

## Summary of laboratory means

Sample 1

	Formaldehyde	Z score	Acetaldehyde	Z score	Propionaldehyde	Z score
Unit	mg/m <sup>3</sup>		mg/m <sup>3</sup>		mg/m <sup>3</sup>	
29	0,076	0,47	0,617	0,50	0,631	0,39
44	0,073	0,02	0,615	0,46	0,680	1,20
123	0,068	-0,67	0,592	0,07	0,623	0,26
155	0,076	0,43	0,651	1,08	0,676	1,13
165	0,073	0,02				
167	0,083	1,39	0,587	-0,01	0,647	0,66
185	0,070	-0,37	0,569	-0,32	0,603	-0,07
186	0,055	-2,47 E	0,417	-2,90 E	0,441	-2,74 E
187	0,080	0,98	0,638	0,86	0,645	0,62
191	0,074	0,15	0,617	0,50	0,634	0,44
208	0,069	-0,53	0,588	0,01	0,591	-0,27
221	0,074	0,15	0,616	0,48	0,561	-0,76
262	0,064	-1,22	0,475	-1,92	0,486	-2,00
267	0,085	1,66	0,658	1,20	0,675	1,12
Method	ISO 5725-2		ISO 5725-2		ISO 5725-2	
Assessment	Z <=2,00		Z <=2,00		Z <=2,00	
Mean	0,073		0,588		0,607	
Reproducibility s.d.	0,008		0,069		0,073	
Rel. reproducibility s.d.	10,57 %		11,72 %		12,02 %	
Reference value	0,074		0,569		0,612	
Target s.d.	0,007		0,059		0,061	
Rel. target s.d.	10,00 %		10,00 %		10,00 %	
Lower limit of tolerance	0,058		0,470		0,486	
Upper limit of tolerance	0,087		0,705		0,729	
Type B outliers	0		0		0	

	Formaldehyde	Z score	Acetaldehyde	Z score	Propionaldehyde	Z score
Type E outliers	1		1		1	
Type F outliers	0		0		0	
No. of laboratories after elimination of outliers type A-D and F (without laboratories that only gave states but no measured values)	14		13		13	
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

	Formaldehyde	Z score	Propionaldehyde	Z score	Butyraldehyde	Z score
Unit	mg/m <sup>3</sup>		mg/m <sup>3</sup>		mg/m <sup>3</sup>	
–	–	--	–	--	–	--
29	0,052	0,82	0,939	0,08		
44	0,043	-1,12	0,983	0,55	0,950	0,46
123	0,052	0,73	0,900	-0,34	0,913	0,06
155	0,049	0,12	0,978	0,49	0,864	-0,48
165	0,047	-0,30				
167	0,055	1,35	0,956	0,26	0,937	0,32
185	0,048	-0,13	0,919	-0,14	0,873	-0,38
186	0,051	0,53	1,016	0,90	1,009	1,12
187	0,053	0,94	0,962	0,32	1,000	1,01
191	0,050	0,32	0,965	0,35	0,964	0,62
208	0,040	-1,74	0,675	-2,76 E	0,654	-2,80 E
221	0,048	-0,09	0,900	-0,34	0,950	0,46
262	0,034	-2,98 E	0,324	-6,52 BE	0,338	-6,28 BE
267	0,056	1,56	0,990	0,62	0,873	-0,38
–	–	--	–	--	–	--
Method	ISO 5725-2		ISO 5725-2		ISO 5725-2	
Assessment	Z <=2,00		Z <=2,00		Z <=2,00	
Mean	0,048		0,932		0,908	
Reproducibility s.d.	0,006		0,088		0,098	
Rel. reproducibility s.d.	12,42 %		9,49 %		10,74 %	
Reference value	0,048		0,922		0,923	
Target s.d.	0,005		0,093		0,091	
Rel. target s.d.	10,00 %		10,00 %		10,00 %	
Lower limit of tolerance	0,039		0,746		0,726	
Upper limit of tolerance	0,058		1,118		1,090	
Type B outliers	0		1		1	

	Formaldehyde	Z score	Propionaldehyde	Z score	Butyraldehyde	Z score
Type E outliers		1		2		2
Type F outliers		0		0		0
No. of laboratories after elimination of outliers type A-D and F (without laboratories that only gave states but no measured values)		14		12		11
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

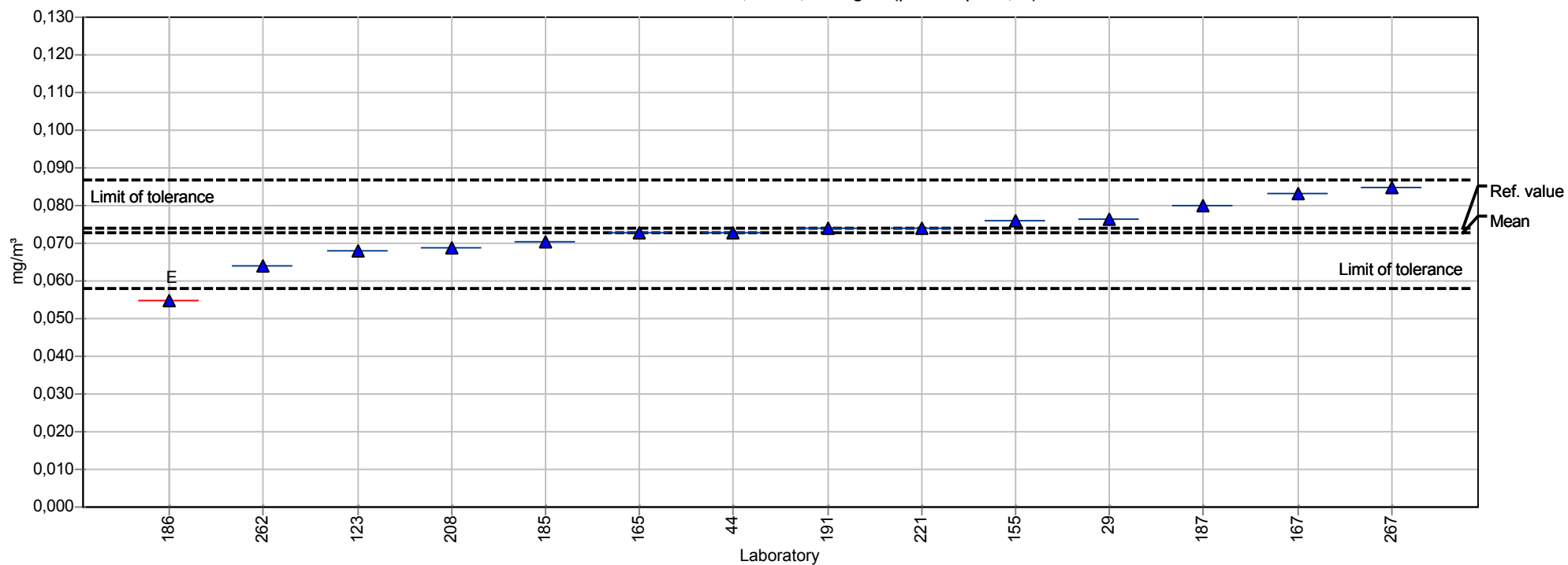
Sample 3

	Formaldehyde	Z score	Acetaldehyde	Z score	Propionaldehyde	Z score	Butyraldehyde	Z score
Unit	mg/m <sup>3</sup>		mg/m <sup>3</sup>		mg/m <sup>3</sup>		mg/m <sup>3</sup>	
29	0,121	0,18	0,495	0,38	0,490	0,10		
44	0,114	-0,41	0,460	-0,35	0,512	0,55	0,502	0,43
123	0,103	-1,33	0,421	-1,17	0,439	-0,95	0,441	-0,84
155	0,113	-0,49	0,493	0,34	0,508	0,47	0,447	-0,71
165	0,114	-0,41						
167	0,130	0,94	0,476	-0,02	0,515	0,61	0,507	0,53
185	0,110	-0,75	0,442	-0,73	0,468	-0,36	0,432	-1,03
186	0,129	0,86	0,501	0,50	0,518	0,68	0,512	0,63
187	0,125	0,52	0,507	0,63	0,509	0,49	0,532	1,05
191	0,117	-0,16	0,483	0,13	0,499	0,28	0,501	0,41
208	0,101	-1,50	0,408	-1,44	0,397	-1,82	0,422	-1,23
221	0,115	-0,33	0,469	-0,16	0,391	-1,94	0,489	0,16
262	0,138	1,61	0,514	0,78	0,527	0,86	0,521	0,82
267	0,134	1,27	0,529	1,10	0,536	1,04	0,471	-0,22
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	0,119		0,477		0,485		0,481	
Reproducibility s.d.	0,011		0,036		0,048		0,037	
Rel. reproducibility s.d.	9,38 %		7,57 %		9,85 %		7,79 %	
Reference value	0,116		0,451		0,483		0,483	
Target s.d.	0,012		0,048		0,049		0,048	
Rel. target s.d.	10,00 %		10,00 %		10,00 %		10,00 %	
Lower limit of tolerance	0,095		0,381		0,388		0,385	
Upper limit of tolerance	0,143		0,572		0,582		0,578	
Type B outliers	0		0		0		0	

	Formaldehyde	Z score	Acetaldehyde	Z score	Propionaldehyde	Z score	Butyraldehyde	Z score
Type E outliers	0		0		0		0	
Type F outliers	0		0		0		0	
No. of laboratories after elimination of outliers type A-D and F (without laboratories that only gave states but no measured values)	14		13		13		12	
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								

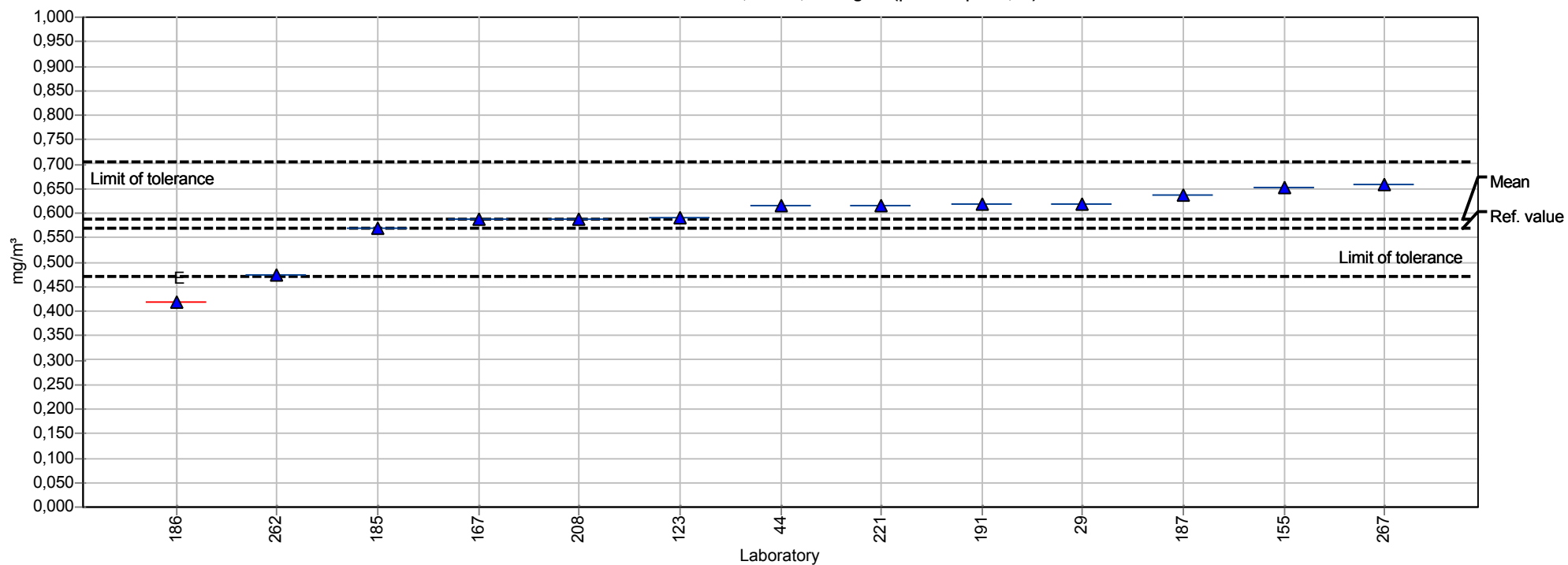
## Summary results

Measurand:	Formaldehyde	Mean:	0,073 mg/m <sup>3</sup>
Sample:	1	Reproducibility s.d.:	0,008 mg/m <sup>3</sup>
Method:	ISO 5725-2	Relative reproducibility s.d.:	10,57%
No. of laboratories:	14	Reference value:	0,074 mg/m <sup>3</sup>
		Range of tolerance:	0,058 - 0,087 mg/m <sup>3</sup> ( Z Score  ≤ 2,00)



## Summary results

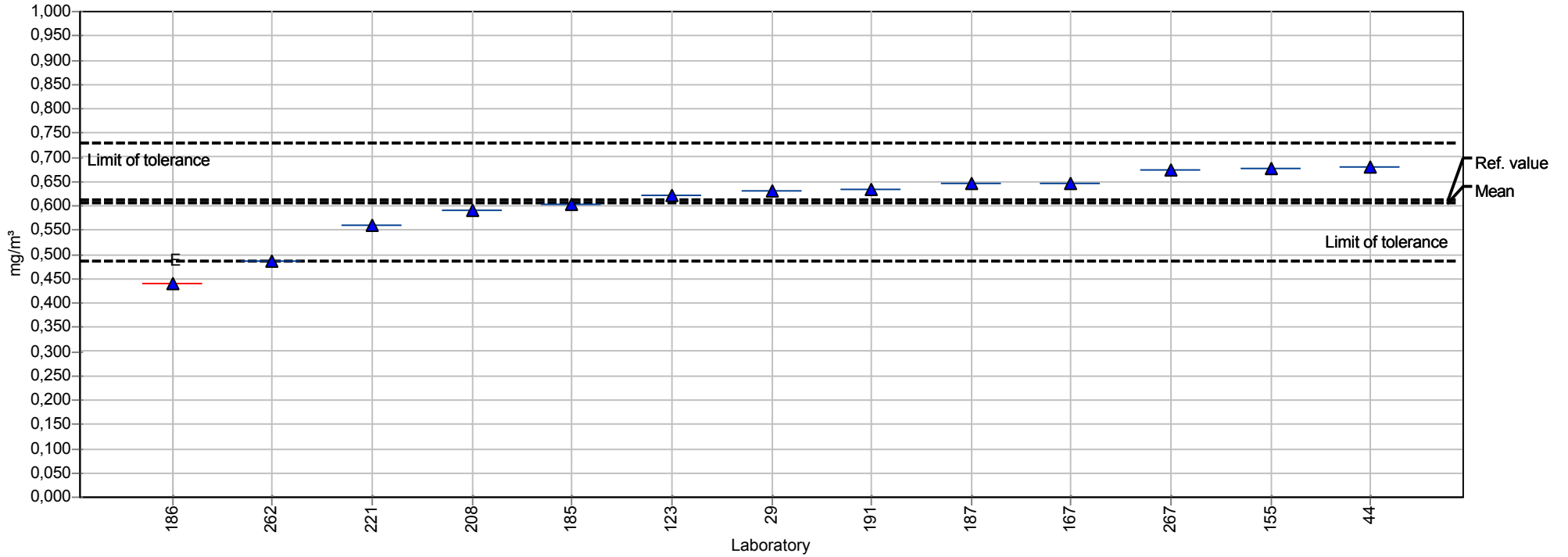
Measurand:	Acetaldehyde	Mean:	0,588 mg/m <sup>3</sup>
Sample:	1	Reproducibility s.d.:	0,069 mg/m <sup>3</sup>
Method:	ISO 5725-2	Relative reproducibility s.d.:	11,72%
No. of laboratories:	13	Reference value:	0,569 mg/m <sup>3</sup>
		Range of tolerance:	0,470 - 0,705 mg/m <sup>3</sup> ( Z Score  ≤ 2,00)





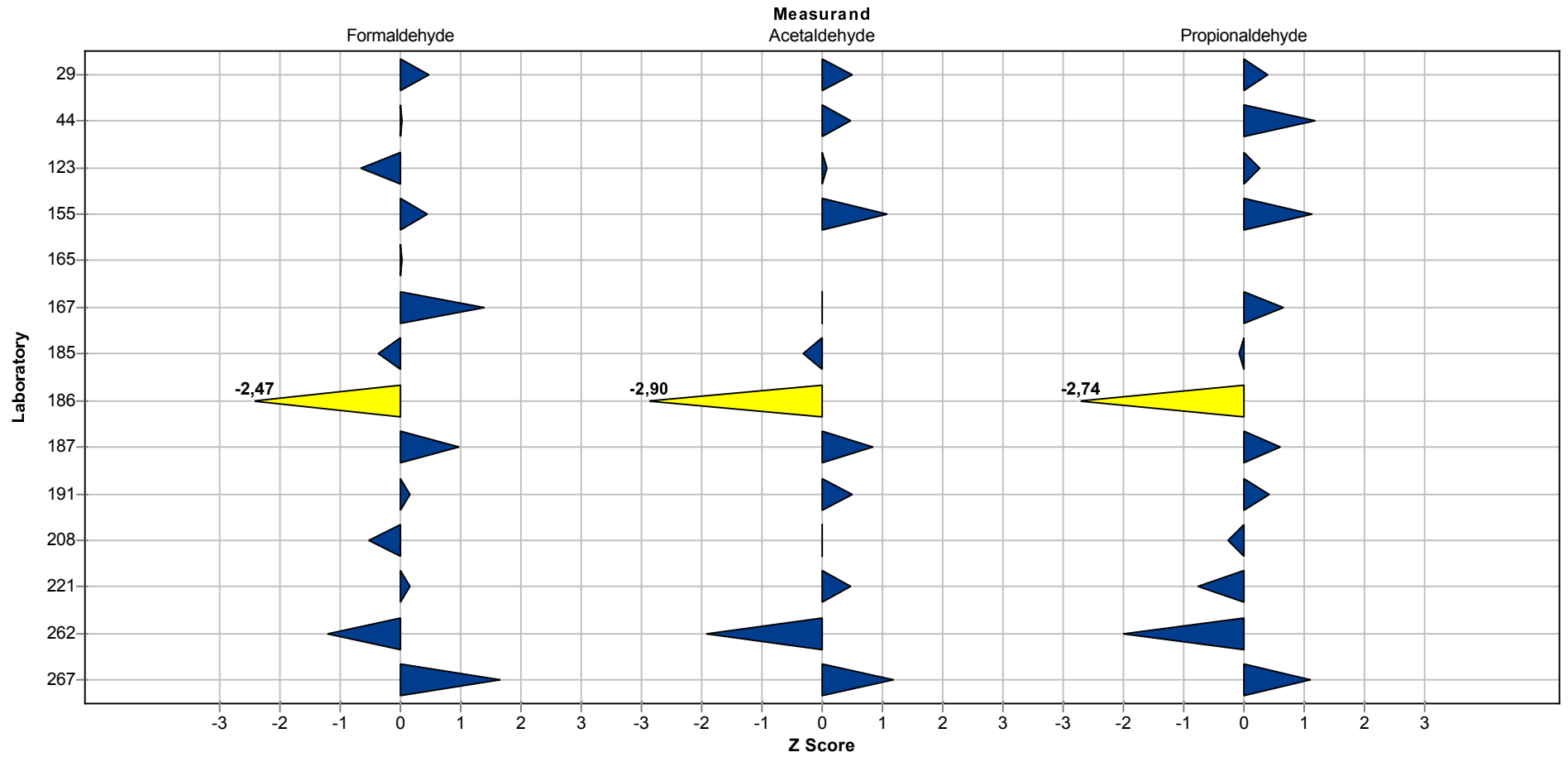
## Summary results

Measurand:	Propionaldehyde	Mean:	0,607 mg/m <sup>3</sup>
Sample:	1	Reproducibility s.d.:	0,073 mg/m <sup>3</sup>
Method:	ISO 5725-2	Relative reproducibility s.d.:	12,02%
No. of laboratories:	13	Reference value:	0,612 mg/m <sup>3</sup>
		Range of tolerance:	0,486 - 0,729 mg/m <sup>3</sup> ( Z Score  <= 2,00)



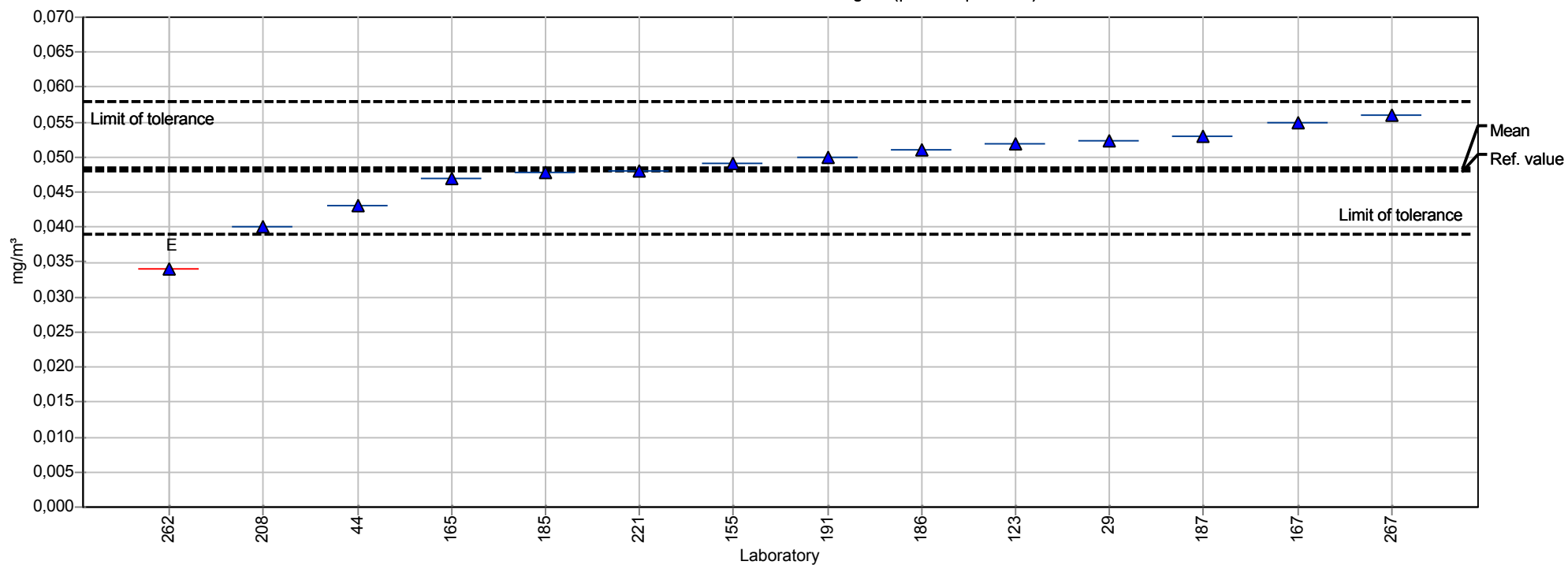
# Sample chart of Z Scores

Sample 1



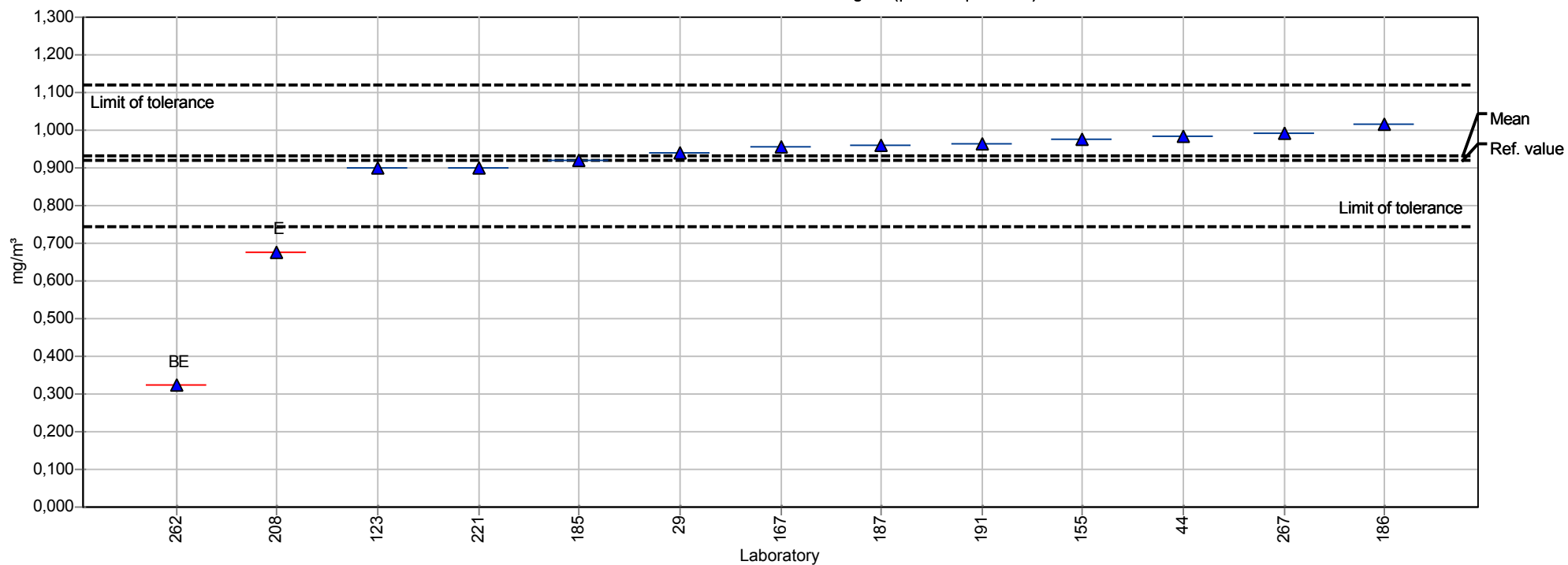
## Summary results

Measurand:	Formaldehyde	Mean:	0,048 mg/m <sup>3</sup>
Sample:	2	Reproducibility s.d.:	0,006 mg/m <sup>3</sup>
Method:	ISO 5725-2	Relative reproducibility s.d.:	12,42%
No. of laboratories:	14	Reference value:	0,048 mg/m <sup>3</sup>
		Range of tolerance:	0,039 - 0,058 mg/m <sup>3</sup> ( Z Score  ≤ 2,00)



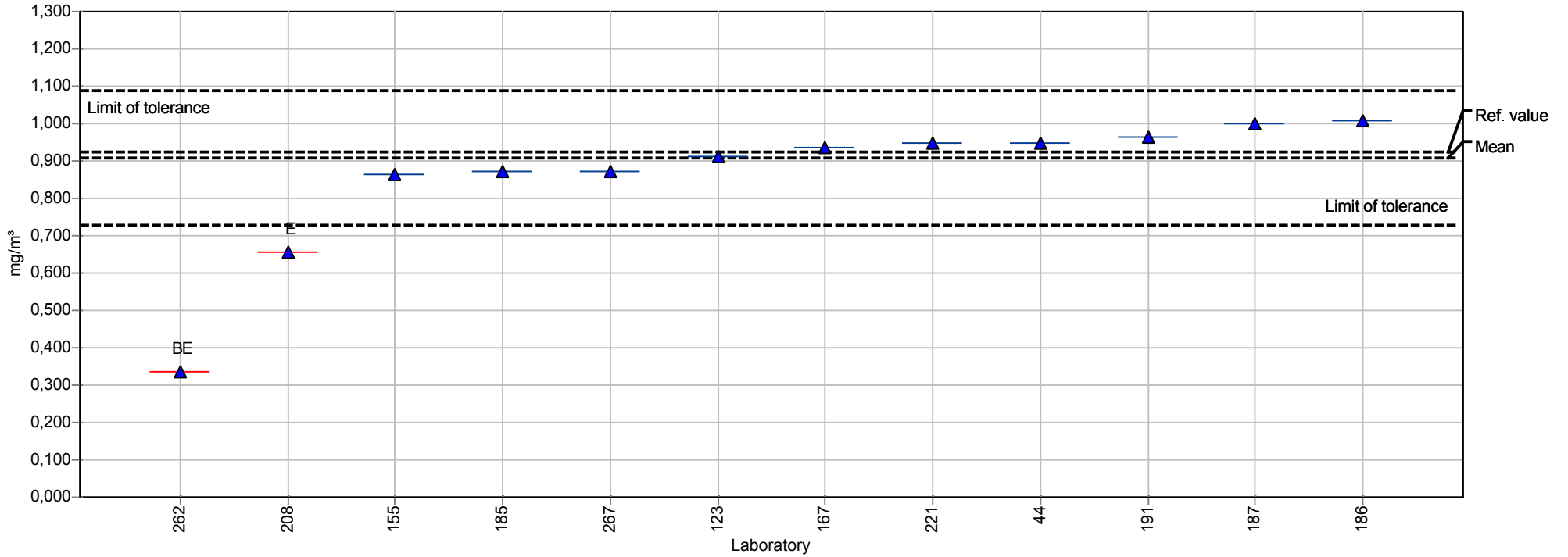
## Summary results

Measurand:	Propionaldehyde	Mean:	0,932 mg/m <sup>3</sup>
Sample:	2	Reproducibility s.d.:	0,088 mg/m <sup>3</sup>
Method:	ISO 5725-2	Relative reproducibility s.d.:	9,49%
No. of laboratories:	12	Reference value:	0,922 mg/m <sup>3</sup>
		Range of tolerance:	0,746 - 1,118 mg/m <sup>3</sup> ( $ Z \text{ Score}  \leq 2,00$ )



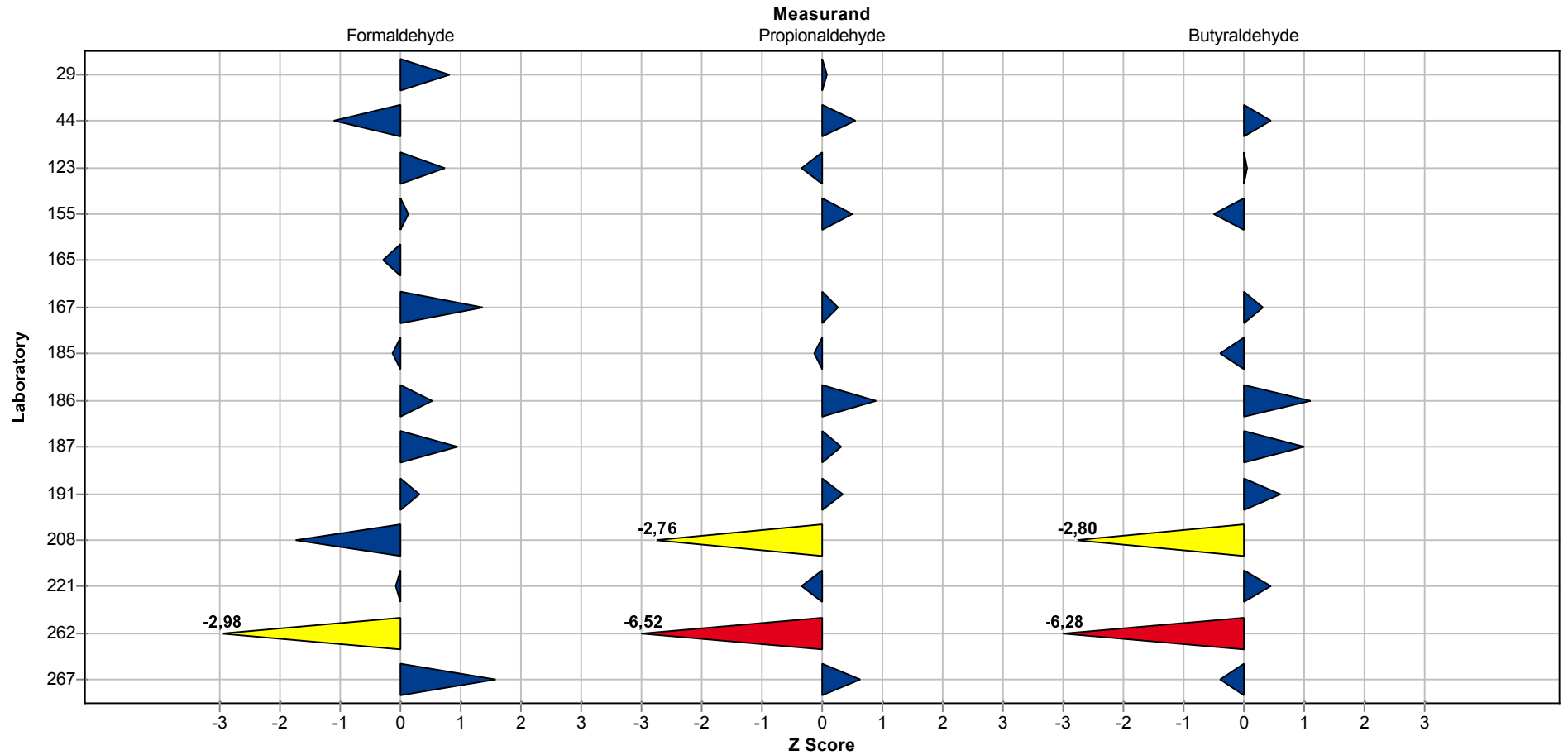
## Summary results

Measurand:	Butyraldehyde	Mean:	0,908 mg/m <sup>3</sup>
Sample:	2	Reproducibility s.d.:	0,098 mg/m <sup>3</sup>
Method:	ISO 5725-2	Relative reproducibility s.d.:	10,74%
No. of laboratories:	11	Reference value:	0,923 mg/m <sup>3</sup>
		Range of tolerance:	0,726 - 1,090 mg/m <sup>3</sup> ( Z Score  <= 2,00)



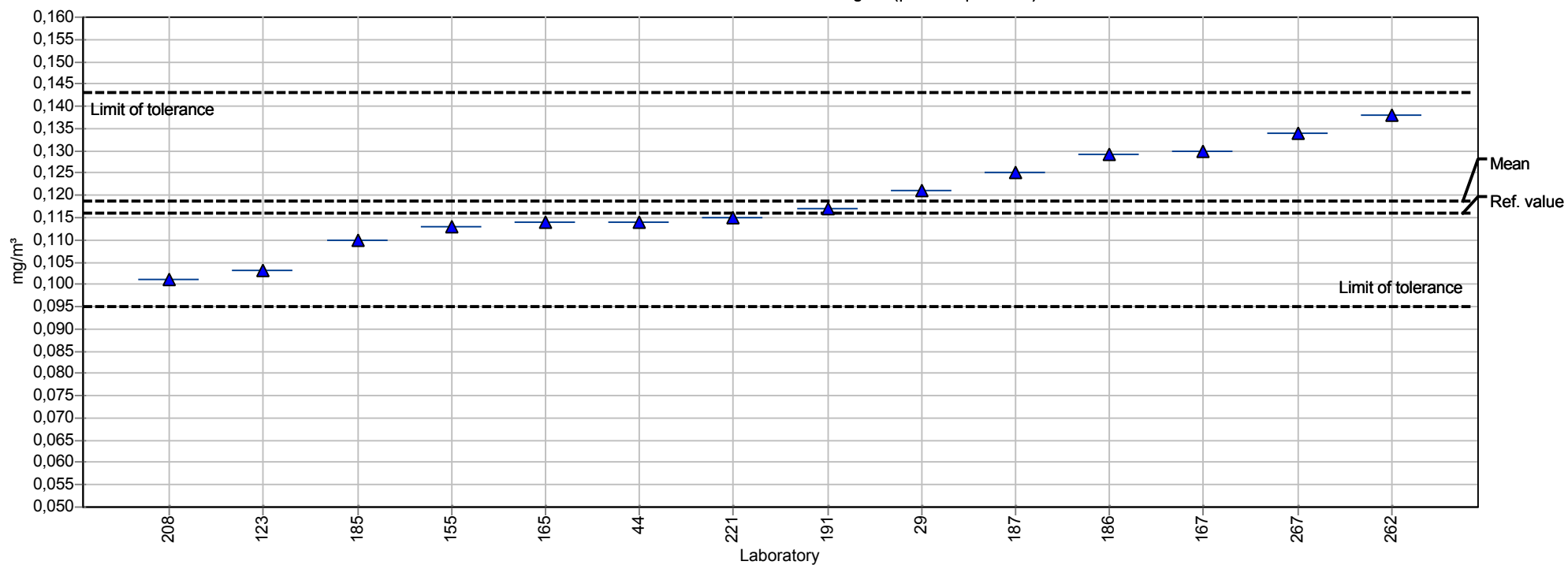
# Sample chart of Z Scores

Sample 2



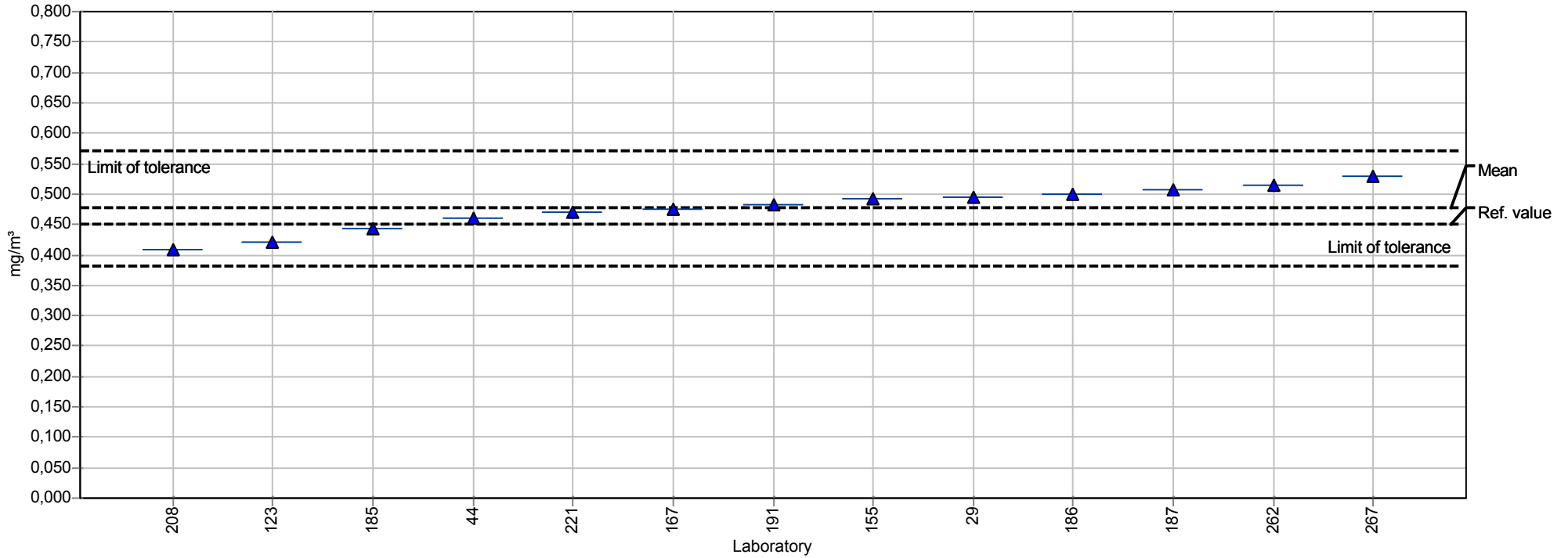
## Summary results

Measurand:	Formaldehyde	Mean:	0,119 mg/m <sup>3</sup>
Sample:	3	Reproducibility s.d.:	0,011 mg/m <sup>3</sup>
Method:	ISO 5725-2	Relative reproducibility s.d.:	9,38%
No. of laboratories:	14	Reference value:	0,116 mg/m <sup>3</sup>
		Range of tolerance:	0,095 - 0,143 mg/m <sup>3</sup> ( Z Score  ≤ 2,00)



## Summary results

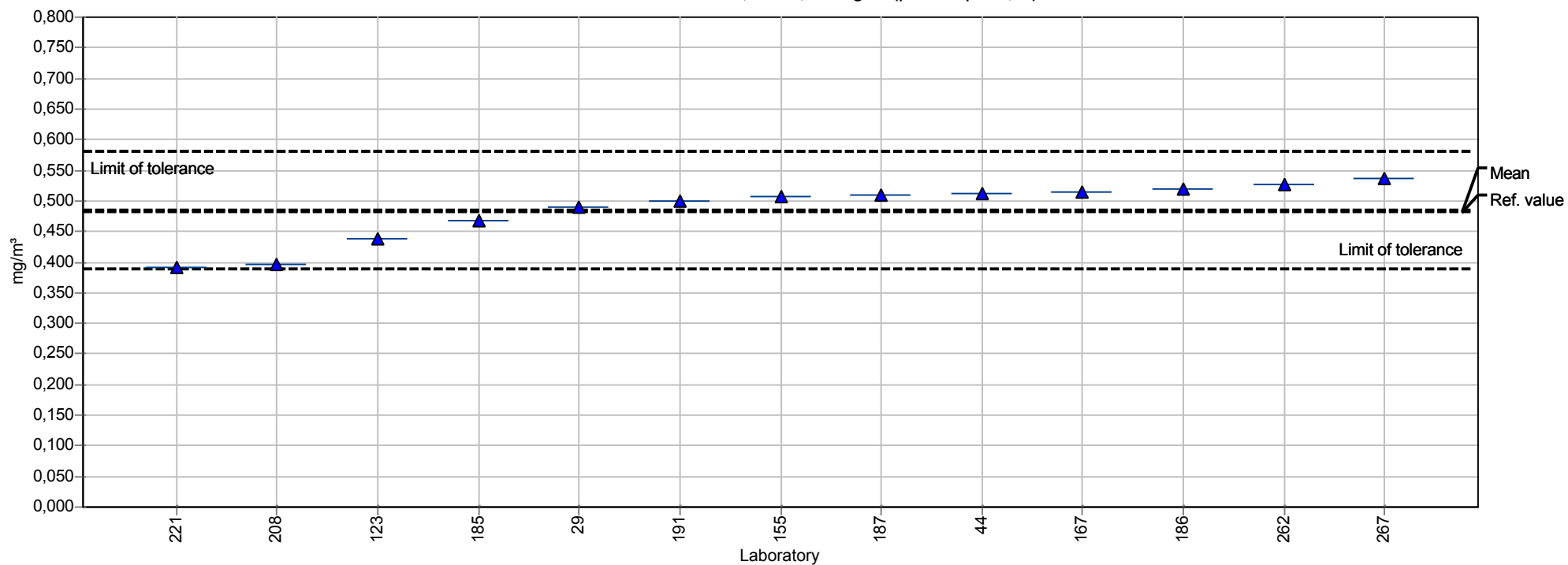
Measurand:	Acetaldehyde	Mean:	0,477 mg/m <sup>3</sup>
Sample:	3	Reproducibility s.d.:	0,036 mg/m <sup>3</sup>
Method:	ISO 5725-2	Relative reproducibility s.d.:	7,57%
No. of laboratories:	13	Reference value:	0,451 mg/m <sup>3</sup>
		Range of tolerance:	0,381 - 0,572 mg/m <sup>3</sup> ( $ Z \text{ Score}  \leq 2,00$ )





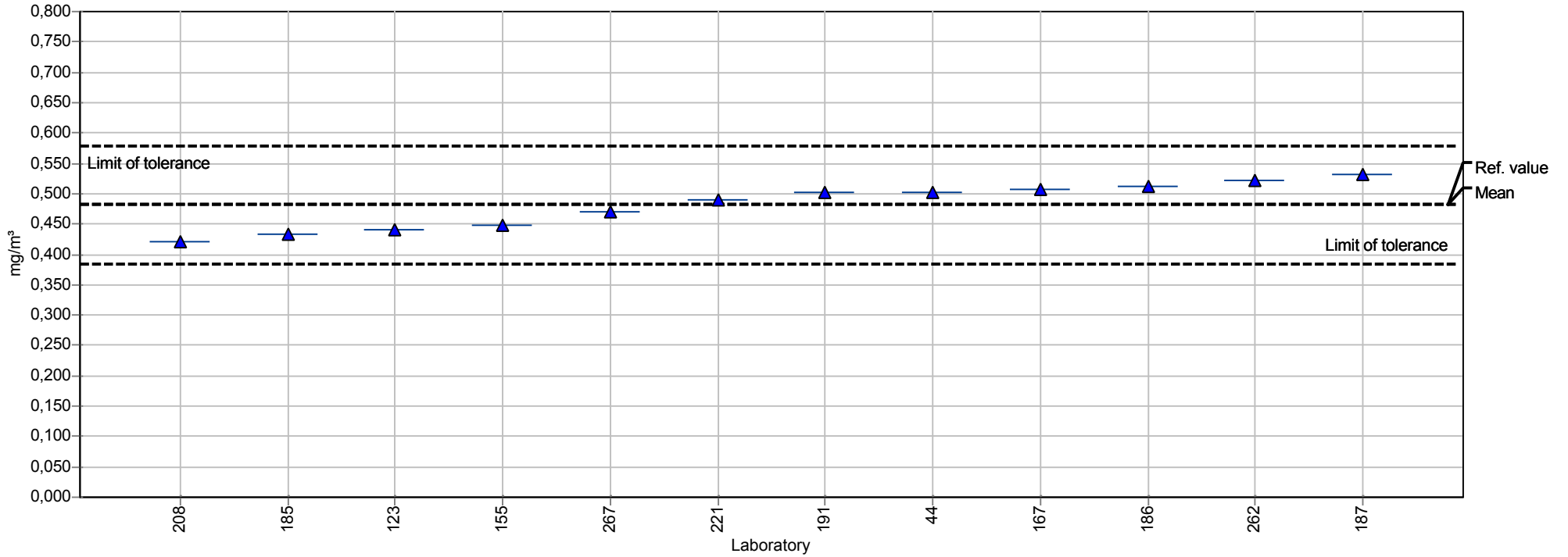
## Summary results

Measurand:	Propionaldehyde	Mean:	0,485 mg/m <sup>3</sup>
Sample:	3	Reproducibility s.d.:	0,048 mg/m <sup>3</sup>
Method:	ISO 5725-2	Relative reproducibility s.d.:	9,85%
No. of laboratories:	13	Reference value:	0,483 mg/m <sup>3</sup>
		Range of tolerance:	0,388 - 0,582 mg/m <sup>3</sup> ( Z Score  ≤ 2,00)



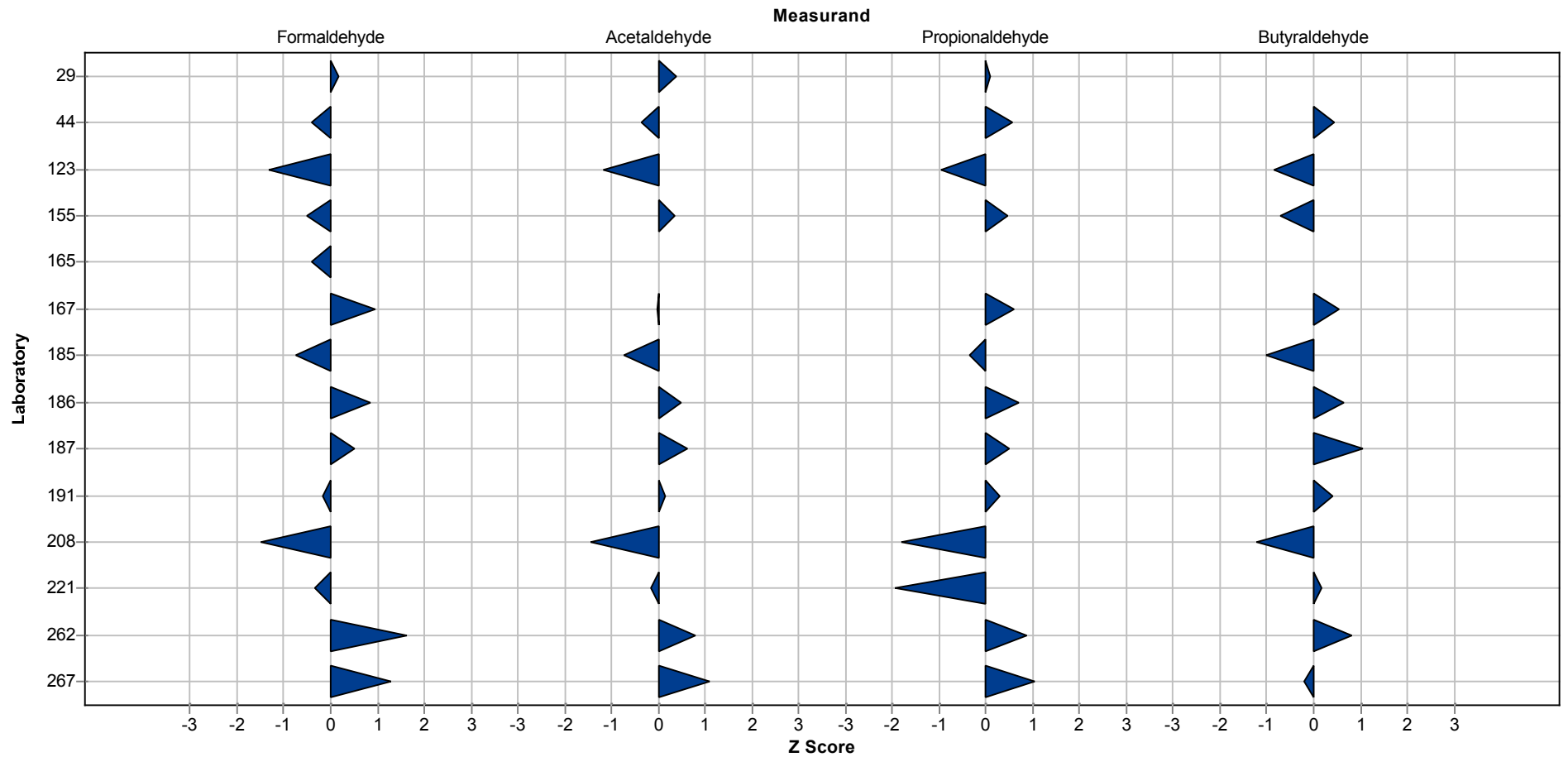
## Summary results

Measurand:	Butyraldehyde	Mean:	0,481 mg/m <sup>3</sup>
Sample:	3	Reproducibility s.d.:	0,037 mg/m <sup>3</sup>
Method:	ISO 5725-2	Relative reproducibility s.d.:	7,79%
No. of laboratories:	12	Reference value:	0,483 mg/m <sup>3</sup>
		Range of tolerance:	0,385 - 0,578 mg/m <sup>3</sup> ( Z Score  <= 2,00)



# Sample chart of Z Scores

Sample 3



## Questions and Answers

Participant	kind of tube	method	date start sample preparation
29	Waters DNPH XPosure	HPLC	11.10.12
44	Waters Sep-Pak DNPH-Silica Cartridge	UPLC/PDA	2012.10.20
123	cartouche waters (ref: Sep-Pak , XPoSure)	hplc/UV	4/10/12
155	Waters DNPH Sampler WAT047205	DIN ISO 16000-3 in Anlehnung	01.10.2012
165	Supelco DNPH S10	ISO 16000-3:2002	23/09/2012
167	Waters Sep-Pak DNPH-Silica Cartridge (WAT037500)	HPLC	2012.10.01
185	Supelco DNPH-Kartusche	HPLC	21.09.12
186	WATERS DNPH CARTRIDGE	16000-3	2012-09-25
187	Set Pak	HPLC CL/012-a	25-10-12
191	WPOSURE DNPH CART. WATERS	16000-3 / ana 033 (internal methdo)	25/09/12
208	Sep-Pak DNPH-silica cartridge	EPA-TO-11A	2.10.2012
221	Waters (vom Veranstalter bereitgestellt)	ISO 16000-3	27.09.2012
262	Tube, Trtd.Silica Gel Lot 7411 Cat.No.226-119	HPLC	27.09.2012
267	LpDNPH Sigma 21014	HPLC	28.9.2012

Participant	solvent	volume of desorption solution	storage time after desorption
29	ACN	2 x 5ml	24h
44	Acetonitrile	5mL	48h
123	CH3CN	5 ml	24 h
155	Acetonitril	5 ml	ein Tag
165	Acetoinitril	3 ml	24 h in Kühlschrank bei 4 °C
167	3 mL Acetonitrile (AcN), filled to 5 mL with distilled water	3 mL Acetonitrile	2 to 3 hours RT
185	Acetonitril	1 ml	1 Woche
186	acetoneitril	5mL	no
187	ACN	5 ml	24 h
191	acetonitrile	5 ml	no
208	acetonitrile	3 mL	no
221	Acetonitril	5 ml	10 min
262	Acetonitril	1ml	nein
267	Acetonitril	5ml	1 Tag / 10 Tage (Butyraldehyd)

Round-robin test Aldehydes 3/2012

Participant	date of analysis	pump/pressure	injection volume
29	12.10.12	Niederdruck	10µl
44	2012.10.25	6800psi	6uL
123	5/10/12	130 bars	25 µl
155	02.10.2012	ca. 130 bar	20 µl
165	24/09/2012		10µl
167	2012.10.01	Perkin Elmers series 200 LC pump Pressure: 2600 PSI (start)	40 microliters
185	29.09.12	Hochdruckpumpe	8 µl
186	2012-09-25	2300psi	20µL
187	25-10-12	--	5 µl
191	25/09/12	gradient pump / 1700 psi	20µl
208	2.10.2012	7500 psi	10 µL
221	27.09.2012	1050 Agilent, 40 bar	20 µl
262	27.09.2012	Hitachi /200 psi	20 µl
267	28.9.2012 / 9.10.2012 (Butyraldehyd)	quaternär, 200bar	25µl

Participant	mobile phase
29	H2O/ACN/THF; ACN
44	Acetonitrile ,water
123	CH3CN/H2O
155	Acetonitril Wasser Tetrahydrofuran
165	43% Wasser-57% Acetonitril
167	Acetonitrile with 0.1% Phosphoric acid and water with 0.1% Phosphoric acid
185	ACN /H2O
186	acetonitril/water/thf
187	ACN/H2O
191	water/THF/MeCN
208	A - water (60), B - ACN (30), C - THF (10)
221	ACN/Wasser
262	Acetonitril/Wasser: THF (9:1)
267	Acetonitril/Wasser resp. Acetonitril/Wasser/THF für Butyraldehyd

Participant	gradient/temperature program	flow rate
29	ja	1,5ml/min

Round-robin test Aldehydes 3/2012

Participant	gradient/temperature program	flow rate
44	25% Acetonitrile+75% water----- 80%Acetonitrile+20% water(11 min)-----25%Acetonitrile+75% water(12min)	0.5mL/min
123	25°C et 0-20mn/60%H2O-40%CH3CN puis 20-48mn / 20%H2O-80%CH3CN puis isochratique 5 min puis retour 60% H2O/40%CH3CN	1 ml/min
155	Gradient Start ACN 30% THF 10% H2O60% Ende: ACN 95% THF 5% H2O 0% Trennsäule Temperatur 35 °C constant	1 ml/min
165		1,3 ml/min
167	Water 45.0 / ACN 55.0 (3 min), up to 100.0 AcN in 4 min + 1 min, back to water 45.0/ AcN 55.0 in 1 min + 5 min 25 degrees Celcius	1.5 mL/min
185	ACN:H2O : 70 : 30 nach 30 min ACN : H2O : 95 : 5	0,5 ml/min
186	35°C	1.5mL/min
187		0.4 ml/min
191	60/10/30 --> 40/0/60 (22min) --> 20/0/80 (25min) --> 10/0/90 (30min)	1,2 ml/min
208	gradient/T = 40 °C	0.42 mL/min
221	0 min 60% ACN, 15 min 100% ACN	0,7 ml/min
262	Minute 0: 35% ACN; Minute 7: 35 %ACN; Minute 18: 80% ACN; Minute 20: 100% ACN; konstant 25°C;	1,5ml/min
267	40/60 H2O-Acetonitril 7min, 100% Acetonitril 20min.	1.5ml/min

Participant	detector	wavelength
29	UVD	365nm
44	PDA	360nm
123	barrette de diode	365,4 nm
155	DAD Agilent LC 1200 System	360 nm und 365 nm und 380 nm
165	UV	360 nm
167	Waters 2487 Dual Lambda Absorbance Detector	360 nm
185	DAD	190, 400, 365
186	PAD	360nm
187	DAD	350 nm
191	UV (waters 2487)	360nm
208	Waters ACQUITY UPLC PDA Detector	360 nm
221	DAD 1100 Agilent	365 nm
262	DAD	365 nm
267	DAD	365

Participant	analytical column/dimension
29	Waters Xbridge Phenyl

### Round-robin test Aldehydes 3/2012

Participant	analytical column/dimension
44	ACQUITYUPLC BEH C18 1.7umx50mm
123	C18 - 25cm*4.6mm , 5µm
155	Agilent Zorbax XDB-C18 4,6mm X 150mm 5µm
165	C18 250 x 4,5 mm
167	Waters Symmetry C18 3.5 micrometers 4.6 x 100 mm Cartridge + Waters microBondapak C18 10 micrometers 3.9 x 20 mm Guard Column
185	Nucleodur C18 250x4 3µm
186	WATERS NOVAPACK C18 / 150mm * 3.9mm * 4µm
187	C18 125 mm x 2.1 mm x 3 µm
191	reversed phase C18 5µm ; 3,9 x 150mm
208	HSS C18 1,8 µm/2,1 x 100 mm
221	Nucleosil 100-10 C 18, 250x4 (MN)
262	YMC-Pack C4 (5µm, 12nm), 150mm x 4,6mm
267	Symmetry C18, 250mm x 4.5mm x 5µm (Waters)

Participant	recovery rate
29	
44	1.0mg/L, 99.2%
123	
155	Kontrolle der vollständigen Desorbition erfolgte durch eine weitere Desorbition des Adsorbers wurde
165	nein
167	100 ± 0,2 %
185	90 - 110 %
186	yes
187	98 per cent
191	
208	no
221	
262	nein
267	