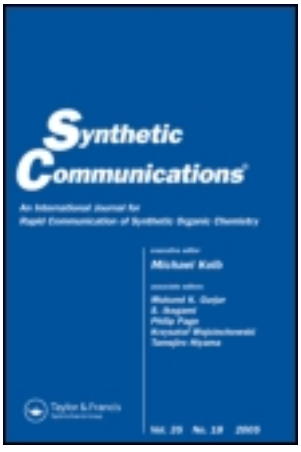

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Microwave-Assisted Expeditious Synthesis of Novel Carbazole-Based 1,3,4-Oxadiazoles

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Microwave-Assisted Expeditious Synthesis of Novel Carbazole-Based 1,3,4-Oxadiazoles

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Abstract: A microwave-assisted expeditious synthetic route to novel carbazole-based 1,3,4-oxadiazoles is described. The reactions of 9-ethylcarbazol-3-carbaldehyde with aroylhydrazines under microwave irradiation first gave intermediates, l-aroyl-2-(9′-ethylcarbazol-3′-yl-methylidene)hydrazines, which were further treated with potassium permanganate in DMF under microwave irradiation to rapidly afford a series of 2-aryl-5-(9′-ethylcarbazol-3′-yl)-1,3,4-oxadiazoles in excellent yield.

Keywords: Aroylhydrazine, carbazole, microwave irradiation, 1,3,4-oxadiazole

INTRODUCTION

1,3,4-Oxadiazoles are important heterocyclic compounds with a wide range of pharmaceutical and biological activities, such as antibacterial,[1] anti-inflammatory,[2] insecticidal,[3] anticonvulsant,[4] and antimitotic[5] activities. However, heterocyclic compounds bearing the 1,3,4-oxadiazole moiety have been used as a new π-conjugation relay to prepare a number of donor–acceptor molecules carrying a π-electron-rich aromatic ring.[6] In addition, carbazole-involved compounds have received increasing interest

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because of their unique optical properties and strong hole-transporting ability in opto-electronic devices.[7,8] Therefore, the compounds bearing both carbazole and 1,3,4-oxadiazole moieties may be good candidates for optical materials or biologically active chemicals.

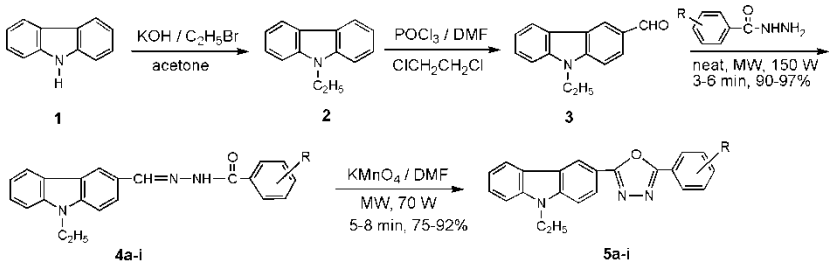
The common synthetic route to 1,3,4-oxidiazoles includes reactions of acid hydrazides (or hydrazine) with acid chlorides[9,10] (or carboxylic acids[11]) or direct cyclization of diacylhydrazines by use of a variety of anhydrous reagents such as thionyl chloride,[12] phosphorous pentoxide,[13] phosphorous oxychloride,[14] triflic anhydride,[15] and polyphosphoric acid.[16] The acylation of tetrazoles[17] and oxidation of acylhydrazones[18,19] are additional synthetic routes to these compounds. Recently, solid-phase syntheses of these compounds were also reported.[20–22] However, these methods often require long reaction times and high temperature, and the reagents used in the reactions are usually corrosive and environmentally unfriendly.

In this article, we report a microwave-assisted rapid synthetic method for novel carbazole-based 1,3,4-oxadiazoles by using potassium permanganate as an efficient cyclization reagent. To the best of our knowledge, this is the first example of the synthesis of small molecules of carbazole-based 1,3,4-oxadiazole.

RESULTS AND DISCUSSION

The reaction of carbazole (**1**) with bromoethane in the presence of potassium hydroxide first gave 9-ethylcarbazole (**2**) in high yield. Compound **2** on treatment with phosphorus oxychloride and N,N-dimethylformamide in the solution of 1,2-dichloroethane at 90°C for 6 h afforded 9-ethylcarbazol-3-carbaldehyde **(3)** in high selectivity (no 3,6-dicarbaldehyde by-product was observed). Compound **3** was irradiated with various aroylhydrazines for 3–6 min at 150 W of microwave power to efficiently give 1-aroyl-2-(9′-ethylcarbazol-3′-ylmethylidene)hydrazines (**4a**–**i**) after simple washing with ethanol. The solution of compounds **4a**–**i** and potassium permanganate in DMF were subjected to microwave irradiation at 70 W of power for only 5–8 min to readily afford 2-aryl-5-(9′-ethylcarbazol-3′-yl)-1,3,4-oxadiazoles (**5a**–**i**) in excellent yield Scheme 1, Table 1. It is noteworthy to mention that the attempt to synthesize compounds **5a**–**i** using other organic solvents, such as ethanol, acetone, and acetonitrile, did not succeed because of the insolubility of potassium permanganate and formation of many by-products. In addition, the same reactions were also investigated by a conventional heating method. However, it took at least 10 h to complete the reactions with satisfactory yield.

The resulting compounds **5a**–**i** are highly soluble in common organic solvents including $CHC1_3$, CH_2C1_2, DMSO, DMF, and EtOH. The structures of new compounds **4a**–**i** and **5a**–**i** were identified by ^{1}H NMR, IR, and elemental analyses. The ^{1}H NMR spectra of compounds **4a**–**i** in DMSO-d_6 show –CH═ group proton peaks at $\delta = 8.60–8.63$ ppm and –NH– group proton peaks at $\delta = 11.75–11.91$ ppm. These two kinds of



Scheme 1.

signals thoroughly disappear in the ^{1}H NMR spectra of compounds **5a–i**, which indicate the formation of oxadiazole ring. The IR spectra of compounds **4a–i** show characteristic absorptions at 3171–3196 cm^{-1}, and 1630–1654 cm^{-1}, attributable to the imino and carbonyl groups, respectively. These adsorptions vanish in the IR spectra of compounds **5a–i**, which also mean the formation of the oxadiazole ring.

In conclusion, we have described a rapid and highly efficient method for the synthesis of a new series of carbazole-based 1,3,4-oxadiazoles under microwave irradiation. This protocol has rapid reaction rate, short reaction time, and high reaction yield.

Table 1. Microwave-assisted synthesis of **4a–i** and **5a–i**

Compd.	R	Time (min)	Mp (°C)	Yield (%)[a]
4a	H	4	237–239	97
4b	4-Cl	5	246–248	95
4c	2-Cl	6	221–222	90
4d	4-NO_2	6	253–254	96
4e	3-NO_2	6	218–220	91
4f	2-CH_3	4	220–221	92
4g	3-CH_3	3	240–241	95
4h	4-Br	5	238–240	95
4i	4-HO	3	296–298	94
5a	H	5	158–159	92
5b	4-Cl	5	204–206	89
5c	2-Cl	8	148–150	84
5d	4-NO_2	5	236–238	86
5e	3-NO_2	5	192–194	79
5f	2-CH_3	6	140–142	88
5g	3-CH_3	6	168–170	75
5h	4-Br	8	198–200	90
5i	4-HO	7	296–298	83

[a] Yields refer to the isolated products.

EXPERIMENTAL

IR spectra were recorded using KBr pellets on a Digilab FTS 3000 FTIR spectrophotometer and ^{1}H NMR spectra on a Mercury Plus-400 instrument using $(CD_3)_2SO$ or $CDCl_3$ as solvents and Me_4Si as internal standard. Elemental analyses were performed on a Vario E1 elemental analysis instrument. Mass spectra were recorded on a QP-1000A GC-MS using the impact mode (70 eV). Melting points were observed in an electrothermal melting-point apparatus. Microwave reactions were conducted in a modified microwave oven fitted with a condenser (LG-WP650, China). Aroylhydrazines,[23] 9-ethylcarbazole (**2**),[24] and 9-ethyl-carbazol-3-carbaldehyde (**3**)[25] were prepared according to literature procedures.

General Procedure for Preparation of 1-Aroyl-2-(9′-ethylcarbazol-3′-yl-methylidene)hydrazines (4a–i)

The aroylhydrazine (6 mmol) and 9-ethylcarbazole-3-carbaldehyde (6 mmol) were thoroughly mixed in a mortar with a pestle, and then the mixture was transferred to a flask and subjected to microwave irradiation at 150 W of power. The completion of the reaction was monitored by thin-layer chromatography (TLC) (ethyl acetate–petroleum ether = 1 : 2 as eluent). Then the resulting mixture was washed with ethanol, and the solid was recrystallized from DMF and water to give the product.

Analytical Data for Compounds 4a–i

4a: White solid. IR (KBr pellet, cm^{-1}): 3176 ($\nu_{N\text{-}H}$), 1634 ($\nu_{C=O}$), 1599, 1553, 1469, 1303, 1232 (carbazole ring). ^{1}H NMR (DMSO-d_6, 400 MHz) δ: 1.32 (t, $J = 7.2$ Hz, 3H, $-CH_3$), 4.46 (q, $J = 7.2$ Hz, 2H, $-CH_2-$), 7.22–8.47 (m, 12H, Ar-H), 8.61 (s, 1H, $-CH=$), 11.79 6 (s, 1H, $-NH-$). MS: m/z, 341 (M^+). Anal. calcd. for $C_{22}H_{19}N_3O$: C, 77.40; H, 5.61; N, 12.31. Found: C, 77.31; H, 5.54; N, 12.24.

4b: White solid. IR (KBr pellet, cm^{-1}): 3186 ($V_{N\text{-}H}$), 1644 ($V_{C=O}$), 1597, 1554, 1473, 1299, 1234 (carbazole ring). ^{1}H NMR (DMSO-d_6, 400 MHz) δ: 1.34 (t, $J = 6.8$ Hz, 3H, $-CH_3$), 4.49 (q, $J = 6.8$ Hz, 2H, $-CH_2-$), 7.24–8.48 (m, 11H, Ar–H); 8.62 (s, lH, $-CH=$), 11.87 (s, lH, $-NH-$). MS: m/z, 375 (M^+). Anal. calcd. for $C_{22}H_{18}ClN_3O$: C, 70.30; H, 4.83; N, 11.18. Found: C,70.22; H, 4.90; N, 11.26.

4c: White solid. IR (KBr pellet, cm^{-1}): 3182 (V_{N-H}), 1646 ($\nu_{C=O}$), 1596, 1553, 1474, 1300, 1235 (carbazole ring). ^{1}H NMR (DMSO-d_6, 400 MHz)

δ: 1.33 (t, $J = 6.8$ Hz, 3H, $-CH_3$), 4.58 (q, $J = 6.8$ Hz, 2H, $-CH_2-$), 7.25–8.49 (m, 11H, Ar–H), 8.61 (s, 1H, –CH═), 11.85 (s, 1H, –NH–). MS: m/z, 375 (M^+). Anal. calcd. for $C_{22}H_{18}ClN_3O$: C, 70.30; H, 4.83; N, 11.18. Found: C, 70.21; H, 4.90; N, 11.11.

4d: Yellow solid. IR (KBr pellet, cm^{-1}): 3195 (ν_{N-H}), 1654 ($\nu_{C=O}$), 1602, 1559, 1478, 1305, 1239 (carbazole ring). ^1H NMR (DMSO-d_6, 400 MHz) δ: 1.41 (t, $J = 7.2$ Hz, 3H, $-CH_3$), 4.53 (q, $J = 7.2$ Hz, 2H, $-CH_2-$), 7.29–8.54 (m, 11H, Ar–H); 8.63 (s, 1H, –CH═), 11.91 (s, 1H, –NH–). MS: m/z, 386 (M^+). Anal. calcd. for $C_{22}H_{18}N_4O_3$: C, 68.38; H, 4.70; N, 14.50. Found: C, 68.47; H, 4.77; N, 14.41.

4e: Yellow solid. IR (KBr pellet, cm^{-1}): 3196 (ν_{N-H}), 1653 ($\nu_{C=O}$), 1604, 1559, 1479, 1304, 1238 (carbazole ring). ^1H NMR (DMSO-d_6, 400 MHz) δ: 1.40 (t, $J = 7.2$ Hz, 3H, $-CH_3$), 4.52 (q, $J = 7.2$ Hz, 2H, $-CH_2-$), 7.26–8.53 (m, 11H, Ar–H); 8.63 (s, 1H, –CH═), 11.90 (s, 1H, –NH–). MS: m/z, 386 (M^+). Anal. calcd. for $C_{22}H_{18}N_4O_3$: C, 68.38; H, 4.70; N, 14.50. Found: C, 68.29; H, 4.65; N, 14.42.

4f: White solid. IR (KBr pellet, cm^{-1}): 3172 (ν_{N-H}), 1631 ($\nu_{C=O}$), 1593, 1550, 1467, 1290, 1228 (carbazole ring). ^1H NMR (DMSO-d_6, 400 MHz) δ: 1.30 (t, $J = 6.8$ Hz, 3H, $-CH_3$), 2.14 (s, 3H, $-CH_3$), 4.44 (q, $J = 6.8$ Hz, 2H, $-CH_2-$), 7.20–8.45 (m, 11H, Ar–H); 8.60 (s, 1H, –CH═), 11.76 (s, 1H, –NH–). MS: m/z, 354 (M^+). Anal. calcd. for $C_{23}H_{20}N_3O$: C, 77.94; H, 5.69; N, 11.86. Found: C, 77.86; H, 5.73; N, 11.77.

4g: White solid. IR (KBr pellet, cm^{-1}): 3171 (ν_{N-H}), 1630 ($\nu_{C=O}$), 1592, 1551, 1466, 1291, 1227 (carbazole ring). ^1H NMR (DMSO-d_6, 400 MHz) δ: 1.29 (t, $J = 6.8$ Hz, 3H, $-CH_3$), 2.15 (s, 3H, $-CH_3$), 4.42 (q, $J = 6.8$ Hz, 2H, $-CH_2-$), 7.19–8.44 (m, 11H, Ar–H); 8.60 (s, 1H, –CH═), 11.75 (s, 1H, –NH–). MS: m/z, 354 (M^+). Anal. calcd. for $C_{23}H_{20}N_3O$: C, 77.94; H, 5.69; N, 11.86. Found: C, 78.06; H, 5.60; N, 11.93.

4h: Yellow solid. IR (KBr pellet, cm^{-1}): 3185 (ν_{N-H}), 1649 ($\nu_{C=O}$), 1599, 1556, 1476, 1300, 1237 (carbazole ring). ^1H NMR (DMSO-d_6, 400 MHz) δ: 1.38 (t, $J = 7.2$ Hz, 3H, $-CH_3$), 4.51 (q, $J = 7.2$ Hz, 2H, $-CH_2-$), 7.26–8.50 (m, 11H, Ar–H); 8.63 (s, 1H, –CH═), 11.90 (s, 1H, –NH–). MS: m/z, 419 (M^+). Anal. calcd. for $C_{22}H_{18}BrN_3O$: C, 62.87; H, 4.32; N, 10.00. Found: C, 62.98; H, 4.24; N, 10.10.

4i: Yellow solid. IR (KBr pellet, cm^{-1}): 3477 (ν_{O-H}), 3183 (ν_{N-H}), 1645 ($\nu_{C=O}$), 1597, 1554, 1475, 1298, 1236 (carbazole ring). ^1H NMR (DMSO-d_6, 400 MHz) δ: 1.37 (t, $J = 7.2$ Hz, 3H, $-CH_3$), 4.50 (q, $J = 7.2$ Hz, 2H, $-CH_2-$), 7.25–8.51 (m, 11H, Ar–H); 8.62 (s, 1H, –CH═), 11.88

(s, 1H, –NH–). MS: m/z, 357 (M^+). Anal. calcd. for $C_{22}H_{19}N_3O_2$: C, 73.93; H, 5.36; N, 11.76. Found: C, 73.81; H, 5.40; N, 11.83.

General Procedure for Preparation of 2-Aryl-5-(9′-ethylcarbazol-3′-yl)-1,3,4-oxadiazoles (5a–i)

The mixture of compounds **4a–i** (3 mmol) and potassium permanganate (3 mmol) was dissolved in 2 mL of DMF. Then the mixture was subjected to microwave irradiation at the 70 W of power for the appropriate time indicated in Table 1. The completion of the reaction was monitored by TLC (ethyl acetate–petroleum ether = 1 : 2 as eluent). Then the resulting mixture was filtered, and the filtrate was evaporated to remove the solvent in vacuo. The residue was recrystallized from ethanol to afford the desired product.

Analytic Data for Compounds 5a–i

5a: White solid. IR (KBr pellet, cm^{-1}): 1598, 1551, 1460, 1332, 1235 (carbazole and oxadiazole rings). ^1H NMR ($CDCl_3$, 400 MHz) δ: 1.44 (t, $J = 6.8$ Hz, 3H, $-CH_3$), 4.36 (q, $J = 6.8$ Hz, 2H, $-CH_2-$), 7.24–8.73 (m, 12H, Ar–H). MS: m/z, 339 (M^+). Anal. calcd. for $C_{22}H_{17}N_3O$: C, 77.86; H, 5.05; N, 12.38. Found: C, 77.78; H, 5.12; N, 12.44.

5b: White solid. IR (KBr pellet, cm^{-1}): 1595, 1540, 1477, 1346, 1260 (carbazole and oxadiazole rings). ^1H NMR ($CDCl_3$, 400 MHz) δ: 1.48 (t, $J = 7.2$ Hz, 3H, $-CH_3$), 4.39 (q, $J = 7.2$ Hz, 2H, $-CH_2-$), 7.25–8.81 (m, 11H, Ar–H). MS: m/z, 373 (M^+). Anal. calcd. for $C_{22}H_{16}ClN_3O$: C, 70.68; H, 4.31; N, 11.24. Found: C, 70.74; H, 4.24; N, 11.30.

5c: White solid. IR (KBr pellet, cm^{-1}): 1595, 1541, 1475, 1344, 1261 (carbazole and oxadiazole rings). ^1H NMR ($CDCl_3$, 400 MHz) δ: 1.47 (t, $J = 7.2$ Hz, 3H, $-CH_3$), 4.38 (q, $J = 7.2$ Hz, 2H, $-CH_2-$), 7.22–8.80 (m, 11H, Ar–H). MS: m/z, 373 (M^+). Anal. calcd. for $C_{22}H_{16}ClN_3O$: C, 70.68; H, 4.31; N, 11.24. Found: C, 70.77; H, 4.22; N, 11.16.

5d: Yellow solid. IR (KBr pellet, cm^{-1}): 1601, 1555, 1465, 1337, 1239 (carbazole and oxadiazole rings). ^1H NMR ($CDCl_3$, 400 MHz) δ: 1.54 (t, $J = 7.2$ Hz, 3H, $-CH_3$), 4.45 (q, $J = 7.2$ Hz, 2H, $-CH_2-$), 7.28–8.87 (m, 11H, Ar–H). MS: m/z, 384 (M^+). Anal. calcd. for $C_{22}H_{16}N_4O_3$: C, 68.74; H, 4.20; N, 14.58. Found: C, 68.69; H, 4.30; N, 14.49.

5e: Yellow solid. IR (KBr pellet, cm^{-1}): 1600, 1554, 1461, 1336, 1237 (carbazole and oxadiazole rings). ^1H NMR ($CDCl_3$, 400 MHz) δ: 1.53 (t, $J = 7.2$ Hz, 3H, $-CH_3$), 4.43 (q, $J = 7.2$ Hz, 2H, $-CH_2-$), 7.27–8.86

(m, 11H, Ar–H). MS: m/z, 384 (M^+). Anal. calcd. for $C_{22}H_{16}N_4O_3$: C, 68.74; H, 4.20; N, 14.58. Found: C, 68.64; H, 4.13; N, 14.62.

5f: White solid. IR (KBr pellet, cm^{-1}): 1591, 1548, 1453, 1326, 1227 (carbazole and oxadiazole rings). ^1H NMR ($CDCl_3$, 400 MHz) δ: 1.41 (t, $J = 6.8$ Hz, 3H, $-CH_3$), 2.18 (s, 3H, $-CH_3$), 4.32 (q, $J = 6.8$ Hz, 2H, $-CH_2-$), 7.19–8.68 (m, 11H, Ar–H). MS: m/z, 353 (M^+). Anal. calcd. for $C_{23}H_{19}N_3O$: C, 78.16; H, 5.42; N, 11.89. Found: C, 78.20; H, 5.35; N, 11.97.

5g: White solid. IR (KBr pellet, cm^{-1}): 1590, 1547, 1452, 1325, 1228 (carbazole and oxadiazole rings). ^1H NMR ($CDCl_3$, 400 MHz) δ: 1.40 (t, $J = 6.8$ Hz, 3H, $-CH_3$), 2.18 (s, 3H, $-CH_3$), 4.30 (q, $J = 6.8$ Hz, 2H, $-CH_2-$), 7.17–8.67 (m, 11H, Ar–H). MS: m/z, 353 (M^+). Anal. calcd. for $C_{23}H_{19}N_3O$: C, 78.16; H, 5.42; N, 11.89. Found: C, 78.09; H, 5.31; N, 11.96.

5h: Yellow solid. IR (KBr pellet, cm^{-1}): 1597, 1553, 1462, 1334, 1236 (carbazole and oxadiazole rings). ^1H NMR ($CDCl_3$, 400 MHz) δ: 1.53 (t, $J = 7.2$ Hz, 3H, $-CH_3$), 4.43 (q, $J = 7.2$ Hz, 2H, $-CH_2-$), 7.26–8.84 (m, 11H, Ar–H). MS: m/z, 417 (M^+). Anal. calcd. for $C_{22}H_{16}BrN_3O$: C, 63.17; H, 3.86; N, 10.05. Found: C, 63.25; H, 3.91; N, 10.12.

5i: Yellow solid. IR (KBr pellet, cm^{-1}): 3489 (ν_{O-H}), 1594, 1549, 1460, 1331, 1233 (carbazole and oxadiazole rings). ^1H NMR ($CDCl_3$, 400 MHz) δ: 1.51 (t, $J = 7.2$ Hz, 3H, $-CH_3$), 4.40 (q, $J = 7.2$ Hz, 2H, $-CH_2-$), 7.25–8.82 (m, 11H, Ar–H). MS: m/z, 355 (M^+). Anal. calcd. for $C_{22}H_{17}N_3O_2$: C, 74.35; H, 4.82; N, 11.82. Found: C, 74.27; H, 4.76; N, 11.89.

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