

IDENTIFICAÇÃO DE ALGUNS CONSTITUINTES QUÍMICOS DE *Indigofera hirsuta* LINN. (FABACEAE) POR CLAE-IES-EM (TOF) E AVALIAÇÃO DA ATIVIDADE ANTIRRADICALAR

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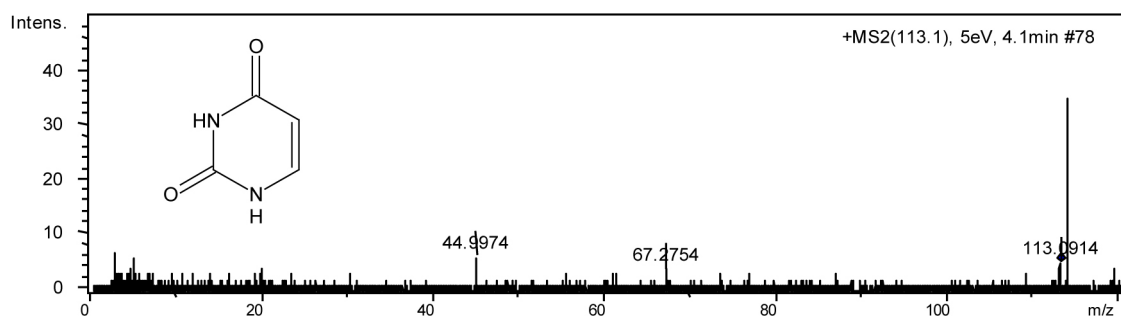


Figura 1S. Espectro de massas (EM/EM) da uracila (1)

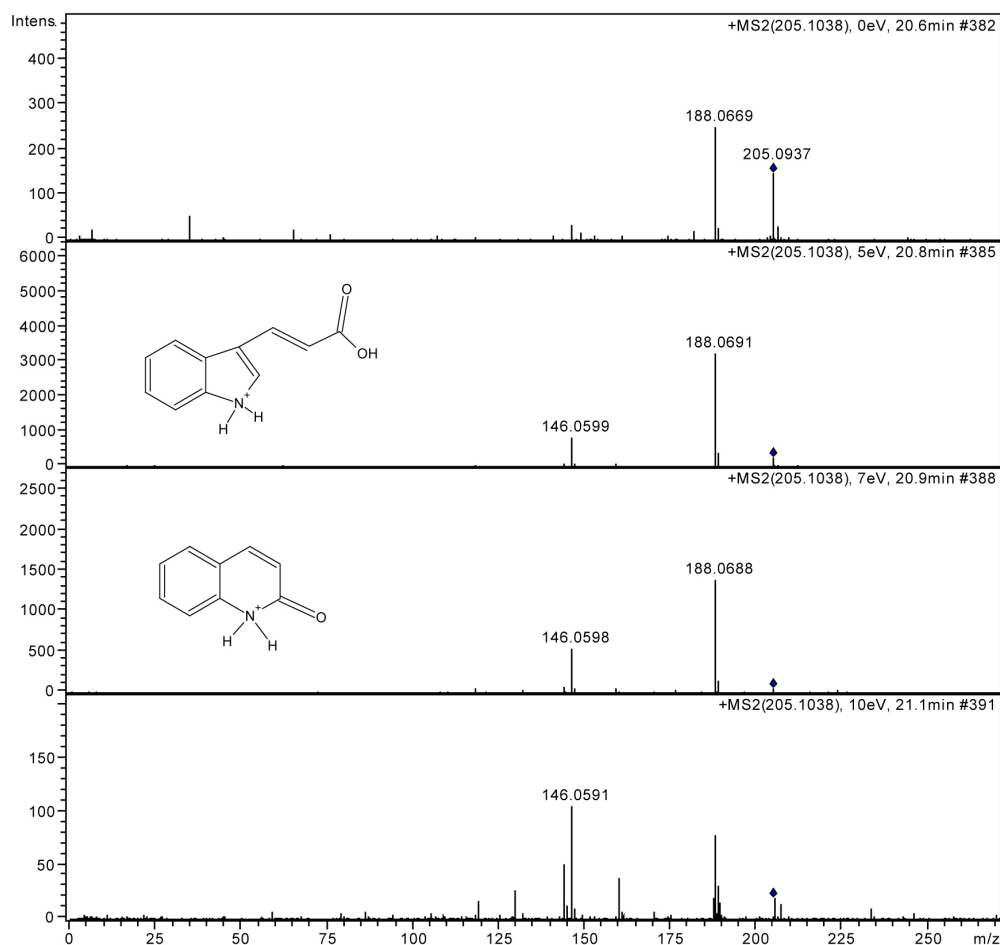


Figura 2S. Espectro de massas (EM/EM) de triptofano (2) em diferentes energias (0, 5, 7 e 10 eV)

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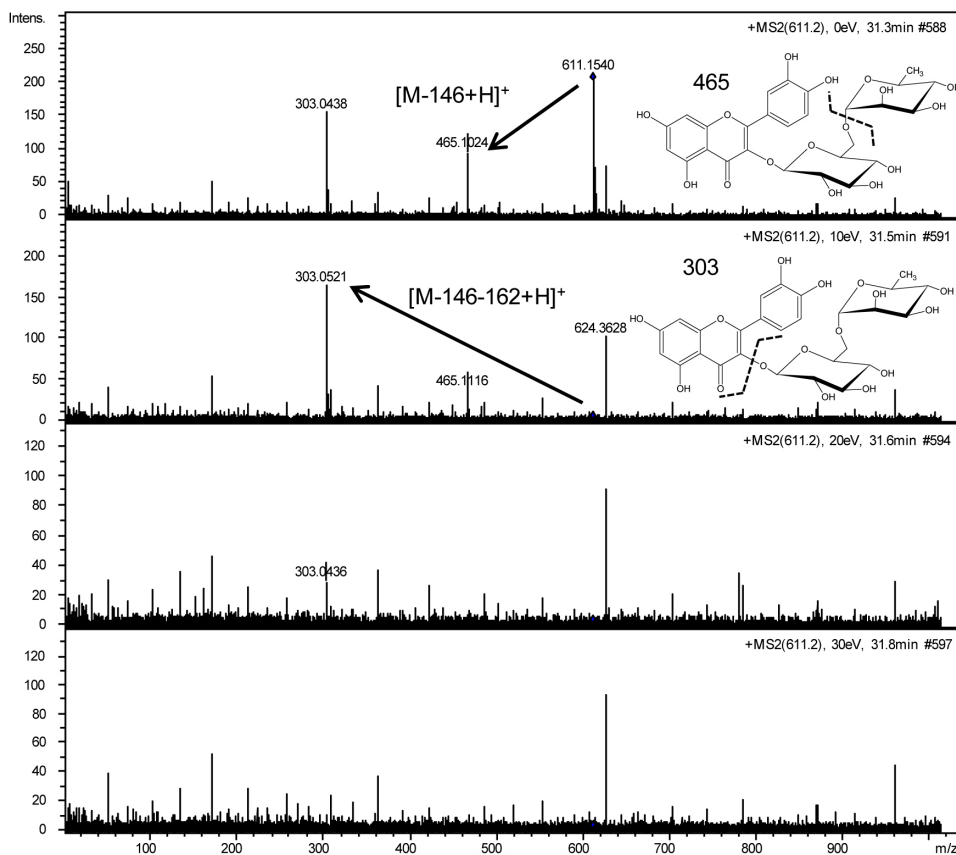


Figura 3S. Espectro de massas (EM/EM) da rutina (**3**) em diferentes energias (0, 10, 20 e 30 eV)

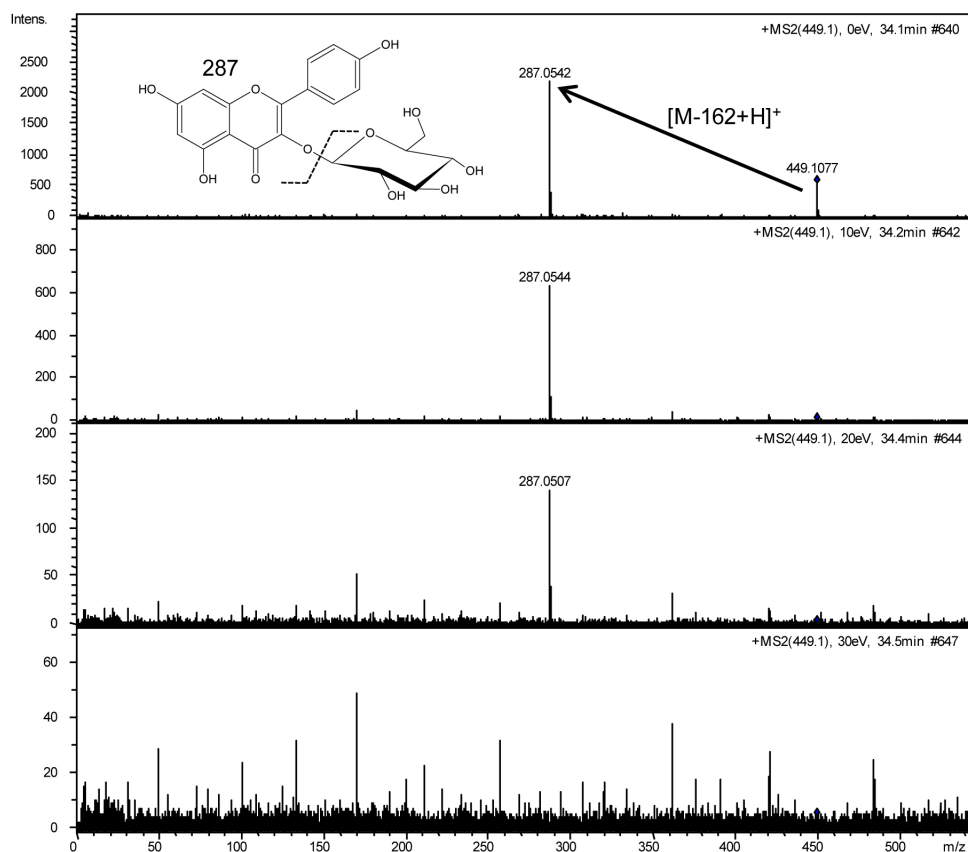


Figura 4S. Espectro de massas (EM/EM) do kaempferol-3-O- β -D-glucopiranosídeo (**4**) em diferentes energias (0, 10, 20 e 30 eV)

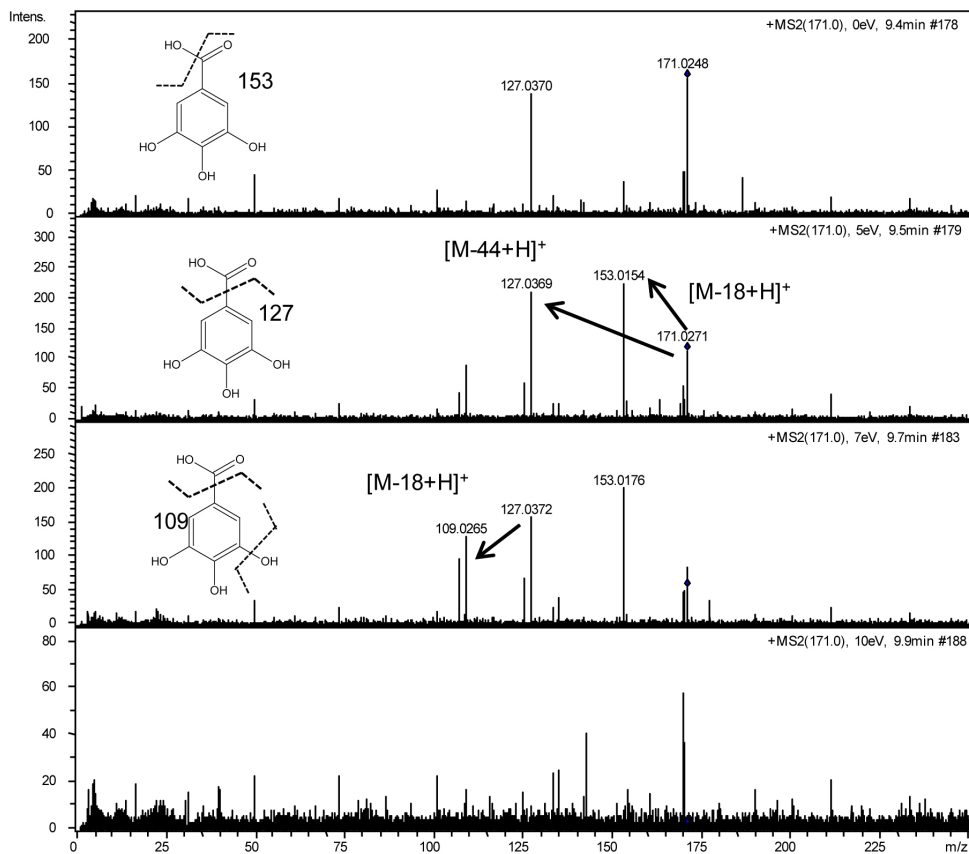


Figura 5S. Espectro de massas (EM/EM) do ácido gálico (5) em diferentes energias (0, 5, 7 e 10 eV)

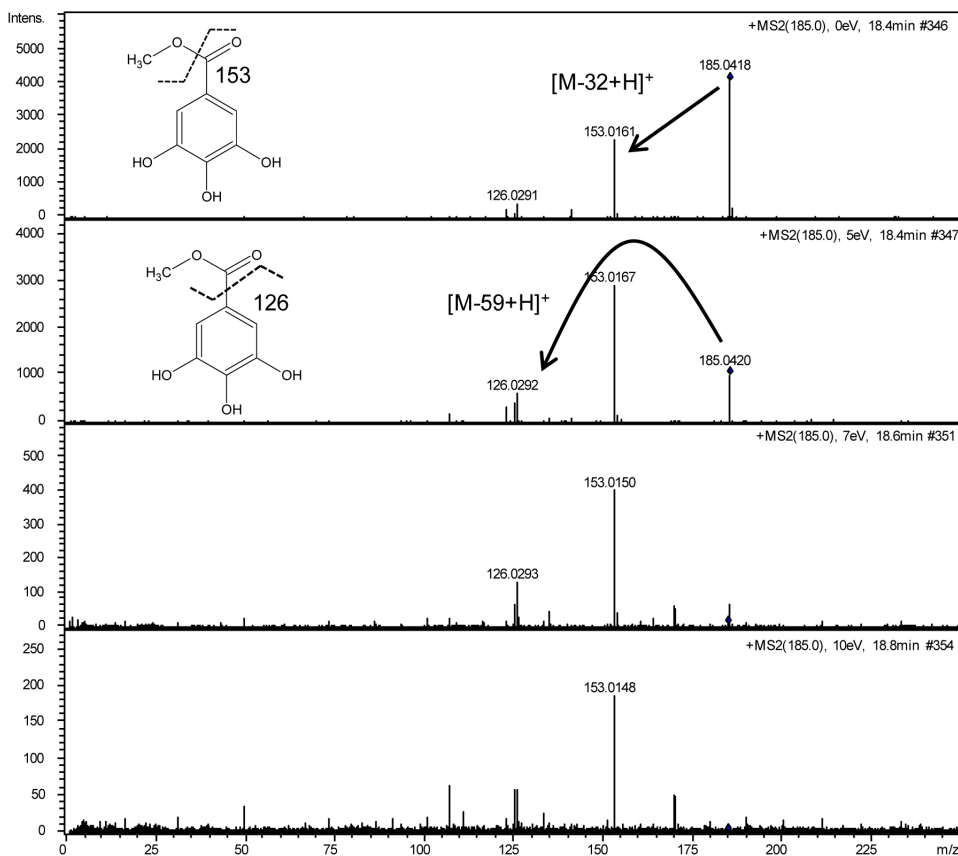


Figura 6S. Espectros de massas (EM/EM) do galato de metila (6) em diferentes energias (0, 5, 7 e 10 eV)

Dados de RMN de ^1H , de ^{13}C (obtidos através de experimentos gHMQC e gHMBC) e de UV e IV das substâncias (1-8):

Triptofano (1): RMN ^1H [500 MHz, DMSO- d_6 , δ (ppm), J (Hz)]: 7,52 (s; H-2); 7,58 (d, J=8,0 Hz; H-4); 6,98 (ddd, J=8,0; J=8,0 e J=0,5 Hz; H-5); 7,07 (ddd, J=8,0; J=8,0 e J=0,5 Hz; H-6); 7,35 (d, J=8,0 Hz; H-7); 3,5 (m; H- α); 3,32 e 2,99 (m; H- β). RMN ^{13}C [125 MHz, DMSO- d_6 , δ (ppm)]: 124,0 (C-2); 110,0 (C-3); 118,0 (C-4; C-5); 121,0 (C-6); 111,0 (C-7); 127,0 (C-3a); 136,0 (C-7a); 54,0 (C- α); 27,0 (C- β). UV: 278 nm. IV (cm^{-1}): ν_{NH_2} em 3288; $\nu_{\text{C-H}}$ em 2920 e ν_{CH_2} em 2853; $\delta_{\text{N-H(ass)}}$ em 1631 cm^{-1} ; $\nu_{\text{C=C(aromático)}}$ e $\nu_{\text{C=N(aromático)}}$ em 1602; $\nu_{\text{C-O}}$ em 1402; $\nu_{\text{C-N}}$ em 1091.

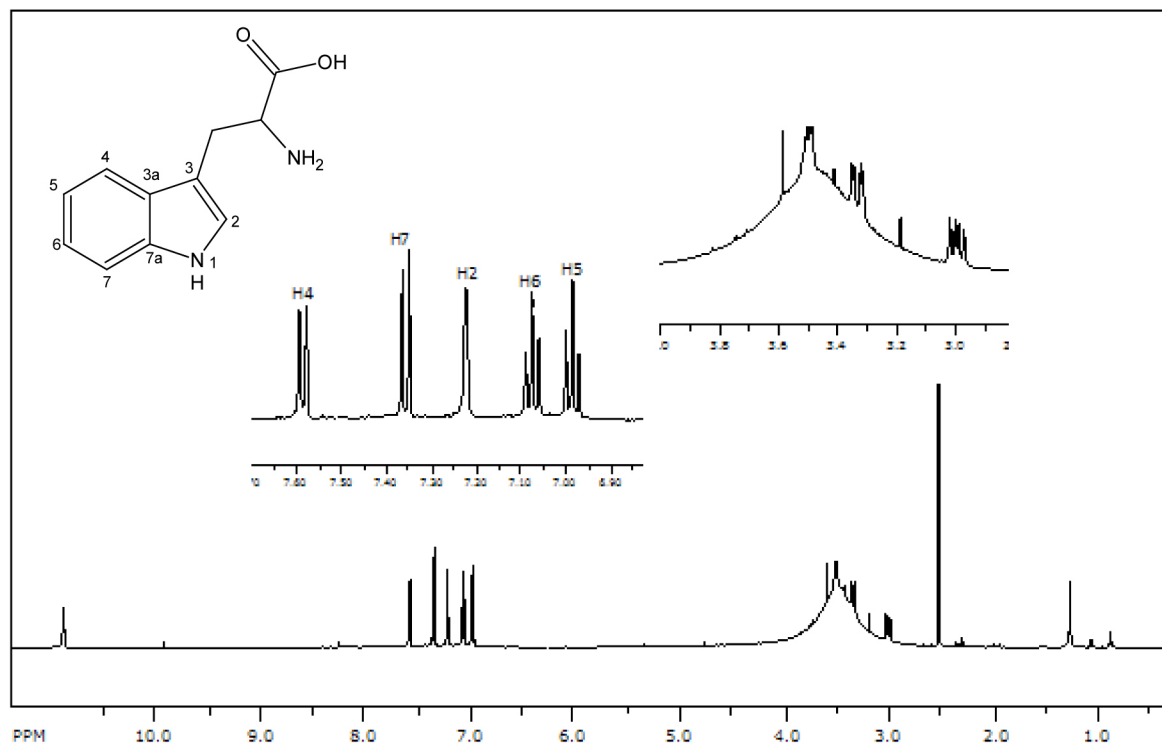


Figura 7S. Espectro de RMN de ^1H do triptofano (1)

Uracila (2): RMN ^1H [500 MHz, DMSO- d_6 , δ (ppm), J (Hz)]: 5,45 (d, J=7,5 Hz, H-5); 7,38 (d, J=7,5 Hz, H-6). RMN ^{13}C [125 MHz, DMSO- d_6 , δ (ppm)]: 151,6 (C-2); 164,4 (C-4); 100,2 (C-5); 142,3 (C-6). UV: 259 nm. IV (cm^{-1}): $\nu_{\text{N-H}}$ em 3370; $\nu_{\text{C=O}}$ em 1702; $\delta_{\text{N-H}}$ em 1578; $\nu_{\text{C-N}}$ em 1392.

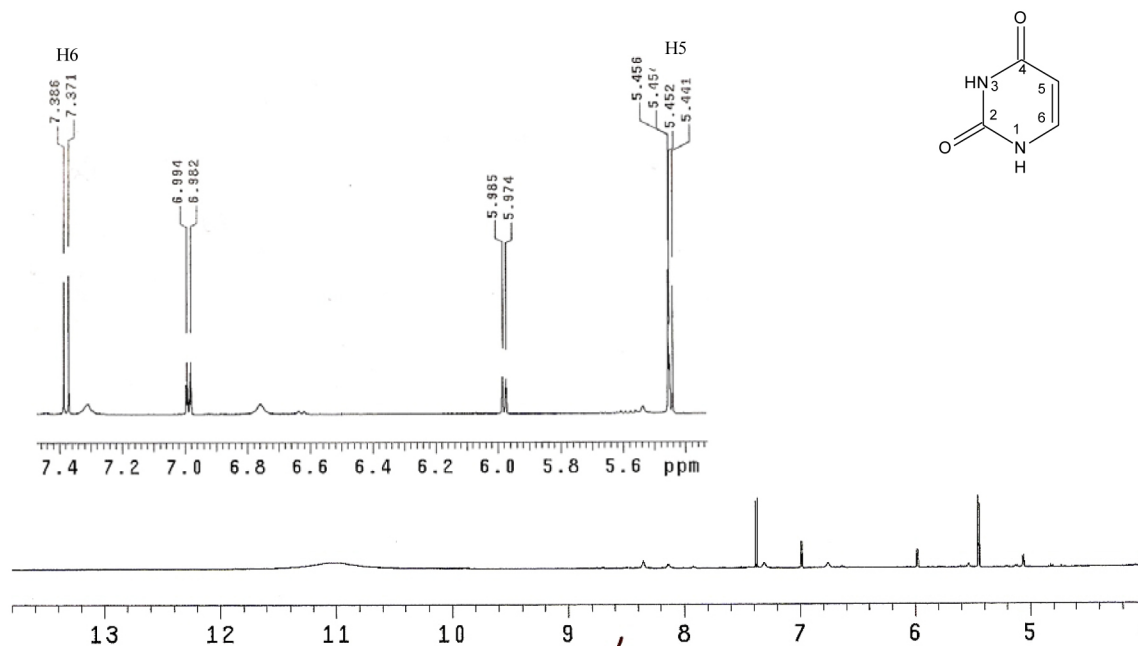


Figura 8S. Espectro de RMN de ^1H da uracila (2)

Rutina (3): RMN ^1H [500 MHz, DMSO- d_6 , δ (ppm), J (Hz)]: 6,20 (d, J=2,1 Hz, H-6); 6,40 (d, J=2,1 Hz, H-8); 7,53 (d, J=2,0 Hz, H-2'); 6,83 (d, J=9,0 Hz, H-5'); 7,75 (dd, J=7,8 e J=2,4 Hz, H-6'); 5,44 (d, J=7,2 Hz, H-1''); 4,38 (d, J=1,0 Hz, H-1'''); 0,99 (d, J=6,0 Hz, H-6'''). RMN ^{13}C [125 MHz, DMSO- d_6 , δ (ppm)]: 156,5 (C-2); 133,2 (C-3); 177,3 (C-4); 161,2 (C-5); 98,7 (C-6); 164,5 (C-7); 93,6 (C-8); 156,4 (C-9); 103,7 (C-10); 121,5 (C-1'); 116,2 (C-2'); 115,2 (C-3'); 148,5 (C-4'); 115,2 (C-5'); 121,0 (C-6'); 101,2 (C-1''); 75,8 (C-2''); 74,0 (C-3''); 69,9 (C-4''); 76,4 (C-5''); 66,9 (C-6''); 100,7 (C-1'''); 70,5 (C-2'''); 70,3 (C-3'''); 71,8 (C-4'''); 68,2 (C-5'''); 17,6 (C-6'''). UV: 258 nm e 354 nm. IR (cm^{-1}): $\nu_{\text{O-H}}$ em 3345, $\nu_{\text{C-H}}$ em 2920, $\nu_{\text{C=O}}$ em 1659, $\nu_{\text{C=C(aromático)}}$ em 1597 e em 1445, $\delta_{\text{O-H}}$ em 1358, $\nu_{\text{C-O}}$ em 1196 cm^{-1} .

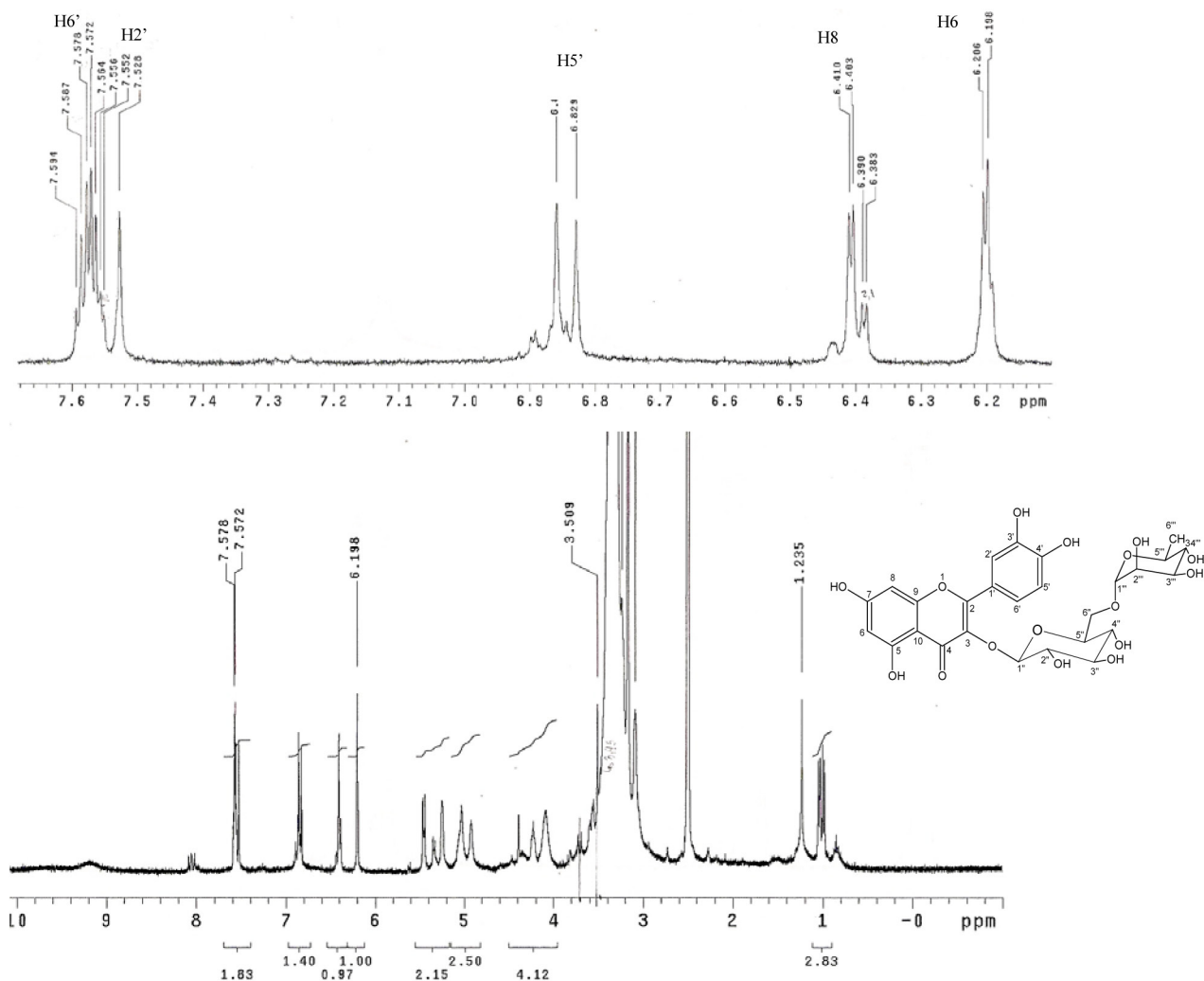


Figura 9S. Espectro de RMN de ^1H da rutina (3)

Kaempferol-3-O- β -D-glucopiranosido (**4**): RMN ^1H [500 MHz, DMSO- d_6 , δ (ppm), J (Hz)]: 6,20 (d, J=2,1 Hz, H-6); 6,43 (d, J=2,1 Hz, H-8); 7,97 (d, J=8,5 Hz, H-2'); 6,84 (d, J=8,5 Hz, H-3'); 6,84 (d, J=8,5 Hz, H-5'); 7,97 (d, J=8,5 Hz, H-6'); 5,28 (d, J=7,5 Hz, H-1''); 3,18 (H-2''); 3,20 (H-3''); 3,10 (H-4''); 3,13 (H-5''); 3,55; 3,35 (H-6''). RMN ^{13}C [125 MHz, DMSO- d_6 , δ (ppm)]: 156,2 (C-2); 133,2 (C-3); 177,4 (C-4); 161,2 (C-5); 98,6 (C-6); 164,1 (C-7); 93,6 (C-8); 156,4 (C-9); 104,0 (C-10); 120,8 (C-1'); 130,8 (C-2'); 115,1 (C-3'); 159,9 (C-4'); 115,1 (C-5'); 130,8 (C-6'); 100,8 (C-1''); 74,2 (C-2''); 76,4 (C-3''); 69,9 (C-4''); 77,4 (C-5''); 60,8 (C-6''). UV: 264 nm e 345 nm. IV (cm^{-1}): $\nu_{\text{O-H}}$ em 3242, $\nu_{\text{C=O}}$ em 1655, $\nu_{\text{C=C(aromático)}}$ em 1607 e em 1505, $\delta_{\text{O-H}}$ em 1354, $\nu_{\text{C-O}}$ em 1175, $\delta_{\text{C-H(aromático)}}$ em 840.

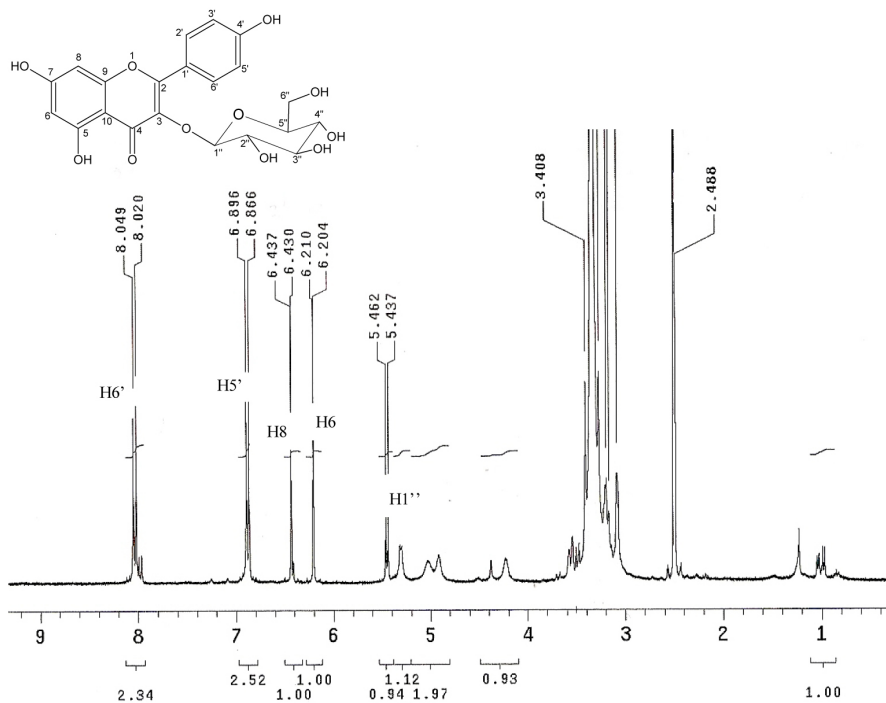


Figura 10S. Espectro de RMN de ^1H do kaempferol-3-O- β -D-glucopiranosido (**4**)

Ácido gálico (**5**): RMN ^1H [500 MHz, DMSO- d_6 , δ (ppm), J (Hz)]: 6,92 (s, H-2 e H-6). RMN ^{13}C [125 MHz, DMSO- d_6 , δ (ppm)]: 120,7 (C-1); 108,8 (C-2); 145,4 (C-3); 138,0 (C-4); 145,4 (C-5); 108,8 (C-6); 167,6 (C-1'). UV: 273 nm. IV (cm^{-1}): $\nu_{\text{O-H}}$ em 3274, $\nu_{\text{C=O(conjugado)}}$ em 1602, $\nu_{\text{C=C(aromático)}}$ em 1535.

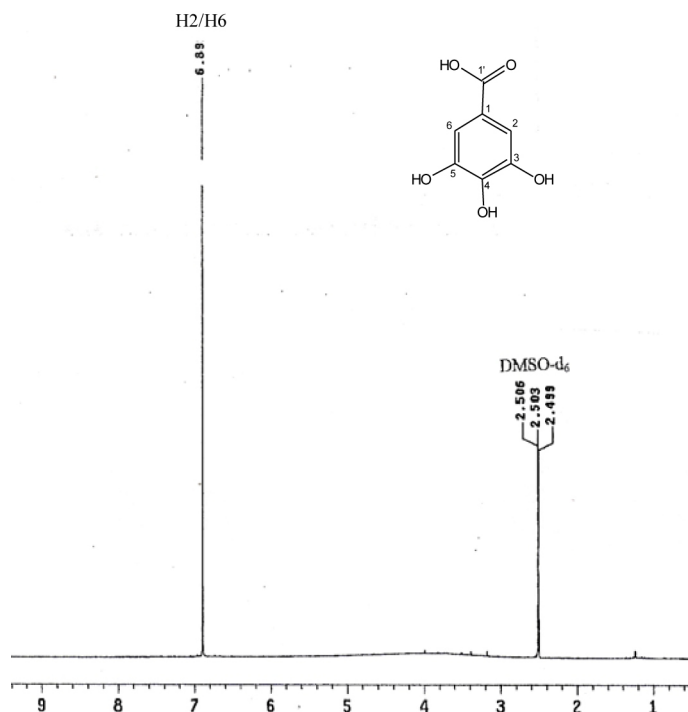


Figura 11S. Espectro de RMN de ^1H do ácido gálico (**5**)

Galato de metila (**6**): RMN ^1H [500 MHz, DMSO- d_6 , δ (ppm), J (Hz)]: 6,93 (s, H-2 e H-6); 3,74 (s, CH_3). RMN ^{13}C [125 MHz, DMSO- d_6 , δ (ppm)]: 119,7 (C-1); 108,8 (C-2); 145,5 (C-3); 138,0 (C-4); 145,5 (C-5); 108,8 (C-6); 165,6 (C-1'); 52,3 (OCH_3). UV: 274 nm. IV (cm^{-1}): $\nu_{\text{O-H}}$ em 3293, $\nu_{\text{C=O}}$ (conjugado) em 1674, $\nu_{\text{C=C}}$ (aromático) em 1540.

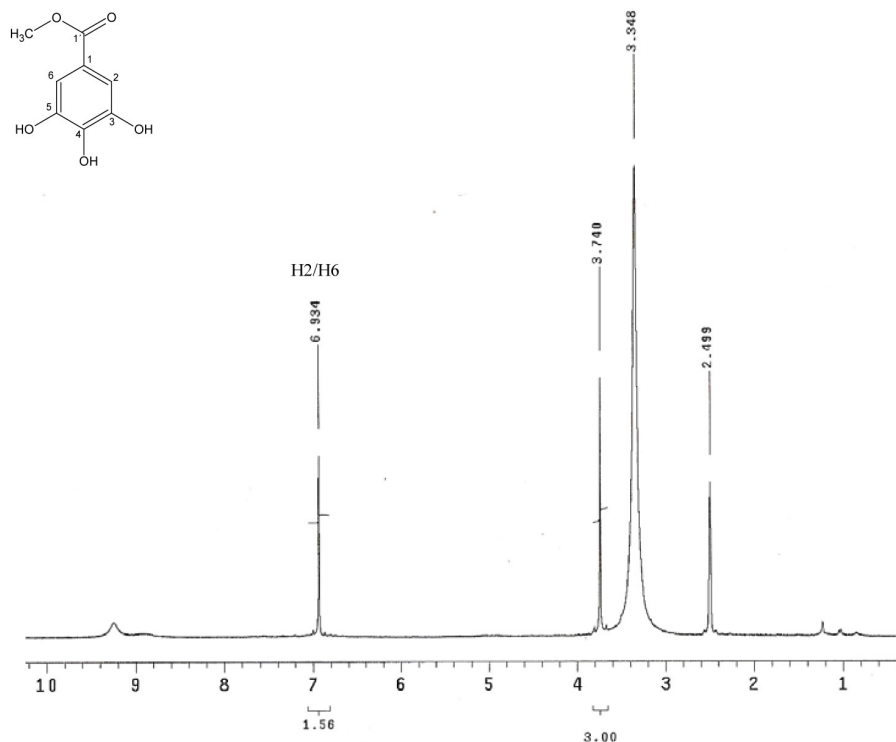


Figura 12S. Espectro de RMN de ^1H do galato de metila (**6**)

Kaempferol (**5**): RMN ^1H [500 MHz, DMSO- d_6 , δ (ppm), J (Hz)]: 6,20 (d, $J=2,0$ Hz, H-6); 6,44 (d, $J=2,0$ Hz, H-8); 8,05 (d, $J=8,5$ Hz, H-2'); 6, 94 (d, $J=8,5$ Hz, H-3'); 6,94 (d, $J=8,5$ Hz, H-5'); 8,05 (d, $J=8,5$ Hz, H-6'). RMN ^{13}C [125 MHz, DMSO- d_6 , δ (ppm)]: 147,0 (C-2); 99,0 (C-6); 164,2 (C-7); 94,0 (C-8); 156,2 (C-9); 103,0 (C-10); 122,0 (C-1'); 130,0 (C-2'); 115,0 (C-3'); 159,0 (C-4'); 115,0 (C-5'); 130,0 (C-6'). UV: 265 nm e 367 nm. IV (cm^{-1}): $\nu_{\text{O-H}}$ em 3317, $\nu_{\text{C=C}}$ (aromático) em 1607, $\delta_{\text{O-H}}$ em 1380 e $\nu_{\text{C=O}}$ em 1170.

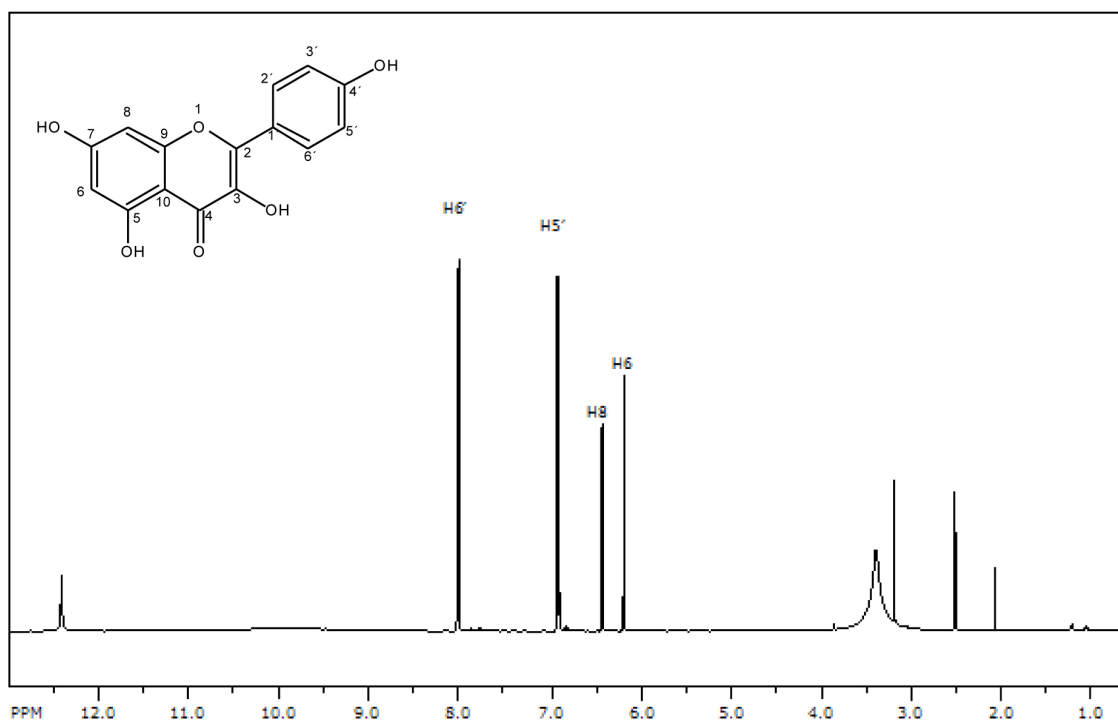


Figura 13S. Espectro de RMN de ^1H do kaempferol (**7**)

Ácido protocatecuico (**8**): RMN ^1H [500 MHz, DMSO-d_6 , δ (ppm), J (Hz)]: 7,32 (d, $J=1,5$ Hz, H-2); 6,76 (d, $J=8,5$ Hz, H-5); 7,27 (dd, $J=8,5$ e $J=1,5$ Hz, H-6). RMN ^{13}C [125 MHz, DMSO-d_6 , δ (ppm)]: 117,1 (C-2); 145,0 (C-3); 150,0 (C-4); 114,4 (C-5); 122,4 (C-6); 167,0 (C-1'). UV: 260 nm e 294 nm. IV (cm^{-1}): $\nu_{\text{C-H(aromático)}}$ em 3182, $\nu_{\text{C=O(conjugado)}}$ em 1673, $\nu_{\text{C=C(aromático)}}$ em 1597.

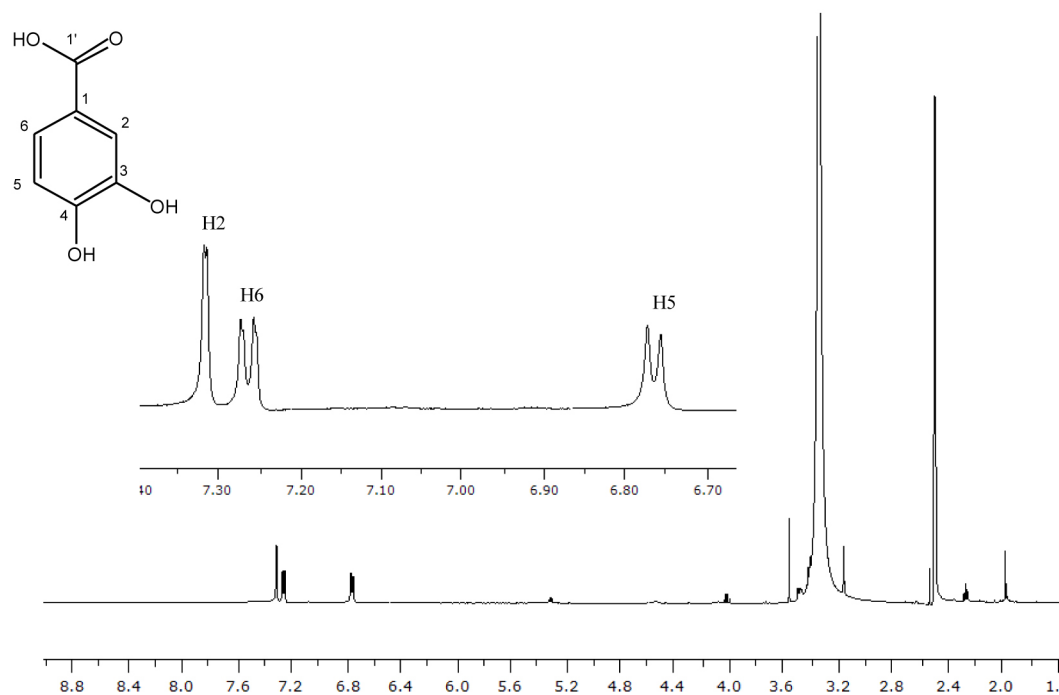


Figura 14S. Espectro de RMN de ^1H do ácido protocatecuico (**8**)