Deuterium Experiment:

[Chemical structure image]

**Compound 2**: Dry potassium carbonate (431 mg, 3.12 mmoles), deuterium oxide (2 ml) and triethylphosphonoacetate (0.265 mg, 1.18 mmoles) are stirred vigorously in a dry flask for 20 hours at room temperature under nitrogen atmosphere\(^1\). Aldehyde (150 mg, 0.78 mmoles) is then introduced and stirring is continued for 24 hours. Extraction (diethylether, 10ml x 3) is performed after addition of water (5 ml), and the combined organic layers were washed with brine and dried over anhydrous Na\(_2\)SO\(_4\). The solvent was removed under reduced pressure. The crude product was purified on silica gel column chromatography using EtOAc-hexane as eluent to furnish the product (185 mg, 91%) as a yellow solid. \(R_f = 0.51\) (EtOAc-hexane 3:97); **IR** (neat): \(v_{max}/\text{cm}^{-1}\) 1713, 1619, 1593, 1476, 1218; \(^1\)H NMR (500 MHz, CDCl\(_3\)) \(\delta\) 1.33 (t, \(J = 7.3\) Hz, 3H), 2.01 (s, 3H), 2.29 (s, 3H), 3.67 (s, 3H), 4.25 (q, \(J = 7.3\) Hz, 2H), 4.90 (br s, 1H), 5.24 (br s, 1H), 6.88 (d, \(J = 7.9\) Hz, 1H), 7.12 (d, \(J = 7.9\) Hz, 1H), 7.82 (s, 1H); \(^13\)C NMR (125 MHz, CDCl\(_3\)) \(\delta\) 14.3, 15.9, 24.6, 59.8, 60.3, 116.7, 122.0(q), 124.3, 125.0, 130.4, 132.0, 139.8, 144.5, 144.8, 157.8, 167.7; **HRMS**: m/z calcd for C\(_{16}\)H\(_{19}\)DNaO\(_3\) [(M+Na)\(^+\)]: 284.1373; Found: 284.1373.

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Cyclization reaction on deuterated diene ester

\[
\text{EtO}_2\text{C}-\text{D} \quad \underset{\text{FeCl}_3, \text{CH}_2\text{Cl}_2}{\text{ } \text{1h, rt}} \quad \text{EtO}_2\text{C}-\text{D}
\]

**Compound 3:** Under inert atmosphere of argon, to a stirred solution of diene ester (50 mg, 0.19 mmol) in CH\textsubscript{2}Cl\textsubscript{2} (50 ml) and FeCl\textsubscript{3} (62 mg, 0.38 mmol) was added at 0°C and stirred for 1 h at RT. The reaction progress was monitored by TLC and after completion the reaction was quenched by sodium bisulphate.

The reaction mixture was extracted with CH\textsubscript{2}Cl\textsubscript{2} and the combined organic layers were washed with brine and dried over anhydrous Na\textsubscript{2}SO\textsubscript{4}. The solvent was removed under reduced pressure. The crude product was purified on silica gel column chromatography using EtOAc-hexane as an eluent to furnish the cyclised product (46 mg, 93%) as a yellow solid. \(R_f = 0.51\) (EtOAc-hexane 3:97); \(\text{IR} \ (\text{neat})\): \(\nu_{\text{max}}/\text{cm}^{-1} \ 2979, 2928, 1704, 1568, 1478, 1419, 1244; \)

\(\text{1H NMR} \ (200 \text{ MHz, CDCl}_3) \ \delta \ 1.38 \ (t, J = 7.0 \text{ Hz, 3H}), \ 1.44 \ (s, 6H), \ 2.29 \ (s, 3H), \ 3.94 \ (s, 3H), \ 4.3 \ (q, J = 7 \text{ Hz, 2H}), \ 7.00 \ (d, J = 7.4 \text{ Hz, 1H}), \ 7.17 \ (d, J = 7.4 \text{ Hz, 1H}), \ 7.79 \ (s, 1H)\); \(\text{13C NMR} \ (100 \text{ MHz, CDCl}_3) \ \delta \ 14.4, 15.8, 24.2, 49.8, 60.0, 61.5, 116.7, 128.5, 130.5, 131.3, 136.0, 145.0, 153.8, 157.0, 164.4; \text{HRMS} \ (\text{C}_{16}\text{H}_{21}\text{O}_3): \ \text{Calc’d} \ 261.1491 \ [(\text{M+H})^+], \ \text{Found} \ 261.1495.\)