

ION MOBILITY SPECTROMETRY: A RAPID TOOL TO ASSESS EGGS FRESHNESS

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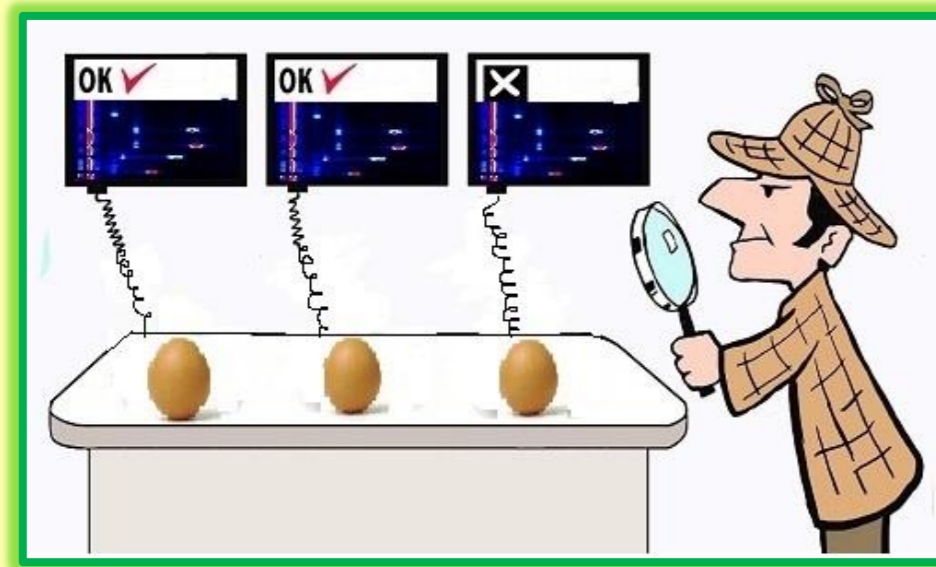
OVERVIEW

- PURPOSE:** develop an easy and rapid method able to assess eggs freshness, easy to use also in the production plants
- METHOD:** GC coupled with an Ion Mobility Spectrometer. High sensitive and reproducible volatile fingerprints. Marker compounds identified with SPME-GC-MS technique
- RESULTS:** several spots highlighted as freshness markers. The chemometric data evaluation identifies clear samples clustering according to the time spent at room temperature. Subsequent compounds identification and GCxIMS spot correlation.
Total analysis time from sampling to the result: approx. **45 minutes**.

INTRODUCTION

In the last decade, the attention on the authenticity and quality of food commodities increased for commercial and safety reasons and that is why, from both industries and research institutes, the demand for the development of rapid methods able to detect frauds increased as well.

Eggs, mostly in the egg products form, are largely used for the creation of different industrial products (egg pasta, bakery etc.) and their freshness is a crucial step for the production of safe and high quality commodities; however, there are not several rapid ways able to assess if egg products are as fresh as declared. In this study, a rapid GC-IMS method is presented as a possible way to quickly solve this problem.



METHODS

SAMPLE PREPARATION: 10 drops of fresh eggproduct directly transferred in a headspace vial. Samples were stored at room temperature and the same sample preparation was repeated after 24h, 48h, 72h and 144h from the receipt of the batch.

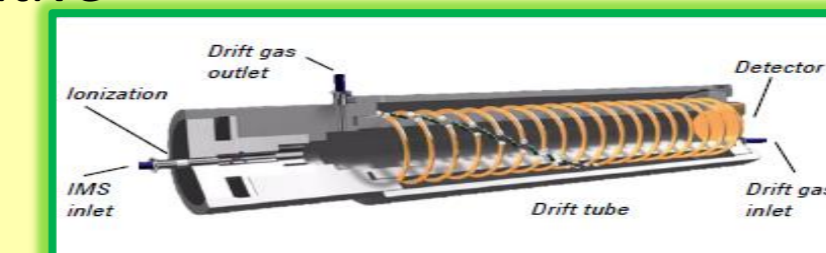
For each time point, three sample preparations were executed

INSTRUMENT: FlavourSpec® (Gas Dortmund – Germany)

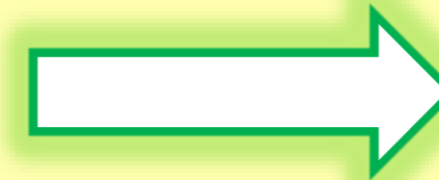
ESSENTIAL PARAMETERS

GC
Column: SE-54-CB-1 15m x 0.53 mm d_i 1 μm @ 40 °C
Incubation time: 20 min @ 40 °C
Injection volume: 0.5 ml
Carrier gas: Nitrogen
Flow gradient: 2 ml/min for 5 mins and then increased to 70 ml/min in 20 min
GC Runtime: 30 min

IMS
Drift gas: Nitrogen
Drift time: 30 ms
Drift gas flow rate: 150 ml/min
Drift tube length: 9.8 cm
Drift Voltage: 5kV
IMS Temperature: 45°C
Ionization mode: positive



COMPOUNDS ID



SPME-GC-MS



SAMPLE PREP.: 2 g of sample weighed directly in the Headspace vials

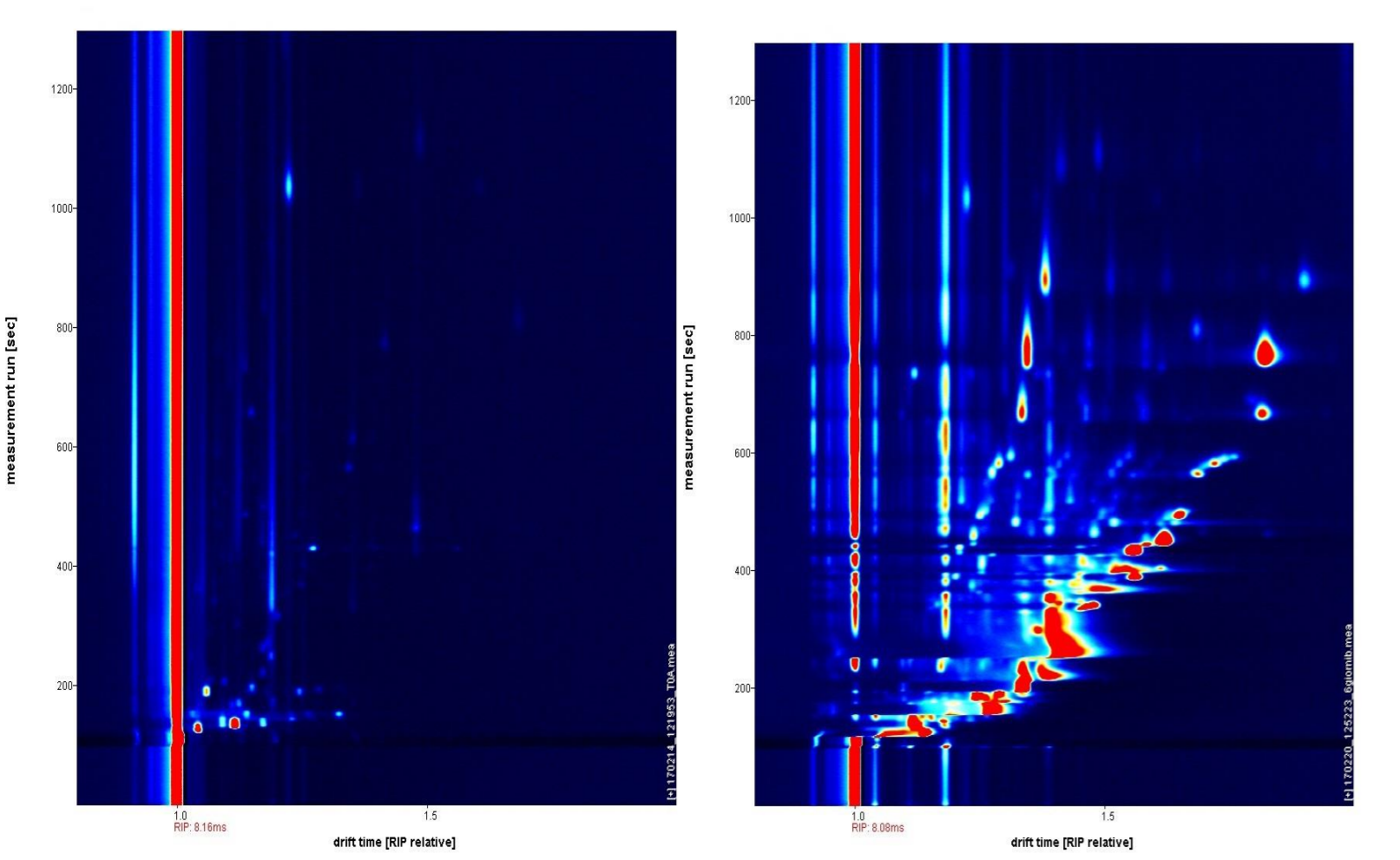
INSTRUMENT: Trace GC Ultra with DSQ II detector (Thermo Fisher Scientific-USA)

ESSENTIAL PARAMETERS

Fiber type: DVB/CAR/PDMS
Incubation time: 60 min
Extract time: 180 min
Column: Restek RXI-5ms 30m x 0.25 mm d_i 0.5 μm
Ionization mode: EI (+)

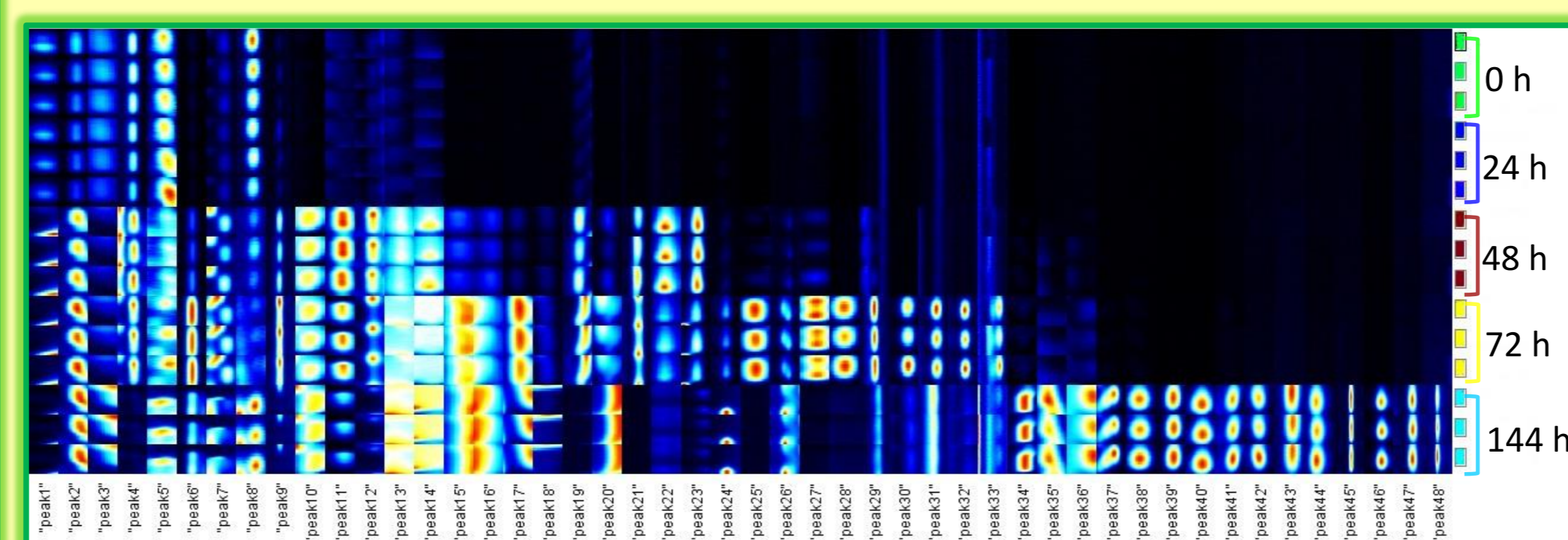
RESULTS

GC X IMS RESULTS STATISTIC ELABORATION COMPOUNDS ID STANDARD CONFIRMATION



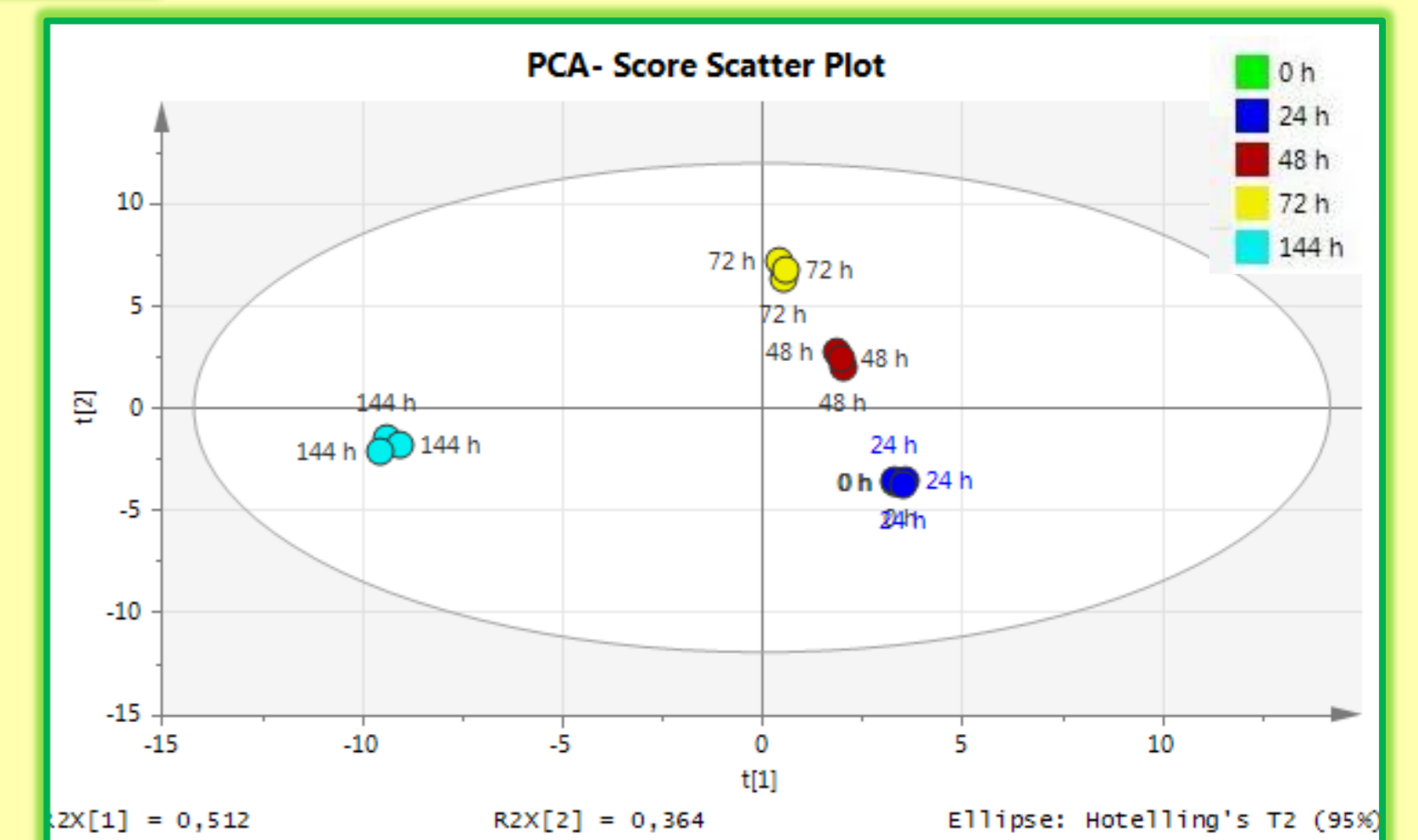
2D GCxIMS graph of fresh egg (left) and after 144h at RT (right)

Specific marker spots can be identified in the final 2D graphs that change their intensity or that can be detected or not, according to the time and storage conditions of the egg products.



Global overview of the 2D spots identified in the eggproduct at different time points

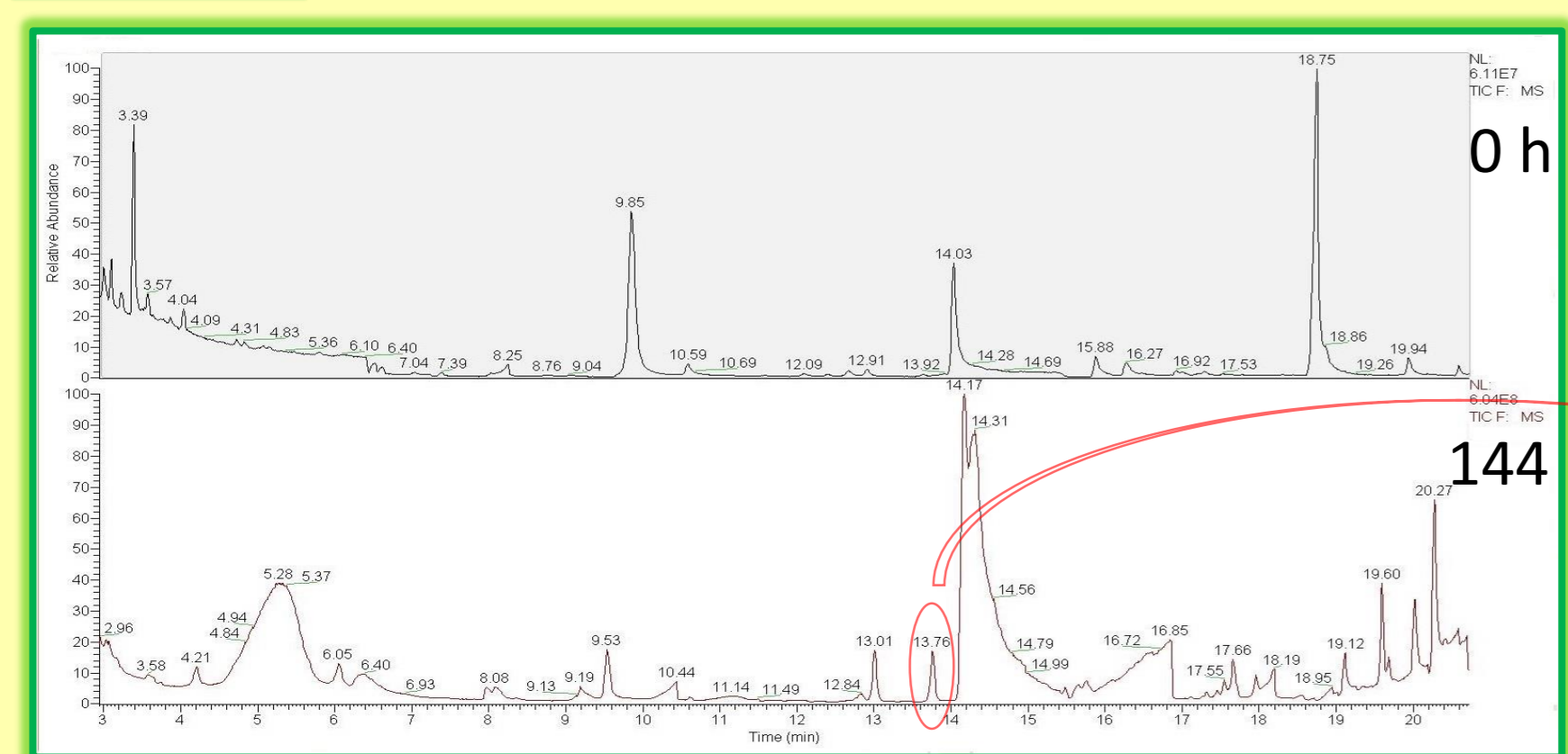
SIMCA GC X IMS RESULTS STATISTIC ELABORATION COMPOUNDS ID STANDARD CONFIRMATION



PCA score plot of the samples (R²X = 0.98 Q² = 0.96)

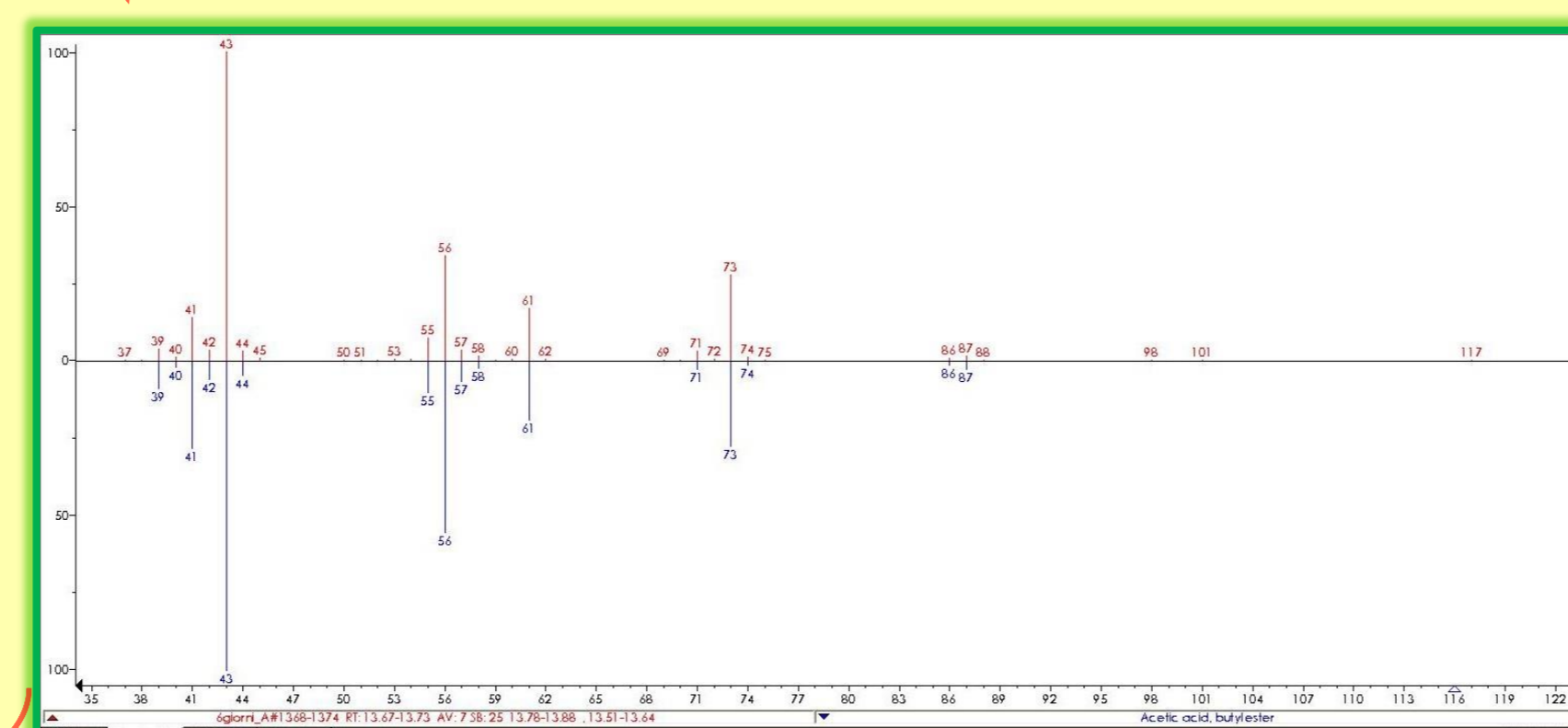
Samples at 48h, 72h and 144h time points are clearly clustered; no significant difference can be detected between the fresh and the 24 hours samples

GC X IMS RESULTS STATISTIC ELABORATION COMPOUNDS ID STANDARD CONFIRMATION



TIC chromatogram of fresh egg (top) and after 144h at RT (bottom)

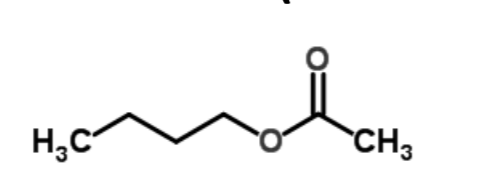
Each egg sample was injected in the GC-MS system, following the same experimental design used for the GC-IMS approach. Several time point marker compounds can be identified thanks to the GC-MS chromatograms and the NIST libraries



Comparison between the experimental (top) and the library recorded (bottom) mass spectra of peak at RT 13.76 min

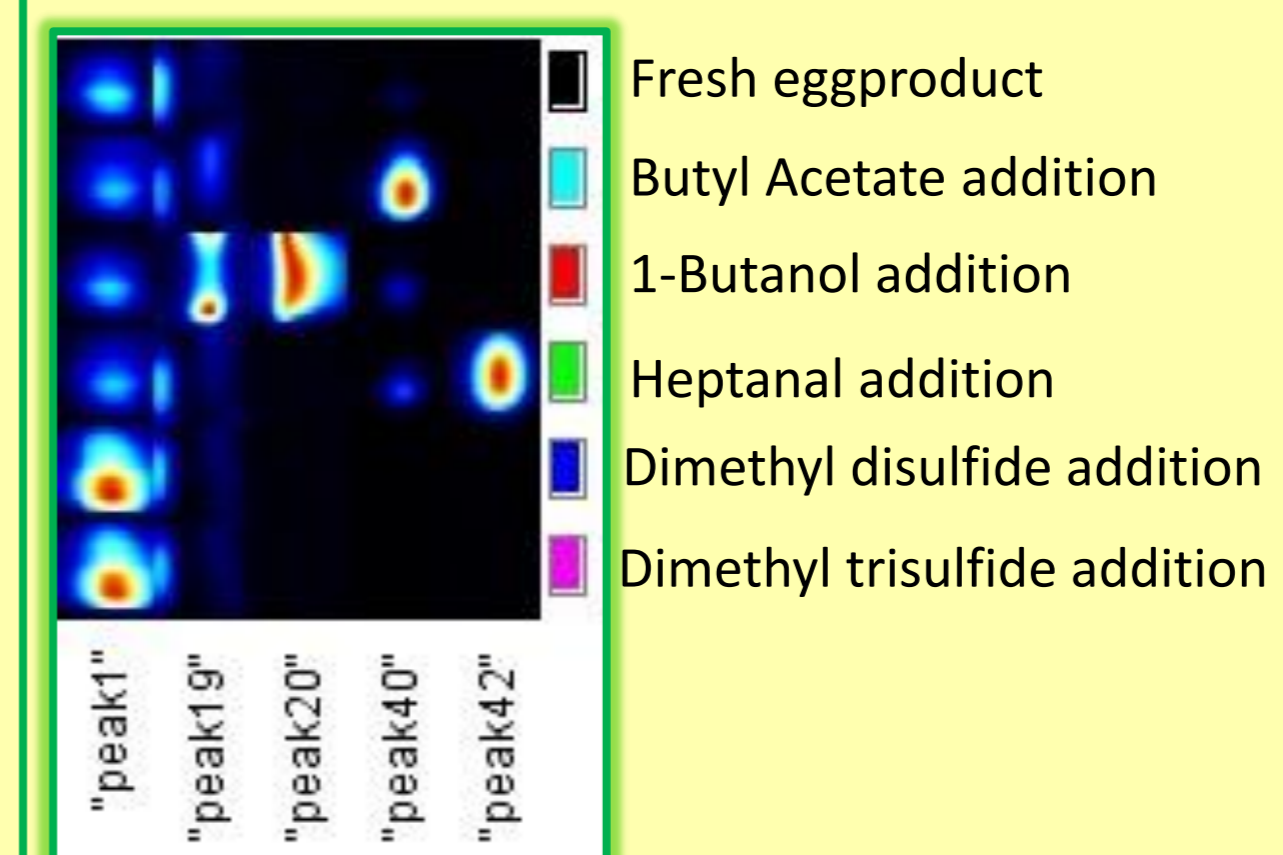
More than 20 marker compounds identified

BUTYL ACETATE (PROB. 90,2%)



GC X IMS RESULTS STATISTIC ELABORATION COMPOUNDS ID STANDARD CONFIRMATION

The available reference standards of the compounds identified with the GC-MS approach were separately spiked into the fresh eggproduct samples and analyzed with the GC-IMS system, with the aim to find a correlation between the marker spots and the target compounds.



Comparison of the most relevant 2D spots after standards addition

Some of the spots previously detected are uniquely correlated with the presence of specific compounds that can be identified as freshness markers.

Due to their similar structure, the sulfur compounds are not separated but in general we can assume that the increase in «peak 1» intensity can be considered a freshness issue

CONCLUSIONS

- This method is able to clearly discriminate the eggproducts according to the time spent at room temperature. Some of the marker compounds were identified; thanks to this technique, fraudulent freshness declarations can be rapidly detected
- The 24 hours samples present the same volatile profile of the fresh ones: this shows that, within this short period of time, eggproducts can be exploited into the production chain without significantly affect the quality and the safety of the corresponding finished products
- After a complete method validation, this GC-IMS technique could be implemented in the production sites

REFERENCES

- M.Suman et al- MOS based artificial olfactory system for the assessment of egg products freshness- Sensors and Actuators B 125, 2007: 40-47
- Liu, Y et al- Measurement of internal quality in chicken eggs using visible transmittance spectroscopy technology- Food Control 18 (1), 2007. 18-22
- A.Costanzo et al- The odour of sex: sex-related differences in volatile compound composition among barn swallow eggs carrying embryos of either sex- PLoS ONE 11(11), 2016