

# **Round-robin tests for in-house measuring laboratories**

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## ***Results and Evaluation***

Round-robin test

„Organic solvents NIOSH 2016“

# Summary of laboratory test results

Sample 1

Unit	o-Xylene Z score		Toluene Z score		Ethylbenzene Z score	
	mg/tube		mg/tube		mg/tube	
10	0,088	0,41	0,370	0,64	0,135	0,96
55	0,088	0,30	0,351	0,09	0,121	-0,18
68	0,085	0,00	0,347	-0,02	0,135	0,96
79	0,072	-1,53	0,309	-1,11	0,101	-1,84
82	0,089	0,47	0,351	0,09	0,123	-0,01
85	0,098	1,53	0,379	0,90	0,148	2,02 E
86	0,084	-0,12	0,373	0,73	0,123	-0,01
90	0,078	-0,82	0,307	-1,17	0,115	-0,66
93	0,089	0,49	0,367	0,55	0,129	0,47
99	0,080	-0,59	0,326	-0,63	0,121	-0,18
100	0,093	1,00	0,365	0,50	0,129	0,47
118	0,091	0,70	0,366	0,52	0,129	0,47
131	0,090	0,59	0,346	-0,05	0,128	0,39
135	0,086	0,09	0,347	-0,03	0,123	0,00
144	0,088	0,41	0,340	-0,23	0,123	-0,04
147	0,087	0,23	0,345	-0,08	0,130	0,56
162	0,082	-0,35	0,340	-0,22	0,120	-0,26
167	0,089	0,47	0,361	0,38	0,130	0,56
168	0,085	0,04	0,363	0,45	0,124	0,05
195	0,067	-2,12 E	0,294	-1,55	0,103	-1,64
199	0,084	-0,12	0,340	-0,22	0,110	-1,07
208	0,084	-0,12	0,350	0,06	0,122	-0,09
220	0,085	0,00	0,330	-0,51	0,130	0,56
224	0,090	0,59	0,366	0,52	0,133	0,80
230	0,075	-1,18	0,260	-2,52 BE	0,099	-1,96
253	0,082	-0,37	0,361	0,38	0,119	-0,34
-	-	--	-	--	-	--
Method	ISO 5725-2		ISO 5725-2		ISO 5725-2	
Assessment	Z <=2,00		Z <=2,00		Z <=2,00	
Mean	0,085		0,348		0,123	
Reproducibility s.d.	0,007		0,022		0,011	
Rel. reproducibility s.d.	7,81 %		6,18 %		8,96 %	
Reference value	0,091		0,367		0,132	
Target s.d.	0,009		0,035		0,012	
Rel. target s.d.	10,00 %		10,00 %		10,00 %	
Lower limit of tolerance	0,068		0,278		0,099	
Upper limit of tolerance	0,102		0,417		0,148	
Type B outliers			1			
Number of laboratories with replicates outside of tolerance limits	1		1		1	
No. of laboratories that submitted results	26		26		26	
No. of laboratories after elimination of outliers type A-D and F (without laboratories that only gave states but no measured values)	26		25		26	
Explanation of outlier types						
A: Single outlier	Grubbs					
B: Differing laboratory mean	Grubbs					

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	o-Xylene Z score	Toluene Z score	Ethylbenzene Z score
C: Excessive laboratory s.d.	Cochran		
D: Excluded manually			
E: mean outside tolerance limits			
F:  Z-Score >3,5			
L: Differing laboratory mean (Grubbs II)	Grubbs für 2		

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# Summary of laboratory test results

Sample 2

Unit	o-Xylene Z score		n-Heptane Z score		n-Octane Z score		Ethylbenzene Z score	
	mg/tube		mg/tube		mg/tube		mg/tube	
10	0,102	0,73	0,366	-0,14	0,382	0,06	0,146	0,59
55	0,099	0,44	0,406	0,93	0,434	1,43	0,137	-0,06
68	0,094	-0,12	0,377	0,15	0,383	0,08	0,151	0,95
79	0,084	-1,18	0,306	-1,75 B	0,317	-1,66	0,117	-1,53
82	0,089	-0,64	0,370	-0,04	0,384	0,11	0,139	0,08
85	0,114	1,99					0,171	2,40 E
86	0,094	-0,12	0,386	0,39	0,425	1,19	0,137	-0,06
90	0,087	-0,85	0,343	-0,76	0,348	-0,84	0,129	-0,64
93	0,098	0,32	0,384	0,34	0,397	0,45	0,142	0,30
99	0,090	-0,54	0,356	-0,41	0,358	-0,57	0,137	-0,06
100	0,105	1,04	0,385	0,37	0,395	0,40	0,149	0,81
118	0,101	0,62	0,372	0,02	0,397	0,45	0,144	0,44
131	0,101	0,62	0,367	-0,12	0,379	-0,02	0,143	0,37
135	0,094	-0,07	0,365	-0,16	0,370	-0,25	0,137	-0,10
144	0,097	0,22	0,360	-0,29	0,370	-0,25	0,136	-0,17
147	0,093	-0,22	0,358	-0,36	0,371	-0,23	0,137	-0,06
162	0,089	-0,64	0,370	-0,04	0,385	0,14	0,133	-0,35
167	0,100	0,52	0,401	0,80	0,381	0,03	0,145	0,52
168	0,096	0,12	0,381	0,26	0,389	0,25	0,140	0,17
195	0,056	-4,11 BE	0,274	-2,62 BE	0,252	-3,36 BE	0,086	-3,76 BE
199	0,091	-0,43	0,340	-0,84	0,385	0,14	0,120	-1,30
208	0,093	-0,22	0,372	0,02	0,367	-0,34	0,134	-0,28
220	0,095	-0,01	0,365	-0,17	0,390	0,27	0,135	-0,21
224	0,099	0,41	0,372	0,02	0,388	0,22	0,146	0,59
230	0,079	-1,69	0,341	-0,82	0,330	-1,31	0,110	-2,02 E
253	0,092	-0,28	0,403	0,85	0,389	0,24	0,133	-0,35
-	-	--	-	--	-	--	-	--
Method	ISO 5725-2		ISO 5725-2		ISO 5725-2		ISO 5725-2	
Assessment	Z <=2,00		Z <=2,00		Z <=2,00		Z <=2,00	
Mean	0,095		0,371		0,380		0,138	
Reproducibility s.d.	0,007		0,018		0,025		0,012	
Rel. reproducibility s.d.	7,53 %		4,85 %		6,63 %		8,59 %	
Reference value	0,103		0,387		0,396		0,150	
Target s.d.	0,010		0,037		0,038		0,014	
Rel. target s.d.	10,00 %		10,00 %		10,00 %		10,00 %	
Lower limit of tolerance	0,076		0,297		0,304		0,110	
Upper limit of tolerance	0,114		0,446		0,456		0,165	
Type B outliers	1		2		1		1	
Number of laboratories with replicates outside of tolerance limits	1		1		1		3	
No. of laboratories that submitted results	26		25		25		26	
No. of laboratories after elimination of outliers type A-D and F (without laboratories that only gave states but no measured values)	25		23		24		25	
Explanation of outlier types								
A: Single outlier	Grubbs							
B: Differing laboratory mean	Grubbs							

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	o-Xylene Z score	n-Heptane Z score	n-Octane Z score	Ethylbenzene Z score
C: Excessive laboratory s.d.	Cochran			
D: Excluded manually				
E: mean outside tolerance limits				
F:  Z-Score >3,5				
L: Differing laboratory mean (Grubbs II)	Grubbs für 2			

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# Summary of laboratory test results

Sample 3

Unit	n-Hexane Z score		n-Octane Z score		n-Propyl acetate Z score	
	mg/tube		mg/tube		mg/tube	
10	0,744	0,63	0,456	0,25	0,240	1,44
55	0,741	0,59	0,461	0,36	0,201	-0,42
68	0,666	-0,48	0,432	-0,29	0,193	-0,80
79	0,667	-0,46	0,429	-0,35		
82	0,725	0,36	0,447	0,04	0,216	0,30
85	0,817	1,68				
86	0,734	0,49	0,493	1,08	0,229	0,92
90	0,631	-0,98	0,401	-0,99	0,180	-1,42
93	0,717	0,25	0,459	0,31	0,206	-0,18
99	0,672	-0,39	0,425	-0,45	0,218	0,40
100	0,737	0,53	0,456	0,25	0,218	0,40
118	0,682	-0,25	0,458	0,29	0,251	1,97
131	0,655	-0,64	0,438	-0,16	0,214	0,20
135	0,644	-0,80	0,426	-0,42	0,211	0,06
144	0,679	-0,30	0,428	-0,38	0,210	0,03
147	0,646	-0,77	0,440	-0,11	0,203	-0,32
162	0,691	-0,12	0,445	0,00	0,186	-1,13
167	0,725	0,36	0,434	-0,25	0,205	-0,22
168	0,708	0,12	0,456	0,26	0,223	0,61
195	0,792	1,32	0,439	-0,14	0,201	-0,42
199	0,680	-0,28	0,456	0,25	0,200	-0,46
208	0,701	0,02	0,449	0,09	0,223	0,63
220	0,660	-0,57	0,460	0,34	0,210	0,01
224	0,735	0,51	0,459	0,31	0,222	0,59
230	0,630	-1,00	0,370	-1,69 B	0,170	-1,89
253	0,711	0,16	0,433	-0,27	0,203	-0,32
-	-	--	-	--	-	--
Method	ISO 5725-2		ISO 5725-2		ISO 5725-2	
Assessment	Z <=2,00		Z <=2,00		Z <=2,00	
Mean	0,700		0,445		0,210	
Reproducibility s.d.	0,047		0,018		0,018	
Rel. reproducibility s.d.	6,70 %		4,09 %		8,53 %	
Reference value	0,711		0,459		0,229	
Target s.d.	0,070		0,045		0,021	
Rel. target s.d.	10,00 %		10,00 %		10,00 %	
Lower limit of tolerance	0,560		0,356		0,168	
Upper limit of tolerance	0,840		0,534		0,252	
Type B outliers			1			
No. of laboratories that submitted results	26		25		24	
No. of laboratories after elimination of outliers type A-D and F (without laboratories that only gave states but no measured values)	26		24		24	
Explanation of outlier types						
A: Single outlier	Grubbs					
B: Differing laboratory mean	Grubbs					
C: Excessive laboratory s.d.	Cochran					
D: Excluded manually						

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n-Hexane Z score	n-Octane Z score	n-Propyl acetate Z score
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E: mean outside tolerance limits

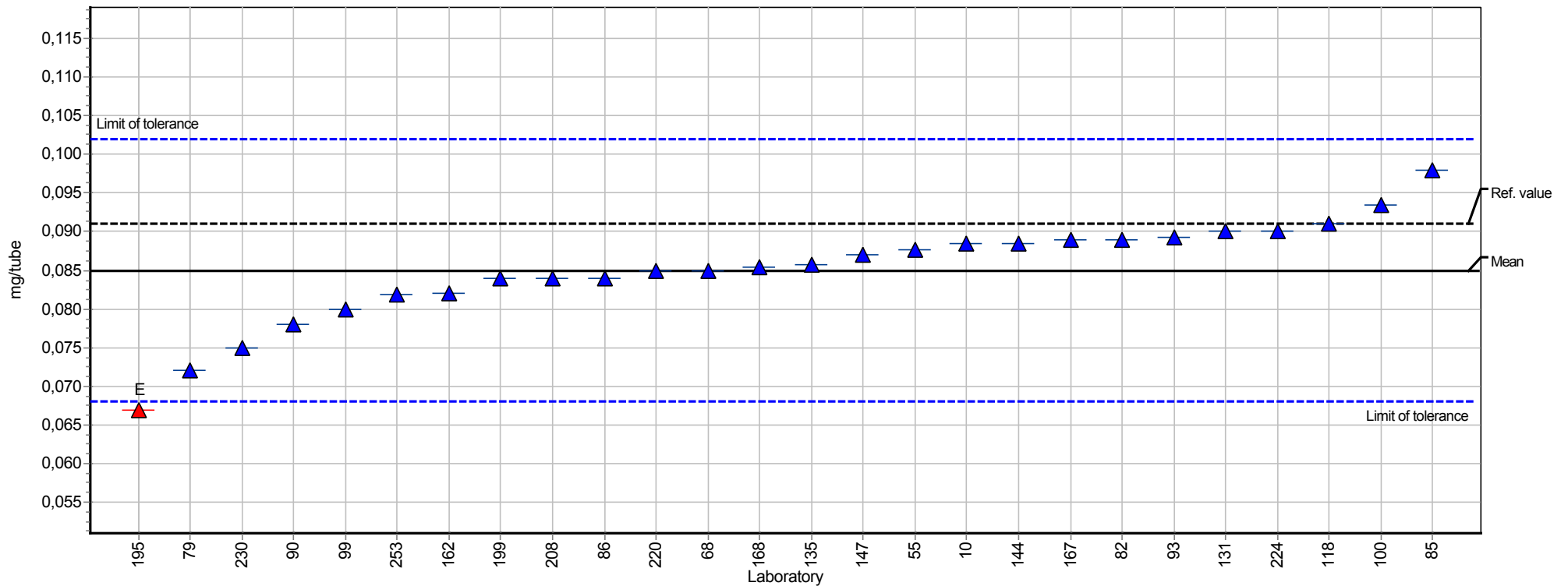
F: |Z-Score|>3,5

L: Differing laboratory mean      Grubbs für 2  
(Grubbs II)

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## Summary results

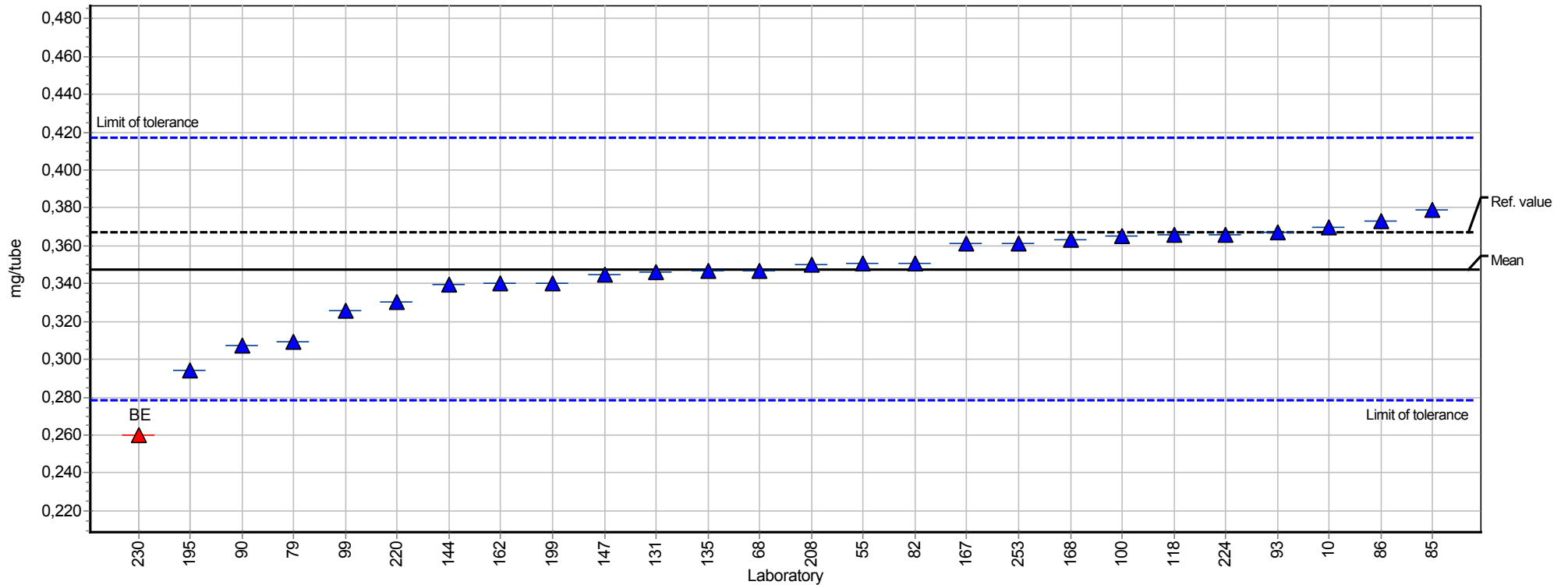
<b>Measurand:</b>	o-Xylene	<b>Mean:</b>	0,085 mg/tube
<b>Sample:</b>	1	<b>Reproducibility s.d.:</b>	0,007 mg/tube
<b>Method:</b>	ISO 5725-2	<b>Relative reproducibility s.d.:</b>	7,81%
<b>No. of laboratories:</b>	26	<b>Reference value:</b>	0,091 mg/tube
<b>Relative target s.d.:</b>	10,00% (Limited)	<b>Range of tolerance:</b>	0,068 - 0,102 mg/tube ( $ Z\text{-Score}  \leq 2,00$ )





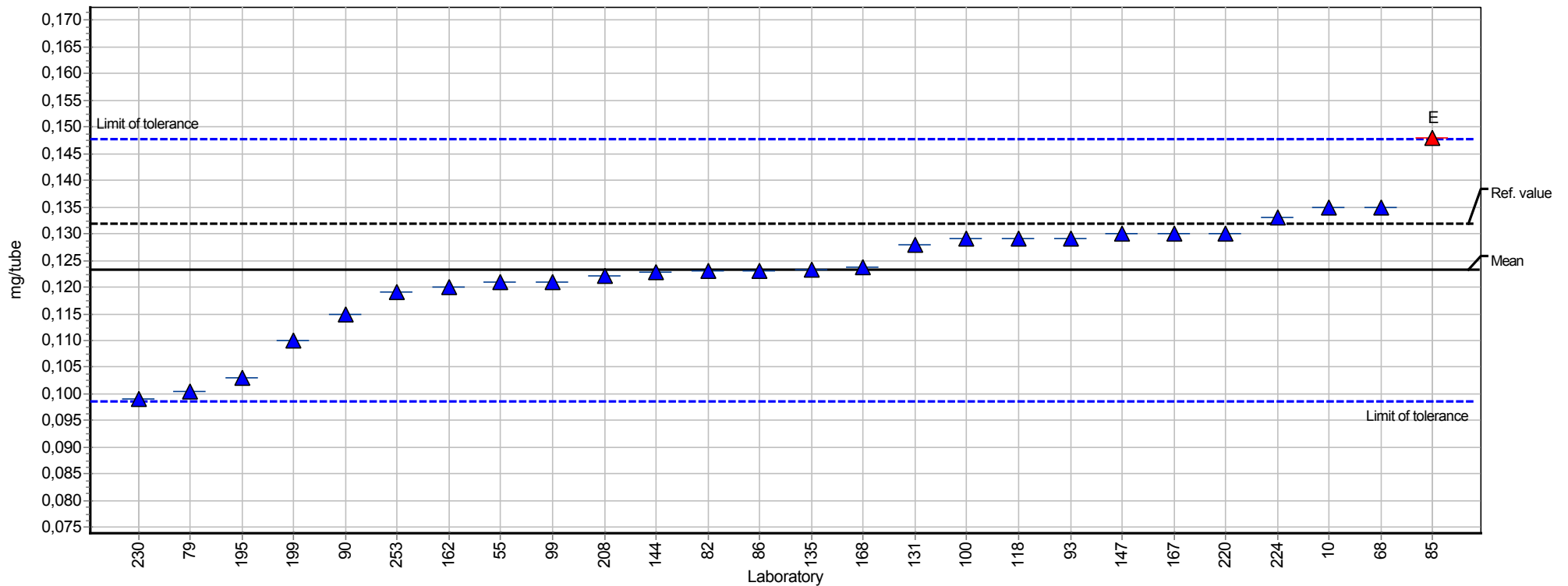
## Summary results

<b>Measurand:</b>	Toluene	<b>Mean:</b>	0,348 mg/tube
<b>Sample:</b>	1	<b>Reproducibility s.d.:</b>	0,022 mg/tube
<b>Method:</b>	ISO 5725-2	<b>Relative reproducibility s.d.:</b>	6,18%
<b>No. of laboratories:</b>	25	<b>Reference value:</b>	0,367 mg/tube
<b>Relative target s.d.:</b>	10,00% (Limited)	<b>Range of tolerance:</b>	0,278 - 0,417 mg/tube ( $ Z\text{-Score}  \leq 2,00$ )



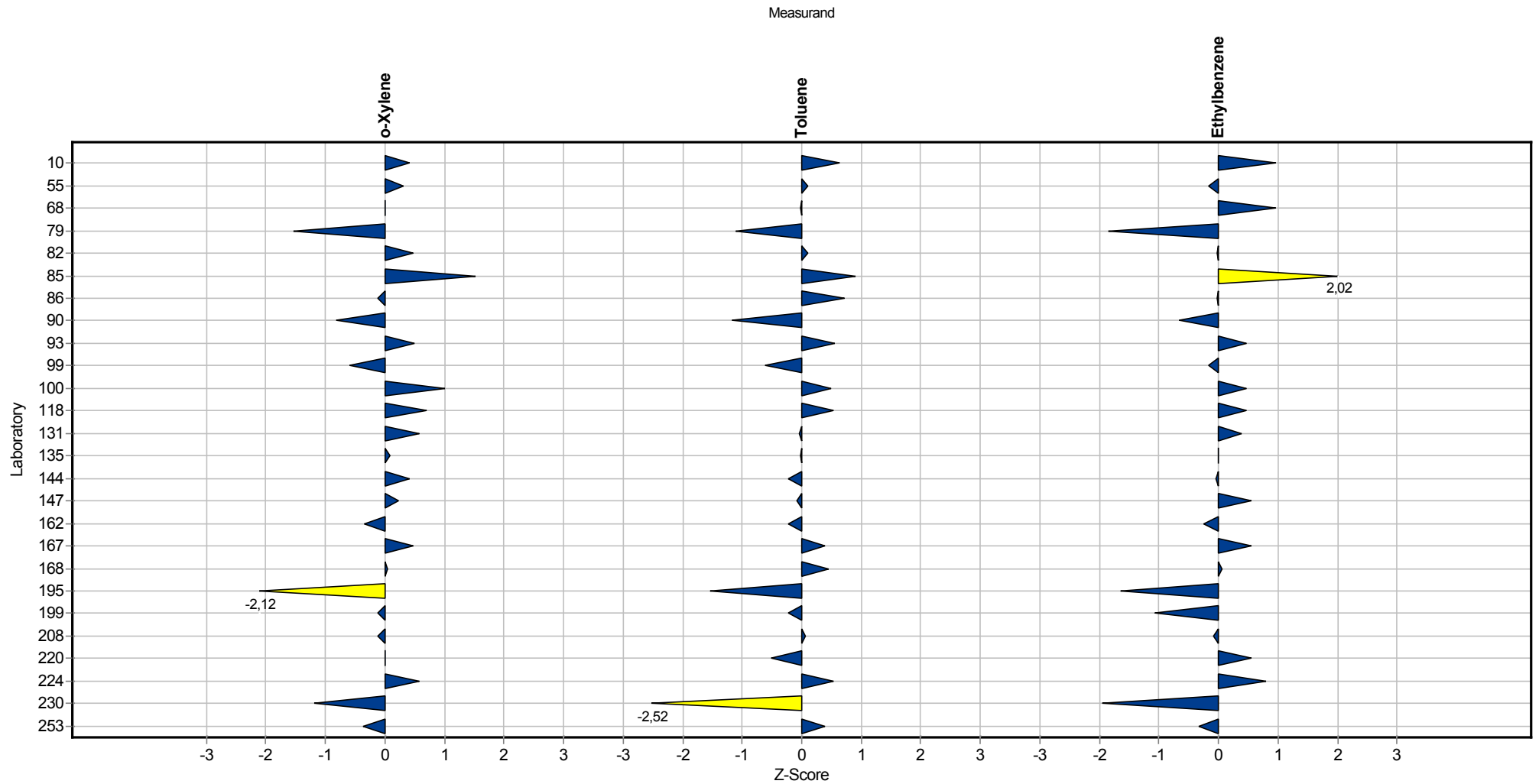
## Summary results

<b>Measurand:</b>	Ethylbenzene	<b>Mean:</b>	0,123 mg/tube
<b>Sample:</b>	1	<b>Reproducibility s.d.:</b>	0,011 mg/tube
<b>Method:</b>	ISO 5725-2	<b>Relative reproducibility s.d.:</b>	8,96%
<b>No. of laboratories:</b>	26	<b>Reference value:</b>	0,132 mg/tube
<b>Relative target s.d.:</b>	10,00% (Limited)	<b>Range of tolerance:</b>	0,099 - 0,148 mg/tube ( $ Z\text{-Score}  \leq 2,00$ )



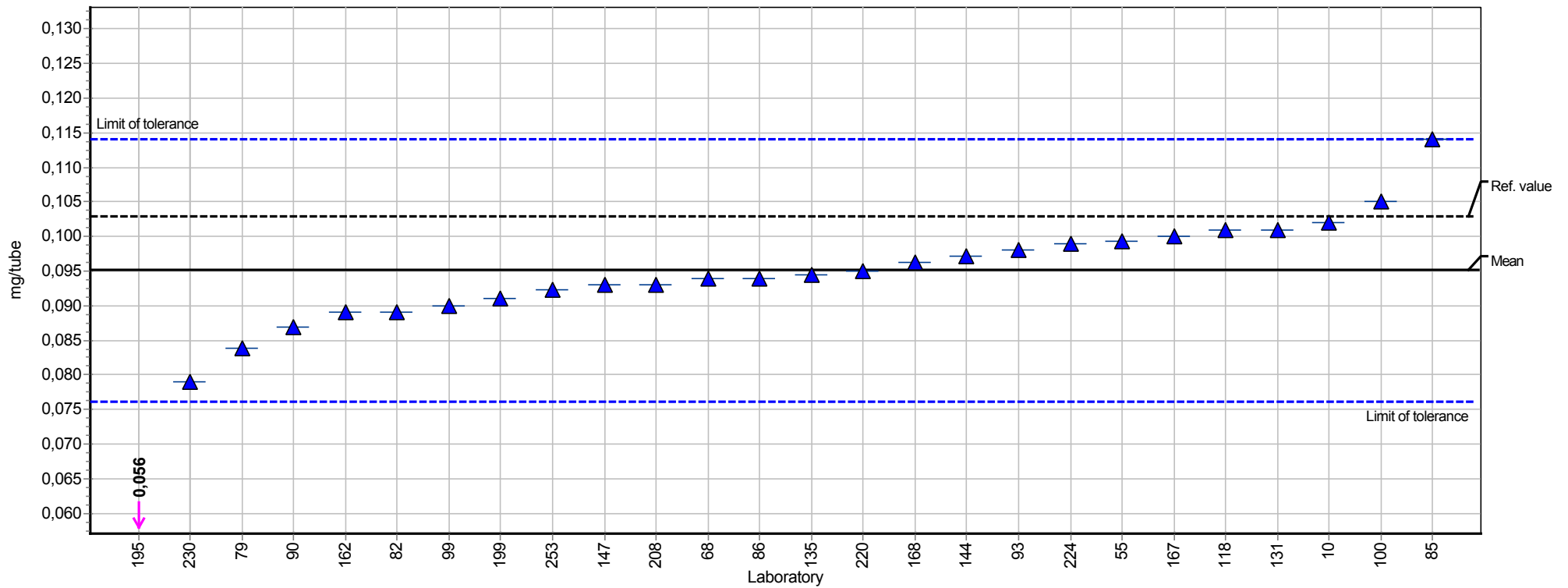
# Sample chart of Z-scores

Sample 1



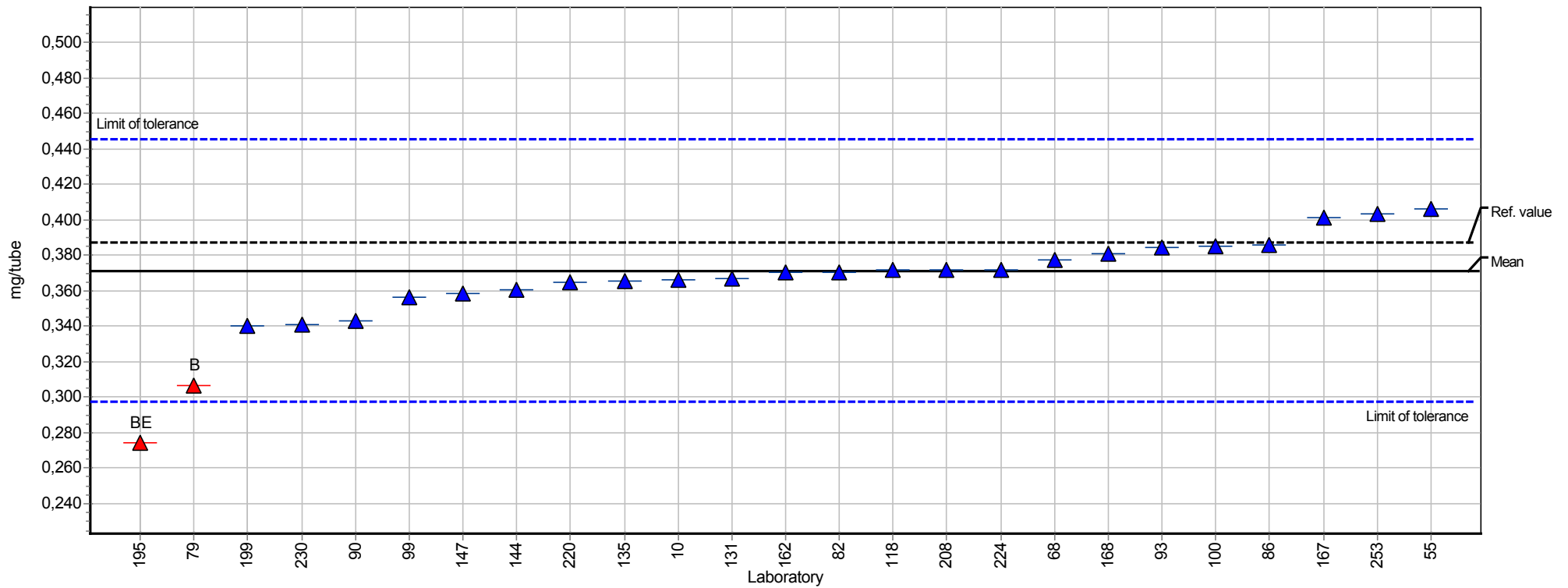
## Summary results

Measurand:	o-Xylene	Mean:	0,095 mg/tube
Sample:	2	Reproducibility s.d.:	0,007 mg/tube
Method:	ISO 5725-2	Relative reproducibility s.d.:	7,53%
No. of laboratories:	25	Reference value:	0,103 mg/tube
Relative target s.d.:	10,00% (Limited)	Range of tolerance:	0,076 - 0,114 mg/tube ( $ Z\text{-Score}  \leq 2,00$ )



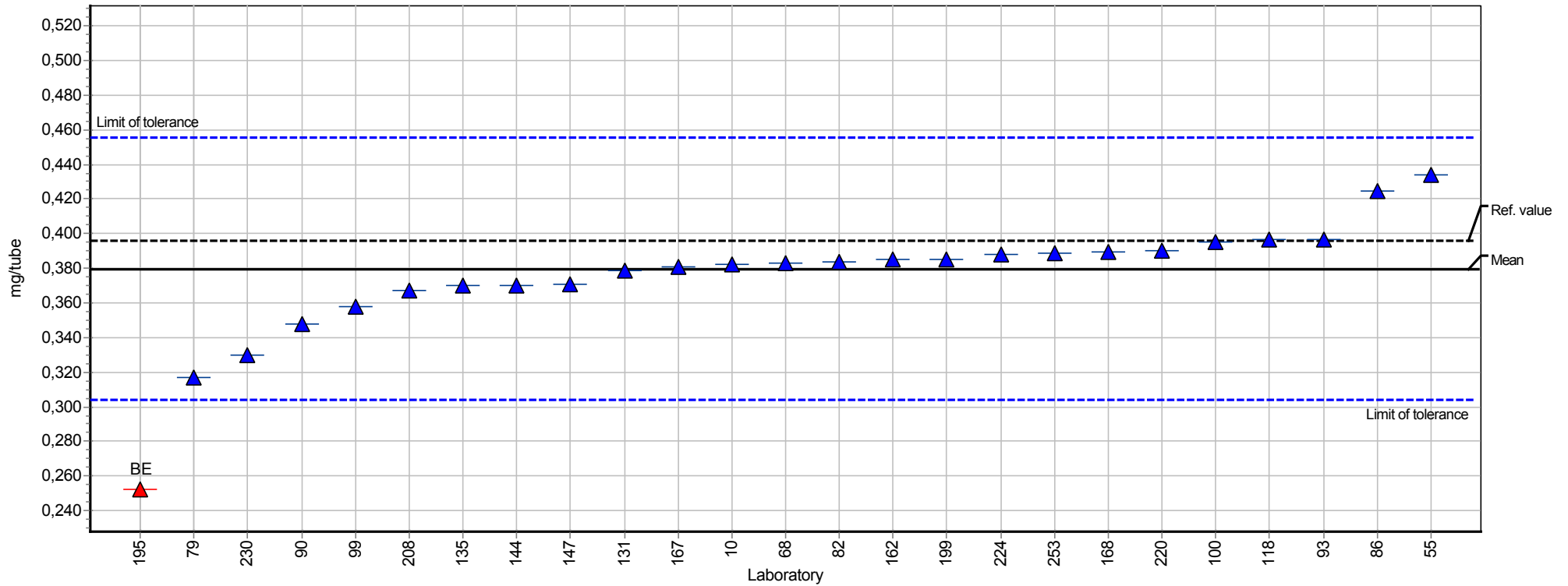
## Summary results

Measurand:	n-Heptane	Mean:	0,371 mg/tube
Sample:	2	Reproducibility s.d.:	0,018 mg/tube
Method:	ISO 5725-2	Relative reproducibility s.d.:	4,85%
No. of laboratories:	23	Reference value:	0,387 mg/tube
Relative target s.d.:	10,00% (Limited)	Range of tolerance:	0,297 - 0,446 mg/tube ( $ Z\text{-Score}  \leq 2,00$ )



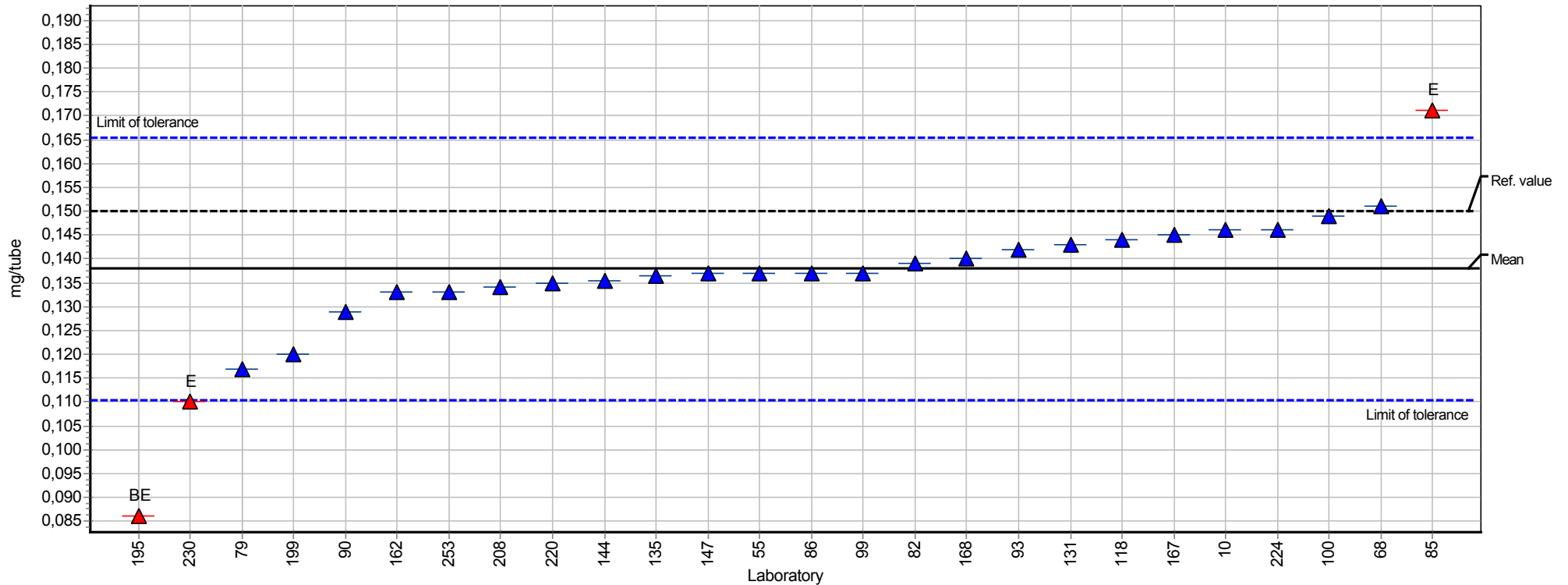
## Summary results

Measurand:	n-Octane	Mean:	0,380 mg/tube
Sample:	2	Reproducibility s.d.:	0,025 mg/tube
Method:	ISO 5725-2	Relative reproducibility s.d.:	6,63%
No. of laboratories:	24	Reference value:	0,396 mg/tube
Relative target s.d.:	10,00% (Limited)	Range of tolerance:	0,304 - 0,456 mg/tube ( $ Z\text{-Score}  \leq 2,00$ )



## Summary results

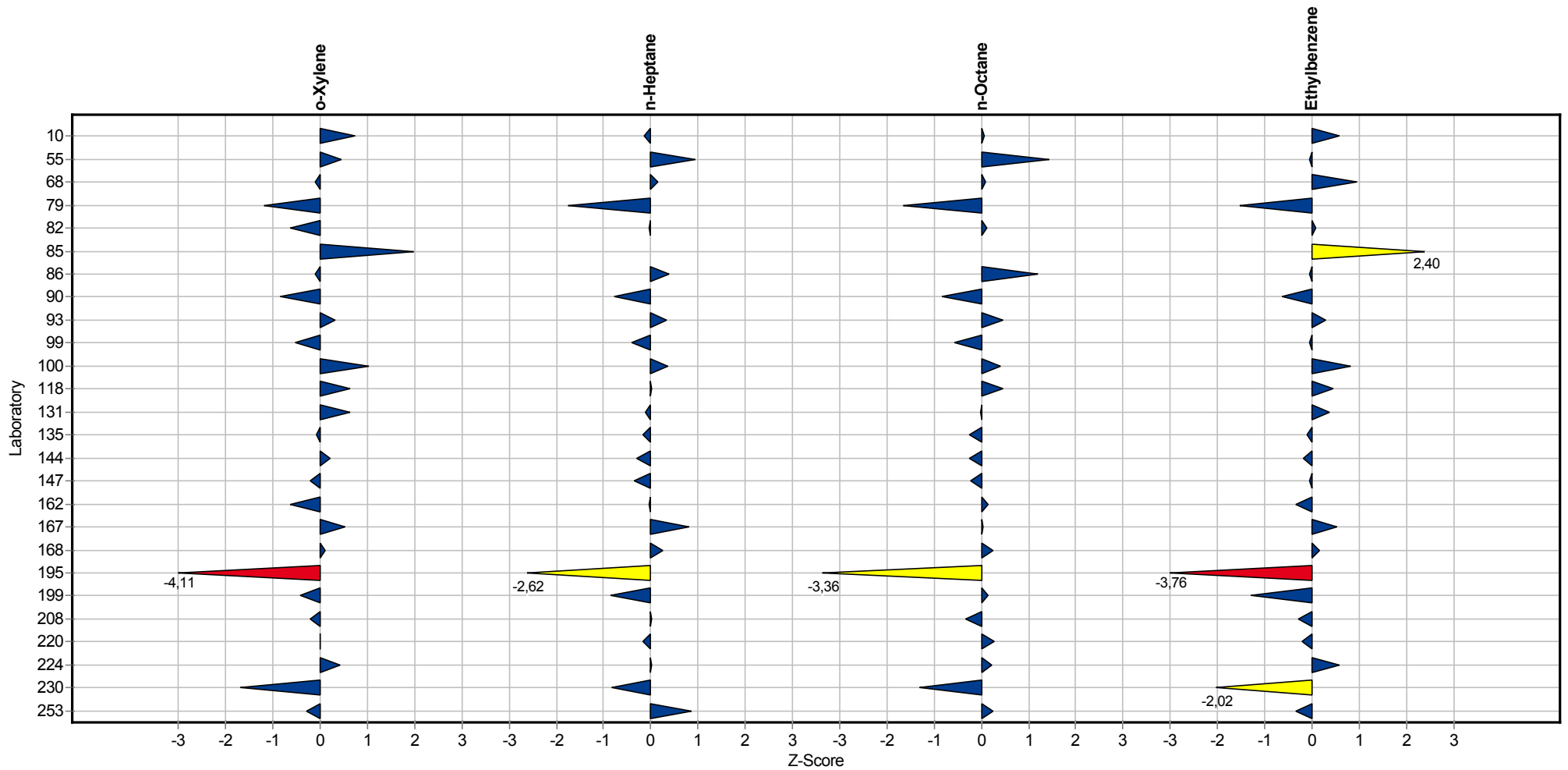
<b>Measurand:</b>	Ethylbenzene	<b>Mean:</b>	0,138 mg/tube
<b>Sample:</b>	2	<b>Reproducibility s.d.:</b>	0,012 mg/tube
<b>Method:</b>	ISO 5725-2	<b>Relative reproducibility s.d.:</b>	8,59%
<b>No. of laboratories:</b>	25	<b>Reference value:</b>	0,150 mg/tube
<b>Relative target s.d.:</b>	10,00% (Limited)	<b>Range of tolerance:</b>	0,110 - 0,165 mg/tube ( $ Z\text{-Score}  \leq 2,00$ )



# Sample chart of Z-scores

Sample 2

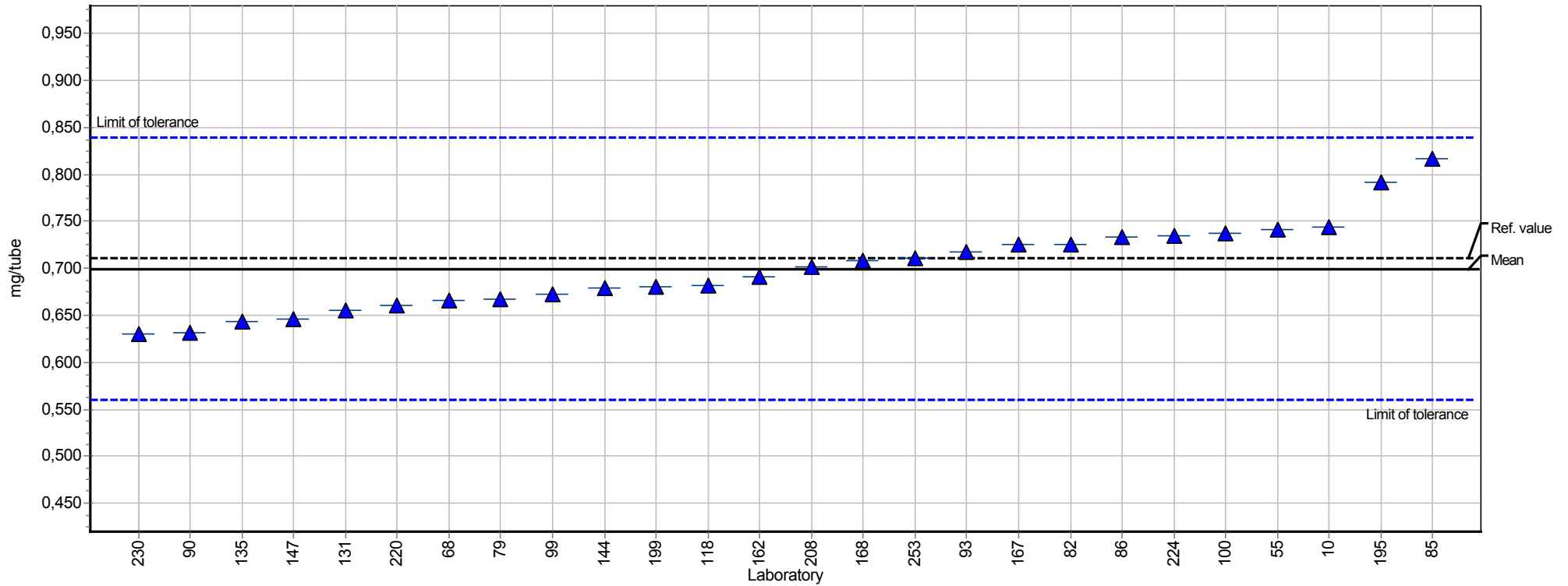
Measurand





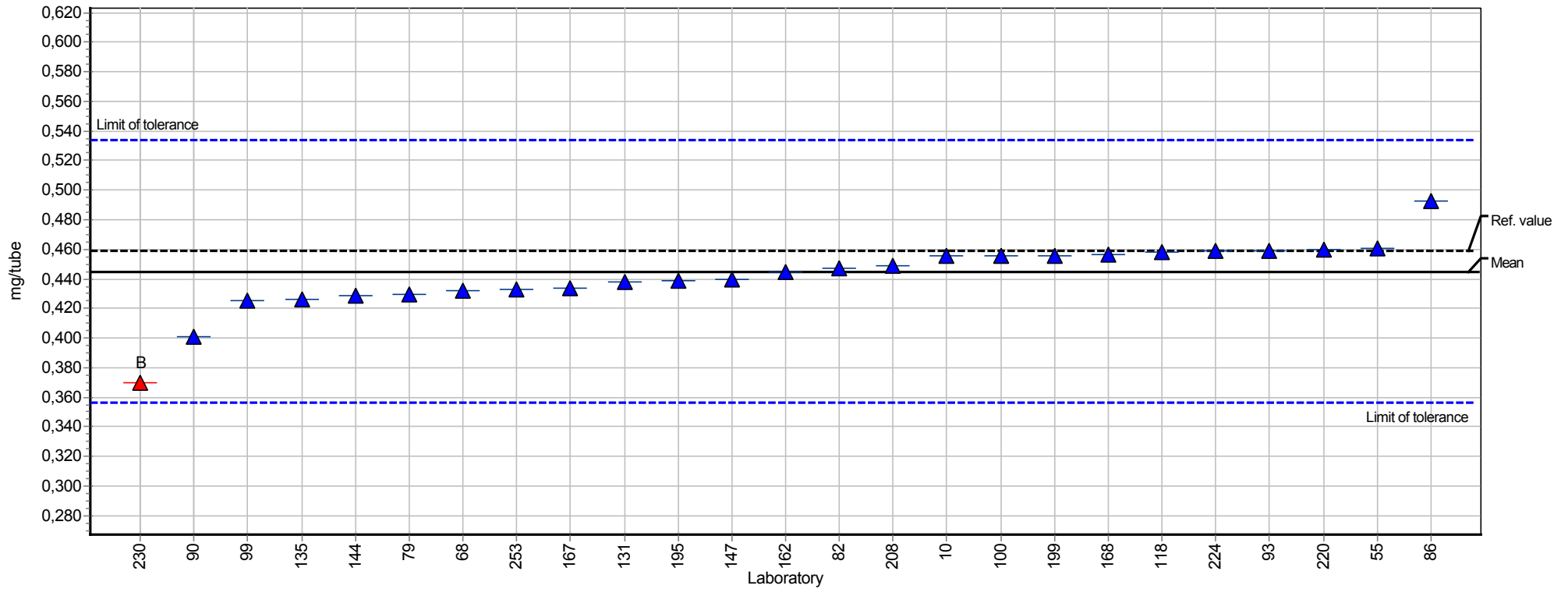
## Summary results

Measurand:	n-Hexane	Mean:	0,700 mg/tube
Sample:	3	Reproducibility s.d.:	0,047 mg/tube
Method:	ISO 5725-2	Relative reproducibility s.d.:	6,70%
No. of laboratories:	26	Reference value:	0,711 mg/tube
Relative target s.d.:	10,00% (Limited)	Range of tolerance:	0,560 - 0,840 mg/tube ( $ Z\text{-Score}  \leq 2,00$ )



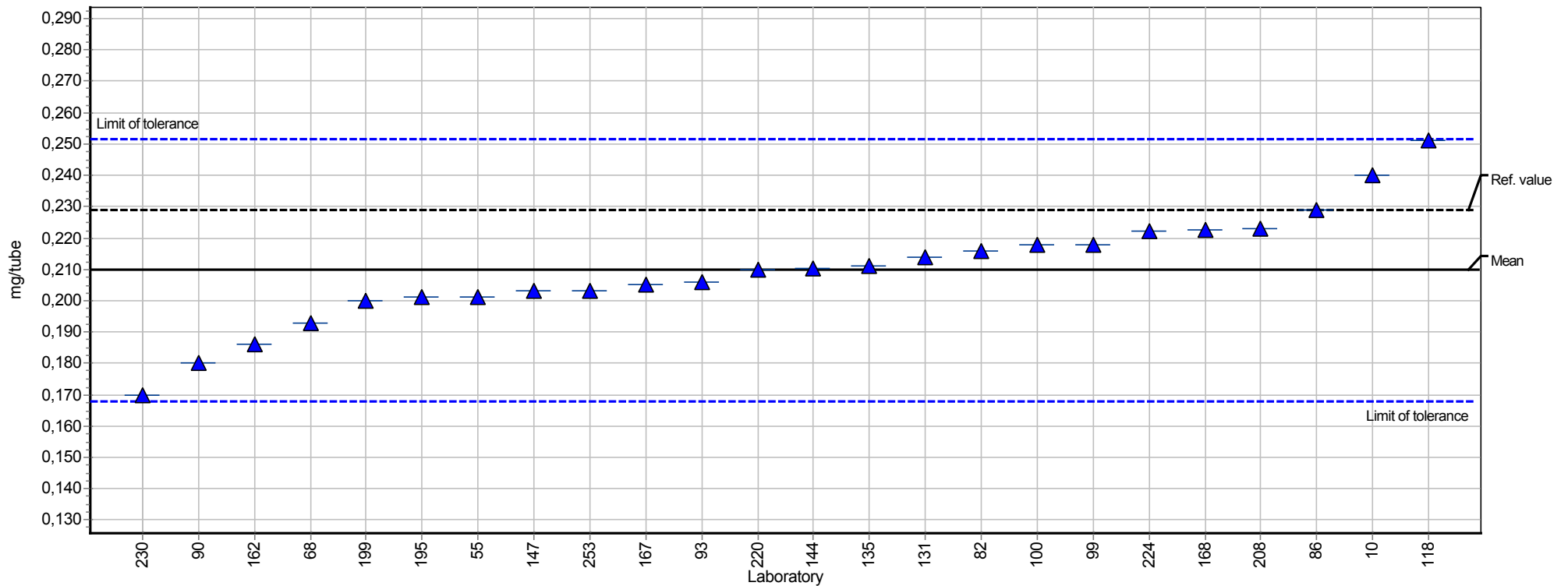
## Summary results

<b>Measurand:</b>	n-Octane	<b>Mean:</b>	0,445 mg/tube
<b>Sample:</b>	3	<b>Reproducibility s.d.:</b>	0,018 mg/tube
<b>Method:</b>	ISO 5725-2	<b>Relative reproducibility s.d.:</b>	4,09%
<b>No. of laboratories:</b>	24	<b>Reference value:</b>	0,459 mg/tube
<b>Relative target s.d.:</b>	10,00% (Limited)	<b>Range of tolerance:</b>	0,356 - 0,534 mg/tube ( $ Z\text{-Score}  \leq 2,00$ )



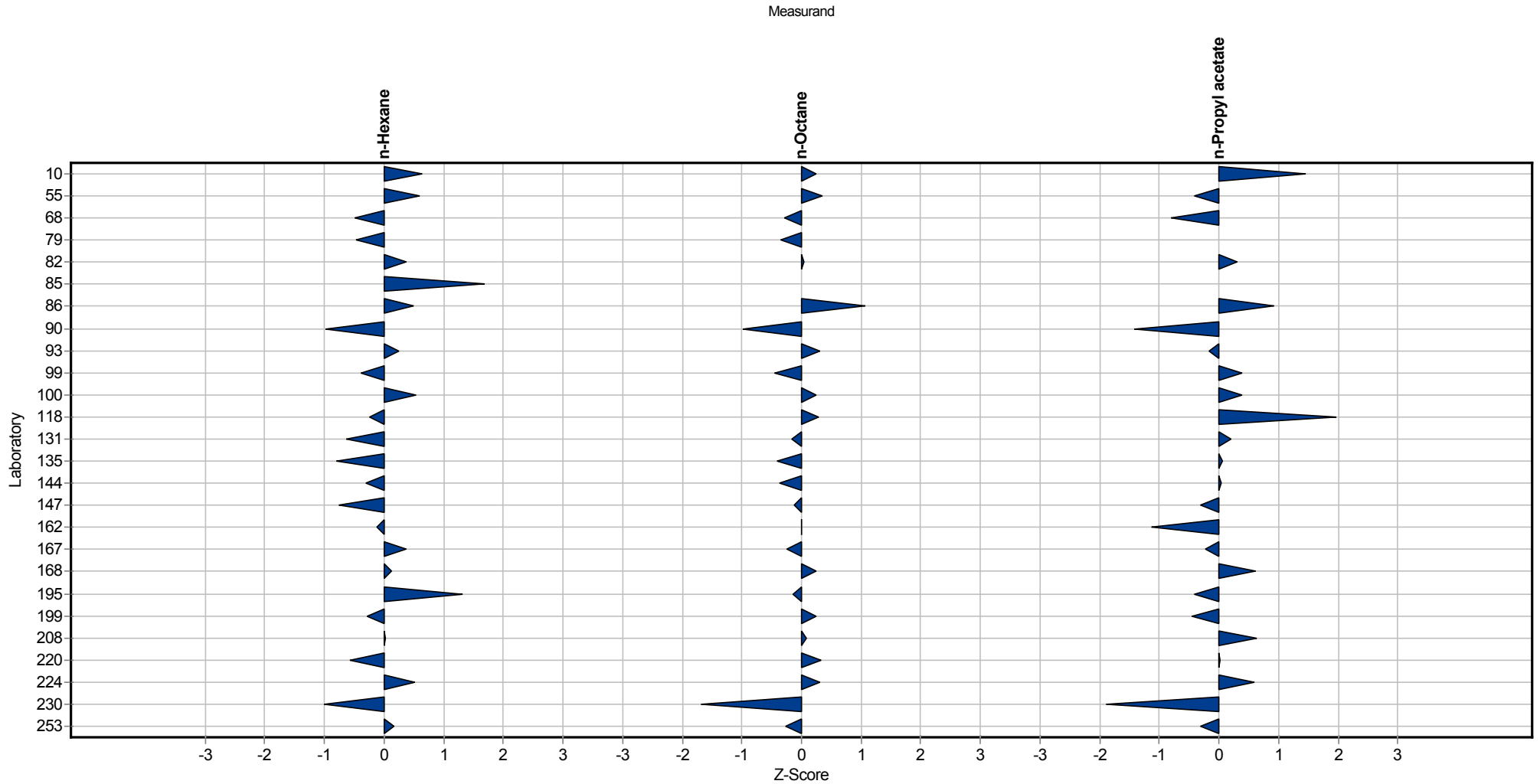
## Summary results

<b>Measurand:</b>	n-Propyl acetate	<b>Mean:</b>	0,210 mg/tube
<b>Sample:</b>	3	<b>Reproducibility s.d.:</b>	0,018 mg/tube
<b>Method:</b>	ISO 5725-2	<b>Relative reproducibility s.d.:</b>	8,53%
<b>No. of laboratories:</b>	24	<b>Reference value:</b>	0,229 mg/tube
<b>Relative target s.d.:</b>	10,00% (Limited)	<b>Range of tolerance:</b>	0,168 - 0,252 mg/tube ( $ Z\text{-Score}  \leq 2,00$ )



# Sample chart of Z-scores

Sample 3



## Questions and Answers

Participant	Analytical method	Desorption solution	Volume of desorption solution	Carrier gas	Injection
10	NFX43-267	carbone disulfide	1 milliliter	helium	split
55	GC-MS (MIM-GA-012)	CS2	2 ml	helium	split
68	Weder DFG noch IFA-Arbeitsmappe	CS2	1 ml	Helium	split
79	BIA 6265	CS2	0,5 ml	Wasserstoff	split
82	Hausmethode, angelehnt an DFG, 3	CS2 / Isopropanol 80:20	1,3 mL	Stickstoff	split
85	interne Standard-Methode	CS2	1,0 mL	Stickstoff	splitless
86	Internal method : Newmethsil	CS2	2 ml	Helium	Split à 80%
90	INRS METROPOL M-188 (ex METROPOL 055)	CS2	2 ml	helium	split
93	INRS Metropol Method	Disulfure de carbone	2 ml	Hydrogène	1/20
99	METROPOL 12 and 21	CS2	5 ML	HELIUM	SPLITLESS
100	Metropol012,021, niosh1500,1501,1450, ...	carbon disulfide	2ml	helium	split
118	in Anlehnung an die VDI Blatt 2	CS2	2mL	Stickstoff	on-column
131	-	CS2	4 ml	He 1.4 ml/min	split 1/20
135	Hausmethode	Schwefelkohlenstoff/Phenoxyethanol (98%/2%)	10	He	Split
144	INRS - Methods METROPOL n° 21 & 55	CS2	1,5 ml	Helium	split
162	Hausmethode	CS2	1 ml	Wasserstoff	split
167	Capillary Gas Chromatography	Carbon disulphide	1,5 mL	Helium	1,0 µL splitless, 250 °C
168	Metropol M-41	CS2	5 ml	He	split
195	Internal	Carbon disulphide + n-propylbenzene as internal standard	1 mL	He	split
199	Hausmethode in Anlehnung an NIOSH-Methode	DCM/CS2/MeOH	5 mL	Helium	splitless
208	Own, based on NIOSH and OSHA methods	2% DMF in carbon disulfide	1,5 ml	Helium	Split
220	Hausmethode, in Anlehnung an VDI 2100-Blatt 2	Benzylalkohol	5 mL	Helium	split
224	inhouse method	carbon disulfide	2 ml	He	split
230	VDI 2100, Blatt 2	Schwefelkohlenstoff	2 ml	Wasserstoff	2 µl, split

Participant	Analytical column	Detector	Data evaluation
10	polar and apolar	FID	external
55	RTX 502.2	MS	internal standard
68	Vocol von Supelco	FID	interner Standard

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Participant	Analytical column	Detector	Data evaluation
79	CP Sil 5CB	FID	interner Standard
82	Agilent HP5, 30m x 0,32mm, 0,25 µm	FID	interner Standard
85	DB-5 / DB-WAX	FID	interner Standard
86	TG5MS 60m*0.25mm - 0.25µm	FID	Internal standard
90	DB5MS 50m ID=0.2mm Film=0.33µm	FID	internal standard
93	Agilent HP5	FID	External standard
99	ZB1/ 30M/ 0.32 mm /5 µm	FID	internal standard
100	DB 624	MS	internal
118	CP Sil 5 CB / CB-Wax 57 CB	FID	interner Standard
131	Equity-1 (100m)	FID	internal standard (n C9)
135	Restek RXi-1 ms	MSD	interne Standards mit externer Kaibrierung
144	Agilent DB5 10m x 0.18mm x 0.18µ	FID	External
162	Agilent CP-Sil PONA; 50m x 0.21 mm ID x 0.50 µm Film	FID	interner Standard
167	Agilent Technologies, Inc., DB-5MS + DG, 30 m, 0,25 mm id, 0,25 µm film thickness	FID, 310 248 °C	Internal standard, chloro benzene
168	ZB-5HT	FID	external standard
195	elite-5 60m x 0.25 mm i.d. x 1.5 um	FID	Internal standard
199	DB-5.625MS	Massendetektor	Externe Standards mit Korrektur über interne Standards
208	Agilnet HP-5, HP-InnoWax	FID	External standard
220	DB-5.625 MS	GC/MS	externe Standards mit Korrektur über interne Standards
224	BPX5 60m x 0.25 mm x 1.0 µm	MS	internal standard
230	Restek Rxi -624 sil MS 30 m, 0,25 mm ID, 1,4 µm df	FID	externer Standard

Participant	Recovery rate	Date of analysis
10	no	23/02/2016
55	yes	09 and 10/03/2016
68	Nein	7./8.3.2016
79		12. KW 2016
82	nein	23.02.2016
85	ja	08.03.2016
86	no	01/03/2016
90	no	26/02/2016
93	no	2/03/2016

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Participant	Recovery rate	Date of analysis
99	no	02/03/2016
100	include	3/4/2016
118		10.03.2016
131	no	14/03/16
135	ja	01.03.2016
144	No but the calibration standards are made with charcoal.	08/03/2016
162	ja	25.02.2016
167	Yes	03.03.2016
168	Yes	09/03/2016
195	yes	15/03/2016
199	nein	04.03.2016
208		22.2.2016
220	Nein	09.03.2016
224	yes	29/2/2016
230	ja	27/29.02.2016