INTRODUCTION

Discovery of qinghaosu\(^1\) (artemisinin), a potent naturally-occurring antimalarial agent, has brought entirely renewed interest to organic peroxides. To establish structure-activity relationships and to discover new lead compounds for antimalarial drugs, a huge number of novel organic peroxides have been designed and synthesized since the late 1970’s.

An issue of central importance in the synthesis of organic peroxides is the introduction of the peroxy bond. To date, almost in all synthetic organic peroxides the peroxy bonds were taken from some inorganic species that already contain O-O bond(s), such as \(^3\)O\(_2\), \(^1\)O\(_2\), and O\(_3\). Hydrogen peroxide (H\(_2\)O\(_2\)) is also a readily available source for peroxy bonds, which has received more and more attention in recent years because of the potential advantages in accessibility, handling, and cost.\(^2\)

![Figure 1](image-url)

**Figure 1.** Incorporation of H\(_2\)O\(_2\) into organic structures with one oxygen alkylated at a time.

This paper is dedicated to Professor Ei-ichi Negishi on the occasion of his 77th birthday.
Incorporation of hydrogen peroxide into an organic framework is often achieved in a stepwise manner (Figure 1), with one oxygen atom bonded to a carbon atom at a time through alkylation or ketalization. Recently, we developed a PMA (phosphomolybdic acid) catalyzed transformation of ketones and ketal into the corresponding gem-dihydroperoxyketalts, which provided a facile way to complete the first step (Figure 1). In efforts to complete the second step (viz., connecting another carbon atom to the hydroperoxyl group) we noticed the Woerpel’s approach to 1,2-dioxolanes (also the original work of Dussault, Figure 2). Although all their products were monocyclic, it seemed that application of such reactions in an intramolecular manner would result in structurally more complex polycyclic peroxides. Prompted by this thought, we carried out the work below.

**Figure 2.** The intermolecular additions of peroxycarbenium ions to alkenes giving monocyclic peroxides developed by Dussault and Woerpel, and the main features of this work (conversion of 2 to 3 and 4).

**RESULTS AND DISCUSSION**

The synthesis merged with the condensation of dimethyl malonate (5) with the known aldehyde 6 (Scheme 1). The resulting α,β-unsaturated species (7) was treated with vinyl Grignard reagent in the presence of CuCl at −78 °C gave the expected Michael addition product 8 in 89% yield. The acidic malonate CH proton was then removed with NaH to afford the corresponding carbanion, which reacted with the commercially available 9 to yield 10. A Heck reaction was then performed under the Pd(OAc)$_2$/K$_2$CO$_3$/Ph$_3$P/MeCN conditions. The resulting 11 was then converted into the corresponding gem-dihydroperoxides 1a via reaction with H$_2$O$_2$ as reported previously with the aid of PMA.
Scheme 1
The hydroperoxide OH’s in 1a were silylated with Et₃SiCl in the presence of imidazole to deliver the bis-silylpoxyketal 2a in 91% yield. The key [3+2] cycloaddition was then examined at −78 °C in CH₂Cl₂ in the presence of SnCl₄. After five hours’ reaction, the desired bridged peroxides, 3a (the less polar isomer) and 4a (the more polar isomer), were isolated in 32% and 14% yields, respectively. The closely related bis-silylpoxyketal 2b (derived in a similar manner by silylation of the known 2t 1b) also underwent facile [3+2] reaction under the same conditions, giving 3b (the less polar isomer) and 4b (the more polar isomer) in 22% and 64% yield, respectively. The products of the cycloadditions (3 and 4) are a pair of diastereomers in each case. Although the relative configurations for 3a and 4a cannot be assigned with certainty, those for 3b and 4b are reliably established with the aid of 2D NOESY experiment; a distinct nOe (the boxed structure on the bottom left corner, Scheme 1) was observed for the polar isomer 4b.

<table>
<thead>
<tr>
<th>Peroxide</th>
<th>IC₅₀ (ng/mL)</th>
<th>IC₅₀ (μM)</th>
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<tr>
<td>3a</td>
<td>336</td>
<td>0.97</td>
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<tr>
<td>4a</td>
<td>936</td>
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<tr>
<td>3b</td>
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<tr>
<td>4b</td>
<td>1516</td>
<td>6.53</td>
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<tr>
<td>Chloroquine diphosphate</td>
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<tr>
<td>Artesunate</td>
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<table>
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<tr>
<th></th>
<th>a, b</th>
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<tr>
<td>a</td>
<td>The in vitro antimalarial data were obtained as described previously (ref. 8).</td>
</tr>
<tr>
<td>b</td>
<td>Data shown are the values from n = 2–3 independent experiments.</td>
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The peroxides obtained were tested in vitro for their antimalarial activity. The results are shown in Table 1. The IC₅₀’s were in the range of 0.97-6.53 μM, with the less polar isomers somewhat more potent than the more polar ones.

In summary, several 1,2-dioxolanes with the peroxy bond installed in a rigid spiro-bridge framework were synthesized by using intramolecular [3+2] addition of in situ generated peroxycarbenium ions to C-C double bonds. All products contain a fused benzene ring to serve as a built-in UV chromophore, which may facilitate e.g., HPLC analysis in case of further biomedical investigations.
EXPERIMENTAL

The $^1$H NMR and $^{13}$C NMR spectra were recorded at ambient temperature using a Varian Mercury 300 or a Bruke Avance 300 instrument (operating at 300 MHz for proton). The FTIR spectra were scanned with a Nicolet Avatar 360 FT-IR. EIMS and EIHRMS were recorded with an HP 5989A and a Finnigan MAT 8430 mass spectrometer, respectively. The ESIMS and ESISHRMS were recorded with a PE Mariner API-TOF and an APEX III (7.0 Tesla) FTMS mass spectrometer, respectively. Dry CH$_2$Cl$_2$ was obtained by distillation over CaH$_2$. Dry THF was distilled over Na/Ph$_2$CO under argon before use. Degassed MeCN was obtained by bubbling N$_2$ into MeCN for 20 min. All other solvents and reagents were used as received from commercial sources. PE = petroleum ether (chromatography solvent, bp 60−90 °C).

**Dimethyl 2-(3-(2-methyl-1,3-dioxolan-2-yl)propylidene)malonate (7).** Piperidine (0.2 mL, 2.0 mmol) and glacial acetic acid (0.12 mL, 2.0 mmol) were added in turn to a solution of dimethyl malonate (3.90 g, 30 mmol) in dry CH$_2$Cl$_2$ (5 mL) stirred in an ice-water bath. After completion of the addition, the bath was removed and the mixture was stirred at ambient temperature overnight. When TLC showed completion of the reaction, sat. aq. NH$_4$Cl (10 mL) was added. The mixture was extracted with Et$_2$O (3×50 mL), washed with brine (3×15 mL), and dried over anhydrous Na$_2$SO$_4$. Removal of the solvent by rotary evaporation and column chromatography (1:6 EtOAc/PE) on silica gel gave 7 as a colorless oil (1.6 g, 6.2 mmol, 62%): $^1$H NMR (300 MHz, CDCl$_3$) δ 7.08 (t, $J = 7.3$ Hz, 1H), 4.00-3.87 (m, 4H), 3.84 (s, 3H), 3.78 (s, 3H), 2.42 (dt, $J = 8.3$, 7.3 Hz, 2H), 1.84 (t, $J = 7.7$ Hz, 2H), 1.32 (s, 3H). $^{13}$C NMR (75 MHz, CDCl$_3$) δ 165.8, 164.3, 150.5, 127.5, 109.1, 64.6, 52.3, 52.2, 37.2, 24.5, 23.9; FT-IR (film) 2955, 2887, 1739, 1642, 1435, 1377, 1256, 1052, 863, 833 cm$^{-1}$; ESI-MS m/z 281.0 ([M+Na]$^+$);EI-HRMS calcd for C$_{12}$H$_{18}$O$_6$(M$^+$) 258.1103, found 258.1105.

**Dimethyl 2-(5-(2-methyl-1,3-dioxolan-2-yl)pent-1-en-3-yl)malonate (8).** CH$_2$=CHMgBr (1.0 M, in Et$_2$O, 1.2 mL, 1.2 mmol) was added dropwise to a mixture of 7 (256 mg, 1.0 mmol) and CuCl (5.0 mg, 0.05 mmol) in dry THF (3 mL) stirred at −78 °C under N$_2$ (balloon). After completion of the addition, the mixture was stirred another 30 min at the same temperature, when TLC showed completion of the reaction. sat. aq. NH$_4$Cl (5 mL) was added. The mixture was extracted with Et$_2$O (3×30 mL), washed with brine (3×15 mL), and dried over anhydrous Na$_2$SO$_4$. Removal of the solvent by rotary evaporation and column chromatography (1:6 EtOAc/PE) on silica gel gave 8 as a colorless oil (236 mg, 0.89 mmol, 89%): $^1$H NMR (300 MHz, CDCl$_3$) δ 5.63 (dt, $J = 17.5$, 9.6 Hz, 1H), 5.12 (d, $J = 1.9$ Hz, 1H), 5.07 (d, $J = 2.6$ Hz, 1H), 4.00-3.84 (m, 4H), 3.74 (s, 3H), 3.69 (s, 3H), 3.40 (d, $J = 9.0$ Hz, 2H), 2.76 (dq, $J = 2.8$, 9.6 Hz, 1H), 1.77-1.50 (m, 2H), 1.46-1.32 (m, 1H), 1.29 (s, 3H); $^{13}$C NMR (75 MHz, CDCl$_3$) δ 168.6, 168.4, 137.6, 117.9, 109.7, 64.6, 64.5, 56.8, 52.4, 52.3, 44.1, 36.4, 26.6, 23.8; FT-IR (film) 2955, 2883, 1739, 1642, 1435, 1377, 1255, 1151, 1039, 924, 850 cm$^{-1}$; ESI-MS m/z 309.0 ([M+Na]$^+$); Anal. Calcd for
C_{14}H_{22}O_{6}: C 58.73, H 7.74. Found: C 58.75, H 7.50.

**Dimethyl 2-(2-bromobenzyl)-2-(5-(2-methyl-1,3-dioxolan-2-yl)pent-1-en-3-yl)malonate (10).** A solution of the 2-bromobenzyl bromide 9 (525 mg, 2.10 mmol) in dry THF (1 mL) was added to a mixture of 8 (200 mg, 0.70 mmol) and NaH (60% in mineral oil, 84 mg, 2.10 mmol, washed thrice with petroleum ether before addition of THF) in dry THF (3 mL) stirred at ambient temperature already for 30 min. Stirring was then continued at the same temperature over night. When TLC showed completion of the reaction, sat. aq. NH₄Cl (5 mL) was added. The mixture was extracted with Et₂O (3× 50 mL), washed with brine (2× 15 mL), and dried over anhydrous Na₂SO₄. Removal of the solvent by rotary evaporation and column chromatography (1:5 EtOAc/PE) on silica gel gave 10 (260 mg, 0.57 mmol, 82%) as a colorless oil: ¹H NMR (300 MHz, CDCl₃) δ 7.50 (d, J = 7.9 Hz, 1H), 7.38 (d, J = 7.8 Hz, 1H), 7.19 (d, J = 7.4 Hz, 1H), 7.04 (d, J = 8.0 Hz, 1H), 5.69 (dt, J = 16.9, 10.3 Hz, 1H), 5.19 (d, J = 2.2 Hz, 1H), 5.09 (d, J = 1.7 Hz, 1H), 4.00-3.84 (m, 4H), 3.63 (s, 3H), 3.61 (s, 3H), 3.53 (d, J = 14.8 Hz, 1H), 3.38 (d, J = 14.8 Hz, 1H), 2.70 (t, J = 9.6 Hz, 1H), 1.92-1.66 (m, 2H), 1.56 (dt, J = 4.4, 13.1 Hz, 1H), 1.39-1.31 (m, 1H), 1.30 (s, 3H); ¹³C NMR (75 MHz, CDCl₃) δ 170.5, 170.4, 137.2, 136.9, 132.5, 131.3, 128.0, 127.1, 126.2, 119.1, 109.8, 64.6, 64.5, 62.4, 52.2, 52.1, 50.4, 38.4, 37.3, 24.8, 23.8; FT-IR (film) 2981, 2880, 1732, 1474, 1435, 1377, 1248, 1045, 852, 751 cm⁻¹; ESI-MS m/z 477.1 ([M+Na]+); MALDI-HRMS calcd for C₂₁H₂₇O₆BrNa ([M+Na]+) 477.08832, found 477.0893.

**Dimethyl 3-(3,3-bis(triethylsilylperoxy)butyl)-4-methylene-3,4-dihydronaphthalene-2,2(1H)-dicarboxylate (11).** A solution of 10 (190 mg, 0.42 mmol), PPh₃ (44 mg, 0.17 mmol), K₂CO₃ (300 mg, 2.52 mmol), and Pd(OAc)₂ (14 mg, 0.06 mmol) in degassed MeCN (8 mL) was placed in liquid N₂. The resulting solid was evacuated with an oil pump and the vaccum was released with argon. The process was repeated three time before the mixture was finally refluxed in a oil bath under argon for 36 h. The mixture was cooled to ambient temperature. Et₂O (50 mL) and water (20 mL) were added. The mixture was extracted with extracted with Et₂O (3×50 mL), washed with brine (2×20 mL), and dried over anhydrous Na₂SO₄. Removal of the solvent by rotary evaporation and column chromatography (1:5 EtOAc/PE) on silica gel gave 11 as a colorless oil (145 mg, 0.38 mmol, 92%): ¹H NMR (300 MHz, CDCl₃) δ 7.53 (d, J = 7.3 Hz, 1H), 7.24-7.08 (m, 3H), 5.56 (s, 1H), 5.05 (s, 1H), 4.00-3.80 (m, 4H), 3.78 (s, 3H), 3.60 (s, 3H), 3.57 (d, J = 17.2 Hz, 1H), 3.25 (d, J = 17.2 Hz, 2H), 1.49-1.82 (m, 3H), 1.38-1.22 (m, 1H), 1.21 (s, 3H); ¹³C NMR (75 MHz, CDCl₃) δ 170.4, 170.3, 142.3, 133.1, 132.0, 128.8, 128.1, 126.4, 124.9, 112.4, 109.7, 64.5, 64.4, 58.4, 52.9, 52.8, 46.7, 36.7, 30.7, 23.6, 23.3; FT-IR (film) 2982, 2882, 1736, 1631, 1478, 1434, 1377, 1259, 1151, 1046, 948, 778, 736 cm⁻¹; ESI-MS m/z 397.1 ([M+Na]+); MALDI-HRMS calcd For C₂₁H₂₈O₆Na ([M+Na]+) 397.16216, found 397.1635.

**Dimethyl 3-(3,3-bis(triethylsilylperoxy)butyl)-4-methylene-3,4-dihydronaphthalene-2,2(1H)-**
dicarboxylate (2a). A solution of 1a (150 mg, 0.40 mmol), Et₃SiCl (202 μL, 1.2 mmol) and imidazole (103 mg, 1.52 mmol) in dry DMF (4 mL) was stirred at ambient temperature for 10 h. Et₂O (50 mL) and water (10 mL) were added. The mixture was extracted with Et₂O (3×50 mL), washed with water (2×20 mL) and brine (2×20 mL), and dried over anhydrous Na₂SO₄. Removal of the solvent by rotary evaporation and column chromatography (1:20 EtOAc/PE) on silica gel gave 11 as a colorless oil (220 mg, 0.36 mmol, 91%): ¹H NMR (300 MHz, CDCl₃) δ 7.55 (d, J = 7.6 Hz, 1H), 7.25-7.10 (m, 3H), 5.57 (s, 1H), 5.08 (s, 1H), 3.80 (s, 3H), 3.62 (s, 3H), 3.57 (d, J = 16.7 Hz, 1H), 3.35-3.318 (m, 2H), 1.94-1.68 (m, 2H), 1.67-1.47 (m, 1H), 1.23 (s, 3H), 0.98 (t, J = 8.0 Hz, 9H), 0.91 (t, J = 8.0 Hz, 9H), 0.68 (q, J = 8.0 Hz, 6H), 0.59 (q, J = 8.0 Hz, 6H). ¹³C NMR (75 MHz, CDCl₃) δ 170.4, 170.3, 142.2, 133.1, 132.0, 128.7, 128.0, 126.4, 125.0, 112.4, 110.5, 58.4, 52.9, 52.7, 46.7, 32.2, 30.8, 23.6, 18.7, 6.67, 3.76, 3.67. FT-IR (film) 2954, 2878, 1742, 1456, 1434, 1233, 1280, 1140, 798, 733 cm⁻¹; ESI-MS m/z 631.4 ([M+Na]⁺); ESI-HRMS calcd for C₃₁H₅₂O₈Si₂Na ([M+Na]⁺) 631.30929, found 631.31085.

Dimethyl 3-methyl-3,4,5,5a-tetrahydro-3,11b-methanonaphtho[1,2-c][1,2]dioxepine-6,6(7H)-dicarboxylate (3a and 4a). A solution of SnCl₄ in dry CH₂Cl₂ (1.0 M, 0.15 mL, 0.15 mmol) was added to a solution of 2a (60 mg, 0.1 mmol) in CH₂Cl₂ (4 mL) stirred at −78 °C under argon (balloon). After completion of the addition, stirring was continued at the same temperature for 5 h before the bath was allowed to warm to ambient temperature over 1 h. When TLC showed completion of the reaction, Et₂O was added, followed by sat. aq. NaHCO₃. The mixture was extracted with Et₂O, washed in turn with water, aq. sat. NaHCO₃, and brine before being dried over anhydrous Na₂SO₄. Removal of the solvent by rotary evaporation and column chromatography (1:6 EtOAc/PE) on silica gel gave the less polar isomer 3a (11 mg, 0.032 mmol, 32%) and the more polar isomer 4a (5 mg, 0.014 mmol, 14%) as a colorless oils. Data for 3a (the less polar isomer): ¹H NMR (300 MHz, CDCl₃) δ 7.21 (d, J = 8.8 Hz, 1H), 7.11-7.01 (m, 2H), 6.94 (d, J = 7.3 Hz, 1H), 3.84 (s, 3H), 3.79 (d, J = 14.1 Hz, 1H), 3.76 (s, 3H), 3.68 (d, J = 8.5 Hz, 1H), 3.04 (d, J = 13.7 Hz, 1H), 2.78 (d, J = 7.8 Hz, 1H), 2.73 (d, J = 11.6 Hz, 1H), 2.59 (dt, J = 9.8, 2.1 Hz, 1H), 2.43-2.25 (m, 1H), 2.00 (dd, J = 15.0, 6.3 Hz, 1H), 1.72 (dd, J = 14.1, 6.7 Hz, 1H), 1.33 (s, 3H). FT-IR (film) 2953, 2929, 1729, 1488, 1450, 1227, 1191, 1112, 997, 894, 766 cm⁻¹; ESI-MS m/z 385.1 ([M+K]⁺); ESI-HRMS calcd for C₁₉H₂₂O₆Na ([M+Na]⁺) 369.13086, found 369.13084.

Data for 4a (the more polar isomer): ¹H NMR (300 MHz, CDCl₃) δ 7.08 (d, J = 8.3 Hz, 1H), 7.03 (d, J = 8.3 Hz, 1H), 6.94-6.84 (m, 2H), 3.83 (dd, J = 5.3, 10.1 Hz, 2H), 3.75 (s, 3H), 3.39 (s, 3H), 3.23 (d, J = 16.2 Hz, 1H), 2.83 (dd, J = 11.0, 2.7 Hz, 1H), 2.61 (d, J = 10.9 Hz, 1H), 2.23-2.05 (m, 1H), 2.02-1.91 (m, 1H), 1.71 (dd, J = 12.3, 6.0 Hz, 1H), 1.52-1.41 (m, 1H), 1.35 (s, 3H). FT-IR (film) 2952, 2929, 1739, 1490, 1452, 1227, 1191, 1152, 893, 872, 759 cm⁻¹; ESI-MS m/z 369.1 ([M+Na]⁺); ESI-HRMS calcd for
\[
C_{19}H_{22}O_6Na ([\text{M+Na}^+]) 369.13086, \text{ found 369.13086.}
\]

\[3,3,9,9-\text{Tetraethyl-6-methyl-6-(2-(4-methylenesichroman-3-y1)ethyl)-4,5,7,8-tetraoxa-3,9-disilaun-decane (2b)}\]. The same procedure for conversion of 1a into 2a above was used (with 1b to replace 1a); yield (chromatography with 1:100 EtOAc/PE): 87%.

Data for 2b (a colorless oil): \(^1\)H NMR (300 MHz, CDCl\(_3\)) \(\delta 7.69-7.60 (m, 1H), 7.27-7.18 (m, 2H), 7.97-7.06 (m, 1H), 5.62 (s, 1H), 5.08 (s, 1H), 4.87 (d, \(J = 15.2 \text{ Hz, } 1H\)), 4.74 (d, \(J = 15.2 \text{ Hz, } 1H\)), 4.28 (t, \(J = 6.8 \text{ Hz, } 1H\)), 2.07-1.80 (m, 4H), 1.36 (s, 3H), 0.97 (t, \(J = 8.2 \text{ Hz, } 1H\)), 0.69 (q, \(J = 8.2 \text{ Hz, } 12H\)).

\(^{13}\)C NMR (75 MHz, CDCl\(_3\)) \(\delta 141.6, 134.4, 131.4, 127.7, 126.9, 124.3, 123.9, 110.8, 107.1, 77.3, 65.6, 30.2, 27.2, 18.8, 6.8, 3.8; FT-IR (film) 2954, 2877, 1458, 1412, 1371, 1239, 1104, 1019, 798, 730 cm\(^{-1}\); ESI-MS m/z 517.2 ([M+Na]+); ESI-HRMS calcd for C\(_{26}\)H\(_{46}\)O\(_5\)Si\(_2\)Na ([M+Na]+) 517.27760, found 517.27837.

\((3R^*,5aR^*,11bS^*)-3\text{-Methyl-4,5,5a,7-tetrahydro-3H-3,11b-methano[1,2]dioxepino[4,3-c]isochromene (3b)}\) and \((3S^*,5aR^*,11bR^*)-3\text{-methyl-4,5,5a,7-tetrahydro-3H-3,11b-methano[1,2]dioxepino[4,3-c]-isochromene (4b)}\). The same procedure for conversion of 2a into 3a and 4a (but with 2b to replace 2a, chromatography with 1:20 EtOAc/PE).

Data for 3b (the less polar isomer, a colorless oil, 22% from 2b): \(^1\)H NMR (300 MHz, CDCl\(_3\)) \(\delta 7.59 (dd, J = 3.8, 5.6 \text{ Hz, } 1H), 7.29-7.20 (m, 2H), 7.00 (t, J = 4.2 \text{ Hz, } 1H), 4.95 (d, J = 14.9 \text{ Hz, } 1H), 4.88 (d, J = 14.9 \text{ Hz, } 1H), 3.80 (d, J = 6.2 \text{ Hz, } 1H), 2.93 (d, J = 11.7 \text{ Hz, } 1H), 2.52-2.35 (m, 1H), 2.27 (d, J = 11.6 \text{ Hz, } 1H), 1.95-1.82 (m, 1H), 1.76 (dd, J = 4.5, 10.4 \text{ Hz, } 2H), 1.40 (s, 3H).

\(^{13}\)C NMR (75 MHz, CDCl\(_3\)) \(\delta 135.3, 133.9, 127.8, 127.0, 123.8, 123.7, 84.3, 83.6, 74.8, 68.7, 54.7, 33.1, 24.7, 22.0. \text{ FT-IR (film) 2934, 2861, 1454, 1372, 1080, 1037, 855, 757 cm}^{-1}; \text{ ESI-MS m/z 255.0 ([M+Na]+); ESI-HRMS calcd for C}_{14}\text{H}_{16}\text{O}_3\text{Na ([M+Na]}^+) 255.09917, found 255.09921.

Data for 4b (the more polar isomer, a colorless oil, 64% from 2b): \(^1\)H NMR (300 MHz, CDCl\(_3\)) \(\delta 7.72-7.61 (m, 1H), 7.37-7.27 (m, 2H), 7.05 (t, J = 4.2 \text{ Hz, } 1H), 4.85 (d, J = 14.8 \text{ Hz, } 1H), 4.76 (d, J = 15.9 \text{ Hz, } 1H), 3.73 (dd, J = 5.8, 11.1 \text{ Hz, } 1H), 2.93 (dd, J = 2.8, 11.8 \text{ Hz, } 1H), 2.34 (d, J = 11.6 \text{ Hz, } 1H), 2.23-2.33 (m, 1H), 2.05-2.16 (m, 1H), 1.94 (dd, J = 6.7, 14.0 \text{ Hz, } 1H), 1.65-1.80 (m, 1H), 1.46 (s, 3H).

\(^{13}\)C NMR (75 MHz, CDCl\(_3\)) \(\delta 137.7, 128.8, 128.4, 127.28, 127.27, 124.4, 83.9, 80.9, 78.8, 68.1, 53.1, 35.6, 25.6, 21.2. \text{ FT-IR (film) 2941, 2851, 1493, 1447, 1376, 1297, 1213, 1108, 1038, 866, 756 cm}^{-1}; \text{ ESI-MS m/z 255.0 ([M+Na]+); ESI-HRMS calcd for C}_{14}\text{H}_{16}\text{O}_3\text{Na ([M+Na]}^+) 255.09917, found 255.09924.

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References (And Notes)


