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Round 02/2018

PAH metabolites in urine

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1 Summary

Within the framework of the HBM4EU project, an External Quality Assurance Scheme (EQUAS) was organized and conducted for the analysis of **P**olycyclic **A**romatic **H**ydrocarbon **met**abolites (PAH met.). PAH met. correspond to 13 biomarkers: 1-naphthol, 2-naphthol, 1,2-DHN, 2-FLUO, 3-FLUO, 9-FLUO, 1-PHEN, 2-PHEN, 3-PHEN, 4-PHEN, 9-PHEN, 1-PYR and 3-BaP in urine.

The study was performed from October 2018 to January 2019.

In total, 42 laboratories were invited for this second round, of which 27 laboratories from 13 countries registered.

The participation in this EQUAS was satisfactory; 27 out of the registered 27 laboratories (100%) submitted their results. In November 2018, six different test samples consisting of 5 ml urine spiked with the biomarker at two different concentrations (PAH met.$_{low}$ and PAH met.$_{high}$) were prepared and sent to the participating expert and candidate laboratories for analysis.

The HBM4EU QAU had selected six expert laboratories for PAH met. in urine. Four expert laboratories were from Europe (HBM4EU consortium) and also participated as candidates. Two expert laboratories were from outside Europe (Japan, USA).

Homogeneity assessment of the control materials confirmed that the materials were adequately homogeneous. The stability test demonstrated no significant loss of the biomarkers in the course of this EQUAS.

The proficiency of the laboratories was assessed through Z-scores calculated using the mean concentration as established by expert laboratories as assigned value, and a fixed fit-for-purpose relative target standard deviation (FFP-RSD$_R$) of 25%. Assigned values and Z-scores could be determined for seven biomarkers at the low level and for seven biomarkers at the high level.

Table 1 below gives an overview of the respective results for the low and the high level of all PAH met. biomarkers.

For two biomarkers at the low level and for two biomarkers at the high level, Z-scores could only be determined using the consensus value of the ICI as described in SOP HBM4EU-SOP-QA-003.

The percentage of satisfactory Z-scores obtained for each biomarker ranges from 62% (1-PYR$_{low}$) to 100% (2-FLUO$_{low}$, 3-FLUO$_{high}$, 1-PHEN$_{high}$ and 2-PHEN$_{low}$).

For four biomarkers (1,2-DHN, 9-FLUO, 9-PHEN and 3-BaP) no kind of Z-score could be provided.

Table 2 shows the results of each participating laboratory for each biomarker and also reflects how many PAH met. biomarkers have been analysed per participant (sum) and how many participants have analysed the low and the high level of the respective biomarkers (TOTAL).

Table 1 Overview of results for PAH met. in urine in 1st EQUAS round 2

number of laboratories with respective results for PAH met. in urine in 1st EQUAS/round 2								
biomarker	participants (+ experts)	quantitative results	evaluation scheme	assigned/ consensus value	satisfactory	questionable	unsatisfactory	ND and no proxy- Z-score
1-naphthol low	17	17	EQUAS	2.008 ng/ml	13 (76%)	1 (6%)	3 (18%)	0
1-naphthol high	17	17	EQUAS	3.090 ng/ml	14 (82%)	1 (6%)	2 (12%)	0
2-naphthol low	20	20	EQUAS	9.355 ng/ml	17 (85%)	2 (10%)	1 (5%)	0
2-naphthol high	20	20	EQUAS	14.271 ng/ml	17 (85%)	2 (10%)	1 (5%)	0
1,2-DHN low	1	0	none	-	no candidates	no candidates	no candidates	1
1,2-DHN high	1	0	none	-	no candidates	no candidates	no candidates	1
2-FLUO low	12	10	EQUAS	0.436 ng/ml	10 (100%)	0	0	0
2-FLUO high	12	10	EQUAS	0.827ng/ml	9 (90%)	1 (10%)	0	0
3-FLUO low	8	6	EQUAS	0.214 ng/ml	5 (83%)	0	1 (17%)	0
3-FLUO high	8 (+2)	6	ICI	0.389 ng/ml	6 (100%)	0	0	0
9-FLUO low	5	4	none	-	no Z-scores	no Z-scores	no Z-scores	0
9-FLUO high	5	4	none	-	no Z-scores	no Z-scores	no Z-scores	0
1-PHEN low	13	11	EQUAS	0.476 ng/ml	9 (82%)	2 (18%)	0	0
1-PHEN high	13	11	EQUAS	0.788 ng/ml	11 (100%)	0	0	0
2-PHEN low	13 (+2)	10	ICI	0.308 ng/ml	10 (100%)	0	0	1
2-PHEN high	13	11	EQUAS	0.732 ng/ml	9 (75%)	3 (25%)	0	0
3-PHEN low	13	11	EQUAS	0.271 ng/ml	9 (82%)	1 (9%)	1 (9%)	0
3-PHEN high	13	11	EQUAS	0.482 ng/ml	9 (82%)	1 (9%)	1 (9%)	0
4-PHEN low	13 (+1)	12	ICI	0.129 ng/ml	11 (92%)	1 (8%)	0	0
4-PHEN high	13 (+1)	12	ICI	0.246 ng/ml	11 (92%)	1 (8%)	0	0
9-PHEN low	10	7	none	-	no Z-scores	no Z-scores	no Z-scores	0
9-PHEN high	10	7	none	-	no Z-scores	no Z-scores	no Z-scores	1
1-PYR low	26	25	EQUAS	0.113 ng/ml	16 (62%)	4 (15%)	6 (23%)	0
1-PYR high	26	26	EQUAS	0.256 ng/ml	19 (73%)	5 (19%)	2 (8%)	0
3-BaP low	8	3	none	-	no Z-scores	no Z-scores	no Z-scores	5
3-BaP high	8	3	none	-	no Z-scores	no Z-scores	no Z-scores	5

Table 2 Results of each participating laboratory

+ = satisfactory, °= questionable, - = unsatisfactory, NA = not analysed, *NA = sum of isomers*, ND = not detected and no proxy Z-score, no = no Z-score available

PAH	1-naph		2-naph		1,2-DHN		2-FLUO		3-FLUO		9-FLUO		1-PHEN		2-PHEN		3-PHEN		4-PHEN		9-PHEN		1-PYR		3-BaP		Sum
Lab	low	high	low	high	lowo	high	low	high	low	high	low	high	low	high	low	high	low	high	low	high	low	high	low	high	low	high	
QR/112	NA	NA	NA	NA	NA	NA	*NA*	*NA*	*NA*	*NA*	no	no	+	+	*NA*	*NA*	*NA*	*NA*	+	+	NA	NA	+	+	NA	NA	16
QR/113	+	+	+	+	NA	NA	+	+	+	+	no	no	°	+	+	°	+	+	°	°	NA	NA	+	°	NA	NA	20
QR/118	-	-	+	+	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	-	+	NA	NA	6
QR/122	+	+	+	+	NA	NA	+	+	NA	NA	NA	NA	+	+	+	+	+	+	+	+	no	no	+	+	no	no	20
QR/123	-	-	°	°	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	+	+	NA	NA	6
QR/124	+	+	+	+	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	+	+	NA	NA	6
QR/125	+	+	+	+	NA	NA	+	+	+	+	no	no	+	+	+	+	+	+	+	+	no	no	+	+	NA	NA	22
QR/126	+	+	+	+	ND	ND	+	°	*NA*	*NA*	*NA*	*NA*	+	+	+	+	+	+	+	+	no	no	-	°	ND	ND	26
QR/127	+	+	+	+	NA	NA	+	+	+	+	NA	NA	+	+	+	+	+	+	+	+	no	no	+	+	ND	ND	22
QR/129	NA	NA	+	+	NA	NA	+	+	+	+	NA	NA	+	+	*NA*	*NA*	*NA*	*NA*	+	+	ND	ND	+	+	ND	ND	20
QR/130	-	°	+	+	NA	NA	+	+	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	no	no	-	°	NA	NA	10
QR/132	NA	NA	+	+	NA	NA	*NA*	*NA*	NA	NA	NA	NA	*NA*	*NA*	+	+	+	+	+	+	NA	NA	+	+	NA	NA	14
QR/140	+	+	+	+	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	+	+	NA	NA	6
QR/141	+	+	+	+	NA	NA	NA	NA	NA	NA	NA	NA	+	+	+	+	+	+	+	+	NA	NA	+	+	NA	NA	14
QR/142	+	+	+	+	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	4
QR/143	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	+	+	NA	NA	2
QR/201	+	+	+	+	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	°	+	no	no	8
QR/202	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	-	-	NA	NA	2
QR/206	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	°	+	NA	NA	2
QR/207	NA	NA	+	+	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	+	+	NA	NA	4
QR/210	+	+	°	°	NA	NA	+	+	NA	NA	NA	NA	+	+	+	°	+	+	*NA*	*NA*	*NA*	*NA*	+	+	NA	NA	18
QR/211	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	°	°	NA	NA	2
QR/217	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	+	+	NA	NA	2
QR/218	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	°	+	NA	NA	2
QR/219	+	+	+	+	NA	NA	+	+	-	+	NA	NA	°	+	ND	+	-	-	+	+	no	no	-	-	ND	ND	22
QR/220	+	+	+	+	NA	NA	+	+	+	+	no	no	+	+	+	°	+	+	+	+	no	no	+	+	no	no	24
QR/221	°	+	-	-	NA	NA	NA	NA	NA	NA	NA	NA	*NA*	*NA*	+	+	°	°	+	+	*NA*	*NA*	-	°	ND	ND	18
TOTAL	17	17	20	20	1	1	12	12	8	8	5	5	13	13	13	13	13	13	13	13	10	10	26	26	8	8	

2 Introduction

Inter-Laboratory Comparison Investigations (ICI) and External Quality Assurance Schemes (EQUAS) are tools to assess the proficiency of laboratories, and the comparability and reliability of analytical methods. Participation in ICI / EQUAS forms an integral part of quality control, in addition to initial and on-going in-house method validation.

This EQUAS study has been organised within the frame of HBM4EU as part of the Quality Assurance program for biomonitoring analyses, following protocols HBM4EU-SOP-QA-001 to 004 which are available through the HBM4EU website. Within HBM4EU, participation in ICI/EQUAS exercises is mandatory for laboratories that will analyse HBM4EU samples.

This report describes the 2nd round for PAH met. in urine and was organised by the Institute and Outpatient Clinic of Occupational, Social and Environmental Medicine (IPASUM) at Friedrich-Alexander University of Erlangen-Nuremberg.

The selection of the most relevant PAH biomarkers was previously done in WP9, and has been described in Deliverable report 9.2 v1.1. Based on this, a set of 13 target biomarkers was compiled to be included in this 1st EQUAS (see **Table 3**).

Table 3 PAH met. biomarkers in urine included in this 1st EUQAS/round 2

Abbreviation	Target biomarker
1-naphthol	1-hydroxynaphthalene
2-naphthol	2-hydroxynaphthalene
1,2-DHN	1.2-dihydroxynaphthalene
2-FLUO	2-hydroxyfluorene
3-FLUO	3-hydroxyfluorene
9-FLUO	9-hydroxyfluorene
1-PHEN	1-hydroxyPHENanthrene
2-PHEN	2-hydroxyPHENanthrene
3-PHEN	3-hydroxyPHENanthrene
4-PHEN	4-hydroxyPHENanthrene
9-PHEN	9-hydroxyPHENanthrene
1-PYR	1-hydroxypyrene
3-BaP	3-hydroxybenzo(a)pyrene

For this 1st EQUAS, expert laboratories had to be selected according to the selection criteria described in HBM4EU-SOP-QA-001 and in agreement with the QAU.

The selection criteria included:

- Number of years of experience with the biomarker/matrix combination of interest.
- Application of highly sensitive and selective analytical techniques for the analysis.
- Application of isotope-labelled standards for quantification.
- Availability of in-house validation reports, data on on-going intra-laboratory performance, ISO17025 accreditation for the biomarker of interest.
- Success rate in inter-laboratory comparisons, external quality assessment schemes or at least comparative results in application studies.

EQUAS does not require a certain number of participants, because the performance evaluation is not based on the participants' results but on assigned values and tolerance ranges as derived from the analysis data from the expert laboratories.

The expert-assigned value is the target value based on analysis results obtained from analysis of the control material by at least three expert laboratories (see HBM4EU-SOP-QA-001). The expert assigned values were calculated by averaging the values obtained by the expert labs.

2.1 Confidentiality

In this report, the identity of the participants and the information provided by them is treated as confidential. However, lab codes of the participants will be disclosed to the HBM-QAU for performance assessment.

3 Control material

3.1 Preparation of control material

For control material, partly burdened material was used. It consists of human urine (from smokers) with the addition of sodium azide. The samples are clear and free of precipitates as much as possible (filtration). The 13 different stock solutions (1-naphthol, 2-naphthol, 1,2-DHN, 2-FLUO, 3-FLUO, 9-FLUO, 1-PHEN, 2-PHEN, 3-PHEN, 4-PHEN, 9-PHEN, 1-PYR and 3-BaP) were diluted into two different concentrations and the addition to the native control material resulted in the intended concentration in control material (PAH met.$_{low}$, PAH met.$_{high}$). The two spiked control materials were aliquoted (5 ml each) into vials with caps (82 x 13 mm, polypropylene, Sarstedt). The tubes were stored in a freezer (≤ -18 °C) until transportation. The two different concentrations (PAH met.$_{low}$, PAH met.$_{high}$) were measured using the analytical method mentioned in **Appendix 5**. The measured concentrations are shown in 3.2 and 3.3 of this report.

3.2 Homogeneity of control material

Six tubes of each concentration (deviation from EQUAS SOP due to pursuit of schedule) of the control material (PAH met.$_{low}$, PAH met.$_{high}$) were randomly selected from the freezer (≤ -18 °C). The thawed samples were re-homogenised by vortex shaking and were analysed in duplicate (analysis method see **Appendix 5**).

Ten tubes of each concentration of 3-BaP were randomly selected from the freezer (≤ -18 °C) and sent to ABF for analysis.

The homogeneity was evaluated according to ISO 13528:2015, Fearn et al [2001] and Thompson [2000] as well as HBM4EU-SOP-QA-002. The results are presented in **Appendix 1**. The conclusion is that no outliers are detected, homogeneity is adequate and the method is suitable.

3.3 Stability of control material

Stability testing was done according to HBM4EU-SOP-QA-002. On the day of preparation of the control materials, randomly selected test samples of PAH met.$_{low}$ and PAH met.$_{high}$ were stored at -80 °C. The assumption is that under these conditions, the biomarkers are stable in urine. On the last day of the deadline for submission of analysis results by the participants (January 18, 2019), six test samples of both materials stored at -80 °C, and six samples of PAH met.$_{low}$ and PAH met.$_{high}$ randomly selected from the -18 °C freezer were thawed and re-homogenised by vortex shaking. Next, all 24 samples were analysed by IPASUM using the analytical method mentioned in **Appendix 5**.

In the same way, 24 samples were sent to ABF for analysis of the biomarker 3-BaP.

The stability was evaluated according to HBM4EU-SOP-QA-002 and using the Excel-sheet “HBM4EU ICI-EQUAS stability test CM v1”. The results are presented in **Appendix 2**. No consequential instabilities and no statistical differences were detected.

4 Organisational details

4.1 Participants

For the organisation of the 1st EQUAS, IPASUM conducted a survey to find expert laboratories for PAH metabolites in urine willing to participate in the project. Then, IPASUM evaluated their eligibility and selected six expert laboratories in agreement with the QAU and according to HBM4EU-SOP-QA-001. Four expert laboratories were from Europe (HBM4EU consortium) and also participated as candidates. Two expert laboratories were from outside Europe (Japan, USA).

IPASUM contacted the six selected expert laboratories and sent them invitation letters by e-mail on October 16, 2018 (see **Appendix 3**). It was indicated that participation would be free of charge and that those who subscribed to this EQUAS would receive a kit containing the test materials needed for analysis. Test results had to be submitted within the stipulated deadline (January 18, 2019).

A list of 42 candidate laboratories from different countries eligible for the analysis of PAH met. had been compiled by the Work Package (WP) Task 9.2 leaders and made available to the institution organising the respective EQUAS.

Instruction letters were thus sent by e-mail to 42 candidate laboratories on November 6, 2018 (see **Appendix 3**). It was indicated that participation would be free of charge and that those who subscribed to the EQUAS would receive a kit containing the test materials needed for analysis. The condition for participation was that the test results had to be submitted within the stipulated deadline (January 18, 2019).

Twenty-seven laboratories from 13 countries out of the 42 laboratories (64%) in the revised candidate list indicated their interest in participating in this EQUAS and sent their registration form to IPASUM, with their agreement to abide by the conditions for participation. These laboratories received an individual laboratory code to report their measurement results.

Twenty-seven of the 27 potential participants (100%) from 13 countries performed the assays and submitted their results.

Twenty-three participants reported their results within the stipulated deadline (January 18, 2019), while four participants reported with a delay (see **Appendix 8**; QR/127 on January 21, QR/112 on January 24, QR/130 on January 25 and QR/220 on January 30, 2019).

4.2 Dispatch and instructions

Test materials were dispatched to the participants under ambient conditions on November 27, 2018. Each participant received six test samples spiked with the biomarker at two levels, three of each concentration. Each sample consisted of approximately 5 ml urine.

Moreover, a letter with instructions on sample handling (instruction letter, see **Appendix 4**), a sample receipt form to be sent back to IPASUM upon receipt of the test material as well as a result submission form and a method information form (see **Appendix 5**) were sent to the participants by e-mail. The latter form was used to extract relevant information related to the analytical method used for quantification.

Participants were asked to perform a single analysis of each sample using the same procedure as will be used for analysis of samples in the frame of HMB4EU and to report results following the instructions given.

5 Data evaluation

5.1 False positives and <LOQ

Classification of false positives and biomarkers reported as "<LOQ-value" or "not detected" (ND) was done as described in HBM4EU-SOP-QA-003.

A result was assigned as false positive if all of the following conditions applied:

1) the biomarker was below the LOQ value as applied by the organiser, the expert laboratories, and the majority of the participants.
2) the biomarker was reported by the participant at a level clearly exceeding the LOQs mentioned under 1.

If a biomarker is reported as "<LOQ-value", AND an assigned value could be established for the biomarker in the control material, a further assessment was done to verify whether this result might be a false negative and to judge whether the LOQ is considered adequate (low enough) for analysis within the frame of HBM4EU. A result is a false negative if the LOQ of a biomarker is well below the assigned value, but the laboratory did not report a quantitative value. The LOQ is considered not adequate (too high) if:

1) the LOQ is substantially above the assigned value
2) the assigned value represents a realistic concentration of real samples in the frame of HBM4EU
3) quantitative determination is feasible by the majority of laboratories

In order to judge "<LOQ" results in a quantitative way, 'proxy-Z-scores' were calculated as described in 5.6.

5.2 Assigned value

For EQUAS studies, the concentration established by expert laboratories is used as the assigned value as described in SOP HBM4EU-SOP-QA-001.

The HBM4EU QAU selected six expert laboratories for PAH met. in urine. Four expert laboratories were from Europe (HBM4EU consortium) and also participated as candidates. Two expert laboratories were from outside Europe (Japan, USA). Some of the expert laboratories did not cover all 13 biomarkers, but this was not an exclusion criterion. The expert laboratories received the same control material and instructions as the participants. Each expert laboratory analysed each of the three control samples (PAH met.$_{low}$, PAH met.$_{high}$) in duplicate. Upon receipt of their results and method information, the acceptability of the results for establishment of the expert value was verified. The expert value was determined as described in HBM4EU-QA-001.

The individual means and standard deviations for each expert laboratory were calculated. Then, the mean of all individual means of the six expert laboratories (mean-of-means, mom) and the standard deviation of the mom (SD_{mom}) were calculated. The relative uncertainty of the mean of the means is given by:

u = RSD / sqrt(N)

with u = relative uncertainty of the mean of the mean concentrations from the expert labs

RSD = relative standard deviation of the mean of the mean concentrations

N = the number of expert labs (after exclusion of outliers if applicable)

The mean-of-means derived from the expert laboratories is considered suitable for use as assigned value in EQUAS studies if $u \leq 0.7 * \sigma_T$ (σ_T = 25% as described in 5.3).

If $u > 0.7 * \sigma_T$, the individual means are checked for outliers. When an individual expert mean is identified as an outlier, it is rejected from the data set and the relative uncertainty is calculated again. If the condition $u \leq 0.7 * \sigma_T$ is still not met, then the uncertainty of the expert-derived mean is too high to be used as assigned value. In this case, no EQUAS assessment of participants' performance is possible for the applicable biomarker. This is also the case if the number of (remaining) individual expert means is less than three.

Only if the EQUAS assessment of the participants' performance is not possible, is the consensus value of the ICI used as assigned value and calculated as described in SOP HBM4EU-SOP-QA-003. In brief, the consensus values and its uncertainty were calculated from the results submitted by the participants using robust statistics to minimize the influence of outliers. Two criteria were evaluated before calculating performance's score:

- if the uncertainty was not significant ($u \leq 0.3\sigma_T$), z-scores were then calculated, otherwise another kind of performance's scores was calculated taking into account the uncertainty.

- if the uncertainty did not meet the following criteria: $u \leq 0.7\sigma_T$, no kind of score was provided.

5.3 Target standard deviation (σ_T)

For calculation of the Z-scores, a fit-for-purpose relative target standard deviation (FFP-RSD_R) of 25% of the assigned value was used as target standard deviation. This was the default indicated in HBM4EU-SOP-QA-003 and considered appropriate based on the outcome of the 1st round.

5.4 ICI/EQUAS standard deviation

To gain insight into the actual inter-laboratory variability of the biomarker analysis in this study, the robust relative standard deviation (RSD_R) was calculated based on the participants' results, as described in HBM4EU-SOP-QA-003. For this, the results of the expert laboratories were not included.

5.5 Z-scores

Z-scores were calculated according to SOP HBM4EU-SOP-QA-003.

$$Z = \frac{x - C}{\sigma_T} \qquad (1)$$

with: Z = Z-score for the submitted analysis result;
x = result submitted by the laboratory;
C = expert-assigned value;
σ_T = target standard deviation, here 0.25*C

In accordance with ISO 13528 and ISO 17043 and the deliverable D 9.4 "*The Quality Assurance/Quality Control Scheme in the HBM4EU project*", Z-scores are classified as presented in **Table 4**.

Table 4: Classification of Z-scores

$\lvert Z \rvert \leq 2$	satisfactory
$2 < \lvert Z \rvert < 3$	questionable
$\lvert Z \rvert \geq 3$	unsatisfactory

5.6 Proxy-Z-scores

'Proxy-Z-scores' are used to judge "<LOQ" results in a quantitative way (see 5.1). The proxy-Z-scores' are calculated using the LOQ-value as result and equation (1). If no LOQ was specified, zero was used.

proxy-Z-scores are classified as follows:

proxy-Z ≤ -3	false negative. Based on the LOQ provided, the laboratory should have been able to detect and quantify the biomarker. Performance is considered 'unsatisfactory'.
proxy-Z ≥3	the LOQ is considered too high to be fit-for-purpose in the frame of HBM4EU analysis. It also means that the LOQ is too high in comparison with other laboratories. (Note: proxy-Z can only be calculated if an assigned value could be established. If this is the case, this inherently means that reliable quantitative determination at a certain low level is feasible). Performance is considered 'unsatisfactory'.
-3 ≤ proxy-Z < -2	possible false negative. Performance is considered 'questionable'.
2 < proxy-Z ≤ 3	the LOQ is relatively high in relation to HBM4EU analysis and compared to other laboratories. Performance is considered 'questionable'.
-2 ≤ proxy-Z ≤ 2	LOQ is within an acceptable range relative to the assigned value, adequate for HBM4EU analysis, and in line with the LOQs of the majority of the participating laboratories. Performance is considered 'satisfactory'.

If a participant reported for the three samples of one control material only one or two quantitative values and two or one results as `not detected´(ND), the quantitative results were considered in the evaluation. For ND results, LOQ values were included in the calculation of the Z-score.

6 Results and discussion

6.1 Results submitted by participants

In total, 27 laboratories from 13 EU countries agreed to participate in this study. All of them submitted results.

Appendix 8 gives an overview of results and LOQs submitted by the participants as well as reasons for delayed submission.

Results reported as `not detected´ (ND):

For **1,2-DHN$_{low}$** and **1,2-DHN$_{high}$**, the sole participant (QR/126) indicated ND and reported a LOQ of 0.500 ng/ml. Due to the lack of other participants no proxy-Z-score could be calculated.

For **2-PHEN$_{low}$**, one participant (QR/219) indicated `not detected´(ND) and reported a LOQ of 0.013 ng/mL. The LOQ could however not be used as input into the ICI evaluation as this caused a too high uncertainty of the consensus value so that no evaluation for all candidate laboratories would have been possible. Thus, no proxy-Z-score for 2-PHEN$_{low}$ from QR/219 can be provided.

For **9-PHEN$_{high}$**, one participant (QR/129) indicated ND and the LOQ was used as input for the ICI evaluation. However, no Z-score could be calculated, because the uncertainty of the consensus value was too high.

For **1-PYR$_{low}$**, one participant (QR/118) indicated ND. A proxy-Z-score could be calculated by using the LOQ value provided by the participant (0.2 ng/ml). For 1-PYR$_{low}$, QR/118 obtained an unsatisfactory result.

For **3-BaP$_{low}$** and **3-BaP$_{high}$**, five participants (QR/126, QR/127, QR/129, QR/219 and QR/221) indicated ND and the LOQ was used as input for the ICI evaluation. However, no proxy-Z-score could be calculated, because the uncertainty of the consensus value was too high.

False positive results: No participant detected a false positive result.

Reporting sums of isomers: Some participants reported values for the sum of two isomers instead of a separate value for each isomer. In these cases, the results could not be considered in the evaluation and were treated as `not analysed´ (*NA*).

Methods: Laboratories were asked to provide details on the method used for analysis. Almost all labs performed an enzymatic deconjugation step, mostly using β-glucuronidase/aryl-sulfatase. Most of the participating laboratories analysed the PAH met. biomarkers by using liquid chromatography with mass spectrometric detection. Four laboratories used GC-MS as analytical instrument. The majority of the participants used an isotope dilution (addition to sample before extraction) as calibration followed by matrix matched (addition to blank matrix before extraction) and solvent standards.

6.2 Assigned values and (target) standard deviations

For most of the biomarkers, the results from the expert laboratories were used to calculate the mean-of-means, which was then used as assigned value (**1-naphthol, 2-naphthol, 2-FLUO, 3-FLUO$_{low}$, 1-PHEN, 2-PHEN$_{high}$, 3-PHEN** and **1-PYR**).

For some biomarkers, the consensus value of the ICI was used as assigned value and was calculated as described in SOP HBM4EU-SOP-QA-003 (**3-FLUO$_{high}$, 2-PHEN$_{low}$, 4-PHEN$_{low}$, 4-PHEN$_{high}$**).

For four biomarkers, no criterion (at least three expert labs or at least seven participants) was met to calculate an assigned value (1**,2-DHN, 9-FLUO, 9-PHEN** and **3-BaP**).

The assigned (EQUAS)/consensus (ICI) value and its uncertainty, the expert (EQUAS)/robust (ICI) standard deviation, the fit-for-purpose (FFP) relative target standard deviation (σ_T) and the study RSD_R for each of the control materials are included in **Appendix 6**.

6.3 Assessment of laboratory performance

Twenty-seven laboratories out of 42 registered candidate laboratories reported results. A summary of the number of laboratories with satisfactory, questionable and unsatisfactory Z-scores for the respective levels of each biomarker, the applied evaluation scheme and the number of ND results without proxy-Z-score is given in **Table 1**.

Table 2 shows the results of each participating laboratory for each biomarker and also reflects how many PAH met. biomarkers have been analysed per participant (sum) and how many participants have analysed the low and the high level of the respective biomarkers (TOTAL).

Z-scores were calculated for the low and the high concentration level of **1-naphthol, 2-naphthol, 2-FLUO, 3-FLUO, 1-PHEN, 2-PHEN**, **3-PHEN, 4-PHEN** and **1-PYR** with graphical representations provided in **Appendix 7**.

The percentage of satisfactory Z-scores obtained for each biomarker ranged from 100% for **2-FLUO$_{low}$, 3-FLUO$_{high}$,1-PHEN$_{high}$** and **2-PHEN$_{low}$** to 62% for **1-PYR$_{low}$** and was 83% on average for the other biomarkers.

The calculation of expert values or consensus values was not possible for **1,2-DHN, 9-FLUO, 9-PHEN** and **3-BaP** due to the limited number of expert labs (smaller than three) or participants (smaller than seven) or a too high uncertainty of the assigned/consensus value, respectively. Thus, in these cases, no Z-scores could be provided.

A direct comparison of the overall performance of the laboratories with that of the first round is not entirely possible, because there were a lot of new laboratories participating in this round and also some laboratories from the first round did not participate in this second round. A high satisfactory performance rate was achieved for **2-FLUO** and **4-PHEN** while **1-PYR** was still more challenging.

6.4 Conclusions and recommendations

The overall participation in the HBM4EU EQUAS Round 2 was successful. Twenty-seven laboratories out of the 42 laboratories (64%) in the revised candidate list confirmed their participation in this EQUAS. All of these 27 registered candidate laboratories reported results, representing a participation rate of 100%.

The tables below (**Table 5** to **Table 17**) provide the LOQs and an overview of the performance of the candidate laboratories in this 1st EQUAS round 2 for all PAH met. biomarkers in urine.

Evaluation of laboratory performance was possible for nine biomarkers. The percentage of satisfactory Z-scores for the individual biomarkers was between 62% and 100%.

For each PAH met. biomarker, between three and eleven additional laboratories reported results in this ICI/EQUAS round compared to the first ICI round. There is only one participating laboratory that analysed all 13 biomarkers of PAH met. in urine, but could only provide the sum of **3-FLUO** and **9-FLUO**. The other laboratories measured a number of biomarkers in a scope between one and twelve. The quantity of satisfactory results was higher in the first round compared to this ICI/EQUAS round. In the first ICI round, no evaluation could be established for five biomarkers (**1,2-DHN,**

3-FLUO, 9-FLUO, 9-PHEN and **3-BaP**) while in this ICI/EQUAS round only for four biomarkers (**1,2-DHN, 9-FLUO, 9-PHEN** and **3-BaP**) no Z-scores could be provided. The reasons were an insufficient number of participating laboratories and no suitable assigned value derived from the expert results.

Table 5 Performance of the candidate laboratories for 1-naphtol in urine

Lab.code	LOQ [ng/ml]	1-naphthol$_{low}$	1-naphthol$_{high}$
QR/112	-	**NA**	**NA**
QR/113	0.059	**satisfactory**	**satisfactory**
QR/118	1.400	**unsatisfactory**	**unsatisfactory**
QR/122	0.100	**satisfactory**	**satisfactory**
QR/123	0.600	**unsatisfactory**	**unsatisfactory**
QR/124	1.000	**satisfactory**	**satisfactory**
QR/125	0.050	**satisfactory**	**satisfactory**
QR/126	0.025	**satisfactory**	**satisfactory**
QR/127	0.010	**satisfactory**	**satisfactory**
QR/129	-	**NA**	**NA**
QR/130	0.200	**unsatisfactory**	**questionable**
QR/132	-	**NA**	**NA**
QR/140	2.000	**satisfactory**	**satisfactory**
QR/141	0.500	**satisfactory**	**satisfactory**
QR/142	0.100	**satisfactory**	**satisfactory**
QR/143	-	**NA**	**NA**
QR/201	6.000	**satisfactory**	**satisfactory**
QR/202	-	**NA**	**NA**
QR/206	-	**NA**	**NA**
QR/207	-	**NA**	**NA**
QR/210	0.4000	**satisfactory**	**satisfactory**
QR/211	-	**NA**	**NA**
QR/217	-	**NA**	**NA**
QR/218	-	**NA**	**NA**
QR/219	0.013	**satisfactory**	**satisfactory**
QR/220	0.250	**satisfactory**	**satisfactory**
QR/221	0.806	**questionable**	**satisfactory**

NA = not analysed

Table 6 Performance of the candidate laboratories for 2-naphtol in urine

Lab.code	LOQ [ng/ml]	2-naphthol$_{low}$	2-naphthol$_{high}$
QR/112	-	**NA**	**NA**
QR/113	0.073	**satisfactory**	**satisfactory**
QR/118	1.400	**satisfactory**	**satisfactory**
QR/122	0.050	**satisfactory**	**satisfactory**
QR/123	0.700	**questionable**	**questionable**
QR/124	1.000	**satisfactory**	**satisfactory**
QR/125	0.050	**satisfactory**	**satisfactory**
QR/126	0.010	**satisfactory**	**satisfactory**
QR/127	0.010	**satisfactory**	**satisfactory**
QR/129	0.001	**satisfactory**	**satisfactory**
QR/130	0.200	**satisfactory**	**satisfactory**
QR/132	0.007	**satisfactory**	**satisfactory**
QR/140	0.500	**satisfactory**	**satisfactory**
QR/141	0.500	**satisfactory**	**satisfactory**
QR/142	0.400	**satisfactory**	**satisfactory**
QR/143	-	**NA**	**NA**
QR/201	6.000	**satisfactory**	**satisfactory**
QR/202	-	**NA**	**NA**
QR/206	-	**NA**	**NA**
QR/207	1.368	**satisfactory**	**satisfactory**
QR/210	0.400	**questionable**	**questionable**
QR/211	-	**NA**	**NA**
QR/217	-	**NA**	**NA**
QR/218	-	**NA**	**NA**
QR/219	0.013	**satisfactory**	**satisfactory**
QR/220	0.250	**satisfactory**	**satisfactory**
QR/221	0.511	**unsatisfactory**	**unsatisfactory**

NA = not analysed

Table 7 Performance of the candidate laboratories for 1,2-DHN in urine

Lab.code	LOQ [ng/ml]	1,2-DHN$_{low}$	1,2-DHN$_{high}$
QR/112	-	NA	NA
QR/113	-	NA	NA
QR/118	-	NA	NA
QR/122	-	NA	NA
QR/123	-	NA	NA
QR/124	-	NA	NA
QR/125	-	NA	NA
QR/126	0.500	ND (no proxy Z-score)	ND (no proxy Z-score)
QR/127	-	NA	NA
QR/129	-	NA	NA
QR/130	-	NA	NA
QR/132	-	NA	NA
QR/140	-	NA	NA
QR/141	-	NA	NA
QR/142	-	NA	NA
QR/143	-	NA	NA
QR/201	-	NA	NA
QR/202	-	NA	NA
QR/206	-	NA	NA
QR/207	-	NA	NA
QR/210	-	NA	NA
QR/211	-	NA	NA
QR/217	-	NA	NA
QR/218	-	NA	NA
QR/219	-	NA	NA
QR/220	-	NA	NA
QR/221	-	NA	NA

NA = not analysed

ND = not detected

Table 8 Performance of the candidate laboratories for 2-FLUO in urine

Lab.code	LOQ [ng/ml]	2-FLUO$_{low}$	2-FLUO$_{high}$
QR/112	0.100	**NA (sum of 2- and 3-FLUO)**	**NA (sum of 2- and 3-FLUO)**
QR/113	0.038	**satisfactory**	**satisfactory**
QR/118	-	**NA**	**NA**
QR/122	0.050	**satisfactory**	**satisfactory**
QR/123	-	**NA**	**NA**
QR/124	-	**NA**	**NA**
QR/125	0.005	**satisfactory**	**satisfactory**
QR/126	0.010	**satisfactory**	**questionable**
QR/127	0.010	**satisfactory**	**satisfactory**
QR/129	0.003	**satisfactory**	**satisfactory**
QR/130	0.200	**satisfactory**	**satisfactory**
QR/132	0.036	**NA (sum of 2- and 3-FLUO)**	**NA (sum of 2- and 3-FLUO)**
QR/140	-	**NA**	**NA**
QR/141	-	**NA**	**NA**
QR/142	-	**NA**	**NA**
QR/143	-	**NA**	**NA**
QR/201	-	**NA**	**NA**
QR/202	-	**NA**	**NA**
QR/206	-	**NA**	**NA**
QR/207	-	**NA**	**NA**
QR/210	0.020	**satisfactory**	**satisfactory**
QR/211	-	**NA**	**NA**
QR/217	-	**NA**	**NA**
QR/218	-	**NA**	**NA**
QR/219	0.013	**satisfactory**	**satisfactory**
QR/220	0.250	**satisfactory**	**satisfactory**
QR/221	-	**NA**	**NA**

NA = not analysed

Table 9 Performance of the candidate laboratories for 3-FLUO in urine

Lab.code	LOQ [ng/ml]	3-FLUO$_{low}$	3-FLUO$_{high}$
QR/112	0.100	NA (sum of 2- and 3-FLUO)	NA (sum of 2- and 3-FLUO)
QR/113	0.038	satisfactory	satisfactory
QR/118	-	NA	NA
QR/122	-	NA	NA
QR/123	-	NA	NA
QR/124	-	NA	NA
QR/125	0.005	satisfactory	satisfactory
QR/126	0.050	NA (sum of 3- and 9-FLUO)	NA (sum of 3- and 9-FLUO)
QR/127	0.050	satisfactory	satisfactory
QR/129	0.003	satisfactory	satisfactory
QR/130	-	NA	NA
QR/132	-	NA	NA
QR/140	-	NA	NA
QR/141	-	NA	NA
QR/142	-	NA	NA
QR/143	-	NA	NA
QR/201	-	NA	NA
QR/202	-	NA	NA
QR/206	-	NA	NA
QR/207	-	NA	NA
QR/210	-	NA	NA
QR/211	-	NA	NA
QR/217	-	NA	NA
QR/218	-	NA	NA
QR/219	0.013	unsatisfactory	satisfactory
QR/220	0.200	satisfactory	satisfactory
QR/221	-	NA	NA

NA = not analysed

Table 10 Performance of the candidate laboratories for 9-FLUO in urine

Lab. code	LOQ [ng/ml]	9-FLUO$_{low}$	9-FLUO$_{high}$
QR/112	0.100	**no Z-score available**	**no Z-score available**
QR/113	0.054	**no Z-score available**	**no Z-score available**
QR/118	-	**NA**	**NA**
QR/122	-	**NA**	**NA**
QR/123	-	**NA**	**NA**
QR/124	-	**NA**	**NA**
QR/125	0.010	**no Z-score available**	**no Z-score available**
QR/126	0.050	**NA (sum of 3- and 9-FLUO)**	**NA (sum of 3- and 9-FLUO)**
QR/127	-	**NA**	**NA**
QR/129	-	**NA**	**NA**
QR/130	-	**NA**	**NA**
QR/132	-	**NA**	**NA**
QR/140	-	**NA**	**NA**
QR/141	-	**NA**	**NA**
QR/142	-	**NA**	**NA**
QR/143	-	**NA**	**NA**
QR/201	-	**NA**	**NA**
QR/202	-	**NA**	**NA**
QR/206	-	**NA**	**NA**
QR/207	-	**NA**	**NA**
QR/210	-	**NA**	**NA**
QR/211	-	**NA**	**NA**
QR/217	-	**NA**	**NA**
QR/218	-	**NA**	**NA**
QR/219	-	**NA**	**NA**
QR/220	0.250	**no Z-score available**	**no Z-score available**
QR/221	-	**NA**	**NA**

NA = not analysed

Table 11 Performance of the candidate laboratories for 1-PHEN in urine

Lab.code	LOQ [ng/ml]	1-PHEN$_{low}$	1-PHEN$_{high}$
QR/112	0.1000	satisfactory	satisfactory
QR/113	0.031	questionable	satisfactory
QR/118	-	NA	NA
QR/122	0.005	satisfactory	satisfactory
QR/123	-	NA	NA
QR/124	-	NA	NA
QR/125	0.005	satisfactory	satisfactory
QR/126	0.010	satisfactory	satisfactory
QR/127	0.010	satisfactory	satisfactory
QR/129	0.003	satisfactory	satisfactory
QR/130	-	NA	NA
QR/132	0.009	NA (sum of 1- and 9-PHEN)	NA (sum of 1- and 9-PHEN)
QR/140	-	NA	NA
QR/141	0.010	satisfactory	satisfactory
QR/142	-	NA	NA
QR/143	-	NA	NA
QR/201	-	NA	NA
QR/202	-	NA	NA
QR/206	-	NA	NA
QR/207	-	NA	NA
QR/210	0.020	satisfactory	satisfactory
QR/211	-	NA	NA
QR/217	-	NA	NA
QR/218	-	NA	NA
QR/219	0.013	questionable	satisfactory
QR/220	0.200	satisfactory	satisfactory
QR/221	-	NA (sum of 1- and 9-PHEN)	NA (sum of 1- and 9-PHEN)

NA = not analysed

Table 12 Performance of the candidate laboratories for 2-PHEN in urine

Lab.code	LOQ [ng/ml]	2-PHEN$_{low}$	2-PHEN$_{high}$
QR/112	-	NA (sum of 2- and 3- PHEN)	NA (sum of 2- and 3- PHEN)
QR/113	0.051	satisfactory	questionable
QR/118	-	NA	NA
QR/122	0.005	satisfactory	satisfactory
QR/123	-	NA	NA
QR/124	-	NA	NA
QR/125	0.005	satisfactory	satisfactory
QR/126	0.0100	satisfactory	satisfactory
QR/127	0.010	satisfactory	satisfactory
QR/129	0.001	NA (sum of 2- and 3-PHEN)	NA (sum of 2- and 3- PHEN)
QR/130	-	NA	NA
QR/132	0.008	satisfactory	satisfactory
QR/140	-	NA	NA
QR/141	0.010	satisfactory	satisfactory
QR/142	-	NA	NA
QR/143	-	NA	NA
QR/201	-	NA	NA
QR/202	-	NA	NA
QR/206	-	NA	NA
QR/207	-	NA	NA
QR/210	0.020	satisfactory	questionable
QR/211	-	NA	NA
QR/217	-	NA	NA
QR/218	-	NA	NA
QR/219	0.013	ND (no proxy Z-score)	satisfactory
QR/220	0.100	satisfactory	questionable
QR/221	0.084	satisfactory	satisfactory

NA = not analysed

ND = not detected

Table 13 Performance of the candidate laboratories for 3-PHEN in urine

Lab.code	LOQ [ng/ml]	3-PHEN$_{low}$	3-PHEN$_{high}$
QR/112	-	NA (sum of 2- and 3- PHEN)	NA (sum of 2- and 3- PHEN)
QR/113	0.032	satisfactory	satisfactory
QR/118	-	NA	NA
QR/122	0.005	satisfactory	satisfactory
QR/123	-	NA	NA
QR/124	-	NA	NA
QR/125	0.005	satisfactory	satisfactory
QR/126	0.010	satisfactory	satisfactory
QR/127	0.01	satisfactory	satisfactory
QR/129	-	NA (sum of 2- and 3- PHEN)	NA (sum of 2- and 3- PHEN)
QR/130	-	NA	NA
QR/132	0.006	satisfactory	satisfactory
QR/140	-	NA	NA
QR/141	0.01	satisfactory	satisfactory
QR/142	-	NA	NA
QR/143	-	NA	NA
QR/201	-	NA	NA
QR/202	-	NA	NA
QR/206	-	NA	NA
QR/207	-	NA	NA
QR/210	0.02	satisfactory	satisfactory
QR/211	-	NA	NA
QR/217	-	NA	NA
QR/218	-	NA	NA
QR/219	0.013	unsatisfactory	unsatisfactory
QR/220	0.1	satisfactory	satisfactory
QR/221	0.051	questionable	questionable

NA = not analysed

Table 14 Performance of the candidate laboratories for 4-PHEN in urine

Lab. code	LOQ [ng/ml]	4-PHEN$_{low}$	4-PHEN$_{high}$
QR/112	0.100	**satisfactory**	**satisfactory**
QR/113	0.022	**questionable**	**questionable**
QR/118	-	**NA**	**NA**
QR/122	0.005	**satisfactory**	**satisfactory**
QR/123	-	**NA**	**NA**
QR/124	-	**NA**	**NA**
QR/125	0.010	**satisfactory**	**satisfactory**
QR/126	0.010	**satisfactory**	**satisfactory**
QR/127	0.010	**satisfactory**	**satisfactory**
QR/129	0.003	**satisfactory**	**satisfactory**
QR/130	-	**NA**	**NA**
QR/132	0.003	**satisfactory**	**satisfactory**
QR/140	-	**NA**	**NA**
QR/141	0.010	**satisfactory**	**satisfactory**
QR/142	-	**NA**	**NA**
QR/143	-	**NA**	**NA**
QR/201	-	**NA**	**NA**
QR/202	-	**NA**	**NA**
QR/206	-	**NA**	**NA**
QR/207	-	**NA**	**NA**
QR/210	0.040	**NA (sum of 4- and 9-PHEN)**	**NA (sum of 4- and 9-PHEN)**
QR/211	-	**NA**	**NA**
QR/217	-	**NA**	**NA**
QR/218	-	**NA**	**NA**
QR/219	0.013	**satisfactory**	**satisfactory**
QR/220	0.100	**satisfactory**	**satisfactory**
QR/221	0.086	**satisfactory**	**satisfactory**

NA = not analysed

Table 15 Performance of the candidate laboratories for 9-PHEN in urine

Lab.code	LOQ [ng/ml]	9-PHEN$_{low}$	9-PHEN$_{high}$
QR/112	-	NA	NA
QR/113	-	NA	NA
QR/118	-	NA	NA
QR/122	0.005	no Z-score available	no Z-score available
QR/123	-	NA	NA
QR/124	-	NA	NA
QR/125	0.010	no Z-score available	no Z-score available
QR/126	0.025	no Z-score available	no Z-score available
QR/127	0.010	no Z-score available	no Z-score available
QR/129	0.100	ND (no proxy Z-score)	ND (no proxy Z-score)
QR/130	0.200	no Z-score available	no Z-score available
QR/132	-	NA	NA
QR/140	-	NA	NA
QR/141	-	NA	NA
QR/142	-	NA	NA
QR/143	-	NA	NA
QR/201	-	NA	NA
QR/202	-	NA	NA
QR/206	-	NA	NA
QR/207	-	NA	NA
QR/210	0.040	NA (sum of 4- and 9-PHEN)	NA (sum of 4- and 9- PHEN)
QR/211	-	NA	NA
QR/217	-	NA	NA
QR/218	-	NA	NA
QR/219	0.025	no Z-score available	no Z-score available
QR/220	0.300	no Z-score available	no Z-score available
QR/221	0.174	NA (sum of 1- and 9- PHEN)	NA (sum of 1- and 9- PHEN)

NA = not analysed

ND = not detected

Table 16 Performance of the candidate laboratories for 1-PYR in urine

Lab.code	LOQ [ng/ml]	1-PYR$_{low}$	1-PYR$_{high}$
QR/112	0.100	satisfactory	satisfactory
QR/113	0.052	satisfactory	questionable
QR/118	0.200	unsatisfactory	satisfactory
QR/122	0.010	satisfactory	satisfactory
QR/123	0.050	satisfactory	satisfactory
QR/124	0.020	satisfactory	satisfactory
QR/125	0.010	satisfactory	satisfactory
QR/126	0.025	unsatisfactory	questionable
QR/127	0.010	satisfactory	satisfactory
QR/129	0.003	satisfactory	satisfactory
QR/130	0.200	unsatisfactory	questionable
QR/132	0.009	satisfactory	satisfactory
QR/140	0.200	satisfactory	satisfactory
QR/141	-	satisfactory	satisfactory
QR/142	-	NA	NA
QR/143	0.010	satisfactory	satisfactory
QR/201	0.050	questionable	satisfactory
QR/202	0.100	unsatisfactory	unsatisfactory
QR/206	0.200	questionable	satisfactory
QR/207	0.102	satisfactory	satisfactory
QR/210	0.050	satisfactory	satisfactory
QR/211	0.025	questionable	questionable
QR/217	0.100	satisfactory	satisfactory
QR/218	0.040	questionable	satisfactory
QR/219	0.013	unsatisfactory	unsatisfactory
QR/220	0.020	satisfactory	satisfactory
QR/221	0.025	unsatisfactory	questionable

NA = not analysed

Table 17 Performance of the candidate laboratories for 3-BaP in urine

Lab.code	LOQ [ng/ml]	3-BaP$_{low}$	3-BaP$_{high}$
QR/112	-	NA	NA
QR/113	-	NA	NA
QR/118	-	NA	NA
QR/122	0.00005	no Z-score available	no Z-score available
QR/123	-	NA	NA
QR/124	-	NA	NA
QR/125	-	NA	NA
QR/126	0.50000	ND (no proxy Z-score)	ND (no proxy Z-score)
QR/127	0.05000	ND (no proxy Z-score)	ND (no proxy Z-score)
QR/129	0.10000	ND (no proxy Z-score)	ND (no proxy Z-score)
QR/130	0.20000	NA	NA
QR/132	-	NA	NA
QR/140	-	NA	NA
QR/141	-	NA	NA
QR/142	-	NA	NA
QR/143	-	NA	NA
QR/201	0.00010	no Z-score available	no Z-score available
QR/202	-	NA	NA
QR/206	-	NA	NA
QR/207	-	NA	NA
QR/210	-	NA	NA
QR/211	-	NA	NA
QR/217	-	NA	NA
QR/218	-	NA	NA
QR/219	0.25000	ND (no proxy Z-score)	ND (no proxy Z-score)
QR/220	0.00010	no Z-score available	no Z-score available
QR/221	0.09900	ND (no proxy Z-score)	ND (no proxy Z-score)

NA = not analysed

ND = not detected

7 References

[1] Analytical Methods Committee, 1989a, Robust statistics - How not to reject outliers Part 1. Basic concepts, Analyst, 114, 1693-1697.

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Appendix 1. Homogeneity data

	1-naphthol				2-naphthol			
	low [ng/ml]		high [ng/ml]		low [ng/ml]		high [ng/ml]	
	replicate 1	replicate 2	replicate 1	replicate 2	replicate 1	replicate 2	replicate 1	replicate 2
1	1.33	1.30	2.64	2.67	10.09	10.09	15.46	15.96
2	1.38	1.35	2.58	2.55	10.02	10.06	15.48	15.54
3	1.32	1.31	2.55	2.60	9.18	10.01	15.21	15.54
4	1.15	1.18	2.87	3.04	9.23	9.31	13.41	13.85
5	1.15	1.17	2.87	3.05	9.25	9.01	14.93	15.23
6	1.38	1.37	2.97	3.09	8.99	9.36	14.87	15.13
7								
8								
9								
10								
Grand mean	1.283		2.790		9.550		15.051	
Cochran`s test								
C	0.273		0.405		0.773		0.350	
Ccrit	0.781		0.781		0.781		0.781	
C < Ccrit?	no outliers detected		no outliers detected		no outliers detected		no outliers detected	
target σ_{FFP}:	0.321		0.698		2.388		3.763	
s_x	0.096		0.214		0.431		0.745	
s_w	0.017		0.082		0.273		0.244	
s_s	0.095		0.206		0.386		0.724	
Critical=0.3 σ_{FFP}	0.096		0.209		0.716		1.129	
s_s < critical?	Homogeneity adequate		Homogeneity adequate		Homogeneity adequate		Homogeneity adequate	
s_w< 0.5*σ_{FFP}?	Method suited		Method suited		Method suited		Method suited	

Appendix 1. Homogeneity data (continued)

	1,2-DHN				2-FLUO			
	low [ng/ml]		high [ng/ml]		low [ng/ml]		high [ng/ml]	
	replicate 1	replicate 2	replicate 1	replicate 2	replicate 1	replicate 2	replicate 1	replicate 2
1	3.69	3.69	6.35	6.61	0.42	0.44	0.82	0.85
2	3.60	3.67	6.52	6.34	0.42	0.44	0.84	0.82
3	3.61	3.69	6.39	6.35	0.46	0.43	0.86	0.86
4	3.61	3.76	6.19	6.48	0.38	0.38	0.70	0.72
5	3.78	3.77	6.36	6.46	0.37	0.39	0.82	0.87
6	3.60	3.74	6.30	6.45	0.39	0.36	0.84	0.80
7								
8								
9								
10								
Grand mean	3.684		6.400		0.407		0.817	
Cochran`s test								
C	0.421		0.385		0.300		0.431	
Ccrit	0.781		0.781		0.781		0.781	
C < Ccrit?	no outliers detected		no outliers detected		no outliers detected		no outliers detected	
target σ_{FFP}:	0.921		1.600		0.102		0.204	
s_x	0.049		0.051		0.032		0.054	
s_w	0.067		0.135		0.016		0.022	
s_s	0.014		0.000		0.030		0.052	
Critical=0.3 σ_{FFP}	0.276		0.480		0.031		0.061	
s_s < critical?	Homogeneity adequate		Homogeneity adequate		Homogeneity adequate		Homogeneity adequate	
s_w< 0.5*σ_{FFP}?	Method suited		Method suited		Method suited		Method suited	

Appendix 1. Homogeneity data (continued)

	3-FLUO				9-FLUO			
	low [ng/ml]		high [ng/ml]		low [ng/ml]		high [ng/ml]	
	replicate 1	replicate 2	replicate 1	replicate 2	replicate 1	replicate 2	replicate 1	replicate 2
1	0.23	0.22	0.32	0.34	0.64	0.60	1.29	1.24
2	0.25	0.26	0.37	0.36	0.64	0.65	1.33	1.38
3	0.26	0.25	0.33	0.36	0.66	0.65	1.31	1.25
4	0.24	0.22	0.28	0.31	0.62	0.66	1.29	1.27
5	0.23	0.24	0.39	0.30	0.58	0.60	1.26	1.19
6	0.22	0.23	0.38	0.32	0.64	0.62	1.20	1.28
7								
8								
9								
10								
Grand mean	0.238		0.338		0.630		1.274	
Cochran`s test								
C	0.444		0.579		0.381		0.315	
Ccrit	0.781		0.781		0.781		0.781	
C < Ccrit?	no outliers detected		no outliers detected		no outliers detected		no outliers detected	
target σ_{FFP}:	0.059		0.085		0.158		0.319	
s_x	0.014		0.024		0.023		0.045	
s_w	0.009		0.034		0.019		0.041	
s_s	0.013		0.000		0.019		0.035	
Critical=0.3 σ_{FFP}	0.018		0.025		0.047		0.096	
s_s < critical?	Homogeneity adequate		Homogeneity adequate		Homogeneity adequate		Homogeneity adequate	
s_w< 0.5*σ_{FFP}?	Method suited		Method suited		Method suited		Method suited	

Appendix 1. Homogeneity data (continued)

	1-PHEN				2-PHEN			
	low [ng/ml]		high [ng/ml]		low [ng/ml]		high [ng/ml]	
	replicate 1	replicate 2	replicate 1	replicate 2	replicate 1	replicate 2	replicate 1	replicate 2
1	0.25	0.26	0.82	0.82	0.66	0.58	0.85	0.82
2	0.26	0.27	0.79	0.81	0.66	0.61	0.78	0.80
3	0.22	0.25	0.73	0.76	0.62	0.66	0.78	0.80
4	0.27	0.29	0.76	0.77	0.55	0.52	0.72	0.76
5	0.26	0.24	0.83	0.85	0.53	0.56	0.75	0.78
6	0.27	0.30	0.84	0.87	0.56	0.62	0.78	0.80
7								
8								
9								
10								
Grand mean	0.262		0.804		0.594		0.785	
Cochran`s test								
C	0.321		0.333		0.403		0.348	
Ccrit	0.781		0.781		0.781		0.781	
C < Ccrit?	no outliers detected		no outliers detected		no outliers detected		no outliers detected	
target σ_{FFP}:	0.065		0.201		0.149		0.196	
s_x	0.019		0.043		0.046		0.032	
s_w	0.015		0.015		0.036		0.020	
s_s	0.015		0.041		0.038		0.028	
Critical=0.3 σ_{FFP}	0.020		0.060		0.045		0.059	
s_s < critical?	Homogeneity adequate		Homogeneity adequate		Homogeneity adequate		Homogeneity adequate	
s_w< 0.5*σ_{FFP}?	Method suited		Method suited		Method suited		Method suited	

Appendix 1. Homogeneity data (continued)

	3-PHEN				4-PHEN			
	low [ng/ml]		high [ng/ml]		low [ng/ml]		high [ng/ml]	
	replicate 1	replicate 2	replicate 1	replicate 2	replicate 1	replicate 2	replicate 1	replicate 2
1	0.25	0.26	0.44	0.44	0.22	0.22	0.36	0.36
2	0.26	0.27	0.44	0.45	0.24	0.21	0.33	0.33
3	0.22	0.25	0.45	0.47	0.21	0.21	0.32	0.32
4	0.27	0.29	0.46	0.48	0.21	0.21	0.35	0.34
5	0.26	0.24	0.48	0.48	0.24	0.21	0.38	0.35
6	0.27	0.30	0.46	0.49	0.21	0.28	0.36	0.38
7								
8								
9								
10								
Grand mean	0.262		0.462		0.233		0.348	
Cochran`s test								
C	0.321		0.500		0.731		0.643	
Ccrit	0.781		0.781		0.781		0.781	
C < Ccrit?	no outliers detected		no outliers detected		no outliers detected		no outliers detected	
target σ_{FFP}:	0.065		0.115		0.056		0.087	
s_x	0.019		0.016		0.013		0.020	
s_w	0.015		0.012		0.024		0.011	
s_s	0.015		0.014		0.000		0.019	
Critical=0.3 σ_{FFP}	0.020		0.035		0.017		0.026	
s_s < critical?	Homogeneity adequate		Homogeneity adequate		Homogeneity adequate		Homogeneity adequate	
s_w< 0.5*σ_{FFP}?	Method suited		Method suited		Method suited		Method suited	

Appendix 1. Homogeneity data (continued)

	9-PHEN				1-PYR			
	low [ng/ml]		high [ng/ml]		low [ng/ml]		high [ng/ml]	
	replicate 1	replicate 2	replicate 1	replicate 2	replicate 1	replicate 2	replicate 1	replicate 2
1	0.31	0.28	0.31	0.28	0.07	0.06	0.17	0.15
2	0.30	0.25	0.31	0.31	0.08	0.07	0.18	0.19
3	0.28	0.24	0.30	0.31	0.06	0.06	0.15	0.18
4	0.25	0.24	0.30	0.33	0.07	0.07	0.22	0.17
5	0.28	0.32	0.30	0.32	0.08	0.06	0.19	0.22
6	0.29	0.31	0.32	0.31	0.07	0.07	0.19	0.17
7								
8								
9								
10								
Grand mean	0.279		0.308		0.068		0.182	
Cochran`s test								
C	0.352		0.375		0.667		0.481	
Ccrit	0.781		0.781		0.781		0.781	
C < Ccrit?	no outliers detected		no outliers detected		no outliers detected		no outliers detected	
target σ_{FFP}:	0.070		0.077		0.017		0.045	
s_x	0.023		0.008		0.005		0.017	
s_w	0.024		0.014		0.007		0.021	
s_s	0.015		0.000		0.001		0.009	
Critical=0.3 σ_{FFP}	0.021		0.023		0.005		0.014	
s_s < critical?	Homogeneity adequate		Homogeneity adequate		Homogeneity adequate		Homogeneity adequate	
s_w< 0.5*σ_{FFP}?	Method suited		Method suited		Method suited		Method suited	

Appendix 1. Homogeneity data (continued)

	3-BaP			
	low [ng/ml]		high [ng/ml]	
	replicate 1	replicate 2	replicate 1	replicate 2
1	0.002060	0.001568	0.003683	0.003247
2	0.002040	0.001539	0.003473	0.003804
3	0.001842	0.001936	0.003773	0.003430
4	0.002097	0.001705	0.003506	0.003402
5	0.001696	0.001787	0.003673	0.002953
6	0.001838	0.002007	0.003362	0.003493
7	0.001952	0.001791	0.003654	0.003555
8	0.001913	0.001863	0.003661	0.003556
9	0.001790	0.001761	0.003554	0.003835
10	0.002068	0.001897	0.003448	0.003170
Grand mean	0.001857		0.003511	
Cochran`s test				
C	0.334		0.454	
Ccrit	0.602		0.602	
C < Ccrit?	no outliers detected		no outliers detected	
target σ_{FFP}:	0.000464		0.000878	
s_x	0.000075		0.000137	
s_w	0.000194		0.000239	
s_s	0.000		0.000	
Critical=0.3 σ_{FFP}	0.000139		0.000263	
s_s < critical?	Homogeneity adequate		Homogeneity adequate	
s_w< 0.5*σ_{FFP}?	Method suited		Method suited	

Appendix 2. Stability data

	1-naphthol				2-naphthol			
	low [ng/ml]		high [ng/ml]		low [ng/ml]		high [ng/ml]	
	-80°C	-18°C	-80°C	-18°C	-80°C	-18°C	-80°C	-18°C
1	1.340	1.220	2.520	2.420	10.330	10.040	16.120	15.410
2	1.450	1.410	2.460	2.510	10.240	9.990	15.270	15.890
3	1.300	1.310	2.400	2.600	10.100	9.840	15.910	15.690
4	1.290	1.300	2.400	2.350	10.080	9.960	15.720	16.040
5	1.220	1.340	2.570	2.620	10.120	10.000	15.810	15.470
6	1.330	1.390	2.520	2.460	9.750	9.800	15.770	15.700
average	1.322	1.328	2.478	2.493	10.103	9.938	15.767	15.700
stdev	0.076	0.069	0.070	0.105	0.198	0.096	0.281	0.240
difference	-0.007		-0.015		0.165		0.067	
critical=0.3 σ_{FFP}	0.099		0.186		0.758		1.183	
consequential instability	no		no		no		no	
t	0.160		0.292		1.838		0.441	
tcrit	2.228		2.228		2.228		2.228	
Significant difference	no		no		no		no	

Appendix 2. Stability data (continued)

	1,2-DHN				2-FLUO			
	low [ng/ml]		high [ng/ml]		low [ng/ml]		high [ng/ml]	
	-80°C	-18°C	-80°C	-18°C	-80°C	-18°C	-80°C	-18°C
1	3.640	3.550	6.480	6.420	0.430	0.430	0.840	0.880
2	3.760	3.590	6.480	6.330	0.440	0.390	0.890	0.840
3	3.660	3.450	6.120	6.090	0.370	0.410	0.870	0.880
4	3.480	3.490	6.120	6.140	0.410	0.420	0.840	0.880
5	3.470	3.370	6.150	6.060	0.420	0.510	0.810	0.850
6	3.490	3.560	6.350	6.400	0.410	0.410	0.840	0.880
average	3.583	3.502	6.283	6.240	0.413	0.428	0.848	0.868
stdev	0.120	0.082	0.175	0.162	0.024	0.042	0.028	0.018
difference	0.082		0.043		-0.015		-0.020	
critical=0.3 σ_{FFP}	0.269		0.471		0.031		0.064	
consequential instability	no		no		no		no	
t	1.372		0.445		0.756		1.468	
tcrit	2.228		2.228		2.228		2.228	
Significant difference	no		no		no		no	

Appendix 2. Stability data (continued)

	3-FLUO				9-FLUO			
	low [ng/ml]		high [ng/ml]		low [ng/ml]		high [ng/ml]	
	-80°C	-18°C	-80°C	-18°C	-80°C	-18°C	-80°C	-18°C
1	0.280	0.250	0.370	0.340	0.780	0.760	1.420	1.430
2	0.260	0.280	0.370	0.410	0.780	0.780	1.450	1.410
3	0.260	0.260	0.370	0.380	0.780	0.780	1.440	1.450
4	0.260	0.280	0.370	0.380	0.750	0.790	1.400	1.460
5	0.280	0.280	0.360	0.410	0.770	0.750	1.460	1.420
6	0.270	0.260	0.340	0.380	0.750	0.790	1.460	1.420
average	0.268	0.268	0.363	0.383	0.768	0.775	1.438	1.432
stdev	0.010	0.013	0.012	0.026	0.015	0.016	0.024	0.019
difference	0.000		-0.020		-0.007		0.007	
critical=0.3 σ_{FFP}	0.020		0.027		0.058		0.108	
consequential instability	no		no		no		no	
t	0.000		1.718		0.740		0.529	
tcrit	2.228		2.228		2.228		2.228	
Significant difference	no		no		no		no	

Appendix 2. Stability data (continued)

	1-PHEN				2-PHEN			
	low [ng/ml]		high [ng/ml]		low [ng/ml]		high [ng/ml]	
	-80°C	-18°C	-80°C	-18°C	-80°C	-18°C	-80°C	-18°C
1	0.580	0.550	0.920	0.980	0.710	0.750	0.890	0.890
2	0.590	0.570	0.960	0.930	0.740	0.750	0.900	0.940
3	0.600	0.540	0.950	0.900	0.730	0.760	0.980	0.950
4	0.540	0.570	0.890	0.920	0.760	0.750	0.900	0.910
5	0.570	0.600	0.930	0.900	0.720	0.710	0.900	0.930
6	0.570	0.560	0.960	0.920	0.730	0.750	0.920	0.980
average	0.575	0.565	0.935	0.925	0.732	0.745	0.915	0.933
stdev	0.021	0.021	0.027	0.029	0.017	0.018	0.033	0.031
difference	0.010		0.010		-0.013		-0.018	
critical=0.3 σ_{FFP}	0.043		0.070		0.055		0.069	
consequential instability	no		no		no		no	
t	0.835		0.609		1.326		0.981	
tcrit	2.228		2.228		2.228		2.228	
Significant difference	no		no		no		no	

Appendix 2. Stability data (continued)

	3-PHEN				4-PHEN			
	low [ng/ml]		high [ng/ml]		low [ng/ml]		high [ng/ml]	
	-80°C	-18°C	-80°C	-18°C	-80°C	-18°C	-80°C	-18°C
1	0.290	0.270	0.430	0.460	0.240	0.260	0.390	0.400
2	0.280	0.210	0.410	0.480	0.240	0.230	0.370	0.410
3	0.210	0.280	0.500	0.410	0.260	0.270	0.380	0.390
4	0.230	0.290	0.410	0.490	0.260	0.260	0.380	0.390
5	0.260	0.230	0.410	0.400	0.240	0.250	0.360	0.410
6	0.260	0.280	0.470	0.400	0.240	0.250	0.400	0.380
average	0.255	0.260	0.438	0.440	0.247	0.253	0.380	0.397
stdev	0.030	0.032	0.038	0.041	0.010	0.014	0.014	0.012
difference	-0.005		-0.002		-0.007		-0.017	
critical=0.3 σ_{FFP}	0.019		0.033		0.019		0.029	
consequential instability	no		no		no		no	
t	0.277		0.072		0.953		2.193	
tcrit	2.228		2.228		2.228		2.228	
Significant difference	no		no		no		no	

Appendix 2. Stability data (continued)

	9-PHEN				1-PYR			
	low [ng/ml]		high [ng/ml]		low [ng/ml]		high [ng/ml]	
	-80°C	-18°C	-80°C	-18°C	-80°C	-18°C	-80°C	-18°C
1	0.360	0.420	0.400	0.370	0.060	0.050	0.140	0.100
2	0.420	0.350	0.400	0.410	0.040	0.040	0.150	0.160
3	0.360	0.450	0.400	0.430	0.070	0.060	0.130	0.150
4	0.350	0.430	0.390	0.430	0.060	0.070	0.140	0.140
5	0.430	0.380	0.350	0.360	0.050	0.060	0.140	0.130
6	0.430	0.360	0.390	0.380	0.060	0.060	0.110	0.140
average	0.392	0.398	0.388	0.397	0.057	0.057	0.135	0.137
stdev	0.039	0.041	0.019	0.031	0.010	0.010	0.014	0.021
difference	-0.007		-0.008		0.000		-0.002	
critical=0.3 σ_{FFP}	0.029		0.029		0.004		0.010	
consequential instability	no		no		no		no	
t	0.291		0.561		0.000		0.164	
tcrit	2.228		2.228		2.228		2.228	
Significant difference	no		no		no		no	

Appendix 2. Stability data (continued)

	3-BaP			
	low [ng/ml]		high [ng/ml]	
	-80°C	-18°C	-80°C	-18°C
1	0.001713	0.001628	0.003533	0.003517
2	0.001696	0.001565	0.003478	0.003376
3	0.001715	0.001629	0.003333	0.003195
4	0.001798	0.001720	0.003370	0.003562
5	0.001644	0.001650	0.003516	0.003325
6	0.001632	0.001593	0.003751	0.003582
average	0.001700	0.001631	0.003497	0.003426
stdev	0.000060	0.000053	0.000148	0.000153
difference	0.000069		0.000071	
critical=0.3 σ_{FFP}	0.000127		0.000262	
consequential instability	no		no	
t	2.118		0.814	
tcrit	2.228		2.228	
Significant difference	no		no	

Appendix 3. Copy of letter of invitation

HBM4EU: Announcement / invitation to participate in ICI / EQUAS study

OH-PAH/Round 2

Title of ICI/EQUAS: PAH metabolites in urine

Dear Colleague,

within the frame of HBM4EU the

Institute and Outpatient Clinic for Occupational-, Social- and Environmental Medicine

Friedrich-Alexander University Erlangen-Nuremberg

Henkestr. 9-11

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Germany

announces the 2nd round of ICI/EQUAS for the determination of PAH metabolites in urine.
The aim of ICI/EQUAS exercises is to provide laboratories with an assessment of their analytical performance and reliability of their data in comparison with other laboratories and/or expert laboratories. This will aid in the quality improvement of analysis in human biomonitoring at each of the laboratories.

Participation is mandatory for laboratories analysing samples in the frame of HBM4EU. Participants meet the quality criteria of the HBM4EU call if they pass 3 of ICI/EQUAS rounds successfully.

Test samples
The matrix will be urine. Accordingly, the participants will receive:

- 2 different materials of urine (3 samples of 5ml each) for determination of PAH metabolites in urine

Target biomarkers
For the biomarkers potentially present in the test samples, please see registration form for OH-PAH/Round 2. We would be pleased if your laboratory could analyse as many metabolites as possible.
LOQs should allow the analysis of PAH metabolites in samples of the general population.

Calendar:

Registration deadline	19-11-2018
Distribution of test samples (projected)	20-11-2018
Deadline for submission of results (projected)	18-12-2018

Registration

For registration, please find attached a registration form for PAH metabolites in urine. Please send it back to us by mail in case you want to register.
Upon registration, the participant will receive a lab-code to be used for submission of results.

Fee

For partners and linked-third parties of HBM4EU, participation is free of charge. Please note that the participant is responsible for custom clearance and associated costs if applicable.

Confidentiality:

All laboratory-specific information will be treated confidentially, and will never be disclosed to third parties (government, accreditation bodies) except the HBM4EU QAU, without permission of the laboratory.

Contact information organiser:

Coordinators:

- Prof. Dr. Thomas Göen
- Barbara Schaller, MD
- Moritz Schäfer
- Karin H. A. Zarrabi

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Appendix 4. Copy of letter/instructions sent together with test samples

HBM4EU: Instruction letter ICI / EQUAS study OH-PAH/Round2

Title of ICI/EQUAS: PAH metabolites in urine

Dear participant,

Thank you for participation in HBM4EU ICI/EQUAS study OH-PAH in urine/Round 2 for the determination of PAH metabolites in urine.

You will receive a parcel containing 6 test samples spiked with the biomarker in 2 levels, 3 of each concentration. Each sample consists of approximately 5 ml urine.

The parcel will be shipped on 27.11.2018 under frozen conditions.

Instructions:

- Upon receipt, please check the content for any damage/leakage of the containers, complete the sample receipt form and return it to the organiser.
- Store the test samples under frozen (-18°C) conditions until analysis.
- Analyse the samples for the biomarkers indicated in the invitation letter ref/ 06.11.2018.
- Thaw the samples and re-homogenise them according to your own procedure.
- Analyse the samples using the same procedure as will be used for analysis of samples in the frame of HBM4EU.
- Carry out a single analysis for each sample.
- For submission of results and method information use the forms provided.
- The deadline for submission of analysis results and method details is 31.12.2018

If you have any questions or need any assistance, please contact:

Karin Zarrabi, Stefanie Nübler or Moritz Schäfer

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Prof. Dr. Thomas Göen (for the ICI/EQUAS organisers)

Appendix 5.

Method information form for participation in ICI/EQUAS OH-PAH in urine (OH-PAH/Round 2)

Laboratory code	**IPASUM**	
ISO17025 accredited	No	
SAMPLE PREPARATION		
amount sample extracted	2 ml	
Deconjugation	Yes	
- chemical	-	
- enzymatic	β-Glucuronidase/Arylsulfatase / 37 °C / 16 h	
Extraction		
- pH adjustment	-	
- LLE;	-	
- SPE; material	Unmodified polystyrene-divinyl benzene copolymer	
Cleanup	**-**	
- LLE; solvent(s)		
- SPE; material		
Derivatisation		
- reagent	N-Methyl-N-(trimethylsilyl)trifluoracetamid (MSTFA)	
INSTRUMENTAL ANALYSIS		
HPLC	**-**	
- injection volume		
- column stationary phase		
- column L (mm) x ID (mm); dp		
- temperature		
- mobile phase A		
- mobile phase B		
- flow rate		
GC		
- injector	Splitless	
- injection volume	1.2 µl	
- column stationary phase	HP-5MS Ultra Inert	
- column L (m) x ID (mm) df (µm)	60 m x 0,25 mm x 0,25 µm	
- carrier	Helium	
- flow rate / inlet pressure	1 ml/min / 20.969 psi	
Detection		
MS	Single quad	
other		
Quantification		
Use of internal standard (IS)	Yes	
- isotopic label	Yes	
- other		
- moment of addition	Before deconjugation	
- response normalised to IS	Yes	
Calibration	Matrix-matched (addition to blank matrix before extraction)	
	Multi-level	
Correction for recovery	No	
Identification criteria used		
- retention time tolerance	Yes	
- number of ions/transitions	Yes	
- ion ratio tolerance	No	

Appendix 6. Assigned values and participant's performance for PAH met. in urine

HBM4EU 02/2018	1-naphthol			
PAH met. (urine)	1-naphthol$_{low}$		1-naphthol$_{high}$	
assigned value from 5 experts	2.008 ng/ml		3.090 ng/ml	
expert standard deviation	0.640 ng/ml		0.742 ng/ml	
uncertainty of assigned value (u)	14%		11%	
relative target standard deviation (σ_T)	25%		25%	
0.7 * σ_T	17.5%		17.5%	
study RSD_R	44%		32%	
laboratory code	value	Z-score	value	Z-score
QR/112	NA	X	NA	X
QR/113	2.242	0.466	3.198	0.140
QR/118	7.300	10.544	8.533	7.045
QR/122	1.413	-1.184	2.285	-1.042
QR/123	11.867	19.643	10.333	9.375
QR/124	1.413	-1.184	2.367	-0.937
QR/125	2.727	1.433	4.003	1.182
QR/126	1.281	-1.448	1.977	-1.441
QR/127	2.329	0.640	3.622	0.689
QR/129	NA	X	NA	X
QR/130	3.670	3.312	4.880	2.316
QR/132	NA	X	NA	X
QR/140	2.316	0.614	3.341	0.324
QR/141	2.164	0.312	3.317	0.294
QR/142	1.271	-1.468	1.854	-1.600
QR/143	NA	X	NA	X
QR/201	2.510	1.001	3.293	0.263
QR/202	NA	X	NA	X
QR/206	NA	X	NA	X
QR/207	NA	X	NA	X
QR/210	1.926	-0.162	2.781	-0.400
QR/211	NA	X	NA	X
QR/217	NA	X	NA	X
QR/218	NA	X	NA	X
QR/219	2.923	1.824	4.073	1.272
QR/220	2.190	0.363	3.153	0.082
QR/221	0.998	-2.012	3.138	0.062

NA = not analysed; X = no Z-score available

Appendix 6. Assigned values and participant's performance for PAH met. in urine (continued)

HBM4EU 02/2018	2-naphthol			
PAH met. (urine)	2-naphthol$_{low}$		2-naphthol$_{high}$	
assigned value from 5 experts	9.355 ng/ml		14.271 ng/ml	
expert standard deviation	1.577 ng/ml		2.221 ng/ml	
uncertainty of assigned value (u)	8%		7%	
relative target standard deviation (σ_T)	25%		25%	
0.7 * σ_T	17.5%		17.5%	
study RSD_R	22%		22%	
Laboratory code	value	Z-score	value	Z-score
QR/112	NA	X	NA	X
QR/113	7.360	-0.853	10.813	-0.969
QR/118	8.333	-0.437	12.967	-0.366
QR/122	11.058	0.728	16.162	0.530
QR/123	3.567	-2.475	5.033	-2.589
QR/124	9.777	0.180	14.900	0.176
QR/125	9.970	0.263	15.020	0.210
QR/126	6.835	-1.078	10.675	-1.008
QR/127	8.029	-0.567	12.925	-0.377
QR/129	9.237	-0.051	13.300	-0.272
QR/130	8.205	-0.492	12.220	-0.575
QR/132	8.820	-0.229	13.233	-0.291
QR/140	8.545	-0.346	12.707	-0.438
QR/141	9.450	0.040	14.470	0.056
QR/142	9.574	0.094	15.157	0.248
QR/143	NA	X	NA	X
QR/201	6.977	-1.017	10.900	-0.945
QR/202	NA	X	NA	X
QR/206	NA	X	NA	X
QR/207	6.359	-1.281	10.428	-1.077
QR/210	2.478	-2.940	4.789	-2.658
QR/211	NA	X	NA	X
QR/217	NA	X	NA	X
QR/218	NA	X	NA	X
QR/219	11.033	0.718	18.200	1.101
QR/220	9.507	0.065	14.490	0.061
QR/221	19.920	4.517	27.933	3.829

NA = not analysed; X = no Z-score available

Appendix 6. Assigned values and participant's performance for PAH met. in urine (continued)

HBM4EU 02/2018	1,2-DHN			
PAH met. (urine)	1,2-DHN$_{low}$		1,2-DHN$_{high}$	
assigned/consensus value	X		X	
no ICI/EQUAS evaluation due to	no candidate values		no candidate values	
Laboratory code	value	Z-score	value	Z-score
QR/112	NA	X	NA	X
QR/113	NA	X	NA	X
QR/118	NA	X	NA	X
QR/122	NA	X	NA	X
QR/123	NA	X	NA	X
QR/124	NA	X	NA	X
QR/125	NA	X	NA	X
QR/126	ND	X	ND	X
QR/127	NA	X	NA	X
QR/129	NA	X	NA	X
QR/130	NA	X	NA	X
QR/132	NA	X	NA	X
QR/140	NA	X	NA	X
QR/141	NA	X	NA	X
QR/142	NA	X	NA	X
QR/143	NA	X	NA	X
QR/201	NA	X	NA	X
QR/202	NA	X	NA	X
QR/206	NA	X	NA	X
QR/207	NA	X	NA	X
QR/210	NA	X	NA	X
QR/211	NA	X	NA	X
QR/217	NA	X	NA	X
QR/218	NA	X	NA	X
QR/219	NA	X	NA	X
QR/220	NA	X	NA	X
QR/221	NA	X	NA	X

NA = not analysed; X = no Z-score available; ND = not detected and no proxy Z-score available

Appendix 6. Assigned values and participant's performance for PAH met. in urine (continued)

HBM4EU 02/2018	2-FLUO			
PAH met. (urine)	2-FLUO$_{low}$		2-FLUO$_{high}$	
assigned value from 4 experts	0.436 ng/ml		0.827ng/ml	
expert standard deviation	0.124 ng/ml		0.238ng/ml	
uncertainty of assigned value (u)	14%		14%	
relative target standard deviation (σ_T)	25%		25%	
0.7 * σ_T	17.5%		17.5%	
study RSD$_R$	28%		23%	
Laboratory code	value	Z-score	value	Z-score
QR/112	NA (sum of 2- and 3-FLUO)	X	NA (sum of 2- and 3-FLUO)	X
QR/113	0.334	-0.936	0.581	-1.191
QR/118	NA	X	NA	X
QR/122	0.617	1.662	1.150	1.558
QR/123	NA	X	NA	X
QR/124	NA	X	NA	X
QR/125	0.543	0.986	0.987	0.770
QR/126	0.644	1.910	1.241	2.001
QR/127	0.469	0.304	0.979	0.731
QR/129	0.525	0.821	1.030	0.979
QR/130	0.320	-1.063	0.720	-0.519
QR/132	NA (sum of 2- and 3-FLUO)	X	NA (sum of 2- and 3-FLUO)	X
QR/140	NA	X	NA	X
QR/141	NA	X	NA	X
QR/142	NA	X	NA	X
QR/143	NA	X	NA	X
QR/201	NA	X	NA	X
QR/202	NA	X	NA	X
QR/206	NA	X	NA	X
QR/207	NA	X	NA	X
QR/210	0.244	-1.761	0.462	-1.767
QR/211	NA	X	NA	X
QR/217	NA	X	NA	X
QR/218	NA	X	NA	X
QR/219	0.610	1.601	1.120	1.414
QR/220	0.630	1.781	1.190	1.753
QR/221	NA	X	NA	X

NA = not analysed; X = no Z-score available

Appendix 6. Assigned values and participant's performance for PAH met. in urine (continued)

HBM4EU 02/2018	3-FLUO			
PAH met. (urine)	3-FLUO$_{low}$		3-FLUO$_{high}$	
assigned value from 3 experts (low)/ consensus value ICI (high)	0.214 ng/ml		0.389 ng/ml	
expert/robust standard deviation	0.062 ng/ml		0.078 ng/ml	
uncertainty of assigned value (u)	16.6 %		0.035 ng/ml	
relative target standard	25%		25%	
0.7 * σ_T	17.5%		17.5%	
study RSD_R	33%		20%	
Laboratory code	value	Z-score	value	Z-score
QR/112	NA (sum of 2- and 3-FLUO)	X	NA (sum of 2- and 3-FLUO)	X
QR/113	0.145	-1.299	0.185	-1.923
QR/118	NA	X	NA	X
QR/122	NA	X	NA	X
QR/123	NA	X	NA	X
QR/124	NA	X	NA	X
QR/125	0.270	1.041	0.363	-0.247
QR/126	NA (sum of 3- and 9-FLUO)	X	NA (sum of 3- and 9-FLUO)	X
QR/127	0.208	-0.116	0.526	1.327
QR/129	0.240	0.481	0.334	-0.531
QR/130	NA	X	NA	X
QR/132	NA	X	NA	X
QR/140	NA	X	NA	X
QR/141	NA	X	NA	X
QR/142	NA	X	NA	X
QR/143	NA	X	NA	X
QR/201	NA	X	NA	X
QR/202	NA	X	NA	X
QR/206	NA	X	NA	X
QR/207	NA	X	NA	X
QR/210	NA	X	NA	X
QR/211	NA	X	NA	X
QR/217	NA	X	NA	X
QR/218	NA	X	NA	X
QR/219	0.382	3.139	0.388	-0.011
QR/220	0.313	1.851	0.427	0.367
QR/221	NA	X	NA	X

NA = not analysed; X = no Z-score available

Appendix 6. Assigned values and participant's performance for PAH met. in urine (continued)

HBM4EU 02/2018	9-FLUO			
PAH met. (urine)	9-FLUO$_{low}$		9-FLUO$_{high}$	
assigned/consensus value	X		X	
no ICI/EQUAS evaluation due to	only 2 experts, < 7 participants		only 2 experts, < 7 participants	
Laboratory code	value	Z-score	value	Z-score
QR/112	0.557	X	0.510	X
QR/113	0.579	X	0.948	X
QR/118	NA	X	NA	X
QR/122	NA	X	NA	X
QR/123	NA	X	NA	X
QR/124	NA	X	NA	X
QR/125	0.807	X	1.400	X
QR/126	NA (sum of 3- and 9-FLUO)	X	NA (sum of 3- and 9-FLUO)	X
QR/127	NA	X	NA	X
QR/129	NA	X	NA	X
QR/130	NA	X	NA	X
QR/132	NA	X	NA	X
QR/140	NA	X	NA	X
QR/141	NA	X	NA	X
QR/142	NA	X	NA	X
QR/143	NA	X	NA	X
QR/201	NA	X	NA	X
QR/202	NA	X	NA	X
QR/206	NA	X	NA	X
QR/207	NA	X	NA	X
QR/210	NA	X	NA	X
QR/211	NA	X	NA	X
QR/217	NA	X	NA	X
QR/218	NA	X	NA	X
QR/219	NA	X	NA	X
QR/220	0.910	X	1.473	X
QR/221	NA	X	NA	X

NA = not analysed; X = no Z-score available

Appendix 6. Assigned values and participant's performance for PAH met. in urine (continued)

HBM4EU 02/2018	1-PHEN			
PAH met. (urine)	**1-PHEN$_{low}$**		**1-PHEN$_{high}$**	
assigned value from 3 experts	**0.476 ng/ml**		**0.788 ng/ml**	
expert standard deviation	**0.093 ng/ml**		**0.154 ng/ml**	
uncertainty of assigned value (u)	**11%**		**11%**	
relative target standard deviation (σ_T)	**25%**		**25%**	
0.7 * σ_T	**17.5%**		**17.5%**	
study RSD$_R$	**23%**		**23%**	
Laboratory code	**value**	**Z-score**	**value**	**Z-score**
QR/112	0.490	0.114	0.850	0.316
QR/113	0.199	-2.332	0.581	-1.050
QR/118	NA	X	NA	X
QR/122	0.522	0.382	1.150	1.838
QR/123	NA	X	NA	X
QR/124	NA	X	NA	X
QR/125	0.497	0.170	0.823	0.181
QR/126	0.419	-0.479	0.784	-0.019
QR/127	0.307	-1.423	0.513	-1.397
QR/129	0.436	-0.340	0.742	-0.234
QR/130	NA	X	NA	X
QR/132	NA (sum of 1- and 9-Phen)	X	NA (sum of 1- and 9-Phen)	X
QR/140	NA	X	NA	X
QR/141	0.592	0.970	1.046	1.309
QR/142	NA	X	NA	X
QR/143	NA	X	NA	X
QR/201	NA	X	NA	X
QR/202	NA	X	NA	X
QR/206	NA	X	NA	X
QR/207	NA	X	NA	X
QR/210	0.428	-0.404	0.648	-0.710
QR/211	NA	X	NA	X
QR/217	NA	X	NA	X
QR/218	NA	X	NA	X
QR/219	0.782	2.563	1.082	1.494
QR/220	0.430	-0.390	0.680	-0.547
QR/221	NA (sum of 1- and 9-Phen)	X	NA (sum of 1- and 9-Phen)	X

NA = not analysed; X = no Z-score available

Appendix 6. Assigned values and participant's performance for PAH met. in urine (continued)

HBM4EU 02/2018	2-PHEN			
PAH met. (urine)	2-PHEN$_{low}$		2-PHEN$_{high}$	
consensus value ICI (low)/assigned value from 3 experts (high)	0.308 ng/ml		0.732 ng/ml	
robust/expert standard deviation	0.134 ng/ml		0.129 ng/ml	
uncertainty (u)	0.048 ng/ml		10%	
relative target standard deviation (σ_T)	25		25%	
0.7 * σ_T	17.5 %		17.5%	
study RSD$_R$	43%		39%	
Laboratory code	value	Z-score	value	Z-score
QR/112	NA (sum of 2- and 3- Phen)	X	NA (sum of 2- and 3- Phen)	X
QR/113	0.133	-1.93	0.226	-2.765
QR/118	NA	X	NA	X
QR/122	0.337	0.32	0.584	-0.811
QR/123	NA	X	NA	X
QR/124	NA	X	NA	X
QR/125	0.297	-0.12	0.487	-1.342
QR/126	0.426	1.30	0.738	0.031
QR/127	0.166	-1.56	0.463	-1.471
QR/129	NA (sum of 2- and 3- Phen)	X	NA (sum of 2- and 3- Phen)	X
QR/130	NA	X	NA	X
QR/132	0.420	1.23	0.667	-0.359
QR/140	NA	X	NA	X
QR/141	0.245	-0.69	0.403	-1.801
QR/142	NA	X	NA	X
QR/143	NA	X	NA	X
QR/201	NA	X	NA	X
QR/202	NA	X	NA	X
QR/206	NA	X	NA	X
QR/207	NA	X	NA	X
QR/210	0.206	-1.12	0.323	-2.234
QR/211	NA	X	NA	X
QR/217	NA	X	NA	X
QR/218	NA	X	NA	X
QR/219	ND	X	0.672	-0.330
QR/220	0.223	-0.93	0.360	-2.034
QR/221	0.331	0.25	0.484	-1.357

NA = not analysed; X = no Z-score available; ND = not detected and no proxy Z-score available

Appendix 6. Assigned values and participant's performance for PAH met. in urine (continued)

HBM4EU 02/2018	3-PHEN			
PAH met. (urine)	3-PHEN$_{low}$		3-PHEN$_{high}$	
assigned value from 4 experts	0.271 ng/ml		0.482 ng/ml	
expert standard deviation	0.092 ng/ml		0.158 ng/ml	
uncertainty of assigned value (u)	17%		16%	
relative target standard deviation (σ_T)	25%		25%	
0.7 * σ_T	17.5%		17.5%	
study RSD$_R$	37%		35%	
Laboratory code	value	Z-score	value	Z-score
QR/112	NA (sum of 2- and 3-Phen)	X	NA (sum of 2- and 3-Phen)	X
QR/113	0.190	-1.202	0.307	-1.459
QR/118	NA	X	NA	X
QR/122	0.399	1.881	0.635	1.264
QR/123	NA	X	NA	X
QR/124	NA	X	NA	X
QR/125	0.403	1.945	0.630	1.224
QR/126	0.298	0.392	0.496	0.110
QR/127	0.262	-0.138	0.418	-0.531
QR/129	NA (sum of 2- and 3-Phen)	X	NA (sum of 2- and 3-Phen)	X
QR/130	NA	X	NA	X
QR/132	0.373	1.503	0.567	0.699
QR/140	NA	X	NA	X
QR/141	0.275	0.049	0.457	-0.214
QR/142	NA	X	NA	X
QR/143	NA	X	NA	X
QR/201	NA	X	NA	X
QR/202	NA	X	NA	X
QR/206	NA	X	NA	X
QR/207	NA	X	NA	X
QR/210	0.184	-1.283	0.296	-1.548
QR/211	NA	X	NA	X
QR/217	NA	X	NA	X
QR/218	NA	X	NA	X
QR/219	0.713	6.514	0.922	3.648
QR/220	0.310	0.569	0.493	0.091
QR/221	0.473	2.977	0.742	2.155

NA = not analysed; X = no Z-score available

Appendix 6. Assigned values and participant's performance for PAH met. in urine (continued)

HBM4EU 02/2018	4-PHEN			
PAH met. (urine)	4-PHEN$_{low}$		4-PHEN$_{high}$	
consensus value ICI	0.129 ng/ml		0.246 ng/ml	
robust standard deviation	0.027 ng/ml		0.052 ng/ml	
uncertainty (u)	0.009 ng/ml		0.018 ng/ml	
relative target standard deviation (σ_T)	25%		25%	
0.7 * σ_T	17.5%		17.5%	
study RSD$_R$	21%		21%	
Laboratory code	value	Z-score	value	Z-score
QR/112	0.113	-0.49	0.150	-1.57
QR/113	0.041	-2.74	0.075	-2.79
QR/118	NA	NA	NA	NA
QR/122	0.124	-0.17	0.253	0.11
QR/123	NA	NA	NA	NA
QR/124	NA	NA	NA	NA
QR/125	0.123	-0.18	0.240	-0.10
QR/126	0.130	0.02	0.280	0.55
QR/127	0.155	0.79	0.321	1.22
QR/129	0.141	0.37	0.248	0.02
QR/130	NA	NA	NA	NA
QR/132	0.107	-0.69	0.213	-0.54
QR/140	NA	NA	NA	NA
QR/141	0.102	-0.85	0.219	-0.45
QR/142	NA	NA	NA	NA
QR/143	NA	NA	NA	NA
QR/201	NA	NA	NA	NA
QR/202	NA	NA	NA	NA
QR/206	NA	NA	NA	NA
QR/207	NA	NA	NA	NA
QR/210	NA (sum of 4- and 9-phen)	NA (sum of 4- and 9-	NA (sum of 4- and 9-phen)	NA (sum of 4- and
QR/211	NA	NA	NA	NA
QR/217	NA	NA	NA	NA
QR/218	NA	NA	NA	NA
QR/219	0.180	1.58	0.280	0.55
QR/220	0.120	-0.28	0.213	-0.54
QR/221	0.152	0.71	0.265	0.30

NA = not analysed; X = no Z-score available

Appendix 6. Assigned values and participant's performance for PAH met. in urine (continued)

Control material	9-PHEN (urine) HBM4EU 02/2018			
PAH met. (urine)	9-PHEN$_{low}$		9-PHEN$_{high}$	
assigned/consensus value	X		X	
no ICI/EQUAS evaluation due to	only 2 experts, no ICI evaluation appropriate		only 2 experts, no ICI evaluation appropriate	
Laboratory code	value	Z-score	value	Z-score
QR/112	NA	X	NA	X
QR/113	NA	X	NA	X
QR/118	NA	X	NA	X
QR/122	0.072	X	0.079	X
QR/123	NA	X	NA	X
QR/124	NA	X	NA	X
QR/125	0.083	X	0.075	X
QR/126	0.301	X	0.395	X
QR/127	0.561	X	0.730	X
QR/129	ND	X	ND	X
QR/130	1.150	X	1.810	X
QR/132	NA	X	NA	X
QR/140	NA	X	NA	X
QR/141	NA	X	NA	X
QR/142	NA	X	NA	X
QR/143	NA	X	NA	X
QR/201	NA	X	NA	X
QR/202	NA	X	NA	X
QR/206	NA	X	NA	X
QR/207	NA	X	NA	X
QR/210	NA (sum of 4- and 9-PHEN)	X	NA (sum of 4- and 9-PHEN)	X
QR/211	NA	X	NA	X
QR/217	NA	X	NA	X
QR/218	NA	X	NA	X
QR/219	0.119	X	0.073	X
QR/220	0.463	X	0.463	X
QR/221	NA (sum of 1- and 9-PHEN)	X	NA (sum of 1- and 9-PHEN)	X

NA = not analysed; X = no Z-score available; ND = not detected and no proxy Z-score available

Appendix 6. Assigned values and participant's performance for PAH met. in urine (continued)

Control material	1-PYR (urine) HBM4EU 02/2018			
PAH met. (urine)	1-PYR_{low}		1-PYR_{high}	
assigned value from 5(low)/ 6(high) experts	0.113 ng/ml		0.258 ng/ml	
expert standard deviation	0.040 ng/ml		0.097 ng/ml	
uncertainty of assigned value (u)	15.9%		16.9%	
relative target standard deviation (σ_T)	25%		25%	
0.7 * σ_T	17.5%		17.5%	
study RSD_R	30%		31%	
Laboratory code	value	Z-score	value	Z-score
QR/112	0.163	1.806	0.343	1.314
QR/113	0.069	-1.554	0.124	-2.086
QR/118	0.200	3.109	0.200	-0.904
QR/122	0.123	0.360	0.259	0.011
QR/123	0.167	1.925	0.277	0.282
QR/124	0.152	1.391	0.317	0.912
QR/125	0.157	1.569	0.310	0.798
QR/126	0.301	6.712	0.395	2.119
QR/127	0.134	0.763	0.290	0.494
QR/129	0.128	0.538	0.233	-0.399
QR/130	0.295	6.486	0.420	2.501
QR/132	0.153	1.451	0.287	0.437
QR/140	0.136	0.846	0.163	-1.482
QR/141	0.135	0.799	0.273	0.220
QR/142	NA	X	NA	X
QR/143	0.121	0.289	0.240	-0.280
QR/201	0.176	2.256	0.340	1.263
QR/202	0.265	5.420	0.452	3.001
QR/206	0.197	2.991	0.307	0.747
QR/207	0.113	0.029	0.196	-0.961
QR/210	0.122	0.337	0.306	0.741
QR/211	0.045	-2.389	0.089	-2.628
QR/217	0.127	0.514	0.268	0.148
QR/218	0.182	2.481	0.389	2.021
QR/219	0.339	8.039	0.662	6.241
QR/220	0.107	-0.208	0.217	-0.646
QR/221	0.204	3.252	0.424	2.563

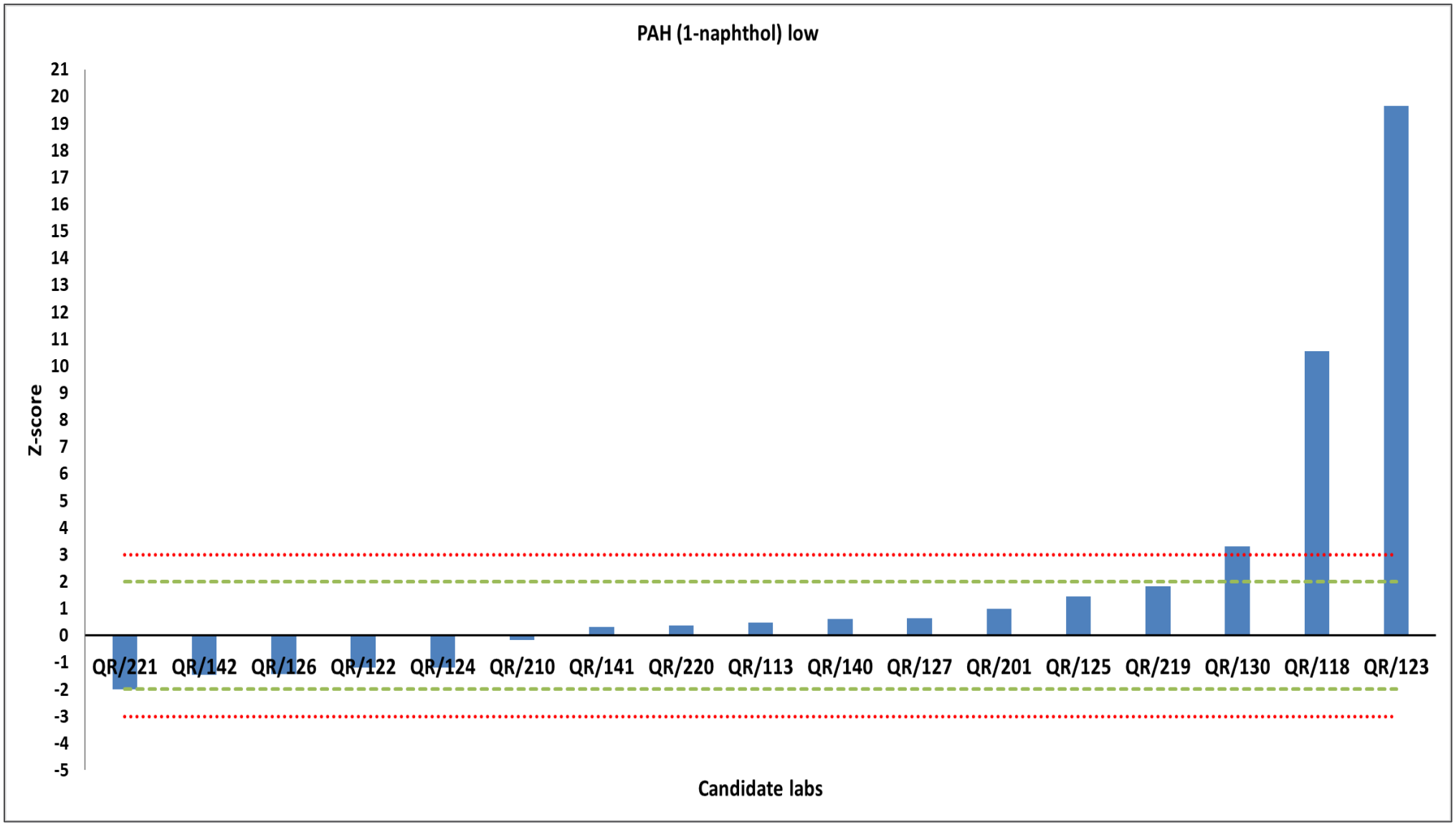
NA = not analysed; X = no Z-score available

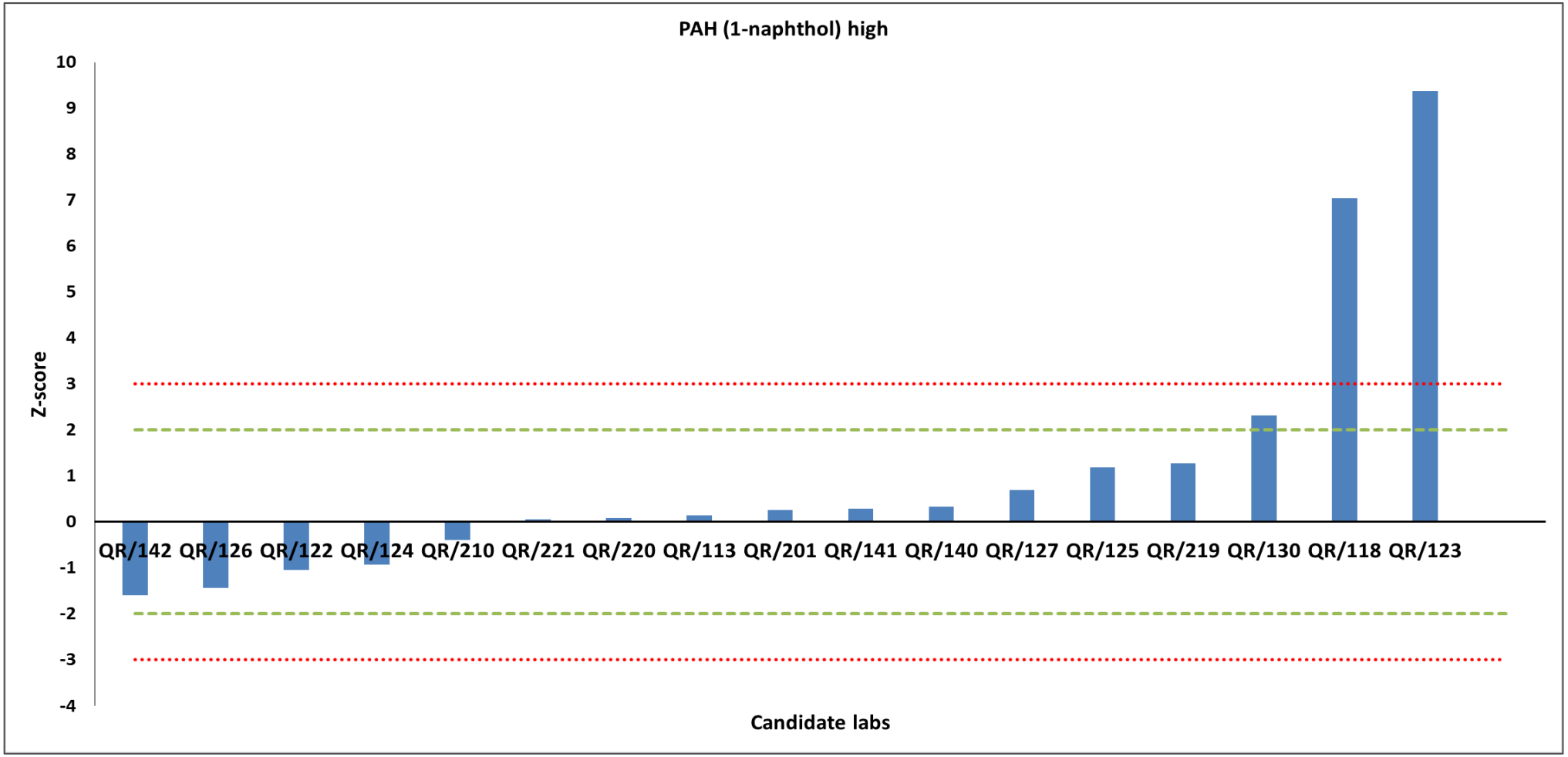
Appendix 6. Assigned values and participant's performance for PAH met. in urine (continued)

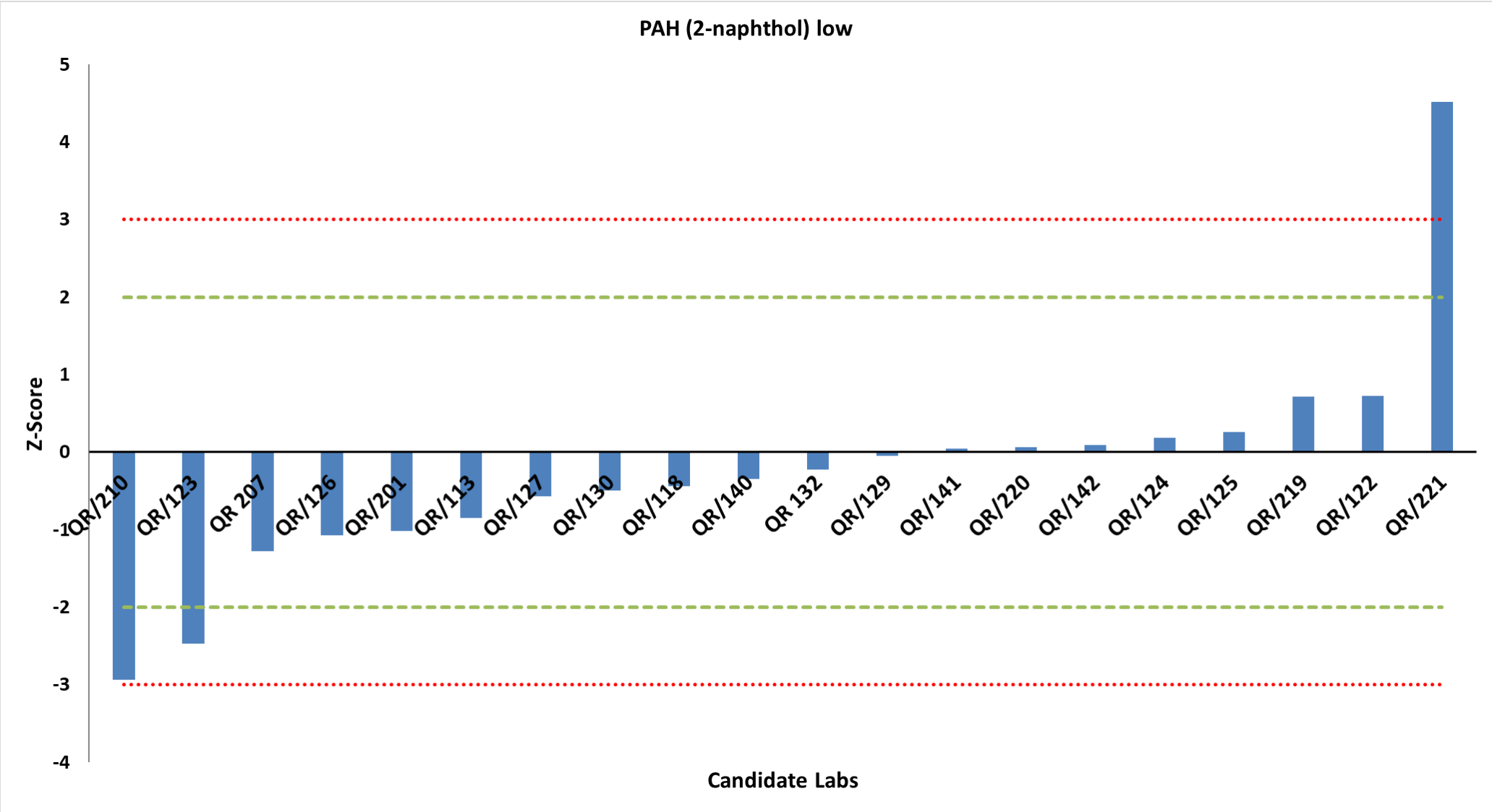
Control material	3-BaP (urine) HBM4EU 02/2018			
PAH met. (urine)	3-BaP$_{low}$		3-BaP$_{high}$	
assigned/consensus value	X		X	
no ICI/EQUAS evaluation due to	only 1 expert, < 7 participants		only 1 expert, < 7 participants	
Laboratory code	value	Z-score	value	Z-score
QR/112	NA	X	NA	X
QR/113	NA	X	NA	X
QR/118	NA	X	NA	X
QR/122	0.001971	X	0.003623	X
QR/123	NA	X	NA	X
QR/124	NA	X	NA	X
QR/125	NA	X	NA	X
QR/126	ND	X	ND	X
QR/127	ND	X	ND	X
QR/129	ND	X	ND	X
QR/130	NA	X	NA	X
QR/132	NA	X	NA	X
QR/140	NA	X	NA	X
QR/141	NA	X	NA	X
QR/142	NA	X	NA	X
QR/143	NA	X	NA	X
QR/201	0.000620	X	0.001323	X
QR/202	NA	X	NA	X
QR/206	NA	X	NA	X
QR/207	NA	X	NA	X
QR/210	NA	X	NA	X
QR/211	NA	X	NA	X
QR/217	NA	X	NA	X
QR/218	NA	X	NA	X
QR/219	ND	X	ND	X
QR/220	0.001167	X	0.002067	X
QR/221	ND	X	ND	X

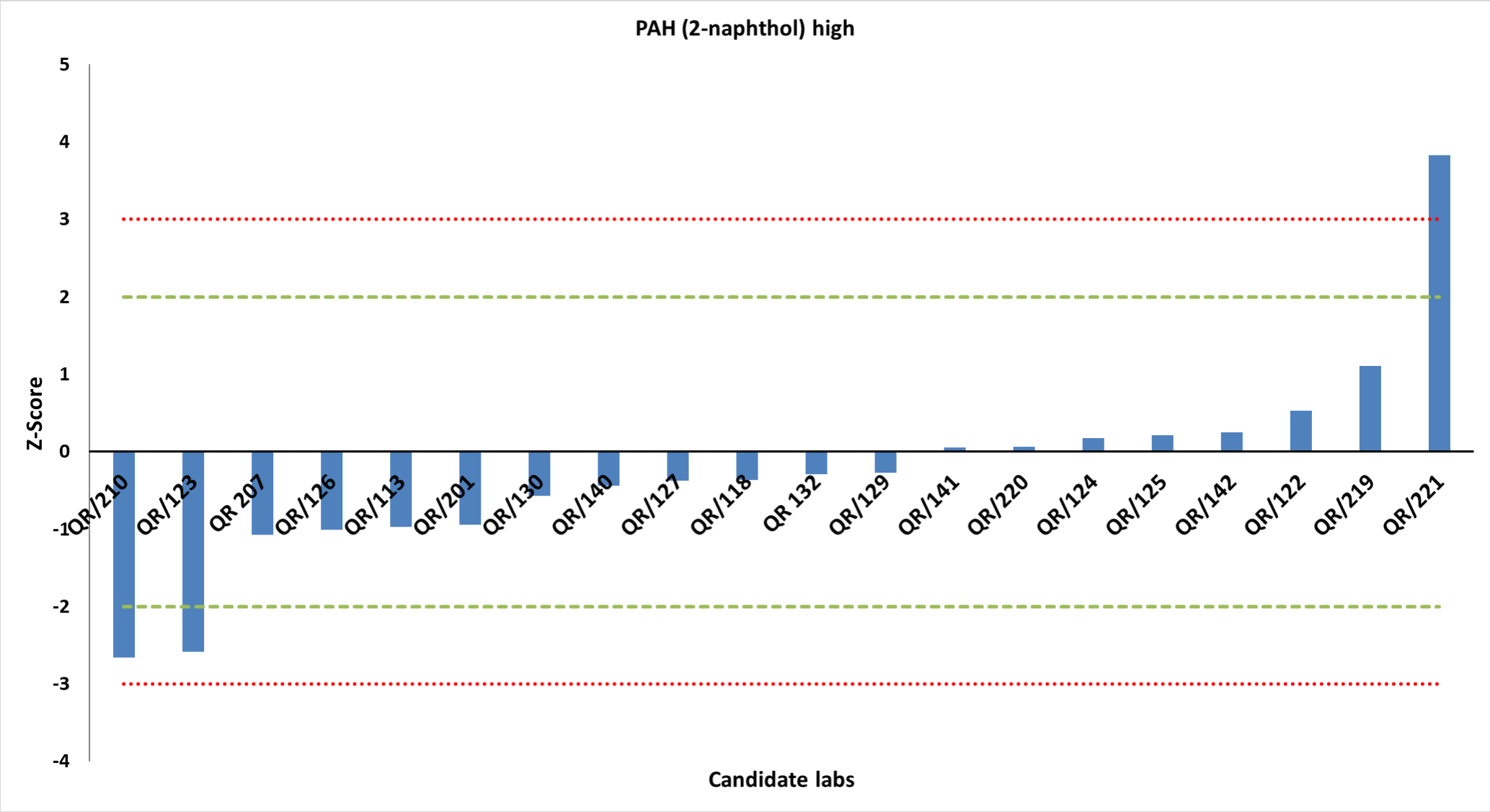
NA = not analysed; X = no Z-score available; ND = not detected and no proxy Z-score available

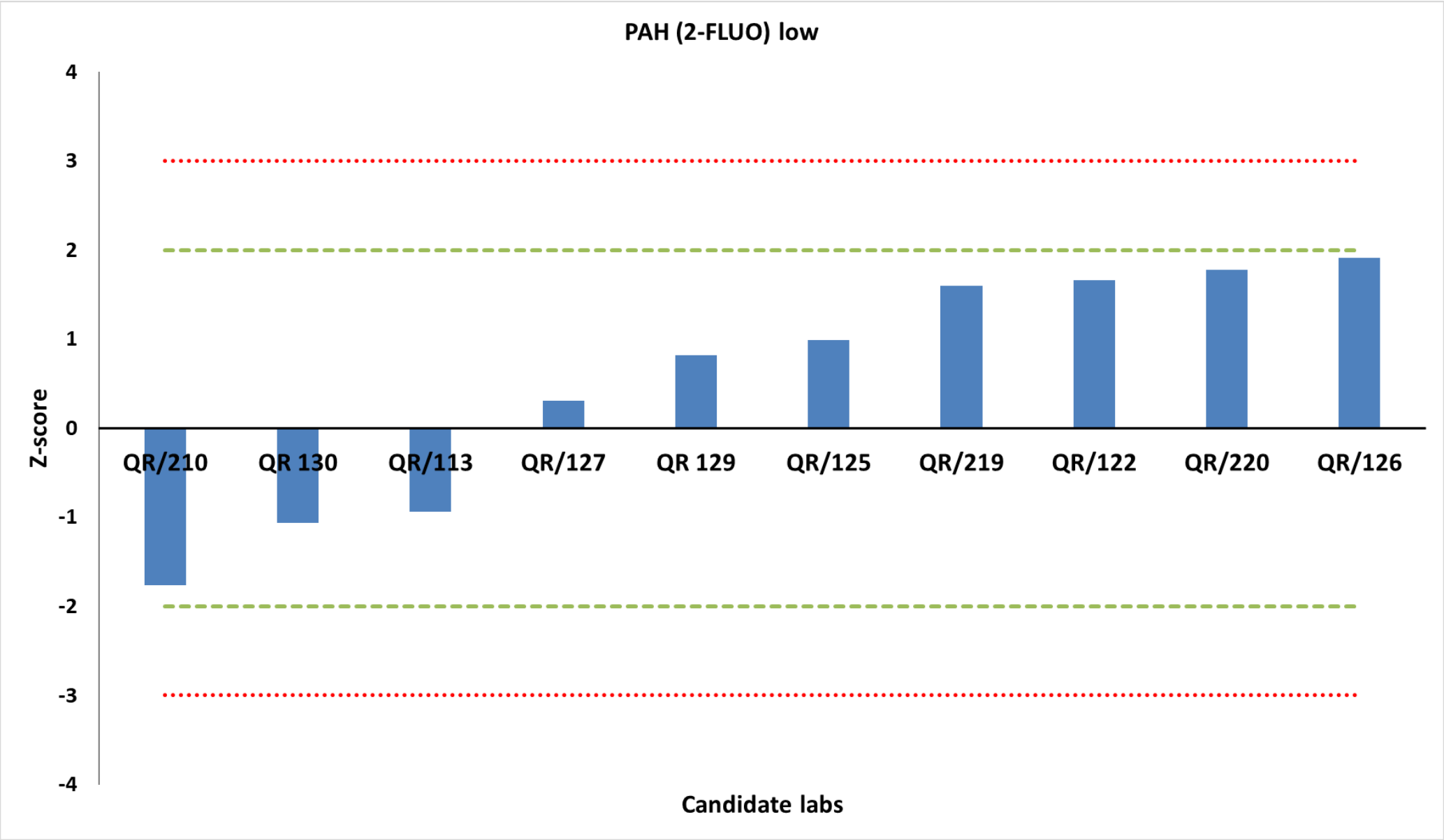
Appendix 7. Graphical representation of the Z-scores

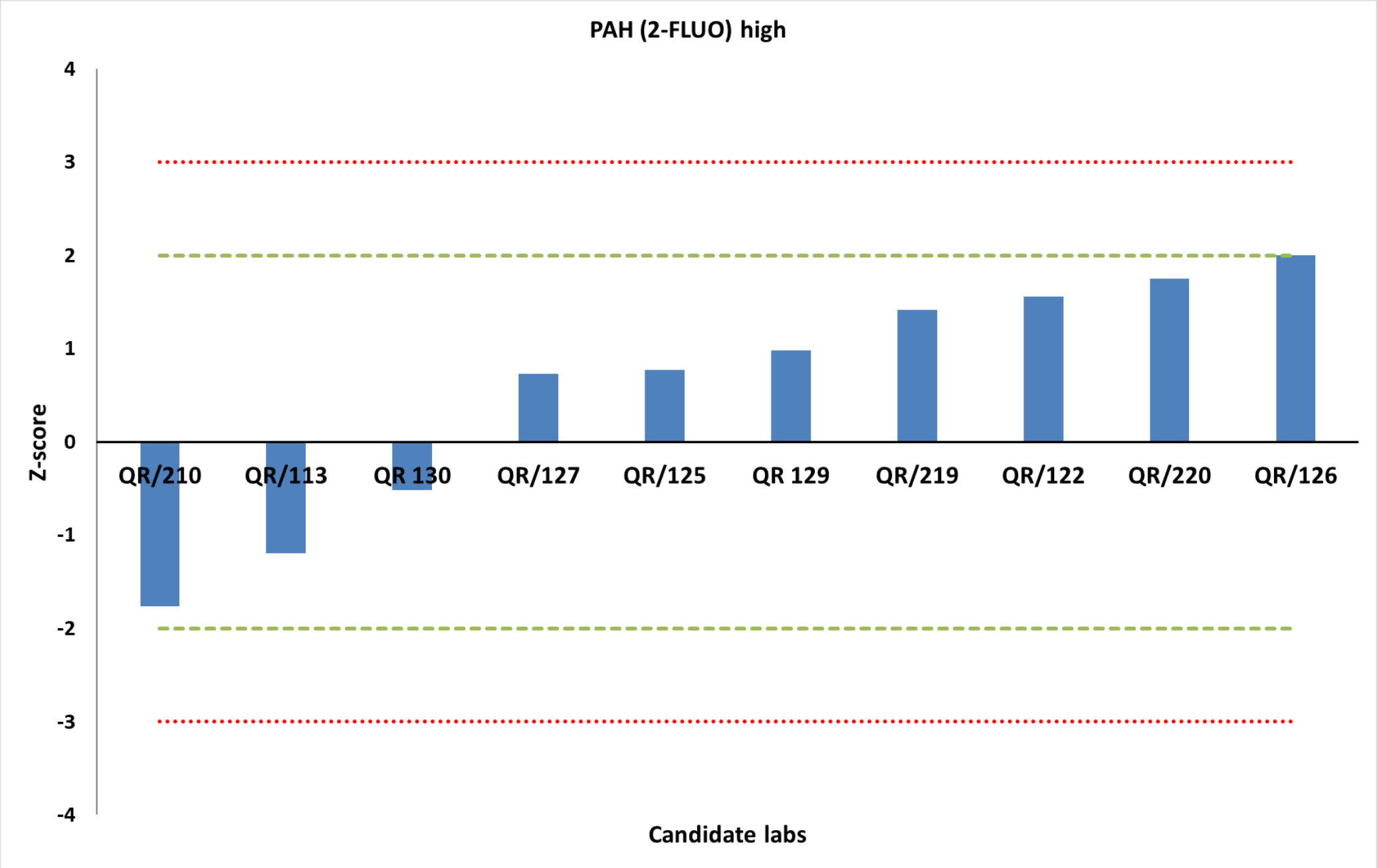


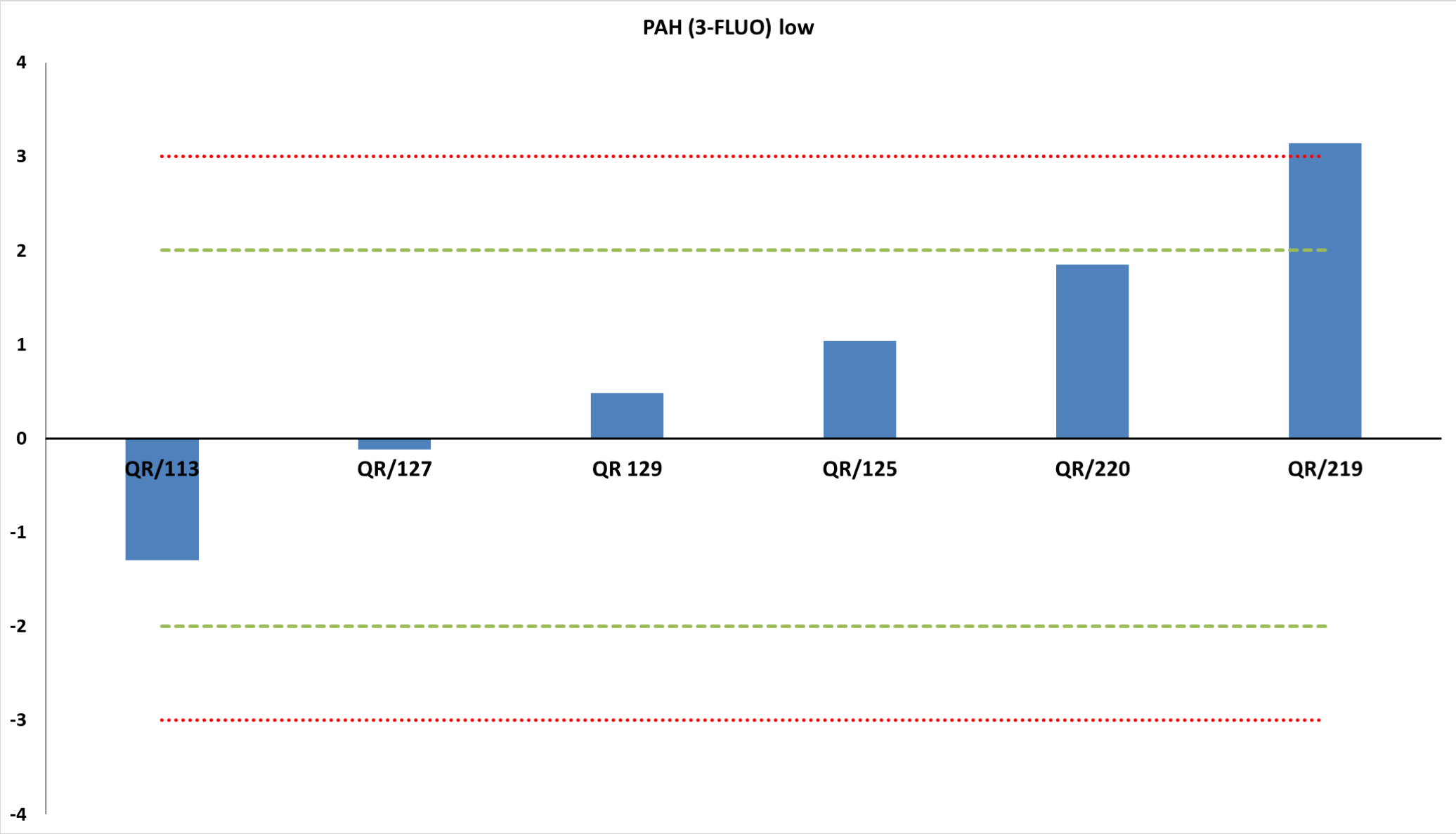

PAH (1-naphthol) high
Z-score
10
9
8
7
6
5
4
3
2
1
0
-1
-2
-3
-4
QR/142
QR/126
QR/122
QR/124
QR/210
QR/221
QR/220
QR/113
QR/201
QR/141
QR/140
QR/127
QR/125
QR/219
QR/130
QR/118
QR/123
Candidate labs

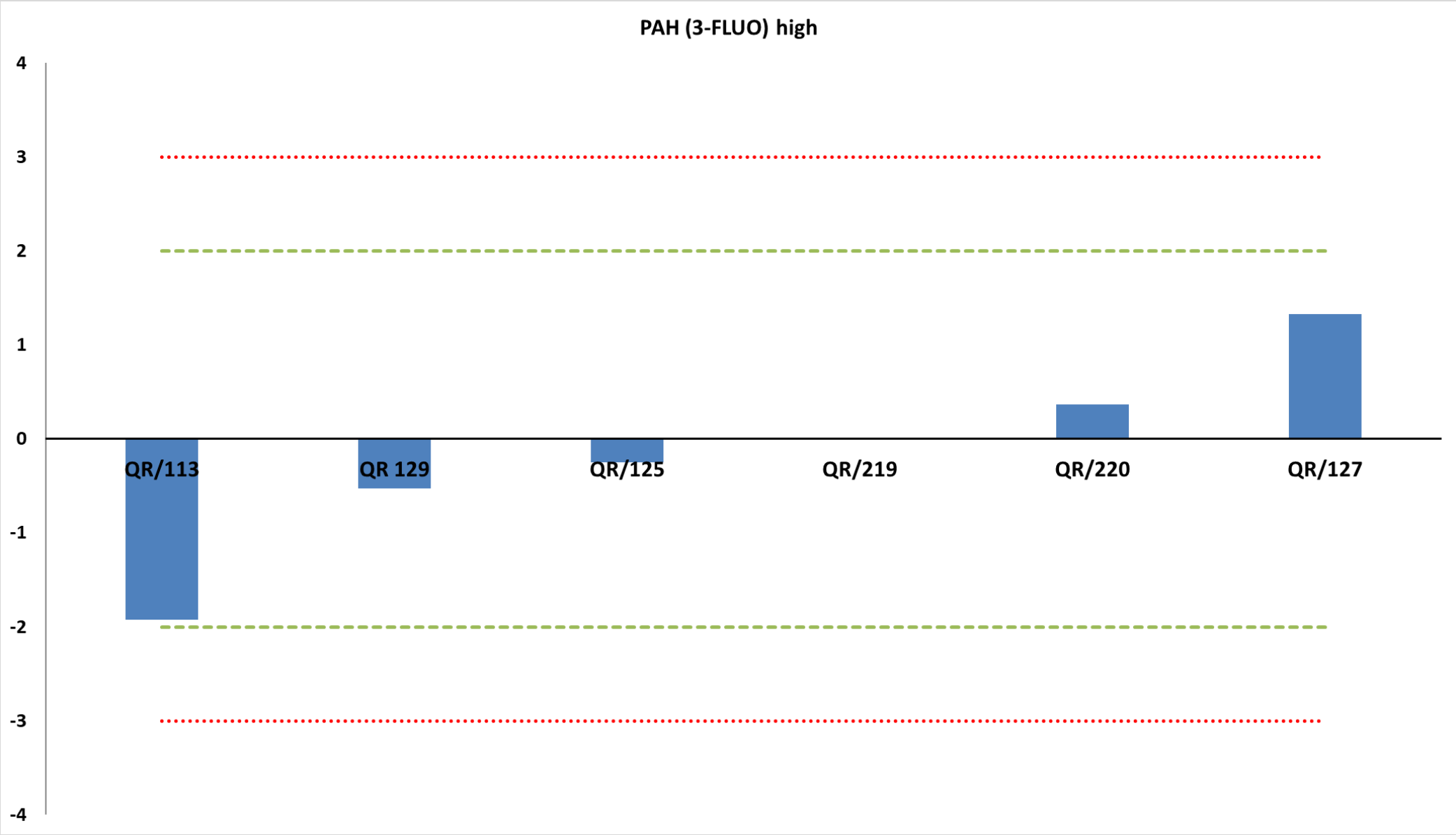

PAH (2-naphthol) low
Z-Score
5
4
3
2
1
0
-1
-2
-3
-4
QR/210
QR/123
QR 207
QR/126
QR/201
QR/113
QR/127
QR/130
QR/118
QR/140
QR 132
QR/129
QR/141
QR/220
QR/142
QR/124
QR/125
QR/219
QR/122
QR/221
Candidate Labs

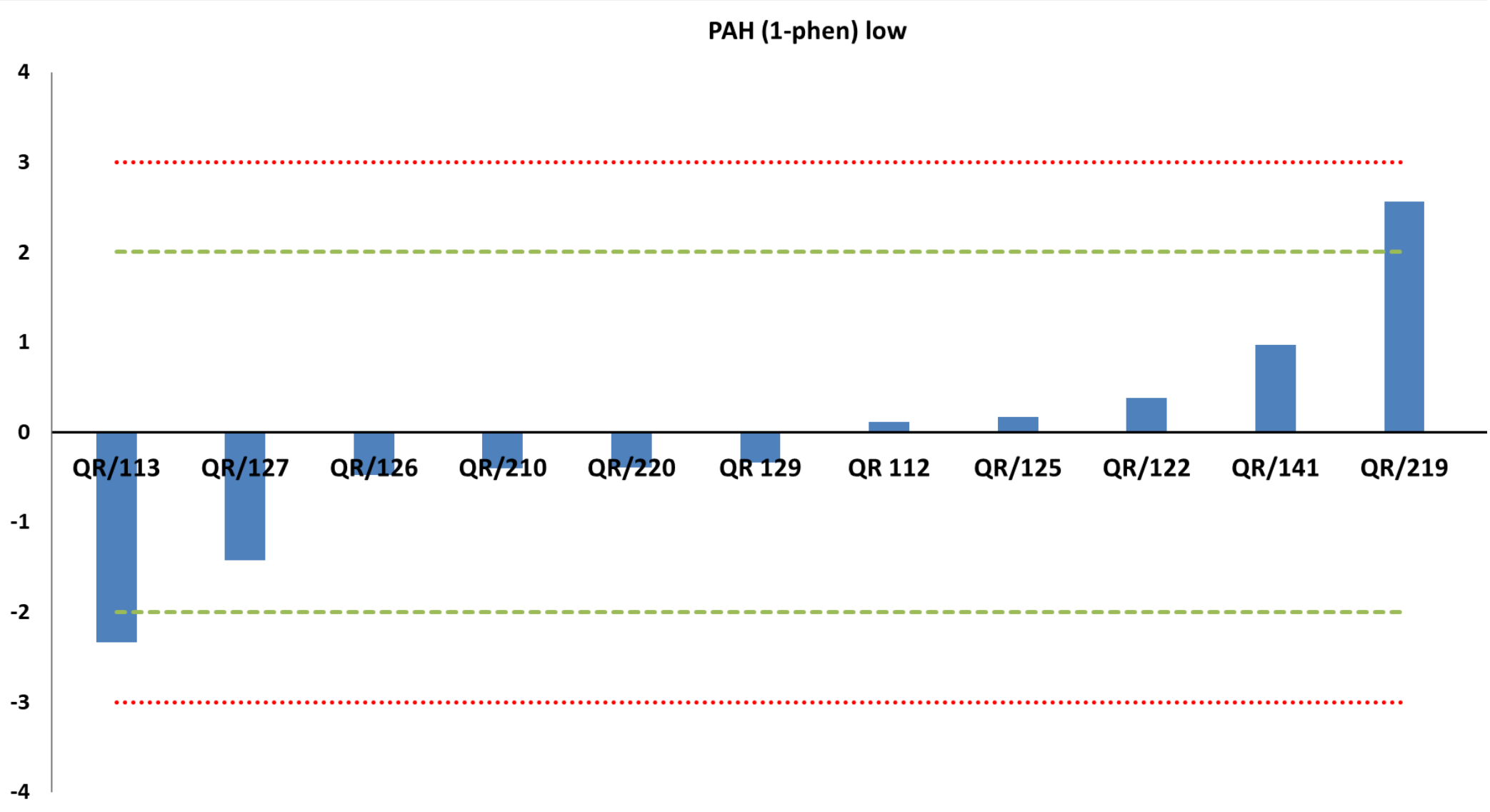

PAH (2-naphthol) high
Z-Score
5
4
3
2
1
0
-1
-2
-3
-4
QR/210
QR/123
QR 207
QR/126
QR/113
QR/201
QR/130
QR/140
QR/127
QR/118
QR 132
QR/129
QR/141
QR/220
QR/124
QR/125
QR/142
QR/122
QR/219
QR/221
Candidate labs

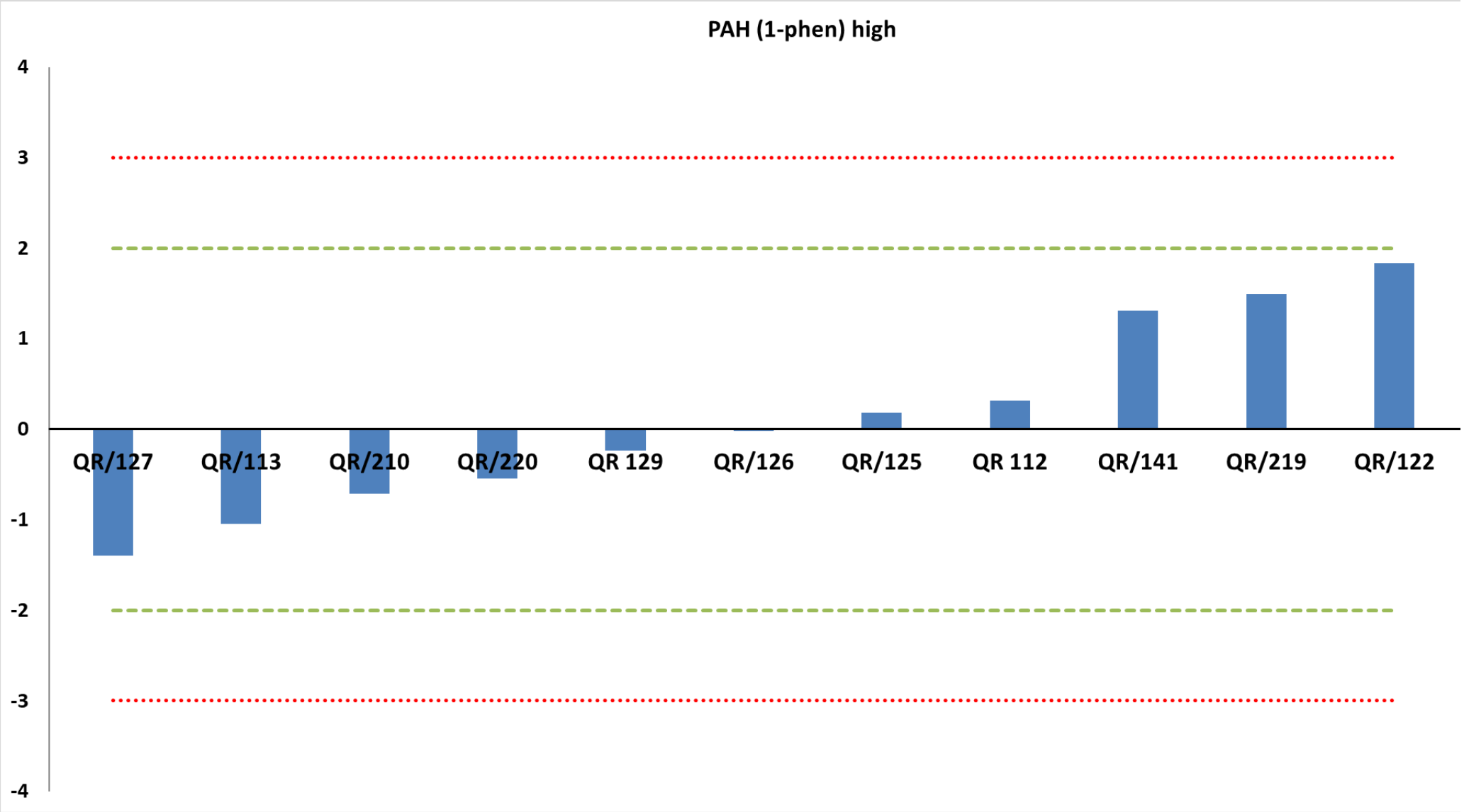

PAH (2-FLUO) low
4
3
2
1
0
-1
-2
-3
-4
Z-score
QR/210
QR 130
QR/113
QR/127
QR 129
QR/125
QR/219
QR/122
QR/220
QR/126
Candidate labs

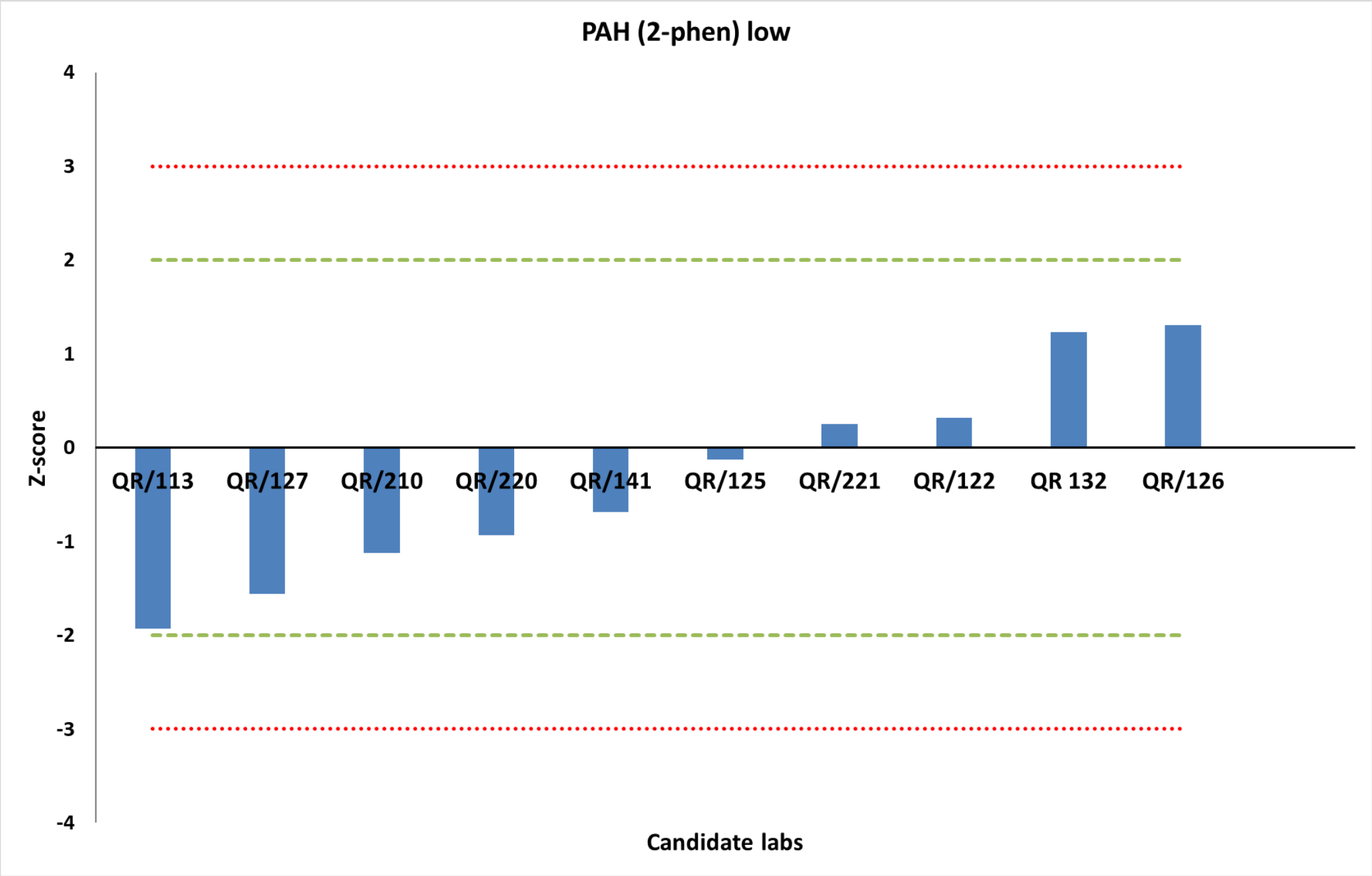

PAH (2-FLUO) high
4
3
2
1
0
-1
-2
-3
-4
Z-score
QR/210
QR/113
QR 130
QR/127
QR/125
QR 129
QR/219
QR/122
QR/220
QR/126
Candidate labs

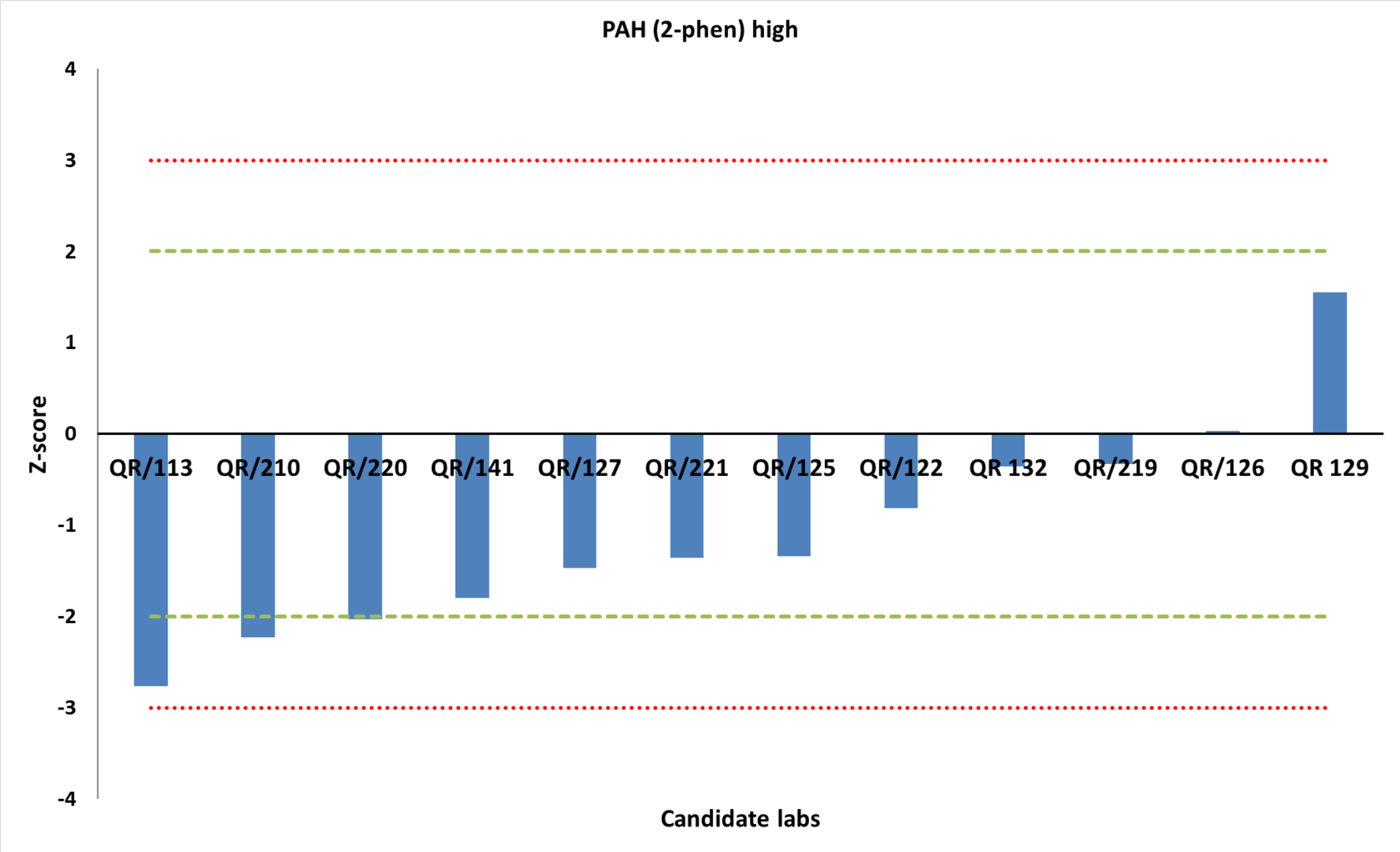

PAH (3-FLUO) low
4
3
2
1
0
-1
-2
-3
-4
QR/113
QR/127
QR 129
QR/125
QR/220
QR/219

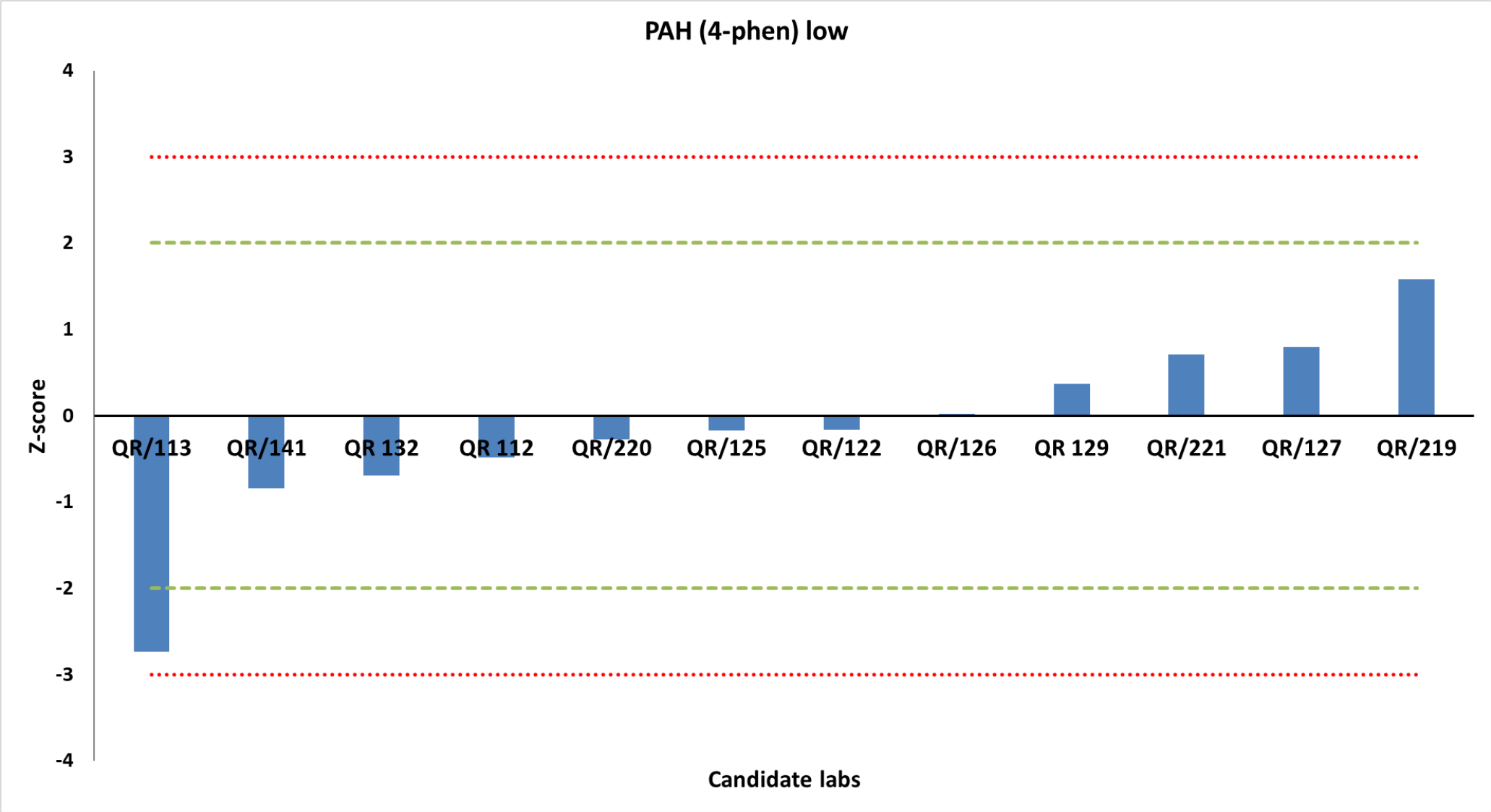

PAH (3-FLUO) high
4
3
2
1
0
-1
-2
-3
-4
QR/113
QR 129
QR/125
QR/219
QR/220
QR/127

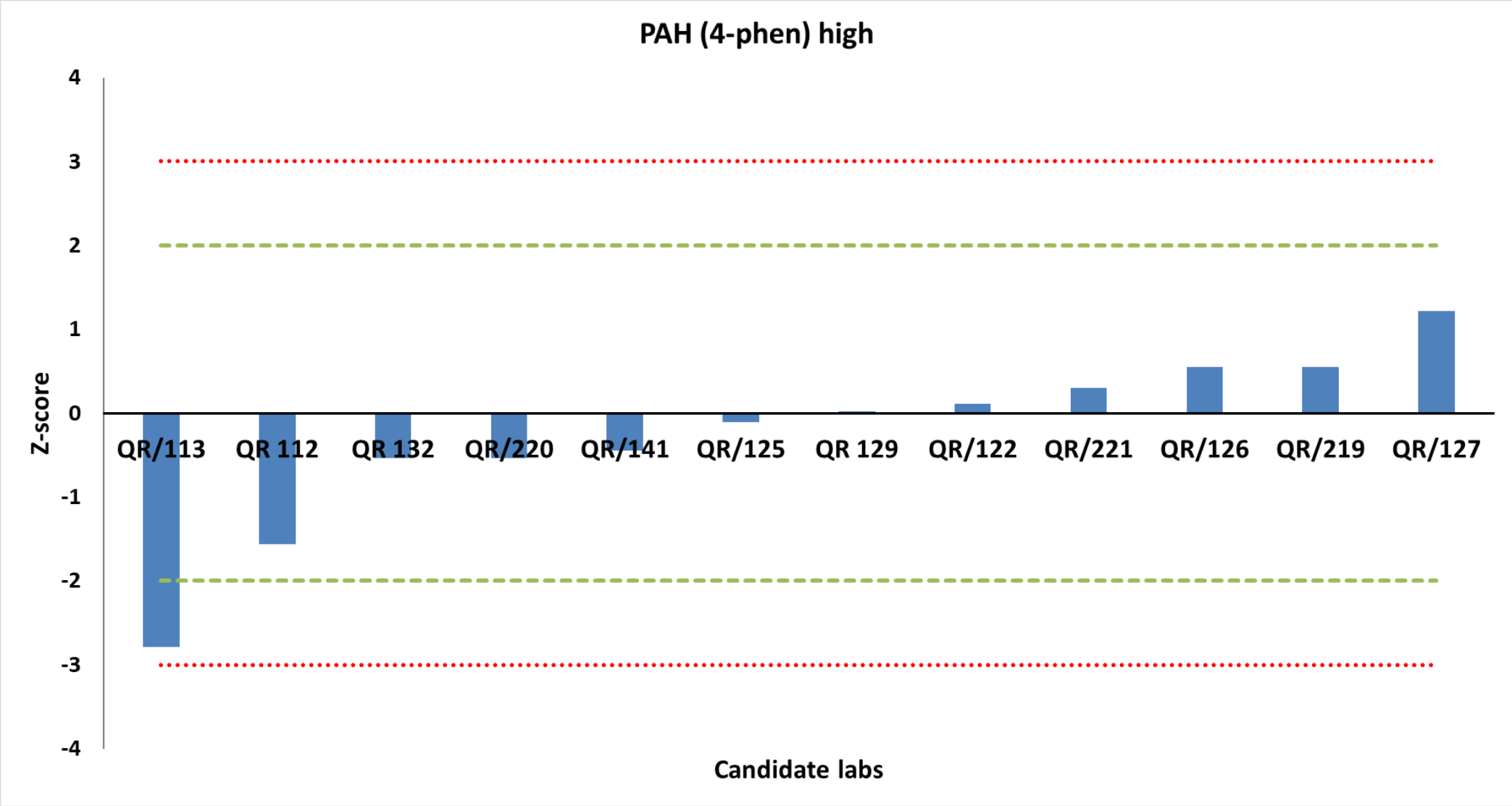

PAH (1-phen) low
4
3
2
1
0
-1
-2
-3
-4
QR/113
QR/127
QR/126
QR/210
QR/220
QR 129
QR 112
QR/125
QR/122
QR/141
QR/219

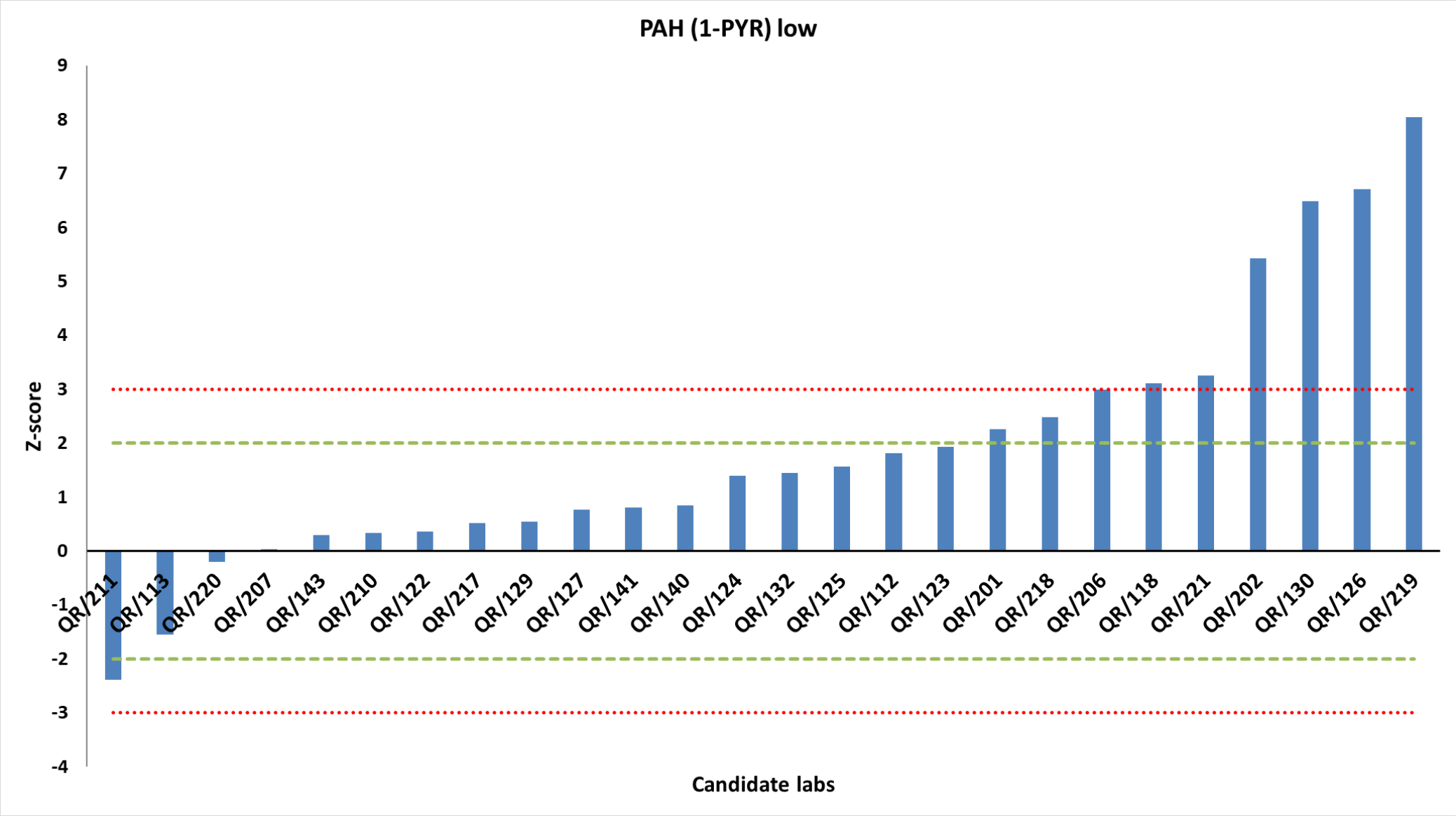

PAH (1-phen) high
4
3
2
1
0
-1
-2
-3
-4
QR/127
QR/113
QR/210
QR/220
QR 129
QR/126
QR/125
QR 112
QR/141
QR/219
QR/122

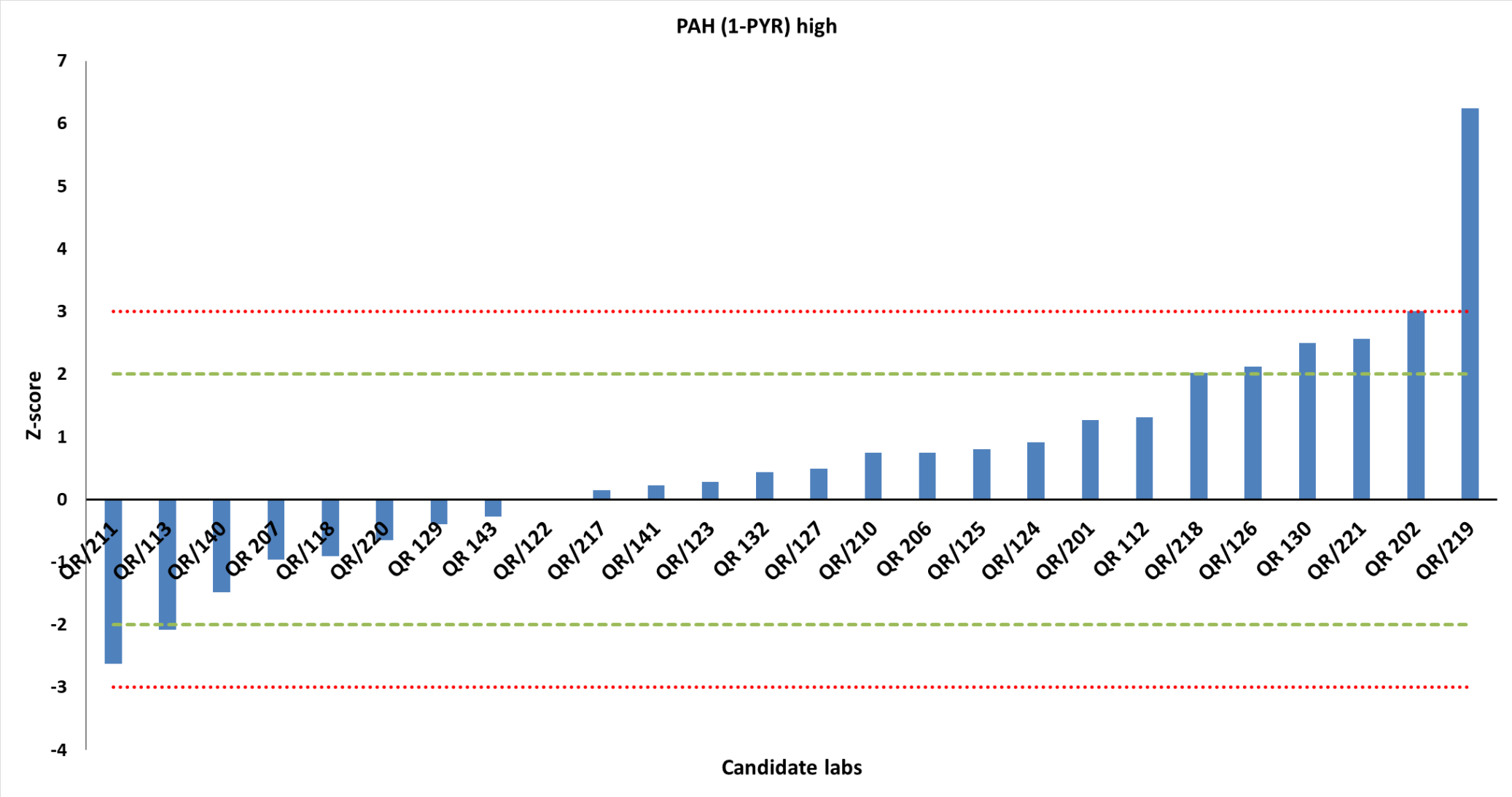

PAH (2-phen) low
4
3
2
1
0
-1
-2
-3
-4
Z-score
QR/113
QR/127
QR/210
QR/220
QR/141
QR/125
QR/221
QR/122
QR 132
QR/126
Candidate labs


PAH (2-phen) high
4
3
2
1
0
-1
-2
-3
-4
Z-score
QR/113
QR/210
QR/220
QR/141
QR/127
QR/221
QR/125
QR/122
QR 132
QR/219
QR/126
QR 129
Candidate labs


PAH (4-phen) low
4
3
2
1
0
-1
-2
-3
-4
Z-score
QR/113
QR/141
QR 132
QR 112
QR/220
QR/125
QR/122
QR/126
QR 129
QR/221
QR/127
QR/219
Candidate labs


PAH (4-phen) high
4
3
2
1
0
-1
-2
-3
-4
Z-score
QR/113
QR 112
QR 132
QR/220
QR/141
QR/125
QR 129
QR/122
QR/221
QR/126
QR/219
QR/127
Candidate labs


PAH (1-PYR) low
Z-score
9
8
7
6
5
4
3
2
1
0
-1
-2
-3
-4
QR/211
QR/113
QR/220
QR/207
QR/143
QR/210
QR/122
QR/217
QR/129
QR/127
QR/141
QR/140
QR/124
QR/132
QR/125
QR/112
QR/123
QR/201
QR/218
QR/206
QR/118
QR/221
QR/202
QR/130
QR/126
QR/219
Candidate labs


PAH (1-PYR) high
Z-score
7
6
5
4
3
2
1
0
-1
-2
-3
-4
QR/211
QR/113
QR/140
QR 207
QR/118
QR/220
QR 129
QR 143
QR/122
QR/217
QR/141
QR/123
QR 132
QR/127
QR/210
QR 206
QR/125
QR/124
QR/201
QR 112
QR/218
QR/126
QR 130
QR/221
QR 202
QR/219
Candidate labs

Appendix 8. Results of the shipped test samples analysed by the participants

Lab.code	1-naphthol in urine [ng/ml]							
	low-1	low-2	low-3	high-1	high-2	high-3	LOQ	delayed reporting
EX/101	1.4	1.5	1.6	2.5	2.4	2.4	0.160	
	1.3	1.8	1.3	2.7	2.6	2.5		
EX/102	1.99	1.93	1.93	3.38	3.40	3.03	0.340	
	1.90	1.96	1.81	3.46	3.33	3.05		
EX/105	3.00	3.00	2.94	4.17	4.19	4.17	0.200	
QR/112	NA	NA	NA	NA	NA	NA	-	
QR/113	1.99	2.26	2.46	2.29	3.59	3.62	0.053	
	1.98	2.40	2.36	2.32	3.64	3.73		
QR/118	7.5	7.2	7.2	8.6	8.6	8.4	1.400	
QR/122	1.674	1.682	1.091	2.772	2.009	1.814	0.100	
	1.627	1.168	1.238	2.664	2.551	1.900		
QR/123	12.10	10.20	13.30	10.60	11.50	8.90	0.600	
QR/124	1.43	1.46	1.35	2.48	2.24	2.38	1.000	
QR/125	2.79	2.70	2.69	3.94	4.03	4.04	0.050	
QR/126	1.228	1.314	1.301	2.095	1.967	1.870	0.025	
QR/127	2.228	2.325	2.434	3.779	3.843	3.245	0.010	technical reasons
QR/129	NA	NA	NA	NA	NA	NA	-	
QR/130	3.73	3.61	NA	4.88	NA	NA	0.200	no reason given
QR/132	NA	NA	NA	NA	NA	NA	-	
QR/140	2.184	2.392	2.371	3.275	3.221	3.526	2.000	
QR/141	2.139	2.195	2.159	3.271	3.374	3.307	0.500	
QR/142	1.241	1.223	1.349	1.852	1.809	1.901	0.100	
QR/143	NA	NA	NA	NA	NA	NA	-	
QR/201	2.4	2.41	2.72	3.43	2.71	3.74	6.000	
QR/202	NA	NA	NA	NA	NA	NA	-	
QR/206	NA	NA	NA	NA	NA	NA	-	
QR/207	NA	NA	NA	NA	NA	NA	-	
QR/210	1.895	1.894	1.990	2.934	2.618	2.791	0.400	
QR/211	NA	NA	NA	NA	NA	NA	-	
QR/217	NA	NA	NA	NA	NA	NA	-	
QR/218	NA	NA	NA	NA	NA	NA	-	
QR/219	2.95	2.91	2.91	4.02	4.08	4.12	0.013	
QR/220	2.17	2.17	2.23	3.25	3.08	3.13	0.250	misunderstanding reg. deadline
QR/221	0.892	1.073	1.029	5.493	2.124	1.797	0.806	

Lab.code	2-naphthol in urine [ng/ml]							
	low-1	low-2	low-3	high-1	high-2	high-3	LOQ	delayed reporting
EX/101	8.4	8.9	9.0	14	14	13	0.110	
	7.1	8.9	7.3	14	14	13		
EX/102	10.83	10.59	10.66	16.05	16.32	16.09	0.290	
	10.81	10.63	10.85	16.35	16.25	16.23		
EX/105	9.37	9.35	9.37	14.4	14.7	14.4	0.300	
QR/112	NA	NA	NA	NA	NA	NA	-	
QR/113	7.68	7.93	6.13	7.49	12.3	12.9	0.074	
	7.88	8.22	6.32	7.79	12.2	12.2		
QR/118	8.6	7.8	8.6	13	12.7	13.2	1.400	
QR/122	10.809	11.018	10.949	16.214	15.902	16.180	0.050	
	10.613	12.298	10.658	15.056	16.225	17.395		
QR/123	0.8	4.90	5.00	4.00	4.90	6.20	0.700	
QR/124	9.55	9.78	10.0	15.2	14.6	14.9	1.000	
QR/125	9.75	9.94	10.22	14.75	15.18	15.13	0.050	
QR/126	6.596	7.186	6.722	10.447	10.910	10.668	0.010	
QR/127	7.590	8.038	8.459	13.235	12.603	12.937	0.010	technical reasons
QR/129	9.27	9.58	8.86	13.1	13.2	13.6	0.001	
QR/130	8.32	8.09	NA	12.22	NA	NA	0.200	no reason given
QR/132	9.12	8.68	8.66	13.5	12.8	13.4	0.007	
QR/140	8.532	8.54	8.563	12.996	12.475	12.651	0.500	
QR/141	9.805	9.341	9.203	14.051	15.143	14.216	0.500	
QR/142	9.614	9.480	9.629	14.872	15.283	15.316	0.400	
QR/143	NA	NA	NA	NA	NA	NA	-	
QR/201	7.17	7.2	6.56	10.2	11.1	11.4	6.000	
QR/202	NA	NA	NA	NA	NA	NA	-	
QR/206	NA	NA	NA	NA	NA	NA	-	
QR/207	6.492	6.394	6.190	10.493	10.243	10.549	1.368	
QR/210	2.819	2.316	2.300	4.816	4.795	4.755	0.400	
QR/211	NA	NA	NA	NA	NA	NA	-	
QR/217	NA	NA	NA	NA	NA	NA	-	
QR/218	NA	NA	NA	NA	NA	NA	-	
QR/219	11.1	10.9	11.1	18.0	17.6	19.0	0.013	
QR/220	9.55	9.37	9.60	14.47	14.55	14.45	0.250	misunderstanding reg. deadline
QR/221	17.895	17.550	24.316	21.229	33.159	29.410	0.511	

Lab.code	1.2-dihydroxynaphthalene in urine [ng/ml]							
	low-1	low-2	low-3	high-1	high-2	high-3	LOQ	delayed reporting
EX/101	NA	NA	NA	NA	NA	NA	-	
	NA	NA	NA	NA	NA	NA		
EX/102	3.64	3.72	3.41	6.30	6.35	6.11	0.780	
	3.65	3.58	3.55	6.52	6.48	6.30		
EX/105	NA	NA	NA	NA	NA	NA	-	
QR/112	NA	NA	NA	NA	NA	NA	-	
QR/113	NA	NA	NA	NA	NA	NA	-	
	NA	NA	NA	NA	NA	NA		
QR/118	NA	NA	NA	NA	NA	NA	-	
QR/122	NA	NA	NA	NA	NA	NA	-	
	NA	NA	NA	NA	NA	NA		
QR/123	NA	NA	NA	NA	NA	NA	-	
QR/124	NA	NA	NA	NA	NA	NA	-	
QR/125	NA	NA	NA	NA	NA	NA	-	
QR/126	ND	ND	ND	ND	ND	ND	0.500	
QR/127	NA	NA	NA	NA	NA	NA	-	
QR/129	NA	NA	NA	NA	NA	NA	-	
QR/130	NA	NA	NA	NA	NA	NA	-	
QR/132	NA	NA	NA	NA	NA	NA	-	
QR/140	NA	NA	NA	NA	NA	NA	-	
QR/141	NA	NA	NA	NA	NA	NA	-	
QR/142	NA	NA	NA	NA	NA	NA	-	
QR/143	NA	NA	NA	NA	NA	NA	-	
QR/201	NA	NA	NA	NA	NA	NA	-	
QR/202	NA	NA	NA	NA	NA	NA	-	
QR/206	NA	NA	NA	NA	NA	NA	-	
QR/207	NA	NA	NA	NA	NA	NA	-	
QR/210	NA	NA	NA	NA	NA	NA	-	
QR/211	NA	NA	NA	NA	NA	NA	-	
QR/217	NA	NA	NA	NA	NA	NA	-	
QR/218	NA	NA	NA	NA	NA	NA	-	
QR/219	NA	NA	NA	NA	NA	NA	-	
QR/220	NA	NA	NA	NA	NA	NA	-	
QR/221	NA	NA	NA	NA	NA	NA	-	

Lab.code	2-hydroxyfluorene in urine [ng/ml]							
	low-1	low-2	low-3	high-1	high-2	high-3	LOQ	delayed reporting
EX/101	NA	NA	NA	NA	NA	NA	-	
	NA	NA	NA	NA	NA	NA		
EX/102	0.36	0.38	0.38	0.79	0.82	0.85	0.070	
	0.38	0.48	0.37	0.80	0.83	0.87		
EX/105	0.398	0.403	0.402	0.760	0.751	0.746	0.030	
QR/112*	NA	NA	NA	NA	NA	NA	0.100	no reason given
QR/113	0.277	0.365	0.348	0.661	0.410	0.661	0.041	
	0.283	0.367	0.363	0.656	0.420	0.678		
QR/118	NA	NA	NA	NA	NA	NA	-	
QR/122	0.631	0.585	0.611	1.146	1.226	1.115	0.050	
	0.585	0.670	0.620	1.171	1.260	0.980		
QR/123	NA	NA	NA	NA	NA	NA	-	
QR/124	NA	NA	NA	NA	NA	NA	-	
QR/125	0.55	0.53	0.55	0.97	1.00	0.99	0.005	
QR/126	0.575	0.674	0.683	1.199	1.227	1.298	0.010	
QR/127	0.463	0.463	0.481	0.925	1.033	0.978	0.010	technical reasons
QR/129	0.531	0.517	0.528	1.05	1.01	1.03	0.003	
QR/130	0.32	0.32	NA	0.72	NA	NA	0.200	no reason given
QR/132*	NA	NA	NA	NA	NA	NA	0.036	
QR/140	NA	NA	NA	NA	NA	NA	-	
QR/141	NA	NA	NA	NA	NA	NA	-	
QR/142	NA	NA	NA	NA	NA	NA	-	
QR/143	NA	NA	NA	NA	NA	NA	-	
QR/201	NA	NA	NA	NA	NA	NA	-	
QR/202	NA	NA	NA	NA	NA	NA	-	
QR/206	NA	NA	NA	NA	NA	NA	-	
QR/207	NA	NA	NA	NA	NA	NA	-	
QR/210	0.263	0.223	0.246	0.471	0.476	0.439	0.020	
QR/211	NA	NA	NA	NA	NA	NA	-	
QR/217	NA	NA	NA	NA	NA	NA	-	
QR/218	NA	NA	NA	NA	NA	NA	-	
QR/219	0.602	0.586	0.643	1.10	1.11	1.15	0.013	
QR/220	0.62	0.63	0.64	1.19	1.20	1.18	0.250	deadline misunderstood
QR/221	NA	NA	NA	NA	NA	NA	-	

*= analysed the sum of 2- and 3-FLUO

Lab.code	3-hydroxyfluorene in urine [ng/ml]							
	low-1	low-2	low-3	high-1	high-2	high-3	LOQ	delayed reporting
EX/101	NA	NA	NA	NA	NA	NA	-	
	NA	NA	NA	NA	NA	NA		
EX/102	0.27	0.25	0.23	0.36	0.38	0.35	0.060	
	0.27	0.27	0.28	0.38	0.36	0.38		
EX/105	0.398	0.403	0.402	0.760	0.751	0.746	0.030	
QR/112*	NA	NA	NA	NA	NA	NA	0.100	
QR/113	0.162	0.154	0.123	0.225	0.130	0.219	0.039	
	0.154	0.156	0.119	0.213	0.137	0.218		
QR/118	NA	NA	NA	NA	NA	NA	-	
QR/122	NA	NA	NA	NA	NA	NA	-	
	NA	NA	NA	NA	NA	NA		
QR/123	NA	NA	NA	NA	NA	NA	-	
QR/124	NA	NA	NA	NA	NA	NA	-	
QR/125	0.27	0.27	0.27	0.36	0.36	0.37	0.005	
QR/126	NA	NA	NA	NA	NA	NA	0.050	
QR/127	0.208	0.242	0.174	0.480	0.569	0.528	0.050	technical reasons
QR/129	0.238	0.243	0.239	0.343	0.338	0.321	0.003	
QR/130	NA	NA	NA	NA	NA	NA	-	
QR/132*	NA	NA	NA	NA	NA	NA	-	
QR/140	NA	NA	NA	NA	NA	NA	-	
QR/141	NA	NA	NA	NA	NA	NA	-	
QR/142	NA	NA	NA	NA	NA	NA	-	
QR/143	NA	NA	NA	NA	NA	NA	-	
QR/201	NA	NA	NA	NA	NA	NA	-	
QR/202	NA	NA	NA	NA	NA	NA	-	
QR/206	NA	NA	NA	NA	NA	NA	-	
QR/207	NA	NA	NA	NA	NA	NA	-	
QR/210	NA	NA	NA	NA	NA	NA	-	
QR/211	NA	NA	NA	NA	NA	NA	-	
QR/217	NA	NA	NA	NA	NA	NA	-	
QR/218	NA	NA	NA	NA	NA	NA	-	
QR/219	0.423	0.216	0.508	0.317	0.551	0.295	0.013	
QR/220	0.32	0.30	0.32	0.43	0.42	0.43	0.200	misunderstanding reg. deadline
QR/221	NA	NA	NA	NA	NA	NA	-	

*= analysed the sum of 2- and 3-FLUO

Lab.code	9-hydroxyfluorene in urine [ng/ml]							
	low-1	low-2	low-3	high-1	high-2	high-3	LOQ	delayed reporting
EX/101	NA	NA	NA	NA	NA	NA	-	
	NA	NA	NA	NA	NA	NA		
EX/102	0.78	0.76	0.70	1.38	1.49	1.33	0.060	
	0.76	0.74	0.74	1.44	1.44	1.38		
EX/105	NA	NA	NA	NA	NA	NA	-	
QR/112	0.53	0.57	0.57	0.48	0.56	0.49	0.100	no reason given
QR/113	0.654	0.616	0.469	1.13	0.634	1.10	0.046	
	0.646	0.620	0.466	1.07	0.666	1.09		
QR/118	NA	NA	NA	NA	NA	NA	-	
QR/122	NA	NA	NA	NA	NA	NA	-	
	NA	NA	NA	NA	NA	NA		
QR/123	NA	NA	NA	NA	NA	NA	-	
QR/124	NA	NA	NA	NA	NA	NA	-	
QR/125	0.81	0.79	0.82	1.37	1.42	1.41	0.010	
QR/126*	NA	NA	NA	NA	NA	NA	0.050	
QR/127	NA	NA	NA	NA	NA	NA	-	
QR/129	NA	NA	NA	NA	NA	NA	-	
QR/130	NA	NA	NA	NA	NA	NA	-	
QR/132	NA	NA	NA	NA	NA	NA	-	
QR/140	NA	NA	NA	NA	NA	NA	-	
QR/141	NA	NA	NA	NA	NA	NA	-	
QR/142	NA	NA	NA	NA	NA	NA	-	
QR/143	NA	NA	NA	NA	NA	NA	-	
QR/201	NA	NA	NA	NA	NA	NA	-	
QR/202	NA	NA	NA	NA	NA	NA	-	
QR/206	NA	NA	NA	NA	NA	NA	-	
QR/207	NA	NA	NA	NA	NA	NA	-	
QR/210	NA	NA	NA	NA	NA	NA	-	
QR/211	NA	NA	NA	NA	NA	NA	-	
QR/217	NA	NA	NA	NA	NA	NA	-	
QR/218	NA	NA	NA	NA	NA	NA	-	
QR/219	NA	NA	NA	NA	NA	NA	-	
QR/220	1.01	0.85	0.87	1.46	1.53	1.43	0.250	misunderstanding reg. deadline
QR/221	NA	NA	NA	NA	NA	NA	-	

*= analysed the sum of 3- and 9-FLUO

Lab.code	1-hydroxyPHENanthrene in urine [ng/ml]							
	low-1	low-2	low-3	high-1	high-2	high-3	LOQ	delayed reporting
EX/101*	NA	NA	NA	NA	NA	NA	0.390	
	NA	NA	NA	NA	NA	NA		
EX/102	0.57	0.54	0.51	0.88	0.87	0.83	0.070	
	0.55	0.54	0.52	0.90	0.89	0.81		
EX/105	0.373	0.367	0.367	0.620	0.617	0.594	0.030	
QR/112	0.50	0.49	0.48	0.82	0.84	0.89	0.100	no reason given
QR/113	0.222	0.201	0.182	0.358	0.232	0.364	0.031	
	0.186	0.206	0.195	0.352	0.234	0.384		
QR/118	NA	NA	NA	NA	NA	NA	-	
QR/122	0.546	0.520	0.533	0.928	0.889	0.866	0.005	
	0.514	0.515	0.504	0.928	0.858	0.869		
QR/123	NA	NA	NA	NA	NA	NA	-	
QR/124	NA	NA	NA	NA	NA	NA	-	
QR/125	0.48	0.49	0.52	0.81	0.83	0.83	0.005	
QR/126	0.353	0.442	0.463	0.838	0.787	0.727	0.010	
QR/127	0.284	0.305	0.332	0.530	0.458	0.550	0.010	technical reasons
QR/129	0.432	0.433	0.443	0.768	0.732	0.725	0.003	
QR/130	NA	NA	NA	NA	NA	NA	-	
QR/132*	NA	NA	NA	NA	NA	NA	0.009	
QR/140	NA	NA	NA	NA	NA	NA	-	
QR/141	0.613	0.592	0.571	1.015	1.102	1.020	0.010	
QR/142	NA	NA	NA	NA	NA	NA	-	
QR/143	NA	NA	NA	NA	NA	NA	-	
QR/201	NA	NA	NA	NA	NA	NA	-	
QR/202	NA	NA	NA	NA	NA	NA	-	
QR/206	NA	NA	NA	NA	NA	NA	-	
QR/207	NA	NA	NA	NA	NA	NA	-	
QR/210	0.429	0.424	0.432	0.618	0.679	0.647	0.020	
QR/211	NA	NA	NA	NA	NA	NA	-	
QR/217	NA	NA	NA	NA	NA	NA	-	
QR/218	NA	NA	NA	NA	NA	NA	-	
QR/219	0.511	0.881	0.953	0.950	0.906	1.39	0.013	
QR/220	0.43	0.43	0.43	0.67	0.67	0.70	0.200	misunderstanding reg. deadline
QR/221*	NA	NA	NA	NA	NA	NA	-	

*= analysed the sum of 1-Phen and 9-Phen

Lab.code	2-hydroxyPHENanthrene in urine [ng/ml]							
	low-1	low-2	low-3	high-1	high-2	high-3	LOQ	delayed reporting
EX/101	0.38	0.38	0.39	0.79	0.78	0.76	0.220	
	0.38	0.54	0.39	0.89	0.85	0.82		
EX/102	0.64	0.63	0.61	0.81	0.78	0.79	0.040	
	0.65	0.65	0.64	0.83	0.80	0.78		
EX/105	NA	NA	NA	NA	NA	NA	-	
QR/112*	NA	NA	NA	NA	NA	NA	-	no reason given
QR/113	0.138	0.138	0.130	0.244	0.163	0.268	0.027	
	0.122	0.140	0.128	0.231	0.170	0.281		
QR/118	NA	NA	NA	NA	NA	NA	-	
QR/122	0.316	0.310	0.395	0.556	0.563	0.697	0.005	
	0.306	0.385	0.307	0.562	0.594	0.531		
QR/123	NA	NA	NA	NA	NA	NA	-	
QR/124	NA	NA	NA	NA	NA	NA	-	
QR/125	0.29	0.29	0.31	0.48	0.49	0.49	0.005	
QR/126	0.375	0.447	0.457	0.712	0.751	0.751	0.010	
QR/127	0.158	0.173	0.168	0.446	0.474	0.469	0.010	technical reasons
QR/129*	NA	NA	NA	NA	NA	NA	0.001	
QR/130	NA	NA	NA	NA	NA	NA	-	
QR/132	0.440	0.400	0.420	0.680	0.640	0.680	0.008	
QR/140	NA	NA	NA	NA	NA	NA	-	
QR/141	0.256	0.241	0.239	0.396	0.419	0.393	0.010	
QR/142	NA	NA	NA	NA	NA	NA	-	
QR/143	NA	NA	NA	NA	NA	NA	-	
QR/201	NA	NA	NA	NA	NA	NA	-	
QR/202	NA	NA	NA	NA	NA	NA	-	
QR/206	NA	NA	NA	NA	NA	NA	-	
QR/207	NA	NA	NA	NA	NA	NA	-	
QR/210	0.212	0.205	0.200	0.331	0.322	0.317	0.020	
QR/211	NA	NA	NA	NA	NA	NA	-	
QR/217	NA	NA	NA	NA	NA	NA	-	
QR/218	NA	NA	NA	NA	NA	NA	-	
QR/219	ND	ND	ND	0.409	0.704	0.903	0.013	
QR/220	0.24	0.21	0.22	0.36	0.37	0.35	0.100	misunderstanding reg.
QR/221	0.394	0.395	0.204	0.566	0.476	0.410	0.084	

*= analysed the sum of 2-PHEN and 3-PHEN

Lab.code	3-hydroxyPHENanthrene in urine [ng/ml]							
	low-1	low-2	low-3	high-1	high-2	high-3	LOQ	delayed reporting
EX/101	0.17	0.21	0.22	0.54	0.51	0.47	0.130	
	0.31	0.40	0.34	0.70	0.71	0.64		
EX/102	0.21	0.22	0.22	0.39	0.39	0.41	0.230	
	0.23	0.23	0.22	0.39	0.38	0.40		
EX/105	NA	NA	NA	NA	NA	NA	-	
QR/112*	NA	NA	NA	NA	NA	NA	-	
QR/113	0.208	0.200	0.182	0.318	0.219	0.355	0.029	
	0.166	0.202	0.181	0.336	0.223	0.388		
QR/118	NA	NA	NA	NA	NA	NA	-	
QR/122	0.399	0.397	0.407	0.640	0.611	0.655	0.005	
	0.393	0.412	0.386	0.629	0.624	0.650		
QR/123	NA	NA	NA	NA	NA	NA	-	
QR/124	NA	NA	NA	NA	NA	NA	-	
QR/125	0.39	0.41	0.41	0.63	0.64	0.62	0.005	
QR/126	0.265	0.315	0.314	0.479	0.519	0.489	0.010	
QR/127	0.297	0.231	0.258	0.438	0.417	0.400	0.010	technical reasons
QR/129	NA	NA	NA	NA	NA	NA	-	
QR/130	NA	NA	NA	NA	NA	NA	-	
QR/132	0.400	0.360	0.360	0.580	0.540	0.580	0.006	
QR/140	NA	NA	NA	NA	NA	NA	-	
QR/141	0.266	0.283	0.275	0.437	0.479	0.454	0.010	
QR/142	NA	NA	NA	NA	NA	NA	-	
QR/143	NA	NA	NA	NA	NA	NA	-	
QR/201	NA	NA	NA	NA	NA	NA	-	
QR/202	NA	NA	NA	NA	NA	NA	-	
QR/206	NA	NA	NA	NA	NA	NA	-	
QR/207	NA	NA	NA	NA	NA	NA	-	
QR/210	0.186	0.175	0.192	0.305	0.296	0.286	0.020	
QR/211	NA	NA	NA	NA	NA	NA	-	
QR/217	NA	NA	NA	NA	NA	NA	-	
QR/218	NA	NA	NA	NA	NA	NA	-	
QR/219	0.413	0.799	0.928	0.798	0.749	1.22	0.013	
QR/220	0.32	0.30	0.31	0.49	0.50	0.49	0.100	misunderstanding reg.
QR/221	0.501	0.485	0.434	0.673	0.765	0.789	0.051	

*= analysed the sum of 2-PHEN and 3-PHEN

Lab.code	4-hydroxyPHENanthrene in urine [ng/ml]							
	low-1	low-2	low-3	high-1	high-2	high-3	LOQ	delayed reporting
EX/101	ND	ND	ND	ND	ND	ND	0.290	
	ND	ND	ND	ND	ND	ND		
EX/102	0.27	0.24	0.23	0.40	0.40	0.36	0.070	
	0.24	0.26	0.27	0.43	0.44	0.37		
EX/105	NA	NA	NA	NA	NA	NA	-	
QR/112	0.14	ND	ND	0.15	0.17	0.13	0.100	no reason given
QR/113	0.0446	0.0446	0.0369	0.0849	0.0585	0.0762	0.022	
	0.0361	0.0443	0.0376	0.0804	0.0572	0.0915		
QR/118	NA	NA	NA	NA	NA	NA	-	
QR/122	0.123	0.122	0.122	0.244	0.247	0.252	0.005	
	0.124	0.120	0.131	0.249	0.269	0.259		
QR/123	NA	NA	NA	NA	NA	NA	-	
QR/124	NA	NA	NA	NA	NA	NA	-	
QR/125	0.12	0.12	0.13	0.24	0.24	0.24	0.010	
QR/126	0.130	0.129	0.130	0.289	0.273	0.279	0.010	
QR/127	0.114	0.198	0.152	0.322	0.273	0.369	0.010	technical reasons
QR/129	0.150	0.149	0.124	0.225	0.262	0.257	0.003	
QR/130	NA	NA	NA	NA	NA	NA	-	
QR/132	0.120	0.100	0.100	0.220	0.200	0.220	0.003	
QR/140	NA	NA	NA	NA	NA	NA	-	
QR/141	0.097	0.098	0.110	0.206	0.231	0.219	0.010	
QR/142	NA	NA	NA	NA	NA	NA	-	
QR/143	NA	NA	NA	NA	NA	NA	-	
QR/201	NA	NA	NA	NA	NA	NA	-	
QR/202	NA	NA	NA	NA	NA	NA	-	
QR/206	NA	NA	NA	NA	NA	NA	-	
QR/207	NA	NA	NA	NA	NA	NA	-	
QR/210*	NA	NA	NA	NA	NA	NA	0.040	
QR/211	NA	NA	NA	NA	NA	NA	-	
QR/217	NA	NA	NA	NA	NA	NA	-	
QR/218	NA	NA	NA	NA	NA	NA	-	
QR/219	0.175	0.167	0.198	0.283	0.279	0.279	0.013	
QR/220	0.12	0.12	0.12	0.21	0.21	0.22	0.100	misunderstanding reg. deadline
QR/221	0.131	0.090	0.235	0.293	0.251	0.250	0.086	

*= analysed the sum of 4-PHEN and 9-PHEN

Lab.code	9-hydroxyPHENanthrene in urine [ng/ml]							
	low-1	low-2	low-3	high-1	high-2	high-3	LOQ	delayed reporting
EX/101*	NA	NA	NA	NA	NA	NA	0.390	
	NA	NA	NA	NA	NA	NA		
EX/102	0.64	0.64	0.64	0.66	0.64	0.66	0.110	
	0.67	0.58	0.67	0.63	0.71	0.58		
EX/105	NA	NA	NA	NA	NA	NA	-	
QR/112	NA	NA	NA	NA	NA	NA	-	
QR/113	NA	NA	NA	NA	NA	NA	-	
	NA	NA	NA	NA	NA	NA		
QR/118	NA	NA	NA	NA	NA	NA	-	
QR/122	0.078	0.069	0.075	0.080	0.080	0.071	0.005	
	0.068	0.072	0.071	0.088	0.079	0.076		
QR/123	NA	NA	NA	NA	NA	NA	-	
QR/124	NA	NA	NA	NA	NA	NA	-	
QR/125	0.08	0.09	0.08	0.07	0.09	0.09	0.010	
QR/126	0.291	0.308	0.305	0.387	0.388	0.411	0.025	
QR/127	0.539	0.578	0.566	0.713	0.686	0.792	0.010	technical reasons
QR/129	ND	ND	ND	ND	ND	ND	0.100	
QR/130	1.2	1.1	NA	1.81	NA	NA	0.200	no reason given
QR/132	NA	NA	NA	NA	NA	NA	-	
QR/140	NA	NA	NA	NA	NA	NA	-	
QR/141	NA	NA	NA	NA	NA	NA	-	
QR/142	NA	NA	NA	NA	NA	NA	-	
QR/143	NA	NA	NA	NA	NA	NA	-	
QR/201	NA	NA	NA	NA	NA	NA	-	
QR/202	NA	NA	NA	NA	NA	NA	-	
QR/206	NA	NA	NA	NA	NA	NA	-	
QR/207	NA	NA	NA	NA	NA	NA	-	
QR/210**	NA	NA	NA	NA	NA	NA	0.040	
QR/211	NA	NA	NA	NA	NA	NA	-	
QR/217	NA	NA	NA	NA	NA	NA	-	
QR/218	NA	NA	NA	NA	NA	NA	-	
QR/219	0.222	0.111	ND	0.086	0.108	ND	0.025	
QR/220	0.49	0.44	0.46	0.42	0.50	0.47	0.300	misunderstanding reg.
QR/221*	NA	NA	NA	NA	NA	NA	0.174	

*= analysed the sum of 1-PHEN and 9-PHEN

**= analysed the sum of 4-PHEN and 9-PHEN

Lab.code	1-hydroxypyrene in urine [ng/ml]							
	low-1	low-2	low-3	high-1	high-2	high-3	LOQ	delayed reporting
EX/101	0.24	0.27	ND	0.35	0.40	0.29	0.240	
	ND	ND	ND	0.42	0.44	0.32		
EX/102	0.06	0.07	0.10	0.21	0.24	0.23	0.090	
	0.09	0.07	0.09	0.20	0.20	0.18		
EX/105	0.170	0.171	0.167	0.340	0.336	0.312	0.200	
QR/112	0.17	0.18	0.14	0.34	0.38	0.31	0.100	no reason given
QR/113	0.0822	0.0779	0.0625	0.153	0.0887	0.128	0.048	
	0.0613	0.0707	0.0582	0.142	0.0792	0.151		
QR/118	ND	ND	ND	0.2	0.2	0.2	0.200	
QR/122	0.124	0.117	0.124	0.269	0.238	0.281	0.010	
	0.123	0.127	0.121	0.246	0.259	0.262		
QR/123	0.15	0.18	0.17	0.27	0.30	0.26	0.050	
QR/124	0.154	0.152	0.149	0.322	0.308	0.322	0.020	
QR/125	0.15	0.15	0.17	0.31	0.31	0.31	0.010	
QR/126	0.291	0.308	0.305	0.387	0.388	0.411	0.025	
QR/127	0.145	0.113	0.144	0.271	0.294	0.306	0.010	technical reasons
QR/129	0.133	0.137	0.113	0.232	0.233	0.233	0.003	
QR/130	0.24	0.35	NA	0.42	NA	NA	0.200	no reason given
QR/132	0.160	0.140	0.160	0.280	0.280	0.300	0.009	
QR/140	0.185	0.112	0.112	0.206	0.118	0.164	0.200	
QR/141	0.154	0.120	0.131	0.269	0.286	0.263	-	
QR/142	NA	NA	NA	NA	NA	NA	-	
QR/143	0.120	0.125	0.117	0.244	0.224	0.253	0.010	
QR/201	0.166	0.188	0.174	0.344	0.326	0.35	0.050	
QR/202	0.295	0.248	0.252	0.481	0.450	0.426	0.100	
QR/206	0.19	0.2	0.2	0.32	0.3	0.3	0.200	
QR/207	0.115	0.116	0.109	0.177	0.203	0.209	0.102	
QR/210	0.126	0.107	0.133	0.349	0.289	0.281	0.050	
QR/211	0.045	0.044	0.047	0.09	0.087	0.089	0.025	
QR/217	0.127	0.132	0.122	0.276	0.252	0.276	0.100	
QR/218	0.170	0.165	0.212	0.385	0.403	0.379	0.040	
QR/219	0.339	0.343	0.334	0.668	0.777	0.540	0.013	
QR/220	0.10	0.11	0.11	0.22	0.21	0.22	0.020	misunderstanding reg.
QR/221	0.192	0.209	0.211	0.428	0.432	0.412	0.025	

Lab.code	3-hydroxybenzo(a)pyrene in urine [ng/ml]							
	low-1	low-2	low-3	high-1	high-2	high-3	LOQ	delayed reporting
EX/101	NA	NA	NA	NA	NA	NA	-	
	NA	NA	NA	NA	NA	NA		
EX/102	NA	NA	NA	NA	NA	NA	-	
	NA	NA	NA	NA	NA	NA		
EX/105	NA	NA	NA	NA	NA	NA	-	
QR/112	NA	NA	NA	NA	NA	NA	-	
QR/113	NA	NA	NA	NA	NA	NA	-	
	NA	NA	NA	NA	NA	NA		
QR/118	NA	NA	NA	NA	NA	NA	-	
QR/122	0.001869	0.002219	0.001826	0.003752	0.003580	0.003536	0.00005	
QR/123	NA	NA	NA	NA	NA	NA	-	
QR/124	NA	NA	NA	NA	NA	NA	-	
QR/125	NA	NA	NA	NA	NA	NA	-	
QR/126	ND	ND	ND	ND	ND	ND	0.50000	
QR/127	ND	ND	ND	ND	ND	ND	0.05000	
QR/129	ND	ND	ND	ND	ND	ND	0.10000	
QR/130	NA	NA	NA	NA	NA	NA	0.20000	
QR/132	NA	NA	NA	NA	NA	NA	-	
QR/140	NA	NA	NA	NA	NA	NA	-	
QR/141	NA	NA	NA	NA	NA	NA	-	
QR/142	NA	NA	NA	NA	NA	NA	-	
QR/143	NA	NA	NA	NA	NA	NA	-	
QR/201	0.000641	0.000619	0.000599	0.001330	0.001340	0.001300	0.00010	
QR/202	NA	NA	NA	NA	NA	NA	-	
QR/206	NA	NA	NA	NA	NA	NA	-	
QR/207	NA	NA	NA	NA	NA	NA	-	
QR/210	NA	NA	NA	NA	NA	NA	-	
QR/211	NA	NA	NA	NA	NA	NA	-	
QR/217	NA	NA	NA	NA	NA	NA	-	
QR/218	NA	NA	NA	NA	NA	NA	-	
QR/219	ND	ND	ND	ND	ND	ND	0.25000	
QR/220	0.001100	0.001200	0.001200	0.002100	0.00200	0.002100	0.00010	misunderstanding reg.
QR/221	ND	ND	ND	ND	ND	ND	0.09900	

ND = not detected, NA = not analysed