# Supplementary Materials

# Selective Extraction of Sinapic Acid Derivatives from Mustard Seed Meal by acting on pH: Toward a High Antioxidant Activity Rich Extract

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**Supplementary Materials and Methods**

NMR analyses were recorded on a Bruker Fourier 300. $^{1}$H NMR spectra of samples were measured on a 300 MHz apparatus at 25 °C; chemicals shifts were reported in parts per million relative to solvent residual peak ($CDC_{13}$ δ = 7.26 ppm; CD3OD, 4.87 ppm). $^{13}$C NMR spectra of samples were recorded at 75 MHz at 25 °C and calibrated on solvent peak ($CDC_{13}$ δ = 77.16 ppm; CD3OD, 49.2 ppm).

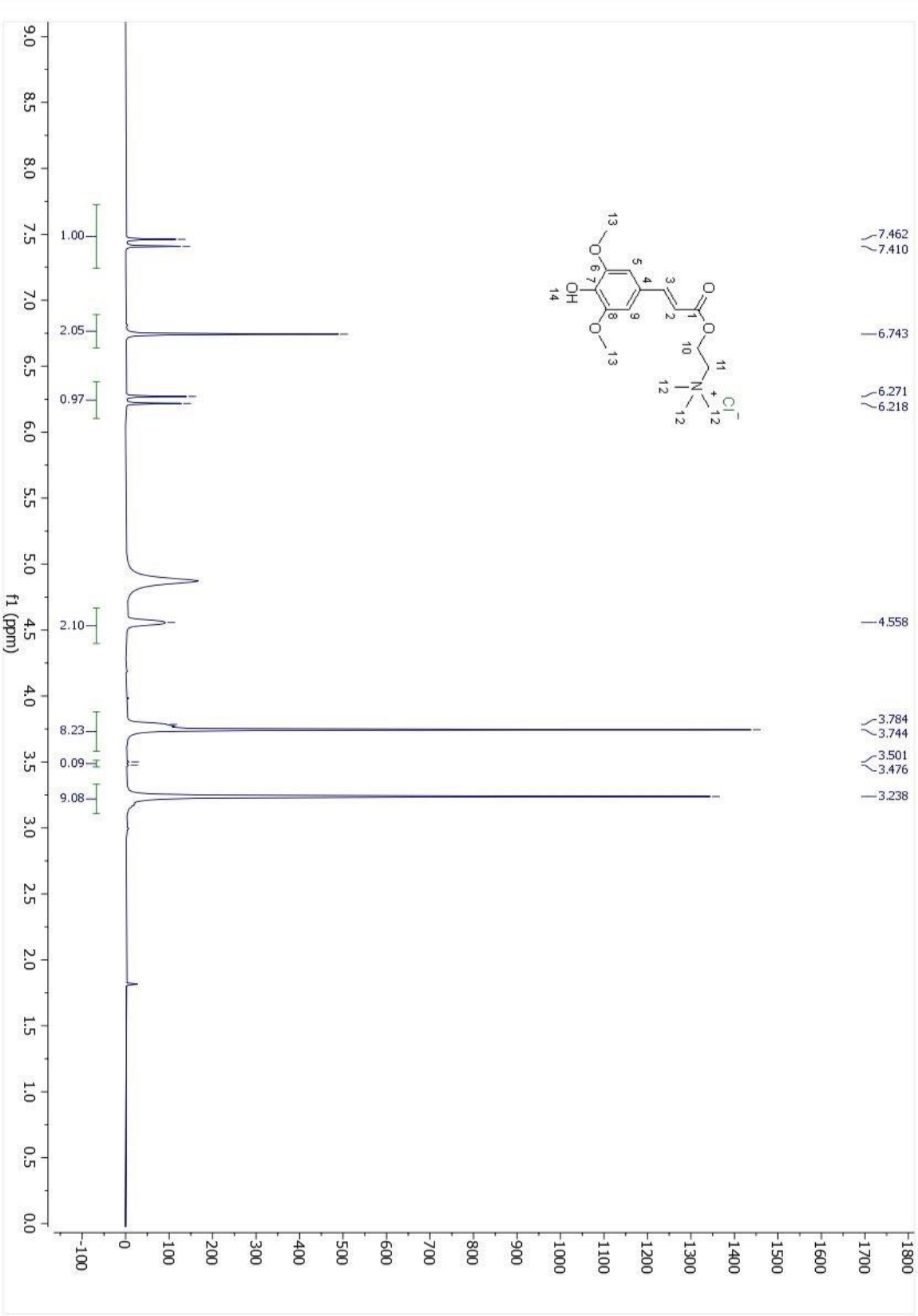
**Supplementary Table and Figures**

Table S1 : SADs composition of the studied extracts

| Ethanol (%v/v) | pH medium | Sinapine (mg/g DM) | Sinapic Acid (mg/g DM) | Ethyl Sinapate (mg/g DM) |
|---|---|---|---|---|
| 0% | pH 12 NaOH | N.D | 9.81 ± 0.69 | N.D |
| | pH 12 Buffer | N.D | 13.22 ± 0.44 | N.D |
| | pH 12 KOH | N.D | 9.79 ± 0.03 | N.D |
| | pH 2 | 11.35 ± 0.37 | 0.08 ± 0.04 | N.D |
| | pH non-controlled | 7.44 ± 0.3 | 0.1 ± 0.08 | N.D |
| 30% | pH 12 NaOH | N.D | 8.29 ± 0.07 | 6.20 ± 0.96 |
| | pH 12 Buffer | N.D | 10.15 ± 0.02 | 5.00 ± 0.04 |
| | pH 12 KOH | N.D | 6.68 ± 0.02 | 6.46 ± 0.12 |
| | pH 2 | 11.32 ± 0.77 | 0.05 ± 0.01 | N.D |
| | pH non-controlled | 9.57 ± 0.28 | 9.57 ± 0.29 | N.D |
| 50% | pH 12 NaOH | N.D | 5.80 ± 0.02 | 8.70 ± 0.42 |
| | pH 12 Buffer | N.D | 8.28 ± 0.01 | 7.78 ± 0.03 |
| | pH 12 KOH | N.D | 5.81 ± 0.22 | 8.21 ± 0.49 |
| | pH 2 | 11.78 ± 0.38 | 0.08 ± 0.01 | N.D |
| | pH non-controlled | 11.35 ± 0.17 | 0.07 ± 0.03 | N.D |
| 70% | pH 12 NaOH | N.D | 7.43 ± 0.42 | 8.97 ± 0.05 |
| | pH 12 Buffer | N.D | 7.31 ± 0.06 | 9.81 ± 0.31 |
| | pH 12 KOH | N.D | 5.13 ± 0.31 | 9.69 ± 0.61 |
| | pH 2 | 15.73 ± 0.54 | 0.08 ± 0.03 | N.D |
| | pH non-controlled | 13.03 ± 0.17 | 0.07 ± 0.04 | N.D |

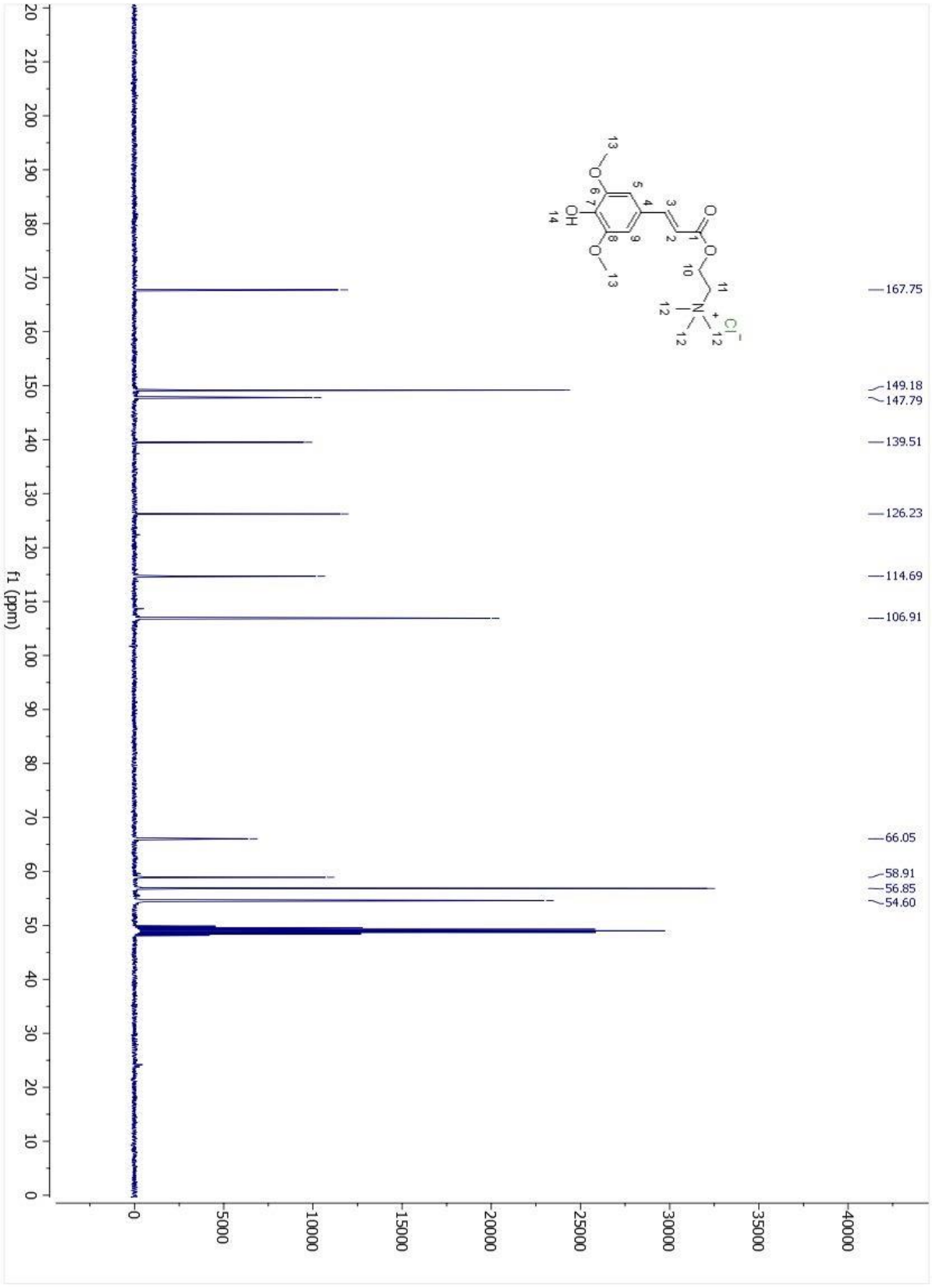
N.D : non-determined

Figure S1 : $^{1}$H-NMR spectrum of sinapine.



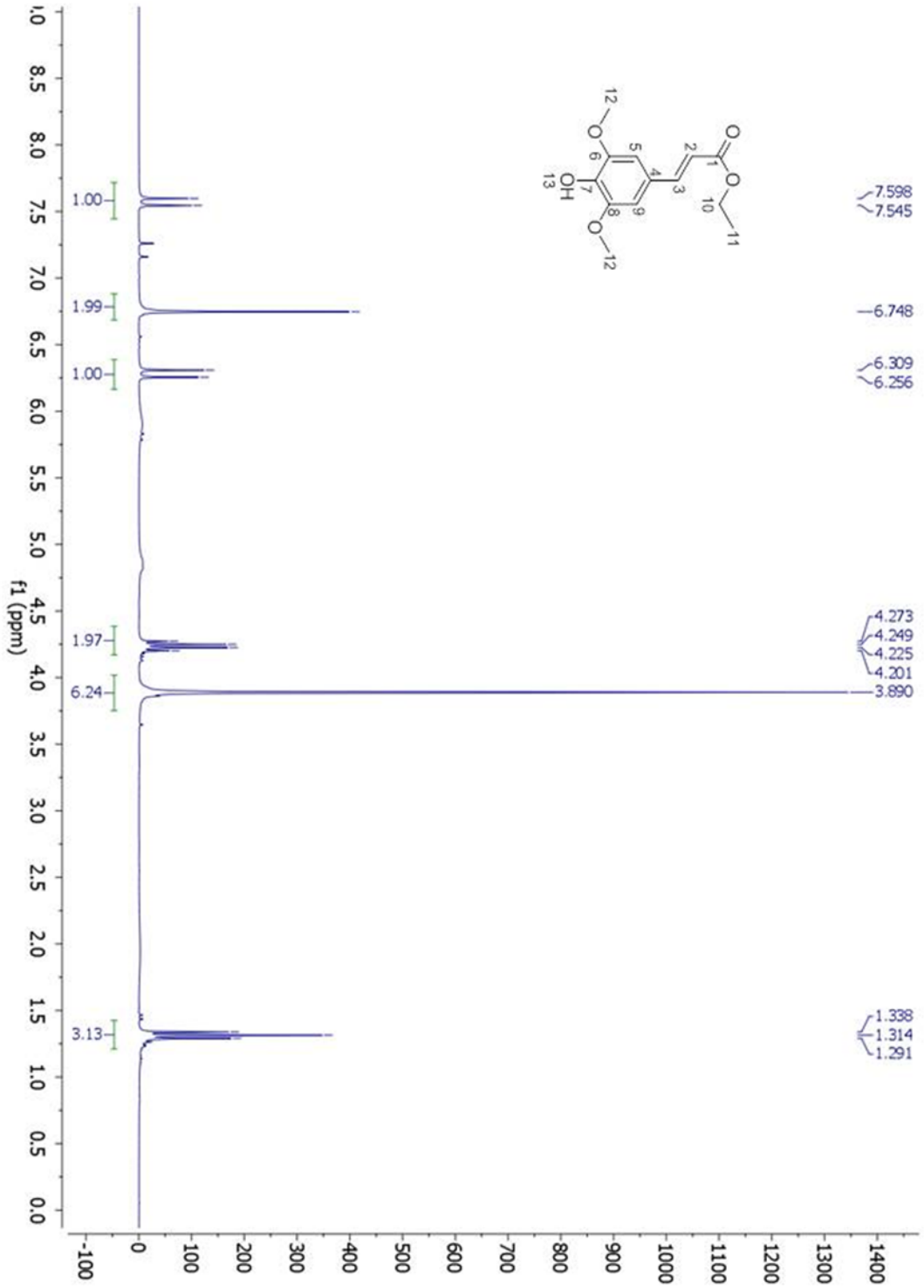

$^{1}$H NMR (300 MHz, $CD_3OD$): δ = 7.44 (d, J = 15.9 Hz, 1H, H-3), 6.74 (s, 2H, H-5 and 9), 6.24 (d, J = 15.9 Hz, $^{1}$H, H-2), 4.56 (m, 2H, H-10), 3.78 (m, 2H, H-11), 3.74 (s, 6H, H-13), 3.24 (s, 9H, H-12).

Figure S2 : $^{13}$C-NMR spectrum of sinapine



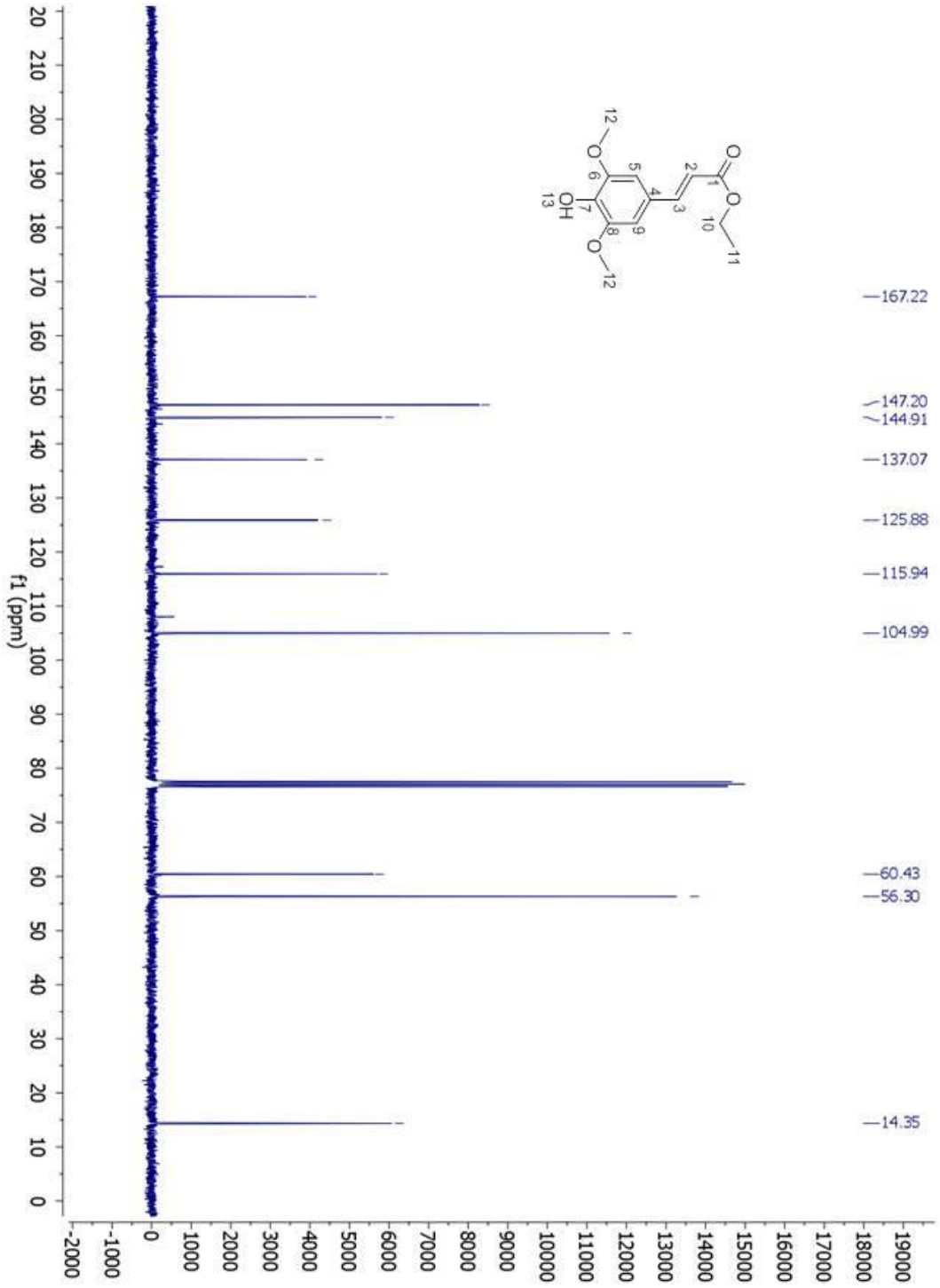

$^{13}$C NMR (75 MHz, $CD_3OD$): δ = 167.7 (s, C-1), 149.2 (s, C-6 and 8), 147.8 (d, C-3), 139.5 (s, C-7), 126.2 (s, C-4), 114.7 (d, C-2), 106.9 (d, C-5 and 9), 66.1 (t, C-11), 59.9 (t, C-10), 56.8 (q, C-12), 54.6 (q, C-13).

Figure S3 : $^{1}$H-NMR spectrum of ethyl sinapate




$^{1}$H NMR (300 MHz, $CDC_{l3}$): δ = 7.57 (d, J = 15.9 Hz, 1H, H-3), 6.75 (s, 2H, H-5 and 9), 6.28 (d, J = 15.9 Hz, 1H, H-2), 4.23 (q, J = 7.2 Hz, 2H, H-10), 3.89 (s, 6H, H-12), 1.31 (t, J = 7.2 Hz, 3H, H-11).

Figure S4 : $^{13}$C-NMR spectrum of ethyl sinapate




$^{13}$C NMR (75 MHz, $CDC_{13}$): δ = 167.2 (s, C-1), 147.2 (s, C-6 and 8), 144.9 (d, C-3), 137.1 (s, C-7), 125.9 (s, C-4), 115.9 (d, C-2), 105.0 (d, C-5 and 9), 60.4 (t, C-10), 59.9, 56.3 (q, C-12), 14.4 (q, C-11).