

Spectrum

Eucaglobulin **1**, a light brown amorphous powder, gave a dark blue colour with FeCl₃, indicating that it had a galloyl group. The FAB-mass spectrum of **1** showed a peak at *m/z* 497 due to [M–H][–], which corresponded to the molecular formula C₂₃H₃₀O₁₂. The ¹HNMR spectrum exhibited one 2H-singlet at δ7.16 ascribe to one galloyl group, one 1H-triplet at δ6.95 due to one unsaturated proton, and glucose proton signals. The configuration at C-1' of the sugar was determined to be β by the coupling constant of the anomeric proton (J=7.7Hz). Additionally, some proton signals in the up-field region were also observed. The ¹³CNMR and DEPT spectra of **1** confirmed the presence of one galloyl group and a β-glucose moiety. Simultaneously, they also revealed that ten carbon signals ascribe to a monoterpene moiety, which include one carbonyl group, two unsaturated carbon atoms, two methyls, three methylene groups, one methine and one quaternary carbon. The assignment of protons and carbons was achieved on the basis of ¹H-¹H COSY and HMQC spectra. According to the results of HMBC spectrum, the position of the galloyl group on the glucose was determined to be at C-1', and the monoterpene moiety was at C-6' through an ester bond (**Figure 1**). Thus, the structure of eucaglobulin was identified as **1**, which is the first example of a gallotannin possessing a monoterpene moiety.

Table1. ¹³CNMR data of Eucaglobulin (400MHz, acetone-d₆)

Carbon	δ (ppm)	Carbon	δ (ppm)
Monoterpene		Glucose	
1	167.6 (s)	1'	95.6 (d)
2	130.8 (s)	2'	73.7 (d)
3	141.0 (d)	3'	77.6 (d)
4	28.1 (t)	4'	70.9 (d)
5	45.0 (d)	5'	75.8 (d)
6	24.1 (t)	6'	64.1 (t)
7	26.0 (t)	Galloyl	
8	71.7 (s)	CO	165.7 (s)
9	27.3 (q)	1''	120.8 (s)
10	26.8 (q)	2'', 6''	110.2 (d)
		3'', 5''	146.1 (s)
		4''	139.4 (s)

Eucaglobulin **1**: C₂₃H₃₀O₁₂, light brown amorphous powder. FABMS *m/z*: 497[(M–1)[–], base peak]. ¹HNMR (acetone-d₆, 400MHz): δ7.16 (2H, s, H-2'', 6''), 6.95 (1H, br. s, H-3), 5.66 (1H, d, J=7.7 Hz, H-1'), 4.42 (1H, dd, J=2.4, 11.6 Hz, H-6'), 4.23 (1H, dd, J=5.6, 11.6 Hz, H-6'' Glc), 3.70 (1H, ddd, J=2.4, 5.6, 9.7 Hz, H-5'), 3.58-3.45 (3H, m, H-2', 3', 4'), 2.42 (2H, br. d, J=15.6Hz, 2×H-7), 2.31 (2H, br. d, J=18.8 Hz, 2×H-4), 1.99 (2H, m, 2×H-6), 1.49 (1H, m, H-5), 1.13 (6H, s, 2×CH₃)

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References

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