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LETTER - SPECTRAL ASSIGNMENT

WILEY

^{1}H and ^{13}C NMR spectral assignments of twenty-six 1-aryl-5-(2-(styryl)phenyl)penta-1,4-dien-3-ones

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1 | INTRODUCTION

Curcumin is a polyphenol produced from *Curcuma longa* and is classified as a diarylheptanoid.[1] Curcumin reportedly shows wound healing effects as well as antimicrobial, antifungal, and antiviral effects and modifies the pathology of Alzheimer's by inhibiting β-amyloid disaggregation. Curcumin also exhibits anticancer effects and acts as a protective agent against cardiac diseases.[2–6] Therefore, the derivatization of curcumin has become the center of interest. Curcumin has the 1,6-diene-3,5-dione skeleton (Figure 1a) as the *keto* form and the 5-hydroxy-1,4,6-triene-3-one skeleton as the *enol* form (Figure 1b). One curcumin derivative is the monocarbonyl analogue, 1,4-dien-3-one (Figure 1c). Monocarbonyl analogues of curcumin exhibit antibacterial activities against *Xanthomonas oryzae pv. Oryzae* and antiviral activities against tobacco mosaic virus[7] and act as activators of the master gene transcription factor EB[8]; they also display neuroprotective effects[9] and antiparasitic activities against *Trypanosoma cruzi.*[10] Another polyphenol produced from grapes, resveratrol (3,4′,5-trihydroxy-*trans*-stilbene; Figure 1d), is known to be a phytoalexin that protects plants from bacteria and fungi.[11] Like curcumin, resveratrol shows diverse biological activities including anticancer, antidiabetes, and antioxidant effects and targets mitochondrial metabolic pathways.[12–15] To exploit the desirable features of both compounds, we designed a combined structure containing both the monocarbonyl moiety of curcumin and stilbene moiety of resveratrol (Figure 1e) and synthesized twenty-six 1-aryl-5-(2-(styryl)phenyl)penta-1,4-dien-3-one compounds. According to recent literature,[16] angular type compounds have shown different biological effects compared with linear analogues. Linear curcumin derivatives have been widely studied,[17] and hence, we focused on angular type derivatives. Even there are multiple possibilities of combination, as described above, our synthesis focused solely on monocarbonyl analogues with various substituents where we tried to find other types of biological activities. Nuclear magnetic resonance (NMR) spectroscopy and high-resolution mass spectrometry are utilized to identify these compounds. The spectral data provided herein can be used for the characterization of synthetic or natural compounds with the monocarbonyl moiety of curcumin and stilbene moiety of resveratrol.

2 | EXPERIMENTAL

2.1 | Syntheses

Synthesis of the 1-aryl-5-(2-(styryl)phenyl)penta-1,4-dien-3-one derivatives was accomplished using Compound III (1 mmol), as shown in Scheme 1, which was obtained through aldol condensation between Compound I[18] and excess acetone (II) in alkali solution. An equivalent molar amount of Compound III and the appropriate hetero/aromatic aldehyde were dissolved in ethanol. Excess 50% (*w*/*v*) potassium hydroxide was added to the reaction mixture and was stirred for 20–48 hr at room temperature. The reaction mixture was poured into ice water and was acidified with 3 N HCl (pH = 2–3) to induce

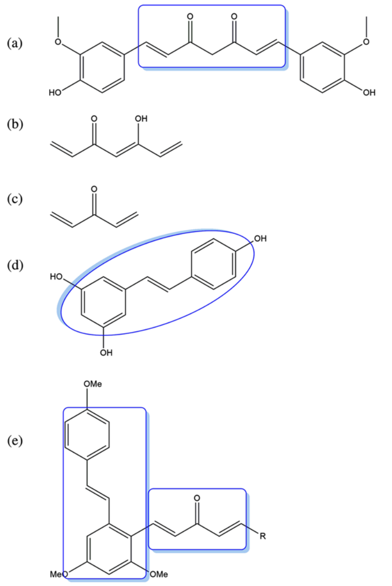
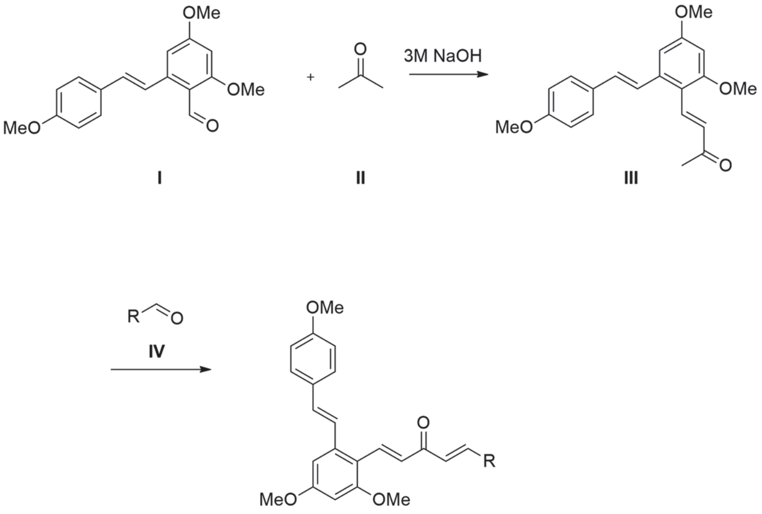


FIGURE 1 Structures of (a) 1,6-diene-3,5-dione, (b) 5-hydroxy-1,4,6-triene-3-one, (c) 1,4-dien-3-one, (d) resveratrol (3,5,4′-trihydroxy-trans-stilbene), where ellipse denotes stilbene moiety, and (e) combined structure containing both monocarbonyl moiety of curcumin and stilbene moiety of resveratrol, where rectangles denote two moieties

precipitation. The precipitate was filtered under vacuum and washed with cold ethanol. Recrystallization in an appropriate solvent gave the pure product. The typical procedure for synthesis of derivative 23 is as follows: Compound I (2.5 mmol, 745 mg) was dissolved in 11 ml of acetone (II, excess) and was combined with 5 ml of 2 M NaOH solution. The reaction mixture was stirred at room temperature for 20 hr. The reaction mixture was poured into 150 ml of ice water for precipitation. The precipitate was vacuum filtered and used for further reaction. Compound III (1 mmol, 338 mg) and 2-furanaldehyde (1 mmol, 96 mg) were dissolved in 10 ml of ethanol. Sodium ethoxide (2 mmol, 136 mg) was added to the reaction mixture and stirred at room temperature for 20 hr. After completion of the reaction, the mixture was poured into ice water and acidified with 3 N HCl (pH = 3) to give a crude solid. The resulting solid was vacuum filtered and purified by recrystallization in ethanol (yield: 25%, mp: 120–122 °C).

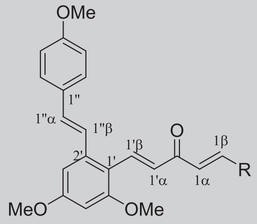
2.2 | NMR spectra

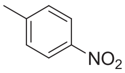
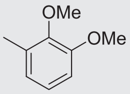
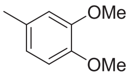
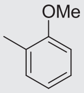
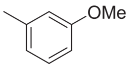
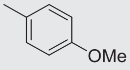
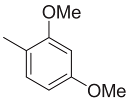
Derivatives 1 and 11 were dissolved in dimethylformamide-*d*7, and 6, 13, 17, 19, 20, 24, and 25 were dissolved in chloroform-*d*; all other compounds were dissolved in dimethylsulfoxide-*d*6. The concentration was adjusted to approximately 50 mM, and the solutions were transferred to 2.5-mm NMR tubes. All NMR data were collected on an Avance 400 spectrometer system (9.4 T; Bruker, Karlsruhe, Germany) at room temperature, and the chemical shifts were referenced to tetramethylsilane. The relaxation delay, 90° pulse, spectral width, number of data points, and digital resolution for the ^{1}H NMR experiments were 1 s, 11.6 μs, 5,500 Hz, 32 K, and 0.34 Hz/point, respectively. The corresponding parameters for the ^{13}C NMR experiments were 3 s, 15.0 μs, 21,000 Hz, 64 K, and 0.64 Hz/point, respectively. Two-dimensional experiments including COSY, HMQC, and HMBC were performed with 2 K × 256 ($t_2 \times t_1$) data points.[19] The long-range coupling time for HMBC was 70 ms. The NMR data were processed using the NMR Pipe program[20] and analyzed using the SPARKY 3 program developed by T. D. Goddard and D. G. Kneller in the University of California at San Francisco.



SCHEME 1 Procedure for synthesis of 1-aryl-5-(2-(styryl)phenyl)penta-1,4-dien-3-one derivatives

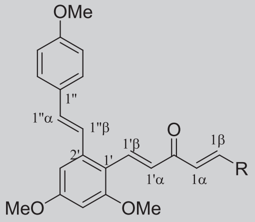
TABLE 1 Structures and names of 1-aryl-5-(2-(styryl)phenyl)penta-1,4-dien-3-one derivatives and corresponding high-resolution mass spectroscopic data, theoretical surface area, volume, and log *p* values

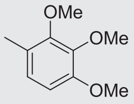
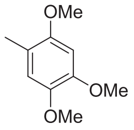
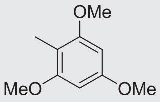
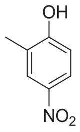
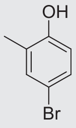
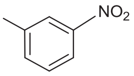


Derivative	R	Mass (calcd/found)	Area (Å^2)	Volume (Å^3)	log P	Name
1		472.1760/472.1757	440.99	393.94	6.61	(1*E*,4*E*)-1-(2-((*E*)-4-methoxystyryl)-4,6-dimethoxyphenyl)-5-(4-nitrophenyl)penta-1,4-dien-3-one
2		487.2121/487.2109	469.67	424.99	5.58	(1*E*,4*E*)-1-(2-((*E*)-4-methoxystyryl)-4,6-dimethoxyphenyl)-5-(2,3-dimethoxyphenyl)penta-1,4-dien-3-one
3		487.2121/487.2074	472.12	426.01	5.58	(1*E*,4*E*)-1-(2-((*E*)-4-methoxystyryl)-4,6-dimethoxyphenyl)-5-(3,4-dimethoxyphenyl)penta-1,4-dien-3-one
4		457.2015/457.2009	445.18	401.33	5.71	(1*E*,4*E*)-1-(2-((*E*)-4-methoxystyryl)-4,6-dimethoxyphenyl)-5-(2-methoxyphenyl)penta-1,4-dien-3-one
5		457.2015/457.1985	446.89	401.10	5.71	(1*E*,4*E*)-1-(2-((*E*)-4-methoxystyryl)-4,6-dimethoxyphenyl)-5-(3-methoxyphenyl)penta-1,4-dien-3-one
6		457.2015/457.2006	447.26	398.49	5.71	(1*E*,4*E*)-1-(2-((*E*)-4-methoxystyryl)-4,6-dimethoxyphenyl)-5-(4-methoxyphenyl)penta-1,4-dien-3-one
7		487.2121/487.2079	469.67	421.73	5.58	(1*E*,4*E*)-1-(2-((*E*)-4-methoxystyryl)-4,6-dimethoxyphenyl)-5-(2,4-dimethoxyphenyl)penta-1,4-dien-3-one

(Continues)

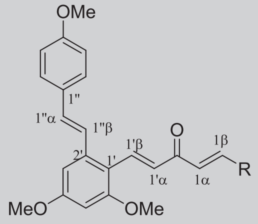
TABLE 1 (Continued)

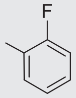
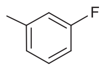
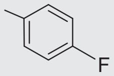
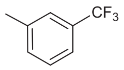
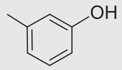
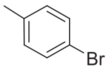
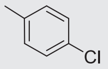
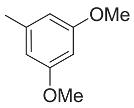


Derivative	R	Mass (calcd/found)	Area (Å^2)	Volume (Å^3)	log P	Name
8		517.2226/517.2209	496.62	451.15	5.45	(1*E*,4*E*)-1-(2-((*E*)-4-methoxystyryl)-4,6-dimethoxyphenyl)-5-(2,3,4-trimethoxyphenyl) penta-1,4-dien-3-one
9		517.2226/517.2220	498.40	449.31	5.45	(1*E*,4*E*)-1-(2-((*E*)-4-methoxystyryl)-4,6-dimethoxyphenyl)-5-(2,4,5-trimethoxyphenyl) penta-1,4-dien-3-one
10		517.2226/517.2211	500.86	454.32	5.45	(1*E*,4*E*)-1-(2-((*E*)-4-methoxystyryl)-4,6-dimethoxyphenyl)-5-(2,4,6-trimethoxyphenyl) penta-1,4-dien-3-one
11		488.1709/488.1718	448.50	401.71	6.22	(1*E*,4*E*)-1-(2-((*E*)-4-methoxystyryl)-4,6-dimethoxyphenyl)-5-(2-hydroxy-5-nitrophenyl) penta-1,4-dien-3-one
12		521.0964/521.0980	446.18	398.97	6.27	(1*E*,4*E*)-1-(2-((*E*)-4-methoxystyryl)-4,6-dimethoxyphenyl)-5-(5-bromo-2-hydroxyphenyl) penta-1,4-dien-3-one
13		472.1760/472.1774	441.55	393.94	6.61	(1*E*,4*E*)-1-(2-((*E*)-4-methoxystyryl)-4,6-dimethoxyphenyl)-5-(3-nitrophenyl)penta-1,4-dien-3-one

(Continues)

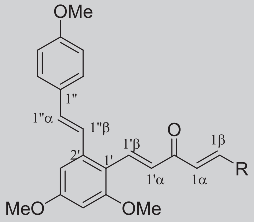
TABLE 1 (Continued)

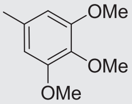
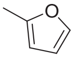
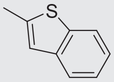
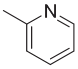
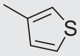


Derivative	R	Mass (calcd/found)	Area (Å^2)	Volume (Å^3)	log P	Name
14		445.1815/445.1806	423.97	377.59	5.99	(1*E*,4*E*)-1-(2-((*E*)-4-methoxystyryl)-4,6-dimethoxyphenyl)-5-(2-fluorophenyl) penta-1,4-dien-3-one
15		445.1815/445.1815	425.15	377.92	5.99	(1*E*,4*E*)-1-(2-((*E*)-4-methoxystyryl)-4,6-dimethoxyphenyl)-5-(3-fluorophenyl) penta-1,4-dien-3-one
16		445.1815/445.1786	424.09	379.74	5.99	(1*E*,4*E*)-1-(2-((*E*)-4-methoxystyryl)-4,6-dimethoxyphenyl)-5-(4-fluorophenyl) penta-1,4-dien-3-one
17		495.1783/495.1771	449.81	404.47	6.75	(1*E*,4*E*)-1-(2-((*E*)-4-methoxystyryl)-4,6-dimethoxyphenyl)-5-(3-(trifluoromethyl)phenyl) penta-1,4-dien-3-one
18		443.1858/443.1833	428.57	384.23	5.44	(1*E*,4*E*)-1-(2-((*E*)-4-methoxystyryl)-4,6-dimethoxyphenyl)-5-(3-hydroxyphenyl) penta-1,4-dien-3-one
19		505.1014/505.1013	439.45	395.11	6.49	(1*E*,4*E*)-1-(2-((*E*)-4-methoxystyryl)-4,6-dimethoxyphenyl)-5-(4-bromophenyl) penta-1,4-dien-3-one
20		461.1520/461.1537	434.95	389.96	6.39	(1*E*,4*E*)-1-(2-((*E*)-4-methoxystyryl)-4,6-dimethoxyphenyl)-5-(4-chlorophenyl) penta-1,4-dien-3-one
21		487.2121/487.2079	473.92	426.39	5.58	(1*E*,4*E*)-1-(2-((*E*)-4-methoxystyryl)-4,6-dimethoxyphenyl)-5-(3,5-dimethoxyphenyl) penta-1,4-dien-3-one

(Continues)

TABLE 1 (Continued)



Derivative	R	Mass (calcd/found)	Area (Å^2)	Volume (Å^3)	log P	Name
22		517.2226/517.2186	495.378	451.90	5.45	(1*E*,4*E*)-1-(2-((*E*)-4-methoxystyryl)-4,6-dimethoxyphenyl)-5-(3,4,5-trimethoxyphenyl) penta-1,4-dien-3-one
23		417.1702/417.1685	403.36	355.66	4.45	(1*E*,4*E*)-1-(2-((*E*)-4-methoxystyryl)-4,6-dimethoxyphenyl)-5-(furan-2-yl) penta-1,4-dien-3-one
24		483.1630/483.1643	452.27	408.54	6.87	(1*E*,4*E*)-1-(2-((*E*)-4-methoxystyryl)-4,6-dimethoxyphenyl)-5-(benzo[*b*]thiophen-2-yl) penta-1,4-dien-3-one
25		428.1862/428.1863	416.44	369.95	4.92	(1*E*,4*E*)-1-(2-((*E*)-4-methoxystyryl)-4,6-dimethoxyphenyl)-5-(pyridin-2-yl) penta-1,4-dien-3-one
26		433.1474/433.1477	412.25	366.70	5.76	(1*E*,4*E*)-1-(2-((*E*)-4-methoxystyryl)-4,6-dimethoxyphenyl)-5-(thiophen-3-yl) penta-1,4-dien-3-one

2.3 | General experimental procedures

To confirm the structures of the 1-aryl-5-(2-(styryl)phenyl)penta-1,4-dien-3-one derivatives determined based on the NMR assignments, ultraperformance liquid chromatography-hybrid quadrupole-time-of-flight mass spectrometry was carried out on a Waters Acquity UPLC system (Waters Corp., Milford, MA).[21] The theoretical log *P*, volume, and area of the synthetic derivatives were calculated using the Sybyl software (Tripos, St. Louis, MO, USA) built on an Intel Core 2 Quad Q6600 (2.4 GHz) Linux personal computer.[22]

3 | RESULTS AND DISCUSSION

Twenty-six 1-aryl-5-(2-(styryl)phenyl)penta-1,4-dien-3-one derivatives were prepared to explain the substituent effects of electron withdrawing, electron donating, and steric. The NMR assignments for all derivatives listed in Table 1 were performed in a similar manner because all of the derivatives have the 1-aryl-5-(2-(styryl)phenyl) penta-1,4-dien-3-one structure in common. As a representative, the NMR assignment for derivative 2, (1*E*,4*E*)-1-(2-((*E*)-4-methoxystyryl)-4,6-dimethoxyphenyl)-5-(2,3-dimethoxyphenyl)penta-1,4-dien-3-one, was as follows:

There were 28 peaks in the ^{13}C NMR spectrum. These peaks were classified as five methoxy, one carbonyl, 13 methines (including six aromatics), and nine quaternary carbons based on the chemical shifts and HMQC spectrum. The ^{1}H spectrum showed five methoxy and 13 methines. The 13 methines were divided into two groups, seven aromatics and six alkenes, based on the coupling constants. Among the aromatics, the two ^{1}H signals at 6.95 ppm (2H, *d*, J = 8.7 Hz) and 7.56 ppm (2H, *d*, J = 8.7 Hz) were assigned as H-3″/5″ and H-2″/6″, respectively, because of their double intensities. The quaternary ^{13}C peak at 159.3 ppm was assigned as C-4″ because it was correlated with the ^{1}H signals of the methoxy (3.77 ppm) and H-2″/6″ (7.56 ppm) groups in the HMBC spectrum. The ^{1}H peaks at 6.60 ppm (1H, *d*, J = 2.3 Hz) and 6.83 ppm (1H, *d*, J = 2.3 Hz) were assigned to H-5′ and H-3′ based on the coupling constants. Further, H-3′ was correlated to the quaternary ^{13}C signal at 114.6 ppm (C-1′) and the alkenyl ^{13}C signal at 124.4 ppm (C-1″β) in the HMBC spectrum. The two methoxy peaks at 3.88 ppm (3H, s) and 3.91 ppm (3H, s) were assigned as 4′-OCH_3 and 6′-OCH_3, respectively. C-4′ (161.6 ppm) was correlated to the H-3′, H-5′, and 4′-OCH_3 signals in the HMBC spectrum. The other aromatic peaks at 7.13 ppm (1H, dd, J = 8.1, 2.4 Hz), 7.12 ppm (1H, dd, J = 8.1, 6.4 Hz), and 7.39 ppm (1H, dd, J = 6.4, 2.4 Hz) were assigned to H-4, H-5, and H-6, respectively. The other two methoxy protons (3.72 and 3.82 ppm) were also assigned based on the HMBC correlations. The six protons in the alkene group all had coupling constants of approximately 16 Hz because they were all *trans* isomers. The signals of the alkene group were assigned using the COSY and HMBC spectra. In the COSY spectrum, the correlation between the signals at 7.08 ppm (1H, *d*, J = 15.9 Hz) and 7.35 ppm (1H, *d*, J = 15.9 Hz) indicated adjacent protons. In the HMBC spectrum, the ^{1}H signal at 7.08 ppm was correlated to C-6″, whereas the ^{1}H signal at 7.35 ppm was correlated to C-3′. Thus, these signals were assigned as H-1″α and H-1″β, respectively. The last four proton signals of the alkenes at 7.08 ppm (1H, *d*, J = 15.9 Hz), 7.27 ppm (1H, *d*, J = 16.1 Hz), 7.82 ppm (1H, *d*, J = 16.1 Hz), and 7.98 ppm (1H, *d*, J = 15.9 Hz) were long-ranged coupled with the carbonyl carbon (188.7 ppm) in the HMBC spectrum, meaning that they were placed near the ketone group. The adjacent protons were determined from the COSY connections, and the positions were determined

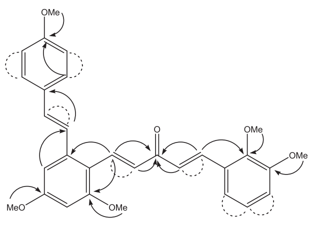


FIGURE 2 Important correlations obtained from the COSY (dotted lines) and HMBC (solid lines) spectra of derivative 2

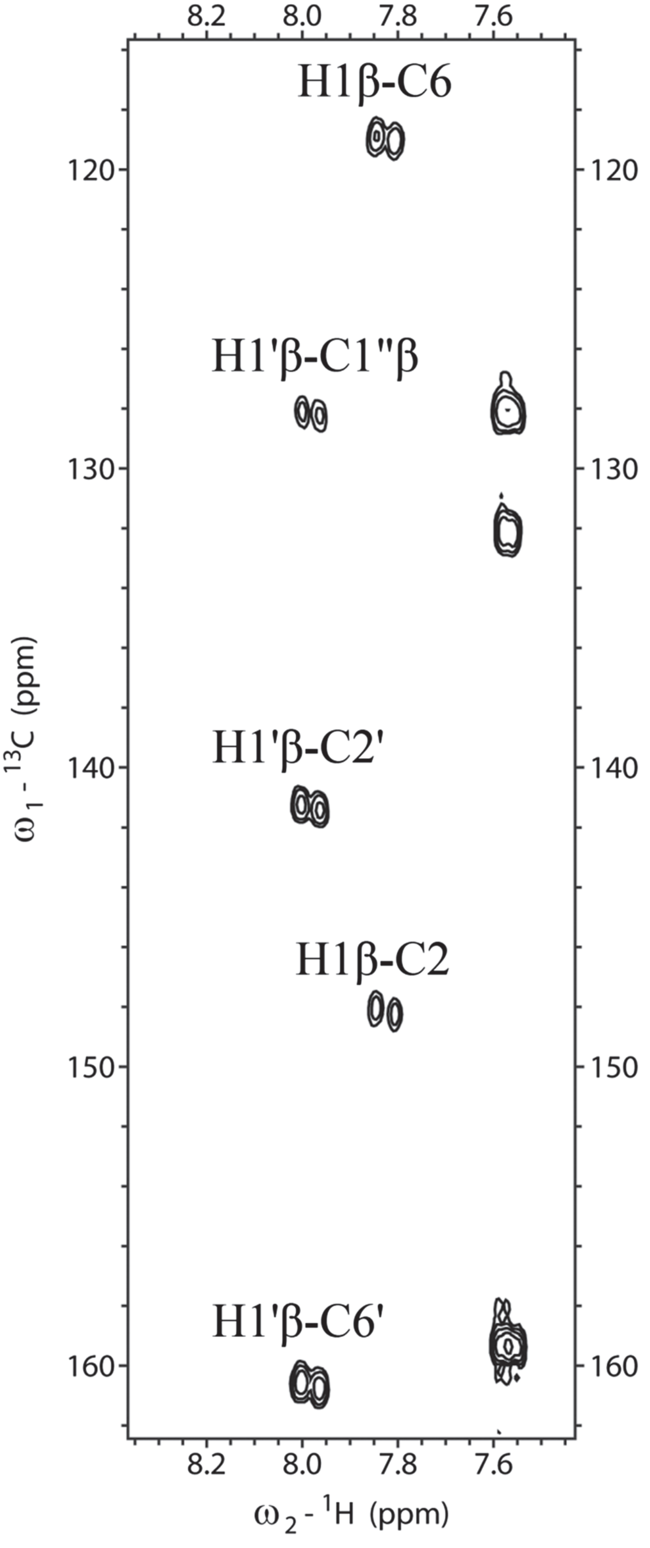


FIGURE 3 Partial HMBC spectrum of derivative 2 showing important long-ranged couplings for the assignment of alkenes

TABLE 2 ^{1}H NMR chemical shifts of 1-aryl-5-(2-(styryl)phenyl)penta-1,4-dien-3-one derivatives 1–26

Position	1	2	3	4	5	6	7	8	9
H-2″	7.67 (d, 8.7)	7.56 (d, 8.7)	7.56 (d, 8.8)	7.56 (d, 8.7)	7.56 (d, 8.7)	7.48 (d, 8.8)	7.55 (d, 8.7)	7.55 (d, 8.5)	7.55 (d, 8.6)
H-3″	7.02 (d, 8.7)	6.95 (d, 8.7)	6.94 (d, 8.8)	6.95 (d, 8.7)	6.94 (d, 8.7)	6.90 (d, 8.8)	6.95 (d, 8.7)	6.95 (d, 8.5)	6.94 (d, 8.6)
H-5″	7.02 (d, 8.7)	6.95 (d, 8.7)	6.94 (d, 8.8)	6.95 (d, 8.7)	6.94 (d, 8.7)	6.90 (d, 8.8)	6.95 (d, 8.7)	6.95 (d, 8.5)	6.94 (d, 8.6)
H-6″	7.67 (d, 8.7)	7.56 (d, 8.7)	7.56 (d, 8.8)	7.56 (d, 8.7)	7.56 (d, 8.7)	7.48 (d, 8.8)	7.55 (d, 8.7)	7.55 (d, 8.5)	7.55 (d, 8.6)
4″-OCH_3	3.86 (s)	3.77 (s)	3.77 (s)	3.77 (s)	3.77 (s)	3.825 (s)	3.77 (s)	3.77 (s)	3.77 (s)
H-1″α	7.20 (d, 16.1)	7.08 (d, 15.9)	7.08 (d, 15.9)	7.08 (d, 16.0)	7.07 (d, 16.0)	6.94 (d, 15.9)	7.08 (d, 16.1)	7.08 (d, 16.1)	7.08 (d, 15.9)
H-1″β	7.52 (d, 16.1)	7.35 (d, 15.9)	7.36 (d, 15.9)	7.35 (d, 16.0)	7.36 (d, 16.0)	7.31 (d, 15.9)	7.33 (d, 16.1)	7.33 (d, 16.1)	7.35 (d, 15.9)
H-3′	6.95 (d, 2.2)	6.83 (d, 2.3)	6.83 (d, 2.1)	6.82 (d, 2.2)	6.82 (d, 2.2)	6.72 (d, 2.0)	6.82 (d, 2.2)	6.82 (d, 2.0)	6.83 (d, 2.0)
H-5′	6.70 (d, 2.2)	6.60 (d, 2.3)	6.60 (d, 2.1)	6.60 (d, 2.2)	6.59 (d, 2.2)	6.43 (d, 2.0)	6.59 (d, 2.2)	6.60 (d, 2.0)	6.59 (d, 2.0)
4′-OCH_3	3.97 (s)	3.88 (s)	3.88 (s)	3.88 (s)	3.87 (s)	3.89 (s)	3.87 (s)	3.88 (s)	3.88 (s)
6′-OCH_3	4.01 (s)	3.91 (s)	3.90 (s)	3.90 (s)	3.90 (s)	3.89 (s)	3.90 (s)	3.90 (s)	3.90 (s)
H-1′β	8.19 (d, 16.0)	7.98 (d, 15.9)	7.94 (d, 15.9)	7.95 (d, 16.0)	7.96 (d, 15.9)	8.09 (d, 15.9)	7.91 (d, 15.9)	7.94 (d, 16.1)	7.92 (d, 15.9)
H-1′α	7.28 (d, 16.0)	7.08 (d, 15.9)	7.08 (d, 15.9)	7.06 (d, 16.0)	7.10 (d, 15.9)	7.07 (d, 15.9)	7.02 (d, 15.9)	7.06 (d, 16.1)	7.05 (d, 15.9)
H-1α	7.61 (d, 16.1)	7.27 (d, 16.1)	7.17 (d, 16.1)	7.26 (d, 16.1)	7.28 (d, 16.1)	6.90 (d, 16.0)	7.12 (d, 16.2)	7.16 (d, 16.1)	7.17 (d, 16.0)
H-1β	7.79 (d, 16.1)	7.82 (d, 16.1)	7.55 (d, 16.1)	7.87 (d, 16.1)	7.55 (d 16.1)	7.61 (d, 16.0)	7.81 (d, 16.2)	7.73 (d, 16.1)	7.88 (d, 16.0)
H-2	8.08 (d, 8.7)	-	7.36 (d, 1.4)	-	7.29 (d, 1.9)	7.45 (d, 8.7)	-	-	-
H-3	8.32 (d, 8.7)	-	-	7.08 (dd, 8.4, 1.0)	-	6.88 (d, 8.7)	6.61 (d, 2.3)	-	6.71 (s)
H-4	-	7.13 (dd, 8.1, 2.4)	-	7.41 (ddd, 8.4, 7.7, 1.6)	7.25 (d, 8.0)	-	-	-	-
H-5	8.32 (d, 8.7)	7.12 (dd, 8.1, 6.4)	6.97 (d, 9.0)	6.98 (ddd, 7.7, 7.7, 1.0)	7.32 (dd, 8.0, 8.0)	6.88 (d, 8.7)	6.57 (dd, 8.6, 2.3)	6.86 (d, 8.9)	-
H-6	8.08 (d, 8.7)	7.39 (dd, 6.4, 2.4)	7.20 (dd, 9.0, 1.4)	7.73 (dd, 7.7, 1.6)	6.99 (dd, 8.0, 1.9)	7.45 (d, 8.7)	7.66 (d, 8.6)	7.53 (d, 8.9)	7.32 (s)
2-OCH_3	-	3.72 (s)	-	3.82 (s)	-	-	3.81 (s)	3.77 (s)	3.82 (s)
3-OCH_3	-	3.82 (s)	3.81 (s)	-	3.78 (s)	-	-	3.75 (s)	-
4-OCH_3	-	-	3.80 (s)	-	-	3.83 (s)	3.82 (s)	3.84 (s)	3.85 (s)
5-OCH_3	-	-	-	-	-	-	-	-	3.75 (s)
Position	**10**	**11**	**12**	**13**	**14**	**15**	**16**	**17**	**18**
H-2″	7.55 (d, 8.6)	7.66 (d, 8.7)	7.56 (d, 8.5)	7.47 (d, 8.7)	7.56 (d, 8.7)	7.565 (d, 8.7)	7.55 (d, 9.0)	7.48 (d, 8.7)	7.57 (d, 8.7)
H-3″	6.95 (d, 8.6)	7.01 (d, 8.7)	6.96 (d, 8.5)	6.90 (d, 8.7)	6.94 (d, 8.7)	6.94 (d, 8.7)	6.94 (d, 9.0)	6.90 (d, 8.7)	6.96 (d, 8.7)
H-5″	6.95 (d, 8.6)	7.01 (d, 8.7)	6.96 (d, 8.5)	6.90 (d, 8.7)	6.94 (d, 8.7)	6.94 (d, 8.7)	6.94 (d, 9.0)	6.90 (d, 8.7)	6.96 (d, 8.7)
H-6″	7.55 (d, 8.6)	7.66 (d, 8.7)	7.56 (d, 8.5)	7.47 (d, 8.7)	7.56 (d, 8.7)	7.565 (d, 8.7)	7.55 (d, 9.0)	7.48 (d, 8.7)	7.57 (d, 8.7)
4″-OCH_3	3.77 (s)	3.85 (s)	3.77 (s)	3.82 (s)	3.77 (s)	3.77 (s)	3.77 (s)	3.83 (s)	3.78 (s)

(Continues)

TABLE 2 (Continued)

Position	10	11	12	13	14	15	16	17	18
H-1″α	7.07 (d, 16.1)	7.19 (d, 16.1)	7.09 (d, 16.0)	6.93 (d, 16.1)	7.07 (d, 15.9)	7.07 (d, 16.1)	7.07 (d, 16.1)	6.94 (d, 16.0)	7.08 (d, 16.1)
H-1″β	7.31 (d, 16.1)	7.52 (d, 16.1)	7.36 (d, 16.0)	7.30 (d, 16.1)	7.35 (d, 15.9)	7.36 (d, 16.1)	7.34 (d, 16.1)	7.31 (d, 16.0)	7.36 (d, 16.1)
H-3′	6.81 (d, 2.0)	6.95 (d, 2.2)	6.83 (d, 1.8)	6.72 (d, 2.3)	6.82 (d, 2.0)	6.82 (d, 2.0)	6.81 (d, 2.0)	6.72 (d, 2.3)	6.83 (d, 2.2)
H-5′	6.59 (d, 2.0)	6.68 (d, 2.2)	6.60 (d, 1.8)	6.43 (d, 2.3)	6.59 (d, 2.0)	6.59 (d, 2.0)	6.59 (d, 2.0)	6.44 (d, 2.3)	6.60 (d, 2.2)
4′-OCH_3	3.87 (s)	3.97 (s)	3.88 (s)	3.89 (s)	3.88 (s)	3.88 (s)	3.87 (s)	3.90 (s)	3.88 (s)
6′-OCH_3	3.90 (s)	4.00 (s)	3.90 (s)	3.90 (s)	3.89 (s)	3.91 (s)	3.90 (s)	3.91 (s)	3.91 (s)
H-1′β	7.87 (d, 16.1)	8.17 (d, 16.1)	7.96 (d, 16.0)	8.14 (d, 15.9)	7.99 (d, 16.0)	7.99 (d, 16.0)	7.96 (d, 15.9)	8.14 (d, 15.9)	7.96 (d, 15.9)
H-1′α	6.98 (d, 16.1)	7.25 (d, 16.1)	7.05 (d, 16.0)	7.12 (d, 15.9)	7.07 (d, 16.0)	7.09 (d, 16.0)	7.08 (d, 15.9)	7.11 (d, 15.9)	7.09 (d, 15.9)
H-1α	7.28 (d, 16.0)	7.68 (d, 16.1)	7.37 (d, 16.0)	7.12 (d, 15.9)	7.35 (d, 16.0)	7.36 (d, 16.0)	7.22 (d, 16.0)	7.07 (d, 16.0)	7.16 (d, 16.0)
H-1β	7.95 (d, 16.0)	8.03 (d, 16.1)	7.79 (d, 16.0)	7.65 (d, 15.9)	7.67 (d, 16.0)	7.57 (d, 16.0)	7.58 (d, 16.0)	7.65 (d, 16.0)	7.51 (d, 16.0)
H-2	-	-	-	8.36 (dd, 1.5, 1.5)	-	7.62 (dd, 10.5, 2.2)	7.77 (dd, 8.6, 5.7)	7.77 (s)	7.13 (s)
H-3	6.28 (s)	7.17 (d, 8.9)	6.94 (d, 9.0)	-	7.28 (dd, 10.6, 7.8)	-	7.24 (dd, 9.6, 8.6)	-	-
H-4	-	8.15 (dd, 8.9, 3.0)	7.38 (dd, 9.0, 2.5)	8.19 (dd, 8.1, 1.5)	7.48 (dddd, 7.8, 7.8, 6.0, 1.8)	7.25 (ddd, 8.4, 8.4, 2.2)	-	7.63 (d, 7.7)	6.85 (dd, 7.8, 2.5)
H-5	6.28 (s)	-	-	7.54 (dd, 8.1, 8.1)	7.26 (dd, 7.8, 7.8)	7.46 (ddd, 8.4, 8.0, 6.0)	7.24 (dd, 9.6, 8.6)	7.49 (dd, 7.7, 7.7)	7.22 (dd, 7.8, 7.8)
H-6	-	8.63 (d, 3.0)	7.88 (d, 2.5)	7.79 (dd, 8.1, 1.5)	7.87 (ddd, 7.8, 7.8, 1.8)	7.52 (d, 8.0)	7.77 (dd, 8.6, 5.7)	7.68 (d, 7.7)	7.10 (dd, 7.8, 2.5)
2-OCH_3	3.81 (s)	-	-	-	-	-	-	-	-
4-OCH_3	3.84 (s)	-	-	-	-	-	-	-	-
6-OCH_3	3.81 (s)	-	-	-	-	-	-	-	-
2-OH	-	-	10.64 (s)	-	-	-	-	-	-
3-OH	-	-	-	-	-	-	-	-	9.60 (s)

Position	19	20	21	22	23	24	25	26
H-2″	7.47 (d, 8.6)	7.47 (d, 8.7)	7.56 (d, 8.5)	7.56 (d, 8.7)	7.55 (d, 8.7)	7.49 (d, 8.7)	7.45 (d, 8.6)	7.55 (d, 8.7)
H-3″	6.90 (d, 8.6)	6.90 (d, 8.7)	6.94 (d, 8.5)	6.94 (d, 8.7)	6.96 (d, 8.7)	6.92 (d, 8.7)	6.88 (d, 8.6)	6.95 (d, 8.7)
H-5″	6.90 (d, 8.6)	6.90 (d, 8.7)	6.94 (d, 8.5)	6.94 (d, 8.7)	6.96 (d, 8.7)	6.92 (d, 8.7)	6.88 (d, 8.6)	6.95 (d,8.7)
H-6″	7.47 (d, 8.6)	7.47 (d, 8.7)	7.56 (d, 8.5)	7.56 (d, 8.7)	7.55 (d, 8.7)	7.49 (d, 8.7)	7.45 (d, 8.6)	7.55 (d, 8.7)
4″-OCH_3	3.83 (s)	3.83 (s)	3.77 (s)	3.76 (s)	3.78 (s)	3.82 (s)	3.80 (s)	3.77 (s)
H-1″α	6.93 (d, 16.1)	6.93 (d, 15.9)	7.07 (d, 16.1)	7.08 (d, 16.1)	7.07 (d, 16.1)	6.94 (d, 16.0)	6.92 (d, 16.1)	7.07 (d, 16.1)
H-1″β	7.29 (d, 16.1)	7.29 (d, 15.9)	7.36 (d, 16.1)	7.37 (d, 16.1)	7.32 (d, 16.1)	7.32 (d, 16.0)	7.30 (d, 16.1)	7.34 (d, 16.1)
H-3′	6.71 (d, 2.2)	6.71 (d, 2.2)	6.82 (d, 1.6)	6.83 (d, 2.3)	6.81 (d, 2.2)	6.72 (d, 2.3)	6.70 (d, 1.8)	6.82 (d, 2.0)
H-5′	6.43 (d, 2.2)	6.43 (d, 2.2)	6.59 (d, 1.6)	6.60 (d, 2.3)	6.59 (d, 2.2)	6.44 (d, 2.3)	6.41 (d, 1.8)	6.59 (d, 2.0)

(Continues)

TABLE 2 (Continued)

Position	19	20	21	22	23	24	25	26
4′-OCH_3	3.89 (s)	3.89 (s)	3.87 (s)	3.88 (s)	3.87 (s)	3.90 (s)	3.87 (s)	3.87 (s)
6′-OCH_3	3.89 (s)	3.89 (s)	3.90 (s)	3.90 (s)	3.90 (s)	3.91 (s)	3.88 (s)	3.89 (s)
H-1′β	8.11 (d, 15.9)	8.11 (d, 15.9)	7.97 (d, 15.9)	7.96 (d, 15.9)	7.92 (d, 15.9)	8.12 (d, 15.9)	8.14 (d, 15.9)	7.94 (d, 15.9)
H-1′α	7.07 (d, 15.9)	7.08 (d, 15.9)	7.09 (d, 15.9)	7.10 (d, 15.9)	7.04 (d, 15.9)	7.08 (d, 15.9)	7.14 (d, 15.9)	7.06 (d, 15.9)
H-1α	6.99 (d, 16.0)	6.98 (d, 16.0)	7.28 (d, 16.0)	7.26 (d, 16.1)	6.90 (d, 15.8)	6.89 (d, 15.9)	7.54 (d, 15.6)	7.09 (d, 15.9)
H-1β	7.55 (d, 16.0)	7.57 (d, 16.0)	7.51 (d, 16.0)	7.55 (d, 16.1)	7.43 (d, 15.8)	7.84 (d, 15.9)	7.64 (d, 15.6)	7.62 (d, 15.9)
H-2	7.34 (d, 8.4)	7.41 (d, 8.5)	6.88 (d, 1.6)	7.06 (s)	-	-	-	7.93 (d, 3.1)
H-3	7.48 (d, 8.4)	7.32 (d, 8.5)	-	-	6.96 (d, 3.4)	7.38 (s)	7.38 (d, 7.4)	-
H-4	-	-	6.55 (dd, 1.6, 1.6)	-	6.65 (dd, 3.4, 1.8)	7.74 (dd, 6.5, 2.6)	7.68 (ddd, 7.4, 7.4, 1.9)	7.61 (dd, 5.0, 3.1)
H-5	7.48 (d, 8.4)	7.32 (d, 8.5)		-	7.85 (d, 1.8)	7.35 (dd, 6.5, 2.4)	7.23 (dd, 7.4, 4.5)	7.63 (d, 5.0)
H-6	7.34 (d, 8.4)	7.41 (d, 8.5)	6.88 (d, 1.6)	7.06 (s)	-	7.36 (dd, 6.7, 2.6)	8.63 (dd, 4.5, 1.9)	-
H-7	-	-	-	-	-	7.78 (dd, 6.7, 2.4)	-	-
3-OCH_3	-	-	3.77 (s)	3.81 (s)	-	-	-	-
4-OCH_3	-	-	-	3.70 (s)	-	-	-	-
5-OCH_3	-	-	3.77 (s)	3.81 (s)	-	-	-	-

Note. Multiplicities and coupling constants are given in parentheses. Multiplicities *d* and *s* represent doublet and singlet, respectively.

TABLE 3 ^{13}C NMR chemical shifts of 1-aryl-5-(2-(styryl)phenyl)penta-1,4-dien-3-one derivatives 1–26

Position	1	2	3	4	5	6	7	8	9	10	11	12	13
C-1″	130.9	129.5	129.6	129.5	129.5	130.1	129.5	129.5	129.5	129.6	131.0	129.5	129.9
C-2″	129.2	128.1	128.1	128.1	128.1	128.2	128.1	128.1	128.1	128.0	129.2	128.1	128.2
C-3″	115.2	114.3	114.3	114.3	114.3	114.4	114.3	114.2	114.2	114.3	115.2	114.3	114.4
C-4″	160.8	159.3	159.3	159.3	159.3	159.8	159.3	159.3	159.3	159.3	160.8	159.3	159.9
C-5″	115.2	114.3	114.3	114.3	114.3	114.4	114.3	114.2	114.2	114.3	115.2	114.3	114.4
C-6″	129.2	128.1	128.1	128.1	128.1	128.2	128.1	128.1	128.1	128.0	129.2	128.1	128.2
4″-OCH$_3$	56.0	55.2	55.2	55.2	55.2	55.5	55.2	55.1	55.2	55.2	56.0	55.2	55.5
C-1″α	133.4	132.0	131.8	132.0	132.0	131.7	131.8	131.9	131.8	131.7	133.1	132.0	132.2
C-1″β	125.6	124.4	124.6	124.6	124.6	125.8	124.6	124.5	124.5	125.1	125.6	124.4	125.4
C-1′	115.9	114.6	114.8	114.7	114.7	116.3	114.8	114.6	114.8	114.8	116.1	114.7	115.7
C-2′	142.9	141.3	141.0	141.2	141.2	141.5	141.0	141.1	141.0	140.9	142.6	141.1	142.1
C-3′	105.2	104.1	104.0	104.1	104.0	103.8	104.0	104.0	104.0	104.1	105.1	103.9	104.1
C-4′	163.3	161.6	161.4	161.5	161.5	161.5	161.4	161.4	161.3	161.3	163.0	161.5	162.0
C-5′	98.7	97.8	97.8	97.8	97.8	97.8	97.8	97.8	97.8	97.8	98.7	97.8	97.8
C-6′	162.2	160.6	160.5	160.6	160.6	160.8	160.4	160.5	160.5	160.4	162.1	160.6	161.1
4′-OCH$_3$	56.3	55.5	55.5	55.5	55.5	55.6	55.5	55.4	55.5	55.5	56.3	55.5	55.6
6′-OCH$_3$	56.6	55.9	55.9	55.9	55.9	55.8	55.9	55.8	55.9	55.9	56.6	55.9	55.9
C-1′β	138.8	136.9	136.3	136.6	136.9	137.2	135.9	136.3	135.9	135.3	137.9	136.7	138.7
C-1′α	129.1	128.1	128.4	128.3	128.1	128.9	128.6	128.2	128.8	129.0	129.9	128.7	128.1
C=O	189.8	188.7	188.6	188.8	188.8	189.8	188.6	188.5	188.6	189.4	190.1	188.8	189.0
C-1α	131.3	127.2	124.3	126.3	126.7	124.5	123.9	125.0	123.5	124.8	128.6	126.4	129.0
C-1β	140.0	135.9	142.4	136.6	141.8	142.5	136.9	136.4	136.5	133.3	137.0	135.7	139.4
C-1	142.8	128.2	127.5	123.0	136.1	127.8	115.9	121.0	114.3	105.0	123.6	123.8	137.0
C-2	130.4	148.1	110.5	158.1	113.1	130.1	159.8	152.8	154.0	161.2	166.2	156.3	122.5
C-3	125.0	152.8	149.0	111.8	159.6	114.4	98.3	141.8	97.6	91.0	118.2	118.4	148.8
C-4	149.3	114.8	151.1	132.0	121.2	161.6	162.8	155.5	152.6	163.2	127.7	133.8	124.4
C-5	125.0	124.3	111.6	120.7	129.9	114.4	106.3	108.4	143.1	91.0	140.2	110.7	130.0
C-6	130.4	118.9	123.4	128.4	116.4	130.1	130.0	123.2	110.8	161.2	125.8	130.3	134.1
2-OCH$_3$	-	60.8	-	55.6	-	-	55.7	61.3	56.3	55.9	-	-	-
3-OCH$_3$	-	55.8	55.6	-	55.2	-	-	60.4	-	-	-	-	-
4-OCH$_3$	-	-	55.6	-	-	55.5	55.7	56.0	55.8	55.5	-	-	-
5-OCH$_3$	-	-	-	-	-	-	-	-	56.2	-	-	-	-
6-OCH$_3$	-	-	-	-	-	-	-	-	-	55.9	-	-	-
Position	**14**	**15**	**16**	**17**	**18**	**19**	**20**	**21**	**22**	**23**	**24**	**25**	**26**
C-1″	129.5	129.5	129.5	130.0	129.5	130.0	130.0	129.5	129.5	129.5	130.0	130.0	129.5
C-2″	128.2	128.1	128.1	128.2	128.1	128.2	128.2	128.1	128.1	128.1	128.2	128.2	128.1
C-3″	114.3	114.2	114.3	114.4	114.3	114.4	114.4	114.3	114.2	114.3	114.4	114.3	114.3
C-4″	159.3	159.3	159.3	159.9	159.3	159.8	159.8	159.3	159.3	159.3	159.8	159.7	159.3
C-5″	114.3	114.2	114.3	114.4	114.3	114.4	114.4	114.3	114.2	114.3	114.4	114.3	114.3
C-6″	128.2	128.1	128.1	128.2	128.1	128.2	128.2	128.1	128.1	128.13	128.2	128.2	128.1
4″-OCH$_3$	55.2	55.1	55.2	55.5	55.1	55.5	55.5	55.1	55.2	55.2	55.5	55.4	55.2
C-1″α	132.1	132.0	131.9	132.1	131.9	131.9	131.9	131.9	131.9	132.0	132.0	132.0	131.9

(Continues)

TABLE 3 (Continued)

Position	14	15	16	17	18	19	20	21	22	23	24	25	26
C-1″β	124.4	124.5	124.6	125.5	124.6	125.7	125.7	124.6	124.5	124.5	125.0	125.3	124.6
C-1′	114.5	114.6	114.7	115.9	114.67	116.0	116.0	114.7	114.7	114.6	116.0	115.8	114.7
C-2′	141.4	141.3	141.2	142.0	141.1	141.8	141.8	141.2	141.1	141.2	141.9	141.9	141.1
C-3′	104.1	104.0	104.0	104.0	104.0	104.0	104.0	104.1	104.0	104.1	103.9	103.8	104.0
C-4′	161.7	161.6	161.5	162.0	161.5	161.8	161.8	161.6	161.5	161.6	161.9	161.8	161.5
C-5′	97.8	97.8	97.8	97.8	97.8	97.8	97.7	97.8	97.8	97.8	97.8	97.7	97.8
C-6′	160.7	160.6	160.6	161.0	160.5	160.9	160.9	160.6	160.6	160.6	161.0	161.0	160.5
4′-OCH_3	55.5	55.5	55.5	55.6	55.5	55.6	55.6	55.5	55.5	55.5	55.8	55.7	55.5
6′-OCH_3	55.9	55.9	55.9	55.9	55.9	55.8	55.8	55.9	55.9	55.9	55.6	55.5	55.9
C-1′β	137.3	137.2	136.8	138.4	136.7	138.1	138.0	136.7	136.6	136.5	138.0	138.2	136.5
C-1′α	128.1	128.1	128.1	128.3	128.1	128.4	128.4	128.07	128.2	128.1	128.6	128.7	128.1
C=O	188.5	188.7	188.6	189.3	188.6	189.5	189.5	188.8	188.7	188.0	189.1	189.9	188.9
C-1α	128.4	127.7	126.3	128.1	126.3	127.0	126.9	126.9	125.8	123.4	127.7	129.8	125.8
C-1β	133.3	140.3	140.6	140.6	142.1	141.1	141.0	142.0	142.3	128.6	135.5	140.8	135.8
C-1	122.4	137.3	131.4	136.1	136.0	134.1	133.7	136.9	130.3	-	-	-	-
C-2	160.8	114.4	130.8	124.8	119.5	129.8	129.5	106.3	106.1	151.0	140.7	153.7	129.8
C-3	116.1	162.5	115.9	131.4	157.7	132.2	129.2	160.7	153.1	116.5	129.3	124.9	138.1
C-4	132.3	117.0	163.2	126.6	117.6	124.5	136.1	102.6	139.6	113.0	124.5	136.8	126.1
C-5	125.0	130.8	115.9	129.5	129.9	132.2	129.2	160.7	153.1	146.0	125.0	124.1	127.7
C-6	129.1	125.0	130.8	131.5	114.73	129.8	129.5	106.3	106.1	-	126.3	150.1	-
C-7	-	-	-	-	-	-	-	-	-	-	122.6	-	-
C-8	-	-	-	-	-	-	-	-	-	-	140.3	-	-
C-9	-	-	-	-	-	-	-	-	-	-	139.9	-	-
3-OCH_3	-	-	-	-	-	-	-	55.4	56.0	-	-	-	-
4-OCH_3	-	-	-	-	-	-	-	-	60.1	-	-	-	-
5-OCH_3	-	-	-	-	-	-	-	55.4	56.0	-	-	-	-
3-CF_3	-	-	-	122.6	-	-	-	-	-	-	-	-	-

from the HMBC correlations (Figure 2). In the HMBC spectrum (Figure 3), the proton signal at 7.98 ppm was correlated to C-2′ and C-6′, as was thus assigned to H-1′β, whereas the other proton signal at 7.82 ppm was correlated to the ^{13}C signals of C-6 (118.9 ppm) and C-2 (148.1 ppm), and the proton was thus assigned as H-1β. The structure of the other derivatives is identical except for the R group, and the NMR assignments followed the same approach as used for derivative 2. The full NMR assignments for all derivatives are presented in Tables 2 and 3.

All derivatives possess identical structures except for the R group. For derivatives 1–22, the R group was a benzene ring with different substituents, and the R group was heteroaromatics for derivatives 23–26, as shown in Table 1. Therefore, the ^1H and ^{13}C chemical shifts related to 1β, which is close to the R group, varied depending on the substituents of the R group. The chemical shifts of C-1β varied from 128.6 to 142.5 ppm. For the derivatives containing a substituent at the *ortho* position of the benzene ring in the R group, the chemical shifts of C-1β were located at relatively low frequency. For example, the chemical shifts of C-1β for derivatives 2, 4, 7, 8, 9, 10, 11, 12, and 14 containing methoxy, hydroxyl, or fluoride at the *ortho* position were lower than 137 ppm. Derivative 23 and 24 with chemical shifts of 128.6 and 135.5 ppm for C-1β contained furan and benzothiofuran as the R group, respectively. For the other derivatives having hydrogen at the *ortho* position, the chemical shifts of C-1β occurred at frequencies higher than 139.4 ppm. On the other hand, the chemical shifts of H-1β tended to be at higher frequency when substituents were present at the *ortho* position. The chemical shift of H-1β for derivative 4 having a methoxy group at the *ortho* position was 7.87 ppm, whereas the chemical shift was 7.55 or 7.61 ppm for derivatives 5 (methoxy at *meta* position) and 6 (methoxy at *para* position), respectively.

Some of the carbon chemical shifts also varied based on the solvent utilized.[23] For example, the chemical shift of C-1′ varied from 114.5 to 116.3 ppm depending on the solvent. In DMSO, the chemical shift ranged from 114.5 to 114.8 ppm, whereas the range varied from 115.7 to 116.3 ppm in chloroform or DMF. The chemical shift of C-1′β also varied depending on the solvent. The peak of C-1′β shifted from 135.3 to 137.3 ppm in DMSO but was observed at 137.2–138.7 ppm in chloroform. In DMF, the chemical shift of C-1′β ranged from 137.9 to 138.8 ppm. These carbons may be more greatly influenced by electrostatic interactions with the solvent than the other carbons. The physicochemical properties of the derivatives such as the area, volume, and log *P* were also predicted using the Sybyl software. These data will be essential for further developments of the drug. Biological activities including anticancer and targets mitochondrial metabolic pathways that 1-aryl-5-(2-(styryl)phenyl)penta-1,4-dien-3-one derivatives are expected to show are remained for further study.

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