

GUIDE FOR FINAL REPORT

Progetto Trieste 2008, II° round – Rev 4 en

The Test Material can be incurred or spiked

Test material U452A, swine urine

Table 1: results of confirmatory analyses performed on test material U452A (spiked sample): stylbenes detection.

dienestrol, assigned value: $2.07 \pm 0.17 \mu\text{g/l}$

This value is the mean of participant's results (excluding outliers and values obtained by screening) = Consensus Value

Lab. code	Result (µg/l)	Recovery %	z-score	Decision limit CCα (µg/l)	Detection capability CCβ (µg/l)	ISO 17025
02	1.4	-	-0.80	1	1	y
03	3.7	-	1.94	0.5	0.7	y
05	≥2	-		2	-	y
06	2.7	100	0.75	1.7	2.0	y
08	2.12	-	0.06	0.3	0.4	n
09	1.6*	-	-0.56	1	-	y
12	ND	-	-1.27	5	-	y
17	ND	-		0.2	0.3	y
19	1.8	-		1	-	n
21	> 0.5	-		1	2	n
24	ND	-		1.7	3.3	n
25	2.1	-	0.04			n
27	2.8	-	0.87	0.5	2.4	y
28	2.70	68.9	0.75	0.39	0.65	y
33	1.69	-	-0.45	0.42	0.52	y
35	1.68**	102	-0.46	0.06	0.10	y
37	2.1	100	0.04	0.19	0.33	y
47	1.5	-	1.23	0.88	0.94	y
59	NS	76.3	-0.05	1.23	1.6	n
63	1.2	100	-1.04	0.3	0.6	n

ND: The lab states it has not found the analyte (false negative)

z-score: parameter which expresses quantitatively the performance of laboratory.

NS: not searched. The lab tested the material but didn't look for DIEN

*qualitative method
**not correct for recovery

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Test material U452A, swine urine

Participating laboratories	27	Labs reporting the presence of DIEN ($n = 23$)	21 (91.3%)
Labs that sent results ($n = 27$)	24 (88.8%)	Labs reporting false positive results ($n = 24$)	1 (4.1%)
Labs that performed DIEN ($n = 24$)	23 (95.8%)	Labs reporting false negative results ($n = 23$)	2 (8.7%)

Mean concentration ($n = 17$)	2.07 $\mu\text{g/l}$	<div style="border: 1px solid black; padding: 5px; display: inline-block;"> Mean concentration, sd e cv here, are calculated using all the quantitative data (including outliers). </div>
Standard deviation ($n = 17$)	0.72 $\mu\text{g/l}$	
Coefficient of variation ($n = 17$)	34.8%	

Table 2: assigned value and target standard deviations.

Analyte	Assigned value ($\mu\text{g/l}$)				Target standard deviation		
	Data points, n	Robust mean	SD	CV	Analytical uncertainty u	b	$\sigma = bX$
dienestrol	17	2.07	0.72	34.80	0.17	0.41	0.84

Number of data used to calculate the assigned value

$CV = SD / rm * 100$

$u = \sigma rm / \sqrt{n}$

$b = \%RDS$ (calculated with Horwitz equation). It is the expected interlaboratory CV

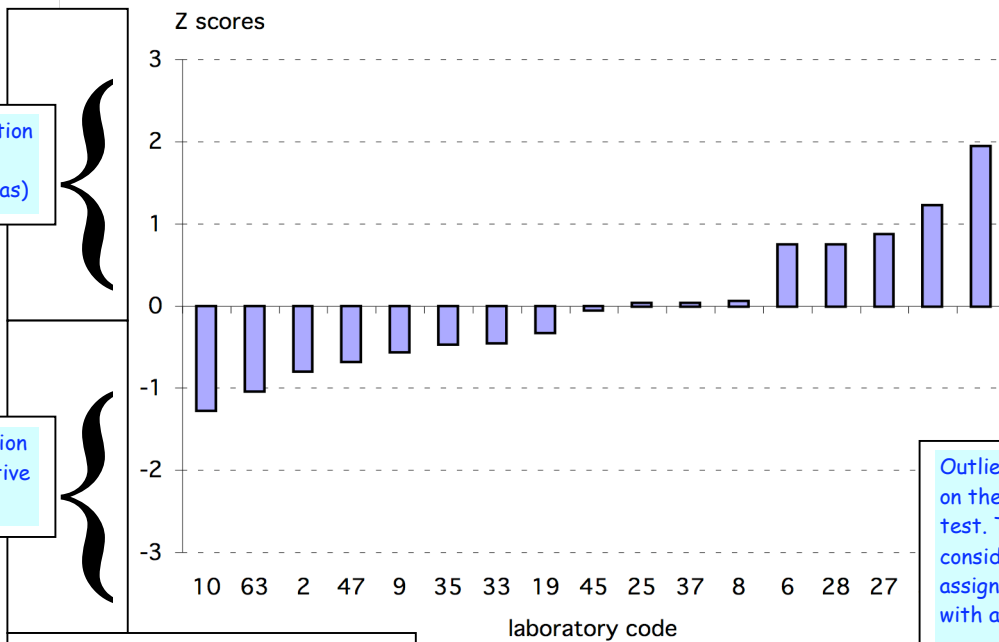
Horrat value

CV (RDS), $n = 17$:	34.80%
Horwitz expected CV:	40.56%
Horrat (= CV / Horwitz CV):	0.85

$u / \sigma = 20.82\%$

If u / σ is more than 50% X the assigned value and the z-score would also be written in *italics*

Test Material U452A, dienestrol, 2.07 $\mu\text{g/l}$



overestimation results (positive bias)

underestimation results (negative bias)

The chart shows the z-scores: the 0 value represents the assigned value; so the lab performance is better as it gets closer to 0.

Outliers: data diverging from others on the basis of Dixon or Grubbs test. The outliers are not considered in calculating the assigned value. Outliers are marked with an asterisk near the lab code

$|z| < 2$ = satisfactory
 $|z| > 3$ = unsatisfactory
 $2 < |z| < 3$ = questionable
 * = outliers

Test material U452A, swine urine

Table 3: decision limits in confirmatory analyses of stylobenes.

Decision limits – CC α ($\mu\text{g/l}$)

CC α = Decision Limit
 Concentration value above which the probability of a false positive have to be less than 1% in the confirms analysis (EU Decision 657/2002)

Lab. code	cis-DES	trans-DES	HEX	DIEN
02	1		1	1
03	NS	0.9	0.2	0.5
05	1			2
06	NS			1.7
08	0.5	-	0.3	0.3
09	NS	1	1	1
10	2	2	1	5
11	-	0.2	0.9	0.2
12		1	1	1
17	0.5	0.5	0.5	1
19		1.3	1.8	1.7
21	NS	0.5	0.5	0.5
24	< 5	NS	NS	NS
25	NS	1	2	2
27	0.3	0.3	0.5	0.5
28	NS	0.37	0.44	0.39
33	NS	0.19	0.2	0.42
35		-		0.06
37	NS	0.08	0.13	0.19
39	0.83	-	0.87	0.88
45	NS	1.23	1.23	1.23
47		0.3	0.4	0.3
59	0.5	-	0.5	NS
63	0.8	0.8	0.4	0.2

The table shows the list of molecules that should have been analyzed (which could be present)

NS: not searched.
 The participant 59 don't look for DIEN

Numbers in *italics* are LOD

Test material U452A, swine urine

Table 4: false positive results and missing analyses.

Lab. code	False positive (µg/l)	Analyte not searched
02	none	-
03	none	cis-DES
05	none	-
06	none	cis-DES
08	none	-
09	none	cis-DES
10	none	-
11	none	cis-DES
12	none	-
17	none	-
19	none	-
21	none	cis-DES
24	-	DES and HEX
25	none	cis-DES
27	none	-
28	none	cis-DES
33	none	cis-DES
35	none	-
37	none	cis-DES
39	none	-
45	none	cis-DES
47	none	-
59	DES (0.9)	DIEN
63	none	-

"None " means that the laboratory had no false positive results

"-": the lab didn't look for other analytes, no false positive could occurs

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Test material U452B-1 and U452B-2, swine

The positive "B" Test Material is the same of the corresponding "A" material. The assigned value is calculated using only data from confirmatory analysis ("A")

Table 5: results of screening analyses performed on test material U452B-1 (spiked sample): stylbenes detection.

dienestrol, assigned value: 2.07 + 0.17 µg/l (U452B-1 = U452A)

Lab code	Response (µg/l)	Evaluation	z-score	False positive (µg/l)	Not searched	ISO 17025
05	NS			-	HEX and DIEN	-
10	Neg.	unsatisfactory		none	-	y
27	Pos. (3)	satisfactory	1.11	none	-	y
35	Neg.	unsatisfactory		-	-	y
38	NS			DES (0.4)	HEX and DIEN	-
46a	Pos.			-	-	-
46b	Neg.	unsatisfactory		none	-	n
49	Pos.	satisfactory				n
53	Pos.	satisfactory				y
57	Neg	unsatisfactory		none	-	-
58	NS			-	HEX and DIEN	-
59	Neg.	unsatisfactory		none	-	y
62	NS			none	HEX and DIEN	n
67	NS			none	HEX and DIEN	y

If the participants provide a quantitative results, a z-score value is assigned

The way to evaluate screening is described in the RF

NS = not searched. The laboratory didn't look for the molecule who was in the Test Material. Anyway, false positive occurrence can be checked.

CRITERIA: see page 5, table 3

Participating laboratories	17	Labs reporting DIEN (n = 9)	4 (44.4%)
Labs that sent results (n = 17)	14 (82.3%)	Labs reporting false neg. results (n = 9)	5 (55.5%)
Labs that performed DIEN test (n = 14)	9 (64.3%)	Labs reporting false pos. results (n = 14)	1 (7.1%)

If quantitative data are available, mean, SD and CV are reported.

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Negative "B" Test Materials are either from untreated animals, or Certify Referente Materials.

Test material U452B-1 and U452B-2, swine urine

Table 6: results of screening analyses performed on test material U452B-2 (negative sample): stylbenes detection.

U452B-2: negative (blank from untreated animals)

Lab code	Response (µg/l)	Evaluation	False positive (µg/l)	Not searched
05	Neg.	satisfactory	none	HEX and DIEN
10	Neg.	satisfactory	none	-
27	Neg.	satisfactory	none	-
35	Pos.	questionable	DES and HEX	DIEN
38	Neg.	satisfactory	none	HEX and DIEN
46b	Pos.	questionable	stylbenes group	-
49	Neg.	satisfactory	none	-
53	Neg.	satisfactory	none	-
57	Pos.	questionable	stylbenes group	-
58	Neg.		none	-
59	Neg.		none	-
62	Neg.		none	HEX and DIEN
67	Neg.	satisfactory	none	HEX and DIEN

The note refers to the of Final Report "Introduction" chapter, where it is possible to check the evaluation criteria applied.

CRITERIA: see page 5, table 3

Participating laboratories	16
Labs that sent results (n = 16)	13 (81.2%)
Labs reporting false pos. results (n = 13)	3 (23.1%)

Test material U452B-1 and U452B-2, swine urine

Table 7: detection capability in screening analyses of stylobenes.

Detection capability – CC β ($\mu\text{g/l}$)

Lab code	DES	HEX	DIEN
05	0.2	NS	NS
10	2		5
27	0.3		0.6
35	0.5		0.5
38	0.3	NS	NS
46a	2	-	-
46b	2	-	-
49	< 1	< 1	< 1
53	1	1	1
57	-	-	-
58	-	NS	NS
59	1	1	1
62	0.5	NS	NS
67	0.025	NS	NS

The list of molecules that should have been analyzed (and could be present in the Test Material).