National Food Chain Safety Office
Pesticide Analytical National Reference Laboratory

NFCSO, Pesticide Analytical National Reference Laboratory, Velence

Conformity or nonconformity, that is the question

L. Benke, CIPAC-Symposium 2019, Braunschweig

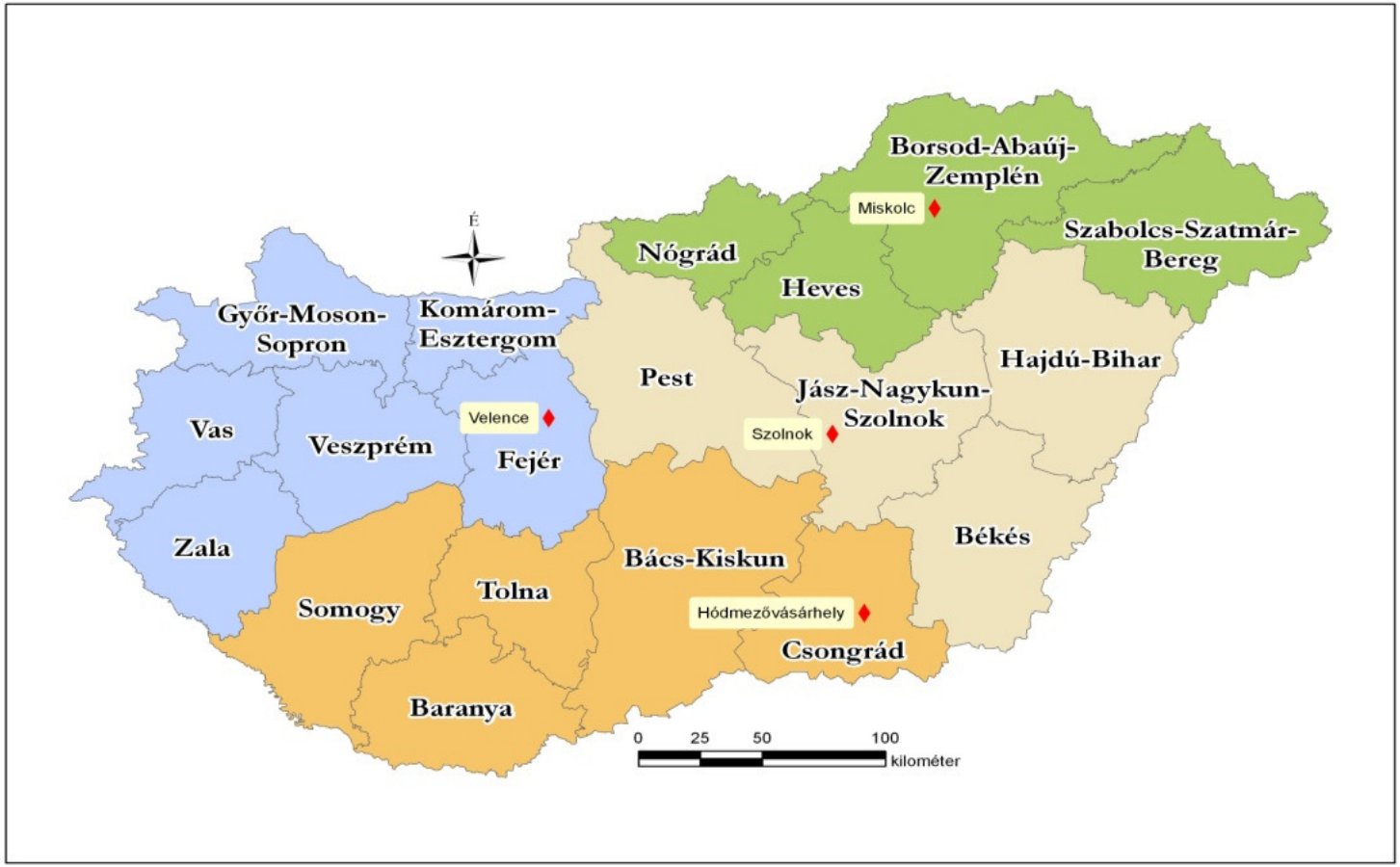
Overview

- Overview of the Hungarian organization
- Official control of PPP-s on the market
- Uncertainty of measurements
- Tolerance limits
- Decision rules for conformity and nonconformity
- Conclusions

Hungarian laboratories

4 regional laboratories,
1 laboratory with two fields of activity: pesticide residues quality control of PPP

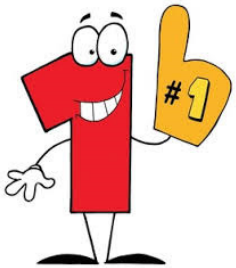
Pesticide analytical laboratories



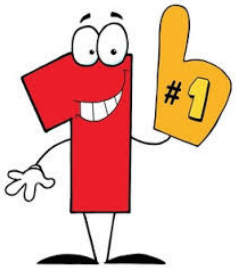
EU-NRL activities, pesticide residues

National Reference Laboratory

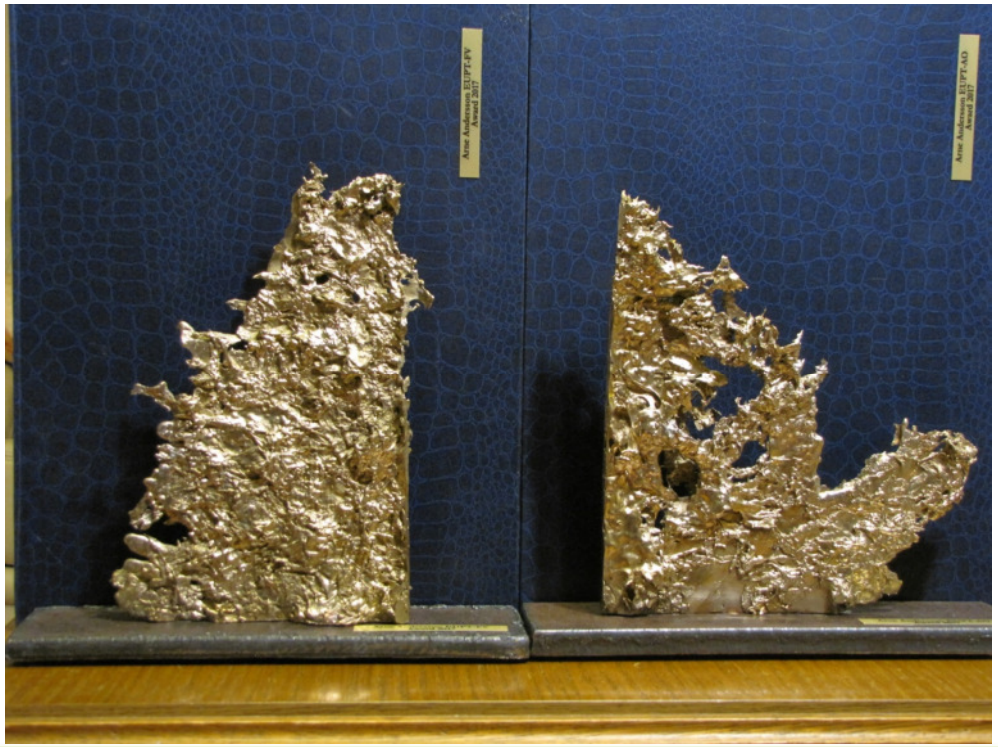
- Reference Laboratories (RL) were established in accordance with REGULATION (EC) No 882/2004 OF THE EUROPEAN PARLIAMENT AND OF THE COUNCIL (29 April 2004) on official controls performed to ensure the verification of compliance with feed and food law, animal health and animal welfare rules
- Since 2006 our laboratory has carried out the tasks of EU-NRL in the field of analysis of pesticide residues in cereals and feeding stuff (EU-NRL-CF)
- Since 2012 in the field of samples of animal origin and high fat content (EU-NRL-AO)
- Since 2016 in the field of fruits and vegetables (EU-NRL-FV) and in the field of single residue methods (EU-NRL-SRM)



- **In 2013 in feed matrix we obtained the 1st place among 132 laboratories, in sample matrices of animal origin the second place, in fruit and vegetables category the 10th place.**
- **In 2015 in fruit and vegetables matrices we finished on the 1st place. 165 laboratories participated in the test. In sample matrices of animal origin we obtained the 4th place.**
- **In 2016 in fruit and vegetables matrices we finished on the 4th place. In the sample matrix of animal origin based on our result we were on the 4th place.**
- **In 2018 in fruit and vegetables matrices we finished on the 3rd place, in animal origin samples we obtained the first place.**



- **In 2017 we won two Arne Andersson awards, one in the field of fruits and vegetables and the other in the field of foodstuffs of animal origin**



Official control of PPP

- **The quality of pesticides is controlled based on a yearly control plan. The sampling is carried out by the plant protection officers, at traders and farmer shops.**

  Through this monitoring program we check, whether the commercially available PPP-s fulfil the quality requirements stated in the registration documents in terms of :

- **active ingredient content**
- **physical and chemical properties**
- **packaging and labelling**

Problems during official control of PPP

- **Active ingredient content**

  Are the obtained values inside the given tolerance interval, is the product compliant or non compliant?

- **Physical and chemical properties**

  Are the obtained values inside the given tolerance interval, is the product compliant or non compliant?

The estimated measurement uncertainty has to be taken into account when verifying conformity or nonconformity with specification.

Problems during official control of PPP

Definition of uncertainty

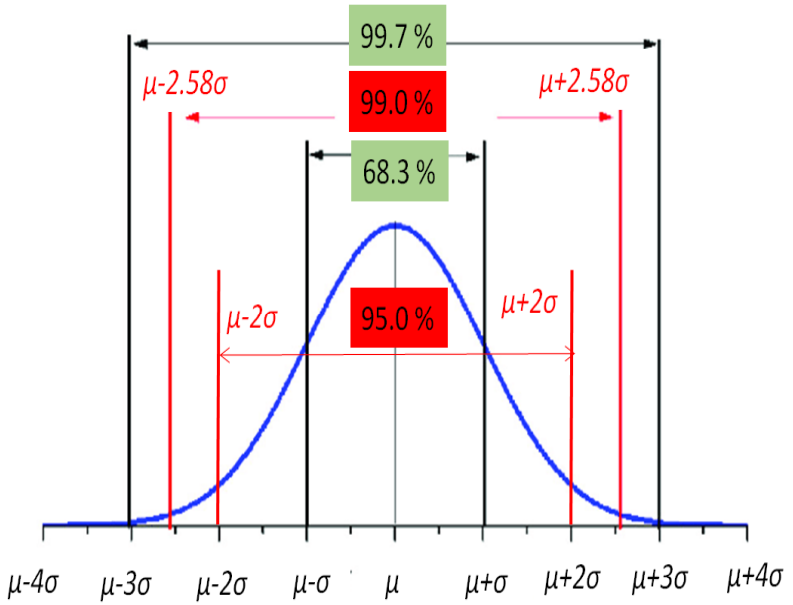
A parameter associated with the result of a measurement, that characterises the dispersion of the values that could reasonably be attributed to the measurand.

Measurement uncertainty is a property of the result of measurement which characterises the spread of the values and tells us how reliable the measurement result is. It is not part of the product design, so the uncertainty of the analytical measurement for checking the quality of the product is not linked to the tolerance interval of the product.

Results should be stated together with the expanded uncertainty U, calculated from the combined standard uncertainty using a coverage factor k=2. Result: x ± U

Problems during official control of PPP

Normal distribution



Problems during official control of PPP

Tolerance definition:
A tolerance is a value by which the designer may allow a manufactured product to deviate from the ideal value whilst still conforming to a specification.
Tolerance is a property of design of a product, the tolerance limit for the active substances in case of PPP refers to the acceptable range of variation of concentration from the nominal value (designed value). Tolerance is a function of the technological process, the quality of the raw materials and other manufacturing parameters.

Problems during official control of PPP

If we have a concentration that is exactly on the limit of tolerance, we would expect to have 50% probability to have the product within the tolerance and 50% probability to have it out of tolerance.

If the true value moves inside the tolerance interval the probability of measuring it to be outside the tolerance gets smaller and if the value moves outside the tolerance the probability of finding it to be inside the tolerance gets smaller.

When we are close to the tolerance limits there is a chance that we wrongly classify the product.

The golden rule of metrology says that the measurement uncertainty shall be less than 10% of the tolerance.

Unfortunately this is not the case in the field of PPP.

Problems during official control of PPP

ISO 14253-1:2017 Part 1 „ Decision rules for verifying conformity or nonconformity with specifications” gives indication for determining the compliance or non compliance with a specification, taking into account the uncertainty of measurement.

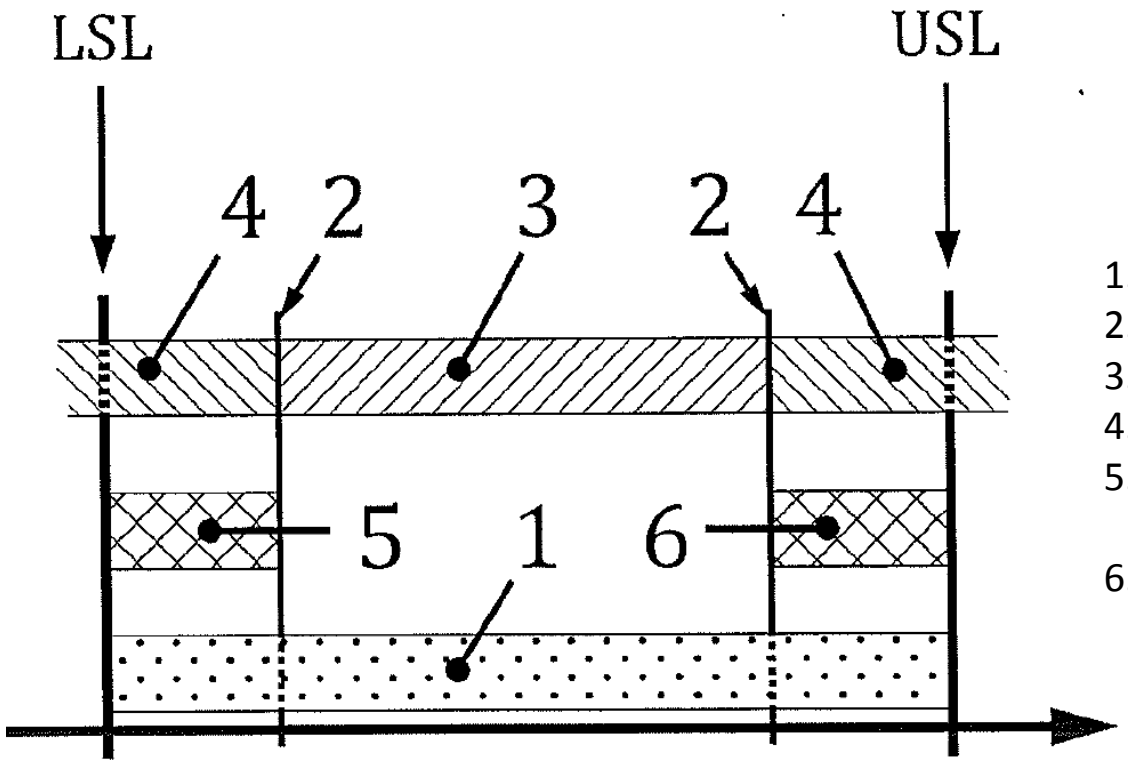
ISO 14253-1:2017 states that in order to prove that a product is conforming to a tolerance, the manufacturer has to measure it to be within that tolerance by more than his measuring uncertainty.

On the other hand for the user to prove that a product is not conforming to a tolerance he has to measure it to be outside that tolerance by more than his measuring uncertainty.

Problems during official control of PPP

How the tolerance limits and decision rules interact in the real measurements?
ISO14253-1 contains two approaches:
when measurements are made to prove conformance of a product (tolerances should be reduced by the measurement uncertainty)
when measurements are made to prove non-conformance of a product (tolerances should be increased by the measurement uncertainty)

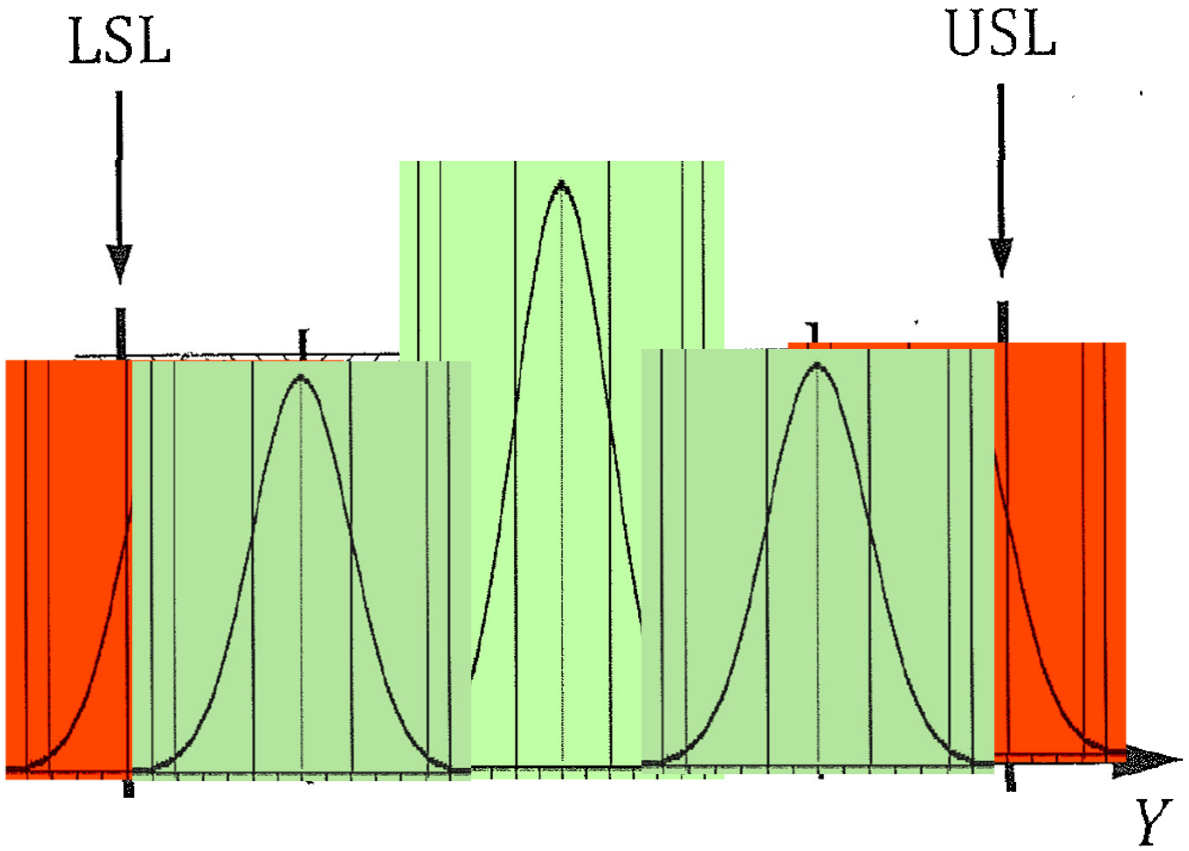
Problems during official control of PPP



1. specification zone
2. acceptance limits
3. acceptance zone
4. rejection zone
5. guard band at lower specification limit (uncertainty)
6. guard band at upper specification limit (uncertainty)

Acceptance zone and rejection zone when verifying conformity

Problems during official control of PPP



Acceptance zone and rejection zone when verifying conformity

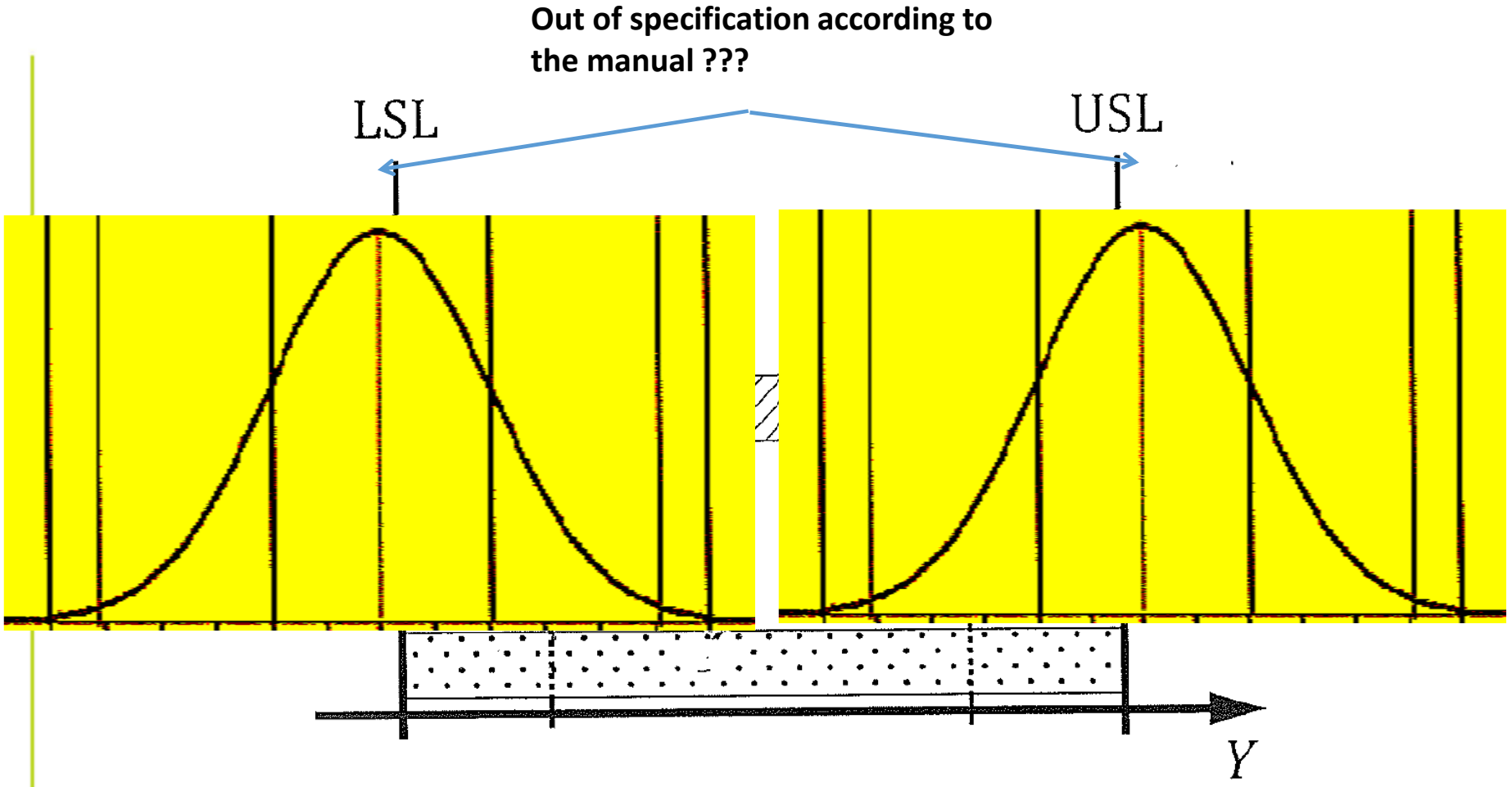
FAO/WHO manual statement

Regarding permissible deviations from the declared content of the active substance the values given in the manual on development and use of FAO and WHO specifications for pesticides should be used:

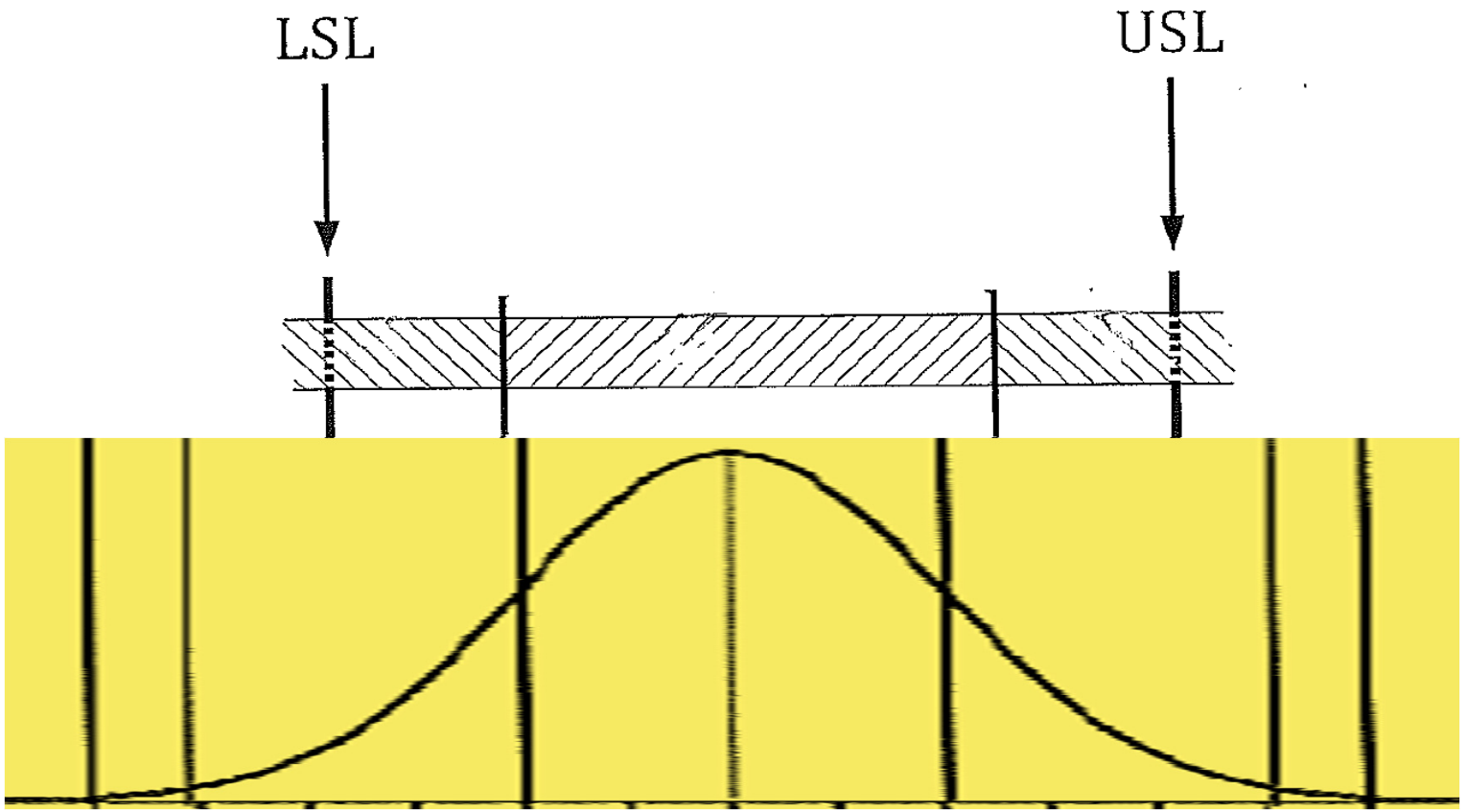
Declared content in g/kg or g/l at 20 ± 2 °C	Tolerance
up to 25	± 15% of the declared content for "homogeneous" formulations (EC, SC, SL, etc.), **or** ± 25% for "heterogeneous" formulations (GR, WG, etc.)
above 25 up to 100	± 10% of the declared content
above 100 up to 250	± 6% of the declared content
above 250 up to 500	± 5% of the declared content
above 500	± 25 g/kg or g/l
Note. In each range the upper limit is included.	

The tolerances refer to the average analytical result and take into account manufacturing, sampling and analytical variations . Thus, if a sample yields a value outside of the tolerance limit, it is generally considered noncompliant and measurement uncertainty is not considered separately.

Problems with FAO/WHO manual tolerance limits

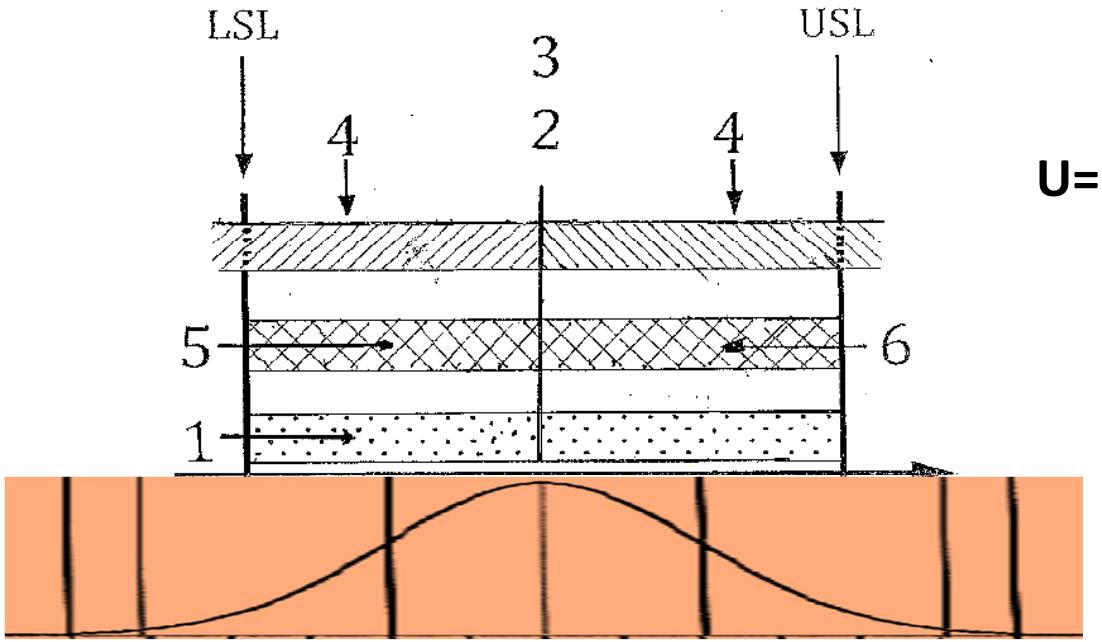


Problems with FAO/WHO manual tolerance limits



Acceptance zone and rejection zone when verifying conformity
At higher concentration levels the FAO/WHO tolerances are too narrow.

Decision example for conformity

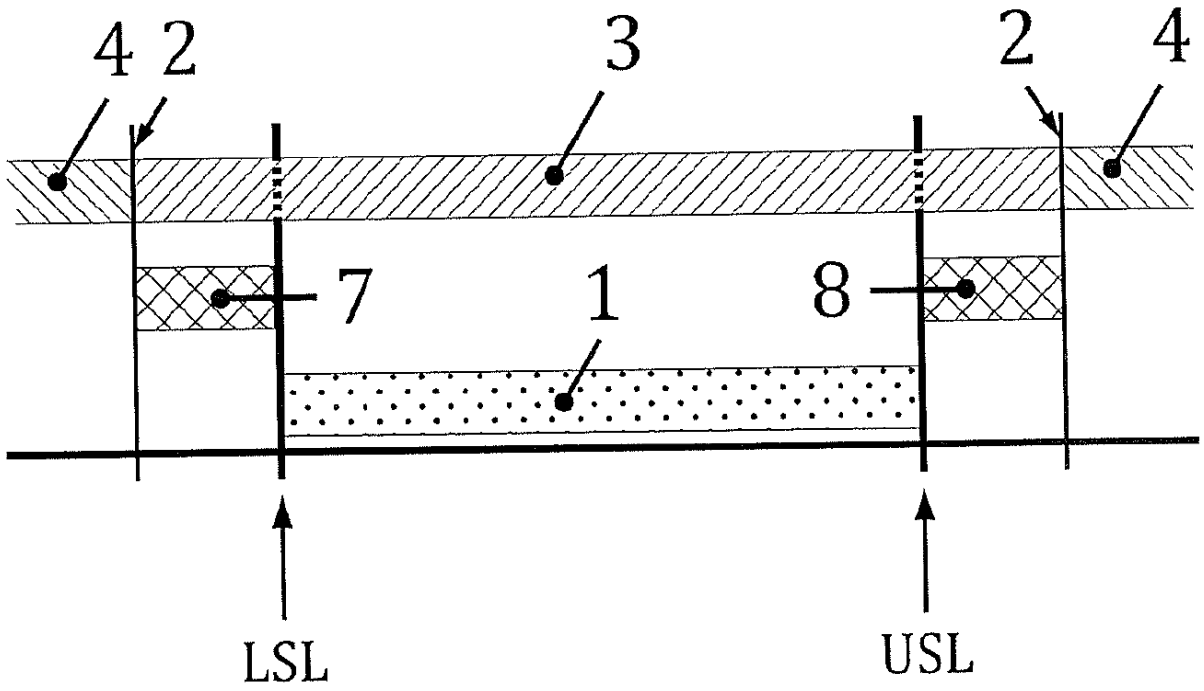


U= 800*0.03125=25g/kg

1- specification zone (tolerance interval, in case of a pesticide with 800g/kg AS +/-25g/kg; lower tolerance limit LSL 775g/kg, upper tolerance limit USL 825g/kg)
2- acceptance limits (LSL+U and USL-U; 775g/kg+25 g/kg and 825g/kg-25g/kg)
3- acceptance zone (acceptance zone=0)
4- rejection zone (non compliance zone lower than 800g/kg and higher than 800g/kg
5- uncertainty of the measurement at the lower tolerance limit
6- uncertainty of the measurement at the upper tolerance limit

Acceptance zone and rejection zone when verifying conformity

Problems during official control of PPP



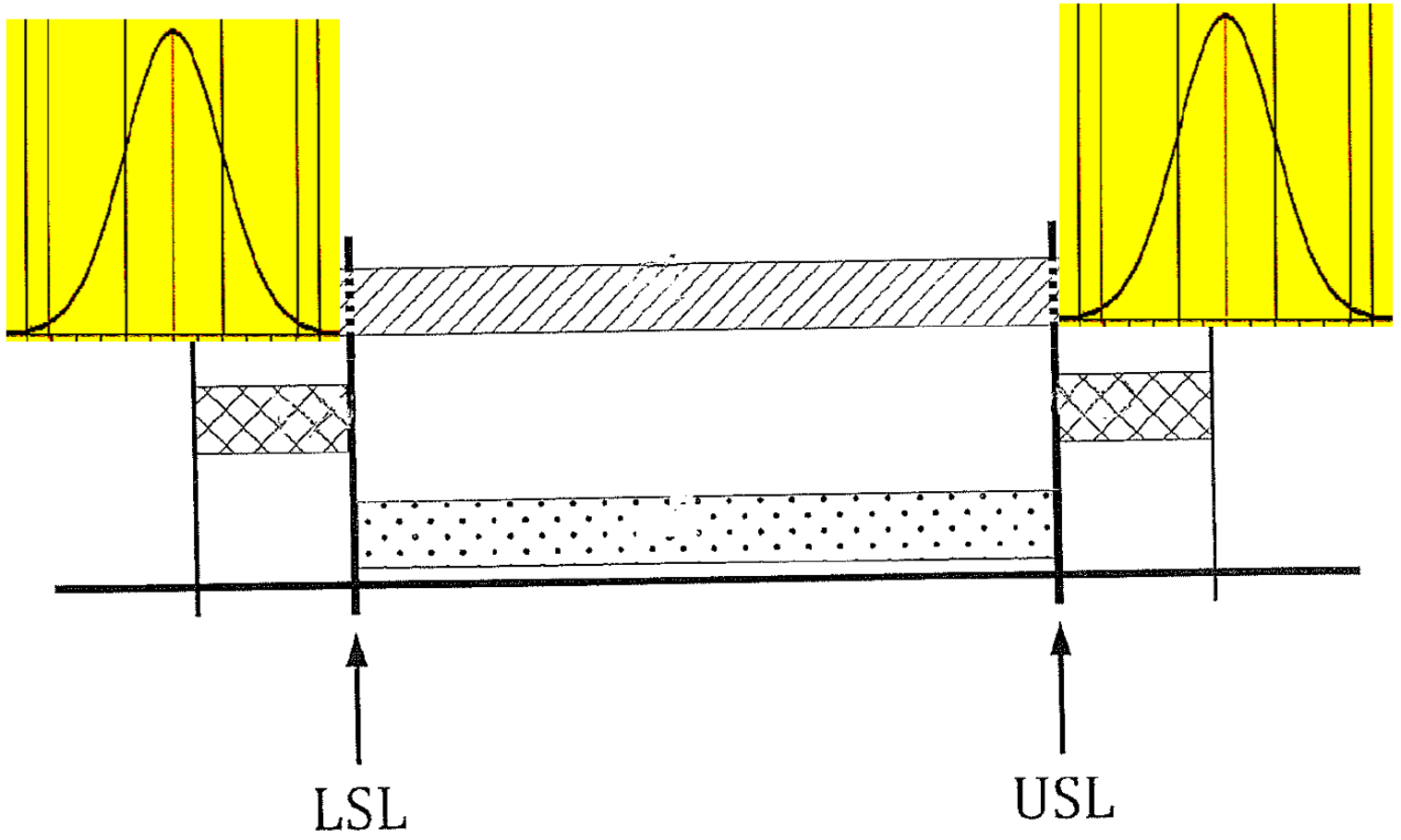
1. specification zone
2. acceptance limits
3. acceptance zone
4. rejection zone
7. guard band at lower specification limit (uncertainty)
8. guard band at upper specification limit (uncertainty)

Rejection zone and acceptance zone when verifying non-conformity

If we have a suspicious sample and we want to prove that the sample is noncompliant we have to apply the nonconformity verifying approach of the ISO standard:

Problems with FAO/WHO manual tolerance limits

To prove that a product is non-conforming the measured value should be outside the tolerance by more than the measurement uncertainty!

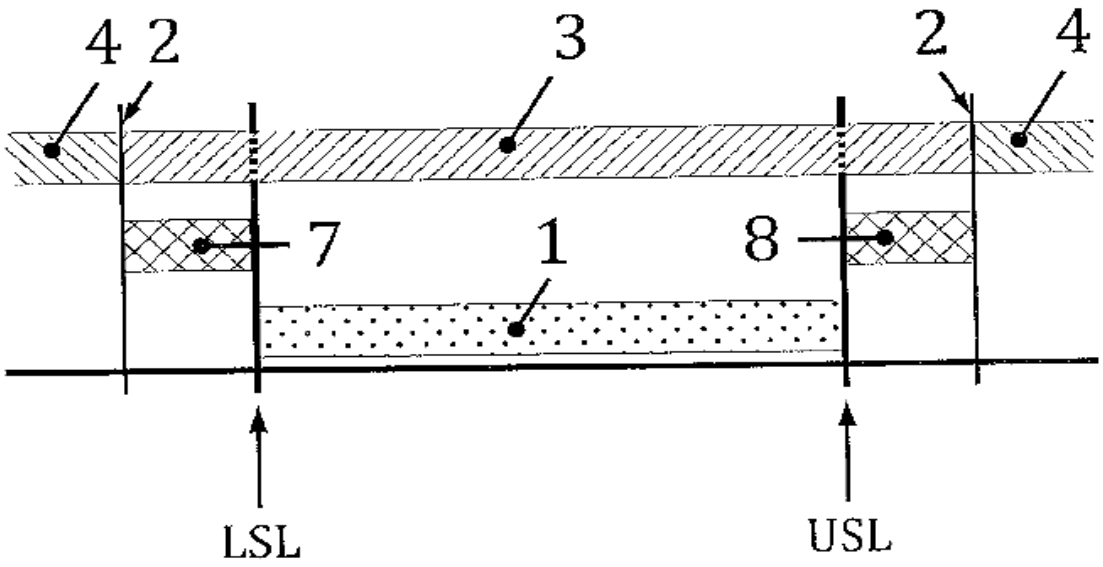


Problems with FAO/WHO manual tolerance limits

The same approach is applied in the field of pesticide residues for interpretation of results for enforcement purposes [SANTE 11813/2017, „Guidance document on analytical quality control and method validation procedures for pesticide residues and analysis in food and feed" page 16; § E 12 .

For official food control by regulatory authorities, compliance with the MRL must be checked by assuming that the MRL is exceeded if the measured value exceeds the MRL by more than the expanded uncertainty (x – U > MRL). The sample is considered noncompliant if x – U > MRL.

Decision example for nonconformity



U= 800*0.03125=25g/kg

1- specification zone (tolerance interval, in case of a pesticide with 800g/kg AS +/-25g/kg; lower tolerance limit LSL 775g/kg, upper tolerance limit USL 825g/kg)
2- acceptance limits (LSL-U and USL+ U; 775g/kg-25 g/kg and 825g/kg+25g/kg)
3- acceptance zone (750g/kg to 850g/kg)
4- rejection zone (non compliance zone lower than 750g/kg and higher than 850g/kg
7- uncertainty of the measurement at the lower tolerance limit
8- uncertainty of the measurement at the upper tolerance limit

Conclusion

ISO 14253-1 decision rules clearly state that any measurement uncertainty reduces the tolerance zone if the conformance with the specification has to be proved and tolerance zone is enlarged by the measurement uncertainty if non-conformance with the specification has to be shown.

nébih
termőföldtől
az asztalig

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Thank you for your attention!