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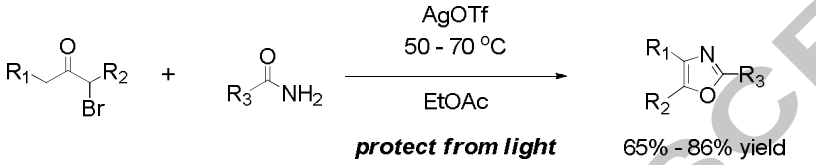

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Synthesis of 2, 4- and 2, 4, 5- Substituted Oxazoles via a Silver Triflate Mediated Cyclization

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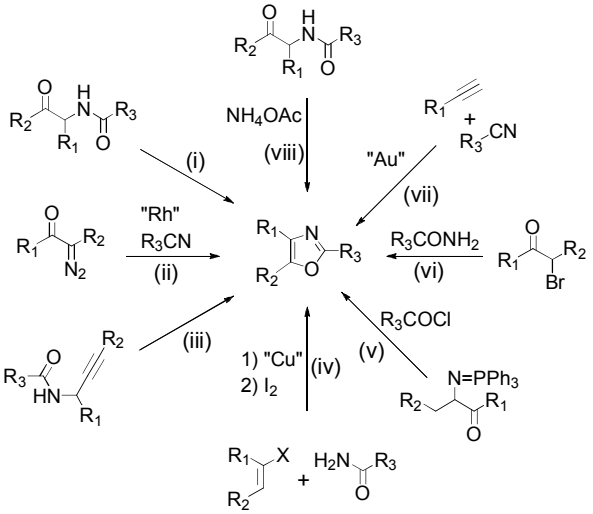
ABSTRACT

2, 4- and 2, 4, 5- substituted oxazoles were prepared from a broad range of bromo-ketones and amides in high yield and purity.

Substituted oxazoles are useful synthetic intermediates found readily in natural products[1] such as Diazonamide A and Leucamide A as well as in several modern pharmaceuticals. There are numerous ways to construct oxazoles but most methodologies are multi-step and/or require harsh reaction conditions.[2] More recent techniques are milder but of limited scope as they tend to require expensive catalysts and/or non-trivial syntheses of key starting materials. Therefore, new methodologies to construct oxazoles from readily available starting materials remain a significant unmet need. Common methods used for the synthesis of oxazoles depicted in Scheme 1 include the Cornforth and Cornforth protocol[3] as well as (i) The initial[4] and modified[5] Robinson-Gabriel synthesis which involves an intramolecular cyclization of an α-acylaminoketone onto a carbonyl followed by dehydration (ii) A rhodium catalyzed synthesis of oxazoles[6a] *via* the reaction of nitriles[6b-c] or amides[6d-e] with α-diazo-β-keto-carbonyl compounds (iii) Cycloisomerization of propargylamides[7-9] (iv) Copper/iodine catalyzed tandem oxidative cyclization[10] (v) The aza-Wittig reaction of vinyliminophosphoranes and acylchlorides[11] (vi) Thermal[12] promoted cyclization of a bromo-ketone and a primary amide (vii) Intermolecular gold catalyzed alkyne oxidation[13] (viii) The Davidson Oxazole synthesis[14] which is the reaction of α-acyloxylketones with ammonium acetate as well as copper catalyzed oxidative cyclization of enamides,[15] transition metal catalyzed cross-coupling,[16] direct C-H arylation/alkenylation,[17] and many others.[18]



Scheme 1. Common methods to construct oxazoles

Cyclization of bromo-ketones (vi, Scheme 1) was attractive to us as we were searching for a reliable and cost effective method to synthesize 2, 4- and 2, 4, 5- substituted oxazoles in high yield and purity. Attempts to generate oxazoles *via* a thermal cyclization were low yielding due to poor thermal stability of the bromo-ketone starting material at the temperatures required for cyclization (>100 °C) especially at larger scales. Our need for an efficient method for the large scale preparation of substituted oxazoles led us to the work of both Ritson[19] and Hoffmann [20] who independently determined that silver salts could promote cyclization. Ritson utilized silver salts to synthesize substituted

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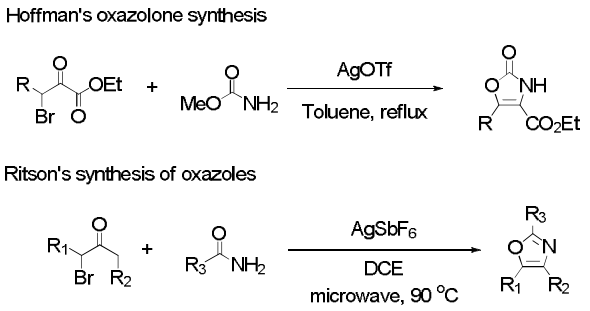
oxazoles (1 equiv $AgSbF_6$, DCE, microwave irradiation, 90 °C, 81 - 100% yield) while Hoffman generated oxazolones from bromo-keto-esters and methylcarbamate (1 equiv AgOTf, toluene, reflux, 30 - 68% yield) as shown below in Scheme 2. The limited availability of $AgSbF_6$, toxicity and carcinogenicity of dichloroethane, generation of stoichiometric quantities of silver bromide waste, serious concerns about the thermal stability of bromo-ketones, and a strong desire to completely eliminate microwave irradiation, led us to deem Ritson's method unsuitable for our purposes, requiring the identification of an alternate technique.



Scheme 2. Silver promoted cyclizations

Hoffman's[20] conditions did not require microwave irradiation and were thus more amenable to larger scale applications. We were pleased to find that Hoffman's approach could also be used to generate substituted oxazoles albeit in low yield (< 20%). Better results were achieved when DCE was used as solvent and the reaction mixture was protected from light, however, the yield was still low (~40%) and the isolation problematic.[21] Further investigation indicated that these difficulties could be traced back to poor stability of the bromo-ketone at the temperatures needed to initiate cyclization and the use of chlorinated solvents (dichloromethane, dichloroethane, or chloroform). Silver triflate reacts with chlorinated solvents (DCM, DCE, or $CHCl_3$) to instantly produce a yellow paste (< 2 min). Charging additional solvent was not beneficial leading us to conclude that chlorinated solvents are not compatible with silver triflate and therefore should be avoided for these types of reactions. It is further postulated that chlorinated solvents react preferentially with silver triflate during the cyclization to generate numerous reactive species which produce large amounts of unidentified impurities and ultimately produce oxazoles in low yield and purity after isolation. A subsequent solvent screen identified both ethyl acetate and toluene as suitable solvents for this transformation. All other solvents (CPME, MiBK, diglyme, DME, dioxane, THF, 2-Me-THF, dimethylcarbonate, ACN, DMF, DMA, DMSO, NMP) produced oxazoles in low yield and purity.

Optimal reaction conditions were identified for the synthesis of oxazole **3** in ethyl acetate by examining the effect of stoichiometry, temperature, and ambient light on the process. As reported in Table 1, excellent conversion to oxazole **3** was achieved in all instances while those examples conducted at lower (ambient) temperature or those employing an equimolar ratio of reagents (**1**:**2**:AgOTf) were slower to react (entries 1, 2). Quantitative conversions could be achieved with a large excess (2 equiv) of either the amide (**2**) or silver triflate but the subsequent workup and isolation of oxazole **3** was more difficult (entries 5, 7). Ambient light must also be excluded to prevent the reaction from stalling. It is important to note that longer reaction times are required for this process than for Ritson's approach (4+h vs 2h). However, we felt this was less important due to the elimination of microwave irradiation and dichloroethane, both of which are undesirable at industrial scales. Additionally, the lower temperatures required to initiate cyclization enable less stable substrates to be used which in turn increases the number of substituted oxazoles that can be synthesized using this methodology.

As discussed above, excess amide and silver triflate is required for best results and therefore an efficient means to purge excess reagent needed to be identified. The amide could easily be removed *via* a series of aqueous washes while the removal of excess silver triflate was more difficult. As expected, attempts to remove excess silver triflate using an extractive workup failed due to uncontrolled precipitation of the silver salts during the extractions. These issues were addressed by treating the reaction mixture with aqueous brine (4-24h, 20 °C) to convert the excess silver triflate into insoluble silver chloride which after filtration quantitatively removed all of the silver salts (AgBr and AgCl).[22] ICP analysis of the isolated product for silver confirmed that all silver had been purged using this technique.[23] Although the stoichiometric quantities of silver waste produced by this process are not ideal, it is our belief that this waste could be recycled to minimize both the cost and environmental impact of the process at industrial scales.

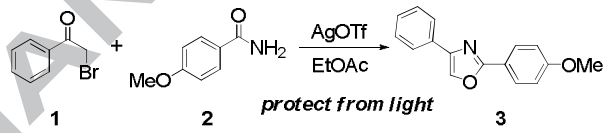


Table 1
Identification of optimal reaction conditions

Entry	Amide (Equiv)	AgOTf (Equiv)	Temp (°C)	Time (hrs)	Conversion (%PAR)
1	1.0	1.0	60	4	93
2	1.25	1.25	20	21	95
3	1.25	1.25	50	4	97
4	1.0	1.25	50	4	94
5	1.5	1.5	60	3	99
6	1.25	1.5	60	3	98
7	2.0	2.0	60	1	99

Alternatives to silver triflate such as silver tetrafluoroborate, silver acetate, or silver oxide, were also explored. As shown in Table 2, silver tetrafluoroborate could also be used for this transformation (**4a**, entry 6) but the reaction rate was slightly slower than with silver triflate. All other silver salts investigated failed to produce significant amounts of oxazole **4a** (entries 1-5), as did copper triflate.[24] In the case of silver acetate, bromide displacement with acetate rather than cyclization was the primary outcome. Chloro-ketones may also be used for this transformation but the reaction rate was significantly slower (**4b**, entry 9) at the temperatures investigated. Lastly, substrates containing chelating groups, such as pyridine, also reacted but require a larger excess of silver triflate (2.25 equiv) and a slight increase in temperature (70 °C) to push the reaction to completion (**4c**, entry 10).

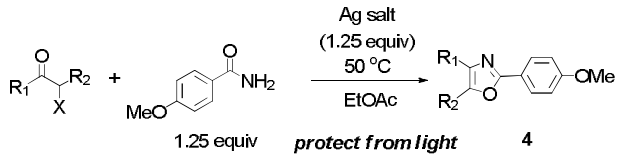


Table 2
Investigation of the effect of silver source and halide

Entry	R_1	R_2	X	Silver Salt	Time (hrs)	Yield[b] (%)	Pdt
1	Ph	H	Br	AgOAc	6	0	
2	Ph	H	Br	AgBr	6	0	
3	Ph	H	Br	AgO	24	1	
4	Ph	H	Br	$AgNO_3$	6	2	**4a**
5	Ph	H	Br	Ag_2CO_3	6	0	
6	Ph	H	Br	$AgBF_4$	6	95	
7	Ph	H	Br	AgOTf	4	97	
8	Me	Me	Br	AgOTf	4	98	**4b**
9	Me	Me	Cl	AgOTf	24	94	
10	4-pyr[a]	H	Br	AgOTf	17	85	**4c**

[a] 2.25 equiv AgOTf and 70 °C required
[b] Solution yield as determined by HPLC

The scope of this reaction was then challenged by evaluation of several bromo-ketones and primary amides of varying reactivity utilizing the optimal reaction conditions identified for bromo acetophenone (1.25 equiv amide, 1.25 equiv AgOTf, 50 °C, EtOAc) and these results are depicted in Table 3. Good results were achieved with the majority of the bromo-ketones and amides evaluated. Exact conditions varied somewhat depending on several factors but generally speaking substrates with thermal stability issues were run at lower temperatures and extended reactions times while those substrates containing chelating groups (e.g. pyridyl-) required slightly higher temperatures to achieve complete conversion. The desired 2, 4- or 2, 4, 5-substituted oxazoles were generated from both electron rich and electron poor amides including aromatic, heteroaromatic, cyclic, and heterocyclic substrates in moderate to good yield (64-85%) and high purity (>95%). We were gratified to note that several sensitive functionalities such as cyclopropyl (oxazoles **9** & **16**), vinyl (oxazole **7**), or cinnamyl (oxazoles **8** & **13**), and even aryl halides (oxazoles **5**, **10**, **11**, & **20**) are well tolerated under the reaction conditions albeit in lower yield (45-65%). Unfortunately, when bromo-ketones such as desyl bromide or bromoethyl pyruvate were employed, the reaction was unsuccessful (oxazoles **21**-**26**). With these more reactive substrates, poor conversion to the desired oxazaole products were obtained due to the formation of numerous unidentified byproducts. Attempts to tune the reactivity through the use of the more stable chloro-ketone, alternative silver salts (AgO) or even copper triflate were unsuccessful.

In conclusion, we have developed a reliable silver triflate promoted cyclization reaction that is suitable for the large scale preparation of 2, 4- and 2, 4, 5- substituted oxazoles from a variety of halo-ketones and primary amides. An efficient isolation/workup was also developed to remove excess amide *via* a series of aqueous washes and silver triflate through the implementation of an aqueous brine treatment and removal of the resulting silver salt byproducts by filtration. We anticipate that this improved process, which no longer requires chlorinated solvents or microwave irradiation, will find extensive industrial applications and further benefit from the utilization of a readily available, non-toxic silver source that can be recycled after use. This methodology has broad applications to industry and enables highly functionalized 2, 4- and 2, 4, 5- oxazoles to be synthesized from easily accessible starting materials producing oxazoles in both high yield and purity.

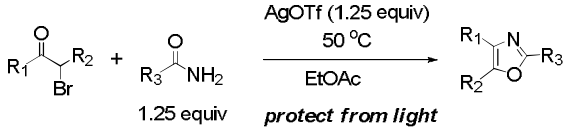
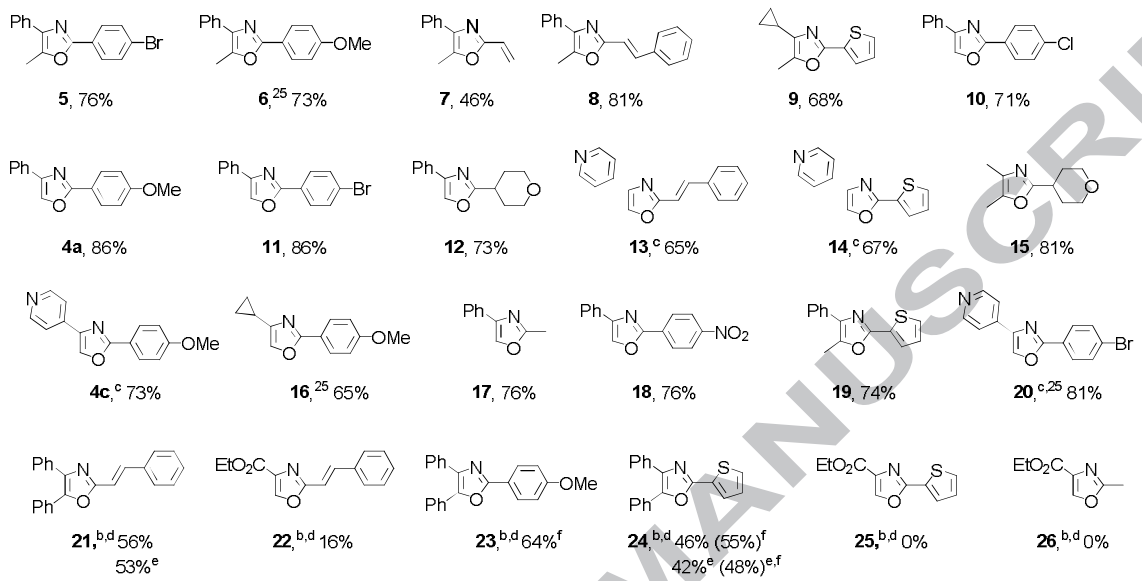


Table 3
Full scope of AgOTf mediated oxazole synthesis[a]



[a] Isolated yields unless otherwise specified
[b] Solution yield as determined by HPLC
[c] 2.25 equiv AgOTf, 70 °C, overnight required for complete conversion
[d] Numerous unidentified impurities also generated
[e] 22°C reaction temperature
[f] Slurry of AgOTf added to solution of amide and bromo-ketone over at least 30 minutes

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21. Polymeric impurities and black tar was produced under these reaction conditions
22. Alternatively, an aqueous potassium bromide solution can be used in the workup to convert the unreacted silver triflate to silver bromide. The silver bromide can then be recovered by filtration and recycled.
23. Typical amounts of residual silver were 10-20ppm (ICP analysis)
24. Copper triflate likely also promotes cyclization but likely requires significantly higher temperatures (>100 °C) in order to achieve a reasonable reaction rate.
25. *General procedure for oxazole cyclization:* A mixture of bromo-ketone (0.6 g, 2.9 mmol), amide (0.55 g, 3.6 mmol, 1.25 equiv), and silver triflate (0.9 g, 3.6 mmol, 1.25 equiv) in ethyl acetate (4 mL) was heated to 50 – 70 °C. After the reaction was deemed complete by HPLC analysis, the mixture was cooled to ~20 °C and diluted with ethyl acetate (3 mL). A solution of sat'd NaCl (3-4 mL) was added and the mixture stirred at ~20 °C for at least 4h. The silver salts (AgBr & AgCl) are removed by filtration and the resulting biphasic solution transferred to a separatory funnel and the layers separated. The organic layer is then washed with water (4 mL), 5% $NaHCO_3$ (4 mL), 1N HCl (4 mL), and water (4 mL). The organic layer is concentrated to dryness and the residue purified by flash column chromatography (5% EtOAc/hexanes) to obtain pure oxazole product. 2-(4-methoxyphenyl)-5-methyl-4-phenyloxazole (**6**) ^{1}H NMR (300 MHz, DMSO-d6) δ 2.61 (s, 3H), 3.90 (s, 3H), 7.12 (m, 2H), 7.37 (m, 1H), 7.48 (m, 2H), 7.75 (m, 2H), 7.95 (m, 2H). ^{13}C NMR (75 MHz, DMSO-d6) δ 12.16, 55.73, 114.91, 119.89, 126.67, 127.65, 127.79, 129.05, 132.26, 135.02, 144.16, 158.88, 161.25. 4-cyclopropyl-2-(4-methoxyphenyl)oxazole (**16**) ^{1}H NMR (300 MHz, DMSO-d6) δ 0.8 – 0.9 (m, 4H), 1.85 (sextet or m, 1H), 3.83 (s, 3H), 7.06 (m, 2H), 7.8 – 7.9 (m, 3H). ^{13}C NMR (75 MHz, DMSO-d6) δ 6.79, 7.26, 55.70, 114.16, 129.69, 134.09, 143.58, 163.19, 167.36. 2-(4-bromophenyl)-4-(pyridine-4-yl)oxazole (**20**) ^{1}H NMR (300 MHz, DMSO-d6) δ 7.38 (m, 1H), 7.8 – 8.1 (m, 6H), 8.6 (m, 1H), 8.8 (s, 1H). ^{13}C NMR (75 MHz, DMSO-d6) δ 120.18, 123.71, 124.85, 128.44, 132.71, 137.70, 138.66, 142.17, 150.04, 161.32.

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