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MICROWAVE-ASSISTED SYNTHESIS OF SUBSTITUTED NAPHTHO[2,3-*c*]FURAN-1,3-DIONE DERIVATIVES

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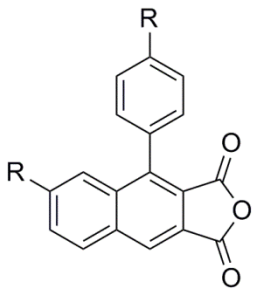
Abstract – The self-condensation reaction of substituted phenylpropiolic acid derivatives (**5a-d**) by microwave irradiation at 100 °C for only three minutes proceeded smoothly to give substituted naphtho[2,3-*c*]furan-1,3-dione derivatives (**1a-d**) as single products in moderate to good yields, respectively.

INTRODUCTION

Naphtho[2,3-*c*]furan-1,3-dione derivatives have attracted attention in a wide range of research areas from pharmaceutical science to materials science, because they have made a great contribution to the preparation of functional molecules such as lactone lignans, environment-sensitive fluorophores, and phthalocyanines. Lactone lignans are important secondary plant metabolites possessing a variety of biological activities.[1,2] Environment-sensitive fluorophores can be used to monitor quantitate formation of protein-protein interactions.[3,4] Phthalocyanines and, especially, their metal complexes are commonly used as dyes, pigments, photoconductive materials in laser printers, and organic semiconductors.[5,6]



R = -H (**1a**); -Me (**1b**); -OMe (**1c**); -Cl (**1d**)

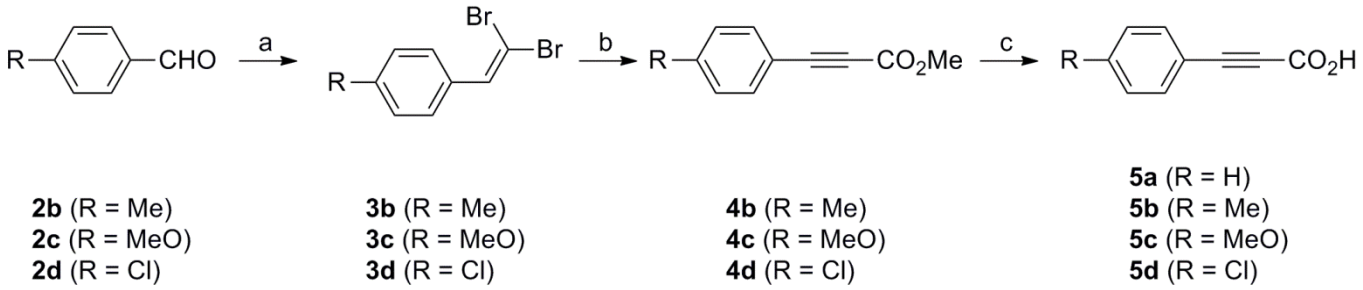
Figure 1. Naphtho[2,3-*c*]furan-1,3-dione derivatives

Therefore, the development of a synthetic method of the naphtho[2,3-*c*]furan-1,3-dione framework would be of significance for exploring novel molecules with attractive functions. Although a number of reaction conditions have already been adopted for the synthesis of this framework, most of them have required vigorous conditions, or resulted in the production of naphtho[2,3-*c*]furan-1,3-dione derivatives in low yields.[7] The low-yield production would be thought to be caused by thermally induced decarbonylation of substituted phenylpropiolic acids, which have been widely used as a starting material.

The use of microwave irradiation in organic synthesis has become a popular technique in the scientific community, because it leads to dramatically reduced reaction times, increased product yields, and enhanced product purities by reducing unwanted side reactions compared to conventional methods.[8,9] We speculated that the use of microwave irradiation should bring us to the effective synthesis of naphtho[2,3-*c*]furan-1,3-dione derivatives. In this paper, we report the microwave-assisted synthesis of substituted naphtho[2,3-*c*]furan-1,3-dione derivatives from substituted phenylpropiolic acids.

RESULTS AND DISCUSSION

We first prepared substituted phenylpropiolic acid derivatives (**5b-d**) from substituted benzaldehydes (**2b-d**), respectively. Scheme 1 provides an outline for the preparation of **2b-d**. Unsubstituted phenylpropiolic acid (**5a**) is commercially available.



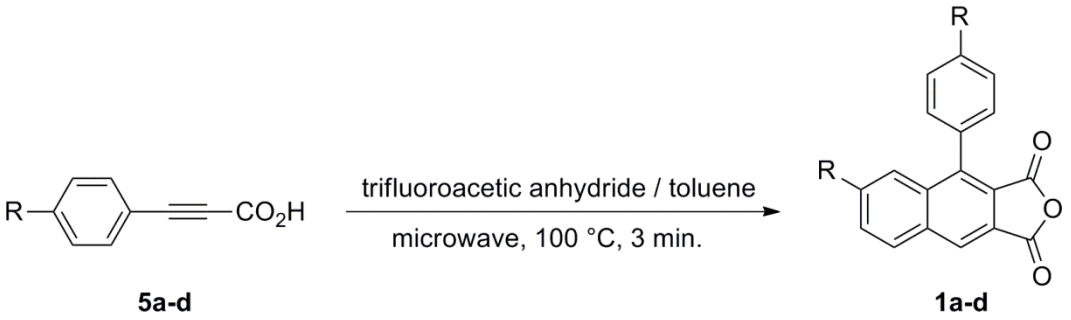
Scheme 1. Preparation of substituted phenylpropiolic acid derivatives (**5b-d**). Reagents and conditions: (a) triphenylphosphine, carbon tetrabromide, CH_2Cl_2, 0 °C (91% for **3b**, 97% for **3c**, and 91% for **3d**); (b) (i) *n*-BuLi, THF, -20 °C, (ii) methyl chloroformate, THF, -20 °C (97% for **4b**, 96% for **4c**, and 95% for **4d**); (c) NaOH, H_2O, MeOH, 0 °C (71% for **5b**, 71% for **5c**, and 60% for **5d**).

We carried out the Corey-Fuchs reaction protocols for the preparation of the alkynyl ester derivatives (**4b-d**), which were phenylpropiolic acid precursors, from benzaldehyde derivatives (**2b-d**).[10] The one-carbon homologation reaction of aldehydes (**2b-d**), which were commercially available, using triphenylphosphine and carbon tetrabromide in dichloromethane at 0 °C proceeded smoothly to yield the

corresponding dibromoolefin derivatives (**3b-d**) in excellent yields, respectively. Treatment of the dibromoolefin derivatives (**3b-d**) with 2.2 equivalents of *n*-butyl lithium in tetrahydrofuran (THF) at -20 °C, followed by trapping the intermediate acetylenic anion with methyl chloroformate at the same temperature, provided the corresponding alkynyl ester derivatives (**4b-d**) in good yields, respectively. Finally, hydrolysis of the alkynyl ester derivatives (**4b-d**) with aqueous sodium hydroxide in methanol, followed by acidification with 1N HCl, provided the desired phenylpropiolic acid derivatives (**5b-d**) in moderate yields, respectively.

The microwave-assisted synthesis of naphtho[2,3-*c*]furan-1,3-dione derivatives by the self-condensation of substituted phenylpropiolic acid derivatives (**5a-d**) was examined at 100 °C (external temperature) for three minutes.[7b,11] The results are summarized in Table 1. The results of the self-condensation reaction of substituted phenylpropiolic acid derivatives (**5a-d**) under the conventional batch (heating in a water bath) method are also shown in Table 1.

Table 1. The microwave-assisted synthesis of naphtho[2,3-*c*]furan-1,3-dione derivatives (**1a-d**)



5a-d **1a-d**

Entry	Compound **5**	Concentration / M	Product	Isolated yield / % by microwave irradiation	Isolated yield / % by conventional batch method
1	**5a** (R = H)	0.10	**1a**	7	trace
2	**5a** (R = H)	0.34	**1a**	72	52
3	**5b** (R = Me)	0.34	**1b**	70	45
4	**5c** (R = MeO)	0.34	**1c**	98	55
5	**5d** (R = Cl)	0.34	**1d**	41	34

The self-condensation reaction of phenylpropiolic acid (**5a**), the concentration of which was adjusted to 0.1 M in toluene, with trifluoroacetic anhydride at 100 °C (external temperature) for three minutes under the conventional heating method resulted in recovery of the starting materials. However, under the condition of

microwave irradiation at 100 °C (external temperature) for three minutes, the self-condensation reaction of **5a** proceeded to give the desired self-condensation product, naphtho[2,3-*c*]furan-1,3-dione (**1a**) in 7% yield, although the starting material **5a** was recovered (entry 1). As seen in entry 2 in Table 1, the self-condensation reaction of **5a** by microwave irradiation under a higher concentration promoted greater efficiency and productivity, and, as a result, we succeeded in obtaining the desired product **1a** in 72% yield. Although the efficiency and productivity of the self-condensation reaction of **5a** by the batch method also was improved, the yield of **1a** by the batch method did not come up to that by microwave irradiation. We also examined the self-condensation reaction of other phenylpropiolic acid derivatives (**5b-d**) in 0.3 M in toluene by microwave irradiation (entries 3-5). It is worth noting that the reaction of phenylpropiolic acid with a methoxy substituent (**5c**) proceeded smoothly to yield the desired substituted naphtho[2,3-*c*]furan-1,3-dione derivative (**1c**) in a quantitative yield (entry 4). The reaction of phenylpropiolic acid with a chloro group (**5d**) resulted in a moderate yield of the desired product (**1d**), along with the recovery of **5d** (entry 5). Therefore, the self-condensation reaction by microwave irradiation showed a remarkable substitution effect, like that by the conventional batch method. Consequently, the reaction by microwave irradiation in this study was found to be superior to that by the conventional batch methods. In general, toluene has been considered an unsuitable solvent for the microwave irradiation synthesis, because it shows a poor absorption of microwaves.[12] Our self-condensation reaction in this study might have been assisted by trifluoroacetic acid, which was produced *in situ* in accordance with the ongoing reaction, because trifluoroacetic acid was a more suitable reagent than toluene for the microwave-assisted synthesis. Then, the microwave heating effects of adding of trifluoroacetic acid to toluene were investigated and summarized in Figure 2.

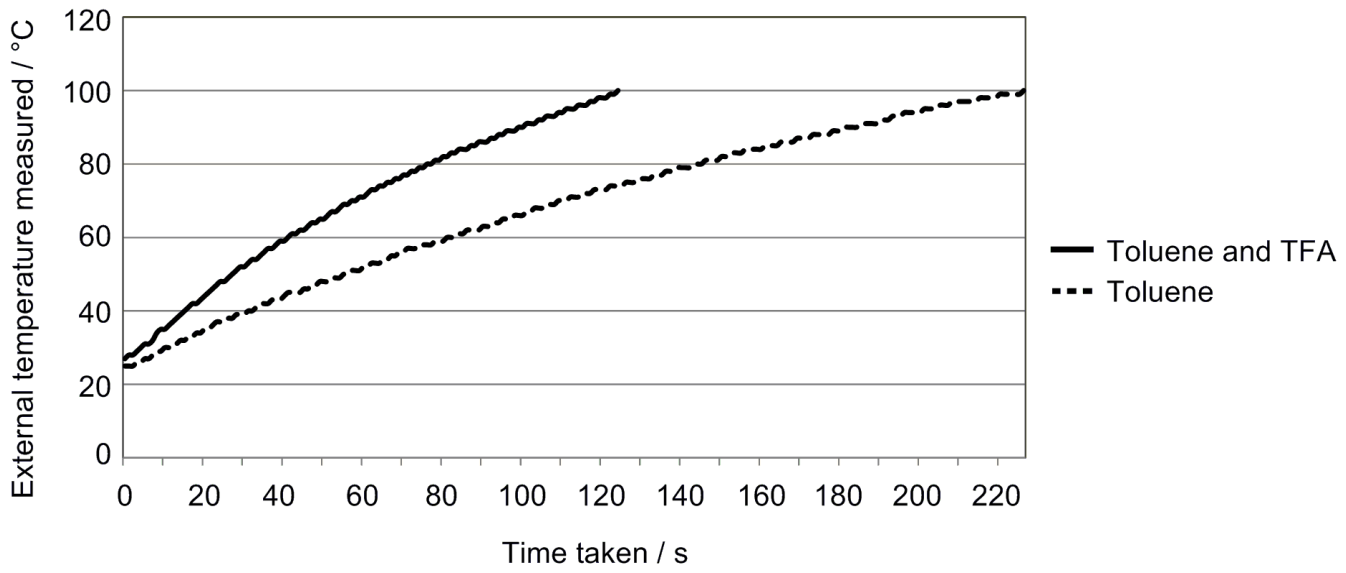


Figure 2. The microwave heating effects of adding of trifluoroacetic acid (TFA) to toluene

In the absence of trifuloroacetic acid, it took 226 s to reach the preset temperature of 100 °C (external temperature). However, in the presence of trifluoroacetic acid, the same temperature was reached 124 s. These results indicate that trifluoroacetic acid has a dramatic effect on the heating of toluene under microwave irradiation. Therefore, we concluded that the self-condensation in this study, which was ascribed to Diels-Alder type reaction,[13] was accelerated by microwave irradiation.

In conclusion, we have demonstrated that the microwave-assisted reaction of substituted phenylpropiolic acid derivatives proceeds smoothly to yield naphtho[2,3-*c*]furan-1,3-dione derivatives in moderate-to -good yields. Microwave-assisted reactions by two different substituted phenylpropiolic acid derivatives would provide us a variety of naphtho[2,3-*c*]furan-1,3-dione derivatives with different substituents, and its regioselectivity by this reaction should be also a matter of deep interest. In addition of the detailed substituent effect, further studies regarding the regioselectivity by the microwave-assisted reactions are in progress.

EXPERIMENTAL

All conventional reactions were carried out under an argon atmosphere with dry solvents under anhydrous conditions unless otherwise noted. All microwave irradiation experiments were carried out in a Discover SP microwave synthesizer of CEM Corporation. Solvents and reagents were purified by literature methods where necessary.[14] All melting points were determined on a Büchi melting point apparatus (B-540) and are uncorrected. Infrared (IR) spectra were recorded on a JASCO FT/IR-4100 FT-IR spectrometer. UV absorption spectra were obtained using a JASCO V-650 spectrophotometer. ^{1}H NMR spectra were recorded on a JEOL JNM-ECA500 (500 MHz) spectrometer; chemical shifts (δ) are reported in parts per million relative to tetramethylsilane (TMS). Splitting patterns are designated as s, singlet; d, doublet; t, triplet; q, quartet; m, multiplet; br, broad; dsp, double of septets. Mass spectra (MS) were recorded on a Shimadzu LCMS-IT-TOF mass spectrometer. Elemental analyses were performed by Mrs. Ayako Sato of A Rabbit Science Japan Corporation. Column chromatography was performed on Kanto Chemical silica gel 60N (spherical, neutral). Phenylpropiolic acid (**5a**) was commercially available.

Typical procedure for the preparation of dibromoolefin derivatives (3).

1-(2,2-Dibromoethenyl)-4-methylbenzene (3b)

A mixture of triphenylphosphine (8.73 g, 33.3 mmol) and carbon tetrabromide (5.52 g, 16.6 mmol) in CH_2Cl_2 was stirred at 0 °C for 1 h. To the resulting reaction mixture was added a solution of 4-methylbenzaldehyde **2b** (1.00 g, 8.32 mmol) in CH_2Cl_2 at 0 °C. After being stirred at the same temperature for 3 h, the resulting precipitate was filtered off and the filtrate was evaporated to dryness under

reduced pressure. The residue was purified by column chromatography (silica gel, hexane-EtOAc) give the desired product **3b** (2.09 g, 91%) as pale yellow oil. Mp 30.0-32.0 °C (lit.,[15] mp 24 °C); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.45 (s, 1H), 7.43 (d, 2H, J = 8.0 Hz), 7.18 (d, 2H, J = 8.0 Hz), 2.34 (s, 3H). [CAS No. 60512-56-3]

1-(2,2-Dibromoethenyl)-4-methoxybenzene (3c)

This compound was prepared from 4-methoxybenzaldehyde according to the method used for the preparation of **3b**. Pale yellow solids; yield 97%; mp 34.0-35.5 °C (lit.,[16] mp 34-36 °C); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.51 (d, 2H, J = 8.9 Hz), 7.41 (s, 1H), 6.89 (d, 2H, J = 8.9 Hz), 3.82 (s, 3H). [CAS No. 60512-57-4]

1-(2,2-Dibromoethenyl)-4-chlorobenzene (3d)

This compound was prepared from 4-chlorobenzaldehyde according to the method used for the preparation of **3b**. Pale yellow solids; yield 91%; mp 32.0-36.0 °C (lit.,[15] mp 34-36 °C); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.47 (d, 2H, J = 8.6 Hz), 7.43 (s, 1H), 7.34 (d, 2H, J = 8.6 Hz). [CAS No. 77295-59-1]

Typical procedure for the preparation of substituted alkynyl ester derivatives (4).

Methyl 4-methylphenylpropiolate (4b)

To a solution of dibromoolefin **3b** (0.500 g, 1.81 mmol) in THF was added dropwise *n*-butyllthium (1.6 M in hexane) (2.80 mL, 3.98 mmol) at -20 °C. After being stirred at the same temperature for 2 h, a solution of methyl chloroformate (0.17 mL, 2.17 mmol) in THF was added dropwise to the reaction mixture at the same temperature. After being stirred at the same temperature for 6 h, the resulting solution was quenched with saturated aqueous NH_4Cl and then extracted with EtOAc. The organic layer was washed with H_2O and brine, respectively, and the dried over anhydrous $MgSO_4$. After removal of the solvent, the residue was purified by column chromatography to give the desired product **4b** (0.307 g, 97%) as pale yellow solids. Mp 64.5-66.0 °C (lit.,[17] mp 63-65 °C); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.48 (d, 2H, J = 8.1 Hz), 7.18 (d, 2H, J = 8.1 Hz), 3.84 (s, 3H), 2.38 (s, 3H). [CAS No. 7515-16-4]

Methyl 4-methoxyphenylpropiolate (4c)

This compound was prepared from dibromoolefin derivative (**3c**) according to the method used for the preparation of **4b**. Colorless solids; yield 96%; mp 43.0-45.0 °C (lit.,[18] mp 43-44 °C); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.54 (d, 2H, J = 8.9 Hz), 6.89 (d, 2H, J = 8.9 Hz), 3.84 (s, 3H), 3.83 (s, 3H). [CAS No. 7515-17-5]

Methyl 4-chlorophenylpropiolate (4d)

This compound was prepared from dibromoolefin derivative (**3d**) according to the method used for the preparation of **4b**. Colorless solids, yield 95%; mp 90.0-92.0 °C (lit.,[17] mp 90-91 °C); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.51 (d, 2H, J = 8.7 Hz), 7.37 (d, 2H, J = 8.7 Hz), 3.03 (s, 3H). [CAS No. 7515-18-6]

Typical procedure for the preparation of substituted phenylpropiolic acid derivatives (5).

(4-Methylphenyl)propiolic acid (5b)

To a solution of methyl 4-methylphenyl propiolate (**4b**) (1.00 g, 5.74 mmol) in MeOH was added dropwise an aqueous NaOH (0.2 N; 32 mL, 6.31 mmol) at 0 °C. After being stirred at room temperature for 2 h, the solvent was evaporated under reduced pressure. After being diluted with H_2O, followed by washing with *t*-BuOMe, the resulting water layer was adjusted to pH 2-3 by 1.0 N HCl. After being extracted with EtOAc, the organic layer was washed with H_2O, and brine, respectively, and then dried over anhydrous $MgSO_4$. After removal of the solvent, the residue was recrystallized to give the desired product (0.651 g, 71%) as colorless solids. Mp 146.5-148.5 °C (lit.,[19] mp 147-148 °C); ^{1}H NMR (500 MHz, $CDCl_3$+D_2O) δ7.50 (d, 2H, J = 8.1 Hz), 7.20 (d, 2H, J = 8.1 Hz), 2.40 (s, 3H). [CAS No. 2227-58-9]

(4-Methoxyphenyl)propiolic acid (5c)

This compound was prepared from alkynyl ester derivative (**4c**) according to the method used for the preparation of **5b**. Colorless solids; yield 71%; mp 145.0-146.0 °C (lit.,[20] mp 144-145 °C); ^{1}H NMR (500 MHz, $CDCl_3$+D_2O) δ7.57 (d, 2H, J = 8.9 Hz), 6.90 (d, 2H, J = 8.9 Hz), 3.85 (s, 3H). [CAS No. 2227-57-8]

(4-Chlorophenyl)propiolic acid (5d)

This compound was prepared from alkynyl ester derivative (**4d**) according to the method used for the preparation of **5b**. Colorless solids; yield 60%; mp 190.5-192.0 °C (lit.,[19] mp 191-193 °C); ^{1}H NMR (500 MHz, $CDCl_3$+D_2O) δ7.54 (d, 2H, J = 8.3 Hz), 7.38 (d, 2H, J = 8.3 Hz). [CAS No. 3240-10-6]

Microwave-assisted synthesis of substituted naphtho[2,3-*c*]furan-1,3-dione derivatives (1)

1-Phenylnaphtho[2,3-*c*]furan-1,3-dione (1a)

A mixture of phenylpropiolic acid **1a** (0.500 g, 3.42 mmol) and trifluoroacetic anhydride (0.482 mL, 3.42 mmol) in toluene (10 mL) was prepared in a 10-mL sealed reaction vessel, which was set in the Discover SP microwave synthesizer (CEM Corporation). After being stirred at 100 °C (external temperature) for 3 min, resulting reaction solution was cooled to room temperature and evaporated under reduced pressure. The resulting residue was diluted with EtOAc, and quenched with saturated aqueous $NaHCO_3$. The organic layer was washed with H_2O and brine, respectively, and then dried over anhydrous $MgSO_4$. After removal of the solvent the residue was purified by column chromatography (silica gel, hexane- EtOAc) to give the desired product **1a** (0.335 g, 72%) as colorless solids. Mp 259.0-260.0 °C (lit.,[21] mp 260-262 °C); ^{1}H NMR (500 MHz, $CDCl_3$) δ8.57 (s, 1H), 8.18 (d, 1H, J = 8.2 Hz), 7.92 (d, 1H, J = 8.5 Hz), 7.80 (ddd, 1H, J = 1.2, 6.9, 8.2 Hz), 7.71 (ddd, 1H, J = 1.1, 6.9, 8.5 Hz), 7.59 (m, 3H), 7.42 (m, 2H). [CAS No. 1985-37-1]

6-Methyl-4-(4-methylphenyl)naphtho[2,3-*c*]furan-1,3-dione (1b)

This compound was prepared from carboxylic acid derivative (**5b**) according to the method used for the

preparation of **1a**. Colorless solids; yield 70%; mp 269.0-270.0 °C (lit.,[7c] mp 268-269 °C); ^{1}H NMR (500 MHz, $CDCl_3$) δ8.49 (s, 1H), 8.05 (d, 1H, *J* = 8.7 Hz), 7.69 (d, 1H, *J* = 1.5 Hz), 7.76 (d, 1H, *J* = 1.5, 8.7 Hz), 7.39 (d, 2H, *J* = 8.2 Hz), 7.29 (d, 2H, *J* = 8.2 Hz), 2.51 (s, 3H), 2.50 (s, 3H). [CAS No. 32050-01-4]

6-Methoxy-4-(4-methoxyphenyl)naphtho[2,3-*c*]furan-1,3-dione (1c)

This compound was prepared from carboxylic acid derivative (**5c**) according to the method used for the preparation of **1a**. Colorless solids; yield 98%; mp 263.0-265.5 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ8.44 (s, 1H), 8.04 (d, 1H, *J* = 8.7 Hz), 7.42 (d, 1H, *J* = 2.5, 8.7 Hz), 7.36 (d, 2H, *J* = 8.8 Hz), 7.23 (d, 1H, *J* = 2.5 Hz), 7.10 (d, 2H, *J* = 8.8 Hz) 3.93 (s, 3H), 3.79 (s, 3H). [CAS No. 15828-76-9]

6-Chloro-4-(4-chlorophenyl)naphtho[2,3-*c*]furan-1,3-dione (1d)

This compound was prepared from carboxylic acid derivative (**5d**) according to the method used for the preparation of **1a**. Colorless solids; yield 41%; mp 264.5-266.5 °C (lit.,[7b] mp 266-267 °C); ^{1}H NMR (500 MHz, $CDCl_3$) δ8.56 (s, 1H), 8.13 (d, 1H, *J* = 9.0 Hz), 7.83 (d, 1H, *J* = 2.2 Hz), 7.76 (d, 1H, *J* = 2.2, 9.0 Hz), 7.59 (d, 2H, *J* = 8.5 Hz), 7.35 (d, 2H, *J* = 8.5 Hz). [CAS No. 7495-50-3]

Synthesis of 1-phenylnaphtho[2,3-*c*]furan-1,3-dione (1a) under the conventional batch method

To a mixture of phenylpropiolic acid **1a** (0.500 g, 3.42 mmol) in tolunene (10 mL) was added trifluoroacetic anhydride (0.482 mL, 3.42 mmol) at room temperature. After being stirred at 100 °C (external temperature) for 3 min in oil bath, the resulting reaction solution was cooled to room temperature and evaporated under reduced pressure. The resulting residue was diluted with EtOAc, and quenched with saturated aqueous $NaHCO_3$. The organic layer was washed with H_2O and brine, respectively, and then dried over anhydrous $MgSO_4$. After removal of the solvent the residue was purified by column chromatography (silica gel, hexane- EtOAc) to give the desired product **1a** (0.241 g, 52%) as colorless solids.

Microwave heating effects of adding of trifluoroacetic acid to toluene

A solution of trifluoroacetic anhydride (0.260 mL, 3.42 mmol) in toluene (10 mL) was prepared in a 10-mL sealed reaction vessel, which was set in the Discover SP microwave synthesizer (CEM Corporation). The irradiation power was set to 300 W, and time taken to reach 100 °C (external temperature) was measured.

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