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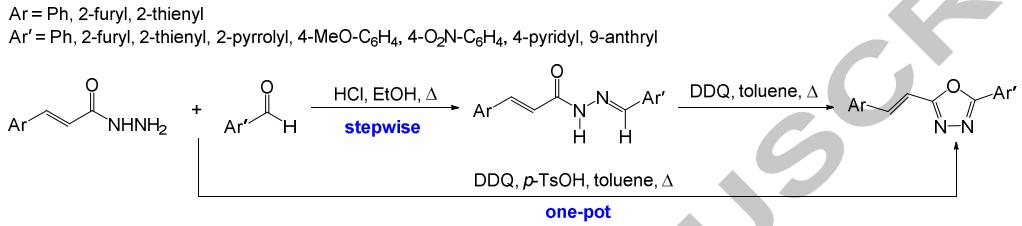

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ABSTRACT

A series of novel 5-aryl-2-(2-arylethenyl)-1,3,4-oxadiazoles were synthesized by the 2,3-dichloro-5,6-dicyanobenzoquinone (DDQ) promoted oxidative cyclization of *N'*-(arylmethylidene)-3-arylacrylohydrazides. A facile and efficient one-pot protocol using the starting 3-arylacrylohydrazides and aromatic aldehydes is also reported. Short reaction times, high yields, a simple work-up procedure and the possibility of regeneration of the oxidizing agent make the reported method a promising alternative for the synthesis of 2,5-diaryl-1,3,4-oxadiazoles.

1. Introduction

Among five-membered aromatic heterocycles, 1,3,4-oxadiazoles have attracted considerable interest in recent decades due to their broad spectrum of pharmaceutical and biological activities.[1] Many compounds containing this scaffold display antimicrobial,[2] antiviral,[3] anti-inflammatory,[4] antihypertensive,[5] analgesic,[6] anticonvulsant,[7] antidiabetic[8] and antitubercular activities.[9] They have also attracted attention in medicinal chemistry as potential therapeutic agents for the treatment of cancer[10] and HIV infections.[11] In addition, 1,3,4-oxadiazole derivatives have been utilized as bioisosteric replacements for carboxylic acid,[12] ester[13] and amide[14] functional groups in biologically active compounds. 2,5-Disubstituted 1,3,4-oxadiazoles have found further application in agriculture as plant growth regulators[15] and crop protection agents.[16] These heterocyclic molecules have also been used to produce heat-resistant polymers, optical brighteners, blowing agents and corrosion inhibitors for mild steel.[17] π-Conjugated arrangements possessing the 1,3,4-oxadiazole moiety, such as 2-(4-biphenylyl)-5-(4-*tert*-butylphenyl)-1,3,4-oxadiazole (PBD), are well known as electron conducting and hole blocking (ECHB) materials in polymer and material science due to their electron deficient nature, high photoluminescence quantum yield as well as both thermal and chemical stability.[18] Therefore, they have been applied as monomers in the production of fluorescent emitting layers for organic electronics, particularly in photovoltaic cells, organic light-emitting diodes (OLEDs) and photosensitive materials.[18]

The myriad of applications of 1,3,4-oxadiazoles has resulted in the development of a number of synthetic methods for their preparation. The most common methods can be divided into two groups: (a) oxidative cyclization of *N*-acylhydrazones using reagents such as CAN,[19] $KMnO_4$,[20] $FeCl_3$,[21] tetravalent lead reagents,[22] HgO/I_2,[23] Br_2/NaOAc,[24] chloramine T,[25] and hypervalent iodine reagents;[26] (b) dehydrative cyclization of *N,N'*-diacylhydrazines using PPA,[27] H_2SO_4,[28] $SOCl_2$,[29] $POCl_3$,[30] P_2O_5,[31] $(CF_3SO_2)_2O$,[32] $BF_3{\cdot}OEt_2$[33] and the Burgess reagent.[34] Besides these reactions, 1,3,4-oxadiazole derivatives have also been prepared by the one-pot reaction of acid hydrazides with carboxylic acids,[35] aromatic aldehydes[19] or orthoesters,[36] the heterocyclization of semicarbazide, thiosemicarbazide and selenosemicarbazide derivatives,[37] photoisomerization of 1,2,4-oxadiazoles,[38] as well as *N*-acylation and subsequent ring opening/closing of tetrazoles.[39] In recent years, the solid phase syntheses of 2,5-disubstituted 1,3,4-oxadiazoles has also been reported.[40] However, many of the above-mentioned methods suffer from drawbacks, such as the use of expensive, hazardous reagents, long reaction times, harsh reaction conditions, limited substrate scope and difficult work-up procedures. Hence, the development of a facile and efficient synthetic protocol to overcome these difficulties is still highly desirable. In continuation of our studies on the synthesis of 1,3,4-oxadiazoles that are conjugated *via* an ethenyl linker to benzene, thiophene, furan and pyridine rings,[36c,41] we describe the efficient oxidative cyclization of *N*-acylhydrazones using DDQ. To the best of our knowledge, the application of DDQ to the preparation of 2,5-disubstituted 1,3,4-oxadiazoles has not been reported.

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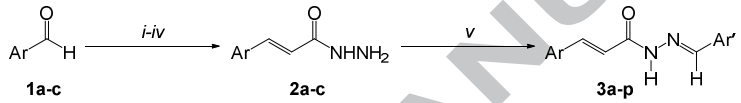
2. Results and discussion

The hydrazides of 3-arylacrylic acids **2a-c** utilized as starting materials were prepared from their respective aldehydes, using a previously reported four-step synthetic protocol. Formation of α,β-unsaturated carboxylic acids *via* the piperidine-catalyzed Knoevenagel-Doebner reaction was followed by neutralization with KOH. The resulting potassium salts were transformed into the corresponding 3-arylacrylic acid hydrazides **2a-c** in a one-pot, two-step reaction sequence by treatment with ethyl chloroformate in acetonitrile, followed by the addition of excess hydrazine hydrate (Scheme 1).

The resulting hydrazides **2a-c** were heated with the appropriate aromatic aldehydes **1a-h** in ethanol, in the presence of catalytic HCl to yield *N*-aroylhydrazones **3a-p** in high yields (77-95%, Table 2). These acyclic products precipitated immediately after mixing the reagents and were purified by recrystallization from ethanol. The structures of all novel compounds **3a-p** were confirmed by elemental analysis and spectroscopic methods (^{1}H and ^{13}C NMR, MS, UV, IR). Analysis of the spectra of *N*-aroylhydrazones **3a-p** in DMSO, showed double the number of signals which was ascribed to the presence of C=C and C=N double bonds, which are a source of geometric isomerism. Similar observations were reported by Palla and co-workers for *N*-acylhydrazones and by Kudelko and Zieliński for *N′*-ethoxymethylene-2-(*N*-Boc-amino)propionohydrazides or *N*-protected *N′*-ethoxymethylene phenylglycine hydrazides.[42]

N′-(4-Methoxybenzylidene)-3-phenylacrylohydrazide (**3e**) was selected as the model substrate in order to optimize the reaction conditions. Initially, various oxidants, including $KMnO_4$, CAN, Oxone and DDQ, were screened for the reaction. Among the oxidants tested, DDQ was found to give the highest yield of the product, 5-(4-methoxyphenyl)-2-(2-phenylethenyl)-1,3,4-oxadiazole **4e** (67%, Table 1, entry 1-4). In addition, the reduced by-product (DDQ-H_2) could be easily removed from the reaction mixture by filtration and recycled by oxidation with MnO_2. It should be noted that the starting *N*-aroylhydrazones contain an unsaturated C=C double bond, that is susceptible to oxidation, which is why effective and selective oxidation in this reaction remains a challenge. The reactions of other oxidizing agents resulted in the formation of side-products, where the desired conjugated arrangement was destroyed. Hence, DDQ was selected as the most efficient and selective oxidizing agent and used throughout this work. Further optimization with respect to the solvent showed that toluene gave the best result in terms of both yield and time (95%, Table 1, entry 4-6). Finally, the optimal procedure was established as follows: reflux in toluene for 5 h in the presence of an equimolar amounts of DDQ.



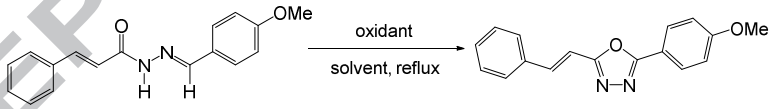
	Ar	Yield (%)
2a	Ph	75
2b	2-furyl	82
2c	2-thienyl	79

Ar = Ph, 2-furyl, 2-thienyl

Ar′ = Ph, 2-furyl, 2-thienyl, 2-pyrrolyl, 4-MeO-C_6H_4, 4-O_2N-C_6H_4, 4-pyridyl, 9-anthryl

Scheme 1. Synthesis of the key *N*-aroylhydrazones **3a-p**. Reagents and conditions: (i) $CH_2(COOH)_2$, pyridine, piperidine, reflux, 2 h; (ii) KOH, H_2O; (iii) ClCOOEt, MeCN, pyridine, reflux, 2 h; (iv) $N_2H_4{\cdot}H_2O$, MeCN, 0 °C, 24 h; (v) aromatic aldehyde, cat. HCl, EtOH, reflux, 2 h.

Table 1. Optimization of the reaction conditions for the synthesis of 5-(4-methoxyphenyl)-2-(2-phenylethenyl)-1,3,4-oxadiazole (**4e**)[a]



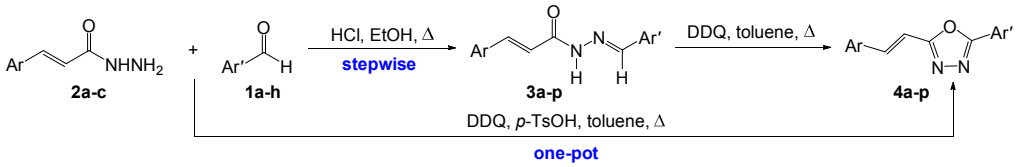
Entry	Oxidant	Solvent	Time (h)	Yield[b] (%)
1	$KMnO_4$	$CHCl_3$-H_2O[c]	20	40
2	CAN	$CHCl_3$	15	22
3	Oxone	$CHCl_3$	25	0
4	DDQ	$CHCl_3$	8	67
5	DDQ	MeCN	8	71
6	DDQ	Toluene	5	95

[a]Reaction conditions: **3e** (5.0 mmol), oxidant (5.0 mmol, for $KMnO_4$ 15.0 mmol), solvent (30 mL).

[b]Isolated yield.

[c]Solvents used in 4:1 (v/v) ratio.

Table 2. Formation of 5-aryl-2-(2-arylethenyl)-1,3,4-oxadiazoles **4a-p** using a stepwise synthesis and proceeding *via* intermediate *N*-aroylhydrazones **3a-p** and using a one-pot synthesis starting from 3-arylacrylohydrazides **2a-c**



Product	Ar	Ar′	Stepwise synthesis[a]			One-pot synthesis[b]
			Hydrazone **3** Yield[c] (%)	1,3,4-Oxadiazole **4** Yield[c] (%)	1,3,4-Oxadiazole **4** Time (h)	1,3,4-Oxadiazole **4** Yield[c] (%)
a	Ph	Ph	95	73	6	75
b	Ph	2-furyl	92	82	5	86
c	Ph	2-thienyl	96	87	5	90
d	Ph	2-pyrrolyl	84	43	10	49
e	Ph	4-MeO-C_6H_4	89	95	5	91
f	Ph	4-O_2N-C_6H_4	86	69	6	75
g	Ph	4-pyridyl	77	26	10	40
h	Ph	9-anthryl	81	75	8	75
i	2-furyl	4-MeO-C_6H_4	86	91	3	88
j	2-furyl	2-furyl	93	86	4	90
k	2-furyl	2-thienyl	95	89	3	94
l	2-furyl	9-anthryl	83	78	6	79
m	2-thienyl	4-MeO-C_6H_4	90	94	3	96
n	2-thienyl	2-furyl	95	89	3	92
o	2-thienyl	2-thienyl	98	96	2	97
p	2-thienyl	9-anthryl	86	80	5	84

[a]Reaction conditions: **3** (5.0 mmol), DDQ (5.0 mmol), toluene (30 mL), reflux, 2-10 h.

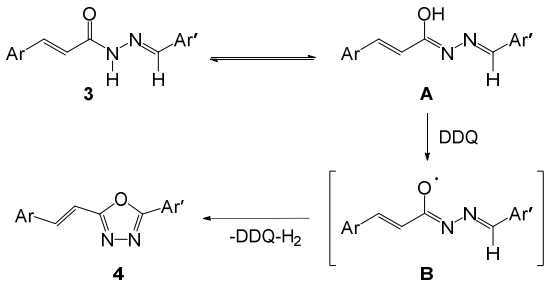
[b]Reaction conditions: **1** (5.0 mmol), **2** (5.0 mmol), *p*-TsOH (0.1 mmol), DDQ (5.0 mmol), toluene (30 mL), reflux, 3 h.

[c]Isolated yield.

With the optimized reaction conditions in hand, *N*′-(arylmethylidene)-3-arylacrylohydrazides **3a-p** were examined to determine the scope and limitations of the reaction (Table 2). To our delight, the reaction displayed a wide substrate scope and proceeded smoothly to afford the 5-aryl-2-(2-arylethenyl)-1,3,4-oxadiazoles **4a-p**, largely, in good yields (Table 2). Initially, *N*-aroylhydrazones **3a,e,f** were tested, which indicated that this synthetic methodology was compatible with a variety of substituents on the benzene ring of the precursor aldehyde. The 2-styryl-1,3,4-oxadiazoles substituted at the 5-position with heteroaryl groups **4b-d,g** were synthesized with yields ranging from 26% to 87%. It should be noted that although heterocycles such as furan and thiophene were found to be stable under the optimized reaction conditions, those possessing a pyrrole **3d** or pyridine ring **3g** gave lower yields due to the formation of by-products as indicated by TLC. *N*′-(9-Anthrylmethylidene)-3-phenylacrylohydrazide (**3h**) was also converted to the corresponding 1,3,4-oxadiazole **4h** in 75% yield.

To further investigate the general utility of this transformation, 3-phenylacrylohydrazide (**2a**) was replaced with other α,β-unsaturated acid hydrazides. Substrates derived from 3-(2-furyl)acrylic acid hydrazide (**2b**) or 3-(2-thienyl)acrylic acid hydrazide (**2c**) were smoothly converted to the corresponding 2,5-disubstituted 1,3,4-oxadiazoles **4i-p** in good to excellent yields (78-96%, Table 2). Comparing the three series of heterocyclic products (Ar = Ph, **4a-h**; Ar = 2-furyl, **4i-l** and Ar = 2-thienyl, **4m-p**), the best results were observed in the derivatives containing the thiophene ring. This trend was also observed for the synthesis of the acyclic *N*-aroylhydrazones **3a-p** (77-98%; Table 2).

In addition, preliminary studies were conducted with aliphatic hydrazides and aliphatic aldehydes to further explore the scope and generality of this reaction. Two *N*-acylohydrazones derived from crotonohydrazide and 4-methoxybenzaldehyde or 3-phenylacrylohydrazide and propionaldehyde were selected. The synthesis of these acyclic intermediates required longer reaction times, giving lower yields with numerous by-products. Similar trends were also observed for the synthesis of the corresponding 1,3,4-oxadiazoles, demonstrating that this methodology does not work as well when either of the counterparts are aliphatic. Further in-depth studies are currently in progress.



Scheme 2. Proposed mechanism for the DDQ-mediated cyclization of *N*-aroylhydrazones.

Additionally, we decided to explore the possibility of conducting the reaction as a one-pot procedure, thus avoiding the purification of the intermediate *N*-aroylhydrazones **3a-p**. A mixture of the appropriate 3-arylacrylic acid hydrazide **2a-c** and aromatic aldehyde **1a-h** in dry toluene was heated at reflux with DDQ in the presence of catalytic *p*-toluenesulfonic acid for 3 h. To our satisfaction, we observed that all tested substrates reacted smoothly to afford the 5-aryl-2-(2-arylethenyl)-1,3,4-oxadiazoles **4a-p** in satisfactory yields (40-97%, Table 2). The developed one-pot procedure gave results that were comparable to those obtained using the stepwise protocol.

Based on literature reports,[43] a plausible mechanism was proposed (Scheme 2). We presume that *N*-aroylhydrazone **3** tautomerizes to hydroxyhydrazone **A**, containing a stable and highly extended π-conjugated system. The reaction of **A** with DDQ generates the resonance stabilized oxygen-centred radical **B**. Subsequent 1,5-homolytic radical cyclization of **B** leads to the formation of the final 1,3,4-oxadiazole ring **4** and reduced DDQ-H_2 which precipitated as a yellow solid. The presence of an electron-donating substituent on the Ar′ ring stabilizes radical **B**, resulting in a higher yield of the desired product (**4e**, 91%, Table 2). Conversely an electron-withdrawing substituent resulted in a decrease in yield (**4f**, 75%, Table 2).

The structures of the 5-aryl-2-(2-arylethenyl)-1,3,4-oxadiazoles **4a-p** were established by elemental analysis and spectroscopic methods. The proton NMR spectrum confirmed the successful synthesis of the desired products by the disappearance of signals for the CH=N and NH protons in the starting *N*-aroylhydrazones **3a-p** and the appearance of diagnostic peaks in the ^{1}H NMR spectra corresponding to two protons of the ethenyl group which were observed as two doublets with coupling constants in the range of 16.0-16.4 Hz. The value of the coupling constants suggested formation of (*E*) geometric isomers of these compounds. The assignment of the ethenyl protons was confirmed by 2D NMR experiments (HMBC and HSQC). The protons adjacent to the carbon at the α position of the ethenyl substituent in 5-aryl-2-(2-arylethenyl)-1,3,4-oxadiazoles **4a-p** appeared in the range of 6.85-7.23 ppm, while the protons in the β position were observed between 7.36 and 7.75 ppm. These two protons were shifted to low fields due to the neighbouring 1,3,4-oxadiazole nitrogen and oxygen atoms. In addition, the ^{13}C NMR spectra showed the disappearance of peaks corresponding to the CH=N and C=O carbon atoms. Characteristic signals belonging to the ethenyl carbon atoms α-CH= and β-CH=, were found between 112-114 ppm and 133-139 ppm, respectively. The C2 ring carbon atom was observed between 157 and 163 ppm, while the location of the second carbon atom C5 was dependent on the aryl substituent and appeared in the range of 163 to 165 ppm.

3. Conclusions

In conclusion, we have demonstrated the effectiveness and selectivity of DDQ for the synthesis of a series of novel, conjugated 5-aryl-2-(2-arylethenyl)-1,3,4-oxadiazoles proceeding *via* the oxidative cyclization of *N*-aroylhydrazones. Commercial availability, high reactivity, recyclability, low cost and low toxicity make DDQ a versatile and useful oxidizing agent for this reaction. In addition, we have developed a facile and efficient method to synthesize 1,3,4-oxadiazoles directly from α,β-unsaturated acid hydrazides and aromatic aldehydes in a one-pot procedure. This methodology offers the advantage of short reaction times, high yields and a wide substrate scope, which makes it a promising alternative to the existing synthetic protocols.

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