



Sampling commodity goods and interpretation of the compliance with maximum limits from the producer/exporter and the competent authority/importer sides

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Objectives

- Interpret the maximum limits from the perspectives of producers and official control.
- Use of combined uncertainty for establishing action and decision limits by the producers and buyers, respectively.
- Illustrate the principles with practical examples.



Basic definitions

- **Performance criterion (PC):**

The frequency and/or concentration of a hazard in a food that must be limited by the application of one or more control measures to provide or contribute to a performance objective.

- **Performance objective (PO):**

The maximum frequency and/or concentration of a hazard in a food at a specified step in the food chain that provides, or contributes to, achievement of the Food Safety Objective (FSO) or Appropriate Level of Protection (ALOP).

- As verifying 100% compliance of the products is practically impossible, the BASELINE project recommended 98% compliance as performance objective, which should be demonstrated at least with 95% probability.
CXG-50: Acceptable quality level $AQL=100-98=2\%$

AQL for a given sampling plan is the rate of non-conforming items at which a lot will be rejected with a low probability, usually 5 %

If the product complies with the legal limits, FSO is achieved.

Basic definitions – legal limits

- **Maximum level (ML)**

For contaminants, naturally occurring toxicants and nutrients, the maximum concentration of a substance recommended by the Codex Alimentarius Commission to be legally permitted in a given commodity.

For food additives, the concentration of permitted maximum use in the given food specified by food standards.

- **Maximum residue limit (MRL) for pesticide residues**

The maximum concentration of a pesticide residue (expressed as milligrams per kilogram) to be legally permitted in or on food commodities and animal feed. MRLs for meat and poultry apply to a bulk sample derived from a single primary sample, whereas MRLs for plant products, eggs and dairy products apply to the average residue in a specified portion of the composite bulk sample derived from 1-10 primary samples.

Control of the commodities

There are two distinctly different situations which needs different sampling plans:

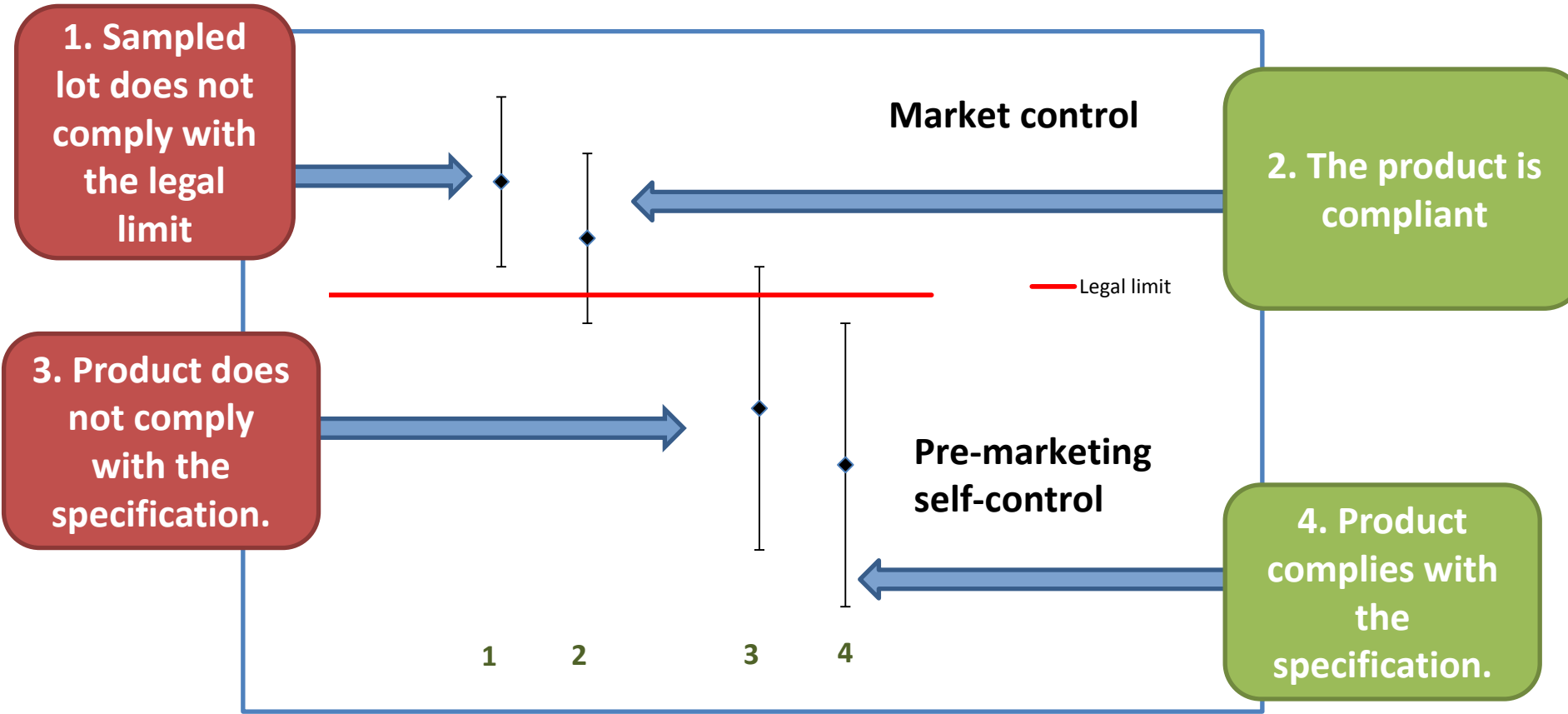
Premarketing self-control

- it has to be certified that at least a specified proportion of the product in terms of the minimum size and mass of bulk/laboratory sample complies with the legal limit
- the combined uncertainty including sampling uncertainty (CV_R) shall be taken into account

Control of commodities on the market

- a lot is considered non-compliant if the measured analyte concentration corrected for recovery, where specified, minus the expanded uncertainty of the results are above the legal limit.
- the combined uncertainty of the measured concentration (CV_L) shall only be taken into account (excluding the sampling uncertainty)

Illustration of the consideration of combined uncertainty of the measurement result



Distributions of contaminants in food

If the measured value is compared to the legal limit the chance of wrongly declaring a lot to be compliant depends on the distribution of the measurand in the tested food.

- if the tested commodity is homogenous in term of the contaminant (aflatoxin M1 in milk), then the uncertainty of the analytical measurement (e.g. 15% for ELISA-based detection of aflatoxin M1) need only to be considered ;
- the pesticide residues in fruits and vegetables, and ochratoxin in pistachio are distributed approximately following lognormal distribution; in case of pesticide residues the CV_R of 35-45% shall be taken into account.
- due to the very patchy distribution of aflatoxins in nuts, cereals, etc. their distribution can be best described with negative binominal function; the CV_R around 60-70% can be expected.



Combined uncertainty of results (S_{Res})

$$S_{Res} = \sqrt{S_S^2 + S_L^2}$$

$$CV_{Res} = \sqrt{CV_S^2 + CV_L^2}$$

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

Ring tests, proficiency tests and internal quality control
provide information only for CV_A

What do we know about the contribution of CV_S , CV_{SS} , CV_{Sp} ??

Internal quality control

Regularly re-analyse replicate test portions at different time intervals.

Select replicate results which are within the 95% critical range.

2 replicates:

$$C_{\max} - C_{\min} = CD = 2.8 CV_{Ltyp} \bar{R}$$

3 replicates

$$C_{\max} - C_{\min} = CD = 3.3 CV_{Ltyp} \bar{R}$$

Determination of CV_L

- Calculate their relative standard deviations from the results of replicate test portions :

$$R_{\Delta i} = 2(R_{i1} - R_{i2}) / (R_{i1} + R_{i2})$$

$$CV_{Lab} = \sqrt{\frac{\sum_{i=1}^n R_{\Delta i}^2}{2n}}$$

$$CV_L = \left(\frac{\sum R_{\Delta i}}{n} \right) / 1.128$$

$v = n$
(# : number of test portions)

For 2 replicates

Effect of particle size

Gy's sampling $RSD=CV_{Sp}$

$$CV_{Sp} = Cd^3 F = Cd^3 \left(\frac{1}{M_{TP}} - \frac{1}{M_{AS}} \right)$$

C : shape factor,

d : upper 95% of particle size,

M_{TP} : extracted test portion,

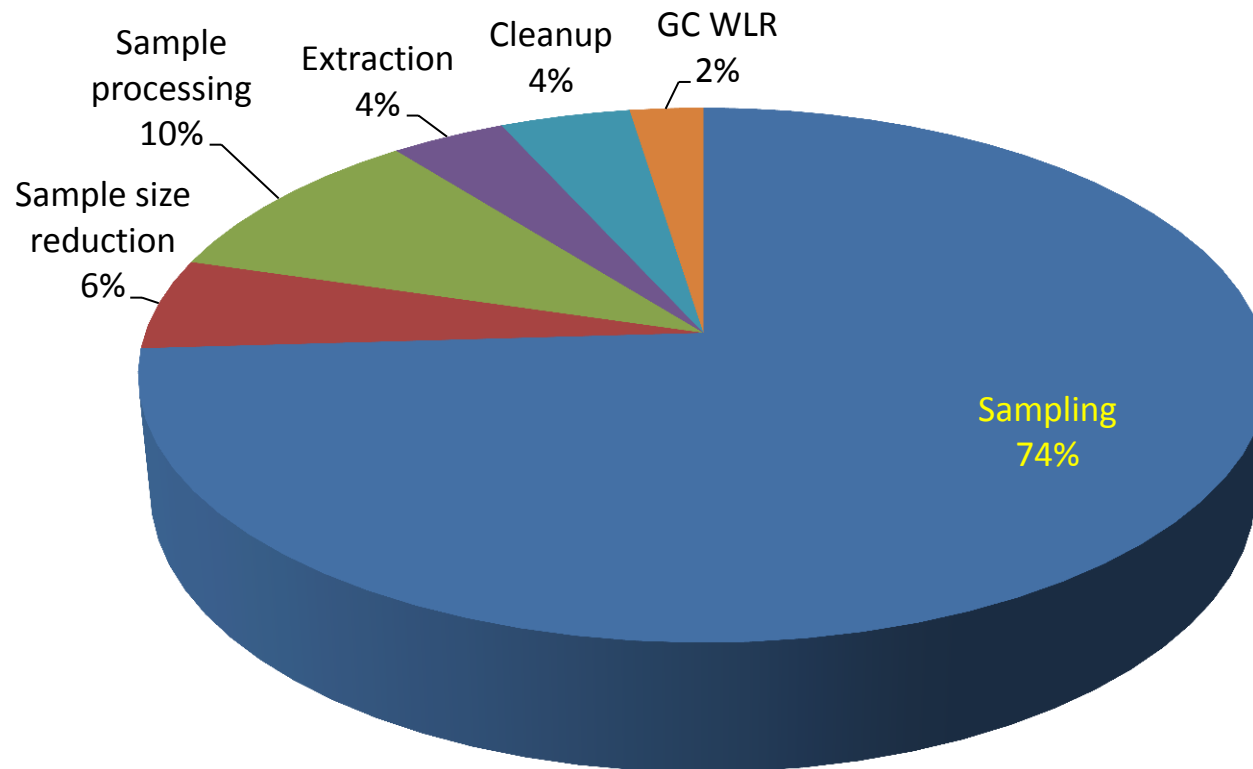
M_{AS} : mass of homogenised portion of sample

$M_{AS}=1000$ g M_{TP} : 25g (**F=0.039**); 10g (**F=0.099**); 5g (**F=0.199**); 2 g (**F=0.499**)

Ingamells' sampling constant:

$$K_s = M_{TP} CV_{Sp}^2$$

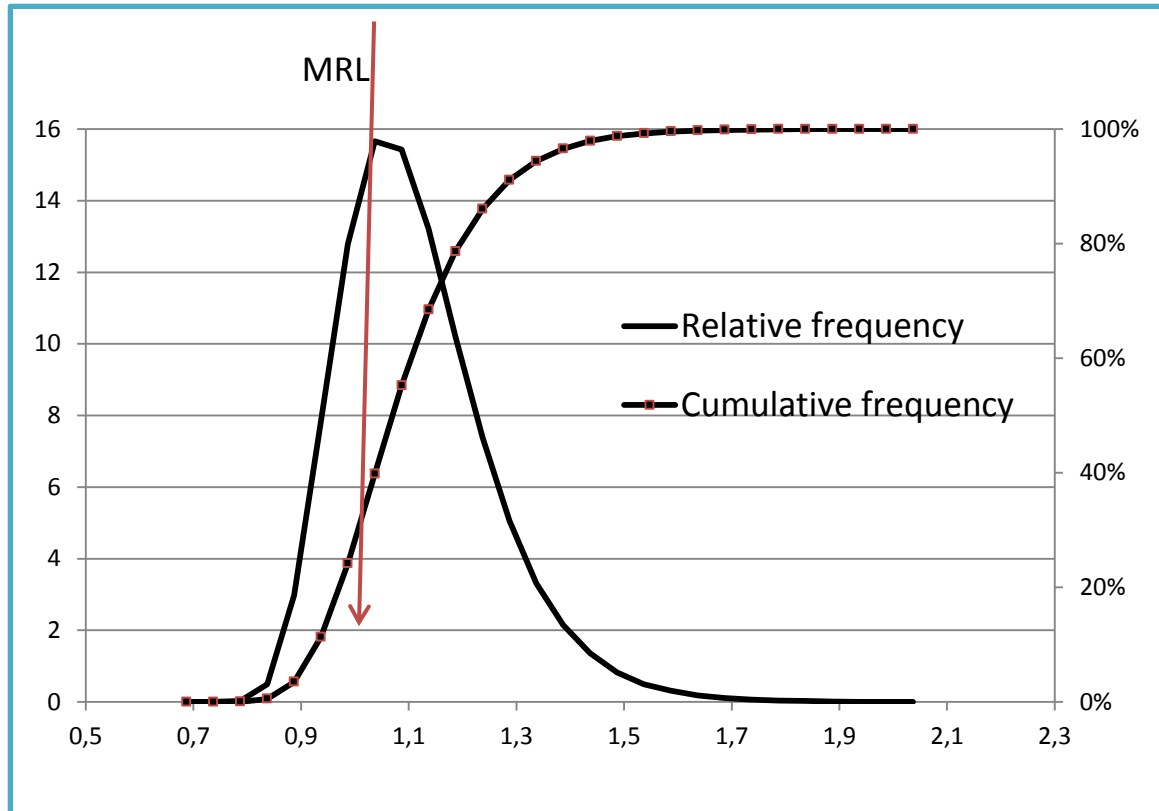
Typical contribution of the steps of pesticide residues determination ($CV_R=0.38$) to the combined uncertainty



The CV_A is only 11%

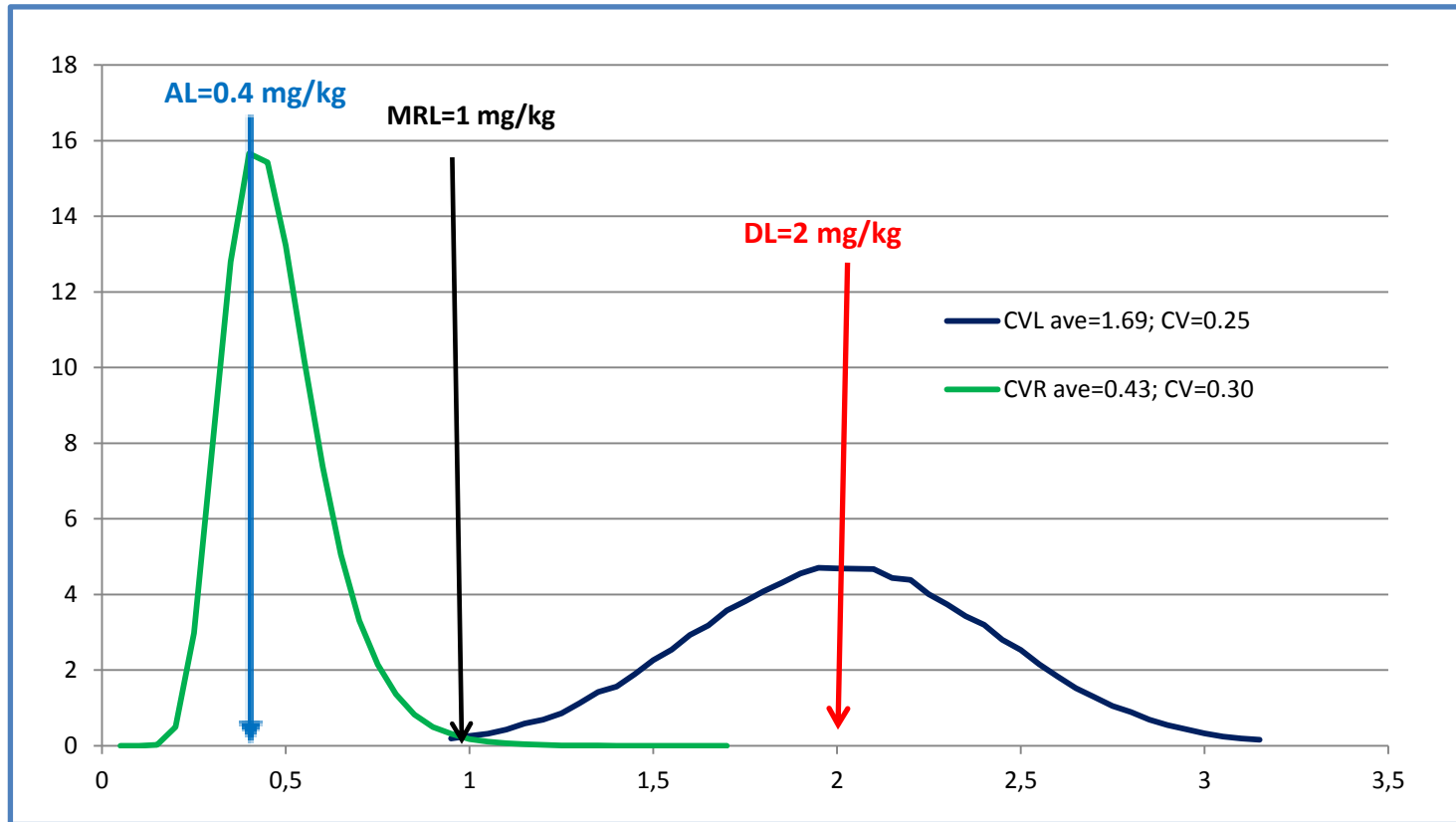
Comparison of measured value the MRL/ML

Distribution of residues in apple composite samples.



If we compare contaminants/residues in composite samples to the ML/MRL we would make wrong decision in over 50-70% of the cases depending on the measurand and sample.

Relationship of the Action limit (AL) for testing and the decision limit (DL) for verifying compliance with an MRL of 1 mg/kg of an apple lot.



Premarket control: Action limit

Post-market control: Decision limit (expanded uncertainty =2x SD, CV = 0.25)

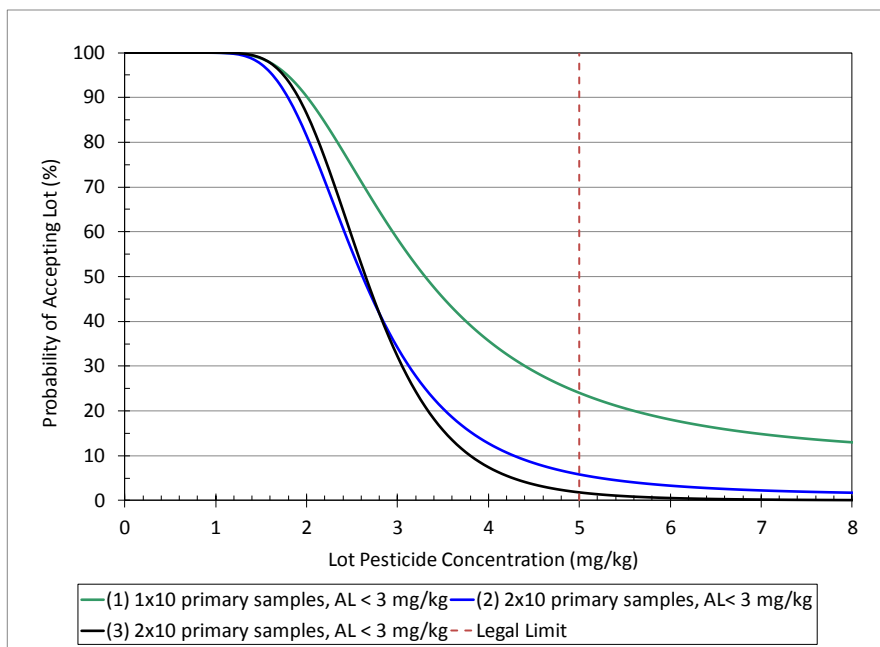
Calculation of Action Limit

The action limits should be calculated taking into account the variability of measurand in composite samples.

Excel templates has been developed to assist producers in selecting appropriate action levels in the pre-market self control.

1. Pesticide residues
2. Mycotoxins

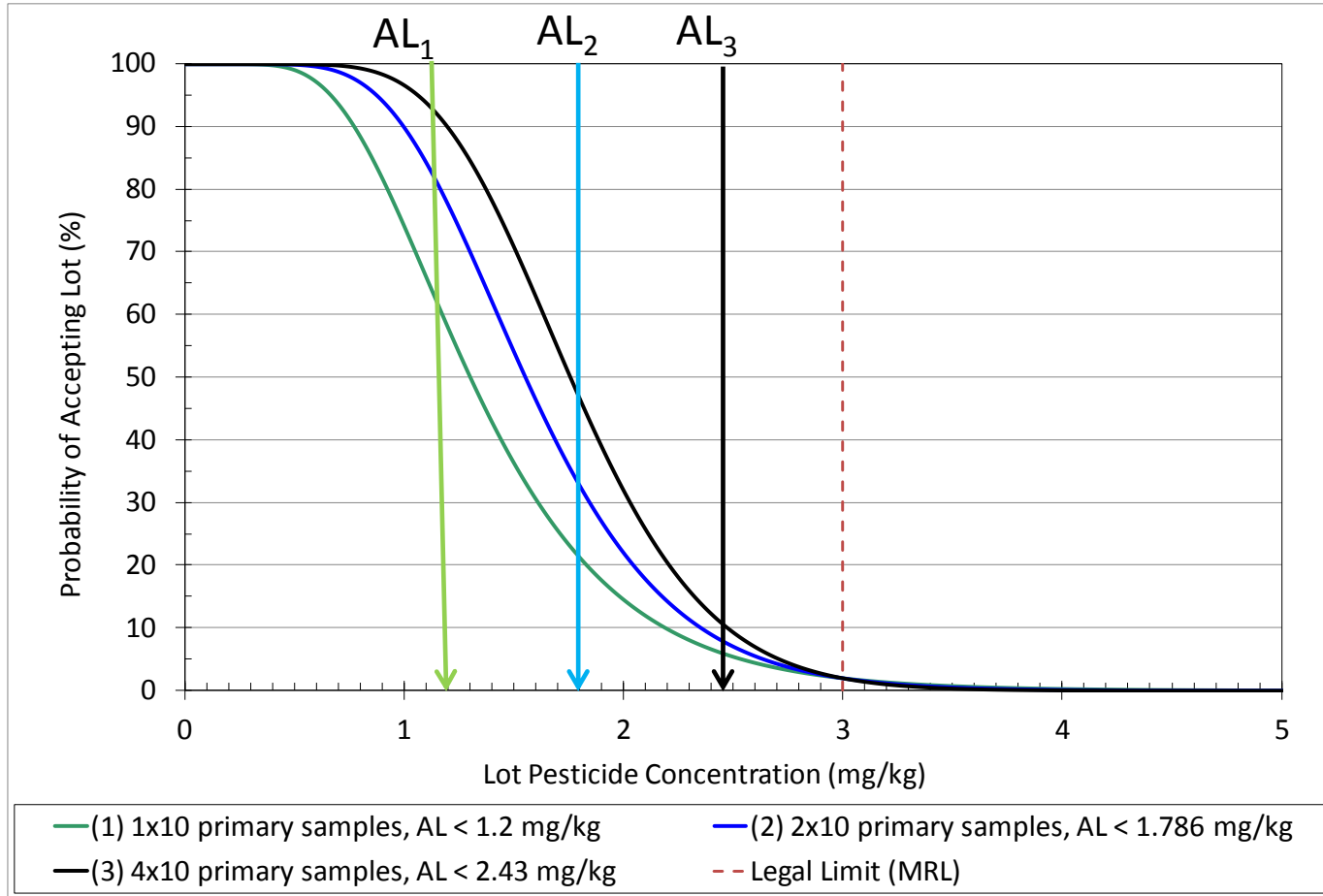
Optimisation of premarketing testing of medium size fruits $CV_S=0.8$, $CV_L=0.16$



	Curve #1	Curve #2	Curve #3
Number of Laboratory Samples	1	2	2
Number of primary samples	10	10	10
CV_L	0.25	0.25	0.25
Number of test portion analysed	1	1	2
Accept Limit <= (mg/kg)	3	3	3

Contributor	Variance ratio %		
	Curve #1	Curve #2	Curve #3
Sampling	50.6	50.6	67.2
Sample prep+anal	49.4	49.4	32.8
	100	100	100

Practical examples



Template for mycotoxins

Mycotoxin, Commodity	Aflatoxin, Corn , Shelled	Aflatoxin, Corn , Shelled	Aflatoxin, Corn , Shelled
Laboratory Sample Size - ns (kg) =	10.00	10.00	10.00
Number Laboratory Samples - scnt (#) =	2	3	4
Test Portion - nss (g) =	50	50	50
Number of aliquots - na =	1	1	1
Accept/Reject Limit (ng/g) =	2	2	2
Regulatory Limit (ng/g) =	5.0		

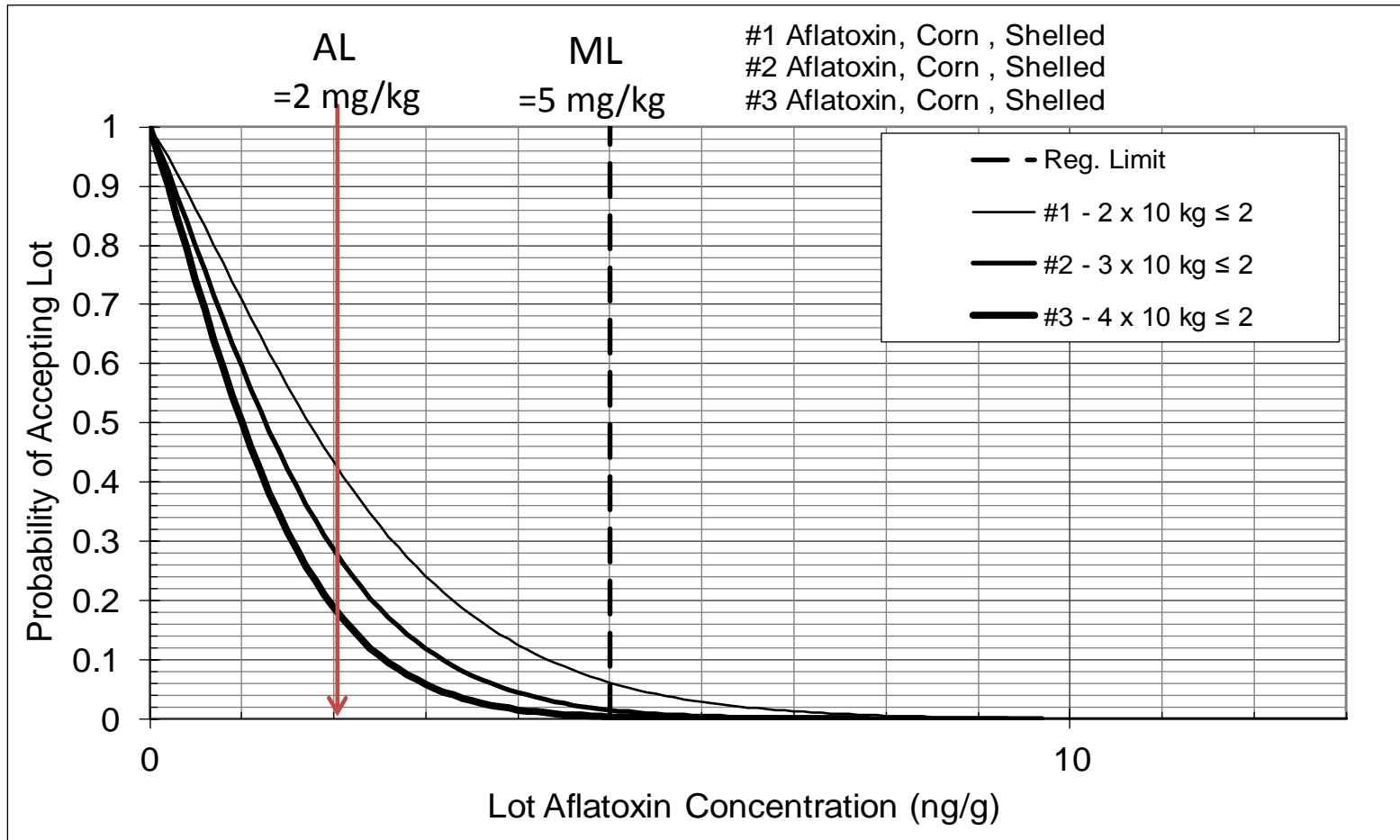
The Mycotoxin Sampling Tool can be accessed at the following website address:

<http://www.fstools.org/mycotoxins/>.

FAO encourages Codex members to use the tool. Feedback on the tool can be sent at food-quality@fao.org

Additional references on related topics can be found on the web at <http://www.bae.ncsu.edu/usda/www/whitaker1.htm>

Practical examples



Examples of estimated action limits for selected pesticide residues

	MRL mg/kg	AL mg/kg
acephate	0.02*	LD≤0.008
azoxystrobin	3	1.2
chlorpyrifos	0.05*	LD≤0.02
cyfluthrin	0.1	0.04
difenoconazole	1	0.4
indoxacarb	0.3	0.12
tetradifon	0.01*	LD≤0.004

Conclusions and recommendations

- The concept of the action limit can be applied for the verification of the compliance of a particular lot, or can be used within an early warning control programme.
- AL depends on CV_R (n , p , ap , CV_L).
- The sample size (number of primary samples, total mass) should correspond to that specified in relevant legislation.
- Producers should define suitable control points when appropriate action levels (Performance Criterion) can be applied.
- The sampling programme should be based on the precise definition of the sampling frame, weighting the potential risk associated with the production of a given product and the random sampling of the products all over the production cycle.
- Under such conditions the analytical results can be used to verify that the production is under control.



Close collaboration of all stakeholders is required for limiting rejection of lots and disputes in food trading

Thank you for your attention.