Application Note

Headspace Analysis of Tea

Introduction

Tea is one of the most common drinks worldwide.

The analysis of the composition of the volatile organic compounds in the headspace of tea allows to classify the tea samples with respect to different criteria like storage age, quality grade or heritage.

Relevant olfactory volatiles in tea are e.g. 3-hexenal, 2,3,-butandione, 2-nonenal and monoterpenes. As the IMS technology (IMS = ion mobility spectrometry) is very sensitive for these kind of compounds, the FlavourSpec® (figure 1) was used for the analysis with respect to the differentiation of tea samples. Data analysis was performed using G.A.S. the software suite.

Figure 1: FlavourSpec® made by G.A.S. mbH

Experimental

All measurements were carried out with the FlavourSpec®, a GC-IMS equipped with an autosampler with headspace option. For an efficient separation an isothermal heated multi capillary column is used (OV-5, 5% diphenyl - 95% dimethyl polysiloxane, 20 cm, 0.2 µm). Ionisation source is Tritium (^{3}H). The activity (300 MBq) is below the threshold of 1 GBq so that no licence is required in all EURATOM countries. The measurement parameters are listed in table 1.

Figure 2: Different Samples of Tea

For analysis purpose 1g of each tea was transferred into a 20ml headspace vials. Each sample (Table 2) was put into two vials for reproducibility testing (index a and b). The vials were then placed onto the FlavourSpec® for automated processing and analysis. The instrument's analysis parameters were set to default (Table 1).

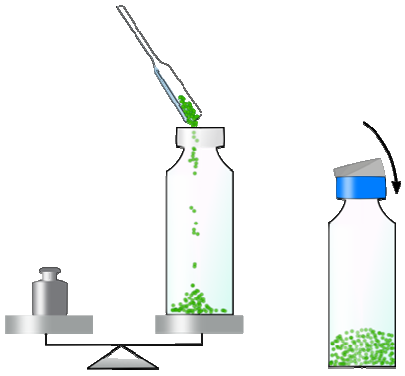

Figure 3: Sample preparation

Table 1: Experimental Parameters

FlavourSpec®		
	Polarity IMS	***Positive***
	T1 (IMS)	**45°C**
	T2 (MCC-OV5, 20cm)	**40°C**
	T3 (Injektor)	**80°C**
	E1 (Driftgas flow rate)	**150 ml/min Nitrogen 5.0**
	E2 (Carriergas flow rate, dynamic)	**25 ml/min** (step to150ml/min after 10min) **Nitrogen 5.0**
	Run time	**20 min**
	Average	**6**
Agitator		
	Incubation temperature	**90 °C**
	Incubation time	**10 min**

Results

Figure 4 exemplary shows the IMS chromatograms of two samples (I and II) of the analysed tea.

Every tea sample gives a characteristic peak pattern.

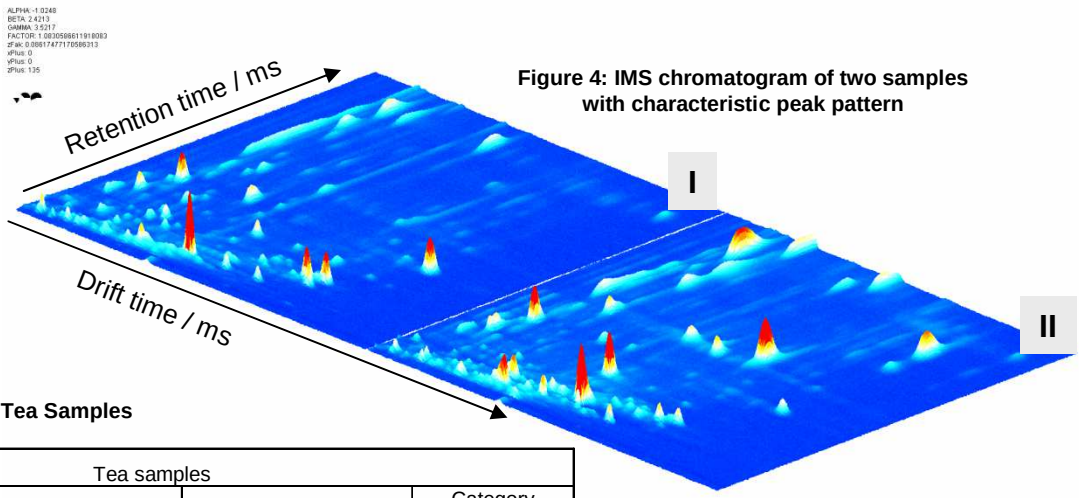

Figure 4: IMS chromatogram of two samples with characteristic peak pattern

Table 2: List of analysed Tea Samples

Tea samples			
NO.	Sample name	Grade	Category number
1	2012 Sichuan Green Tea	Special Grade	G-1
2	2013 Ningde Green Tea	Special Grade	G-4
3	2013 Anhui Green Tea	Special Grade	G-5
4	2013 Fuding Jasmine Tea	Special Grade	S-1
5	2013 Wuyi Black Tea	Special Grade	B-1
6	2013 Jianou Narcissus Tea	Level A	O-2
7	2013 Anxi Tie Guanyin Tea	Special Grade	O-12
8	2013 Anxi Tie Guanyin Tea	Level A	O-13
9	2013 Anxi Tie Guanyin Tea	Level B	O-14
10	Pu'er Tea-20 years	Level A	D-4
11	Pu'er Tea-15 years	Level B	D-5
12	Pu'er Tea-8 years	Level C	D-6
13	Pu'er Tea-5 years	Level D	D-7
14	White Tea	Level A	W-1

In order analyse/classify the samples the intensities of 101 signals were selected by marking evaluation areas on characteristic signals. The criterion for selection is the variation of individual signals in the chromatograms of the different samples. Thus only those compound signals are selected that vary in between the samples (Figure 5).

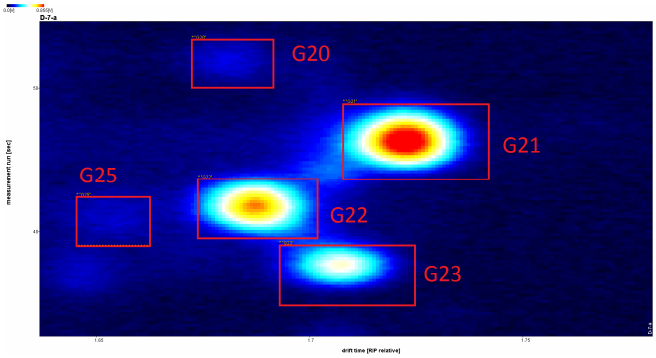

Figure 5: Exemplary clipping of chromatogram of sample D7 with indicated evaluation areas

The following figure gives an overview on the variation of the selected signals by plotting the selected evaluation areas for all samples using the *LAV plug-in 'Gallery-Plot'*. Note that for each tea two equal samples are analysed. The tea classes are obvious. Each of the reproducibility measurements validates the high reproducibility of the system (Figure 7).

The intensities of all signal peak areas automatically can be determined for all samples. Due to the huge number of areas and samples the presentation of the result has to be omitted here. Exemplarily, the intensities of the signal areas for 5 selected peaks (G28, S2, B8, D9 and W3) are presented in table 3.

Table 3: Automatically retrieved intensities of five selected evaluation areas (values given are in mV)

	"G28"	"S2"	"B8"	"D9"	W3
G-1-a	171	69	102	184	363
G-1-b	165	69	91	194	409
G-4-a	251	63	107	181	363
G-4-b	224	79	104	194	348
G-5-a	340	63	113	184	348
G-5-b	340	62	102	184	348
D-4-a	78	71	93	828	297
D-4-b	78	68	98	786	323
D-5-a	76	58	98	1000	297
D-5-b	79	61	100	847	313
D-6-a	90	64	100	652	318
D-6-b	94	64	93	630	323
D-7-a	106	61	93	557	297
D-7-b	99	73	93	595	292
O-12-a	914	55	139	238	681
O-12-b	1000	58	123	213	661
O-13-a	822	58	111	216	601
O-13-b	865	57	114	222	601
O-14-a	818	54	114	216	469
O-14-b	755	59	113	219	489
O-2-a	992	52	176	216	560
O-2-b	998	55	173	216	575
W-1-a	39	58	160	267	1000
W-1-b	40	62	141	248	979
B-1-a	208	59	1000	229	575
B-1-b	190	61	964	222	595
S-1-a	116	750	134	194	590
S-1-b	130	1000	132	197	606

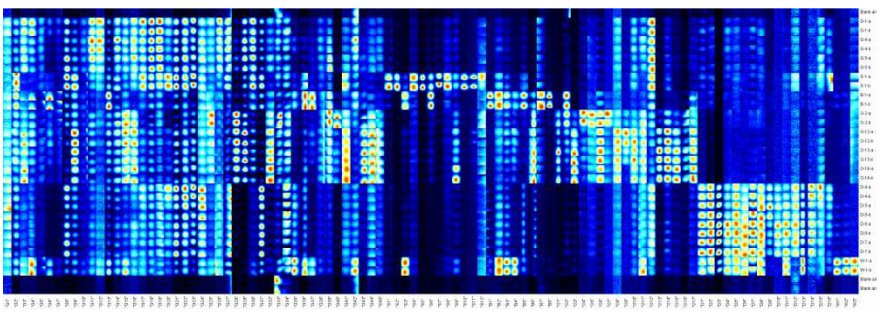

Figure 6: 'Gallery-Plot' of the evaluation area selection: Samples in rows, areas in columns

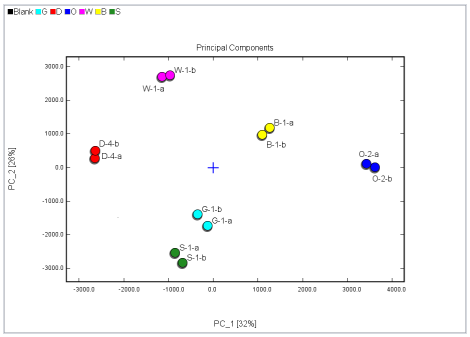

Figure 7: Principal Component Analysis (PCA) of selected samples based on the signal intensities allows a classification.

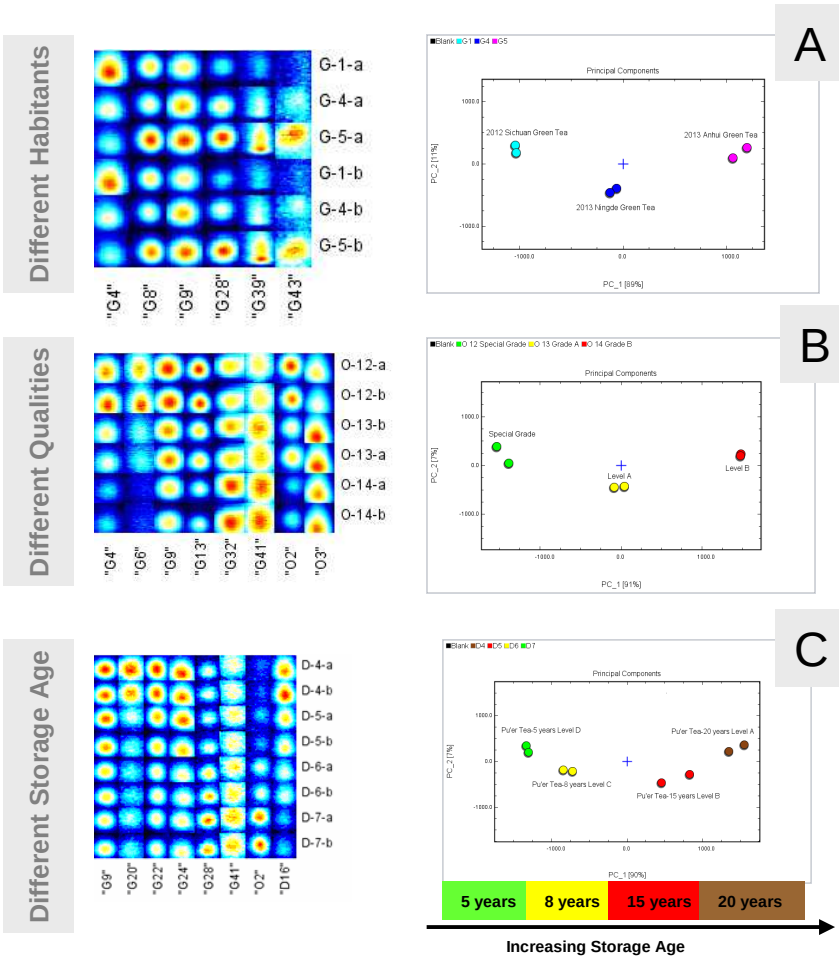

Figure 8: Gallery plot and PCA of sample set

Summary

The headspace composition of all tea samples was successfully analysed using a FlavourSpec®. The individual compounds are 2-dimensionally separated by gas-chromatography plus ion-mobility-spectrometry. A set of 101 individual signals representing headspace compounds was analysed in order to successfully determine variations between the tea samples.

A more detailed analysis was carried out within the different samples groups:

- Different habitants (green teas)
- Different qualities (Anxitie guanyin teas)
- Different storage ages (Pu'er teas)

Therefore, by visual data mining (using the G.A.S. 'Dynamic PCA' software) those areas are selected, that exhibit a significant difference in intensity.

The results are displayed in figure 8. The tea classes can easily be separated by occurrence/absence and concentration, resp., of specific volatile compounds.

- The green teas (*2012 Sichuan Green Tea*, *2013 Ningde Green Tea* and *2013 Anhui Green Tea*) from different habitats can easily be differentiated based on the FlavourSpec® analysis (figure 8A).

- The quality grade of the *2013 Anxi Tie Guanyin Tea* can precisely be determined by FlavourSpec® analysis (figure 8B).

- The storage age of the *Pu'er Teas* can clearly be determined by FlavourSpec® headspace analysis (figure 8C).

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