Objective

Coffee is one of the major commodities of agricultural origin traded on international markets.
The organoleptic quality of coffee is very important for consumers.
This application note describes the analysis of the sensory features of coffee and the building of a quality control model for routine evaluation 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

Ten batches of coffee stored under different conditions (stressed or not) were analyzed with HERACLES electronic nose.

Table 1: Sample list

Sample	Quality	Storage
Ga	good	reference
Gb	good	reference
Gc	good	reference
Gd	good	reference
Ge	good	reference
Gf	good	reference
Ba	bad	sample stressed
Bb	bad	sample stressed
Bc	bad	sample stressed
Bd	bad	sample stressed

Table 2: Heracles analytical conditions

Parameter	Value
Sample quantity	0.5 g±0.05 in a 20mL vial
Headspace generation	20 min at 70°C
Injected volume	5000 µL
Trap temperature	40°C
Acquisition duration	110 s
Time between 2 analyses	9 min

Chromatograms

*RT MXT-5 (±0.1s)	*RT MXT-1701 (±0.1s)	**RI MXT-5 (±20)	**RI MXT-1701 (±20)	Possible compounds	Sensory attributes
16.8	16.1	448	487	Methanethiol	Cheese, fishy, garlic
17.5	18.5	463	557	Ethanol	Alcohol
19.3	19.8	499	595	Acetone	Solvent, acetone
22.2	22.3	557	636	Butanal	Malty, chocolate
23.7	25.7	589	689	2,3-butanedione	Butter, caramelized, creamy
28.1	39.5	643	829	Carboxylic acid	-
30.8	34.0	674	776	Pent-1-en-3-ol	Butter, green, milky, pungent
32.8	35.8	697	794	Pentan-2-ol	Fruity, green
37.8	40.5	743	838	3-penten-2-one	-
39.7	42.3	760	855	2-pentanal	Fruity, green, oily, soapy
47.7	49.5	835	926	Ethyl 2-methylbutyrate	Fruity, green, sweet
48.7	55.4	844	987	Furfural	Almond, bread, sweet
50.9	58.7	866	1027	2-furanmethanol	Bread, caramelized, creamy
56.7	68.7	925	1159	2-furanone	-
61.3	66.6	976	1129	2,4-heptadienal	Fired

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

The comparison of chromatograms obtained with HERACLES instrument (fig. 2 & 3) showed slight differences of volatile profiles between coffee batches of the two qualities (stressed or not).

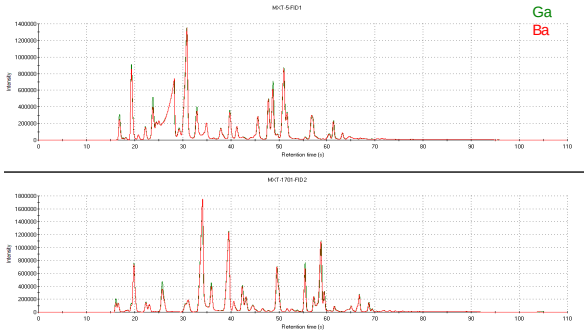

Fig. 2: Comparison of chromatograms of 1 good quality coffee batch and 1 coffee batch stored in stressed conditions

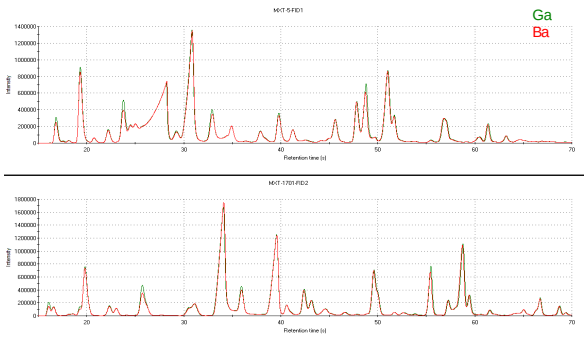

Fig. 3: Zoom on chromatograms of 1 good quality coffee batch and 1 coffee batch stored in stressed conditions

Odor map

A general odor map was established (fig.4) upon selecting the most important chemical compounds on HERACLES e-nose chromatograms.
This odor map, based on Principal Components Analysis (PCA), shows a clear separation of stressed samples from good samples, according to their volatile compounds composition.

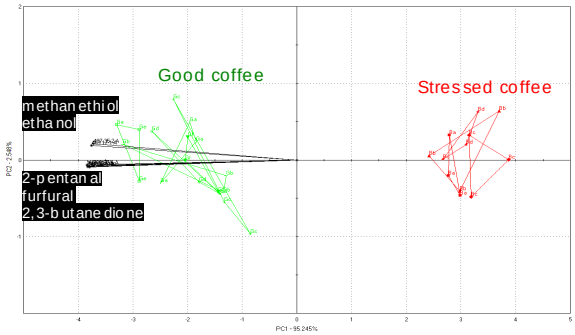

Fig. 4: Principal Components Analysis of coffee obtained with HERACLES e-nose measurements

Chemical characterization

Using the AroChemBase and Kovats indices on RXT-5 & RXT-1701 columns, indications about the chemical composition could be determined.
The main volatile compounds are listed in table 3, most of them correspond to alcohols, aldehydes and ketones. The compounds responsible for the discrimination between qualities have been identified as methanethiol,

ethanol, 2,3-butanedione, 2-pentanal and furfural.

Quality Control Chart

A quality control model based on Statistical Quality Control (SQC) algorithm was built (Fig.5) using good samples as the reference quality.
All bad batches are projected out of the green area and considered as non-conform to the reference quality.

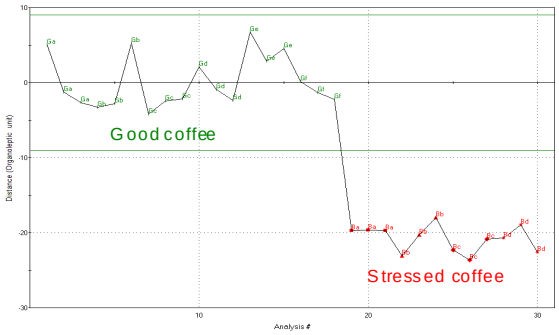

Fig. 5: Coffee quality control chart obtained with HERACLES e-nose measurements

Conclusion

This application note shows that HERACLES electronic nose can be successfully employed to control the quality of coffee samples and detect aged products.
Thus the e-nose can be a very powerful tool to rapidly assess the sensory quality of coffee batches upon receipt and to select suppliers.