## (土)-7-methoxy-2-methyl-1-oxaspiro[4,5]deca-6,9-diene-8-one

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The discussion and purpose for the synthesis of this compound has been reported elsewhere [1]. To a cold $\left(0^{\circ} \mathrm{C}\right)$ solution of $( \pm)$-1-(4-hydroxy-3-methoxyphenyl)-3-butanol ( $106 \mathrm{mg}, 0.54 \mathrm{mmol}$ ) in acetone ( 10 mL ) was added in one portion $\mathrm{Pb}(\mathrm{OAc})_{4}(655 \mathrm{mg}, 1.5 \mathrm{mmol}, 2.8 \mathrm{eq})$. The resulting orange mixture was stirred at $0^{\circ} \mathrm{C}$ for 2 h . The precipitate was filtered through celite and ethylene glycol ( 5 drops) was added. The solution was stirred at room temperature for 20 h and filtered through celite. The solvent was evaporated in vacuo to afford a racemic mixture of diastereomers ( $58 / 42$ ratio). Chromatography on silica gel ( $20 \%$ EtOAc/hexanes) afforded a mixture of diastereoisomers as a colorless oil ( $65 \mathrm{mg}, 62 \%$ ). Spectroscopic data were obtained from the diastereomeric mixture.

IR (neat) $\mathrm{cm}^{-1}: 1682(\mathrm{CO}), 1675(\mathrm{CO})$.
${ }^{1} \mathrm{H}-\mathrm{NMR}\left(\mathrm{CDCl}_{3}\right)$ d: Major: $1.35\left(\mathrm{~d}, 3 \mathrm{H}, \mathrm{J}=6.1 \mathrm{~Hz}, \mathrm{CH}_{3}\right), 1.79(\mathrm{~m}, 1 \mathrm{H}, \mathrm{H}-3 \mathrm{a}), 2.17(\mathrm{~m}, 3 \mathrm{H}, \mathrm{H}-3 \mathrm{~b}, \mathrm{H}-4)$, $3.68\left(\mathrm{~s}, 3 \mathrm{H}, \mathrm{OCH}_{3}\right), 4.38(\mathrm{~m}, 1 \mathrm{H}, \mathrm{H}-2), 5.75(\mathrm{~d}, 1 \mathrm{H}, \mathrm{J}=2.7 \mathrm{~Hz}, \mathrm{H}-6), 6.13(\mathrm{~d}, 1 \mathrm{H}, \mathrm{J}=10.0 \mathrm{~Hz}, \mathrm{H}-9), 6.80$ (dd, 1H, J=2.7, 10.0 Hz, H-10); Minor: 1.37 (d, 3H, J=6.1 Hz, CH3), 1.79 (m, 1H, H-3a), 2.17 (m, 3H, $\mathrm{H}-3 \mathrm{~b}, \mathrm{H}-4), 3.69\left(\mathrm{~s}, 3 \mathrm{H}, \mathrm{OCH}_{3}\right), 4.38(\mathrm{~m}, 1 \mathrm{H}, \mathrm{H}-2), 5.70(\mathrm{~d}, 1 \mathrm{H}, \mathrm{J}=2.7 \mathrm{~Hz}, \mathrm{H}-6), 6.14$ (d, 1H, J=10.0 Hz, $\mathrm{H}-9), 6.86(\mathrm{dd}, 1 \mathrm{H}, \mathrm{J}=2.7,10.0 \mathrm{~Hz}, \mathrm{H}-10)$.
${ }^{13} \mathrm{C}-\mathrm{nmr}\left(\mathrm{CDCl}_{3}\right)$ d: Major: $21.6\left(\mathrm{CH}_{3}\right), 34.2(\mathrm{C}-3), 38.1(\mathrm{C}-4), 54.9\left(\mathrm{OCH}_{3}\right), 76.7(\mathrm{C}-5), 79.6(\mathrm{C}-2)$, 117.3 (C-6), 125.9 (C-9), 149.8 (C-7), 151.3 (C-10), 181.7 (CO); Minor: $21.5\left(\mathrm{CH}_{3}\right), 34.0(\mathrm{C}-3), 37.8$ (C-4), $54.9\left(\mathrm{OCH}_{3}\right), 76.8(\mathrm{C}-5), 79.6(\mathrm{C}-2), 117.8(\mathrm{C}-6), 125.9(\mathrm{C}-9), 149.8(\mathrm{C}-7), 150.7(\mathrm{C}-10), 181.7$ (CO).

MS m/e (rel \%): Major: 194 [M+] (100), 179 (34), 166 (29), 151 (61), 139 (33), 123 (33), 111 (44), 85 (73); Minor: 194 [M+] (100), 177 (8), 153 (85), 147 (16), 124 (11).

Anal. calc. for $\mathrm{C}_{11} \mathrm{H}_{14} \mathrm{O}_{3}$ : C 68.01, H 7.27; found: C $67.99, \mathrm{H} 7.52$.

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## Reference

1. Plourde G.L. Tetrahedron Letters 2002, 43, 3597-3599.
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