Optimisation of Headspace High Concentration Capacity method followed by comprehensive two-dimensional Gas Chromatography Mass Spectrometry applied to coffee brew aroma analysis

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7. Annexes

Annex 1 Averaged values of features for sample volume = 1 and 4 mL, and for extraction time of 10, 30, 40 and 60 min (HiSorb)

Sample volume	**1 mL**				**4 mL**			
Extraction time	**10 min**	**30 min**	**40 min**	**60 min**	**10 min**	**30 min**	**40 min**	**60 min**
8.487_2.500_49	18400	185000	213000	172000	24100	215000	202000	240000
10.719_2.000_106	7430	8260	7110	9620	8200	30300	6980	6700
20.956_2.000_144	5410	6740	5040	6040	5070	6910	7920	18400
17.544_1.000_86	7610	16100	15500	53400	7720	19300	14500	21900
24.763_2.500_108	11700	18500	25600	26900	10500	23300	45300	32600
7.700_2.500_234	6580	4720	7470	4660	4750	6500	4290	4870
6.912_2.000_79	13800	16900	6310	12000	6410	14400	11300	9120
20.431_2.500_122	15600	131000	123000	173000	19900	157000	136000	194000
24.369_2.000_153	5720	5940	7000	5030	6430	6500	14600	18000
10.719_1.500_106	6080	8190	6440	6910	8550	28300	7220	7890
7.831_2.500_45	21900	47400	56100	22500	27400	42200	32800	41800
25.550_1.500_145	9130	55200	28300	31500	8950	30500	41900	47300
13.475_2.500_195	5630	6150	3960	3720	4640	6580	3920	6230
11.506_3.000_291	4290	4790	4480	5390	6580	3810	3570	4390
21.088_1.500_109	35100	275000	316000	311000	44800	322000	289000	357000
10.456_2.500_99	14600	17100	16300	14500	13700	18300	86900	59800
26.863_3.000_175	3910	12500	10700	33100	4140	9090	11700	45600
17.675_3.000_117	7470	4830	7220	17200	5800	5550	8190	8600
12.688_1.500_88	5940	4520	6620	5770	6660	20500	8460	11400
8.881_2.500_66	6020	7800	18800	10000	6540	5700	10900	12200
19.250_1.500_350	4520	6460	5820	5690	7010	5730	5110	4610
13.344_1.500_95	440000	2140000	2250000	2590000	444000	2870000	3400000	4330000
21.219_2.000_109	32600	181000	256000	293000	34800	239000	230000	299000
22.531_0.500_327	4280	12000	5420	5230	5580	6170	5940	5420
23.844_2.000_134	8020	15900	7230	25300	9000	26500	10700	11200
25.025_2.000_88	5810	9000	7060	17900	6620	5320	7080	11200
14.919_1.000_88	9550	29100	30000	27700	9030	32400	15200	54700
12.819_2.500_165	3980	5110	5880	5100	6330	5350	4120	4060
18.069_2.000_66	29400	169000	173000	163000	37600	188000	190000	238000
15.050_1.500_64	12800	22700	30200	58600	15100	26700	26300	49500
14.919_0.500_206	7340	6650	6480	4800	7420	4570	5070	20500
23.581_3.000_60	8650	10800	11000	16400	6680	7610	20700	17400
25.288_2.000_220	6880	5360	15200	7220	5020	4810	5280	6340

14.263_1.000_87	6420	25400	9010	11200	6340	7050	12400	8730
19.775_1.000_210	6440	6070	5200	5580	3800	5330	4700	5380
11.900_2.000_116	22600	93500	133000	122000	16800	135000	183000	196000
20.038_3.000_133	16100	62900	36700	72000	10500	51200	52800	123000
21.875_3.000_136	8320	21200	17900	44700	11700	16000	25000	19400
20.431_1.500_89	6970	10100	11100	10100	8120	22900	9290	13000
11.900_3.000_59	6150	8650	26400	10200	8790	12600	9900	11300
17.019_2.500_316	5040	8300	5130	5680	5190	5130	5540	3800
23.450_1.500_185	4800	6260	6350	5090	10500	5420	5320	10100
11.900_1.500_43	226000	1070000	1140000	1340000	204000	1340000	1520000	1690000
21.350_1.000_154	7190	12900	13900	22000	7880	9520	20400	46900
17.938_0.500_137	9300	33800	19600	51800	12400	49800	70100	48900
20.300_1.000_142	6390	5860	9750	18700	5890	5780	7390	8410
19.381_1.500_145	4390	5380	5450	5780	5490	11700	5170	7950
14.525_1.500_142	6030	19400	4890	7100	5020	10000	5680	5510
12.163_1.000_290	4380	6570	7950	6540	5920	5070	4840	5150
8.487_2.000_49	10400	125000	137000	117000	10600	151000	157000	167000
19.513_1.500_140	12000	35000	42500	97500	16400	46500	64400	80100
25.550_2.500_93	10100	25500	26300	25300	10400	20900	22400	54700
20.825_1.500_187	6360	5250	14600	7260	7410	5280	7100	6750
17.019_0.500_193	5080	12300	6570	7750	5460	6170	7110	5100
17.806_1.000_144	5550	5340	6590	5250	5030	5740	13500	7120
13.475_1.000_236	4880	5520	3740	5880	7210	7080	5100	6460
14.919_1.500_110	903000	8450000	9810000	10500000	1290000	11700000	14100000	17400000
18.856_2.000_92	13400	50900	77900	65400	14900	77600	51400	56600
21.875_1.000_323	5180	3340	3870	7260	5110	3980	4780	3680
22.531_1.500_150	8500	24000	26000	33200	9820	39300	18600	50700
10.981_3.000_108	7420	5770	5930	4990	5890	18600	17200	10400
21.219_1.000_110	19600	87300	39600	92700	18700	67500	101000	69600
17.413_1.500_332	5790	5540	5920	5470	3980	5340	5030	5760
15.969_1.500_42	585000	3790000	3920000	5430000	591000	5110000	5860000	7460000
24.631_1.000_60	7860	9880	13700	19100	5790	8100	6660	11400
11.113_3.000_108	6330	6540	5580	3610	4840	16400	5060	8860
14.525_1.000_142	6160	19500	5760	7460	4660	8280	6150	6250
23.975_1.500_178	5820	12200	7880	13700	6130	12600	13600	33200
9.931_2.500_63	5120	7140	14200	5640	5700	7560	5650	9680
16.888_0.500_196	5000	5810	6140	6370	6580	6000	5030	5020
21.088_2.000_109	26800	260000	285000	306000	37900	280000	266000	357000
11.375_2.500_111	9780	16800	17100	46700	8170	29900	48500	95000

15.313_1.000_62	10400	16800	8650	14100	6270	32200	14000	18800
20.300_1.500_142	5730	5240	7040	21900	7760	6540	14400	8930
23.844_1.500_266	5090	5530	5950	5390	5360	5820	11400	5570
22.400_1.000_113	7380	21200	9040	12200	7900	7960	10600	10400
11.375_2.000_336	3950	3620	3700	4620	6100	5910	4670	4350
21.219_2.500_109	28500	149000	173000	249000	27100	173000	184000	225000
13.344_1.000_66	47200	150000	151000	204000	55500	213000	316000	440000
19.119_3.000_134	13000	63800	52900	29000	13200	50700	63200	52600
12.819_1.000_78	14500	40800	27400	38500	19400	78100	31300	35600
7.437_0.500_182	6090	4100	7320	3710	5050	5470	4450	6610
22.138_2.000_67	18900	42800	28400	42600	19500	32500	23000	53500
22.006_2.500_313	4500	5250	6680	4670	6180	5380	4300	5130
8.487_1.500_78	29600	143000	119000	144000	33700	153000	104000	194000
14.788_1.500_130	15900	35400	34500	46500	14400	66900	83300	84200
25.681_2.000_164	6710	9740	13700	11200	9700	10300	12100	35200
20.563_2.000_123	29700	129000	200000	246000	37000	139000	196000	250000
22.269_3.000_259	5220	5780	8140	4110	5260	4910	7230	5800
16.888_2.000_264	5660	5110	7570	5520	4990	10600	5310	6700
12.031_3.000_254	4500	5110	6170	5110	6780	5940	5560	4670
17.019_1.500_125	18300	87800	119000	137000	17600	137000	129000	179000
21.350_1.500_154	8290	13500	16500	18000	8390	10800	22200	50600
25.288_1.000_179	6880	5310	7520	20300	5540	5440	5590	7910
17.544_2.000_47	7450	23700	7790	11000	7920	7700	12100	8640
22.269_1.500_327	5270	6370	8160	5900	4840	4530	4850	4390
21.481_2.000_62	9110	18600	13900	14800	7770	21300	11200	32600
21.088_1.000_219	6670	6630	8360	10900	13400	5560	3770	9590
15.969_1.000_42	424000	3010000	3160000	4430000	454000	4100000	4820000	6000000
21.219_1.500_151	8080	30500	24700	50300	10800	37100	23000	78700

Annex 2 Averaged values of features for 1 mL of sample extracted for 30 min at temperature = 30, 40, 50, 60 and 75 °C (HiSorb)

Extraction temperature	**30 °C**	**40 °C**	**50 °C**	**60 °C**	**75 °C**
8.881_3_241	26200	36800	154000	208000	253000
9.931_2.5_209	9540	16400	14800	25900	33800
10.325_2.5_68	22700	62200	287000	511000	555000
10.325_3_94	6400	5670	6200	32500	107000
10.456_3_99	15300	27000	62700	46000	63200
10.719_2.5_178	5180	7560	5240	5680	5270
11.375_0.5_330	29600	81700	366000	628000	647000
12.688_2.5_185	13200	16600	44700	378000	828000
12.95_2_308	10300	10100	19200	13000	28400
13.213_2.5_42	33700	66400	66300	44200	36300
13.344_2_266	110000	243000	975000	1570000	1690000
13.475_1.5_80	15000	13400	21800	79300	257000
14.131_2.5_85	8070	10900	29100	128000	175000
14.788_3_184	8690	4280	6730	4790	4990
14.919_1_49	28500	40100	128000	197000	187000
14.919_2.5_81	16400	11600	20800	32000	147000
14.919_2_95	8000	10500	11300	25000	23000
15.181_2.5_57	4690	6710	6680	4530	5790
15.444_1.5_152	16000	29200	55100	108000	98600
15.706_2_46	5930	6820	8370	12800	36700
15.838_1.5_92	7540	13100	93300	83300	71800
15.838_2_42	6740	4350	5950	6950	6310
15.969_1.5_53	6440	4490	5920	29000	81000
15.969_1_121	104000	185000	534000	660000	647000
15.969_2_51	5390	4910	7140	4370	8050
16.1_1.5_64	9010	5170	5720	7390	7210
16.231_2.5_54	41800	97300	1420000	3640000	3930000
16.494_3_241	7740	10500	13600	52300	108000
16.756_1.5_239	891000	2460000	9490000	11300000	10800000
16.888_2.5_250	11900	8170	14300	15100	13600
17.019_1.5_64	18800	23200	34000	73000	121000
17.15_2.5_86	15900	22300	52900	76500	61900
17.281_2.5_95	4840	6650	6530	4320	6000
17.938_2_112	10800	7270	13500	13000	15900
18.069_1.5_67	87700	125000	378000	711000	751000

18.069_1_67	15900	17900	21100	42500	189000
18.069_2_109	63200	239000	879000	982000	802000
18.2_2.5_65	8020	4500	7460	5880	6220
18.331_1.5_42	6610	9990	54400	157000	276000
18.594_1.5_67	21200	39700	102000	138000	127000
18.856_1_97	8670	8310	31800	108000	97700
19.119_1.5_54	5440	8220	4200	4840	5690
19.25_2.5_120	8140	11300	12900	41700	104000
19.381_2.5_80	99400	129000	599000	761000	673000
19.644_2.5_109	9490	12300	21900	110000	161000
19.906_1_71	7700	4650	5650	4970	6160
20.169_2.5_149	50300	69900	214000	381000	446000
20.3_1.5_293	5700	8170	9850	23600	73500
20.3_2_142	9750	22900	39700	29200	23700
20.431_1.5_39	4550	6600	4890	7520	5460
20.694_1_175	10700	17600	22300	60100	83500
20.694_2.5_45	34500	53900	134000	134000	127000
20.825_2.5_57	6530	8620	12300	18300	58100
20.956_2.5_147	4860	7280	4880	4600	4210
21.088_1.5_137	5590	6380	22200	143000	241000
21.35_3_109	50000	60000	133000	187000	123000
21.481_2.5_134	5580	6280	11100	8370	12800
22.138_0.5_43	7880	4520	7140	5080	5810
22.4_1_331	19200	26700	30000	64100	88500
22.663_2_322	12400	764000	4250000	4720000	4100000
23.188_1.5_122	7080	9980	10400	11500	25100
23.188_1_94	4880	8690	4970	6250	5740
23.188_2.5_293	6160	9510	16400	31800	86100
23.188_2_146	14300	22400	64300	77900	67100
23.319_1.5_122	9450	9300	67600	280000	235000
23.319_1_51	5310	7630	5980	4470	4950
23.319_2.5_152	8610	8310	25400	58500	71500
23.319_2_91	7830	6900	9590	16700	122000
23.581_2_53	39400	53800	149000	145000	149000
23.713_0.5_66	6900	5090	5720	4790	5420
23.713_1.5_53	15700	16600	105000	184000	206000
23.713_2.5_348	6000	5560	6820	25700	69200
23.713_2_51	9480	11700	34200	193000	297000
23.844_1.5_82	8830	4540	5070	5290	6110

24.238_0.5_89	13100	21200	70400	110000	92300
24.238_1.5_78	19200	23000	40800	71100	127000
24.238_2.5_117	9720	6160	20400	15200	21100
24.238_2_77	5260	7720	6400	4970	4720
24.369_1.5_135	12700	14100	129000	266000	396000
24.369_1_135	34500	67800	205000	202000	181000
24.894_1_235	7400	7290	7420	13400	8170
25.025_1.5_284	6590	4530	5580	4870	4680
25.288_0.5_160	19500	23400	43100	148000	258000
25.288_2.5_93	55000	55400	105000	129000	110000
25.288_2_161	6280	10300	15900	19300	48500
25.419_0.5_92	6380	7920	5930	6930	5310
25.419_1.5_122	23400	19400	67200	114000	116000
25.419_1_122	12700	6560	13200	37900	88900
25.419_2.5_174	17900	27500	57000	48300	33900
25.419_2_275	7530	5800	6830	4690	4440
25.55_2.5_159	6830	9660	10700	24900	59400
25.681_2.5_79	9390	9790	41200	67200	70300
25.944_2.5_81	56900	83300	117000	96200	123000
25.944_2_81	6240	5430	6110	4990	6470
26.206_2_106	13800	12800	54700	120000	177000
26.6_2_53	8750	19200	93300	122000	86800
26.863_2_159	18800	13400	20200	46400	38000
26.994_1.5_159	95500	72000	85000	76100	100000
27.125_3_303	16300	23700	32700	86600	440000
27.388_2_120	10500	10300	30000	65900	76800

Annex 3 Averaged values of features for 1 mL of sample extracted 30', 3×10' and 2×30' at 60 °C for HiSorb, SPME PDMS and SPME DVB/CAR/PDMS ("Triphasic")

Tools	**HiSorb**			**SPME PDMS**			**SPME Triphasic**		
Extraction conditions	**1 × 30'**	**3 × 10'**	**2 × 30'**	**1 × 30'**	**3 × 10'**	**2 × 30'**	**1 × 30'**	**3 × 10'**	**2 × 30'**
9.406_1.000_163	7520	34300	30700	11500	12600	9240	82400	33800	36200
9.406_1.500_96	26100	21300	20900	14300	7970	16800	40000	29200	16900
9.538_1.500_304	5790	4060	6520	5940	7080	5360	4910	6640	4700
9.669_3.000_71	24900	54200	43000	15200	40300	27300	8430	21300	24400
9.800_2.000_45	292000	1310000	1120000	11300	13600	17900	14600	22400	15600
9.800_2.500_100	388000	1150000	1200000	7630	6960	10800	9230	12900	11900
10.325_2.500_63	40600	209000	185000	9610	7220	8810	8580	8820	4840
10.325_3.000_68	104000	94700	101000	10600	12600	9010	9660	8810	12600
10.456_2.000_50	62800	209000	242000	16100	18400	21600	18400	37200	21700
10.588_1.500_93	159000	158000	145000	8520	7800	5700	17100	73200	56800
10.719_2.500_94	343000	1430000	1060000	9250	58900	46700	147000	352000	163000
10.719_3.000_39	252000	2310000	1970000	99600	104000	116000	130000	1100000	209000
10.850_1.000_241	4550	5340	4900	6550	7050	3790	5950	5970	5630
10.850_2.500_87	11300	6850	8420	4400	6150	7220	7600	17800	10300
11.375_1.500_82	98600	248000	225000	11200	12700	15800	20000	162000	40500
11.375_2.000_82	151000	348000	304000	9150	11200	14100	14700	294000	95100
11.375_2.500_82	127000	197000	162000	7170	11800	11800	14900	181000	124000
11.638_1.500_95	187000	251000	185000	9230	32500	20300	97700	160000	179000
12.031_3.000_169	5850	5870	5640	5030	9690	6030	5240	6710	5000
12.425_2.000_71	35600	58500	77900	18200	52000	39700	16500	21500	30400
12.556_1.500_151	164000	169000	133000	6160	6840	4740	7640	11000	7490
12.688_1.000_135	35900	97500	58700	5210	6340	5610	5910	6260	9520
12.688_1.500_96	106000	172000	169000	14400	15800	14600	34700	141000	86400
12.688_2.000_151	92400	135000	77500	5850	5130	5910	6010	7200	12400
12.819_1.500_288	5650	6220	5840	5480	6680	5680	4900	6370	3810
13.213_2.000_94	198000	326000	314000	11000	14000	20800	21800	80000	36500
13.475_2.500_93	148000	276000	276000	11400	8130	9460	13600	51300	30300
13.475_3.000_51	191000	195000	207000	40300	38300	31700	54000	132000	129000
13.606_1.000_51	166000	144000	158000	34100	34200	39300	54300	103000	63100
13.606_3.000_57	74000	116000	121000	30500	68300	56600	22600	48200	48900
13.869_1.000_107	162000	172000	187000	13100	34600	27500	48100	99000	67100
14.525_2.500_108	145000	183000	178000	29400	35500	25400	52300	48300	66300
14.788_2.000_281	10600	11800	15800	8700	8320	7120	24500	27500	31300
14.919_2.500_49	77500	305000	290000	8430	9300	5330	17000	149000	150000

15.050_2.500_186	4250	5990	5730	8530	7830	5130	5360	5430	6070
15.050_3.000_106	218000	315000	367000	9270	10900	6660	142000	223000	261000
15.181_1.500_110	196000	300000	314000	8050	12100	5600	172000	211000	227000
15.181_2.000_110	205000	334000	321000	9870	10600	6160	167000	205000	224000
15.181_3.000_51	163000	143000	189000	21600	32100	28700	110000	154000	163000
15.444_1.500_264	5000	5810	5840	6320	5850	6960	5080	6260	11500
15.444_3.000_110	175000	215000	223000	5790	15000	8610	122000	172000	180000
15.575_2.000_110	161000	235000	224000	5540	12900	10900	111000	149000	140000
15.706_1.500_55	210000	626000	430000	21100	28600	20100	31900	112000	77900
15.706_2.000_41	799000	1640000	1500000	61900	61600	54500	71600	347000	192000
15.706_3.000_219	6840	7210	6400	12500	7800	5580	6680	6760	7210
15.838_1.500_40	650000	1810000	2260000	152000	156000	163000	153000	153000	159000
15.838_3.000_67	139000	170000	175000	12300	19300	13900	34100	116000	58700
15.969_1.500_81	1760000	3790000	3960000	17100	28600	23800	98300	366000	169000
15.969_3.000_329	5800	5500	4860	6450	6950	4430	4900	4870	6010
16.363_2.000_71	115000	185000	349000	12900	27500	24500	16700	40700	31100
17.150_2.500_332	5060	5900	5820	7070	4620	5490	6420	4840	4160
17.413_2.000_121	90400	139000	190000	14500	21800	18600	19000	40900	26100
17.544_2.000_68	43200	105000	88200	13700	13900	10200	18500	38100	24800
18.331_0.500_52	131000	122000	153000	13400	25000	19900	47700	103000	90300
18.331_2.000_314	5380	5580	4930	4540	5420	7690	4020	5650	5590
18.463_3.000_136	192000	152000	194000	9560	27400	14300	68400	152000	113000
18.856_3.000_239	5860	6050	4920	5910	6040	5740	4500	14600	7300
19.250_2.500_93	132000	193000	328000	8000	13300	10900	25300	47800	69400
19.381_3.000_53	177000	176000	183000	24700	24700	20500	62500	83700	106000
20.038_2.000_56	131000	182000	220000	17700	27000	19900	39300	67700	59900
20.825_2.500_252	5050	6190	6450	4910	8060	4150	6450	4700	5000
20.956_0.500_95	109000	216000	173000	9470	17900	14700	47100	91900	85900
20.956_2.500_137	32900	69400	108000	6650	6700	6830	12300	12500	26100
21.219_3.000_109	207000	191000	216000	11500	12500	9420	48200	118000	157000
21.350_2.000_94	112000	132000	182000	6160	13700	13000	30400	61400	74500
21.350_2.500_111	82600	132000	169000	6230	10200	7910	21300	48400	52600
21.481_1.500_109	154000	203000	195000	9480	20600	11400	47700	114000	113000
21.744_1.500_319	4720	4680	6110	4790	4460	6780	6230	6540	4660
22.269_2.500_62	13600	14700	13500	6150	11500	6500	6850	13200	21500
22.400_2.000_311	4520	4250	5490	5260	8270	5230	5370	5450	4350
22.400_3.000_341	25600	7870	5220	5390	5680	4920	4790	5960	5610
23.188_2.000_122	84000	156000	277000	8150	10000	6150	12900	14700	37500
23.188_2.500_263	4800	6800	4410	5020	7230	4640	7250	5570	5560

23.319_1.500_78	56500	98300	142000	11800	20400	18000	16100	25600	30400
23.319_2.000_63	47200	78400	153000	7620	9270	11100	10400	14400	34600
23.450_1.000_326	5800	4780	6020	4480	6320	7740	5790	4770	6520
24.106_2.000_37	23000	28400	31300	16400	15200	14700	8010	26700	13500
24.238_1.500_77	91000	227000	660000	11800	13900	14400	15600	20600	46900
24.238_2.000_77	154000	325000	867000	9680	12700	14500	17000	21700	51100
24.238_2.500_75	18400	50000	108000	7900	8130	7010	7070	8110	10700
24.369_2.500_135	227000	269000	270000	8350	7320	7960	10000	25300	86900
24.500_2.000_206	8160	10600	8490	6500	11900	5780	5360	5630	5300
24.894_0.500_284	5490	6170	6020	7090	6920	4800	4470	6120	6220
25.681_0.500_333	5280	4520	4310	6820	5470	4860	4770	7600	6520
25.681_2.000_295	4830	6130	5790	5030	7070	6370	4060	5520	5540
25.944_0.500_303	5420	5380	5240	7090	5680	6510	5080	4410	7390
26.469_2.500_322	5720	4980	5790	5350	6240	4820	6340	4230	7890
27.388_2.000_163	11200	14500	69300	6630	11700	5780	5030	10100	7190
27.388_2.500_163	14400	18200	88800	6280	8960	4870	7230	8510	6100
28.175_0.500_57	79800	69800	101000	27800	109000	34900	47600	40200	46700
28.175_1.000_85	16100	22500	24300	8680	31600	12400	15600	16000	19000
28.306_0.500_81	67600	83400	70500	15300	21400	15700	18300	20400	57600
28.306_2.000_279	5820	5210	6330	5530	8300	4850	4940	4790	6300
28.569_1.000_86	8000	7470	8160	5370	10300	5120	8300	7060	5650
30.931_1.000_255	4810	5760	4790	5360	5490	7910	5330	5780	6440
31.063_2.500_221	6430	6280	7070	8810	7400	4470	5800	5970	5690
31.588_0.500_303	5810	6350	5360	3800	6070	5850	5870	6140	5150
31.588_1.000_115	20700	115000	72100	8420	7670	6300	6530	8360	5830
31.981_2.000_206	7650	7160	10300	6610	7080	10300	5130	5370	15200
32.506_1.000_171	7310	7160	6380	5570	15800	8180	6400	5980	5650

Annex 4 Values of selected value for industrial coffee sample under different brand and packaging for HiSorb (1 mL 3x10' at 60°C) (PQN (median) normalised and log 10 transformed data)

brand	man	man	subt	subt	mag	mag	chia	chia	puis	puis	kiv	kiv	sum	man_puis	man_puis	man_subt	man_subt	subl_mok	subl_mok	dis_dk	dis_dk	equi	equi
packaging	**caps_bio**	**pack**	**caps_alu**	**pack**	**caps_alu**	**pack**	**caps_bio**	**pack**	**caps_alu**	**pack**	**caps_bio**	**pack**	**caps_bio**	**caps_bio**	**pack**	**caps_bio**	**pack**	**caps_alu**	**pack**	**caps_alu**	**pack**	**caps_alu**	**pack**
7.962_2.500_59	4.91	3.90	3.93	4.18	4.32	4.01	5.03	4.12	4.10	4.16	5.01	4.13	5.12	5.14	4.17	5.06	4.25	4.33	4.20	4.08	4.19	4.06	4.42
15.575_2.000_238	3.73	3.66	3.88	3.76	3.86	3.75	3.83	3.72	3.78	3.67	3.75	3.66	3.82	3.72	3.65	3.80	3.73	3.83	3.72	3.82	3.75	3.86	3.74
37.231_1.500_234	3.84	3.82	3.71	4.01	3.60	3.80	3.82	3.82	3.71	3.76	3.84	3.85	3.76	3.73	3.84	3.85	3.85	3.73	3.86	3.69	3.82	3.69	3.78
25.025_2.000_101	5.23	3.96	4.13	3.89	4.13	4.01	5.20	3.96	4.03	4.23	5.33	4.09	5.33	5.10	4.00	5.24	4.14	3.93	3.95	4.30	4.23	3.89	4.06
8.356_0.500_59	4.60	4.14	4.18	3.89	3.94	3.98	4.62	3.83	4.30	4.19	4.73	4.31	4.63	4.72	4.29	4.71	4.01	4.28	4.04	3.95	4.00	4.13	4.02
7.962_2.000_59	4.90	4.06	4.16	4.17	4.38	4.01	5.14	4.16	4.25	4.21	5.09	4.29	5.17	5.25	4.20	5.02	4.29	4.21	4.13	4.13	4.07	4.25	4.27
21.481_3.000_304	3.73	3.76	3.79	3.73	3.86	3.62	3.76	3.75	3.83	3.76	3.72	3.76	3.69	3.78	3.75	3.71	3.75	3.85	3.61	3.83	3.65	3.80	3.72
5.206_2.000_71	4.59	3.78	4.00	4.21	4.00	3.98	4.49	3.98	4.15	4.20	4.71	4.14	4.66	4.71	3.96	4.79	4.09	4.25	4.08	4.12	3.78	4.07	4.17
8.094_0.500_59	4.70	4.30	4.20	4.37	4.13	4.21	4.75	4.15	4.07	4.10	4.99	4.52	4.90	4.82	4.15	5.04	3.95	4.30	4.38	4.11	4.16	4.12	4.13
8.225_0.500_59	4.59	4.09	4.16	4.01	4.13	4.10	4.56	3.96	4.18	4.14	4.86	4.44	4.74	4.82	4.15	4.89	3.92	4.32	3.99	3.92	4.02	4.34	4.04
8.356_1.000_59	4.59	3.99	4.39	4.09	4.32	3.76	4.52	4.00	4.34	4.02	4.70	4.06	4.64	4.68	4.17	4.87	4.04	4.28	3.85	3.72	4.07	4.05	4.08
7.831_3.000_58	5.02	4.88	4.86	4.88	4.78	4.73	5.07	4.68	4.72	4.77	5.08	4.73	5.01	5.14	4.89	5.09	4.80	4.68	4.74	4.81	4.71	4.78	4.75
8.881_2.000_151	4.01	4.01	4.11	3.98	4.11	3.89	4.01	3.93	4.06	3.86	3.97	3.91	3.89	3.92	3.82	3.94	3.73	4.09	3.92	4.04	3.98	4.07	3.95
25.550_3.000_275	3.77	3.70	3.54	3.73	3.61	3.80	3.72	3.71	3.64	3.71	3.59	3.71	3.66	3.72	3.77	3.72	3.70	3.60	3.72	3.65	3.78	3.40	3.74
8.356_2.500_59	4.64	4.15	3.96	3.94	4.05	3.95	4.46	4.05	3.88	3.96	4.70	3.92	4.72	4.72	3.80	4.83	4.06	4.02	4.03	3.89	4.04	4.07	4.25
5.075_2.000_71	4.80	4.03	4.18	3.98	4.03	4.14	4.64	4.19	4.21	4.20	4.99	4.24	4.96	4.94	4.18	5.06	4.02	4.26	4.30	4.23	3.94	4.29	4.18
8.094_2.500_59	4.74	4.14	4.07	3.95	3.84	3.97	4.75	3.96	3.92	4.10	5.08	4.17	4.87	4.86	4.03	5.05	4.15	4.04	4.11	4.05	4.10	4.27	4.06

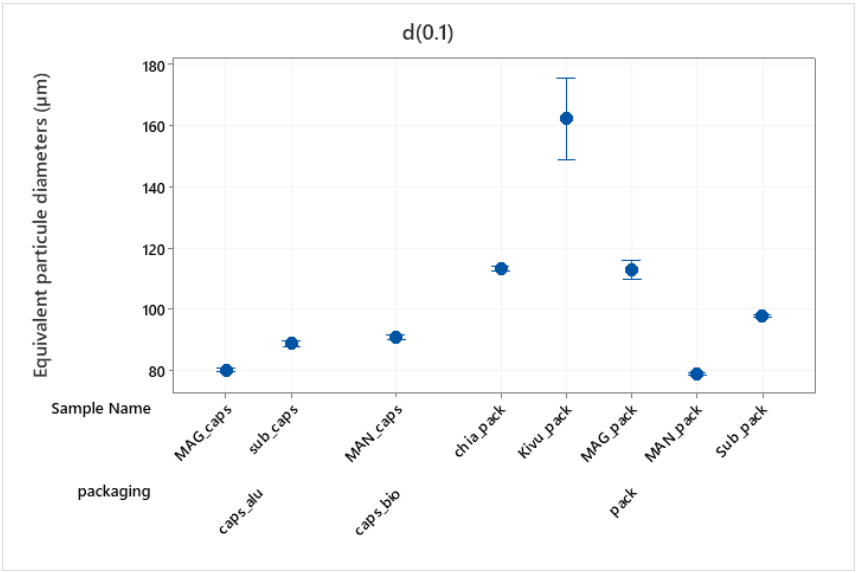
8.487_3.000_59	4.42	4.04	4.23	4.08	4.06	4.04	4.63	4.22	4.14	3.95	4.65	4.17	4.61	4.81	4.18	4.63	4.09	4.39	4.20	3.71	4.09	4.17	4.29
4.812_2.000_41	5.44	5.10	5.00	4.99	5.11	5.06	5.15	4.95	5.12	5.04	5.43	5.03	5.26	5.38	5.02	5.56	4.91	5.00	5.13	4.93	5.13	5.08	5.03
15.969_2.500_116	4.78	5.57	5.68	5.41	5.61	5.25	5.22	5.56	5.63	5.65	4.94	5.44	5.02	5.36	5.50	5.12	5.39	5.49	5.71	5.66	5.71	5.63	5.65
7.831_1.500_59	5.29	4.22	4.31	4.24	4.38	4.39	5.15	4.31	4.48	4.22	5.31	4.29	5.30	5.40	4.52	5.27	4.24	4.07	4.36	4.19	4.48	4.45	4.40
18.594_3.000_112	4.55	4.55	4.77	4.47	4.70	4.51	4.58	4.51	4.88	4.66	4.54	4.56	4.55	4.57	4.53	4.59	4.59	4.76	4.65	4.82	4.67	4.67	4.64
18.988_3.000_155	3.80	3.72	3.91	3.71	3.87	3.70	3.75	3.70	3.77	3.67	3.85	3.66	3.91	3.71	3.61	3.61	3.71	3.89	3.52	3.86	3.76	3.97	3.69

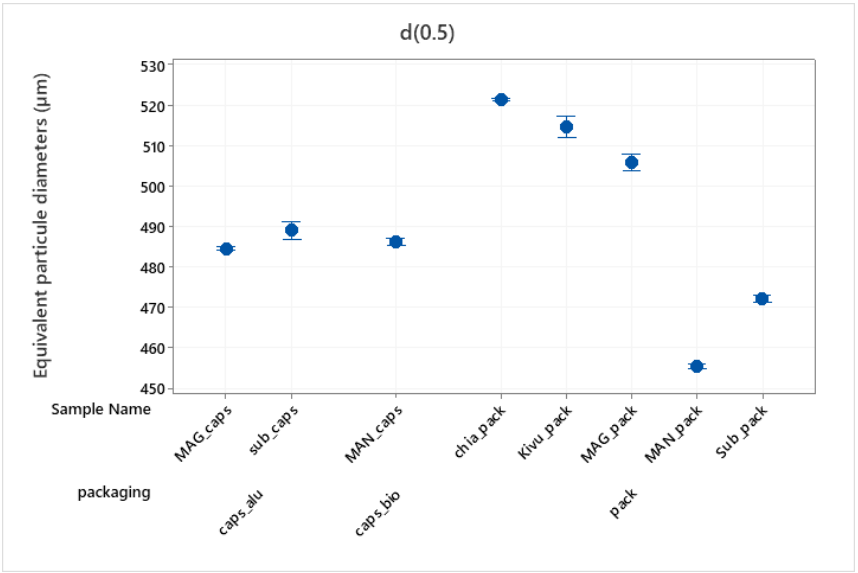
Annex 5 Values of selected value for industrial coffee sample under different brand and packaging for SPME DVB/CAR/PDMS (1 mL 3x10' at 60°C) (PQN (median) normalised and log 10 transformed data)

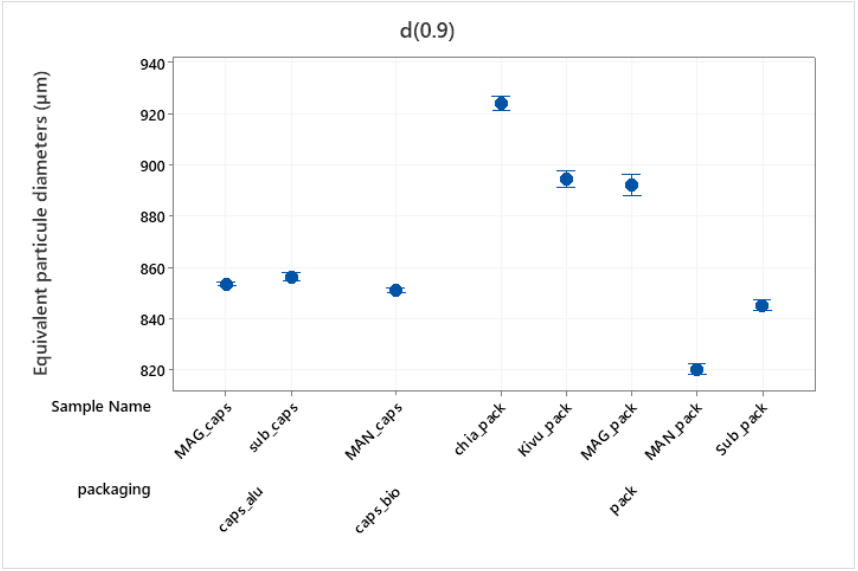
brand	man	man	subt	subt	mag	mag	chia	chia	puis	puis	kiv	kiv	sum	man_puis	man_puis	man_subt	man_subt	subl_mok	subl_mok	dis_dk	dis_dk	equi	equi
packaging	**caps_bio**	**pack**	**caps_alu**	**pack**	**caps_alu**	**pack**	**caps_bio**	**pack**	**caps_alu**	**pack**	**caps_bio**	**pack**	**caps_bio**	**caps_bio**	**pack**	**caps_bio**	**pack**	**caps_alu**	**pack**	**caps_alu**	**pack**	**caps_alu**	**pack**
9.538_1.500_271	3.80	3.72	3.57	3.73	3.65	3.77	3.74	3.84	3.68	3.73	3.75	3.80	3.80	3.68	3.73	3.77	3.76	3.70	3.76	3.63	3.82	3.60	3.79
36.313_2.500_346	3.69	3.68	3.67	3.82	3.65	3.75	3.69	3.81	3.61	3.77	3.69	3.76	3.84	3.88	3.69	3.91	3.71	3.67	3.86	3.60	3.69	3.57	3.84
8.094_3.000_59	4.75	4.09	4.16	4.00	3.99	4.11	4.54	3.80	4.36	4.12	4.60	4.08	4.59	4.63	4.01	4.54	3.66	3.70	3.92	3.91	4.02	4.02	3.92
25.156_3.000_101	4.35	3.76	3.59	3.78	3.87	3.71	4.35	3.92	3.98	3.72	4.26	3.74	4.35	4.18	3.76	4.37	4.02	3.78	3.77	3.64	3.77	4.11	3.76
8.094_1.000_59	4.53	3.85	3.75	4.09	3.91	3.91	4.59	4.13	3.98	4.28	4.54	4.06	4.42	4.76	3.99	4.61	3.98	3.96	3.91	4.15	4.22	3.87	3.98
7.962_2.000_59	4.73	4.20	3.86	4.13	4.14	4.06	4.76	4.03	4.11	4.00	4.91	3.81	4.83	4.82	3.88	4.90	3.90	3.96	4.12	3.92	3.78	4.01	4.13
8.225_1.000_59	4.38	3.95	4.24	3.92	3.74	3.86	4.45	3.94	4.07	3.87	4.43	3.88	4.33	4.53	3.96	4.61	4.05	3.97	4.00	3.78	3.88	3.91	3.91
8.225_1.500_59	4.38	4.01	3.92	4.01	3.83	3.68	4.43	4.00	3.93	3.98	4.38	3.96	4.32	4.31	3.85	4.70	3.85	3.80	4.03	3.84	3.81	3.89	3.82
33.163_1.000_139	3.74	3.76	3.91	3.78	3.91	3.81	3.82	3.57	3.94	3.89	3.66	3.80	3.85	3.83	3.70	3.76	3.85	4.00	3.82	3.89	3.81	3.88	3.84
12.425_2.000_177	3.71	3.70	3.49	3.85	3.62	3.72	3.79	3.74	3.64	3.73	3.81	3.76	3.81	3.73	3.68	3.87	3.66	3.53	3.72	3.39	3.67	3.65	3.76
8.881_3.000_91	4.21	3.83	3.67	3.94	3.67	3.87	4.03	3.96	3.97	3.80	4.02	3.78	4.03	4.05	3.84	4.10	3.84	3.84	3.93	3.81	3.87	3.74	4.01
28.306_2.500_335	3.85	3.76	3.76	3.69	3.78	3.77	3.85	3.77	3.71	3.70	3.83	3.78	3.79	3.85	3.69	3.84	3.62	3.66	3.69	3.54	3.78	3.68	3.77
27.781_2.500_91	3.85	4.27	4.12	4.27	4.31	4.24	3.73	4.43	4.18	4.20	4.01	4.20	4.08	3.92	4.30	3.91	4.38	4.08	4.43	4.18	4.31	4.13	4.31
8.750_2.500_59	4.28	3.82	4.12	4.16	3.89	3.80	4.26	4.00	4.11	3.86	4.28	4.01	4.32	4.49	4.10	4.32	4.03	3.93	3.97	3.93	3.97	4.06	3.88
8.487_3.000_59	4.17	3.88	4.01	4.03	3.94	4.07	4.29	3.74	3.88	3.81	4.11	3.93	4.18	4.30	3.86	4.40	3.98	3.84	3.94	3.89	3.83	3.98	3.97
33.163_3.000_186	3.75	3.83	3.73	3.77	3.72	3.77	3.81	3.86	3.60	3.76	3.65	3.77	3.64	3.76	3.80	3.78	3.86	3.69	3.85	3.65	3.91	3.71	3.80
20.825_2.500_78	4.50	4.69	4.56	4.59	4.63	4.67	4.28	4.55	4.64	4.64	4.30	4.67	4.54	4.46	4.76	4.45	4.69	4.59	4.63	4.65	4.73	4.80	4.71

7.437_1.000_115	3.62	3.49	4.08	3.72	3.95	3.71	3.85	3.78	3.89	3.81	3.81	3.64	3.78	3.65	3.75	3.81	3.82	3.92	3.82	3.90	3.75	3.87	3.79
19.250_2.000_81	5.01	5.16	5.13	5.15	5.07	5.17	4.97	5.19	5.23	5.20	5.05	5.19	5.12	5.04	5.15	4.94	5.17	5.10	5.32	5.23	5.22	5.27	5.23
12.031_1.500_59	4.05	4.22	3.99	4.31	4.00	4.02	4.11	4.27	3.90	4.13	4.26	4.31	4.09	4.28	4.01	4.13	4.18	3.92	4.20	4.00	4.06	3.91	4.12
35.000_2.000_84	3.85	3.99	3.97	4.14	3.88	3.96	3.87	4.07	4.12	3.90	3.79	4.00	3.81	3.82	3.87	3.71	3.92	3.96	4.05	3.92	4.02	3.98	3.98
26.731_1.000_326	3.87	3.81	3.65	3.73	3.71	3.75	3.84	3.71	3.54	3.87	3.74	3.81	3.78	3.81	3.84	3.81	3.84	3.61	3.83	3.61	3.75	3.68	3.77
13.475_2.000_325	3.79	3.72	3.65	3.77	3.67	3.80	3.82	3.85	3.57	3.76	3.79	3.83	3.75	3.81	3.73	3.73	3.75	3.56	3.72	3.66	3.74	3.70	3.72

Annex 6 Equivalent diameters distribution (bar represents the standard deviation)







Exploring the capability of the HiSorb probe for characterization of volatile and semi-volatile compounds in brewed coffee by GC×GC-qMS

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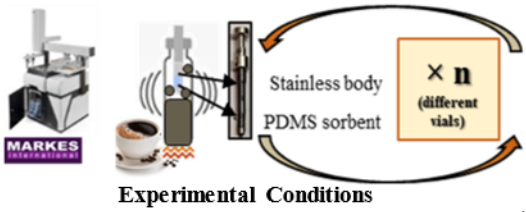
Introduction

Volatile and semi-volatile components are highly relevant to characterize different samples, among which food. These compounds contribute to the peculiar aroma profile of foods and are widely used to assess quality and authenticity. The analysis of volatile and semi-volatile compounds requires the sampling of the headspace (HS) above the sample, which, now a day is mainly performed using high-concentration -capacity HS (HCC-HS) approaches, such as solid-phase microextraction (SPME), stir-bar sorptive extraction (SBSE). More recently, HiSorb has been introduced to fill the gap between SPME and SBSE. In fact, it can be easily automated (as for SPME) but. present a significantly higher sorbent volume which allows a much higher trapping capacity.

The aim of this work was to exploit the potentiality of HiSorb for the analysis of volatile and semi-volatile from brew coffee and compared to the traditional triphasic SPME fiber more widely applied. Time and temperature conditions were carefully optimized, and the possibility of performing multiple cumulative trapping was investigated as well. The analyses were performed on a multidimensional comprehensive gas chromatographic system coupled with a quadrupole mass spectrometer (GC×GC-qMS) and equipped with a flow modulator

Material and Method

HiSorb



Experimental Conditions

- Extraction time: 10, 30, 40 and 60 °C at 50 °C using 1 and 4 mL of brewed coffee
- Temperature: 30, 40 ,50, 60 and 75 °C during 30 min using 1 mL of brewed coffee
- classical extraction vs multiple cumulative extraction (HiSorb and SPME)

GC× GC-qMS

^{1}D column: BPX-5MS 20 m × 0.18 mm × 0.18 µm
^{2}D column: BPX-50ms 5 m × 0.25 mm × 0.25 µm
Oven: 40 °C (3 min); 6 °C/min to 250 °C
Carrier gas: He; Flow ^{1}D: 0.6 mL/min ; ^{2}D 16 mL/min
Modulation period: 3.5 s; Flush time: 150 ms
2D Software: ChromSpace&ChromCompare + (SepSolve)

Results and Discussion

Expansion of an example chromatogram is shown in ***Figure 1***. A series a key compounds from coffee were selected to evaluate and optimize the conditions of extraction (time on ***Figure 2*** and temperature on ***Figure 3***).

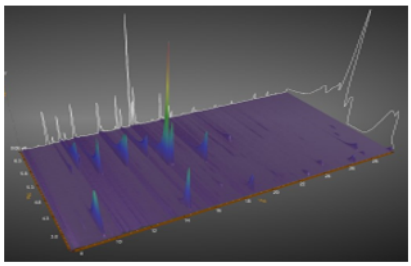


Figure 1*: Expansion of HiSorb extraction by GC×GC-MS*

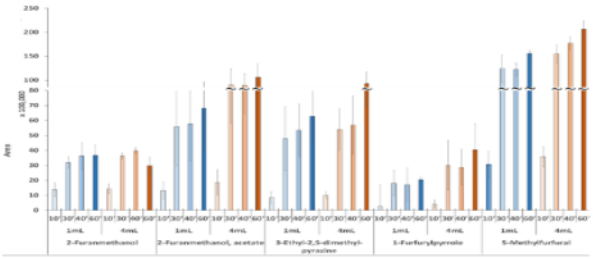


Figure 2*: Time profile at 50 °C using two different sample volumes (1 mL in blue and 4 mL in orange)*

Figure 4 show the potentiality of HiSorb compared to DVB/CAR/PDMS SPME using classical extraction and multiple cumulative extraction.

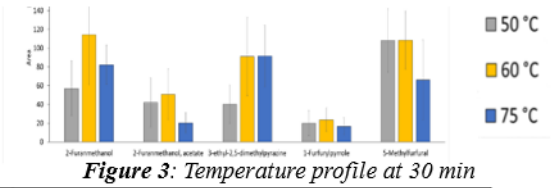


Figure 3*: Temperature profile at 30 min*

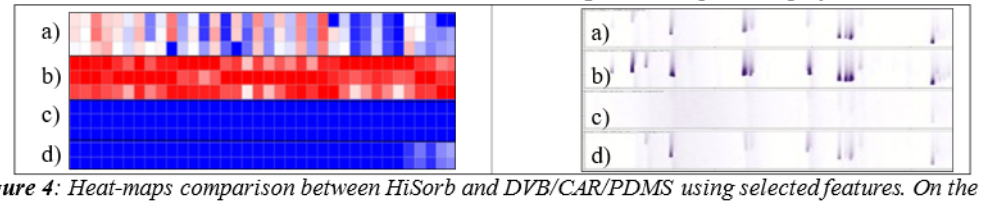


Figure 4*: Heat-maps comparison between HiSorb and DVB/CAR/PDMS using selected features. On the right, example of the corresponding chromatograms. (a: HiSorb 30 min; b) HiSorb 3 x 10 min; c) SPME 30 min; d) 3 x 10 min)*

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