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## Figure S1. Fractionation scheme



Figure S2. Structure and NMR data of eckol



<sup>1</sup>H NMR (DMSO- $d_6$ , 600 MHz)  $\delta$  9.48 (1H, s, OH-9), 9.42 (1H, s, OH-4), 9.15 (2H, s, OH-2,7), 9.12 (2H, s, OH-3', -5'), 6.14 (1H, s, H-3), 5.96 (1H, d, J = 2.7 Hz, H-8), 5.80 (1H, t, J = 1.9 Hz, H-4'), 5.79 (1H, d, J = 2.7 Hz, H-6), 5.72 (2H, d, J = 2.1 Hz, H-2', -6'); <sup>13</sup>C NMR (150 MHz, DMSO- $d_6$ ) 123.1 (C-1), 145.9 (C-2), 98.1 (C-3), 141.8 (C-4), 122.2 (C-4a), 142.5 (5a), 93.7 (C-6), 152.9 (C-7), 98.4 (C-8), 146.0 (C-9), 122.6 (9a), 137.1 (10a), 160.3 (C-1'), 93.6 (C-2'6'), 158.7 (C-3'5'), 96.1 (C-4')



HPLC analysis of Eckol



## Figure S5. <sup>13</sup>C-NMR spectra of eckol



## Figure S6. <sup>1</sup>H-NMR spectra of eckol





## Figure S8. Proposed fragmentation pattern of the structures tentatively identified in EA fraction



Figure S9. (A) Extracted Ion Chromatograms (EIC) of EA fraction obtained by molecular features in Agilent Masshunter. [M-H]- (m/z): 1, 371; 2, 743; 3, 741; 4, 497; 5, 495; 6, 373; 7, 601; 8, 477. (B) Total ion chromatogram (TIC) and EIC of EA fraction. The x-axis represents retention time (min), and the y-axis represents signal intensity.

