AN042N – Odor Quality Control of Hydroalcoholic Gel

Keywords

Hydroalcoholic gel, odor quality control, electronic nose, fast GC, Sensory ID

Application Benefits

- Easy comparison of overall odor quality of different batches of hydroalcoholic gel
- Investigation of defect origin

Introduction

The hydroalcoholic gel used as a hand disinfectant is an ideal, fast and effective solution against bacteria.

One of the main components of hydroalcoholic gel is ethanol, which can be produced via petrochemical process or from vegetal materials. Some off-odors can come from ethanol and it is very important to carefully control its odor quality to avoid any complaint from consumers on the final hydroalcoholic gel. This application note proposes to evaluate different qualities of ethanol from vegetal origin with a Fast Gas Chromatography Electronic Nose to check its sensory conformity.

Materials & Methods

Samples

Eight samples of ethanol representing 2 qualities were analyzed: 4 good (GS1 to GS4) and 4 bad (BS1 to BS4).

Fig. 1: Ultra Fast GC based HERACLES NEO Electronic Nose (Alpha MOS, France)

HERACLES NEO Smell analyzer

The HERACLES NEO Electronic Nose (Figure 1) is based on ultra fast gas chromatography. It features 2 metal columns of different polarities (non polar MXT-5 and slightly polar MXT-1701, 10m length, 180 µm diameter, *Restek*) in parallel and coupled to 2 Flame Ionization Detectors (FID). Two chromatograms are obtained simultaneously, allowing a sharper identification of the chemical compounds. It allows headspace or liquid injection modes.
The integrated solid adsorbent trap thermo-regulated by Peltier cooler (0-280°C) achieves an efficient pre-concentration of light volatiles and shows a great sensitivity.
With fast column heating rates (up to 480°C/min), results are delivered within seconds and the usual analysis cycle time is 8 minutes.

The electronic nose is coupled to an autosampler (PAL3 RSI, CTC Analytics) to automate sampling and injection.
The instrument is monitored by Alpha Soft software. In addition to classical chromatography functionalities, it provides chemometrics data processing tools such as sample fingerprint analysis for comparison, qualitative and quantitative models, quality control charts.

AroChemBase: Kovats Index library for chemical & sensory characterization

The HERACLES NEO e-nose is additionally equipped with AroChemBase module (Alpha MOS, France) that can be used within AlphaSoft E-Nose software. It consists of a library of chemical compounds with name, formula, CAS number, molecular weight, Kovats retention Index, sensory attributes and related bibliography. It allows pre-screening the chemical compounds and giving sensory features by directly clicking on the chromatograms' peaks.

Smell analysis

Analytical conditions

The analytical parameters optimized for this analysis are described in Table 1.

Parameters	
Sample mass	1 g + water (2mL)
Vial	20 mL
Acquisition duration	110 s
Incubation	40°C (20 min)
Injection volume	5 mL

Table 1: HERACLES NEO e-nose analytical parameters

A standard mixture of n-alkanes (n-pentane to n-hexadecane) is used to calibrate the system, to allow retention time conversion into Kovats indices.

Volatile profiles

The volatile profile of 2 ethanol samples of different qualities is displayed on the chromatograms (Figure 2): significant differences in peak proportions can be observed.

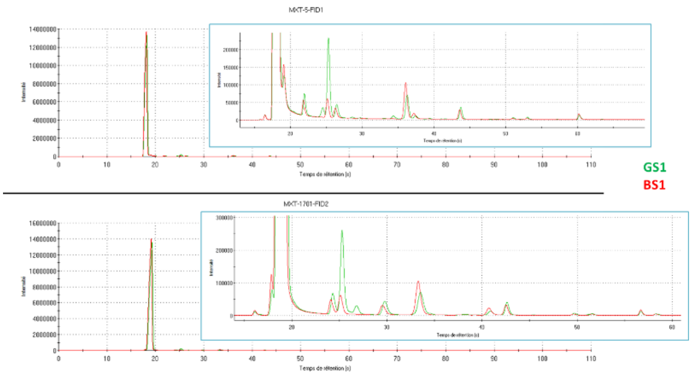

Fig. 2: Heracles NEO e-nose volatile profiles of ethanol samples

An odor map based on Principal Component Analysis can be generated using the most discriminant volatile compounds (Figure 3). The 2 qualities of ethanol are clearly discriminated.

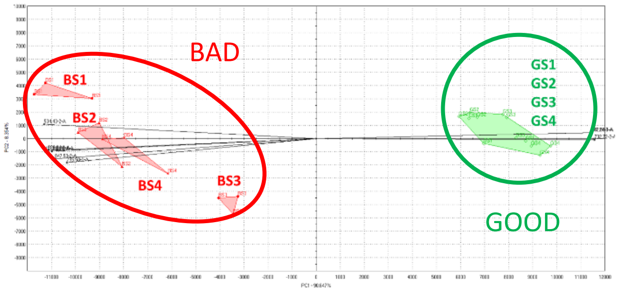

Fig.3: Odor map of ethanol samples (good and bad) based on a Principal Component Analysis (PCA)

Quality Control: Sensory ID model

AlphaSoft has several quality control models to assess the sensory quality and ensure conformity to a reference. The Sensory ID model is a patented method for global processing of chromatograms without the need for peaks integration. It gives an accurate and complete sensory fingerprint by detecting if a peak is outside the acceptance area, if a new peak is appearing or is missing and if peaks have lower or higher intensity (Figure 4).

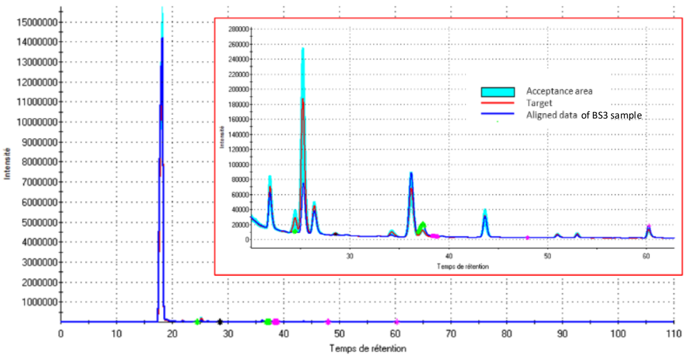

Fig.4: Target chromatogram and acceptance area (blue) for reference samples on MXT-5 column. Projection of BS3 sample

This model allows an efficient tracking of chemical and sensory defects that can occur during production, with easy-to-understand pass/fail results. In this study, good batches GS1, GS2, GS3 were used as reference (Figure 5). Other samples were then projected as unknown.

The acceptable area is displayed in green, if a sample is projected inside it is considered as conform (pass) to the reference whereas outside, samples are considered as non-acceptable (fail).

The Sensory ID model confirms that bad batches are outside the conformity area.

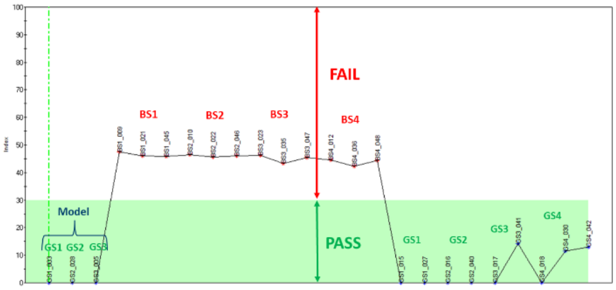

Fig.5: Sensory ID card of ethanol samples analyzed with Heracles e-nose

Investigation of Chemical Composition of the Odor

The nature of volatile compounds involved in the aroma was investigated using AroChemBase.
Table 2 gives a list of the main molecules found in ethanol samples and Figure 4 gives the proportion of some of these molecules in the different ethanol qualities.

Conclusion

HERACLES NEO e-nose can assess accurately and thoroughly the odor profile and provide quality control models for ethanol and hydroalcoholic gel testing. It allows to investigate the origin of a sensory defect to help improve production process.

This study suggests that HERACLES NEO e-nose can be a valuable decision tool within the alcohol and hydroalcoholic gel production area, with many **applications in product development or quality assurance**:

- **Sensory control** of raw materials or in final product
- **Investigation of consumer claims**
- **Detection of traces of contamination**
- **Influence of process change on sensory quality.**

Comparison with target peak intensity in SensoryID model — Acceptable | Lower | Higher | Absent

RT MXT5 (± 0.1s)	RT MXT1701 (± 0.1s)	RI MXT5 (± 30)	RI MXT1701 (± 30)	Possible matching compound	Sensory attribute (s)	BS1	BS2	BS3	BS4
16.5	16.1	448	485	methanol	*Alcohol, pungent*				
19.1	17.9	504	535	furan	*eEher*				
24.5	26.7	607	702	2-butanol	*Pleasant, strong odor, sweet, wine*				
25.2	25.2	617	680	Ethyl acetate	*Ether, fruity, orange, green, solvent*				
26.4	29.7	630	732	2-methyl-1-propanol	*Alcohol, fusel, glu, mold, stale*				
32.8	39.3	706	832	3-pentanol	*Fruity, green, hazelnut, oily*				
34.2	34.2	716	782	methyl metacrylate	*Acrid, aromatic, fruity*				
36.1	33.4	734	770	2-methoxyfuran					
37.2	40.8	745	847	3-methyl-1-butanol	*Alcohol, bitter, burnt, cheese, fermented, onion*				
37.2	40.8	745	847	2-methyl-1-butanol	*Banana, butter, fusel, malt, ripe onion, winy*				
47.9	47.0	850	914	ethyl 2-methylbutyrate	*Apple, blackberry, cognac, sour, sweet*				
51.0	49.7	882	947	isoamyl acetate	*Banana, fresh, solvent*				
52.5	51.6	898	970	butyl 2-propenoate	*Sour (pungent)*				
60.2	56.7	999	1042	psi-cumene	*Sour (pungent)*				
60.2	58.3	999	1070	butyl butanoate	*Banana, cherry, fruity, green, sweet*				
65.8	59.7	1085	1089	gamma-terpinene	*Citrus, herbaceous, oily*				
66.9	60.5	1101	1101	undecane	*Alkane, fusel*				
72.4	66.1	1201	1201	dodecane	*Alkane, fusel*				
86.3	80	1502	1504	pentadecane	*Alkane, fusel, sweet*				

Table 2: main compounds detected in ethanol samples and indication on peak intensity vs target quality