Wageningen Food Safety Research

IMPORT CONTROL IN THE NETHERLANDS

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WAGENINGEN
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Joint EURL/NRL Workshop, Fellbach, Germany, 18-20 October, 2023

WFSR in short

Food safety and authenticity
Research institute within
Wageningen University & Research

Co-workers
> 400 including PhD students
and foreign guests

History
of > 120 years

Annual turnover
37 M€

Located
on the Wageningen Campus

80% for
**Ministry of Agriculture,
Nature and Food Quality**

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Our activities

Reference institute
NRL and EURL

Method development

Measuring and detecting substances

Safe food production

Effects of substances on humans and animals

Training and consultancy

Food fraud and composition

24/7

EU Regulation No 669/2009 → **EU 2019/1793**

29.10.2019 EN Official Journal of the European Union L 277/89

COMMISSION IMPLEMENTING REGULATION (EU) 2019/1793

of 22 October 2019

on the temporary increase of official controls and emergency measures governing the entry into the Union of certain goods from certain third countries implementing Regulations (EU) 2017/625 and (EC) No 178/2002 of the European Parliament and of the Council and repealing Commission Regulations (EC) No 669/2009, (EU) No 884/2014, (EU) 2015/175, (EU) 2017/186 and (EU) 2018/1660

(Text with EEA relevance)

ANNEX I

Food and feed of non-animal origin from certain third countries subject to a temporary increase of official controls at border control posts and control points

ANNEX II

Food and feed from certain third countries subject to special conditions for the entry into the Union due to contamination risk by mycotoxins, including aflatoxins, pesticide residues, pentachlorophenol and dioxins, microbiological contamination, Sudan dyes, Rhodamine B and plant toxins

EU Regulation No 669/2009 → **EU 2019/1793**

ANNEX I

Food and feed of non-animal origin from certain third countries subject to a temporary increase of official controls at border control posts and control points

Row	Country of origin	Food and feed (intended use)	CN code (1)	TARIC sub-division	Hazard	Frequency of identity and physical checks (%)
5	**Colombia (CO)**	Granadilla and passion fruit (*Passiflora ligularis* and *Passiflora edulis*) ***(Food)***	ex 0810 90 20	**30**	Pesticide residues (3)	10
6	**Dominican Republic (DO)**	— Sweet peppers (*Capsicum annuum*)	0709 60 10 0710 80 51		Pesticide residues (3) (17)	50
		— Peppers of the genus *Capsicum* (other than sweet) ***(Food – fresh, chilled or frozen)***	ex 0709 60 99 ex 0710 80 59	**20** **20**		

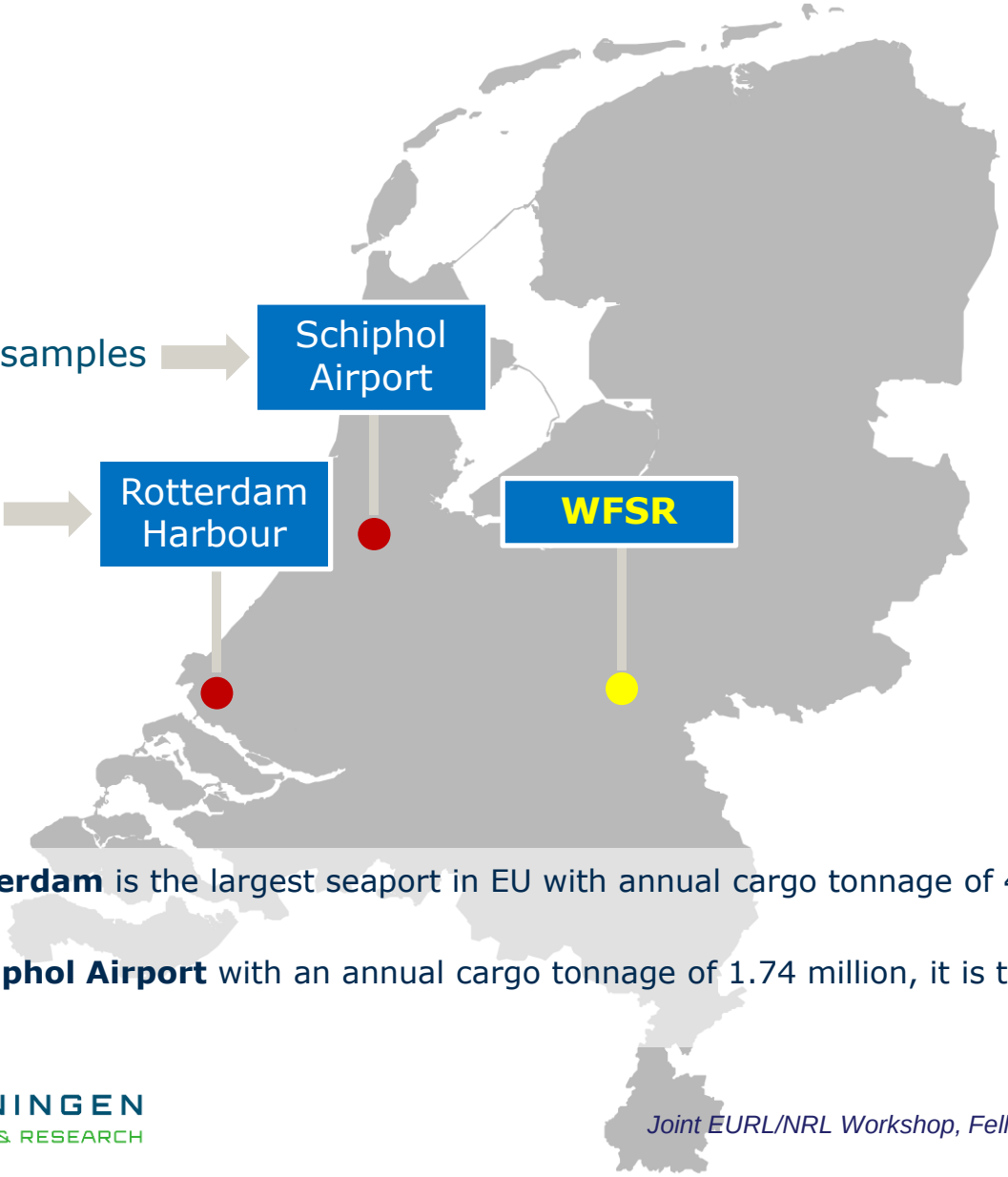
EU Regulation No 669/2009 → **EU 2019/1793, ANNEX I**

(1) Where only certain products under any CN code are required to be examined, the CN code is marked 'ex'.
(2) The sampling and the analyses shall be performed in accordance with the sampling procedures and the analytical reference methods set out in point 1(a) of Annex III.
(3) Residues of at least those pesticides listed in the control programme adopted in accordance with Article 29(2) of Regulation (EC) No 396/2005 of the European Parliament and of the Council of 23 February 2005 on maximum residue levels of pesticides in or on food and feed of plant and animal origin and amending Council Directive 91/414/EEC (OJ L 70, 16.3.2005, p. 1) that can be analysed with multi-residue methods based on GC-MS and LC-MS (pesticides to be monitored in/on products of plant origin only).
(4) The sampling and the analyses shall be performed in accordance with the sampling procedures and the analytical reference methods set out in point 1(b) of Annex III.
(5) Residues of Tolfenpyrad.
(6) Residues of Dicofol (sum of p, p' and o,p' isomers), Dinotefuran, Folpet, Prochloraz (sum of prochloraz and its metabolites containing the 2,4,6-trichlorophenol moiety expressed as prochloraz), Thiophanate-methyl and Triforine.
(7) Residues of Diafenthiuron.
(8) Residues of Formetanate (sum of formetanate and its salts expressed as formetanate (hydrochloride)), Prothiofos and Triforine.
(9) Residues of Prochloraz.
(10) Residues of Diafenthiuron, Formetanate (sum of formetanate and its salts expressed as formetanate (hydrochloride)) and Thiophanate-methyl.
(11) Reference methods: EN 1988-1:1998, EN 1988-2:1998 or ISO 5522:1981.
(12) Residues of Dithiocarbamates (dithiocarbamates expressed as CS2, including maneb, mancozeb, metiram, propineb, thiram and ziram), Phenthoate and Quinalphos.
(13) Residues of Ethylene Oxide (sum of ethylene oxide and 2-chloro-ethanol, expressed as ethylene oxide). In case of food additives, the applicable maximum residue level (MRL) is 0,1 mg/kg (limit of quantification (LOQ)). Prohibition of use of Ethylene Oxide provided for in Commission Regulation (EU) No 231/2012 of 9 March 2012 laying down specifications for food additives listed in Annexes II and III to Regulation (EC) No 1333/2008 of the European Parliament and of the Council (OJ L 83, 22.3.2012, p. 1).
(14) For the purposes of this Annex, 'Sudan dyes' refers to the following chemical substances: (i) Sudan I (CAS Number 842-07-9); (ii) Sudan II (CAS Number 3118-97-6); (iii) Sudan III (CAS Number 85-86-9); (iv) Scarlet Red or Sudan IV (CAS Number 85-83-6). Residues of Sudan dyes, using a method of analysis with an LOQ, shall be lower than 0,5 mg/kg).
(15) Both finished products and raw materials containing any botanicals intended for the production of food supplements declared under CN codes mentioned in column 'CN code'.
(16) Hereinafter understood as the State of Israel, excluding the territories under the administration of the State of Israel after 5 June 1967, namely the Golan Heights, the Gaza Strip, East Jerusalem and the rest of the West Bank.
(17) Residues of Acephate.

EU Regulation No 669/2009 → **EU 2019/1793, ANNEX I**

Country of origin	Food and feed	Frequency of identity and physical checks (%)
Brazil (BR)	Peanut, peanut butter and oilcake	30
China (CN)	Tea, whether or not flavoured	20
Colombia (CO)	Granadilla and passion fruit	10
Dominican Republic (DO)	Sweet peppers	50
Egypt (EG)	Sweet peppers and oranges	30
	Sugar apple	20
Israel (IL)	Basil, mint, guava	10
India (IN)	Betel leaves	30
	Okra, drumsticks, yardlong beans, locust beans, guar gum, cumin seeds	20
	Rice	10
Kenya (KE)	Beans	10
	Peppers	20
South Korea (KR)	Food supplements	30
	Instant noodlescontaining spices/seasonings or sauces	20
Sri Lanka (LK)	Gotukola and mukunuwenna	50
Madagascar (MG)	Black eyed beans	10
Mexico (MX)	Green papaya	20
Malaysia (MY)	Jackfruit	50
Pakistan (PK)	Rice	5
	Peppers	20
Rwanda (RW)	Peppers	20
Thailand (TH)	Peppers	30
Turkey (TR)	Lemons, Grapefruits, Pomegranates	30
	sweet pepper	20
	Cumin seeds, dried oregano, sesamum seeds, locust bean	20
Uganda (UG)	Peppers	50
Vietnam (VN)	Peppers	50
	Instant noodlescontaining spices/seasonings or sauces	20

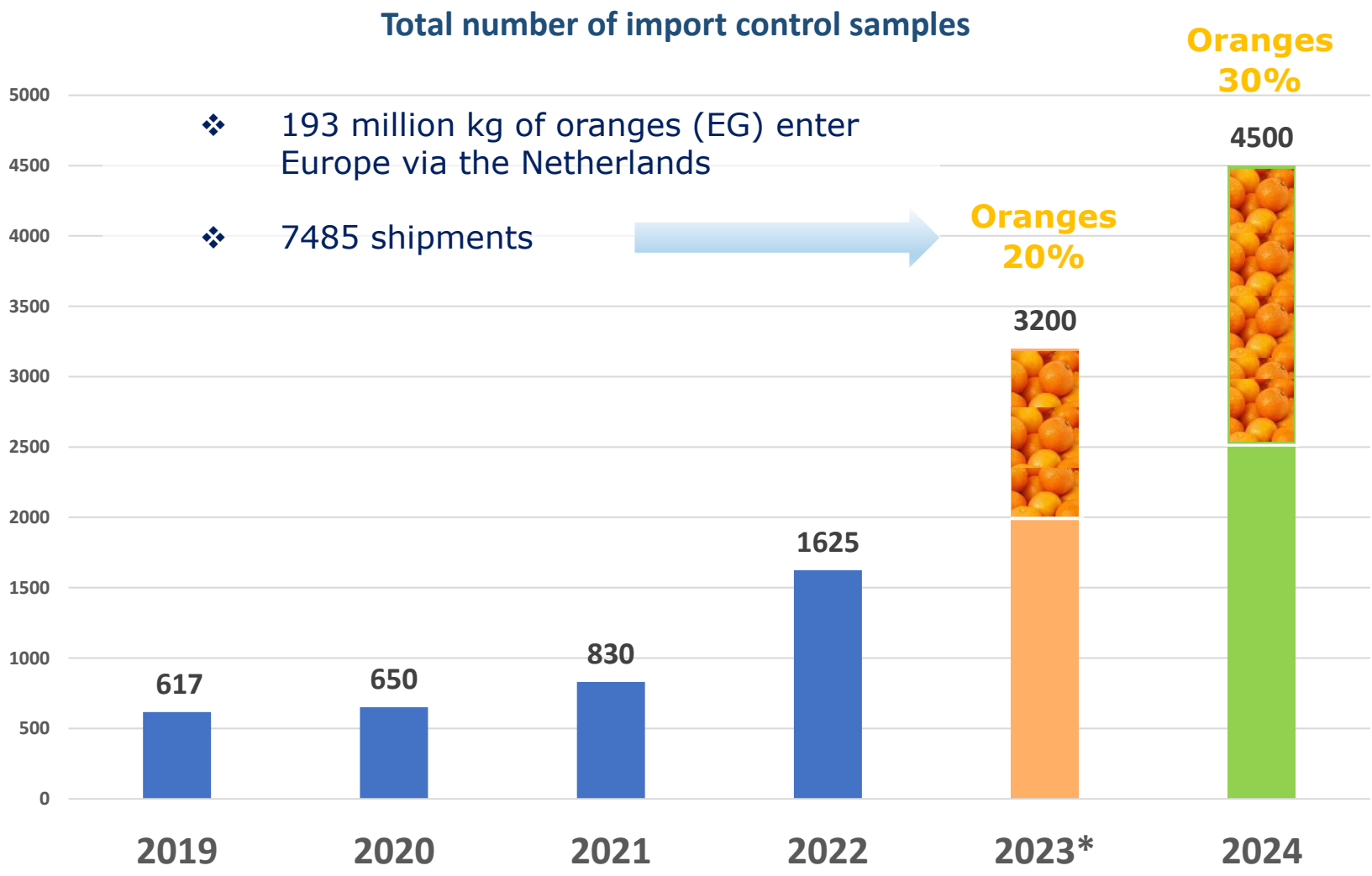
Import control – Netherland as designated point of entry



The Port of Rotterdam is the largest seaport in EU with annual cargo tonnage of 468.7 million tones (2021)

Amsterdam Schiphol Airport with an annual cargo tonnage of 1.74 million, it is the 4th busiest in Europe.

Import control - number of samples analyzed in recent years



* - realised until September

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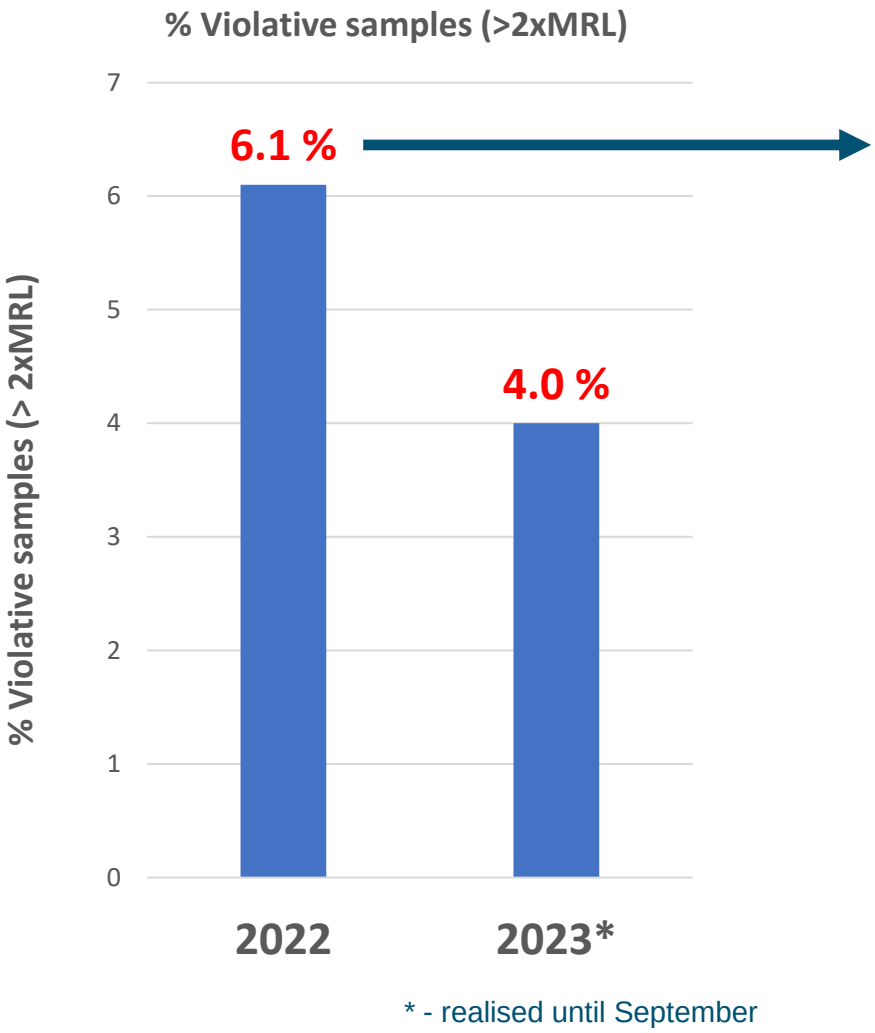
% Violative samples (>2xMRL) in years 2022 – 2023

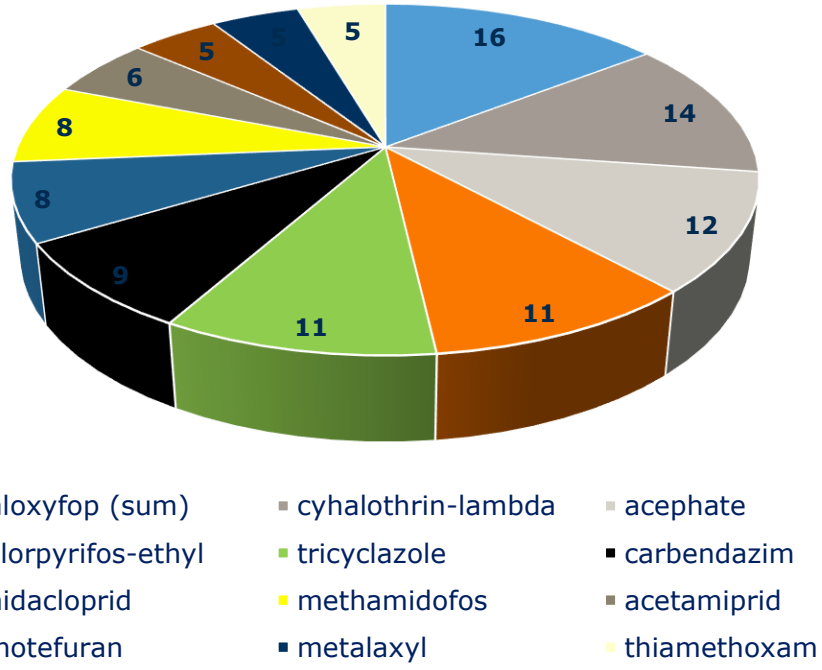


* - realised until September

<u>Coming from 14 different countries:</u>
Brazil, China, Colombia, Egypt, Ecuador, Guatemala, India, Kenya, Uganda, Pakistan, Thailand, Turkey, Vietnam, South Africa

<u>60 different pesticides were detected >2xMRL</u>

<u>12 most frequently detected pesticides:</u>



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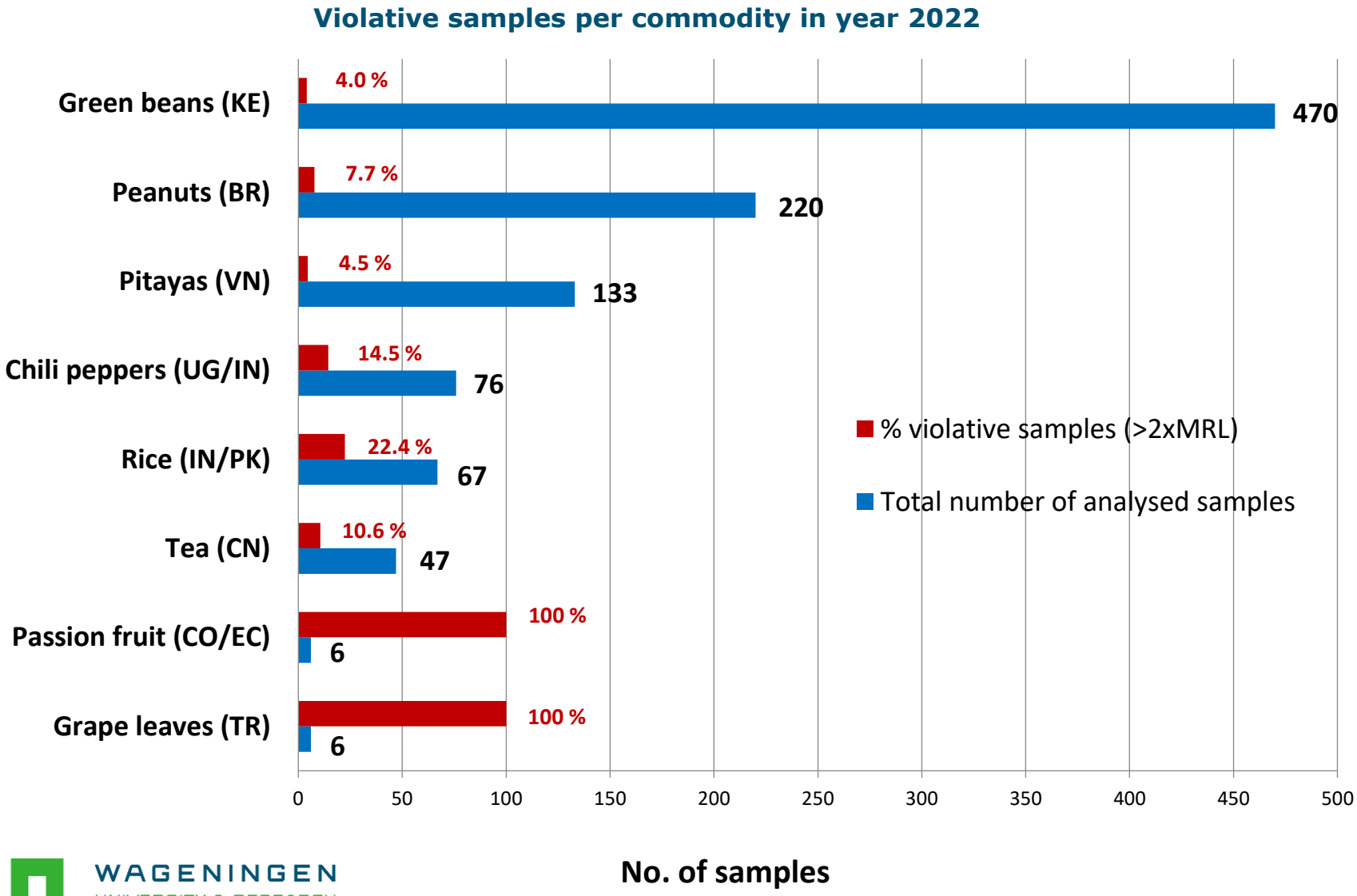
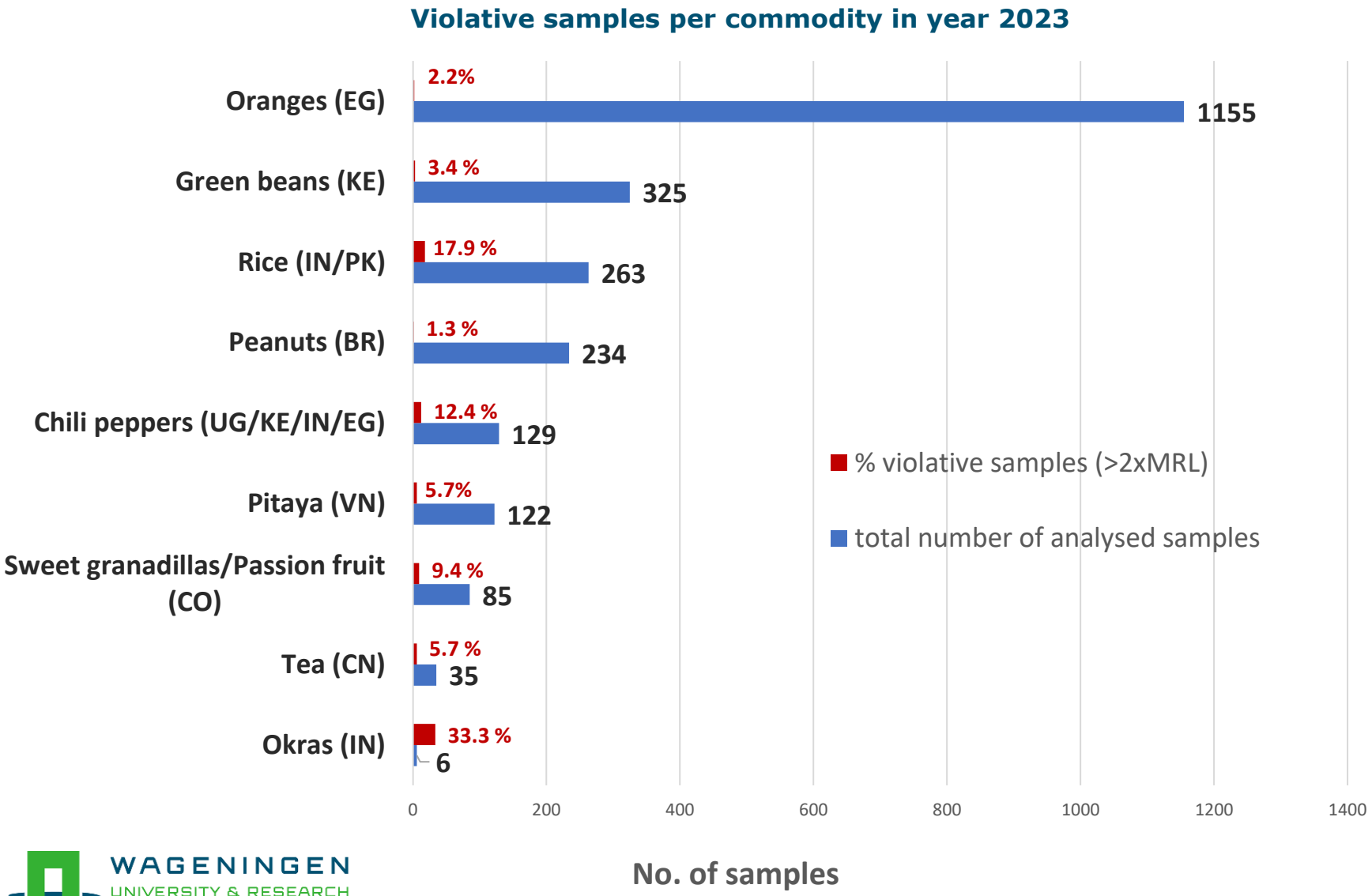
% Violative samples (>2xMRL) in years 2022



% Violative samples (>2xMRL) in years 2023



Violative samples for Ethyle-oxide in the period Jan-June 2023

Country of origin	Product	No. of analyzed samples	No. of non-compliances
Vietnam	Noodles	16	n.d.
South Korea	Noodles	130	n.d.
India	Sesam seeds	17	n.d.
	Guar gum	14	n.d.
	Okra	1	n.d.
	Ginger, saffron, turmeric (curcuma), thyme, bay leaves, curry and other spices	38	1
	Sauces and preparations thereof; mixed condiments and mixed seasonings; mustard flours and meals and prepared mustard	25	1
	Pepper	44	n.d.
	Nutmeg and cardamoms	6	n.d.
	Cloves	1	n.d.
	Seeds of anise, badian, fennel, coriander, cumin or caraway, juniper berries	41	1
	Food supplements containing botanicals	20	n.d.
China	Xanthan gum	22	n.d.
Uganda	Peppers of the genus Capsicum (other than sweet)	1	n.d.

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In total 376 samples → only 3 non-compliance!!!

Analytical methods applied for import samples at the WFSR

Analytical method/scope	Matrices analysed	Total number of samples in 2023
Multiresidue methods:		
❖ **315 pesticides LC-MS/MS** ❖ **229 pesticides GC-MS/MS**	➢ yeard long beans (KE) ➢ peppers (UG/KE/IN/EG) ➢ oranges (EG) ➢ peanuts (BR) ➢ tea (CN) ➢ passion fruit (CO/EC) ➢ okra and cumin seeds (IN) ➢ papaya (VN) ➢ rice (IN/PK)	**~2800**
SRM-methods:		
Ethylene-oxide + 2-chloroethanol	➢ sesam seeds (IN) ➢ okra (IN) ➢ herbs & spices (IN / KR) ➢ noodles (VN / KR) ➢ food suplements (KR / IN)	**~800**
Dithiocarbamates via CS_2	➢ peppers and pitahaya (VN)	**~100**

Major import control challenges

Issues:

- diversity of samples
- short delivery and turnaround time
- unpredictability of what we will receive (stand-by of staff/instruments)

- huge workload this year due to oranges samples from Egypt

During orange campaign we encountered number of analytical issues

Method and workflows needed some adjustment!!!

Import Control = A Need For Speed!

Multi-residue methods:
- LC-MS: 315 pesticides in 15 min.
- GC-MS: 229 pesticides in 22 min.

Limiting factor = analysis time!!!

Single-residue methods:
- Ethyle-oxide/2-chloroethanol: in 12 min.
- Dithiocarbametes via CS_2: in 11 min.

Limiting factor = extraction methods!!!

	Import F&V	Oranges
Nr. samples	Max 6	Max 20
Nr. Inj.	Ca. 15	Ca. 42
Sequence	6 hours	18 hours
Report Time	**Before 18:00**	**72 hours**

Reporting: before 18:00, every day!

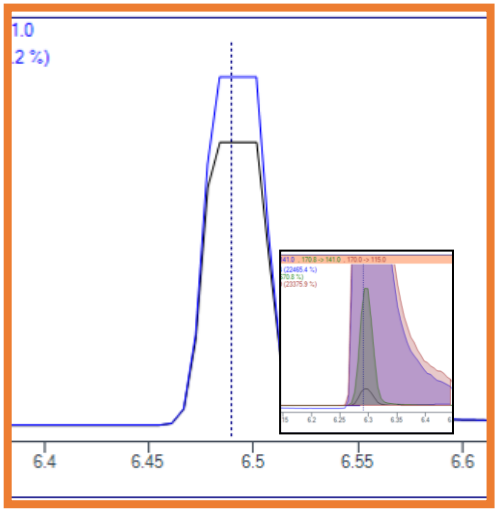
You need a robust and reliable method to reach this strict deadline!

First analysis:
- Max 3-point calibration
- 1 QC sample in the batch
- Several Internal Standards (Injection and Procedure)

Second analysis → MRL violation (>2 x MRL)
- Always standard addition approach!!!

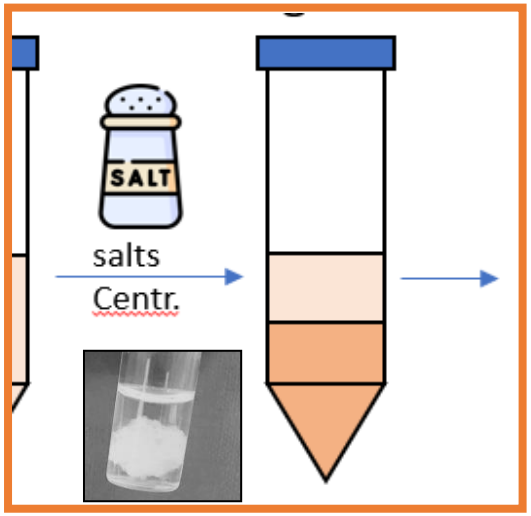
Analytical Challenges – orange sample

I



Low/High pesticide concentrations

II



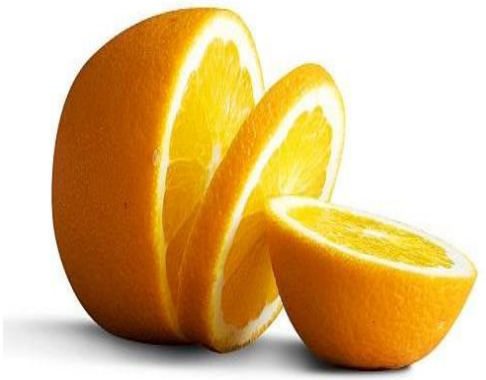
Sample preparation

III

Instrument issues: GC needles

Challenge I – high concentrations

Pesticide	MRL mg/kg	Technique
Chlorpyrifos	0.01	GC-MS/MS
Fludioxonil	10	LC-MS/MS
Imazalil	4	LC-MS/MS
Pyrimethanil	8	LC-MS/MS
Thiabendazole	7	LC-MS/MS
Phenylphenol-2	10	GC-MS/MS



LC-MS – analysis

- Calibration line in solvent 1-50 ng/mL
- Matrix conc. 0.25 g/mL
- If found `high': remeasure diluted

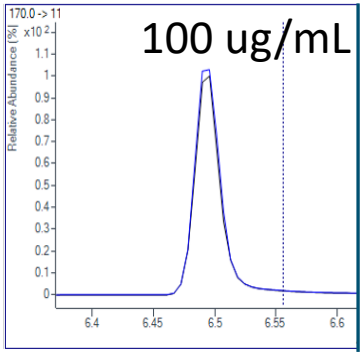
GC-MS – analysis

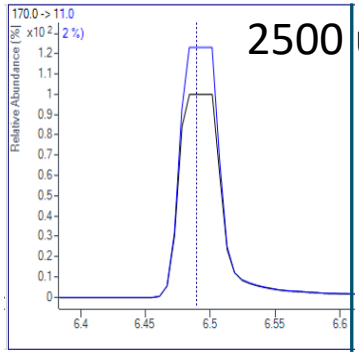
- Matrix matched calibration 1-100 ng/mL
- Matrix conc. 1 g/mL

ALL oranges had to be diluted!

- 2 processing batches
- Double measurement time

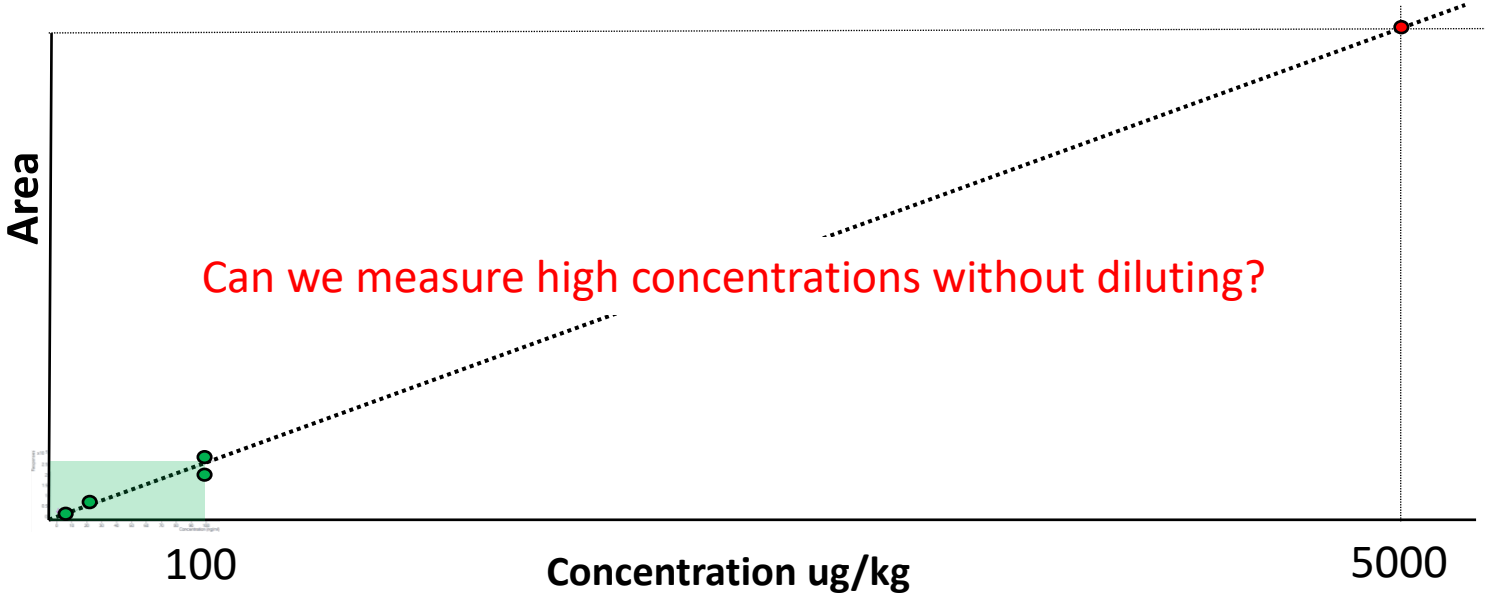
Challenge I – high concentrations





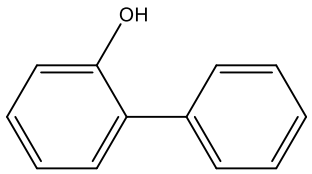
The detector is saturated ☹

Phenylphenol-2

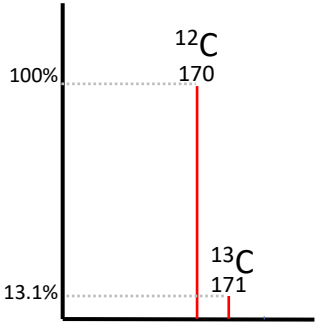


Challenge I – high concentrations

Phenylphenol-2

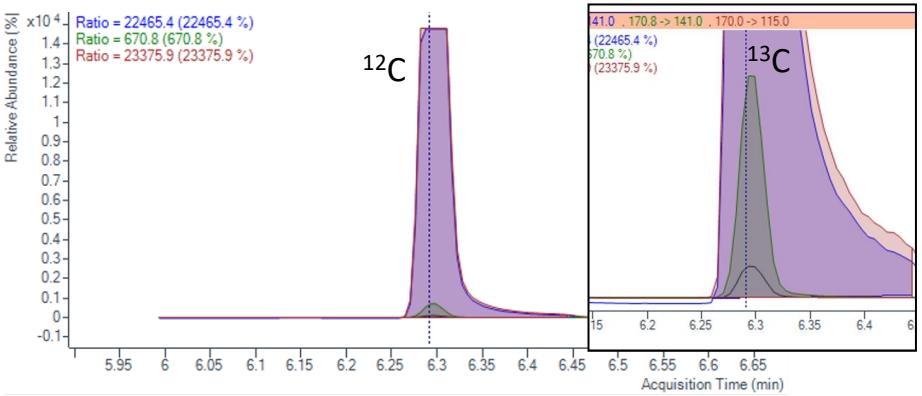


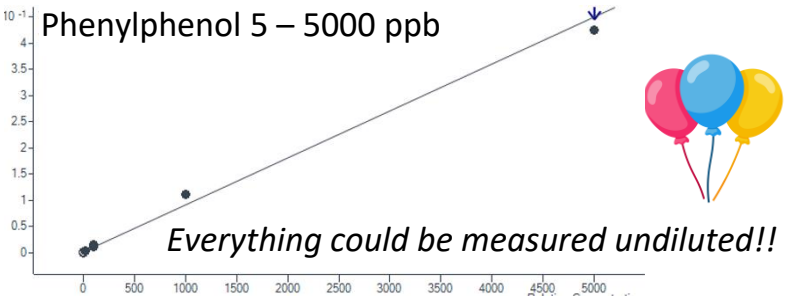
m/z: 170.07 (100.0%), 171.08 (13.1%), 172.08 (1.0%)



MRM - transition	
12C	**13C**
170 -> 141	171 -> 142
170 -> 115	171 -> 116

13C-abundance:
Every C-atom has a 1.1%
chance of being a ^{13}C-isotope

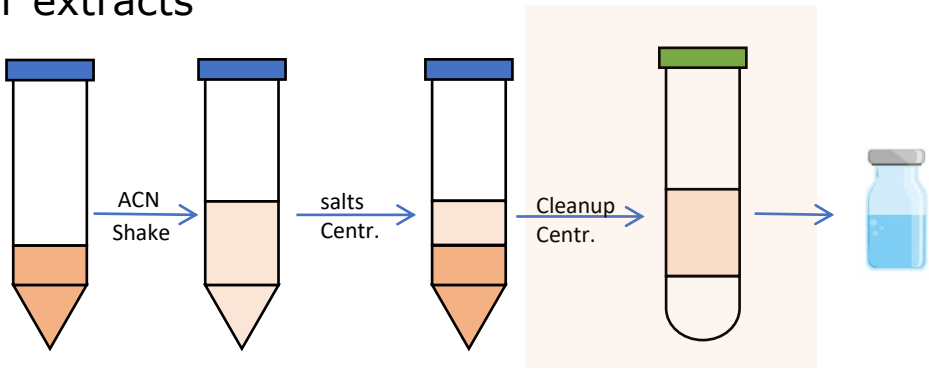


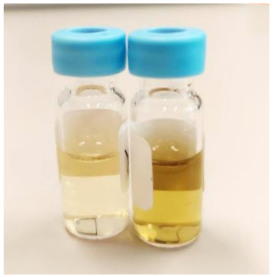


Everything could be measured undiluted!!

Challenge II – sample preparation

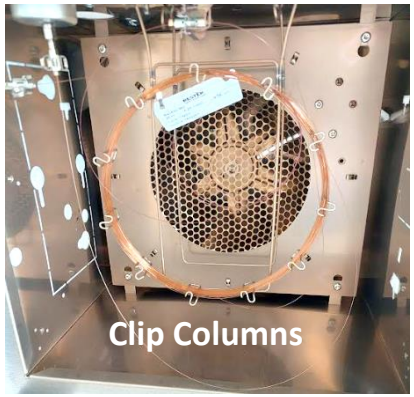
1) Cleaner extracts





Clean-up effect

2) Routine maintenance

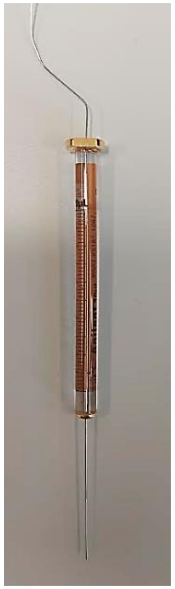


Challenge II – sample preparation

- ✓ Tried to filter the extracts → did not solve this issue
- ✓ Transfer extracts to another sample vial after couple of hours
- ✓ Modify clean-up procedure

Challenge III – Clogging Needles

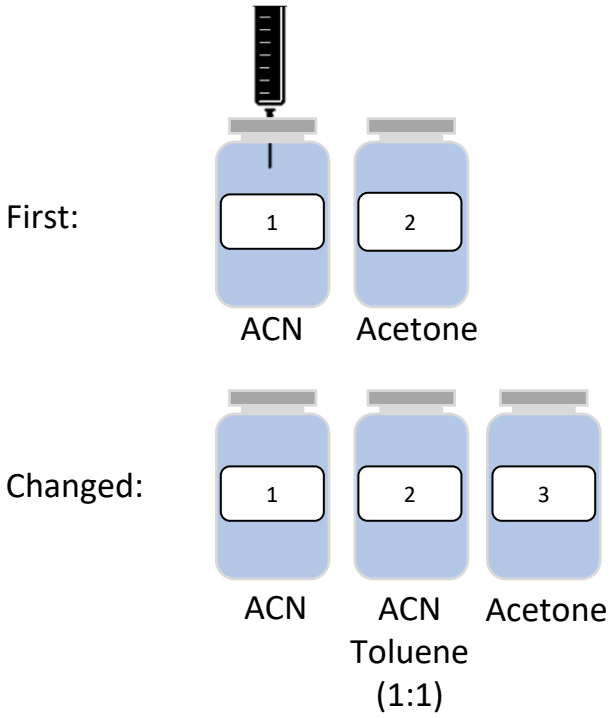
GC needles don't like oranges…..



- Completely stuck plungers → sequences aborted
- Sometimes `varnishy' layer visible
- Agilent PAL-plungers are very bendy

Challenge III – Clogging Needles

1) Changed Rinse solvents



2) Changed autosampler

Robust plungers

Thank you for your attention

Acknowledgement:
Both pesticides teams!!!

Any questions?

www.wur.eu/food-safety-research

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