VALIDATION AND UNCERTAINTY ASSESSMENT OF SAMPLING STEPS IN COMPLEX FOOD MATRIX ANALYSIS

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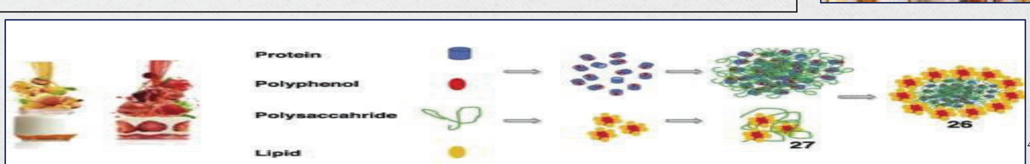
Outline

- Introduction
- Sampling and Sampling Plans
- ISO 17025 and Sampling
- Validation and Uncertainty Estimation of Sampling Step
- Combined Measurement Uncertainty
- Compliance Assessment/Interpretation of Results
- Conclusion

1.Introduction

➢ Foods as a complex matrices

- Foods are defined by major nutrients: **proteins, fats, carbohydrates**
- They also contain **minor components**: salts, vitamins, minerals (e.g., sodium, calcium, iron), and additives
- These components are **not free or uniformly distributed**, but embedded in complex **food microstructures** (the **food matrix**).
- **Residues and contaminants** are also present in the matrix in a **non-uniform (heterogeneous)** form



2.Sampling and Sampling Procedure

➢ Measurement Techniques

- ➢ **Ex situ measurement techniques** involve:
  - ✓ **Primary sampling** (e.g., selecting a batch of lettuce heads)
  - ✓ **Chemical analysis** conducted on the extracted sample
- ➢ **In situ measurement techniques** (e.g., portable XRF):
  - ✓ **Direct measurement** from the target without extracting a physical sample

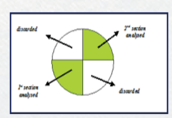
Common Steps in Analysis of Complex Food Matices

External operations

Sampling → 

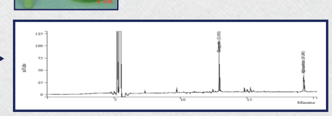
Laboratory operations

Sample preparation → 

Sample size reduction → 

Sample processing → 

Exctraction and clean-up → 

Determination → 

Reporting → 1.5±0.5 mg/kg

➢ Sampling and Food Analysis

- Accuracy and validity of analytical results rely on sample quality
- Sampling selects a representative portion from a larger batch or target
- The purpose is to ensure the sample reflects the overall composition accurately
- **A sample** consists of one or more units from a population or a portion of a larger material quantity

- Analytical measurements in food analysis use small samples representing the whole material
- Proper sampling is essential due to variability and heterogeneity in food matrices and lots
- A representative sample that reflects the parent population is needed for reliable results
- Selecting an appropriate, internationally recognized sampling procedure is a crucial first step
- Sampling procedure is the method used to collect and form a sample

(Thompson, 1998, IUPAC, 1990; Codex, 1999; EC, 2002Lyken et al., 1957; Thompson and Ramsey, 1995; Nocerino, et al., 2005).

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Methods of Sampling for Food Analyses

✓For the control of levels of mycotoxins: Commission Regulation (EC) 401/2006

✓For the control of levels of lead, cadmium, mercury, inorganic tin, 3-MCPD and benzo(a)pyrene in foodstuffs: Commission Regulation (EC) No 333/2007

✓For the control of levels of dioxins, dioxin-like PCBs and non-dioxin-like PCBs in certain foodstuffs: Commission Regulation (EU) 2017/644 of 5 April 2017 laying down methods of sampling and analysis for the control of levels of dioxins, dioxin-like PCBs and non-dioxin-like PCBs in certain foodstuffs and repealing Regulation (EU) No 589/2014

✓For the control of levels of nitrates: Commission Regulation (EC) 1882/2006

✓For the control levels of pesticide: Commission Directive 2002/63/EC of 11 July 2002 Sampling for the official control of pesticide residues in and on products of plant and animal

✓For the control of microroganism: Consolidated text: Commission Regulation (EC) No 2073/2005 of 15 November 2005 on microbiological criteria for foodstuffs

✓General Guidelines on Sampling (CAC/GL 50- 2004)

➢ISO has released sampling standards both for specific products and general application;

✓ ISO 18593:2004 Microbiology of food and animal feeding stuffs -- Horizontal methods for sampling techniques from surfaces using contact plates and swabs

✓ ISO 5667-1-24 6:2014 Water quality – Sampling

✓ ISO 28590:2017 Sampling procedures for inspection by attributes -- Introduction to the ISO 2859 series of standards for sampling for inspection by attributes

✓ ISO 28591:2017 Sequential sampling plans for inspection by attributes

✓ISO 28592:2017Double sampling plans by attributes with minimal sample sizes, indexed by producer's risk quality (PRQ) and consumer's risk quality (CRQ)

✓ISO 28593:2017 Acceptance sampling procedures by attributes -- Accept-zero sampling system based on credit principle for controlling outgoing quality

✓……..

➢ Random Sampling

✓Random sampling gives each item in the food population an equal chance of being selected for analysis

➢ Stratified sampling

✓Stratified sampling divides the population into subgroups based on key variation factors

✓Samples are randomly chosen from each subgroup to ensure representativeness and reduce bias

✓This method is ideal for monitoring pesticide residues in plant commodities and soil

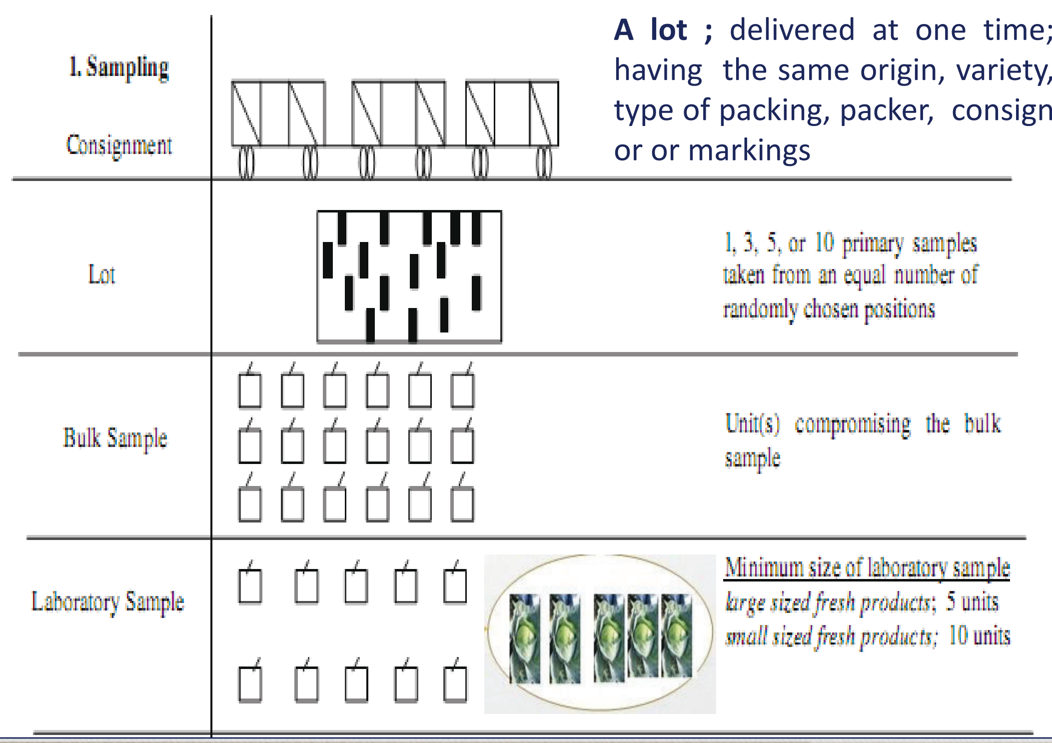
✓Stratification factors can include crop type, cultivation method, growing season, or soil type

✓Random samples are independently collected from each stratum

(Thompson, 1998, IUPAC, 1990; Codex, 1999; EC, 2002Lyken et al., 1957; Thompson and Ramsey, 1995; Nocerino, et al., 2005).

Sampling Procedure: Example

A- For pesticide residue analysis to comply with MRL (CAC 1999; EC 2002)



Minimum number of primary samples (CAC 33, 1999; EC 2002/63)

	Minimum number of primary samples to be taken from the lot
a) Meat and poultry	
a non-suspect lot	1
a suspect lot	determined according to Table 2 given in the guideline (CAC, 1999)
b) Other products	
i) Products, packaged or in bulk, which can be assumed to be well mixed or homogenous	1
ii) Products, packaged or in bulk, which may not be well mixed or homogenous	
either:	
Weight of lot, kg	
<50	3
50-500	5
>500	10
or	
Number of cans, cartons or other containers in the lot	
1-25	1
26-100	5
>100	10

Description of primary samples and minimum size of laboratory samples for fresh food commodities (CAC, 1999).

	Examples	Nature of primary samples to be taken	Minimum size of each laboratory sample
Class A, primary food commodities of plant origin			
1. All fresh fruits and vegetables			
Small sized fresh products units generally >25 g	berries peas olives	Whole units, or packages, or units taken with a sampling device	1 kg
Medium sized fresh products units generally 25-500 g	apples oranges	Whole units	1 kg (at least 10 units)
Large sized fresh products units generally>250 g	cabbages cucumbers grapes (bunches)	Whole units	2 kg (at least 5 units

[1] details for other commodities can be found in Table 4 of the Codex guideline (CAC, 1999).

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B- For mycotoxins *(EC 401/2006; EC Guidance Documentt for aflatoxin sampling 2010)*

General survey of the method of sampling for dried figs, groundnuts and nuts

Table 1

Subdivision of lots into sublots depending on product and lot weight

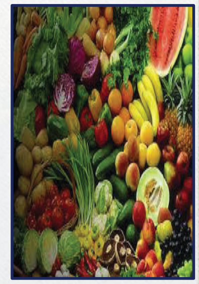
Commodity	Lot weight (tonnes)	Weight or number of sublots	Number of incremental samples	Aggregate sample weight (kg)
Dried figs	≥ 15	15-30 tonnes	100	30
	< 15	—	10-100 (*)	≤ 30
Groundnuts, pistachios, brazil nuts and other nuts	≥ 500	100 tonnes	100	30
	> 125 and < 500	5 sublots	100	30
	≥ 15 and ≤ 125	25 tonnes	100	30
	< 15	—	10-100 (*)	≤ 30

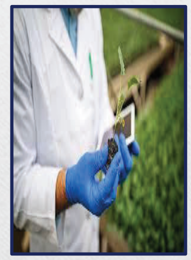
(*) Depending on the lot weight — see Table 2 of this part of this Annex.

3.Validation and Uncertainty Estimation of Sampling Step

Validation, Uncertainty and Sampling

- Sampling validation is vital for accurate, representative results in complex food matrices
- Analytical result reliability depends on validating all measurement steps, especially sampling
- Traditional validation often ignores the sampling process, focusing only on the analytical method
- Measurement uncertainty was mainly considered for the lab phase in the past
- Awareness of sampling uncertainty is growing and becoming increasingly recognized





ISO 17025:2017 and Sampling

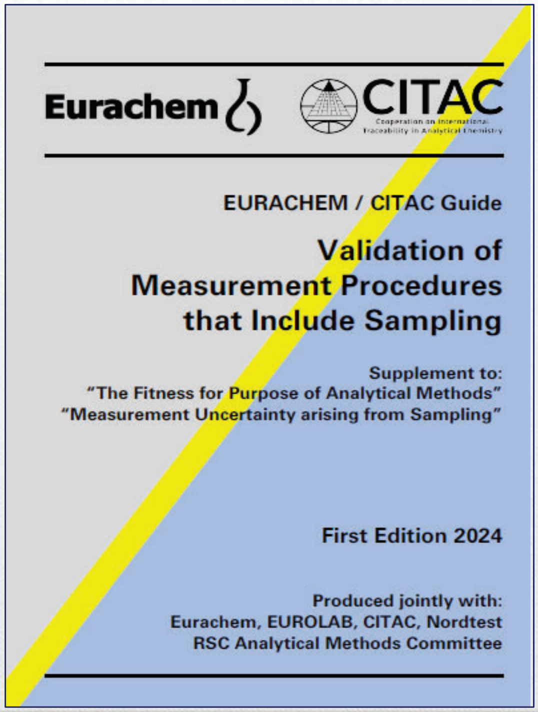
3. Terms and Definition

- Laboratory; Body that performs one or more of the following activies
  - ✓ test; calibration; and sampling, associated with subsequent testing or calibration

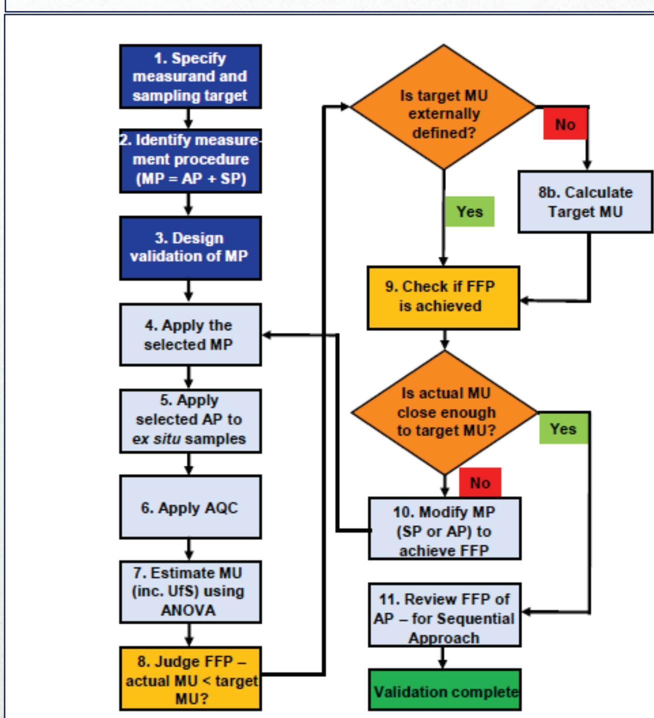
- **7.3 Sampling, 7.3.1**
  - ✓ The laboratory shall have a sampling plan and method when it carries out sampling of substances, materials or products for subsequent testing or calibration
  - ✓ Sampling plans shall, whenever reasonable, be based on appropriate statistical methods.

- **7.2.2.1** Validation can include sampling step as well
- **7.6.1** Laboratories shall identify the contributions to measurement uncertainty. When evaluating measurement uncertainty, all contributions that are of significance, including those arising from sampling, shall be taken into account using appropriate methods of analysis.

Validation and Sampling: VaMPIS Procedure

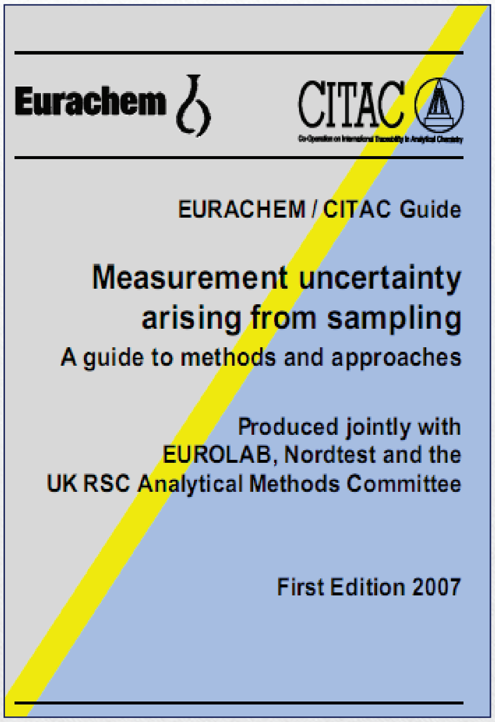

Eurachem
CITAC
Cooperation on International Traceability in Analytical Chemistry
EURACHEM / CITAC Guide
Validation of Measurement Procedures that Include Sampling
Supplement to:
"The Fitness for Purpose of Analytical Methods"
"Measurement Uncertainty arising from Sampling"
First Edition 2024
Produced jointly with:
Eurachem, EUROLAB, CITAC, Nordtest
RSC Analytical Methods Committee

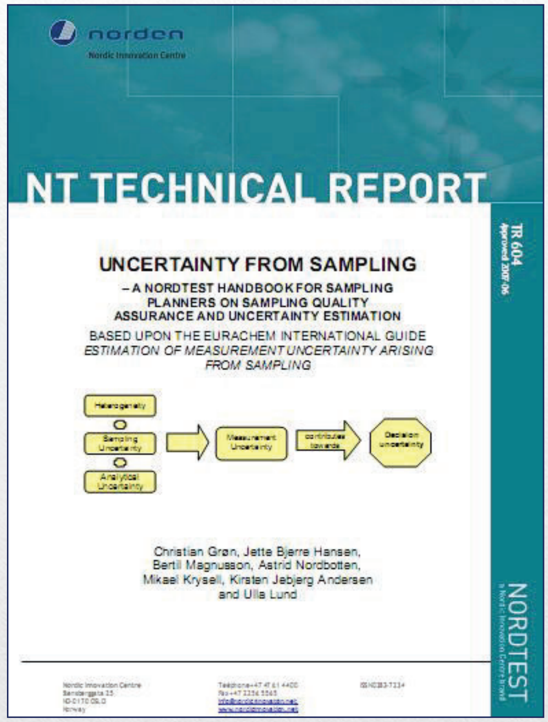
Validation and Sampling: VaMPIS Procedure



- Measurement uncertainty reflects both random and systematic errors across the whole process.
- The **VaMPIS** approach compares this uncertainty to a target to check if the procedure is suitable.
- The target uncertainty includes sampling and is set by regulators or users.
- The **Optimum Uncertainty** method can help define this target.

Sampling Uncertainty





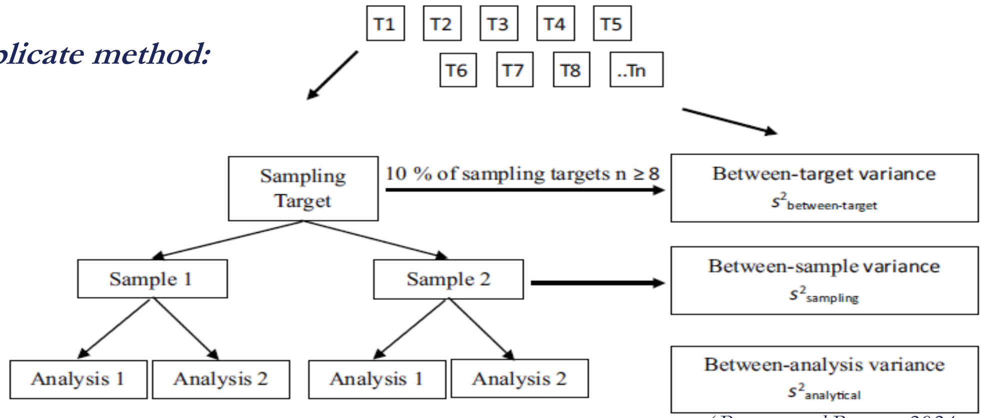
Approaches to estimate sampling uncertainty

- **Modeling approach:** Uses a predefined model to identify, quantify, and combine all sources of uncertainty for an overall estimate
- **Empirical approach:** Relies on repeated sampling and analysis to measure variability due to analyte heterogeneity and sampling differences

  ✓**Duplicate method:** A single sampler takes duplicate samples from at least 8 sampling targets, representing at least 10% of the lot but no fewer than eight targets

(*EUROCHEM CITAC Guide 2007)*

Duplicate method:



(Ramsey and Rostron 2024

- *Calculations:* ANOVA (Analysis of Variance) and Range Satistic
- Increasing duplicate samples from 8 to 16 slightly lowers sampling uncertainty confidence intervals *(Lyn et al. 2007b)*
- At least 6 replicate samples from 8–12 lots are recommended to keep sampling uncertainty within a 50% 95% confidence range *(Farkas et al. 2014)*

5.Sampling Uncertainty in Food Safety Analyses

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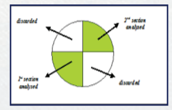
Combined Measurement Uncertainty

CV_R

- CV_S — Sampling

- Sample preparation

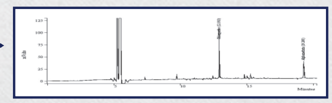
- CV_{Size} — Sample size reduction



- CV_{SS} — Sample processing

- CV_A — Extraction and clean-up

- Chromatographic determination



- Reporting → 1.5±0.5 mg/kg

$$S_R = \sqrt{(S_S)^2 + \left((S_{Sizereduction})^2 + (S_{Sp})^2 + (S_A)\right)^2} \quad \Leftrightarrow \quad CV_R = \sqrt{(CV_S)^2 + (CV_L)^2}$$

External steps

Analytical Steps in laboratory (S_L)

$$S_R = \sqrt{(S_S)^2 + (S_L)^2}$$

$$CV_L = \sqrt{(CV_{Sizereduction})^2 + (CV_{Sp})^2 + (CV_{Ex})^2 + (CV_{Clean-up})^2 + (CV_{ch})^2}$$

$$CV_{Lc} = \sqrt{CV_{SS}^2 + CV_{Sp}^2 + CV_A^2}$$

Uncertainty of laboratory phase

$U = 2 \times S_R$

Result =x±U (mg/kg)

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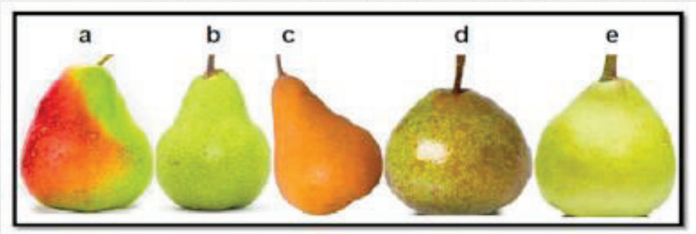
Sources of Errors during Sampling of Food

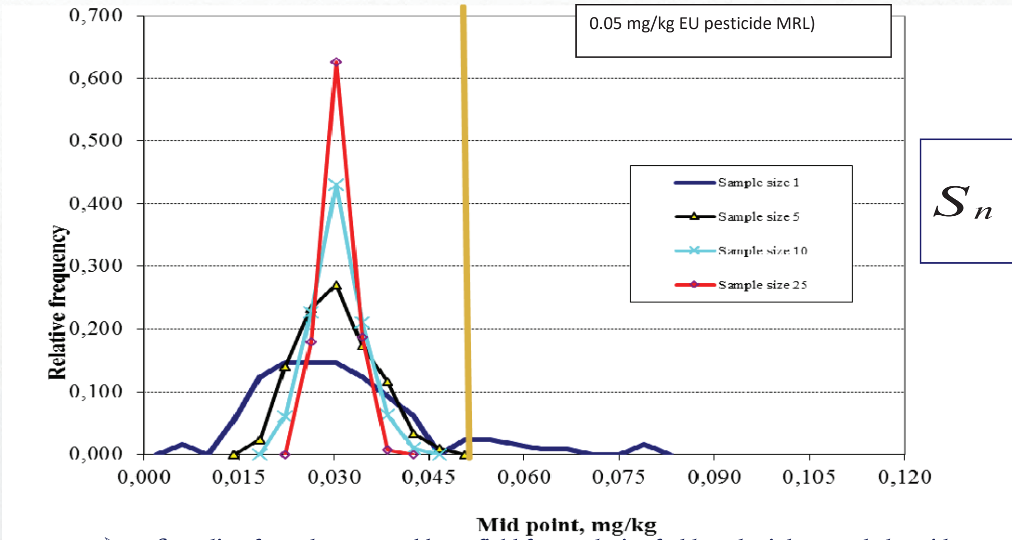
- Sampling target selection
- Type of the commodity
- Sampling plan and method
- Effects of specific sampling strategy (e.g, random, strafied random, proportional etc.)
- Effects of movement of bulk medium (Particularly density or size selection)
- Physical state of bulk (solid, liquid, gas)
- Effect of sampling process on composition (e.g differential adsorption in sampling system)
- Contamination of the sample
- Degradation, evaporation of analyte
- Mislabelling
- Transportation and preservation of sample
- Temperature and pressure effects
- Varying temperature during shipping and storage

(Ramsey and Ellison, 2007; Ambrus et al.2023)

- Distribution of the analayte in the lot
- Heterogeneity
- Variability
  - ✓**Variations** in sample composition arise from heterogeneity, contamination, analyte loss, or incorrect sampling plans
  - ✓Such variations occur between random samples from a food lot
  - ✓These variations contribute to uncertainty in the sampling step and must be considered



➢ Size of the primary sample/incremental sample



➢ *Sampling from the same cabbage field for analysis of chlrorphyriphos-methyl residue*

$$S_n = \frac{S_i}{\sqrt{n}}$$

- ✓ Sampling uncertainty decreases as the sample size (number of primary samples, n) increases
- ✓ Sampling uncertainty depends on the variability (CV_1) of residues in crop units or single sample increments

Typical CV values of pesticide residues in market and field samples.

	CV^1 of samples of size n			
Sample size (n)	*1*	*5*	*10*	*25*
Market samples	1.1	0.47	0.34	0.21
Field samples	0.8	0.356	0.25	0.16

[1]Except Brassica leafy and leafy vegetables.

(Horvat et al.2013)

➢Residue levels in individual crop units from the same field can vary significantly—by up to 80–100 times (within-field variability).

➢Therefore, increasing the number of samples ('n') reduces result uncertainty and enhances the accuracy of the estimated average residue

➢ **Aflatoxin**

➢ Sampling uncertainty is the largest contributor to total variance in aflatoxin analysis

➢ This is due to high variability among contaminated units

➢ Sampling contributed about 99% of the total variance in aflatoxin analysis of a 10 kg hazelnut sample (10 ng/g).

➢ Sample preparation accounted for 0.42% of the variance

➢ Another factor contributed 0.14% of the variance (Whitaker *et al.*, 2007a;-b)

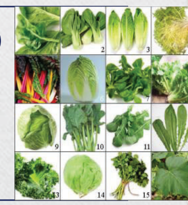
➢ Sampling uncertainty can contribute 80–90% of the combined uncertainty in **pesticide residu**e analysis results (*Ambrus and Lantos 2002; Ambrus 2011;Farkas vd 2014; Ambrus 2009*)

✓ Middle and small size crops (n≤250 g, apple): **CV_s= %25 (n=10)**

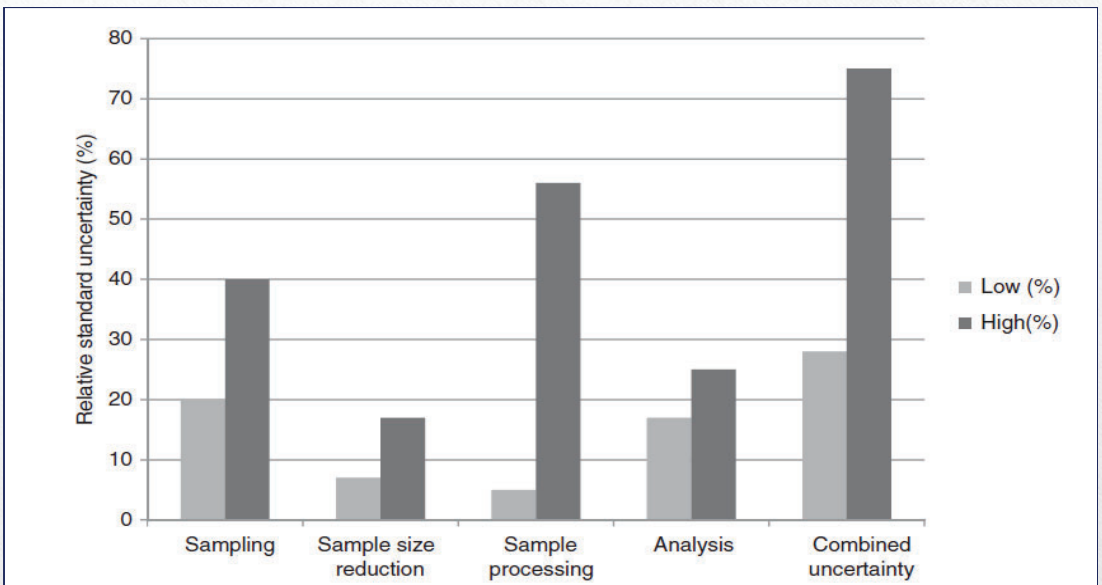
✓ Large size crops (n>250 g, cabbage): **CV_s= %33 (n=5)**

✓ Root vegetable crops (carrot): **CV_s=%19 (n=10)**

✓ Green leafy vegetables (n>250 g, lettuce): **CV_s=%20 (n=5)**



The contribution of individual steps to combined measurement uncertainty in pesticide residue analysis for fruit and vegetables



(*Yolci Omeroglu 2013c; Ambrus vd. 2011;Farkas vd 2014; Ambrus 2009*)

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The contribution of individual steps to combined measurement uncertainty in pesticide residue analysis for **homogenous** paprika samples

CV sampling, subsampling and analysis for each pesticide.

Pesticide	C (mg/kg)	CV_s	CV_{sp}	CV_{an}	CV_{res}
Fludioxinil	0.090	5.9%	6.6%	2.2%	9.1%
Cypermetrine	0.012	3.7%	7.2%	5.9%	10.0%
Acetamiprid	0.072	4.2%	7.1%	2.7%	8.7%
Spirotetramat	0.016	5.9%	4.9%	4.2%	8.8%

(Veiga-del Bono, et al. 2023)

4.Sampling Uncertainty and Compliance Assessment

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Compliance Assessment

➢Article 14(6) of Regulation (EC) 178/2002 provides that "where any food which is

✓unsafe is part of a batch, lot or consignment of food of the same class or description, it shall be presumed that all the food in that batch, lot or consignment is also unsafe, unless following a detailed assessment there is no evidence that the rest of the batch, lot or consignment is unsafe".

- **Each lot should be sampled separately** to ensure accurate assessment
- **Residue variation between fields** is typically **2–3 times greater** than within a single field due to differences in average residue levels
- In **mixed consignments**, non-compliant lots may go **undetected**
- Therefore, **sampling of mixed lots should be avoided** whenever possible

(Hill, 2000; Ambrus, 2004)

MRL and ML values

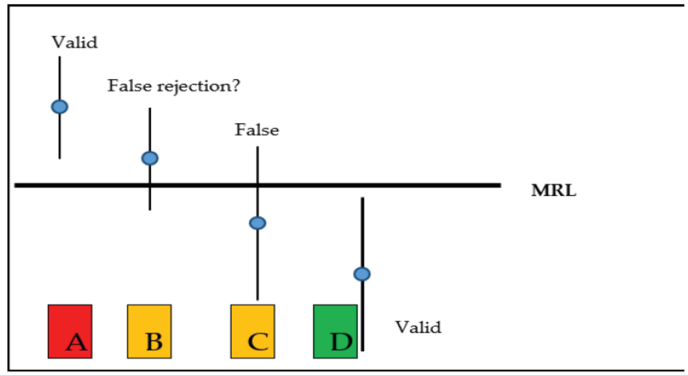
➢**The Maximum Residue Level (MRL)** and **Maximum Level (MLs)** are defined as the maximum legally permissible average concentration of chemical contaminants/residue in a composite sample with specified minimum mass and size in terms of the number of primary samples (single-sample increments)

➢The sample sizes defined in the related regulation

(Farkas et al., 2015;EC 1881/2006, 396/2005, etc.)

Pesticide Residue Analyses as an Example

- **A is rejected** and **D is accepted** based on clear criteria
- For **cases B and C**, a decision requires **pre-defined decision rules**.
- Without a decision rule, the **outcome remains uncertain**



- **Acceptance or rejection** of results depends on **national legislation** when **no Codex guidance** is available
- **Codex dispute guidelines** recommend that **trading partners agree in advance** on acceptance criteria
- **Different sampling plans** are needed for compliance testing **before and after marketing**
- **Accurate compliance assessment** requires considering the **uncertainty of measured residue concentrations**

- **MRL** refers to the **average residue** in the specified portion of a **composite sample**, for both local and imported commodities.
- **Sample mass and number of primary samples** must follow the **Codex Standard** or **Commission Directive 2002/63**.
- Current application for compliance testing; for rejection of a lot the **measured residue (C_R)** must be **≥ decision limit**, calculated using only the **laboratory phase uncertainty (CV_L)**.

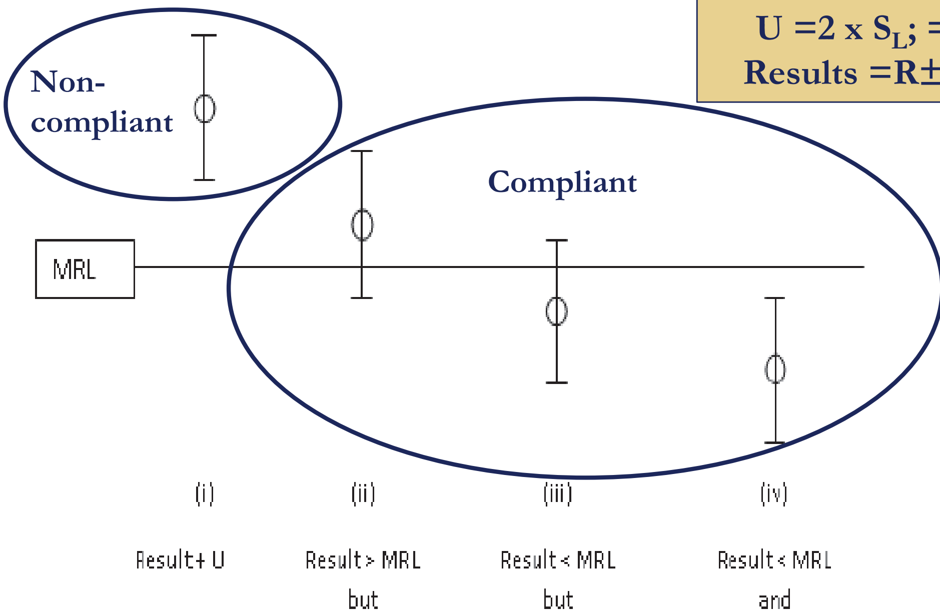
$$C_R - (2 \times CV_L \times C_R) \leq MRL$$

- **CV_L** should be **no more than 25%**, as per **SANTE (2021) Guideline**.
- Laboratories must use their own **inter-laboratory reproducibility (CV_L)**, not exceeding 25%, when applying the decision limit for rejection (**R > 2×MRL** compliance criterion)

Commodotiy on the Market

$$CV_{Lc} = \sqrt{CV_{SS}^2 + CV_{Sp}^2 + CV_A^2}$$

U =2 x S_L; =2 xCV_LxR
Results =R±U (mg/kg)



R > 2*MRL criterion.

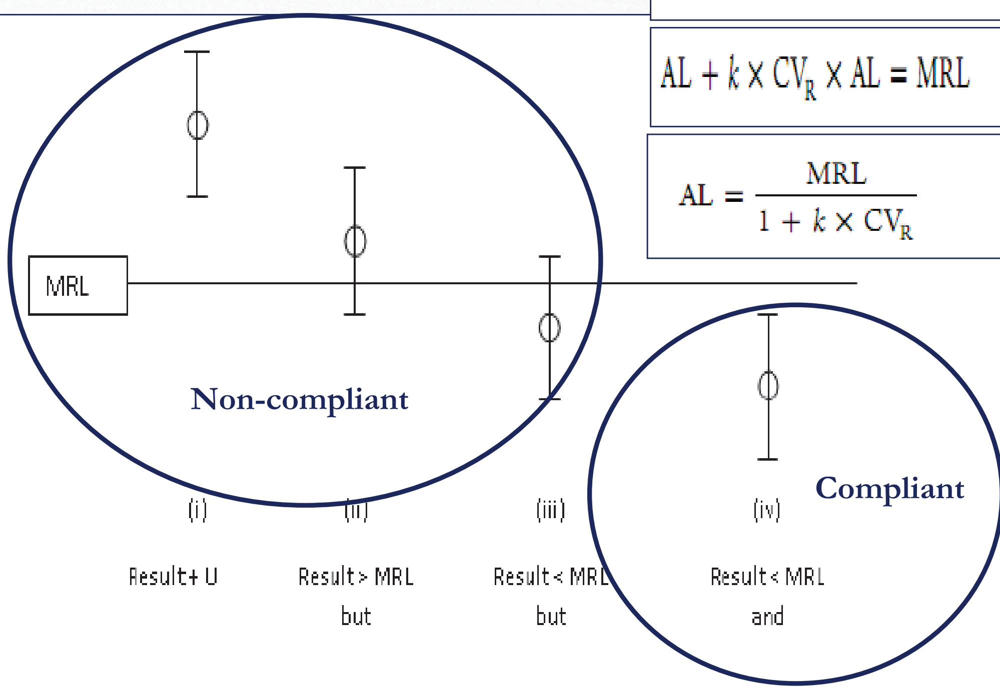
Desicion Limit=DL= 2MRL (for pesticide residue)

Pre-marketed control

$$CV_R = \sqrt{(CV_S)^2 + (CV_L)^2}$$

$$AL + k \times CV_R \times AL = MRL$$

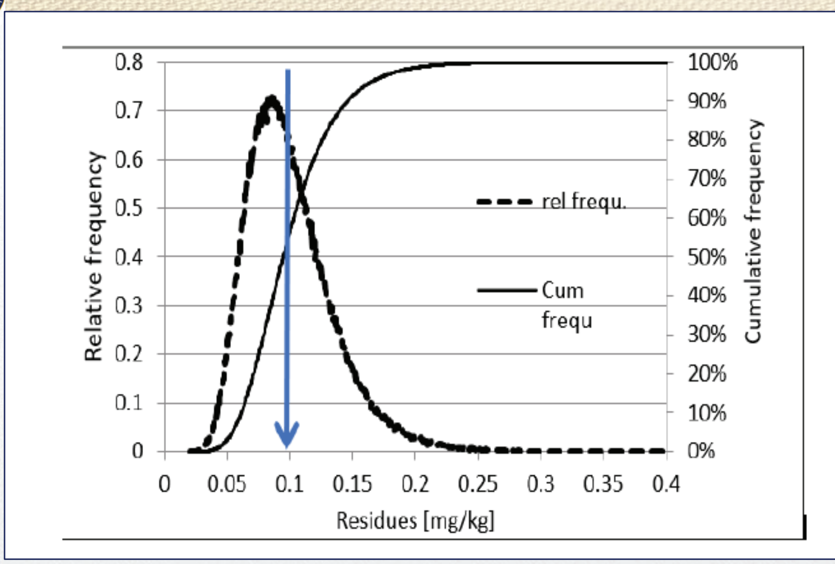
$$AL = \frac{MRL}{1 + k \times CV_R}$$



Whitker model for AL for mycotoxin:http://www.fstools.org/mycotoxins/.

Pre-marketed control

- Compliance with MRLs should be verified with high certainty before placing commodities on the market or exporting them
- **Combined uncertainty**, including **sampling uncertainty**, must be considered in evaluations.
- **Sampling uncertainty decreases** as the **number of primary samples** increases
- **Composite samples with up to 25 primary samples** are recommended, if effective homogenization in the lab is possible
- The **combined uncertainty (CV_R),** including sampling (CV_S), is used to calculate an **action limit (AL)**
- **The AL must not be exceeded to** certify MRL compliance during **premarketing self-control programs**



- Comparing residue in composite samples directly to the MRL can lead to a 44% error rate—too high for safe export decisions.
- Instead, use the **combined uncertainty (CV_R)** to assess compliance.

Distribution of residues in apple composite samples taken from a lot with mean residue of 0.11 mg/kg and CVR of 0.35. The MRL of 0.1 mg/kg is indicated by the blue arrow (Ambrus et al.2023)

- Introduce an **Action Limit (AL)**, set **below the MRL**, to improve reliability.
- A default **AL of 0.3–0.4 × MRL** is generally safe and avoids complex calculations.
- The AL concept applies to **pesticides, mycotoxins, and gluten**.
- For pre-export checks:
  - ✓Take **two replicate samples**.
  - ✓If the first exceeds AL, test the second.
  - ✓**Both must be below 0.5 × MRL** to ensure ~95% acceptance by the importing country

Conclusion

- The maximum allowable limits for **residue and contaminants** in food would at low levels (µg/kg or µg/l)
- **Careful sampling** is crucial, especially from lots weighing over 1 tonne
- Poor sampling can render all **analytical work ineffective** and waste resources
- **Sampling is a critical step** when checking legal compliance of a commodity
- **Sampling uncertainty is greater** than uncertainties from sample preparation and analysis
- EURACHEM Guidlines can be used for validation and uncertainty estimation of sampling step
- **Optimum Uncertainty** method can be used for specifying target uncertainty
- **Sampling uncertainty must be considered** in pre-market checks
- **Action Limits (ALs)** can be determined based on:
  - ✓Commodity type
  - ✓Sample size
  - ✓Acceptable violation rate.

References

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