SUPPLEMENTARY INFORMATION

This document contains nine tables of supporting information. The tables contain the in-source CID mass measurement data and mass measurement data for DART-HRMS analysis of six Ayahuasca brews.

Table S-1. Mass measurement data of in-source CID spectra obtained at 60 V for *M. hostilis* and dimethyltryptamine. The corresponding spectra are shown in Figure 3.

	Formula	Measured	Calculated	Diff.[†]	Rel. Int.[‡]
M. hostilis	$C_{12}H_{16}N_2 + H^+$	189.1381	189.1392	1.1	3.9
	$C_{10}H_{10}NO$	160.0738	160.0762	2.4	13.1
	$C_{10}H_{10}N$	144.0810	144.0813	0.3	79.3
	C_3H_8N	58.0683	58.0657	-2.6	100.0
DMT	$C_{12}H_{16}N_2 + H^+$	189.1381	189.1392	1.1	21.6
	$C_{10}H_{10}NO$	160.0757	160.0762	0.5	9.3
	$C_{10}H_{10}N$	144.0817	144.0813	-0.4	72.6
	C_3H_8N	58.0639	58.0657	1.8	100.0

[†]Differences are reported in millimass units (mmu). Measured masses fell within 5 mmu of the calculated masses.
[‡]Relative intensities are reported in percent.

Table S-2. Mass measurement data of in-source CID spectra obtained at 60 V for *P. viridis* and dimethyltryptamine. The corresponding spectra are shown in Figure 3.

	Formula	Measured	Calculated	Diff.[†]	Rel. Int.[‡]
P. viridis	$C_{12}H_{16}N_2 + H^+$	189.1410	189.1392	-1.8	9.6
	$C_{10}H_{10}NO$	160.0759	160.0762	0.3	8.0
	$C_{10}H_{10}N$	144.0801	144.0813	1.2	60.0
	C_3H_8N	58.0667	58.0657	-1.0	100.0
DMT	$C_{12}H_{16}N_2 + H^+$	189.1381	189.1392	1.1	21.6
	$C_{10}H_{10}NO$	160.0757	160.0762	0.5	9.3
	$C_{10}H_{10}N$	144.0817	144.0813	-0.4	72.6
	C_3H_8N	58.0639	58.0657	1.8	100.0

[†]Differences are reported in millimass units (mmu). Measured masses fell within 5 mmu of the calculated masses.
[‡]Relative intensities are reported in percent.

Table S-3. Mass measurement data of in-source CID spectra obtained at 60 V for *D. cabrerana* and dimethyltryptamine. The corresponding spectra are shown in Figure 3.

	Formula	Measured	Calculated	Diff.[†]	Rel. Int.[‡]
D. cabrerana	$C_{12}H_{16}N_2 + H^+$	189.1400	189.1392	-0.8	12.3
	$C_{10}H_{10}NO$	160.0757	160.0762	0.5	9.0
	$C_{10}H_{10}N$	144.0801	144.0813	1.2	70.8
	C_3H_8N	58.0671	58.0657	-1.4	100.0
DMT	$C_{12}H_{16}N_2 + H^+$	189.1381	189.1392	1.1	21.6
	$C_{10}H_{10}NO$	160.0757	160.0762	0.5	9.3
	$C_{10}H_{10}N$	144.0817	144.0813	-0.4	72.6
	C_3H_8N	58.0639	58.0657	1.8	100.0

[†]Differences are reported in millimass units (mmu). Measured masses fell within 5 mmu of the calculated masses.
[‡]Relative intensities are reported in percent.

Table S-4. Mass measurement data of in-source CID spectra obtained at 90 V for *P. harmala* and harmaline. The corresponding spectra are shown in Figure 4.

	Formula	Measured	Calculated	Diff.[†]	Rel. Int.[‡]
P. harmala	$C_{13}H_{14}N_2O + H^+$	215.1151	215.1184	3.3	100.0
	$C_9H_{14}N_2O_4$	200.0949	200.0953	0.4	82.1
	$C_{11}H_{12}NO$	174.0901	174.0919	1.8	87.0
	$C_8H_{14}NO_3$	172.0968	172.0974	0.6	55.2
	$C_7H_{11}O_4$	159.0677	159.0657	-2.0	14.0
	C_4H_6N	68.0500	68.0500	0.0	18.5
Harmaline	$C_{13}H_{14}N_2O + H^+$	215.1191	215.1184	-0.7	100.0
	$C_9H_{14}N_2O_4$	200.0923	200.0953	3.0	77.0
	$C_{11}H_{12}NO$	174.0907	174.0919	1.2	87.4
	$C_8H_{14}NO_3$	172.0976	172.0974	-0.2	34.0
	$C_7H_{11}O_4$	159.0664	159.0657	-0.7	10.7
	C_4H_6N	68.0522	68.0500	-2.2	17.6

[†]Differences are reported in millimass units (mmu). Measured masses fell within 5 mmu of the calculated masses.

‡Relative intensities are reported in percent.

Table S-5. Mass measurement data of in-source CID spectra obtained at 90 V for *B. caapi* and harmaline. The corresponding spectra are shown in Figure 4.

	Formula	Measured	Calculated	Diff.†	Rel. Int.‡
B. caapi	$C_{13}H_{14}N_2O + H^+$	215.1145	215.1184	3.9	14.2
	$C_9H_{14}N_2O_4$	200.0916	200.0953	3.7	5.6
	$C_{11}H_{12}NO$	174.0899	174.0919	2.0	6.2
	$C_8H_{14}NO_3$	172.0932	172.0974	4.2	1.8
	$C_7H_{11}O_4$	159.0689	159.0657	-3.2	0.4
	C_4H_6N	68.0504	68.0500	-0.4	1.6
Harmaline	$C_{13}H_{14}N_2O + H^+$	215.1191	215.1184	-0.7	100.0
	$C_9H_{14}N_2O_4$	200.0923	200.0953	3.0	77.0
	$C_{11}H_{12}NO$	174.0907	174.0919	1.2	87.4
	$C_8H_{14}NO_3$	172.0976	172.0974	-0.2	34.0
	$C_7H_{11}O_4$	159.0664	159.0657	-0.7	10.7
	C_4H_6N	68.0522	68.0500	-2.2	17.6

†Differences are reported in millimass units (mmu). Measured masses fell within 5 mmu of the calculated masses.
‡Relative intensities are reported in percent.

Table S-6. Mass measurement data of in-source CID spectra obtained at 90 V for *B. caapi* and harmine. The corresponding spectra are shown in Figure 4.

	Formula	Measured	Calculated	Diff.†	Rel. Int.‡
B. .caapi	$C_{13}H_{12}N_2O + H^+$	213.0995	213.1028	3.3	100.0
	$C_{13}H_{11}NO_2$	199.0805	199.0759	4.6	23.1
	$C_{12}H_{10}N_2O$	198.0779	198.0793	1.4	82.0
	$C_{12}H_{11}O$	171.0855	171.0810	-4.5	9.6
	$C_{11}H_{10}N_2$	170.0837	170.0817	-2.0	58.1
Harmine	$C_{13}H_{12}N_2O + H^+$	213.0997	213.1028	2.9	100.0
	$C_{13}H_{11}NO_2$	199.0800	199.0759	4.1	29.9
	$C_{12}H_{10}N_2O$	198.0787	198.0793	0.6	91.1
	$C_{12}H_{11}O$	171.0857	171.0810	-4.7	17.0
	$C_{11}H_{10}N_2$	170.0845	170.0817	-2.8	74.8

†Differences are reported in millimass units (mmu). Measured masses fell within 5 mmu of the calculated masses.
‡Relative intensities are reported in percent.

Table S-7. Mass measurement data of in-source CID spectra obtained at 90 V for *P. harmala* and harmine. The corresponding spectra are shown in Figure 4.

	Formula	Measured	Calculated	Diff.[†]	Rel. Int.[‡]
P. harmala	$C_{13}H_{12}N_2O + H^+$	213.1019	213.1028	0.9	45.7
	$C_{12}H_{11}N_2O$	199.0865	199.0759	0.6	11.8
	$C_{12}H_{10}N_2O$	198.0811	198.0793	-1.8	50.9
	$C_{11}H_{10}N_2$	170.0833	170.0817	-1.6	36.7
Harmine	$C_{13}H_{12}N_2O + H^+$	213.0997	213.1028	2.9	100.0
	$C_{12}H_{11}N_2O$	199.0800	199.0759	4.1	29.9
	$C_{12}H_{10}N_2O$	198.0787	198.0793	0.6	91.1
	$C_{11}H_{10}N_2$	170.0845	170.0817	-2.8	74.8

[†]Differences are reported in millimass units (mmu). Measured masses fell within 5 mmu of the calculated masses.
[‡]Relative intensities are reported in percent.

Table S-8. Mass measurement data of in-source CID spectra obtained at 90 V for *P. harmala* and vasicine. The corresponding spectra are shown in Figure 4.

	Formula	Measured	Calculated	Diff.[†]	Rel. Int.[‡]
P. harmala	$C_{11}H_{12}N_2O + H^+$	189.1044	189.1027	-1.6	23.7
	$C_{11}H_{11}N_2$	171.0933	171.0922	-1.1	76.3
	$C_{10}H_{10}N$	144.0816	144.0813	-0.3	18.5
	$C_{10}H_9N$	143.0755	143.0735	-2.0	18.5
	C_8H_8N	118.0678	118.0657	-2.1	53.7
	C_7H_7	91.0538	91.0548	1.0	7.4
	C_4H_6N	68.0464	68.0500	3.6	22.0
Vasicine	$C_{11}H_{12}N_2O + H^+$	189.0980	189.1027	2.7	66.3
	$C_{11}H_{11}N_2$	171.0897	171.0922	1.5	100.0
	$C_{10}H_{10}N$	144.0798	144.0813	1.5	49.4
	$C_{10}H_9N$	143.0707	143.0735	2.8	51.7
	C_8H_8N	118.0654	118.0657	0.3	99.4
	C_7H_7	91.0539	91.0548	0.9	19.5
	C_4H_6N	68.0497	68.0500	0.3	4.2

[†]Differences are reported in millimass units (mmu). Measured masses fell within 5 mmu of the calculated masses.
[‡]Relative intensities are reported in percent.

Table S-1. Mass measurement data of Ayahuasca brews at 20 V. The corresponding spectra are shown in Figure 5.

	Brew Components		Compound	Formula	Measured	Calculated	Diff.[†]	Rel. Int.[‡]
	DMT	Harmala						
Ayahuasca Brew 1	*P. viridis*	*B. caapi*	*N*-Methyltryptamine[a]	$C_{11}H_{14}N_2 + H^+$	175.1232	175.1235	0.3	1.7
			Harmane[a]	$C_{12}H_{10}N_2 + H^+$	183.0889	183.0922	3.3	2.0
			Dimethyltryptamine[b]	$C_{12}H_{16}N_2 + H^+$	189.1368	189.1392	2.4	100.0
			Harmine[b]	$C_{13}H_{12}N_2O + H^+$	213.1027	213.1028	0.1	69.6
			Harmaline[b]	$C_{12}H_{14}N_2O + H^+$	215.1158	215.1184	2.6	2.1
Ayahuasca Brew 2	*M. hostilis*	*P. harmala*	Deoxypeganine[a]	$C_{11}H_{12}N_2 + H^+$	173.1082	173.1079	-0.3	3.8
			N-Methyltryptamine[a]	$C_{11}H_{14}N_2 + H^+$	175.1234	175.1235	0.1	5.1
			Dimethyltryptamine[b]	$C_{12}H_{16}N_2 + H^+$	189.1358	189.1392	3.4	100.0
			Harmine[b]	$C_{13}H_{12}N_2O + H^+$	213.1012	213.1028	1.6	51.4
			Harmaline[b]	$C_{12}H_{14}N_2O + H^+$	215.1175	215.1184	0.9	73.7
Ayahuasca Brew 3	*P. viridis*	*P. harmala*	Deoxypeganine[a]	$C_{11}H_{12}N_2 + H^+$	173.1099	173.1079	-2.0	3.3
			N-Methyltryptamine[a]	$C_{11}H_{14}N_2 + H^+$	175.1230	175.1235	0.5	1.6
			Dimethyltryptamine[b]	$C_{12}H_{16}N_2 + H^+$	189.1342	189.1392	5.0	100.0
			Harmine[b]	$C_{13}H_{12}N_2O + H^+$	213.1002	213.1028	2.6	36.8
			Harmaline[b]	$C_{12}H_{14}N_2O + H^+$	215.1163	215.1184	2.1	57.2
Ayahuasca Brew 4	*M. hostilis*	*B. caapi*	*N*-Methyltryptamine[a]	$C_{11}H_{14}N_2 + H^+$	175.1236	175.1235	-0.1	2.8
			Harmane[a]	$C_{12}H_{10}N_2 + H^+$	183.0884	183.0922	3.8	1.1
			Dimethyltryptamine[b]	$C_{12}H_{16}N_2 + H^+$	189.1381	189.1392	1.1	77.1
			Harmine[b]	$C_{13}H_{12}N_2O + H^+$	213.1036	213.1028	-0.8	100.0
			Harmaline[b]	$C_{12}H_{14}N_2O + H^+$	215.1156	215.1184	2.8	5.9
Ayahuasca Brew 5	*D. cabrerana*	*B. caapi*	*N*-Methyltryptamine[a]	$C_{11}H_{14}N_2 + H^+$	175.1265	175.1235	-3.0	1.1
			Harmane[a]	$C_{12}H_{10}N_2 + H^+$	183.0884	183.0922	3.8	1.9
			Dimethyltryptamine[b]	$C_{12}H_{16}N_2 + H^+$	189.1385	189.1392	0.7	100.0
			Harmine[b]	$C_{13}H_{12}N_2O + H^+$	213.1008	213.1028	2.0	94.2
			Harmaline[b]	$C_{12}H_{14}N_2O + H^+$	215.1131	215.1184	5.3	13.8
			Tetrahydroharmine[a]	$C_{12}H_{16}N_2O + H^+$	217.1329	217.1341	1.2	2.1
Ayahuasca Brew 6	*D. cabrerana*	*P. harmala*	Deoxypeganine[a]	$C_{11}H_{12}N_2 + H^+$	173.1062	173.1079	1.7	3.2
			N-Methyltryptamine[a]	$C_{11}H_{14}N_2 + H^+$	175.1235	175.1235	-0.9	1.4
			Dimethyltryptamine[b]	$C_{12}H_{16}N_2 + H^+$	189.1392	189.1392	0.2	100.0
			Harmine[b]	$C_{13}H_{12}N_2O + H^+$	213.1028	213.1028	0.8	36.7
			Harmaline[b]	$C_{12}H_{14}N_2O + H^+$	215.1184	215.1184	0.0	50.0

[a]The corresponding mass was consistent with the formula of the indicated compound which has previously been isolated from the species listed. [b]The presence of this compound was confirmed through comparison of in-source collision-induced dissociation (CID) spectrum of the plant material with the in-source CID spectrum of an authentic standard. [†]Differences are reported in millimass units (mmu). Measured masses fell within 5 mmu of the calculated masses. [‡]Relative intensities are reported in percent.