AntiFraud Initiative

Good implementation practices for Articles 28 and 29 of Regulation (EU) 2018/848

Handles, keys and levers for investigation of residue cases in EU organic production

Open discussion on the concept and the content

Brussels, 25 and 26 January 2024

AntiFraud Initiative

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1 The Role of Analytical Laboratories

2 Analytical Methods

3 Result Interpretation

4 Selecting Criterias for Lab Service

5 Special Issues

The Content

1 The Role of Analytical Laboratories

-Main roles along relevant client-groups

Authorities

- Analytical Support in routine monitoring

.....along Regulation (EU) 2023/731

Control Bodies

- Targeted Support in routine monitoring

Operators

- Risk assessment by clearance analysis

The Content

1 The Role of Analytical Laboratories

- Main objectives of the assignment -

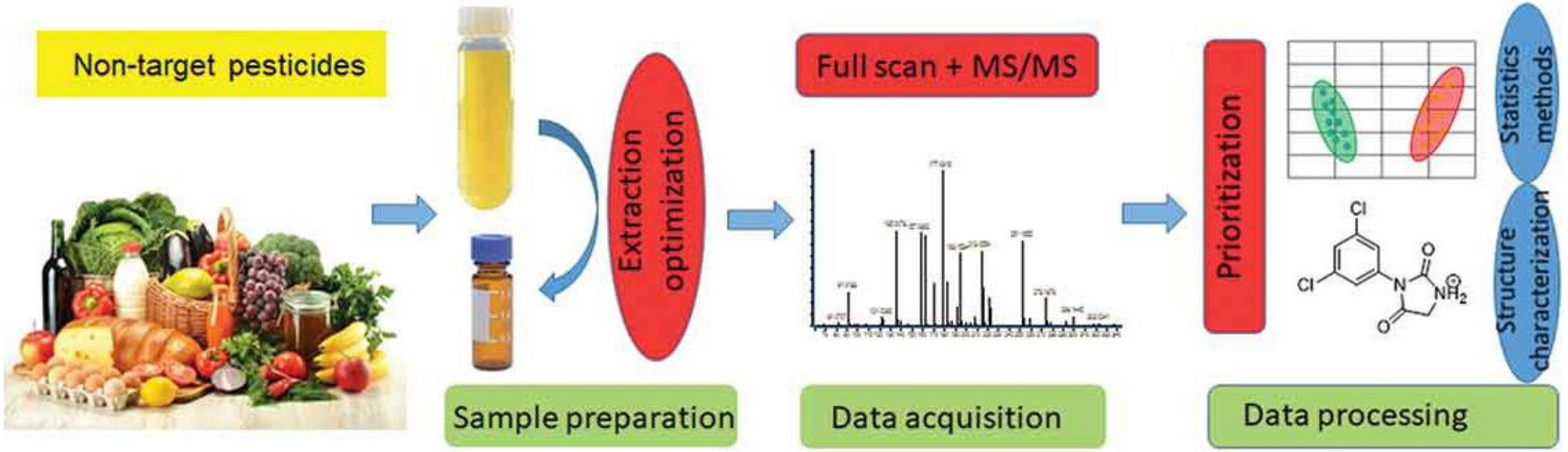
- Uncovering of suspected cases by analytical (non target) screening
- Verification of suspected cases by target analytics
- Guidance of sampling
- Interpretational support

→ Making invisible process attributes visible

The Content

2 Analytical Methods

- **Sample preparation**
- **Multi residue methods MRM**
- **Single residue methods SRM**



https://doi.org/10.1080/19440049.2020.1753890

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2 Analytical Methods

a) Sample preparation

- Sample preparation for food (Regulation (EU) 396/2005

  sample quantity is well defined (Dir. 2002/63)

  sample preparation has to follow up Annex 1 of the regulation,

  which aims in: -comparability of results

  -representativity of parts

  …..

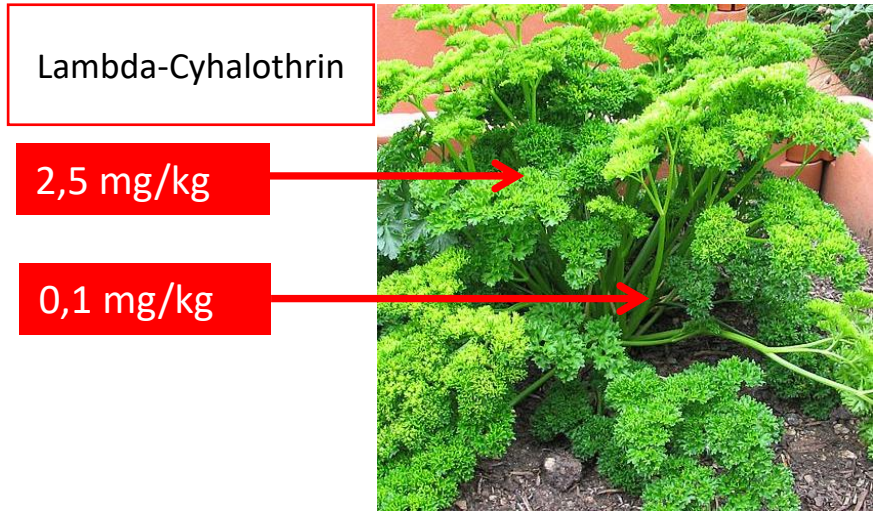
- Sample preparation for process control (i.e. Reg. (EC) 848/2018)

  sample quantity often is result of risk sampling

  matrix type is often not the end produce (leaves of potatoes)

  sample preparation may follow up a worst-case scenario (concentration)

  …..



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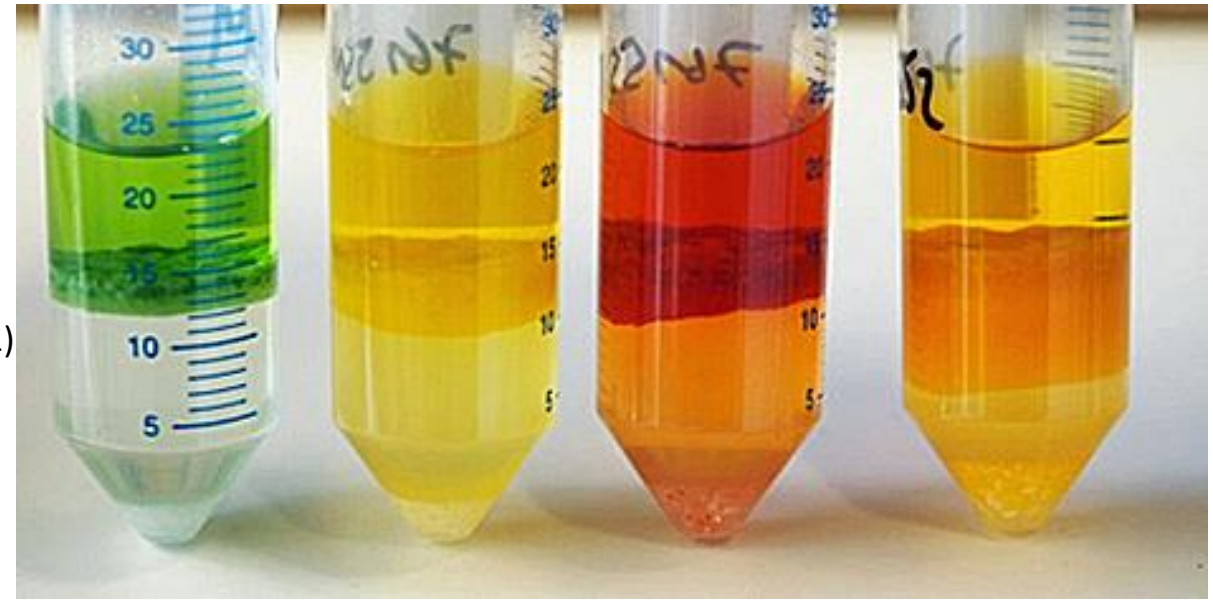
2 Analytical Methods



b) Multi residue methods MRM

- QuEChERS Multimethod
  - non target screening method
  - Type of extraction
  - number of analytes detected (i.e.226 in Reg. (EU) 2023/731)
  - Type of analytes detected
  - Validation level of analyte/matrix combination
  - LOD of each analyte with relevance
  - included scopes (food, feedstuff, consumer goods…)
- QuPPe Multimethod
  - Mostly targeted demands
  - Included anlytes: i.e. Ethephon, Chlormequat, Mepiquat, Phosphonoic acid, Perchlorate, Chlorate, Maleic acid , Cynuric acid, Bromid

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Multi method means as well multiple methods !!

There are 11 matrix groups for food and 8 matrix groups for feed defined

See SANTE/11312/2021 vs 2

Commodity groups	***Typical commodity categories wthin the group***	***Typical representative commodities within the category***
1. High water content	Pome fruit	Apples, pears
	Stone fruit	Apricots, cherries, peaches,
	Other fruit	Bananas
	Alliums	Onions, leeks
	Fruiting vegetables/cucurbits	Tomatoes, peppers, cucumbers, melons
	Brassica vegetables	Cauliflowers, Brussels-sprouts, cabbages, broccoli
	Leafy vegetables and fresh herbs	Lettuce, spinach, basil
	Stem and stalk vegetables	Celery, asparagus
	Fresh legume vegetables	Fresh peas with pods, peas, mange tout, broad beans, runner beans, French beans
	Fresh Fungi	Champignons, chanterelles
	Root and tuber vegetables	Sugar beet, carrots, potatoes, sweet potatoes
2. High acid content and high water content12	Citrus fruit	Lemons, mandarins, tangerines, oranges
	Small fruit and berries	Strawberries, blueberries, raspberries, black currants, red currants, white currants, grapes
3. High sugar and low water content13	Honey, dried fruit	Honey, raisins, dried apricots, dried plums, fruit jams
4a. High oil content and very low water content	Tree nuts	Walnuts, hazelnuts, chestnuts
	Oil seeds	Oilseed rape, sunflower, cotton-seed, soybeans, peanuts, sesame etc.
	Pastes of tree nuts and oil seeds	Peanut butter, tahina, hazelnut paste
4b. High oil content and intermediate water content	Oily fruits and products	Olives, avocados and pastes thereof

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2 Analytical Methods

b) Single residue methods SRM

- Multi element analysis
- DDAC und BAC
- Dithiocarbamates
- Pesticides requiring hydrolysis
- .....

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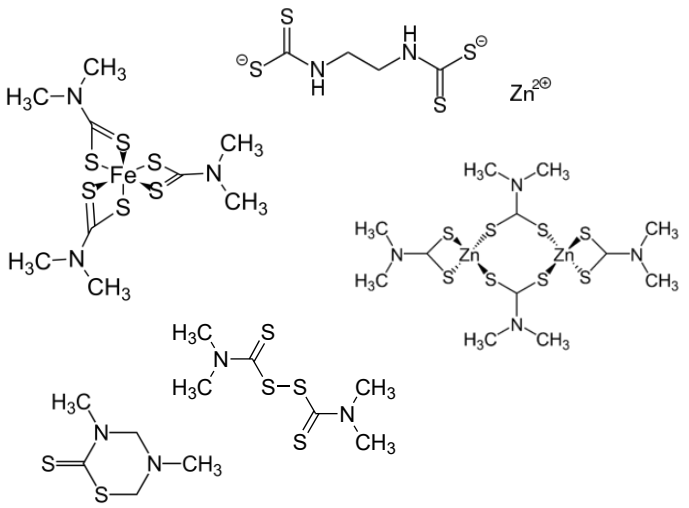
2 Analytical Methods

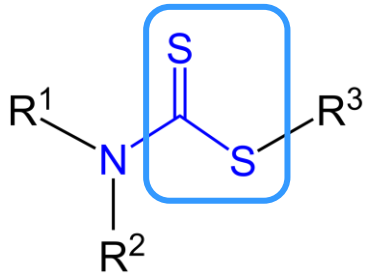
b) Single residue methods SRM

- Dithiocarbamates as they are :
  - Dazomet Disulfiram, Ferbam, Mancozeb, Maneb, Metam, Methylmetiram, Metiram, Nabam, Propineb, Thiram, Zineb, Ziram

Analysis via their common degradation product: carbon disulfide (CS2)



Makes it difficult to track back into possible application scenarios



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2 Analytical Methods

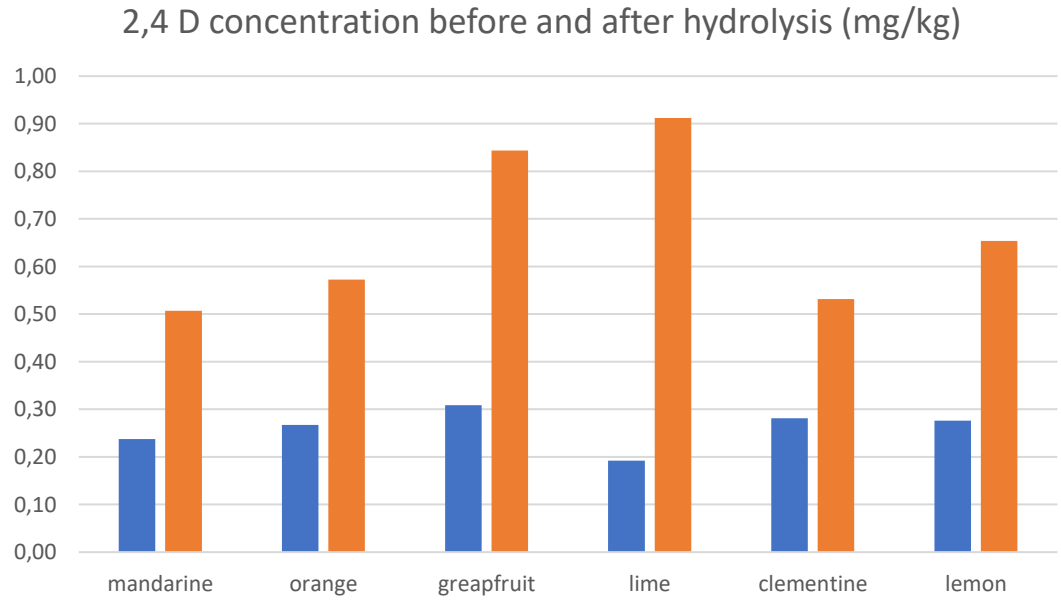
b) Single residue methods SRM

- Pesticides requiring hydrolysis, some of them are:
  - 2,4 D, MCPA, Haloxyfop, Fluzazifop ……

Some substances undergo conjugation within organisms (detoxification process)

Not available for MRM and thus have tobe hydrolysed to release the primary compound

MRM (blue) vs. SRM (orange) in citrus



The Content

3 Result Interpretation

a) Analytical background of the single analysis (SANTE/2020/12830, Rev.2)

- Applicability
- Limit of detection (LOD)
- Limit of quantification (LOQ)
- Precision
- Repeatability
- Reproducibility
- Recovery
- Selectivity
- Linearity
- Measurement uncertainty

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3 Result Interpretation

a) Analytical background of the single analysis

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depends on sensitivity analyte/matrix, i.e. azadirachtin 0,02 mg/kg

type of chromatography generates different results GC vs. LC

more quantification problem, hard to explain to customers

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3 Result Interpretation

b) Sampling background of the single analysis

- Sample size
- Type of sample matrix
- Logistic history of sample
- Processing degree
- Phenotyp of the plant
- ……

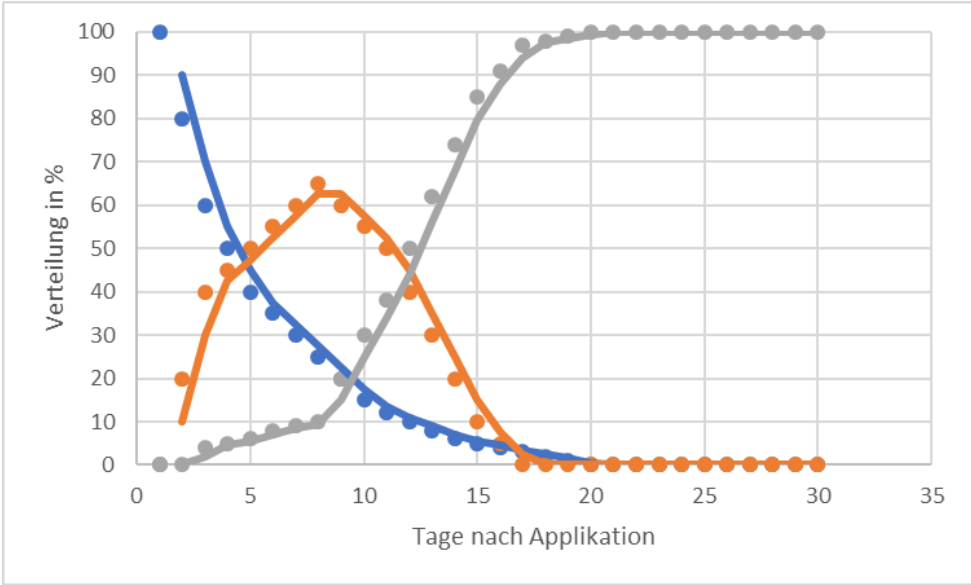
physiological age
technical process level (i.e. plantlet)

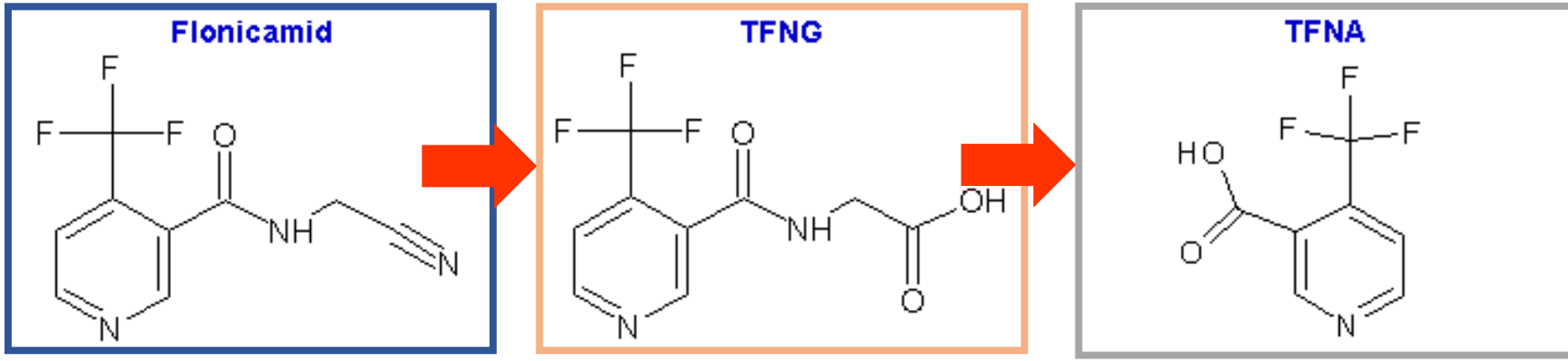
The Content

3 Result Interpretation

c) Analytes and their metabolites , isomeres

- **Conditions of metabolisation**
- Simultaneity of the occurrence of analytes
- Agronomic plausibility
- Recalculation of processed food (rehydration)





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3 Result Interpretation

c) Analytes and their metabolites , isomeres

- Conditions of metabolisation
- **Simultaneity of the occurrence of analytes**
- Agronomic plausibility
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Exemples of combined pesticides

Commercial product	active substance 1	active substance 2
Luna sensation	Fluopyram	Trifloxystrobin
Luna care	Fluopyram	Fosetyl
Signum	Boscalid	Pyraclostrobin
Switch	Cyprodinil	Fludioxonil
Signum	Boscalid	Pyraclostrobin
Folpan	Folpet	Metalaxyl-M
Curamat	Tebuconazol	Trifloxystrobin

A. Christou et al. Environmental Research 170 (2019) 422–432

The Content

4 Selecting Criterias for Lab Service

a) Accreditation
b) Designation as official laboratory
c) Expertise
d) Accessibility
e) Methodology
f) Turnaround time
g) Level of independence
h) Pricing

The Content

4 Selecting Criterias for Lab Service

a) **Accreditation**

b) **Designation as official laboratory**

c) **Expertise**

d) **Accessibility**

e) **Methodology**

f) **Turnaround time**

g) **Level of independence**

h) **Pricing**

soft criterias

- matrix experience
- experience with organic samples
- experience with sampling
- exchange networks (EURL, lab circles)
- qualifications (BNN)

- accessible contact person
- „smart“ exchange possibilities
- overlapping interests

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5 Special Issues

a) Sensitive & complex issues

- multi residue methods advantage : screening and thus fast and cost saving
- Multi residue methods disadvantage : i.e. 300.000 analyte/matrix combinations cannot be validated
- ….which makes quality assurance systems more important
- Multi source compounds (phtalimides, phosphonic acvid etc.)
- Naturally occurring compounds (giberrellic acid,….)
- Detection of esters and conjugates

b) Challenges

- competition about LOD and LOQ
- quality assurance kills time => costs money
- multi residue methods are developed by authority labs NOT for private sector
- Lack of information about samples
- Inclusion of metabolites

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Thank you