

Characterization of precipitation from citrus vinegar during ageing: Chemical constituents, formation mechanism and anti-proliferative effect

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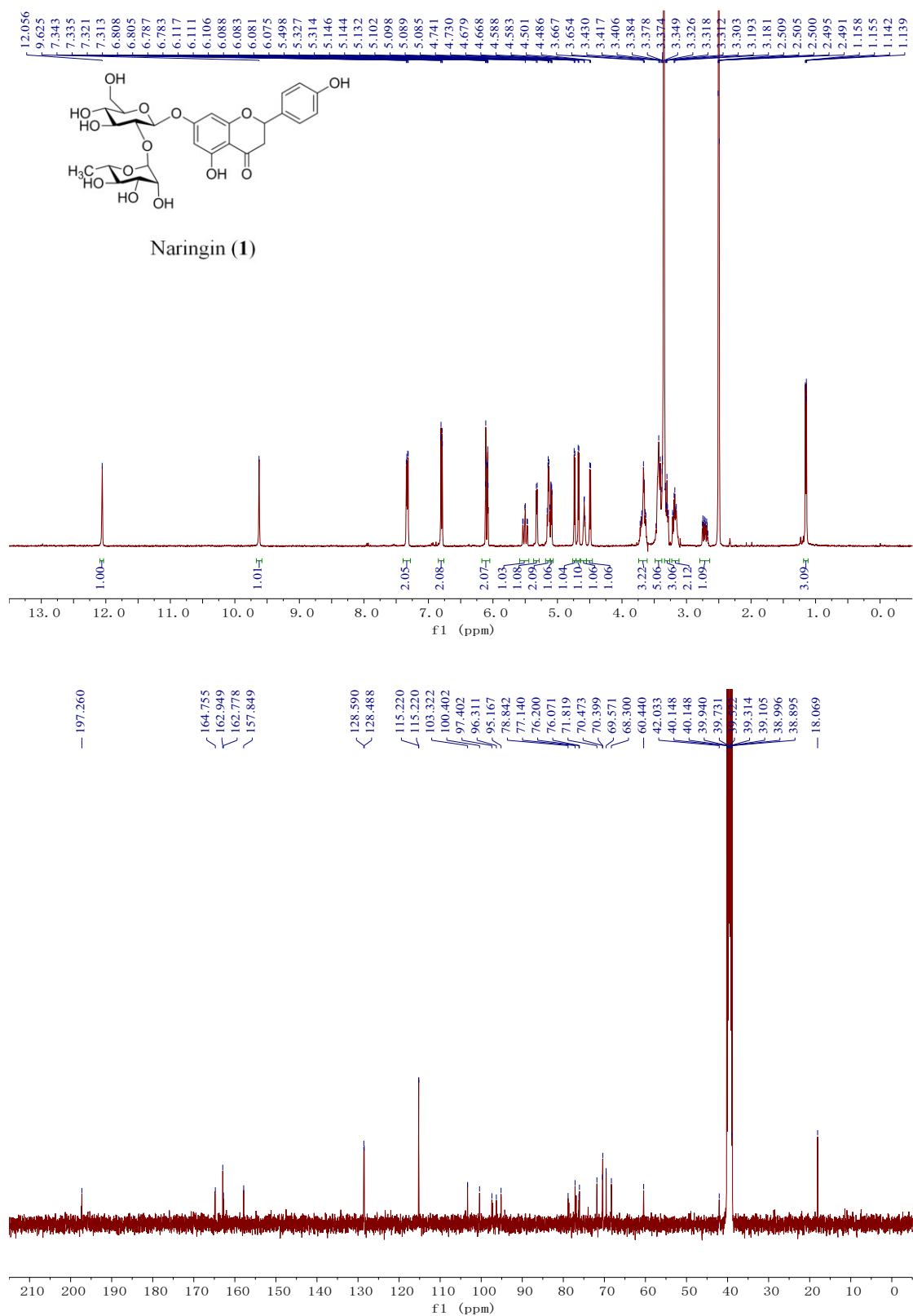


Fig.S1. ¹H and ¹³C NMR spectrum (400 MHz, DMSO-*d*₆) of naringin(1)

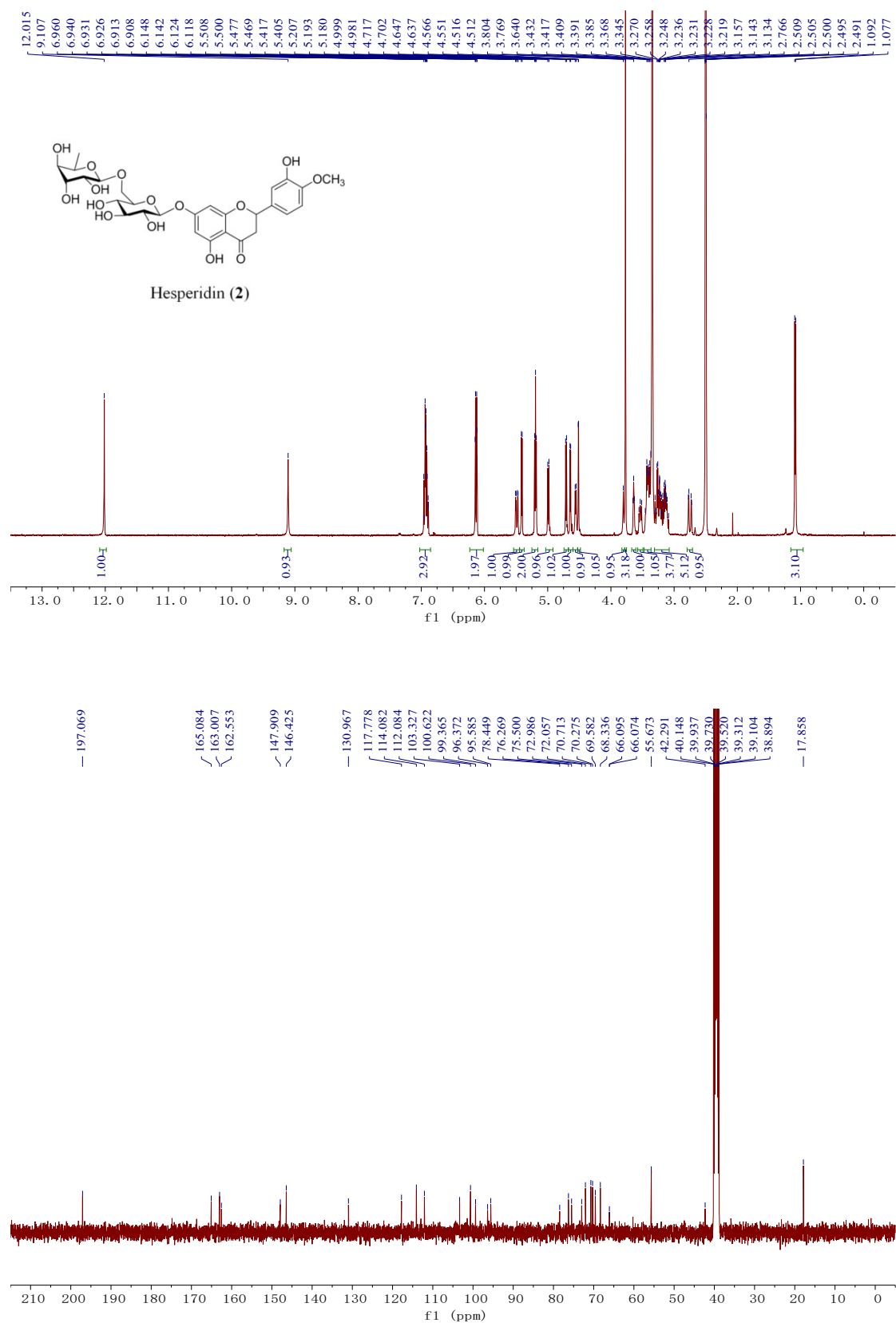


Fig.S2. ¹H and ¹³C NMR spectrum (400 MHz, DMSO-*d*₆) of hesperidin(2)

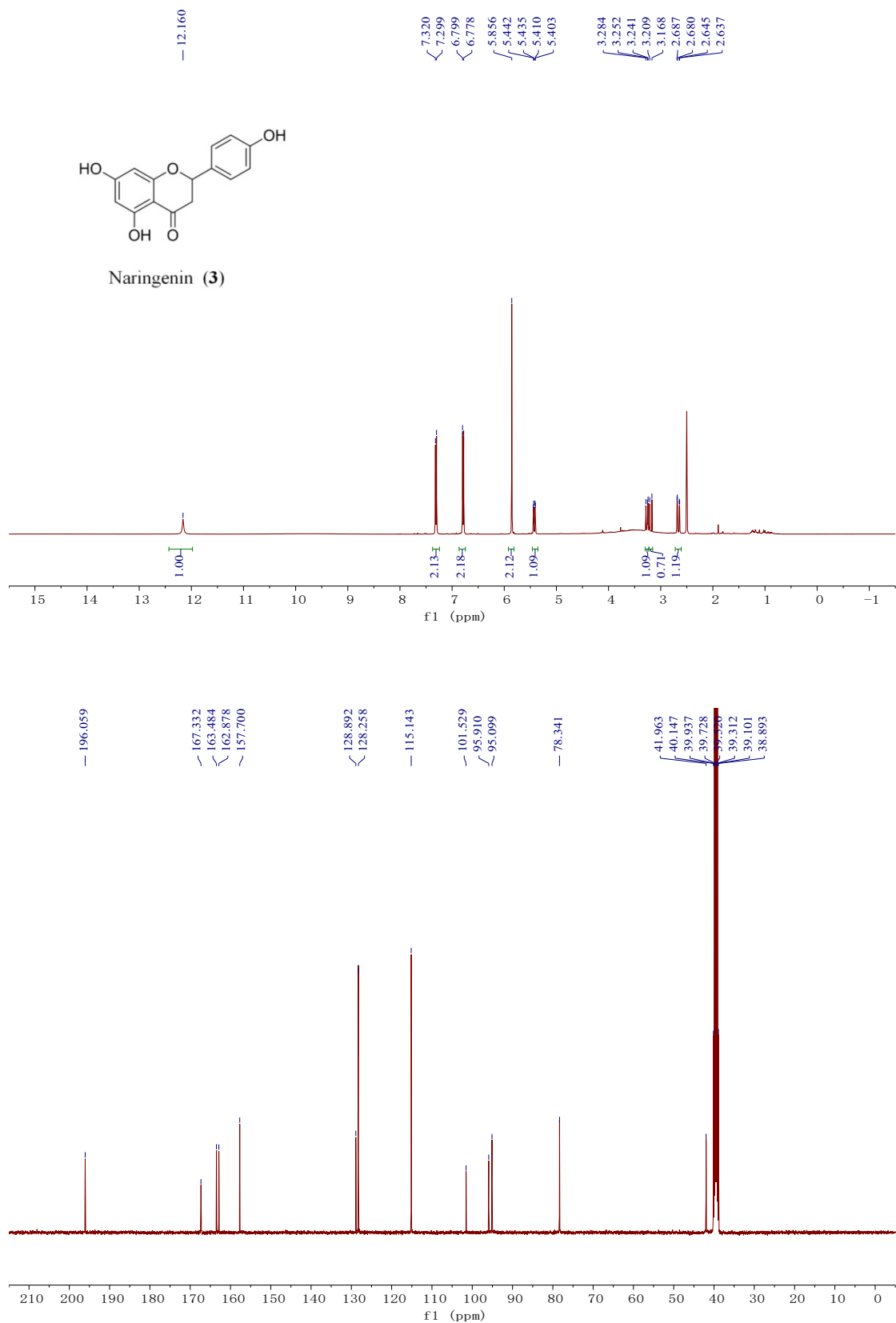


Fig.S3. ¹H and ¹³C NMR spectrum (400 MHz, DMSO-*d*₆) of naringenin(**3**)

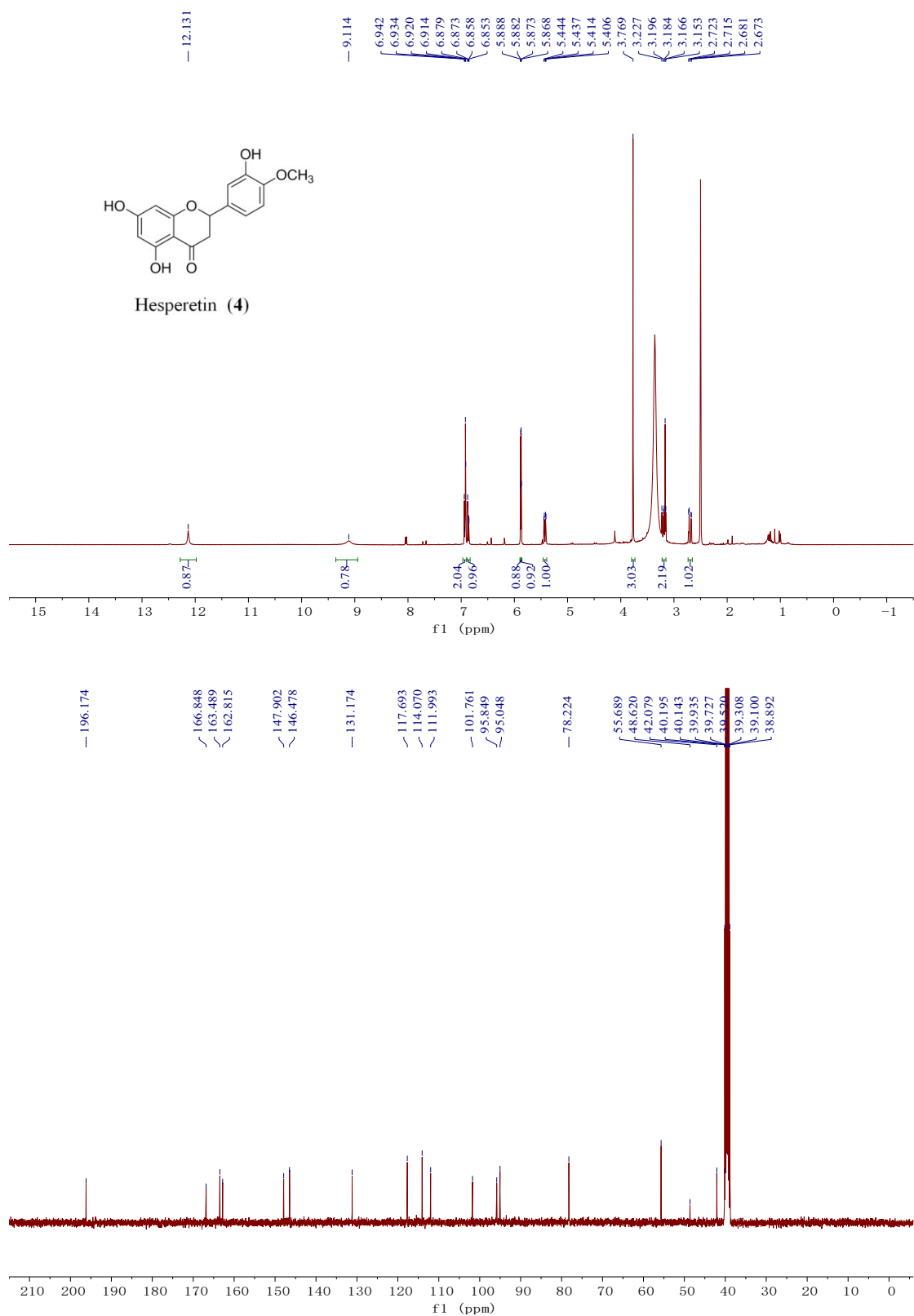


Fig.S4. ¹H and ¹³C NMR spectrum (400 MHz, DMSO-*d*₆) of hesperetin(**4**)

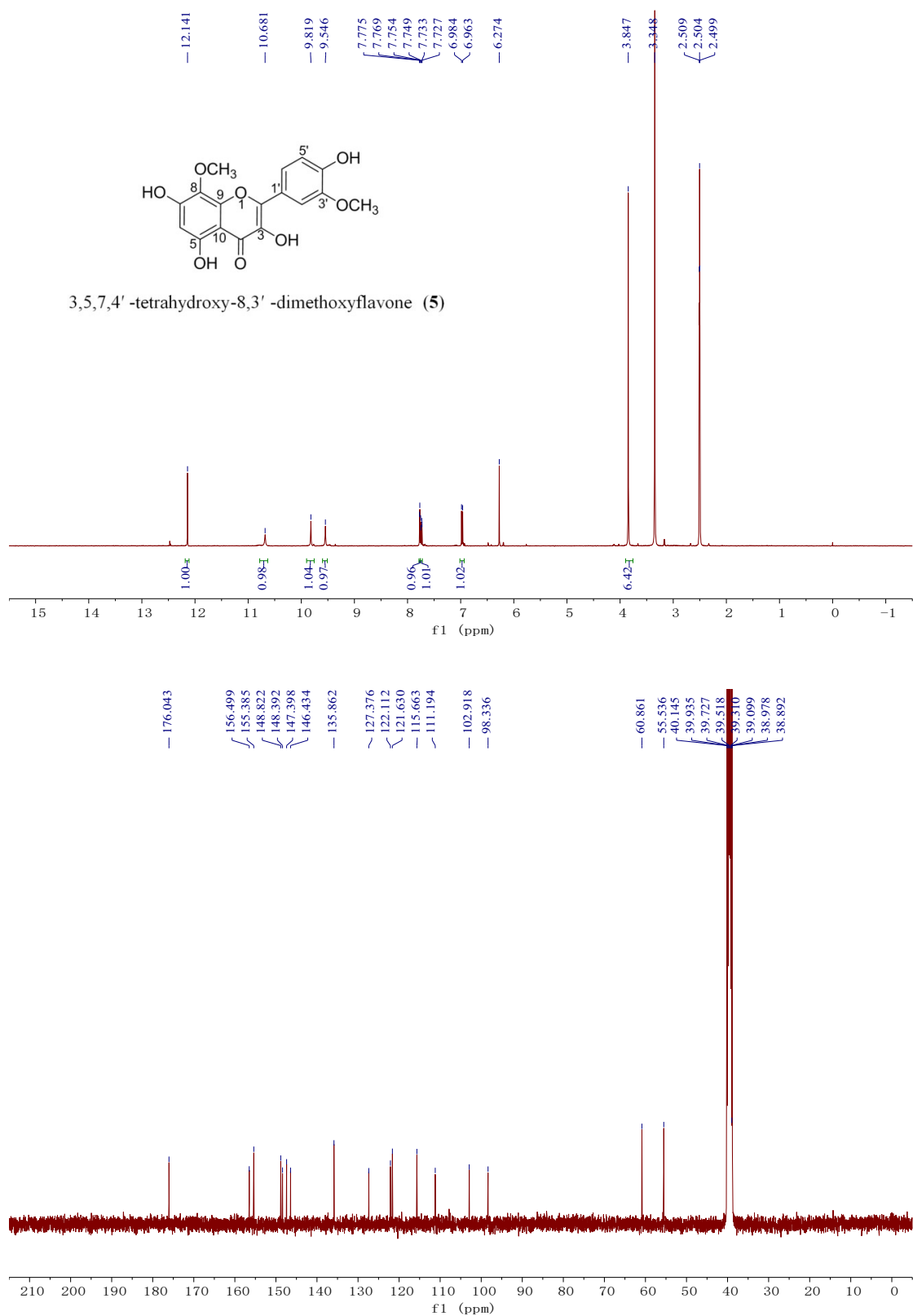


Fig.S5. ¹H and ¹³C NMR spectrum (400 MHz, DMSO-*d*₆) of 3,5,7,4'-tetrahydroxy-8, 3'-dimethoxyflavone(**5**)

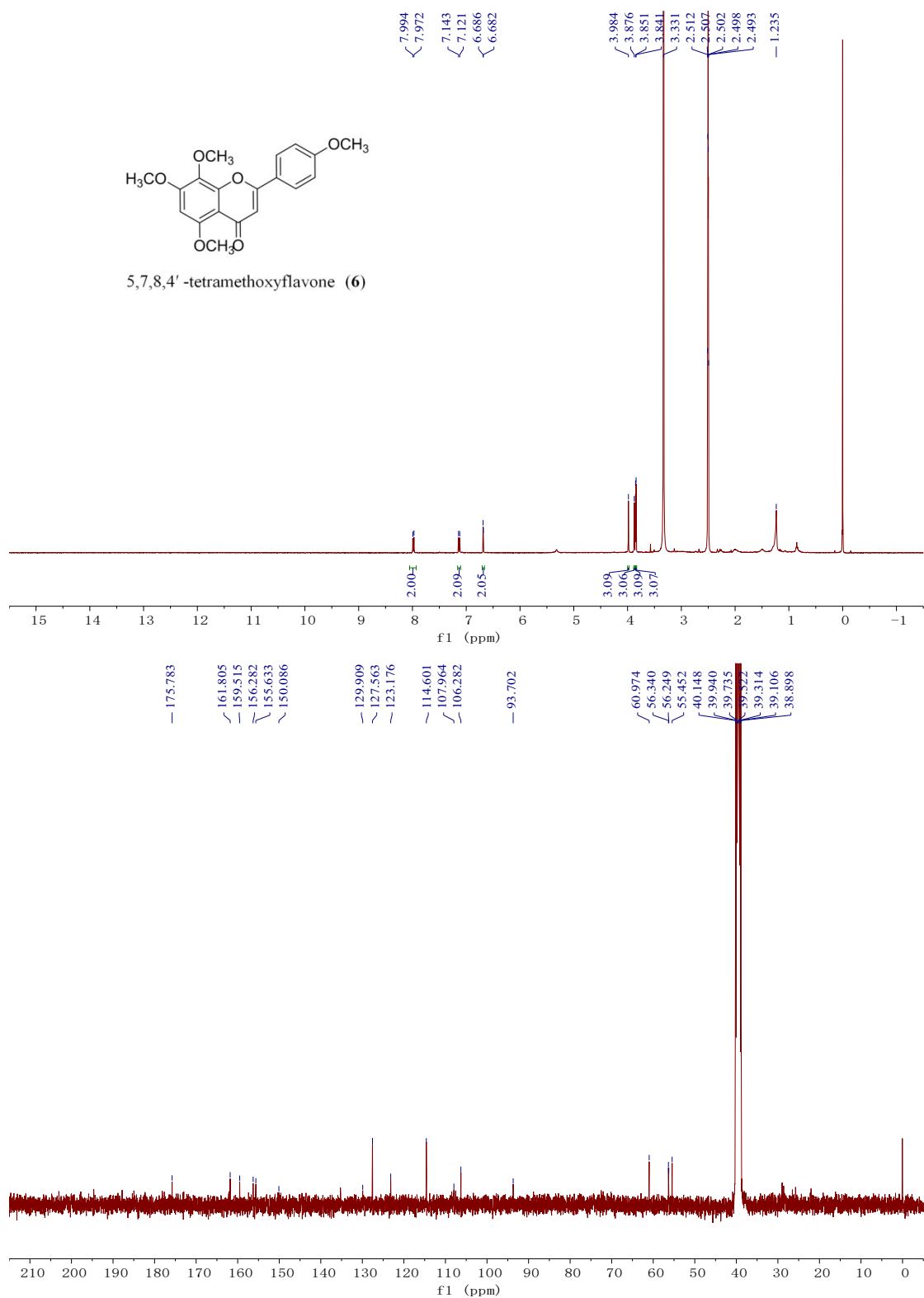


Fig.S6. ¹H and ¹³C NMR spectrum (400 MHz, DMSO-*d*₆) of 5,7,8,4'-tetramethoxyflavone(6)

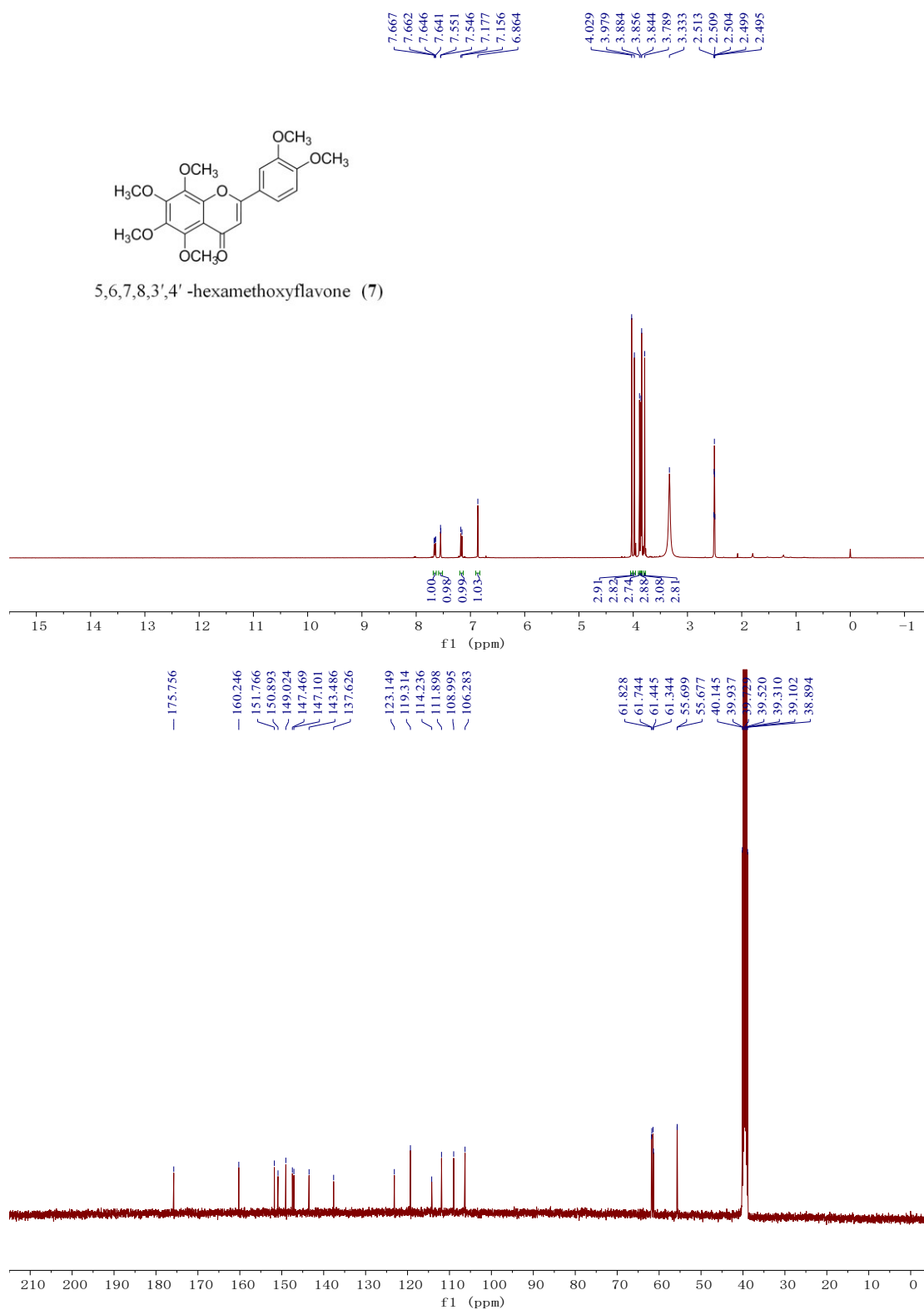


Fig.S7. ¹H and ¹³C NMR spectrum (400 MHz, DMSO-*d*₆) of 5,6,7,8,3',4'-hexamethoxyflavone(7)

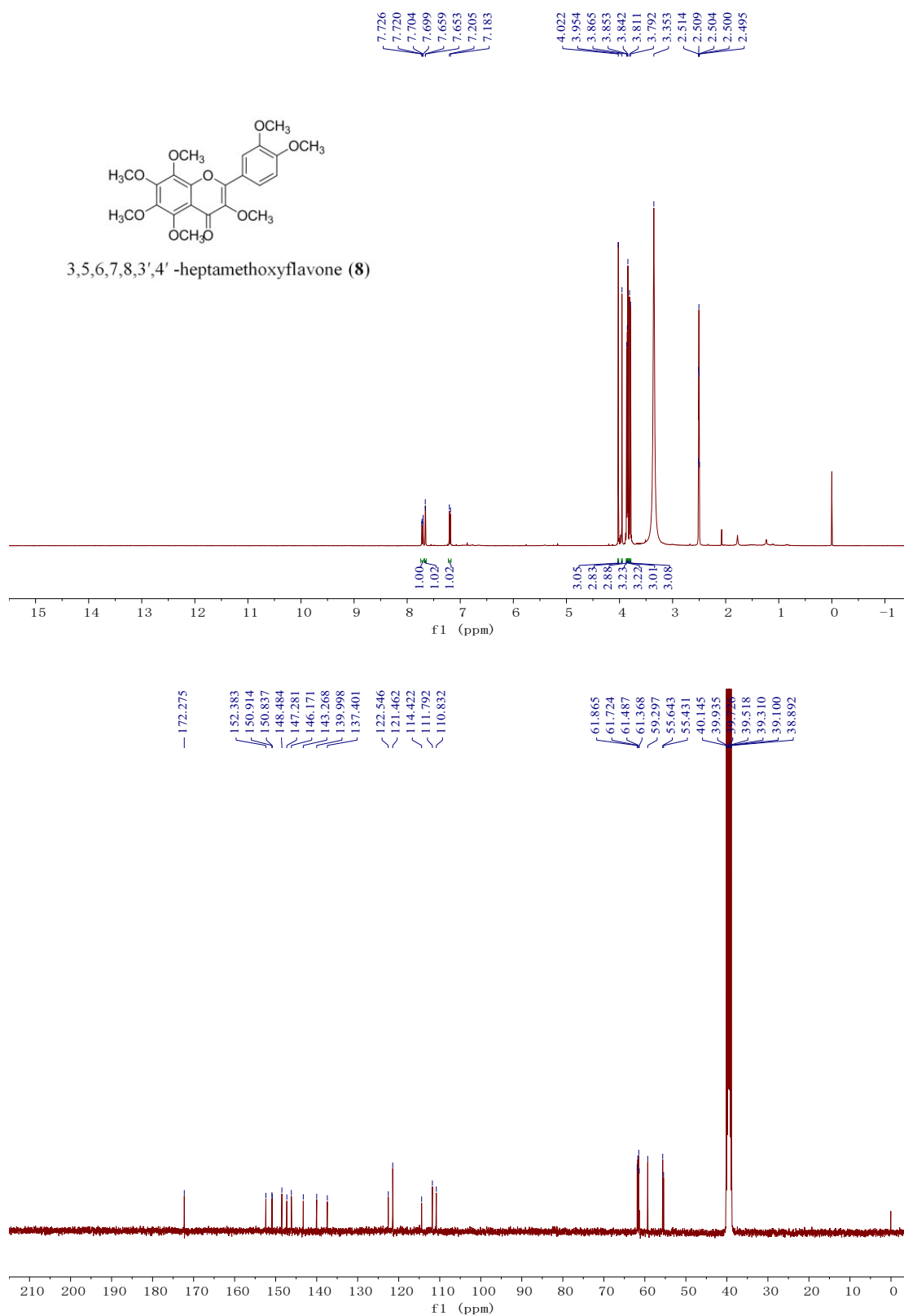


Fig.S8. ¹H and ¹³C NMR spectrum (400 MHz, DMSO-*d*₆) of 3,5,6,7,8,3',4'-heptamethoxyflavone(**8**)

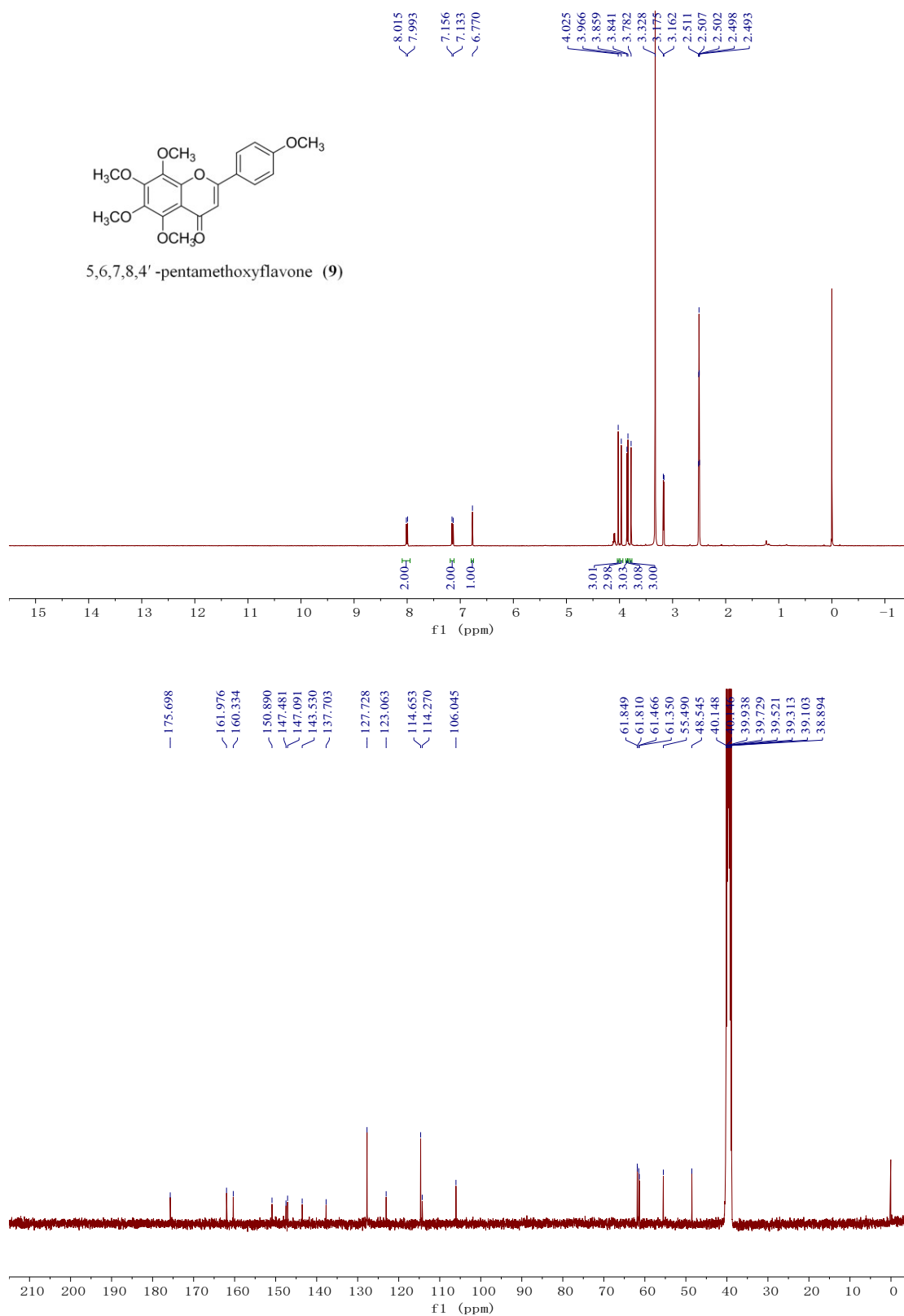


Fig.S9. ¹H and ¹³C NMR spectrum (400 MHz, DMSO-*d*₆) of 5,6,7,8,4'-pentamethoxyflavone(9)

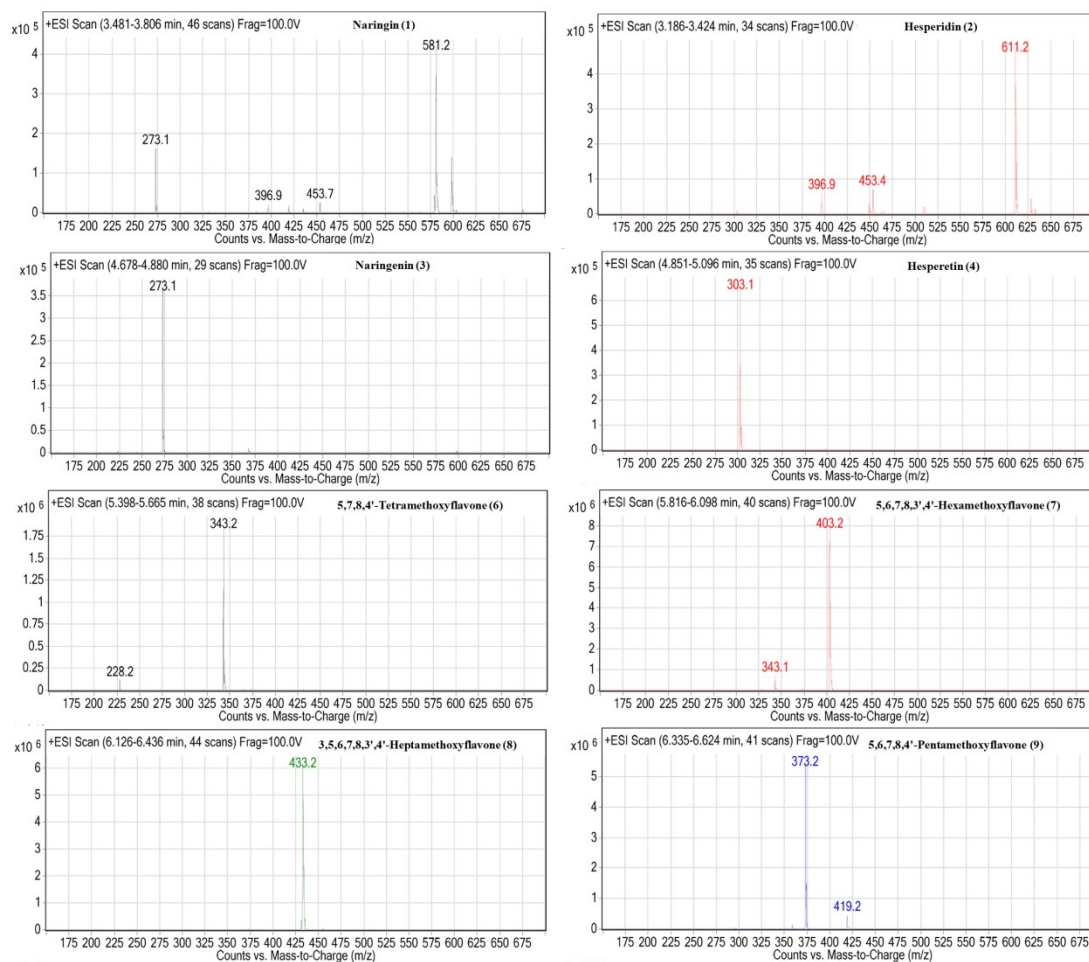


Fig.S10. The MS spectrum of principal components of precipitates.

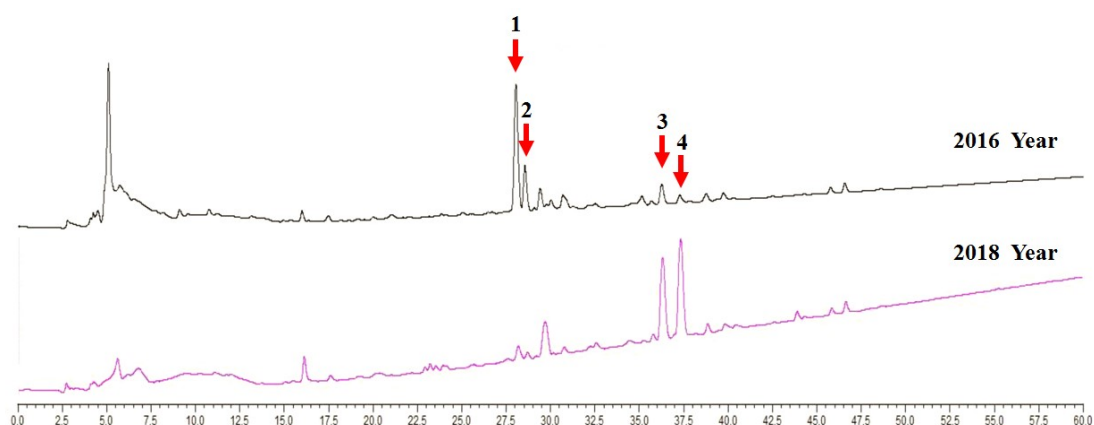


Fig.S11. The HPLC analysis of naringin (1), hesperidin (2), naringenin (3) and hesperetin (4) in new citrus vinegar produced in 2016 and aged 2 year (2018) citrus vinegar.

Table S1

Changes in Naringin (1), Hesperidin (2), Naringenin (3) and Hesperetin (4) Contents during Citrus Vinegar Production.

Sample	Naringin (mg/L)	Hesperidin (mg/L)	Naringenin (mg/L)	Hesperetin (mg/L)
New citrus vinegar (produced in 2016)	72.35 ± 0.97	39.54 ± 0.64	8.53 ± 0.25	4.98 ± 0.11
Aged 2 year citrus vinegar (resampled in 2018)	3.35 ± 0.05	2.74 ± 0.34	18.30 ± 0.66	27.59 ± 1.26
Precipitates (2016)	n. m.	n. m.	103 ± 3 *	77 ± 7 *
Precipitates (2018)	n. m.	n. m.	215 ± 11 *	149 ± 15 *

n. m. : Not measured; * Contents value is measured in mg/g.

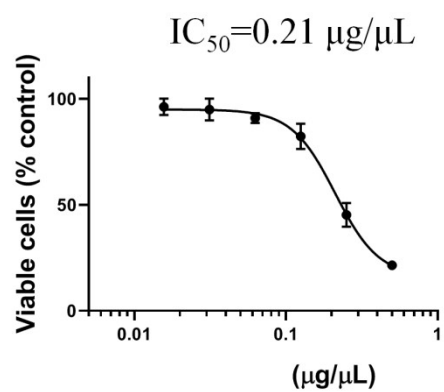


Fig. S12. Anti-proliferative effects of precipitates extract from model III on MCF-7 cells