APPLICATION NOTE

Gas Chromatography
Ion Mobility Spectrometry

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Fast Monitoring of Tobacco Aroma Compounds and Additives

INTRODUCTION

Cigarette retail values in 2014 were worth over US$700 billion and over 5.6 trillion cigarettes were sold to more than one billion smokers worldwide. Tobacco and cigarettes belong to the world's most important convenience goods and sophisticated quality control regarding several aspects is a MUST. In a most recent application study it was shown that the FlavourSpec instrument of G.A.S. can give very detailed information on the headspace-composition. It was possible to demonstrate how the tobacco-flavour compounds migrate to the packaging material and to the cigarette filter material. Furthermore identification of a humidifying agent, which is added to keep a defined moisture level in the tobacco in order to extend the product shelf life, could be determined. Adding too much of the agent can induce an undesired smell of the product.

The experimental GC-IMS measurement method leads to detailed insights into the composition of the tobacco flavour.

Experimental Conditions

Table 1: GC Conditions	
Column	FS-SE-54-CB-1
Temperature	40 °C
Flow program 20 min	linear 2 → 100 mL/min
Carrier gas	N_2

Table 2: IMS Conditions	
Radiation source	Tritium
Temperature	45 °C
Flow rate	150 mL/min
Carrier gas	N_2
Mode	positive

Table 3: Sampling Conditions	
Incubation	Automated conditioning in FlavourSpec built-in agitator: 20 min @ 60 °C
Sample volume	500 µL (splitless headspace injection)
Syringe temperature	80 °C
Injection speed	0.5 mL/min

Table 4: Samples	
Samples	1. cigarette filter 2. cigarette part 1 3. cigarette part 2 4. packaging part 1 5. packaging part 2
Sample preparation	One piece of cigarette sample was cut into 3 parts: filter, cigarette part 1 (2) and part 2 (3). Further the outer (4) and inner (5) packaging material was analyzed. The samples were transferred into a 20 mL headspace vial, without any pre-treatment.

***Figure 1*: Samples as-is**

The cigarette and packaging samples were directly transferred into the headspace vial. A sample pre-treatment is not necessary.

Instrumentation

The FlavourSpec made by G.A.S. offers a gas-chromatographic separation and high sensitive detection via ion mobility spectrometry. The automated built-in agitator and coupled autosampler enable an efficient and fast workflow.

Related Information (Click Links)

- FlavourSpec
- Laboratory Analytical Viewer
- GCxIMS Library Search Software

Results

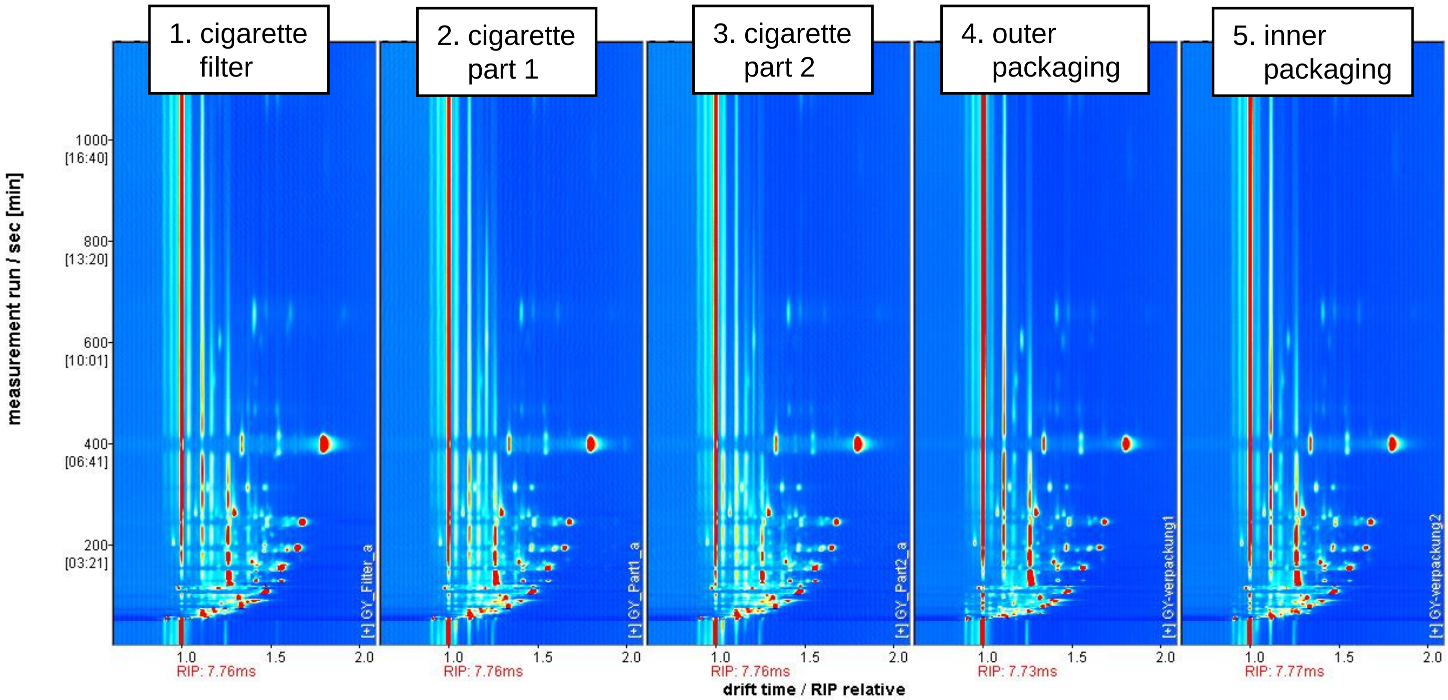

Figure 2: 2-dimensional representation of GC-IMS data. A cigarette filter, two pieces of the cigarette itself, the outer and inner packaging material were measured.

I. Migration and Identification of Flavour Compounds

The observed 2-D GC-IMS data clearly show a complex fingerprint for each sample (Figure 2), representing the rich headspace composition. The complex fingerprint of the headspace contains over 45 signals from volatile compounds, which were evaluated in the following step (Figure 3). G.A.S. has developed a sophisticated identification software module called 'GC-IMS Library Search' which allows to identify unknown compounds based on the NIST GC-retention indices and proprietary IMS drift times.

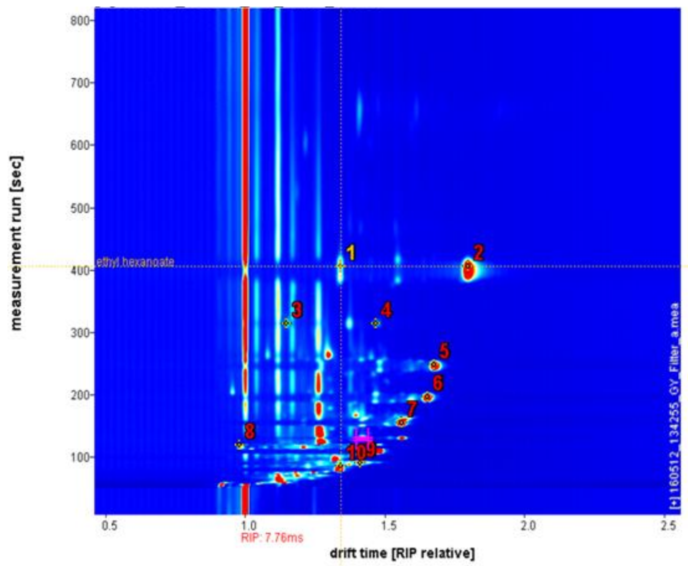

Figure 4: Identification of individual compounds acc. to GC-IMS Library Search (s. Table 5).

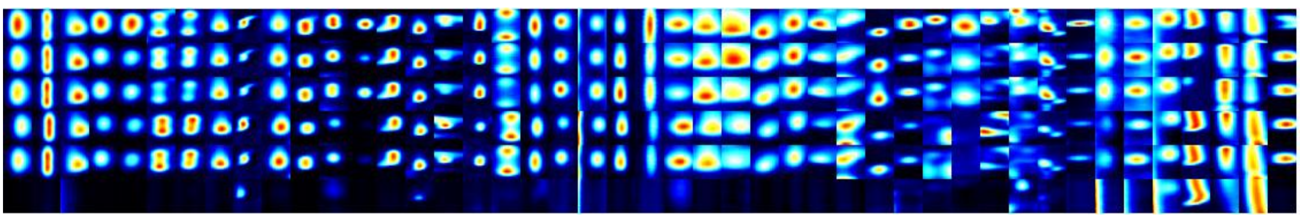

Figure 3: Gallery Plot of 45 selected signals of the tobacco's fingerprints derived by measurements of samples 1-5 and one additional measurements of propylene glycole (6).

GC-IMS: Innovative Quality Control on Tobacco

***Table 5:* Identified compounds in tobacco filter**

ID	Compound	CAS #	Formula	MW	RI	Rt/s	Dt/RIPrel
1	ethyl hexanoate	C123660	C8H16O2	144.2	1004.7	400.979	1.3435
2	ethyl hexanoate	C123660	C8H16O2	144.2	1004.7	400.979	1.8022
3	Benzaldehyde	C100527	C7H6O	106.1	948.1	315.139	1.152
4	Benzaldehyde	C100527	C7H6O	106.1	948.1	315.139	1.4689
5	ethyl pentanoate	C539822	C7H14O2	130.2	900.4	249.175	1.6801
6	Ethyl 3-methylbutanoate	C108645	C7H14O2	130.2	845.9	196.081	1.657
7	ethyl butyrate	C105544	C6H12O2	116.2	795.8	155.859	1.5613
8	Dimethyl disulfide	C624920	C2H6S2	94.2	728.1	120.463	0.9837
9	3-methylbutanal	C590863	C5H10O	86.1	651.9	91.503	1.4128
10	Ethyl Acetate	C141786	C4H8O2	88.1	627.3	85.068	1.3435

Table 5 contains several compounds of the tobacco filter, identified by the use of the 'GC-IMS Library Search'. Furthermore the fingerprint analysis reveals the presence of an eminent signal which has been identified as the humidifying agent, propylene glycole, which in the fragrance and flavour industry is often used as an universal solvent for flavours (Figure 3).

II. Cigarette Authentication by Detection of Flavour Additives

GC-IMS also allows to distinguish between different origins of markets. In Western markets, by regulation, it is prohibited to add flavour compounds to the tobacco leaves. The comparison of a cigarette with Western market origin compared to Asian market origin is given in Figure 5. Visually it is clear that the Western cigarette product generates a less concentrated headspace while the Asian cigarette product also reveals a much higher concentration of volatiles and a strongly pronounced presence of the humidifying agent (Figure 5).

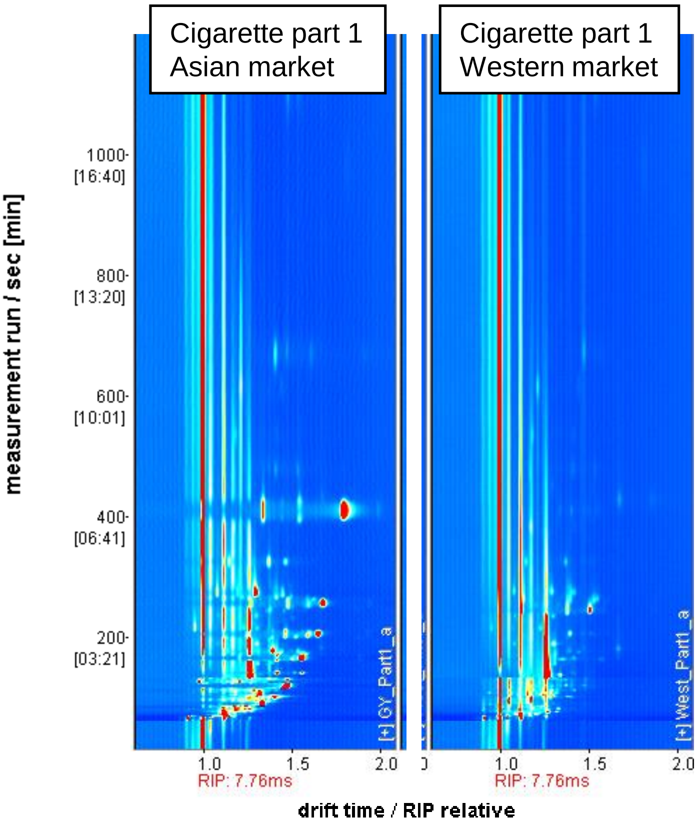

Figure 5: Fingerprints of cigarettes produced in Asian and Western market.

Conclusions

Quality control tasks for the tobacco products are various and very challenging analytical assignments. GC-IMS provides an innovative and highly sensitive approach and proves to demonstrate short analysis time, a simplified workflow without sample pre-treatment, and very short run-times between different measurements, whereby the following important subjects can be addressed:

- Determination of the aroma load/quality (blending) and stability
- Quality control of the finished product (cigarette/cigar)
- Observing product lifetime, eg. impact of outgasing of volatiles originating from packaging (inserts), printing inks, etc. onto the cigarettes

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