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SUPPLEMENTARY DATA

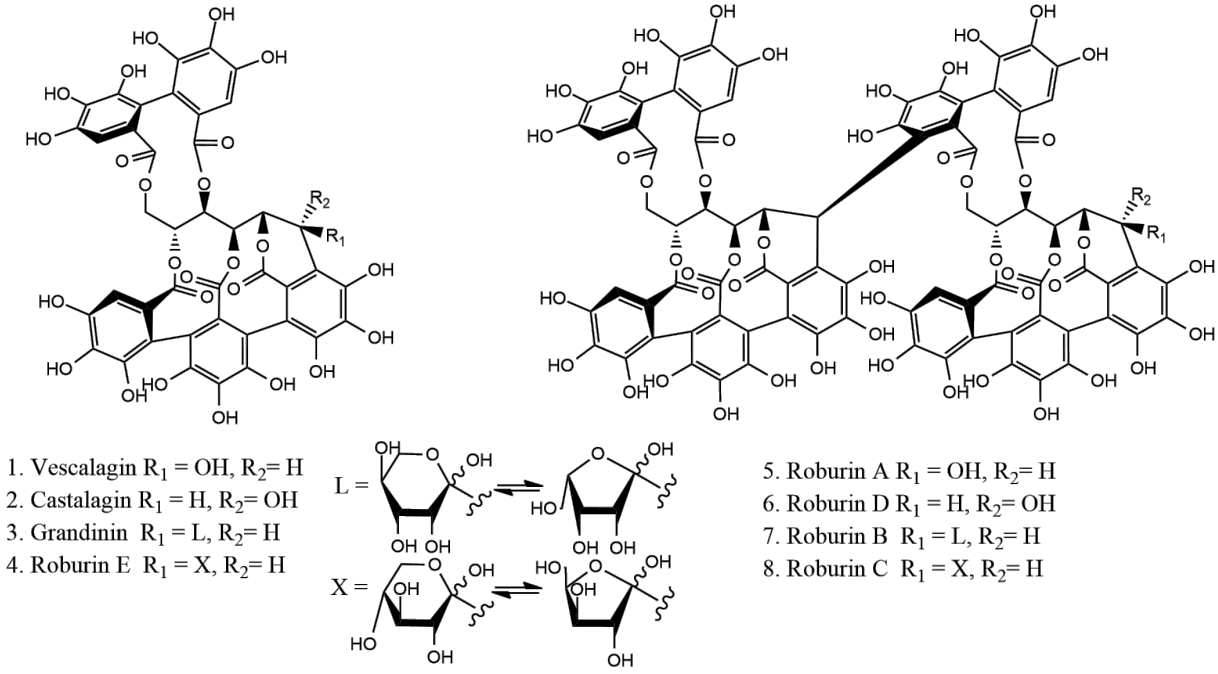


Figure S1. Structures of the native *C*-glucosidic ellagitannins.

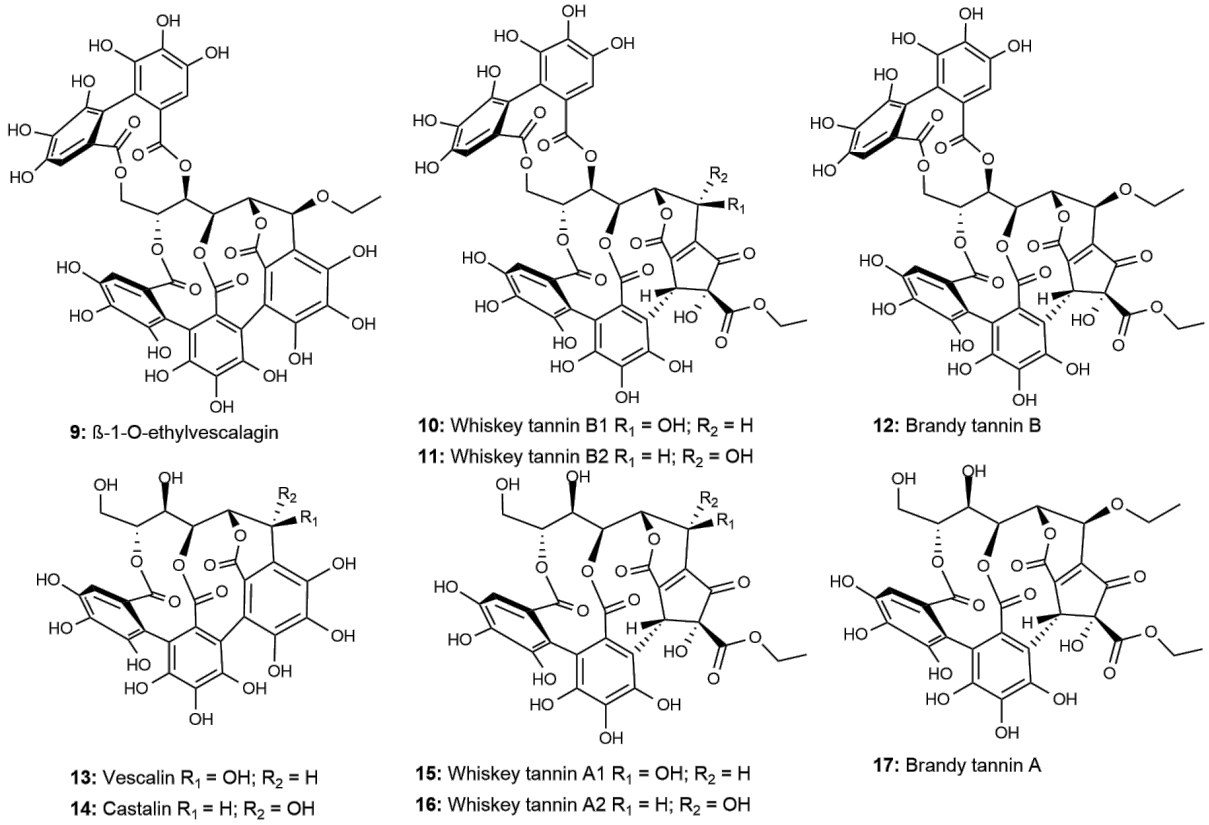


Figure S2. Structures of ellagitannin derivatives found in different matrices.

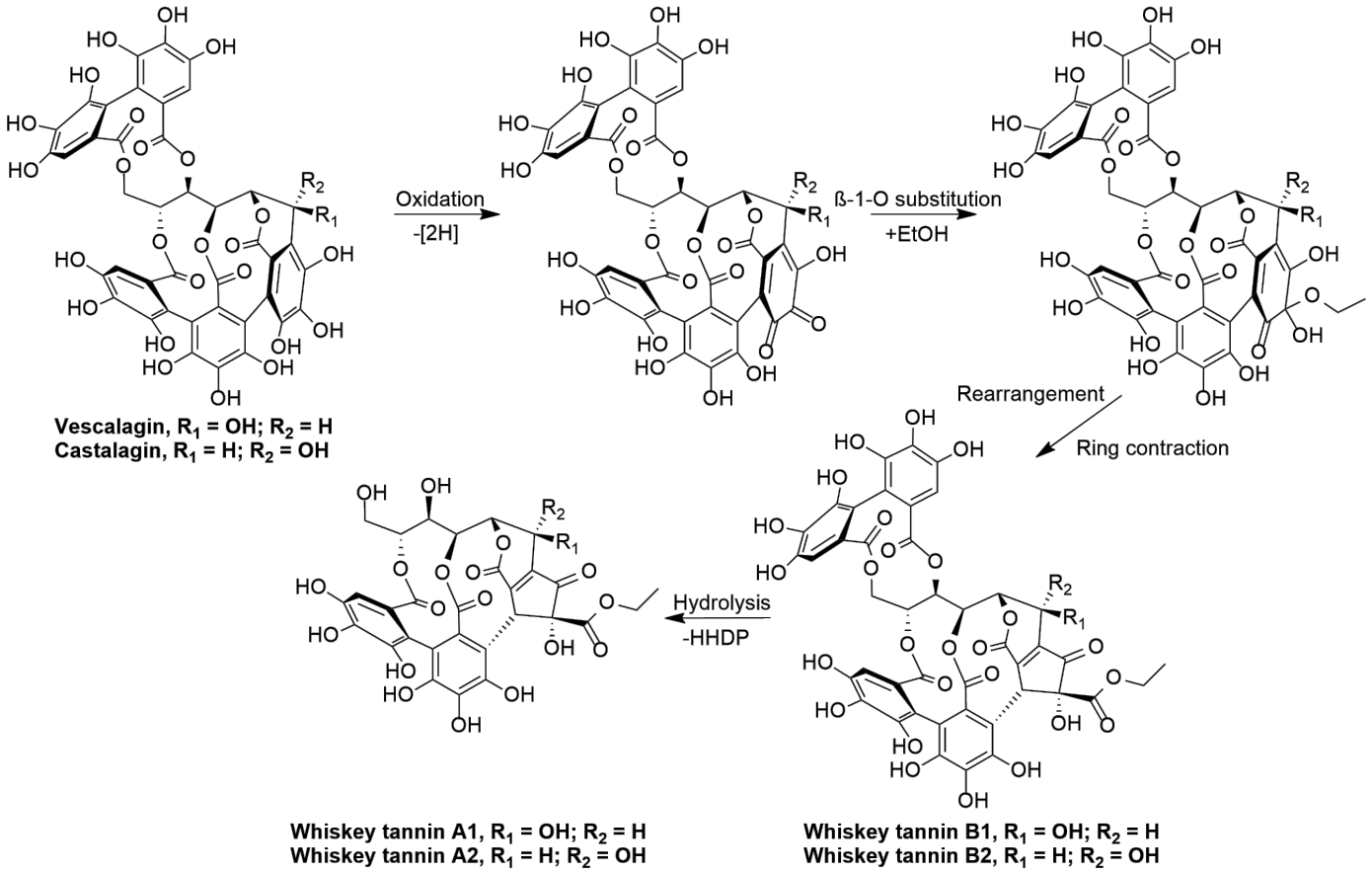


Figure S3. Structures and formation mechanisms of whiskey tannins B1/B2 and whiskey tannins A1/A2.

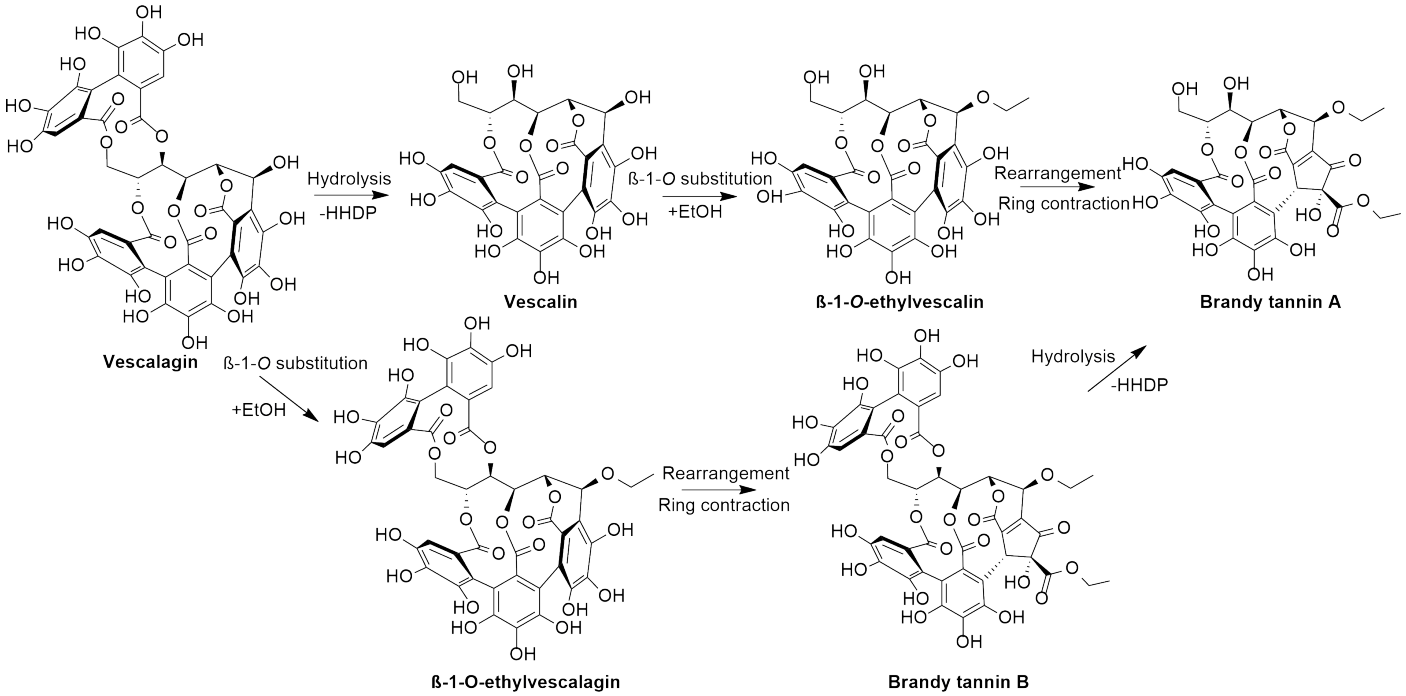


Figure S4. Structures and formation mechanisms of brandy tannins A and B from vescalagin.

Table S1. HPLC retention times in minutes, molecular weight and mass fragmentation patterns of the reference compounds (1–17).

Compounds	Retention time (min)	m/z molecular weight	MS/MS fragment ions
Vescalagin **(1)**	13,9	933	915,613,301
Castalagin **(2)**	20,4	933	915,613,302
Grandinin **(3)**	9,5	1065	915,613,303
Roburin E **(4)**	12,0	1065	915,613,304
Roburin A **(5)**	5,9	1849	933,924,915,301
Roburin D **(6)**	9,3	1849	933,924,915,302
Roburin B **(7)**	5,5	1981	933,924,915,303
Roburin C **(8)**	5,9	1981	933,924,915,304
ß-1-O-Ethylvescalagin **(9)**	19,3	961	915,480,301
Whiskey tannin B1 **(10)**	27,1	977	933,675,631,301
Whiskey tannin B2 **(11)**	28,0	977	933,675,631,302
Brandy tannin B **(12)**	29,7	1005	703,301
Vescalin **(13)**	2,0	631	-
Castalin **(14)**	2,9	631	-
Whiskey tannin A1 **(15)**	9,8	675	-
Whiskey tannin A2 **(16)**	16,6	675	-
Brandy tannin A **(17)**	28,8	703	-

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Table S2. Statistical analysis of the evolution of C-glucosidic ellagitannins and their derivatives in two different toasting barrels (LT+AAS and MT+AAS). * Represent significant differences within each compound: * $p < 0.05$; ** $p < 0.03$; grey cell = no significant differences, nd = not detected, WT B1= Whiskey Tannin B1, WT B2= Whiskey Tannin B2, WT A1= Whiskey Tannin A1, WT A2= Whiskey Tannin A2, BT A = Brandy Tannin A. Letters a-g show significant differences among the distinct time point for each ellagitannin ($p < 0.05$).

		LT+												MT+											
		M1	M2	M3	M4	M5	M6	M7	M9	M10	M11	M12		M1	M2	M3	M4	M5	M6	M7	M9	M10	M11	M12	
Vescalagin	groups	f	g	f	e	de	b	a	bc	cd	bc	de		b	d	bc	bc	b	ab	a	bc	c	c	c	
	significance level						**												*						
Castalagin	groups	g	f	e	d	c	b	a	a	b	ab	b		g	f	ef	e	d	ab	a	ab	c	c	bc	
	significance level						**												**						
Roburin A	groups	f	e	de	cd	bc	ab	ab	ab	ab	ab	ab													
	significance level						*																		
Roburin D	groups	c	c	c	c	bc	a	a	a	a	ab	a		c	bc	b	b	b	b	a	b	ab	a	a	
	significance level						*												*						
Grandinin	groups	f	ef	ef	d	d	ab	ab	bc	cd	cd	cd													
	significance level						**																		
Roburin E	groups	e	d	cd	cd	cd	ab	ab	ab	ab	ab	b		cd	d	cd	bc	c	a	a	b	b	ab	b	
	significance level						*												*						
Roburin B	groups																								
Roburin C	groups													e	cd	de	e	c	ab	a	a	bc	b	ab	
	significance level																		*						
EthylVesca	groups																								
WT B1	groups																								
	significance level																		nd						
WT B2	groups	d	c	bc	ab	a	a	a	ab	ab	a	a													
	significance level						*												nd						
WT A1	groups	f	e	de	cd	bc	ab	bc	a	a	b	a													
	significance level						**																		
WT A2	groups																								
Vescalin	groups																								
Castalin	groups																								
BT A	groups																								
	significance level						nd																		

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Table S3. Concentrations of ellagitannin classes over a year in light-plus and medium-plus toasting barrels (LT+AAS/MT+AAS). nd = not detected, concentrations are expressed as vescalagin equivalents (mg/L).

Month	Toasting type	Monomers	Dimers	Substitution products	Oxidative products	Hydrolysis products
M1	LT+AAS	31,10 ± 0,05	7,91 ± 0,05	1,21 ± 0,05	1,76 ± 0,05	1,44 ± 0,05
	MT+AAS	35,96 ± 0,05	11,61 ± 0,05	1,45 ± 0,05	nd	0,73 ± 0,05
M2	LT+AAS	42,20 ± 2,70	10,43 ± 0,14	2,47 ± 0,25	7,89 ± 1,03	4,77 ± 2,21
	MT+AAS	50,76 ± 1,90	12,98 ± 0,87	2,42 ± 0,24	nd	5,83 ± 0,86
M3	LT+AAS	62,22 ± 1,27	12,98 ± 0,97	3,05 ± 0,31	9,99 ± 1,10	5,19 ± 1,10
	MT+AAS	59,55 ± 4,43	13,35 ± 0,44	2,95 ± 0,46	nd	5,22 ± 0,48
M4	LT+AAS	91,64 ± 3,81	15,02 ± 1,74	3,77 ± 0,83	12,92 ± 1,67	13,49 ± 1,29
	MT+AAS	65,42 ± 1,72	14,47 ± 0,89	3,02 ± 0,26	nd	6,30 ± 0,96
M5	LT+AAS	108,36 ± 5,47	18,31 ± 1,14	3,69 ± 0,22	13,80 ± 1,68	14,07 ± 1,51
	MT+AAS	81,33 ± 5,51	15,88 ± 1,80	2,99 ± 0,27	nd	7,64 ± 0,69
M6	LT+AAS	144,91 ± 2,12	27,90 ± 1,90	3,67 ± 0,42	15,58 ± 0,76	21,72 ± 1,38
	MT+AAS	102,64 ± 4,09	17,50 ± 1,24	3,67 ± 0,20	nd	15,76 ± 1,19
M7	LT+AAS	159,46 ± 3,37	31,05 ± 2,68	3,86 ± 0,40	14,75 ± 1,03	22,12 ± 1,71
	MT+AAS	109,63 ± 2,73	21,25 ± 1,22	4,13 ± 0,29	nd	16,51 ± 1,81
M9	LT+AAS	153,56 ± 5,54	28,17 ± 1,84	3,91 ± 0,27	13,52 ± 1,15	20,42 ± 1,93
	MT+AAS	100,00 ± 3,66	17,62 ± 2,47	3,69 ± 0,50	nd	17,90 ± 2,21
M10	LT+AAS	140,14 ± 4,60	30,04 ± 4,53	3,37 ± 0,46	14,21 ± 0,88	19,53 ± 1,10
	MT+AAS	87,83 ± 4,50	19,32 ± 2,05	3,42 ± 0,22	nd	16,39 ± 2,14
M11	LT+AAS	143,54 ± 3,07	25,26 ± 0,98	5,67 ± 0,28	14,28 ± 0,30	22,89 ± 1,71
	MT+AAS	89,72 ± 4,38	20,03 ± 1,59	3,49 ± 0,47	nd	16,97 ± 1,70
M12	LT+AAS	134,30 ± 2,90	28,76 ± 4,87	3,47 ± 0,04	14,88 ± 1,00	22,53 ± 1,04
	MT+AAS	90,17 ± 0,99	21,01 ± 1,60	3,57 ± 0,27	nd	18,77 ± 2,52