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Supplemental information

Combined –omics framework reveals how ant symbionts benefit the Neotropical ant-plant *Tococa quadrialata* at different levels

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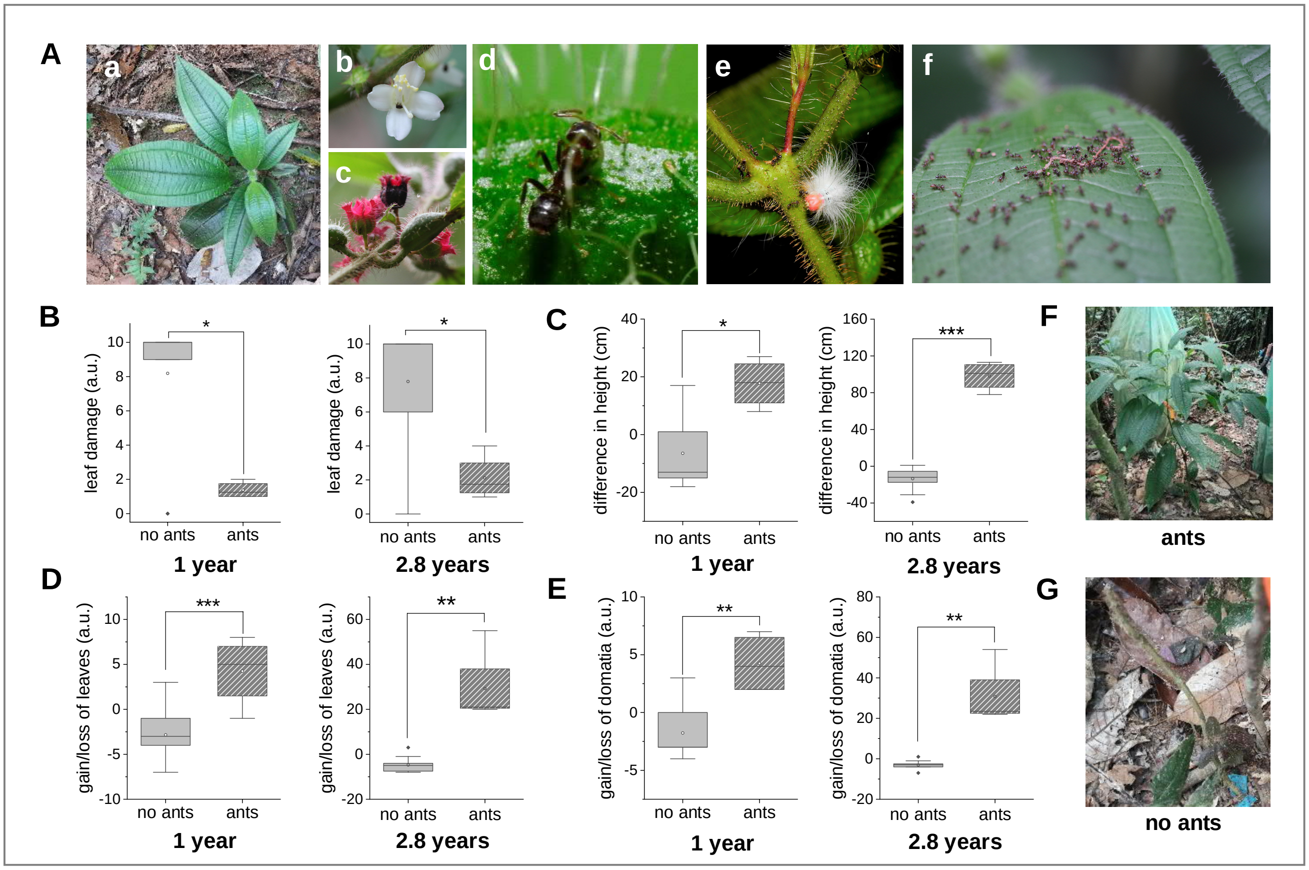

Figure S1: **Tococa plants suffer from herbivory and reduced growth in the absence of their mutualistic ants.** Related to Figure 1. Data were taken from a naturally occurring population of *Tococa quadrialata* plants in the Tambopata national reserve (Madre de Dios, Peru). Growth and performance were monitored for 2.8 years. Boxplots (25^{th} percentile, median, mean (open circle) and 75^{th} percentile) show plant growth parameters. **A:** The symbiosis between *Azteca* cf. *tonduzi* ants and *T. quadrialata*: a typical example of a young *T. quadrialata* plant (a) is shown and its flowers (b), and fruit development (c). *A.* cf. *tonduzi* workers shown patrolling the leaf (d); they attack anything placed on the plant, including invertebrates or inanimate objects, whether or not they feed on the plant (e, f; see SI File 1), and often eat the intruder (f). **B**: The level of naturally occurring herbivore damage was higher on uncolonized (no ants) compared to ant-inhabited (ants) plants (*: $p<0.05$, Wilcoxon rank sum test, n=4-13) at both examined timepoints. Ant-colonized plants grew taller (**C**) and had an increased number of leaves (**D**), and domatia (**E**) in comparison to uncolonized plants. Only ant-colonized plants exhibited net growth (*: $p<0.05$; **: $p<0.01$; ***: $p<0.001$, Wilcoxon rank sum test or Student's t-test, n=4-13). Example of ant-colonized (**F**) and uncolonized (**G**) plants after 2.6 yrs of monitoring. Both plants were of similar size when the monitoring started (1F: 10 cm tall, 4 leaves, 2 domatia; 1G: 8 cm tall, 8 leaves, 4 domatia), but while the ant-colonized plant gained 55 leaves over the 2.8 years, the uncolonized plant suffered through cycles of leaf gain, leaf loss, and regrowth (pers. observation). a.u.: arbitrary units.

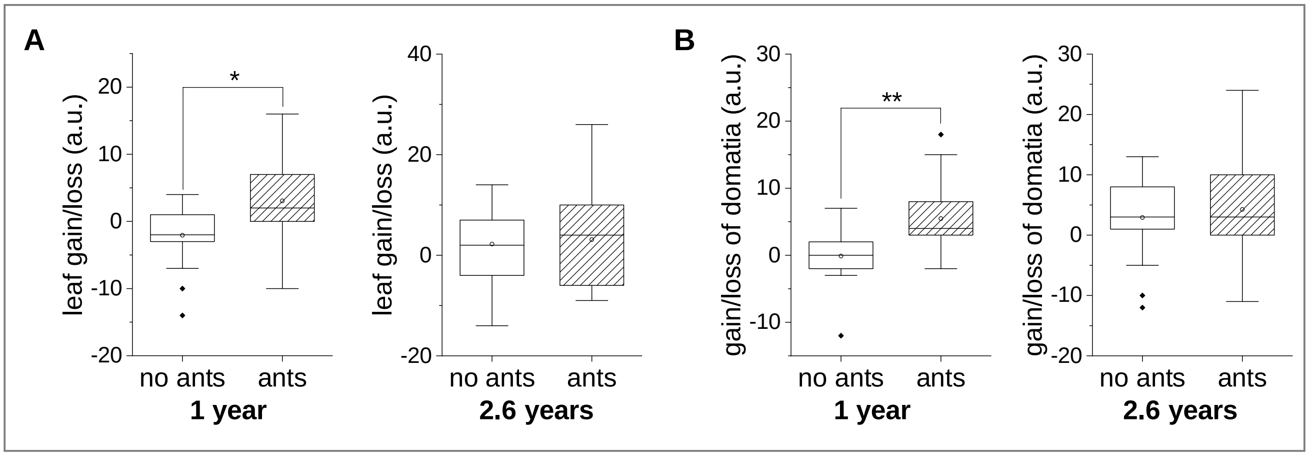

Figure S2: Growth of *T. quadrialata* plants protected from herbivory by nets. Related to Figure 1. Experimental design: A set of young *T. quadrialata* plants colonized by *Azteca* cf. *tonduzi* ants was split into two groups, and ants were removed from one group. Herbivore damage and ant recolonization were prevented by covering all plants with nets. The production and loss of leaves and formation of domatia by colonized (ant) and ant-deprived (no ants) plants were monitored for 2.6 years. It was accounted for leaves that were removed due to experiments. Leaf (**A**) and domatia (**B**) gain after 1 and 2.6 years of growth is presented as boxplots (25^{th} percentile, median, mean (open circle) and 75^{th} percentile) and was calculated by comparison to the respective numbers at the beginning of the monitoring period. Negative values indicate a net loss. a.u.: arbitrary units; Student's t-test, n=13-19, *:$p<0.05$; **: $p<0.01$.

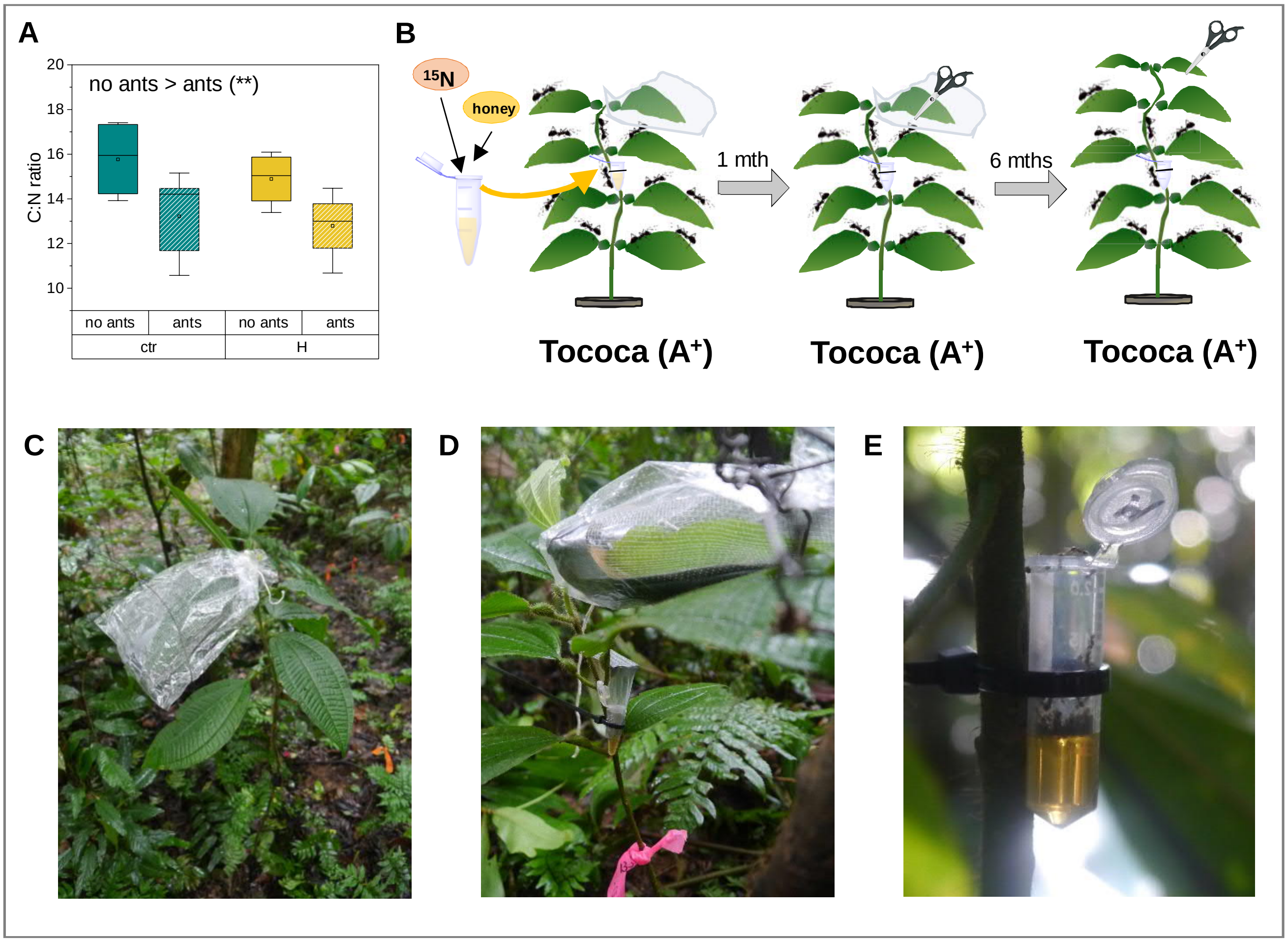

Figure S3: Effect of ant colonization on carbon:nitrogen ratio and setup for ^{15}N labeling experiment. Related to Figure 1 and STAR Methods. **A:** The C:N ratio of ant-colonized (ants) and ant-deprived (no ants) *T. quadrialata* plants was determined for leaves from the herbivory experiment 1 yr after the start of ant exclusion and the results shown in boxplots (25th percentile, median, mean (open circle) and 75th percentile). Colonization by ants led to a lower C:N ratio (**:$p_{colonization}<0.01$, $F_{1,16}=9.789$, Two-way ANOVA, n = 4-5), herbivory treatment ($F_{13,14}=0.73$, p=0.41) or the interaction of herbivory and ant colonization ($F_{14,15}=0.08$, p=0.78) did not affect the ratio. **B**: Experimental setup: ^{15}N-labeled glycine and honey were mixed in a tube that was attached to a colonized *T. quadrialata* plant (A^+). One of the youngest leaves of the plant was enclosed with a perforated plastic bag beforehand to prevent ants from accessing the leaf. After a month, a sample of the protected leaf was taken for isotope analysis. After 6 months, a newly grown leaf was sampled for isotope analysis as well. **C**-**E**: pictures of the plants and setup in the field, showing the cover (**C**, **D**), the attached tube with honey (**D**, **E**) and *Azteca* cf. *tonduzi* ants consuming the ^{15}N enriched honey (**E**).

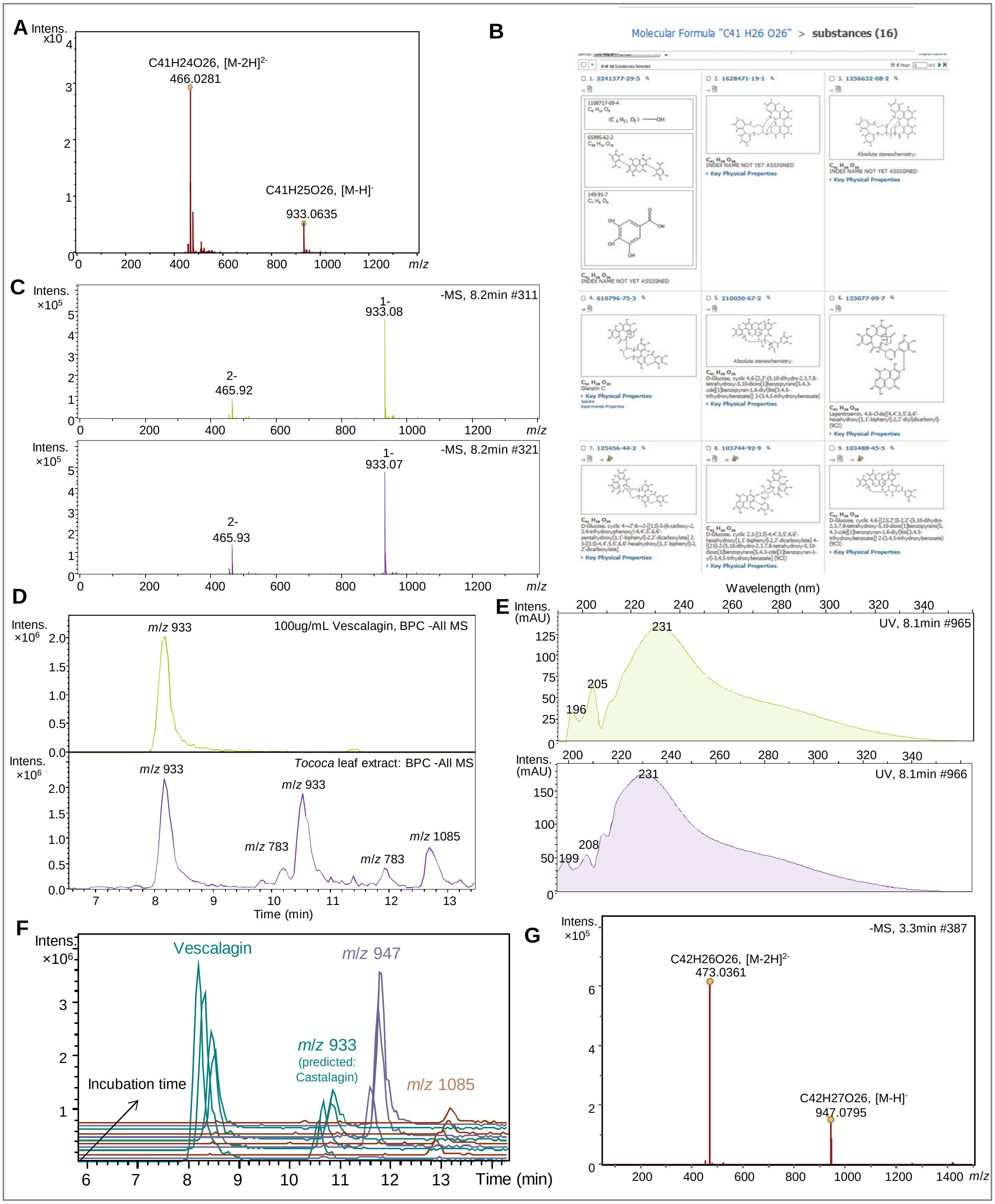

Figure S4: Identification of the ellagitannin vescalagin by high resolution MS and UV spectroscopy in comparison to literature data and an authentic standard. Related to Figure 2. High resolution MS allowed the calculation of the sum formula of this unknown feature (**A**), which enabled a search in the Scifinder data base for possible candidate structures (**B**). All 16 known possible structures with the sum formula $C_{41}H_{26}O_{26}$ belonged to the group of ellagitannins. Comparison of the mass spectra (**C**), chromatographic properties (**D**), and UV spectra (**E**) of the unknown feature (purple) with an authentic vescalagin standard (green) allowed the identification of vescalagin. **F**: Spiking with the standard revealed that vescalagin was not stable in the methanolic plant extract, but spontaneously converted to a compound with *m/z* 947 over time (z-axis). Extracted ion chromatograms of vescalagin (*m/z* 933) and *m/z* ratios of other predicted ellagitannins of a *Tococa* extract incubated for 0, 1.5, 3, 4.5 h at room temperature are shown. **G**: Based on the sum formula prediction gained by high resolution MS of the derivative, the spontaneous conversion product is an O-methylated vescalagin derivative, from now on referred to as 'MeVescalagin'.

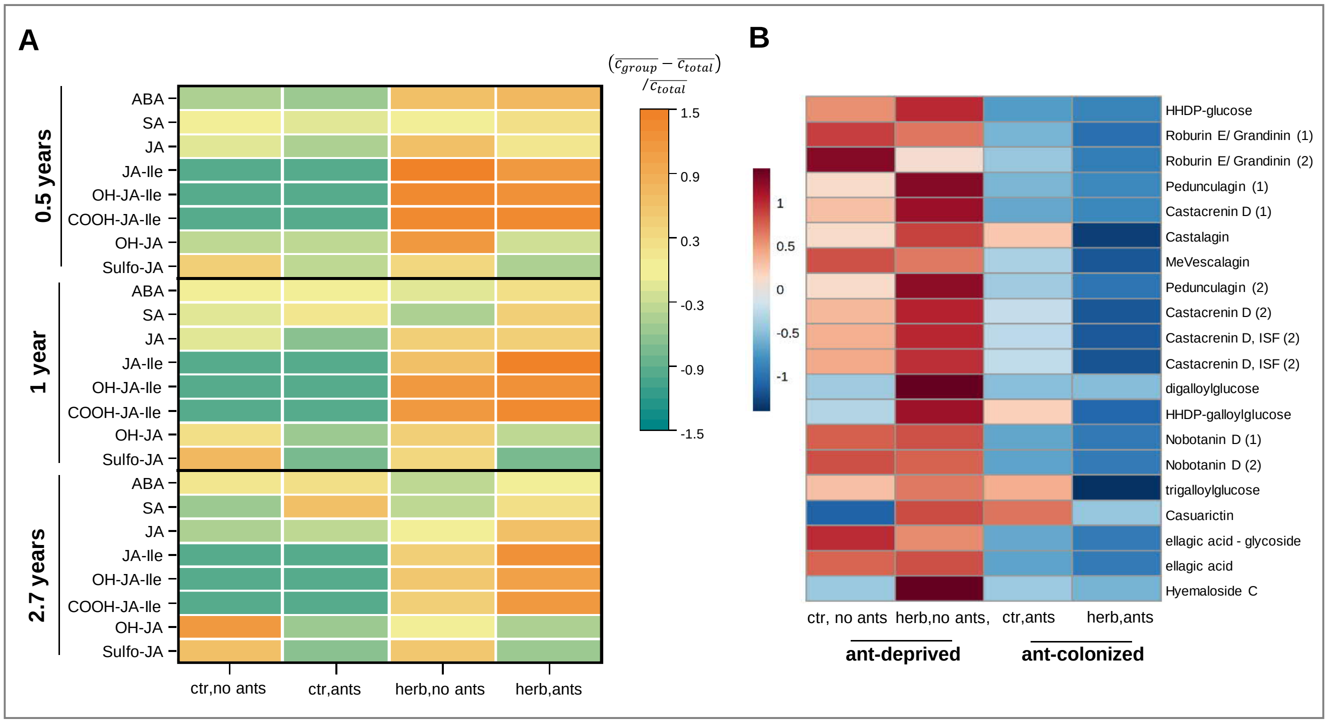

Figure S5: Effects of herbivory and ant colonization on phytohormones and ellagitannins in *Tococa quadrialata* plants. Related to Figure 3. Leaves of ant-colonized (ants) and ant-deprived (no ants) plants net-protected from herbivory otherwise were exposed to 24 h of feeding by *Spodoptera spp*. caterpillars (herb) 0.5 year (n = 6-8), 1 year (n = 4-5) and 2.7 years (n = 5-6) after installation of the nets. **A**: The phytohormones abscisic acid (ABA), salicylic acid (SA), jasmonic acid (JA) as well as prominent JA metabolites were quantified in all herbivore experiments, and their relative concentration visualized as a heatmap, showing that some phytohormones respond to herbivory whilst others were altered by ant-removal. **B**: Untargeted metabolomic analysis of the herbivore experiment 1 year after the plot was set up with mass spectrometer operating in negative mode indicated that many features predicted to be ellagitannin-related compounds (predicted compound name given, more information in Table S4) were enriched in ant-deprived compared to ant-colonized plants. The herbivory treatment, on the other hand, did not affect these features.

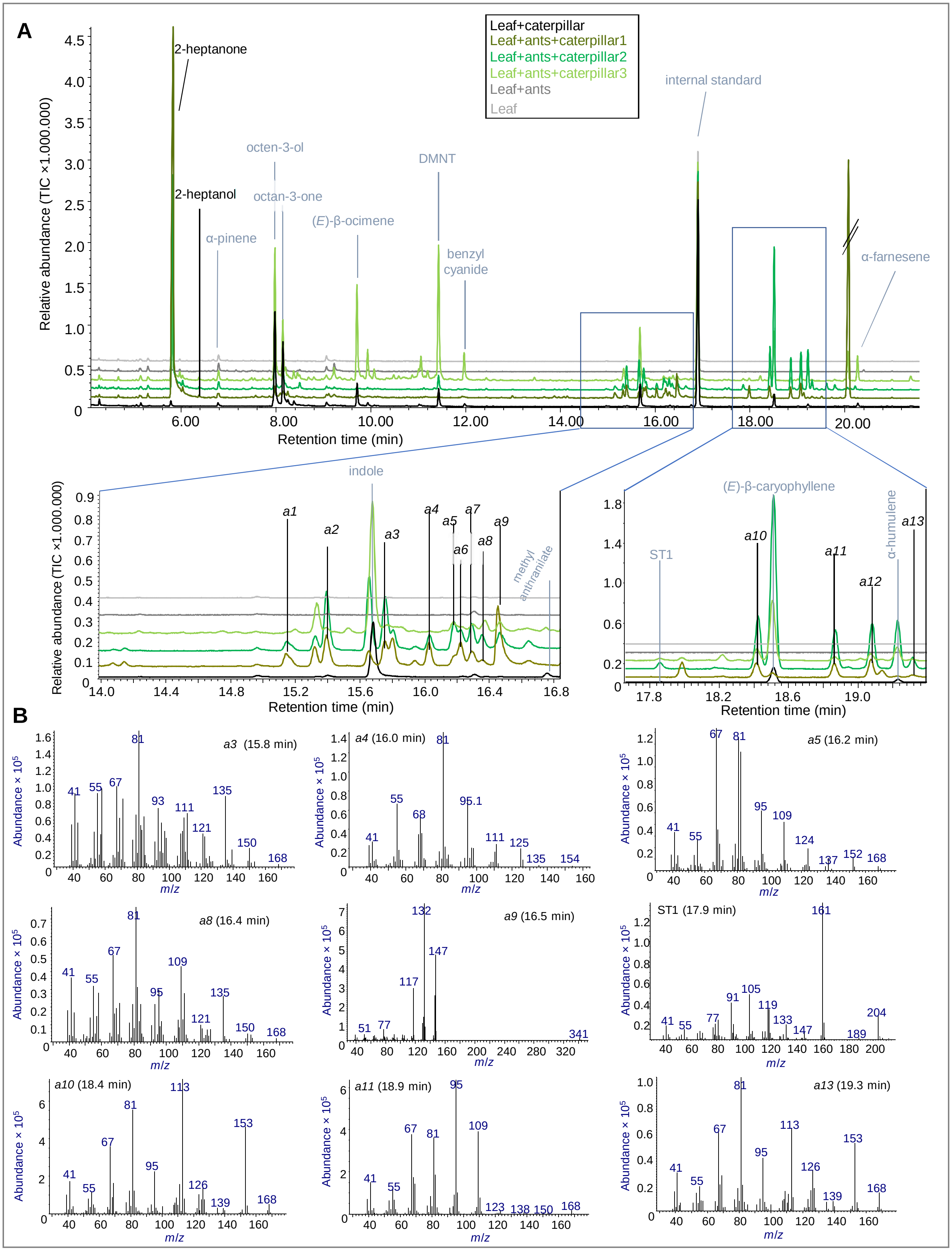

Figure S6: Identification of ant-specific and plant volatiles. Related to Figure 3. **A:** Comparison of the volatile blends of ant-colonized and ant-deprived *T. quadrialata* leaves after insect damage revealed not only herbivore induced compounds (gray label, see Figure 3F), but also that some compounds were specific for herbivory-damaged plants colonized by ants (black label). Components identified with authentic standards are labeled with their respective name; unknown ant-specific volatiles are numbered according to their retention time *a1*-*a13*. **B**: The mass spectra of some of the unknown compounds are shown. In some cases, the spectra together with their retention time allowed compound prediction with the help of the NIST17 library and literature (Do Nascimento et al., 1998, Ohmura et al., 2009): *a2,3,7,8*: iridodial-isomers*; a10*: isodihydronepetalactone; *a11, a12*: Iridomyrmecin; *a13*: dihydronepetalactone; ST1: *unknown sesquiterpene* (see also Table S2).

Table S1: Statistical analysis of the accumulation of free amino acids upon herbivory or jasmonic acid (JA) treatment of ant-colonized and ant-deprived uncolonized *T. quadrialata* plants.

Compound	Explanatory variable					
	Treatment		**Ant colonization**		**Interaction (treat:ants)**	
	F-value/ *L-ratio*	p-value	F-value/*L-ratio*	p-value	F-value/*L-ratio*	p-value
Herbivory experiment (0.5 yr) (n=6-8)						
Phe#	20.34	<0.001 ***	0.274	0.606	0.563	0.460
Trp	20.81	<0.001 ***	0.001	0.971	1.412	0.246
Tyr#	2.302	0.142	1.451	0.240	1.658	0.210
Val#	8.754	0.007 **	1.104	0.304	0.439	0.514
Ile#	13.09	0.001 **	0.340	0.565	0.942	0.342
Leu#	*11.89*	<0.001 ***	*0.255*	0.613	*0.453*	0.501
Pro	0.580	0.454	0.014	0.906	0.220	0.643
Thr#	1.260	0.273	0.295	0.592	0.174	0.680
Glu	12.17	0.002 **	1.968	0.174	0.033	0.858
Asp	0.008	0.928	1.020	0.323	0.008	0.929
Met	1.638	0.213	0.687	0.415	0.099	0.756
Ala	1.462	0.238	0.000	0.989	0.031	0.862
Herbivory experiment (1 yr) (n=4-5)						
Phe#	45.22	<0.001 ***	1.040	0.325	0.111	0.744
Trp#	29.77	<0.001 ***	1.673	0.217	0.009	0.925
Tyr#	12.64	0.003 **	2.726	0.121	0.113	0.742
Val#	18.17	<0.001 ***	3.243	0.093	0.000	0.999
Ile#	24.41	<0.001 ***	2.633	0.127	0.029	0.868
Leu#	26.72	<0.001 ***	3.748	0.073	0.192	0.668
Pro#	6.361	0.023 *	2.857	0.113	0.015	0.90
Thr#	5.412	0.034 *	2.548	0.133	0.275	0.608
Glu	0.732	0.407	8.053	0.012 *	0.080	0.781
Asp	0.631	0.440	9.290	0.008 **	0.525	0.481
Met	0.961	0.343	0.765	0.396	0.628	0.441
Ala	2.344	0.148	5.354	0.034 *	0.184	0.675
Herbivory experiment (2.6 yrs) (n=5-6)						
Phe#	*27.64*	<0.001 ***	*7.264*	0.007 **	*0.645*	0.422
Trp#	*19.61*	<0.001 ***	*2.914*	0.088	*1.190*	0.275
Tyr#	27.68	<0.001 ***	13.39	0.002 **	1.706	0.209
Val#	25.23	<0.001 ***	6.295	0.022 *	1.496	0.238
Ile#	11.49	0.003 **	4.151	0.058	0.624	0.440
Leu#	22.04	<0.001 ***	8.447	0.009 **	2.035	0.172
Pro#	22.68	<0.001 ***	2.303	0.147	0.934	0.347
Thr	4.787	0.041 *	1.652	0.216	0.562	0.464
Glu	2.862	0.109	3.605	0.075	0.002	0.969
Asp	0.011	0.916	7.525	0.013 *	0.356	0.559
Met	0.194	0.665	2.717	0.118	0.112	0.741
Ala#	0.147	0.707	7.641	0.012 *	0.446	0.513
JA spraying experiment (2.7 yrs) (n=4-5)						
Phe#	89.85	<0.001 ***	8.912	0.001 **	1.658	0.220
Trp#	62.31	<0.001 ***	12.44	0.003 **	0.467	0.506
Tyr	22.73	<0.001 ***	7.320	0.017 *	0.007	0.933
Val#	44.51	<0.001 ***	12.29	0.004 **	1.200	0.293
Ile#	42.01	<0.001 ***	13.56	0.003 **	1.222	0.289
Leu#	30.62	<0.001 ***	14.57	0.002 **	1.418	0.255
Pro#	36.95	<0.001 ***	7.849	0.014 *	1.647	0.222
Thr	8.579	0.011 *	10.99	0.004 **	0.112	0.743
Glu	0.023	0.887	12.54	0.003 **	2.530	0.136
Asp	0.876	0.367	3.592	0.0805	2.283	0.155
Met	8.325	0.012 *	4.953	0.043 *	0.009	0.927
Ala	0.501	0.491	9.067	0.009 **	3.176	0.098

Related to Figure 3. Depending on whether a Two-Way ANOVA or a gls model was used F-values or Likelihood (L)-ratios are given. L-ratios are in italics. Models were simplified to the minimal model. # indicates log-transformed data; *: p<0.05; **: p<0.01; ***: p<0.001.

Table S2: Details about the volatiles emitted in *Tococa* leaves of colonized and ant-deprived plants including names, Kovats indices for retention time, amounts and statistical analysis.

Compound	KI		rel. amount (pg/h/cm^2, mean ± SEM)				statistics (Kruskal-Wallis rank sum test)		
	Det.	Lit.	noants_C	ants_C	noants_H	ants_H	χ^2	df	p value
Terpenoids									
α-pinene	933	933	26.7±6.9	26.6±4.2	29.1±4.4	31.3±5.2	1.5644	3	0.6675
(E)-β-ocimene	1049	1052	1.5±0.7 ^{a}	1.2±0.3 ^{a}	155.5±54.5 ^{b}	306.9±150.1 ^{b}	39.262	3	<0.001
DMNT	1118	1114	2.7±1.7 ^{a}	2.3±1 ^{a}	119.5±35.1 ^{b}	282±99.2 ^{b}	40.898	3	<0.001
α-copaene	1380	1380	0.2±0.2 ^{a}	0.5±0.4 ^{a}	4.6±1.5 ^{b}	11.1±3.9 ^{b}	17.722	3	<0.001
unknown sesquiterpene1	1393	-	0.3±0.2 ^{a}	0.4±0.3 ^{a}	4.3±1.1 ^{b}	8.7±2.9 ^{b}	18.975	3	<0.001
(*E*)-β-caryophyllene	1425	1425	1.7±0.5 ^{a}	1.2±0.6 ^{a}	170.5±59.1 ^{b}	268.4±102.5 ^{b}	30.402	3	<0.001
α-humulene	1459	1455	1.4±0.6 ^{a}	0.8±0.5 ^{a}	36.2±11.7 ^{b}	62.9±23.9 ^{b}	23.739	3	<0.001
germacrene D	1487	1487	0±0 ^{a}	0±0 ^{a}	4.5±1.3 ^{b}	8.3±2.7 ^{b}	22.7	3	<0.001
α-farnesene	1510	1507	0±0 ^{a}	0.1±0.1 ^{a}	20.7±6.2 ^{b}	56.9±22.7 ^{b}	34.265	3	<0.001
nerolidol	1567	1565	0±0 ^{a}	0±0 ^{a}	10.8±4.2 ^{b}	28.1±13.7 ^{b}	32.394	3	<0.001
Nitrogenous compounds									
benzyl cyanide	1141	1148	0.8±0.7 ^{a}	0.5±0.3 ^{a}	15.3±7.5 ^{a}	26.4±9.9 ^{b}	18.997	3	<0.001
indole	1296	1290	0±0 ^{a}	0±0 ^{a}	61.9±24.6 ^{b}	140±72.6 ^{b}	34.297	3	<0.001
methyl anthranilate	1347	1354	0±0 ^{a}	0±0 ^{a}	9.4±4.6 ^{b}	35.8±17.3 ^{b}	17.857	3	<0.001
Fatty acid derived compounds									
2-heptanone§	894	896	4.6±1.2 ^{a}	6.4±1.9 ^{a}	7±1.9 ^{a}	772.9±243.9 ^{b}	22.053	3	<0.001
2-heptanol§	903	900	1.3±0.5 ^{a}	1.4±0.5 ^{a}	5.2±1.9 ^{a}	57.5±18.4 ^{b}	22.564	3	<0.001
oct-1-en-3-ol	981	983	1.8±0.8 ^{a}	3.7±1.7 ^{a}	302.7±83.3 ^{b}	553.3±149.4 ^{b}	46.484	3	<0.001
3-octanone	988	988	11.4±5.4 ^{a}	6.2±2.4 ^{a}	136±38 ^{b}	215.1±64.4 ^{b}	30.92	3	<0.001
octan-3-ol	998	996	0±0 ^{a}	0±0 ^{a}	32.6±9.8 ^{b}	75.5±25.8 ^{b}	48.396	3	<0.001
Others									
a1 *(3-methyl-hexahydrophthalide)*§	1273	-	0.8±0.4	0.7±0.2 ^{a}	1.4±0.4 ^{a}	13.4±5 ^{b}	13.34	3	0.0040
a2 *(iridodial)*§	1281	-	2.9±0.5 ^{ab}	1.4±0.4 ^{a}	3.8±0.7 ^{b}	19±7.6 ^{b}	8.4578	3	0.0374
a3 *(iridodial)*§	1299	-	0.8±0.4 ^{a}	1.1±0.3 ^{a}	1.9±0.6 ^{a}	28.2±11.4 ^{b}	12.601	3	0.0056
a4§	1301	-	0±0 ^{a}	0±0 ^{a}	0.2±0.2 ^{a}	6.2±2.8 ^{b}	20.013	3	<0.001
a5§	1307	-	0±0 ^{a}	0±0 ^{a}	0±0 ^{a}	8.3±3.8 ^{b}	22.723	3	<0.001
a6§	1311	-	1.1±0.3 ^{a}	1±0.3 ^{a}	1±0.3 ^{a}	17.3±6.2 ^{b}	10.431	3	0.0152
a7 *(iridodial)*§	1316	-	0.7±0.5 ^{ab}	0.5±0.2 ^{ab}	0.3±0.2 ^{a}	5.1±2.9 ^{b}	8.4023	3	0.0384
a8 *(iridodial)*§	1320	-	2.2±1 ^{a}	2.5±1 ^{a}	3.1±0.8 ^{a}	15±4.4 ^{b}	11.278	3	0.0103
a9 *(2-(1-Methylcyclopropyl) aniline)*§	1331	1331	0±0 ^{a}	0.1±0.1 ^{a}	0.4±0.3 ^{a}	17.2±6.8 ^{b}	15.133	3	0.0017
a10 (*isodihydro-nepetalactone)*§	1420	1414	2.4±1.9 ^{a}	1.1±0.6 ^{a}	0.5±0.2 ^{a}	7.6±3.9 ^{b}	11.128	3	0.0111
a11 *(iridomyrmecin)*§	1442	1463	0±0 ^{a}	0±0 ^{a}	0.2±0.2 ^{a}	22.1±8.6 ^{b}	28.552	3	<0.001
a12 *(iridomyrmecin)*§	1452	1463	0±0 ^{a}	0±0 ^{a}	1.5±0.7 ^{a}	34.6±14.2 ^{b}	27.769	3	<0.001
a13 *(dihydro-nepetalactone)*§	1463	1414 -30	0±0 ^{a}	0±0 ^{a}	0.1±0.1 ^{a}	7.7±3.1 ^{b}	25.601	3	<0.001

Related to Figure 3. Specific chemicals present at concentrations above 800 pg cm^{-2} in at least two of the samples are depicted. Compounds only identified by library (NIST17) and literature (Do Nascimento et al., 1998; Ohmura et al., 2009) search without comparison to an authentic standard are in *italics*. To further confirm the identification or provide additional information on the compounds, the retention time indices were determined (Det.) with the temperature-programmed Kovats index (KI) and the values compared to NIST and literature (Lit). For mass spectra of these compounds, see Figure S6. § indicates that the compound is most likely produced by the ants. The Dunn-test was used as a *post hoc* test. Different letters indicate significant differences between groups (p<0.025); n=14-21.

Table S3: Statistical analysis of the changes in various defense hormones caused by herbivore damage and ant-colonization.

Compound	Explanatory variable					
	Treatment (herbivory or JA)		**Ant-colonization**		**Interaction (treat:ants)**	
	F-value/ *L-ratio*	p-value	F-value/*L-ratio*	p-value	F-value/*L-ratio*	p-value
Herbivory experiment (0.5 yr) (n=6-8)						
ABA[#]	9.771	0.004 **	0.423	0.522	0.036	0.852
SA[#]	0.702	0.410	0.438	0.514	0.000	0.995
JA	12.05	0.002 **	2.727	0.112	0.264	0.612
JA-Ile[#]	*68.73*	0.003 **	*0.000*	0.989	*0.036*	0.851
OH-JA-Ile[#]	*37.91*	<0.001 ***	*4.275*	0.039 *	*1.028*	0.311
COOH-JA-Ile[#]	*48.90*	<0.001 ***	*2.247*	0.134	*0.369*	0.543
OH-JA[#]	3.963	0.057	2.173	0.153	1.816	0.190
Sulfo-JA	1.224	0.280	37.16	<0.001 ***	0.194	0.664
Herbivory experiment (1 yr) (n=4-5)						
ABA[#]	0.288	0.600	4.971	0.040 *	0.030	0.866
SA	0.027	0.873	7.928	0.012 *	2.046	0.175
JA[#]	6.207	0.024 *	0.755	0.399	2.254	0.156
JA-Ile[#]	*16.24*	<0.001 ***	*2.890*	0.089	*0.880*	0.347
OH-JA-Ile[#]	*20.73*	<0.001 ***	*1.672*	0.196	*1.713*	0.191
COOH-JA-Ile[#]	*24.14*	<0.001 ***	*1.186*	0.276	*0.391*	0.532
OH-JA	0.146	0.708	9.129	0.008 **	0.071	0.793
Sulfo-JA[$]	0.868	0.367	30.21	<0.001 ***	1.027	0.328
Herbivory experiment (2.6 yrs) (n=5-6)						
ABA[#]	6.142	0.023 *	5.745	0.027 *	0.886	0.359
SA	0.285	0.600	15.11	<0.001 ***	2.668	0.120
JA[#]	5.403	0.031 *	0.490	0.493	0.369	0.551
JA-Ile[#]	*48.80*	<0.001 ***	*0.931*	0.335	*1.512*	0.219
OH-JA-Ile[#]	*21.19*	<0.001 ***	*0.120*	0.729	*3.450*	0.063
COOH-JA-Ile[#]	*17.69*	<0.001 ***	*0.499*	0.480	*4.357*	0.037 *
OH-JA[$]	2.961	0.102	16.30	<0.001 ***	6.194	0.023 *
Sulfo-JA[#]	0.022	0.885	14.00	0.001 **	0.240	0.630
JA spraying experiment (2.7 yrs) (n=4-5)						
ABA[#]	1.097	0.314	7.493	0.015 *	1.376	0.262
SA	0.847	0.374	3.873	0.068	2.091	0.172
JA[#]	386.4	<0.001 ***	0.069	0.797	4.470	0.054
JA-Ile[#]	*24.18*	<0.001 ***	*0.099*	0.753	*2.449*	0.118
OH-JA-Ile	*28.13*	<0.001 ***	*0.771*	0.380	*1.090*	0.297
COOH-JA-Ile	*19.76*	<0.001 ***	*3.571*	0.059	*0.329*	0.567
OH-JA[$]	7.125	0.018 *	1.503	0.242	0.062	0.807
Sulfo-JA	3.812	0.073	5.776	0.030 *	0.021	0.886

Related to Figure 3. Depending on whether a Two-Way ANOVA or a gls model was used F-values or Likelihood (L)-ratios are given. L-ratios are in italics. Models were simplified to the minimal model. # indicates log-transformed data, $ sqrt-transformed data; *: p<0.05; **: p<0.01; ***: p<0.001.

Table S4: Additional information about the features assigned as ellagitannins or related compounds that are shown in Figure 2 and Figure S5.

RT	*m/z* (negative mode)	**other *m/z* of the same compound** (e.g. in source fragments)	Sum formula	possible compound
Measurements 1 year after ant exclusion				
1.18	481.0625		$C_{20}H_{18}O_{14}$	HHDP-glucose
2.20	532.0497 $[M-2H]^{2-}$		$C_{46}H_{34}O_{30}$	Roburin E/ Grandinin (1)
2.55	532.0497 $[M-2H]^{2-}$		$C_{46}H_{34}O_{30}$	Roburin E/ Grandinin (2)
2.81	783.0687		$C_{34}H_{24}O_{22}$	Pedunculagin (1)
2.86	542.0340 $[M-2H]^{2-}$		$C_{48}H_{30}O_{30}$	Castacrenin D (1)
3.03	466.0285 $[M-2H]^{2-}$		$C_{41}H_{26}O_{26}$	Castalagin
3.25	473.0364 $[M-2H]^{2-}$		$C_{42}H_{28}O_{26}$	MeVescalagin
3.37	783.0687		$C_{34}H_{24}O_{22}$	Pedunculagin (2)
3.45	542.0341 $[M-2H]^{2-}$	457.0233 $[M-GA-2H]^{2-}$ 169.0143 $[GA-H]^{1-}$	$C_{48}H_{30}O_{30}$	Castacrenin D (2)
3.58	483.0780		$C_{20}H_{20}O_{18}$	digalloylglucose
3.75	633.0734		$C_{27}H_{22}O_{18}$	HHDP-galloylglucose
4.18	392.0387 $[M-2H]^{2-}$		$C_{34}H_{26}O_{22}$	Tellimagrandin/Nobotanin D
4.47	635.0891		$C_{27}H_{24}O_{18}$	trigalloylglucose
4.50	392.0387 $[M-2H]^{2-}$		$C_{34}H_{26}O_{22}$	Nobotanin D/Tellimagrandin
4.54	467.0362 $[M-2H]^{2-}$		$C_{41}H_{28}O_{26}$	Casuarictin
4.89	447.0569		$C_{20}H_{16}O_{12}$	Ellagic acid glycoside
5.28	300.9991		$C_{14}H_6O_8$	Ellagic acid
5.51	875.0945		$C_{40}H_{30}O_{23}$	Hyemaloside C
Measurements 2.6 years after ant exclusion				
2.82	783.0690		$C_{34}H_{24}O_{22}$	Pedunculagin (1)
3.05	466.0284 $[M-2H]^{2-}$		$C_{41}H_{26}O_{26}$	Castalagin
3.26	473.0363 $[M-2H]^{2-}$		$C_{42}H_{28}O_{26}$	MeVescalagin
3.40	783.0685		$C_{34}H_{24}O_{22}$	Pedunculagin (2)
3.46	542.0339 $[M-2H]^{2-}$	457.0231 $[M-GA-2H]^{2-}$ 169.0143 $[GA-H]^{1-}$	$C_{48}H_{30}O_{30}$	Castacrenin D
3.57	483.0781		$C_{20}H_{20}O_{18}$	digalloylglucose
3.76	633.0736		$C_{27}H_{22}O_{18}$	HHDP-galloylglucose
4.46	635.0893		$C_{27}H_{24}O_{18}$	trigalloylglucose
4.50	392.0387 $[M-2H]^{2-}$		$C_{34}H_{26}O_{22}$	Nobotanin D
4.57	467.0359 $[M-2H]^{2-}$		$C_{41}H_{28}O_{26}$	Casuarictin
5.27	300.9991		$C_{14}H_6O_8$	Ellagic acid
5.52	875.0950		$C_{40}H_{30}O_{23}$	Hyemaloside C

Related to Figure 2. *m/z* corresponds to $[M-H]^-$, if not noted otherwise. Compound names assigned based on SciFinder hits for the sum formula and comparison to literature (Fracassetti et al., 2013; Moilanen et al., 2013; Serna and Martinez, 2015; Yoshida et al., 1991). GA: gallic acid moiety, RT: retention time, HHDP: hexahydroxydiphenoyl.