

# Supporting Information for

## Multi-year interlaboratory exercises for the analysis of illicit drugs and metabolites in wastewater: development of a quality control system

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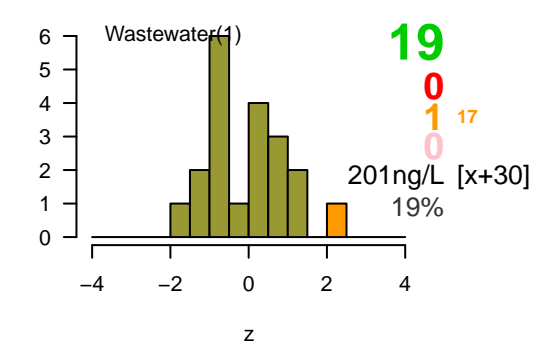
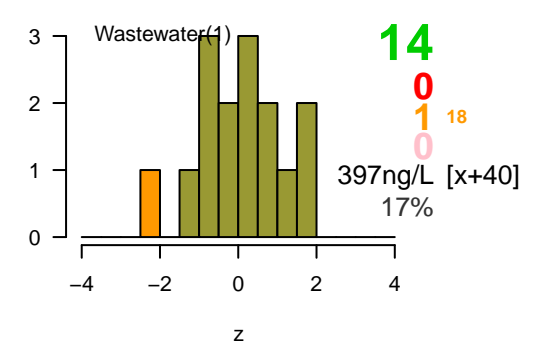
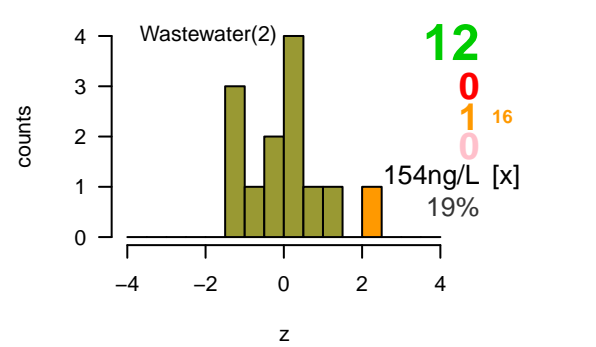
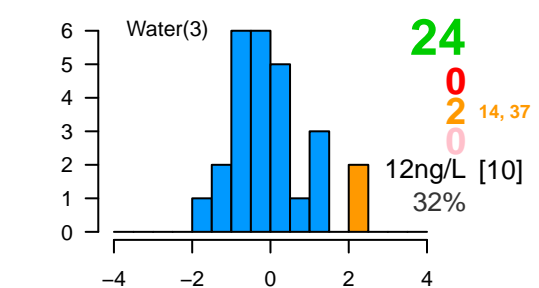
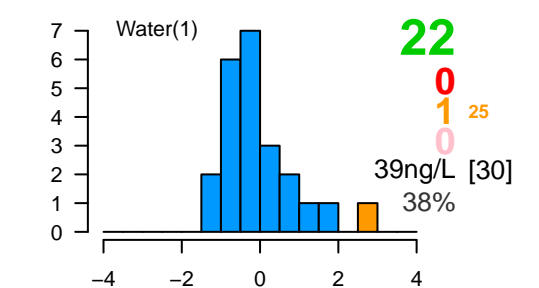
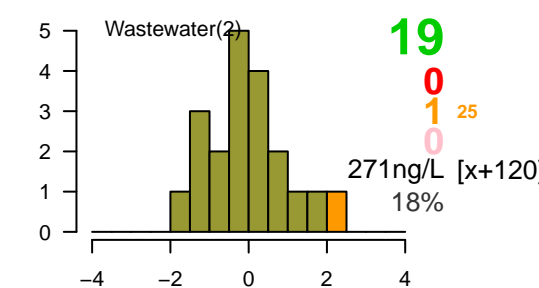
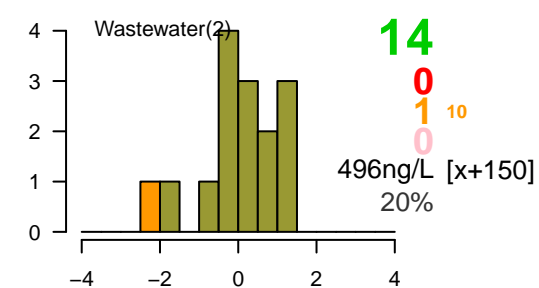
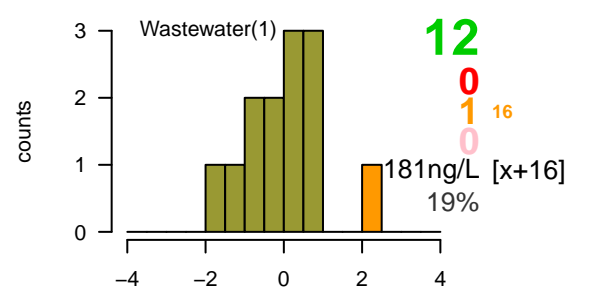
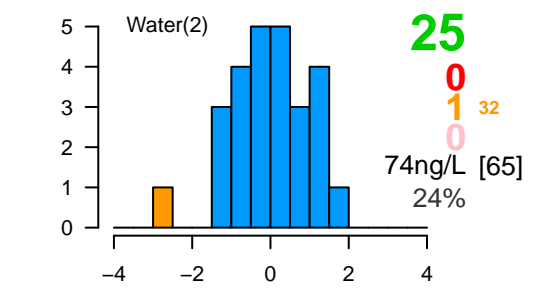
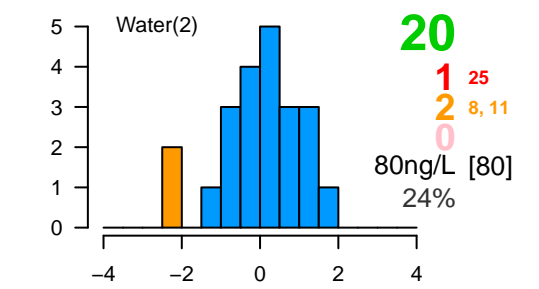
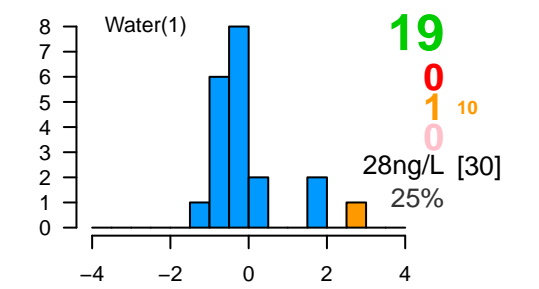
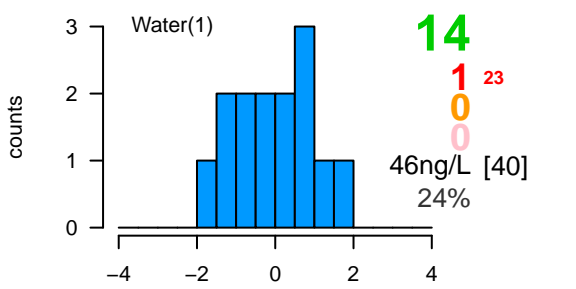
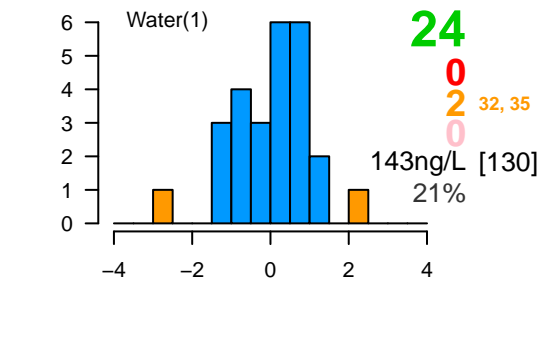
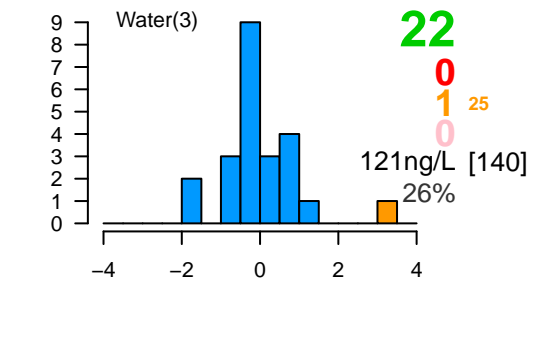
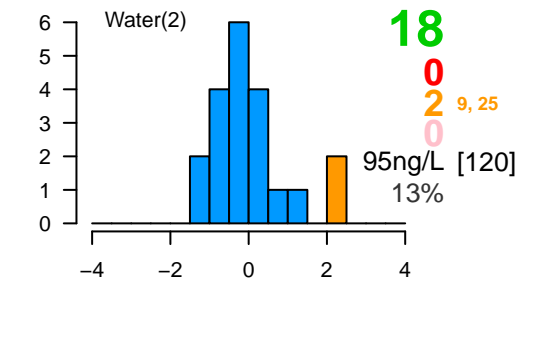
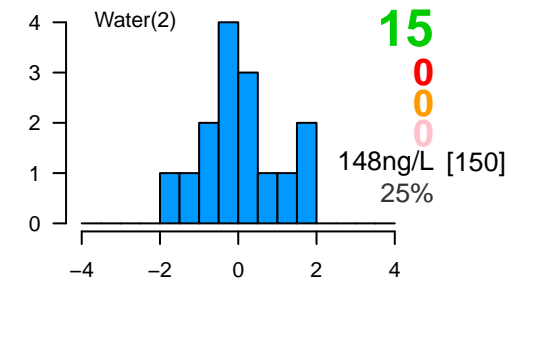
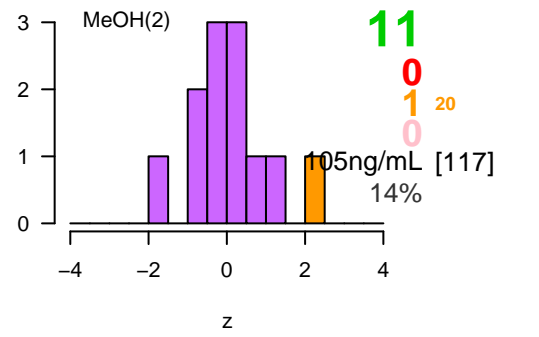
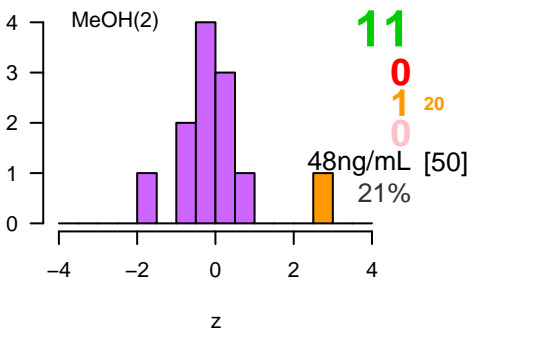
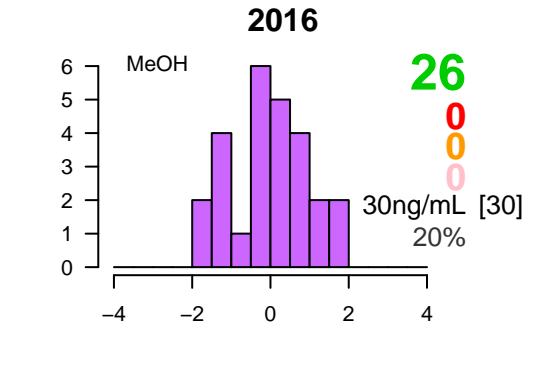
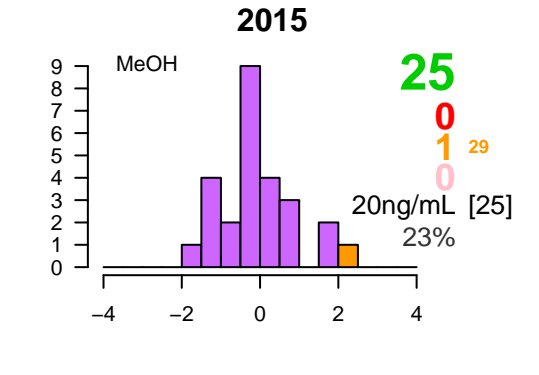
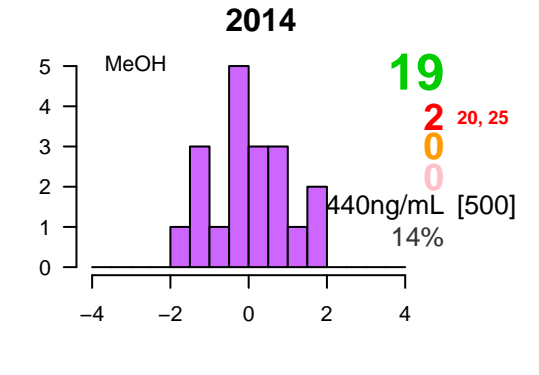
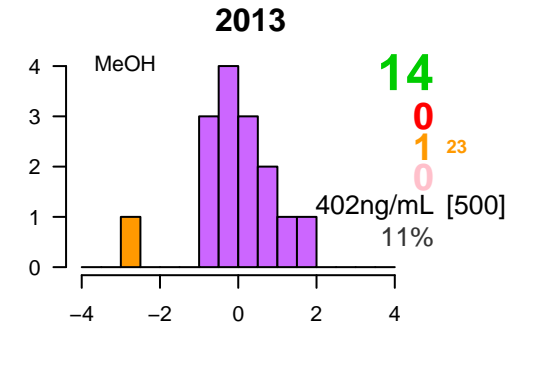
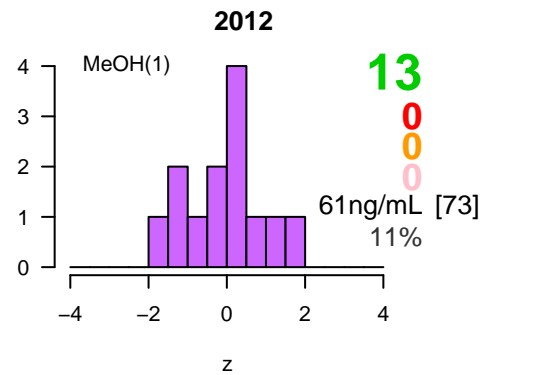
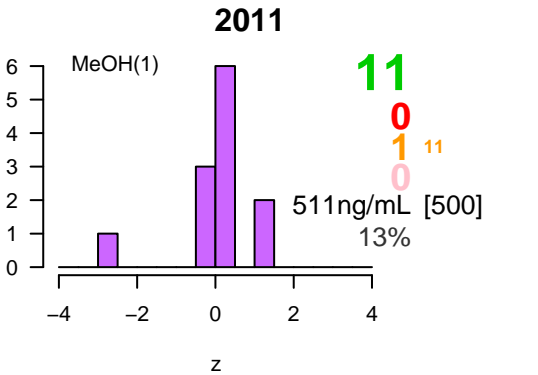
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### Content

| Substance                    | Overviews | Details   |
|------------------------------|-----------|---|
| Benzoylcgonine (BE).....     | p3 .....  | MeOH..... p10<br>Water (W) ..... p11<br>Wastewater (WW) ..... p12 |
| Cocaine (COC).....           | p4 .....  | MeOH..... p13<br>Water (W) ..... p14<br>Wastewater (WW) ..... p15 |
| MDMA.....                    | p5 .....  | MeOH..... p16<br>Water (W) ..... p17<br>Wastewater (WW) ..... p18 |
| Amphetamine (AMP) .....      | p6 .....  | MeOH..... p19<br>Water (W) ..... p20<br>Wastewater (WW) ..... p21 |
| Methamphetamine (METH) ..... | p7 .....  | MeOH..... p22<br>Water (W) ..... p23<br>Wastewater (WW) ..... p24 |
| THC-COOH.....                | p8 .....  | MeOH..... p25<br>Water (W) ..... p26<br>Wastewater (WW) ..... p27 |
| 6-MAM .....                  | p9 .....  | MeOH..... p28<br>Water (W) ..... p29<br>Wastewater (WW) ..... p30 |



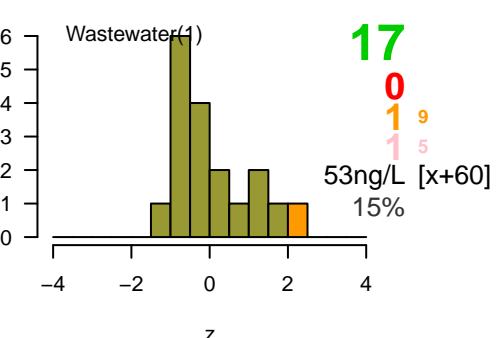
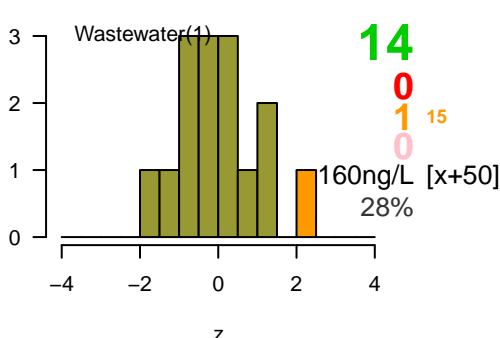
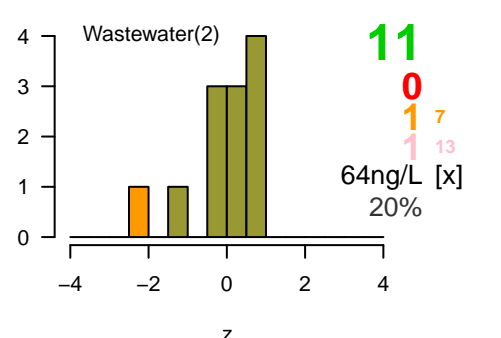
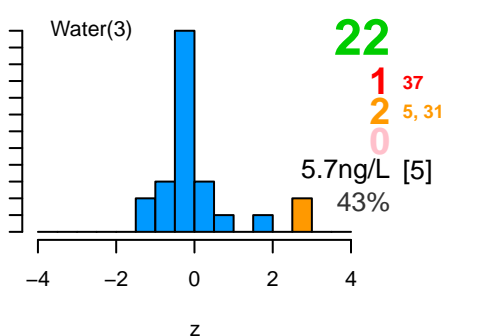
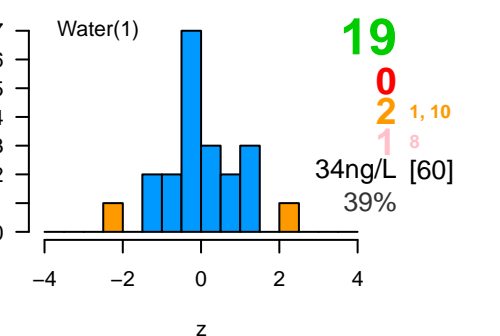
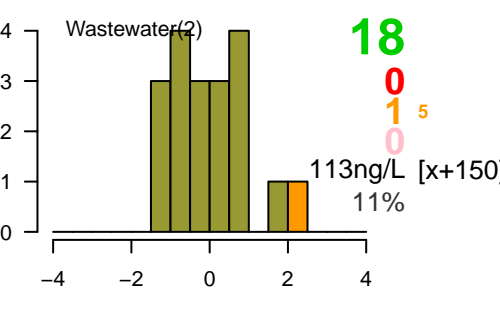
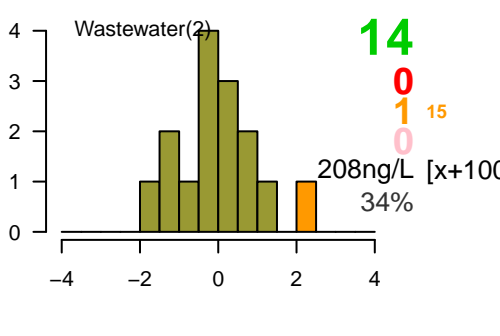
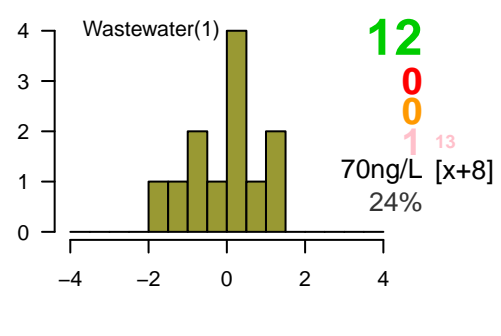
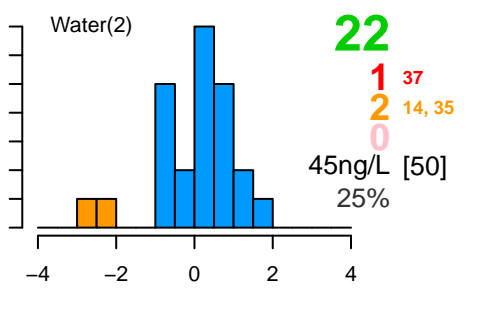
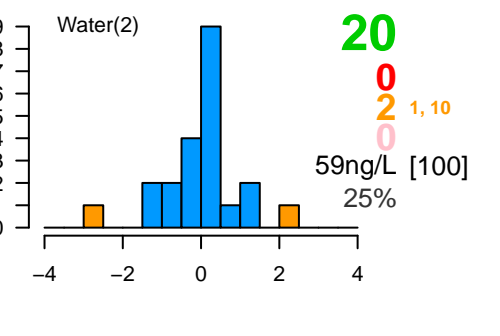
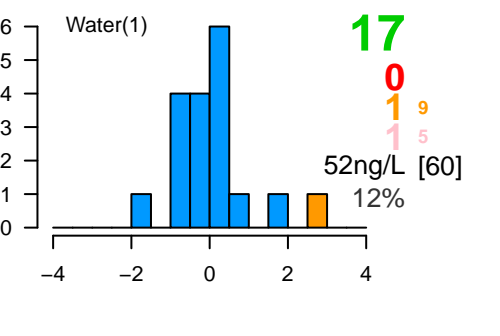
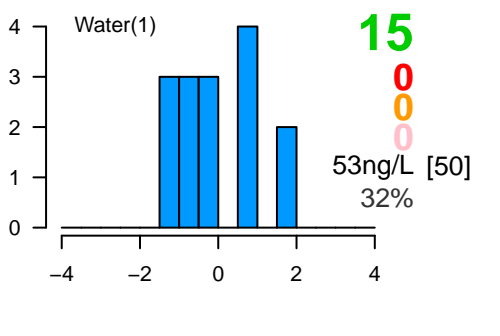
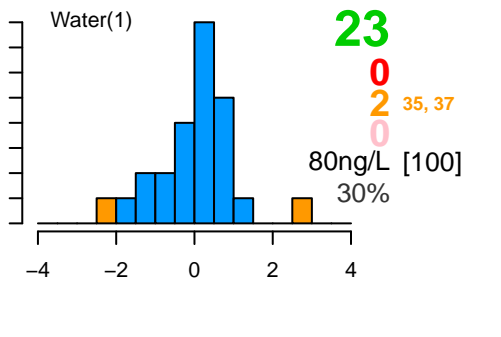
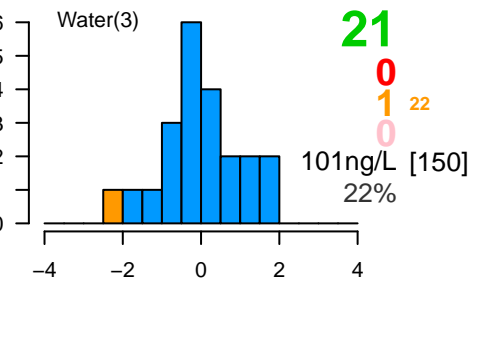
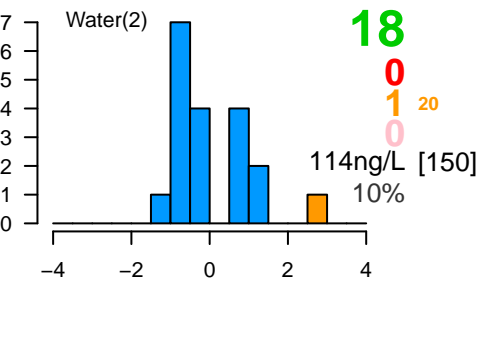
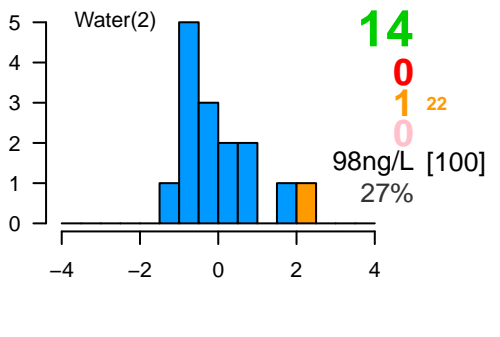
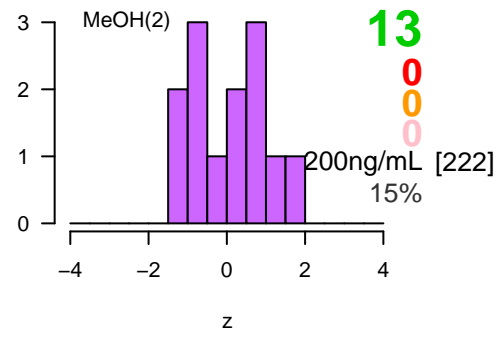
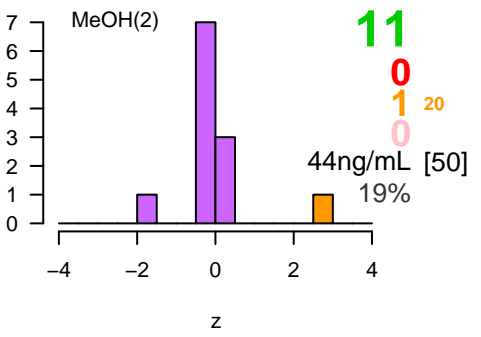
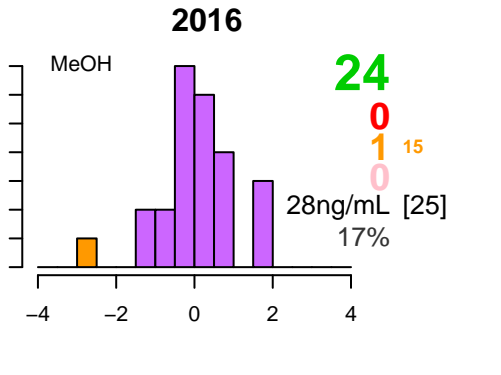
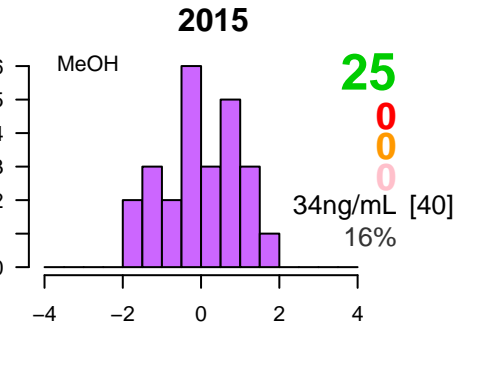
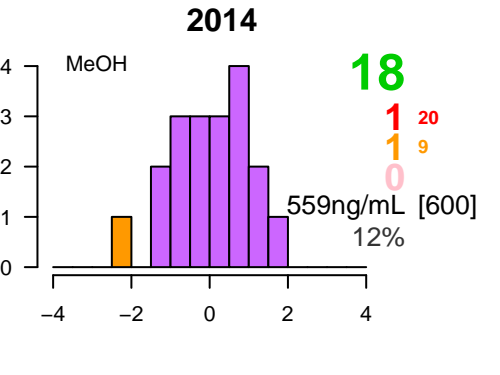
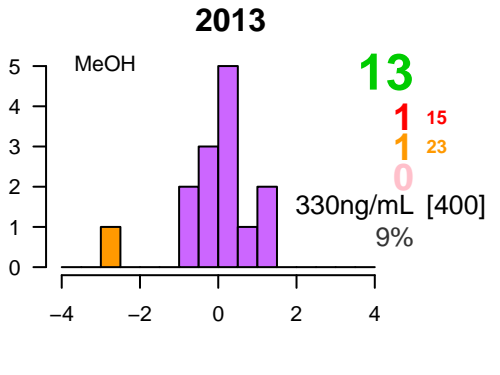
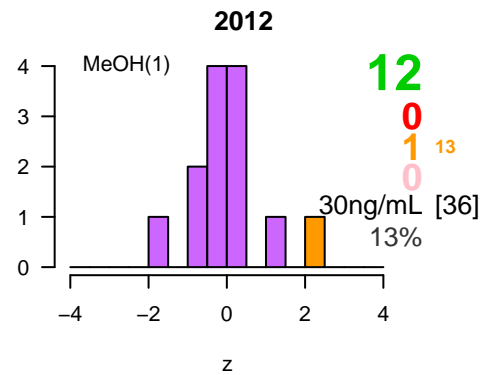
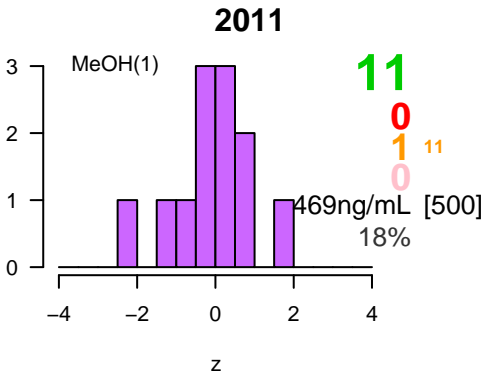
# Benzylecgonine

nr. of labs

passed

outliers lab ID(s)  
 $|z| > 2$  lab ID(s)  
 $< LOQ$  lab ID(s)

mean of all labs  $|z| < 2$  [spike level]  
 rsd of all labs  $|z| < 2$



# Cocaine

nr. of labs

passed

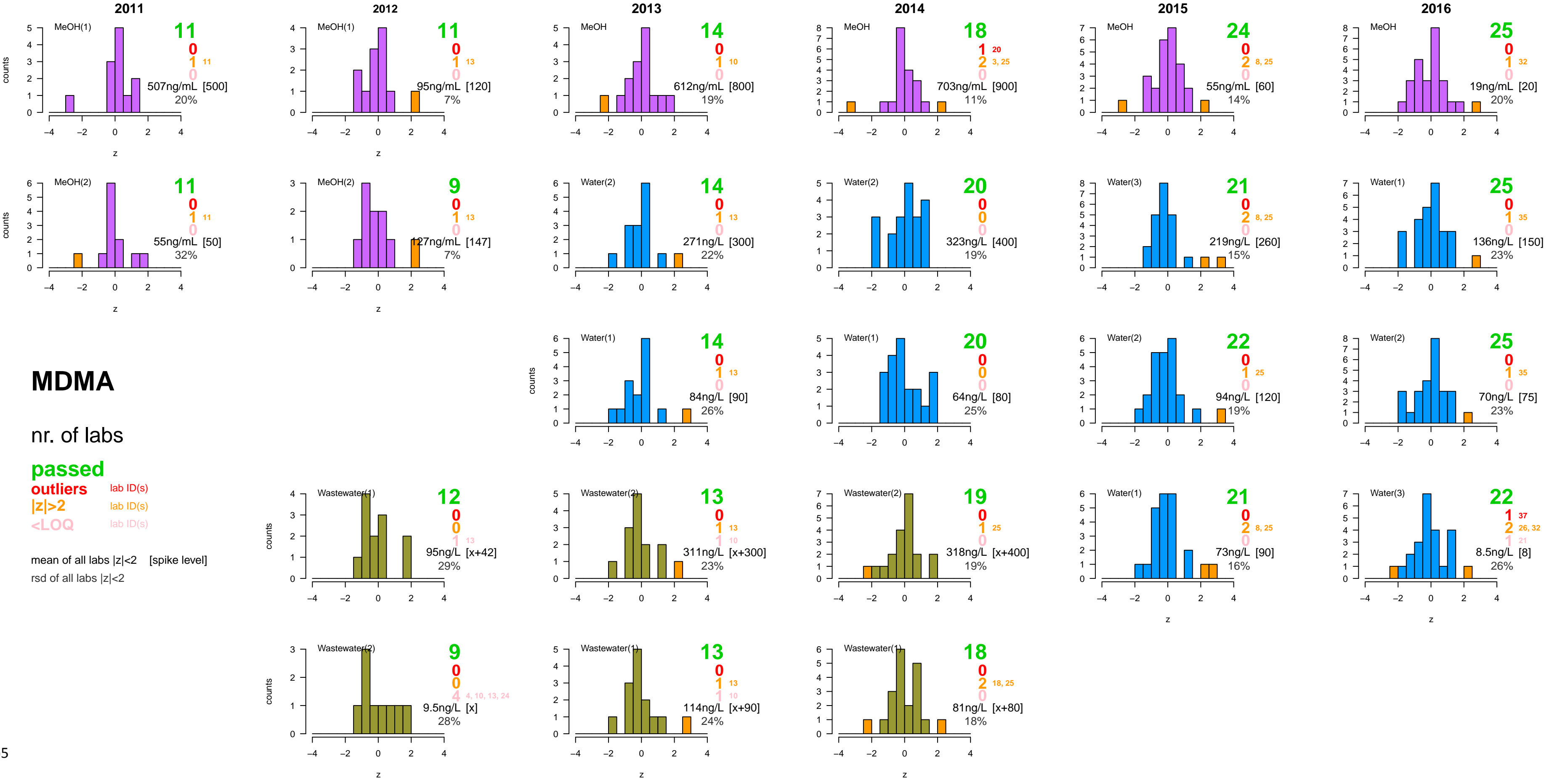
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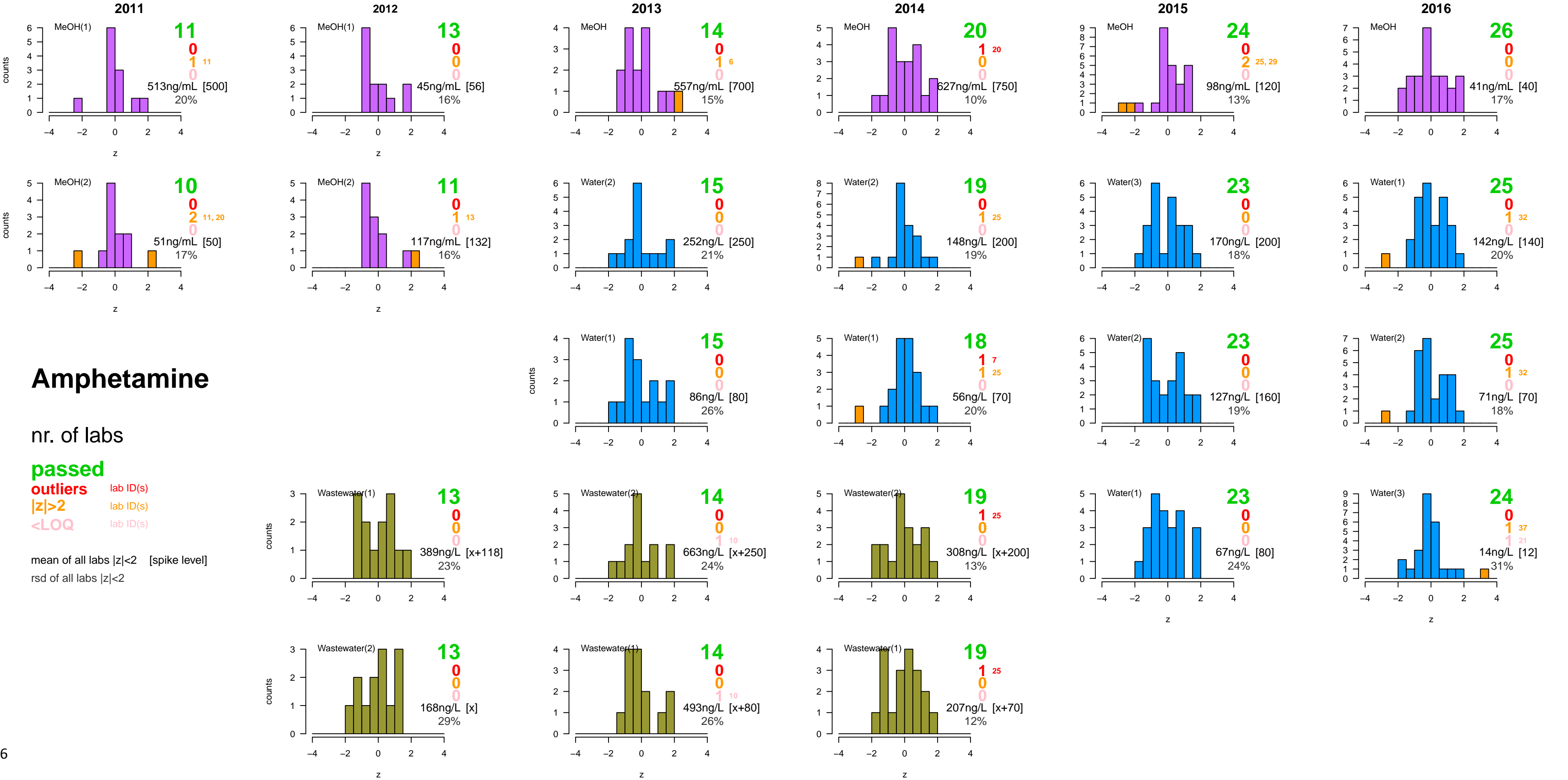
|z|>2 lab ID(s)

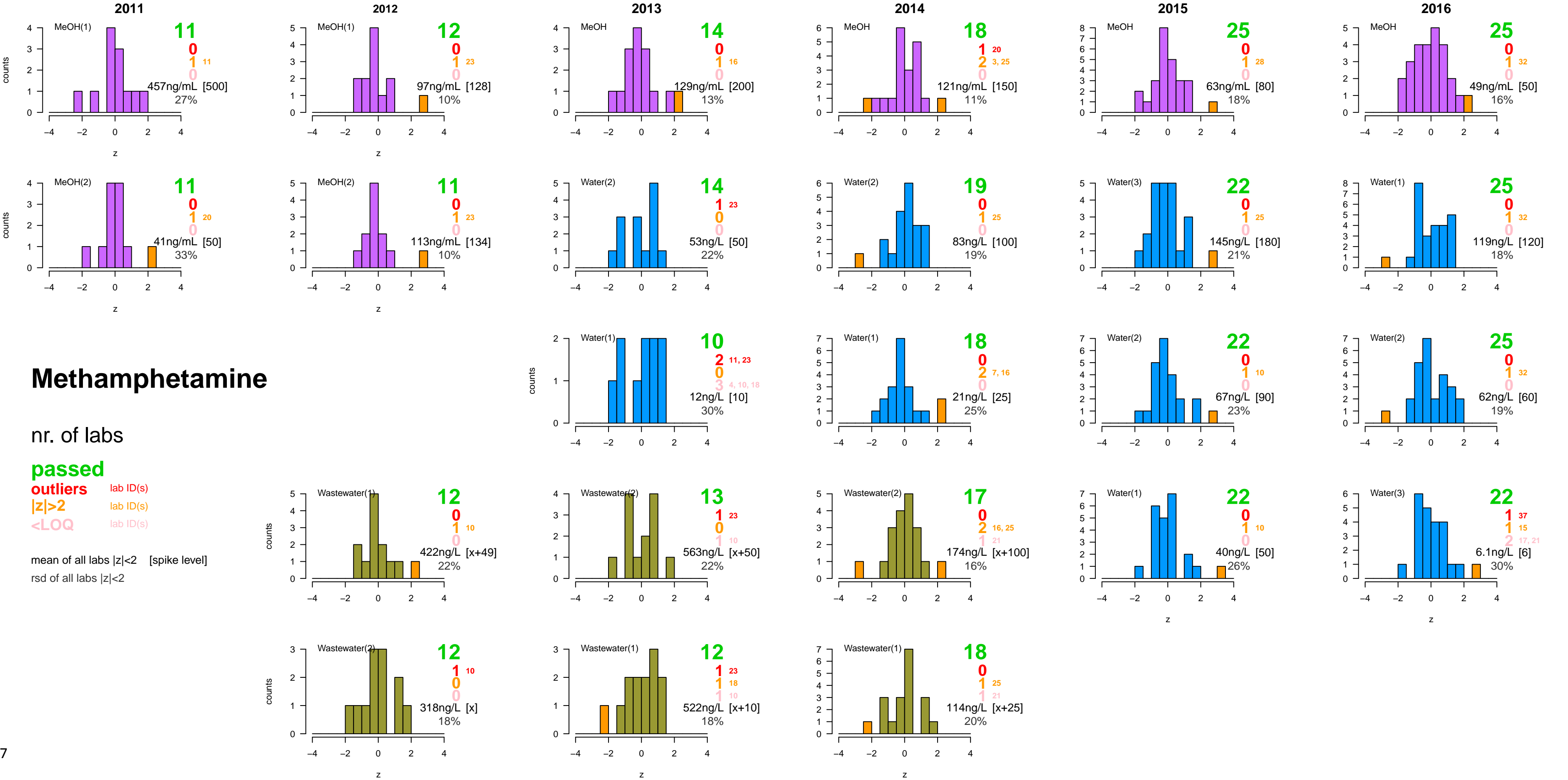
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mean of all labs |z|<2 [spike level]

rsd of all labs |z|<2





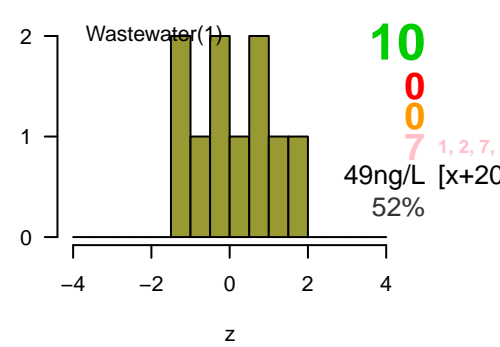
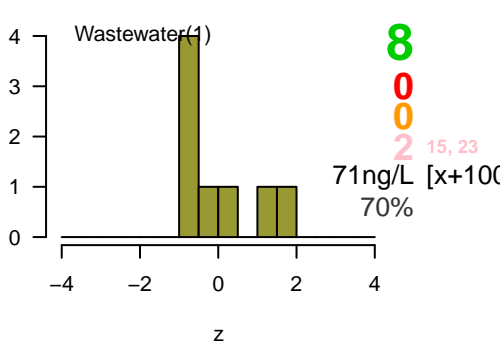
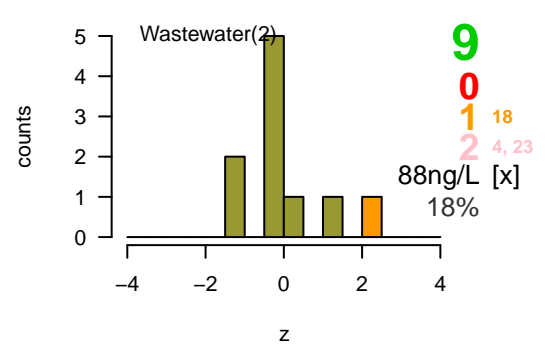
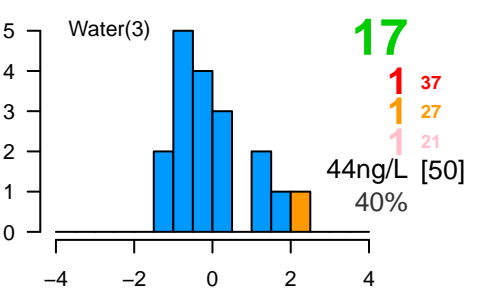
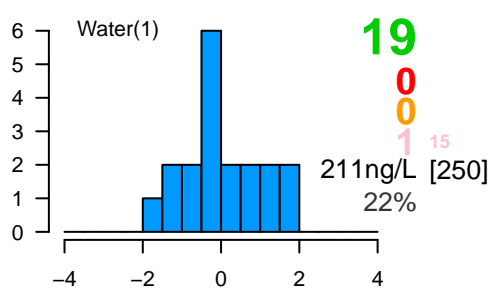
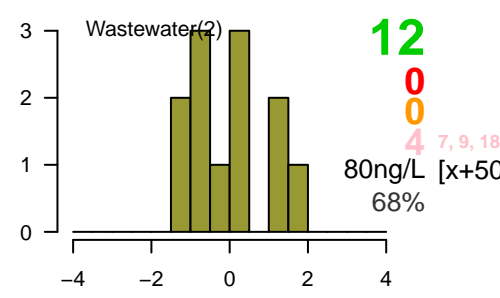
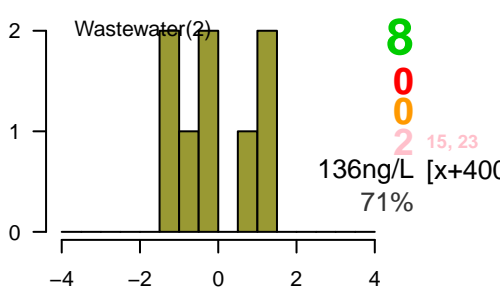
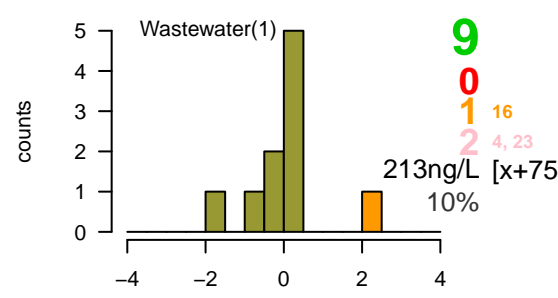
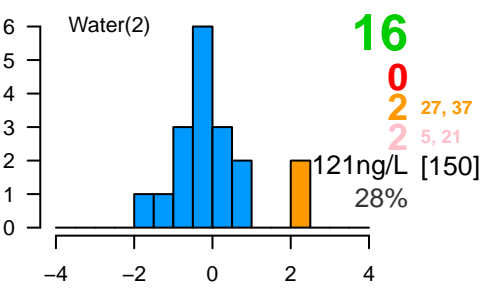
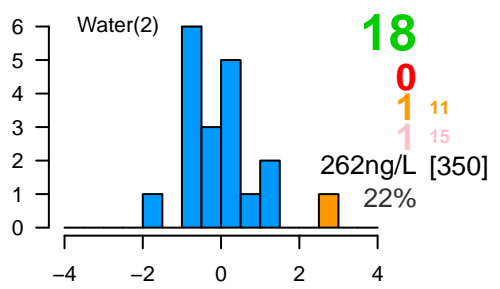
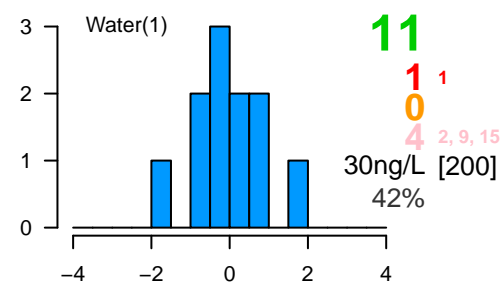
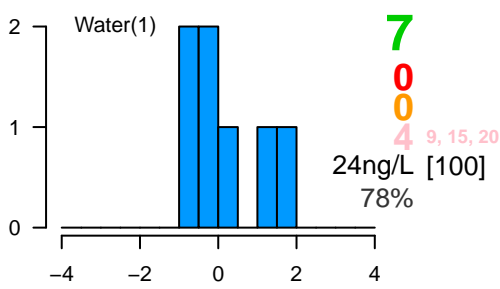
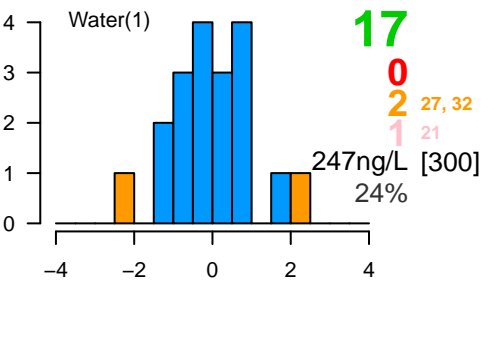
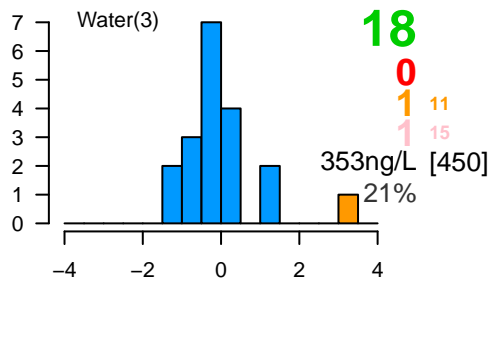
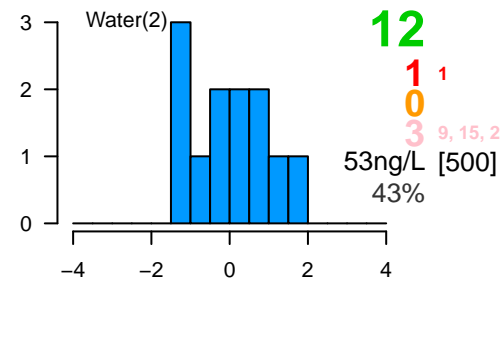
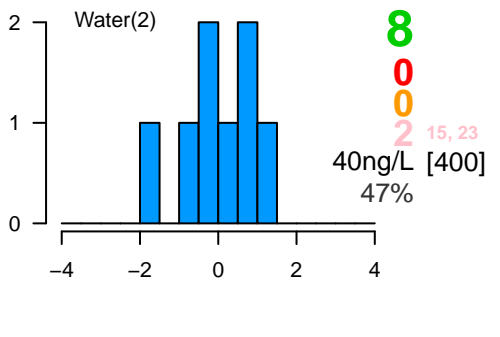
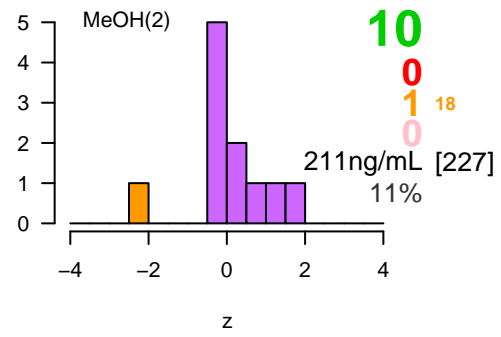
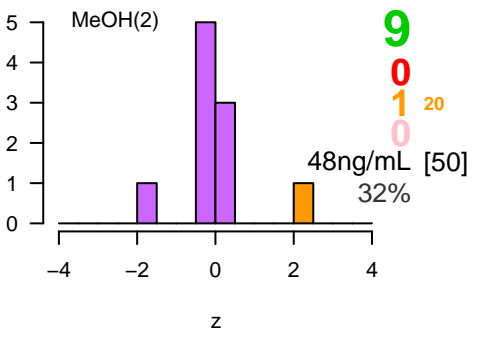
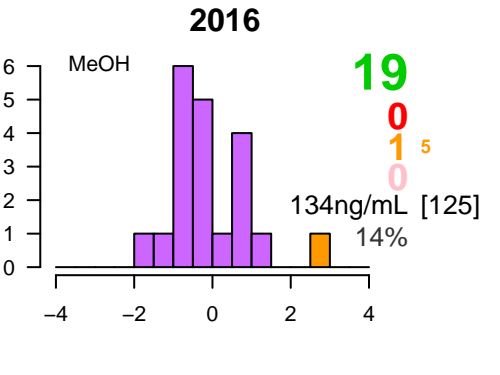
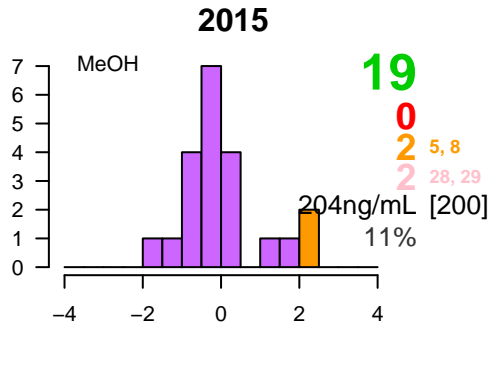
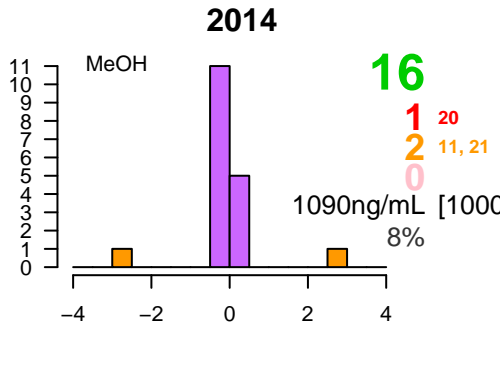
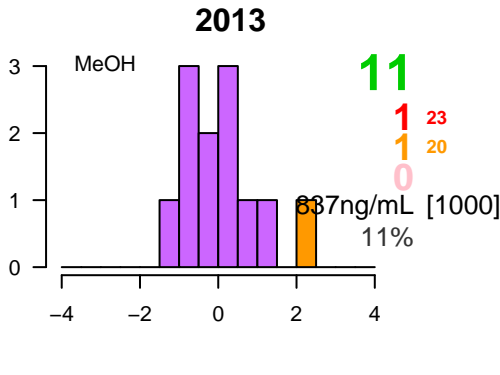
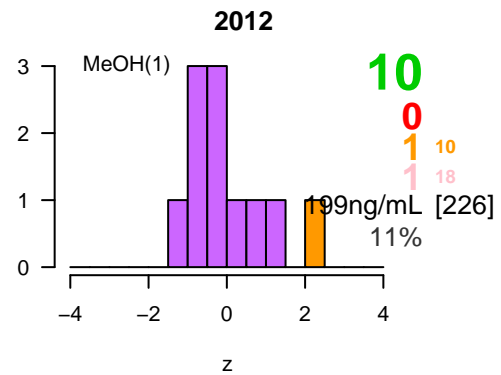
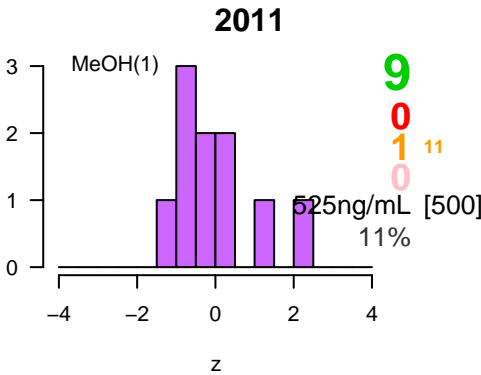


# Methamphetamine

nr. of labs

passed  
 outliers lab ID(s)  
 $|z| > 2$  lab ID(s)  
 $< LOQ$  lab ID(s)

mean of all labs  $|z| < 2$  [spike level]  
 rsd of all labs  $|z| < 2$



# THC-COOH

nr. of labs

passed

outliers lab ID(s)

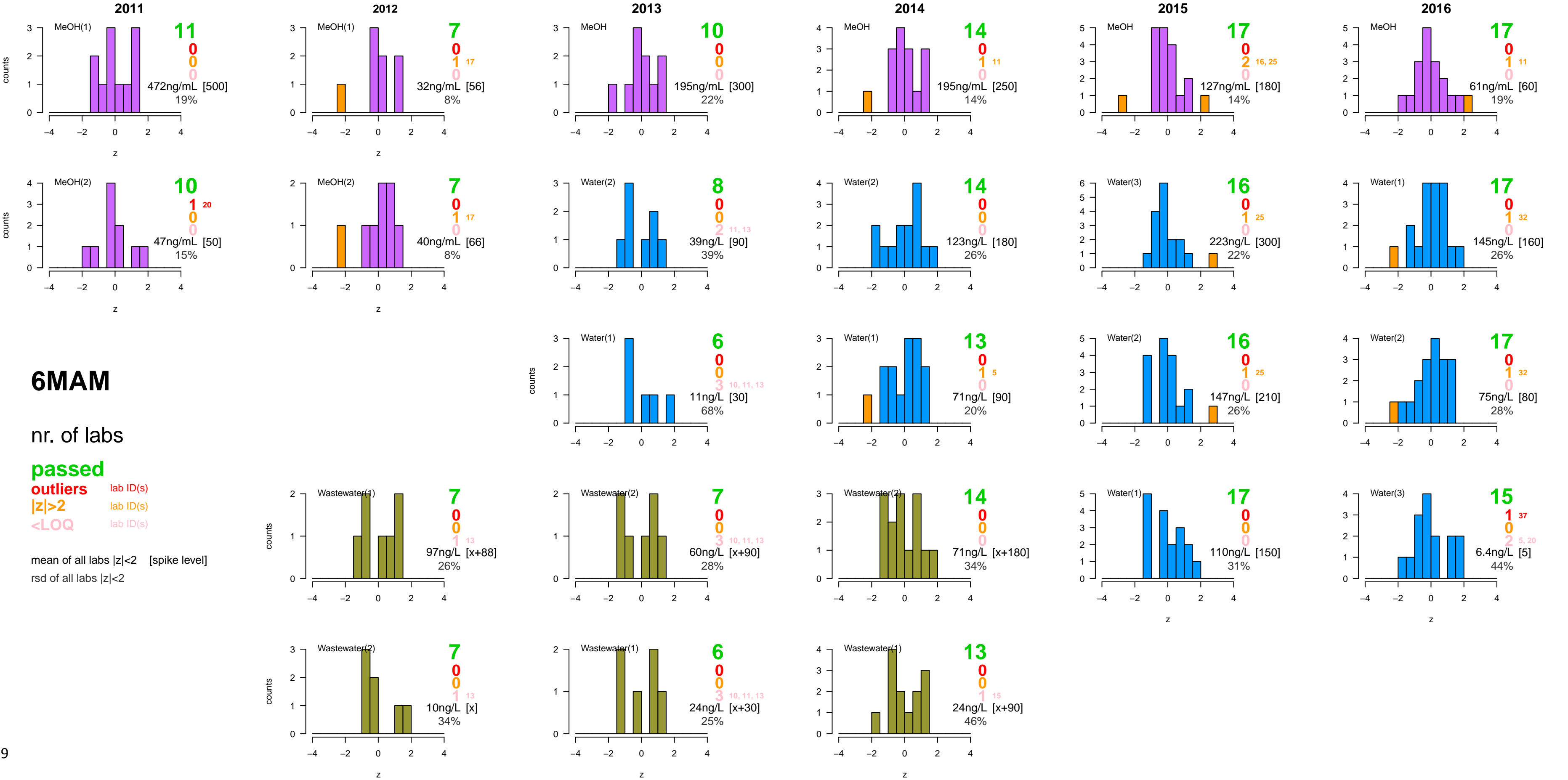
|z|>2 lab ID(s)

<LOQ lab ID(s)

mean of all labs |z|<2 [spike level]

rsd of all labs |z|<2





# BE MeOH(1)

normalized concentrations (with mean of means per year after removing outliers)

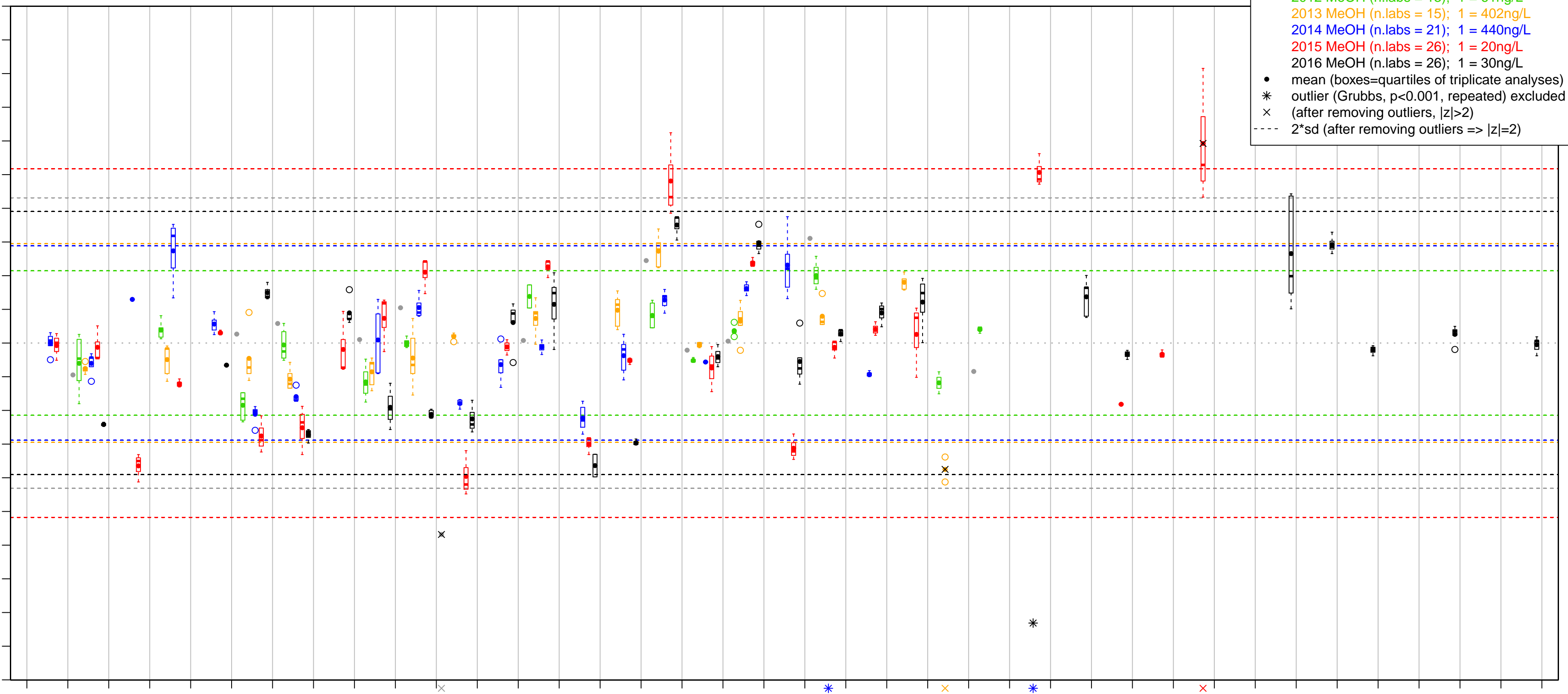
2.0  
1.5  
1.0  
0.5  
0.0

- 2011 MeOH (n.labs = 12); 1 = 511ng/L
- 2012 MeOH (n.labs = 13); 1 = 61ng/L
- 2013 MeOH (n.labs = 15); 1 = 402ng/L
- 2014 MeOH (n.labs = 21); 1 = 440ng/L
- 2015 MeOH (n.labs = 26); 1 = 20ng/L
- 2016 MeOH (n.labs = 26); 1 = 30ng/L
- mean (boxes=quartiles of triplicate analyses)
- \* outlier (Grubbs,  $p < 0.001$ , repeated) excluded
- × (after removing outliers,  $|z| > 2$ )
- - - 2\*sd (after removing outliers =>  $|z| = 2$ )

1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17 18 19 20 21 22 23 24 25 26 27 28 29 30 31 32 33 34 35 36 37

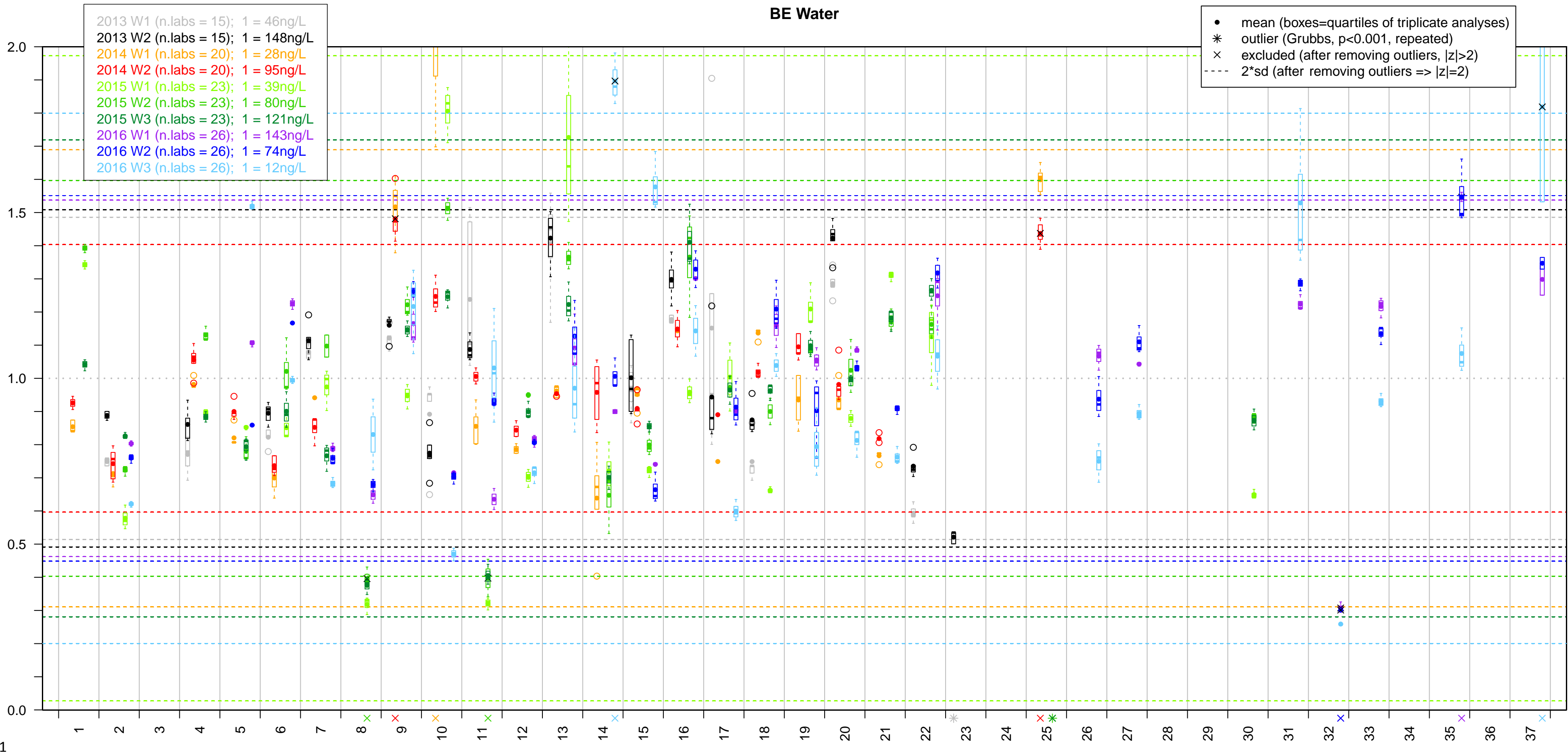
Lab ID

p10



# BE Water

normalized concentrations (with mean of means per year after removing outliers)



p11

Lab ID

# BE Wastewater

normalized concentrations (with mean of means per year after removing outliers)

2.0  
1.5  
1.0  
0.5  
0.0

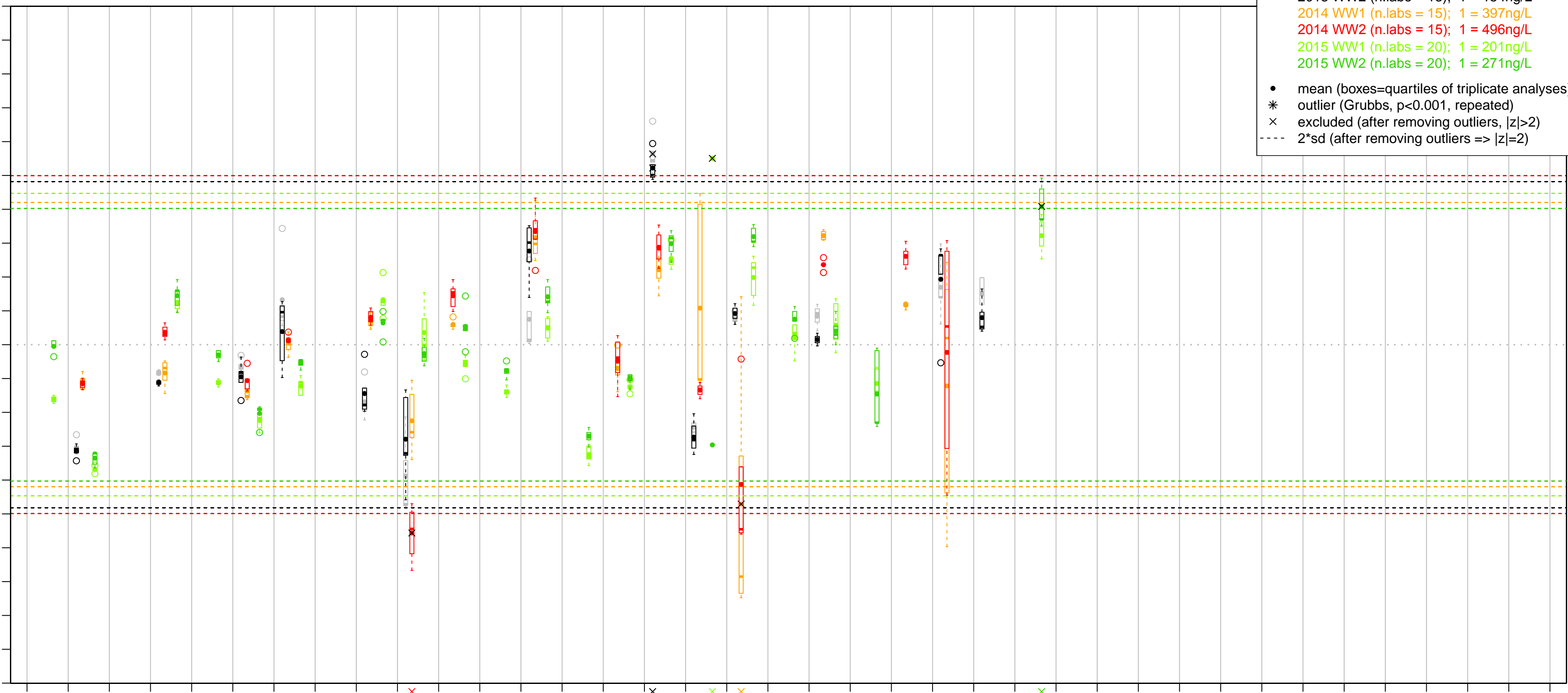
2013 WW1 (n.labs = 13); 1 = 181ng/L  
2013 WW2 (n.labs = 13); 1 = 154ng/L  
2014 WW1 (n.labs = 15); 1 = 397ng/L  
2014 WW2 (n.labs = 15); 1 = 496ng/L  
2015 WW1 (n.labs = 20); 1 = 201ng/L  
2015 WW2 (n.labs = 20); 1 = 271ng/L

- mean (boxes=quartiles of triplicate analyses)
- \* outlier (Grubbs,  $p < 0.001$ , repeated)
- × excluded (after removing outliers,  $|z| > 2$ )
- - -  $2 \cdot sd$  (after removing outliers  $\Rightarrow |z| = 2$ )

1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17 18 19 20 21 22 23 24 25 26 27 28 29 30 31 32 33 34 35 36 37

Lab ID

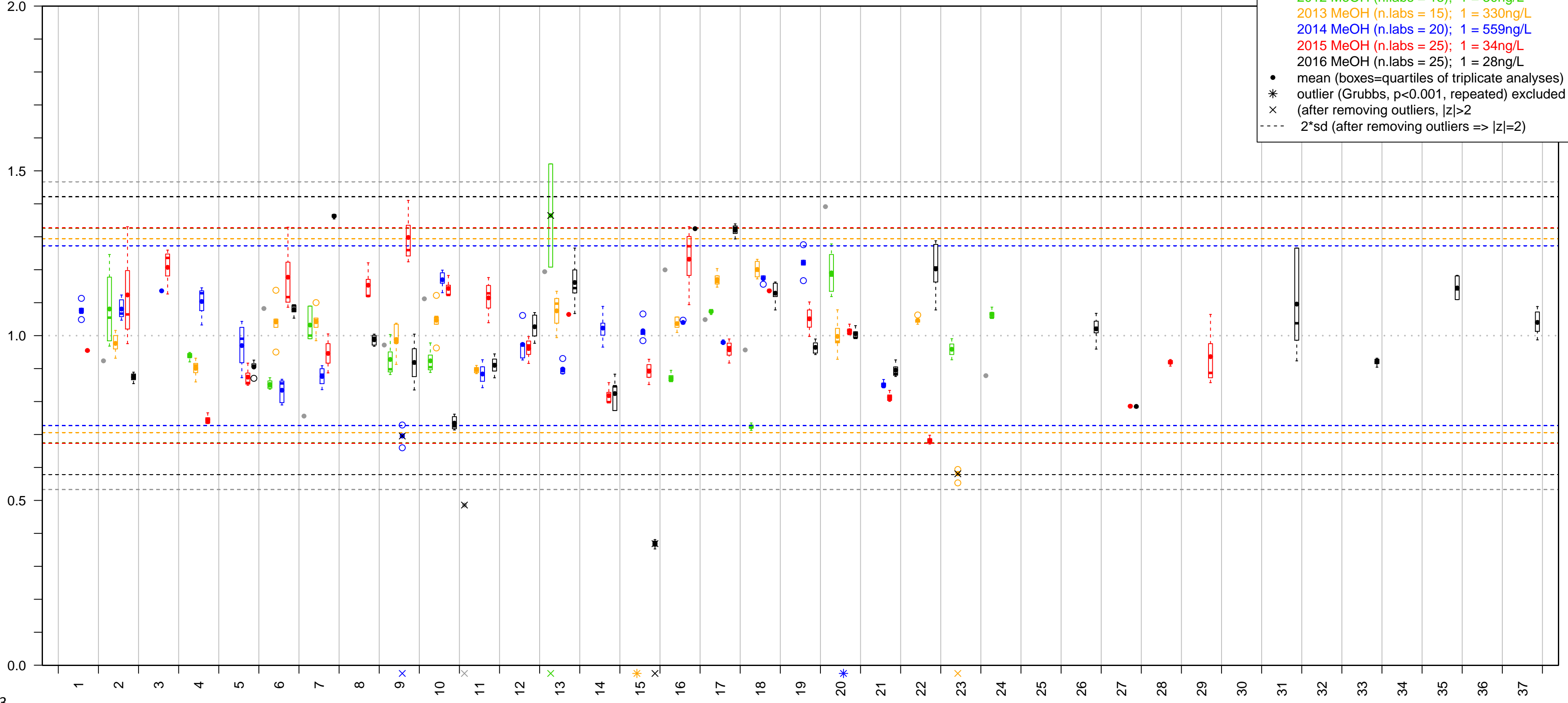
p12



# COC MeOH(1)

normalized concentrations (with mean of means per year after removing outliers)

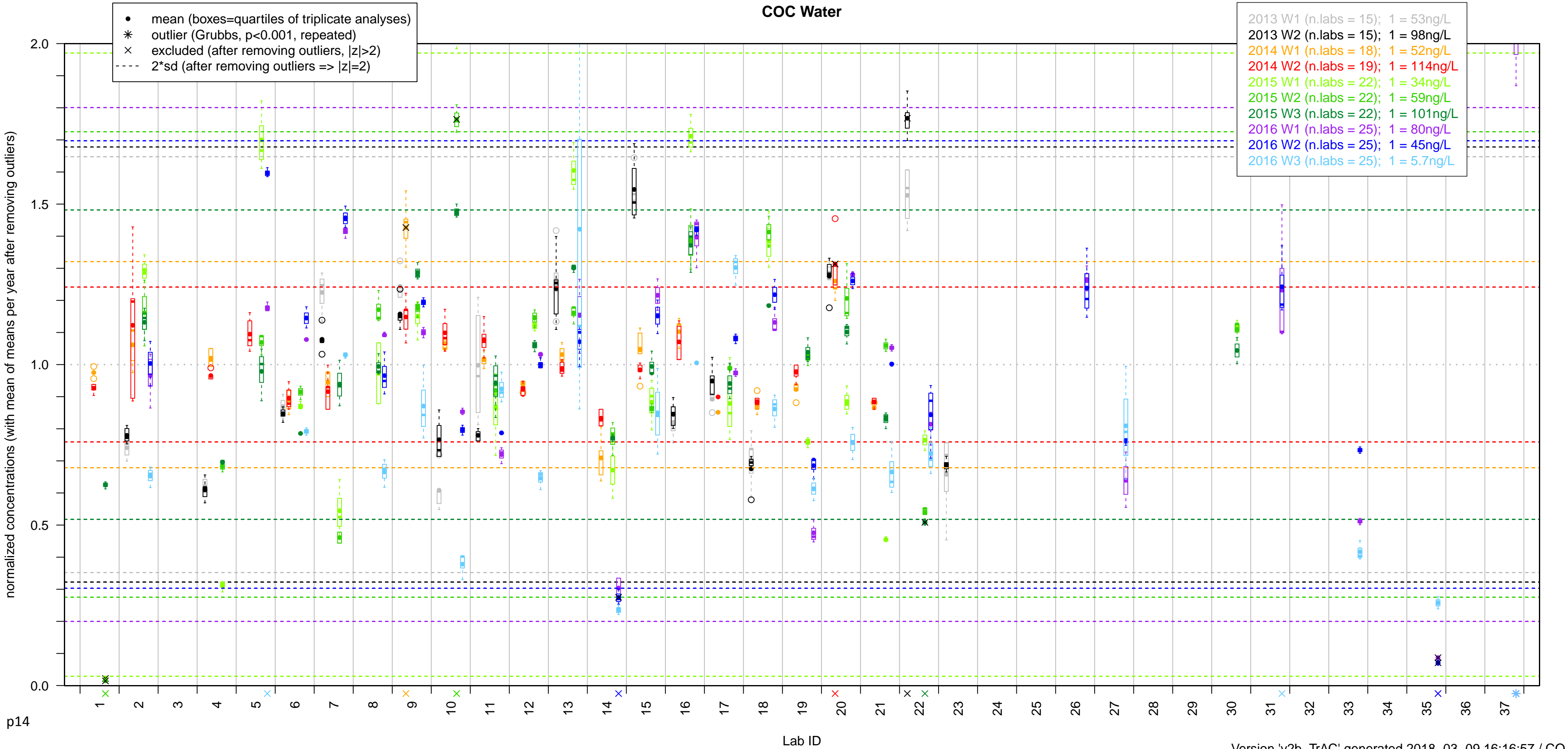
- 2011 MeOH (n.labs = 12); 1 = 469ng/L
- 2012 MeOH (n.labs = 13); 1 = 30ng/L
- 2013 MeOH (n.labs = 15); 1 = 330ng/L
- 2014 MeOH (n.labs = 20); 1 = 559ng/L
- 2015 MeOH (n.labs = 25); 1 = 34ng/L
- 2016 MeOH (n.labs = 25); 1 = 28ng/L
- mean (boxes=quartiles of triplicate analyses)
- \* outlier (Grubbs,  $p < 0.001$ , repeated) excluded
- × (after removing outliers,  $|z| > 2$ )
- - - 2\*sd (after removing outliers =>  $|z| = 2$ )



p13

Lab ID

# COC Water



p14

# COC Wastewater

normalized concentrations (with mean of means per year after removing outliers)

2.0  
1.5  
1.0  
0.5  
0.0

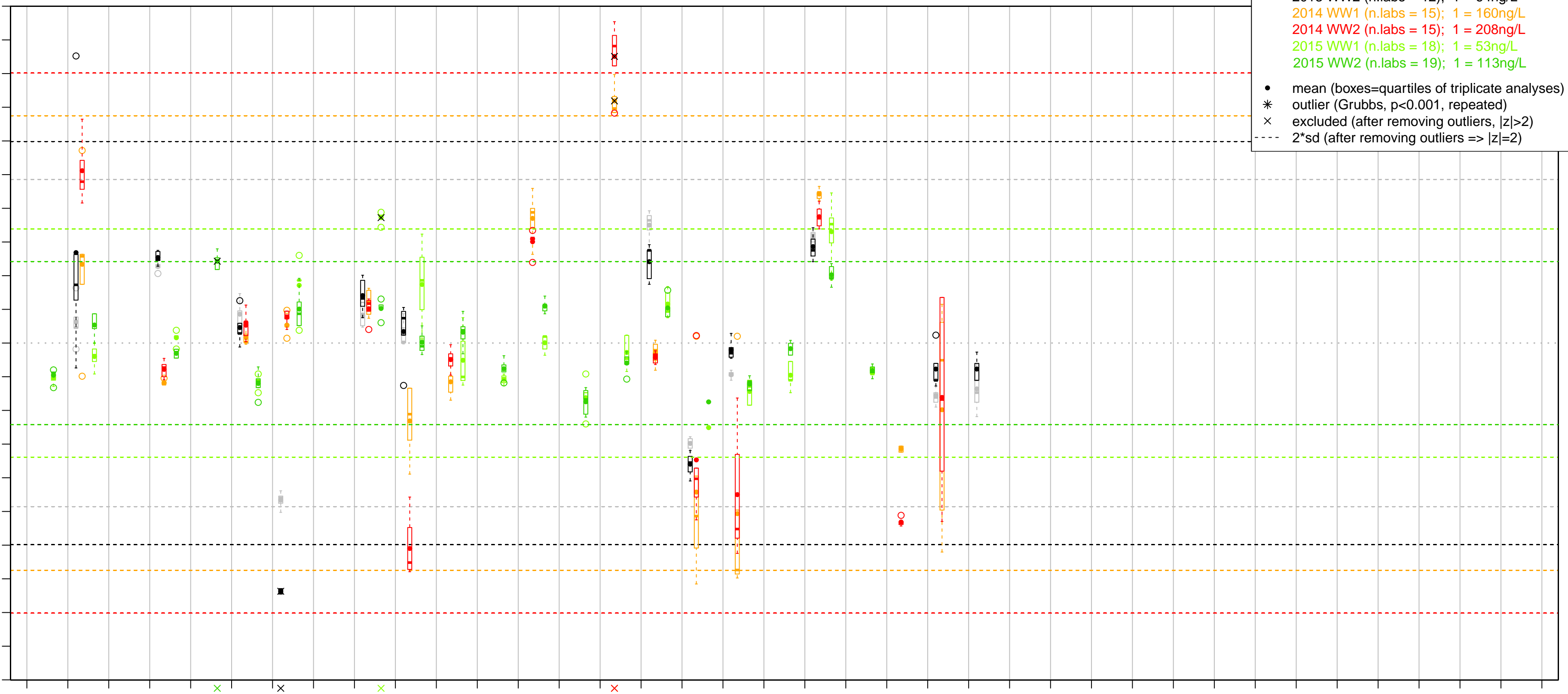
2013 WW1 (n.labs = 12); 1 = 70ng/L  
2013 WW2 (n.labs = 12); 1 = 64ng/L  
2014 WW1 (n.labs = 15); 1 = 160ng/L  
2014 WW2 (n.labs = 15); 1 = 208ng/L  
2015 WW1 (n.labs = 18); 1 = 53ng/L  
2015 WW2 (n.labs = 19); 1 = 113ng/L

- mean (boxes=quartiles of triplicate analyses)
- \* outlier (Grubbs, p<0.001, repeated)
- × excluded (after removing outliers, |z|>2)
- - - 2\*sd (after removing outliers => |z|=2)

1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17 18 19 20 21 22 23 24 25 26 27 28 29 30 31 32 33 34 35 36 37

Lab ID

p15



# MDMA MeOH(1)

normalized concentrations (with mean of means per year after removing outliers)

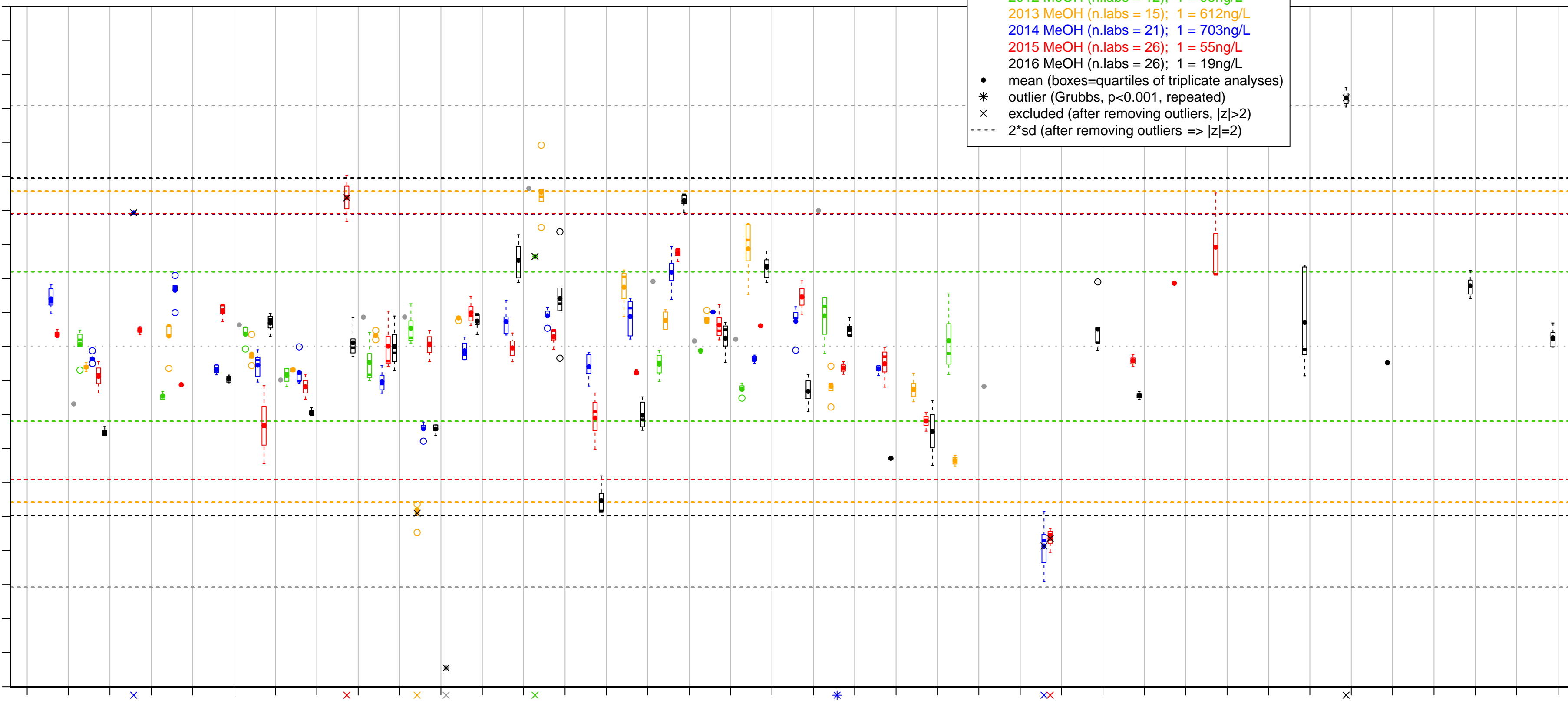
2.0  
1.5  
1.0  
0.5  
0.0

- 2011 MeOH (n.labs = 12); 1 = 507ng/L
- 2012 MeOH (n.labs = 12); 1 = 95ng/L
- 2013 MeOH (n.labs = 15); 1 = 612ng/L
- 2014 MeOH (n.labs = 21); 1 = 703ng/L
- 2015 MeOH (n.labs = 26); 1 = 55ng/L
- 2016 MeOH (n.labs = 26); 1 = 19ng/L
- mean (boxes=quartiles of triplicate analyses)
- \* outlier (Grubbs,  $p < 0.001$ , repeated)
- × excluded (after removing outliers,  $|z| > 2$ )
- - -  $2 \cdot sd$  (after removing outliers  $\Rightarrow |z| = 2$ )

1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17 18 19 20 21 22 23 24 25 26 27 28 29 30 31 32 33 34 35 36 37

Lab ID

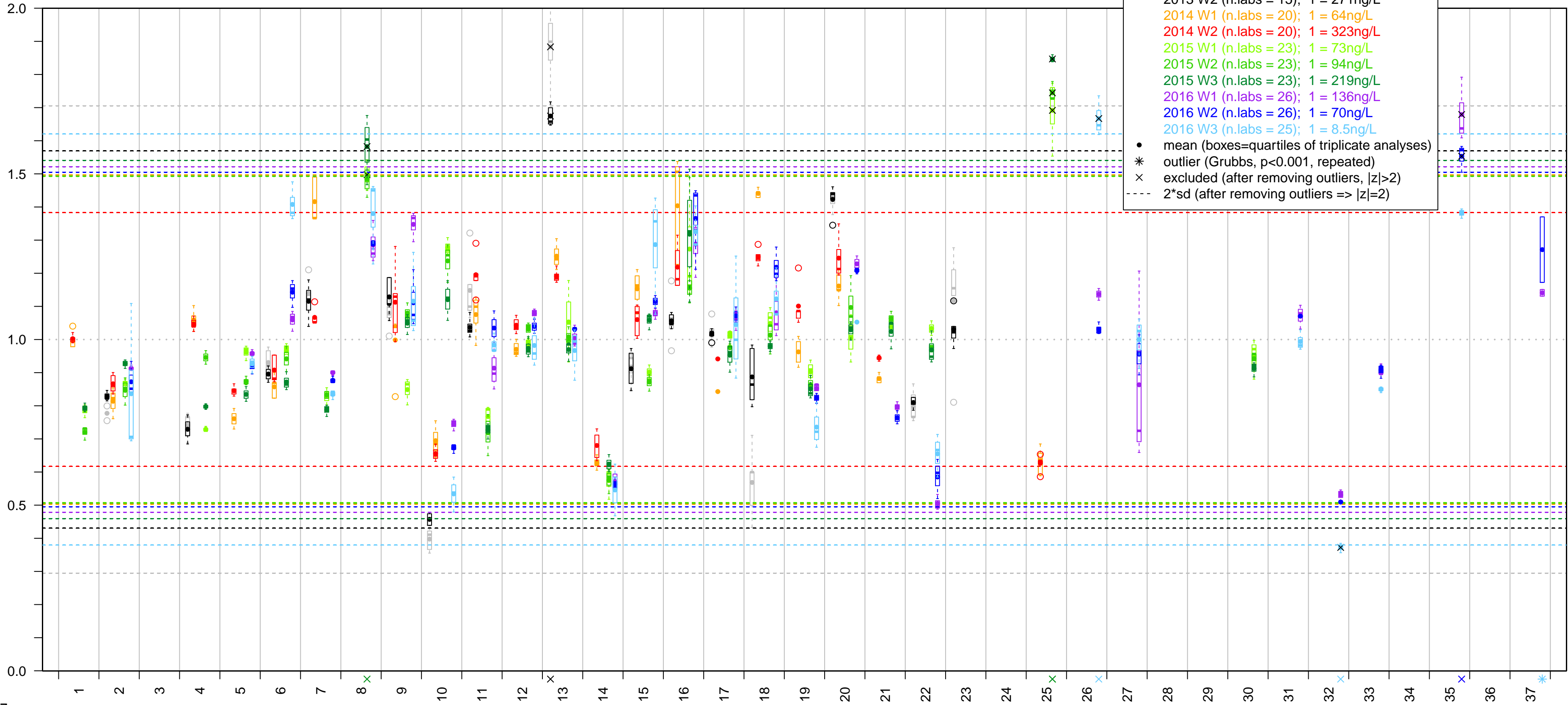
p16





# MDMA Water

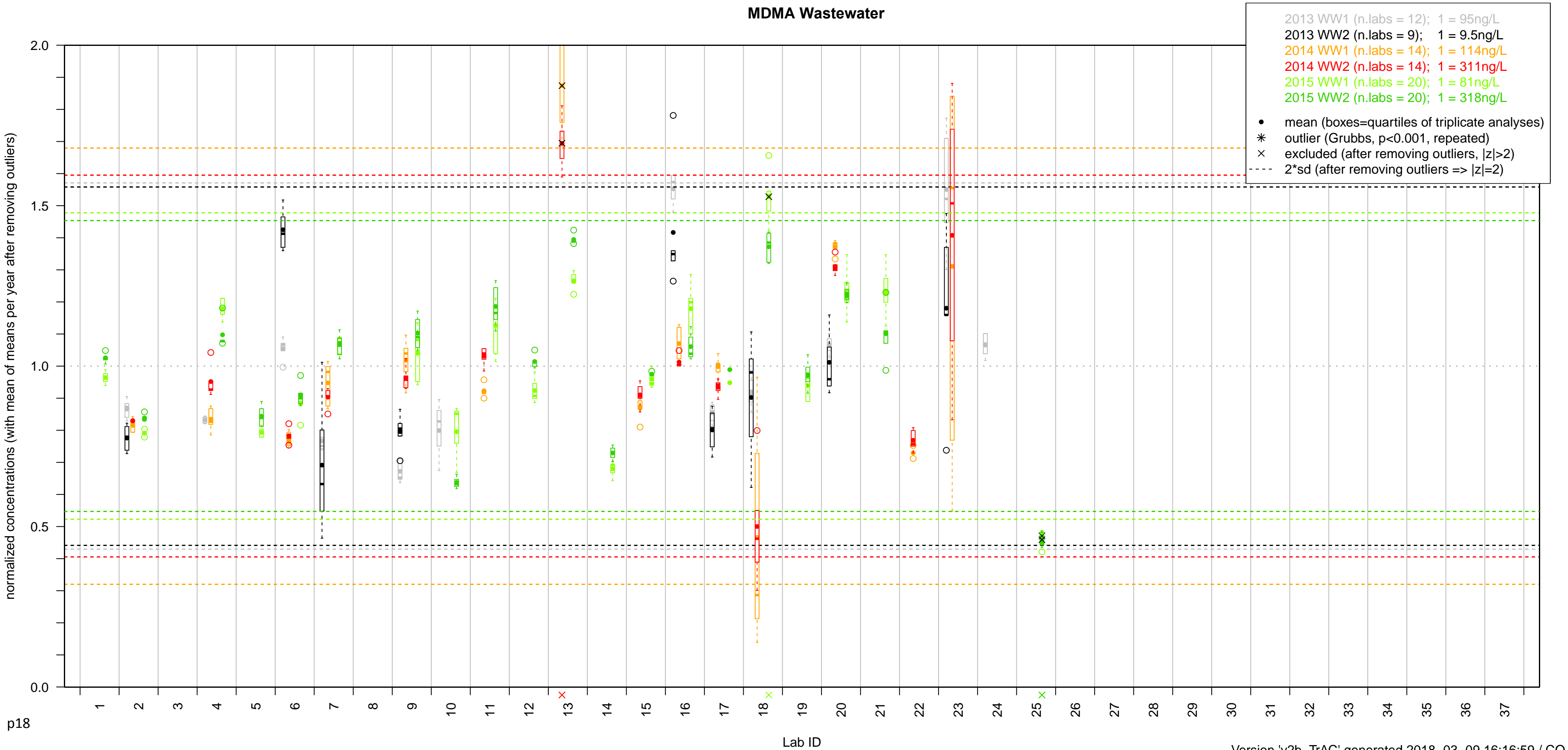
normalized concentrations (with mean of means per year after removing outliers)



p17

Lab ID

# MDMA Wastewater



# AMP MeOH(1)

normalized concentrations (with mean of means per year after removing outliers)

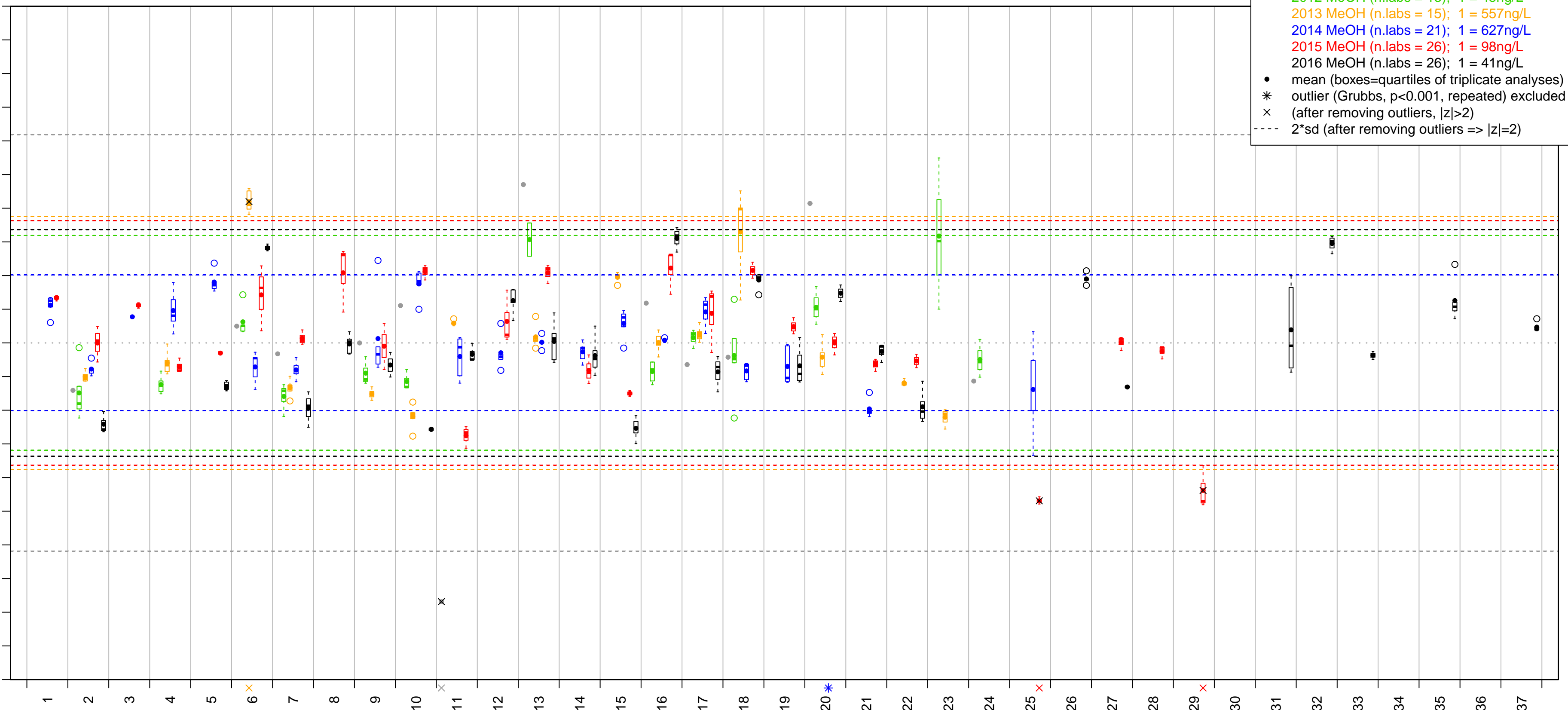
2.0  
1.5  
1.0  
0.5  
0.0

- 2011 MeOH (n.labs = 12); 1 = 513ng/L
- 2012 MeOH (n.labs = 13); 1 = 45ng/L
- 2013 MeOH (n.labs = 15); 1 = 557ng/L
- 2014 MeOH (n.labs = 21); 1 = 627ng/L
- 2015 MeOH (n.labs = 26); 1 = 98ng/L
- 2016 MeOH (n.labs = 26); 1 = 41ng/L
- mean (boxes=quartiles of triplicate analyses)
- \* outlier (Grubbs,  $p < 0.001$ , repeated) excluded
- x (after removing outliers,  $|z| > 2$ )
- - - 2\*sd (after removing outliers =>  $|z| = 2$ )

1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17 18 19 20 21 22 23 24 25 26 27 28 29 30 31 32 33 34 35 36 37

Lab ID

p19



# AMP Water

normalized concentrations (with mean of means per year after removing outliers)

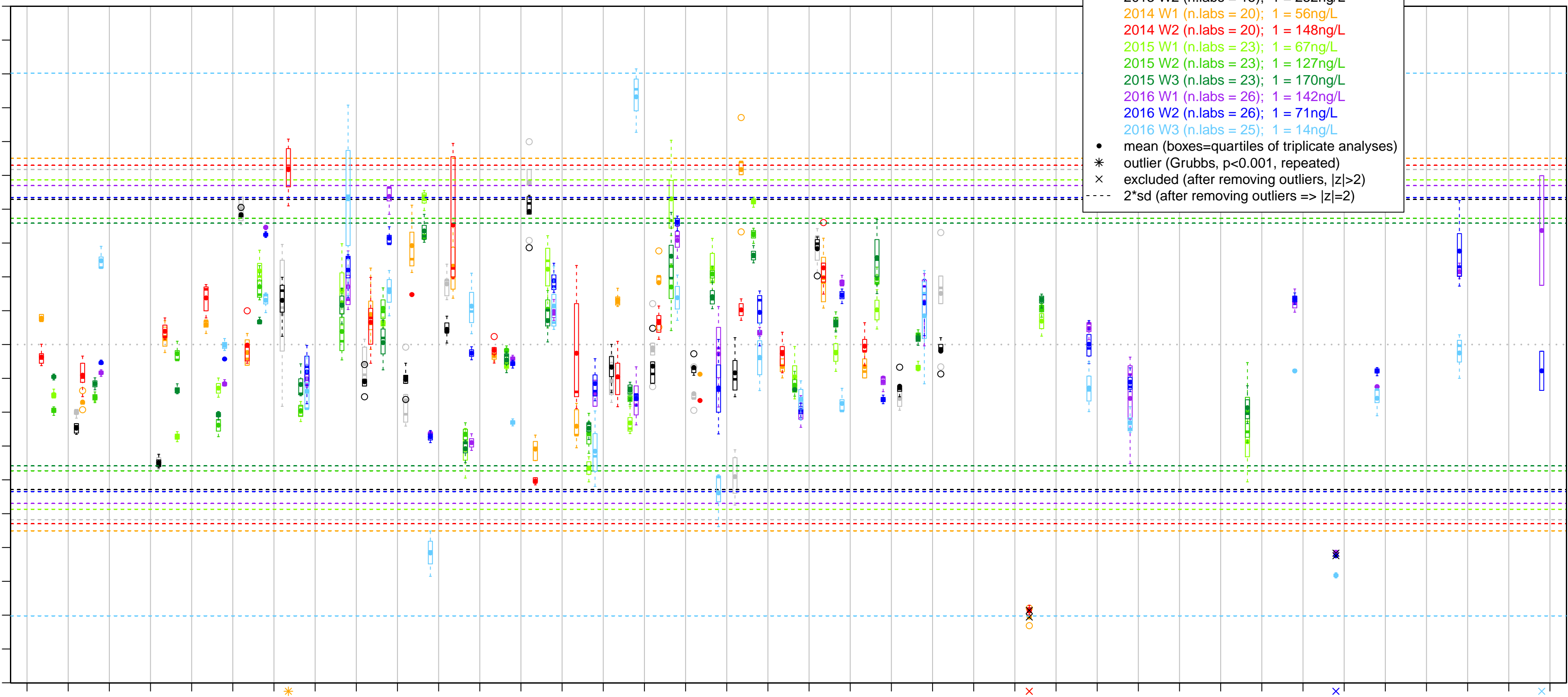
2.0  
1.5  
1.0  
0.5  
0.0

- 2013 W1 (n.labs = 15); 1 = 86ng/L
- 2013 W2 (n.labs = 15); 1 = 252ng/L
- 2014 W1 (n.labs = 20); 1 = 56ng/L
- 2014 W2 (n.labs = 20); 1 = 148ng/L
- 2015 W1 (n.labs = 23); 1 = 67ng/L
- 2015 W2 (n.labs = 23); 1 = 127ng/L
- 2015 W3 (n.labs = 23); 1 = 170ng/L
- 2016 W1 (n.labs = 26); 1 = 142ng/L
- 2016 W2 (n.labs = 26); 1 = 71ng/L
- 2016 W3 (n.labs = 25); 1 = 14ng/L
- mean (boxes=quartiles of triplicate analyses)
- \* outlier (Grubbs,  $p < 0.001$ , repeated)
- × excluded (after removing outliers,  $|z| > 2$ )
- - - 2\*sd (after removing outliers =>  $|z| = 2$ )

1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17 18 19 20 21 22 23 24 25 26 27 28 29 30 31 32 33 34 35 36 37

Lab ID

p20

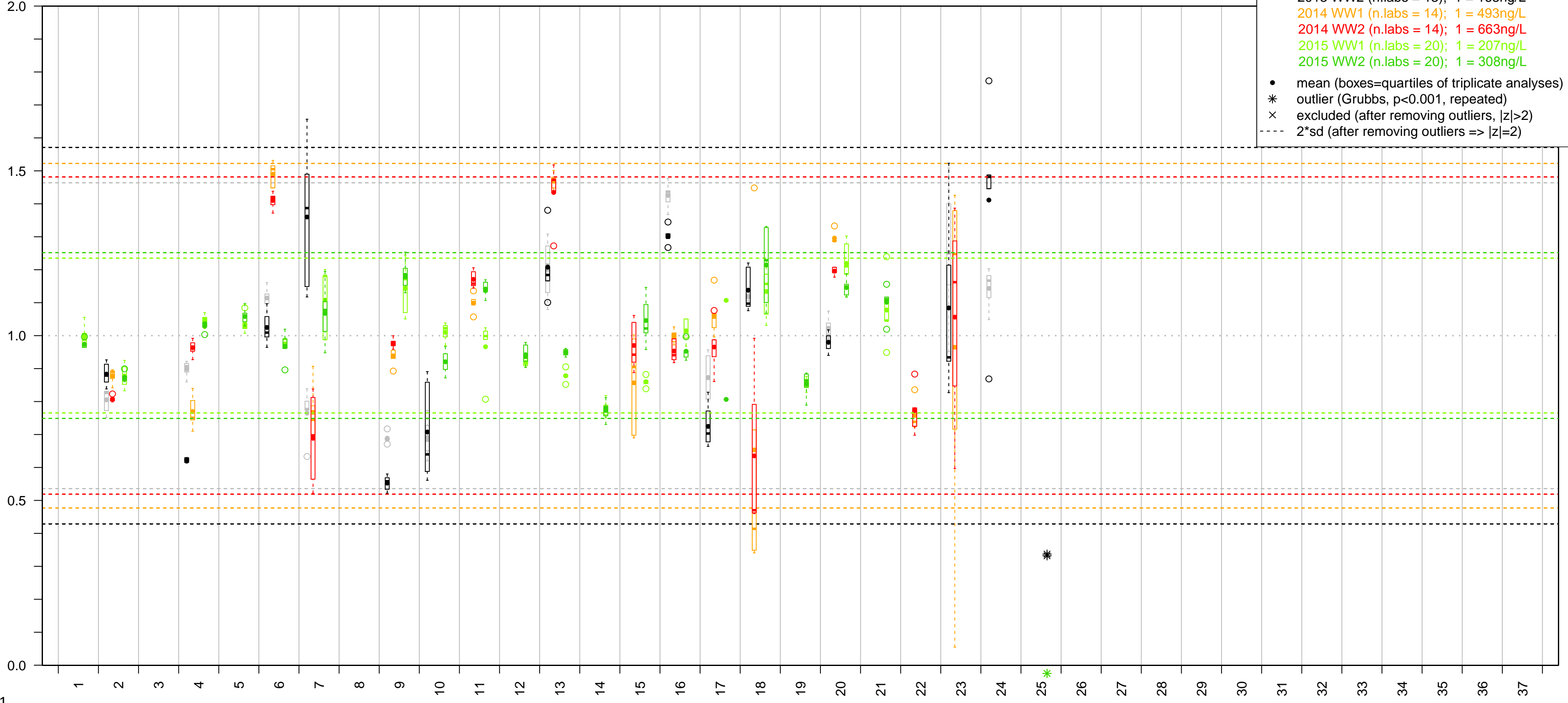


# AMP Wastewater

normalized concentrations (with mean of means per year after removing outliers)

2013 WW1 (n.labs = 13); 1 = 389ng/L  
 2013 WW2 (n.labs = 13); 1 = 168ng/L  
 2014 WW1 (n.labs = 14); 1 = 493ng/L  
 2014 WW2 (n.labs = 14); 1 = 663ng/L  
 2015 WW1 (n.labs = 20); 1 = 207ng/L  
 2015 WW2 (n.labs = 20); 1 = 308ng/L

- mean (boxes=quartiles of triplicate analyses)
- \* outlier (Grubbs,  $p < 0.001$ , repeated)
- × excluded (after removing outliers,  $|z| > 2$ )
- 2\*sd (after removing outliers =>  $|z| = 2$ )



p21

Lab ID

# METH MeOH(1)

normalized concentrations (with mean of means per year after removing outliers)

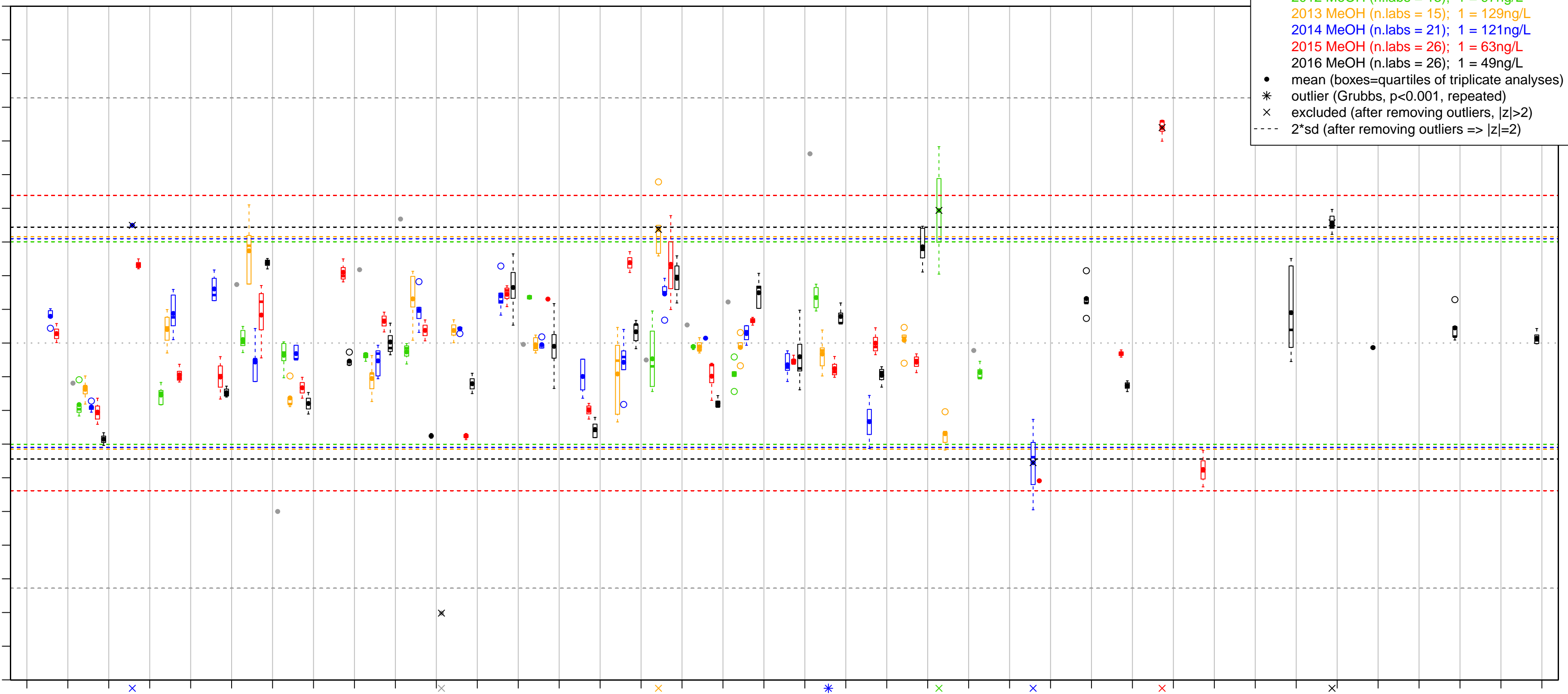
2.0  
1.5  
1.0  
0.5  
0.0

- 2011 MeOH (n.labs = 12); 1 = 457ng/L
- 2012 MeOH (n.labs = 13); 1 = 97ng/L
- 2013 MeOH (n.labs = 15); 1 = 129ng/L
- 2014 MeOH (n.labs = 21); 1 = 121ng/L
- 2015 MeOH (n.labs = 26); 1 = 63ng/L
- 2016 MeOH (n.labs = 26); 1 = 49ng/L
- mean (boxes=quartiles of triplicate analyses)
- \* outlier (Grubbs,  $p < 0.001$ , repeated)
- × excluded (after removing outliers,  $|z| > 2$ )
- - - 2\*sd (after removing outliers =>  $|z| = 2$ )

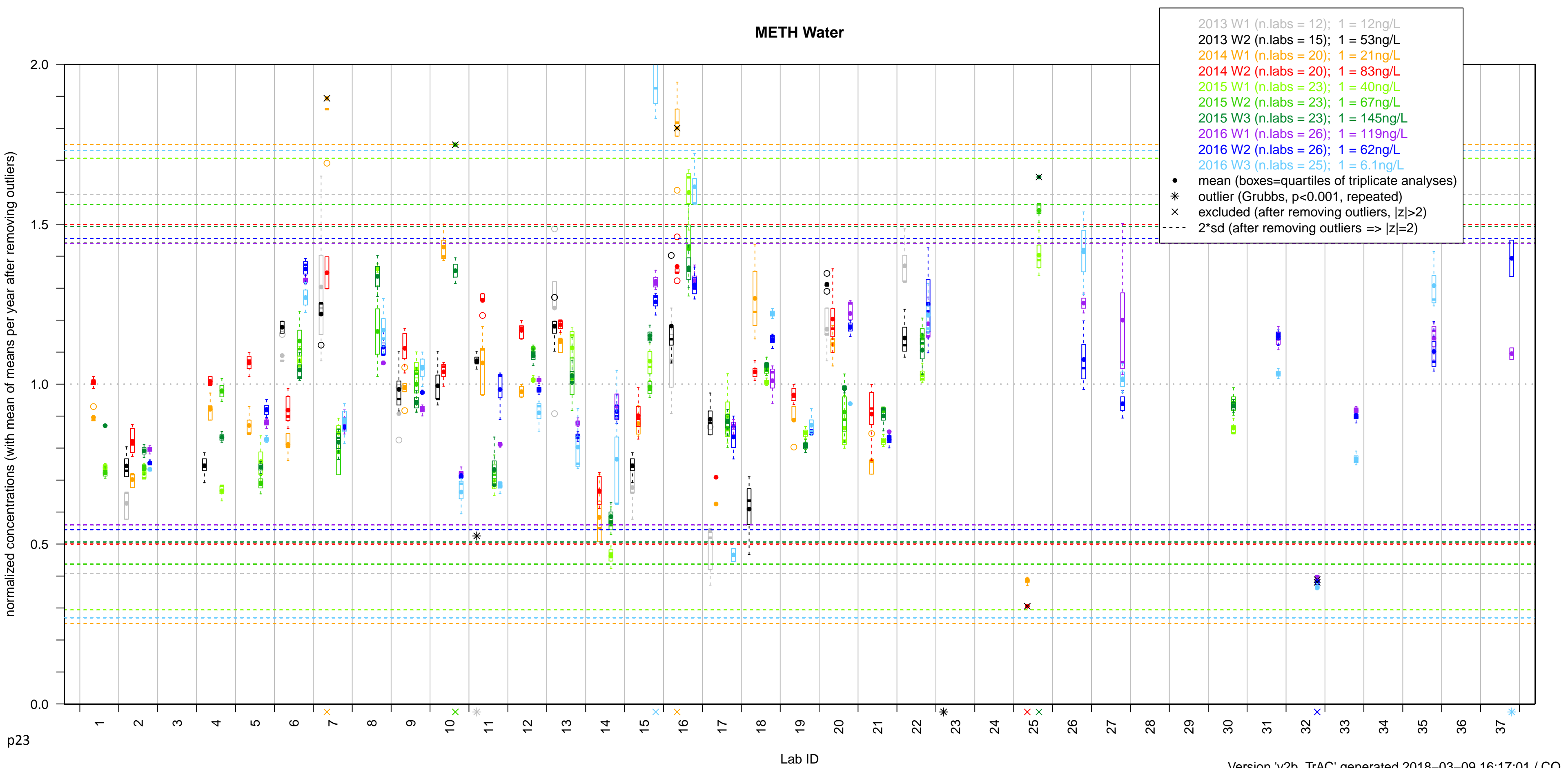
1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17 18 19 20 21 22 23 24 25 26 27 28 29 30 31 32 33 34 35 36 37

Lab ID

p22



# METH Water



# METH Wastewater

normalized concentrations (with mean of means per year after removing outliers)

2.0  
1.5  
1.0  
0.5  
0.0

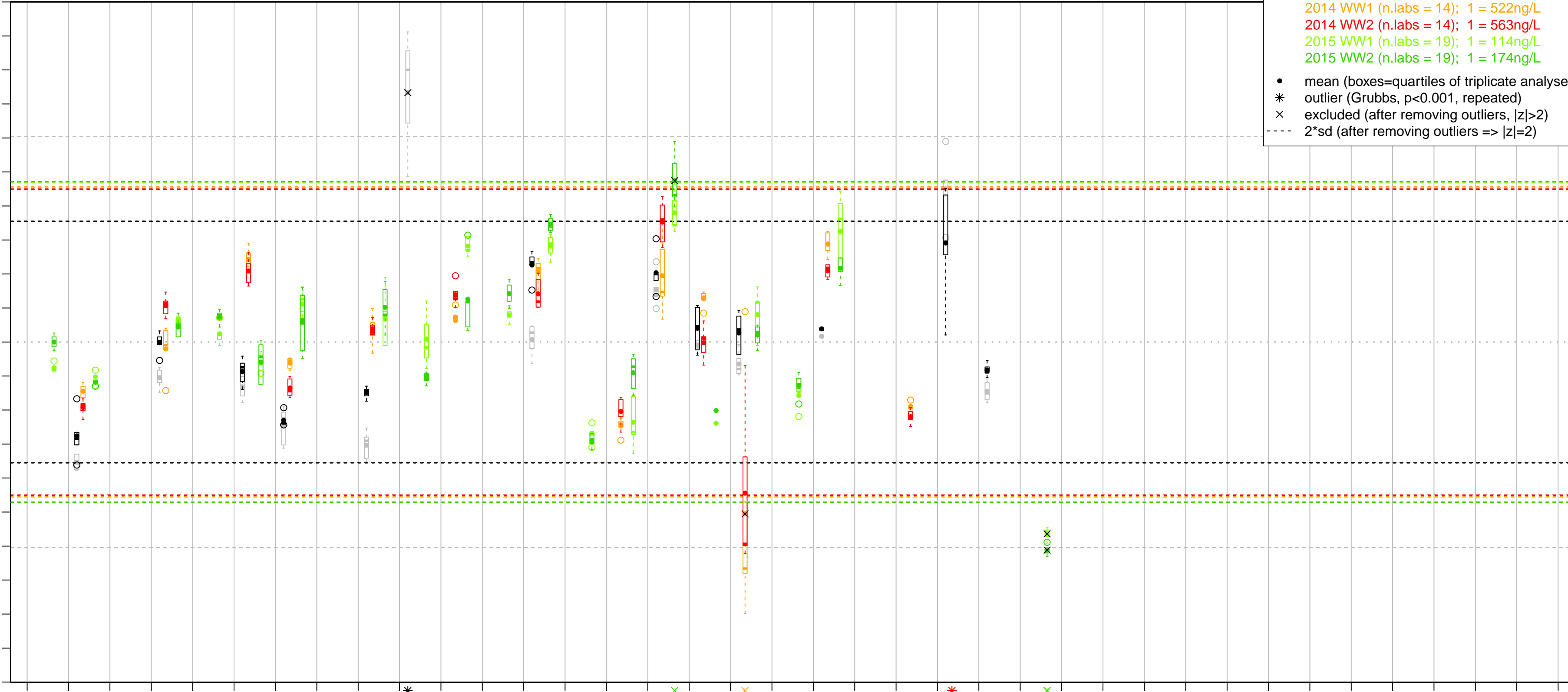
2013 WW1 (n.labs = 13); 1 = 422ng/L  
2013 WW2 (n.labs = 13); 1 = 318ng/L  
2014 WW1 (n.labs = 14); 1 = 522ng/L  
2014 WW2 (n.labs = 14); 1 = 563ng/L  
2015 WW1 (n.labs = 19); 1 = 114ng/L  
2015 WW2 (n.labs = 19); 1 = 174ng/L

- mean (boxes=quartiles of triplicate analyses)
- \* outlier (Grubbs,  $p < 0.001$ , repeated)
- × excluded (after removing outliers,  $|z| > 2$ )
- - - 2\*sd (after removing outliers =>  $|z| = 2$ )

1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17 18 19 20 21 22 23 24 25 26 27 28 29 30 31 32 33 34 35 36 37

Lab ID

p24

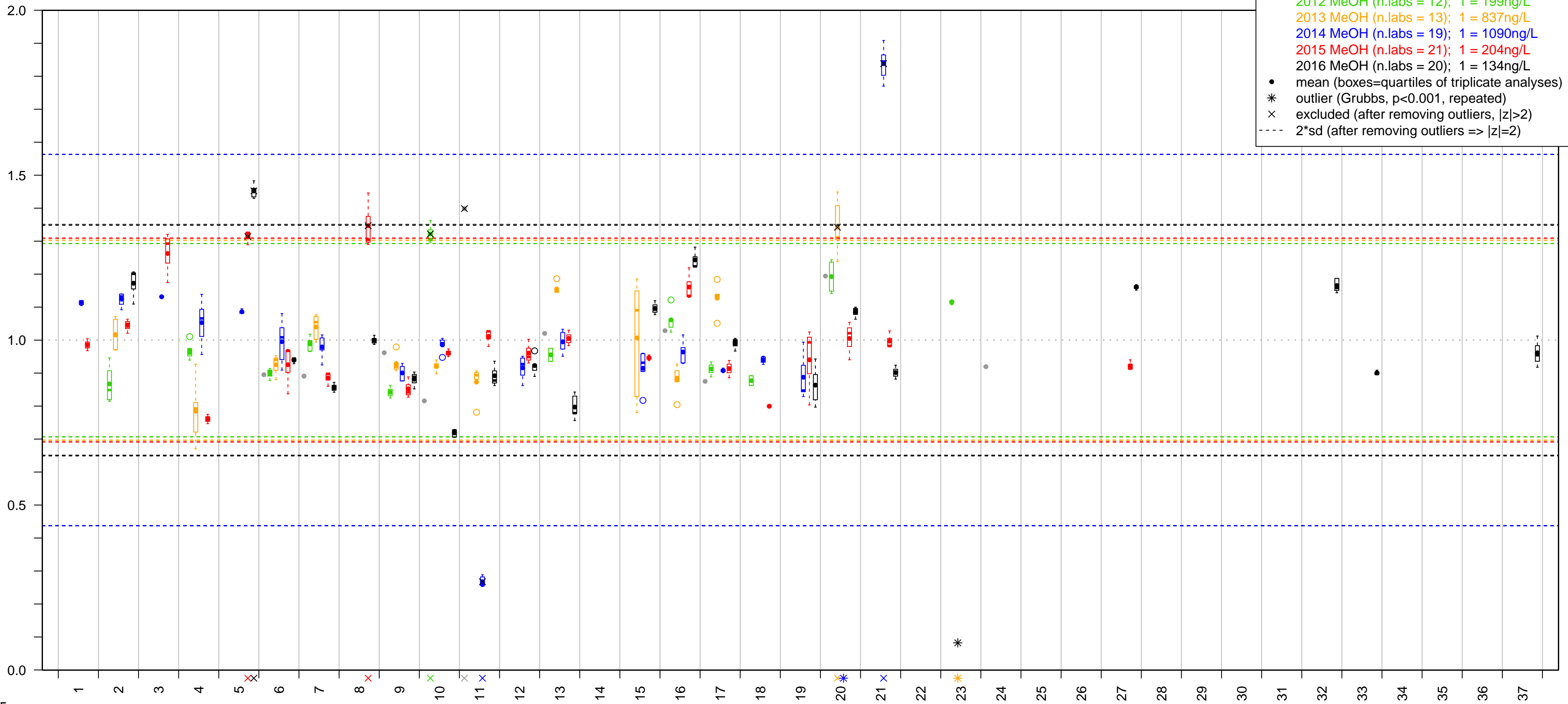




# THC-COOH MeOH(1)

normalized concentrations (with mean of means per year after removing outliers)

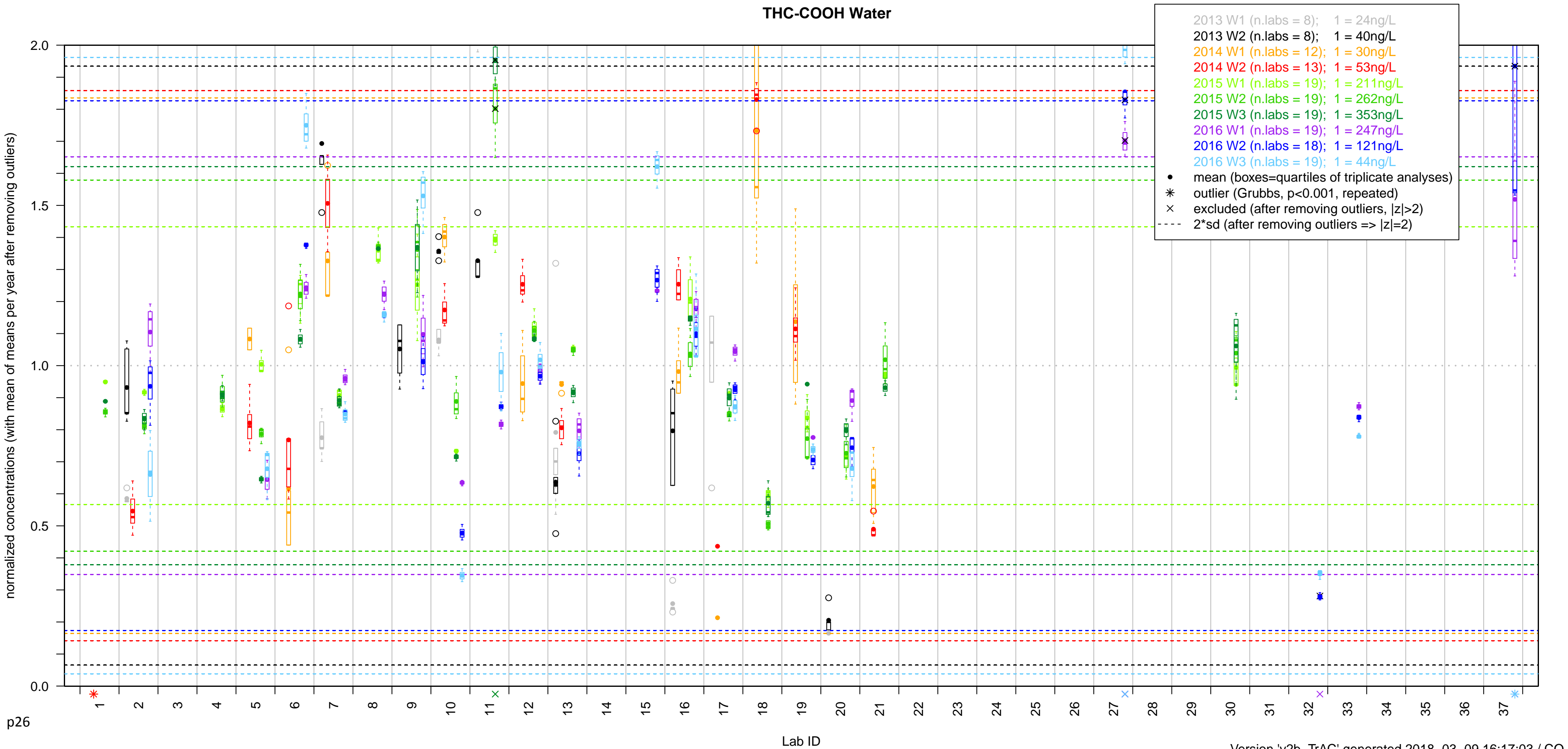
- 2011 MeOH (n.labs = 10); 1 = 525ng/L
- 2012 MeOH (n.labs = 12); 1 = 199ng/L
- 2013 MeOH (n.labs = 13); 1 = 837ng/L
- 2014 MeOH (n.labs = 19); 1 = 1090ng/L
- 2015 MeOH (n.labs = 21); 1 = 204ng/L
- 2016 MeOH (n.labs = 20); 1 = 134ng/L
- mean (boxes=quartiles of triplicate analyses)
- \* outlier (Grubbs,  $p < 0.001$ , repeated)
- × excluded (after removing outliers,  $|z| > 2$ )
- - - 2\*sd (after removing outliers =>  $|z| = 2$ )



p25

Lab ID

# THC-COOH Water

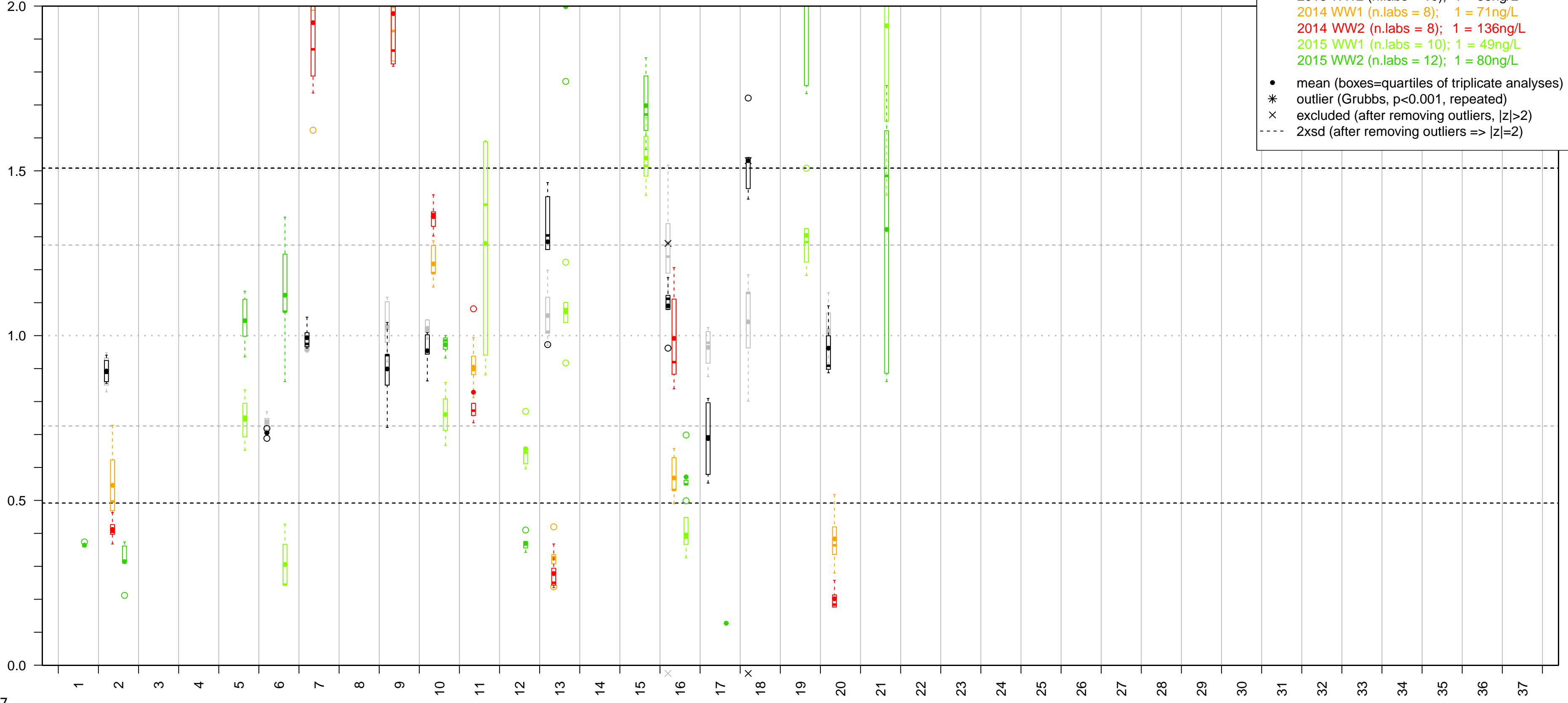


### THC-COOH Wastewater

normalized concentrations (with mean of means per year after removing outliers)

2013 WW1 (n.labs = 10); 1 = 213ng/L  
 2013 WW2 (n.labs = 10); 1 = 88ng/L  
 2014 WW1 (n.labs = 8); 1 = 71ng/L  
 2014 WW2 (n.labs = 8); 1 = 136ng/L  
 2015 WW1 (n.labs = 10); 1 = 49ng/L  
 2015 WW2 (n.labs = 12); 1 = 80ng/L

- mean (boxes=quartiles of triplicate analyses)
- \* outlier (Grubbs,  $p < 0.001$ , repeated)
- × excluded (after removing outliers,  $|z| > 2$ )
- - - 2xstd (after removing outliers  $\Rightarrow |z| = 2$ )



p27

Lab ID

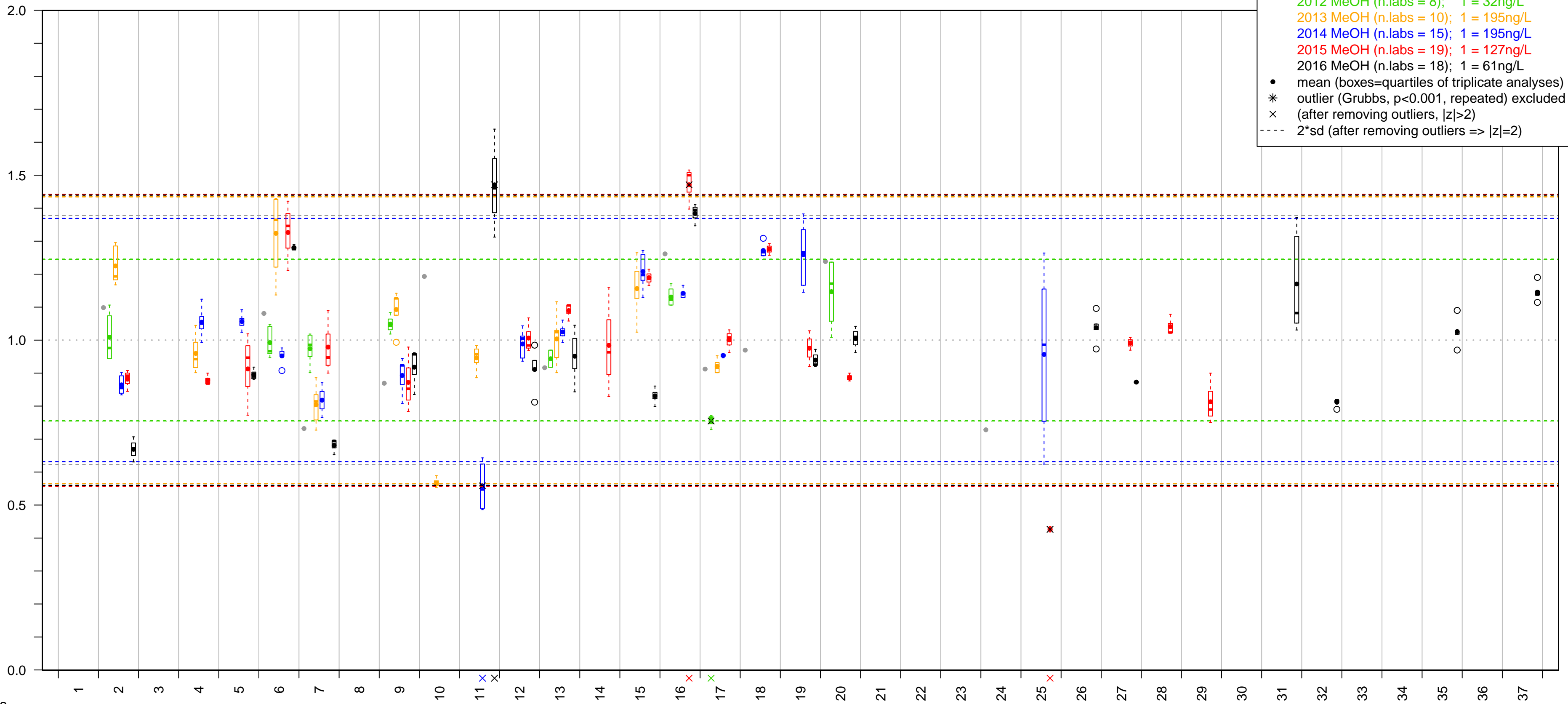
# 6-MAM MeOH(1)

normalized concentrations (with mean of means per year after removing outliers)

2011 MeOH (n.labs = 11); 1 = 472ng/L  
 2012 MeOH (n.labs = 8); 1 = 32ng/L  
 2013 MeOH (n.labs = 10); 1 = 195ng/L  
 2014 MeOH (n.labs = 15); 1 = 195ng/L  
 2015 MeOH (n.labs = 19); 1 = 127ng/L  
 2016 MeOH (n.labs = 18); 1 = 61ng/L

- mean (boxes=quartiles of triplicate analyses)
- \* outlier (Grubbs,  $p < 0.001$ , repeated) excluded
- × (after removing outliers,  $|z| > 2$ )
- - - 2\*sd (after removing outliers =>  $|z| = 2$ )

p28



Lab ID

### 6-MAM Water

normalized concentrations (with mean of means per year after removing outliers)

2.0  
1.5  
1.0  
0.5  
0.0

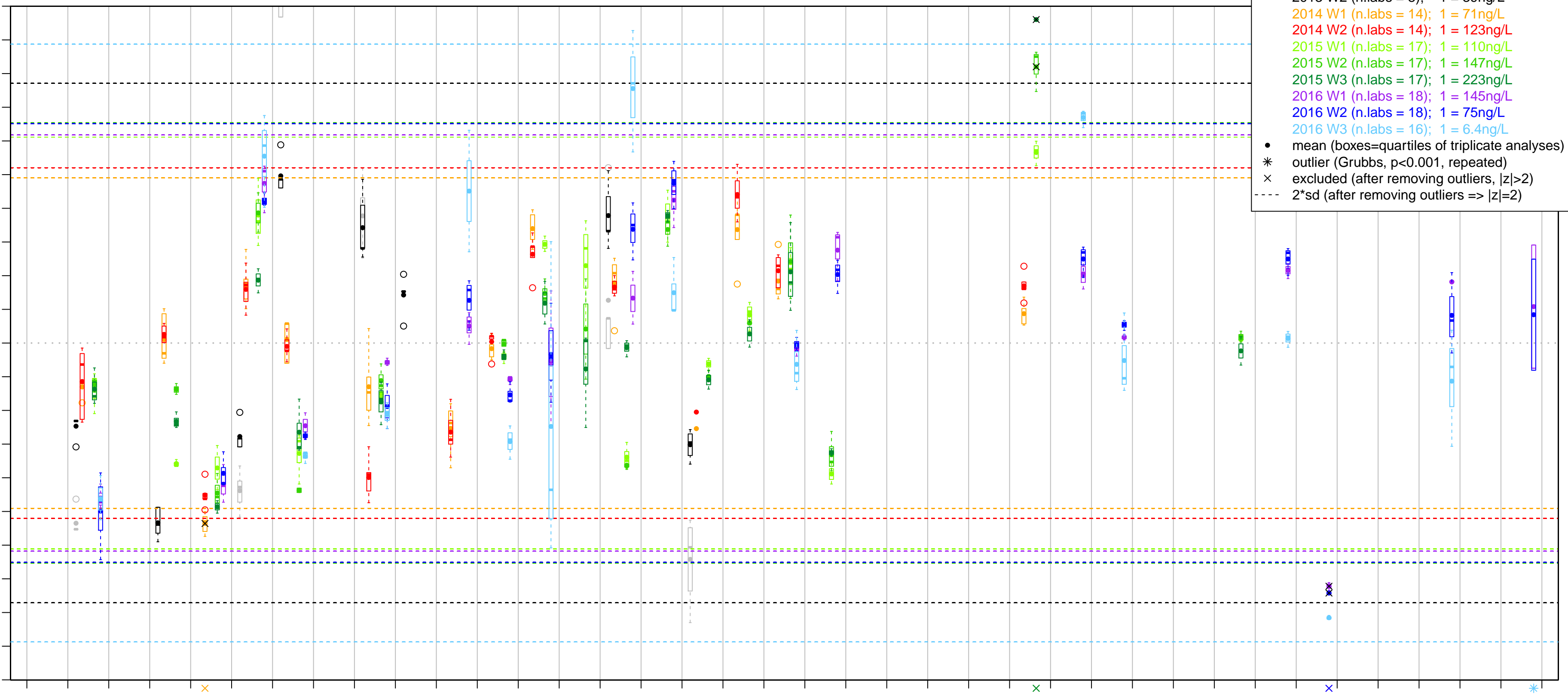
|                        |             |
|------------------------|-------------|
| 2013 W1 (n.labs = 6);  | 1 = 11ng/L  |
| 2013 W2 (n.labs = 8);  | 1 = 39ng/L  |
| 2014 W1 (n.labs = 14); | 1 = 71ng/L  |
| 2014 W2 (n.labs = 14); | 1 = 123ng/L |
| 2015 W1 (n.labs = 17); | 1 = 110ng/L |
| 2015 W2 (n.labs = 17); | 1 = 147ng/L |
| 2015 W3 (n.labs = 17); | 1 = 223ng/L |
| 2016 W1 (n.labs = 18); | 1 = 145ng/L |
| 2016 W2 (n.labs = 18); | 1 = 75ng/L  |
| 2016 W3 (n.labs = 16); | 1 = 6.4ng/L |

- mean (boxes=quartiles of triplicate analyses)
- \* outlier (Grubbs, p<0.001, repeated)
- × excluded (after removing outliers, |z|>2)
- 2\*sd (after removing outliers => |z|=2)

1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17 18 19 20 21 22 23 24 25 26 27 28 29 30 31 32 33 34 35 36 37

Lab ID

p29



# 6-MAM Wastewater

