Objective

Entering the composition of many processed foods as a base ingredient, the wheat must be of high and constant quality. The presence of tainted wheat can indeed contaminate large batches of other raw material in storage or even prevail into the finished product if not removed from the process. The evaluation of wheat contamination is commonly carried out by visual assessment and human smelling at reception of raw materials. An instrumental analytical method can significantly simplify and improve usual quality control process.
This application note describes the analysis of wheat and the detection of off-flavor with an electronic nose.

Equipment

HERACLES Flash GC Electronic Nose

The HERACLES Electronic Nose (Alpha MOS, France – Fig. 1) is based on the technology of ultra fast chromatography. It features two metal columns of different polarities (non polar RXT-5 and slightly polar RXT-1701, length = 10m, diameter = 180µm, Restek) mounted in parallel and coupled to 2 Flame Ionization Detectors (FID). Therefore, 2 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 (0-260°C) achieves an efficient pre-concentration of light volatiles and shows a great sensitivity (in the pg range).
With fast column heating rates (up to 600°C/min), results are delivered within seconds and the analysis cycle time is around 5 to 9 minutes.

Fig. 1: Ultra Fast GC based HERACLES Electronic Nose

The electronic nose is coupled to an autosampler (HS 100, CTC Analytics) to automate sampling and injection.
The instrument is operated through Alpha Soft software. In addition to classical chromatography functionalities, it provides chemometrics data processing tools such as sample fingerprint analysis and comparison, qualitative and quantitative models, quality control charts.

AroChembase: Kovats Index library for chemical & sensory characterization

HERACLES e-nose was 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.

Samples & analytical conditions

Eight samples of wheat were analyzed with HERACLES electronic nose.

Table 1: Sample list

Code	Quality	Info
GA	Good	Whole
GB	Good	Whole
GC	Good	Whole
GD	Good	Whole
TE	Tainted	Ground
TF	Tainted	Whole
TG	Tainted	Whole
TH	Tainted	Whole

Each sample has been ground using a coffee grinder for 15 s before analysis on Heracles e-nose.

Table 2: Heracles analytical conditions

Parameter	Value
Sample quantity	5 g ± 0.01 in a 20mL vial
Headspace generation	20 min at 90°C
Syringe temperature	100°C
Injected volume	5000 µL
Injection speed	100 µL/s
Injector temperature	220°C
Injector vent	30 mL/min
Trap temperature	60°C
Trap pressure	60 kPa (2s) - 0.25kPa/s - 90 kPa
Trapping time	70 s
Trap desorption	240°C
Columns temperature program	60°C (2s) to 80°C by 1°C/s then to 280°C (0s) by 2°C/s
FID temperature	280°C
Acquisition duration	120 s
Time between 2 analyses	9 min

Chromatograms

The comparison of chromatograms (Fig. 2) obtained with HERACLES e-nose shows significant differences in terms of VOCs concentration between good and bad batches of wheat. Several volatile compounds have been detected at much higher concentration in tainted wheat than in good samples.

Chemical Characterization

Using the AroChemBase software and Kovats indices obtained on MXT-5 & MXT-1701 columns, indications about chemical composition can be determined.
The main volatile compounds are listed in table 3. Most of them correspond to aldehydes and alcohols that are related with fatty acid oxidation and microbial metabolism.

Odor map

This odor map (Fig. 3) obtained by selecting the most significant chemical compounds, shows a clear separation of tainted samples from good samples, according to their volatile compounds composition.
Geosmin together with alcohols, aldehydes and alkanes are considered as discriminant compounds explaining the organoleptic differences.

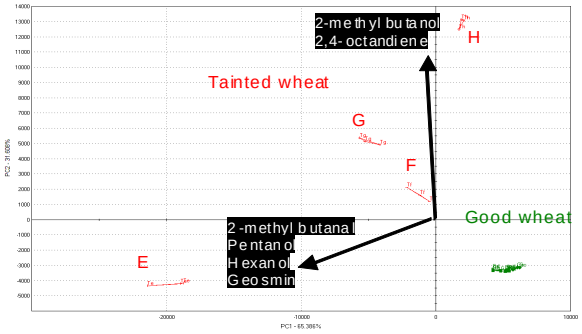

Fig. 3: Principal Components Analysis of wheat obtained with HERACLES e-nose measurements

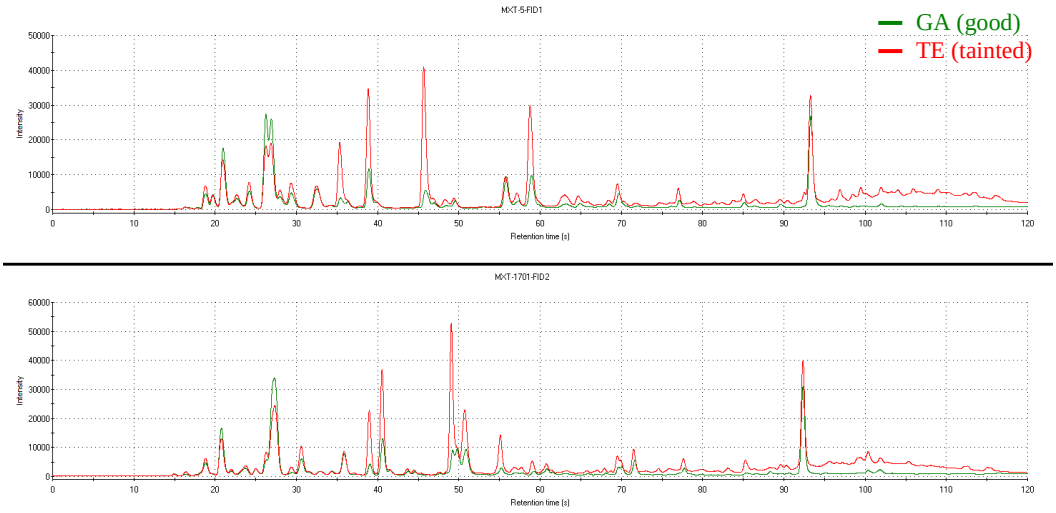

Fig 2: Chromatograms of 2 batches of wheat (GA & TE) on the 2 columns of HERACLES

*RT MXT-5 (±0.1s)	*RT MXT-1701 (±0.1s)	**RI MXT-5 (±20)	**RI MXT-1701 (±20)	Possible compounds	Sensory attributes
24.2	26.39	623	731	2-methyl propan-1-ol	Alcoholic, winey
26.2	27.2	654	744	3-methyl butanal	Green, malty
26.9	27.2	664	744	2-methyl butanal	Green, malty
29.3	30.6	701	788	Pentanal / 2,3-pentanedione	Butter, caramelized
32.6	35.9	735	845	3-methyl butan-1-ol	Alcoholic, fermented
32.6	37.7	736	864	2-methyl butan-1-ol	Winey, balsamic
35.5	38.9	766	877	Pentanol	Fruity, green
38.8	40.5	804	894	Hexanal	Fatty, green
40.9	33.6	827	824	2,4-octadiene	Glue, warm
45.7	49.1	870	980	Hexan-1-ol	Floral, green
55.8	49.7	969	985	β-pinene	Green, turpentine
58.8	50.8	999	996	n-decane	Alkane
64.7	56.6	1063	1058	2-methyl decane	-
93.2	92.2	1412	1499	Methyl undecanoate	Brandy, winey
96.5	95.3	1461	1546	Geosmin	Beet, earthy

Table 3: Extract of possible volatile compounds identified by their Kovats indices in the headspace of wheat
*Retention Time **Retention Index (Kovats Index)

Quality control model

A quality control model (SQC algorithm) using good samples as the reference has been built (Fig. 4). This model shows that all tainted samples are out of range.

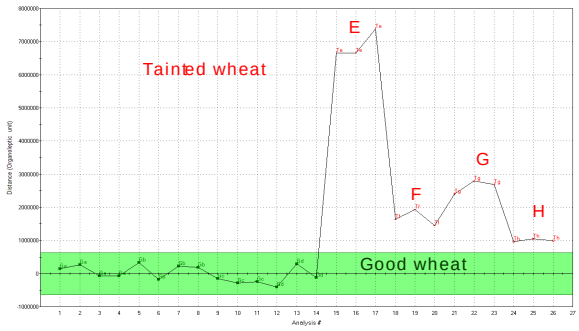

Fig. 4: Statistical Quality Control (SQC) Chart model with good samples as reference

Conclusion

The Heracles e-nose has proved to be a very powerful tool to rapidly assess the sensory quality of wheat.

The AroChemBase software indicates that the concentration of several alcohols is correlated with the off-odour. These molecules probably originate from microorganism metabolism that is responsible for wheat decay.