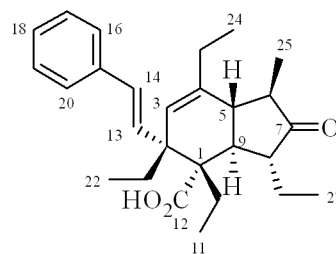


# Data for Synthetic Spiculoic Acid A



$R_f = 0.21$  (70% PE 30–40, 30% Et<sub>2</sub>O).

$[\alpha]_D^{22} = -97$  (c 0.16, CH<sub>2</sub>Cl<sub>2</sub>).

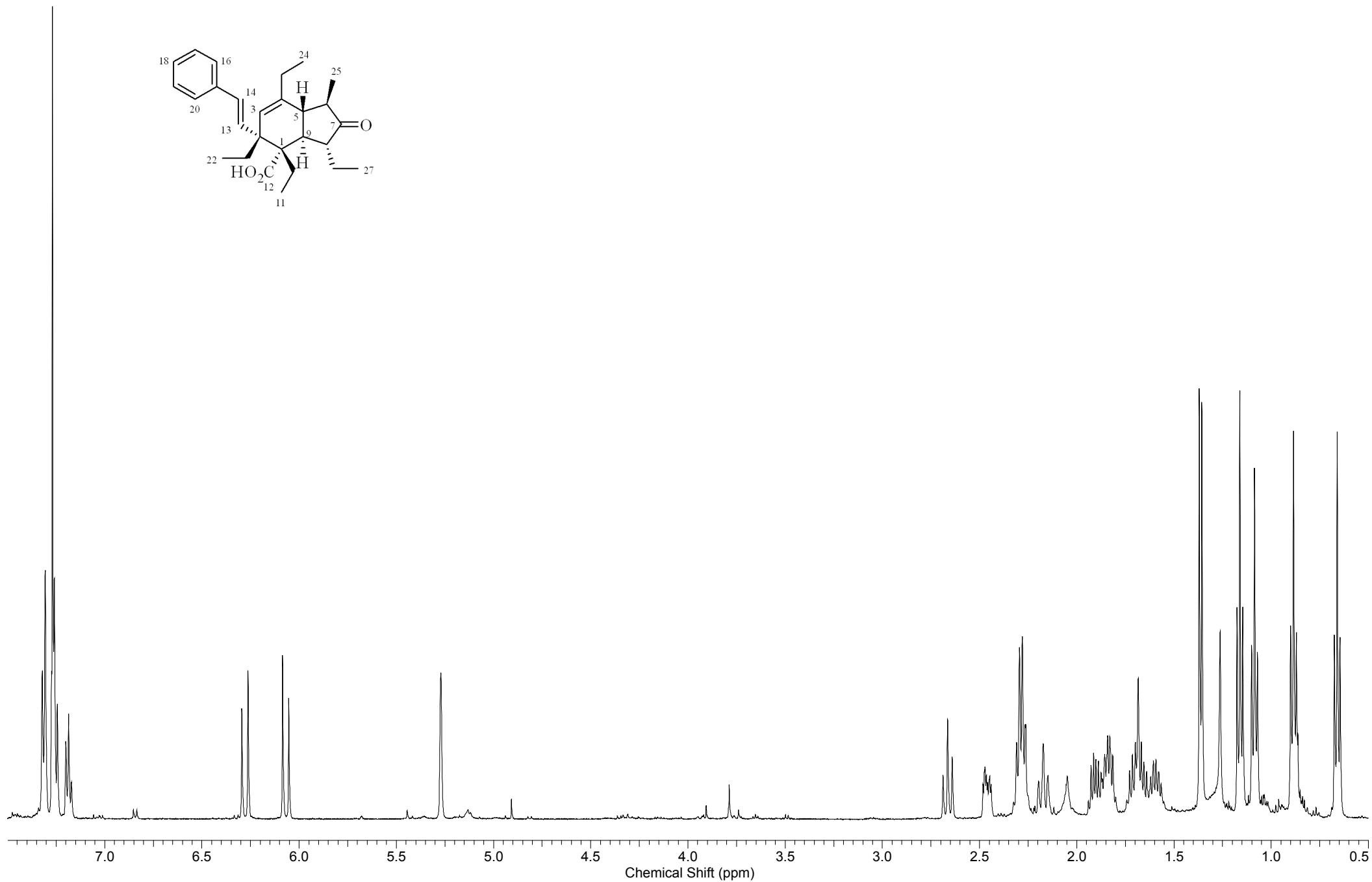
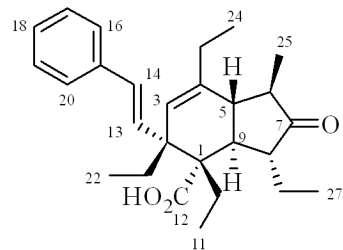
$m/z$  Probe ES<sup>-</sup> (NH<sub>3</sub>) 407.3 ([MH]<sup>-</sup>, 100%).

Accurate Mass (ESI) found [M–H]<sup>-</sup> = 407.2581, C<sub>27</sub>H<sub>35</sub>O<sub>3</sub>.

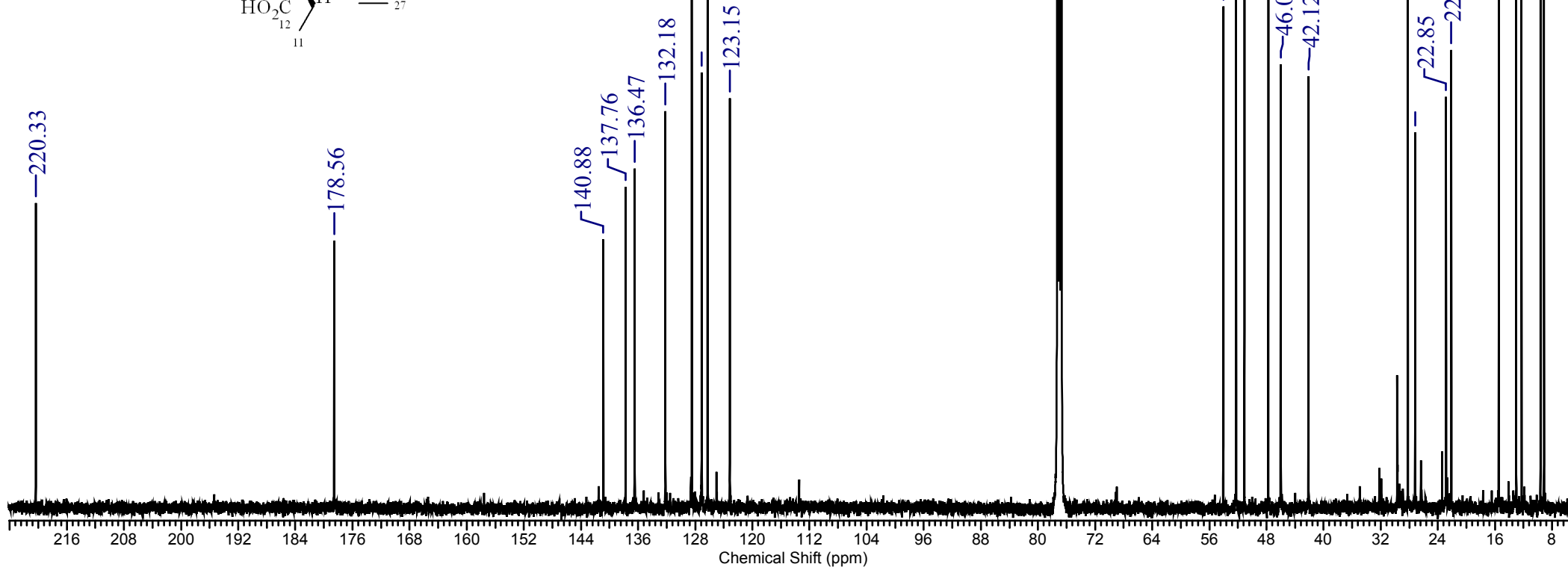
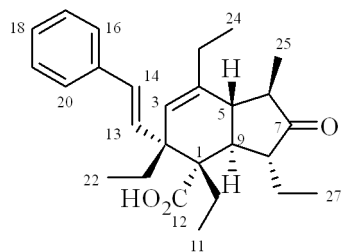
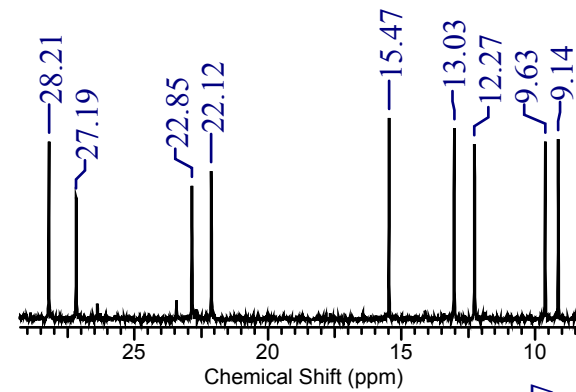
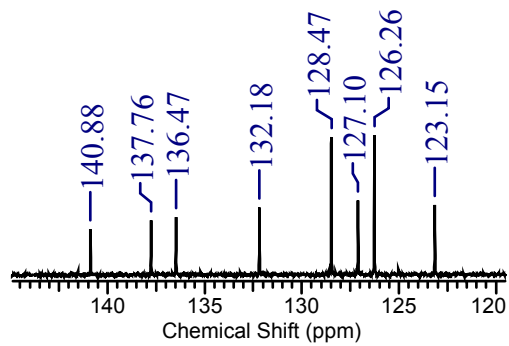
$\delta_H$  (500MHz, CDCl<sub>3</sub>) 0.66 (3H, t,  $J$  7.3Hz, H-27), 0.88 (3H, t,  $J$  7.3Hz, H-22), 1.08 (3H, t,  $J$  7.3Hz, H-11), 1.16 (3H, t,  $J$  7.3Hz, H-24), 1.36 (3H, d,  $J$  6.8Hz, H-25), 1.55–1.63 (1H, m, 1H of H-26), 1.63–1.74 (2H, m, 1H of H-10, 1H of H-21), 1.80–1.87 (2H, m, 1H of H-21, 1H of H-26), 1.96 (1H, dq,  $J_1$  11.8 Hz,  $J_2$  6.7Hz, H-6), 2.17 (1H, t,  $J$  11.8Hz, H-5), 2.26–2.31 (3H, m, 1H of H-10, H-23), 2.45 (1H, ddd,  $J_1$  11.8Hz,  $J_2$  5.3Hz,  $J_3$  3.5Hz, H-8), 2.66 (1H, t,  $J$  11.8Hz, H-9), 5.27 (1H, d,  $J$  2.4Hz, H-3), 6.06 (1H, d,  $J$  15.9Hz, H-13), 6.28 (1H, d,  $J$  15.9Hz, H-14), 7.19 (1H, t,  $J$  7.3Hz, H-18), 7.24–7.28 (2H, m, H-17,19), 7.31 (2H, d,  $J$  7.6Hz, H-16,20).

$\delta_C$  (125.8MHz, CDCl<sub>3</sub>) 9.14 (1C, C-22), 9.63 (1C, C-27), 12.27 (1C, C-11), 13.03 (1C, C-24), 15.47 (1C, C-25), 22.12 (1C, C-26), 22.85 (1C, C-10), 27.18 (1C, C-21), 28.21 (1C, C-23), 42.12 (1C, C-9), 46.00 (1C, C-5), 47.75 (1C, C-6), 51.10 (1C, C-2), 52.26 (1C, C-8), 54.05 (1C, C-1), 123.2 (1C, C-3), 126.3 (2C, C-16,20), 127.1 (1C, C-18), 128.5 (2C, C-17,19), 132.2 (1C, C-14), 136.5 (1C, C-13), 137.8 (1C, C-15), 140.9 (1C, C-4), 178.6 (1C, C-12), 220.3 (1C, C-7).

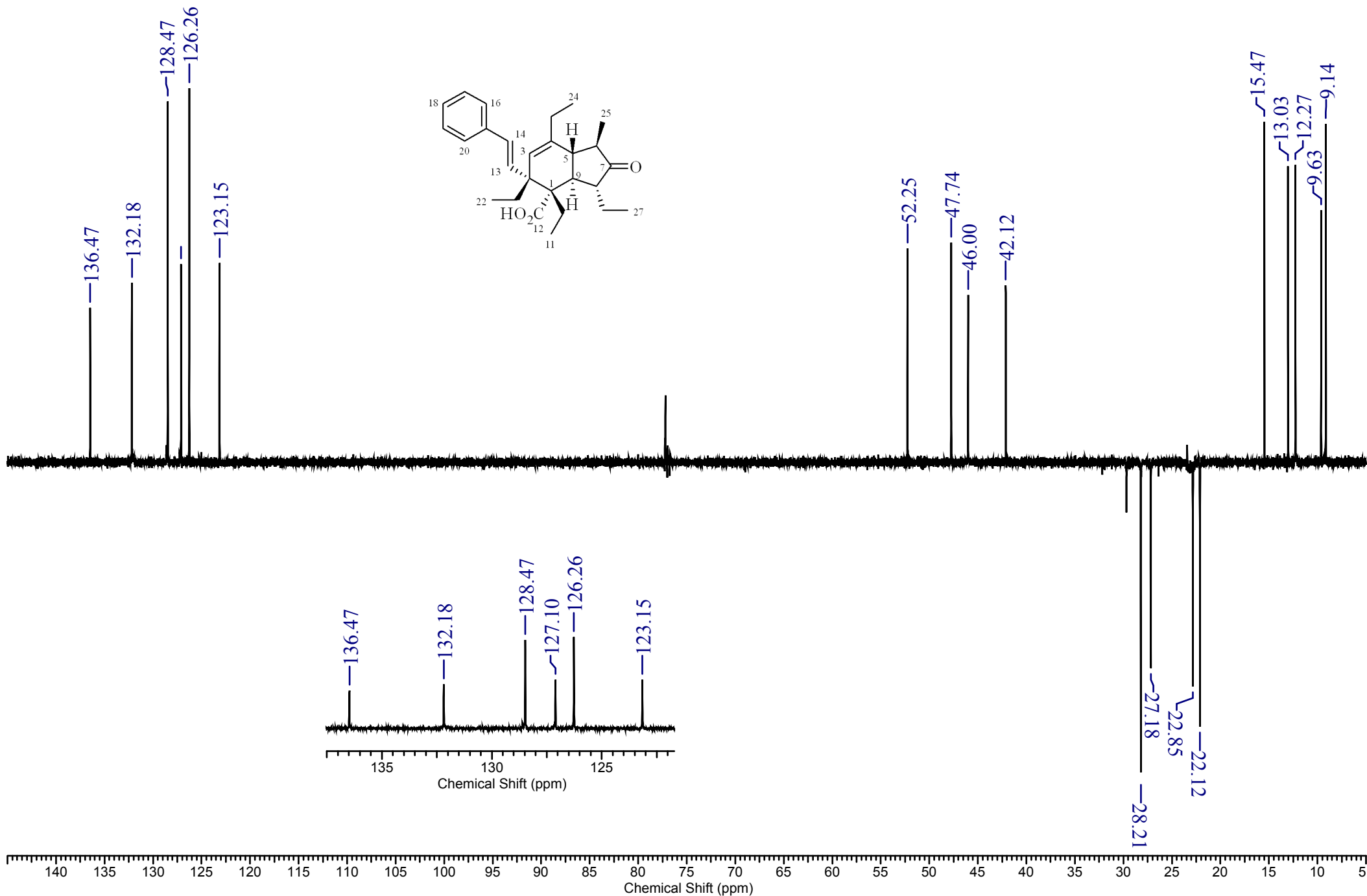
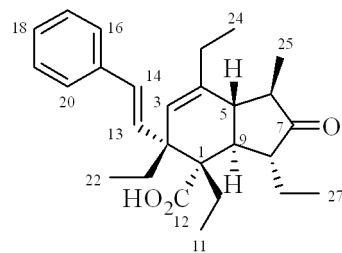
# Spiculoic Acid A: 500MHz, 1H



# Spiculoic Acid A: 500MHz, 13C

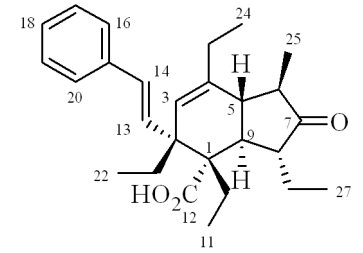
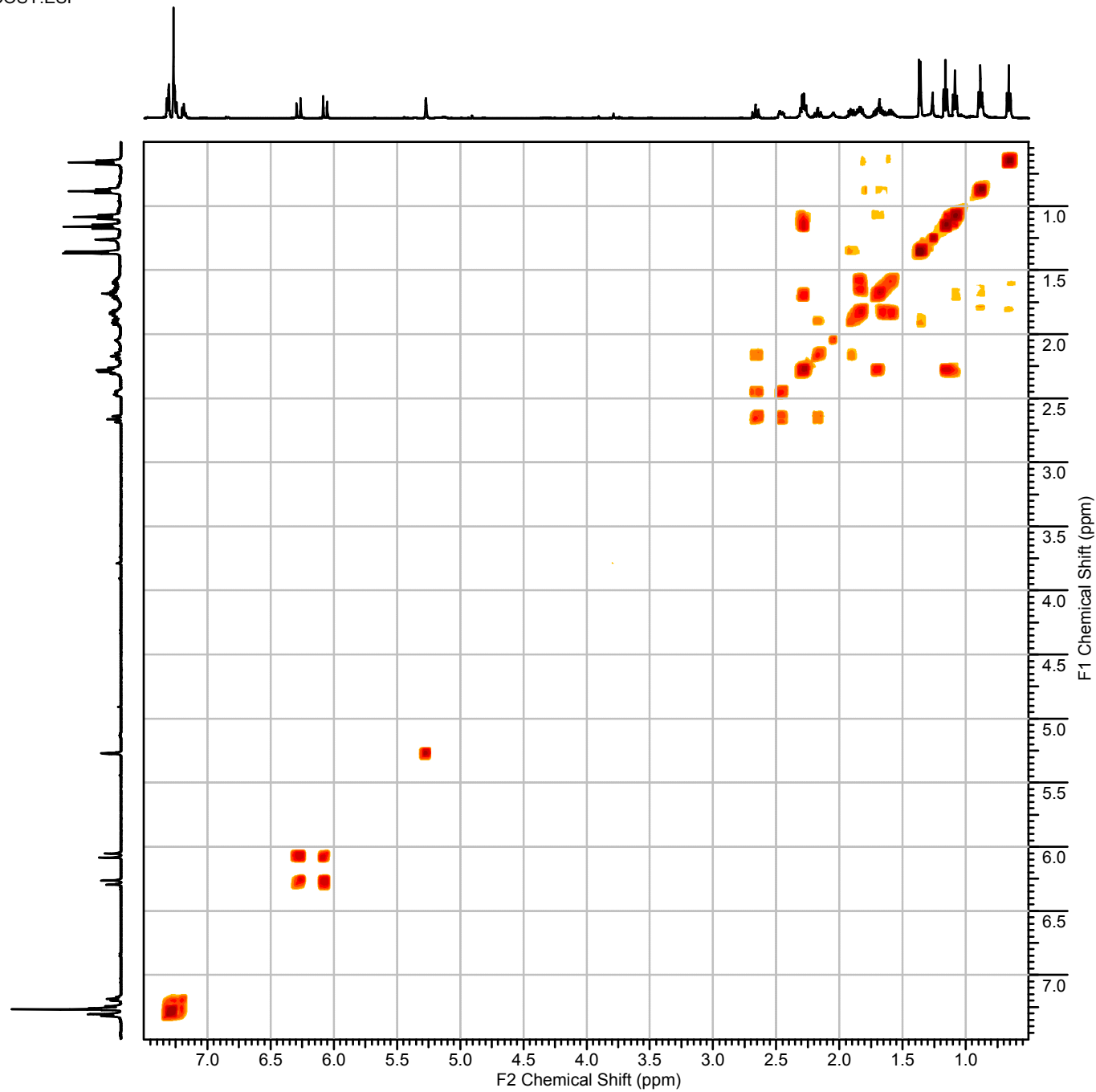


# Spiculoic Acid A: 500MHz, DEPT



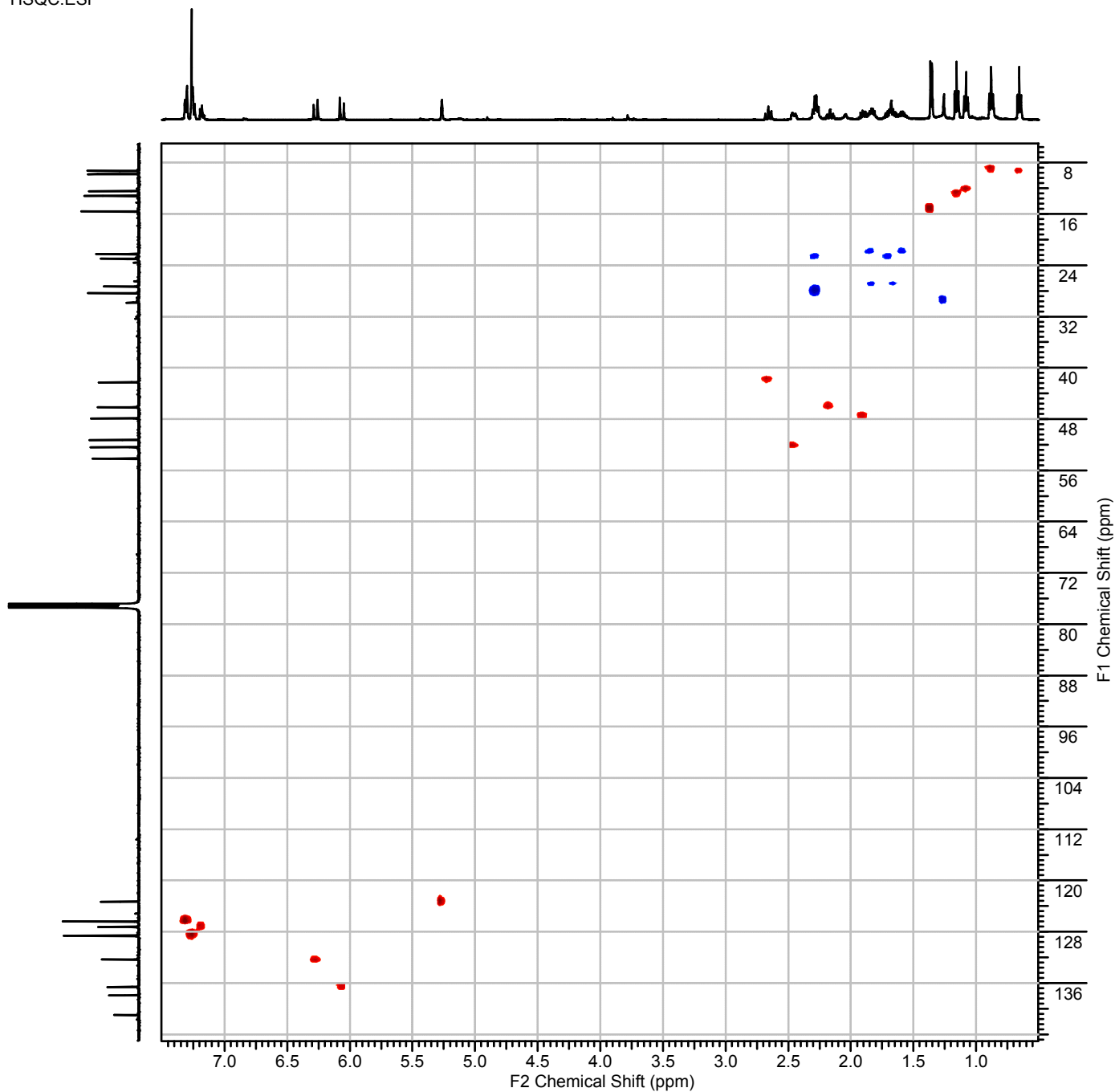
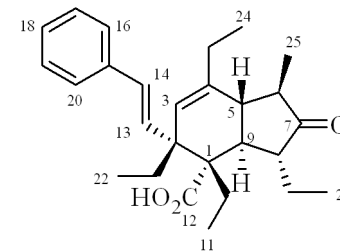
# Spiculoic Acid A: 500MHz, COSY

COSY.ESP



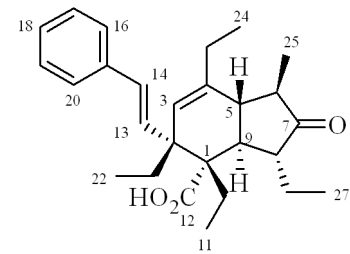
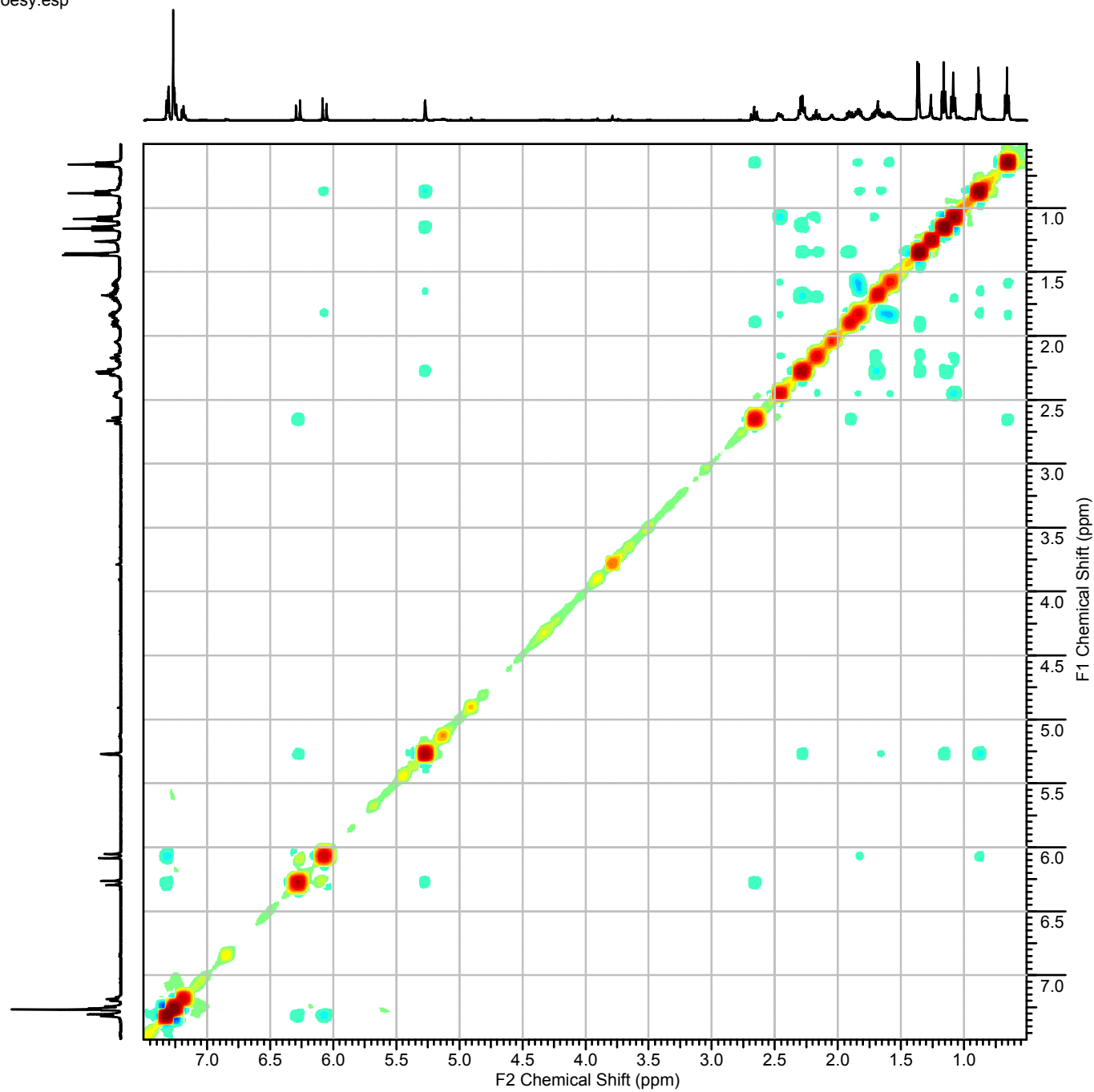
# Spiculoic Acid A: 500MHz, HSQC

HSQC.ESP



# Spiculoic Acid A: 500MHz, NOESY

noesy.esp



# Spiculoic Acid A: 500MHz, TOCSY @ 2.66ppm

