Assessment of results against limits -practical example-

Laying down enforcement limits for chemical analysis (Xmax and Xmin)

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Presentation

- Introduction Inspectorate
- Why limits of enforcement
- Approach of laying down limits
- Results of the approach-**examples**

Introduction/Tasks

- Enforcement and promotion of compliance with regulations
- Investigations health hazards situations
- Advising policy making authorities
- Investigations consumers complaints
- Developments and publication of methods

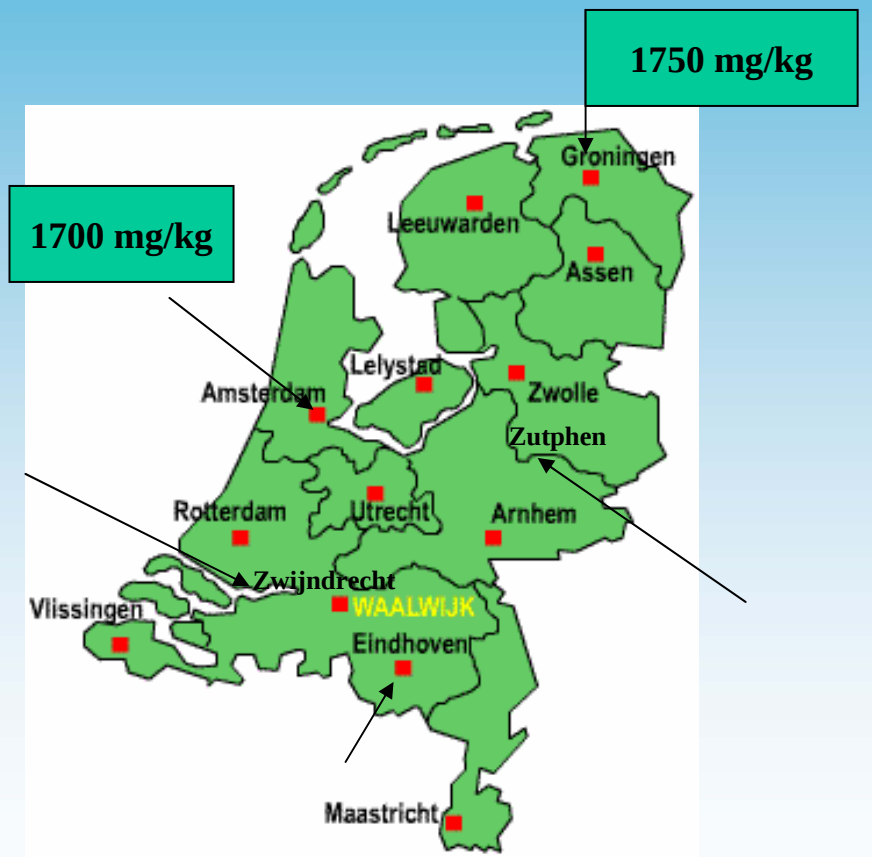
Workfield

- Food and commodities act
- Meat inspection act
- Livestock act
- Experiment on animals act
- Dangerous machinery act
- Pesticide act
- Environmentally hazardous substances act
- Tobacco act

Special tasks

* Primary and horticultural products
* Composite food products
* Veterinary products
* Non-food product safety
* Non-food chemistry

5 Regional Inspectorates
Different enforcement limits



Enforcement limits WHY ?

- Be sure, 5 laboratories of the inspectorate enforce on the same way, with the same quality, with a confidence of 95%

- **Work with the same (concern-)uncertainty**

Uncertainty

- No analytical method gives the exact value or content of an measurand (analyte).
- We make (little) mistakes such as:
  1. Systematically errors
  2. Random errors

Validation

- Recovery
- Specificity
- Reproducibility
- Limit of detection
- Uncertainty

Test always: Robustness

Tools for enforcement limits

- Organise proficiency test
- Use reference materials

Criteria CHEK proficiency studies

- Test accredited methods
- Desires of the regions
- Possibilities

What kind of proficiency tests?

- Food
- Non Food
- Microbiological

Examples of proficiency tests

- Histamin in fish
- Alcohol/methanol in wine
- Aflatoxins in nutproduct
- Glutamic acid in soup
- Parabens in cosmetics
- Hydrochinon in cosmetics
- Salmonella spp in milk

Keuringsdienst van Waren

VWA Voedsel en Waren Autoriteit

CHEK

Certified Secondary Reference material (CSRM)

- example

Specification

Glutamic acid in chips

Product	
Batch	: Proficiency study 292
Matrix	: Bolognese chips
Date of production	: June 2004
Storage life	: June 2005
Store temperature	: - 20° C
Glutamic acid	: 0.639% m/m
Uncertainty	: 0.034% m/m

Description of the sample

10 gram homogeneous well-crushed and minced Bolognese chips in vacuum-sealed sachets.

How to use

The material is intended for analytical purposes. Samples should be stored unopened < -20°C.

Used methods

A detailed technical report of the analytical procedures and treatment of the analytical data is available (Proficiency study 292, oktober 2004).

Analytical measurement and uncertainty HOW?

- R => proficiency studies

  (reproducibility between labs)

- R => Shewhart charts

  (within lab reproducibility)

- Formula of Horwitz

Estimation of R
HOW ?

Proficiency studies

-> S_R= standard deviation estimates for interlaboratory reproducibility

-> 2.8 S_R= interlaboratory reproducibility

-> 2.8 S_R = R

CHEK organised more then 300 proficiency studies

Estimation of R
HOW?

Use data of SHEWHART charts

-> S_l= standard deviation estimates for intralaboratory reproducibility

-> 1.6*2.8* S_l= interlaboratory reproducibility

-> 1.6*2.8* S_l= R

Estimation of R
HOW?

Use the formula of Horwitz

(calculated from > 100 proficiency studies)

$$R = X_{limit} * 2.8 * 2^{(1-0.5 \log c)}/100$$

c: absolute concentration, f.e. 10% is c=0.1)

x_{limit}: legal limit

criterion: use a validated method

Enforcement limit = X min/max

Xmax: the value of a certain parameter, resulting from analyses, above which is a sample regarded as rejectable with a probability of 95% (upper limit)

Xmin: under limit

Xmax and Xmin

$$X_{max/min} = X_{limit} \pm \frac{0.84\ R_l}{2} \quad \text{(duplicates)}$$

$$X_{max/min} = X_{limit} \pm \frac{0.84\ R_l}{V2} \quad \text{(1 analysis)}$$

X_{limit} = legally required maximum/minimum value

literature:

ASTM standard D 3244-74 (2002)

NEN 3206

Example Xmax/min Horwitz

$R = X_{limit} * 2.8 * 2^{(1-0.5 \log c)}/100$

$X_{limit} = 1\ \%$

$C = 10^{-2}$

$R = 1*2.8*2^2/100 = 0.112$

$Xmax = 1 + 0.59* 0.112 = 1.066\%$

Criterion: use a validated method

Examples Xmax/min proficiency studies

component	matrix	Legal limit	R	X max/min (1 analysis)
Acesulfaam-K	Lemonade	400 mg/l	40.5 mg/l	423.9 mg/l
Benzoic acid	Salad	1500 mg/kg	152 mg/kg	1590 mg/kg
Cadmium	Canned tomatoes	0.1 mg/k	0.040 mg/kg	0.124 mg/kg
Alcohol	Beer	4 %(m/m)	0.15 % (m/m)	3.915 % (m/m)

Examples Xmax/min Interlaboratory reproducibility

component	matrix	(Legal) limit	R (R_l * 1.6)	X max/min (1 analysis)
Propanol	Winter-product	20%	4.27%	22.52%
d-limonene	Household-product	1%	0.14%	1.08%
Activ chlor	Desinfectant	30%	0.88	30.52%

Summary

Calculation of the enforcement limits:

- Use R calculated/estimated from:

1. Proficiency studies
2. Shewhart charts (reference materials)
3. Horwitz

Conclusion


OH ALICE... YOU'RE THE ONE FOR ME
BUT BOB... IN A QUANTUM WORLD HOW CAN WE BE SURE?
Physics World


10.00
11.00
13.00
15.00
16.00
18.00
23.00
00.00