



Federal Office of
Consumer Protection
and Food Safety



Concept for evaluating results close to the participants' LOQ in PTs for elements in Food



- **BVL Department 5 is designated as National Reference Laboratory (NRL) for metals and nitrogenous compounds in food and feed**
- **NRLs are part of the European EURL-NRL-network according to regulation (EU) No. 2017/625**
- **NRL responsibilities according to article 101 (amongst others):**
 - Harmonisation and improvement of methods of laboratory analysis
 - Organisation of proficiency test for official control laboratories
- **Purpose of laboratory analysis**
 - Control of maximum levels in foods
 - Generation of contamination data for ongoing risk assessment (monitoring)



COMMISSION REGULATION (EC) No 333/2007

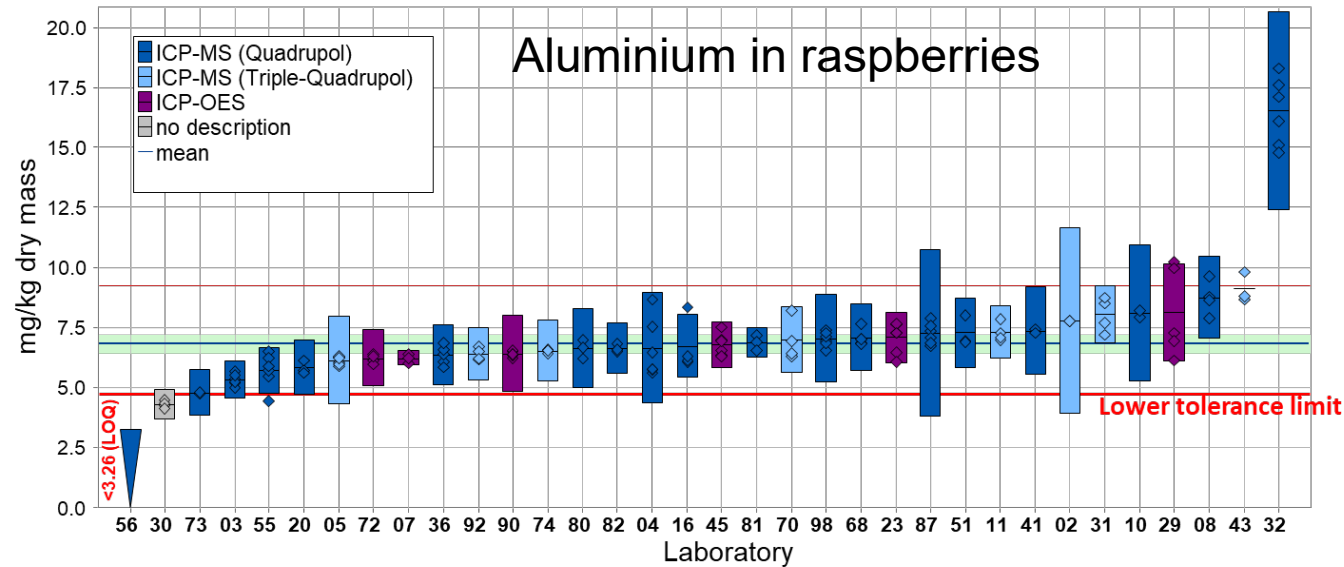
of 28 March 2007

laying down the methods of sampling and analysis for the official control of the levels of lead, cadmium, mercury, inorganic tin, 3-MCPD and benzo(a)pyrene in foodstuffs

- **Performance criteria for analysis of contaminants in food given by regulation (EC) No. 333/2007**
 - Recovery and measurement uncertainty
 - Repeatability
 - Reproducibility
 - Limit of Quantification (LOQ)
 - Free of contamination (free of the metal to be determined)
 - Specificity (free from matrix and spectral interferences)



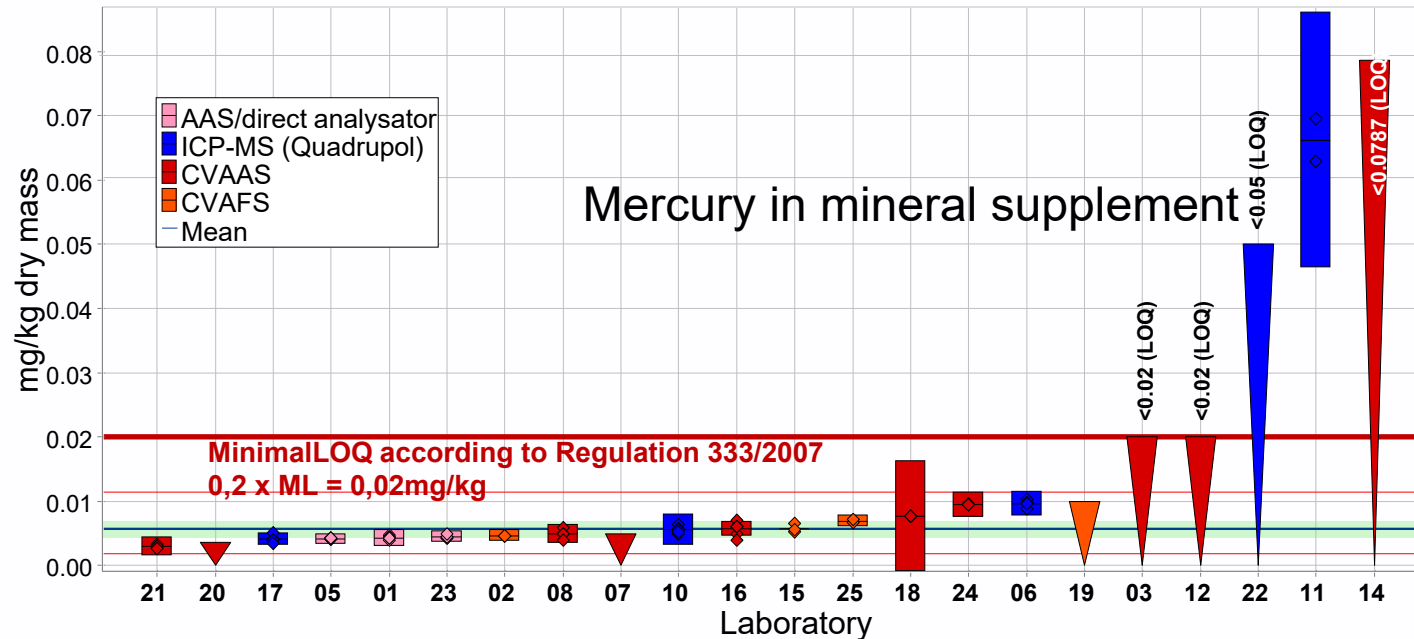
Inappropriate low LOQ (easy case)



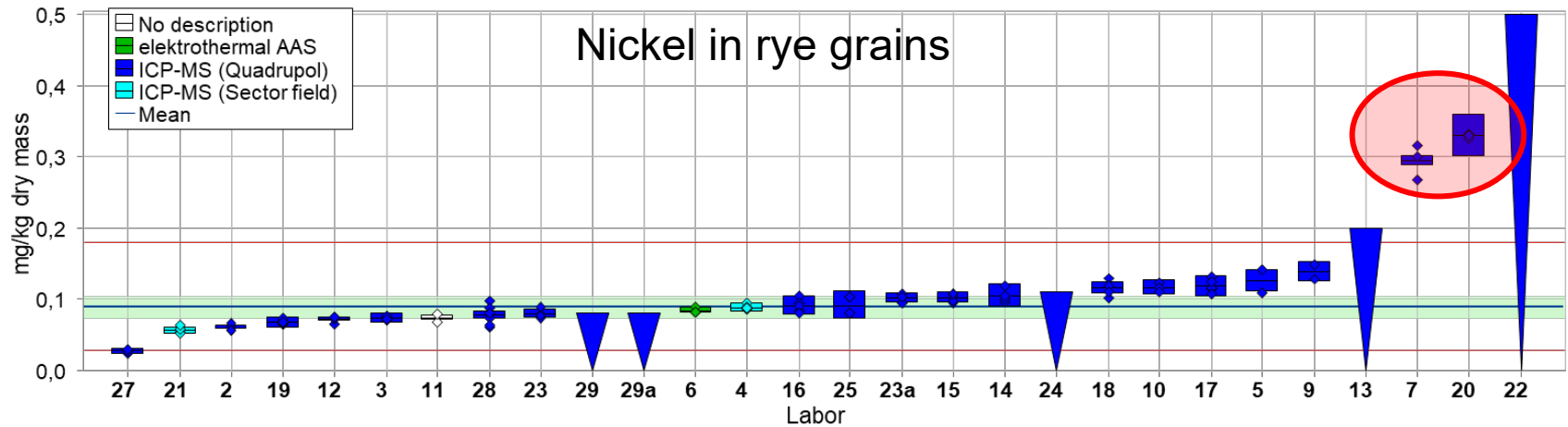
- Regular reportable PT
- a laboratory stating the content being under their LOQ **BUT** this LOQ located under lower tolerance limit
- Slightly different concepts for scoring this case (we set z-score to „-3,0“)
- Risk of false negative results = risk for consumer protection



Inappropriate high LOQ



- Still regular reportable PT (general performance at HorRat 1.8)
- Mercury content lower than legal minimum LOQ
- Two laboratories report higher LOQs than required for analysis according to regulation (EU) 333/2007
- Scoring similar to too low LOQ ??
- ⇒ risk for monitoring programs getting too many left censored data



- **Still regular reportable PT (general performance at HorRat 1.8)**
- ⇒ **LOQ level for future performance criteria**
- **Two participants report results about threefold of the mean**
- ⇒ **resulting from stainless steel parts in the apparatus used in sample preparation (was investigated in follow up actions)**

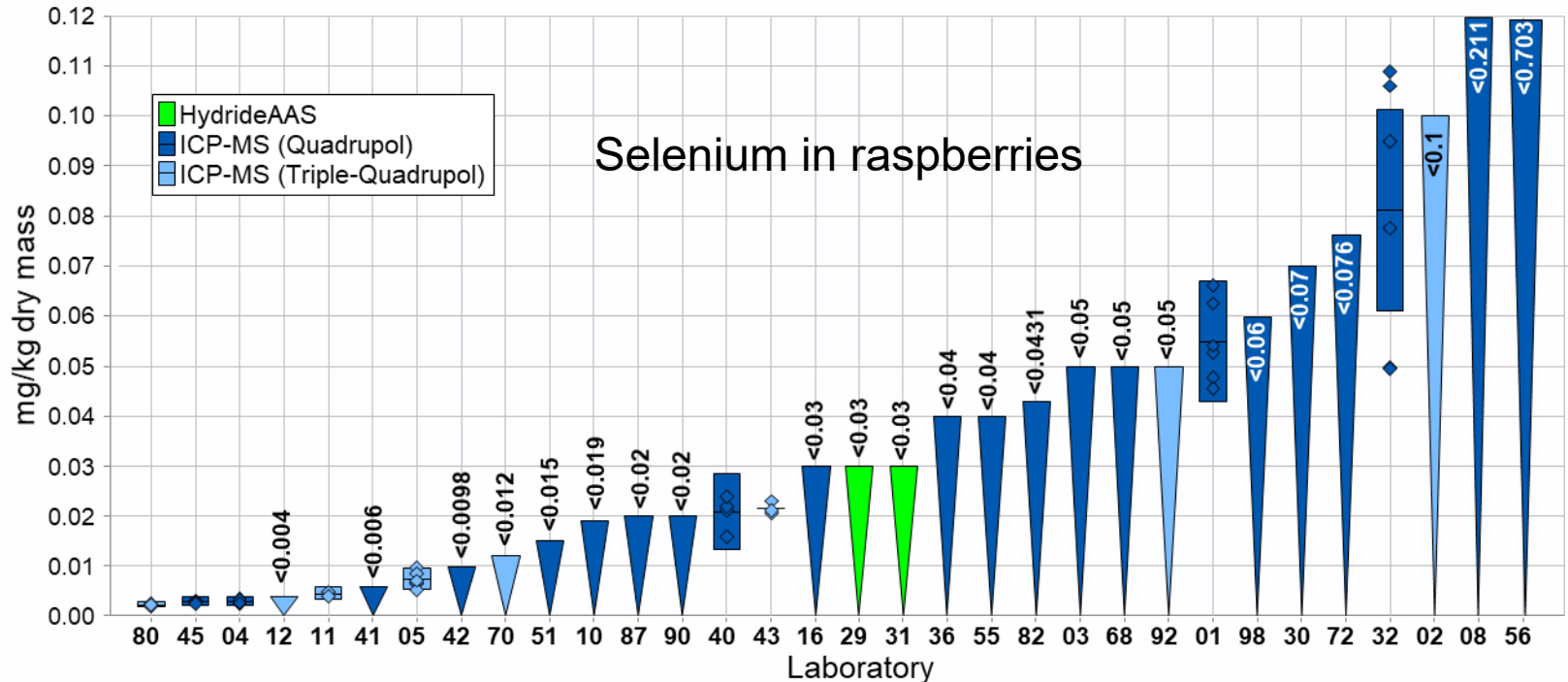


PTs testing the participants' LOQ

- **Since 2017 we do not spike every analyte on the target list**
- **Therefore, a few contaminants could be found in their natural, usually very low content**
- **For official control labs, this is a frequent situation and, therefore, the idea was welcomed by the official laboratories**
- **In many of these situations, we received by the majority „<LOQ“-results**
- **A minority of laboratories still reported quantitative results**
- **How to give a proper feedback???**



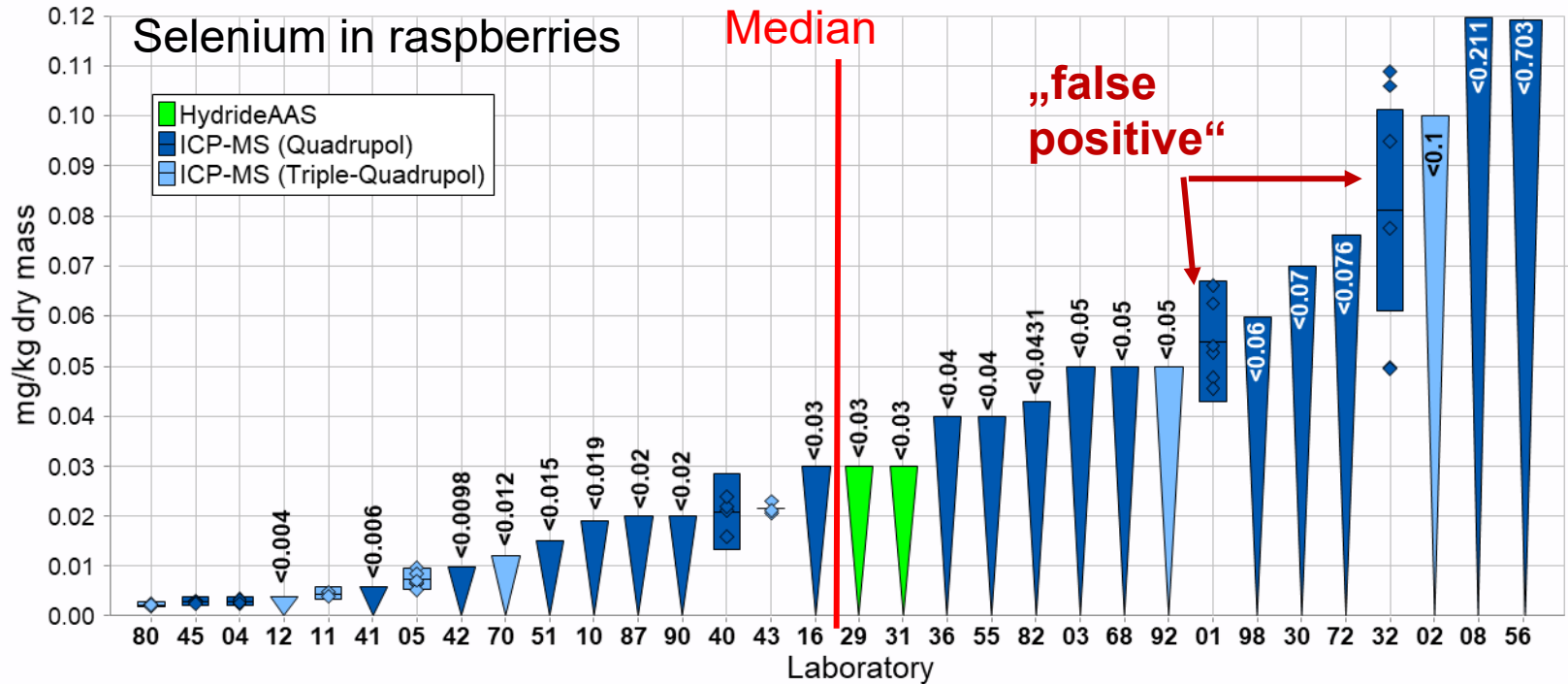
Analytes not reportable



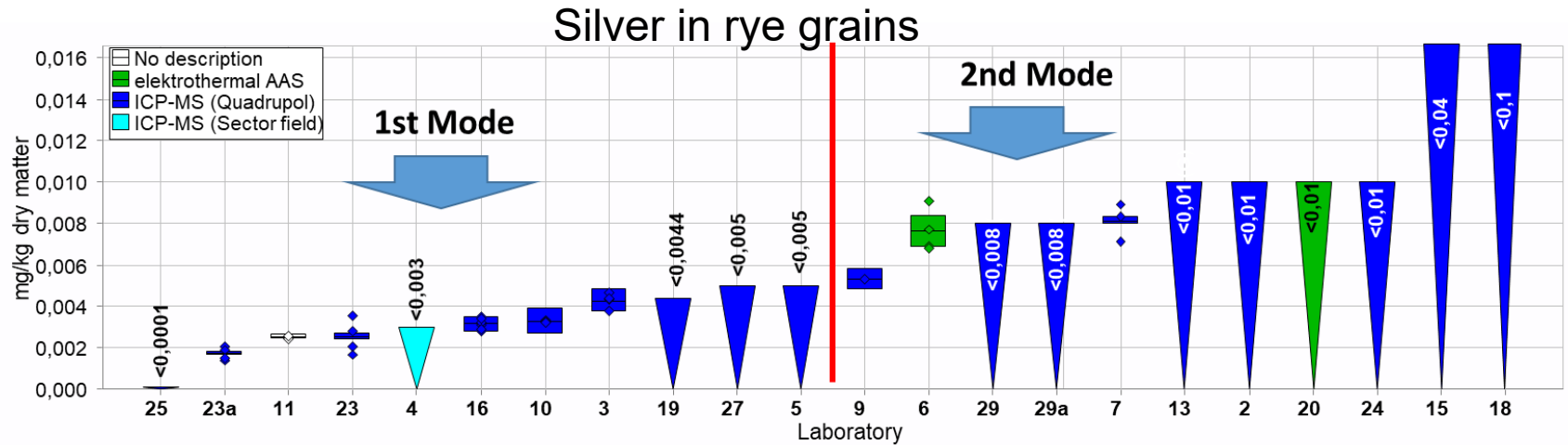
- PT not evaluable in the classic way (only few quantitative results accompanied by many „<LOQ“ results)
- Heterogeneity and contamination could be excluded
- Selenium analysis by ICP-MS prone to spectral interferences caused by matrix components (e.g. Calcium)



Approach for feedback to participants



- All results sorted by the value (real value or LOQ value)
- Determination of median
- All quantitative results higher than the median are considered as „false positive“ while the other quantitative results are rated as a more or less „true statement“



- All quantitative results lower than LOQ applied in the homogeneity test (0,01 mg/kg)
- Two modes visible in KDE \Rightarrow homogeneity cannot be assumed
- The narrower the spread of results (here: factor 4 between min. and max. quantitative result), the more difficult is the distinction between „true statement“ and „false positive“



- **PTs with measurands in the proximity of the LOQ are beneficial to detect certain laboratories' issues with contamination, specificity or LOQ determination**
- **In most cases classic approaches are applicable to give appropriate feedback to the participant**
- **In cases of a majority of non quantitative results, common procedures need to be amended by other concepts to safeguard proper feedback**



Thank you for your attention!

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