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Original article

An efficient synthesis of 2-(guaiazulen-1-yl)furan derivatives *via* intramolecular Wittig reactions

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ABSTRACT

An efficient and mild synthesis of 2-(guaiazulen-1-yl)furans, starting from easily accessible 1-(3-aryl-2-cyanopropenoyl)guaiazulenes, tributylphosphine and acyl chlorides, is described. The strategy employs the intramolecular Wittig protocol as a key step to append the crucial furan ring, leading to the highly functional furans in good yields.

1. Introduction

Furans occupy an important place in the heterocyclic family of compounds because of their prevalence as a key structural component in a myriad of natural and pharmaceutical products [1]. Polyfunctionalized furans are of great importance because numerous interesting compounds bearing this heterocyclic ring exhibit a wide array of activities and are also building blocks in organic synthesis [2]. Although the Wittig reaction is frequently used in the synthesis of a wide range of heterocycles [3], the interest in intramolecular Wittig methodology in multisubstituted furan synthesis has been growing [4].

Guaiazulene (7-isopropyl-1,4-dimethylazulene) is a known active component of the essential oil of *Guaiacum officinalis* L., and the subject of a number of reports describing its anti-allergenic and anti-inflammatory activities [5]. Azulene derivatives have attracted interest in medicine as antiulcer drugs [6], anticancer agents [7], and as antioxidant therapeutics for neurodegenerative conditions [8]. To date a variety of heterocyclic-fused and substituted azulenes have been synthesized by several methods and reported on from the viewpoint of chemical properties and physiological activities [9].

As part of our current studies on the development of new routes to heterocyclic-substituted azulenes systems [10], we herein report on a novel preparation of a number of azulene substituted furans **4** starting from 1-(3-aryl-2-cyanopropenoyl)guaiazulenes **2**, acid chlorides **3** and tributylphosphine (Bu_3P), in the presence of Et_3N under mild reaction conditions (Scheme 1).

2. Experimental

A mixture of acid chloride **3** (1.1 equiv) and Bu_3P (1.1 equiv) was dissolved in dry THF (20 mL). A solution of **2** (0.5 mmol) in dry THF (5.0 mL) was added, which was followed by the addition of Et_3N (1.2 equiv). The reaction mixture was stirred for the indicated time at room temperature. The progress of the reaction was monitored by TLC. Upon completion, the reaction was quenched with aqueous saturated $NaHCO_3$ solution, and the mixture was extracted with EtOAc (3× 10 mL). Thereafter, the solvent was removed by evaporation in vacuo. The residue was purified by column chromatography on silica gel (160–200 mesh) using *n*-hexane/EtOAc (4:1) as eluent to afford 2-(guaiazulen-1-yl)furans (**4a–l**).

3. Results and discussion

First, to optimize the conditions, 1-(3-phenyl-2-cyanopropenoyl)guaiazulene **2a** (preparation by Knoevenagel condensation of the 1-cyanoacetylguaiazulene with aldehydes [10c]) and *p*-anisoyl chloride **3a** were selected as testing substrates to react with Bu_3P under different reaction conditions (Table 1). We found that amines, such as pyrrolidine and Et_3N, are beneficial for the

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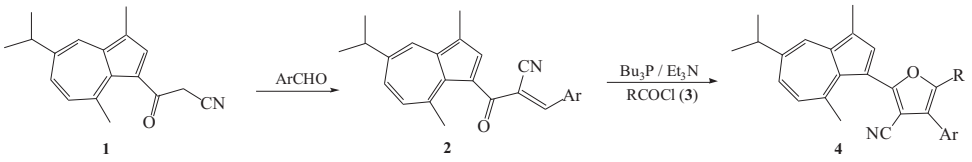


Scheme 1. Syntheses of 2-(guaiazulen-1-yl)furans.

Table 1
Optimization of reaction conditions on the 1-(3-phenyl-2-cyanopropenoyl)-guaiazulene **2a** and *p*-anisoyl chloride **3a**.[a]

Entry	Solvent	Amine	Amount of amine (equiv)	Time (h)	Yield (%)[b]
1	CH_3CN	Pyrrolidine	1.2	3	56
2	CH_3CN	Et_3N	1.2	3	62
3	CH_2Cl_2	Pyrrolidine	1.2	4	64
4	CH_2Cl_2	Et_3N	1.2	3	75
5	Toluene	Et_3N	1.2	4	63
6	THF	Et_3N	1.0	3	83
7	THF	Et_3N	1.2	2	91
8	THF	Et_3N	1.5	2	86

[a] Reactions were performed with **2a** (0.5 mmol), **3a** (1.1 equiv), and Bu_3P (1.2 equiv) in dry solvent (20 mL) under nitrogen, at r.t.
[b] Yield of isolated products.

Table 2
Synthesis of 2-(guaiazulen-1-yl)furans **4**.

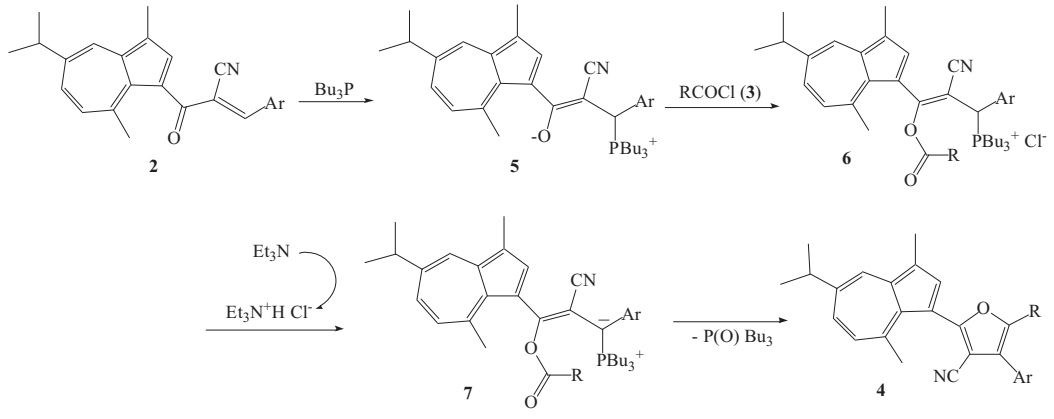
Entry	Ar (**2**)	R (**3**)	Time (h)	Product[a]	Yield (%)[b]
1	Ph (**2a**)	Ph (**3a**)	2	**4a**	91
2	Ph (**2a**)	4-$CH_3C_6H_4$ (**3b**)	2	**4b**	89
3	Ph (**2a**)	4-$CH_3OC_6H_4$ (**3c**)	2	**4c**	94
4	4-$CH_3C_6H_4$ (**2b**)	4-$CH_3OC_6H_4$ (**3c**)	2	**4d**	90
5	4-$CH_3OC_6H_4$ (**2c**)	4-$CH_3OC_6H_4$ (**3c**)	1	**4e**	95
6	4-ClC_6H_4 (**2d**)	4-$CH_3OC_6H_4$ (**3c**)	4	**4f**	83
7	4-$CH_3C_6H_4$ (**2b**)	4-$NO_2C_6H_4$ (**3d**)	8	**4g**	65
8	4-$CH_3OC_6H_4$ (**2c**)	4-$NO_2C_6H_4$ (**3d**)	7	**4h**	70
9	4-$CH_3OC_6H_4$ (**2c**)	2-Furyl (**3e**)	3	**4i**	84
10	Ph (**2a**)	CH_3 (**3f**)	6	**4j**	76
11	4-$CH_3C_6H_4$ (**2b**)	CH_3 (**3f**)	6	**4k**	84
12	4-$CH_3OC_6H_4$ (**2c**)	C_2H_5 (**3g**)	8	**4l**	80

[a] All products were characterized by ^1H NMR and IR spectral data [11].
[b] Yield of isolated products.

formation of **4a** (entries 1–8). The best result was achieved when Et_3N was present in our designed reaction (entry7).

The reaction was optimized by screening solvents, such as CH_2Cl_2, CH_3CN, toluene and THF. As a result, we determined that the reaction best proceeded in THF.

Next, we investigated the utility of the reaction and our protocol with different acyl chlorides **3** (Table 2). It was observed that aryl-substituted acid chlorides **3a–e** (R = Ph, 4-$CH_3C_6H_4$, 4-$CH_3OC_6H_4$, 4-$NO_2C_6H_4$, and 2-furyl) with electron donating substituents (**3a–c**) or a heterocyclic substituent (**3e**) (entries 1–6, 9) were converted to the corresponding adducts (**4a–f**) in higher yields and shorter reaction times than those with an electron-withdrawing substitutent (**3d**) (entries 7, 8). It should be noted that under the same reaction conditions, the alkyl-substituted acid chlorides (**3f**, **3g**) (R = CH_3, C_2H_5), gave the corresponding furan **4j–l** in yields of 76%–84% within 6–8 h (entries 10–12).



Scheme 2. A proposed mechanism for the formation of **4**.

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On the basis of the experimental results, a plausible reaction mechanism was proposed (Scheme 2). First, the regioselective Michael addition of Bu_3P toward **2** took place, providing the corresponding zwitterion **5**. The intermediate **5** was *in situ* acylated with an acid chloride **3**, leading to the formation of **6**. Then deprotonation of **6** by Et_3N occurred, and the resulting ylide **7** underwent an intramolecular Wittig reaction, affording the corresponding furan **4**.

4. Conclusion

In conclusion, we have successfully developed a facile and efficient method to prepare a series of 2-(guaiazulen-1-yl)furans with good yields *via* intramolecular Wittig reactions from readily available 1-(3-aryl-2-cyanopropenoyl)guaiazulene, tributylphosphine and acyl chlorides in the presence of Et_3N.

Acknowledgments

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[11] Physical and spectral data **4a**: Mp: 154–156 °C; IR (KBr): ν 2208 cm^{-1} (CN); ^1H NMR (400 MHz, $CDCl_3$): δ 1.40 (d, 6H, J = 6.9 Hz, $CH(CH_3)_2$), 2.66 (s, 3H, CH_3), 2.76 (s, 3H, CH_3), 3.11–3.16 (m, 1H, $CH(CH_3)_2$), 7.18 (d, 1H, J = 10.5 Hz), 7.40–7.48 (m, 8H), 7.53–7.56 (m, 3H), 7.90 (s, 1H), 8.29 (s, 1H). Anal. Calcd. for $C_{32}H_{27}NO$: C 87.04, H 6.16, N 3.17; found: C 87.15, H 6.23, N 3.26. **4b**: Mp: 165–167 °C; IR (KBr): ν 2213 cm^{-1} (CN); ^1H NMR (400 MHz, $CDCl_3$): δ 1.40 (d, 6H, J = 6.9 Hz, $CH(CH_3)_2$), 2.40 (s, 3H, CH_3), 2.66 (s, 3H, CH_3), 2.75 (s, 3H, CH_3), 3.10–3.15 (m, 1H, $CH(CH_3)_2$), 7.21 (d, 1H, J = 10.5 Hz), 7.35–7.48 (m, 7H), 7.50–7.58 (m, 3H), 7.90 (s, 1H), 8.25 (s, 1H). Anal. Calcd. for $C_{33}H_{29}NO$: C 87.00, H 6.42, N 3.07; found: C 87.16, H 6.51, N 3.14. **4c**: Mp: 189–191 °C; IR (KBr): ν 2208 cm^{-1} (CN); ^1H NMR (400 MHz, $CDCl_3$): δ 1.41 (d, 6H, J = 6.9 Hz, $CH(CH_3)_2$), 2.67 (s, 3H, CH_3), 2.75 (s, 3H, CH_3), 3.11–3.17 (m, 1H, $CH(CH_3)_2$), 3.80 (s, 3H, OCH_3), 6.81 (d, 2H, J = 8.9 Hz), 7.18 (d, 1H, J = 10.5 Hz), 7.40–7.48 (m, 5H), 7.51–7.57 (m, 3H), 7.93 (s, 1H), 8.27 (s, 1H). Anal. Calcd. for $C_{33}H_{29}NO_2$: C 84.05, H 6.20, N 2.97; found: C 84.23, H 6.31, N 3.15. **4d**: Mp: 171–173 °C; IR (KBr): ν 2213 cm^{-1} (CN); ^1H NMR (400 MHz, $CDCl_3$): δ 1.40 (d, 6H, J = 6.9 Hz, $CH(CH_3)_2$), 2.42 (s, 3H, CH_3), 2.66 (s, 3H, CH_3), 2.74 (s, 3H, CH_3), 3.10–3.15 (m, 1H, $CH(CH_3)_2$), 3.79 (s, 3H, OCH_3), 6.80 (d, 2H, J = 8.7 Hz), 7.18 (d, 1H, J = 10.5 Hz), 7.24–7.27 (m, 4H), 7.42–7.47 (m, 4H), 7.52 (d, 1H, J = 10.5 Hz), 7.92 (s, 1H), 8.26 (s, 1H). Anal. Calcd. for $C_{34}H_{31}NO_2$: C 84.09, H 6.43, N 2.88; found: C 84.17, H 6.59, N 3.04. **4e:** Mp: 161–163 °C; IR (KBr): ν 2216 cm^{-1} (CN); ^1H NMR (400 MHz, $CDCl_3$): δ 1.39 (d, 6H, J = 6.9 Hz, $CH(CH_3)_2$), 2.66 (s, 3H, CH_3), 2.74 (s, 3H, CH_3), 3.08–3.17 (m, 1H, $CH(CH_3)_2$), 3.79 (s, 3H, OCH_3), 3.87 (s, 3H, OCH_3), 6.81 (d, 2H, J = 8.7 Hz), 6.98 (d, 2H, J = 8.7 Hz), 7.17 (d, 1H, J = 10.8 Hz), 7.44–7.53 (5H, m), 7.92 (s, 1H), 8.27 (s, 1H). Anal. Calcd. for $C_{34}H_{31}NO_3$: C 81.41, H 6.23, N 2.79; found: C 81.58, H 6.41, N 2.96. **4f:** Mp: 153–155 °C; IR (KBr): ν 2219 cm^{-1} (CN); ^1H NMR (400 MHz, $CDCl_3$): δ 1.38 (d, 6H, J = 6.9 Hz, $CH(CH_3)_2$), 2.67 (s, 3H, CH_3), 2.74 (s, 3H, CH_3), 3.11–3.18 (m, 1H, $CH(CH_3)_2$), 3.81 (s, 3H, OCH_3), 6.82 (d, 2H, J = 9.0 Hz), 7.19 (d, 1H, J = 10.8 Hz), 7.41–7.44 (m, 5H), 7.50 (d, 2H, J = 8.7 Hz), 7.92 (s, 1H), 8.28 (s, 1H). Anal. Calcd. for $C_{33}H_{28}ClNO_2$: C 78.33, H 5.58, N 2.77; found: C 78.47, H 5.73, N 2.89. **4g**: Mp: 121–123 °C; IR (KBr): ν 2215 cm^{-1} (CN); ^1H NMR (400 MHz, $CDCl_3$): δ 1.37 (d, 6H, J = 6.8 Hz, $CH(CH_3)_2$), 2.48 (s, 3H, CH_3), 2.61 (s, 3H, CH_3), 2.85 (s, 3H, CH_3), 3.12–3.17 (m, 1H, $CH(CH_3)_2$), 6.89 (d, 2H, J = 8.0 Hz), 7.29–7.31 (m, 4H), 7.35 (d, 1H, J = 10.8 Hz), 7.64 (d, 1H, J = 10.8 Hz), 7.88 (s, 1H), 7.95 (d, 2H, J = 8.4 Hz), 8.31 (s, 1H). Anal. Calcd. for $C_{33}H_{28}N_2O_3$: C 79.18, H 5.64, N 5.60; Found: C 79.34, H 5.79, N 5.81. **4h:** Mp: 115–117 °C; IR (KBr): ν 2219 cm^{-1} (CN); ^1H NMR (400 MHz, $CDCl_3$): δ 1.37 (d, 6H, J = 6.8 Hz, $CH(CH_3)_2$), 2.62 (s, 3H, CH_3), 2.83 (s, 3H, CH_3), 3.11–3.15 (m, 1H, $CH(CH_3)_2$), 3.83 (s, 3H, OCH_3), 6.91 (d, 2H, J = 7.6 Hz), 7.01 (d, 2H, J = 8.8 Hz), 7.34 (d, 1H, J = 10.8 Hz), 7.30–7.32 (m, 2H), 7.62 (d, 1H, J = 10.8 Hz), 7.88 (s, 1H), 8.03 (d, 2H, J = 8.8 Hz), 8.30 (s, 1H). Anal. Calcd. for $C_{33}H_{28}N_2O_4$: C 76.73, H 5.46, N 5.42; found: C 76.86, H 5.63, N 5.57. **4i**: Mp: 139–140 °C; IR (KBr): ν 2225 cm^{-1} (CN); ^1H NMR (400 MHz, $CDCl_3$): δ 1.38 (d, 6H, J = 6.3 Hz, $CH(CH_3)_2$), 2.67 (s, 3H, CH_3), 2.71 (s, 3H, CH_3), 3.12–3.16 (m, 1H, $CH(CH_3)_2$), 3.84 (s, 3H, OCH_3), 6.53–6.55 (m, 1H), 6.82–6.83 (m, 1H), 6.91 (d, 2H, J = 8.1 Hz), 7.17 (d, 1H, J = 9.9 Hz), 7.51 (d, 1H, J = 9.9 Hz), 7.53–7.54 (m, 1H), 7.62 (d, 2H, J = 8.1 Hz), 7.90 (s, 1H), 8.26 (s, 1H). Anal. Calcd. for $C_{31}H_{27}NO_3$: C 80.67, H 5.90, N 3.03; found: C 80.75, H 6.11, N 3.09. **4j**: Mp: 154–156 °C; IR (KBr): ν 2218 cm^{-1} (CN); ^1H NMR (400 MHz, $CDCl_3$): δ 1.35 (d, 6H, J = 6.8 Hz, $CH(CH_3)_2$), 1.53 (s, 3H, CH_3), 2.62 (s, 3H, CH_3), 2.86 (s, 3H, CH_3), 3.13–3.16 (m, 1H, $CH(CH_3)_2$), 7.37–7.40 (m, 2H), 7.63–7.65 (m, 5H), 7.89 (s, 1H), 8.32 (s, 1H). Anal. Calcd. for $C_{27}H_{25}NO$: C 85.45, H 6.64, N 3.69; found: C 85.57, H 6.80, N 3.73. **4k:** Mp: 169–171 °C; IR (KBr): ν 2215 cm^{-1} (CN); ^1H NMR (400 MHz, $CDCl_3$): δ 1.37 (d, 6H, J = 6.8 Hz, $CH(CH_3)_2$), 1.54 (s, 3H, CH_3), 2.48 (s, 3H, CH_3), 2.62 (s, 3H, CH_3), 2.86 (s, 3H, CH_3), 3.12–3.17 (m, 1H, $CH(CH_3)_2$), 7.19 (d, 1H, J = 10.8 Hz), 7.20–7.25 (m, 4H), 7.53 (d, 1H, J = 10.8 Hz), 7.85 (s, 1H), 8.24 (s, 1H). Anal. Calcd. for $C_{28}H_{27}NO$: C 85.46, H 6.92, N 3.56; found: C 85.63, H 7.11, N 3.74. **4l:** Mp: 135–137 °C; IR (KBr): ν 2212 cm^{-1} (CN); ^1H NMR (400 MHz, $CDCl_3$): δ 1.05 (t, 3H, J = 7.6 Hz, CH_2CH_3), 1.38 (d, 6H, J = 6.8 Hz, $CH(CH_3)_2$), 1.41 (q, 2H, J = 7.6 Hz, CH_2CH_3), 2.62 (3H, s, CH_3), 2.85 (3H, s, CH_3), 3.12–3.18 (m, 1H, $CH(CH_3)_2$), 3.82 (s, 3H, OCH_3), 7.19 (d, 1H, J = 9.2 Hz), 7.19–7.22 (m, 4H), 7.52 (d, 1H, J = 9.2 Hz), 7.85 (s, 1H), 8.24 (s, 1H). Anal. Calcd. for $C_{29}H_{29}NO_2$: C 82.24, H 6.90, N 3.31; found: C 82.38, H 7.03, N 3.47.

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