

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

**Institut für Arbeitsschutz der Deutschen Gesetzlichen Unfallversicherung (IFA)  
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## ***Results and evaluation***

Organic solvents 2014

## Summary of laboratory means

Sample 1

	n-Heptane	Z score	Ethylbenzene	Z score	n-Propyl acetate	Z score
Unit	mg/tube		mg/tube		mg/tube	
10	0,882	0,53	0,086	-1,03	0,459	0,33
28	0,858	0,24	0,074	-2,30 E	0,534	2,00 E
31	0,805	-0,39	0,101	0,48	0,444	-0,01
55	0,848	0,12	0,099	0,27	0,441	-0,08
68	0,778	-0,71	0,102	0,58	0,429	-0,35
73	0,750	-1,05	0,110	1,41	0,460	0,35
79	0,807	-0,36	0,109	1,32		
82	0,414	-5,06 BE	0,048	-5,02 BE	0,325	-2,69 E
85			0,110	1,41		
162	0,790	-0,57	0,092	-0,46	0,392	-1,18
164			0,090	-0,62		
167	0,902	0,77	0,094	-0,25	0,473	0,64
208	0,776	-0,74	0,096	-0,04	0,451	0,15
223	0,787	-0,60	0,093	-0,35	0,410	-0,78
224	0,907	0,83	0,088	-0,87	0,434	-0,24
253	0,788	-0,59	0,096	-0,04	0,419	-0,57
256	0,828	-0,11	0,100	0,37	0,420	-0,55
257	0,948	1,32	0,115	1,93	0,577	2,98 E
260	0,948	1,32	0,079	-1,80		
-	-	--	-	--	-	--
Method	ISO 5725-2		ISO 5725-2		ISO 5725-2	
Assessment	Z <=2,00		Z <=2,00		Z <=2,00	
Mean	0,838		0,096		0,445	
Reproducibility s.d.	0,063		0,011		0,058	

	n-Heptane	Z score	Ethylbenzene	Z score	n-Propyl acetate	Z score
Rel. reproducibility s.d.	7,56 %		11,21 %		12,98 %	
Reference value	0,759		0,096		0,438	
Target s.d.	0,084		0,010		0,044	
Rel. target s.d.:	10,00 %		10,00 %		10,00 %	
Type B outliers	1		1		0	
Type F outliers	0		0		0	
No. of laboratories that submitted results	17		19		15	
No. of laboratories after elimination of outliers type A-D and F (w ithout laboratories that only gave states but no measured values)	16		18		15	
Explanation of outlier types						
A: Single outlier	Grubbs					
B: Differing laboratory mean	Grubbs					
C: Excessive laboratory s.d.	Cochran					
D: Excluded manually						
E: score outside tolerance limits						
F:  Score >3,5						

## Summary of laboratory means

Sample 2

Unit	Ethyl acetate		n-Octane		n-Propyl acetate		cyclohexane	
	mg/tube	Z score	mg/tube	Z score	mg/tube	Z score	mg/tube	Z score
10	0,498	-0,05	0,414	-0,21	0,650	0,63	0,304	-0,30
28	0,502	0,03	0,434	0,27	0,715	1,69	0,306	-0,23
31	0,451	-0,99	0,423	0,00	0,589	-0,37	0,295	-0,59
55	0,503	0,05	0,428	0,12	0,596	-0,25	0,315	0,05
68	0,441	-1,19	0,422	-0,02	0,557	-0,89	0,303	-0,33
73	0,520	0,39	0,400	-0,54	0,610	-0,02	0,310	-0,11
79			0,503	1,89 B			0,361	1,50
82	0,479	-0,43	0,426	0,07	0,603	-0,14	0,290	-0,75
85							0,300	-0,43
162	0,415	-1,70	0,430	0,17	0,529	-1,35	0,293	-0,65
167	0,518	0,35	0,389	-0,80	0,634	0,37	0,301	-0,40
208	0,472	-0,57	0,415	-0,19	0,594	-0,29	0,299	-0,46
223	0,444	-1,13	0,414	-0,21	0,555	-0,92	0,303	-0,33
224	0,527	0,53	0,463	0,95	0,592	-0,32	0,333	0,62
253	0,513	0,25	0,425	0,05	0,617	0,09	0,319	0,18
256	0,567	1,33	0,438	0,36	0,580	-0,52	0,333	0,62
257	0,540	0,79			0,752	2,30 E	0,364	1,61
260	0,615	2,29 E						
-	-	--	-	--	-	--	-	--
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	0,500		0,423		0,612		0,313	
Reproducibility s.d.	0,051		0,017		0,058		0,022	
Rel. reproducibility s.d.	10,12 %		4,12 %		9,56 %		7,05 %	

	Ethyl acetate	Z score	n-Octane	Z score	n-Propyl acetate	Z score	cyclohexane	Z score
Reference value	0,462		0,416		0,574		0,293	
Target s.d.	0,050		0,042		0,061		0,031	
Rel. target s.d.:	10,00 %		10,00 %		10,00 %		10,00 %	
Type B outliers	0		1		0		0	
Type F outliers	0		0		0		0	
No. of laboratories that submitted results	16		15		15		17	
No. of laboratories after elimination of outliers type A-D and F (w ithout laboratories that only gave states but no measured values)	16		14		15		17	
Explanation of outlier types								
A: Single outlier	Grubbs							
B: Differing laboratory mean	Grubbs							
C: Excessive laboratory s.d.	Cochran							
D: Excluded manually								
E: score outside tolerance limits								
F:  Score >3,5								

## Summary of laboratory means

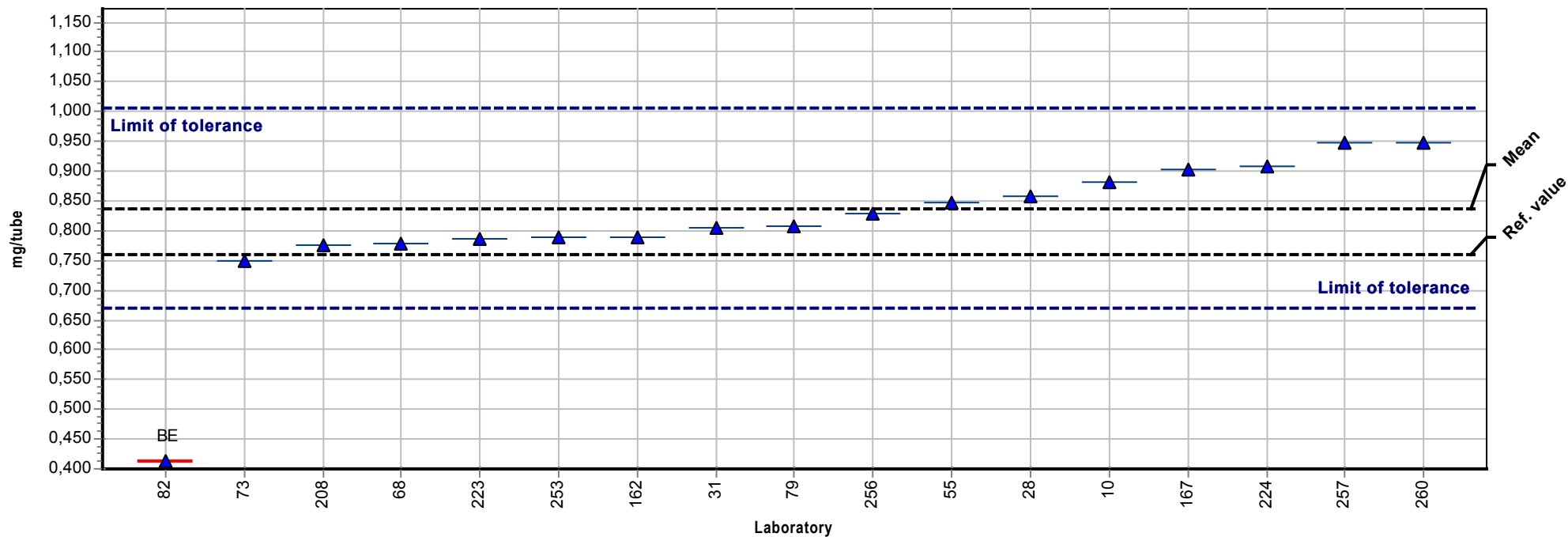
Sample 3

Unit	o-Xylene		Toluene		Ethylbenzene	
	mg/tube	Z score	mg/tube	Z score	mg/tube	Z score
8	0,119	2,41 E	0,908	1,38	0,181	1,98
10	0,085	-1,19	0,826	0,35	0,134	-1,13
28	0,071	-2,63 E	0,846	0,60	0,122	-1,94
31	0,106	1,05	0,781	-0,21	0,164	0,86
55	0,099	0,33	0,824	0,32	0,152	0,06
68	0,094	-0,20	0,763	-0,44	0,155	0,26
73	0,100	0,43	0,730	-0,85	0,150	-0,07
79	0,123	2,81 E	0,981	2,29 E	0,188	2,47 E
82	0,097	0,11	0,787	-0,14	0,156	0,33
85	0,110	1,47	0,850	0,65	0,170	1,26
162	0,088	-0,83	0,759	-0,49	0,143	-0,53
164	0,085	-1,12	0,677	-1,51	0,124	-1,81
167	0,090	-0,62	0,720	-0,98	0,140	-0,73
208	0,097	0,11	0,775	-0,29	0,147	-0,27
223	0,093	-0,30	0,751	-0,59	0,143	-0,53
224	0,083	-1,35	0,767	-0,39	0,136	-1,00
253	0,093	-0,30	0,807	0,11	0,147	-0,27
256	0,094	-0,20	0,830	0,40	0,156	0,33
257	0,106	1,05	0,850	0,65	0,176	1,65
260	0,086	-1,03	0,729	-0,87	0,137	-0,93
-	-	--	-	--	-	--
Method	ISO 5725-2		ISO 5725-2		ISO 5725-2	
Assessment	Z <=2,00		Z <=2,00		Z <=2,00	
Mean	0,096		0,798		0,151	

	<b>o-Xylene</b>	<b>Z score</b>	<b>Toluene</b>	<b>Z score</b>	<b>Ethylbenzene</b>	<b>Z score</b>
Reproducibility s.d.	0,012		0,070		0,018	
Rel. reproducibility s.d.	13,01 %		8,75 %		11,87 %	
Reference value	0,099		0,757		0,149	
Target s.d.	0,010		0,080		0,015	
Rel. target s.d.:	10,00 %		10,00 %		10,00 %	
Type B outliers	0		0		0	
Type F outliers	0		0		0	
No. of laboratories that submitted results	20		20		20	
No. of laboratories after elimination of outliers type A-D and F (w without laboratories that only gave states but no measured values)	20		20		20	
Explanation of outlier types						
A: Single outlier	Grubbs					
B: Differing laboratory mean	Grubbs					
C: Excessive laboratory s.d.	Cochran					
D: Excluded manually						
E: score outside tolerance limits						
F:  Score >3,5						

## Summary results

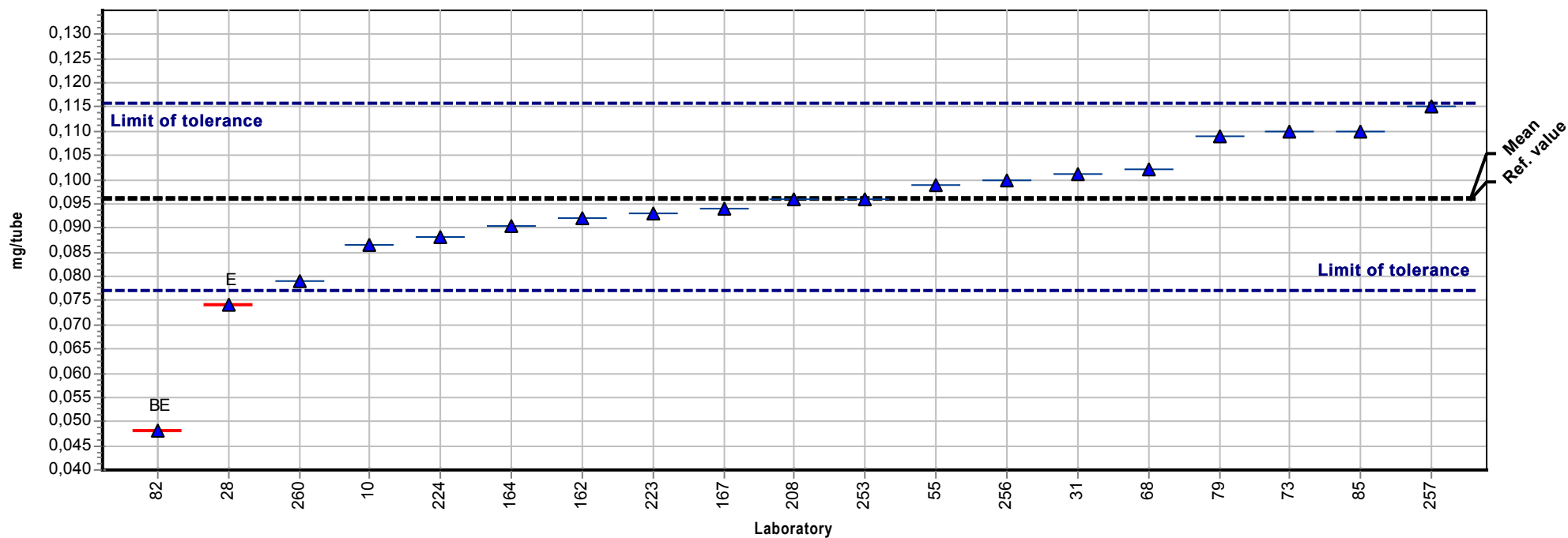
<b>Measurand:</b>	n-Heptane	<b>Mean:</b>	0,838 mg/tube
<b>Sample:</b>	1	<b>Reprod. s.d.:</b>	0,063 mg/tube
<b>Method:</b>	ISO 5725-2	<b>Rel. reprod. s.d.:</b>	7,56%
<b>Rel. target s.d.:</b>	10,00% (Limited)	<b>Reference value:</b>	0,759 mg/tube
<b>No. of laboratories:</b>	16	<b>Range of tolerance:</b>	0,670 - 1,005 mg/tube ( $ Z\text{-Score}  \leq 2,00$ )





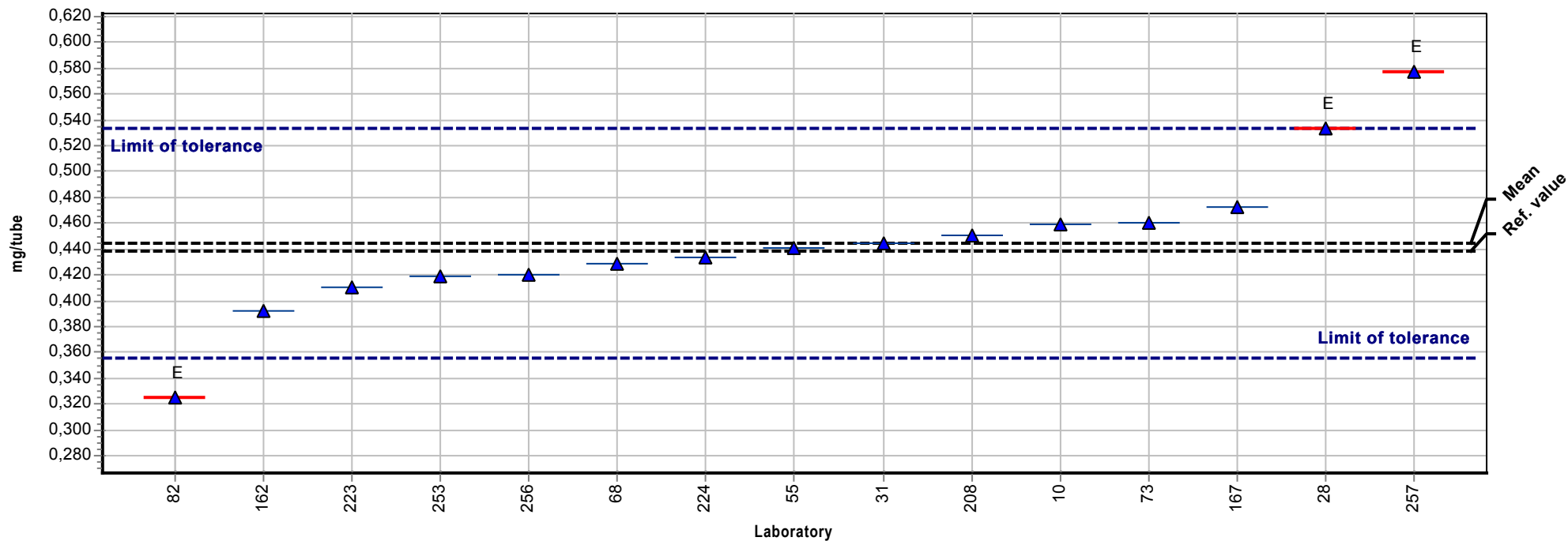
## Summary results

<b>Measurand:</b>	Ethylbenzene	<b>Mean:</b>	0,096 mg/tube
<b>Sample:</b>	1	<b>Reprod. s.d.:</b>	0,011 mg/tube
<b>Method:</b>	ISO 5725-2	<b>Rel. reprod. s.d.:</b>	11,21%
<b>Rel. target s.d.:</b>	10,00% (Limited)	<b>Reference value:</b>	0,096 mg/tube
<b>No. of laboratories:</b>	18	<b>Range of tolerance:</b>	0,077 - 0,116 mg/tube ( $ Z\text{-Score}  \leq 2,00$ )



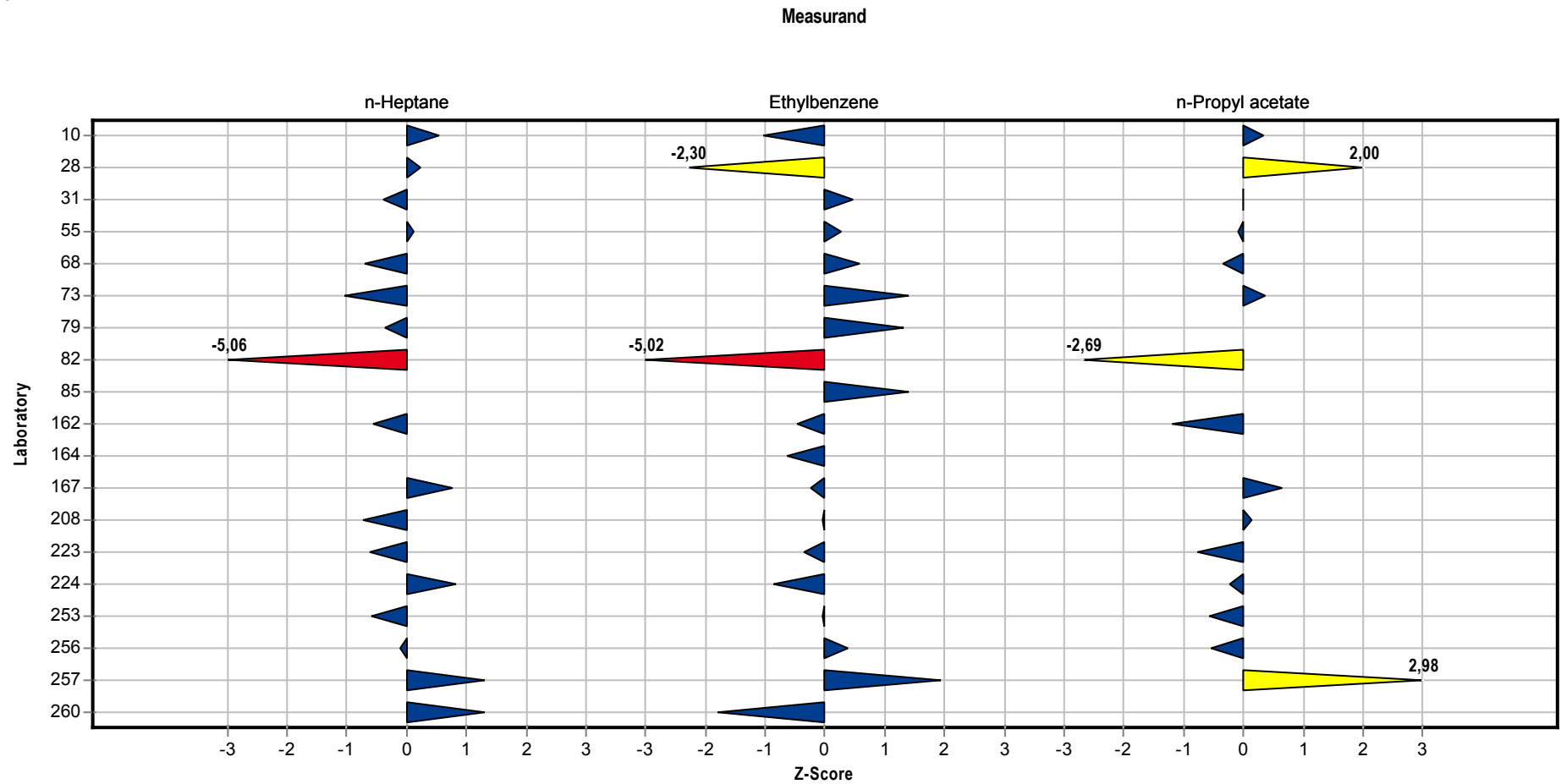
## Summary results

<b>Measurand:</b>	n-Propyl acetate	<b>Mean:</b>	0,445 mg/tube
<b>Sample:</b>	1	<b>Reprod. s.d.:</b>	0,058 mg/tube
<b>Method:</b>	ISO 5725-2	<b>Rel. reprod. s.d.:</b>	12,98%
<b>Rel. target s.d.:</b>	10,00% (Limited)	<b>Reference value:</b>	0,438 mg/tube
<b>No. of laboratories:</b>	15	<b>Range of tolerance:</b>	0,356 - 0,533 mg/tube ( $ Z\text{-Score}  \leq 2,00$ )



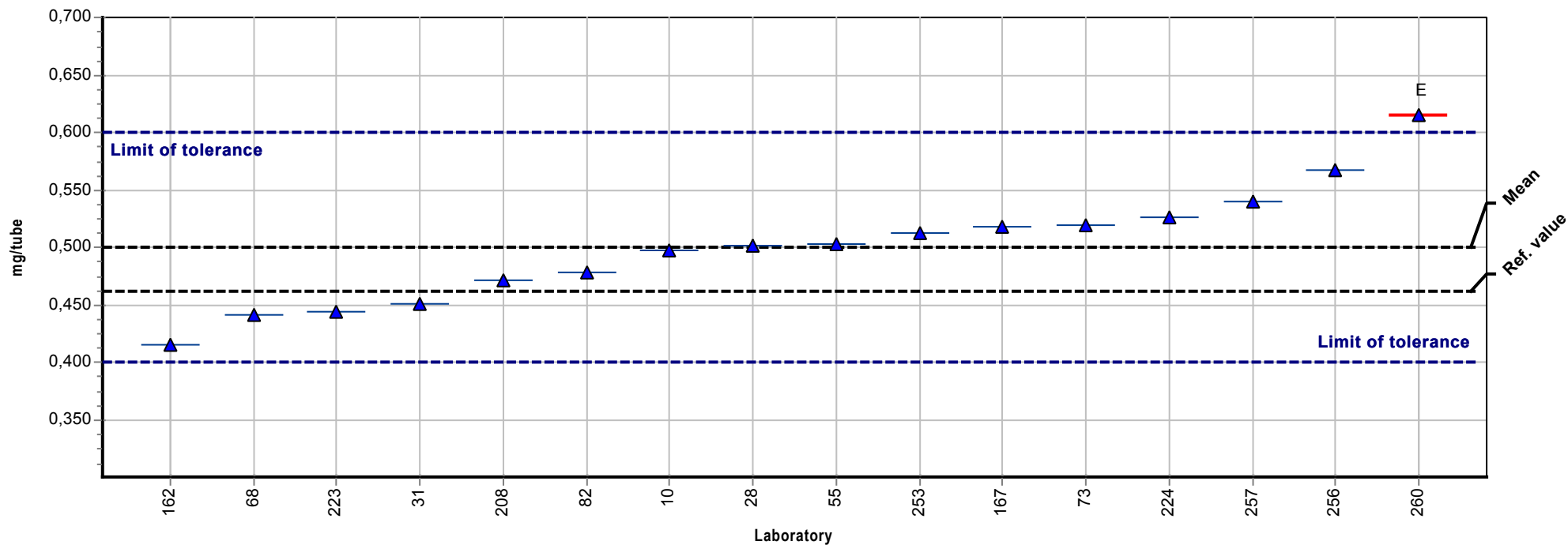
# Sample chart of Z-Scores

Sample: 1



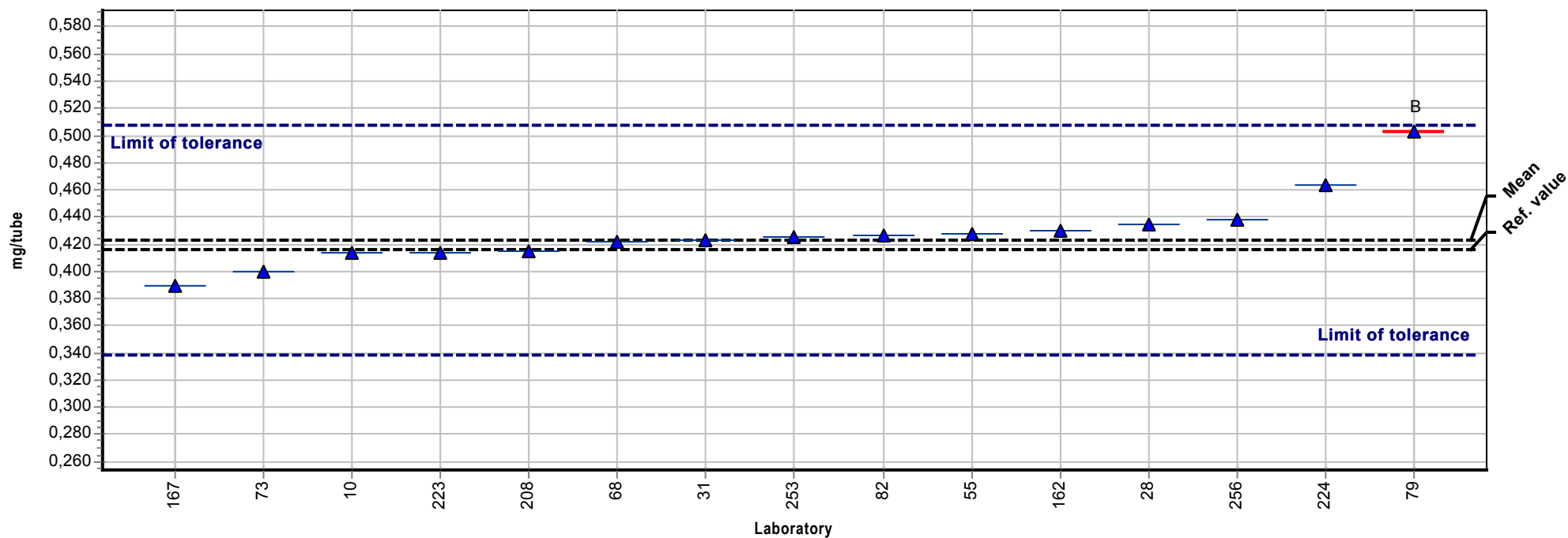
## Summary results

<b>Measurand:</b>	Ethyl acetate	<b>Mean:</b>	0,500 mg/tube
<b>Sample:</b>	2	<b>Reprod. s.d.:</b>	0,051 mg/tube
<b>Method:</b>	ISO 5725-2	<b>Rel. reprod. s.d.:</b>	10,12%
<b>Rel. target s.d.:</b>	10,00% (Limited)	<b>Reference value:</b>	0,462 mg/tube
<b>No. of laboratories:</b>	16	<b>Range of tolerance:</b>	0,400 - 0,600 mg/tube ( $ Z\text{-Score}  \leq 2,00$ )



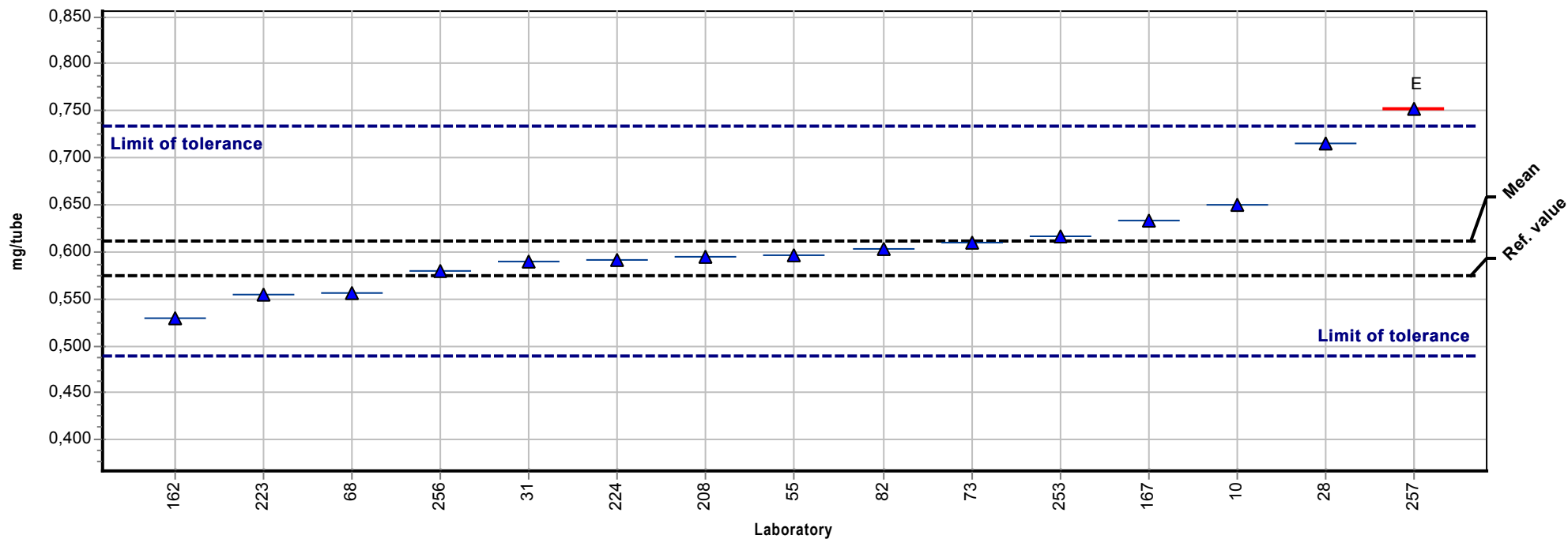
## Summary results

<b>Measurand:</b>	n-Octane	<b>Mean:</b>	0,423 mg/tube
<b>Sample:</b>	2	<b>Reprod. s.d.:</b>	0,017 mg/tube
<b>Method:</b>	ISO 5725-2	<b>Rel. reprod. s.d.:</b>	4,12%
<b>Rel. target s.d.:</b>	10,00% (Limited)	<b>Reference value:</b>	0,416 mg/tube
<b>No. of laboratories:</b>	14	<b>Range of tolerance:</b>	0,338 - 0,508 mg/tube ( $ Z\text{-Score}  \leq 2,00$ )



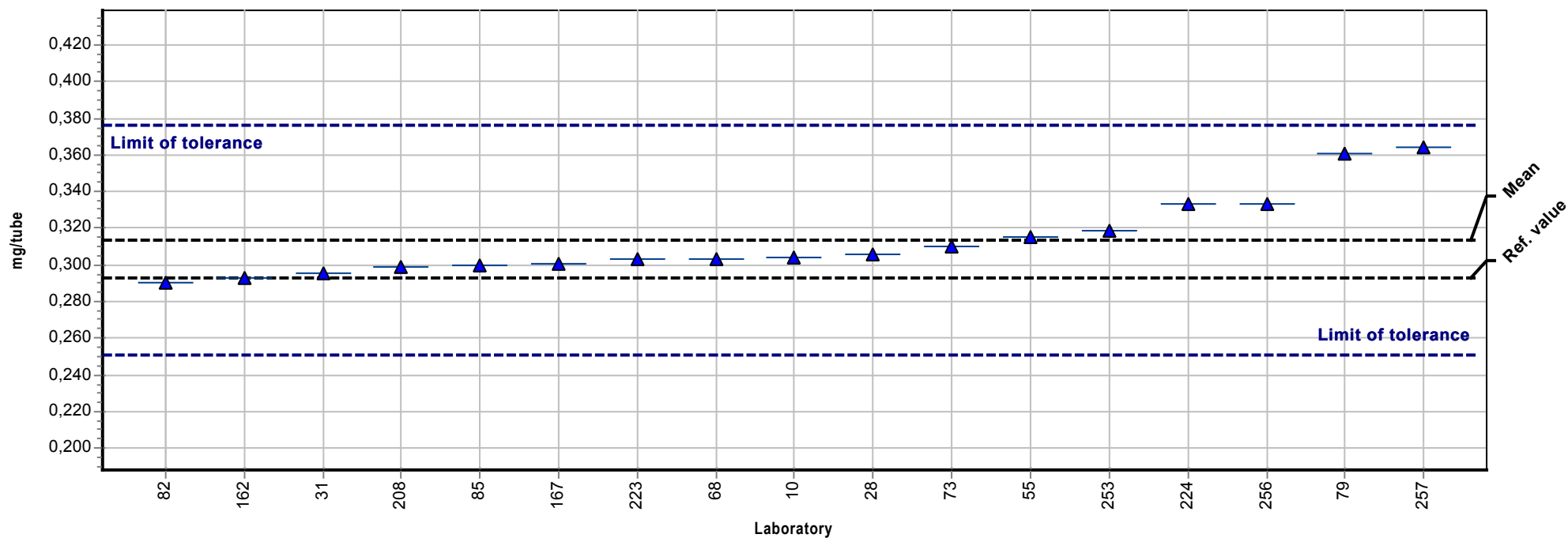
## Summary results

Measurand:	n-Propyl acetate	Mean:	0,612 mg/tube
Sample:	2	Reprod. s.d.:	0,058 mg/tube
Method:	ISO 5725-2	Rel. reprod. s.d.:	9,56%
Rel. target s.d.:	10,00% (Limited)	Reference value:	0,574 mg/tube
No. of laboratories:	15	Range of tolerance:	0,489 - 0,734 mg/tube ( $ Z\text{-Score}  \leq 2,00$ )



## Summary results

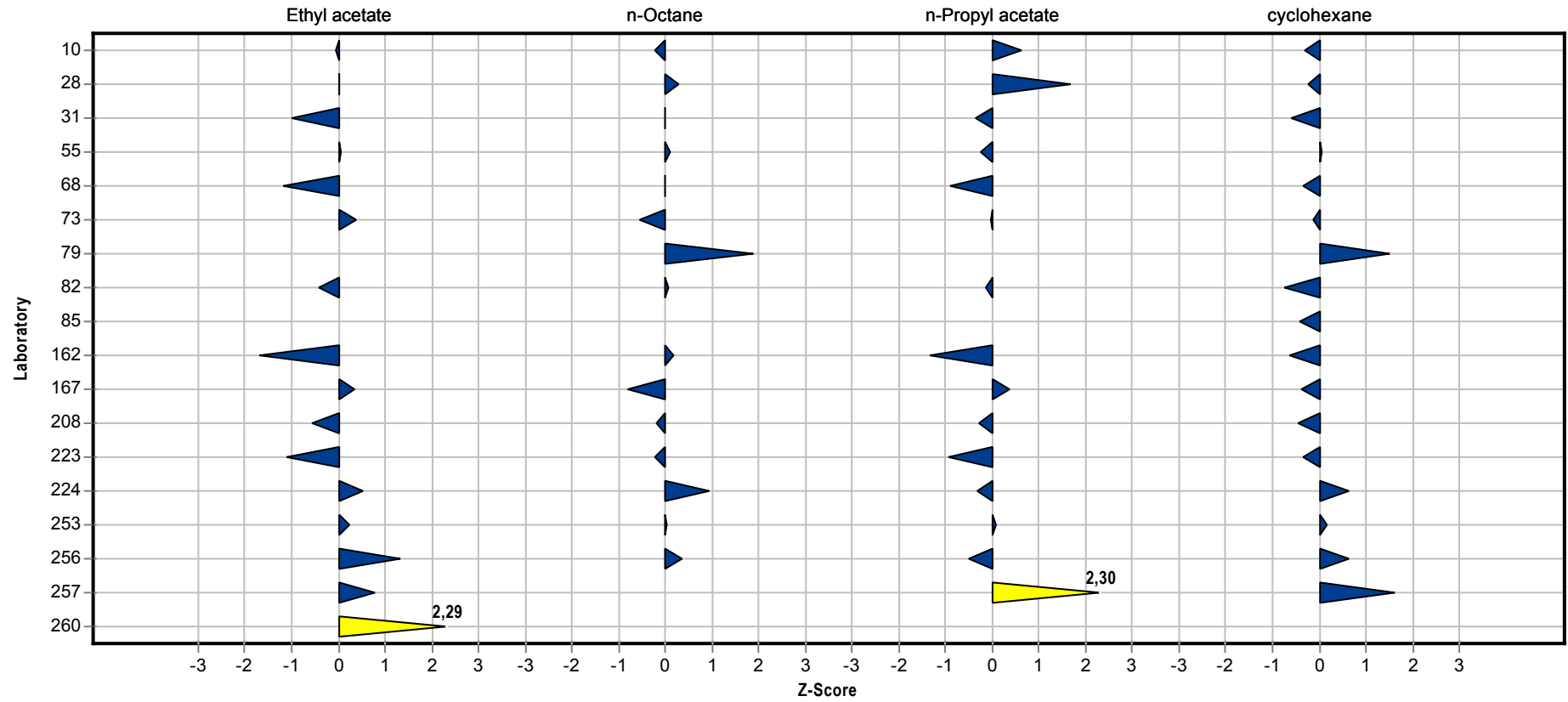
Measurand:	cyclohexane	Mean:	0,313 mg/tube
Sample:	2	Reprod. s.d.:	0,022 mg/tube
Method:	ISO 5725-2	Rel. reprod. s.d.:	7,05%
Rel. target s.d.:	10,00% (Limited)	Reference value:	0,293 mg/tube
No. of laboratories:	17	Range of tolerance:	0,251 - 0,376 mg/tube ( $ Z\text{-Score}  \leq 2,00$ )



# Sample chart of Z-Scores

Sample: 2

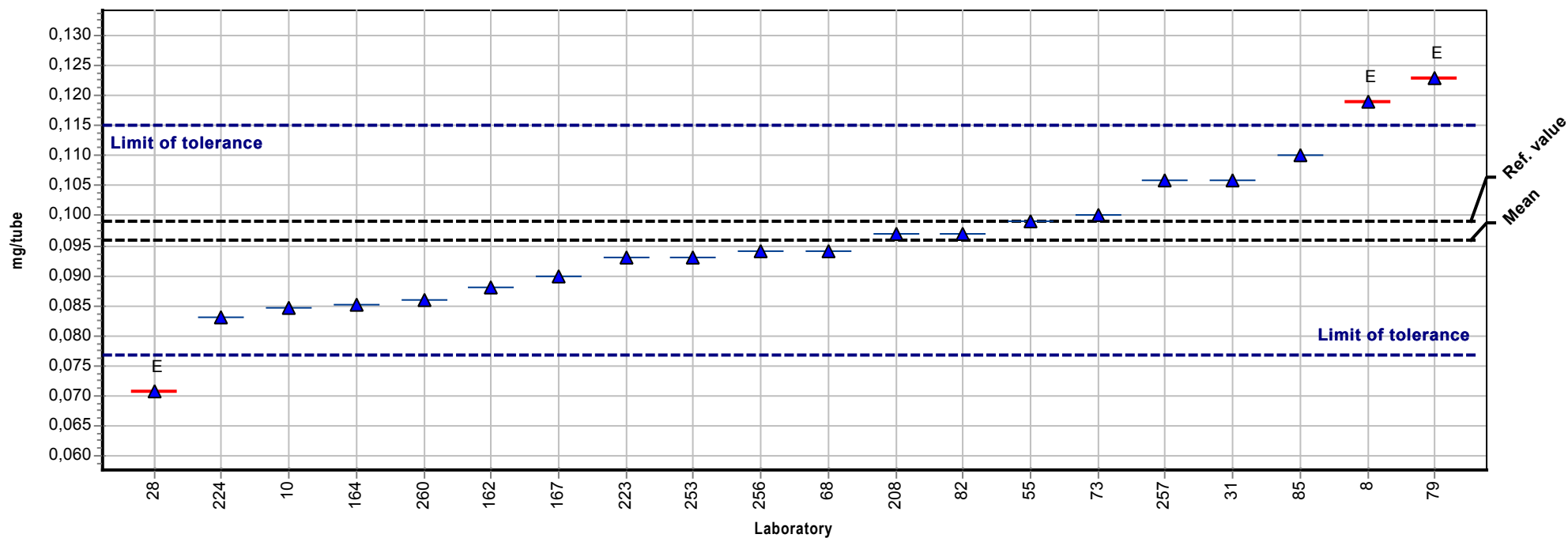
Measurand





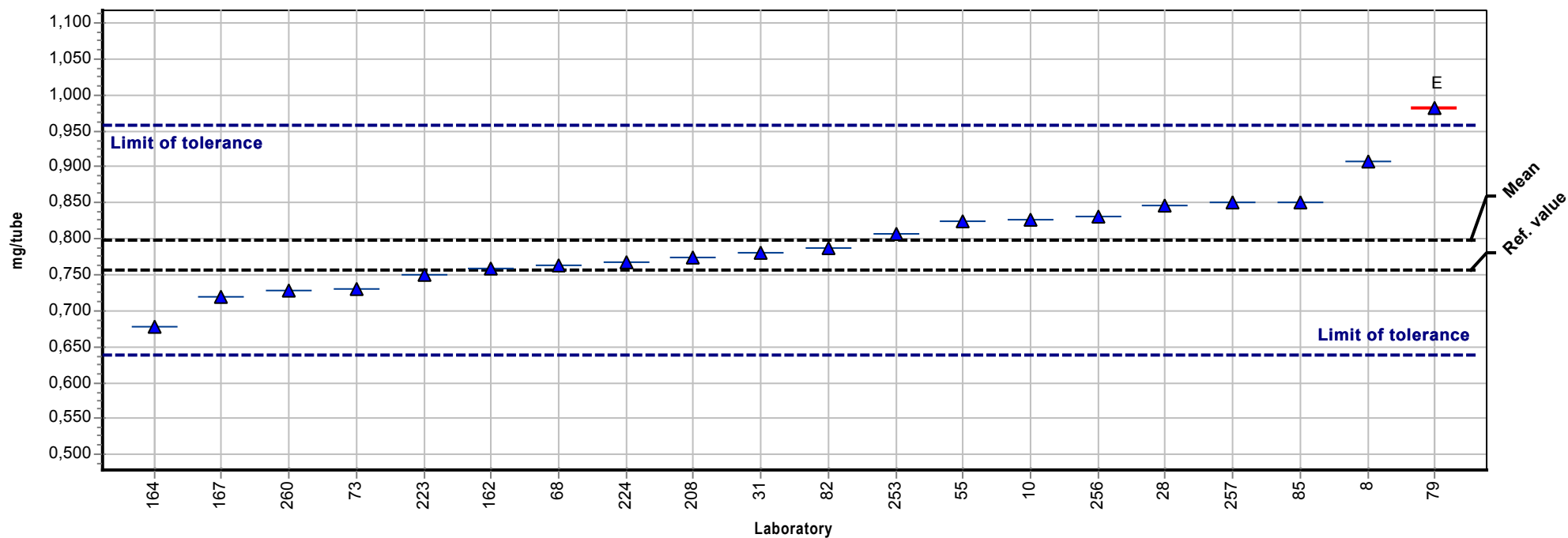
## Summary results

<b>Measurand:</b>	<b>o-Xylene</b>	<b>Mean:</b>	<b>0,096 mg/tube</b>
<b>Sample:</b>	<b>3</b>	<b>Reprod. s.d.:</b>	<b>0,012 mg/tube</b>
<b>Method:</b>	<b>ISO 5725-2</b>	<b>Rel. reprod. s.d.:</b>	<b>13,01%</b>
<b>Rel. target s.d.:</b>	<b>10,00% (Limited)</b>	<b>Reference value:</b>	<b>0,099 mg/tube</b>
<b>No. of laboratories:</b>	<b>20</b>	<b>Range of tolerance:</b>	<b>0,077 - 0,115 mg/tube ( Z-Score  &lt;= 2,00)</b>



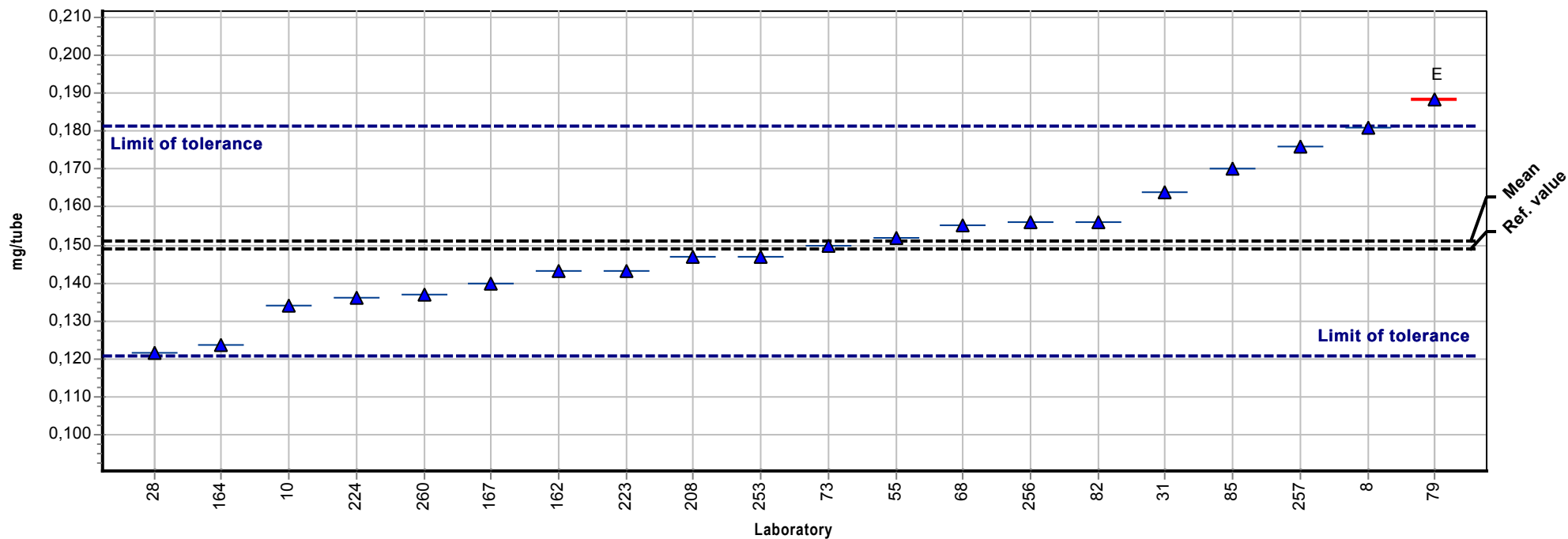
## Summary results

<b>Measurand:</b>	<b>Toluene</b>	<b>Mean:</b>	<b>0,798 mg/tube</b>
<b>Sample:</b>	<b>3</b>	<b>Reprod. s.d.:</b>	<b>0,070 mg/tube</b>
<b>Method:</b>	<b>ISO 5725-2</b>	<b>Rel. reprod. s.d.:</b>	<b>8,75%</b>
<b>Rel. target s.d.:</b>	<b>10,00% (Limited)</b>	<b>Reference value:</b>	<b>0,757 mg/tube</b>
<b>No. of laboratories:</b>	<b>20</b>	<b>Range of tolerance:</b>	<b>0,638 - 0,958 mg/tube ( Z-Score  &lt;= 2,00)</b>



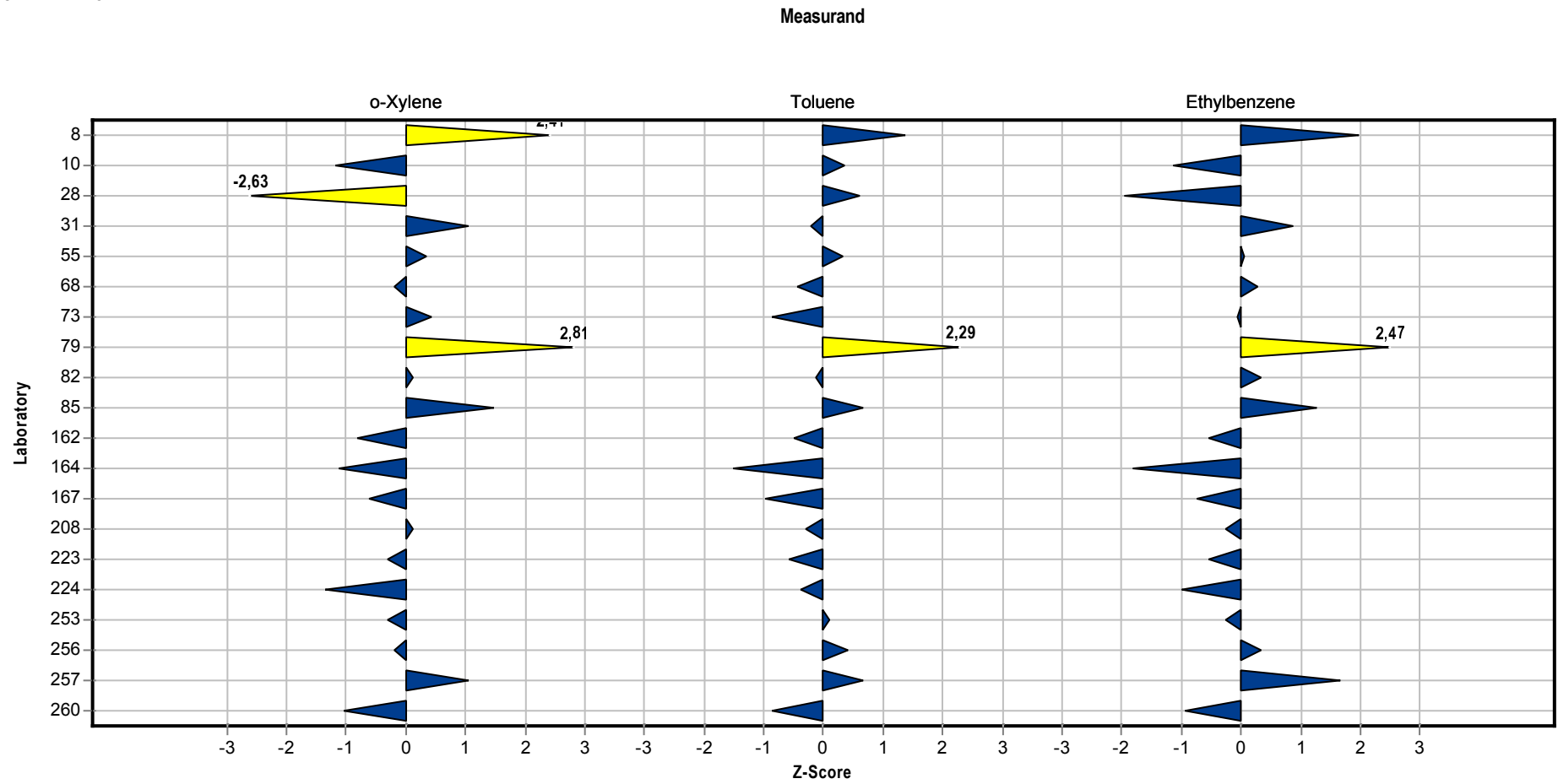
## Summary results

<b>Measurand:</b>	Ethylbenzene	<b>Mean:</b>	0,151 mg/tube
<b>Sample:</b>	3	<b>Reprod. s.d.:</b>	0,018 mg/tube
<b>Method:</b>	ISO 5725-2	<b>Rel. reprod. s.d.:</b>	11,87%
<b>Rel. target s.d.:</b>	10,00% (Limited)	<b>Reference value:</b>	0,149 mg/tube
<b>No. of laboratories:</b>	20	<b>Range of tolerance:</b>	0,121 - 0,181 mg/tube ( $ Z\text{-Score}  \leq 2,00$ )



# Sample chart of Z-Scores

Sample: 3



## Questions and Answers

Participant	Analytical method	Desorption solution
8	Analyse de gaz et vapeurs organiques par GC/MS selon la norme NF X 43-267	CS2
10	ISO16200-1	CS2
28	NF X 43 267	Carbon Disulfide
31	?	CH2Cl2:CS2:Methanol 60:35:5
55	No, intern method GC-MS	CS2
68	Weder DFG noch IFA-Arbeitsmappe	CS2
73	keine Bestimmte, Desorptionsmittel musste für alle geeignet sein	CS2 + ISTD Nonan 1 mg/ml
79	BIA 6265	CS2
82	Hausmethode, angelehnt an DFG, Nr.: 3	CS2 / Isopropanol 80/20
85	interne Standard-Methode	CS2
162	Hausmethode	Schwefelkohlenstoff
164	DIN EN 13649	Schwefelkohlenstoff
167	Capillary Gas Chromatography	Carbon disulfide
208	own, based on NIOSH and OSHA methods	CS2
223	DIN EN 13649 (2011/05)	Schwefelkohlenstoff
224	inhouse method	Carbon Disulfide
253	GC/MS	CS2
256	eigene	CS2
257	Gas chromatography	Carbone sulfide
260	NF X 43-267	CS2

Participant	Volume of desorption solution	Carrier gas	Injection
8	4	He	1µL
10	1/1ml	Air	Split
28	2ml	He	Split
31	2,5 mL	Helium	1 µL
55	2 ml	Helium	split
68	1 ml	Helium	split

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Participant	Volume of desorption solution	Carrier gas	Injection
73	ca. 2 ml	Helium	split
79	0,5ml	Wasserstoff	split
82	1 mL	Stickstoff	Splitt
85	1 ml	Stickstoff	Splitless
162	1 ml	Wasserstoff	Split
164	1,6 ml	Helium	splitless
167	1.5 mL	Helium	1 µL
208	2 ml	He	split, 1µl
223	4 ml	Stickstoff bzw . Helium	split bzw . on-column
224	2 mL	He	split
253	1,5 ml	Helium	1 myl splitless
256	3 ml	Helium	split
257	1 ml	Helium	2 µl
260	1 ml	helium	Split

Participant	Analytical column
8	30m x 0,25mm ID x 0,25um DF
10	Supelcow ax 10 & SPB5
28	VF5MS 30x0.25x1
31	60m RXI-5 und 60m Innow ax
55	RTX 502.2 60m x 0.32mm x 1.8 µm
68	Vocol von Supelco
73	DB5-MS 60 m
79	CP Sil 5CB
82	Agilent HP-5 30m; 0,32mm; 0,25µm
85	DB-5 / DB-Wax
162	Varian CP SII PONA CB 50m x 0,21 mm ID x 0,5µm Filmdicke, Säule2 (für Probe1): Restek Stabilw ax 60m x 0,25mm ID 0,50 m Filmdicke
164	ZB 624 von Phenomenex; Dimensionen: Lange 60 m; ID: 0,25 mm; Filmdicke 1,40 µm
167	Zebron Guardian ZB-5MS, 30 m, 0,25 mm id, 0,25 µm film thickness
208	Agilent HP-5 (30m x 0,320mm x 1,00 µm), Agilent HP-Innow ax (30m x 0,320mm x 0,50µm)
223	Supelcow ax 100 m x 0,25 µm Filmdicke bzw . Petrocol DH 50.2, 50 m x 0,2 mm, 0,5 µm Filmdicke

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Participant	Analytical column
224	BPX5
253	HP-1MS 30m x 0.250 mm, 1 µm film
256	Rtx-624,0.18mm,1µm Film
257	
260	Perkin -Elite 5MS

Participant	Detector	Data evaluation	Recovery rate	Date of analysis
8	Mass	20/032014	Yes for Toluene KD=0.88	20/03/2014
10	FID	external	no	03/24/2014
28	MS	Internal standard	No	11/03/2014
31	FID	Agilent OpenLab C.01.03	ja	26.02.2014
55	MS	internal standard	yes	17/03/2013 en 19/03/2014
68	FID	interner Standard	Nein	17./18.3.2014
73	FID	interner Standard	nein	10.03.2014
79	FID	interner Standard		12. KW 2014
82	FID	Interner Standard	Nein	11.03.2014
85	FID	interner Standard	ja	05.03.2014
162	FID	Interner Standard	ja	01.03.2014
164	MS	externer Standard	nein	05.03. bis 26.03.2014
167	FID, 310 °C			13.03.2014
208	FID	external standard		10.3.2014
223	FID	über internen Standard	100 %	21.03. und 25.03.2014
224	MS	internal standard	no	12/3/2014
253	MSD	Chemstation	80-100%	22 March 2014
256	MS	interner Standard	ja	27.02. - 03.04.2014
257	Masse			
260	MS	internal standard	No	06/03/2014