 58% but (**9a**) was the only product (entry 6, Table 2). The base also affected the yield and the diastereoselectivity of the reaction (entries 5 and 7, Table 2): in our case, TPA gave higher yields and better stereoselectivity than TEA.[13] A run conducted as in entry 7 but in 1,2-dichloroethane as solvent at 70 °C afforded compound (**9a**) alone, but with a yield of only 36%.

A similar trend was observed with (**7b**) (entries 8–10, Table 2); the bicyclic compound (**8b**) was also detected (about 3–5%), which is in line with the greater stabilisation of the mesoionic tautomer (β) by the phenyl group in comparison with the methyl group.

The same reactions of compounds (**7a**,**b**) with imine (**3**) conducted in toluene solution and acetic anhydride, or in dichloromethane solution and Mukaiyama reagent, led to only NMR-detectable traces of the corresponding spiro-β-lactams, with respectively acetanilide and 3-acetyl-thiazolidine-2-carboxylic acid phenylamide as the main products. Imine (**3**) is scarcely reactive in these kinds of reactions with bicyclic mesoionic compounds.[1]

3. Conclusion

In this study, we generated new mesoionic compounds (2*H*,3*H*-thiazolo[3,2-*c*]oxazol-7-ones) (β) and ketenes ((3-acyl-1,3-thiazolidin-2-ylidene)methanone) (β′) from *N*-acetyl and *N*-benzoyl-thiazolidine-2-carboxylic acids (**7a**,**b**). By reacting these intermediates with imine (**2**), we selectively obtained new imidazo[5,1-*b*]thiazoles and spiro-β-lactams in better yields than the similar mesoionic compounds (α, X=S), possibly because of the different position of the sulphur atom in the starting cyclic amino acid.

The effect of the different position of the sulphur atom on the reactivity of the spiro-β-lactams (**9a**–**b**–**10a**–**b**) is now under investigation.

4. Experimental

4.1. General

Melting points were measured using a Büchi apparatus and are uncorrected. The ^{1}H and ^{13}C NMR spectra were recorded using a Bruker AC 300 spectrometer. Chemical shifts (δ) are given in ppm in relation to TMS; the solvent was $CDCl_3$ unless otherwise specified. All of the coupling constants (*J*) are in Hertz. The MS spectra were determined using a VG Analytical 7070 EQ mass spectrometer with an attached VG analytical 11/250 data system. The IR spectra were determined using a Perkin–Elmer 1725X FT-IR spectrometer.

Compounds (**2**)[14] and (**7a**,**b**)[5] were prepared according to the reported methods. Mukaiyama's reagent (**11**) was obtained from commercial sources.

4.2. General procedure for the reactions of (7a,b) with (2) and acetic anhydride

Acetic anhydride (25 mmol) was added dropwise under nitrogen to a stirred solution of (**7a**) or (**7b**) (5 mmol) in anhydrous toluene (15 ml). The mixture was heated at 80 °C for 1 h, and then a solution of imine (**2**) (5 mmol) in toluene (10 ml) was added dropwise and the heating continued for 28 h. After evaporation of the solvent, the residue was taken up in dichloromethane (50 ml), and the solution was washed with 10% sodium bicarbonate (2×20 ml) and water. The organic phase was dried (Na_2SO_4) and the solvent evaporated off. The crude mixture was separated using column chromatography (silica gel, toluene/ethyl acetate: 95:5), and the products were recrystallised and identified by means of analytical and spectroscopic data. The relative yields are reported in Table 1.

4.2.1. 2,3-Dihydro-5-methyl-7-phenyl-imidazo[5,1-*b*]thiazole (8a). Colorless solid, mp 130–131 °C (Et_2O/EtOH); ^{1}H NMR δ 2.34 (3H, s, CH_3), 3.83 (2H, t, *J*=6.7 Hz, SCH_2), 4.00 (2H, t, *J*=6.7 Hz, NCH_2), 7.10–7.60 (5H, m, Ph). IR (cm^{-1}, Nujol) 1660, 1600, 1548, 1519, 770. ^{13}C NMR δ 13.5 (CH_3), 38.5 (SCH_2), 44.4 (NCH_2), 124.2–140.9 (5-C, 7-C, 8-C, C_{Ph}). Anal. calcd for $C_{12}H_{12}N_2S$: C, 66.63; H, 5.59; N, 12.95. Found: C, 66.62; H, 5.47; N, 12.85. MS (*m*/*z*) 216, 201, 188, 147, 121.

4.2.2. *cis*-5-Acetyl-3-phenyl-2-(phenylsulphonyl)-8-thia-2,5-diazaspiro[3.4]octan-1-one (9a). Colorless solid, mp 177–178 °C d. (iPrOH); ^{1}H NMR δ 2.05 (3H, s, CH_3), 2.59 (1H, m, SCH), 2.92 (1H, ddd, *J*=11.4, 7.6, 5.9 Hz, SCH), 3.60 (1H, ddd, *J*=11.4, 7.6, 5.9 Hz, NCH), 3.82 (1H, m, NCH), 5.52 (1H, s, CH), 7.30–7.90 (10H, m, Ph). ^{13}C NMR (DMSO) δ 23.2 (CH_3), 30.1 (SCH_2), 51.4 (NCH_2), 67.6 (CH), 84.6 ($C_{spir.}$), 127.3–136.6 (C_{Ph}), 164.8 (CO), 168.8 (CO). IR (cm^{-1}, Nujol) 1802 (lactam CO), 1657 (amide CO). Anal. calcd for $C_{19}H_{18}N_2O_4S_2$: C, 56.70; H, 4.50; N, 6.96. Found: C, 56.62; H, 4.47; N, 6.89. MS (*m*/*z*) 402, 245, 216.

4.2.3. *trans*-5-Acetyl-3-phenyl-2-(phenylsulphonyl)-8-thia-2,5-diazaspiro[3.4]octan-1-one (10a). Colorless solid, mp 160–161 °C d. (iPrOH); ^{1}H NMR δ 1.63 (3H, s, CH_3), 3.07 (2H, m, SCH_2), 3.27 (1H, m, NCH), 3.75 (1H, m, NCH), 5.10 (1H, s, CH), 7.00–8.00 (10H, m, Ph). ^{13}C NMR (DMSO) δ 28.5 (CH_3), 30.7 (SCH_2), 53.2 (NCH_2), 72.9 (CH), 80.9 ($C_{spir.}$), 124.8–142.2 (C_{Ph}), 163.1 (CO), 167.8 (CO). IR (cm^{-1}, Nujol) 1790 (lactam CO), 1668 (amide CO). Anal. calcd for $C_{19}H_{18}N_2O_4S_2$: C, 56.70; H, 4.50; N, 6.96. Found: C, 56.58; H, 4.36; N, 6.81. MS (*m*/*z*) 402, 245, 216.

4.2.4. 2,3-Dihydro-5,7-diphenyl-imidazo[5,1-*b*]thiazole (8b). Colorless solid, mp 155–156 °C (Et_2O); ^1H NMR δ 3.88 (2H, t, J=7.0 Hz, SCH_2), 4.34 (2H, t, J=7.0 Hz, NCH_2), 7.10–7.75 (10H, m, Ph). ^{13}C NMR (DMSO) δ 39.2 (SCH_2), 47.0 (NCH_2), 124.5–134.46 (5-C, 7-C, 8-C, C_{Ph}). IR (cm^{-1}, Nujol) 1602, 1540, 1446, 766. Anal. calcd for $C_{17}H_{14}N_2S$: C, 73.37; H, 5.07; N, 10.06. Found: C, 73.19; H, 4.92; N, 10.00. MS (*m*/*z*) 278, 250, 147, 121, 103.

4.2.5. *cis*-5-Benzoyl-3-phenyl-2-(phenylsulphonyl)-8-thia-2,5-diazaspiro[3.4]octan-1-one (9b). Colorless solid, mp 176–177 °C d. (toluene); ^1H NMR δ 2.60 (1H, ddd, J=11.6, 5.0, 2.0 Hz, SCH), 3.0 (1H, m, SCH), 3.67 (1H, m, NCH), 3.84 (1H, ddd, J=11.6, 5.0, 2.0 Hz, NCH), 5.64 (1H, s, CH), 7.20–8.00 (15H, m, Ph). ^{13}C NMR δ 30.7 (SCH_2), 54.6 (NCH_2), 66.7 (CH), 85.1 ($C_{spir.}$), 127.3–137.5 (C_{Ph}), 165.4 (CO), 169.2 (CO). IR (cm^{-1}, Nujol) 1794 (lactam CO), 1638 (amide CO). Anal. calcd for $C_{24}H_{20}N_2O_4S_2$: C, 62.05; H, 4.34; N, 6.03. Found: C, 62.10; H, 4.26; N, 5.94. MS (*m*/*z*) 464, 323, 281, 245.

4.2.6. *trans*-5-Benzoyl-3-phenyl-2-(phenylsulphonyl)-8-thia-2,5-diazaspiro[3.4]octan-1-one (10b). Colorless solid, mp 163–165 °C d. (toluene); ^1H NMR δ 2.85 (1H, ddd, J=11.0, 5.23, 1.7 Hz, SCH), 3.04 (1H, m, SCH), 3.3 (1H, m, NCH), 3.66 (1H, m, NCH), 5.18 (1H, s, CH), 6.70–8.10 (15H, m, Ph). ^{13}C NMR (DMSO) δ 31.7 (SCH_2), 54.2 (NCH_2), 73.8 (CH), 81.4 ($C_{spir.}$), 125.9–143.1 (C_{Ph}), 163.7 (CO), 168.4 (CO). IR (cm^{-1}, Nujol) 1808 (lactam CO), 1644 (amide CO). Anal. calcd for $C_{24}H_{20}N_2O_4S_2$: C, 62.05; H, 4.34; N, 6.03. Found: C, 61.98; H, 4.29; N, 5.91. MS (*m*/*z*) 464, 323, 281, 245.

4.3. General procedure for the reactions of (7a,b) with (2) and Mukaiyama's reagent

A suspension of (**7a**) or (**7b**) (1.8 mmol), 2-chloro-*N*-methylpyridinium iodide (**11**) (2.1 mmol) and tripropylamine (1.8 mmol) in anhydrous methylene chloride (25 ml) were heated at reflux temperature under a nitrogen atmosphere for 10 h. A solution of the imine (**2**) (1 mmol) in anhydrous dichloromethane (10 ml) and tripropylamine (3.6 mmol) were added and the reaction mixture was refluxed for other 10 h. After cooling, the solution was washed with water, 5% HCl aqueous solution, and then with water. The organic layer was dried over Na_2SO_4 and the solvent was removed under reduced pressure. The crude products were purified by column chromatography (silica gel, toluene/ethyl acetate, 95:5) and recrystallized as indicated above. The relative yields are reported in Table 2.

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