Spectroscopic analysis for feed quality and safety control at site and in lab

Vincent Baeten
v.baeten@cra.wallonie.be

Quality and Authentication of Products Unit

Knowledge and valorization of agricultural products Department
Walloon Agricultural Research Centre – CRA-W
Gembloux, Belgium

Quality and Authentication of Products Unit (Unit QAP)

Walloon Agricultural Research Centre

Dr Vincent Baeten

Scientific Director Unit QAP

v.baeten@cra.wallonie.be

Wallonie recherche CRA-W

« An active and innovative Unit involved in the development of sustainable methods to reinforce and assess the quality and authentication of agricultural and agro-food products »

2020 - …

EURL

Funded by the European Union

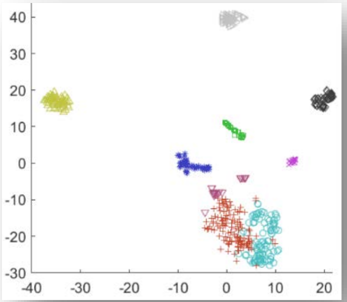
Wallonie recherche CRA-W

Quality and Authentication of Products Unit – CRA-W

4 MAIN RESEARCH FIELDS

PRECISION AGRICULTURE | PRECISION LIVESTOCK FARMING | RISK MANAGEMENT | UNDERSTANDING PRODUCTS

Field & orchard analysis	Farm & industry analysis	Laboratory analysis	Laboratory analysis
PhenWheat II (2022-2024)	**MobiLAB (2022-2024)**	**DepiPEST (2022-2024)**	**ValCerWal (2022-2024)**
NIR imaging solutions for phenotyping of wheat	Mobile laboratory dedicated for the demonstration and dissemination of photonics and digital solutions	MIR & Raman (+ chroma., U10) for the detection of fraud in pesticides products	NIR & NIR imaging (+ optical sorting /chemistry U11) for cereal batch allotment



sensor FINT

farmYng

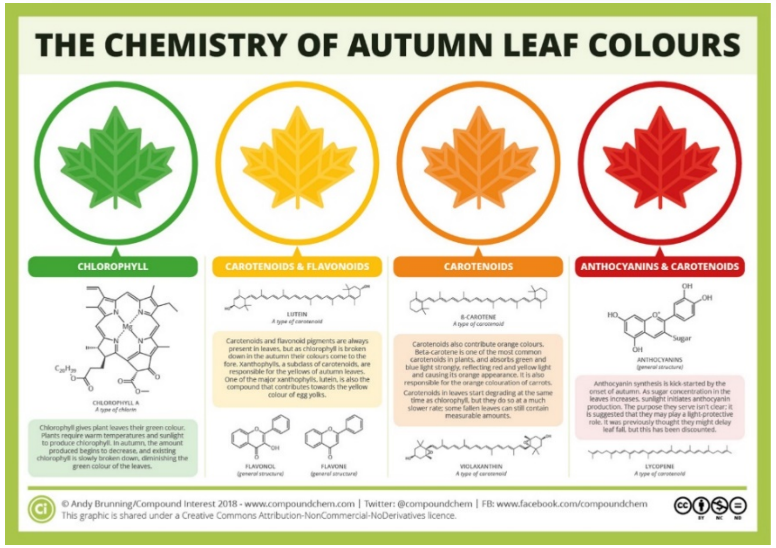
BROMEDIR

PHENET
PHENOTYPING & ENVIROTYPING SOLUTIONS FOR AGROECOLOGY

InnOBreed

Wallonie recherche CRA-W

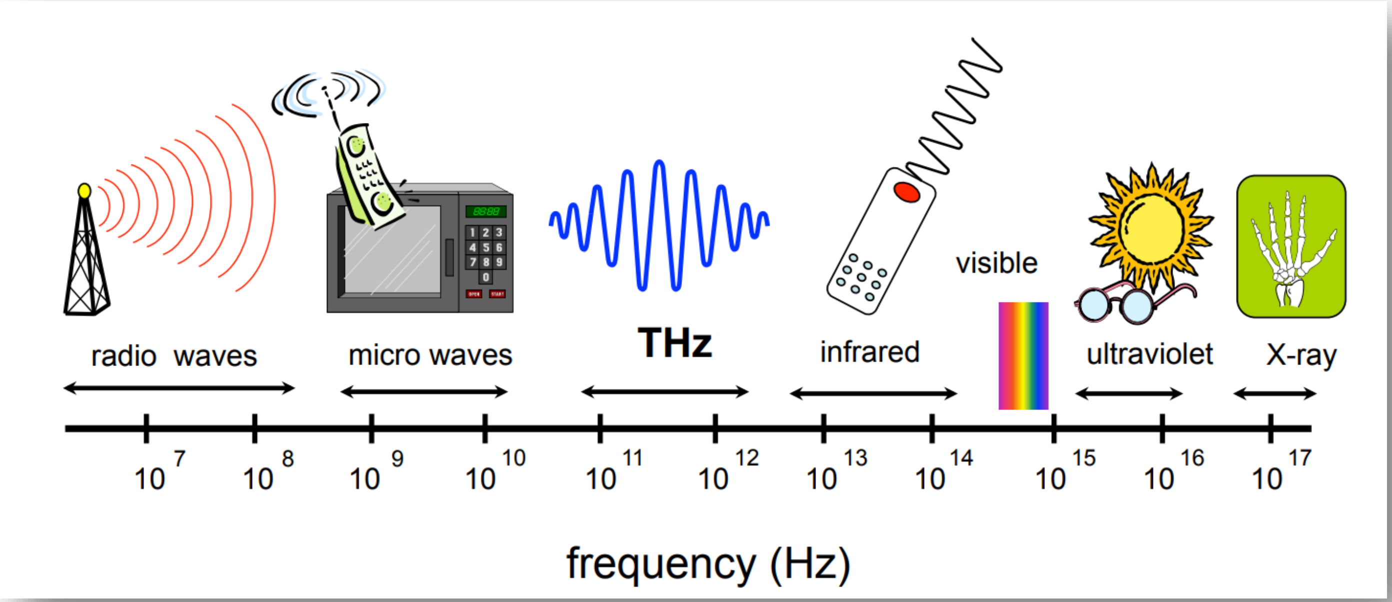
The waves at our services



Source : https://www.encyclopedie-environnement.org

We are using light to understand what we observe

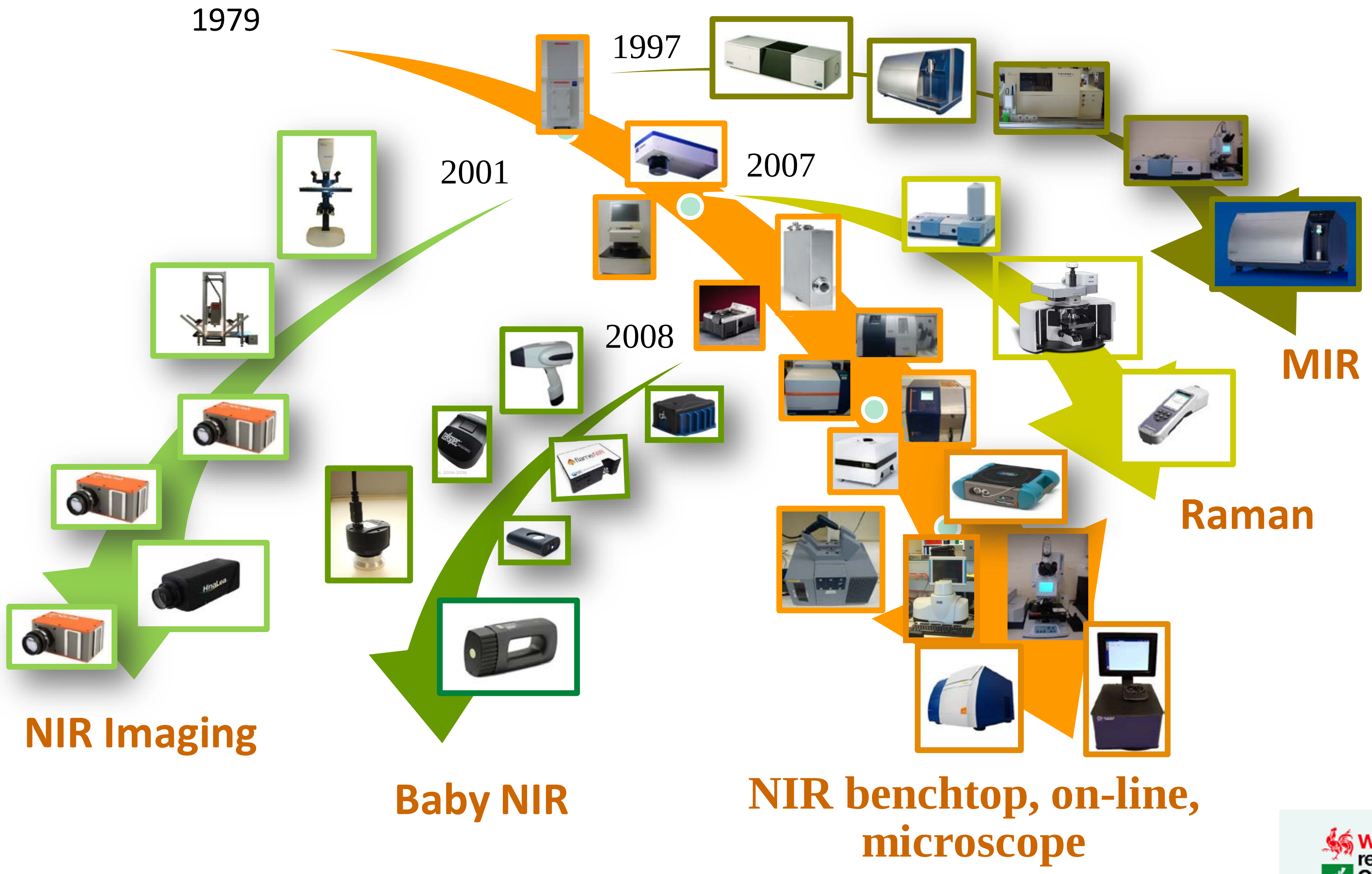
Colors = changes in the composition or texture of the object



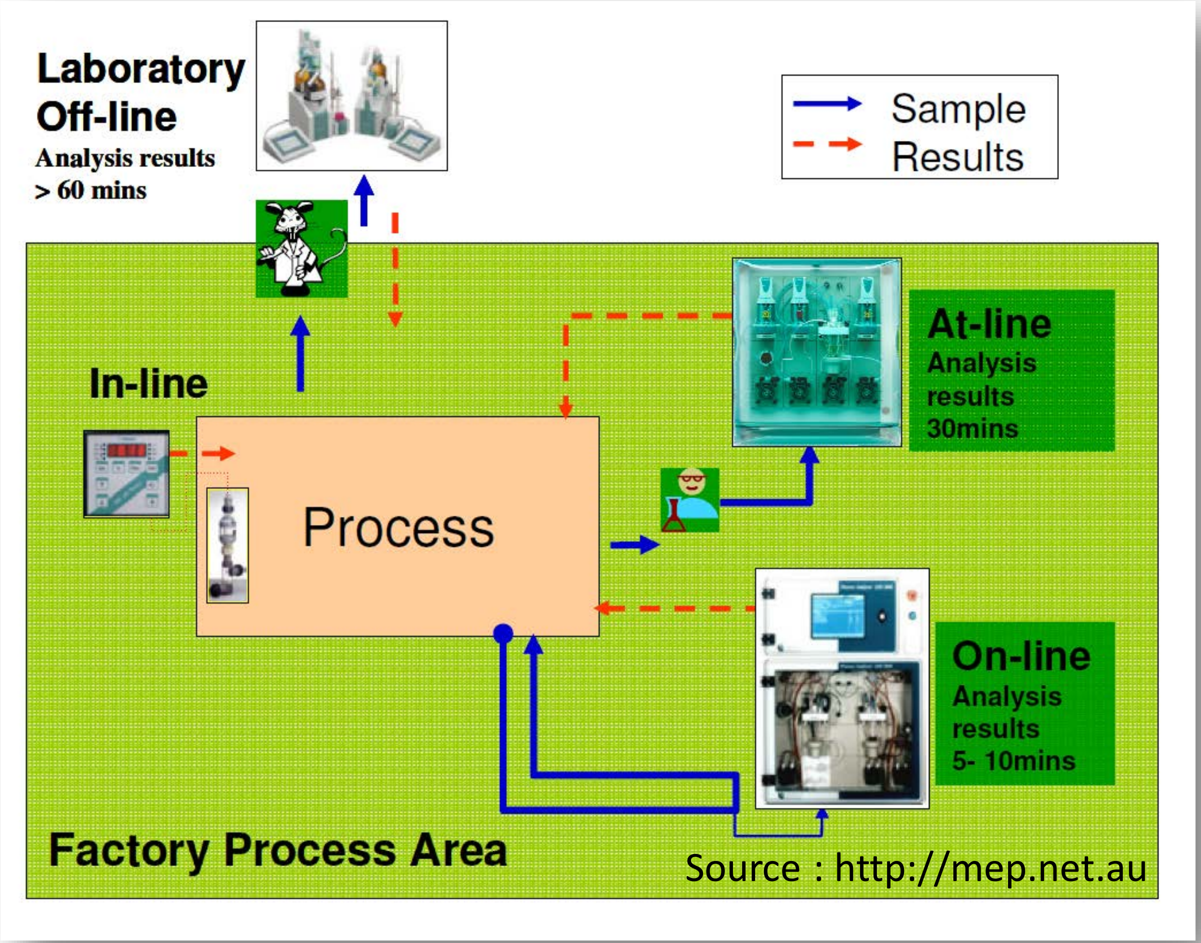
Source : R. Gente, N. Born, A. Rehn, M. Koch CROP.SENSe.net Symposium, Bonn, 29.9.2014.

Wallonie recherche CRA-W

Spectroscopy devices at CRA-W



TREND → « lab to the sample approach » = the data travel instead of the samples !



***Off-line –** spot & laboratory analysis*
feedback in hours to days

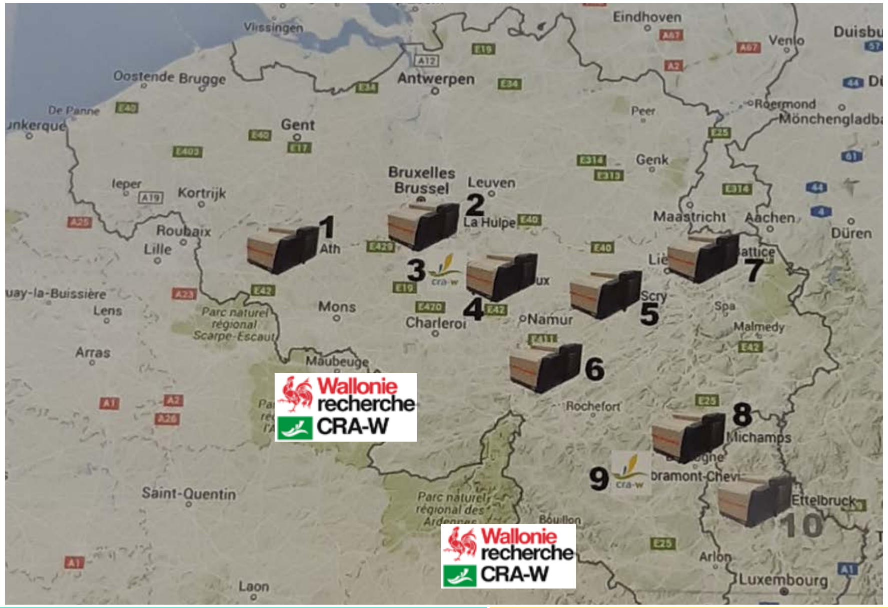
***At-line –** spot & on-site analysis*
feedback in minutes

***On-line –** regular interval & automatic analysis*
feedback in minutes to seconds

***In-line –** continuous & automatic analysis*
instaneous feedback

A network analysis and advices for the Walloon agriculture sector

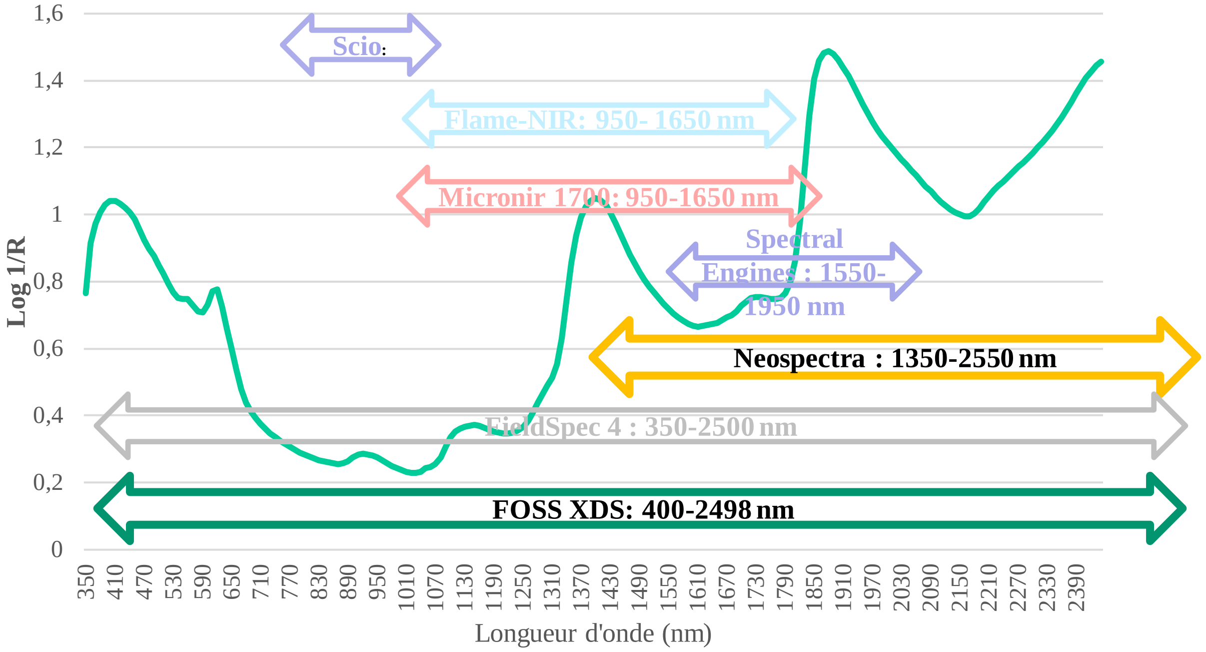
- Created in 1989
- 9 laboratories in Wallonia
  + 1 in Luxembourg
- 11 spectrometers connected to CRA-W (= master)
- Each spectrometer is standardized every year on the master
- Several parameters/criteria for different matrices
- Interlaboratory studies



ISO 17025 & ISO 17043

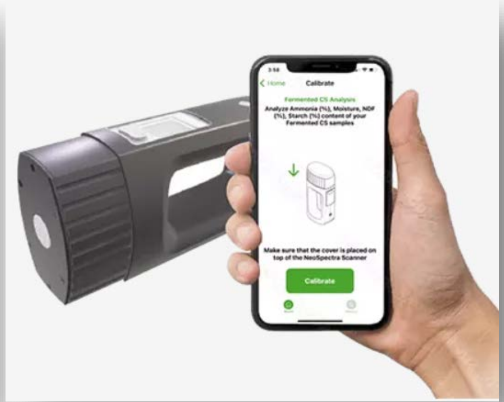
Reagent/chemical	Reference values	NIR valus
Reagent	☹	☺ (95 % reduction)
Time / sample / technician	4 hours	5 minutes
Samples / day/ technician	5-10	100

Network of spectrometers : other strategies !



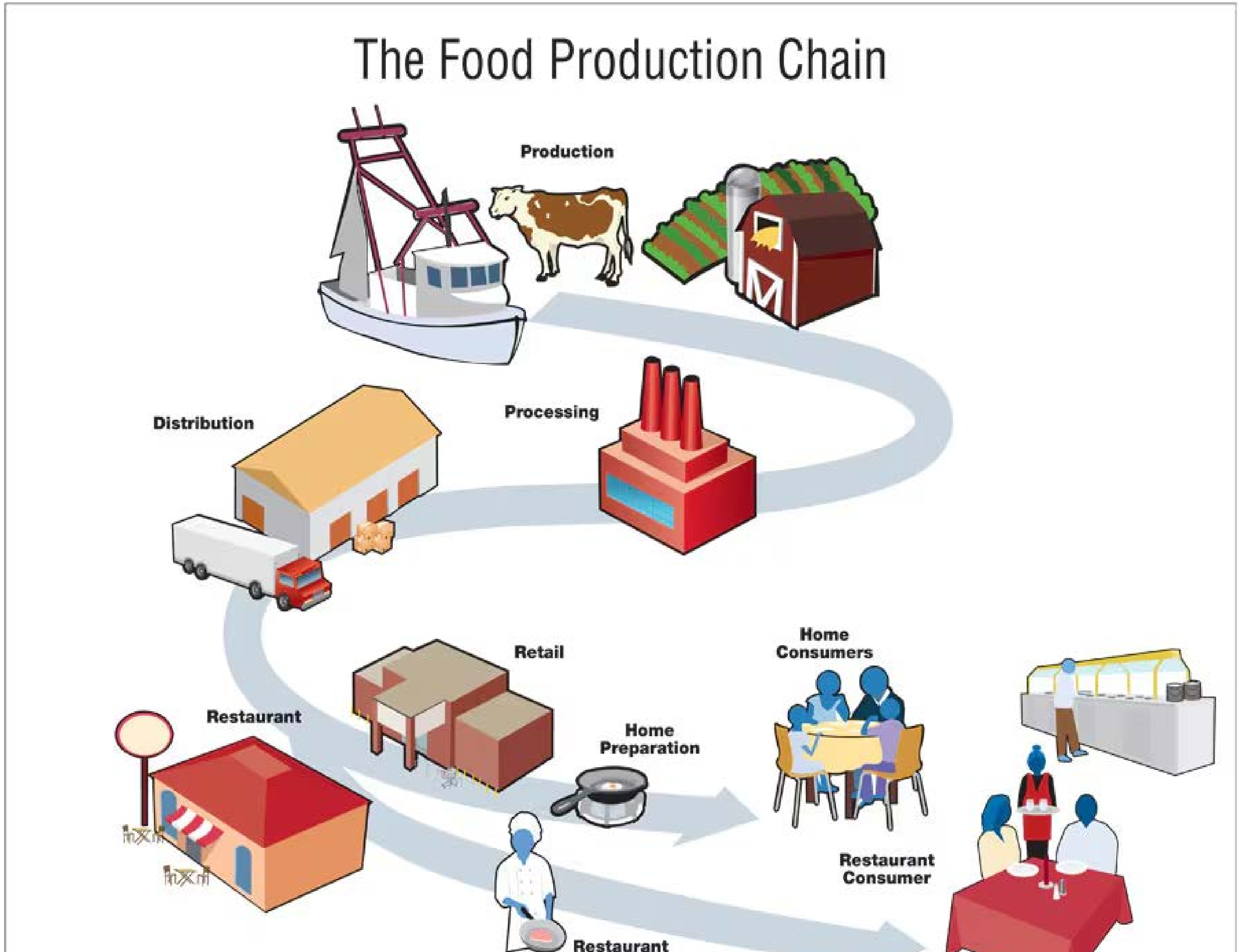
Neospectra (Siware)

1350-2500 nm



WALOPEA project

We need a global approach to enhance the quality and safety of our food production chain

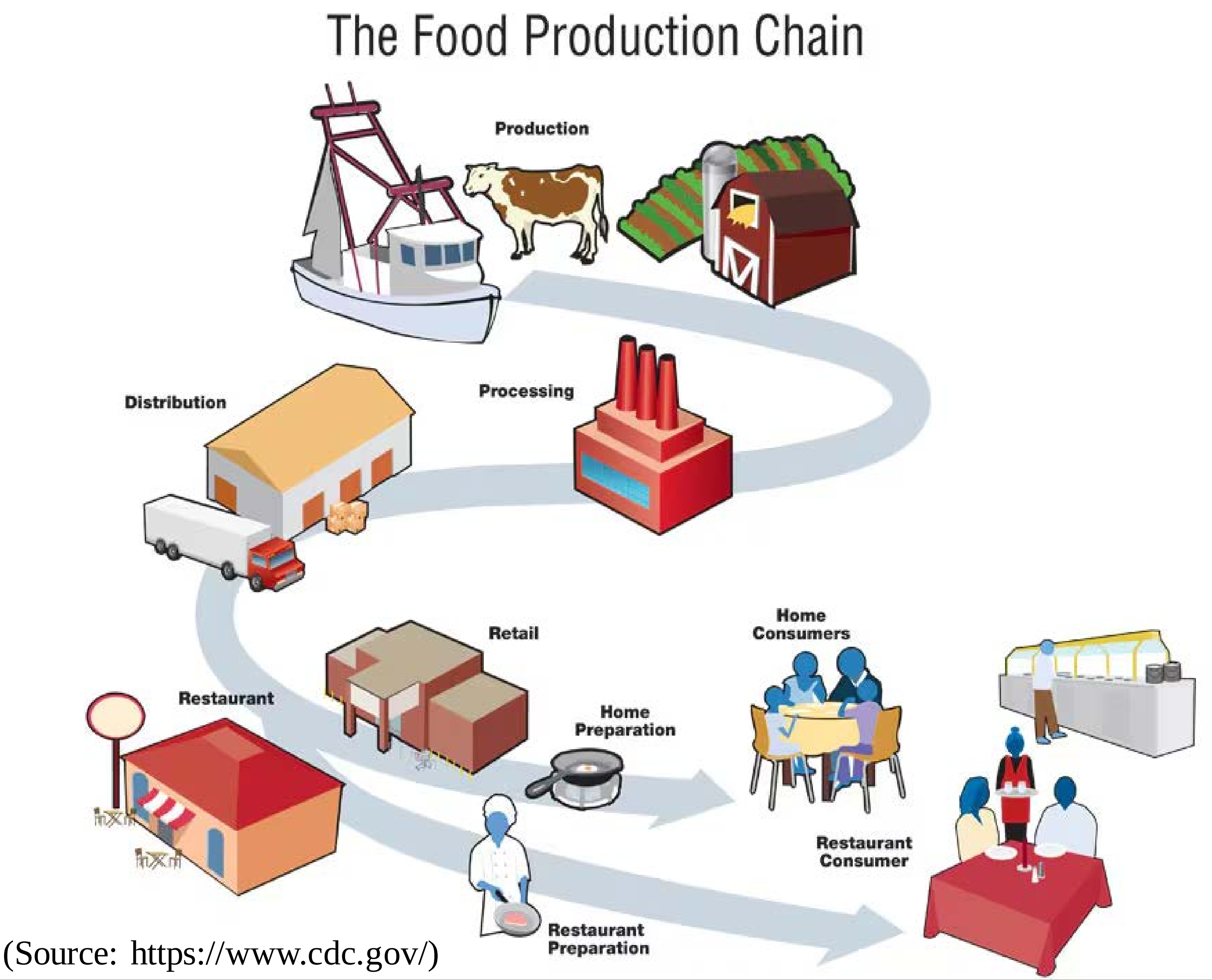


(Source: https://www.cdc.gov/)

- ***At fields, meadows, farms, orchards & greenhouses level***
- ***At processing and transforming plants level***
- ***At distribution and retail facilities level***
- ***At consumer level***

In addition to the official control

We need a global approach to enhance the quality and safety of our food production chain

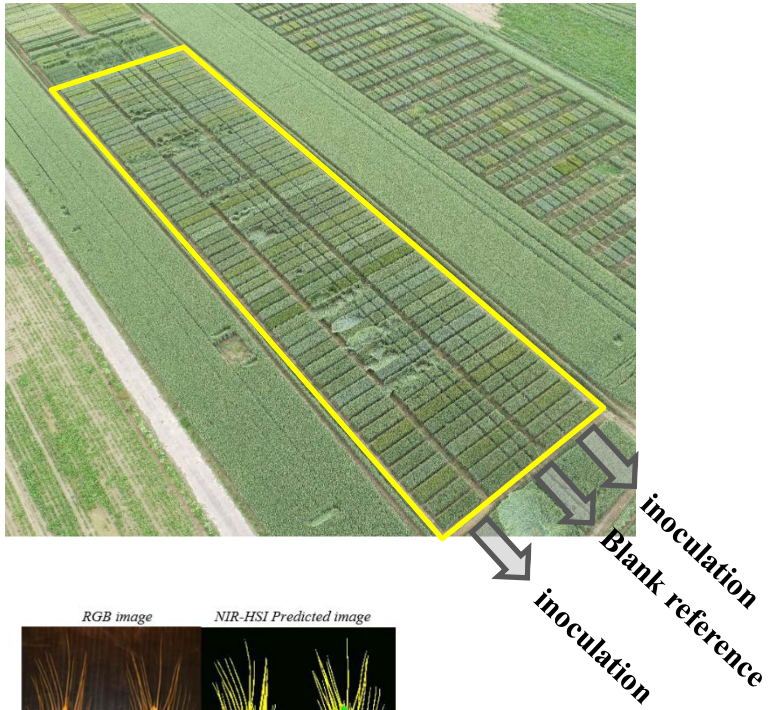


(Source: https://www.cdc.gov/)

- ❑ ***At fields, meadows, farms, orchards & greenhouses level***
- ❑ *At processing and transforming plants level*
- ❑ *At distribution and retail facilities level*
- ❑ *At consumer level*

In addition to the official control

At fields, meadows, farms, orchard & greenhouses level : evaluation of fusarium blight infection



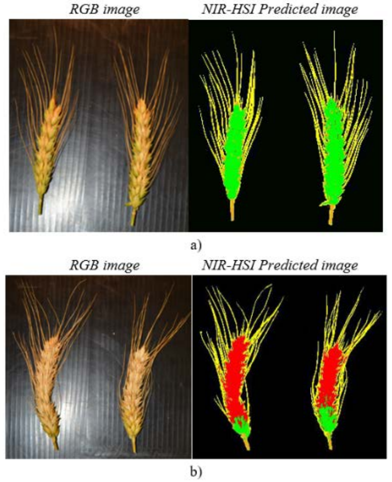
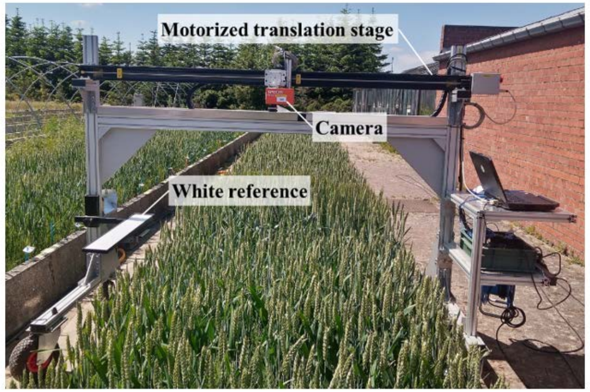


Figure 5-5: Pictures and predictions of NIR hyperspectral images of (a) healthy ears and (b) FHB-infected ears. Pixels predicted as FHB-infected ears are in red, pixels predicted as healthy ears are in green, pixels predicted as stems are in orange, pixels predicted as awns are in yellow and pixels predicted as background are in black.



Vincke et al. (2023). Near infrared hyperspectral imaging method to assess Fusarium Head Blight infection on winter wheat ears (2023) Microchemical Journal, 191, art. no. 108812.

Wallonie recherche CRA-W

At fields, meadows, farms, orchard & greenhouses level : evaluation of fusarium blight infection

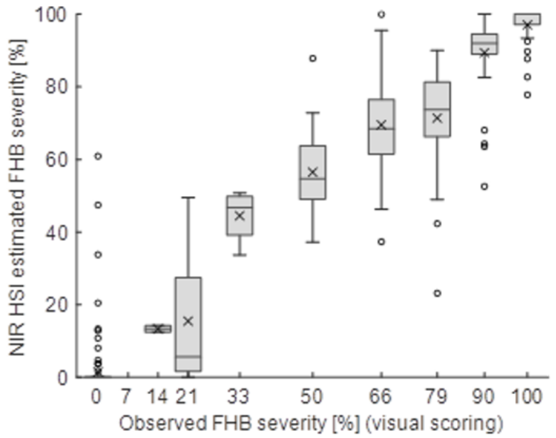


Figure 5-6: Boxplot of the PLS-DA estimated FHB severity of each ear against its corresponding value for the visual scoring of FHB severity.

In the lab

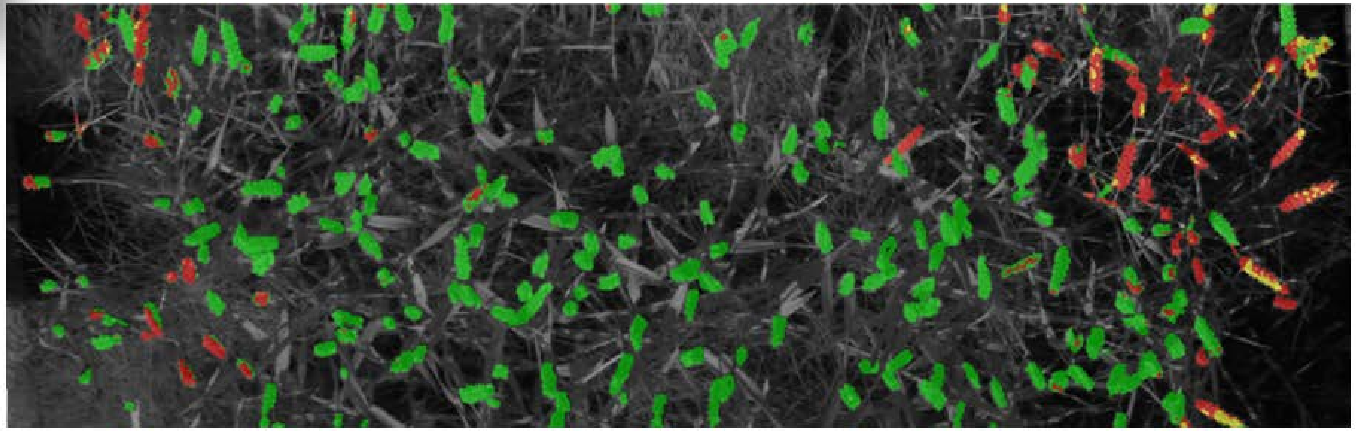


Figure 6-9: Prediction result for the image of plot 14 acquired on June 28th.

In the field

At fields, meadows, farms, orchard & greenhouses level : evaluation of forage at farm level

PRECISION LIVESTOCK FARMING

€FFORT

Transition of dairy farming toward a better efficient use of fodder resources

In the farm

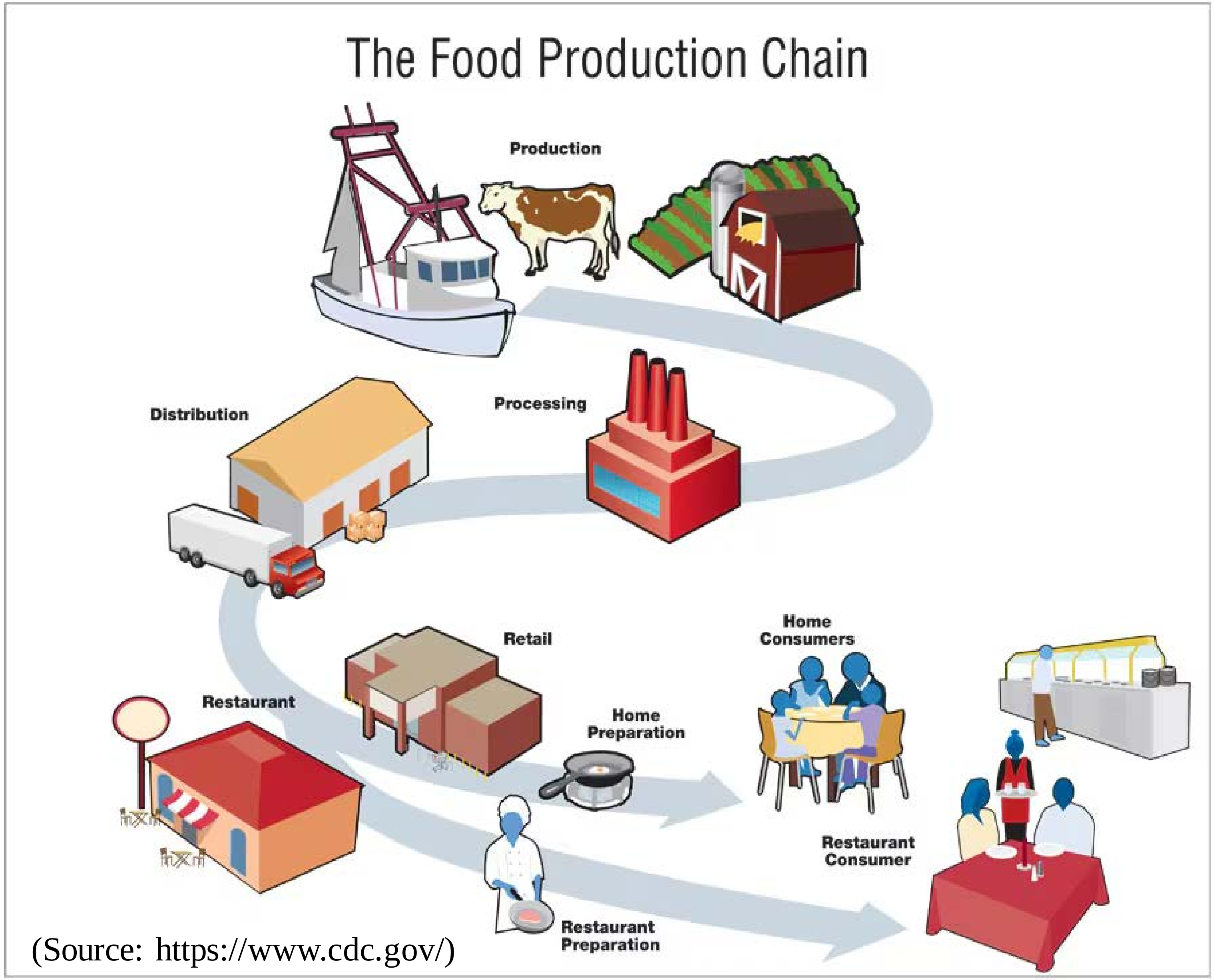
Sampling of corn silage

Sampling of haylage bale

t° and humidity influences
Sampling issues !!!

Fernández et al. (2022). Performance of three handheld NIR spectrometers for predicting grass silage quality. Biotechnology, Agronomy, Society and Environment, 26 (Special Issue), pp. 1-10.

We need a global approach to enhance the quality and safety of our food production chain



(Source: https://www.cdc.gov/)

- ❑ *At fields, meadows, farms, orchards & greenhouses level*
- ❑ ***At processing and transforming plants level***
- ❑ *At distribution and retail facilities level*
- ❑ *At consumer level*

In addition to the official control

At processing and transforming plants level : analysis a the ports of entry

Detection of adulteration at industrial level

STEP 1
Loading of 5 tons of soybean meal

STEP 2
Loading of 5 tons of soybean meal
+
“Massive” adulteration

STEP 3
Loading of 5 tons of soybean meal

STEP 4
Loading of 5 tons of soybean meal
+
“Smooth” adulteration

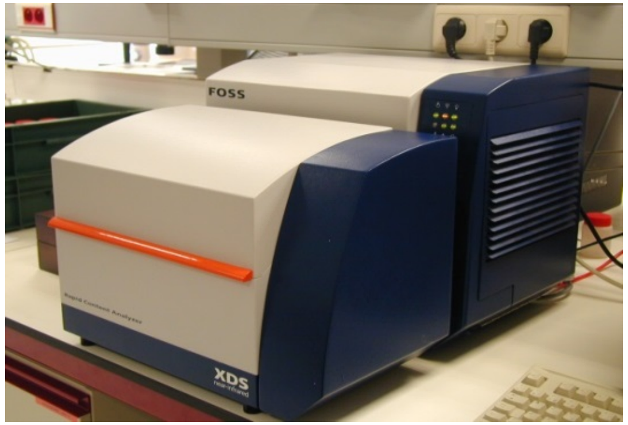
STEP 5
Loading the remaining part of the soybean meal

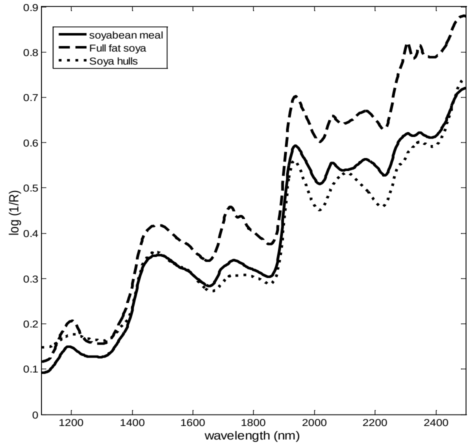


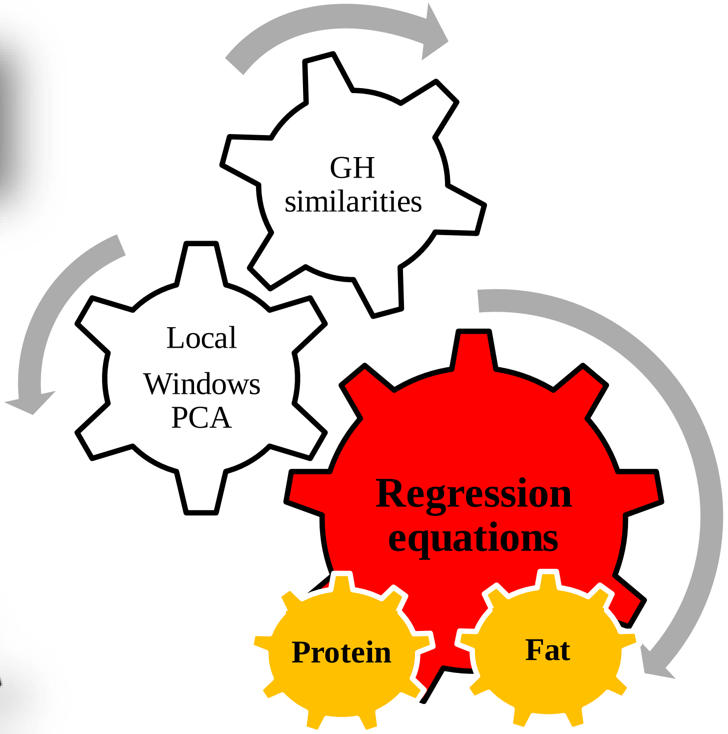


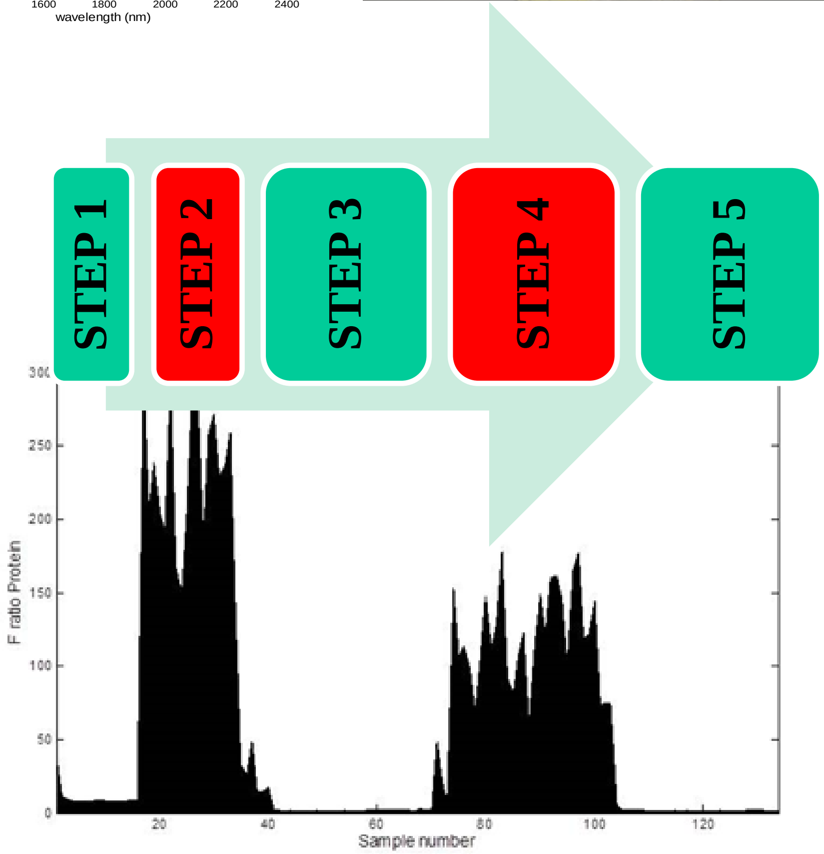
Fernández et al. (2015). NIR fingerprint screening for early control of non-conformity at feed mills. Food Chemistry, 189, art. no. 16461, pp. 2-12.

At processing and transforming plants level : analysis a the ports of entry

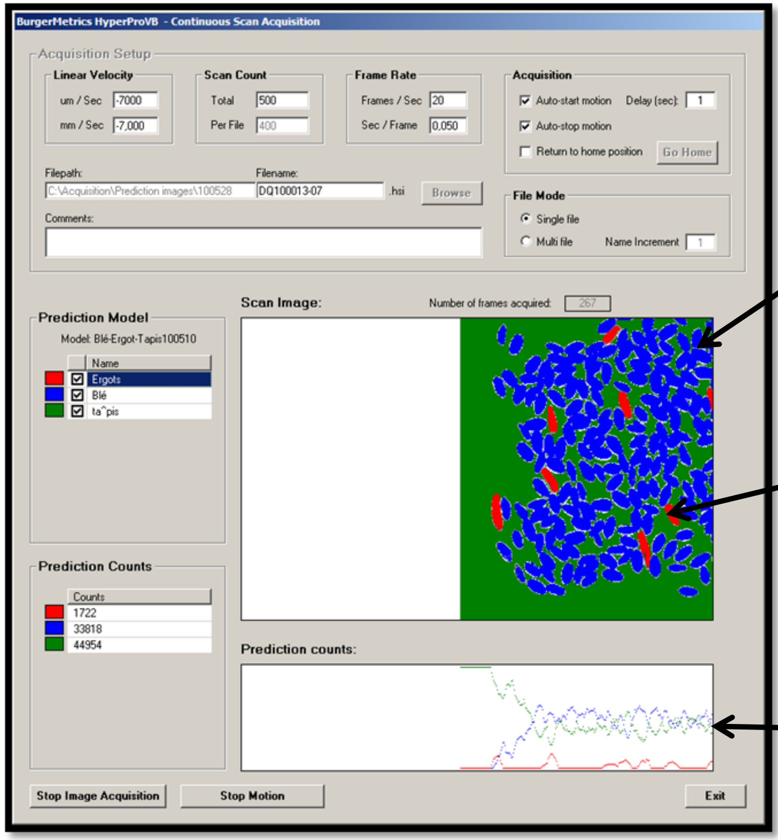








Protein determination



Wheat kernels

Ergot Bodies

The number of pixels counted for each class of the model is also provided.

- **LOD <<< 500 ppm**
- **Several analysis by kernel or by ergot body**
- **Fast → 30-40 kg / hour = LOD 1 ppm**

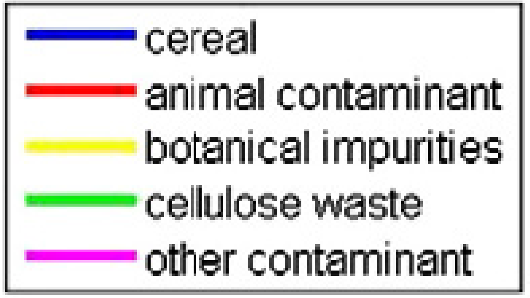
(Ref method = several days)

Analytical parameters used and on-line prediction results of the PLSDA (Partial Least Squares Discriminant Analysis) model

Ergot detection in wheat by NIR imaging

Wallonie recherche CRA-W

At processing and transforming plants level : analysis a the ports of entry



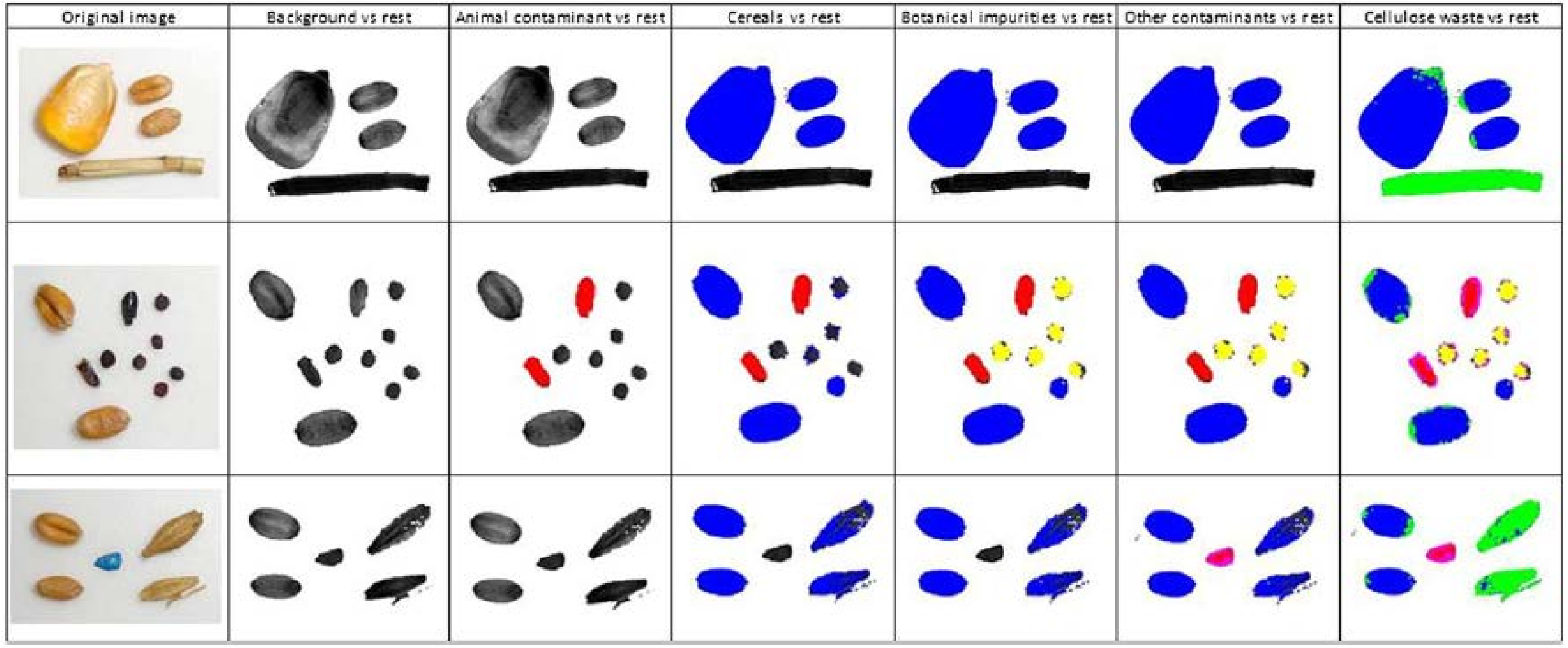
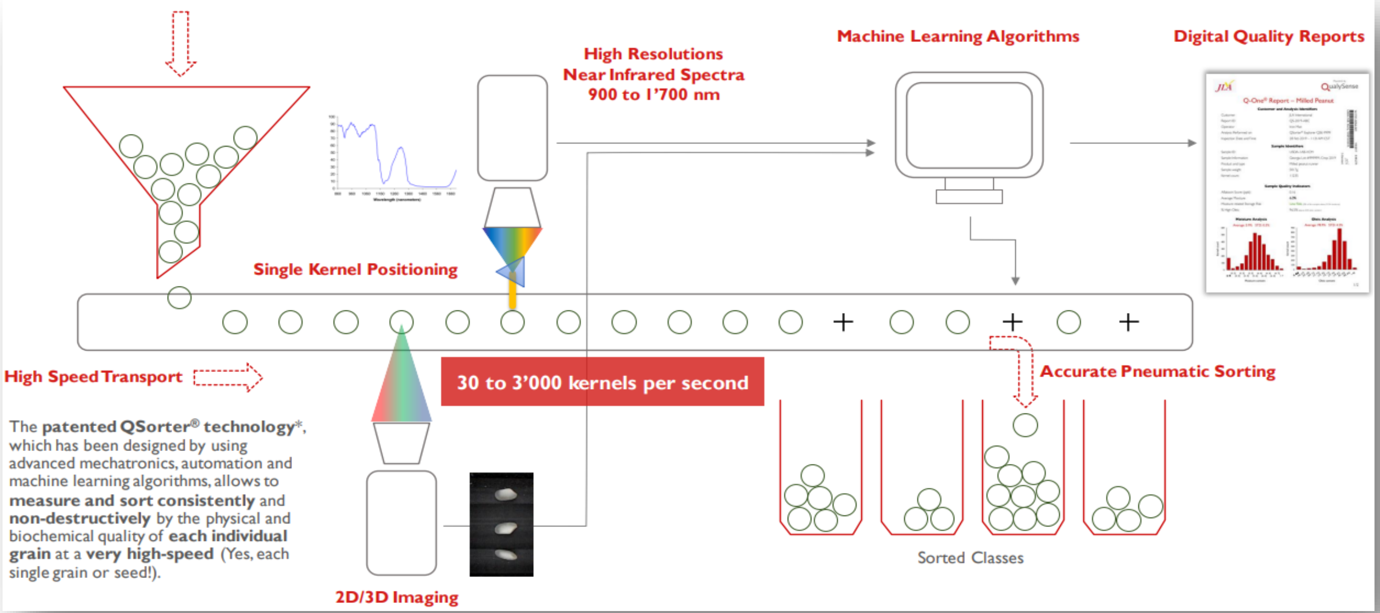


Fig. 2. Three examples of the application of each of the equations of the classification tree nodes. Each example includes the original image and the prediction images. Pixels are coloured as follows: detected as cereal are indicated in blue, detected as animal contaminant in red, detected as botanical impurities in yellow, detected as cellulose waste in green and detected as other contaminant in pink. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

NIR Imaging – **multi-detection** of contaminants/impurities

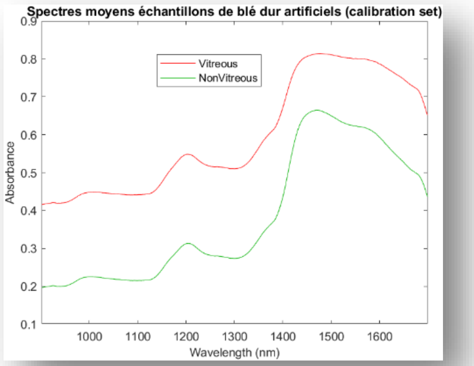
At processing and transforming plants level : analysis a the ports of entry

Qsorter – kernel sorting (installed at CRA-W in 2022 – VALCERWAL project)



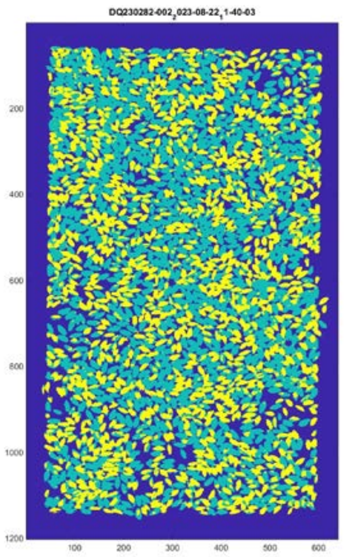
Batch segmentation

Individual kernel sorting



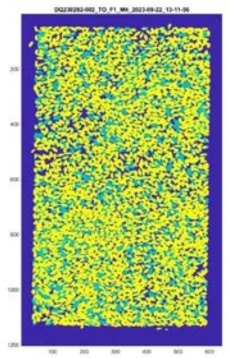
(Contact : Bruno Godin, b.godin@cra.wallonie.be & Pierre-Yves Werrie, p.werrie@cra.wallonie.be)

At processing and transforming plants level : analysis a the ports of entry

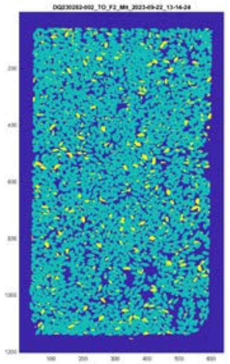


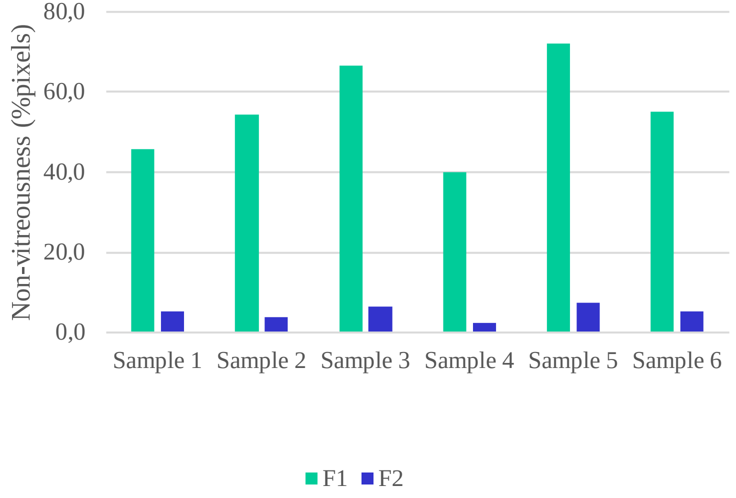
NIR spectra 800 – 1800 nm
& RGB image

F1



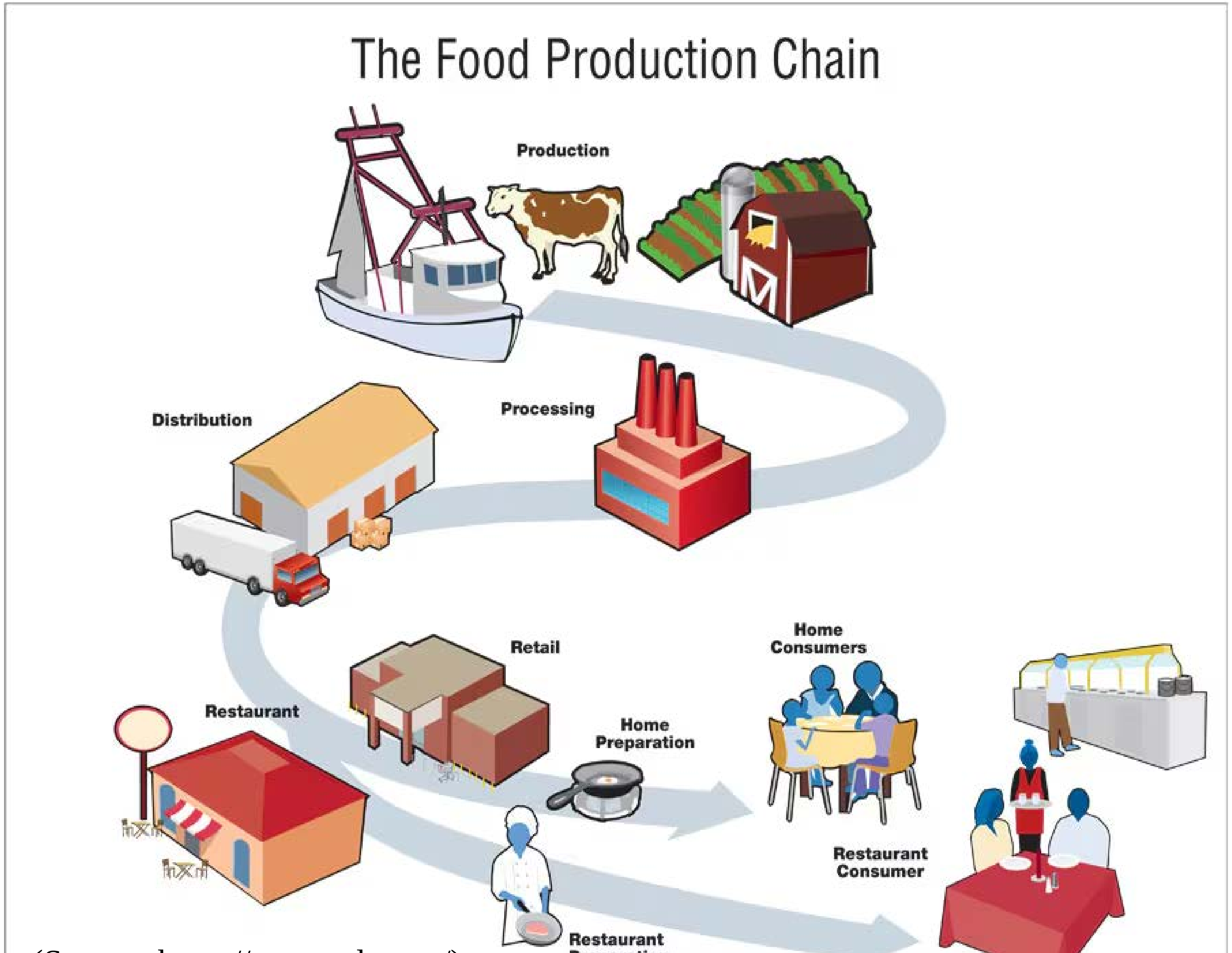
F2





- Sorting of raw material – maximization of the valorization of the production

We need a global approach to enhance the quality and safety of our food production chain



(Source: https://www.cdc.gov/)

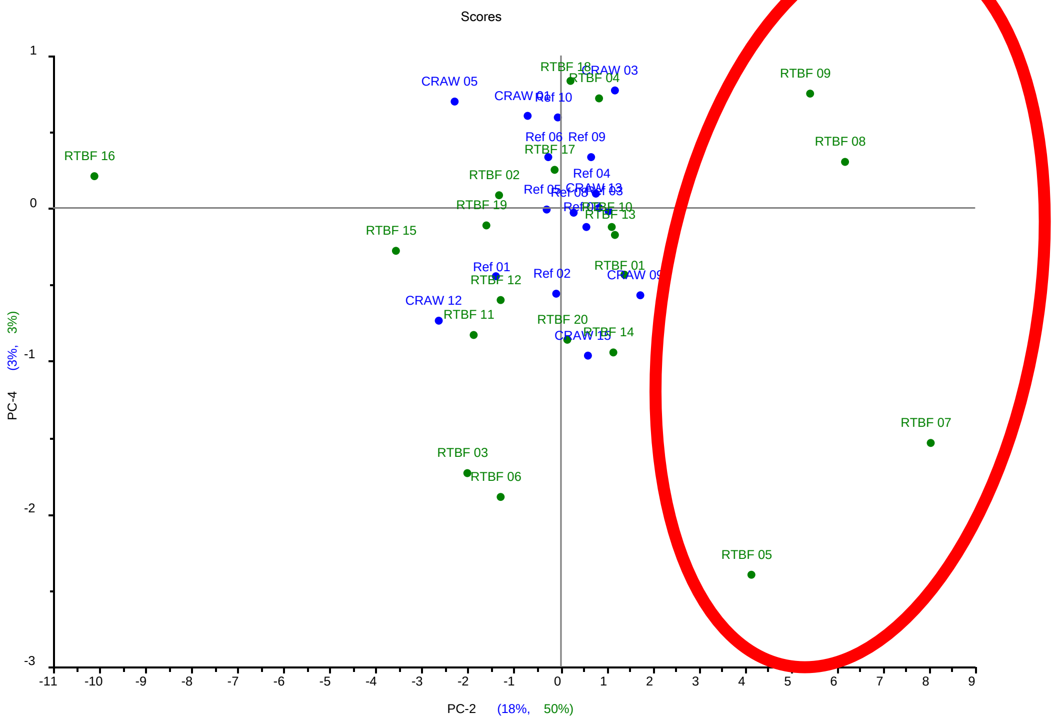
- ❑ *At fields, meadows, farms, orchards & greenhouses level*
- ❑ *At processing and transforming plants level*
- ❑ ***At distribution and retail facilities level***
- ❑ *At consumer level*

In addition to the official control

At distribution and retail facilities level : the challenge of the detection of mislabeling and fraud

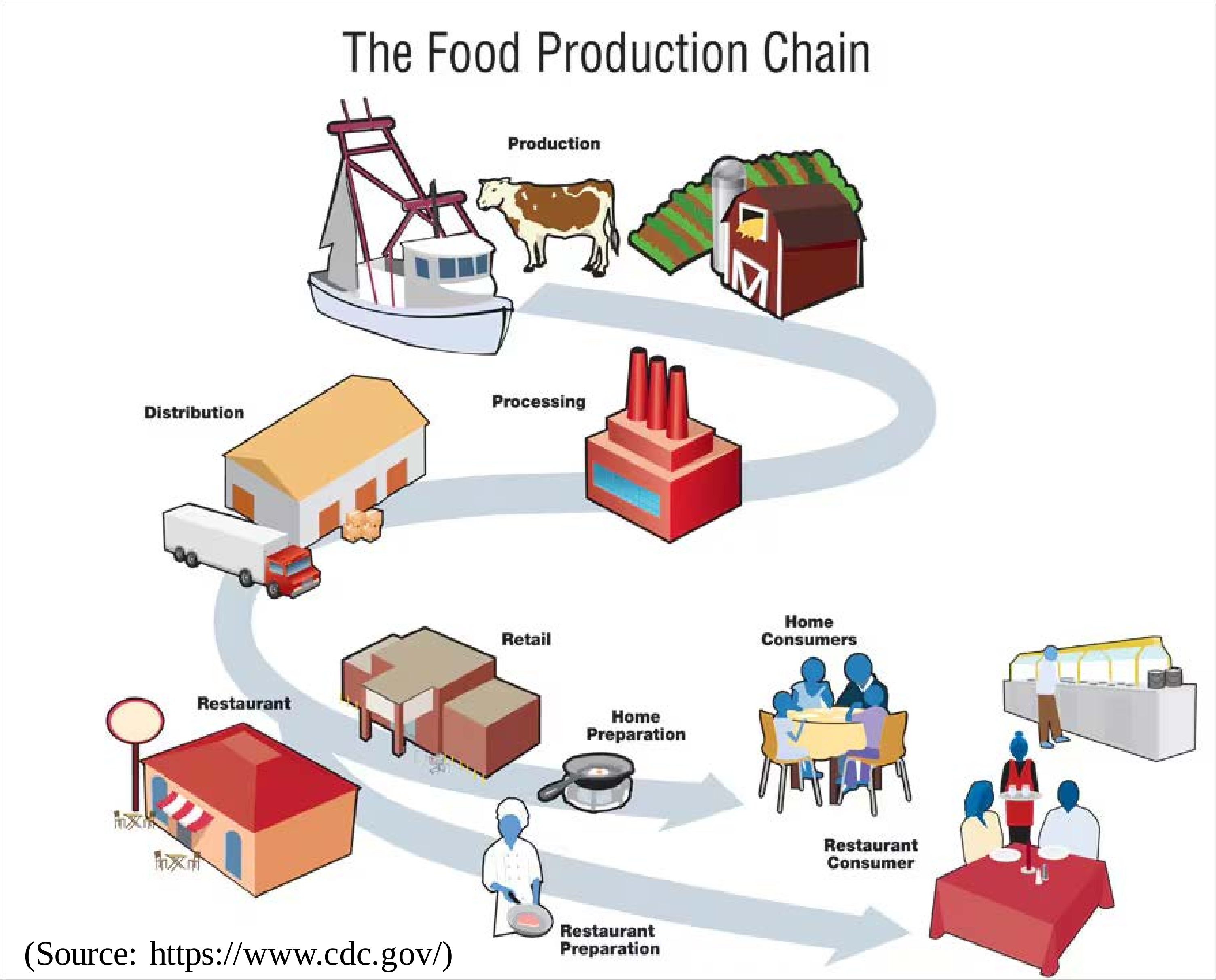
Reduced survey (2023) of pepers sold in Belgium

Untarget approach



Vermeulen et al. (2017). Authentication and Traceability of Agricultural and Food Products Using Vibrational Spectroscopy In: Food Traceability and Authenticity: Analytical Techniques, Montet D. and Ray RC. USA, Biology series, CRC press, 450.

We need a global approach to enhance the quality and safety of our food production chain



(Source: https://www.cdc.gov/)

- ❑ *At fields, meadows, farms, orchards & greenhouses level*
- ❑ *At processing and transforming plants level*
- ❑ *At distribution and retail facilities level*
- ❑ ***At consumer level***

In addition to the official control

Wallonie recherche CRA-W

At consumer level : protection of the consumer

Journal of Analytical Toxicology, 2020;44:851–860
doi:10.1093/jat/bkaa101
Special Issue

OXFORD

Special Issue

Comparison of Spectroscopic Techniques Combined with Chemometrics for Cocaine Powder Analysis

Joy Eliaerts[1,*], Natalie Meert[1], Pierre Dardenne[2], Vincent Baeten[2], Juan-Antonio Fernandez Pierna[2], Filip Van Durme[1], Karolien De Wael[3] and Nele Samyn[1]

[1]National Institute of Criminalistics and Criminology (NICC), Department drugs and toxicology, Brussels, Belgium, [2]Walloon Agricultural Research Centre, Department of Product Valorization, Gembloux, Belgium and [3]University of Antwerp, Department of Bioengineering, Antwerp, Belgium

*Author to whom correspondence should be addressed. E-mail: joy.eliaerts@just.fgov.be

Abstract

Spectroscopic techniques combined with chemometrics are a promising tool for analysis of seized drug powders. In this study, the performance of three spectroscopic techniques [Mid-InfraRed (MIR), Raman and Near-InfraRed (NIR)] was compared. In total, 364 seized powders were analyzed and consisted of 276 cocaine powders (with concentrations ranging from 4 to 99 w%) and 88 powders without cocaine. A classification model (using Support Vector Machines [SVM] discriminant analysis) and a quantification model (using SVM regression) were constructed with each spectral dataset in order to discriminate cocaine powders from other powders and quantify cocaine in powders classified as cocaine positive. The performances of the models were compared with gas chromatography coupled with mass spectrometry (GC–MS) and gas chromatography with flame-ionization detection (GC–FID). Different evaluation criteria were used: number of false negatives (FNs), number of false positives (FPs), accuracy, root mean square error of cross-validation (RMSECV) and determination coefficients (R^2). Ten colored powders were excluded from the classification data set due to fluorescence background observed in Raman spectra. For the classification, the best accuracy (99.7%) was obtained with MIR spectra. With Raman and NIR spectra, the accuracy was 99.5% and 98.9%, respectively. For the quantification, the best results were obtained with NIR spectra. The cocaine content was determined with a RMSECV of 3.79% and a R^2 of 0.97. The performance of MIR and Raman to predict cocaine concentrations was lower than NIR, with RMSECV of 6.76% and 6.79%, respectively and both with a R^2 of 0.90. The three spectroscopic techniques can be applied for both classification and quantification of cocaine, but some differences in performance were detected. The best classification was obtained with MIR spectra. For quantification, however, the RMSECV of MIR and Raman was twice as high in comparison with NIR. Spectroscopic techniques combined with chemometrics can reduce the workload for confirmation analysis (e.g., chromatography based) and therefore save time and resources.

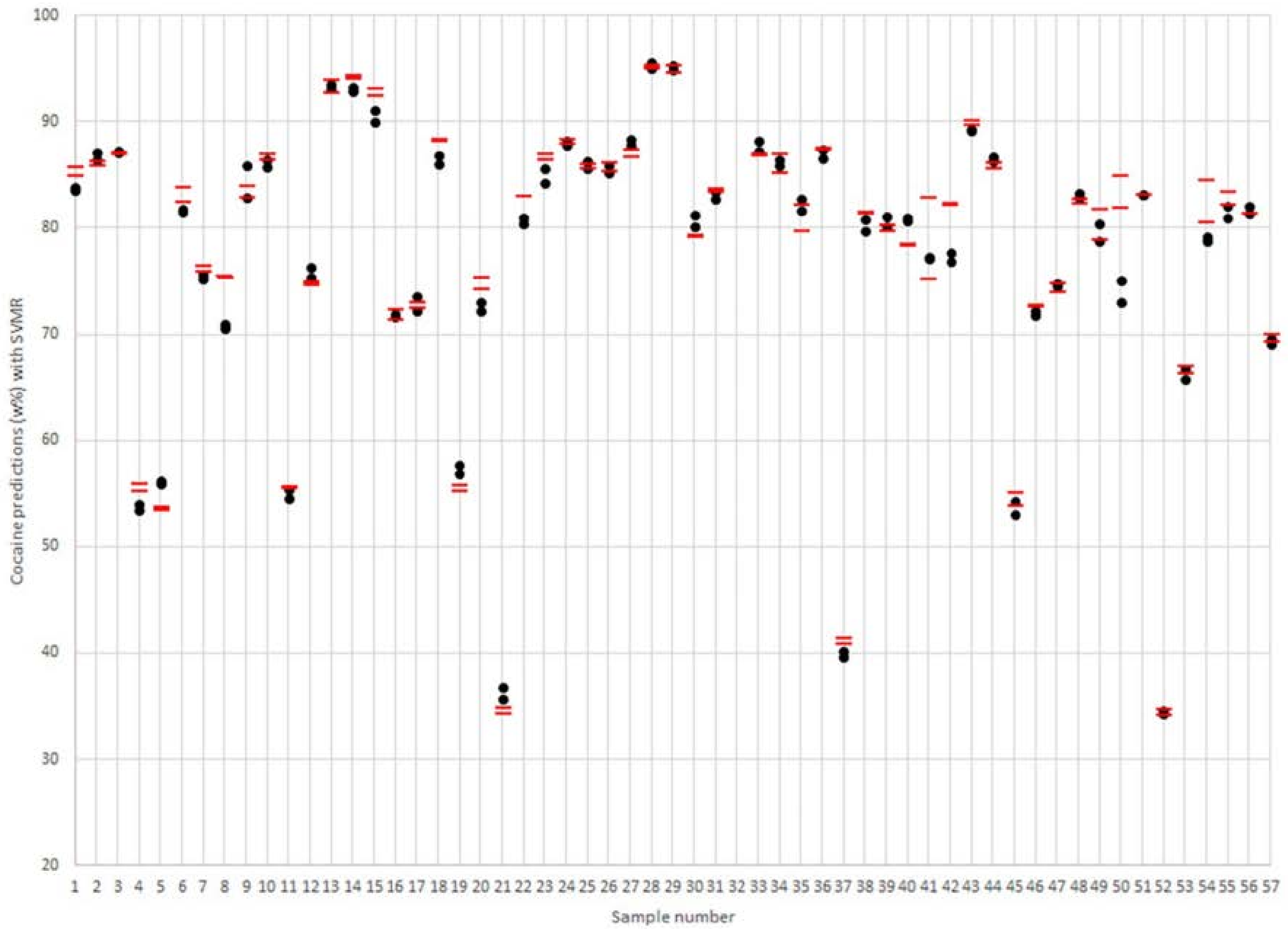


Figure 9. Replicate MIR analyses of 57 cocaine street samples (4 measurements/day per sample). LEGEND: Red stripes: two consecutive measurements of the same powder on the ATR crystal, black points: measurements after placing new powder of the same sample on the ATR crystal.

Wallonie recherche CRA-W

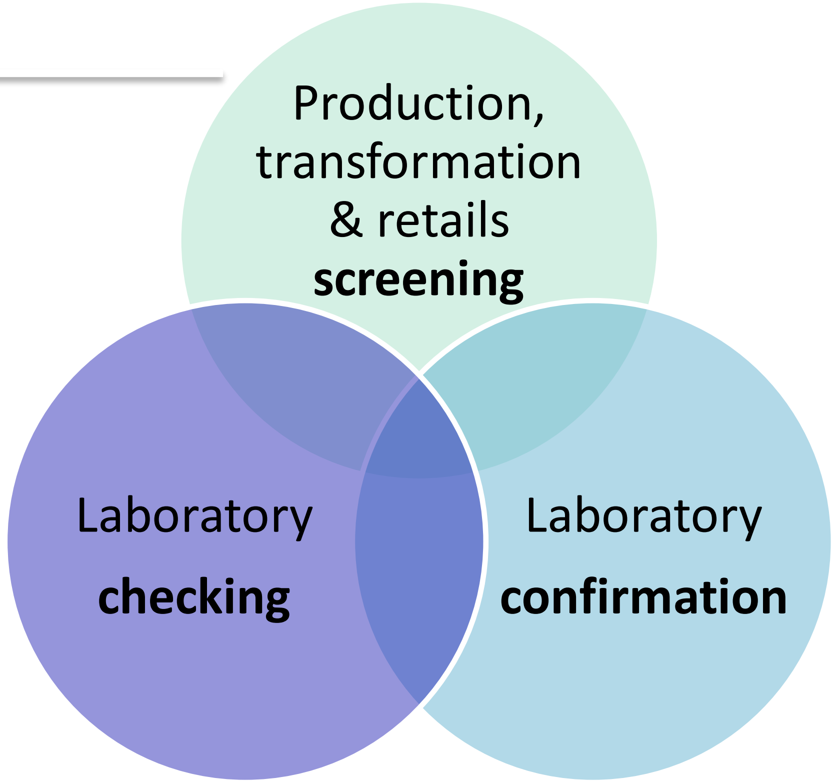
Take home message !

- ❑ ***Three-tiers spectroscopic approach for the integrity of the food chain***

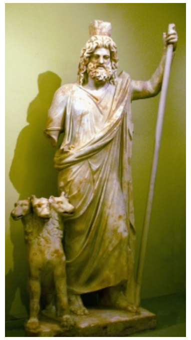
  - ***Step 1 :*** *Screening at production (plant/field/industry), transformation and retails levels*
    (NIRS on-line & handheld; Raman)

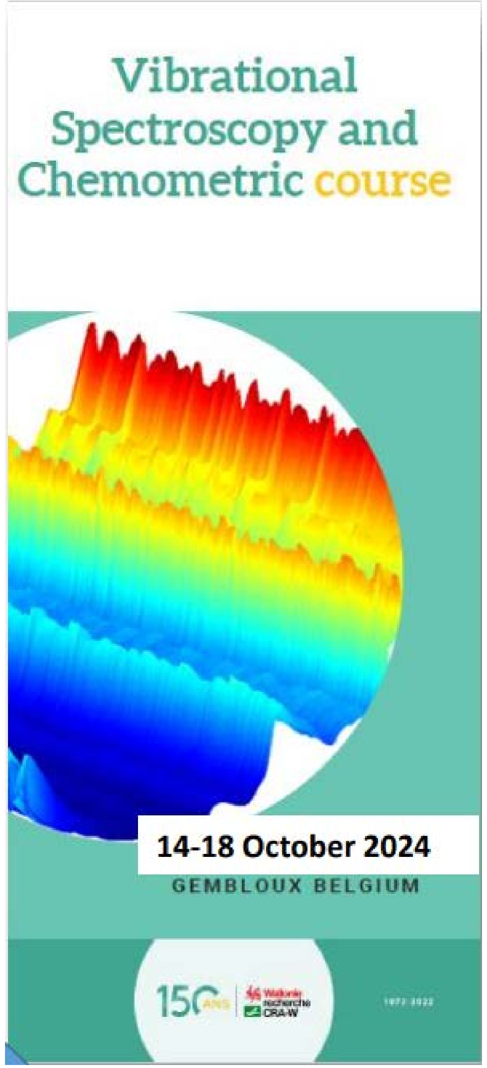
  - ***Step 2 :*** *Checking at the laboratory level*
    (NIRS laboratory; NIR HSI)

  - ***Step 3 :*** *Confirmation at the laboratory level*
    (MIR & Raman laboratory; NIR HSI)



The Cerberus approach for the protection of the food chain !




Vibrational
Spectroscopy and
Chemometric course
14-18 October 2024
GEMBLOUX BELGIUM

Merci !