Vinyl Sulfoxonium Ylide: A New Vinyl Carbenoid Transfer Reagent for the Synthesis of Heterocycles

Janakiram Vaitla*[a,b]
Annette Bayer[b]
Kathrin H. Hopmann*[a,b]

[a] Hylleraas Centre for Quantum Molecular Sciences, Department of Chemistry, University of Tromsø–The Arctic University of Norway, 9037 Tromsø, Norway.

[b] Department of Chemistry, University of Tromsø–The Arctic University of Norway, 9037 Tromsø, Norway.

janakiray.vaitla@uit.no
kathrin.hopmann@uit.no

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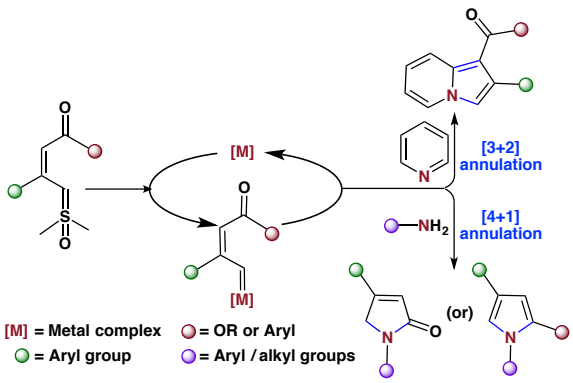

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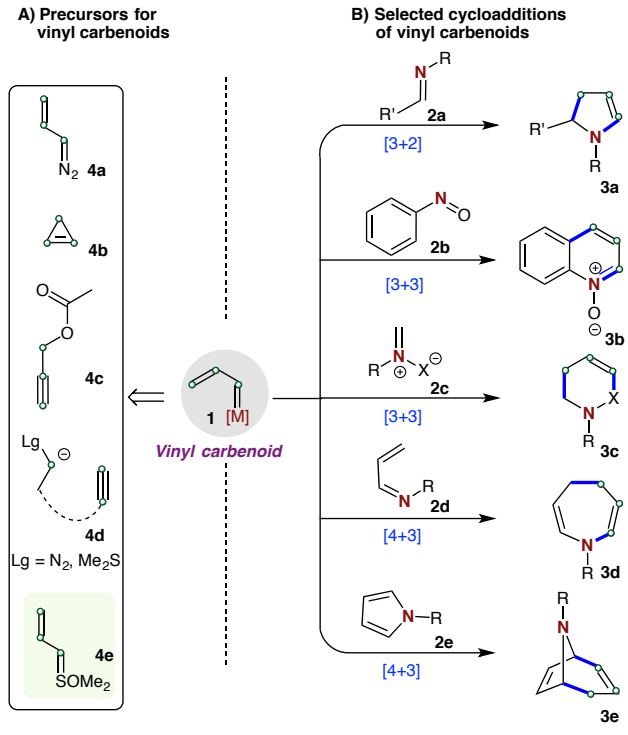
Abstract Sulfoxonium ylides have recently gained prominence as safe carbenoid precursors in metal-catalyzed reactions. The stability and reactivity of sulfoxonium ylides depends on the substitution of the ylide carbon. The reactivity of vinyl substituted sulfoxonium ylides is different and offers several advantages over known stabilized sulfoxonium ylides in the case of carbenoid transfer reactions. Herein, we provide an overview of early efforts in this area, with particular emphasis on our own recent development of sulfoxonium ylide-derived vinyl carbenoid transformations for *N*–Heterocycles.

Key words Sulfoxonium ylides, metal carbenoids, transition metal catalysis, insertion reactions, heterocycles

Introduction

N–Heterocycles are important structural frameworks for numerous natural products and pharmaceuticals.[1] Cycloaddition of vinyl carbenoids with different nitrogen-based dipole or dipolarophiles is a powerful strategy to access medium-sized *N*–heterocycles.[2] Due to the presence of an active carbenoid carbon with an adjacent C=C bond, vinyl carbenoid **1** acts as an efficient 3-carbon synthon in various synthetic transformations leading to diverse *N*-heterocycles (Scheme 1). Vinyl carbenoids can undergo [3+2] cycloaddition with imines **2a**,[3] [3+3] cycloadditions with nitrones **2b**[4] or azomethine imines **2c**,[5] and [4+3] cycloadditions with alkenyl imines **2d**[6] or pyrroles **2e**[7] (Scheme 1).

The vinyl diazo compounds **4a** are commonly used for the generation of vinyl carbenoids through elimination of nitrogen gas.[8] Since diazo compounds are considered to be potentially explosive for large scale applications,[9] the use of alternative strategies for vinyl carbenoid generation such as ring opening of cyclopropenes **4b**,[10] rearrangement of propargylic esters **4c**,[11] and the attack of alkynes by a nucleophilic entity containing a leaving group **4d**,[12] has attracted attention from the synthetic community (Scheme 1). This Synpact article focuses on the use of vinyl sulfoxonium ylides **4e** as precursors of **1**, as recently described by us.[13]



Scheme 1 Generation[8-12] and application[3-7] of vinyl carbenoids

Lately, the use of sulfoxonium ylides as carbenoid surrogates has been explored extensively.[14] Most of the sulfoxonium ylides are bench-stable crystalline compounds, do not produce gas, and are not explosive.[15] Moreover, sulfoxonium ylides possess high thermal stability when compared to diazo compounds.[16] Over the past few years, sulfoxonium ylide-based metal carbenoids have been applied in C–C, C–N, C–O, and C–S bonds formation reactions.[14b] Significantly, the iridium-catalyzed N—H insertion of carbenoids derived from sulfoxonium ylides has been optimized for production of drug candidates on an industrial scale.[17] Despite the promising applicability of sulfoxonium ylide-based carbenoids, a number of key challenges became apparent to us from reports in the field:

1) Sulfoxonium ylides can efficiently produce metal carbenoids with noble metals (Rh, Ir, Au, Pt, etc.), however, base metals (e.g. Cu, Fe) were reported to be unsuccessful.[18]

2) Carbenoid transfer reactions of sulfoxonium ylides other than α-keto sulfoxonium ylides remained scarce.[19]

3) Unlike diazo-derived metal carbenoids, the reactivity of metal carbenoids derived from sulfoxonium ylides had not been explored beyond insertion reactions.

4) In the presence of metals, sulfoxonium ylides could show undesired homocoupling even at room temperature.[20]

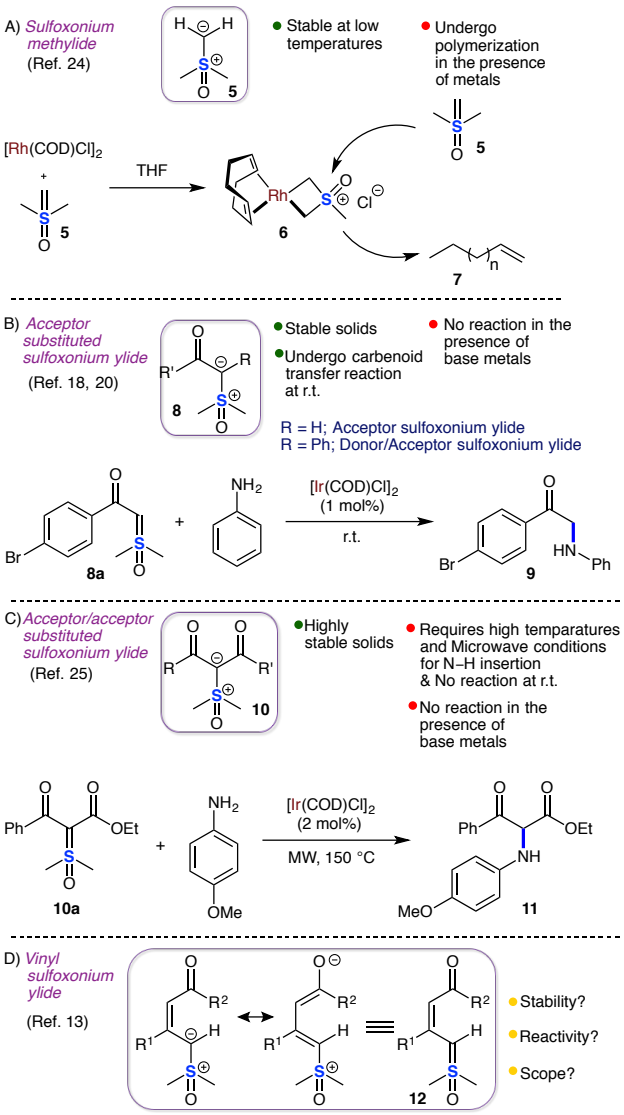
Clearly, further development of sulfoxonium ylides with a focus on inexpensive and abundant catalyst systems is highly desirable.[21] This objective led us to investigate the reactivity and application of vinyl sulfoxonium ylides **4e** in sustainable catalysis.[13] The present Synpacts article comprises *i*) a classification of sulfoxonium ylides based on their stability and reactivity, *ii*) a description of our working hypothesis for vinyl sulfoxonium ylides, *iii*) a review of our efforts in optimization of the reaction parameters, and *iv*) the scope of the resulting sustainable carbenoid transfer reactions.

Classification of Sulfoxonium Ylides

Sulfoxonium ylides are 1,2-dipolar compounds with a formal carbanion neighboring a positive sulfoxide group. Similar to diazo compounds,[22] the stability and reactivity of sulfoxonium ylides depends on the carbanionic character of the ylide; thus, electron-withdrawing groups (such as keto, ester or amide groups) stabilize sulfoxonium ylides.

Dimethyl sulfoxonium methylide (**5**) (known as the Corey–Chaykovsky reagent) is a moderately stable sulfoxonium ylide, due to the absence of stabilizing groups. A solution of **5** is unstable at r.t.; however, it is stable in THF solvent under inert atmosphere at 0 °C.[23] In 2012, Bruin and coworkers reported that **5** can be used as a one-carbon metal-carbene synthon (M=CH_2) in a homopolymerization reaction (Scheme 2A).[24] The mixing of the dimethyl sulfoxonium ylide **5** with a Rh(diene) complex lead to the rhodium complex **6**, which can function as active catalyst for the polymerization of **5** to **7**.

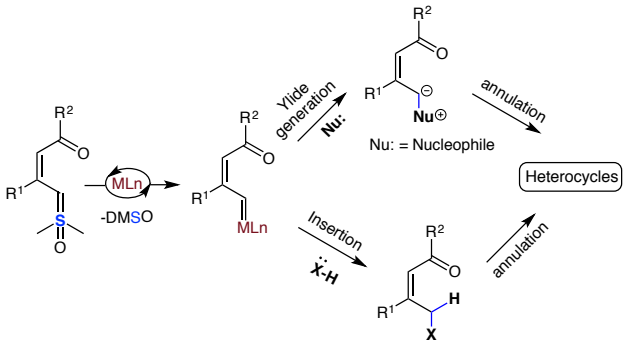
The groups of Baldwin and Mangion investigated the reactivity of acceptor stabilized sulfoxonium ylides **8** in carbenoid transfer reactions (Scheme 2B).[18, 20] The exposure of **8** to transition metal complexes afforded carbenoids, which underwent rapid N–H insertion to give **9**. A range of primary and secondary amines, thiols and alcohols could be applied, providing the insertion products in good yields. Recently, our group investigated the synthesis and reactivity of acceptor/acceptor stabilized sulfoxonium ylide **10** (Scheme 2c).[25] Although **10** provided good yields of a variety of N–H insertion products, the high stability of these ylides required elevated temperatures to proceed the reaction under Ir-catalysis. Under the same conditions, O–H insertion with phenols was unsuccessful. These observations indicated to us that the reactivity of sulfoxonium ylides could be tuned through the substituents on the ylide carbon.



Scheme 2 Classification of sulfoxonium ylides

In general, the generation and reactivity of the carbenoid can be expected to depend on the electronic nature of the Lewis acidic metal catalyst, the nucleophilicity of the ylide carbon and the leaving group (N_2, Me_2SO, Me_2S). In presence of strong electron-withdrawing groups, the electron density decreases at the carbanion of the ylide. As a result, highly active catalysts are required for carbenoid transfer reactions.[26] We envisioned that installation of a weak electron-withdrawing group at the carbanion of the sulfoxonium ylide could attenuate its stability, and thereby increase its reactivity. In this regard, we anticipated that Michael acceptor-stabilized sulfoxonium ylides **12** (Scheme 2D), which have an additional π-system between the ylide and the electron withdrawing group, could alleviate the strong stabilization at the ylide carbon.[27] If successful, such ylides could

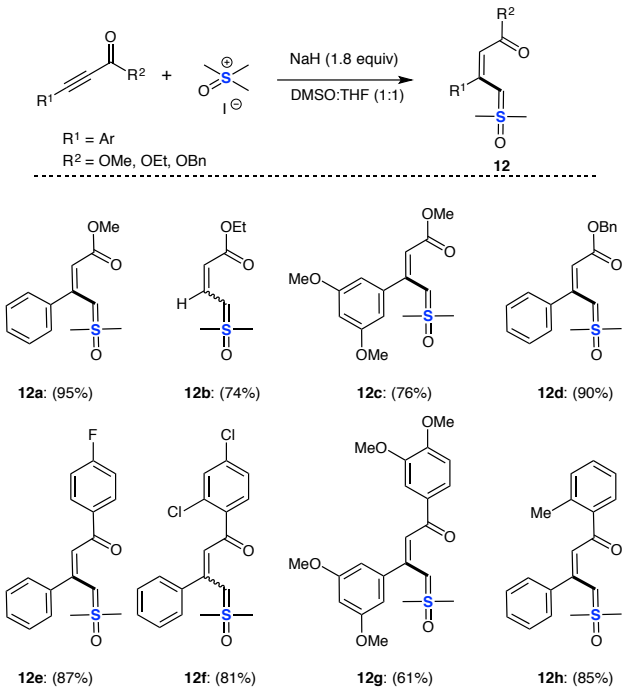
possibly afford vinyl carbenoids from less reactive metals, may be even base metals like iron. Iron is earth-abundant, relatively cheap, and nontoxic, making it an attractive alternative to other transition metals in catalysis, [28] however, iron-catalyzed vinyl carbenoid transformations had not yet been explored.[29] We speculated that the reactive vinyl carbenoid species could be trapped by nucleophiles or amines followed by cyclization to give heterocycles (Scheme 3).



Scheme 3 Our working hypothesis for generation and application of sulfoxonium-derived vinyl carbenoids.[13]

Synthesis of Vinyl Sulfoxonium Ylides

The vinyl sulfoxonium ylides **12** can be prepared by addition of dimethyl sulfoxonium methylide **5** (generated from trimethylsulfoxonium iodide with NaH) to aryl-substituted propiolates or ynones (Scheme 4).[30] With respect to the stability of **12**, ketone functional groups provide the more stable vinyl sulfoxonium ylides **12e-h** (bench stable solids) than ester functional groups, which give the less stable **12a-d** (stable at 2-8 °C).

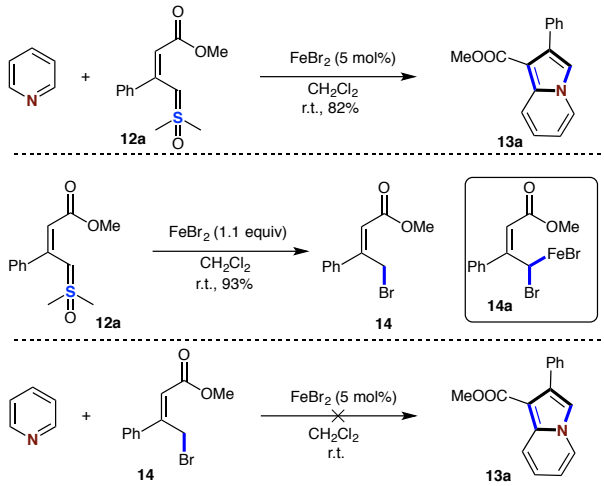


Scheme 4 Selected examples of vinyl sulfoxonium ylides synthesis.[13]

Preparation of **12** containing electron-poor aryl substituents (ester, cyano, nitro, keto substituted aryl groups) was unsuccessful. We speculate that the observed Z-selectivity of the vinyl sulfoxonium ylides is due to steric interactions between the aryl group (R^1) and the carbonyl functionality. In the case of **12b**, due to the absence of an aryl group, the reaction between dimethyl sulfoxonium methylide and ethyl propiolate led to a 1:10 mixture of *E*- and *Z*-diastereomers.

[3+2] annulation of Vinyl Sulfoxonium Ylides

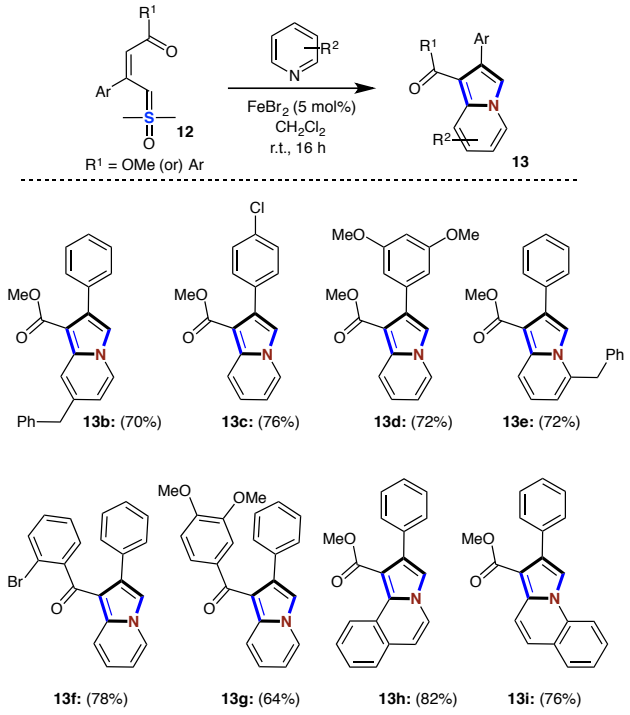
To verify the hypothesis in Scheme 3, we screened appropriate conditions by using vinyl sulfoxonium ylide **12a**, pyridine, and various iron-based catalysts [Hemin, (TPP)FeCl, $FeBr_2$, $FeCl_3$, $FeBr_3$, $Fe(OTf)_3$] for the carbenoid transfer reaction. After examining a range of iron and other metal (Cu, Ir, Ni, Zn, In) catalysts, we found that by using $FeBr_2$ (5 mol%) as the catalyst, the desired product **13a** was isolated in good yield (Scheme 5). Surprisingly, reaction of vinyl sulfoxonium ylide and stoichiometric amounts of $FeBr_2$ (1.1 equiv) gave allyl-brominated compound **14,** which led us to evaluate the mechanistic implications of intermediates involved in our working hypothesis (Scheme 3). Suspicious of the possible involvement of **14a** (via metal halo exchange of carbene)[31] in the catalytic cycle, we carried out several control experiments. Reaction of allyl bromide **14** with pyridine in the presence of 5 mol% of $FeBr_2$ gave pyridinium salt, but not indolizine **13a**. This result suggests that allyl bromide intermediate **14a** may not be involved in the catalytic cycle for formation of **13a** (Scheme 5). Despite the lack of strong spectroscopic evidence for the Fe-carbene, HRMS of the reaction mixture (using 5 mol% of $FeBr_2$) showed dimerized ylide, consistent with a mechanistic cycle involving a metal carbene.[32] The reaction afforded trace amount of **13a** in the presence of other Lewis acids such as $Fe(OTf)_3$, $In(OTf)_3$, $Zn(OTf)_2$, which indicated that the reactions proceed via a carbenoid pathway rather than simple Lewis acid activation. Based on additional computational studies performed in our group (vide infra, Scheme 10), we concluded that the observed allyl bromide **14** is generated from an off-cycle Fe-alkylbromide intermediate **14a**, which is in rapid equilibrium with the on-cycle Fe-carbene.[13]



Scheme 5 Investigation of intermediates involved in the carbenoid transfer reaction.[13]

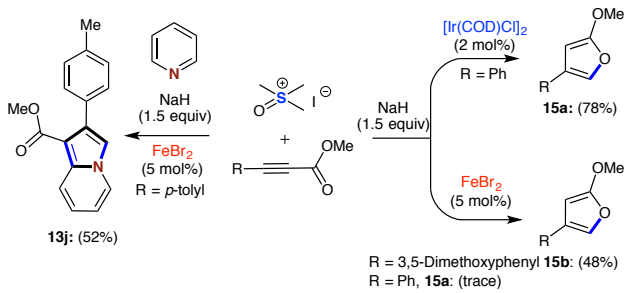
The substrate scope was subsequently examined under the $FeBr_2$-catalysed conditions (Scheme 6). A wide range of vinyl sulfoxonium ylides and pyridines were effectively converted to their corresponding indolizines **13**. Vinyl sulfoxonium ylides with ester functional groups were more reactive and afforded better yields of the corresponding indolizines (**13b**−**13e**) than

ketone functional groups (**13f–13g**). The reaction was also successful in affording pyrroloisoquinolines (**13h**, 82%), which is the framework of lamellarins (a family of anticancer marine alkaloids).[33] Also pyrroloquinolines could be formed (**13i**, 76%), which are known to exhibit biological activities, including caspase activation and apoptosis induction.[34]



Scheme 6 Selected examples of indolizine synthesis from vinyl sulfoxonium ylides.[13]

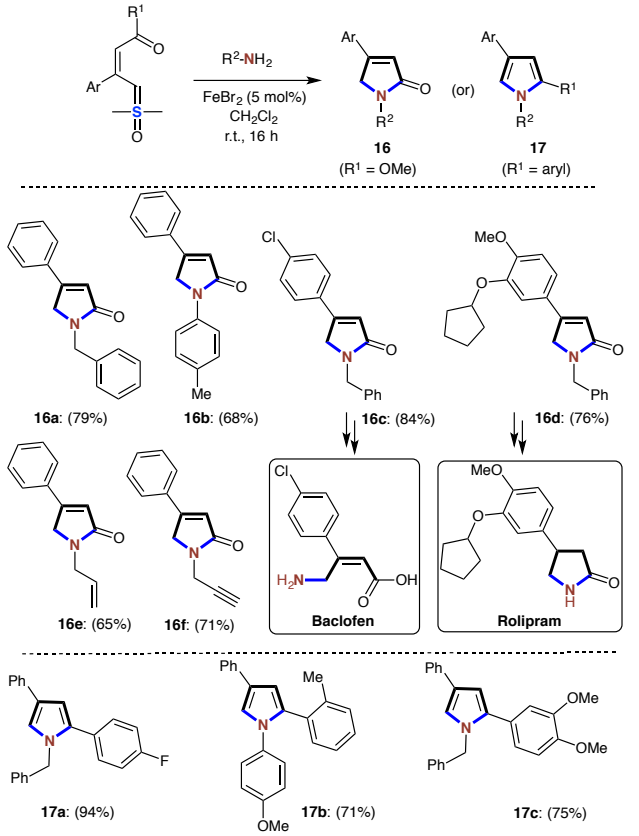
We then turned our interest to a three component synthesis of indolizine **13** (Scheme 7) directly from trimethyl sulfoxonium iodide and alkyne without isolation of the sulfoxonium ylide **12**. In order to test this approach, sequential addition of alkyne and pyridine to the in situ generated dimethylsulfoxonium methylide, followed by addition of the $FeBr_2$ catalyst, which indeed gave indolizine **13j**, albeit in moderate yield. Interestingly, in absence of pyridine, the reaction afforded furan **15a**, but only with $[Ir(cod)Cl]_2$ (2mol%) instead of $FeBr_2$ catalyst. Under $FeBr_2$ catalysis, furan formation only proceeded with alkynes containing EDG groups on aryl substituents. This result support our hypothesis that electron donating aryl groups on the ylide may make the ylide carbon more reactive in the presence of iron catalysts.



Scheme 7 One-pot generation and application of vinyl sulfoxonium ylides.[13]

[4+1] annulation of Vinyl Sulfoxonium Ylides

Next, to extend the scope of the vinyl carbenoids, we studied [4+1] annulation of vinyl sulfoxonium ylides with primary amines via N–H insertion of the carbenoid (Scheme 8). Despite the recent efforts on carbenoid insertion reactions using sulfoxonium ylides, to our knowledge, there had not been any report on N–H insertion under base metal catalysis.

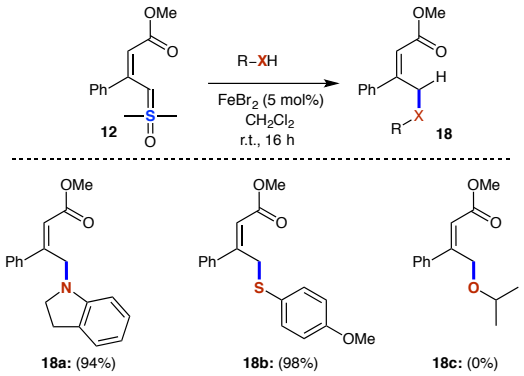


Scheme 8 Selected examples for synthesis of 3-pyrrolin-2-ones and pyrroles from vinyl sulfoxonium ylides.[13]

Reaction of primary amines with vinyl sulfoxonium ylides **12** having ester groups provided 3-pyrrolin-2-ones **16a–f** in the presence of $FeBr_2$. Remarkably, allyl and propargyl amines were also converted into the corresponding 3-pyrrolin-2-ones derivatives **16e** and **16f**, without detectable cyclopropanation of the unsaturated bonds by the metal carbenoid intermediates. Notably, the product 3-pyrrolin-2-one **16c** can be converted in 2 steps to Baclofen,[35] a pharmaceutical employed in treatment of spasticity, whereas 3-pyrrolin-2-one **16d** can be converted in 2 steps to Rolipram,[36] which is a potential antidepressant drug. Similarly, reaction of primary amines with ylides having ketone functional groups afforded 2,4-disubstituted pyrroles (**17a–c**), which are known to display a range of biologically important activities as HDAC inhibitors, dopamine D4 receptor modulators, and naturally occurring antibiotics.[37] Whereas a previous approach for generating pyrroles from sulfoxonium ylide-derived carbenoids required a precious metal catalyst at high temperatures,[38] this approach (Scheme 8) converted primary amines and vinyl sulfoxonium ylides into pyrroles using a cheap and sustainable catalyst under mild reaction conditions.[13]

To further explore the X–H insertion activity of the vinyl carbenoids, ylide **12** was treated with secondary amines and thiols in the presence of $FeBr_2$, which provided the corresponding insertion products in excellent yields (Scheme 9). However, the

reaction failed to give O—H insertion products, if ethanol or isopropanol were used as solvent.

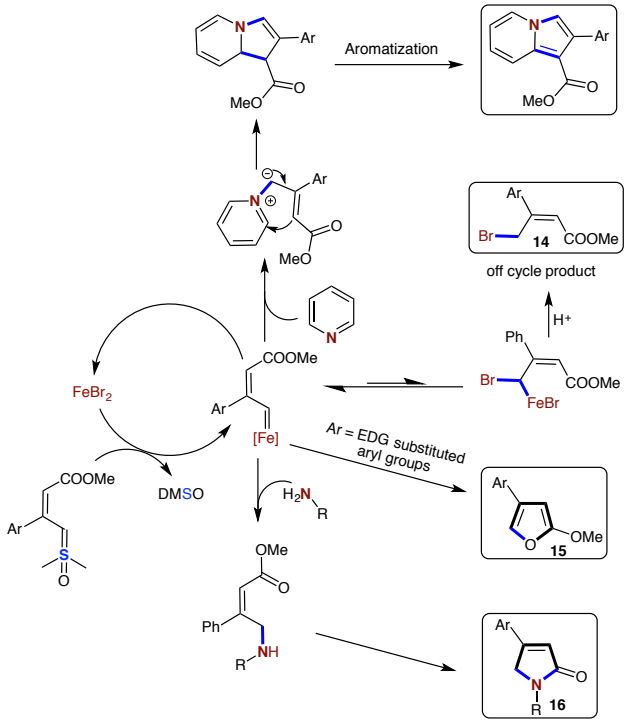


Scheme 9 Examples of X—H insertion reactions of vinyl sulfoxonium ylide.[13]

On basis of our experimental observations, computational studies and literature precedents,[39] we propose an overall mechanism for the observed carbenoid-mediated annulations (Scheme 10). Initially, $FeBr_2$ reacts with the vinyl sulfoxonium ylide and generates an iron carbenoid complex by losing DMSO. Then the vinyl carbenoid can undergo attack by nucleophiles, which can be bromide (from the $FeBr_2$ complex) or an added nucleophile, such as pyridine. According to DFT calculations,[13] Br attack on the carbenoid carbon has a low barrier and leads to an unstable metal–bromide inserted intermediate **14a**, which is an off-cycle species in rapid equilibrium with the iron-carbenoid complex. Attack of pyridine on the iron carbenoid generates pyridinium ylide, which undergoes intramolecular cyclization, followed by aromatization, leading to indolizine **13**.[40] Primary amines instead undergo N—H insertion of the carbenoid followed by annulation, to give 3-pyrrolin-2-one **16** or pyrroles **17**. In absence of external nucleophiles, the metal carbenoid can generate furan **15** via intramolecular oxygen attack on the metal carbene; this requires EDG substituted aryl groups to be successful under iron catalysis, but proceeds readily with iridium as catalyst.

Conclusion

In summary, we have discussed the background and rationale for our recent work on $FeBr_2$-catalyzed carbenoid transfer reactions of vinyl sulfoxonium ylides. The one pot generation and utilization of vinyl sulfoxonium ylides in carbenoid transfer reactions provides a facile strategy, which is more convenient than a multistep synthesis involving enal diazo carbenoids.[41] The reaction of vinyl sulfoxonium ylide-derived carbenoids with pyridines leads to indolizines via [3+2] annulation, whereas primary amines give 3-pyrrolin-2-ones and pyrroles via [4+1] annulation. The usefulness and versatility of this method was demonstrated through the synthesis of precursors for Baclofen and Rolipram. The synthetic strategy presented here is expected to have high impact on vinyl carbenoid chemistry and to find wide applications in organic synthesis.



Scheme 10 Our proposed mechanism for carbenoid transfer reactions.[13]

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Biosketches

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Janakiram Vaitla (Right) obtained his Ph.D. in 2015 from National Chemical Laboratory (NCL-CSIR, India) under the supervision of Prof. Dr. Ganesh Pandey, working on the total synthesis of bioactive complex natural products. He joined as a postdoc in the research group of Dr. Bayer and Dr. Hopmann at UiT – The Arctic University of Norway, where he started to work in the field of experimental investigation of selectivity-determining factors in transition metal-catalyzed reactions. He is currently working as a researcher in the same group and his current research focuses on the development of carbon dioxide conversion, homogeneous catalysis and sulfoxonium ylide derived metal carbenoids.
Annette Bayer (Middle) received a Dr. Scient. degree in Chemistry in 2002 from UiT – The Arctic University of Norway. After a postdoctoral research visit at Uppsala University, Sweden, she returned to UiT to start her independent career as Associate Professor in Chemistry in 2003. Her research interests cover natural product based drug discovery, development of β-lactamase inhibitors and method development for carbon dioxide conversion.
Kathrin H. Hopmann (Left) studied Chemistry and Molecular Biology at Aarhus University, Denmark. She obtained her PhD in 2008 from KTH - the Royal Institute of Technology in Stockholm, Sweden. In her post doc studies (2008-11, UiT Norway) and her subsequent researcher position, she has focused on chirality and enantioselective catalysis. Currently, she is working as Associate Professor at the University of Tromsø (UiT), Norway, where she is associated with the Centre of Excellence *Hylleraas Centre for Quantum Molecular Sciences*. Her research focuses on experimental and computational studies of transition metal-catalyzed processes, including conversion of CO_2 to higher value products.