Synthesis of 3-Alkoxy/Aryloxy-β-lactams Using Diazoacetate Esters as Ketene Precursors Under Photoirradiation

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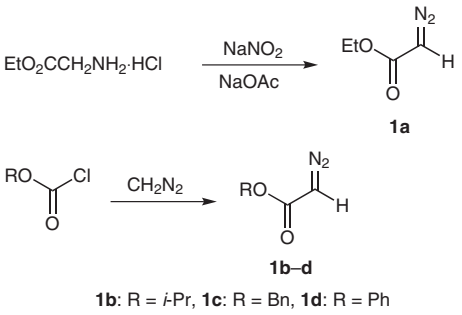
Abstract: 3-Alkoxy/aryloxy-β-lactams are synthesized in satisfactory to good yields from the reaction of imines and alkyl/aryl diazoacetates under photoirradiation conditions. Typically, *trans*-β-lactams are obtained as the major products from linear imines using the current method. By contrast, the corresponding thermal reaction of imines and alkoxy/aryloxyacetyl chlorides, or their equivalents, in the presence of triethylamine affords *cis*-β-lactams as the major or exclusive products. The formation of *trans*-β-lactams from linear imines is attributed to isomerization of the imines from their *trans*-isomers into *syn*-isomers under UV irradiation. The reported method represents a metal-free and neutral approach for the synthesis of 3-alkoxy/aryloxy-β-lactams.

Key words: diazoacetate, imine, ketene, β-lactam, photoirradiation, Staudinger reaction

Diazoacetate esters are important synthetic intermediates in organic chemistry.[1] They are used as carbene precursors for the cyclopropanation of olefins[1,2] and the aziridination of imines,[3] and for insertion reactions with various single bonds[1,4] (C–H, N–H, O–H, S–H, C–S, etc.), and accompany the Wolff rearrangement to generate ketenes, with subsequent nucleophilic addition to the ketenes in some cases.[1] They have also been utilized as ylide precursors in nucleophilic additions[1,5] and as 1,3-dipoles in [2+3] cycloadditions.[6] Although α-diazo ketones[7] and phenyl diazothioacetate[8] have been applied as ketene precursors for the synthesis of β-lactams, diazoacetates have seldom served as ketene precursors; examples include cycloaddition with alkenes to afford 2-alkoxy/aryloxycyclobutanones,[9] and one in which the cycloaddition of ethyl diazoacetate with a cyclic imine gave a 3-ethoxy-β-lactam derivative.[10] 3-Alkoxy/aryloxy-β-lactams have been prepared previously from the reaction of imines and alkoxy/aryloxyketenes, generated by elimination of the corresponding alkoxy/aryloxyacetyl chlorides in the presence of a tertiary amine as a base, or from alkoxy/aryloxyacetic acid with activating agents.[11] An alternative method for the generation of alkoxy/aryloxyketenes is via photolysis of chromium–alkoxy/aryloxycarbene complexes.[12] In the reported methods, *cis*-3-alkoxy/aryloxy-β-lactams were generally obtained as the major or exclusive products. However, *trans*-acetoxy-β-lactams are very important synthetic intermediates,[13] especially for the synthesis of antibiotics such as penem derivatives.[14] Additionally, recent reports have described their anticancer activity.[15] Although Brady and Dad,[16] as well as Banik and Becker,[17] have reported the synthesis of *trans*-3-alkoxy-β-lactams from the cycloddition of imines and alkoxyacetic acids or their chlorides, respectively, their methods were limited to the preparation of *trans*-3-alkoxy-β-lactams possessing *N*-polycyclic aromatic substituents. It is thus desirable to develop an efficient and direct method to synthesize *trans*-alkoxy/aryloxy-β-lactams. It has been reported that *trans*-3-alkyl/aryl-β-lactams were generated as the major or sole products from reactions with α-diazo ketones as the ketene precursors under photoirradiation.[7] In our recent investigation on the Staudinger reaction involving monosubstituted ketenes with electron-acceptor substituents,[18] we observed that ethoxycarbonylketene, generated from ethyl 3-diazo-2-oxopropanate, can lose a molecule of carbon monoxide to produce ethoxyketene, which further reacts with imines under photoirradiation conditions to give 3-alkoxy-β-lactams. This prompted us to attempt to use alkyl and aryl diazoacetates directly as ketene precursors to prepare *trans*-alkoxy/aryloxy-β-lactams under photoirradiation. Herein, we present the use of alkyl and aryl diazoacetates for the synthesis of alkoxy/aryloxy-β-lactams, in particular their *trans*-isomers.

Alkyl and aryl diazoacetates can be prepared conveniently from the corresponding glycine esters via diazotization with nitrous acid, or from the corresponding chloroformates using diazomethane. Ethyl diazoacetate (**1a**) was prepared from ethyl glycinate hydrochloride via diazotization with nitrous acid.[19] Isopropyl, benzyl, and phenyl diazoacetates **1b–d** were synthesized from the corresponding chloroformates and diazomethane[20] (Scheme 1).



Scheme 1 Preparation of alkyl/aryl diazoacetates **1a–d**

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Table 1 Reaction of Diazoacetates **1a–d** and Various Imines[a]

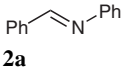
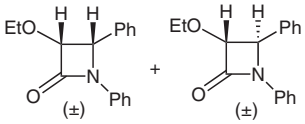
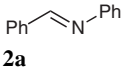
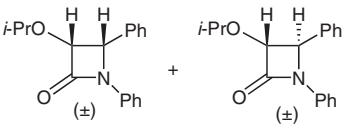
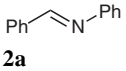
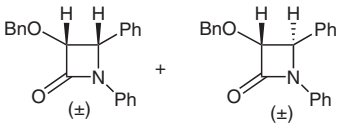
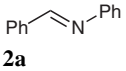
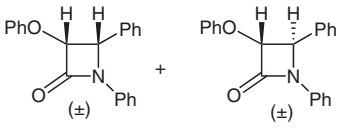
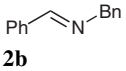
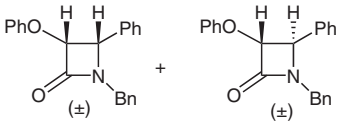
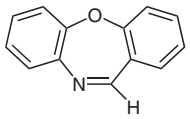
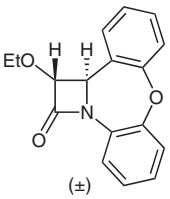
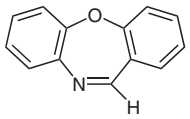
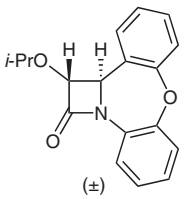
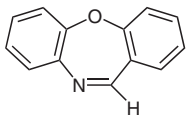
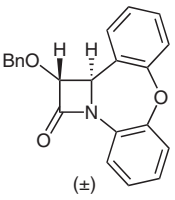
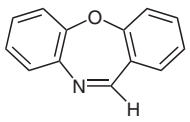
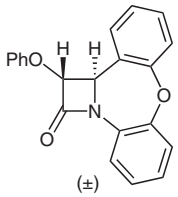
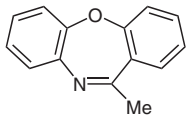
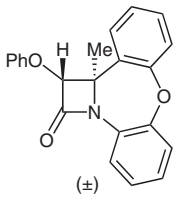
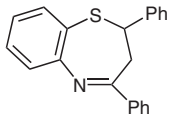
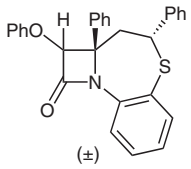
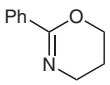
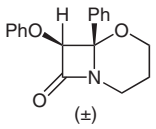
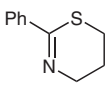
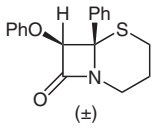
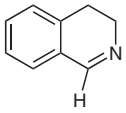
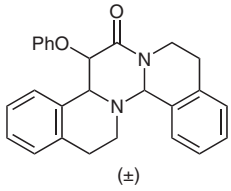
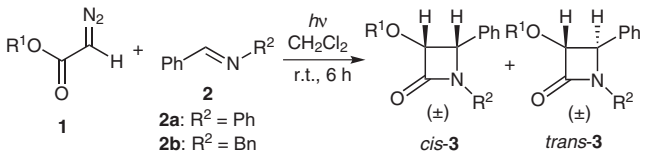
Entry	Diazoacetate	Imine	β-Lactam	Yield (%)[b]	*cis*:*trans*[c]
1	**1a**	 **2a**	 *cis*-**3aa** + *trans*-**3aa**	80	24:76
2	**1b**	 **2a**	 *cis*-**3ba** + *trans*-**3ba**	45	22:78
3	**1c**	 **2a**	 *cis*-**3ca** + *trans*-**3ca**	75	30:70
4	**1d**	 **2a**	 *cis*-**3da** + *trans*-**3da**	99	33:67
5	**1d**	 **2b**	 *cis*-**3db** + *trans*-**3db**	78	67:33
6	**1a**	 **4a**	 **5aa**	67	–
7	**1b**	 **4a**	 **5ba**	59	–
8	**1c**	 **4a**	 **5ca**	70	–

Table 1 Reaction of Diazoacetates **1a–d** and Various Imines[a] (continued)

Entry	Diazoacetate	Imine	β-Lactam	Yield (%)[b]	*cis*:*trans*[c]
9	**1d**	 **4a**	 **5da**	76	–
10	**1d**	 **4b**	 **5db**	62	–
11	**1d**	 **6**	 **7**	52	–
12	**1d**	 **8a**	 **9a**	32	–
13	**1d**	 **8b**	 **9b**	54	–
14	**1d**	 **10**	 **11**	78	–

[a] Reaction conditions: diazoacetate (2 mmol), imine (1 mmol), CH_2Cl_2 (10 mL), UV irradiation, r.t., N_2 atm, 6 h.
[b] Yield of isolated product (combined yield of *cis* and *trans* products).
[c] Ratio determined by ^1H NMR spectral analysis of the reaction mixture.

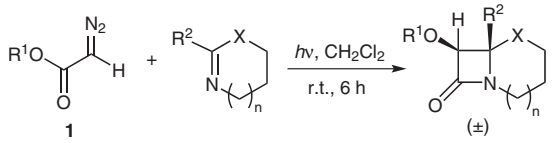
We initially optimized the conditions for the preparation of the alkoxy/aryloxy-β-lactams using the reaction of phenyl diazoacetate (**1d**) with benzylideneaniline (**2a**) as a model reaction, in anhydrous solvents, under UV photoirradiation at room temperature (Scheme 2). The desired products were obtained in good to excellent yields in dichloromethane, chloroform, diethyl ether and THF, and in low yields in benzene, toluene and xylene. Dichloromethane was the best choice as solvent affording a 99% yield of β-lactam **3** (Table 1, entry 4). Different reaction temperatures and modes of reagent addition (including intermittent, stepwise, and one-pot addition) did not have a significant impact on the yield. Of the diazoacetates **1a–d**, phenyl diazoacetate (**1d**) produced the desired products in the highest yields, but also with the lowest diastereoselectivity (Table 1, entry 4), whereas isopropyl diazoacetate (**1b**) gave the lowest yield of β-lactam product, but with the highest diastereoselectivity (Table 1, entry 2). The different diazoacetates did not have any significant influence on the diastereoselectivity, the *cis*:*trans* ratios varied from 33:67 (phenyl) to 22:78 (isopropyl). Phenyl diazoacetate (**1d**) also reacted with benzylidene benzylamine (**2b**) to afford *cis*- and *trans*-phenoxy-β-lactams **3db** in a ratio of 67:33 (Table 1, entry 5). The *trans*-alkoxy/aryloxy-β-lac-



Scheme 2 Reaction of diazoacetates **1** and linear imines **2**

tams were obtained as the major products in the case of linear imine **2a**. The described procedure provides a direct method for the preparation of *trans*-alkoxy/aryloxy-β-lactams via the Staudinger reaction.

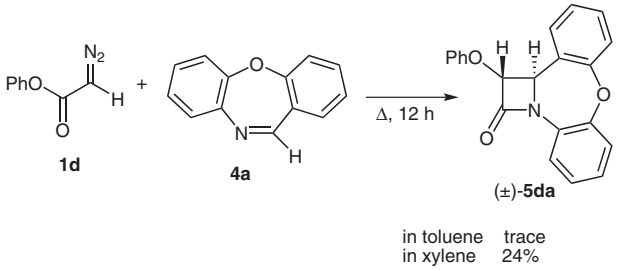
This method was extended to reactions with various cyclic imines, **4a**,**b**, **6**, **8a**,**b** and **10** (Scheme 3). Reactions of cyclic imine **4a** with diazoacetates **1a–d** yielded the desired products, all with *trans* configuration, in yields ranging from 59% to 76% (Table 1, entries 6–9). However, sterically hindered cyclic imines gave rise to the desired products in relatively low yields (Table 1, compare entries 9 and 10–13). Spectral analyses indicated that the structure of β-lactam product **7**, generated from imine **6** and diazoacetate **1d**, was identical to the product obtained from the reaction of imine **6** and phenoxyacetyl chloride in the presence of triethylamine under thermal conditions.[21] By contrast, the reaction of cyclic imine **10** and diazoacetate **1d** produced [2+2+2] cycloadduct **11** as a pair of enantiomers, with no other diastereomers present, as was evident from the ^{1}H and ^{13}C NMR spectra (Table 1, entry 14). It is likely that cycloadduct **11** was generated from one molecule of phenoxyketene and two molecules of imine **10**. Similarly, the [2+2+2]-type cycloadducts were also observed in the reactions of α-diazo ketones and imines.[7a,e] These results indicate that most of the cyclic imines studied give rise to the desired β-lactam products using the current method.



Scheme 3 Reaction of diazoacetates **1a–d** and various cyclic imines

To compare the current method with the thermal Wolff rearrangement,[7f,g] we attempted the reaction of phenyl diazoacetate (**1d**) with cyclic imine **4a** under thermal conditions. Only a trace amount of the desired product **5da** was obtained in toluene, whilst a yield of 24% of **5da** was achieved in xylene, both reactions occurring at reflux temperature over 12 hours. Hence the use of UV irradiation is beneficial for the Wolff rearrangement of diazoacetates (Scheme 4).

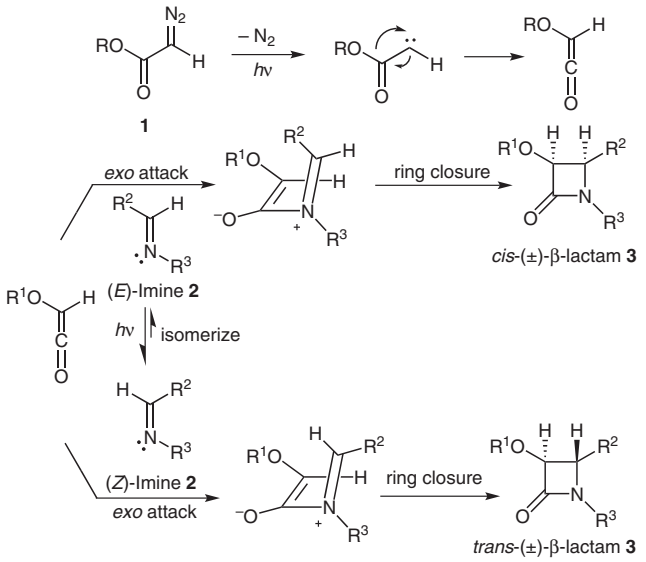
Regarding the reaction mechanism, the alkyl/aryl diazoacetate **1** undergoes a photo-induced Wolff rearrangement via an alkoxy/aryloxy group shift to generate the corresponding alkoxy/aryloxyketene, which further reacts with the imine to give the desired 3-alkoxy/aryloxy-β-lactam by way of the Staudinger reaction. The first step in the



Scheme 4 Thermal reaction of diazoacetate **1d** and imine **4a**

Staudinger reaction is believed to be nucleophilic addition of an imine to a ketene from the less hindered side. The addition between alkoxyketenes and imines is also supported by the 'torquoelectronic effect', as put forward by Hegedus to explain the stereochemical outcome of chromium carbene–imine cycloadditions.[22] That is, conrotatory ring closure of iminium intermediates gives β-lactams through favorable outward rotation of the alkoxy/aryloxy groups. Under thermal conditions, *cis*-β-lactams were obtained as the major or exclusive products with *trans*-imines. However, in the current method (under photoirradiation conditions), both *cis*- and *trans*-β-lactams were obtained from *trans*-imines, indicating that the linear imine and/or the iminium moiety of the zwitterionic intermediate (formed by addition of the imine to the ketene) undergoes isomerization. On the basis of our previous investigation and proposal,[7f] alkoxy/aryloxy groups are strong electron-donating groups and demonstrate very fast ring closure rates in Staudinger reactions (more than 100 times faster than phthalimido, arylthio, aryl and alkyl groups). Typically, isomerization of the alkoxyketene–imine addition intermediates during formation of the 3-alkoxy/aryloxy-β-lactams is not observed with alkoxy/aryloxy ketenes generated from the corresponding acid chlorides, or when chromium carbenes are used under thermal conditions.[11,12] Only the iminium intermediates generated from imines with strong electron-withdrawing N-substituents can partially isomerize in the alkoxy/aryloxy ketene-participating Staudinger reaction.[7f,g] Thus, it is believed that the iminium moiety of the zwitterionic intermediates, generated from alkoxy/aryloxy ketenes, does not isomerize in the current reactions. On the other hand, it has been well documented that linear *E*-imines isomerize preferentially to their *syn*-isomers under UV irradiation conditions.[23] Therefore, it can be concluded that in the current method, *cis*-β-lactams are obtained from the *E*-imines directly, while some *E*-imines isomerize to their *Z*-isomers under UV irradiation, which gives rise to *trans*-β-lactams (Scheme 5).

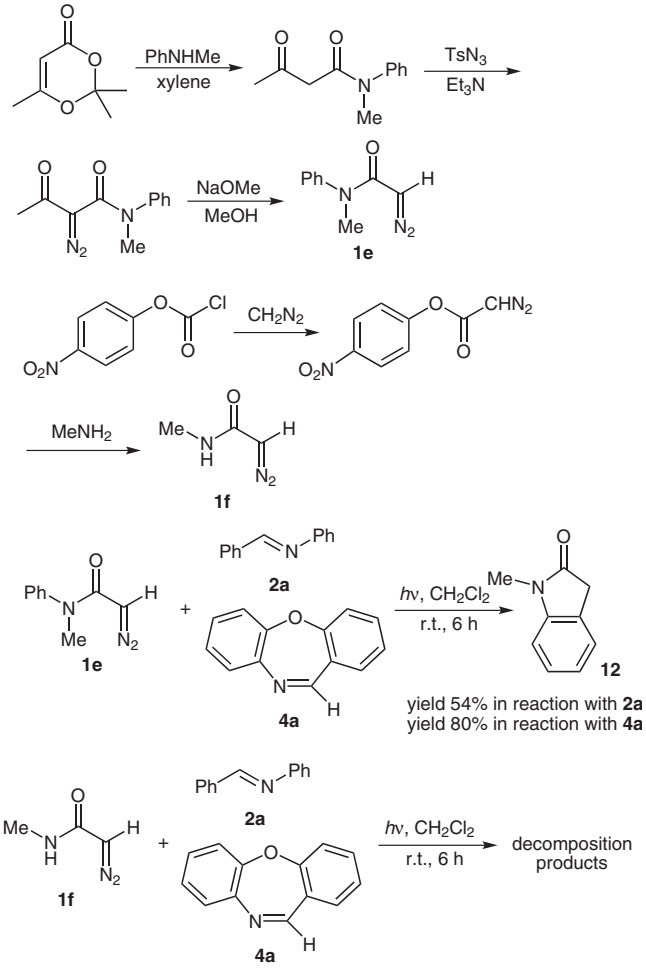
After successful application of alkyl and aryl diazoacetates as ketene precursors in the synthesis of 3-alkoxy/aryloxy-β-lactams, we hoped to extend this method to diazoacetamides in order to prepare 3-mono/dialkylamino-β-lactams. 2-Diazo-*N*-methyl-*N*-phenylacetamide (**1e**) was prepared from *N*-methyl-3-oxo-*N*-phenylbutamide, itself synthesized from 2,2,6-trimethyl-1,3-dioxin-4-one and *N*-methylaniline in xylene at reflux temperature, via



Scheme 5 Proposed mechanism for the reaction of diazoacetates **1** and linear imines **2**

diazo transformation with tosyl azide and subsequent hydrolysis with sodium methoxide.[24] 2-Diazo-*N*-methylacetamide (**1f**) was prepared from methylamine aminolysis of 4-nitrophenyl diazoacetate,[20] which was obtained from 4-nitrophenyl chloroformate and diazomethane. However, in the photo-induced reactions with imines **2a** and **4a**, diazoacetamide **1e** produced, in both cases, 1-methylindolin-2-one (**12**) as the product of intramolecular carbene insertion, instead of the desired β-lactam products. Similar reactions of diazoacetamide **1f** yielded decomposition products. These results indicated that diazoacetamides could not be used as mono/dialkylaminoketene precursors for the preparation of 3-mono/dialkylamino-β-lactams (Scheme 6).

In summary, 3-alkoxy/aryloxy-β-lactams have been synthesized conveniently in satisfactory to good yields by way of the photo-induced Staudinger reaction of imines and alkyl/aryl diazoacetates. Initially, the diazoacetates generated alkoxy/aryloxyketenes, via the photo-induced Wolff rearrangement, which underwent subsequent Staudinger reactions with various imines to give the desired β-lactam products. Compared with the corresponding thermal Staudinger reaction, in which the ketenes produced through base-mediated elimination of the corresponding alkoxy/aryloxyacetyl chlorides, or their equivalents, gave *cis*-β-lactams as the major or sole products, the current method provides in most cases, *trans*-β-lactams predominantly from linear imines. The formation of *trans*-β-lactams from linear imines is attributed to isomerization of the imines from their *trans*-isomers into their *syn*-isomers under UV irradiation. The present method represents a neutral approach to synthesize 3-alkoxy/aryloxy-β-lactams via the Staudinger reaction.



Scheme 6 Reaction of diazoacetamides **1e**,**f** with linear imine **2a** and cyclic imine **4a**

Melting points were measured on a Yanaco MP-500 melting point apparatus and are uncorrected. IR spectra were determined on a Nicolet 5700 FT-IR spectrometer. ^{1}H and ^{13}C NMR spectra were recorded on Varian Mercury 200 (200 MHz), Varian Mercury Plus 300 (300 MHz), or Bruker AMX 400 (400 MHz) spectrometers in $CDCl_3$ with TMS as the internal standard. HRMS data were obtained on a Bruker APEX IV Fourier transform ion cyclotron resonance mass spectrometer. Ethyl diazoacetate was prepared from ethyl glycinate hydrochloride via diazotization with nitrous acid.[19] Isopropyl, benzyl and phenyl diazoacetates **1b**–**d**, and 4-nitrophenyl diazoacetate were synthesized from the corresponding chloroformates and diazomethane.[20] 2-Diazo-*N*-methylacetamide (**1f**) was prepared via aminolysis of 4-nitrophenyl diazoacetate with methylamine.[20] Column chromatography was carried out on Haiyang brand silica gel (200–300 mesh). The analytical data of all known diazoacetates and acetamides were identical to those reported in the literature.[19,20,24] CH_2Cl_2 was dried over CaH_2 and freshly distilled prior to use. **CAUTION:** Although no explosions occurred in any of our experiments, diazomethane is toxic and potentially explosive. Diazoacetates and diazoacetamides are also potentially explosive. The operations must be carried out in a well-ventilated hood with an adequate safety shield.

2-Diazo-*N*-methyl-*N*-phenylacetamide (1e)

A soln of 2,2,6-trimethyl-1,3-dioxin-4-one (7.358 g, 50 mmol) and *N*-methylaniline (4.822 g, 45 mmol) in xylene (50 mL) was heated at reflux temperature for 2 h. After removal of the solvent, the residue was subjected to silica gel column chromatography [EtOAc–PE (30–60 °C), 1:4] to afford *N*-methyl-3-oxo-*N*-phenylbutanamide as a red oil, yield: 7.29 g (85%).

TsN_3 (7.89 g, 40 mmol) in CH_2Cl_2 (15 mL) was added to a soln of *N*-methyl-3-oxo-*N*-phenylbutanamide (7.29 g, 38 mmol) and Et_3N (7.71 g, 76 mmol) in CH_2Cl_2 (30 mL). The soln was stirred for 2.5 h at r.t. and then washed with 35% aq KOH soln [KOH (18 g) in H_2O (500 mL)] (5 × 100 mL) and H_2O (2 × 100 mL). The resulting aqueous mixture was extracted with Et_2O (2 × 100 mL), and the combined organic phase was then dried over anhyd Na_2SO_4 and concd under reduced pressure. The product was separated chromatographically on a silica gel column [EtOAc–PE (30–60 °C), 1:4] to give 2-diazo-*N*-methyl-3-oxo-*N*-phenylbutanamide as yellow oil, yield: 7.43 g (90%).

2-Diazo-*N*-methyl-3-oxo-*N*-phenylbutanamide (4.817 g, 22 mmol) was dissolved in MeOH (25 mL) and the resulting soln treated with an equimolar soln of NaOMe in MeOH (10 mL) at r.t. The mixture was heated at reflux temperature for 3 h, cooled, and poured onto H_2O. The product was extracted with Et_2O (3 × 50 mL) and the combined organic layer dried over anhyd Na_2SO_4 and evaporated. The residue was purified by flash column chromatography on a silica gel column [EtOAc–PE (60–90 °C), 1:4] to give 2-diazo-*N*-methyl-*N*-phenylacetamide as a yellow oil, yield: 3.54 g (92%).

^{1}H NMR (300 MHz, $CDCl_3$): δ = 3.33 (s, 3 H, CH_3), 4.53 (s, 1 H, CH), 7.20–7.45 (m, 5 H, ArH).

Photoirradiation of Diazoacetic Acid Derivatives and Imines; General Procedure

A soln of diazoacetate or diazoacetamide (2 mmol) and imine (1 mmol) in CH_2Cl_2 (10 mL) was irradiated using a medium pressure Hg lamp at r.t. under an N_2 atm for 6 h. After removal of the solvent, the residue was separated chromatographically on a silica gel column [EtOAc–PE (60–90 °C), 1:20] to afford the product.

(±)-*cis*-3-Ethoxy-1,4-diphenylazetidin-2-one (*cis*-3aa)

Colorless crystals; yield: 51 mg (19%); mp 154–155 °C.

IR (KBr): 1745 (C=O) cm^{-1}.

^{1}H NMR (300 MHz, $CDCl_3$): δ = 0.87 (t, *J* = 6.9 Hz, 3 H, CH_3), 3.16 (dq, *J* = 9.0, 6.9 Hz, 1 H of CH_2O), 3.45 (dq, *J* = 9.0, 6.9 Hz, 1 H of CH_2O), 4.93 (d, *J* = 4.8 Hz, 1 H, CH), 5.20 (d, *J* = 4.5 Hz, 1 H, CH), 7.03–7.40 (m, 10 H, ArH).

^{13}C NMR (75.5 MHz, $CDCl_3$): δ = 15.5, 62.0, 66.4, 83.5, 117.4, 124.3, 128.0, 128.4, 128.5, 129.0, 133.4, 137.1, 164.4.

HRMS (ESI): *m*/*z* [M + H]$^+$ calcd for $C_{17}H_{18}NO_2$: 268.1332; found: 268.1329.

(±)-*trans*-3-Ethoxy-1,4-diphenylazetidin-2-one (*trans*-3aa)

Colorless crystals; yield: 162 mg (61%); mp 113–114 °C.

IR (KBr): 1754 (C=O) cm^{-1}.

^{1}H NMR (300 MHz, $CDCl_3$): δ = 1.28 (t, *J* = 7.0 Hz, 3 H, CH_3), 3.71 (dq, *J* = 9.3, 7.0 Hz, 1 H of CH_2O), 3.81 (dq, *J* = 9.3, 7.0 Hz, 1 H of CH_2O), 4.47 (d, *J* = 1.8 Hz, 1 H, CH), 4.91 (d, *J* = 1.8 Hz, 1 H, CH), 7.05–7.42 (m, 10 H, ArH).

^{13}C NMR (75.5 MHz, $CDCl_3$): δ = 15.5, 63.8, 66.7, 90.1, 117.5, 124.3, 126.0, 128.7, 129.0, 129.2, 136.4, 137.1, 164.5.

HRMS (ESI): *m*/*z* [M + Na]$^+$ calcd for $C_{17}H_{17}NNaO_2$: 290.1151; found: 290.1151.

(±)-*cis*-3-Isopropoxy-1,4-diphenylazetidin-2-one (*cis*-3ba)

Colorless crystals; yield: 28 mg (10%); mp 154–155 °C.

IR (KBr): 1738 (C=O) cm^{-1}.

^{1}H NMR (300 MHz, $CDCl_3$): δ = 0.64 (d, *J* = 6.0 Hz, 3 H, CH_3), 1.09 (d, *J* = 6.0 Hz, 3 H, CH_3), 3.40 (sept, *J* = 6.0 Hz, 1 H, CH), 5.02 (d, *J* = 4.5 Hz, 1 H, CH), 5.18 (d, *J* = 4.5 Hz, 1 H, CH), 7.03–7.37 (m, 10 H, ArH).

^{13}C NMR (50 MHz, $CDCl_3$): δ = 21.1, 22.1, 62.5, 72.7, 81.9, 117.4, 124.2, 128.2, 128.3, 128.4, 129.0, 133.8, 137.2, 165.0.

HRMS (ESI): *m*/*z* [M + H]$^+$ calcd for $C_{18}H_{20}NO_2$: 282.1489; found: 282.1491.

(±)-*trans*-3-Isopropoxy-1,4-diphenylazetidin-2-one (*trans*-3ba)

Colorless oil; yield: 98 mg (35%).

IR (KBr): 1760 (C=O) cm^{-1}.

^{1}H NMR (300 MHz, $CDCl_3$): δ = 1.18 (d, *J* = 6.0 Hz, 3 H, CH_3), 1.25 (d, *J* = 6.3 Hz, 3 H, CH_3), 3.85 (sept, *J* = 6.0 Hz, 1 H, CH), 4.49 (d, *J* = 1.8 Hz, 1 H, CH), 4.84 (d, *J* = 1.8 Hz, 1 H, CH), 7.00–7.41 (m, 10 H, ArH).

^{13}C NMR (50 MHz, $CDCl_3$): δ = 22.4, 22.6, 64.8, 73.6, 88.9, 117.4, 124.1, 126.0, 128.6, 128.9, 129.2, 136.3, 137.1, 165.0.

HRMS (ESI): *m*/*z* [M + H]$^+$ calcd for $C_{18}H_{20}NO_2$: 282.1489; found: 282.1483.

(±)-*cis*-3-Benzyloxy-1,4-diphenylazetidin-2-one (*cis*-3ca)

Colorless crystals; yield: 74 mg (22%); mp 149–151 °C (Lit.[25] 148–150 °C).

^{1}H NMR (200 MHz, $CDCl_3$): δ = 4.27 (d, *J* = 11.2 Hz, 1 H of CH_2O), 4.38 (d, *J* = 11.2 Hz, 1 H of CH_2O), 5.02 (d, *J* = 4.8 Hz, 1 H, CH), 5.23 (d, *J* = 4.8 Hz, 1 H, CH), 6.92–7.41 (m, 15 H, ArH).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 62.0, 72.5, 82.7, 117.5, 124.4, 128.0, 128.1, 128.3, 128.7, 129.1, 133.5, 136.3, 137.1, 164.3.

(±)-*trans*-3-Benzyloxy-1,4-diphenylazetidin-2-one (*trans*-3ca)

Colorless crystals; yield: 173 mg (53%); mp 148–150 °C (Lit.[26] 148–150 °C).

^{1}H NMR (300 MHz, $CDCl_3$): δ = 4.56 (d, *J* = 1.8 Hz, 1 H, CH), 4.65 (d, *J* = 11.7 Hz, 1 H of CH_2O), 4.87 (d, *J* = 1.8 Hz, 1 H, CH), 4.90 (d, *J* = 11.7 Hz, 1 H of CH_2O), 7.02–7.34 (m, 15 H, ArH).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 63.9, 73.2, 89.8, 117.6, 124.4, 126.1, 128.25, 128.30, 128.6, 128.7, 129.1, 129.2, 136.2, 136.9, 137.2, 164.4.

(±)-*cis*-3-Phenoxy-1,4-diphenylazetidin-2-one (*cis*-3da)

Colorless crystals; yield: 103 mg (33%); mp 194–195 °C (Lit.[27] 193–195 °C).

^{1}H NMR (200 MHz, $CDCl_3$): δ = 5.41 (d, *J* = 5.0 Hz, 1 H, CH), 5.58 (d, *J* = 5.0 Hz, 1 H, CH), 6.59–7.47 (m, 15 H, ArH).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 62.1, 81.2, 115.8, 117.6, 122.2, 124.6, 128.1, 128.4, 128.8, 129.2, 129.3, 132.6, 137.0, 157.0, 163.1.

(±)-*trans*-3-Phenoxy-1,4-diphenylazetidin-2-one (*trans*-3da)

Colorless crystals; yield: 209 mg (66%); mp 112–113 °C (Lit.[28] 110–113 °C).

^{1}H NMR (200 MHz, $CDCl_3$): δ = 5.02 (d, *J* = 1.6 Hz, 1 H, CH), 5.12 (d, *J* = 1.6 Hz, 1 H, CH), 6.85–7.50 (m, 15 H, ArH).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 64.0, 87.4, 115.5, 117.7, 122.4, 124.7, 126.5, 129.18, 129.24, 129.5, 129.7, 135.6, 137.0, 157.1, 162.7.

(±)-*cis*-1-Benzyl-3-phenoxy-4-phenylazetidin-2-one (*cis*-3db)

Colorless crystals; yield: 171 mg (52%); mp 117–118 °C (Lit.[29] 116–117 °C).

^{1}H NMR (400 MHz, $CDCl_3$): δ = 3.87 (d, *J* = 14.8 Hz, 1 H of CH_2), 4.75 (d, *J* = 4.4 Hz, 1 H, CH), 4.90 (d, *J* = 14.8 Hz, 1 H of CH_2), 5.40 (d, *J* = 4.4 Hz, 1 H, CH), 6.69–7.33 (m, 15 H, ArH).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 44.2, 61.5, 82.2, 115.6, 122.0, 128.0, 128.3, 128.66, 128.68, 128.9, 129.2, 132.7, 134.8, 156.9, 165.6.

(±)-*trans*-1-Benzyl-3-phenoxy-4-phenylazetidin-2-one (*trans*-3db)
Colorless crystals; yield: 85.5 mg (26%); mp 103–104 °C.

IR (KBr): 1752 (C=O) cm^{-1}.

^1H NMR (400 MHz, $CDCl_3$): δ = 3.80 (d, *J* = 14.8 Hz, 1 H of CH_2), 4.41 (d, *J* = 1.2 Hz, 1 H, CH), 4.91 (d, *J* = 14.8 Hz, 1 H of CH_2), 5.02 (d, *J* = 1.2 Hz, 1 H, CH), 6.77–7.45 (m, 15 H, ArH).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 44.5, 63.1, 87.5, 115.4, 122.1, 127.0, 128.0, 128.6, 128.9, 129.2, 129.3, 129.6, 134.6, 135.3, 157.1, 165.3.

HRMS (ESI): *m*/*z* $[M + H]^+$ calcd for $C_{22}H_{20}NO_2$: 330.1489; found: 330.1494.

(±)-*trans*-1-Ethoxy-1,12b-dihydroazeto[1,2-*d*]dibenzo[*b*,*f*][1,4]oxazepin-2-one (5aa)
Colorless oil; yield: 188 mg (67%).

IR (KBr): 1754 (C=O) cm^{-1}.

^1H NMR (300 MHz, $CDCl_3$): δ = 1.36 (t, *J* = 6.9 Hz, 3 H, CH_3), 3.84 (dq, *J* = 9.0, 6.9 Hz, 1 H of CH_2O), 3.97 (dq, *J* = 9.0, 6.9 Hz, 1 H of CH_2O), 5.05 (d, *J* = 2.1 Hz, 1 H, CH), 5.61 (d, *J* = 1.8 Hz, 1 H, CH), 6.99–8.08 (m, 8 H, ArH).

^{13}C NMR (75.5 MHz, $CDCl_3$): δ = 15.2, 60.5, 66.5, 84.5, 120.3, 121.6, 121.7, 124.8, 125.3, 126.0, 129.3, 129.7, 130.4, 144.1, 150.7, 158.6, 162.9.

HRMS (ESI): *m*/*z* $[M + Na]^+$ calcd for $C_{17}H_{15}NNaO_3$: 304.0944; found: 304.0937.

(±)-*trans*-1,12b-Dihydro-1-isopropoxyazeto[1,2-*d*]dibenzo[*b*,*f*][1,4]oxazepin-2-one (5ba)
Colorless oil; yield: 174 mg (59%).

IR (KBr): 1757 (C=O) cm^{-1}.

^1H NMR (300 MHz, $CDCl_3$): δ = 1.33 (d, *J* = 6.0 Hz, 3 H, CH_3), 1.39 (d, *J* = 6.3 Hz, 3 H, CH_3), 4.05 (sept, *J* = 6.0 Hz, 1 H, CH), 5.06 (d, *J* = 2.1 Hz, 1 H, CH), 5.56 (d, *J* = 1.8 Hz, 1 H, CH), 6.95–8.08 (m, 8 H, ArH).

^{13}C NMR (75.5 MHz, $CDCl_3$): δ = 22.4, 22.7, 61.5, 73.5, 83.1, 120.2, 121.5, 121.8, 124.7, 125.3, 125.8, 129.3, 129.7, 130.3, 144.1, 158.6, 163.3.

HRMS (ESI): *m*/*z* $[M + H]^+$ calcd for $C_{18}H_{18}NO_3$: 296.1281; found: 296.1280.

(±)-*trans*-1-Benzyloxy-1,12b-dihydroazeto[1,2-*d*]dibenzo[*b*,*f*][1,4]oxazepin-2-one (5ca)
Colorless oil; yield: 240 mg (70%).

IR (KBr): 1750 (C=O) cm^{-1}.

^1H NMR (300 MHz, $CDCl_3$): δ = 4.83 (d, *J* = 11.7 Hz, 1 H of CH_2O), 4.95 (d, *J* = 11.7 Hz, 1 H of CH_2O), 5.07 (d, *J* = 2.1 Hz, 1 H, CH), 5.59 (d, *J* = 2.1 Hz, 1 H, CH), 6.94–8.07 (m, 13 H, ArH).

^{13}C NMR (75 MHz, $CDCl_3$): δ = 60.5, 72.5, 83.4, 120.1, 121.5, 121.6, 124.7, 125.1, 125.2, 125.8, 127.9, 128.2, 128.5, 129.0, 129.5, 130.2, 136.5, 144.0, 158.4, 162.7.

HRMS (ESI): *m*/*z* $[M + H]^+$ calcd for $C_{22}H_{18}NO_3$: 344.1281; found: 344.1292.

(±)-*trans*-1,12b-Dihydro-1-phenoxyazeto[1,2-*d*]dibenzo[*b*,*f*][1,4]oxazepin-2-one (5da)
Colorless crystals; yield: 174 mg (76%); mp 157–158 °C.

IR (KBr): 1755 (C=O) cm^{-1}.

^1H NMR (300 MHz, $CDCl_3$): δ = 5.62 (d, *J* = 2.2 Hz, 1 H, CH), 5.84 (d, *J* = 2.2 Hz, 1 H, CH), 7.00–8.08 (m, 13 H, ArH).

^{13}C NMR (75 MHz, $CDCl_3$): δ = 60.6, 83.0, 116.4, 120.3, 121.6, 121.9, 122.9, 125.1, 125.4, 125.9, 128.6, 129.5, 129.8, 130.5, 144.2, 157.3, 158.6, 161.6.

HRMS (ESI): *m*/*z* $[M + H]^+$ calcd for $C_{21}H_{16}NO_3$: 330.1125; found: 330.1129.

(±)-*trans*-1,12b-Dihydro-12b-methyl-1-phenoxyazeto[1,2-*d*]dibenzo[*b*,*f*][1,4]oxazepin-2-one (5db)
Colorless crystals; yield: 213 mg (62%); mp 43–44 °C.

IR (KBr): 1755 (C=O) cm^{-1}.

^1H NMR (300 MHz, $CDCl_3$): δ = 2.11 (s, 3 H, CH_3), 5.65 (s, 1 H, CH), 7.05–8.18 (m, 13 H, ArH).

^{13}C NMR (50 MHz, $CDCl_3$): δ = 22.7, 67.8, 84.9, 116.7, 120.9, 121.5, 122.5, 122.8, 125.0, 125.2, 125.5, 128.7, 129.8, 130.1, 133.9, 143.6, 157.0, 157.7, 162.5.

HRMS (ESI): *m*/*z* $[M + H]^+$ calcd for $C_{22}H_{18}NO_3$: 344.1281; found: 344.1280.

***rel*-(2*S*,2a*R*,4*R*)-2,2a,3,4-Tetrahydro-2-phenoxy-2a,4-diphenylazeto[1,2-*d*]benzo[*b*][1,4]thiazepin-1-one (7)**
Colorless crystals; yield: 233.5 mg (52%); mp 190–191 °C (Lit.[21] 190–191 °C).

IR (KBr): 1765 (C=O) cm^{-1}.

^1H NMR (300 MHz, $CDCl_3$): δ = 3.23 (dd, *J* = 14.1, 11.1 Hz, 1 H of CH_2), 3.68 (d, *J* = 14.1 Hz, 1 H of CH_2), 4.02 (d, *J* = 11.1 Hz, 1 H, CH), 5.40 (s, 1 H, CH), 6.68–8.03 (m, 19 H, ArH).

^{13}C NMR (50 MHz, $CDCl_3$): δ = 44.9, 48.4, 71.0, 88.4, 115.5, 122.2, 126.8, 126.9, 128.1, 128.2, 128.4, 129.2, 129.3, 129.8, 132.5, 134.6, 137.6, 140.8, 152.9, 156.7, 164.0.

HRMS (ESI): *m*/*z* $[M + H]^+$ calcd for $C_{29}H_{24}NO_2S$: 450.1522; found: 450.1522.

(±)-*trans*-7-Phenoxy-6-phenyl-5-oxa-1-azabicyclo[4.2.0]octan-8-one (9a)
Colorless crystals; yield: 94.5 mg (32%); mp 140–141 °C (Lit.[30] 139–140 °C).

IR (KBr): 1776 (C=O) cm^{-1}.

^1H NMR (300 MHz, $CDCl_3$): δ = 1.49 (m, 1 H of CH_2), 2.03 (m, 1 H of CH_2), 3.08 (m, 1 H of CH_2N), 3.79 (m, 1 H of CH_2N), 4.03 (m, 2 H, CH_2O), 5.24 (s, 1 H, CH), 6.69–7.50 (m, 10 H, ArH).

^{13}C NMR (50 MHz, $CDCl_3$): δ = 24.1, 36.5, 62.7, 89.1, 90.8, 115.2, 121.8, 127.7, 128.1, 128.7, 129.0, 132.7, 156.4, 164.5.

HRMS (ESI): *m*/*z* $[M + H]^+$ calcd for $C_{18}H_{18}NO_3$: 296.1281; found: 296.1277.

(±)-*trans*-7-Phenoxy-6-phenyl-5-thia-1-azabicyclo[4.2.0]octan-8-one (9b)
Colorless crystals; yield: 168 mg (54%); mp 130–131 °C (Lit.[26] 130–131 °C).

^1H NMR (400 MHz, $CDCl_3$): δ = 1.89 (m, 2 H), 2.73 (m, 2 H), 3.04 (m, 1 H), 4.15 (m, 1 H), 5.39 (s, 1 H), 6.62–7.60 (m, 10 H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 23.8, 26.0, 37.6, 70.2, 91.0, 115.4, 122.2, 128.1, 128.5, 128.8, 129.2, 134.6, 156.5, 162.5.

4b,5,9,13b,15,16-Hexahydro-5-phenoxy-6*H*,8*H*-pyrimido[2,1-*a*:4,3-*a*′]diisoquinolin-6-one (11)
Colorless crystals; yield: 309 mg (78%); mp 183–184 °C.

IR (KBr): 1653 (C=O) cm^{-1}.

^1H NMR (300 MHz, $CDCl_3$): δ = 2.57 (m, 1 H of CH_2), 2.79 (m, 2 H of CH_2), 3.01 (m, 3 H of CH_2), 3.36 (m, 1 H of CH_2), 4.68 (d,

J = 4.8 Hz, 1 H, CHN), 4.80 (d, J = 5.1 Hz, 1 H, CHO), 4.97 (m, 1 H of CH_2), 5.88 (s, 1 H, CHN_2), 6.67–7.53 (m, 13 H, ArH).

^{13}C NMR (50 MHz, $CDCl_3$): δ = 28.3, 28.5, 36.9, 38.3, 61.7, 74.9, 76.4, 117.5, 121.9, 125.4, 126.2, 126.9, 127.0, 127.4, 127.7, 128.6, 128.8, 132.4, 132.7, 135.7, 136.5, 160.3, 167.3.

HRMS (ESI): m/z $[M + H]^+$ calcd for $C_{26}H_{25}N_2O_2$: 397.1911; found: 397.1921.

1-Methyl-2-indolinone (12)
Yellow crystals; yield: 118 mg (80%); mp 91–92 °C (Lit.[31] 88–89 °C).

^{1}H NMR (300 MHz, $CDCl_3$): δ = 3.20 (s, 3 H, CH_3), 3.50 (s, 2 H, CH_2), 6.80–7.31 (m, 4 H, ArH).

^{13}C NMR (75.5 MHz, $CDCl_3$): δ = 26.0, 35.6, 107.9, 122.2, 124.2, 124.3, 127.7, 145.0, 174.9.

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