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First Synthesis of (±)-Basidifferquinone C, an Inducer for Fruiting-Body Formation in *Polyporus arcularius*

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Basidifferquinones, isolated from *Streptomyces* sp., are potent inducers of fruiting-body formation in the basidiomycete, *Polyporus arcularius*. The first synthesis of (±)-basidifferquinone C was accomplished by starting from 3,5-dihydroxy-2-naphthoic acid.

Key words: basidifferquinone; fruiting-body inducer; anthraquinone; Diels-Alder reaction

Fruiting-body development in basidiomycetes is the most striking expression of differentiation and morphogenesis among fungi; it is known to be induced not only by environmental and physical stimuli, but also by various chemicals.[1–6] In 1990, basidifferquinone, an inducer for fruiting-body formation in *Polyporus arcularius*, was isolated from a culture medium of *Streptomyces* sp. B-412 by Azuma *et al.*[7,8] They then also reported in 1993 the isolation of two basidifferquinone relatives.[9] Thus, the originally isolated basidifferquinone was renamed basidifferquinone A (BDQ A, **1**), and the others were named basidifferquinone B (BDQ B, **2**) and basidifferquinone C (BDQ C, **3**), respectively, as shown in Scheme 1.[9] Despite their interesting biological activity and structural uniqueness, no synthetic studies on BDQs have so far been disclosed, apart from a preliminary communication by the present authors.[10] We report here the first synthesis of (±)-**3** with experimental details.

Results and Discussion

As already discussed in our preliminary communication, the construction of the C-ring portion of BDQs proved to be much more problematic than expected.[10] We therefore designed a new and simple synthetic plan as shown in Scheme 1. This plan allowed us to circumvent the difficulty of C-ring construction. The target compound, BDQ C (**3**), should be prepared by a Diels-Alder reaction between naphthoquinone derivative **A** and a properly functionalized diene. For the synthesis of **A**, an appropriate precursor was **B**, because oxidation of **B** to **A** was feasible. To construct the lactone ring of **B**, we envisaged adopting *ortho*-lithiation[11] of **C** and subsequent trapping with PhCHO. Amide **C** could be prepared from commercially available starting material **D**.

Scheme 2 illustrates our synthesis of (±)-**3**. The starting material was 3,5-dihydroxy-2-naphthoic acid **4** (= **D**). This was converted to corresponding protected amide **7** (= **C**) in three standard steps: methoxymethyl (MOM) protection (82%), hydrolysis (92%), and amidation (89%). The *ortho*-lithiation of **7** was successfully performed by treating with *n*-BuLi in the presence of *N*,*N*,*N*′,*N*′-tetramethylethylenediamine (TMEDA),[12] and generated 1-lithio-**7** was trapped with benzaldehyde to give an adduct which was then immediately heated in refluxing toluene to afford desired lactone **8** (= **B**) in 59% yield (70% based on recovered SM). In this case, *n*-BuLi was better than *s*-BuLi as a base, and TMEDA was preferable as an additive. The oxidation of **8** was successfully achieved by treating with ceric ammonium nitrate (CAN) to give **9** (= **A**) in quantitative yield.

With naphthoquinone **9** in hand, we then attempted to carry out the Diels-Alder reaction between **9** and Danishefsky-Kitahara diene **10**[13,14] under Tietze's conditions.[15] The Diels-Alder reaction occurred in CH_2Cl_2 at room temperature to give the adducts. These adducts were then successively treated with SiO_2 and K_2CO_3 to afford the desired anthraquinone **11** and its regioisomer **11**′ (8-OH isomer) in 39% yield as an inseparable mixture. The ratio of **11**:**11**′ was estimated to be 55:45 based on ^{1}H-NMR analysis. The mixture of **11** and **11**′ was then converted to a mixture of **12** and **12**′ (55:45) in 99% yield by cleavage of the MOM protecting group. The structures of **12**/**12**′ were confirmed by HMBC spectroscopy as depicted in Scheme 2. The overall yield of the mixture of **12** and **12**′ was 19% in seven steps based on starting material **4**.

We next focused on the synthesis of (±)-BDQ C (**3**). After the preparation of diene **13**,[15,16] a Diels-Alder reaction between **9** and **13** was effected in a similar manner. Although desired product **14** and its regioisomer

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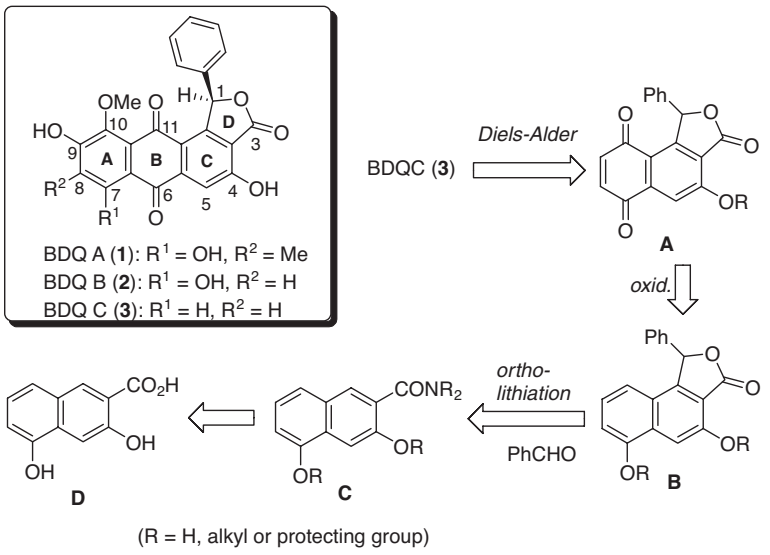
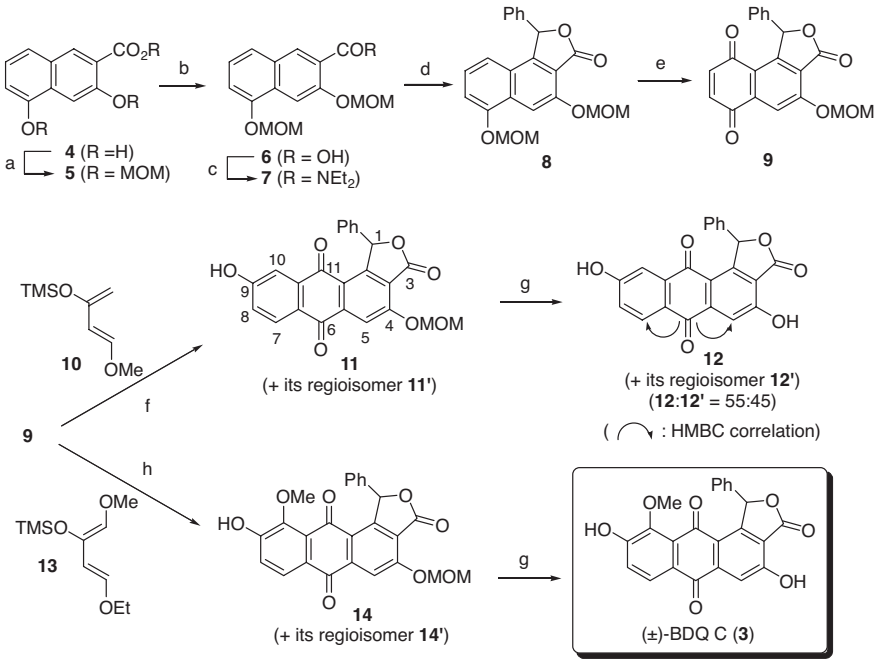


Scheme 1. Structures of BDQs and Synthetic Plan for BDQ C.



Scheme 2. Synthesis of (±)-**3**.

Reagents and conditions: (a) MOMCl, $^{i}Pr_2NEt$, DMF (82%); (b) KOH, aq. MeOH (92%); (c) EtOCOCl, Et_3N, THF then Et_2NH (89%); (d) i) *n*-BuLi, TMEDA, THF −78 °C, then PhCHO; ii) toluene reflux [59% (70% based on the recovered SM)]; (e) CAN, aq. CH_3CN (quant.); (f) i) CH_2Cl_2, r.t., 4.5 h; ii) SiO_2, THF; iii) K_2CO_3, aq. THF (39%; **11**:**11**′ = 55:45); (g) *p*-TsOH, MeOH, $CHCl_3$ (99% for **12**/**12**′; 95% for **3**); (h) i) CH_2Cl_2, r.t., 2 h, then SiO_2; ii) K_2CO_3, aq. THF (26%; **14**:**14**′ = 1:1)

14′ (7-OMe 8-OH isomer) were obtained, the reproducibility, yield and regioselectivity were all worse in comparison to the case with **10**. After a considerable number of attempts to optimize the reaction conditions, we found the following: i) removal of CH_2Cl_2 before the SiO_2 treatment was not necessary and should be avoided to obtain a better yield; ii) a shorter reaction time was of some benefit; iii) a lower temperature did not contribute to a better yield; and iv) the Lewis acid-mediated Diels-Alder reaction and MOM-deprotection of **9** gave no substantive results.[10] These effects may mainly be the results of the intrinsic instability of the substrates, intermediates, and/or products. Regarding the best result, we succeeded in obtaining a mixture of **14** and **14**′ (1:1) in 26% yield. It should be noted that both the isolated yield (17–26%) and the ratio of **14**:**14**′ (1:2–1:1) fluctuated considerably depending on certain unknown factors, even under the foregoing optimized conditions. The regiochemistry of **14**/**14**′ was tentatively estimated on the basis of the NMR spectral similarity between **14**/**14**′ and **12**/**12**′; this was later confirmed by the fact that **14** could be converted to (±)-**3**. The obtained mixture of **14** and **14**′ was carefully purified by preparative TLC to give pure **17** which was then deprotected by treating with *p*-TsOH to give (±)-**3** in 95% yield. The various spectral data for synthetic (±)-**3** were in good accordance with those of the natural product. The overall yield was 6% in seven steps. Finally, a bioassay employing synthetic samples, (±)-**3**, and a mixture of **12** and **12**′, showed that both of these

were active as a fruiting-body inducer for *Polyporus arcularius*, although the latter was less active than the former.

In conclusion, we accomplished the first synthesis of (±)-BDQ C by employing a Diels-Alder strategy. It was shown that synthesized (±)-BDQ C was biologically active as a fruiting-body inducer. Our established synthetic method was relatively simple and straightforward; it will be applicable for the synthesis of other BDQs and their derivatives.

Experimental

IR spectra were measured with a Shimadzu IR-408 spectrometer. ^{1}H-NMR spectra were recorded at 300 MHz with a Jeol JNM-AL300 spectrometer. TMS or the residual solvent peak in $CDCl_3$ (at $\delta_H = 7.26$), DMSO-d_6 (at $\delta_H = 2.49$), or C_5D_5N (at $\delta_H = 7.21$) was used as the internal standard. ^{13}C-NMR spectra were recorded at 75 MHz with the Jeol JNM-AL300 spectrometer, the peak for $CDCl_3$ (at $\delta_C = 77.0$), DMSO-d_6 (at $\delta_C = 39.5$), or C_5D_5N (at $\delta_C = 123.5$) being used as the internal standard. Mass spectra were measured with a Jeol JMS-SX102A spectrometer.

*Methoxymethyl 3,5-bis(methoxymethoxy)-2-naphthoate (**5**).* To an ice-cooled solution of **4** (5.11 g, 25.0 mmol) in DMF (120 ml), $(i\text{-Pr})_2$NEt (18 ml, 0.10 mol) and MOMCl (7.6 ml, 0.10 mol) were successively added. After stirring at room temperature overnight, the reaction mixture was quenched with saturated aq. NH_4Cl and extracted with EtOAc. The organic layer was successively washed with water and brine, dried ($MgSO_4$), and concentrated under reduced pressure. The residue was chromatographed on SiO_2 to give **5** (6.92 g, 20.6 mmol, 82%) as a white solid. Mp 47–48 °C. IR ν_{max} (nujol) cm^{-1}: 1740 (C=O). NMR δ_H ($CDCl_3$): 3.53 (3H, s), 3.57 (3H, s), 3.59 (3H, s), 5.36 (2H, s), 5.40 (2H, s), 5.52 (2H, s), 7.16 (1H, d, $J = 7.8$ Hz), 7.28 (1H, t, $J = 7.8$ Hz), 7.46 (1H, d, $J = 7.8$ Hz), 7.88 (1H, s), 8.32 (1H, s). NMR δ_C ($CDCl_3$): 56.1, 56.2, 57.5, 90.8, 94.7, 94.9, 105.7, 110.2, 121.8, 122.2, 124.7, 128.3, 129.0, 132.3, 151.8, 152.7, 165.4. HRFABMS m/z $[M]^+$: calcd. for $C_{17}H_{20}O_7$, 336.1209; found, 336.1208.

*3,5-Bis(methoxymethoxy)-2-naphthoic acid (**6**).* To a solution of **5** (11.0 g, 32.7 mmol) in MeOH (100 ml), 1.5 M aq. KOH (100 ml) was added and the stirring was continued for 1 h. After evaporating MeOH, the residue was diluted with 1 M HCl and extracted with $CHCl_3$. The organic layer was successively washed with water and brine, dried ($MgSO_4$), and concentrated under reduced pressure. The residue was recrystallized from hexane:ether = 5:1 to give **6** (8.76 g, 30.0 mmol, 92%) as pale-yellow needles. Mp: 107–108 °C. IR ν_{max} (nujol) cm^{-1}: 1700 (C=O). NMR δ_H ($CDCl_3$): 3.56 (3H, s), 3.62 (3H, s), 5.40 (2H, s), 5.56 (2H, s), 7.23 (1H, d, $J = 7.8$ Hz), 7.36 (1H, t, $J = 7.8$ Hz), 7.55 (1H, d, $J = 7.8$ Hz), 7.95 (1H, s), 8.73 (1H, s). NMR δ_C ($CDCl_3$): 56.3, 57.2, 94.9, 96.0, 105.5, 111.1, 118.7, 122.5, 125.7, 128.9, 129.8, 135.5, 151.8, 152.0, 165.8. HRFABMS m/z $[M]^+$: calcd. for $C_{15}H_{16}O_6$, 292.0947; found, 292.0944.

N,N-*Diethyl-3,5-bis(methoxymethoxy)-2-naphthamide (**7**).* To an ice-cooled solution of **6** (3.30 g, 11.3 mmol) in THF (100 ml), Et_3N (6.3 ml, 45 mmol) and $ClCO_2Et$ (1.3 ml, 14 mmol) were added. After stirring at 0 °C for 15 min, Et_2NH (1.5 ml, 14 mmol) was added, and the stirring was continued at 0 °C for 15 min. The reaction mixture was quenched with saturated aq. NH_4Cl and extracted with EtOAc. The organic layer was successively washed with water and brine, dried ($MgSO_4$), and concentrated under reduced pressure. The residue was chromatographed on SiO_2 to give **7** (3.48 g, 10.0 mmol, 89%) as colorless crystals. Mp 62–63 °C. IR ν_{max} (nujol) cm^{-1}: 1730 (C=O). NMR δ_H ($CDCl_3$): 1.05 (3H, t, $J = 7.2$ Hz), 1.29 (3H, t, $J = 7.2$ Hz), 3.20 (2H, q, $J = 7.2$ Hz), 3.21–3.78 (2H, m), 3.52 (3H, s), 3.55 (3H, s), 5.30–5.41 (4H, m), 7.12 (1H, d, $J = 7.8$ Hz), 7.28 (1H, t, $J = 7.8$ Hz), 7.42 (1H, d, $J = 7.8$ Hz), 7.67 (1H, s), 7.83 (1H, s). NMR δ_C ($CDCl_3$): 12.8, 14.0, 38.8, 42.7, 56.2, 56.3, 94.6, 94.8, 104.1, 108.8, 121.2, 124.5, 126.49, 126.51, 129.2, 129.9, 150.7, 152.0, 168.2. HRFABMS m/z $[M + H]^+$: calcd. for $C_{19}H_{26}O_5N$, 348.1811; found, 348.1810.

*4,6-Bis(methoxymethoxy)-1-phenylnaphtho[1,2-c]furan-3(1H)-one (**8**).* To a solution of TMEDA (6.1 ml, 41 mmol) and *n*-BuLi (2.77 M in THF; 13 ml, 36 mmol) in THF (150 ml), **7** (9.41 g, 27.1 mmol) in THF (30 ml) was added dropwise at −78 °C under Ar. After stirring for 2 h, PhCHO (5.5 ml, 54 mmol) was added dropwise to this mixture. After stirring at −78 °C for 30 min, the reaction mixture was quenched with saturated aq. NH_4Cl and extracted with EtOAc. The organic layer was successively washed with water and brine, dried ($MgSO_4$), and concentrated under reduced pressure to give a residue which was then heated under reflux in toluene (60 ml) for 3 h. After removing toluene under reduced pressure, the residue was recrystallized from hexane:ether = 1:1 to give recovered **7** (1.51 g, 4.35 mmol) and **8** [6.07 g, 16.0 mmol, 59% (70% based on recovered **8**)] as pale-yellow crystalline powder. Mp 165–166 °C. IR ν_{max} (nujol) cm^{-1}: 1760 (C=O). NMR δ_H ($CDCl_3$): 3.55 (3H, s), 3.63 (3H, s), 5.39 (2H, dd, $J = 6.9$, 7.8 Hz), 5.54 (2H, dd, $J = 6.9$, 8.7 Hz), 6.58 (1H, s), 7.05 (1H, m), 7.16–7.22 (2H, m), 7.24–7.28 (2H, m), 7.34–7.37 (3H, m), 7.98 (1H, s). NMR δ_C ($CDCl_3$): 56.2, 56.5, 81.7, 94.8, 94.9, 106.3, 111.2, 116.0, 117.4, 123.8, 125.6, 128.1, 128.9, 129.5, 130.3, 136.0, 150.6, 151.1, 152.7, 168.0. HRFABMS m/z $[M + H]^+$: calcd. for $C_{22}H_{21}O_6$, 381.1338; found, 381.1338.

*4-Methoxymethoxy-1-phenylnaphtho[1,2-c]furan-3,6,9(1H)-trione (**9**).* To a solution of **8** (980 mg, 2.57 mmol) in CH_3CN (80 ml), a solution of CAN (4.25 g, 10.3 mmol in 30 ml H_2O) was added. After stirring at room temperature for 15 min, a further solution of CAN (1.41 g, 2.57 mmol in 10 ml H_2O) was added. After stirring for 15 min, the reaction mixture was diluted with water and extracted with EtOAc. The organic layer was successively washed with water and brine, dried ($MgSO_4$), and concentrated under reduced pressure. The residue was recrystallized from hexane:ether = 1:1 to give **9** (901 mg, 2.57 mmol, quant.) as red-orange crystalline powder. Mp 167–168 °C. IR ν_{max} (nujol) cm^{-1}: 1760 (C=O), 1660 (C=O). NMR δ_H ($CDCl_3$): 3.52 (3H, s), 5.49 (2H, q-like, $J = 6.6$ Hz), 6.73 (1H, d, $J = 10.2$ Hz), 6.76 (1H, s), 6.87 (1H, d, $J = 10.2$ Hz), 7.11–7.26 (5H, m), 7.86 (1H, s). NMR δ_C ($CDCl_3$): 57.2, 83.2, 95.2, 112.9, 119.3, 120.4, 128.0, 128.5, 129.1, 135.2, 137.9, 138.4, 139.1, 152.8, 159.6, 165.9, 182.4, 183.7. HRFABMS m/z $[M + H]^+$: calcd. for $C_{20}H_{15}O_6$, 351.0869; found, 351.0868.

*9-Hydroxy-4-methoxymethoxy-1-phenylanthra[1,2-c]furan-3,6,11(1H)-trione (**11**) and 8-hydroxy-4-methoxymethoxy-1-phenylanthra[1,2-c]furan-3,6,11(1H)-trione (**11′**).* To a solution of **9** (66 mg, 0.19 mmol) in CH_2Cl_2 (5 ml), **10** (70 µl, 0.36 mmol) was added at room temperature under Ar. After stirring for 4.5 h, the reaction mixture was concentrated under reduced pressure. To this residue, THF (5 ml) and SiO_2 (3 g) were added, and stirring was continued for 30 min. The reaction mixture was filtered, and the filtrate was concentrated under reduced pressure. To this residue, K_2CO_3 (24 mg, 0.19 mmol) in aq. THF (50 vol%; 6 ml) was added. After stirring at room temperature for 30 min, the reaction mixture was diluted with water and extracted with EtOAc. The organic layer was successively washed with water and brine, dried ($MgSO_4$), and concentrated under reduced pressure. The residue was chromatographed on SiO_2 to give an inseparable mixture of **11** and **11′** (55:45; 28 mg, 0.068 mmol, 39%) as yellow crystalline powder. Mp 202–204 °C. IR ν_{max} (nujol) cm^{-1}: 3300 (O–H), 1730 (C=O), 1670 (C=O). NMR δ_H (DMSO): 3.51 (3H, s), 5.63 (2H, dd, $J = 12.3$, 6.9 Hz), 6.99 and 7.00 [total 1H, 2 × s (55:45)], 7.10–7.43 (7H, m), 7.81 and 8.02 [total 1H, 2 × d (45:55), $J = 8.7$ Hz], 7.94 and 7.95 [total 1H, 2 × s (45:55)], 11.0–11.1 (1H, m). NMR δ_C (DMSO): 56.68, 56.70, 83.0, 83.1, 89.5, 95.0, 112.0, 112.4, 112.8, 118.5, 118.9, 121.55, 121.60, 121.7, 121.9, 124.7, 124.9, 127.98, 128.02, 128.5, 128.7, 128.8, 129.7, 130.3, 134.7, 135.0, 136.3, 136.6, 139.9, 140.3, 152.5, 152.7, 158.7, 159.2, 163.0, 163.6, 165.46, 165.50, 179.1, 180.22, 180.24, 181.9. HRFABMS m/z $[M + H]^+$: calcd. for $C_{24}H_{17}O_7$, 417.0974; found, 417.0975.

*4,9-Dihydroxy-1-phenylanthra-[1,2-c]furan-3,6,11(1H)-trione (**12**) and 4,8-dihydroxy-1-phenylanthra-[1,2-c]furan-3,6,11(1H)-trione (**12′**).* A solution of a mixture of **11** and **11′** (36 mg, 0.087 mmol), and *p*-TsOH (*ca.* 1 mg) in MeOH (10 ml) was heated under reflux for 2 h. After removing MeOH under reduced pressure, the residue was chromatographed on SiO_2 to give an inseparable mixture of **12** and **12′**

(55:45; 32 mg, 0.086 mmol, 99%) as orange-yellow crystalline powder. Mp > 250 °C (decomp.). IR ν_{max} (nujol) cm^{-1}: 3300 (O–H), 1730 (C=O), 1660 (C=O). NMR δ_H (DMSO): 6.92 and 6.94 [total 1H, 2 × s (55:45), 7.11–7.41 (7H, m), 7.69 (1H, s), 7.79 and 7.99 [total 1H, 2 × s (45:55), $J = 8.7$ Hz]. NMR δ_C (DMSO): 82.9, 83.0, 111.9, 112.2, 115.0, 115.2, 116.3, 116.7, 118.9, 119.1, 121.2, 121.8, 124.7, 125.0, 127.9, 128.4, 128.6, 129.4, 130.0, 134.7, 135.2, 136.7, 136.9, 139.6, 139.9, 152.9, 153.4, 161.4, 162.2, 162.7, 163.4, 166.2, 178.8, 179.7, 180.5, 182.2. HRFABMS m/z $[M + H]^+$: calcd. for $C_{22}H_{13}O_6$, 373.0712; found, 373.0712.

*9-Hydroxy-10-methoxy-4-methoxymethoxy-1-phenylanthra[1,2-c]furan-3,6,11(1H)-trione (**14**) and 8-hydroxy-7-methoxy-4-methoxymethoxy-1-phenylanthra[1,2-c]furan-3,6,11(1H)-trione (**14′**).* To a solution of **9** (45 mg, 0.13 mmol) in CH_2Cl_2 (5 ml), **13** (10 µl, *ca.* 0.43 mmol) was added at room temperature under Ar. After stirring for 2 h, SiO_2 (2 g) was added to the reaction mixture, and stirring was continued for 30 min. After filtration, the filtrate was concentrated under reduced pressure. To this residue, K_2CO_3 (20 mg, 0.13 mmol) in aq. THF (50 vol%; 4 ml) was added. After stirring at room temperature for 30 min, the reaction mixture was diluted with water and extracted with EtOAc. The organic layer was successively washed with water and brine, dried ($MgSO_4$), and concentrated under reduced pressure. The residue was chromatographed on SiO_2 to give a mixture of **14** and **14′** (15 mg, 0.034 mmol, 26%). This mixture was further purified by careful preparative TLC to give pure **14** ($R_f = 0.33$, toluene/EtOAc = 1 : 1) and **14′** ($R_f = 0.30$, toluene/EtOAc = 1:1), as orange-yellow crystalline powder. **14**: Mp >210 °C (decomp.). IR ν_{max} (nujol) cm^{-1}: 3300 (O–H), 1730 (C=O), 1660 (C=O). NMR δ_H (C_5D_5N): 3.52 (3H, s), 3.55 (3H, s), 5.68 (2H, dd, $J = 9.9$, 6.9 Hz), 7.22–7.34 (3H, m), 7.46 (1H, d, $J = 8.4$ Hz), 7.50–7.57 (2H, m), 8.21 (1H, d, $J = 8.4$ Hz), 8.38 (1H, s). NMR δ_C (C_5D_5N): 57.0, 61.0, 83.9, 95.7, 112.9, 113.5, 119.8, 122.2, 126.14, 126.18, 126.9, 128.86, 128.92, 129.1, 137.2, 140.5, 148.7, 153.6, 159.5, 159.8, 166.5, 181.1, 181.4. HRFABMS m/z $[M + H]^+$: calcd. for $C_{25}H_{19}O_8$, 447.1080; found, 447.1077. **14′**: Mp > 160 °C (decomp.). IR ν_{max} (nujol) cm^{-1}: 3300 (O=H), 1740 (C=O), 1670 (C=O). NMR δ_H (C_5D_5N): 3.54 (3H, s), 4.05 (3H, s), 5.70 (2H, dd, $J = 10.8$, 6.9 Hz), 7.25–7.37 (3H, m), 7.39 (1H, d, $J = 8.4$ Hz), 7.50–7.58 (2H, m), 7.94 (1H, d, $J = 8.4$ Hz), 8.39 (1H, s). NMR δ_C (C_5D_5N): 57.0, 61.0, 84.0, 95.7, 113.5, 119.7, 122.1, 122.6, 125.7, 126.4, 127.0, 128.8, 128.9, 129.1, 137.5, 142.2, 148.8, 153.3, 159.3, 159.7, 166.5, 180.0, 181.8. HRFABMS m/z $[M + H]^+$: calcd. for $C_{25}H_{19}O_8$, 447.1080; found, 447.1078.

*4,9-Dihydroxy-10-methoxy-1-phenylanthra[1,2-c]furan-3,6,11(1H)-trione: (±)-basidifferquinone C (**3**).* A solution of **14** (1.8 mg, 4.0 µmol), and *p*-TsOH (*ca.* 0.1 mg) in MeOH (2 ml) was heated under reflux for 2 h. After removing MeOH under reduced pressure, the residue was chromatographed on SiO_2 to give **3** (1.5 mg, 3.8 µmol, 95%) as orange-yellow crystalline powder. Mp > 250 °C (decomp.). IR ν_{max} (nujol) cm^{-1}: 3300 (O=H), 1730 (C=O), 1660 (C=O). NMR δ_H (C_5D_5N): 3.53 (3H, s), 7.22–7.33 (4H, m), 7.43 (1H, d, $J = 8.4$ Hz), 7.53–7.56 (2H, m), 8.13 (1H, s), 8.21 (1H, d, $J = 8.4$ Hz). NMR δ_C (C_5D_5N): 61.0, 84.1, 116.2, 117.9, 120.9, 121.7, 125.9, 126.6, 127.3, 128.8, 128.9 (two coincident peaks), 137.7, 140.4, 148.6, 154.4, 159.5, 164.1, 168.5, 181.3, 181.9. HRFABMS m/z $[M + H]^+$: calcd. for $C_{23}H_{15}O_7$, 403.0818; found, 403.0811.

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