

Supplementary Table S1. List of genotypes included in this study

Cultivar name	Plant species	Fruit type	Harvesting period (Days after Full Bloom)	TSS (°Brix)	TA (%)
Wase	<i>Citrus unshiu</i> cv. Wase	Mandarin	20, 80, 110, 200	13.80	0.94
Thomson	<i>Citrus sinensis</i> cv. Thomson navel	Navel orange	20, 80, 110, 200	11.80	1.52
Duncan	<i>Citrus paradisi</i> cv. Duncan	Grapefruit	20, 80, 110, 200	11.10	2.78
Moro	<i>Citrus sinensis</i> cv. Moro	Blood orange	20, 80, 110, 200	11.20	1.45
Aurantium	<i>Citrus aurantium</i> L.	Sour orange	20, 80, 110, 200	9.70	5.60
Palestine	<i>Citrus limettioides</i> cv. Palestine	Lime	20, 80, 110, 200	9.80	0.11
Bakraei	<i>Citrus reticulata</i> Blanco × <i>Citrus limettioides</i> Tan.	Mandarin	20, 80, 110, 200	7.80	0.07
Page	(<i>Citrus paradisi</i> cv. Duncan × <i>Citrus reticulata</i> cv. Dancy) × <i>Citrus clementina</i>	Mandarin	20, 80, 110, 200	12.80	1.47

Supplementary Table S2. Identification of metabolites as extracted from PLS-DA. VIP: Variable Importance; ID level: a) co-injection with standard, b) mass spectrometry data matches those in databases (metlin or HMDB), c) tentatively annotated based on mass spectrometry data and database search (See Material and Methods section for details). (*) p-value after one-way ANOVA using genotype as factor.

Annotation	Positive electrospray (ESI+)					Negative electrospray (ESI-)					ID level	<i>p-value*</i>
	mz precursor	MS/MS	Rt [min]	Fragment annotation	VIP	mz	MS/MS	Rt [min]	Fragment annotation	VIP		
Naringenin	273.0700	153.0300	9.03	[M-C8H8O]+	-	271.0624	151.0055 119.0604	9.03	[M-C8H7O]- [M-C7H3O4]-	1.33	a	<0.001
Myricetin	319.054	153.041	7.30	[M-C4H6O7]+	1.20	317.0342	179.99 151.0136 137.0183 109.0171	7.27	[M-C6H7O4]- [M-C2H4]- [M-O]- [M-CHO2]-	1.44	a	<0.001
Nobiletin	403.1396	388.1245 358.0729 330.0801 301.0784 211.043 183.049	10.96	[M-CH3]+ [M-C2H6]+ [M-CO]+ - - -	1.34	nd					a	<0.001
Diosmin	609.1805	303.0976 177.0500 153.0200	7.72	[M-Hexose]+ [M-C6H3O3]+ [M-C9H10O2]+	1.03	607.1660		6.89	[M-H]-	0.95	a	<0.001
Isosinensetin	373.1289	343.1197	9.75	[M-C2H6]+	-	nd					c	<0.001
Sinensetin	373.1290	343.1199	10.39	[M-C2H6]+	1.16	nd					c	<0.001
Tangeretin	373.1291	343.1199	11.60	[M-C2H6]+	1.42	nd					c	<0.001

Eriodictyol rutinoside #1	451.1247	289.0738	5.35	[M-Hexose]-	1.15	595.1615	5.39	[M-H]-	1.01	c	<0.001	
						287.0569	5.33	[M- rutinoside]+				
Eriodictyol rutinoside #2	597.1788	289.0732	6.14	[M- rutinoside]+	0.97	595.1662	287.0574	6.17	[M- rutinoside]-	0.97	c	<0.001
Isorhamnetin-3- O-rutinoside	625.1758	479.1195	6.76	[M- Deoxyhexosie] +	1.50	623.1614		6.73	[M-H]-	1.36	b	<0.001
		317.0669		[M-Hexose]+	1.46	315.0519		6.74	[M- rutinoside]-	1.23		
Isorhamnetin rutinoside hexoside	787.2267	317.0568	5.40	[M- rutinoside]+	1.14	785.2158		5.36	[M-H]-	1.25	b	<0.001
		479.1185		[M-hexoside]+	1.29							
Isorhamnetin rutinoside deoxyhexoside	771.2325	317.0663	6.02	[M-rutinoside- deoxyhexoside] +	1.40	769.2181		6.06	[M-H]-	1.33	b	<0.001
Kaempferol diDeoxyhexoside hexoside	741.2224	287.0559	5.98	[M- Deoxyhexosid e-Hexoside] +	1.47	739.2099		6.07	[M-H]-	1.32	b	<0.001
		595.1649		[M- Deoxyhexosid e] +								
Kaempferol Deoxyhexoside hexoside	595.1647	287.0565	6.63	[M- Deoxyhexosid e-Hexoside] +	1.09	593.1558	285.0419	6.64	[M-Hexoside- Deoxyhexosid e]-	1.30	b	<0.001

		449.1100	6.64	[M-deoxyhexoside] +]	1.20							
Kaempferol Caffeoyl Hexoside Deoxyhexoside	757.2167	611.1477	5.34	[M-Deoxyhexose] +	1.38	755.2044	641.1742	5.33	-	1.31	b	<0.001
		449.1133 287.0644		[M-Caffeoyl] [M-Hexose]	+							
Kaempferol hidoxymethyl glutaryl (HMG)- glucoside tentative	287.0565		7.13	Kaempferol moiety	1.15	591.1360	161.0583	7.08	hydroxymeth yl glutarate moiety	0.92	c	<0.001
Quercetin hexoside rutinoside	773.2128	465.1050	4.99	[M-Rutinoside] +	1.31	771.2002		4.99	[M-H]-	1.28	b	<0.001
		303.0500		[M-Hexose] +								
Poncirin	595.2021	577.1910	7.91	[M-H ₂ O] +	1.12	593.1874	285.0776	7.91	[M-rutinoside]-	0.97	b	<0.001
		449.1449		[M-Deoxyhexose] +								
		433.1497 287.0922		[M-Hexose] [M-Hexose]	+							
Narirutin	581.1857	435.1293	5.51	[M-Deoxyhexose] +	1.12	579.1449		5.77	[M-H]-	1.31	a, b	<0.001
		273.0766		[M-Hexose] +								
Naringin	581.1864		6.64	[M+H] +	1.07	579.1711		6.65	[M-H]-	0.93	a	<0.001

		435.1291		6.63	[M-Deoxyhexose] +	1.15								
		273.0768		6.67	[M-Hexose] +	1.00								
Deacetyl Nomilinic glycoside tentative	acid	693.2733	491.2279	6.50	[M-Hexose] +	0.98	669.2754	6.53	[M-H] -	0.96	c	<0.001		
Deacetyl glycoside	Nomilin	675.2619	473.2176	6.96	[M-Hexose] +	1.01	651.2673	6.96	[M-H] -	0.96	c	<0.001		
Nomilin		515.2286	487.2336	11.14	[M-CO] +	0.94	513.2118	11.14	[M-H] -	1.03	b	<0.001		
Nomilinic glycoside	acid	712.3161	455.2066	7.39	[M-CH ₃ COOH] +	1.23	711.2874	7.36	[M-H] -	1.02	b	<0.001		
			515.2279		[M-CH ₃] +									
			533.2380		[M-Hexose] +									
Nomilin lactone	A-ring	533.2381		10.53		1.04	531.2236	515.1915	10.53	[M-CH ₃] -	1.03	b	<0.001	
								489.2155		[M-CH ₃ CO] -	1.11			
								469.1865		[M-CH ₂ O ₂] -	1.07			
								427.2129		[M-CH ₂ O ₃] -	1.16			
								411.1453		[M-CH ₂ O ₄] -	1.03			
Nomilin glycoside tentative		735.2791	533.2380	7.35	[M-Hexose]	1.07	711.2874	7.36	[M-H] -	1.02	c	<0.001		
			515.2279	7.37	[M-Hexose] - CO] +	1.15								
			488.2377	7.39	-	1.21								
			469.2226	7.37	[M-CH ₂ O ₂] +	1.19								
			457.2096	7.39	-	1.18								

		455.2066	7.40	[M-C2H4O2]+	1.09
		427.2121	7.39	[M-CO]+	1.23
		419.1991	7.39	-	1.18
Obacunone	455.2076	-	11.14	[M+H]+	0.91
Obacunoic acid	473.2170	429.2274	10.26	[M-CO]+	1.50
Obacunone glycoside	455.208		7.71	[M-Hexose]+	1.12
Ichangin	489.2120		9.65	[M+H]+	1.38
Limonoate A-ring lactone tentative	489.2120		9.81	-	
Limonin	471.2018	381.2074	10.56	[M+H-CO2]+	1.24
		383.1955		[M+H-COCH2]+	
		397.2022		[M+H-CO]+	
		425.1964		[M+H-HCOOH]+	
		443.2070		[M+H-CO]+	
Limonin 17 beta D glucopyranoside	674.2515	453.1923	6.38	[M+H-H2O]+	
		425.1964		[M+H-HCOOH]+	1.10
		443.2072		[M+H-CO]+	1.19
		453.1916		[M+H-H2O]+	1.10
Tryptophan	205.0988	471.2014	4.18	[M-Hexose]	1.10
		144.0837		[M+H-CO2]+	1.13

nd					c	<0.001
nd					b	<0.001
633.2555	499.1799	7.73	[M-Hexose+HCOOH]-	1.41	c	<0.001
	517.2292		[M-Hexose fragment]-			
487.2120	427.1880	9.61	[M-C2H4O2]-	1.21	c	<0.001
487.2120	427.1880	9.81	[M-C2H4O2]-	-	c	<0.001
	383.1847		-			
469.1935		10.53		-	a	<0.001
649.2501	605.2612	6.38	[M-CO2]-	1.03	a, b	<0.001
203.0840	142.0670	4.16	[M-NH3]-	1.12	b	<0.001

		146.0618		[M+H-COCH ₂] ⁺		116.0523	4.17	-				
		170.0605		[M+H-H ₂ O] ⁺		159.0943	4.16	[M-CO ₂] ⁻				
		188.0718		[M-NH ₃] ⁺								
Phenylalanine	166.0869	103.0539	2.30	[M-NH ₃] ⁺ [M-HCO ₂] ⁺	1.34	164.0712	2.33			0.93	b	<0.001
Absciscic acid	247.1348	201.1286 205.1240 219.1395 229.1249	8.18	[M-HCOOH] ⁺ [M-COCH ₂] ⁺ [M-CO] ⁺ [M-H ₂ O] ⁺	1.07	263.1303	8.35		0.95	a		<0.001
Absciscic acid glycosyl ester	427.1023	201.1285 205.1228 219.1416 229.1251 247.1345	6.60	[M-HCOOH] ⁺ [M-COCH ₂] ⁺ [M-CO] ⁺ [M-H ₂ O] ⁺ [M-Hexose-H ₂ O] ⁺	1.46	425.1821	285.0419	6.66	[M-Hexose+Na] ⁻	1.07	a	<0.001
12-oxophytodienoic acid	293.2105	275.2005	13.05	[M-H ₂ O] ⁺	1.12	291.2331	247.1989	13.05	[M-HCO ₂] ⁻	0.90	a	<0.001
Dihydroconiferyl alcohol	183.1031	147.0462 165.0560	6.35	[M-2xH ₂ O] ⁺ [M-H ₂ O] ⁺	1.42	165.1235	181.0878	119.0517	6.35	[M-C ₂ H ₆ O ₂] ⁻ [M-CO ₂] ⁻ [M-H ₂ O] ⁻	1.32	<0.001
caffeic acid	181.0599	163.0495	5.40	[M-H ₂ O] ⁺	1.16	179.0379	135.0490	5.47	[M-CO ₂] ⁻	1.35	a	<0.001
sinapinic acid	225.0763	119.0526 145.0329	6.65	[M-C ₂ H ₂] ⁺ [M-CH ₂ O] ⁺	1.26	223.0626	208.047	6.71	[M-CH ₃] ⁻ [M-CO ₂] ⁻	1.22	a	<0.001
							179.066					

		175.0412		[M-CH4O]+	
		207.0668		[M-H2O]+	
chlorogenic acid	355.1035	163.0417	5.08	[M-C7H12O6]+	1.03
Glucosyl salicylate	nd				

	164.051		[M-CH3]-			
353.0883	191.0213	4.96	[M-Hexose]-	1.32	a	<0.001
299.0776	137.0264	3.88	[M-Hexose]+	0.98	a, b	<0.001