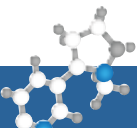


# EUPT-SRM18

## Pesticide Residues in Honey



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P. Schreiter / A.-K. Schäfer  
A. Barth / S. Fieberg / G. Cerchia



## Selection of Commodity & Pesticides to be spiked

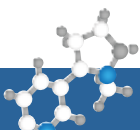
### COMMODITY & DATE Selection

**Decided in AdvG-Meeting in June 2022:**

**Commodity: HONEY** (→ Later decided to use different material for SRM18 and AO18)

**Timing: LAST in EUPT-season (shipment in May)**





## Registration for EUPT-SRM18:

17 March – 6 April, 2023

## Sample Shipment:

19 May, 2023

## Results Submission Period:

22\* May – 16 June, 2023 (\*Webtool Opening)

## Preliminary Report:

to be released by 14 July, 2023

|  <b>EURL-SRM</b>  EU Reference Laboratories for Residues of Pesticides<br>Single Residue Methods                                                                                        |                                                 |
|---------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|-------------------------------------------------|
| <b>CALENDAR for the EUPT – SRM18</b><br><b>Matrix: Honey</b><br><small>(released on 25/02/2023)</small>                                                                                                                                                                                                                                                       |                                                 |
| Activity                                                                                                                                                                                                                                                                                                                                                      | Dates                                           |
| <b>Announcement of the EUPT-SRM18</b><br><small>opening of the EUPT-SRM18 Website with links to all relevant documents</small>                                                                                                                                                                                                                                | 18 February 2023                                |
| <b>Registration Period for EUPT-SRM18</b><br><small>via "EUPT-Registration Website"</small><br><small>Laboratories classified as "SRM18/17" to participate in the EUPT-SRM18 MUST enter the EUPT-Registration Website and either register OR give explanations for non-participation</small>                                                                  | 17 March – 6 April 2023, 23:59 h CEST*          |
| <b>Dispatch of EUPT-SRM18-Specific Protocol</b>                                                                                                                                                                                                                                                                                                               | by 5 May 2023                                   |
| <b>Shipment of EUPT-SRM18 Test Item</b>                                                                                                                                                                                                                                                                                                                       | 19 May 2023                                     |
| <b>Confirming Sample Receipt and Acceptance</b><br><small>via "EUPT-SRM18 Result Submission Webtool"</small>                                                                                                                                                                                                                                                  | From 22 May 2023 onwards                        |
| <b>Submission of Results (Pesticide scope, Results, Method Info)</b><br><small>via "EUPT-SRM18 Result Submission Webtool"</small>                                                                                                                                                                                                                             | 22 May – 16 June:<br>11 h (11 a.m.) CEST        |
| <b>Submission of Additional/Missing Information</b><br><small>e.g. Method Info on tentatively false negative results<br/>via "EUPT-SRM18 Result Submission Webtool"</small>                                                                                                                                                                                   | 18 – 22 June 2023                               |
| <b>Dispatch of Preliminary Report</b><br><small>containing results as well as preliminary assigned values and 2 scores only</small>                                                                                                                                                                                                                           | Within 5 weeks<br>after the submission deadline |
| <b>Collection of reasons for underperformance and missing information on methods</b>                                                                                                                                                                                                                                                                          | July & August 2023                              |
| <b>Dispatch of Final Report</b>                                                                                                                                                                                                                                                                                                                               | Dec. 2023                                       |
| <small>*Please make sure to register for the EUPT from 17 March to the deadline 06 April 2023 via "EUPT-Registration Website". Any wish for registration after this deadline or not using the registration website cannot be considered.</small>                                                                                                              |                                                 |
| <b>REMARK:</b><br>Please note that the dates given above may be subject to minor changes. In case of changes importantly affecting the participants, the participants will be informed via e-mail. However, please still check periodically our website for possible updates in case the email does not get through to you.<br>Contact: eurl-srm@ec.europa.eu |                                                 |
| The EUPT-SRM Team                                                                                                                                                                                                                                                                                                                                             |                                                 |

## Selection of Commodity & Pesticides to be spiked

### ANALYTE Selection - Factors considered for INCLUSION IN TARGET LIST...

#### a) Listing in EU monitoring documents:

- MACP-Regulation:

- **Mandatory:** Chlormequat, Mepiquat, Nicotine (*MACP 2024 ff*), Fosetyl, Phosphonic acid, Haloxyfop, Fluazifop, 2,4-D, Glyphosate
- **Extra:** Bromide (*MRL too low for many labs*), Copper (*No MRL, also wish of COM*)

- Monitoring Working Document :

- **Mandatory (from Honey List in WD) :** BAC, DDAC, Chlorate, Matrine, Oxymatrine, Amitraz (dilemma: parent, metab., full RD?), Fluazifop, Glyphosate, 2,4-D,  
**Optional (from Honey List in WD):** Chloridazon desphenyl
- **Optional (other entries):** AMPA, NAGly, MCPA, Quizalofop, Trinexapac

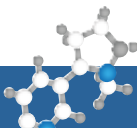
#### b) COM-wish: Copper

#### c) Relevance to matrix (**HONEY**) – Intermediate Results of own Pilot Monitoring

#### d) Suggestions/Voting by EUPT-Scientific Committee

#### e) Capacities and Capability of Participants (and EURL): Keep no. of methods moderate: Amitraz after hydrolysis not analyzed by many labs (previous survey)





## TARGET PESTICIDES List:

for the EUPT-SRM18 (2023), Honey  
(updated on 08.05.2023)

Analytes are sorted alphabetically in the same order as in the Webtool.

For grouping into **Mandatory** and **Optional** (incl. **Extra**): See sheet "Grouped into Mandatory-Optional"

Only **mandatory** (=compulsory) analytes will be considered in the scope-based classification, **optional** (=voluntary) and **extra** analytes not.  
Please also refer to the EUPT General Protocol.

a new category "Extra (E)" is introduced:  
Extra analyte, not for lab evaluation (07.05.2023)

| M: Mandatory<br>O: Optional<br>E: Extra | Analytes                               | Notes                                                                       | Listed in                | MRRL<br>[mg/kg] |
|-----------------------------------------|----------------------------------------|-----------------------------------------------------------------------------|--------------------------|-----------------|
| M                                       | 2,4-D (free acid)                      | No hydrolysis required                                                      | WD-Honey and MACP-Reg.   | 0.01            |
| O                                       | AMPA                                   |                                                                             | WD-Annex II (future RD)  | 0.01            |
| M                                       | BAC-C12 chloride                       | Benzyltrimethylammonium-chloride                                            | WD-Honey                 | 0.01            |
| M                                       | BAC-C14 chloride                       | Benzyltrimethyltetradecylammonium-chloride                                  | WD-Honey                 | 0.01            |
| E                                       | Bromide (anion)                        | Analysis of natural content for information only (no z-score calculations)  | MACP-Reg.                | none            |
| M                                       | Chlorate (anion)                       |                                                                             | WD-Honey                 | 0.01            |
| O                                       | Chloridazon desphenyl                  |                                                                             | WD-Honey                 | 0.02            |
| M                                       | Chlormequat chloride                   | Expressed as chloride salt                                                  | MACP-Reg.                | 0.01            |
| E                                       | Copper                                 | Analysis of natural content for information only (no z-score calculations)  | MACP-Reg.                | none            |
| M                                       | DDAC-C10 chloride                      | Didecyltrimethylammonium-chloride                                           | WD-Honey                 | 0.01            |
| <del>M</del>                            | <del>DMF</del>                         | <del>N-(2,4-Dimethylphenyl)-formamide (Amitraz-metabolite)</del>            | <del>WD-Honey</del>      | <del>0.01</del> |
| <del>M</del>                            | <del>DMPP</del>                        | <del>N-(2,4-Dimethylphenyl)-N'-methylformamidine (Amitraz-metabolite)</del> | <del>WD-Honey</del>      | <del>0.01</del> |
| M                                       | Fluazifop (free acid, sum of isomers)  | No hydrolysis required                                                      | WD-Honey and MACP-Reg.   | 0.01            |
| M                                       | Fosetyl                                |                                                                             | MACP-Reg.                | 0.01            |
| M                                       | Glyphosate                             |                                                                             | WD-Honey and MACP-Reg.   | 0.01            |
| M                                       | Haloxifop (free acid, sum of isomers)  | No hydrolysis required                                                      | MACP-Reg.                | 0.01            |
| M                                       | Matrine                                |                                                                             | WD-Honey                 | 0.01            |
| O                                       | MCPA (free acid)                       | No hydrolysis required                                                      | WD-Annex III             | 0.01            |
| M                                       | Mepiquat chloride                      | Expressed as chloride salt                                                  | MACP-Reg.                | 0.01            |
| O                                       | N-Acetyl-glyphosate                    |                                                                             | WD-Annex II (future RD)  | 0.01            |
| M                                       | Nicotine                               |                                                                             | MACP-Reg. (2024 onwards) | 0.01            |
| M                                       | Oxymatrine                             |                                                                             | WD-Honey                 | 0.01            |
| O                                       | Perchlorate                            |                                                                             | Contaminant              | 0.01            |
| M                                       | Phosphonic acid                        |                                                                             | MACP-Reg.                | 0.02            |
| O                                       | Quizalofop (free acid, sum of isomers) | No hydrolysis required                                                      | WD-Annex III             | 0.01            |
| O                                       | Trinexapac (free acid)                 | No hydrolysis required                                                      | WD (4.1 and Annex II)    | 0.01            |

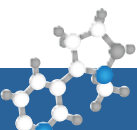
Changed to category "Extra" (07.05.2023)

New! (05.04.2023)

New on the TPL in the category "Extra"! (08.05.2023)

removed from the TPL (05.04.2023)

removed from the TPL (05.04.2023)



## OVERVIEW:

### COMPOUNDS APPLIED IN LAB AND INCURRED ONES

|                        | residues<br>incurred | compounds<br>spiked in the<br>lab | Compound form used<br>for spiking in the lab |
|------------------------|----------------------|-----------------------------------|----------------------------------------------|
| 2,4-D (free acid)      | -                    | ✓                                 | 2,4-D (free acid)                            |
| BAC-C14 Chloride       | -                    | ✓                                 | BAC-C14 Chloride                             |
| Chlorate (anion)       | -                    | ✓                                 | Sodium-Chlorate                              |
| DDAC-C10 chloride      | -                    | ✓                                 | DDAC-C10 chloride                            |
| Fluazifop (free acid)  | -                    | ✓                                 | Fluazifop (free acid)                        |
| Glyphosate             | -                    | ✓                                 | Glyphosate                                   |
| Matrine                | -                    | ✓                                 | Matrine                                      |
| Nicotine               | -                    | ✓                                 | Nicotine                                     |
| Oxymatrine             | -                    | ✓                                 | Oxymatrine                                   |
| Phosphonic acid        | -                    | ✓                                 | Phosphonic acid                              |
| Perchlorate (anion)    | -                    | ✓                                 | Sodium-Perchlorate                           |
| Trinexapac (free acid) | -                    | ✓                                 | Trinexapac (free acid)                       |
| Bromide (anion)        | traces               | -                                 | -                                            |
| Copper                 | traces               | -                                 | -                                            |

## Preparation of PT-Item



25 kg honey

Filling into Container  
and Spiking



Homogenization with  
Rapido<sup>®</sup> stirrer



Express-shipment  
to Participants

Preparation for Shipment



Bottling into Special Bucket with Tap



# Homogeneity TEST

## COMPULSORY Compounds

|                                                                        | 2,4-D                 | BAC-C14         | Chlorate        | DDAC-C10       | Fluazifop       |
|------------------------------------------------------------------------|-----------------------|-----------------|-----------------|----------------|-----------------|
| Sample                                                                 | Concentration [mg/kg] |                 |                 |                |                 |
| No. 001                                                                | 0.0523 / 0.0532       | 0.104 / 0.106   | 0.109 / 0.108   | 0.143 / 0.144  | 0.0627 / 0.0617 |
| No. 004                                                                | 0.0525 / 0.0542       | 0.105 / 0.106   | 0.106 / 0.108   | 0.144 / 0.145  | 0.0636 / 0.0635 |
| No. 020                                                                | 0.0541 / 0.0538       | 0.108 / 0.106   | 0.107 / 0.109   | 0.149 / 0.149  | 0.0631 / 0.0630 |
| No. 048                                                                | 0.0535 / 0.0539       | 0.108 / 0.106   | 0.107 / 0.110   | 0.142 / 0.145  | 0.0618 / 0.0637 |
| No. 067                                                                | 0.0530 / 0.0527       | 0.103 / 0.103   | 0.109 / 0.107   | 0.139 / 0.145  | 0.0625 / 0.0620 |
| No. 074                                                                | 0.0531 / 0.0534       | 0.106 / 0.104   | 0.106 / 0.108   | 0.150 / 0.145  | 0.0640 / 0.0614 |
| No. 89                                                                 | 0.0536 / 0.0526       | 0.106 / 0.106   | 0.107 / 0.109   | 0.145 / 0.143  | 0.0635 / 0.0610 |
| No. 099                                                                | 0.0539 / 0.0539       | 0.105 / 0.103   | 0.108 / 0.109   | 0.142 / 0.143  | 0.0627 / 0.0606 |
| No. 124                                                                | 0.0543 / 0.0540       | 0.106 / 0.105   | 0.110 / 0.108   | 0.146 / 0.145  | 0.0630 / 0.0620 |
| No. 140                                                                | 0.0529 / 0.0542       | 0.106 / 0.108   | 0.106 / 0.107   | 0.150 / 0.151  | 0.0624 / 0.0648 |
| Mean (AV)                                                              | 0.0535 (0.0523)       | 0.105 (0.119)   | 0.108 (0.102)   | 0.145 (0.149)  | 0.0627 (0.0598) |
| RSD (%)                                                                | 0.9                   | 1.3             | 0.8             | 1.9            | 1.0             |
| $S_{sam}^2 < (c) ?$                                                    | 2.1E-4 < 0.0040       | 0.0011 < 0.0079 | 2.5E-4 < 0.0081 | 0.0023 < 0.011 | 0 < 0.0047      |
| Homogeneity test                                                       | passed                | passed          | passed          | passed         | passed          |
| $S_{sam}^2$ : between-samples STD<br>(c): Check Value (0.3* sigma-PT): |                       |                 |                 |                |                 |



# Homogeneity TEST

## COMPULSORY Compounds

|                                                                        | Glyphosate            | Matrine         | Nicotine        | Oxymatrine      | Phosphonic acid |
|------------------------------------------------------------------------|-----------------------|-----------------|-----------------|-----------------|-----------------|
| Sample                                                                 | Concentration [mg/kg] |                 |                 |                 |                 |
| No. 011                                                                | 0.104 / 0.106         | 0.0934 / 0.0947 | 0.0883 / 0.0910 | 0.0733 / 0.0769 | 0.211 / 0.207   |
| No. 034                                                                | 0.104 / 0.102         | 0.0887 / 0.0942 | 0.0917 / 0.0957 | 0.0725 / 0.0705 | 0.209 / 0.212   |
| No. 050                                                                | 0.106 / 0.101         | 0.0886 / 0.0909 | 0.0923 / 0.0922 | 0.0727 / 0.0700 | 0.196 / 0.191   |
| No. 073                                                                | 0.105 / 0.106         | 0.0912 / 0.0894 | 0.0912 / 0.0921 | 0.0719 / 0.0697 | 0.200 / 0.210   |
| No. 096                                                                | 0.103 / 0.104         | 0.0885 / 0.0919 | 0.0932 / 0.0917 | 0.0767 / 0.0723 | 0.207 / 0.199   |
| No. 112                                                                | 0.102 / 0.108         | 0.0907 / 0.0903 | 0.0922 / 0.0926 | 0.0702 / 0.0719 | 0.194 / 0.195   |
| No. 143                                                                | 0.103 / 0.107         | 0.0905 / 0.0920 | 0.0899 / 0.0914 | 0.0712 / 0.0697 | 0.211 / 0.201   |
| No. 156                                                                | 0.106 / 0.104         | 0.0916 / 0.0936 | 0.0921 / 0.0902 | 0.0777 / 0.0738 | 0.203 / 0.206   |
| No. 183                                                                | 0.104 / 0.109         | 0.0922 / 0.0908 | 0.0966 / 0.0933 | 0.0671 / 0.0724 | 0.208 / 0.193   |
| No. 200                                                                | 0.103 / 0.099         | 0.0861 / 0.0914 | 0.0938 / 0.0936 | 0.0744 / 0.0729 | 0.207 / 0.211   |
| Mean (AV)                                                              | 0.104 (0.102)         | 0.0910 (0.0873) | 0.0923 (0.0877) | 0.0724 (0.0681) | 0.204 (0.202)   |
| RSD (%)                                                                | 1.5                   | 1.7             | 1.7             | 3.0             | 2.8             |
| $S_{sam}^2 < (c) ?$                                                    | 0 < 0.0078            | 1.9E-4 < 0.0068 | 3.2E-5 < 0.007  | 0.0015 < 0.0054 | 0.0043 < 0.015  |
| Homogeneity test                                                       | passed                | passed          | passed          | passed          | passed          |
| $S_{sam}^2$ : between-samples STD<br>(c): Check Value (0.3* sigma-PT): |                       |                 |                 |                 |                 |

# Homogeneity TEST

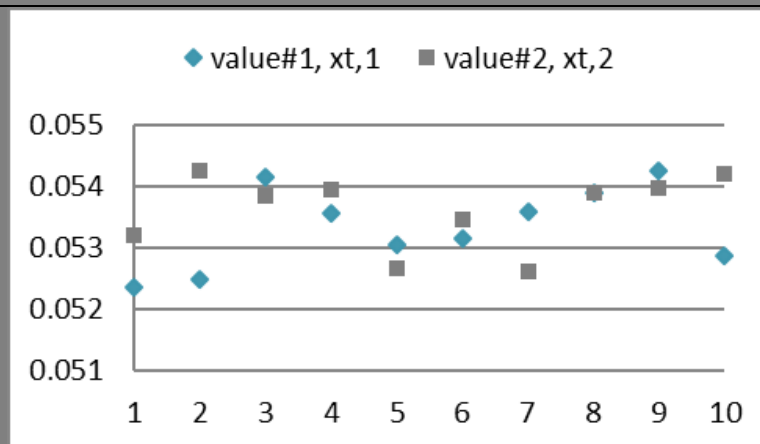
## OPTIONAL Compounds

|                                                                        | Perchlorate           | Trinexapac      |
|------------------------------------------------------------------------|-----------------------|-----------------|
| Sample                                                                 | Concentration [mg/kg] |                 |
| No. 001                                                                | 0.0631 / 0.0587       | 0.108 / 0.101   |
| No. 004                                                                | 0.0605 / 0.0603       | 0.128 / 0.119   |
| No. 020                                                                | 0.0613 / 0.0607       | 0.115 / 0.097   |
| No. 048                                                                | 0.0618 / 0.0603       | 0.111 / 0.103   |
| No. 067                                                                | 0.0627 / 0.0628       | 0.107 / 0.101   |
| No. 074                                                                | 0.0573 / 0.0600       | 0.095 / 0.107   |
| No. 89                                                                 | 0.0621 / 0.0618       | 0.113 / 0.106   |
| No. 099                                                                | 0.0619 / 0.0613       | 0.118 / 0.098   |
| No. 124                                                                | 0.0621 / 0.0602       | 0.105 / 0.085   |
| No. 140                                                                | 0.0616 / 0.0612       | 0.106 / 0.099   |
| Mean (AV)                                                              | 0.0611 (0.0550)       | 0.106 (0.118)   |
| RSD (%)                                                                | 1.8                   | 7%              |
| $S_{sam}^2 < (c) ?$                                                    | 5.76E-4 < 0.0046      | 0.0040 < 0.0080 |
| Homogeneity test                                                       | passed                | passed          |
| $S_{sam}^2$ : between-samples STD<br>(c): Check Value (0.3* sigma-PT): |                       |                 |

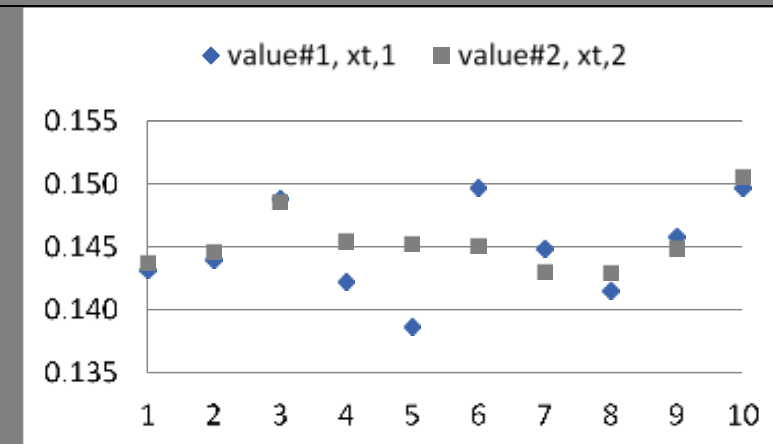
# Homogeneity TEST

**Distribution of results**  
**from 10 bottles**  
**(2 portions each),**  
**exemplary**

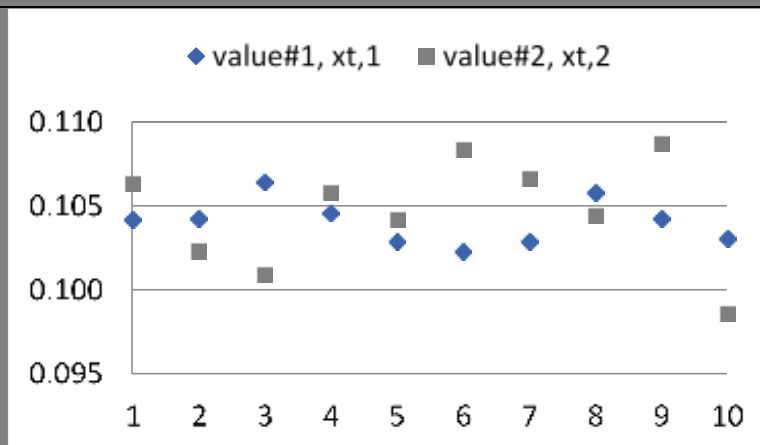
2,4-D



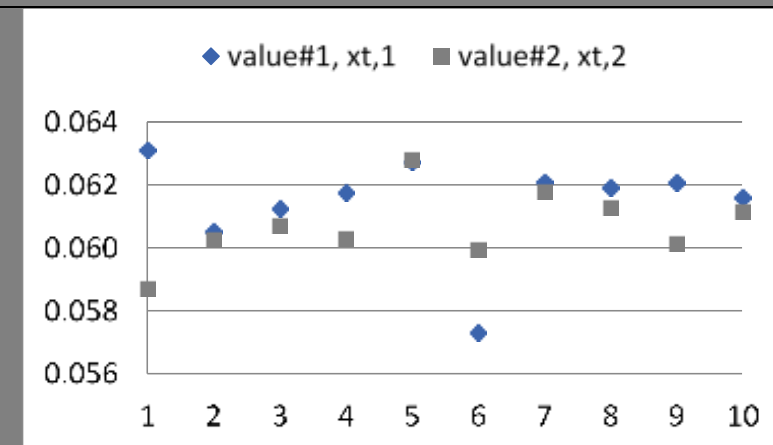
DDAC-C10



Glyphosate



Perchlorate



# Stability TEST

## COMPULSORY Compounds

|                                               | 2,4-D      |            |            | BAC-C14    |            |            | Chlorate   |            |            | DDAC-C10   |            |            | Fluazifop  |            |            |
|-----------------------------------------------|------------|------------|------------|------------|------------|------------|------------|------------|------------|------------|------------|------------|------------|------------|------------|
| Date of extraction                            | 20.04.2023 | 25.05.2023 | 15.06.2023 | 20.04.2023 | 25.05.2023 | 15.06.2023 | 27.04.2023 | 25.05.2023 | 15.06.2023 | 20.04.2023 | 25.05.2023 | 15.06.2023 | 20.04.2023 | 25.05.2023 | 15.06.2023 |
| No. 004                                       | 0.0534     | 0.0513     | 0.0552     | 0.105      | 0.105      | 0.0990     | 0.139      | 0.127      | 0.135      | 0.144      | 0.143      | 0.139      | 0.0636     | 0.0595     | 0.0616     |
| No. 067                                       | 0.0528     | 0.0513     | 0.0548     | 0.103      | 0.109      | 0.0984     | 0.136      | 0.129      | 0.134      | 0.142      | 0.141      | 0.141      | 0.0623     | 0.0594     | 0.0607     |
| No. 099                                       | 0.0539     | 0.0519     | 0.0548     | 0.104      | 0.106      | 0.0961     | 0.135      | 0.127      | 0.13       | 0.142      | 0.144      | 0.135      | 0.0616     | 0.0605     | 0.0616     |
| Mean [mg/kg]                                  | 0.0534     | 0.0515     | 0.0549     | 0.104      | 0.106      | 0.0978     | 0.136      | 0.128      | 0.133      | 0.143      | 0.143      | 0.138      | 0.0625     | 0.0598     | 0.0613     |
| % Diviation<br>(vs. 1 <sup>st</sup> Anylisis) | -          | -3.53%     | 2.95%      | -          | 2.28%      | -5.99%     | -          | -6.5%      | -2.8%      | -          | -0.01%     | -3.16%     | -          | -4.29%     | -1.92%     |
| Diff (mean)                                   | -          | 0.0019     | 0.0016     | -          | 0.0024     | 0.0062     | -          | 0.0088     | 0.0038     | -          | 2E-05      | 0.0045     | -          | 0.0027     | 0.0012     |
| 0.3*FFP-SD                                    |            | 0.0039     | 0.0039     |            | 0.0089     | 0.0089     |            | 0.012      | 0.012      |            | 0.011      | 0.011      |            | 0.0045     | 0.0045     |
| Stability test                                |            | passed     | passed     |            | passed     | passed     |            | passed     | passed     |            | passed     | passed     |            | passed     | passed     |

|                                               | Glyphosate |            |            | Matrine    |            |            | Nicotine   |            |            | Oxymatrine |            |            | Phosphonic acid |            |            |
|-----------------------------------------------|------------|------------|------------|------------|------------|------------|------------|------------|------------|------------|------------|------------|-----------------|------------|------------|
| Date of extraction                            | 27.04.2023 | 25.05.2023 | 15.06.2023 | 27.04.2023 | 25.05.2023 | 15.06.2023 | 27.04.2023 | 25.05.2023 | 15.06.2023 | 27.04.2023 | 25.05.2023 | 15.06.2023 | 27.04.2023      | 25.05.2023 | 15.06.2023 |
| No. 004                                       | 0.103      | 0.100      | 0.107      | 0.0915     | 0.0986     | 0.0926     | 0.0937     | 0.0938     | 0.0937     | 0.0713     | 0.0697     | 0.0727     | 0.210           |            | 0.205      |
| No. 067                                       | 0.104      | 0.0927     | 0.110      | 0.0902     | 0.0892     | 0.0888     | 0.0924     | 0.0896     | 0.0911     | 0.0711     | 0.0694     | 0.0710     | 0.203           |            | 0.207      |
| No. 099                                       | 0.105      | 0.0984     | 0.106      | 0.0926     | 0.0961     | 0.0940     | 0.0911     | 0.0879     | 0.0937     | 0.0697     | 0.0716     | 0.0700     | 0.204           |            | 0.212      |
| Mean [mg/kg]                                  | 0.104      | 0.0970     | 0.107      | 0.0914     | 0.0946     | 0.0918     | 0.0924     | 0.0905     | 0.0929     | 0.0707     | 0.0702     | 0.0712     | 0.206           |            | 0.208      |
| % Diviation<br>(vs. 1 <sup>st</sup> Anylisis) | -          | -6.75%     | 3.34%      | -          | 3.52%      | 0.42%      | -          | -2.13%     | 0.45%      | -          | -0.70%     | 0.76%      | -               |            | 0.89%      |
| Diff (mean)                                   | -          | 0.007      | 0.0035     | -          | 0.0032     | 0.0004     | -          | 0.0020     | 0.0004     | -          | 0.0005     | 0.0005     | -               |            | 0.0018     |
| 0.3*FFP-SD                                    |            | 0.0077     | 0.0077     |            | 0.0065     | 0.0065     |            | 0.0074     | 0.0074     |            | 0.0051     | 0.0051     |                 |            | 0.015      |
| Stability test                                |            | passed     | passed     |            | passed     | passed     |            | passed     | passed     |            | passed     | passed     |                 |            | passed     |

Instrumental  
issues, no  
measurement  
possible at that  
point

All stability test samples were extracted at the indicated day and measured alltogether directly.

# Stability TEST

## OPTIONAL Compounds

|                                               | Perchlorate |            |            | Trinexapac |            |            |
|-----------------------------------------------|-------------|------------|------------|------------|------------|------------|
| Date of extraction                            | 27.04.2023  | 25.05.2023 | 15.06.2023 | 20.04.2023 | 25.05.2023 | 15.06.2023 |
| No. 004                                       | 0.0604      | 0.0602     | 0.0603     | 0.1236     | 0.1115     | 0.1194     |
| No. 067                                       | 0.0628      | 0.0604     | 0.0615     | 0.1083     | 0.1208     | 0.1087     |
| No. 099                                       | 0.0616      | 0.0609     | 0.0613     | 0.1023     | 0.0999     | 0.1092     |
| Mean [mg/kg]                                  | 0.0616      | 0.0605     | 0.0610     | 0.1114     | 0.1107     | 0.1124     |
| % Diviation<br>(vs. 1 <sup>st</sup> Anaylsis) | -           | -1.81%     | -0.96%     | -          | -0.60%     | 0.89%      |
| Diff (mean)                                   | -           | 0.0011     | 0.0006     | -          | 0.0007     | 0.001      |
| 0.3*FFP-SD                                    |             | 0.0041     | 0.0041     |            | 0.0089     | 0.0089     |
| Stability test                                |             | passed     | passed     |            | passed     | passed     |

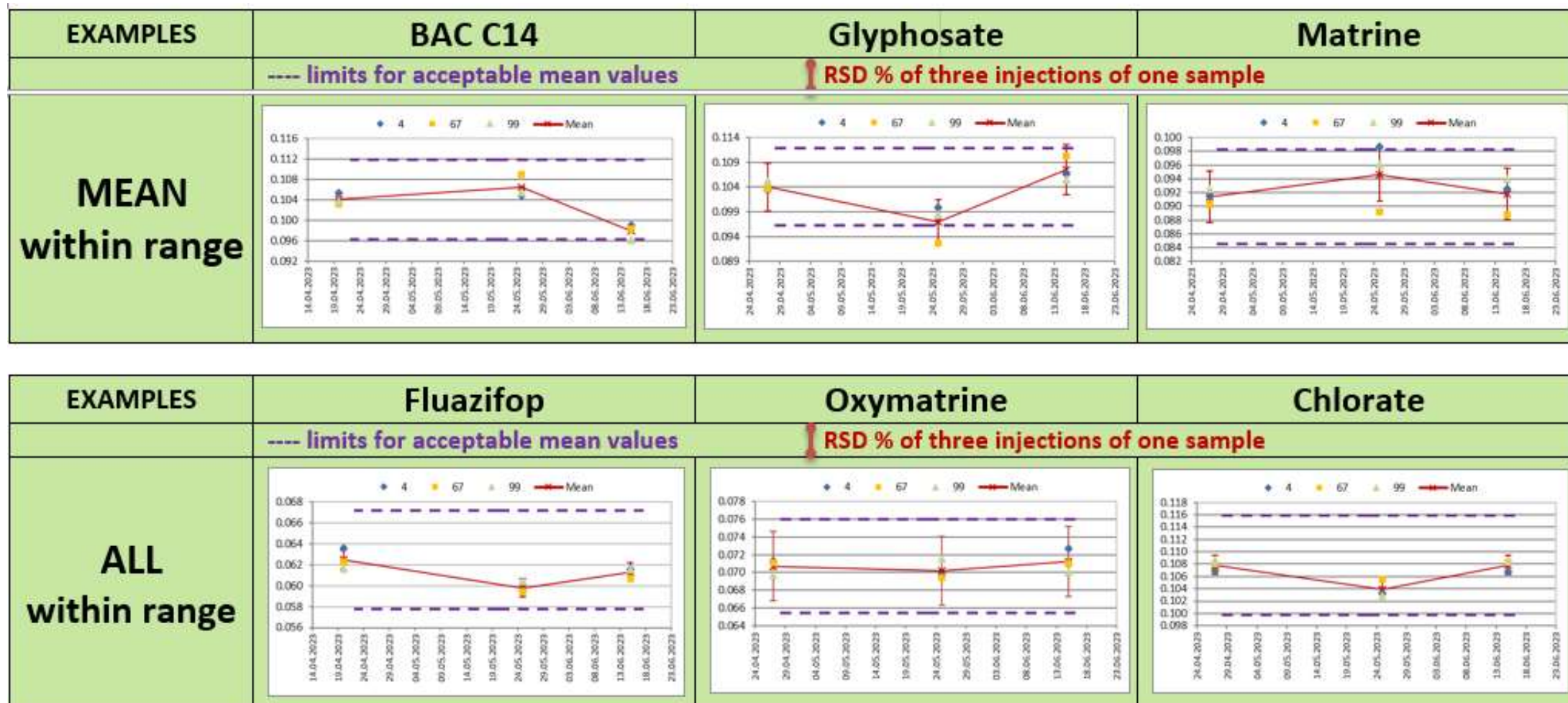
All stability test samples were extracted at the indicated day and measured alltogether at the end.

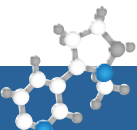


# Stability TEST

## Standard Deviation derived from quintuplicate injection of one sample

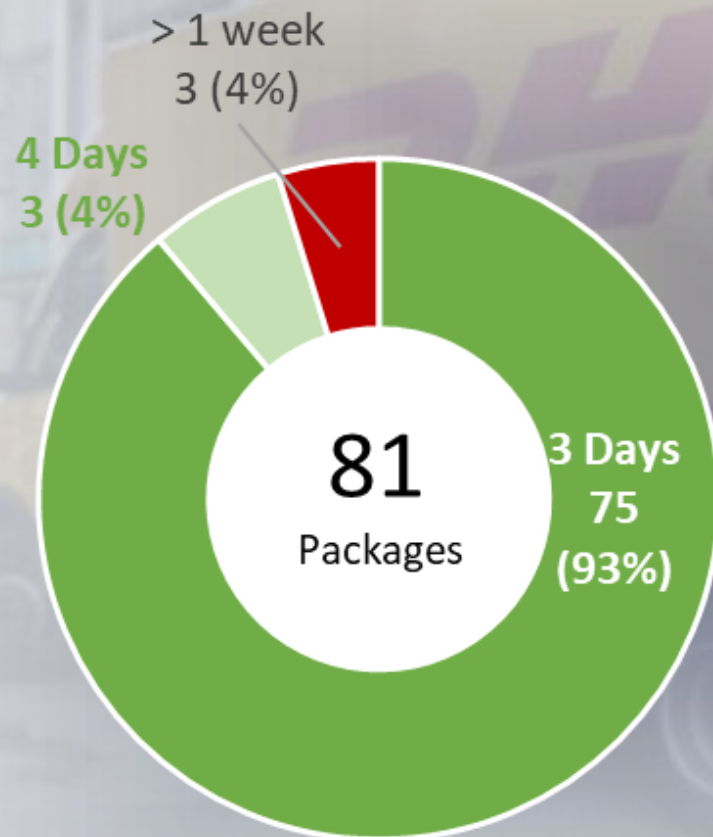
Mean values within range, individual values outside





## Stability during shipping

### Shipment Duration



### Dispatched on Friday without cooling

- Arrival on Mon. (3 days): 93 %
- Arrival on Tue. (4 days): 4 %
  - 3 EU (2x ES, 1x CY): remote Location
- All packages to EU and EFTA-labs arrived within 4 days.
- ≥ 1 Week: EU Candidate and 3rd Countries
  - VN (7 days): was kept frozen at local DHL
  - RS (13 days)
  - RS (> 1 month): (counted as non-participating)
- Test material for 1 participating labs in Latin America was handed over at LAPRW

|                                           |                                                                                     | Labs submitting results | Registered WITHOUT submitting results |
|-------------------------------------------|-------------------------------------------------------------------------------------|-------------------------|---------------------------------------|
| EU OfLs                                   |   | 69                      | 2* + 1 <sup>‡</sup> + 2               |
| EFTA OfLs                                 |   | 2                       | 0                                     |
| 3 <sup>rd</sup> Countries + EU Candidates |  | 4                       | 2                                     |
| SUM                                       |                                                                                     | 75                      | 7                                     |

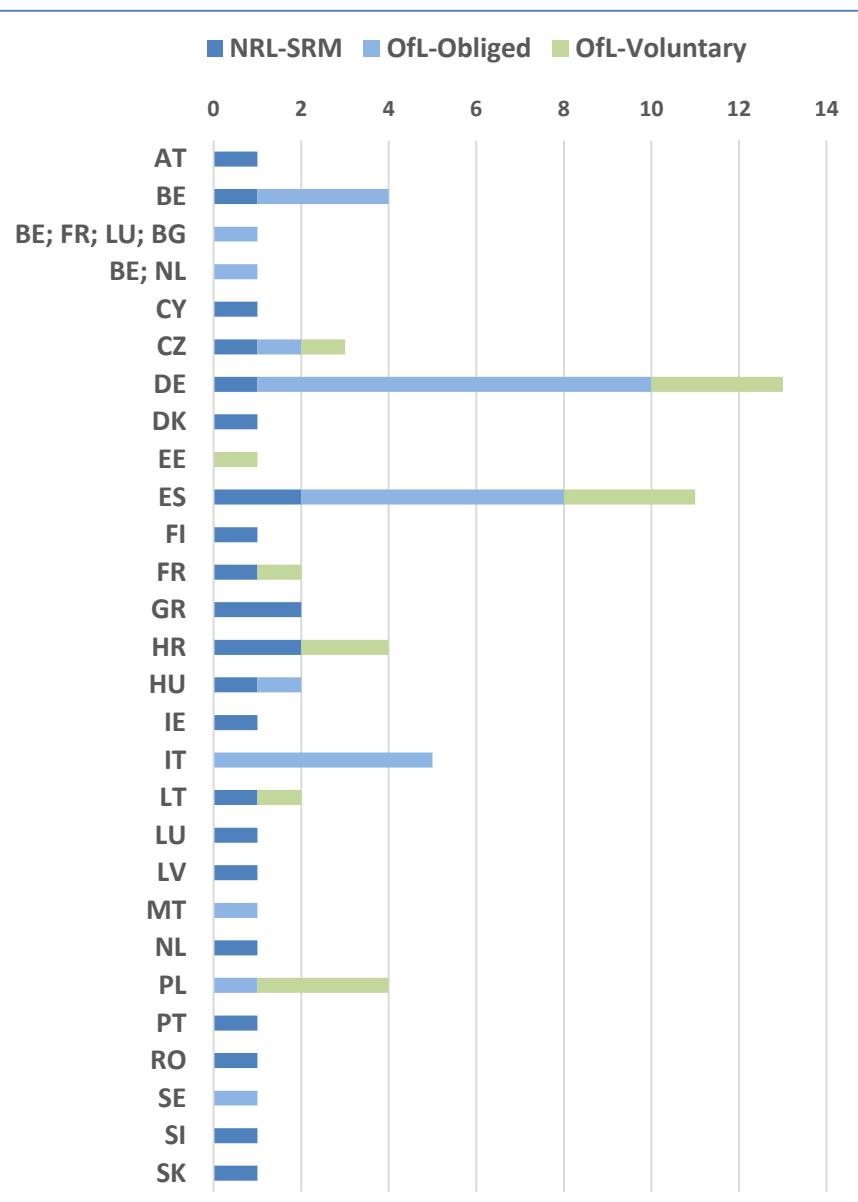
Non-participating EU OfLs:

2\* labs informed during SRM18 that their instrument was broken,

1<sup>‡</sup> lab informed after the PT that the standards were delivered too late to complete the PT,

2 labs have not yet stated their reasons for non-submission.

# Participating labs / countries



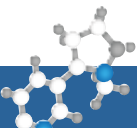
| EFTA-Countries | Participated in EUPT-SRM18 |
|----------------|----------------------------|
| CH             | 1                          |
| NO             | 1                          |
| EFTA-Total     | 2                          |

| EU Candidate & 3rd countries | Participated in EUPT-SRM18 |
|------------------------------|----------------------------|
| RS                           | 1                          |
| UY                           | 1                          |
| VN                           | 1                          |
| UK                           | 1                          |
| EU Cand. & 3rd C. Total      | 4                          |

7 labs having not submitting results not shown.

No NRL-SRM participant from 3 countries:

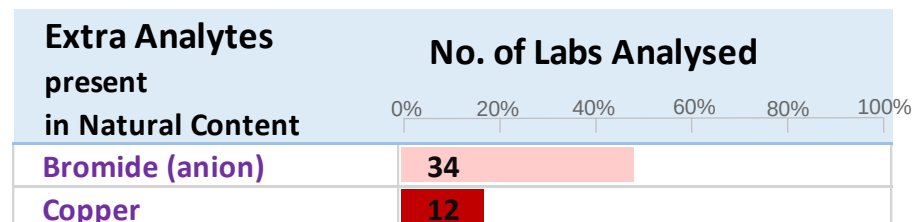
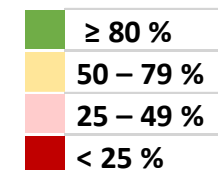
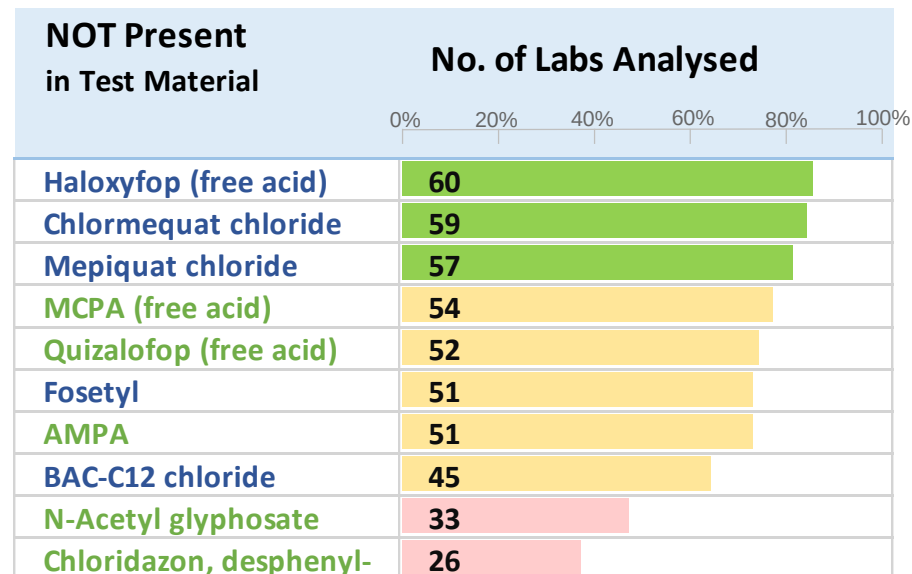
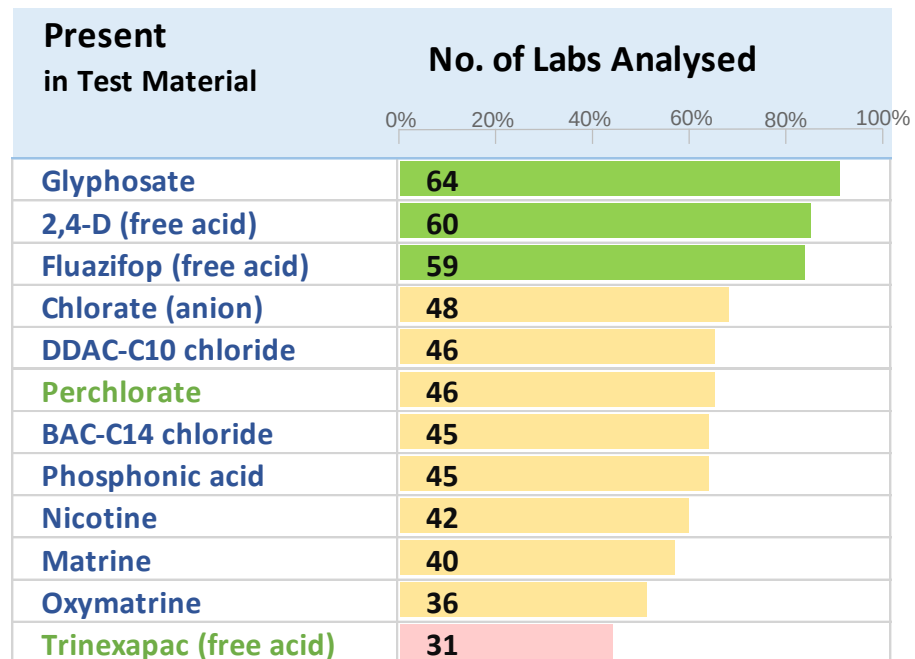
- EE (major lab Re-organisation)
- IT (Failed to submit results)
- SE (Only few compounds of TPL covered)
- MT (no lab, subcontracting NRL to ?)



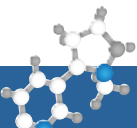
# Frequency of analysis

## COMPULSORY, OPTIONAL and Extra Compounds

Percentage based on 71 (69 EU + 2 EFTA) labs registered and submitting results



EU+EFTA



# COMPULSORY Compounds

EU+EFTA

## False Negatives (FN) 14

|            | Analyte               | MRRL<br>[mg/kg] | AV<br>[mg/kg] | No. of<br>FNs | Lab<br>Code | RL<br>[mg/kg] | Judgement     |
|------------|-----------------------|-----------------|---------------|---------------|-------------|---------------|---------------|
| COMPULSORY | BAC-C14 chloride      | 0.01            | 0.119         | 3             | 3           | 0.01          | FN            |
|            |                       |                 |               |               | 58          | 0.01          | FN            |
|            |                       |                 |               |               | 77          | 0.01          | FN            |
|            | Chlorate (anion)      | 0.01            | 0.102         | 3             | 18          | 0.01          | FN            |
|            |                       |                 |               |               | 45          | 0.01          | FN            |
|            |                       |                 |               |               | 69          | 0.01          | FN            |
|            | DDAC-C10 chloride     | 0.01            | 0.149         | 2             | 3           | 0.01          | FN            |
|            |                       |                 |               |               | 77          | 0.01          | FN            |
|            | Fluazifop (free acid) | 0.01            | 0.0598        | 1             | 58          | 0.01          | FN            |
|            | Glyphosate            | 0.01            | 0.102         | 1             | 45          | 0.01          | FN            |
|            | Matrine               | 0.01            | 0.0873        | 1             | 52          | 0.05          | FN            |
|            | Nicotine              | 0.01            | 0.0877        | 2             | 69          | 0.01          | FN            |
|            |                       |                 |               |               | 82          | 0.1           | FN* (RL > AV) |
|            | Phosphonic acid       | 0.03            | 0.202         | 1             | 45          | 0.03          | FN            |



# False negatives

## OPTIONAL Compounds

EU+EFTA

## False Negatives (FN) 14

| Analyte    |                        | MRRL<br>[mg/kg] | AV<br>[mg/kg] | No. of<br>FNs | Lab<br>Code | RL<br>[mg/kg] | Judgement |
|------------|------------------------|-----------------|---------------|---------------|-------------|---------------|-----------|
| COMPULSORY | Perchlorate            | 0.01            | 0.0550        | 4             | 11          | 0.01          | FN        |
|            |                        |                 |               |               | 45          | 0.01          | FN        |
|            |                        |                 |               |               | 64          | 0.01          | FN        |
|            |                        |                 |               |               | 69          | 0.01          | FN        |
|            | Trinexapac (free acid) | 0.01            | 0.118         | 10            | 3           | 0.01          | FN        |
|            |                        |                 |               |               | 5           | 0.01          | FN        |
|            |                        |                 |               |               | 16          | 0.01          | FN        |
|            |                        |                 |               |               | 26          | 0.01          | FN        |
|            |                        |                 |               |               | 45          | 0.01          | FN        |
|            |                        |                 |               |               | 57          | 0.01          | FN        |
|            |                        |                 |               |               | 61          | 0.01          | FN        |
|            |                        |                 |               |               | 69          | 0.01          | FN        |
|            |                        |                 |               |               | 71          | 0.01          | FN        |
|            |                        |                 |               |               | 79          | 0.01          | FN        |

5x „Wrong Standard used (Trinexapac-ethyl)  
 1x „Lack of Experience“  
 1x „Transcription Error“  
 3x No answer yet

# False Negatives by lab

| Lab-Co | Trinexapac (free acid) | Fluazifop (free acid) | BAC-C14 chloride | DDAC-C10 chloride | Chlorate (anion) | Perchlorate | Phosphonic acid | Glyphosate | Matrine | Oxymatrine | Nicotine | Overall |
|--------|------------------------|-----------------------|------------------|-------------------|------------------|-------------|-----------------|------------|---------|------------|----------|---------|
| 45*    | 1                      |                       |                  |                   | 1                | 1           | 1               | 1          |         |            |          | 5       |
| 69     | 1                      |                       |                  |                   | 1                | 1           |                 |            |         |            | 1        | 4       |
| 70     |                        |                       |                  |                   |                  |             |                 | 1          | 1       | 1          |          | 3       |
| 3      | 1                      |                       | 1                | 1                 |                  |             |                 |            |         |            |          | 3       |
| 77     |                        |                       | 1                | 1                 |                  |             |                 |            |         |            |          | 2       |
| 58     |                        | 1                     | 1                |                   |                  |             |                 |            |         |            |          | 2       |
| 71     | 1                      |                       |                  |                   |                  |             |                 |            |         |            |          | 1       |
| 82*    |                        |                       |                  |                   |                  |             |                 |            |         |            | 1        | 1       |
| 79     | 1                      |                       |                  |                   |                  |             |                 |            |         |            |          | 1       |
| 26     | 1                      |                       |                  |                   |                  |             |                 |            |         |            |          | 1       |
| 5      | 1                      |                       |                  |                   |                  |             |                 |            |         |            |          | 1       |
| 52     |                        |                       |                  |                   |                  |             |                 |            | 1       |            |          | 1       |
| 16     | 1                      |                       |                  |                   |                  |             |                 |            |         |            |          | 1       |
| 11*    |                        |                       |                  |                   |                  | 1           |                 |            |         |            |          | 1       |
| 18     |                        |                       |                  |                   | 1                |             |                 |            |         |            |          | 1       |
| 57*    | 1                      |                       |                  |                   |                  |             |                 |            |         |            |          | 1       |
| 61     | 1                      |                       |                  |                   |                  |             |                 |            |         |            |          | 1       |
| 64     |                        |                       |                  |                   |                  | 1           |                 |            |         |            |          | 1       |

Extra Compounds,  
Present (natural content)

EU+EFTA

| Analyte        | MRRL [mg/kg] | not detected LabCode | RL [mg/kg] |
|----------------|--------------|----------------------|------------|
| Extra Analytes | none         | 5                    | 0.5        |
|                |              | 11                   | 0.5        |
|                |              | 14                   | 1          |
|                |              | 15                   | 0.5        |
|                |              | 17                   | 0.5        |
|                |              | 26                   | 5          |
|                |              | 32                   | 2          |
|                |              | 33                   | 0.5        |
|                |              | 37                   | 1          |
|                |              | 38                   | 0.5        |
|                |              | 41                   | 0.2        |
|                |              | 42                   | 0.5        |
|                |              | 44                   | 0.5        |
|                |              | 45                   | 0.5        |
|                |              | 48                   | 1          |
|                |              | 50                   | 2.5        |
|                |              | 51                   | 0.5        |
|                |              | 52                   | 2.5        |
|                |              | 54                   | 0.5        |
|                |              | 55                   | 0.5        |
|                |              | 57                   | 0.5        |
|                |              | 62                   | 0.5        |
|                |              | 63                   | 0.5        |
|                |              | 66                   | 0.5        |
|                |              | 67                   | 0.05       |
|                |              | 73                   | 0.5        |
|                |              | 74                   | 1          |
|                |              | 78                   | 0.5        |
| Copper         | none         | 17                   | 0.5        |
|                |              | 74                   | 1          |

Copper:

2x FNs

10 numerical results (median 0.048 mg/kg)

No MRL established

Bromide:

28x “FNs”

6 numerical results (median 0.09 mg/kg)

MRL: 0.05\* (should be higher)

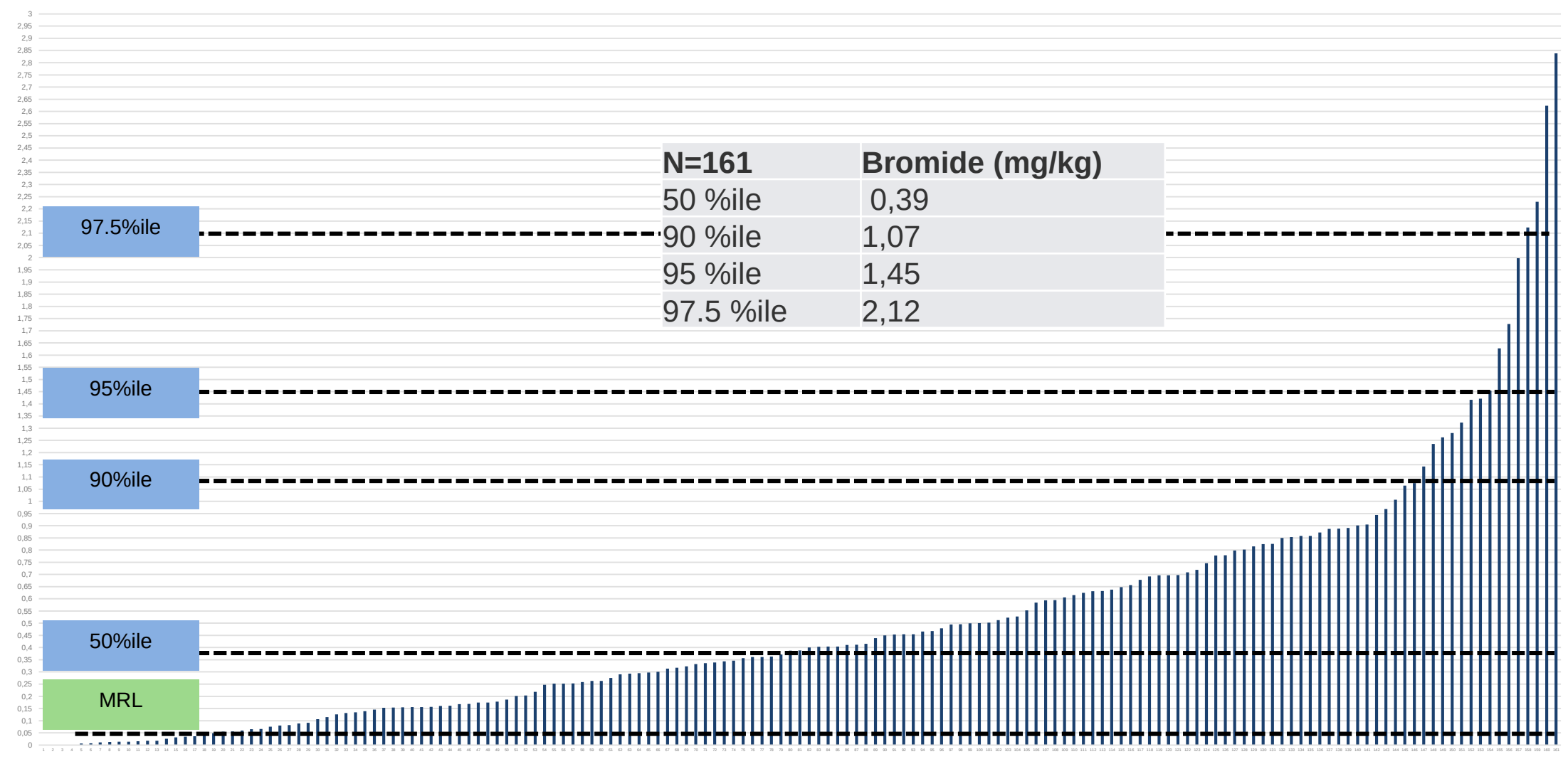
(if based on OfL-LOQs should be e.g. 0.5\* mg/kg)

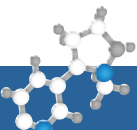
(if based on real levels should be at 1.5 or 2\* mg/kg)

| RLs (mg/kg) | No Labs |
|-------------|---------|
| 0,05        | 1       |
| 0,2         | 1       |
| 0,5         | 18      |
| 1           | 4       |
| 2           | 1       |
| 2,5         | 2       |
| 5           | 1       |
| SUM         | 28      |

Statistical establishment of AVs for Bromide-ion and Cu not possible.

Bromide Levels in Honey

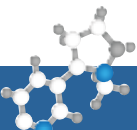




EU+EFTA

# No False Positive Results in the EUPT-SRM18!





# RESULTS OVERVIEW

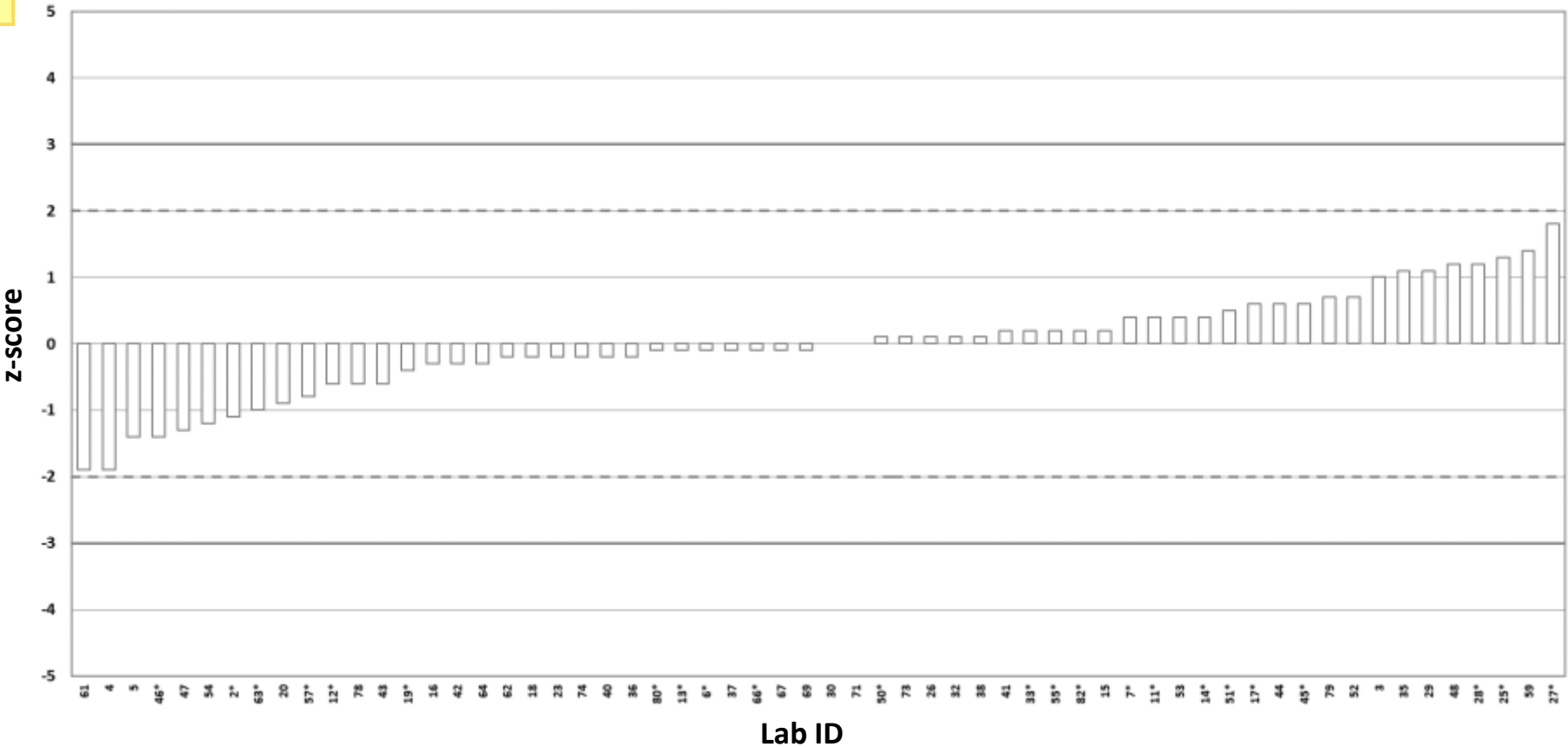
## COMPULSORY Compounds



2,4-D

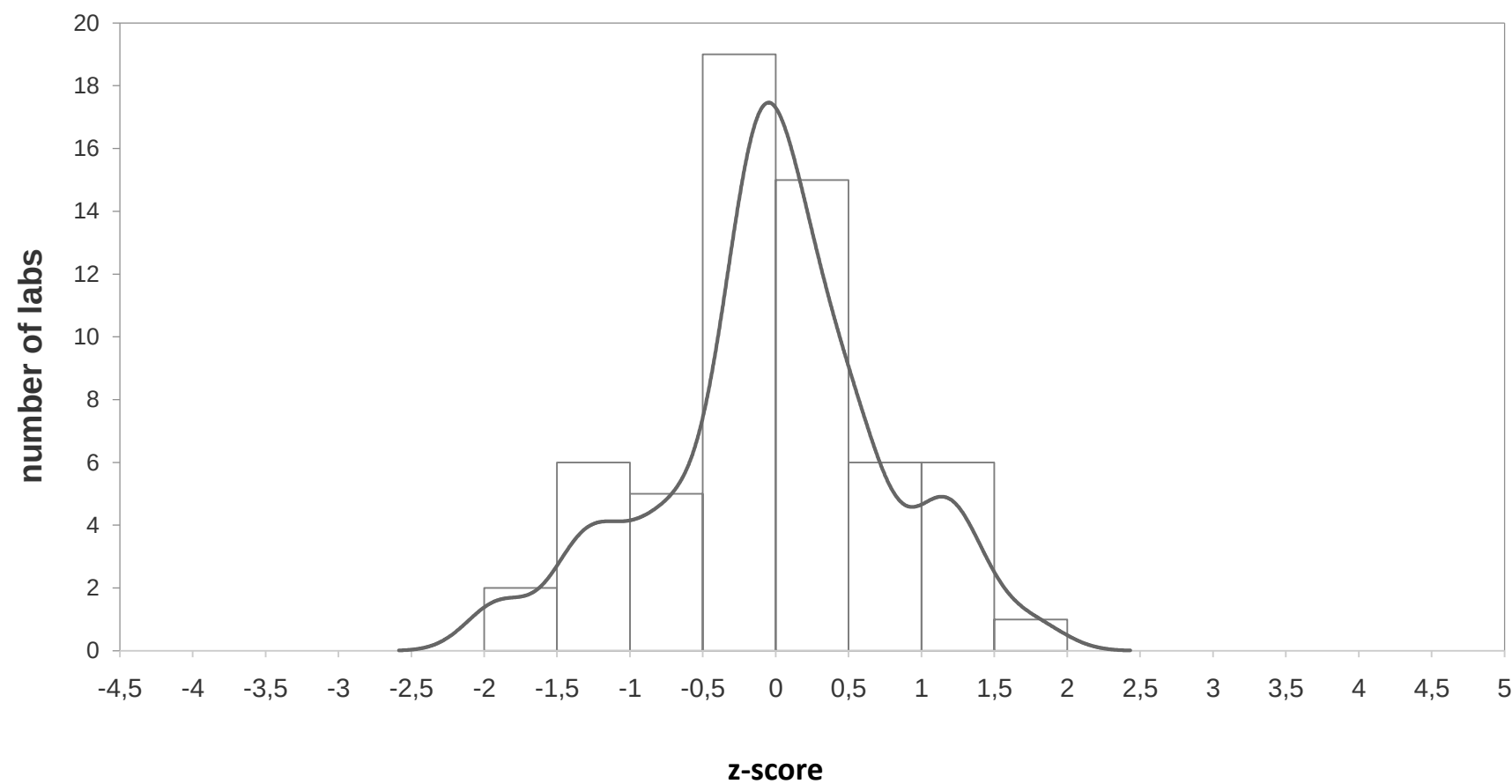
|            |                |
|------------|----------------|
| Results    | 60             |
| False Neg. | 0              |
| AV         | 0.0523 [mg/kg] |
| CV*        | 18.9%          |
| MRRL       | 0.01 [mg/kg]   |
| UAV-test   | passed         |

|                                                                                     |                            |              |    |        |
|-------------------------------------------------------------------------------------|----------------------------|--------------|----|--------|
|  | $ z\text{-score}  \leq 2$  | Acceptable   | 60 | (100%) |
|  | $2 <  z\text{-score}  < 3$ | Questionable | 0  | (0%)   |
|  | $ z\text{-score}  \geq 3$  | Unacceptable | 0  | (0%)   |
|  | False Negative             |              | 0  | (0%)   |



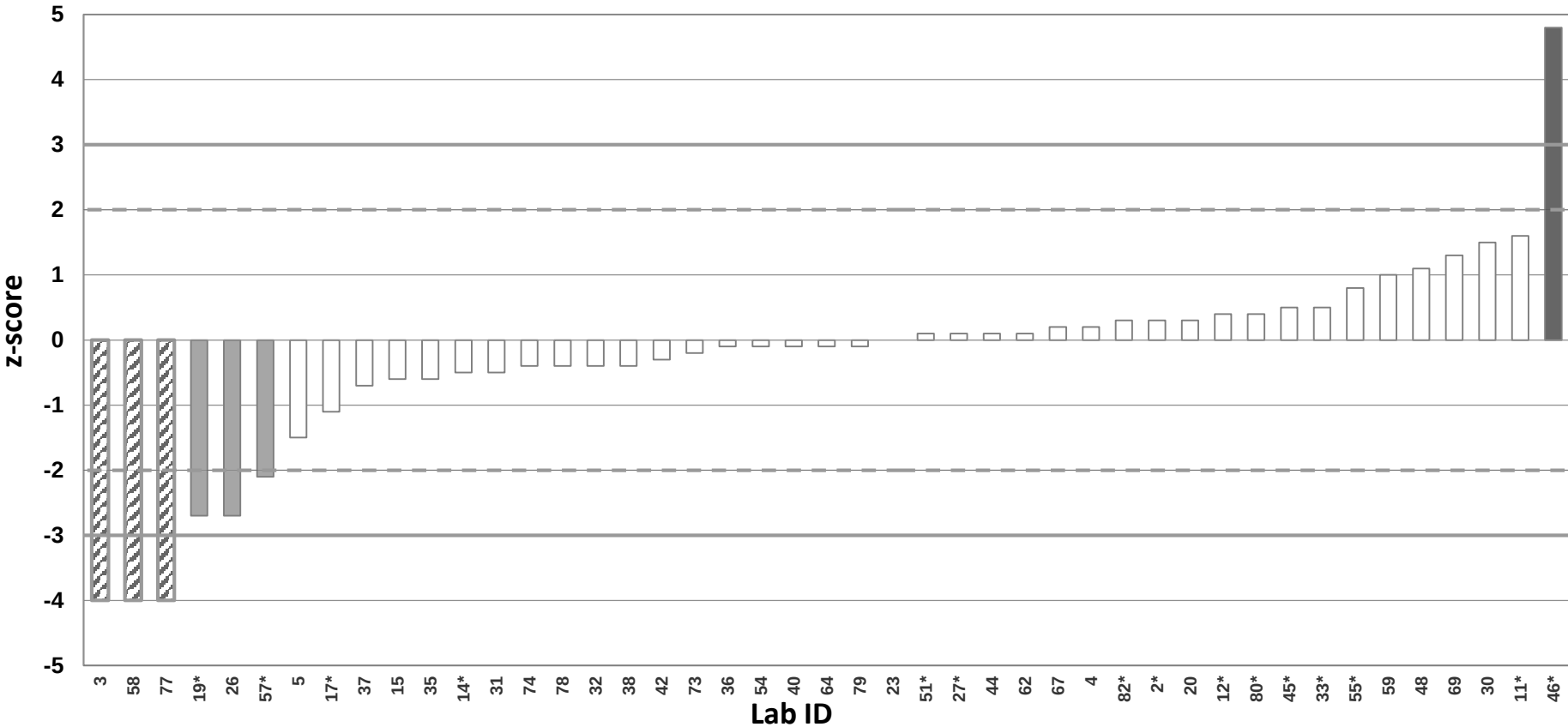
## 2,4-D

|            |                |
|------------|----------------|
| Results    | 60             |
| False Neg. | 0              |
| AV         | 0.0523 [mg/kg] |
| CV*        | 18.9%          |
| MRRL       | 0.01 [mg/kg]   |
| UAV-test   | passed         |



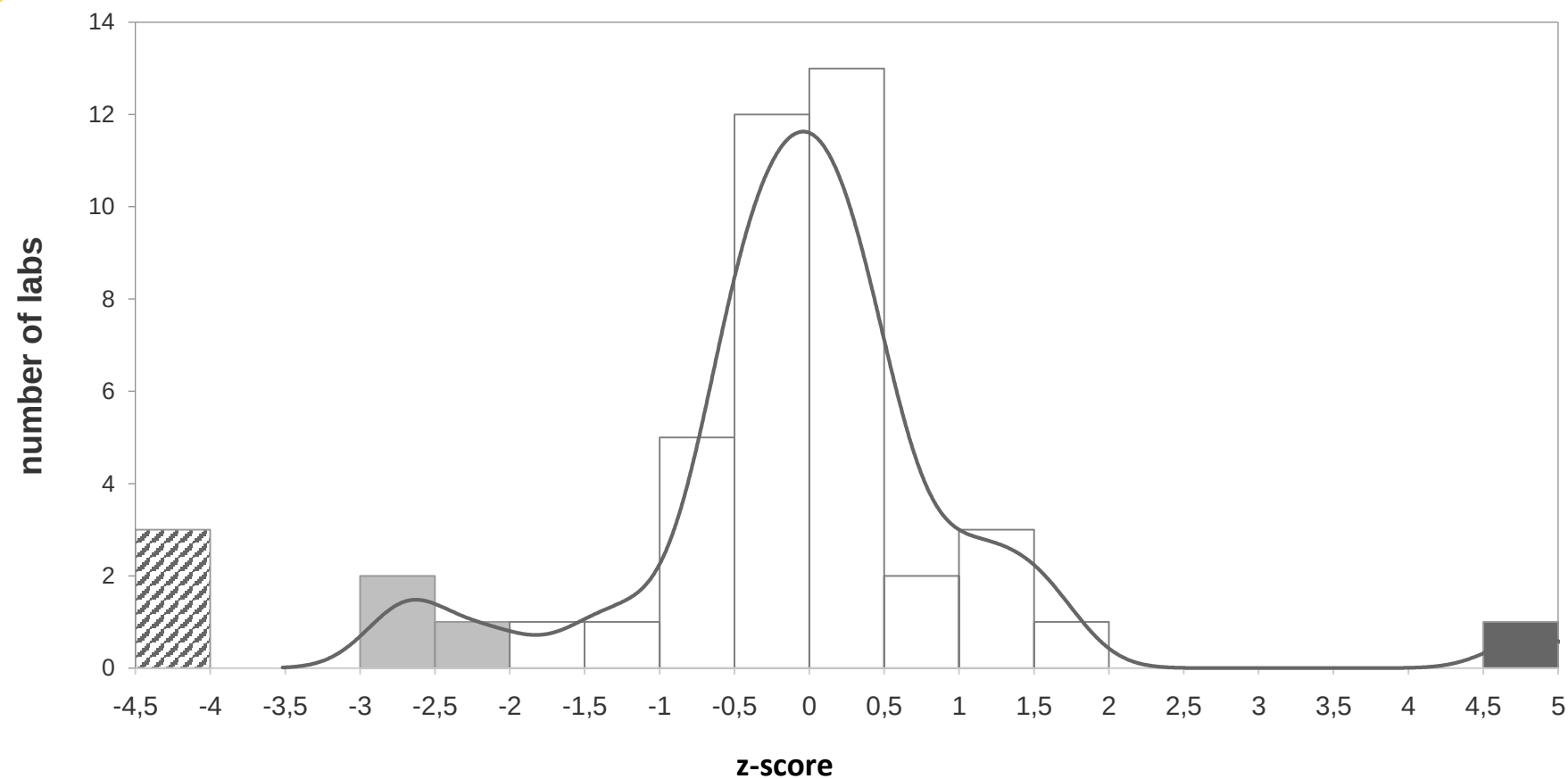
|            |               |
|------------|---------------|
| Results    | 42            |
| False Neg. | 3             |
| AV         | 0.119 [mg/kg] |
| CV*        | 19.0 %        |
| MRRL       | 0.01 [mg/kg]  |
| UAV-test   | passed        |

|                                                                                     |                            |              |    |       |
|-------------------------------------------------------------------------------------|----------------------------|--------------|----|-------|
|  | $ z\text{-score}  \leq 2$  | Acceptable   | 38 | (84%) |
|  | $2 <  z\text{-score}  < 3$ | Questionable | 3  | (7%)  |
|  | $ z\text{-score}  \geq 3$  | Unacceptable | 1  | (2%)  |
|  | False Negative             |              | 3  | (7%)  |



## BAC-C14

|            |               |
|------------|---------------|
| Results    | 42            |
| False Neg. | 3             |
| AV         | 0.119 [mg/kg] |
| CV*        | 19.0 %        |
| MRRL       | 0.01 [mg/kg]  |
| UAV-test   | passed        |

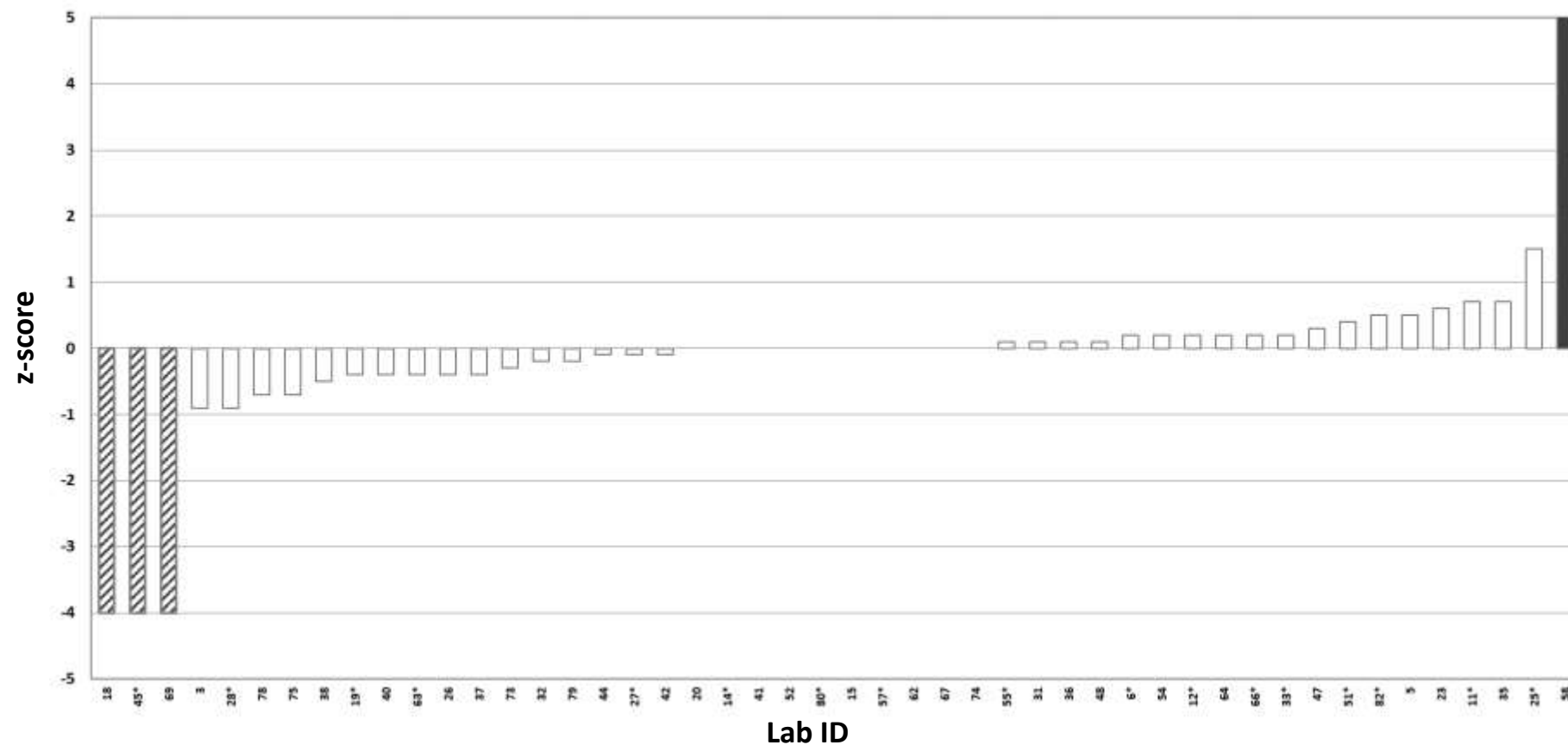


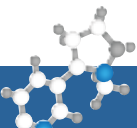
# Chlorate

|            |               |
|------------|---------------|
| Results    | 45            |
| False Neg. | 3             |
| AV         | 0.102 [mg/kg] |
| CV*        | 9.8 %         |
| MRRL       | 0.01 [mg/kg]  |
| UAV-test   | passed        |

|   |                            |              |    |       |
|---|----------------------------|--------------|----|-------|
| █ | $ z\text{-score}  \leq 2$  | Acceptable   | 44 | (92%) |
| █ | $2 <  z\text{-score}  < 3$ | Questionable | 0  | (0%)  |
| █ | $ z\text{-score}  \geq 3$  | Unacceptable | 1  | (2%)  |
| █ | False Negative             |              | 3  | (6%)  |

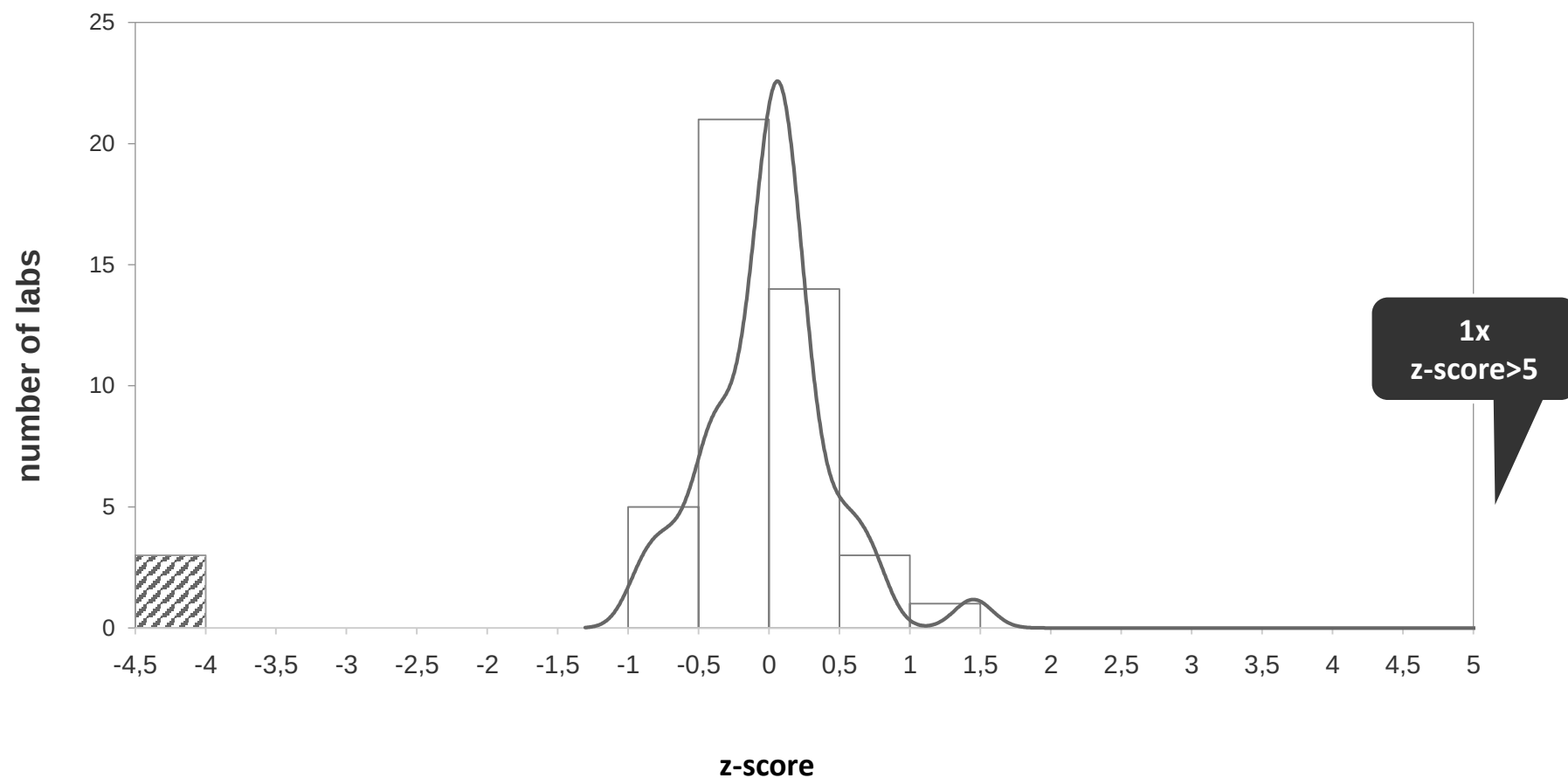
1x>5

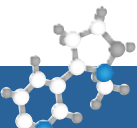




# Chlorate

|            |               |
|------------|---------------|
| Results    | 45            |
| False Neg. | 3             |
| AV         | 0.102 [mg/kg] |
| CV*        | 9.8 %         |
| MRRL       | 0.01 [mg/kg]  |
| UAV-test   | passed        |





## Impact of using ILIS

| CHLORATE    | Total                                                                                   | ILIS-Yes                                                                                | ILIS-No                                                                                  |
|-------------|-----------------------------------------------------------------------------------------|-----------------------------------------------------------------------------------------|------------------------------------------------------------------------------------------|
| Values      | 47                                                                                      | 28                                                                                      | 19                                                                                       |
| Median      | 0,103                                                                                   | 0,103                                                                                   | 0,102                                                                                    |
| Robust Mean | 0,102                                                                                   | 0,102                                                                                   | 0,102                                                                                    |
| CV*         |  10%   |  8%  |  14%  |
| RSD         |  28% |  9% |  43% |

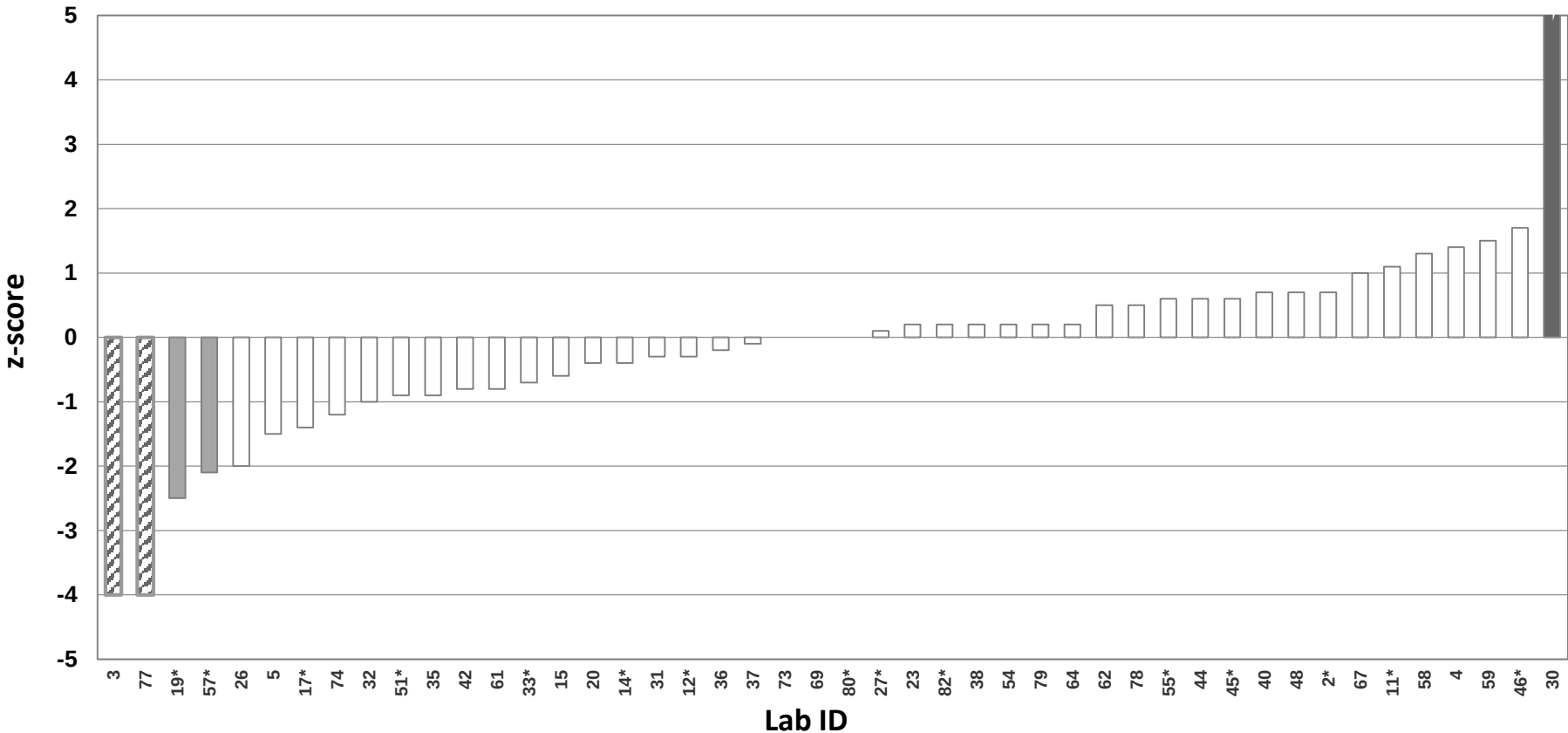


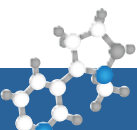
DDAC-C10

|            |               |
|------------|---------------|
| Results    | 44            |
| False Neg. | 2             |
| AV         | 0.149 [mg/kg] |
| CV*        | 25.0 %        |
| MRRL       | 0.01 [mg/kg]  |
| UAV-test   | passed        |

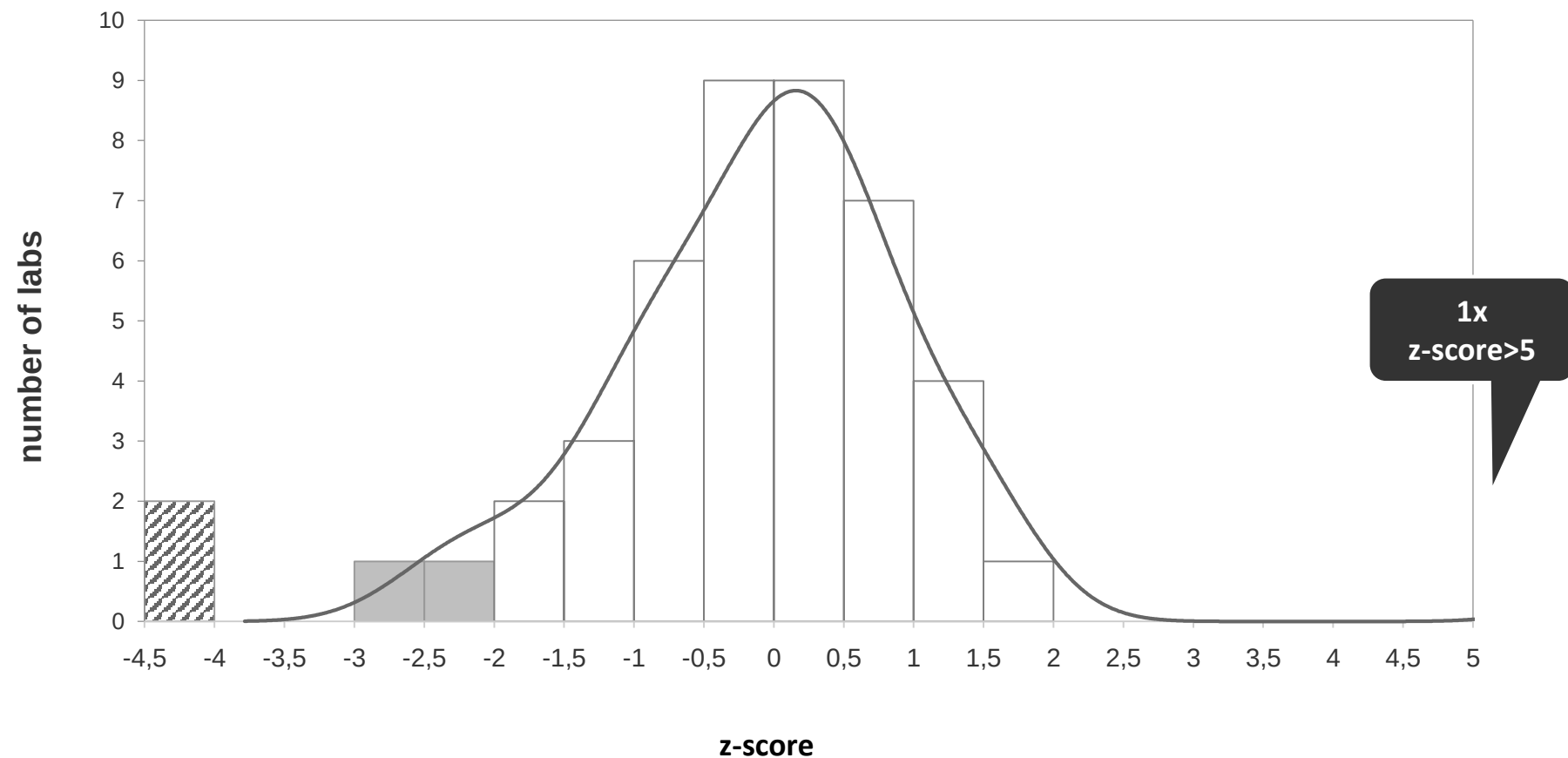
|                                                                                     |                            |              |    |       |
|-------------------------------------------------------------------------------------|----------------------------|--------------|----|-------|
|  | $ z\text{-score}  \leq 2$  | Acceptable   | 41 | (89%) |
|  | $2 <  z\text{-score}  < 3$ | Questionable | 2  | (4%)  |
|  | $ z\text{-score}  \geq 3$  | Unacceptable | 1  | (2%)  |
|  | False Negative             | Unacceptable | 2  | (5%)  |

1x>5





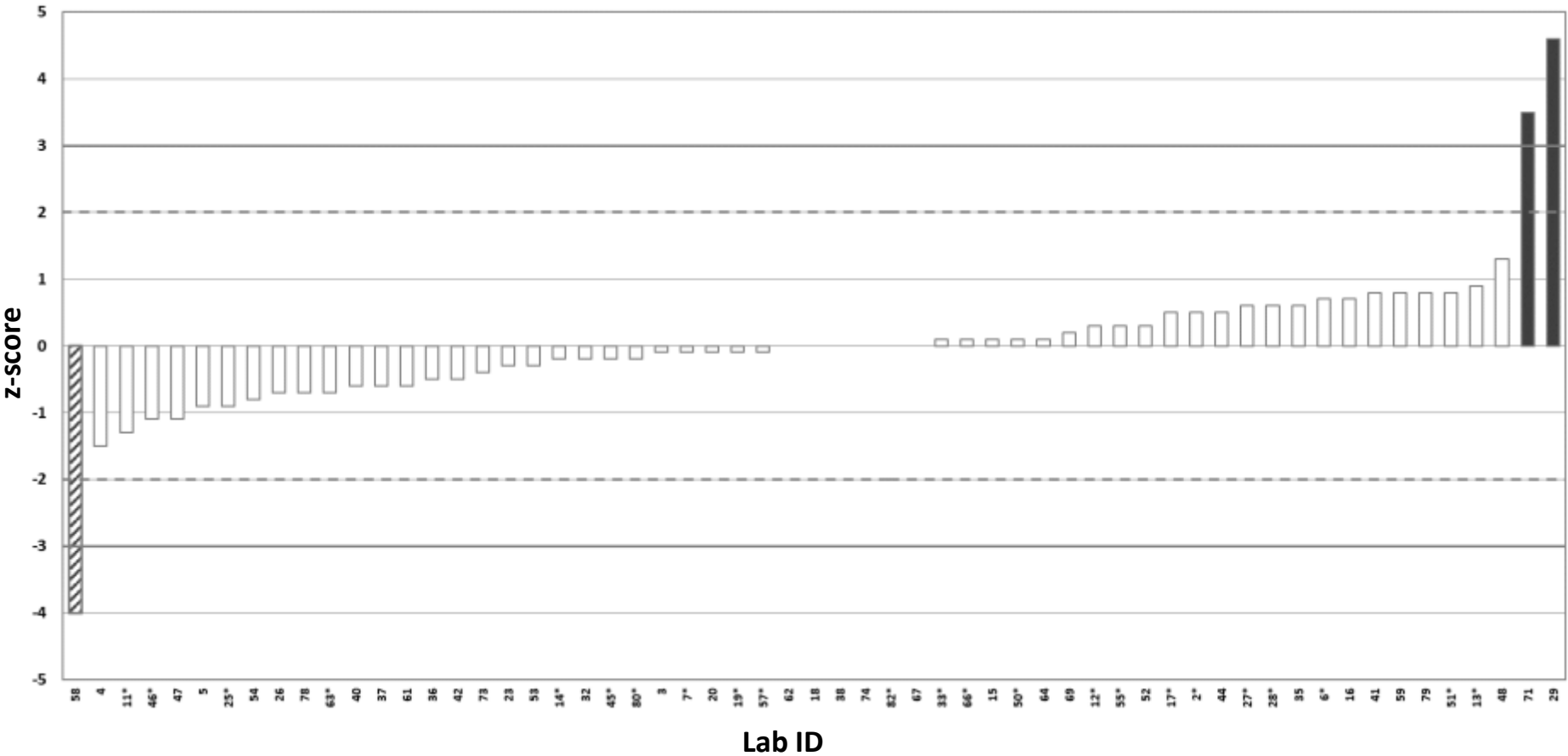
|            |               |
|------------|---------------|
| Results    | 44            |
| False Neg. | 2             |
| AV         | 0.149 [mg/kg] |
| CV*        | 25.0 %        |
| MRRL       | 0.01 [mg/kg]  |
| UAV-test   | passed        |

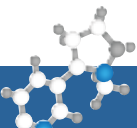


Fluazifop free acid

|            |                |
|------------|----------------|
| Results    | 58             |
| False Neg. | 1              |
| AV         | 0.0598 [mg/kg] |
| CV*        | 16.5 %         |
| MRRL       | 0.01 [mg/kg]   |
| UAV-test   | passed         |

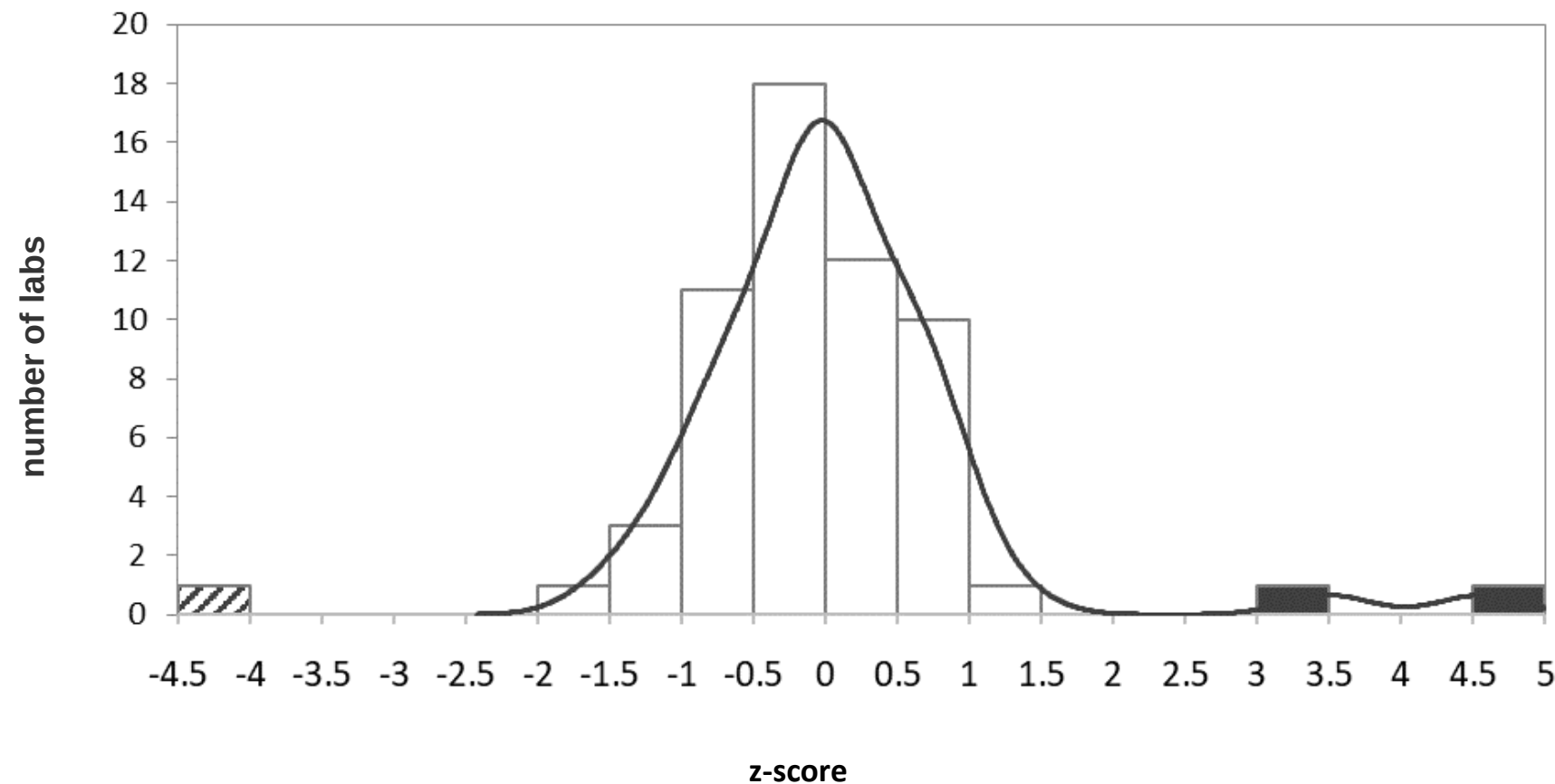
|                                                                                     |                            |              |    |       |
|-------------------------------------------------------------------------------------|----------------------------|--------------|----|-------|
|  | $ z\text{-score}  \leq 2$  | Acceptable   | 56 | (95%) |
|  | $2 <  z\text{-score}  < 3$ | Questionable | 0  | (0%)  |
|  | $ z\text{-score}  \geq 3$  | Unacceptable | 2  | (3%)  |
|  | False Negative             |              | 1  | (2%)  |





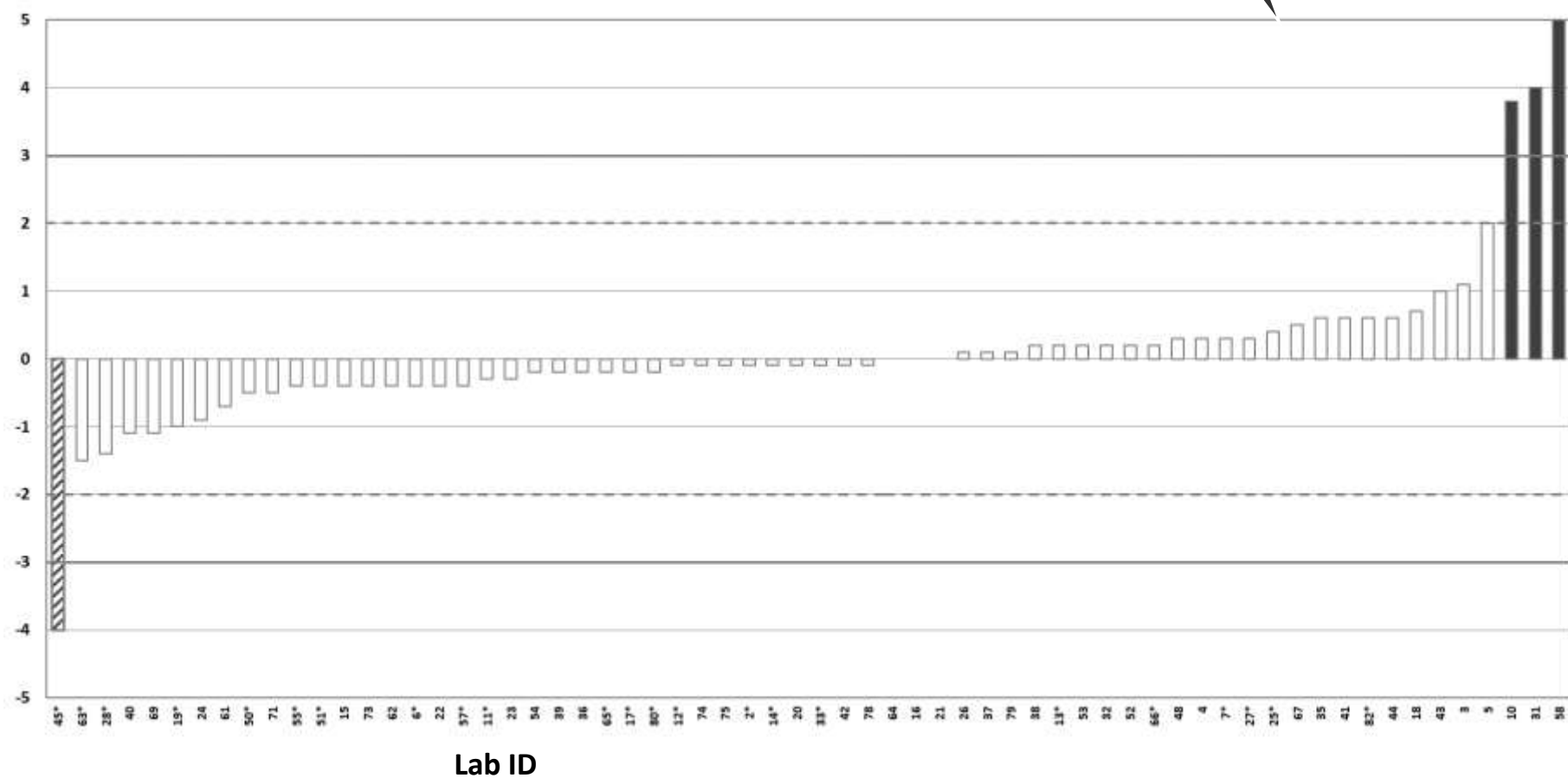
# Fluazifop free acid

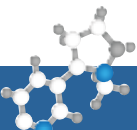
|            |                |
|------------|----------------|
| Results    | 58             |
| False Neg. | 1              |
| AV         | 0.0598 [mg/kg] |
| CV*        | 16.5 %         |
| MRRL       | 0.01 [mg/kg]   |
| UAV-test   | passed         |



# GLYPHOSATE

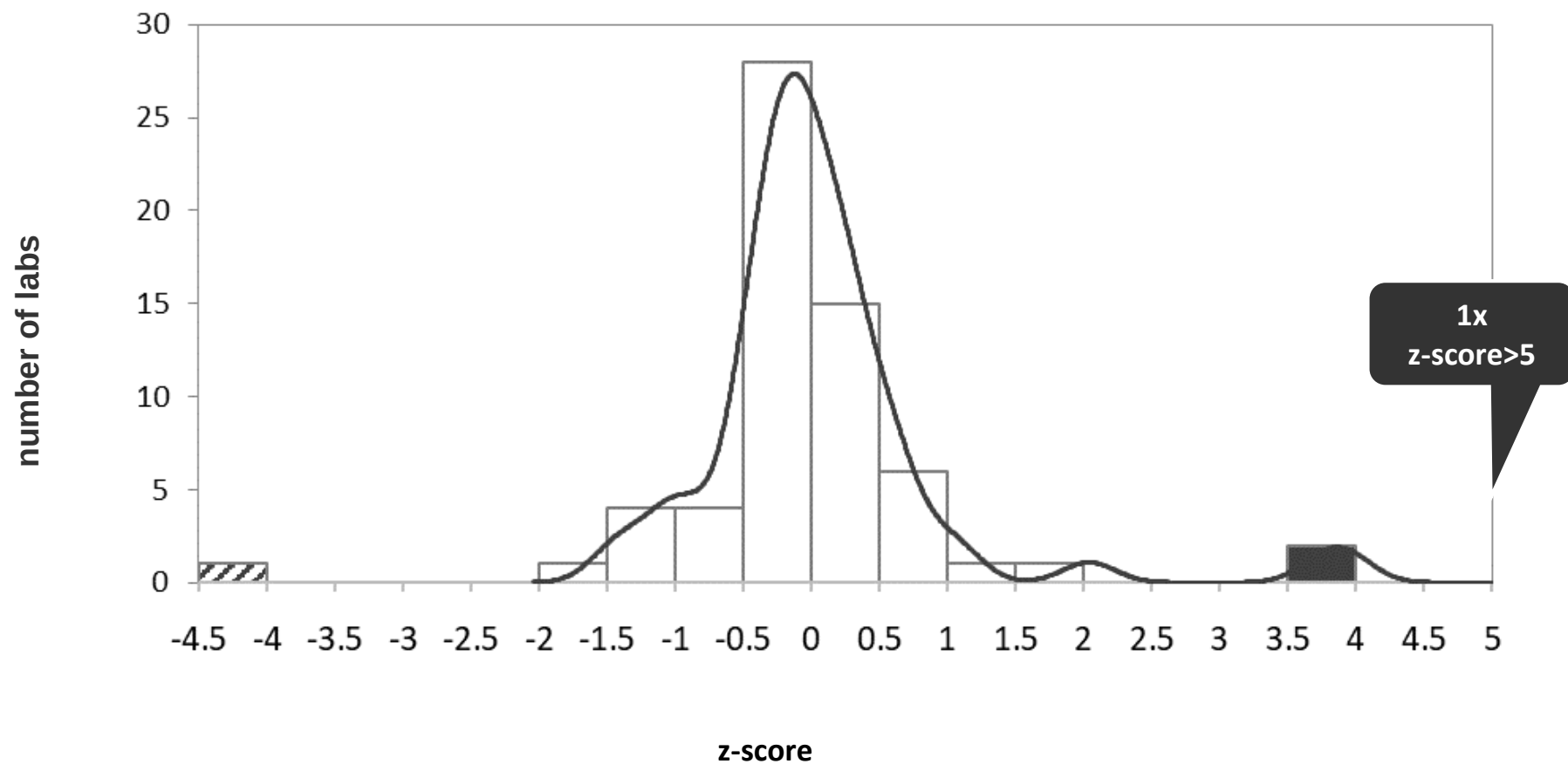
|                                                                                     |                            |                         |    |       |
|-------------------------------------------------------------------------------------|----------------------------|-------------------------|----|-------|
|  | $ z\text{-score}  \leq 2$  | Acceptable              | 60 | (77%) |
|  | $2 <  z\text{-score}  < 3$ | Questionable            | 0  | (9%)  |
|  | $ z\text{-score}  \geq 3$  | 1x<br>z>5<br>Acceptable | 3  | (13%) |
|  | False Negative             |                         | 1  | (1%)  |

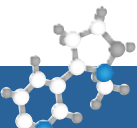




# Glyphosate

|            |               |
|------------|---------------|
| Results    | 63            |
| False Neg. | 1             |
| AV         | 0.102 [mg/kg] |
| CV*        | 12.8 %        |
| MRRL       | 0.01 [mg/kg]  |
| UAV-test   | passed        |

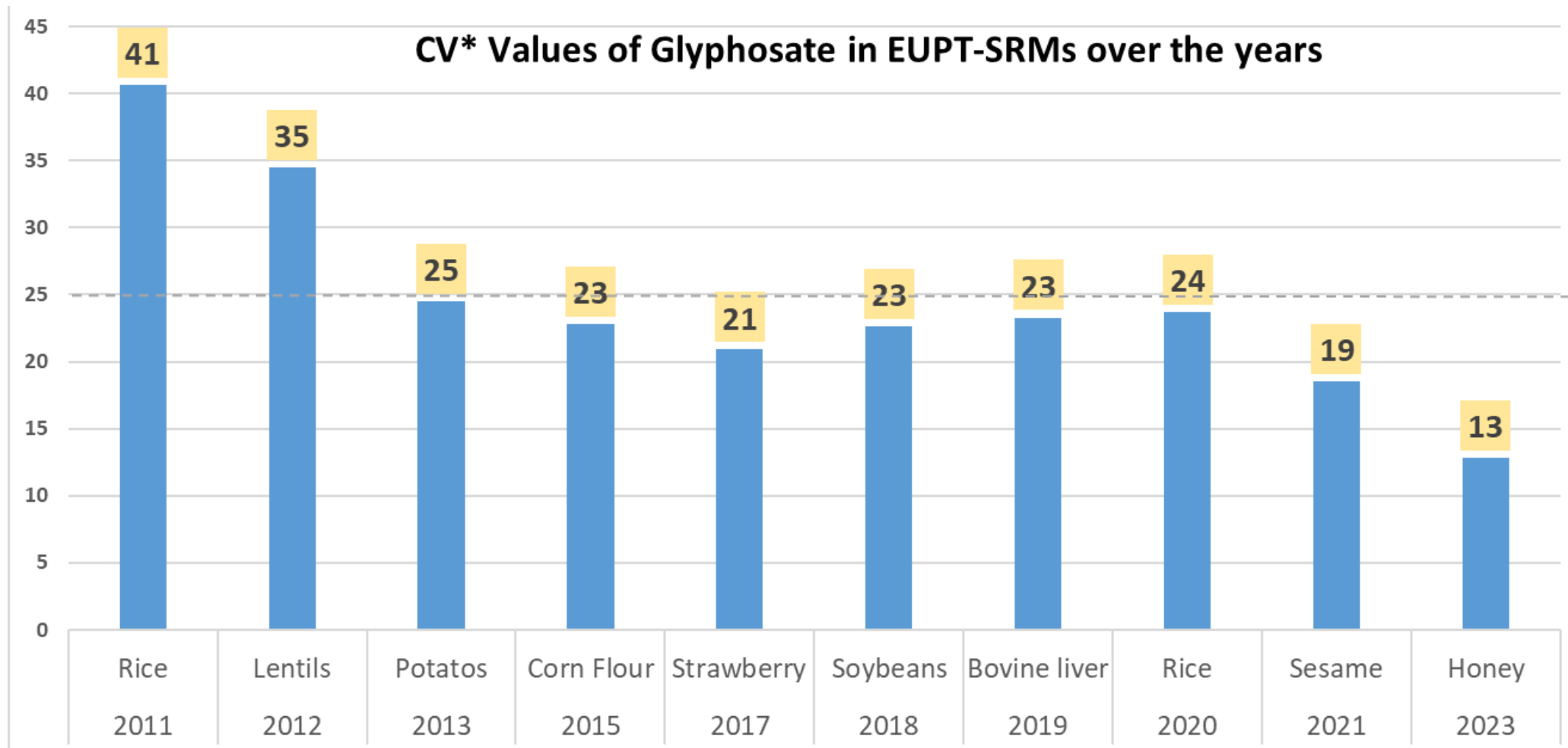




## Impact of using ILIS

| GLYPHOSATE  | Total | ILIS-Yes | ILIS-No |
|-------------|-------|----------|---------|
| Values      | 66    | 46       | 20      |
| Median      | 0,100 | 0,100    | 0,100   |
| Robust Mean | 0,102 | 0,102    | 0,100   |
| CV*         | 13%   | 13%      | 16%     |
| RSD         | 30%   | 23%      | 43%     |

# Long-term Performance - Glyphosate





# Glyphosate and Chlormequate (since SRM4)

## Long-term Performance - Glyphosate

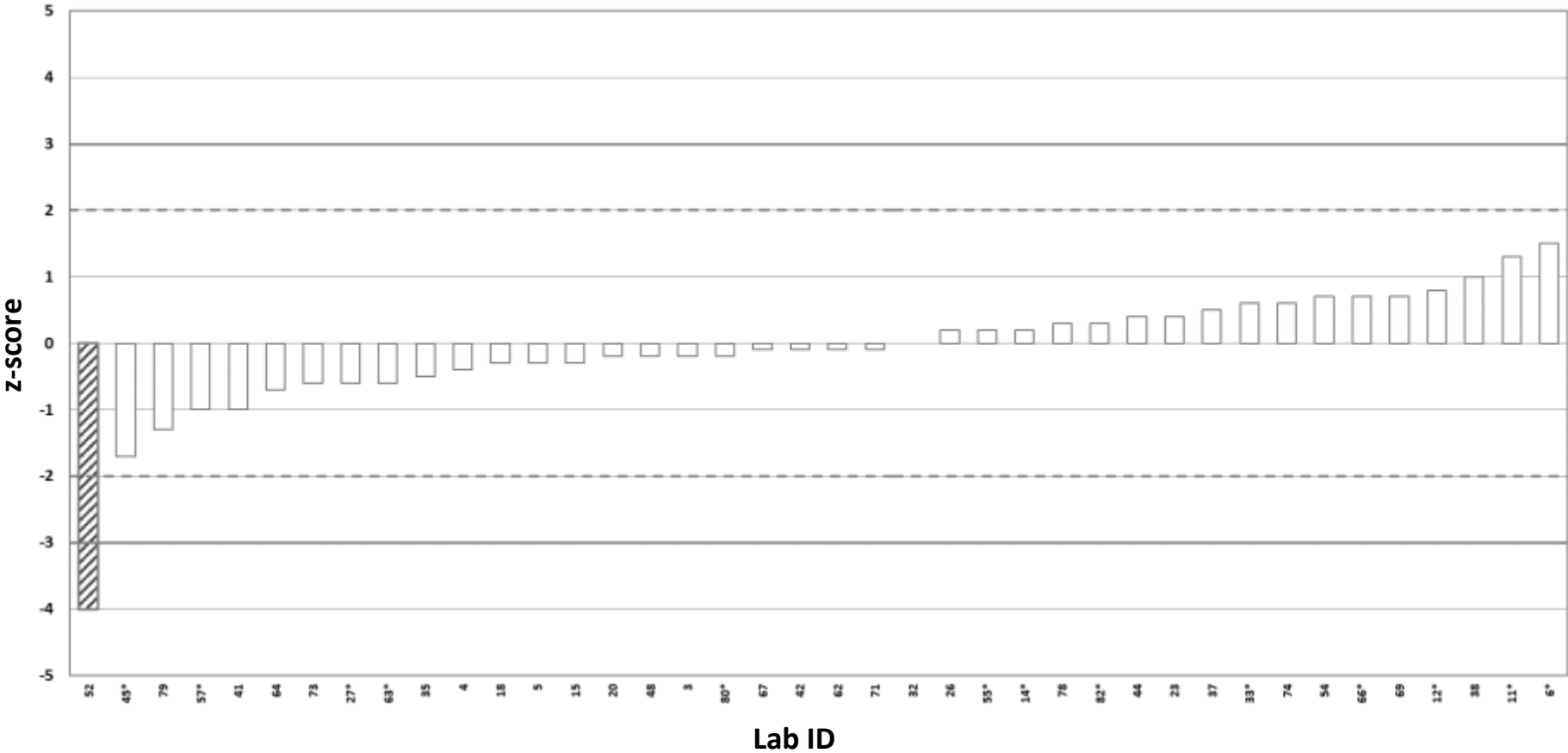
|                | SRM4 | SRM6  | SRM7    | SRM8     | SRM10 | SRM12         | SRM13     | SRM14 | SRM15 | SRM16  | SRM18 |
|----------------|------|-------|---------|----------|-------|---------------|-----------|-------|-------|--------|-------|
| Matrix         | Oats | Rice  | Lentils | Potatoes | Maiz  | Straw-berries | Soy-beans | Liver | Rice  | Sesame | Honey |
| Glyphosate     |      |       |         |          |       |               |           |       |       |        |       |
| No. Results    | 6    | 34    | 39      | 45       | 62    | 86            | 83        | 43    | 84    | 88     | 66    |
| % of Particip. | 13%  | 34%   | 35%     | 41%      | 60%   | 67%           | 76%       | 75%   | 78%   | 88%    | 90%   |
| CV* [%]        | --   | 40.6% | 34.5%   | 24.5%    | 22.8% | 20.9%         | 22.6%     | 23.3% | 23.7% | 18.5%  | 12.8% |

Avg CV\*= 22.7 %

Matrine

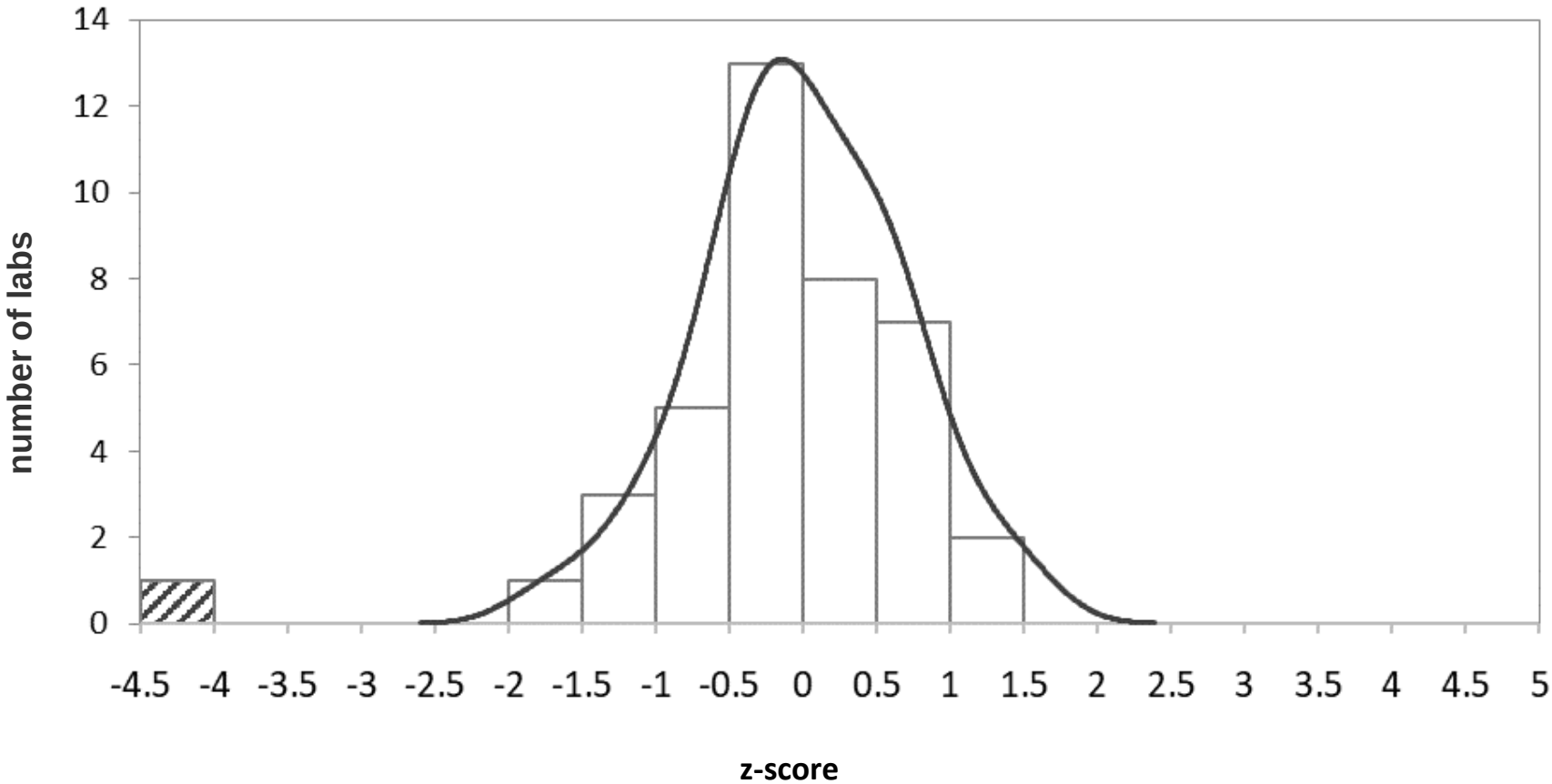
|            |                |
|------------|----------------|
| Results    | 39             |
| False Neg. | 1              |
| AV         | 0.0873 [mg/kg] |
| CV*        | 16.9 %         |
| MRRL       | 0.01 [mg/kg]   |
| UAV-test   | passed         |

|  |                            |              |    |       |
|--|----------------------------|--------------|----|-------|
|  | $ z\text{-score}  \leq 2$  | Acceptable   | 39 | (97%) |
|  | $2 <  z\text{-score}  < 3$ | Questionable | 0  | (0%)  |
|  | $ z\text{-score}  \geq 3$  | Unacceptable | 0  | (0%)  |
|  | False Negative             |              | 1  | (3%)  |



Matrine

|            |                |
|------------|----------------|
| Results    | 39             |
| False Neg. | 1              |
| AV         | 0.0873 [mg/kg] |
| CV*        | 16.9 %         |
| MRRL       | 0.01 [mg/kg]   |
| UAV-test   | passed         |

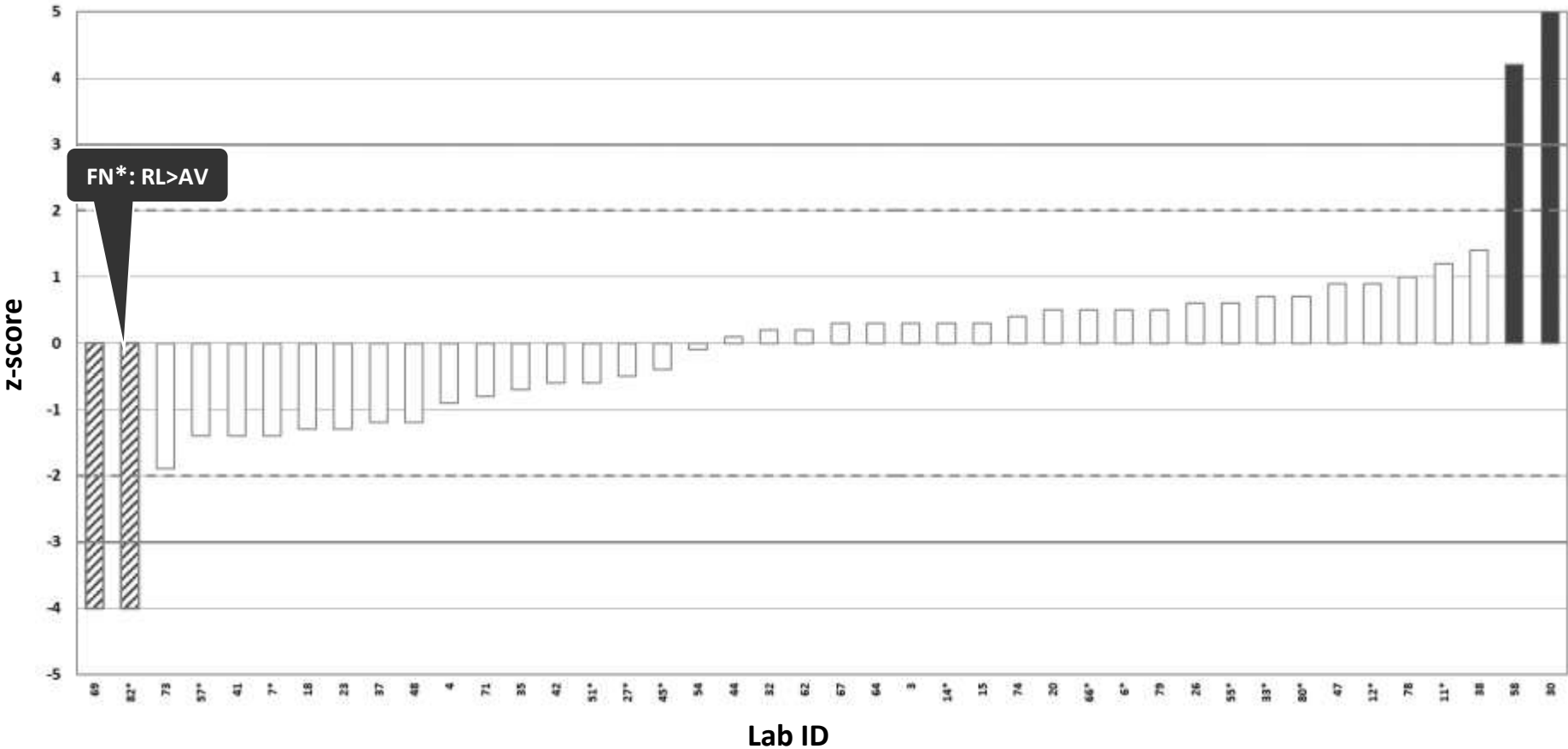


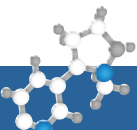
Nicotine

|            |                |
|------------|----------------|
| Results    | 40             |
| False Neg. | 1 + 1 (FN*)    |
| AV         | 0.0877 [mg/kg] |
| CV*        | 25.3 %         |
| MRRL       | 0.01 [mg/kg]   |
| UAV-test   | passed         |

|  |                            |              |    |       |
|--|----------------------------|--------------|----|-------|
|  | $ z\text{-score}  \leq 2$  | Acceptable   | 38 | (90%) |
|  | $2 <  z\text{-score}  < 3$ | Questionable | 0  | (0%)  |
|  | $ z\text{-score}  \geq 3$  | Unacceptable | 2  | (5%)  |
|  | False Negative             |              | 2  | (5%)  |

1x  
z>5

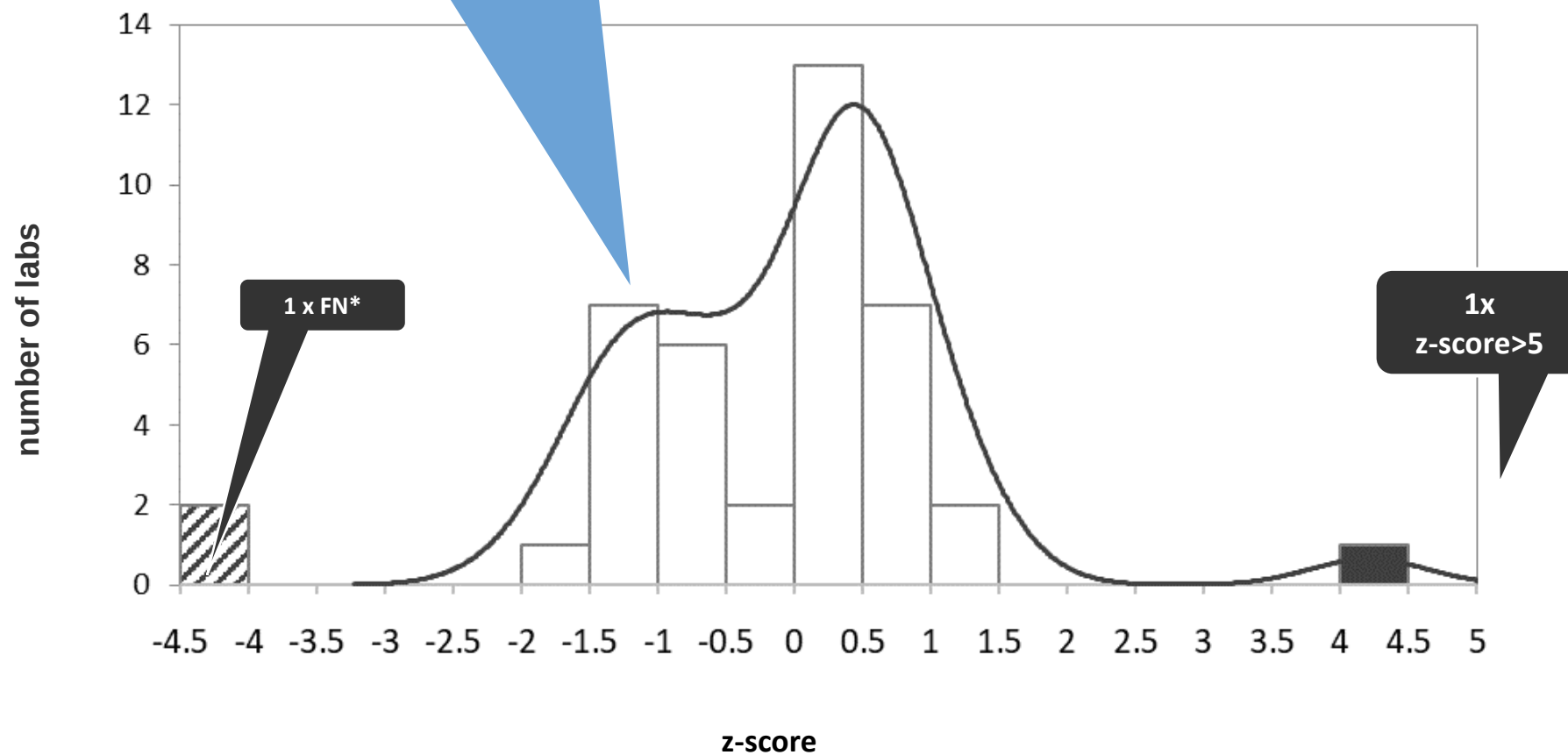


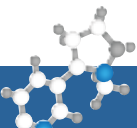


# Nicotine

|            |                |
|------------|----------------|
| Results    | 40             |
| False Neg. | 1 + 1 (FN*)    |
| AV         | 0.0877 [mg/kg] |
| CV*        | 25.3 %         |
| MRRL       | 0.01 [mg/kg]   |
| UAV-test   | passed         |

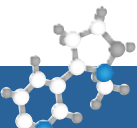
No obvious analytical reason for the apparent bimodality could be found





## Impact of using ILIS

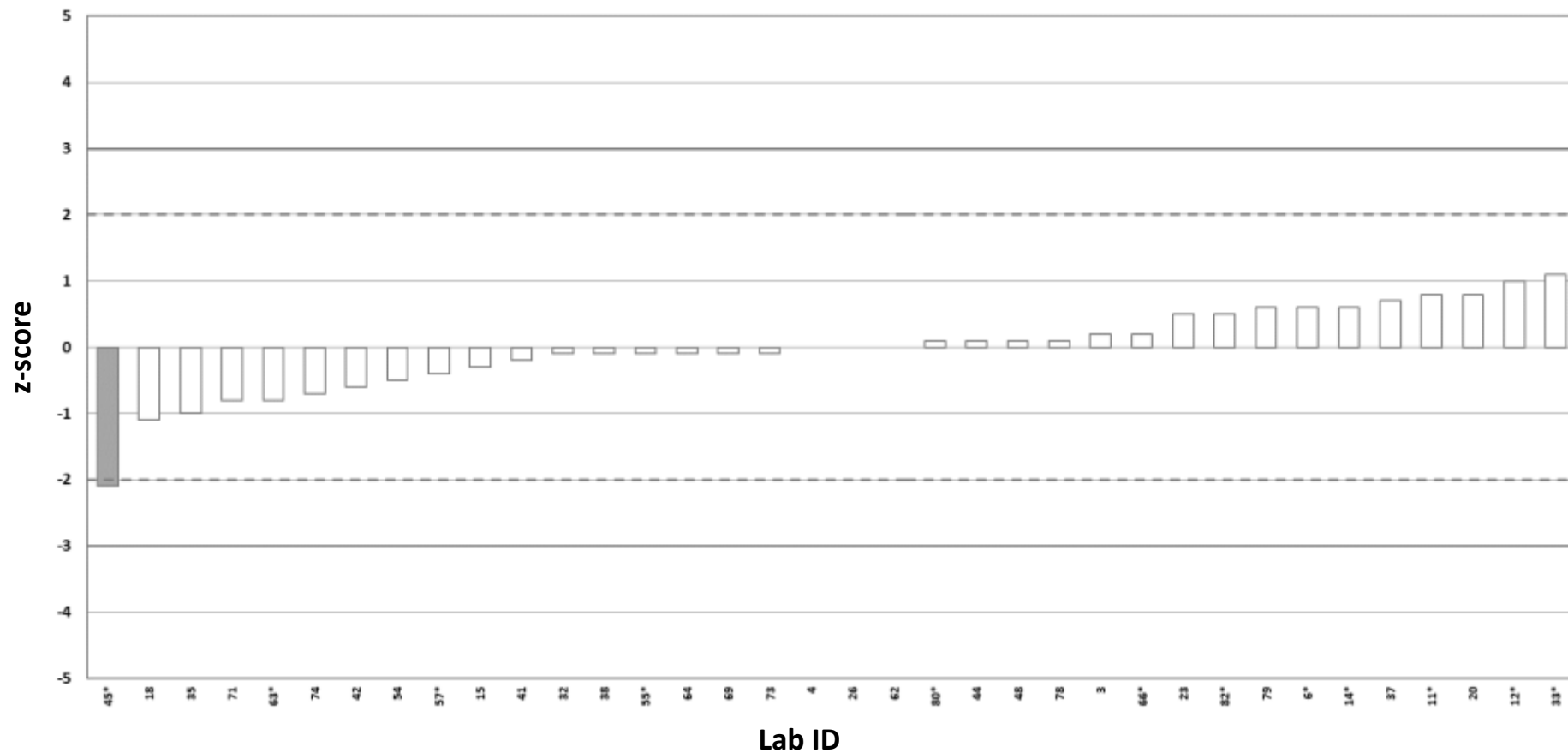
| NICOTINE    | Total | ILIS-Yes | ILIS-No |
|-------------|-------|----------|---------|
| Values      | 42    | 12       | 30      |
| Median      | 0,094 | 0,092    | 0,095   |
| Robust Mean | 0,088 | 0,085    | 0,090   |
| CV*         | 26%   | 23%      | 28%     |
| RSD         | 49%   | 21%      | 56%     |

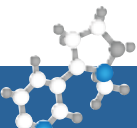


# Oxymatrine

|            |                |
|------------|----------------|
| Results    | 36             |
| False Neg. | 0              |
| AV         | 0.0681 [mg/kg] |
| CV*        | 15.4 %         |
| MRRL       | 0.01 [mg/kg]   |
| UAV-test   | passed         |

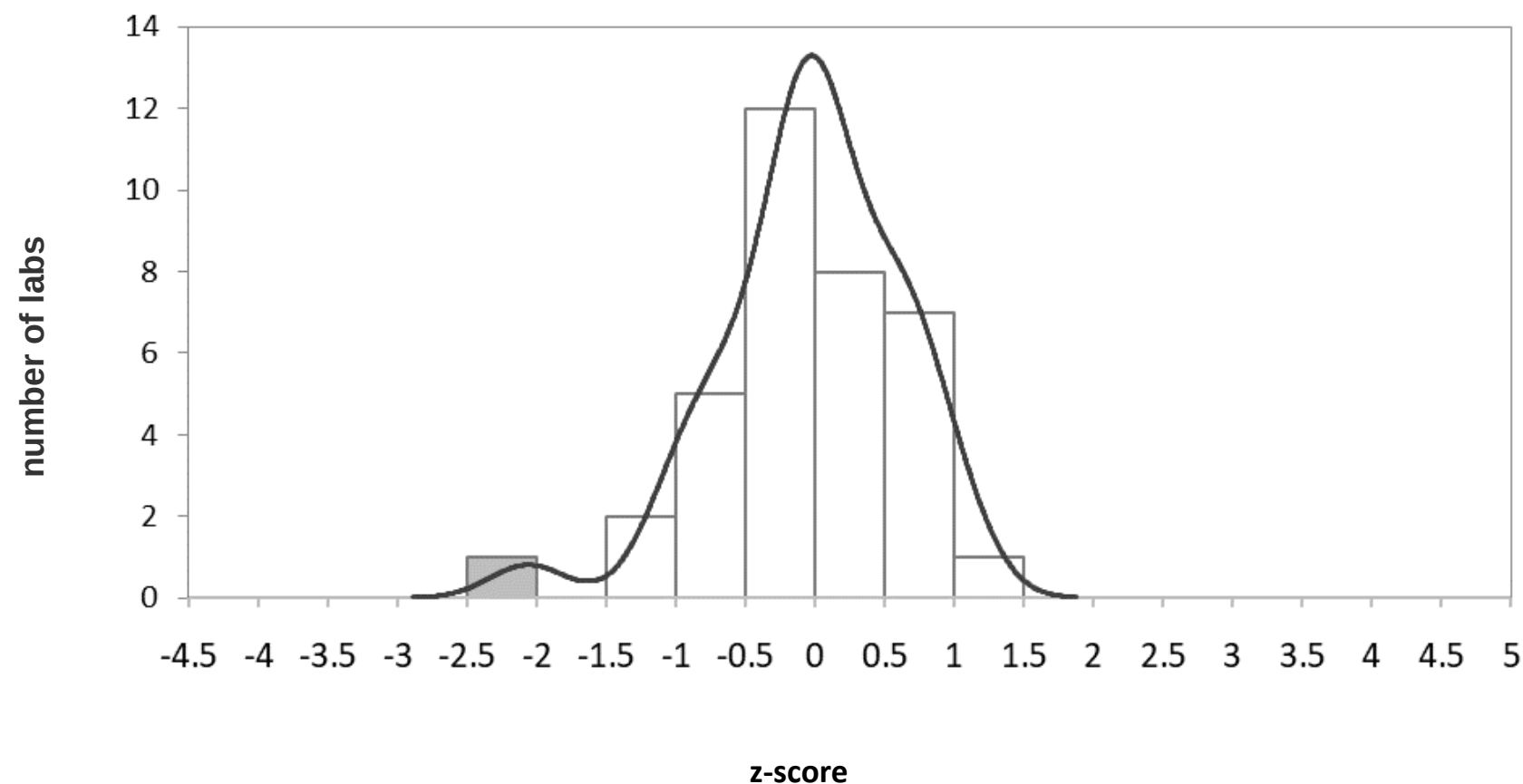
|                                                                                     |                            |              |    |       |
|-------------------------------------------------------------------------------------|----------------------------|--------------|----|-------|
|  | $ z\text{-score}  \leq 2$  | Acceptable   | 35 | (97%) |
|  | $2 <  z\text{-score}  < 3$ | Questionable | 1  | (3%)  |
|  | $ z\text{-score}  \geq 3$  | Unacceptable | 0  | (0%)  |
|  | False Negative             | Unacceptable | 0  | (0%)  |





# Oxymatrine

|            |                |
|------------|----------------|
| Results    | 36             |
| False Neg. | 0              |
| AV         | 0.0681 [mg/kg] |
| CV*        | 15.4 %         |
| MRRL       | 0.01 [mg/kg]   |
| UAV-test   | passed         |





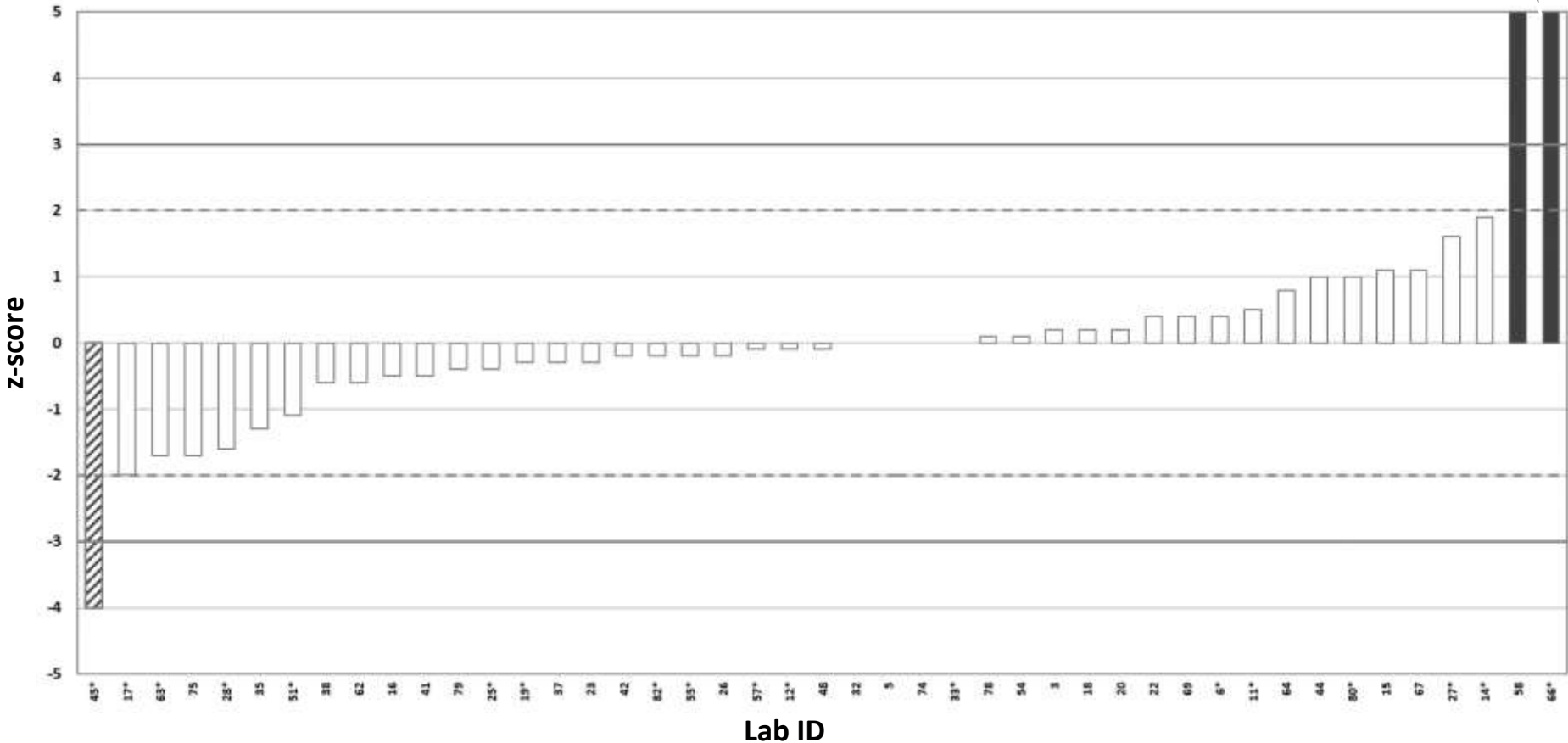
Phosphonic acid

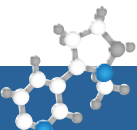
|            |               |
|------------|---------------|
| Results    | 44            |
| False Neg. | 1             |
| AV         | 0.202 [mg/kg] |
| CV*        | 20.9 %        |
| MRRL       | 0.03 [mg/kg]  |
| UAV-test   | passed        |

|  |                            |              |    |       |
|--|----------------------------|--------------|----|-------|
|  | $ z\text{-score}  \leq 2$  | Acceptable   | 42 | (90%) |
|  | $2 <  z\text{-score}  < 3$ | Questionable | 0  | (0%)  |
|  | $ z\text{-score}  \geq 3$  | Unacceptable | 2  | (5%)  |
|  | False Negative             |              | 1  | (5%)  |

2x

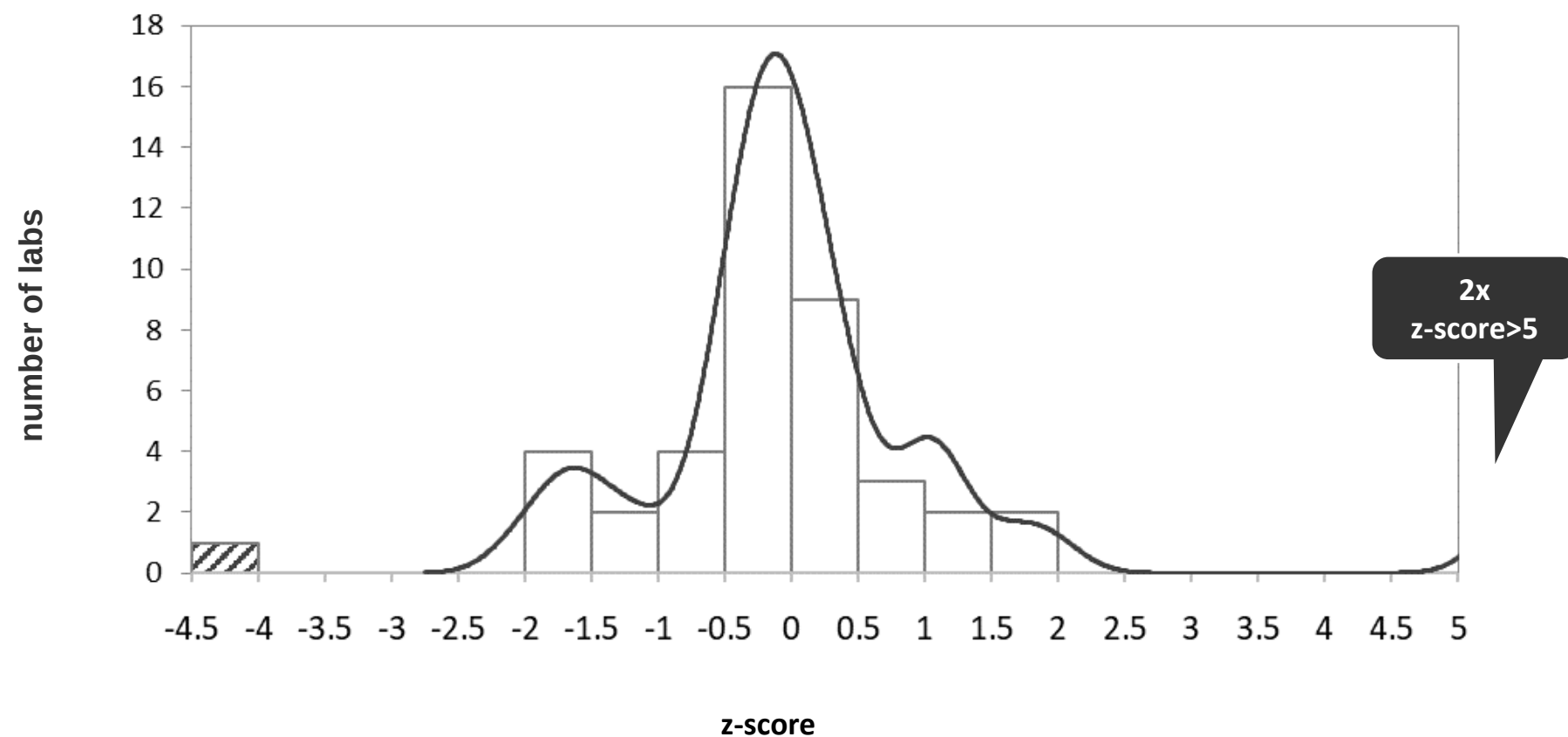
z>5

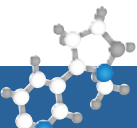




# Phosphonic acid

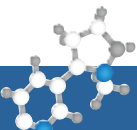
|            |               |
|------------|---------------|
| Results    | 44            |
| False Neg. | 1             |
| AV         | 0.202 [mg/kg] |
| CV*        | 20.9 %        |
| MRRL       | 0.03 [mg/kg]  |
| UAV-test   | passed        |





## Impact of using ILIS

| PHOSPHONIC ACID | Total | ILIS-Yes | ILIS-No |
|-----------------|-------|----------|---------|
| Values          | 47    | 23       | 24      |
| Median          | 0,197 | 0,199    | 0,196   |
| Robust Mean     | 0,200 | 0,197    | 0,206   |
| CV*             | 21%   | 15%      | 30%     |
| RSD             | 36%   | 18%      | 46%     |



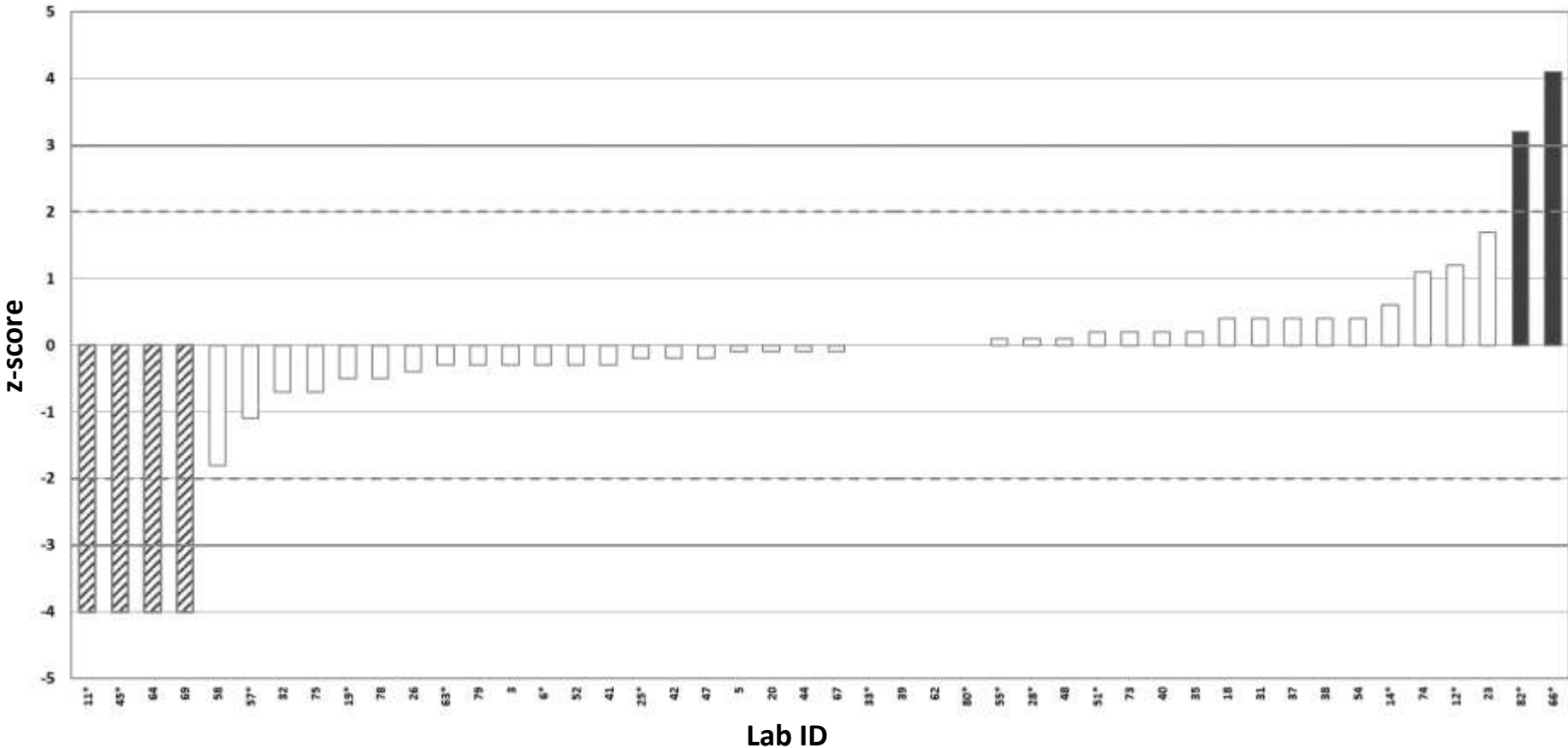
# RESULT OVERVIEW

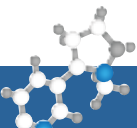
OPTIONAL Compounds

Perchlorate anion

|            |               |
|------------|---------------|
| Results    | 42            |
| False Neg. | 4             |
| AV         | 0.055 [mg/kg] |
| CV*        | 11.6 %        |
| MRRL       | 0.01 [mg/kg]  |
| UAV-test   | passed        |

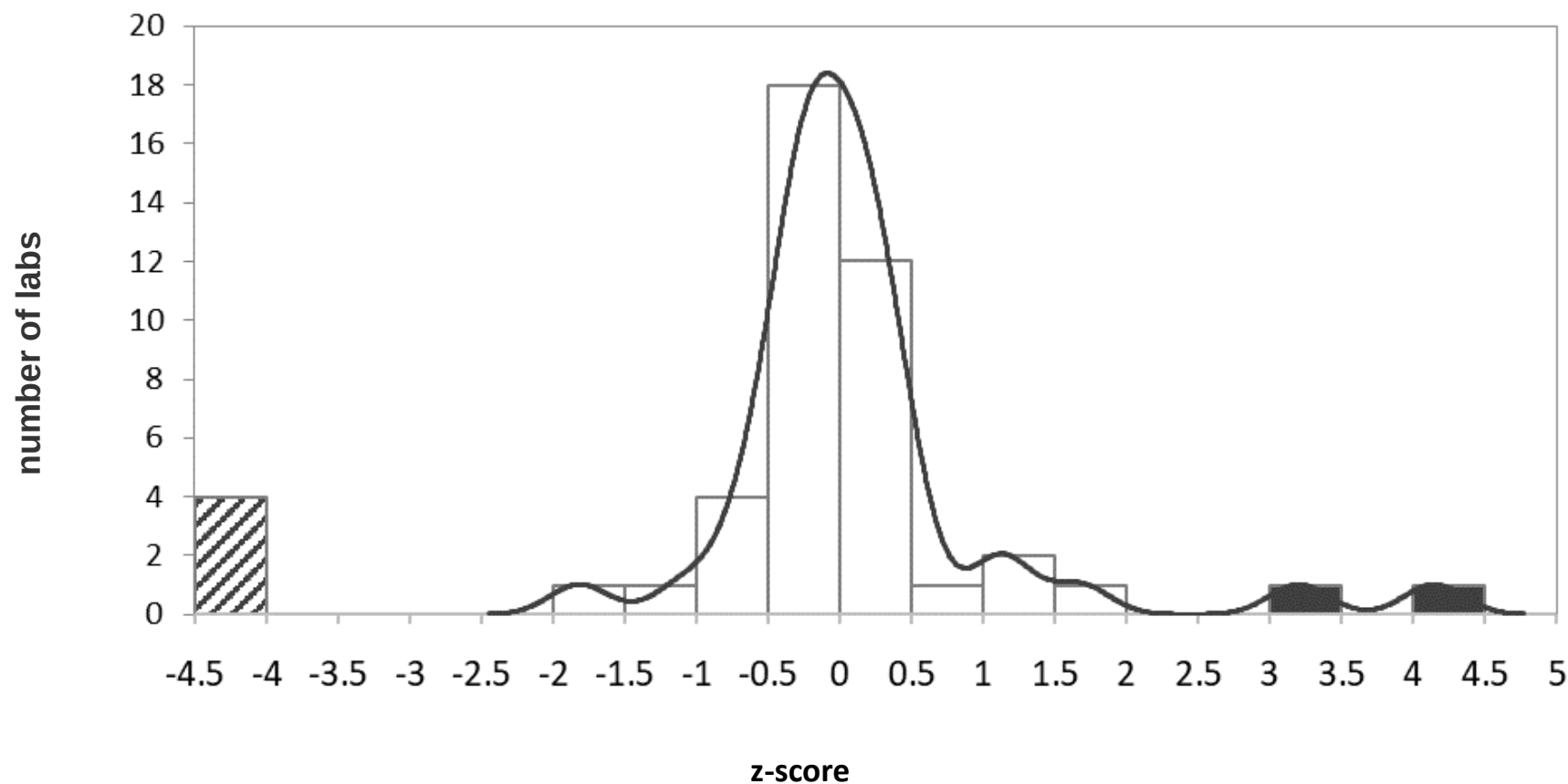
|                                                                                     |                            |              |    |       |
|-------------------------------------------------------------------------------------|----------------------------|--------------|----|-------|
|  | $ z\text{-score}  \leq 2$  | Acceptable   | 40 | (87%) |
|  | $2 <  z\text{-score}  < 3$ | Questionable | 0  | (0%)  |
|  | $ z\text{-score}  \geq 3$  | Unacceptable | 2  | (4%)  |
|  | False Negative             | Unacceptable | 4  | (9%)  |

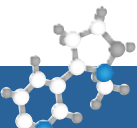




# Perchlorate (anion)

|            |               |
|------------|---------------|
| Results    | 42            |
| False Neg. | 4             |
| AV         | 0.055 [mg/kg] |
| CV*        | 11.6 %        |
| MRRL       | 0.01 [mg/kg]  |
| UAV-test   | passed        |





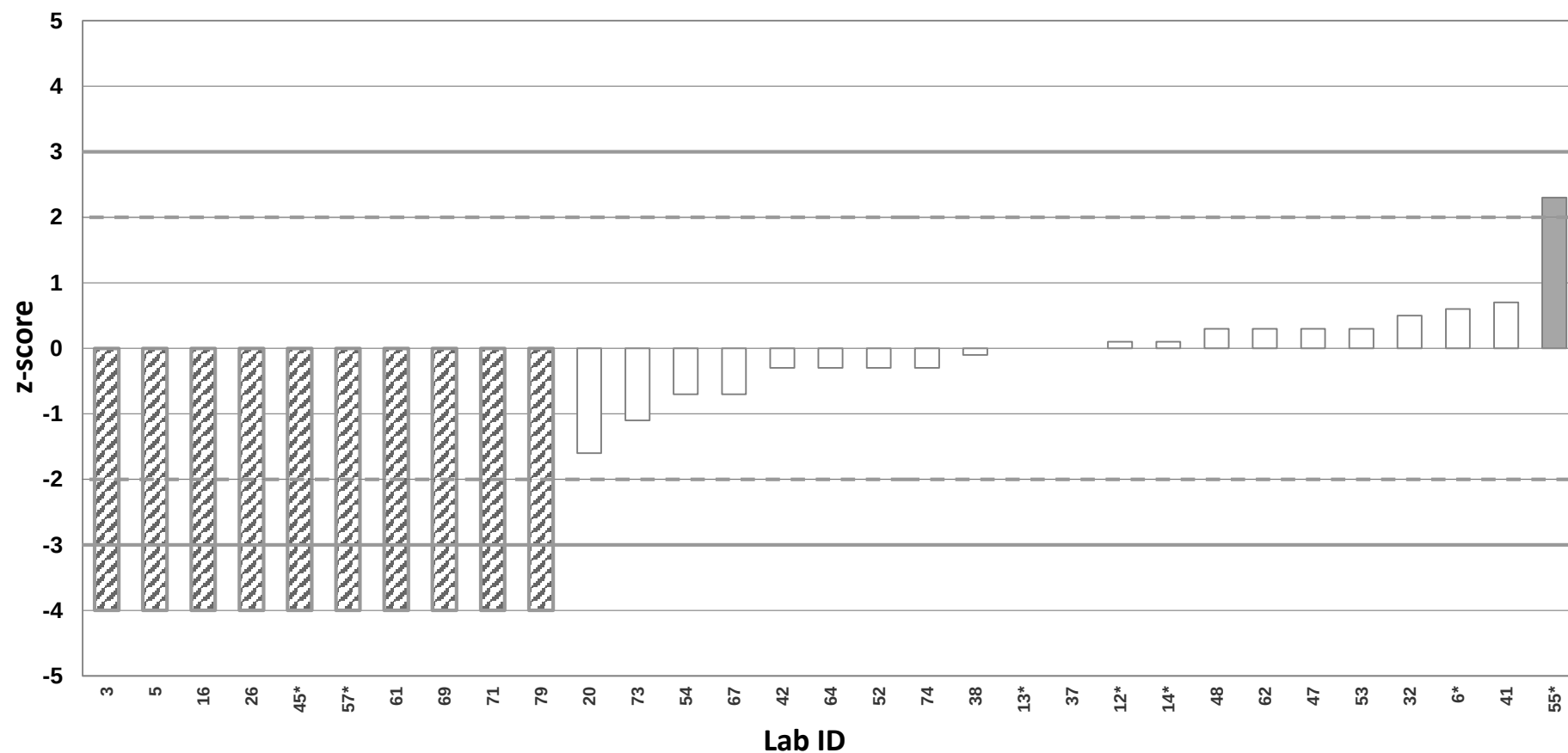
## Impact of using ILIS

| PERCHLORATE | Total | ILIS-Yes | ILIS-No |
|-------------|-------|----------|---------|
| Values      | 44    | 28       | 16      |
| Median      | 0,055 | 0,055    | 0,053   |
| Robust Mean | 0,055 | 0,055    | 0,054   |
| CV*         | 11%   | 8%       | 19%     |
| RSD         | 24%   | 19%      | 34%     |

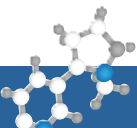
# Trinexapac free acid

|            |               |
|------------|---------------|
| Results    | 21            |
| False Neg. | 10            |
| AV         | 0.118 [mg/kg] |
| CV*        | 14.7 %        |
| MRRL       | 0.01 [mg/kg]  |
| UAV-test   | passed        |

|                                                                                     |                            |              |    |       |
|-------------------------------------------------------------------------------------|----------------------------|--------------|----|-------|
|  | $ z\text{-score}  \leq 2$  | Acceptable   | 20 | (70%) |
|  | $2 <  z\text{-score}  < 3$ | Questionable | 1  | (0%)  |
|  | $ z\text{-score}  \geq 3$  | Unacceptable | 0  | (4%)  |
|  | False Negative             | Unacceptable | 10 | (33%) |

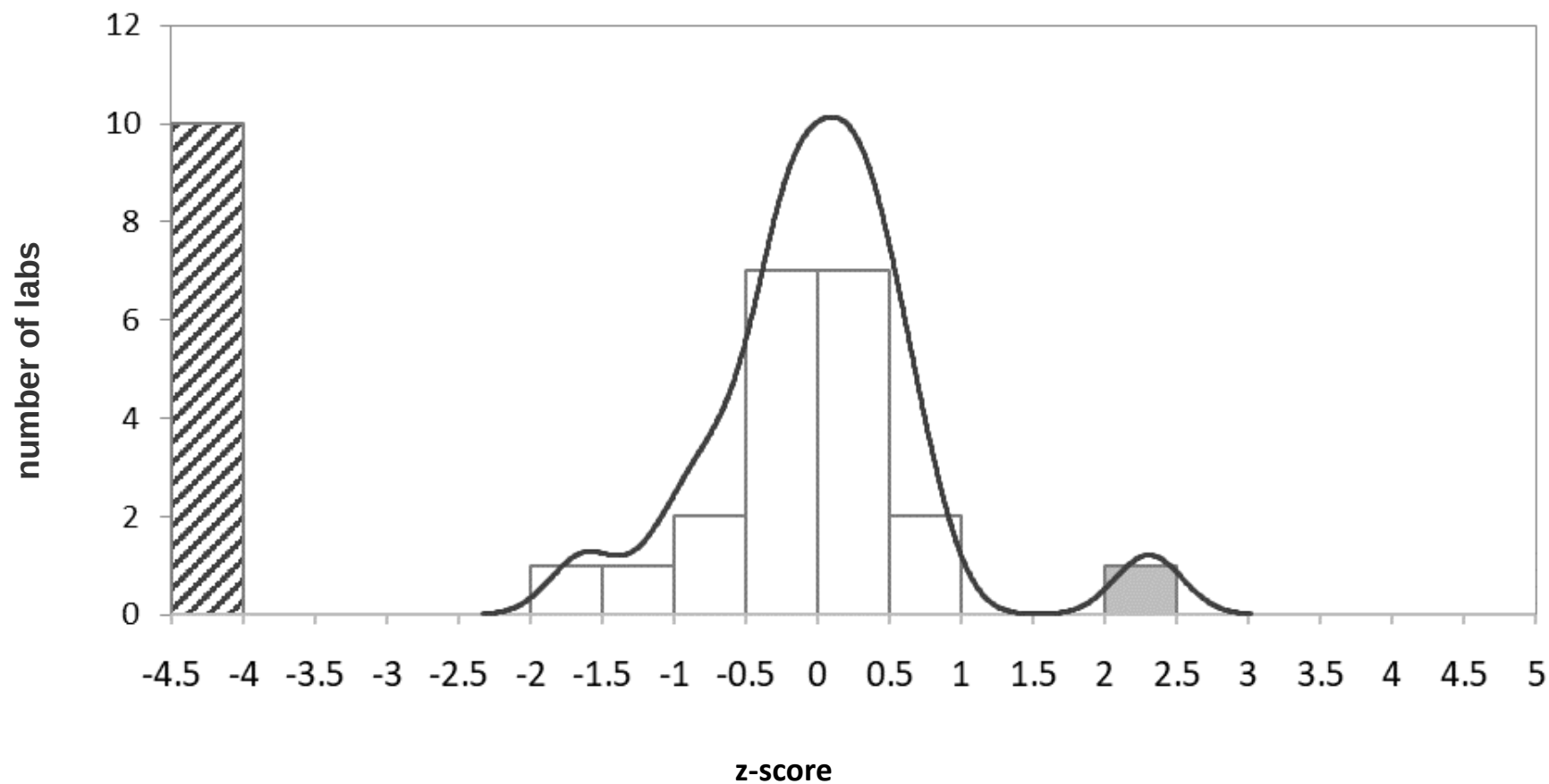


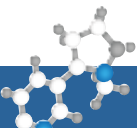




# Trinexapac free acid

|            |               |
|------------|---------------|
| Results    | 21            |
| False Neg. | 10            |
| AV         | 0.118 [mg/kg] |
| CV*        | 14.7 %        |
| MRRL       | 0.01 [mg/kg]  |
| UAV-test   | passed        |





# Uncertainty of assigned value

## COMPULSORY / OPTIONAL / Extra Compounds

EU+EFTA

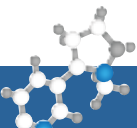
| Compound               | No. of FNs  <br>Outlier             | No. of<br>Numerical<br>Results | AV<br>[mg/kg] | CV*<br>[%] | Uncertainty<br>of AV (UAV)<br>[mg/kg] | UAV-<br>Tolerance<br>[mg/kg] | Judgement |
|------------------------|-------------------------------------|--------------------------------|---------------|------------|---------------------------------------|------------------------------|-----------|
| 2,4-D (free acid)      | 0   0                               | 60                             | 0.052         | 18.9%      | 0.0016                                | 0.0039                       | passed    |
| BAC-C14 chloride       | 3   1                               | 42                             | 0.119         | 19.0%      | 0.0044                                | 0.0089                       | passed    |
| Chlorate (anion)       | 3   1                               | 45                             | 0.102         | 9.8%       | 0.0019                                | 0.0077                       | passed    |
| DDAC-C10 chloride      | 2   1                               | 44                             | 0.149         | 25.0%      | 0.0071                                | 0.0112                       | passed    |
| Fluazifop (free acid)  | 1   2                               | 58                             | 0.060         | 16.5%      | 0.0017                                | 0.0045                       | passed    |
| Glyphosate             | 1   4                               | 63                             | 0.102         | 12.8%      | 0.0021                                | 0.0077                       | passed    |
| Matrine                | 1   0                               | 39                             | 0.087         | 16.9%      | 0.0029                                | 0.0065                       | passed    |
| Nicotine               | 2   1                               | 40                             | 0.088         | 25.3%      | 0.0044                                | 0.0066                       | passed    |
| Oxymatrine             | 0   0                               | 36                             | 0.068         | 15.4%      | 0.0022                                | 0.0051                       | passed    |
| Phosphonic acid        | 1   2                               | 44                             | 0.202         | 20.9%      | 0.0082                                | 0.0152                       | passed    |
| Perchlorate            | 4   2                               | 42                             | 0.055         | 11.6%      | 0.0013                                | 0.0041                       | passed    |
| Trinexapac (free acid) | 10   0                              | 21                             | 0.118         | 14.7%      | 0.0047                                | 0.0089                       | passed    |
| Compound               | No. of not<br>detected  <br>Outlier | No. of<br>Numerical<br>Results | AV<br>[mg/kg] | CV*<br>[%] | Uncertainty<br>of AV (UAV)<br>[mg/kg] | UAV-<br>Tolerance<br>[mg/kg] | Judgement |
| Bromide ion            | 28   ?                              | 6                              | –             | –          | –                                     | –                            |           |
| Copper                 | 2   ?                               | 10                             | –             | –          | –                                     | –                            |           |

# Overall Performance I

## COMPULSORY / OPTIONAL / Extra Compounds

EU+EFTA

| Compound               | No. of Labs<br>(all) | FNs<br>(therein) | AAZ | CV*<br>[%] | 0%  20%  40% 60%  80% 100% |
|------------------------|----------------------|------------------|-----|------------|-------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|
| 2,4-D (free acid)      | 60                   | 0                | 0.6 | 18.9%      |                                                                                                                                                                                                  |
| BAC-C14 chloride       | 45                   | 3                | 1.0 | 19.0%      |                                                                                                                                                                                                  |
| Chlorate (anion)       | 48                   | 3                | 0.6 | 9.8%       |                                                                                                                                                                                                  |
| DDAC-C10 chloride      | 46                   | 2                | 1.0 | 25.0%      |                                                                                                                                                                                                  |
| Fluazifop (free acid)  | 59                   | 1                | 0.7 | 16.5%      |                                                                                                                                                                                                  |
| Glyphosate             | 64                   | 1                | 0.7 | 12.8%      |                                                                                                                                                                                                  |
| Matrine                | 40                   | 1                | 0.6 | 16.9%      |                                                                                                                                                                                                  |
| Nicotine               | 42                   | 2                | 1.1 | 24.8%      |                                                                                                                                                                                                 |
| Oxymatrine             | 36                   | 0                | 0.5 | 15.4%      |                                                                                                                                                                                                |
| Phosphonic acid        | 45                   | 1                | 0.9 | 20.9%      |                                                                                                                                                                                                |
| Perchlorate            | 60                   | 0                | 0.9 | 18.9%      |                                                                                                                                                                                                |
| Trinexapac (free acid) | 31                   | 10               | 1.6 | 14.7%      |                                                                                                                                                                                                |
| Bromide ion            | 34                   | 28               | 1.1 | —          |                                                                                                                                                                                                                                                                                     |
| Copper                 | 12                   | 2                | 1.5 | —          |                                                                                                                                                                                                                                                                                     |



### Rules for Category A:

EU+EFTA

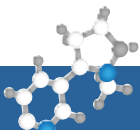
- Analysed for at least 13 out of 15 compulsory pesticides
- Correctly found at least 9 out of 10 compulsory pesticides present in test item
- No FPs among compulsory analytes

|            | No. of Labs | [%]  |
|------------|-------------|------|
| Category A | 28          | 39 % |
| Category B | 43          | 61 % |



# Use of ILIS

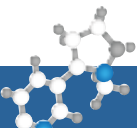
| Analytes               | NO | YES Spiked at... |               |                   |     |     | Total Results |
|------------------------|----|------------------|---------------|-------------------|-----|-----|---------------|
|                        |    | the beginning    | Final Extract | intermediate step | SUM | %   |               |
| Glyphosate             | 20 | 42               | 4             | 2                 | 48  | 71% | 68            |
| Perchlorate            | 19 | 26               | 3             |                   | 29  | 60% | 48            |
| Chlorate (anion)       | 20 | 27               | 3             |                   | 30  | 60% | 50            |
| Phosphonic acid        | 24 | 21               | 3             |                   | 24  | 50% | 48            |
| Nicotine               | 31 | 13               |               |                   | 13  | 30% | 44            |
| Matrine                | 34 | 9                |               |                   | 9   | 21% | 43            |
| 2,4-D (free acid)      | 56 | 7                |               |                   | 7   | 11% | 63            |
| DDAC-C10 chloride      | 43 | 5                |               |                   | 5   | 10% | 48            |
| Oxymatrine             | 35 | 4                |               |                   | 4   | 10% | 39            |
| BAC-C14 chloride       | 45 | 2                |               |                   | 2   | 4%  | 47            |
| Fluazifop (free acid)  | 61 | 1                |               |                   | 1   | 2%  | 62            |
| Trinexapac (free acid) | 32 |                  |               |                   | 0   | 0%  | 32            |



## Poor performance

- **25 EU/EFTA OfLs, 49 cases**  
(therein 28 cases of FNs)
- **Additionally „FN“ for Extra Compounds (natural content)**

| Bromide Ion | 28 cases | No MRRL set |
|-------------|----------|-------------|
| Copper      | 2 cases  | No MRRL set |



## ■ 21 EU/EFTA OfLs gave feedback for poor performance in 42 cases

| No. cases | Reasons                                                                                                                                                         |
|-----------|-----------------------------------------------------------------------------------------------------------------------------------------------------------------|
| 13        | <i>Lack of experience</i>                                                                                                                                       |
| 10        | <i>Others/Miscellaneous (e.g. Column equilibration, 1 injection in screening)</i>                                                                               |
| 9         | <i>Analytical procedure was appropriate but it was not properly performed (e.g. important component - e.g. water- not used, extraction time too short/long)</i> |
| 5         | <i>Misinterpretation / Misevaluation of measurement data / <b>Wrong target pesticide assumed</b></i>                                                            |
| 5         | <i>Inappropriate / erroneous calibration approach</i>                                                                                                           |
| 4         | <i>Analytical procedure was inappropriate</i>                                                                                                                   |
| 4         | <i>Calculation error<br/>(e.g. use of wrong factor, to express residue as required in PT; to address dilutions etc.)</i>                                        |
| 4         | <i>Deficient QC-measures that would have helped to recognize that method generates FNs, FPs or strongly biased results (e.g. no recovery test)</i>              |



# Thank You for Your Attention