Explaining Regiodivergent Vinylations with Vinylbenziodoxolones**

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**Abstract**: Vinylbenziodoxolones have recently been identified as efficient hypervalent iodine(III) reagents for electrophilic vinylations under transition metal-free conditions. Their unique reactivity allows synthesis of either internal or terminal alkenes, depending on the nucleophile class. This paper constitutes the first mechanistic investigation of VBX vinylations, and makes use of NMR studies, deuterium labelling and computations to rationalize the observed regio- and stereochemical outcome. Internal alkene formation in S-vinylation was found to proceed through the ligand coupling mechanism typical of diaryliodonium salts, whereas terminal alkene formation in P-vinylations took place via a phosphinous acid-coordinated VBX complex, which underwent concerted deprotonation and Michael-type addition. Subsequent base-assisted protonation and E2 elimination delivered the terminal alkene. The findings can be used to predict the regioselectivity in vinylations of other nucleophile classes.

**Introduction**

Alkenes are versatile building blocks in polymer chemistry, organic synthesis and drug discovery, and are biologically relevant functionalities common in pharmaceuticals.[1] Regiochemical control for selective synthesis of either the terminal or internal alkene, as well as stereochemical control of the E/Z-selectivity in internal alkenes is of paramount importance in many applications.[2] Hence, many regio- and stereoselective strategies to access alkenes have been developed over the past decades and generally involve transition metal-catalyzed cross coupling, C–H activation or metathesis.[2,3] With the increased focus on sustainable chemistry,[4] the development of transition metal-free, regio- and stereoselective alkenylations that proceed under mild conditions is highly important.[5]

Vinylbenziodoxolones (VBX) have recently been reported as novel hypervalent iodine(III) reagents sharing the same benziodoxolone core as the popular ethynylbenziodoxolones (EBX) and Togni reagents.[6] VBX compounds have already been employed as electrophilic vinylation reagents under transition metal-catalyzed, photocatalytic and metal-free conditions with a variety of nucleophiles (Scheme 1a).[7] VBX shows interesting reactivity under mild conditions without need for excess reagents, and tolerates a variety of functional groups. Core-substituted Me₂-VBX was reported to give improved results in some cases.[8] In parallel, the chemistry of β-heteroatom-substituted VBX reagents and their benziodoxole counterparts has also been developed.[9]

Methodology for transition metal-free vinylations from our lab highlights the intriguing reactivity of VBX reagents. The efficient, room temperature S-vinylation of thiols 2 resulted in formation of internal alkenes 3 with retention of the VBX alkene configuration (Scheme 1b).[8–10] To the contrary, metal-free P-vinylation of phosphine oxides 4 or H-phosphinates delivered terminal alkenes 5 with complete...
regioselectivity (Scheme 1b).\textsuperscript{[11]} Terminal alkene formation was also favored with a C-nucleophile.\textsuperscript{[9a]}

Literature reports of reactions with vinyl(aryl)iodonium salts, which are related iodine(III) reagents, indicate that various mechanisms can be operative under metal-free conditions. Vinylic S\textsubscript{1} or S\textsubscript{2} or reactions via alkylidene carbene intermediates are common, whereas Michael additions are less likely.\textsuperscript{[12]} Vinyldene carbene formation has also been proposed in reactions with alkynyliodonium salts\textsuperscript{[13]} and EBX.\textsuperscript{[14]} Mechanistic studies of the latter revealed that thiol alkynylation can proceed via ligand coupling or Michael-type addition depending on the electronics of the reagent.\textsuperscript{[14]}

To increase the utility of VBX as an efficient vinylating agent, tools to understand and predict the principles for product regiochemistry with other nucleophile classes.

**Results and Discussion**

**S-Vinylation of Thiols**

Earlier experimental observations were the starting point of this mechanistic study.\textsuperscript{[6]} Vinylation of S-nucleophiles led exclusively to internal alkenes 3 (E:Z ratio generally >20:1), indicating that VBX does not react through a vinylic S\textsubscript{2} mechanism\textsuperscript{[15]} (Scheme 1b). Alkenes 3 were formed also in the presence of radical traps. Furthermore, core-substituted Me\textsubscript{2}VXB proved superior to the standard VBX as formation of disulfide (ArS)\textsubscript{2} byproduct was suppressed.\textsuperscript{[9]}

On account of experimental observations, literature mechanisms with vinyliodonium salts\textsuperscript{[6a, 18]} and the reported mechanistic investigations of S-alkylations with EBX\textsuperscript{[10b, 14, 16]} we proposed three possible pathways leading to (E)-3a and 2-iodobenzoate (Scheme 2). Thiophenol (2a) is first deprotonated by BuOK, after which four-coordinated I–S intermediate A can form through ligand association (pathway I). This intermediate either undergoes direct ligand coupling to stereospecifically yield product (E)-3a (pathway 1a), or via release of the carbonate ligand to intermediate B (pathway 1b).

A direct nucleophilic attack on the α- or β-carbon of the vinyl moiety could also lead to product 3a, although the (E)-selectivity could be compromised through such routes. Pathway II depicts an α-attack of the thiolate to yield intermediate C, which could rotate to intermediate D followed by β-elimination to give (E)-3a. Finally, attack on the β-carbon would give intermediate E via pathway III. This could undergo a concerted elimination and 1,2-shift of either the phenyl or sulfide moiety to yield vinyl sulfide 3a directly (pathway IIIa), or stepwise elimination of iodobenzoate to yield alkylcarbene F, followed by a 1,2-shift (pathway IIIb). A concerted asynchronous alternative in between these two extremes is also possible.

We followed the reaction of VBX 1a with 2a in THF-d\textsubscript{8} by \textsuperscript{1}H-NMR to investigate the formation of any intermediates and to rationalize the observed influence of the base addition order.\textsuperscript{[17]} When 2a was mixed with BuOK in an NMR tube, signals of the thiophenolate were immediately observed and the vinylation started instantly when 1a was added. A similar NMR experiment with 1a and 2a in the absence of base showed no interaction between the two, supporting our assumption that the thiophenolate forms prior to S–I coordination. The reaction started upon addition of the base, and thiophenolate was the only intermediate observed in both experiments.

To gain further understanding of the mechanism, deuterium-labelled VBX reagents 1a-D\textsubscript{1} and 1a-D\textsubscript{2} were synthesized (Scheme 3a,b).\textsuperscript{[6a, 18]} Thiophenol 2b was employed as the model substrate to facilitate the NMR analysis of the reaction outcome. Reactions with 1a-D under the standard conditions resulted in product 3b-D as the only regioisomer, together with the corresponding disulfide by-products B and C.

![Scheme 2](Image)

Scheme 2. Mechanistic pathways for S-vinylation with VBX.
product in 11 % yield (Scheme 3c). The reaction with 1b-D₂ delivered product 3b-D₂ in moderate yield together with the disulfide byproduct (Scheme 3d). To reach these products through pathway II, complete selectivity in β-elimination from intermediate D rather than C would be needed, otherwise an E:Z mixture would be obtained.

DFT calculations were then performed at the M06-2X level of theory to investigate all three pathways. A low energy reaction profile was found for pathway Ia, starting with exothermic I–S bond formation to intermediate A’ (Figure 1). The long bond lengths between the S–I and S–Cu atoms, (3.04 and 3.17 Å respectively) as well as the 167° S–I–C₅₇ angle, are similar to the tilted intermediate found in S-alkynylation with EBX.[14a] Ligand coupling through transition state TS₁ leads stereospecifically to the vinylated product (E)-3a and iodobenzoate. The low energy of TS₁ (18.2 kcal mol⁻¹) is in accordance with the experimentally observed high reaction rate at room temperature. Pathway III was found to be higher in energy by 13.7 kcal mol⁻¹, as depicted in Figure S15. Intermediates B and C could not be located; the structures converged to intermediate A’, indicating that the reaction proceeds through pathway Ia. We also computed the reaction profiles with Me₂-VBX 1b, and o-Me-VBX 1c, which resulted in very similar energies (Figures S13–S14). The benefit of reagent 1b might be explained by the lower electrophilicity of the iodine compared to 1a, thus resulting in cleaner reactions without formation of disulfide byproduct.

**P-Vinylation of Phosphine Oxides**

Contrary to the S-vinylation, the P-vinylation of diarylphosphine oxides 4 led exclusively to terminal alkenes 5 (Scheme 1b), and preliminary mechanistic studies revealed that a radical pathway was unlikely.[11] A ligand coupling mechanism can be ruled out, as this would lead to the internal alkene product. With inspiration from previous P-functionalizations with vinyl,[10] and alkynylidonium salts[20] and recent mechanistic studies with EBX[14b,21] we proposed five pathways leading to terminal alkene 5a and 2-iodobenzoate (Scheme 4). In the presence of base, diphenylphosphine oxide (4a) and VBX 1a can react through an ω-attack to give intermediate G, which could undergo double 1,2-shifts to reach the terminal alkene 5a via carbene H (pathway IV). Alternatively, the regioselectivity can be rationalized through a phospha-Michael-type reaction, which is a versatile and powerful tool for P–C bond formation.[22] Pathway V depicts a direct β-attack, yielding anionic intermediate I, which can form 5a through two alternative pathways. Due to the high leaving group ability of the iodine(III) moiety, I could decompose to carbene J, which would yield 5a through a 1,2-hydride shift. Alternatively, anion I could undergo a concerted 1,2-hydride shift and elimination to 5a. Finally, protonation of intermediate I would give intermediate K with subsequent base-mediated β-elimination to 5a. Alternatively, the four-coordinated I–P complex L forms prior to intramolecular Michael-type addition to yield intermediate I (pathway VI).

Phosphine oxide 4a exists in equilibrium with the more reactive and nucleophilic phosphinic acid 4a’,[23] which can participate in Michael additions under basic conditions.[22a,24] Pathway VII depicts deprotonation of 4a’ followed by reaction with 1a to yield I–O bonded intermediate M, which is well aligned for a Michael-type addition to give I. In pathway VIII, 4a’ and 1a instead form the weakly I–O

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coordinated intermediate N. This species could either undergo simultaneous deprotonation and \( \beta \)-attack to I, or undergo a (3+2) cycloaddition to intermediate K, followed by base-mediated \( \beta \)-elimination to 5a.

NMR experiments in THF-\( d_8 \) were carried out to observe any reaction intermediates.\(^{[13]} \) In the absence of VBX 1a, no reaction was observed between DBU and 4a. When 1a was added, product 5a started forming immediately, together with an intermediate that could later be identified (see below). Likewise, no adduct could be observed upon mixing 1a and 4a in the absence of DBU. These results indicate a simultaneous reaction between 1a, 4a and DBU rather than initial deprotonation of the nucleophile.

Deuterium-labelled VBX reagents 1a-D and 1a-D\(_2\) were utilized in a series of experiments to study whether any rearrangement or protonation took place (Scheme 5). The reaction of 4a with 1a-D delivered monodeuterated products 5a-D in a 7:1 Z:Z mixture as the only vinylation products observed (Scheme 5a). To verify these results, the reaction was repeated with \( p \)-methoxy-substituted phosphine oxide 4b and 1a-D, which simplified the NMR analysis (Figure S15–S17).

The vinylation of 4a with 1a-D\(_2\) resulted in a 1:1 mixture of 5a-D isomers and no di-deuterated products were observed (Scheme 5b). A crossover experiment was performed with 1a-D\(_2\) and 1d, in which the expected products 5a-D and 5c were formed together with a trace amount of 5c-D (Scheme 5c). Finally, deuterated phosphine oxide 4a-D was employed with VBX 1a, which resulted in the expected product 5a as major product, with some deuterium incorporation to 5a-D, now with the (E)-isomer as major isomer (Scheme 5d).

Based on these results, we propose that the reaction does not proceed through carbenes H or J, as these intermediates would give 5a-D\(_2\) with reagent 1a-D\(_2\). Likewise, a concerted hydride shift and elimination from I is ruled out. The results from Schemes 5c, d indicate that the base is involved in protonation, which aligns well with pathways V–VIII proceeding through intermediates I and K.

These pathways were analyzed in more detail to rationalize the observed stereoselectivity in formation of 5a-D (Scheme 6). The reaction of phosphine oxide 4a with 1a-D would generate anionic intermediate 1-D, which could either be protonated from above or below by DBU–H.
reach two diastereomeric intermediates. Rotation around the single bond to obtain anti-periplanar alignment followed by β-elimination yields (Z)-5a-D and (E)-5a-D, respectively. With the assumption that anti-elimination (E2) is favored over syn-elimination in this system, the observed high (Z)-selectivity in Scheme 5a indicates that protonation of 1-D preferentially takes place from the same face as the phosphorous addition. This is also in agreement with the opposite stereoselectivity observed in Scheme 5d, where +DBU-D is used in the protonation step. The formation of 5a as the major product in that reaction indicates that protonation with +DBU–H, which forms in the E2-elimination, is much faster than with +DBU–D. This is expected as a competitive proton/deuterium transfer should display a significant primary kinetic isotope effect.

DFT calculations were then performed to investigate the most plausible mechanisms in Scheme 4 based on the experimental observations. The lowest energy pathway was found to follow pathway VIII in Scheme 4, with isomerization of phosphine oxide 4a to phosphinous acid 4a’ followed by formation of uncharged O–I intermediate $N'$ with a hydrogen-bonded DBU molecule (Figure 2, blue pathway). Deprotonation proceeds simultaneously with the Michael addition via 5-membered TS$_1$ (20.5 kcal mol$^{-1}$) as the rate determining step to give anionic intermediate I. Protonation by +DBU–H preferentially takes place from above, to yield syn-K' (Figure S25), which rotates to anti-K'. As expected, subsequent E2-elimination is favored over the corresponding β-elimination from syn-K', (Figure S26) and provides 5a and 2-iodobenzoate. A direct (3+2) cyclo-
addition from $\mathbf{N}$ to $anti-\mathbf{K}$ was found to have a TS energy of 40.7 kcal mol$^{-1}$ (Figure S31).

A low-energy pathway was also found roughly following pathway VI, where charged O–I intermediate $\mathbf{M}'$ is formed from phosphine oxide $4\mathbf{a}$ and $1\mathbf{a}$ via $\mathbf{T}_\mathbf{S}_\mathbf{e}$ (Figure 2, black pathway). Coordination to the protonated base $\mathbf{DBU-H}$ stabilizes the partial negative charge on the oxygen. The mechanism proceeds by Michael-type addition via transition state $\mathbf{T}_\mathbf{S}_\mathbf{I}$ (26.9 kcal mol$^{-1}$) to anionic intermediate $\mathbf{I}$, after which the reaction proceeds as above. Computations of the other pathways from intermediate $\mathbf{I}$ in Scheme 4, including 1,2-hydride shift or carbene formation, revealed high-energy intermediates that are discussed in Figure S27 and S28.

Two ligand coupling pathways were investigated to support the observed complete regioselectivity for terminal over internal alkene product. The latter product could be formed through a 4-membered ligand coupling TS from O–I intermediate $\mathbf{M}'$ (Figure S23), or a three-membered ligand coupling TS via intermediate $\mathbf{L}$ (Figure S30), both with considerably higher TS energies than $\mathbf{T}_\mathbf{S}_\mathbf{I}$ (Figure 2).

Further investigations were performed to support the formation of intermediate $\mathbf{K}$. Upon shorter reaction time, the synthesis of $5\mathbf{a-D}$ from $4\mathbf{a}$ and $1\mathbf{a-D}$, was accompanied by isolation of another product, which was identified as compound $6\mathbf{D}_2$ (Scheme 7). This finding was highly unexpected, as iodine(III) compounds with alkyl ligands are generally unstable.[25]

Compound $6\mathbf{D}_2$ could be converted to product $5\mathbf{a-D}$ upon treatment with DBU, which supports that $\mathbf{K}$ is an intermediate in the reaction. Upon kinetic-NMR studies of reactions of $4\mathbf{a}$ with $1\mathbf{a}$, the corresponding intermediate $6\mathbf{H}_2$ could be detected in minor amounts (Figure S20). The varying $Z:E$ ratios obtained for $5\mathbf{a-D}$ can be explained by different selectivity in protonation of I–D vs. I–D$^-$ from above or below, as the energy difference for syn- vs anti-elimination is much higher.

The mechanistic differences between VBX vinylations of thiols and phosphorus nucleophiles can be rationalized by invoking the preferential formation of a 5-membered TS over a 3-membered TS, which is generally required in ligand coupling mechanisms. This is in line with our previous findings in arylation of enolates with diaryliodonium salts, where a 5-membered pathway via an I–O intermediate was preferred over a 3-membered ligand coupling TS from the corresponding I–C intermediate.[26] Based on this conclusion, other ambident nucleophiles would also be expected to coordinate to the iodine with subsequent Michael-type attack to yield terminal alkenes, whereas mono-nucleophilic species should give internal vinylation products. This is in line with the C-vinylation of nitrocyclohexanone, which gave the terminal product as the major regioisomer.[34] Strong nucleophiles could prefer I–Nu coordination followed by ligand coupling or $\alpha$-attack to give the internal product, as observed in vinylation of in situ-generated sulfenate anions.[34] The possibility to control the regioselectivity by modulating the nucleophile structure should be of great interest in organic synthesis, and our continued studies of controlling VBX reactivity will be reported in due time.

**Conclusion**

A combined experimental and theoretical study of transition metal-free vinylations with the recently discovered hypervalent iodine(III) reagent VBX has revealed two different pathways leading either to the internal or the terminal alkene. Deuterium-labelling studies and computations support that the S-vinylation of thiols proceeds through deprotonation followed by a ligand coupling to provide the experimentally observed internal alkenes with retained $E$-configuration. The P-vinylation of diarylphosphine oxides instead begins with I–O coordination of the corresponding phosphinous acid to VBX. A simultaneous deprotonation and Michael-type addition then gives an anionic intermediate, which is transformed to the terminal alkene through a base-assisted protonation and E2 elimination. A general regioselectivity trend for VBX vinylations under metal-free conditions is predicted, where ambident nucleophiles will deliver terminal alkenes, whereas monodent or strong nucleophiles will provide internal alkenes.

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**Conflict of Interest**

The authors declare no conflict of interest.

**Data Availability Statement**

The data that support the findings of this study are available in the supplementary material of this article.
Keywords: Alkenes · Density Functional Calculations · Deuterium Labelling · Hypervalent Compounds · Regioselectivity

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