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Polycyclic Indoline-Benzodiazepines through Electrophilic Additions of α-Imino Carbenes to Tröger Bases

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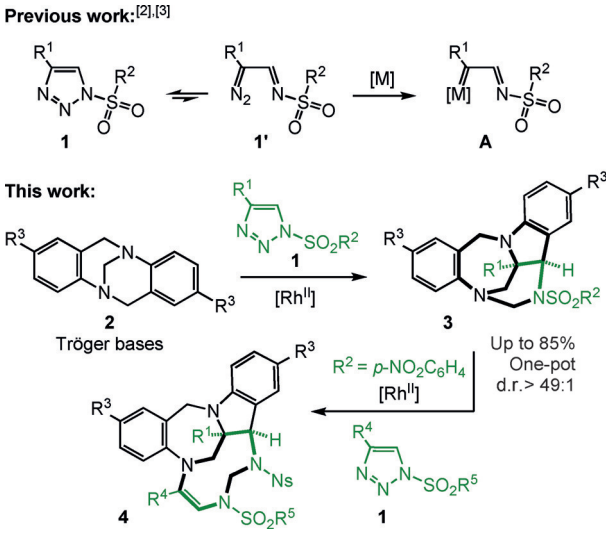
Abstract: *Polycyclic indoline-benzodiazepines can be accessed through the intermolecular reaction of Tröger bases with N-sulfonyl-1,2,3-triazoles. Under Rh^{II} catalysis, α-imino carbenes are generated and a subsequent cascade of [1,2]-Stevens, Friedel–Crafts, Grob, and aminal formation reactions yield the polycyclic heterocycles as single isomers (d.r. > 49:1, four stereocenters including two bridgehead N atoms). Further ring expansion by insertion of a second α-imino carbene leads to elaborated polycyclic 9-membered-ring triazonanes.*

N-Sulfonyl-1,2,3-triazoles **1** are structural motifs that are used regularly in synthetic, biological, and medicinal chemistry.[1] Significantly, triazoles **1** are in equilibrium with α-diazo imines of type **1′**, which decompose under metal catalysis to afford α-imino carbenes **A** (Scheme 1, top).[2] In recent years, many synthetically useful and original processes have been developed using these electrophilic intermediates, from migrations to ylide-forming reactions and subsequent transformations.[3,4] In terms of ammonium ylide chemistry,[5] few studies have been reported.[6] Of interest to the current study, the synthesis of tetrasubstituted indolines through reactions of *o*-vinylanilines is noteworthy.[7] Herein, in a new development, the intermolecular reactivity of *N*-sulfonyl-1,2,3-triazoles **1** with Tröger bases (TB) **2** is reported (Scheme 1, bottom). Under Rh^{II} catalysis (2 mol%),[8] densely functionalized polycyclic heterocycles **3** are obtained (yields up to 85%). After the initial addition of carbenes **A** to TB, the transformation involves a cascade of [1,2]-Stevens, Friedel–Crafts, Grob and aminal formation reactions. Resulting chalice-like products **3** are generated with high stereoselectivity (d.r. > 49:1, four stereocenters including two bridgehead nitrogen atoms). When the aminal nitrogen is protected by a nosyl group, insertion of a second α-imino carbene is possible, leading to nine-membered triazonanes **4**, albeit in moderate yields (29–34%). In view of their unusual heterocyclic structures, products **3** and **4** should be of interest in a variety of fields, and medical chemistry in particular. These derivatives combine in a single framework both indoline and benzodiazepine skeletons, which are privileged motifs in natural product and medical chemistry (Figure 1).[9,10]



Scheme 1. Generation of α-imino carbenes and application to the synthesis of elaborated polycyclic indoline-benzodiazepines.

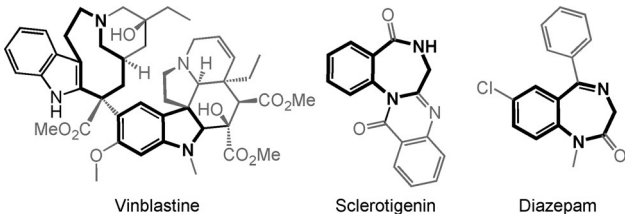


Figure 1. Typical examples of an indoline (left) and benzodiazepines (right).

Recently, it was shown that TB **2** (Scheme 1, bottom),[11] classical diaza heterocycles prepared in one step by condensation of primary anilines with formaldehyde, react with α-keto carbenes to afford ethano-TB, after aminal bridge expansion.[12] Drawing from the direct analogy that exists

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between α-keto and α-imino carbenes, the reactivity of **2** with intermediates **A** was tested. Initial experiments were performed using Tröger base **2a** (R^3 = Me) and *N*-tosyl-4-phenyltriazole **1A** under dirhodium catalysis ($Rh_2(oct)_4$, 1 mol%). While the reaction was mostly unproductive, a product was isolated in low yield (8%).

This compound **3aA** presented interesting characteristics in ^{1}H and ^{13}C-NMR spectroscopy and an additional mass of 271 Da compared to **2a** (equivalent to one carbene moiety). The structure of **3aA** was determined with certainty after X-ray diffraction analysis (Figure 2). It presents an interesting

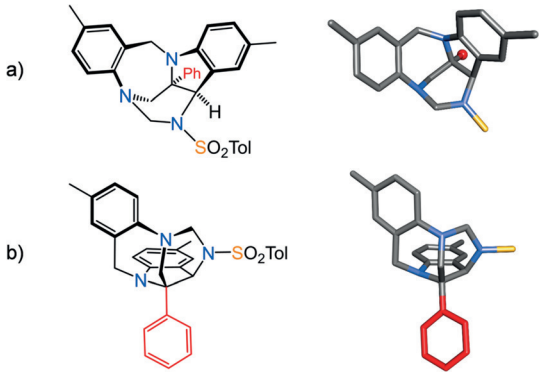


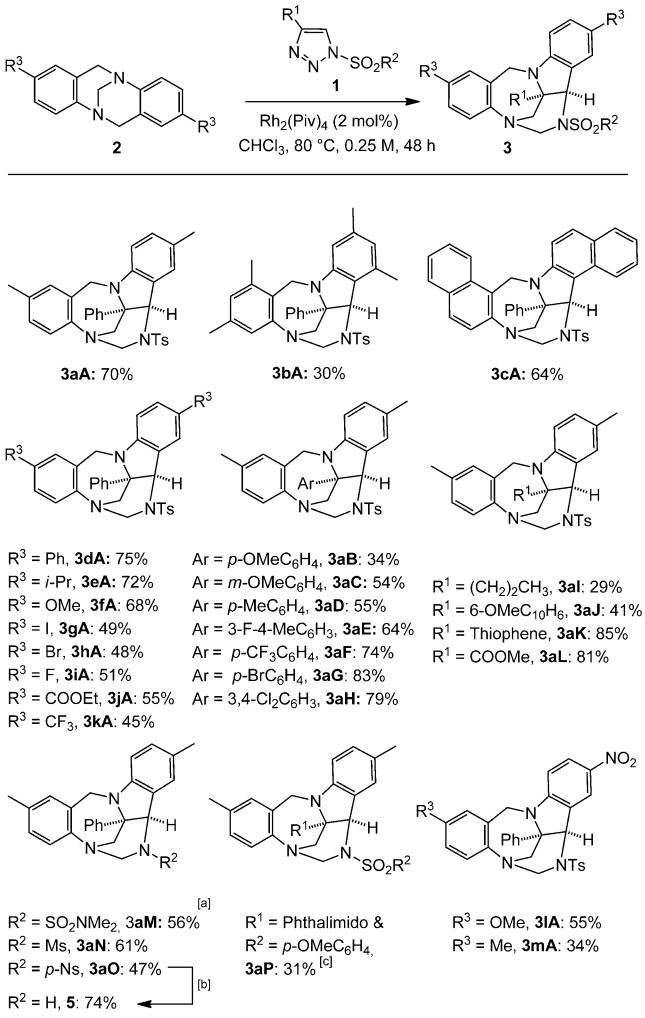
Figure 2. Representations of indoline-benzodiazepine **3aA** and stick views of the crystal structure.[24] a) View from above the rim (top). b) Lateral view. Part of the tosyl group is removed for clarity in the stick models. The apical phenyl group is indicated in red and replaced with a single ball in the top right representation.

chalice-like geometry with 5-, 6-, and 7-membered rings forming a rigid bowl-shape motif above substituent R^1 (here Ph), which extends from the center of the cup in an apical fashion. In Figure 2, both top and lateral views of **3aA** are represented since the geometry of compounds **3** is difficult to appreciate with a single representation.[13] Overall, an interesting fusion of indoline and benzodiazepine motifs is revealed.

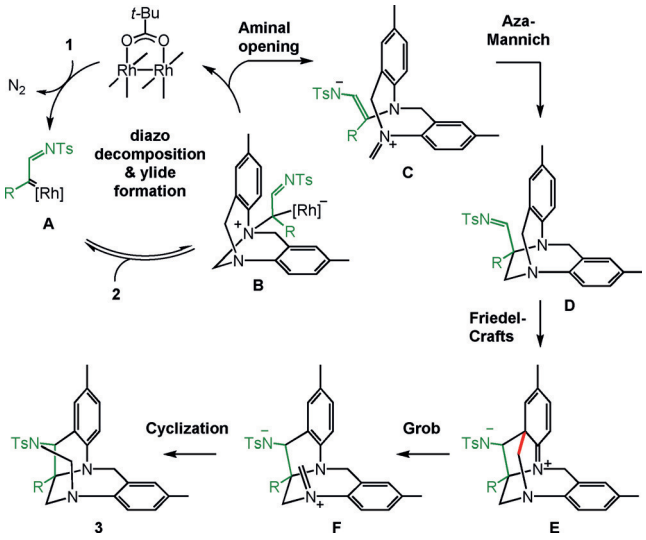
With this result and characterization in hand, optimization studies were conducted to improve the synthesis of **3aA** and the results are reported in Table S1 in the Supporting Information. Using $CHCl_3$ as the solvent and $Rh_2(Piv)_4$ (2 mol%) as the catalyst improved the outcome. Complete consumption of **2a** is achieved after 48 h at 80°C when using a slight excess of **1A** (1.5 equiv, 0.25 M). Product **3aA** is then formed in 70% yield as a single stereoisomer.[14] With these conditions, disubstituted and naphthyl derivatives **3bA** and **3cA** were obtained in 30% and 64% yields, respectively. In the first case, the lower yield might be due to the strain created by the methyl groups at immediate proximity to the central bicyclic core.[15] Modulation of the substituents at the *para* position to the nitrogen atoms of TB substrates led to products **3dA** to **3kA** in moderate to good yields (45–75%). The reactions proceed more effectively with electron donating substituents (**3dA**–**3fA**) than with electron-withdrawing groups (**3gA**–**3kA**). A higher nucleophilic character of the nitrogen atoms is beneficial for the first elemental step of the mechanism (see below, Scheme 3, **A**→**B**).[16]

The reaction was also performed with a series of *N*-sulfonyl triazoles (**1B**–**1P**) and TB **2a** as substrates. An inverse electronic demand was then beneficial. In fact, with electron-donating substituents on the triazole precursors, products **3aB**–**3aD** are obtained in low to moderate yields (34–55%), while higher yields (74–83%) are afforded in the presence of electron-withdrawing groups (**3aF**–**3aH**). This electronic preference was confirmed with four other derivatives (**3aI**–**3aL**); thiophene and methyl ester substituents promoted the reaction effectively while *n*-propyl and 6-methoxynaphthalene residues led to lower yields.

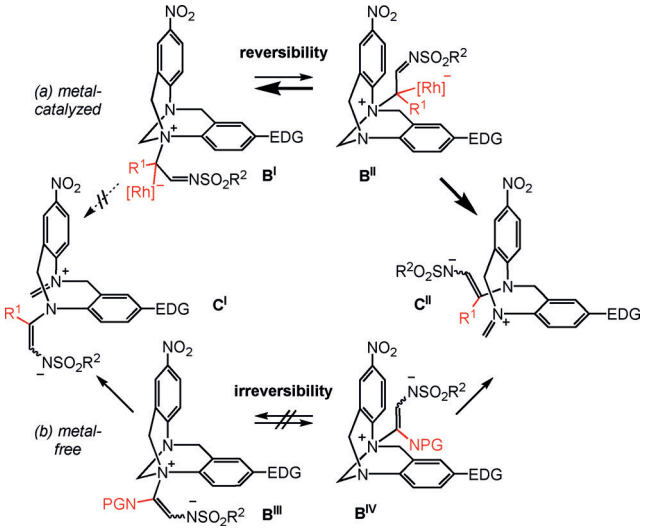
The influence of the sulfonyl group substituent was then further investigated. Dimethylsulfamoyl-, mesyl (Ms)-, and *p*-nosyl (Ns)-substituted triazoles were reacted and afforded products **3aM**, **3aN**, and **3aO** in 56%, 61% and 47% yields, respectively. With **3aO** in hand, removal of the *p*-nosyl group was possible by a treatment with thiophenol/K_2CO_3 to afford **5** in 74% yield (Scheme 2).[17] Care was also taken to verify that the process can occur under metal-free conditions. Using Davies' *N*-sulfonyl-4-phthalimido triazole **1P**,[18] product **3aP**



Scheme 2. Scope of the reaction. [a] Reaction performed at 100°C. [b] PhSH, K_2CO_3, CH_3CN/DMSO (49:1), 50°C, 2 h. [c] Thermal activation only.



Scheme 3. Mechanistic rationale.



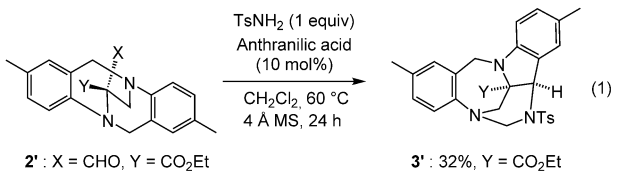
Scheme 4. a) Reversible ylide formation under metal-catalyzed conditions and preferred formation of electronically-stabilized iminium $\mathbf{C^{II}}$. b) Formation of iminium intermediates $\mathbf{C^{I}}$ and $\mathbf{C^{II}}$ under (kinetic) metal-free conditions. EDG = Electron-donating group (OMe or Me), NPG = *N*-phthalimido proup.

was formed under thermal activation, albeit in only 31% yield. Finally, **3lA** and **3mA** were also prepared; these compounds formed as single regioisomers and are of importance for the mechanistic discussion (see below).

A rationale for the observed reactivity is outlined in Scheme 3. In the presence of Rh^{II} catalysts and $Rh_2(Piv)_4$ in particular, *N*-sulfonyl triazoles **1** generate α-imino carbenes **A** by nitrogen extrusion.[2] These electrophilic intermediates react with Tröger bases **2** to form nitrogen ylides **B**. The quaternization of the nitrogen atom leads to ring opening of the aminal bridge to form intermediates of type **C**. These species react intramolecularly in an aza-Mannich reaction and cyclize to form sulfonyl imines **D**. The overall process from **1** to **D** corresponds to a formal [1,2]-Stevens rearrangement.[12a] Then, induced by the spatial proximity between the electrophilic imine and the electron-rich aniline, a Friedel–Crafts cyclization occurs to form intermediate **E**. In this rigid moiety, the alignment of the nitrogen lone pair with the σ* orbital of the adjacent C–C bond (highlighted in red) leads to a Grob fragmentation[19] and the formation of intermediates **F**. A final intramolecular trapping of the new iminium species by the nitrogen of the sulfonamide group leads to final aminal formation and products **3**.

In the above rationale, the ylide formation (**A**→**B**) is presented as a reversible step. This was established by the regioselective formation of **3lA** and **3mA**, in which nitrogen atoms *para* to the nitro group of **2l** and **2m**—the more electron poor and less nucleophilic N atoms—have reacted with carbenes **A**.[20] This result, in contradiction with the general reactivity (Scheme 2),[16] can be however rationalized by a Curtin–Hammet analysis.[12a] It is proposed that carbenes **A** react preferentially with the more electron-rich nitrogen atoms (*para* to the MeO or Me groups, step **A**→$\mathbf{B^{I}}$) but the subsequent aminal opening is disfavored since it would lead to electronically unstable intermediates $\mathbf{C^{I}}$ (Scheme 4, top). Opportunely, upon ylide equilibration, reaction of **A** with the less nucleophilic (disfavored) nitrogen occurs (→$\mathbf{B^{II}}$). The subsequent aminal opening forms intermediates $\mathbf{C^{II}}$ and their higher stability constitutes the kinetic and thermodynamic driving force of the process. Importantly, in the reactions with **1P** performed under thermal activation,[18] TB **2l** and **2m** react to afford 1:1 mixtures of regioisomers (see Schemes S1–S2 in the Supporting Information). In these metal-free processes, intermediates $\mathbf{B^{III}}$ and $\mathbf{B^{IV}}$ are probably formed in equimolar ratios and do not interconvert (Scheme 4, bottom). The presence of the Rh^{II} catalyst is thus essential to ensure the reversibility of the ylide formation.

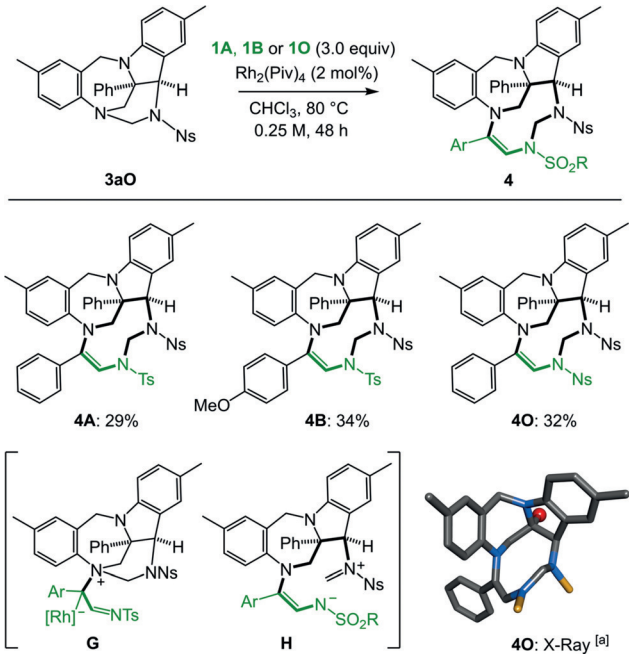
Of importance for the mechanistic understanding, aldehyde-substituted ethano-TB **2′** was also prepared (see the Supporting Information) and reacted with tosyl amine in the presence of anthranilic acid (10 mol%).[21] The corresponding iminium **D** could not be observed in ^{1}H NMR spectroscopy, but a slow and steady formation of polycyclic product **3′** (32%) was observed [Eq. (1)]. This experiment suggests that,



2′ : X = CHO, Y = CO_2Et **3′** : 32%, Y = CO_2Et

once formed, intermediates **D** react rapidly in the Friedel–Crafts and subsequent reactions to form products **3**.

Finally, with *p*-nosyl-substituted derivative **3aO** in hand, the possibility to form benzodiazepine-indoline adducts containing a 9-membered 1,3,6-triazonane ring was recognized (Scheme 5).[22] Treatment of **3aO** with an excess of **1A**, **1B**, or **1O** (3 equiv) in $CHCl_3$ (0.25 M) at 80 °C (48 h) and $Rh_2(Piv)_4$ as catalyst (2 mol%) afforded products **4A**, **4B** and **4O** in moderate yields (29–34%). The structure of compounds **4** was unambiguously determined by X-ray crystal-



Scheme 5. Synthesis of benzodiazepine-indoline triazonanes **4** and probable intermediates **G** and **H**. [a] Part of the sulfonyl groups are removed for clarity; the apical phenyl group is replaced with a red single ball.[24]

lography. Mechanistically, the most nucleophilic and less-hindered tertiary nitrogen atom of **3O** reacts with carbenes **A** (generated in situ) to yield ylides **G** (Scheme 5, bottom). Then, aminal opening (formation of iminium intermediates **H**) and intramolecular nucleophilic closures lead to products **4**.

In summary, polycyclic indoline-benzodiazepines **3** were generated in a single step through the direct intermolecular reaction of TB with *N*-sulfonyl-1,2,3-triazoles. Initiated by the formation of α-imino carbenes under Rh^{II} catalysis, a cascade of [1,2]-Stevens, Friedel–Crafts, Grob, and aminal formation reactions occurs to yield the triaza polycycles as single isomers (d.r. > 49:1, four stereocenters including two bridgehead N atoms). Mechanistic insight was obtained, demonstrating, for instance, the importance of metal-bound ylides to explain the regioselectivity of certain reactions. Further ring expansions by insertion of a second α-imino carbene are possible, resulting in elaborated polycyclic 9-membered ring triazonanes **4**. Products of type **3** or **4** present interesting skeleton and geometries that ought to be tested in medicinal chemistry, and their preparation is difficult to imagine by other routes.[23]

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Conflict of interest

The authors declare no conflict of interest.

Keywords: benzodiazepines · cascade reactions · indolines · rhodium · α-imino carbenes

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[9] In relation to compounds **3** and **4**, vinblastine is an interesting example since it contains not only the indoline skeleton but also a 9-membered ring in the second fragment.

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[13] A third stereochemical representation of compounds **3** is provided in Scheme 3.

[14] In TB and derivatives, the bridgehead nitrogen atoms are stereogenic and configurationally stable under neutral conditions.

[15] With TB-carrying substituents *ortho* to the nitrogen atoms, only traces of the desired products were observed with very small conversion of starting materials.

[16] With two *p*-nitro substituents, only traces of conversion are observed.

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[24] CCDC 1832809 (**4O**), 1832810 (**3aA**), and 1832811 (**3IA**) contain the supplementary crystallographic data for this paper. These can be obtained free of charge from The Cambridge Crystallographic Data Centre.

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