Highly Efficient Synthesis of Tricyclic Perimidines under Microwave Heating

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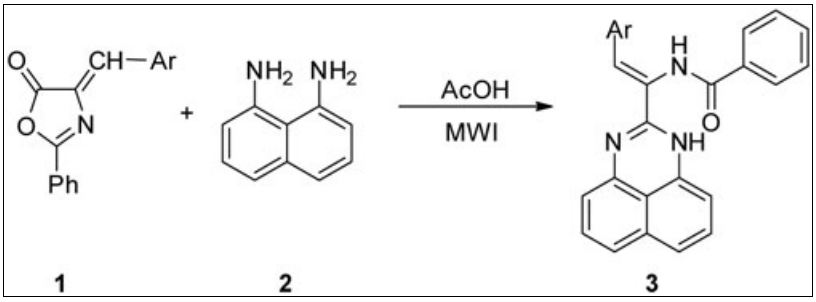

A new and efficient access to (*Z*)-*N*-(2-argio-1-(1*H*-perimidin-2-yl)vinyl)benzamide derivatives from readily available substrates in HOAc is described with aid of microwave irradiation. The results of our study provide a simple, straightforward synthetic route to these interesting classes of 2-substituted perimidines analogs in excellent yields.

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INTRODUCTION

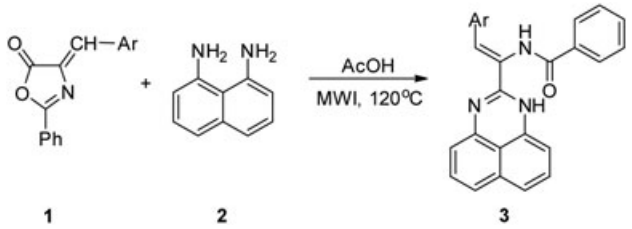
The 1*H*-perimidine system has been first obtained by de Aguiar in 1874 [1], and its synthesis was reported by Sachs in 1909 [2]. Perimidines, a condensed three-ring system with electronic properties, are unusual among azines where the lone pair of a pyrrole-like nitrogen participates in the π-system of the molecule. Its charge-distribution pattern shows a transfer of electron density from the heterocyclic to the naphthalene moiety [3]. Therefore, perimidines have the characteristics of both π-deficient and π-excessive systems [4]. The interest in perimidine derivatives stems have long been used in dyestuffs [3] and in the manufacture of polyester fibers [3], and more recently as a source of a novel carbene ligand [5]. Simultaneously, their biological activity has also attracted attention, such as, their potential to act as antifungal, antimicrobial, antiulcer, and antitumor agents [3,6,7]. Consequently, there has been an ongoing interest in the synthesis of perimidine ring structures [3–8]. However, they are commonly either flawed in some aspect or narrow in their range of activity.

Recently, most synthetic routes [3] to 2-substituted perimidines are based on the reactions of 1,8-diaminonaphthalene (DAN) with various carbonyl compounds [9,10]. Carboxylic acids, acyl halides, and anhydrides afford the monoamide derivatives, which undergo acid-catalyzed cyclization to 2-substituted perimidines. The corresponding reaction with aldehydes affords 2,3-dihydroperimidines, which are readily dehydrogenated to 2-substituted perimidines [11,12]. So far, to the best of our knowledge, the synthesis of (1-perimidin-2-yl)vinyl)benzamide derivatives through two-component reaction between (*Z*)-4-(4-arzylidene)-2-phenyloxazol-5(4*H*)-one [13] and DAN has not been reported.

In the past several years, our groups have developed microwave-assisted organic synthesis (MAOS) that can provide easy accesses to multifunctionalized heterocyclic structures of chemical and pharmaceutical interest.[14] Especially, we established a new microwave-assisted domino reaction for an efficient synthesis of multifunctionalized quinazoline derivatives [14]. The reaction is easily performed by simply mixing readily available starting materials, aromatic aldehydes, cyclopentanone, and cyanoacetamide with K_2CO_3 in ethylene glycol under microwave (MW) irradiation. Interestingly, when aliphatic aldehydes were used to replace their aromatic counterparts for the above reaction, the reaction was found to undergo along another pathway leading to the formation of multifunctionalized tricyclo[6.2.2.$0^{1,6}$]dodecanes [14]. Recently, we have also found that the microwave-assisted reaction of Meldrum's acid, aromatic aldehydes, and electron-rich heteroaryl-amines in aqueous phase under microwave irradiation (MW) led to the multifunctionalized spiro{[1,3]dioxanes-pyridine}-4,6-dione with high chemoselectivity, regioselectivity, and stereoselectivity and good yields [14].

As part of our current studies on the development of new routes in heterocyclic synthesis [14] and expanding the

Scheme 1



families containing perimidine unit, herein we like to report a practically simple, economical, and high-yielding route to a wide variety of 2-substituted perimidine derivatives through microwave-assisted reaction between 4-arylmethylene-2-phenyloxazol-5(4*H*)-ones **1** and DAN **2** at 120°C in glacial acetic acid (Scheme 1).

RESULTS AND DISCUSSION

To choose the most appropriate solvent, the MW-assisted reaction of (*Z*)-4-(4-chlorobenzylidene)-2-phenyloxazol-5(4*H*)-one (**1**, 1.0 mmol) and naphthalene-1,8-diamine (**2**, 1.0 mmol) was examined using ethanol (EtOH), ethylene glycol, *N*,*N*-dimethylformamide (DMF), water, and glacial acetic acid (HOAc) as the solvent (1.5 mL) at 100°C, respectively (Scheme 2). All the reactions were carried out at the maximum power of 250 W (initial power 100 W). The incomplete reaction was observed in ethanol, ethylene glycol, or water (Table 1, entries 1–3). The use of DMF gave rise to the increase of yield to 54%. As shown in Table 1, the reaction in HOAc gave the best result (Table 1, entry 5).

To further optimize reaction conditions, the same reaction was performed in HOAc at temperatures ranging from 100 to 140°C. Initially, the product **3c** was easily obtained with yield 59% at 100°C. The yield of product **3c** was increased as the temperature was increased from 100 to 120°C (Table 1, entries 5–7). However, further increase of the temperature from 130 to 140°C (Table 1, entries 8–9) failed to improve the yield of **3c**. Therefore, 120°C was chosen as the reaction temperature for all further microwave-assisted reactions (Scheme 2).

Table 1

Optimization for the synthesis of **3c** under MW.

Entry	Solvent	*T* (°C)	Time (min)	Yield (%)
1	EtOH	100	6	39
2	Glycol	100	6	45
3	Water	100	6	30
4	DMF	100	6	54
5	HOAc	100	6	59
6	HOAc	110	6	72
7	HOAc	120	6	88
8	HOAc	130	6	82
9	HOAc	140	6	69

Under the optimal conditions [HOAc, 120°C], reactions of different (*Z*)-4-(argiomethylene)-2-phenyloxazol-5(4*H*)-one with naphthalene-1,8-diamine were performed to generate a series of new 2-substituted perimidine analogs. As shown in Table 2, we made a research for the aldehyde substrate scope, and the results indicated that aromatic aldehydes bearing both electron-withdrawing (such as nitro) and electron-donating (such as methoxy) readily provided compounds **3** in good yields (Table 2, entries 1–11). Moreover, heterocyclic aldehydes such as thiophene-2-carbaldehyde (Table 2, entry 12) still displayed a high reactivity under this standard condition. It is worth noting that this chemistry is significant, because there is no literature precedent for the synthesis of (1*H*-perimidin-2-yl)vinyl)benzamide. All the products were characterized by IR, ^{1}H NMR, and HRMS.

A reasonable mechanism for the formation of the products **3** was outlined in Scheme 3. The reaction underwent initial nucleophilic addition and subsequent ring opening to generate intermediate **5**. The intermediate **5** in acetic acid should be mostly protonated at the NH_2 group

Scheme 2

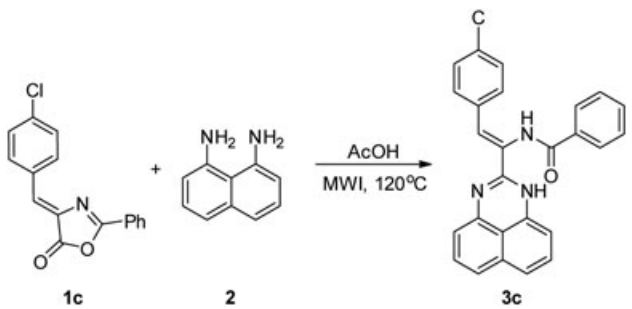
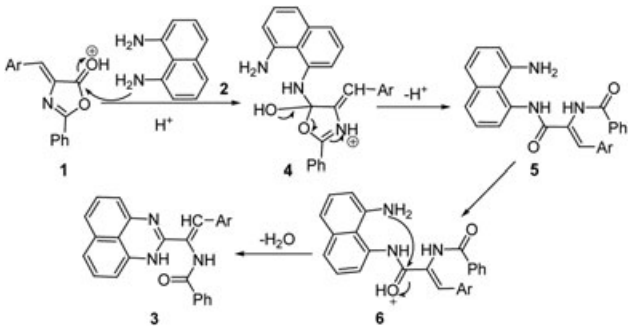


Table 2

Physical and analytical data of compounds **3**.

Entry	Compd.	Ar	Time (min)	Yield (%)	Mp (°C)
1	**3a**	C_6H_5	7	85	267.2–268.4
2	**3b**	4-FC_6H_4	6	79	257.5–259.6
3	**3c**	4-ClC_6H_4	6	88	262.1–264.0
4	**3d**	2-ClC_6H_4	8	86	257.3–258.5
5	**3e**	2,4-$Cl_2C_6H_4$	8	86	257.3–258.5
6	**3f**	4-BrC_6H_4	7	90	270.0–271.8
7	**3g**	4-$O_2NC_6H_4$	5	82	247.3–248.9
8	**3h**	4-MeC_6H_4	7	89	265.3–266.4
9	**3i**	2,3-$(MeO)_2C_6H_3$	9	77	235.3–237.4
10	**3j**	3,4-$(MeO)_2C_6H_3$	7	82	261.7–263.2
11	**3k**	3,4,5-$(MeO)_3C_6H_2$	7	78	211.3–212.6
12	**3l**	Thiophen-2-yl	9	84	257.2–259.5

Scheme 3



of the *ortho*-diamine. Small equilibrium concentrations of protonations of amide carbonyls will also be present, particularly at 120°, to enable cyclization and dehydration to prepare compounds **3**.

We have successfully developed microwave-assisted reactions to facilitate the rapid construction of (1*H*-perimidin-2-yl)vinyl)benzamide skeleton from readily obtainable and inexpensive materials. Particularly valuable features of this procedure included the good to excellent yields and operational simplicity as well as increased safety for small-scale high-speed synthesis. Furthermore, this series of (1*H*-perimidin-2-yl)vinyl) benzamide may prove new classes of chemical properties and physiological activity, which is in progress in our laboratory.

EXPERIMENTAL

All reactions were performed in a monomodal Emrys™ Creator from Personal Chemistry, Uppsala, Sweden. Melting points were determined in open capillaries and are uncorrected. IR spectra were recorded on a FTIR-tensor 27 spectrometer in KBr. ^{1}H-NMR spectra were measured on a DPX 400 MHz spectrometer using TMS as an internal standard and DMSO-d_6 as solvent. HRMS (ESI) was determined by using the micrO-TOF-Q II HPLC/MS instrument (BRUKER).

General procedure for the synthesis of (1*H*-perimidin-2-yl) vinyl)benzamide 3 under microwave irradiation. In a 10-mL Emrys reaction vial, 4-arylmethylene-2-phenyloxazol-5(4*H*)-ones (1 mmol) with 1,8-diaminonaphthalene (1.0 mmol) in HOAc (1.5 mL) were mixed and then capped. The automatic mode stirring helped the mixing and uniform heating of the reactants. The mixture was irradiated by microwave at 120°C for a given time. On completion, monitored by TLC, the reaction mixture was cooled to room temperature and then poured into cold water, filtered to give the crude products, which were further purified by recrystallization from 95% EtOH. The reaction time and the yields are listed in Table 2. The analytical data of new products are as following:

(Z)-N-(1-(1H-Perimidin-2-yl)-2-phenylvinyl) benzamide (3a). IR (KBr, ν, cm^{-1}):3415, 3234, 3049, 1650, 1638, 1594, 1510, 1479, 1412, 1372, 1275, 1164, 1029, 904, 823, 769, 688 cm^{-1}. ^{1}H-NMR (400 MHz, DMSO-d_6) (δ, ppm): 10.53 (s, 1H, NH), 10.09 (s, 1H, NH), 8.03 (d, J = 7.2 Hz, 2H, ArH), 7.64–7.52 (m, 5H, ArH), 7.40–7.22 (m, 4H, ArH), 7.14–6.97 (m, 4H, ArH), 6.53–6.47 (m, 2H, ArH). HRMS (ESI) *m/z*: calc. for $C_{26}H_{19}N_3O$: 388.1444 [M-H]$^-$, found: 388.1469 [M-H]$^-$.

(Z)-N-(2-(4-Fluorophenyl)-1-(1H-perimidin-2-yl)vinyl) benzamide (3b). IR (KBr,ν, cm^{-1}): 3415, 3237, 3056, 1638, 1594, 1508, 1478, 1412, 1375, 1287, 1237, 1159, 824, 768, 696. ^{1}H-NMR (400 MHz, DMSO-d_6) (δ, ppm): 10.54 (s, 1H, NH), 10.09 (s, 1H, NH), 8.03 (d, J = 7.2 Hz, 2H, ArH), 7.69–7.52 (m, 5H, ArH), 7.26–6.97 (m, 7H, ArH), 6.52–6.46 (m, 2H, ArH). HRMS (ESI) *m/z*: calc. for $C_{26}H_{18}FN_3O$: 406.1370 [M-H]$^-$, found: 406.1374 [M-H]$^-$.

(Z)-N-(2-(4-Chlorophenyl)-1-(1H-perimidin-2-yl)vinyl) benzamide (3c). IR (KBr,ν, cm^{-1}) 3415, 3237, 3056, 1638, 1594, 1508, 1478, 1412, 1374, 1288, 1164, 1092, 959, 822, 770, 699 cm^{-1}. ^{1}H-NMR (400 MHz, DMSO-d_6) (δ, ppm): 10.56 (s, 1H, NH), 10.07 (s, 1H, NH), 7.95 (d, J = 7.6 Hz, 2H, ArH), 7.60–7.48 (m, 5H, ArH), 7.32–6.98 (m, 7H, ArH), 6.55–6.47 (m, 2H, ArH). HRMS (ESI) *m/z*: calc. for $C_{26}H_{18}ClN_3O$: 422.1052 [M-H]$^-$, found: 422.1074 [M-H]$^-$.

(Z)-N-(2-(2-Chlorophenyl)-1-(1H-perimidin-2-yl)vinyl) benzamide (3d). IR (KBr,ν, cm^{-1}): 3417, 3217, 3048, 1650, 1638, 1595, 1522, 1486, 1374, 1289, 1107, 1031, 904, 820, 755, 690cm^{-1}. ^{1}H-NMR (400 MHz, DMSO-d_6) (δ, ppm): 10.57 (s, 1H, NH), 10.08 (s, 1H, NH), 7.95 (d, J = 7.2 Hz, 2H, ArH), 7.59–7.49 (m, 5H, ArH), 7.32–7.24 (m, 3H, ArH), 7.15–6.98 (m, 4H, ArH), 6.55–6.46 (m, 2H, ArH). ^{13}C-NMR (100MHz, DMSO-d_6) (*d*, ppm): 166.0, 152.4, 138.4, 135.1, 133.2, 133.1, 131.8, 129.8, 129.6, 129.5, 128.9, 128.3, 127.9, 127.1, 122.8, 121.9, 119.2, 117.6, 113.8, 102.7. HRMS (ESI) *m/z*: calc. for $C_{26}H_{18}ClN_3O$: 422.1052 [M-H]$^-$, found: 422.1056 [M-H] $^-$.

(Z)-N-(2-(2,4-Dichlorophenyl)-1-(1H-perimidin-2-yl)vinyl) benzamide (3e). IR (KBr,ν, cm^{-1}): 3415, 3209, 3048, 1638, 1594, 1522, 1470, 1373, 1286, 1151, 1099, 907, 822, 764, 695 cm^{-1}. ^{1}H-NMR (400 MHz, DMSO-d_6) (δ, ppm): 10.61 (s, 1H, NH), 10.12 (s, 1H, NH), 7.95 (d, J = 7.6 Hz, 2H, ArH), 7.68 (s, 1H, ArH), 7.58–7.49 (m, 4H, ArH), 7.38 (d, J = 8.4 Hz, 1H, ArH), 7.14–6.99 (m, 5H, ArH), 6.53-6.45 (m, 2H, ArH). HRMS (ESI) *m/z*: calc. for $C_{26}H_{17}Cl_2N_3O$: 456.0665 [M-H]$^-$, found: 456.0670 [M-H]$^-$.

(Z)-N-(2-(4-Bromophenyl)-1-(1H-perimidin-2-yl)vinyl) benzamide (3f). IR (KBr,ν, cm^{-1}): 3415, 3238, 3054, 1650, 1637, 1596, 1514, 1479, 1374, 1282, 1164, 1075, 1008, 900, 822, 770, 707, 638 cm^{-1}. ^{1}H-NMR (400 MHz, DMSO-d_6) (δ, ppm): 10.58 (s, 1H, NH), 10.10 (s, 1H, NH), 8.02 (d, J = 7.2 Hz, 2H, ArH), 7.61–7.52 (m, 7H, ArH), 7.16–7.01 (m, 5H, ArH), 6.49 (s, 2H, ArH). ^{13}C-NMR (100MHz, DMSO-d_6) (*d*, ppm): 165.9, 152.7, 145.0, 138.5, 135.1, 134.0, 133.6, 131.8, 131.4, 131.1, 130.4, 128.4, 127.9, 125.9, 121.8, 121.5, 119.1, 117.5, 113.7, 102.5. HRMS (ESI) *m/z*: calc. for $C_{26}H_{18}BrN_3O$: 466.0549 [M-H] $^-$, found: 466.0554 [M-H] $^-$.

(Z)-N-(2-(4-Nitrophenyl)-1-(1H-perimidin-2-yl)vinyl) benzamide (3g). IR (KBr,ν, cm^{-1}): 3415, 3319, 3049, 1666, 1636, 1594, 1575, 1518, 1477, 1340, 1283, 1111, 1020, 921, 823, 769, 690 cm^{-1}. ^{1}H-NMR (400 MHz, DMSO-d_6) (δ, ppm): 10.71 (s, 1H, NH), 10.32 (s, 1H, NH), 8.19 (d, J = 8.4 Hz, 2H, ArH), 8.03–7.83 (m, 3H, ArH), 7.72–7.48 (m, 4H, ArH), 7.23–7.01 (m, 5H, ArH), 6.48 (s, 2H, ArH). HRMS

(ESI) *m/z*: calc. for $C_{26}H_{18}N_4O_3$: 433.1295 $[M-H]^-$, found: 433.1313 $[M-H]^-$.

(Z)-N-(1-(1H-Perimidin-2-yl)-2-(p-tolyl)vinyl)benzamide (3h). IR (KBr,ν, cm^{-1}): 3415, 3240, 3051, 1638, 1594, 1520, 1478, 1374, 1289, 1185, 1029, 823, 770, 696 cm^{-1}. ^{1}H-NMR (400 MHz, DMSO-d_6) (δ, ppm): 10.51 (s, 1H, NH), 10.05 (s, 1H, NH), 8.03 (d, *J* = 7.6 Hz, 2H, ArH), 7.61–7.51 (m, 5H, ArH), 7.20–6.97 (m, 7H, ArH), 6.52–6.47 (m, 2H, ArH), 2.29 (s, 3H, CH_3). ^{13}C-NMR (100 MHz, DMSO-d_6) (*d*, ppm): 165.9, 152.8, 145.1, 138.2, 135.1, 133.7, 131.8, 131.7, 129.3, 129.1, 128.8, 128.4, 127.9, 127.6, 121.8, 118.9, 117.4, 113.6, 102.5, 20.9. HRMS (ESI) *m/z*: calc. for $C_{27}H_{21}N_3O$: 402.1601 $[M-H]^-$, found: 402.1609 $[M-H]^-$.

(Z)-N-(2-(2,3-Dimethoxyphenyl)-1-(1H-perimidin-2-yl) vinyl)benzamide (3i). IR (KBr,ν, cm^{-1}): 3414, 3211, 3048, 1636, 1593, 1523, 1476, 1371, 1273, 1166, 1075, 1002, 933, 822, 769, 692 cm^{-1}. ^{1}H-NMR (400 MHz, DMSO-d_6) (δ, ppm): 10.56 (s, 1H, NH), 9.99 (s, 1H, NH), 7.98 (d, *J* = 7.6 Hz, 2H, ArH), 7.61–7.50 (m, 4H, ArH), 7.29 (s, 1H, ArH), 7.16–6.99 (m, 6H, ArH), 6.54–6.48 (m, 2H, ArH), 3.81 (d, *J* = 5.2 Hz, 6H, $2OCH_3$). HRMS (ESI) *m/z*: calc. for $C_{28}H_{23}N_3O_3$: 448.1655 $[M-H]^-$, found: 448.1671 $[M-H]^-$.

(Z)-N-(2-(3,4-Dimethoxyphenyl)-1-(1H-perimidin-2-yl) vinyl)benzamide (3j). IR (KBr,ν, cm^{-1}): 3416, 3213, 3050, 1636, 1593, 1514, 1479, 1372, 1265, 1143, 1025, 824, 769, 699 cm^{-1}. ^{1}H-NMR (400 MHz, DMSO-d_6) (δ, ppm): 10.43 (s, 1H, NH), 10.03 (s, 1H, NH), 8.09 (d, *J* = 7.6 Hz, 2H, ArH), 7.61–7.54 (m, 3H, ArH), 7.29–6.97 (m, 8H, ArH), 6.54–6.48 (m, 2H, ArH), 3.76 (s, 3H, OCH_3), 3.54 (s, 3H, $2OCH_3$). HRMS (ESI) *m/z*: calc. for $C_{28}H_{23}N_3O_3$: 448.1655 $[M-H]^-$, found: 448.1659 $[M-H]^-$.

(Z)-N-(1-(1H-Perimidin-2-yl)-2-(3,4,5-trimethoxyphenyl) vinyl)benzamid (3k). IR (KBr,ν, cm^{-1}): 3416, 3240, 3049, 1638, 1593, 1505, 1479, 1373, 1292, 1128, 1005, 824, 771, 698 cm^{-1}. ^{1}H-NMR (400 MHz, DMSO-d_6) (δ, ppm): 10.46 (s, 1H, NH), 10.09 (s, 1H, NH), 8.10 (d, *J* = 7.6 Hz, 2H, ArH), 7.60–7.52 (m, 3H, ArH), 7.14 (s, 1H, ArH), 7.16–6.97 (m, 6H, ArH), 6.56–6.49 (m, 2H, ArH), 3.66 (s, 3H, OCH_3), 3.61 (s, 6H, $2OCH_3$). ^{13}C-NMR (100 MHz, DMSO-d_6) (*d*, ppm): 165.9, 152.6, 137.9, 135.1, 133.5, 131.8, 129.9, 128.9, 128.6, 128.3, 127.9, 127.8, 121.8, 118.9, 117.5, 113.5, 107.1, 102.5, 60.1, 55.6. HRMS (ESI) *m/z*: calc. for $C_{29}H_{25}N_3O_4$: 478.1761 $[M-H]^-$, found: 478.1765 $[M-H]^-$.

(Z)-N-(1-(1H-Perimidin-2-yl)-2-(thiophen-2-yl)vinyl) benzamide (3l). IR (KBr,ν, cm^{-1}): 3415, 3258, 3053, 1650, 1638, 1594, 1476, 1373, 1275, 1164, 1029, 905, 824, 771, 704, 617 cm^{-1}. ^{1}H-NMR (400 MHz, DMSO-d_6) (δ, ppm): 10.43 (s, 1H, NH), 9.95 (s, 1H, NH), 8.13 (d, *J* = 7.6 Hz, 2H, ArH), 7.79 (s, 1H, ArH), 7.67–7.49 (m, 6H, ArH), 7.15–6.97 (m, 4H, ArH), 6.55–6.49 (m, 2H, ArH). HRMS (ESI) *m/z*: calc. for $C_{29}H_{25}N_3O_4$: 394.1013 $[M-H]^-$, found: 394.1040 $[M-H]^-$.

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