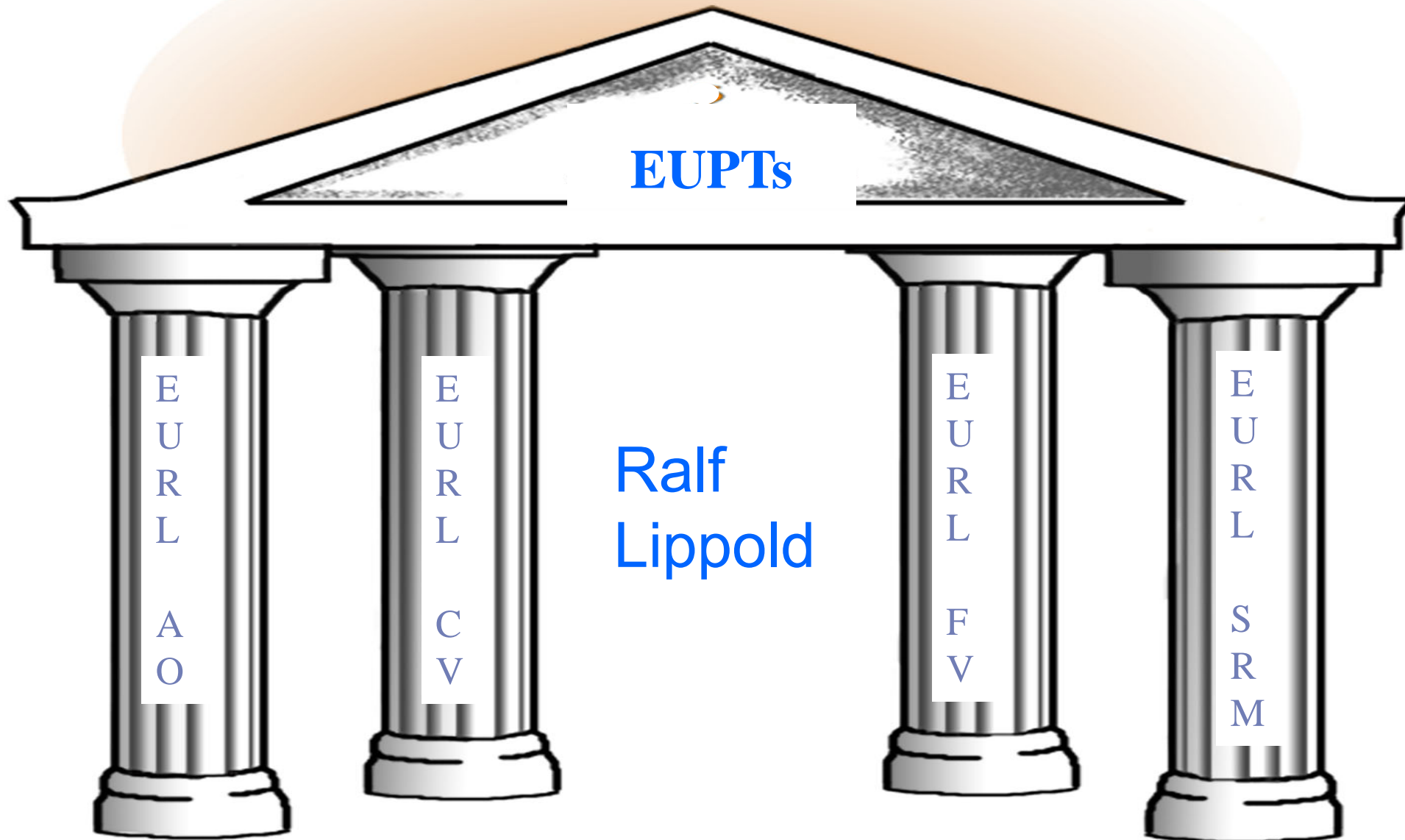
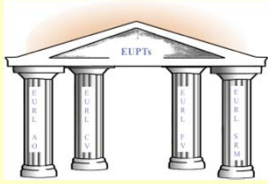


# EUPTs for Pesticides





## Scope

### Proficiency test(s) organised by the Commission (EUPTs)

- Mandatory participation for all laboratories analysing samples for the official control of pesticide residues (396/2005/EC)
- Mandatory participation for all NRLs

### Aim of EUPTs

- Obtaining information regarding **quality, accuracy** and **comparability** of the pesticide residue data
- Laboratories provided with an assessment of their **analytical performance** and the **reliability** of their data

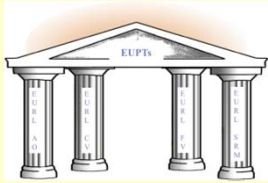


# Harmonisation

## Organisation of EUPTs by the 4 EURLs

- for Fruits and Vegetables (-FV),
- for Cereals and Feeding Stuff (-CF),
- for Single Residue Methods (-SRM) and
- for Food of Animal Origin and Commodities with high Fat Content (-AO)

## 1 Protocol with general procedures for all European Union proficiency tests (EUPTs)



# Organisation of EUPTs

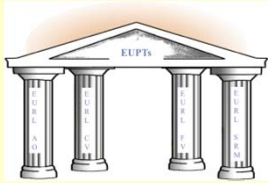
## Local organisation teams

- Consisting of members of the responsible EURLs
- Responsible for all administrative and technical matters concerning the organisation of the EUPT

## One common Scientific Committee (EUPT-Panel):

- Subgroup 1: Advisory Group (ADG)
- Subgroup 2: Independent Quality Control Group (QCG)

Scientists appointed by EURLs and approved by DG-SANCO



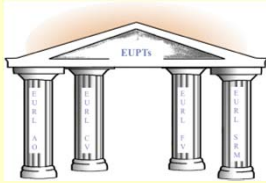
## Role of the Scientific Committee

### Helping in decisions concerning the design of the EUPTs:

- selection of pesticides to be included in the Target Pesticide List
- establishment of the minimum required reporting levels (MRRLs)
- evaluation and statistical treatment of the results

### Additional functions of the Quality Control Group:

- supervises the quality of the EUPT
- assists the EURLs with confidential aspects:
  - choice of the pesticides and
  - levels to be present in the test material



# Role of the EUPT-Panel

**Flamenco Night :**  
**ADVG Dancers**

4th Joint Workshop of the European Union Reference Laboratories for Residues of Pesticides

**EURL** EU REFERENCE LABORATORIES FOR RESIDUES OF PESTICIDES

4th Edition, Revised 03 Jan, 2014

**GENERAL PROTOCOL**  
**for EU Proficiency Tests on Pesticide Residues**  
**in Food and Feed**

**Introduction**

This protocol contains general procedures valid for all European Union Proficiency Tests (EUPTs) organised on behalf of the European Commission, DG-SANCO<sup>1</sup> by the four European Union Reference Laboratories (EURLs) responsible for pesticide residues in food and feed. These EUPTs are directed at laboratories belonging to the Network<sup>2</sup> of National Reference Laboratories (NRLs) and Official Laboratories (OLs) of the EU Member States. OLs from EFTA countries and EU Candidate countries are also welcome to participate in the EUPTs. OLs from Third countries may be permitted to participate on a case-by-case basis.

The following four EURLs for pesticide residues were appointed by DG-SANCO based on regulation 853/2004/EC<sup>3</sup>:

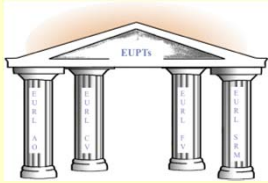
- EURL for Fruits and Vegetables (EURL-FV)
- EURL for Cereals and Feedingstuffs (EURL-CF)
- EURL for Food of Animal Origin and Commodities with High Fat Content (EURL-AO) and
- EURL for pesticides requiring Single Residue Methods (EURL-SRM)

The aim of these EUPTs is to obtain information regarding the quality, accuracy and comparability of pesticide residue data in food and feed reported to the European Union within the framework of the national control programmes and the EU multiannual co-ordinated control programme<sup>4</sup>. Participating laboratories will be provided with an assessment of their analytical performance that

<sup>1</sup> DG-SANCO - European Commission, Health and Consumer Protection Directorate General  
<sup>2</sup> For more information about the EURL/NL/OL network please refer to the EURL web-portal under: <http://www.eurl-pesticides.eu>  
<sup>3</sup> Regulation (EC) No 853/2004 of the European Parliament and of the Council on official controls performed to ensure the compliance of operators with food and feed law, animal health and animal welfare rules, published at OJ of the EU L31 of 23.03.2004  
<sup>4</sup> European Commission Proficiency Tests for Pesticide Residues in Fruits and Vegetables, Trends in Analytical Chemistry, 2010, 29 (1), 75 - 83.

[www.eurl-pesticides.eu](http://www.eurl-pesticides.eu)





# Submission of Results and Information



Home - Windows Internet Explorer bereitgestellt von LGL

http://pesticides.food.dtu.dk/apex/f?p=150:1:7972799385457

File Bearbeiten Ansicht Favoriten Extras ? X

Favoriten Home

Welcome: JOHN DOE@TEST.NU Feedback Logout

 **EURL-AO**  EU Reference Laboratories for Residues of Pesticides

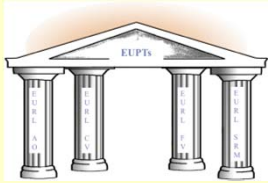
## Main page EUPT AO-09

Links to subpages:	European Commission's Proficiency Test on Pesticide Residues in Food of Animal Origin - EUPT AO-09, 2014	Contact Persons:
<b>0. Test item receipt</b> Acknowledge receipt of parcel with test sample.	Welcome to the result submission pages of EUPT AO 09. This website is accessible from 23 April to 02 June 2014. Sub-pages 1-3 will close on 23 May 2014, 13.00 CET, whereas sub-page 4, concerning additional information, will be accessible from 27 May to 02 June 2014.  As soon as you receive the package with the test items, please enter sub-page <b>0. Test item receipt</b> to notify the organizer.	Björn Hardebusch, Raif Lippold,  European Union Reference Laboratory for Pesticides in Food of Animal Origin, State Institute for Chemical and Veterinary Analysis of Food, P.O. Box 100462, D-79123 Freiburg / Germany  <a href="mailto:eurl-pesticides@cvuafr.bwl.de">eurl-pesticides@cvuafr.bwl.de</a>
<b>1. Pesticide scope</b> Specify which pesticides you analysed for	To submit your EUPT AO-09 results, please use sub-pages 1-3. Each sub-page contains instructions on how to enter the data, and each sub-page must be saved separately.	<b>Export to Excel</b>
<b>2. Results</b> Enter your analytical results	Enter the sub-pages in order 1-3  <b>1. Pesticide scope.</b> Here you should indicate which of the 88 pesticides on the list you have analysed for, which you have detected and if they are within your routine scope.	<b>When completed, you can download your results in an Excel file</b>  <a href="#">View and download your results.</a>
<b>3. Methods</b> Describe the methods used for detected pesticides	<b>2. Results.</b> Here you can enter your results for the pesticides you have detected in the samples - concentrations and recoveries.  <b>3. Methods.</b> Here you can enter information about the methods you have used. For each detected pesticide, please indicate details about the analytical procedure, e.g. sample weight, extraction solvents, clean-up, calibration, GC- and LC-detectors.	
<b>4. Additional information requested</b> <a href="#">Describe the methods used for false negatives etc.</a>	Finalise with sub-page  <b>4. Additional information requested.</b> The webpage will be open to everybody after the deadline on 27 May. Here you are requested to enter or update information about the methods you have used for all pesticides present in the test item. This includes both detected pesticides and false negatives. If no information on the method used is given, the organiser has the right not to accept the analytical results reported.	

Remember to save each sub-page separately before you leave it! You can enter the pages as often as you wish until the website is closed. You can e.g. enter all data for the GC pesticides one day (on sub-page 1 to 3) and the LC results another day. Just remember to enter data in the right order from sub-page 1 to 3, because data on sub-page 1 is used on sub-page 2, etc. All data correction must be done before the deadline.

Fertig



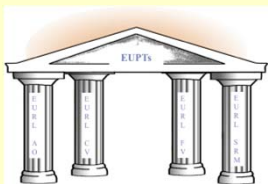


## Treatment and Assessment of Results

### Estimation of the true concentration ( $\mu$ )

- Calculation of the median value for every compound
- Median used as the assigned value
- EUPT Panel may decide to use only part of the population of results to establish the median (e.g. using only results with z-scores  $\leq 5.0$ ).
- From 2014 onwards: robust mean according algorithm A as assigned value





## Treatment and Assessment of Results

### Target standard deviation ( $\delta$ )

- calculated using a Fit-For-Purpose Relative Standard Deviation (FFP-RSD) approach
- based on experience from previous EUPTs FFP-RSD is set at 25% <sup>(1)</sup>
- **$\delta = b * \mu$**

with  $b = \text{FFP-RSD} = 0.25$

- (1) The expanded measurement uncertainty U for the comparison of results from different pesticide laboratories is set to 50 % (Document SANCO 12471/2013 - Guidance Document on Analytical Quality Control and Method Validation Procedures for Pesticide Residues Analysis in Food and Feed)



# Treatment and Assessment of Results

## z-Scores

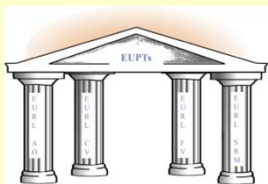
- $z_i = (x_i - \mu_i) / \delta_i$

$x_i$  - value reported by the laboratory

$\mu_i$  - the assigned value (median)

$\delta_i$  - standard deviation [level of the median for each pesticide (i)]

- z-scores of  $> 5$  will be reported as “+5”

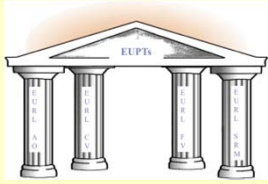


# Treatment and Assessment of Results

## Interpretation of z-Scores

z-Score	Evaluation
$ z  \leq 2$	The analysis fulfils the requirements - satisfactory
$2 <  z  \leq 3$	The analysis should be examined - questionable
$ z  > 3$	The analysis does not fulfil the requirements - unsatisfactory

- For false negative results z-scores will be calculated using the MRRL
- In cases  $RL < MRRL$  the RL will be used
- No calculation of z-scores for false positive results

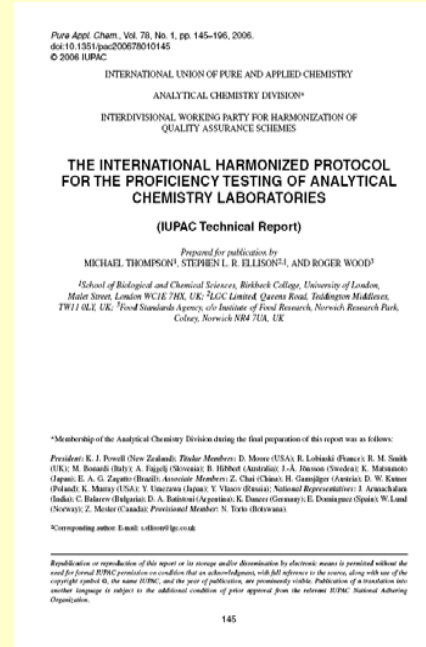
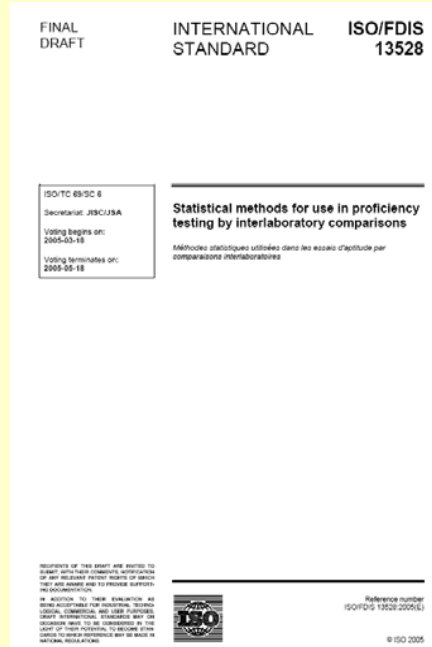


# Treatment and Assessment of Results

## ***Category A and B classification***

- **Category A Laboratories**
  - quantified at least 90% of the pesticides present in the sample
  - reported no false positives
  - sought all the pesticides mandatory present in the test material
- These laboratories demonstrate 'sufficient scope'

# EUPTs organized by EURL-AO



# EUPTs organized by EURL-AO

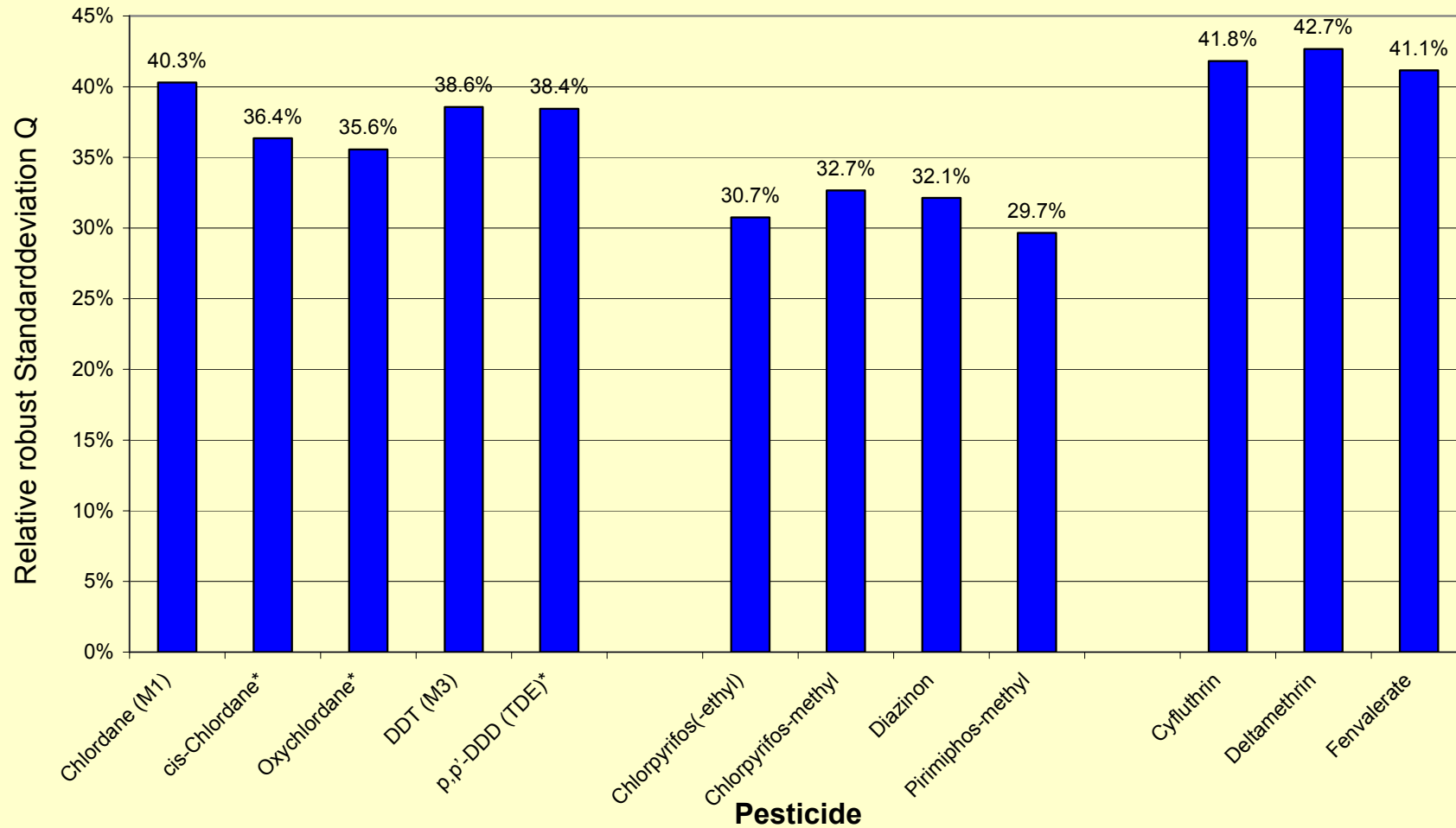
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## *Test Items*

- Matrices Taken from the Multi Annual Coordinated Control Programme (MACCP) of the European Union
  - Commodity Group milk: butter (2009), cream (2012)
  - Commodity group eggs: hen´s egg (2008 + 2014)
  - Commodity group meat (2010 – pork; 2011 + 2013 poultry):
  - Vegetable oil (2006 + 2007)
- Target Analytes
  - Pesticides to be analysed within the MACCP
  - Extension by additional pesticides

# Results of EUPT AO 06 – Poultry Meat

Overview about Results (EUPT AO 06 - Poultry Meat)



## List of mandatory compounds

## Conception

Analyte	MRRL [mg/kg]	Analyte	MRRL [mg/kg]	Analyte	MRRL [mg/kg]
Abamectin B1 $\alpha$ (F)	0.005	Diazinon (F)	0.005	cis-Heptachlor epoxide (F)	0.002
Abamectin B1 $\beta$ (F)	0.005	Deltamethrin (F)	0.01	trans-Heptachlor epoxide (F)	0.002
Aldrin (F)	0.002	Dieldrin (F)	0.002	Malathion (parent only)	0.005
Azinphos-ethyl (F)	0.005	alpha-Endosulfan (F)	0.002	Methidathion	0.005
Bifenthrin (F)	0.005	beta-Endosulfan (F)	0.002	4.4'-Methoxychlor (F)	0.005
cis-Chlordane (F)	0.002	Endosulfan sulfate (F)	0.002	Parathion(-ethyl) (F)	0.005
trans-Chlordane (F)	0.002	Endrin (F)	0.002	Parathion-methyl (parent only)	0.005
Oxychlordane (F)	0.002	Fenthion (F)	0.005	Phosmet (parent only)	0.02
Chlorobenzilate (F)	0.01	Fenthion sulfoxide (F)	0.005	Phoxim (F)	0.02
Chlorfenvinphos (F)	0.005	Fenthion sulfone (F)	0.005	Pendimethalin (F)	0.02
Chlorpyrifos(-ethyl) (F)	0.005	Fenthion oxon (F)	0.005	Permethrin (sum)	0.01
Chlorpyrifos-methyl (F)	0.005	Fenthion oxon sulfoxide (F)	0.005	Pirimiphos-methyl (F)	0.01
Cyfluthrin (sum of isomers) (F)	0.01	Fenthion oxon sulfone (F)	0.005	Profenofos (F)	0.005
Cypermethrin (sum of isomers) (F)	0.01	Fenvalerate (F) (sum of RS/SR and RR/SS isomers)	0.005	Pyrazophos (F)	0.005
lambda-Cyhalothrin (F) (sum of isomers)	0.01	Hexachlorobenzene (HCB) (F)	0.002	Quintozene (parent only) (F)	0.002
p.p'-DDE (F)	0.002	alpha-HCH (F)	0.002	Resmethrin (F)	0.01
p.p'-DDD (TDE) (F)	0.002	beta-HCH (F)	0.002	Tecnazene (F)	0.005
p.p'-DDT (F)	0.002	gamma-HCH (Lindane) (F)	0.002	Triazophos (F)	0.005
o.p'-DDT (F)	0.002	Heptachlor (F)	0.002	Vinclozolin (parent only)	0.02



## List of voluntary compounds

## Conception

Analyte	MRRL [mg/kg]	Analyte	MRRL [mg/kg]
Bixafen (parent only)	0.01	Flusilazole (F) (parent only)	0.01
Boscalid (F) (parent only)	0.01	Indoxacarb (F) (sum of isomers)	0.01
Carbendazim (Carbendazim only)	0.01	Metazachlor (parent only)	0.01
Chlorpropham (F) (parent only)	0.01	Prochloraz (parent only)	0.01
Cyproconazole (F)	0.01	Prothioconazole-desthio	0.01
Epoxiconazole (F)	0.01	Spinosyn A (F)	0.01
Etofenprox (F)	0.01	Spinosyn D (F)	0.01
Famoxadone	0.01	tau-Fluvalinate (F)	0.01
Fenpropidin (parent only)	0.01	Tebuconazole	0.01
Fenpropimorph (parent only)	0.01	Tetraconazole (F)	0.01
Fluquinconazole (F)	0.01	Thiacloprid (F)	0.01
Additional pesticide spiked (not in target list):		Metaflumizone	

## Spiked compounds

## Conception

2013 Mandatory		MRL (VO 396/2005) mg/kg	MRRL mg/kg	spiking level (target)	Ratio spiking level to MRL	Ratio spiking level to MRRL	Ratio spiking level to MRRL
Group	Analyte	poultry		mg/kg		µg/kg meat	(worst case: 70% recovery)
Organo-chlorines	Oxychlorane* (F)	0.05	0.002	0.025	0.500	12.5	8.8
	o,p'-DDT (F)	1	0.002	0.030	0.300	15.0	10.5
	beta-Endosulfane* (F)	0.05	0.002	0.045	0.900	22.5	15.8
	Endosulfansulfate* (F)	0.05	0.002	0.060	1.200	30.0	21.0
	trans-Heptachlorepoxid (F)	0.2	0.002	0.025	1.250	12.5	8.8
	HCH beta (F)	0.1	0.002	0.015	1.500	7.5	5.3
Organophosphorous	Diazinon* (F)	0.05	0.005	0.065	1.300	13.0	9.1
	Chlorpyrifos(-ethyl)* (F)	0.05	0.005	0.055	1.100	11.0	7.7
	Phosmet	0.1	0.02	0.150	1.500	7.5	5.3
Pyrethroids	Cypermethrin* (F)	0.05	0.01	0.080	1.600	8.0	5.6
	Deltamethrin (F)	0.1	0.01	0.095	9.500	9.5	6.7
	lambda-Cyhalothrin* (F)	0.02	0.01	0.115	5.750	11.5	8.1
2013 Voluntary		MRL (VO 396/2005) mg/kg	MRRL mg/kg	spiking level (target)	Ratio spiking level to MRL	Ratio spiking level to MRRL	Ratio spiking level to MRRL
"new compounds"	Metaflumizone	0.02	0.01	0.080	4.000	8.0	5.6
	Prochloraz	0.1	0.01	0.095	0.950	9.5	6.7
	Tebuconazole	0.1	0.01	0.075	0.750	7.5	5.3

## Data - Participants

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- 113 Participating Laboratories (2012: 106)
  - 27 different countries
  - 27 Member States were represented by NRLs
  - 1 NRL (EFTA-Countries)
  - 85 OFLs from different Member States and Norway

## Data - Participants

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- 108 (2012: 102) Laboratories reported results
  - 28 different countries
  - 26 Member States were represented by NRLs (no result from Denmark)
  - 1 NRL (EFTA-Countries)
  - 81 OFLs from different Member States and Switzerland

## Data - Participants

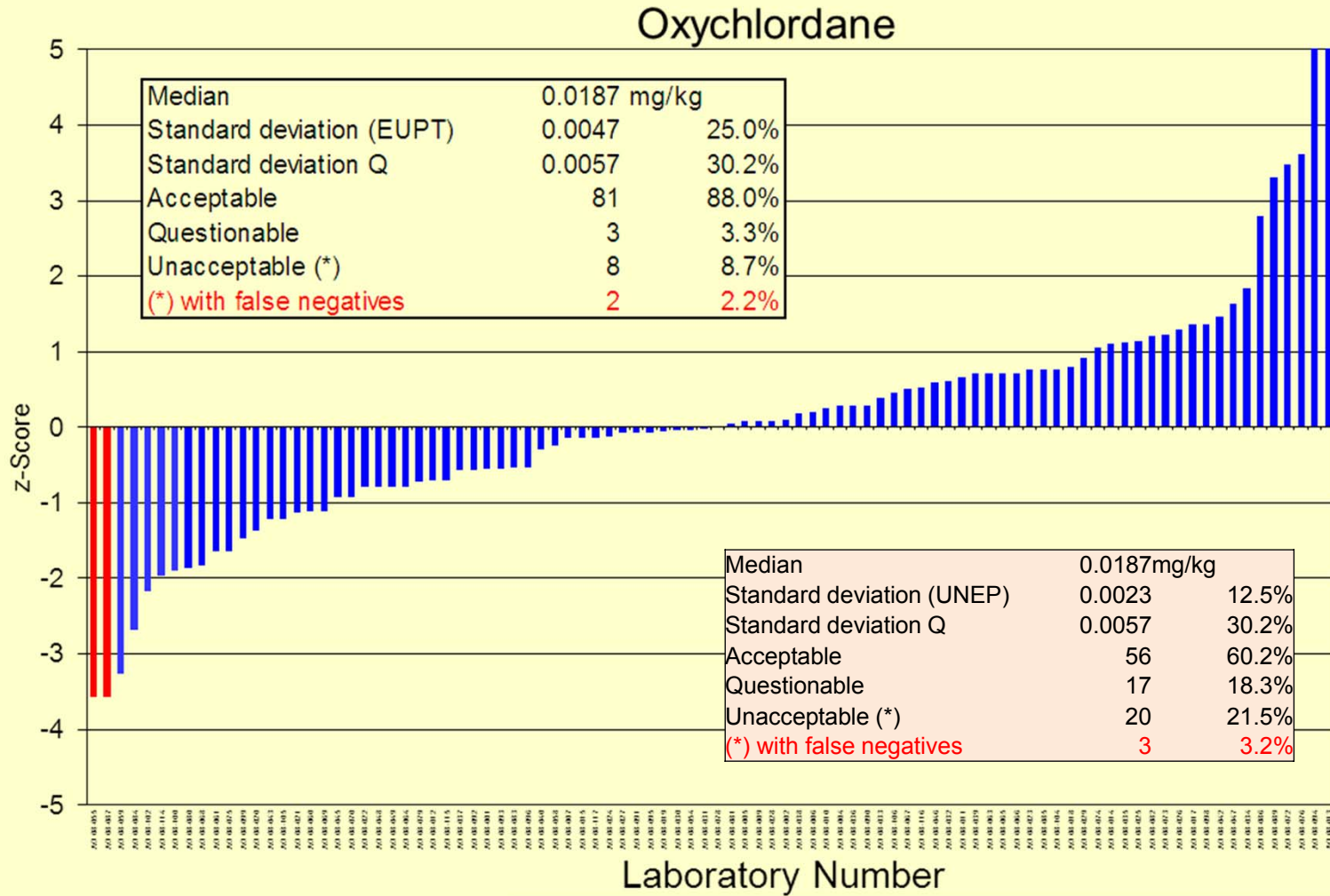
Austria	1	Germany	20 (+1)	Poland	9 (+1)
Belgium	5	Greece	3	Portugal	2 (+1)
Bulgaria	1	Hungary	5 (+2)	Romania	5 (+1)
Cyprus	1	Ireland	2	Slovakia	3 (+1)
Czech Republic	2	Italy	11 (-1)	Slovenia	1
Denmark	1 (-2)	Latvia	1	Spain	10 (+4)
Estonia	2	Lithuania	1	Sweden	2
Finland	1	Netherlands	3 (-1)	Switzerland	1
France	11 (-2)	Norway	1	United Kingdom	3
<b>Summary</b>		<b>European Union</b>	<b>25</b>	<b>EFTA</b>	<b>2</b>

## False Positive Results

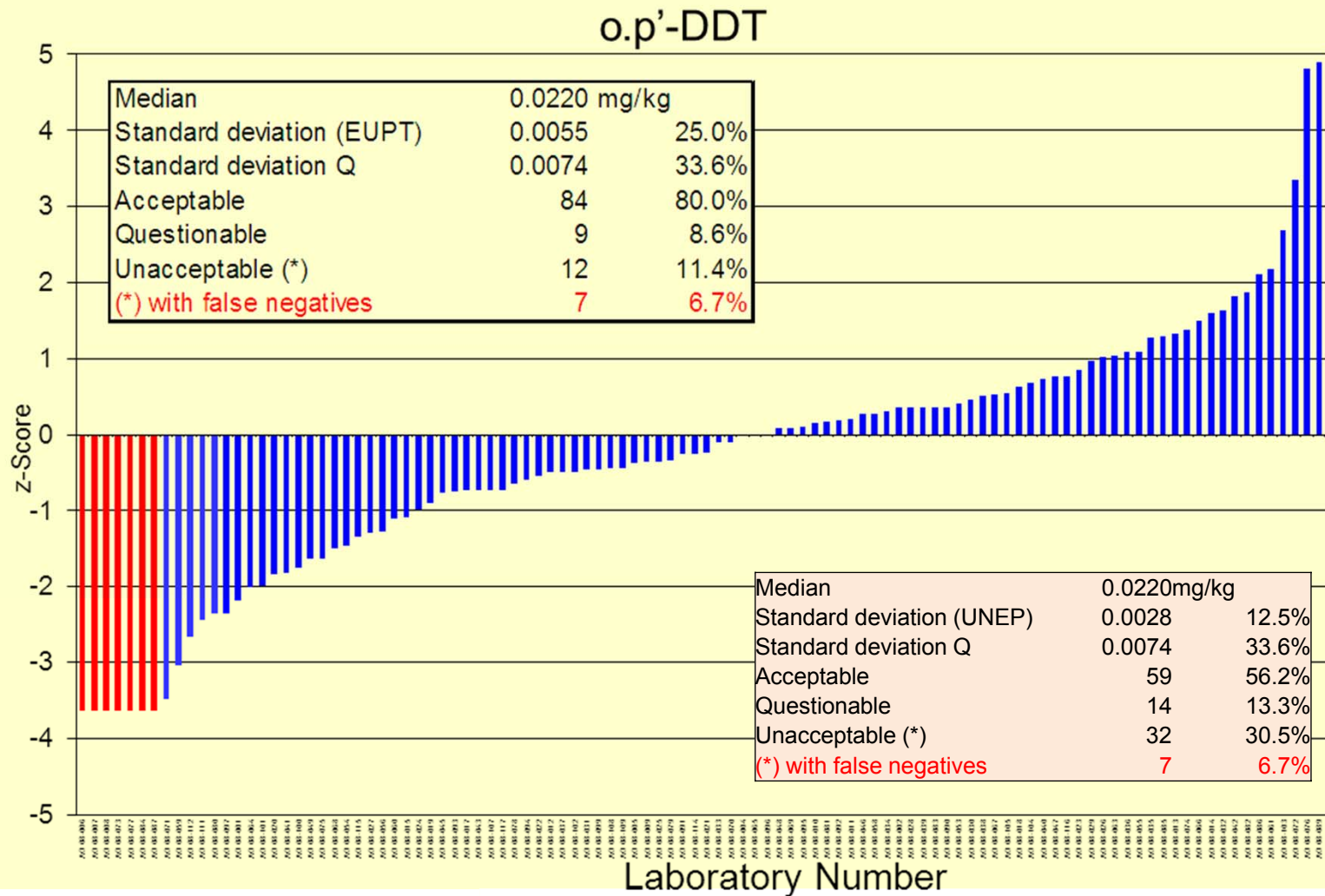
- Results exceeded the MRRL values will be considered as false positives.
  - Results below the corresponding MRRL concentrations will be ignored.
- 25 false positive results from 25 different laboratories were observed.

Pesticide	Number	Lab-Number (concentration - mg/kg)
Aldrin	1	108 (0.052)
p,p'-DDD (TDE)	5	6 (0.0078), 45 (0.0127), 80 (0.0090), 105 (0.0050), 106 (0.0259)
p,p'-DDT	2	71 (0.0054), 84 (0.0080)
Dieldrin	1	26 (0.0028)
alpha-Endosulfan	1	61 (0.0021)
alpha-HCH	1	7 (0.0040)
gamma-HCH (Lindane)	2	8 (0.130), 46 (0.0140)
cis-Heptachlor epoxide	11	13 (0.0105), 22 (0.0130), 42 (0.0276), 56 (0.0163), 58 (0.0043), 74 (0.0148), 75 (0.0110), 90 (0.0180), 93 (0.0175), 109 (0.0144), 111 (0.0085)
Cyproconazole	1	59 (0.0639)

# EUPT-AO 08: Results (Mandatory Pesticides)



# EUPT-AO 08: Results (Mandatory Pesticides)





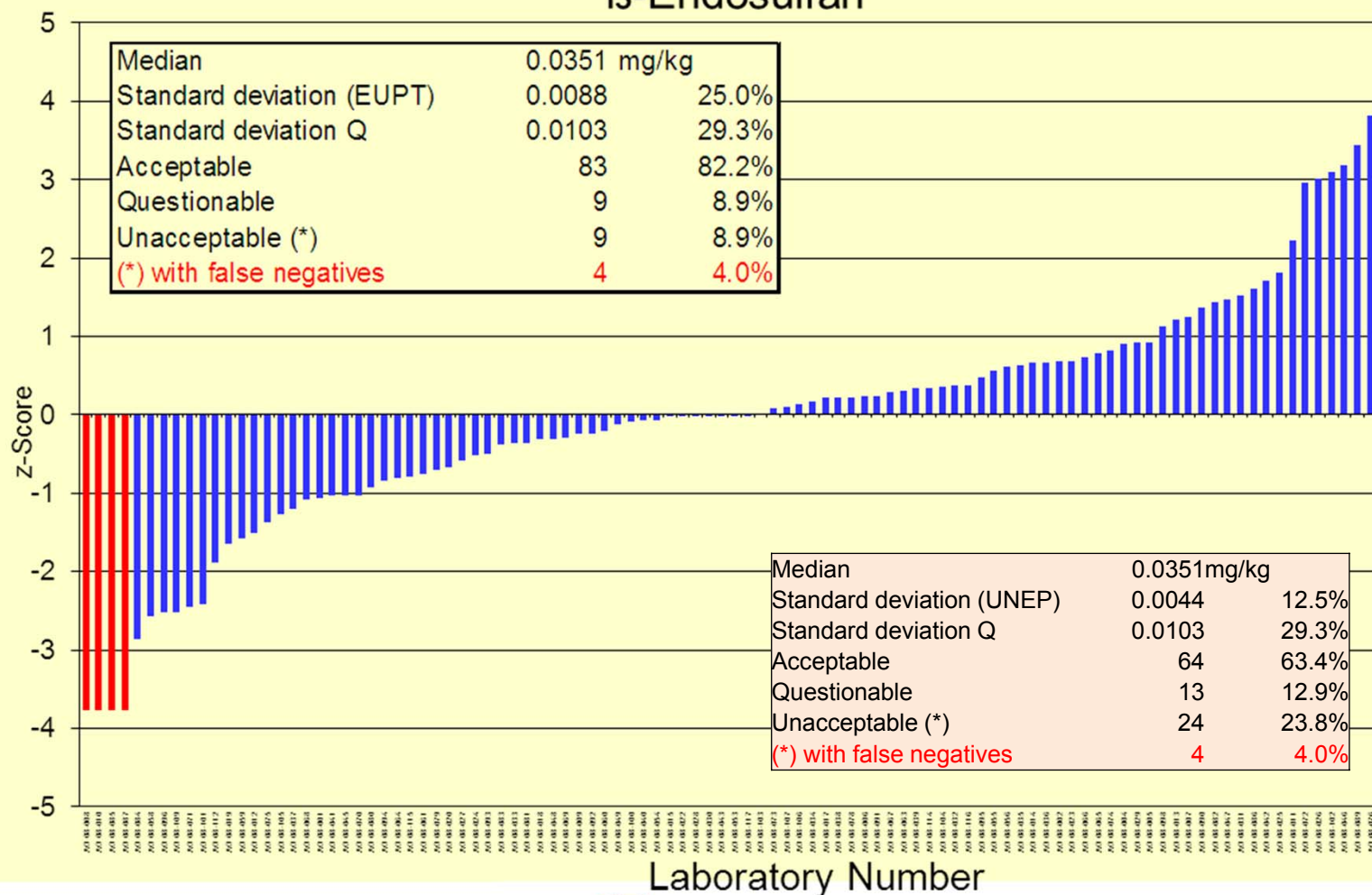
## The “o.p’-DDT” case

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- In EUPT AO 08 in total 7 labs reported o.p’-DDT as „nd“ (6, 7, 8, 73, 77, 84, 87)
  - 1 lab reported p.p’-DDD (TDE) instead (6 (0.0078))
  - 1 lab reported p.p’-DDT instead (84 (0.0080))

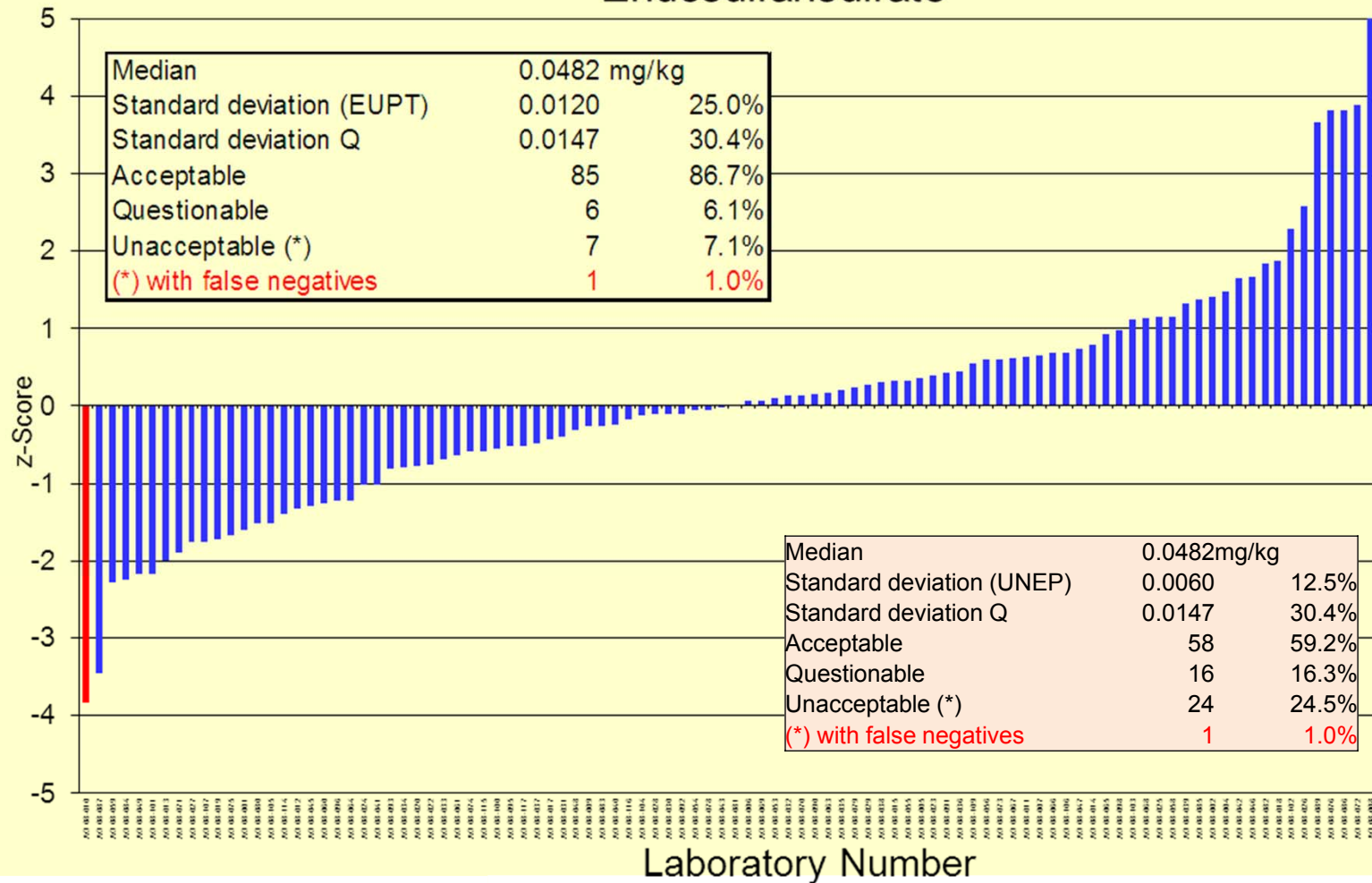
# EUPT-AO 08: Results (Mandatory Pesticides)

## β-Endosulfan

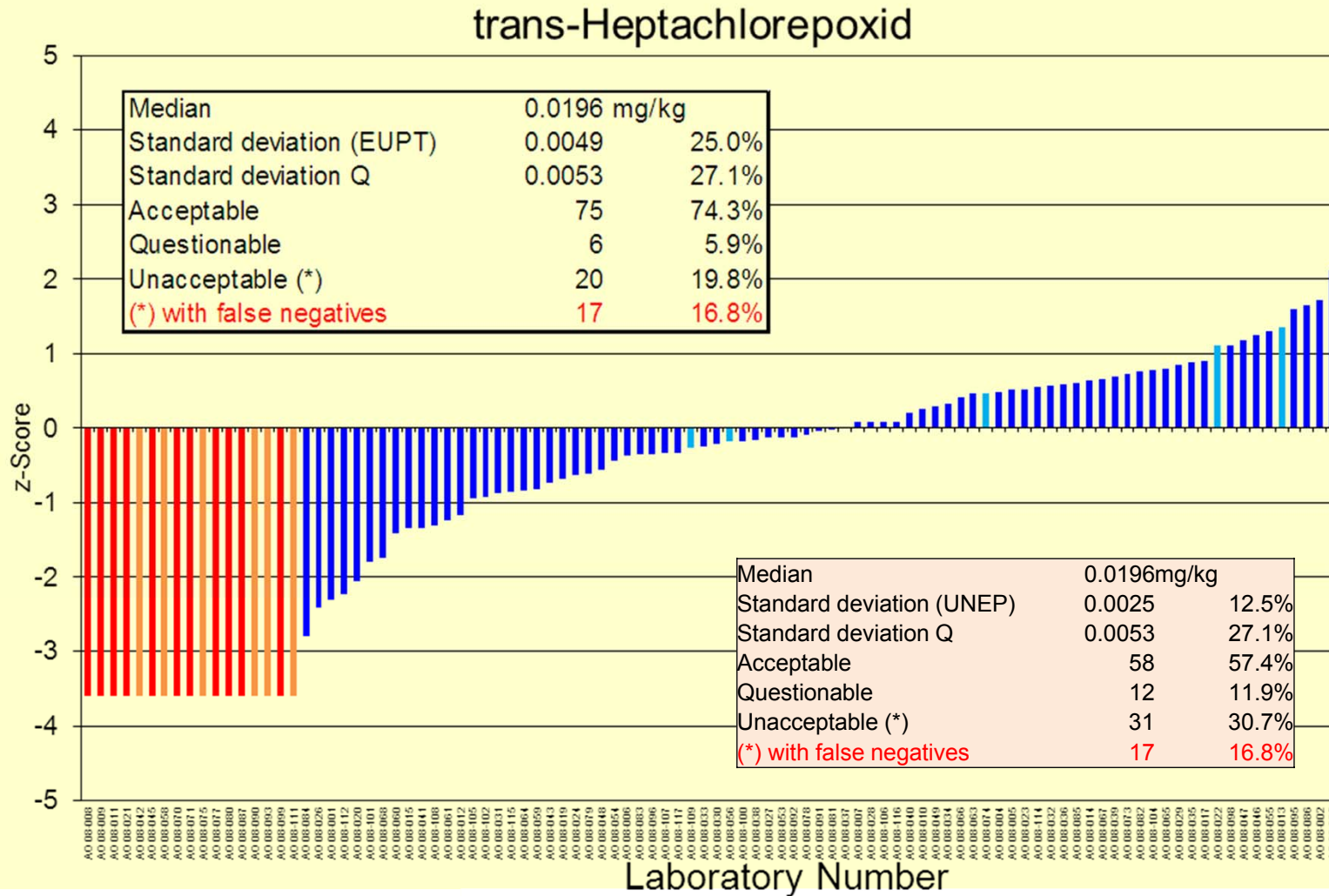


# EUPT-AO 08: Results (Mandatory Pesticides)

## Endosulfansulfate



# EUPT-AO 08: Results (Mandatory Pesticides)

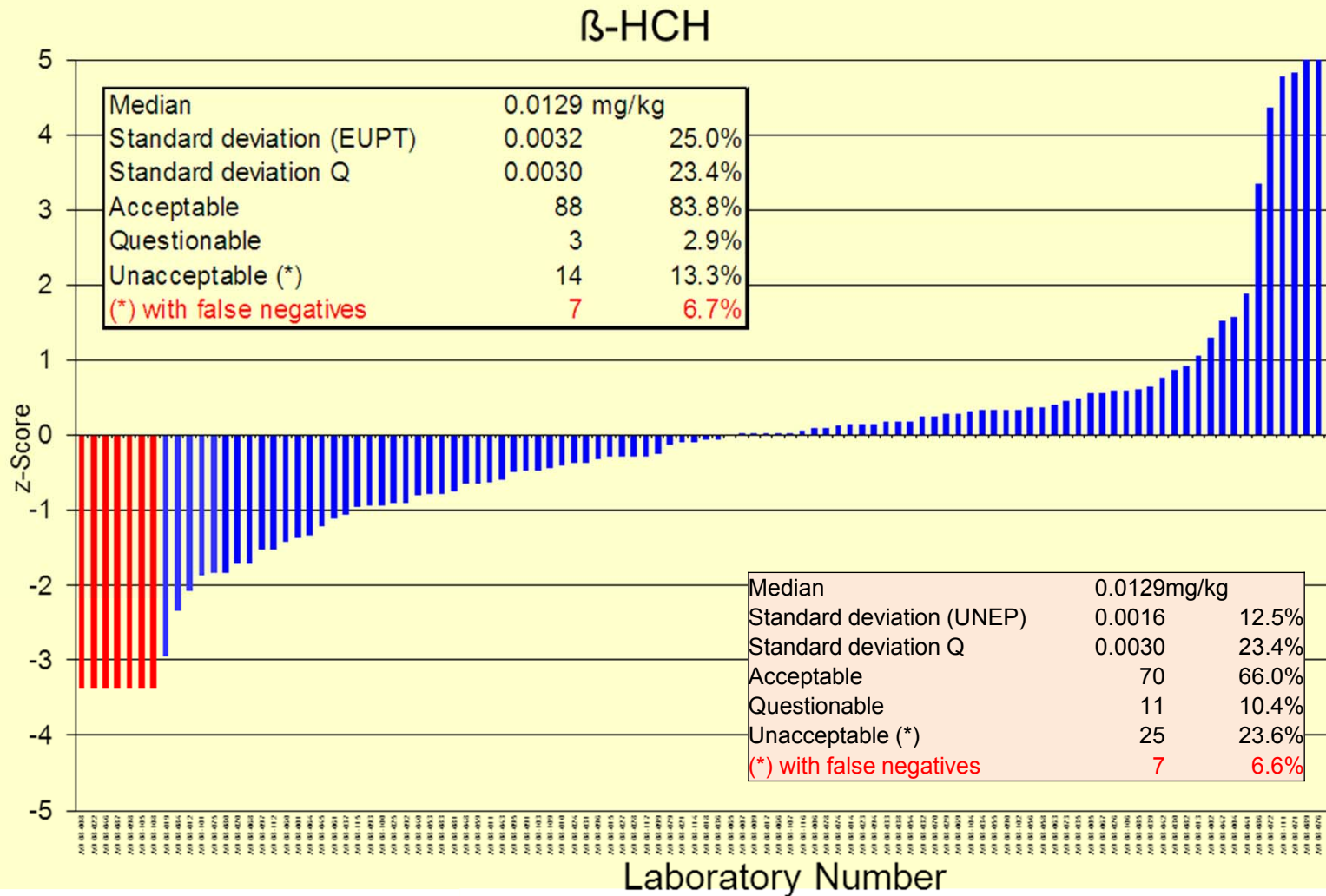


## The “cis-heptachlor epoxide” case

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- In EUPT AO 08 in total 11 labs reported cis-heptachlor epoxide
- Blank material was free of cis-heptachlor epoxide
  - 6 labs reported no trans-heptachlor epoxide (42, 58, 75, 90, 93, 111)
  - 5 labs reported both, cis- and trans-heptachlor epoxide (13, 22, 56, 74, 109)

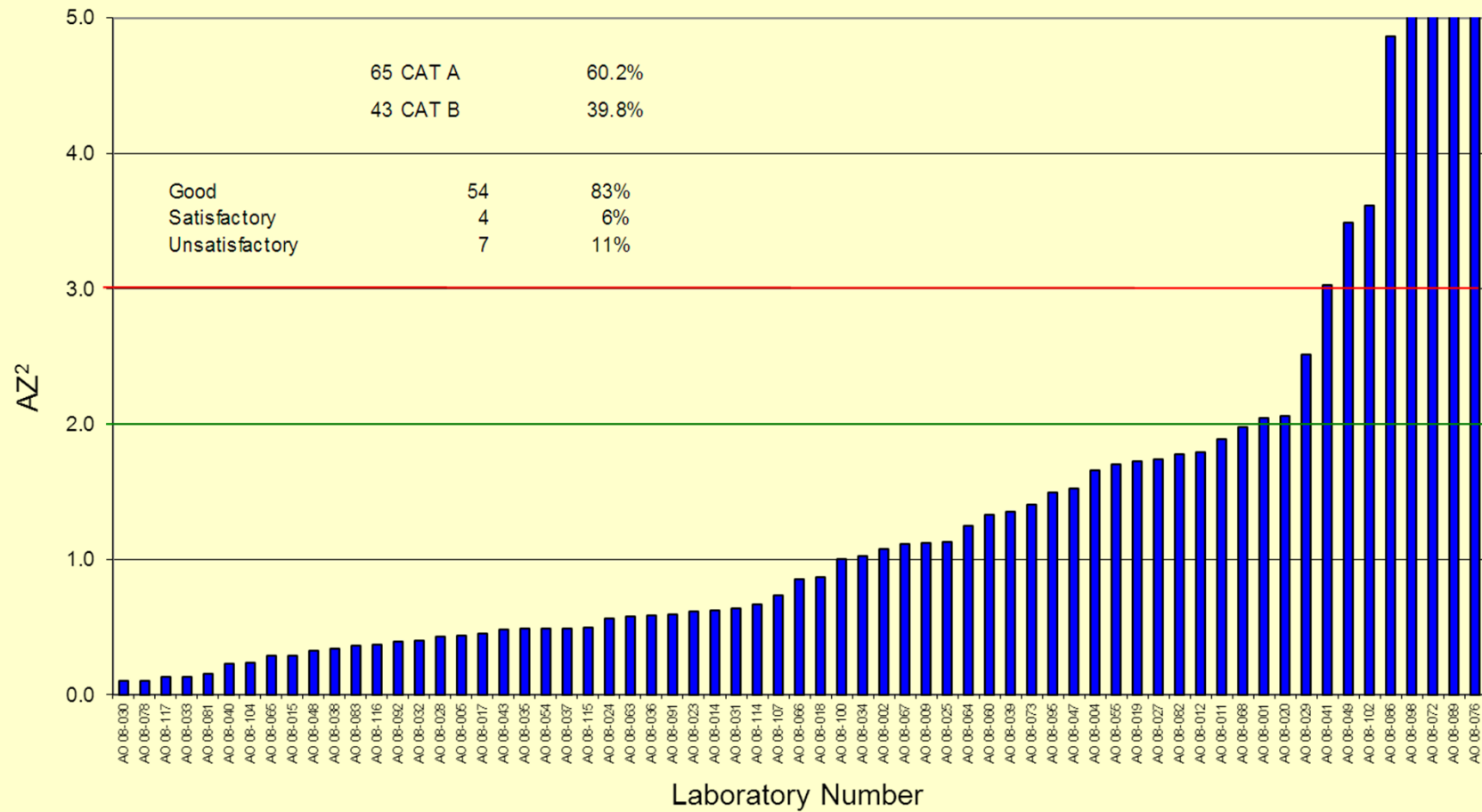
# EUPT-AO 08: Results (Mandatory Pesticides)





# Results (Mandatory Pesticides)

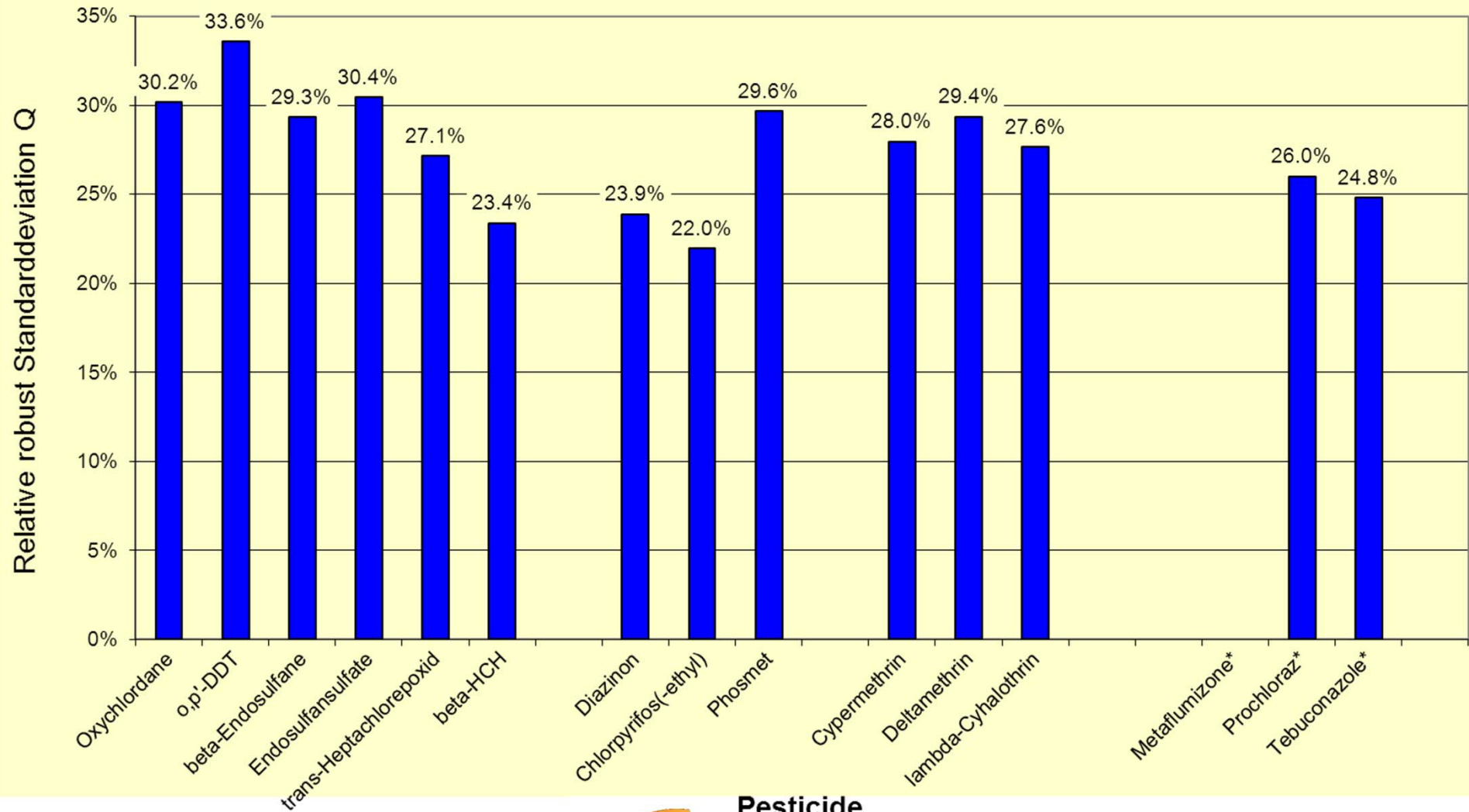
Category A: Average of Squared z-Scores





# Results

## Overview about Results (EUPT AO 08 - Raw Poultry Meat)



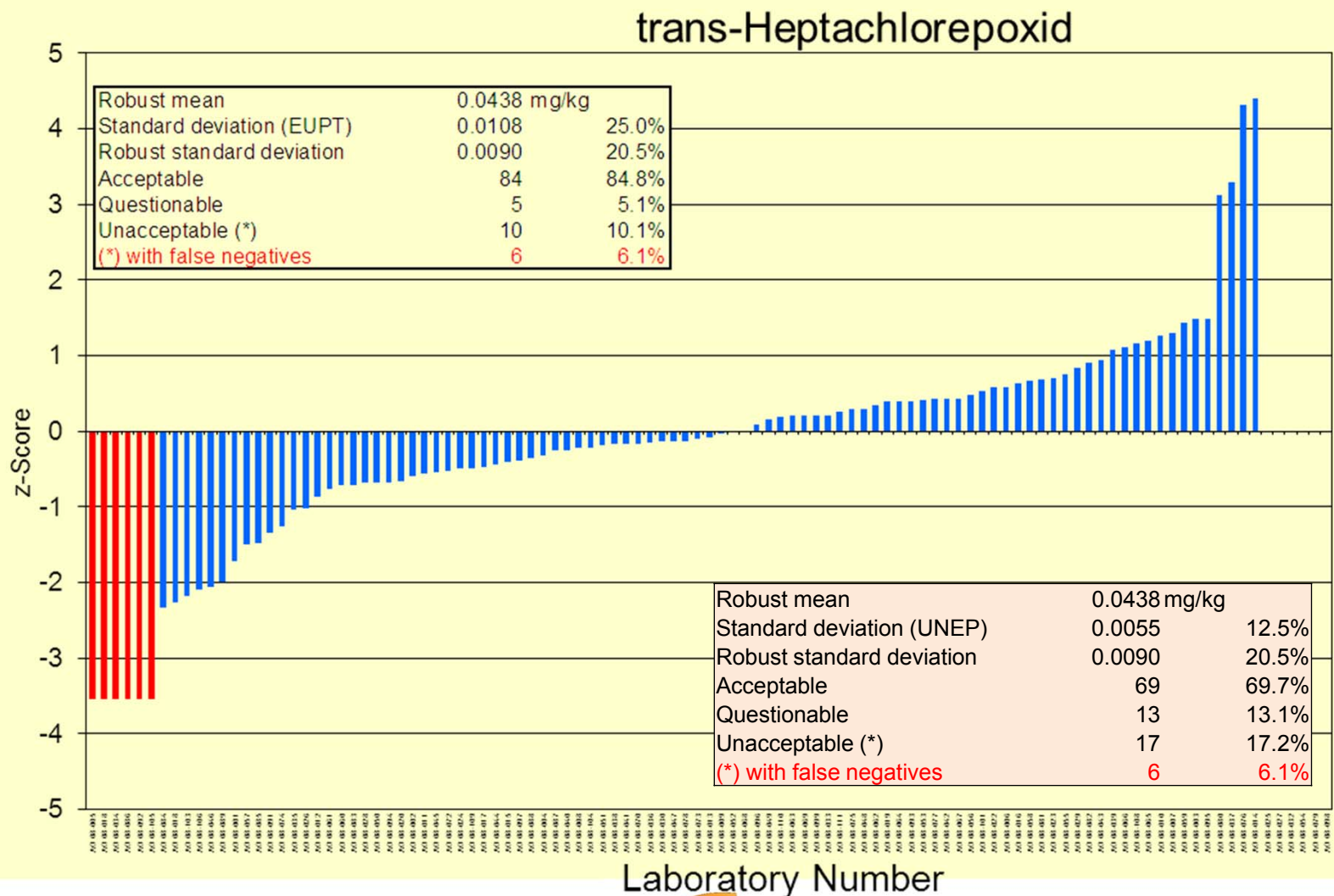
### Pesticide



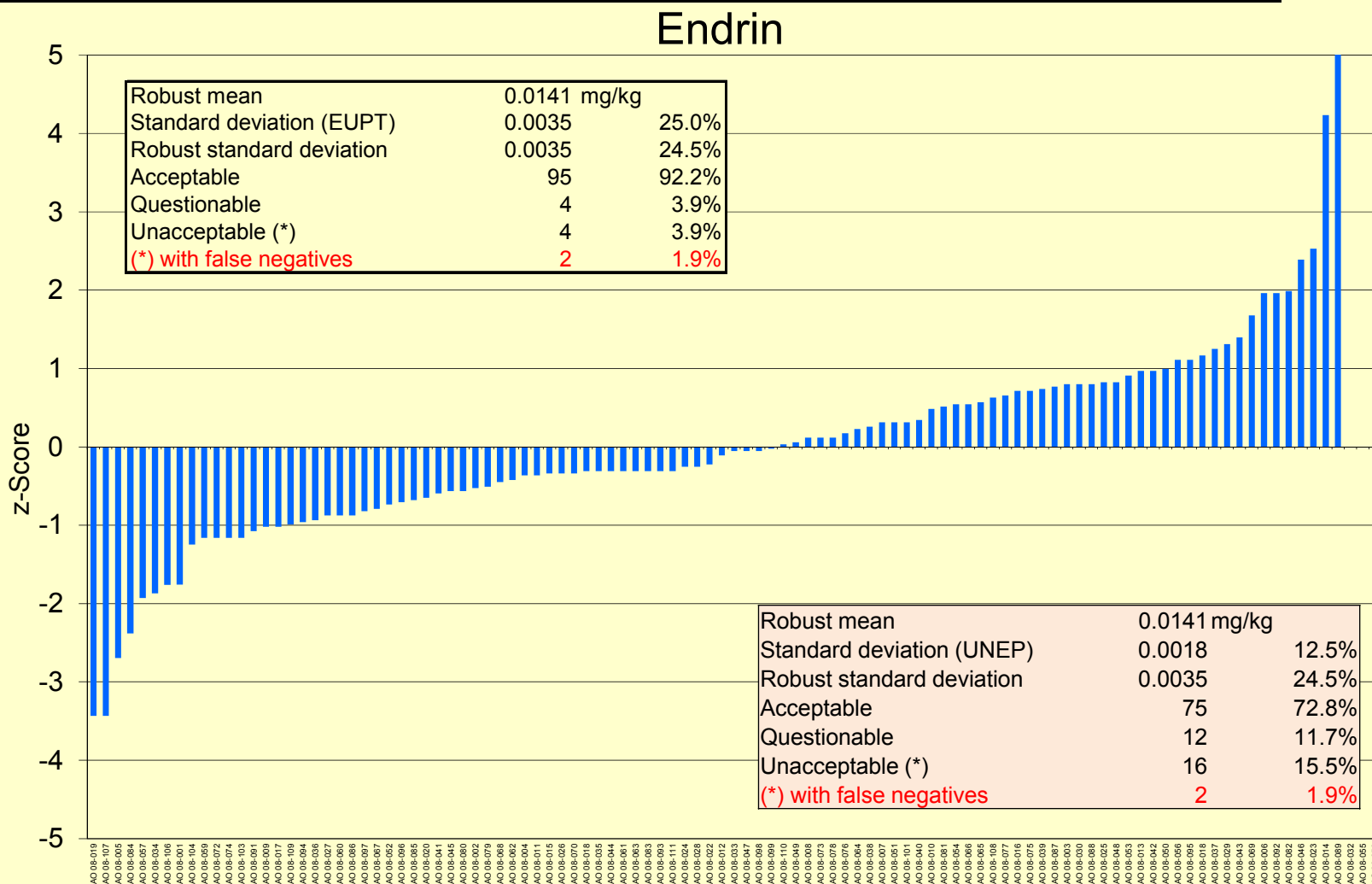
EUROPEAN UNION REFERENCE LABORATORY

PESTICIDE RESIDUES IN FOOD OF ANIMAL ORIGIN & COMMODITIES WITH HIGH FAT CONTENT

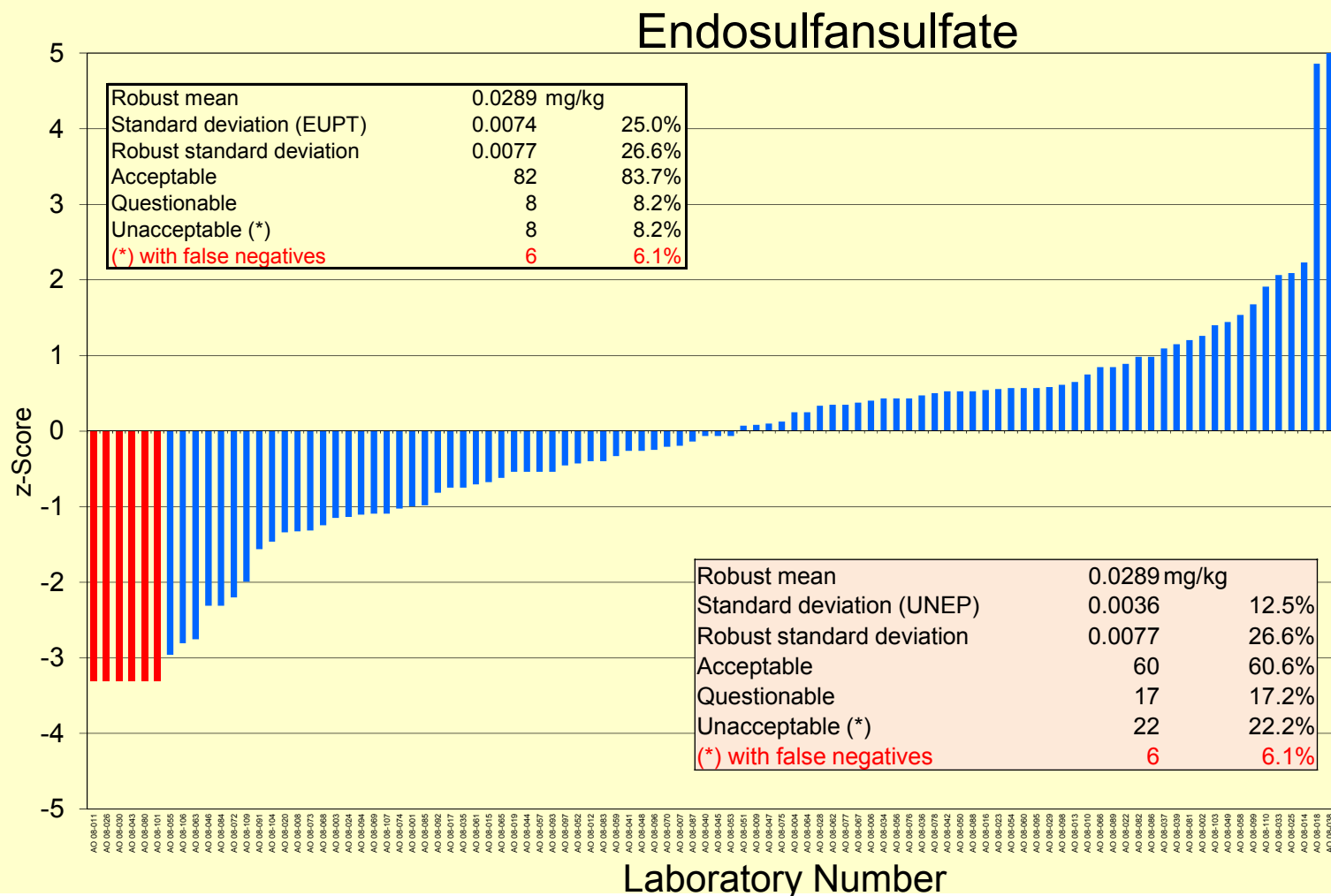
# EUPT-AO 09: Results (Mandatory Pesticides)



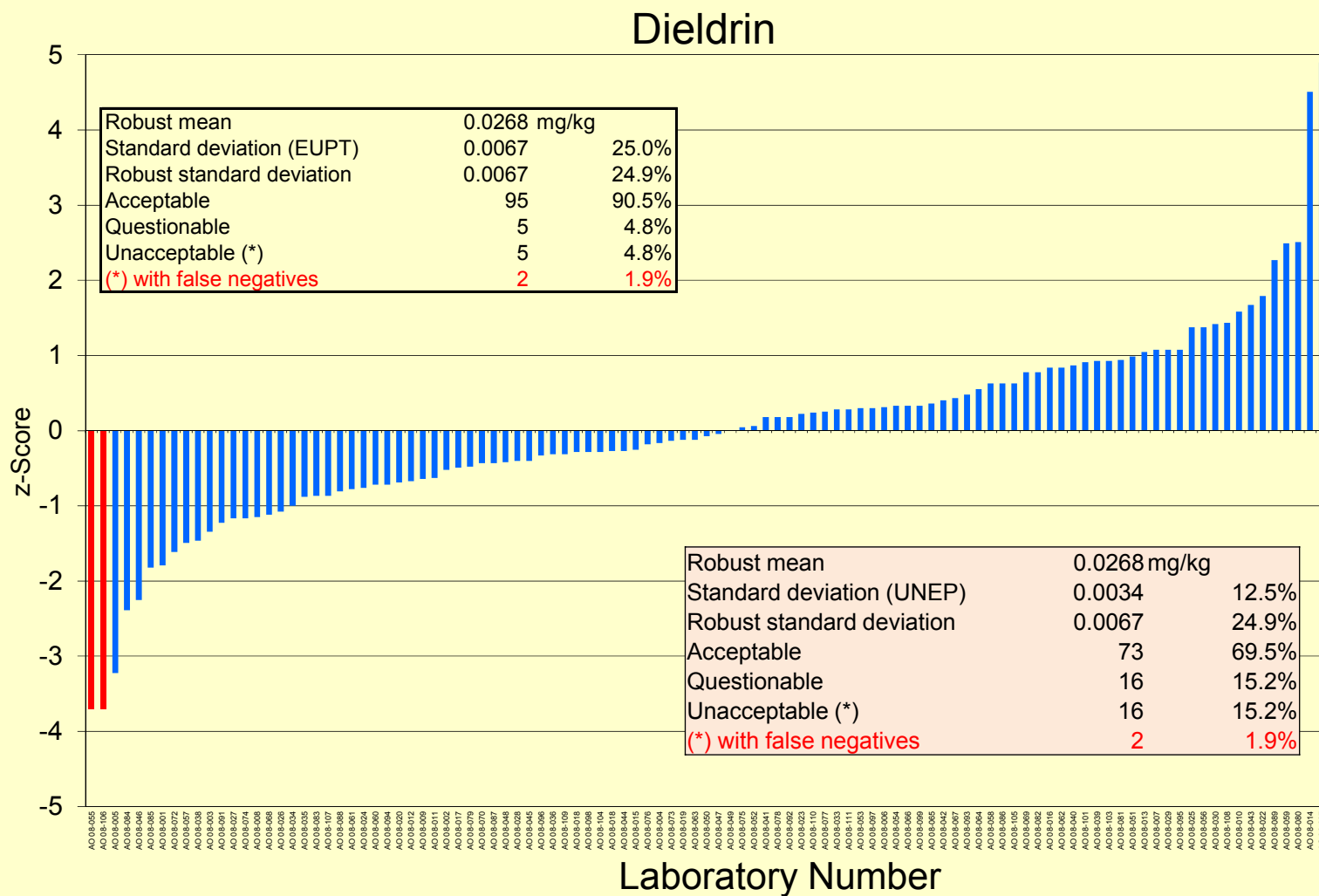
# EUPT-AO 09: Results (Mandatory Pesticides)



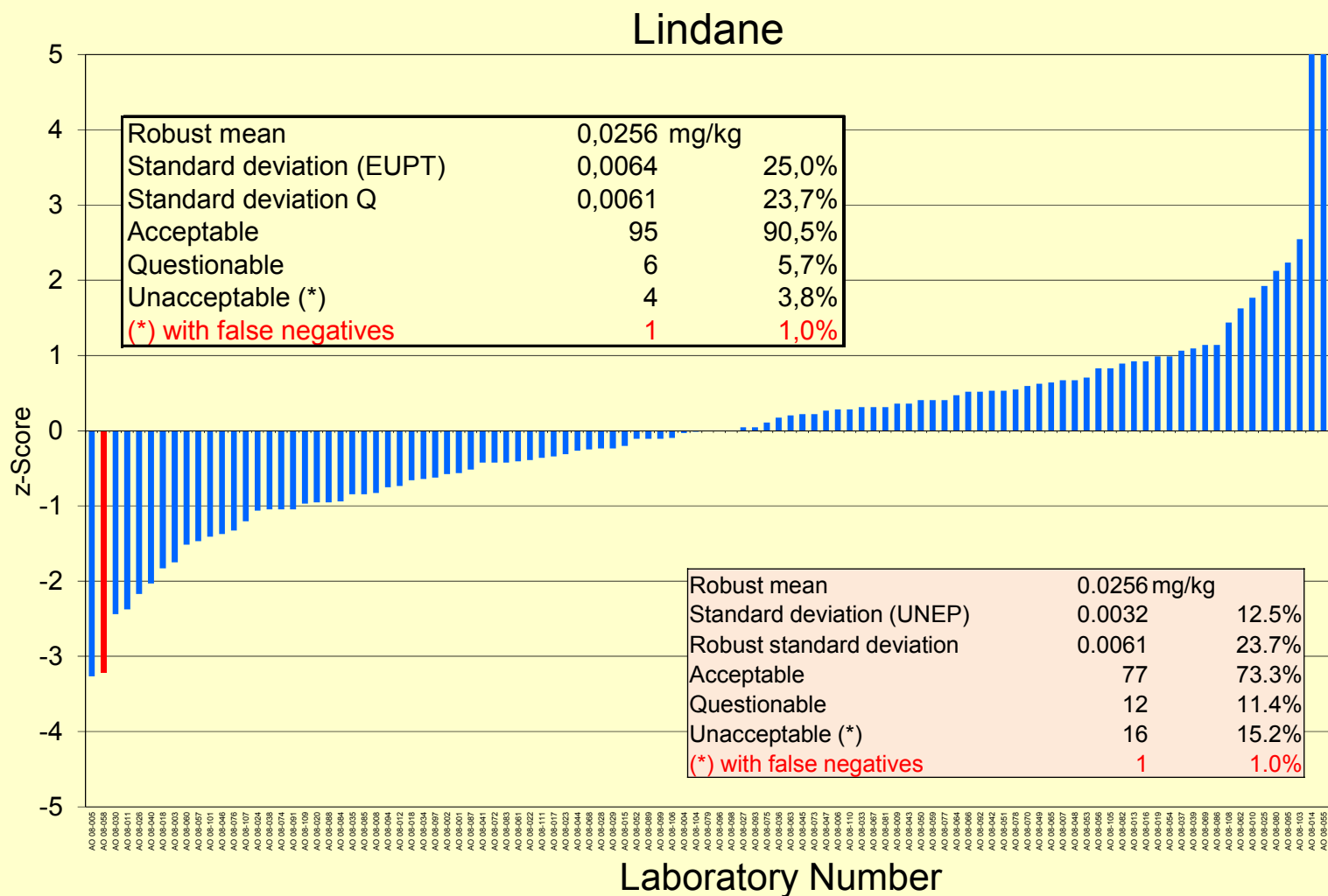
# EUPT-AO 09: Results (Mandatory Pesticides)



# EUPT-AO 09: Results (Mandatory Pesticides)



# EUPT-AO 09: Results (Mandatory Pesticides)



# Summary

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- EURLs offer one EUPT on a yearly basis, at least
- Number of participating laboratories increased (EUPT AO:  $\pm 110$ )
- Number of analytes increases (EUPT AO:  $\pm 85$ )
- Scope of laboratories increases
- Quality of results increases
  
- But, due to large workload of the laboratories -> number of errors during reporting increases!