

# Proficiency testing for in-house and external measuring stations - results and evaluation

## Proficiency testing scheme aldehydes with own sampling

**09 - 10 October 2018**

## Summary of laboratory test results

Sample 1

	Acetaldehyde	Z score	Butyraldehyde	Z score	Formaldehyde	Z score
Unit	mg/m <sup>3</sup>		mg/m <sup>3</sup>		mg/m <sup>3</sup>	
40	1,161	0,03	0,292	0,17	0,167	0,13
46	1,262	0,90	0,347	2,09 E	0,167	0,13
76	1,095	-0,55	0,245	-1,45	0,180	0,89
104	1,220	0,54	0,290	0,10	0,160	-0,30
107	1,235	0,67	0,310	0,80	0,165	0,01
109	1,160	0,02	0,305	0,62	0,157	-0,48
111	1,250	0,79	0,269	-0,63	0,165	0,01
135	1,164	0,05	0,294	0,24	0,159	-0,36
138	1,210	0,45	0,300	0,45	0,170	0,31
172	0,971	-1,61	0,227	-2,09 E	0,132	-1,99 B
210	1,100	-0,50	0,295	0,28	0,160	-0,30
248	1,208	0,43	0,260	-0,94	0,157	-0,48
289	1,018	-1,21	0,298	0,36	0,172	0,43
-	-	--	-	--	-	--
Method	ISO 5725-2		ISO 5725-2		ISO 5725-2	
Assessment	Z <=2,00		Z <=2,00		Z <=2,00	
No. of laboratories that submitted results	13		13		13	
Mean	1,158		0,287		0,165	
Reproducibility s.d.	0,090		0,031		0,007	
Rel. reproducibility s.d.	7,73 %		10,67 %		4,12 %	
Reference value	1,148		0,283		0,150	
Target s.d.	0,116		0,029		0,016	
Rel. target s.d.	10,00 %		10,00 %		10,00 %	
Lower limit of tolerance	0,926		0,230		0,132	
Upper limit of tolerance	1,390		0,344		0,198	
Type B outliers					1	
No. of laboratories after elimination of outliers type A-D and F (without)	13		13		12	

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	Acetaldehyde	Z score	Butyraldehyde	Z score	Formaldehyde	Z score
laboratories that only gave states but no measured values)						
Explanation of outlier types						
A: Single outlier			Grubbs			
B: Differing laboratory mean			Grubbs			
C: Excessive laboratory s.d.			Cochran			
D: Excluded manually						
E: mean outside tolerance limits						
F: $ Z\text{-Score}  > 3,5$						

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## Summary of laboratory test results

Sample 2

	Acetaldehyde	Z score	Butyraldehyde	Z score	Formaldehyde	Z score
Unit	mg/m <sup>3</sup>		mg/m <sup>3</sup>		mg/m <sup>3</sup>	
40	0,979	0,06	1,079	0,18	0,072	0,32
46	1,067	0,97	1,314	2,40 E	0,073	0,46
76	0,927	-0,47	0,911	-1,40	0,105	5,05 BE
104	1,030	0,59	1,100	0,38	0,070	0,03
107	1,007	0,35	1,119	0,56	0,068	-0,25
109	0,957	-0,16	1,088	0,27	0,065	-0,68
111	1,049	0,78	0,995	-0,61	0,071	0,18
135	0,989	0,17	1,098	0,36	0,069	-0,11
138	1,030	0,59	1,100	0,38	0,070	0,03
172	0,805	-1,73	0,832	-2,15 E	0,054	-2,26 BE
210	0,960	-0,13	1,100	0,38	0,072	0,32
248	1,007	0,35	0,956	-0,98	0,066	-0,54
289	0,841	-1,35	1,083	0,22	0,072	0,25
-	-	--	-	--	-	--
Method	ISO 5725-2		ISO 5725-2		ISO 5725-2	
Assessment	Z <=2,00		Z <=2,00		Z <=2,00	
No. of laboratories that submitted results	13		13		13	
Mean	0,973		1,060		0,070	
Reproducibility s.d.	0,077		0,117		0,003	
Rel. reproducibility s.d.	7,95 %		11,07 %		3,68 %	
Reference value	0,960		1,090		0,061	
Target s.d.	0,097		0,106		0,007	
Rel. target s.d.	10,00 %		10,00 %		10,00 %	
Lower limit of tolerance	0,778		0,848		0,056	
Upper limit of tolerance	1,168		1,272		0,084	
Type B outliers					2	
No. of laboratories after elimination of outliers type A-D and F (without)	13		13		11	



## Summary of laboratory test results

Sample 3

	Acetaldehyde	Z score	Butyraldehyde	Z score	Formaldehyde	Z score	Propionaldehyde	Z score
Unit	mg/m <sup>3</sup>		mg/m <sup>3</sup>		mg/m <sup>3</sup>		mg/m <sup>3</sup>	
40	0,386	-0,11	0,860	0,18	0,139	0,67	0,744	0,39
46	0,425	0,89	1,048	2,40 E	0,136	0,44	0,779	0,88
76	0,374	-0,42	0,729	-1,37	0,149	1,41	0,620	-1,33
104	0,400	0,25	0,840	-0,06	0,110	-1,56	0,700	-0,22
107	0,403	0,33	0,879	0,40	0,131	0,06	0,769	0,74
109	0,395	0,12	0,875	0,36	0,129	-0,10	0,743	0,38
111	0,431	1,05	0,786	-0,70	0,134	0,29	0,689	-0,38
135	0,393	0,07	0,872	0,32	0,131	0,06	0,735	0,27
138	0,410	0,51	0,900	0,65	0,130	-0,02	0,750	0,48
172	0,341	-1,26	0,669	-2,08 E	0,108	-1,71	0,609	-1,49
210	0,390	0,00	0,920	0,89	0,140	0,75	0,770	0,75
248	0,399	0,23	0,755	-1,07	0,127	-0,25	0,671	-0,63
289	0,325	-1,67	0,852	0,08	0,130	-0,02	0,728	0,17
-	-	--	-	--	-	--	-	--
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	
No. of laboratories that submitted results	13		13		13		13	
Mean	0,390		0,845		0,130		0,716	
Reproducibility s.d.	0,030		0,095		0,011		0,055	
Rel. reproducibility s.d.	7,61 %		11,27 %		8,51 %		7,70 %	
Reference value	0,372		0,870		0,120		0,701	
Target s.d.	0,039		0,084		0,013		0,072	
Rel. target s.d.	10,00 %		10,00 %		10,00 %		10,00 %	
Lower limit of tolerance	0,312		0,676		0,104		0,573	
Upper limit of tolerance	0,468		1,014		0,156		0,859	
No. of laboratories after elimination of outliers type A-D and F (without laboratories that only gave states but no	13		13		13		13	

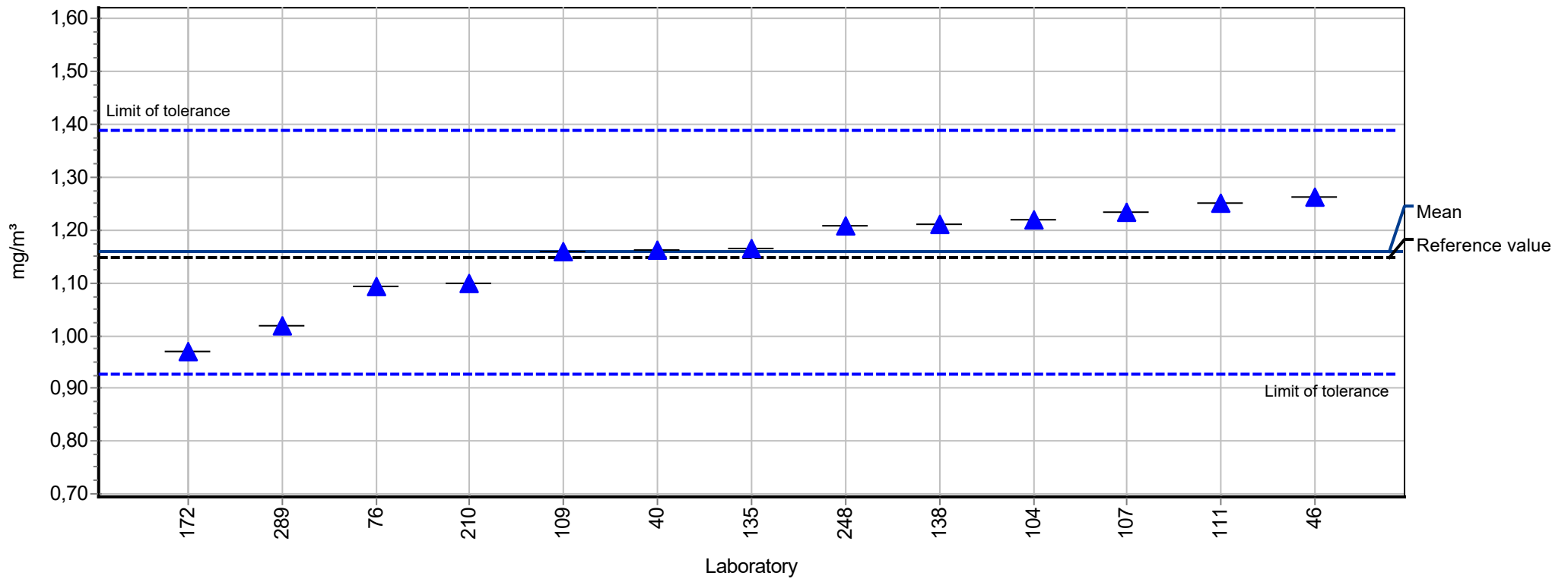
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	Acetaldehyde	Z score	Butyraldehyde	Z score	Formaldehyde	Z score	Propionaldehyde	Z score
measured values)								
Explanation of outlier types								
A: Single outlier			Grubbs					
B: Differing laboratory mean			Grubbs					
C: Excessive laboratory s.d.			Cochran					
D: Excluded manually								
E: mean outside tolerance limits								
F:  Z-Score >3,5								

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## Summary results

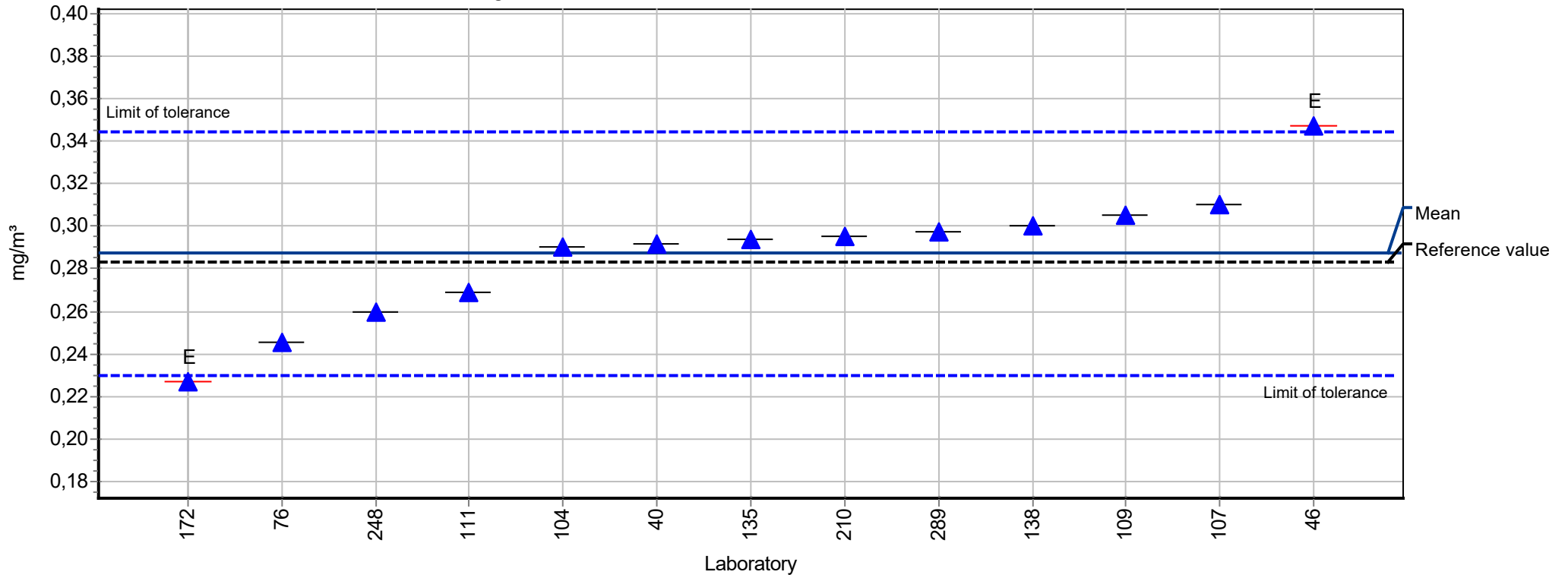
Measurand:	Acetaldehyde	Mean:	1,158 mg/m <sup>3</sup>
Sample:	1	Reprod. s.d.:	0,090 mg/m <sup>3</sup>
Method:	ISO 5725-2	Rel.reprod. s.d.:	7,73%
Rel.target s.d.:	10,00% (Limited)	Reference value:	1,148 mg/m <sup>3</sup>
No. of laboratories:	13	Range of tolerance:	0,926 - 1,390 mg/m <sup>3</sup> ( Z-Score  <= 2,00)





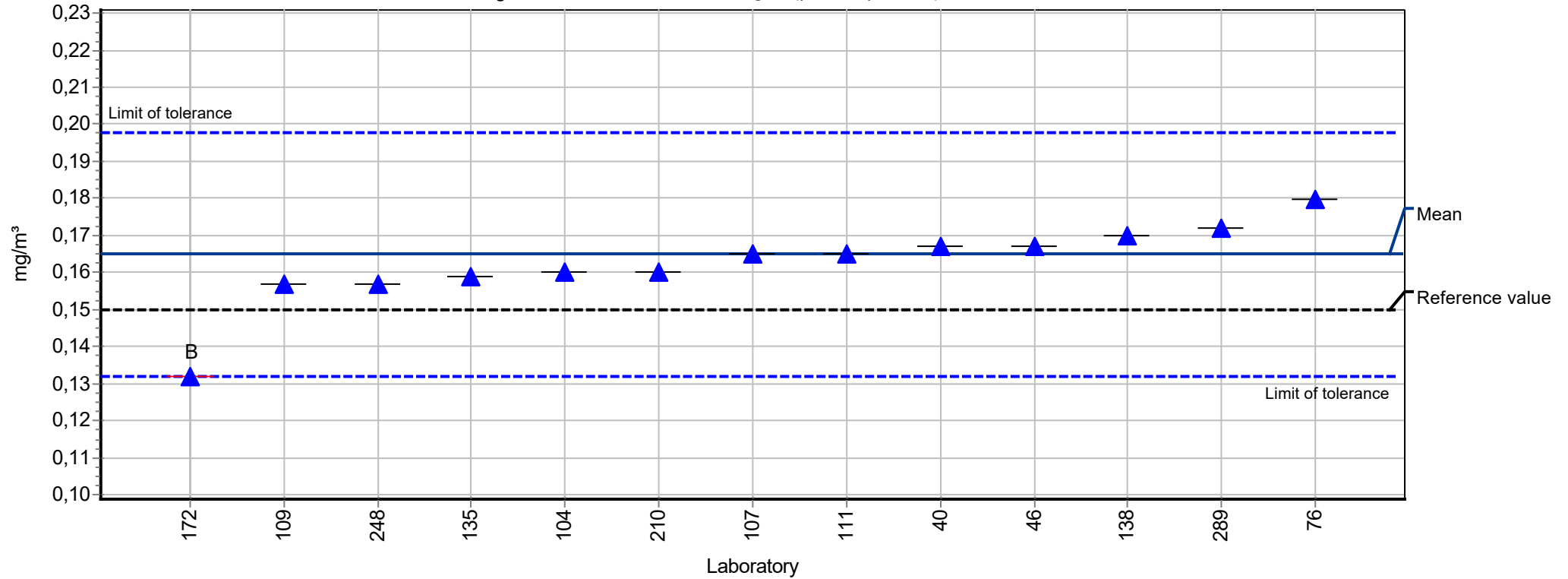
## Summary results

Measurand:	Butyraldehyde	Mean:	0,287 mg/m <sup>3</sup>
Sample:	1	Reprod. s.d.:	0,031 mg/m <sup>3</sup>
Method:	ISO 5725-2	Rel.reprod. s.d.:	10,67%
Rel.target s.d.:	10,00% (Limited)	Reference value:	0,283 mg/m <sup>3</sup>
No. of laboratories:	13	Range of tolerance:	0,230 - 0,344 mg/m <sup>3</sup> ( Z-Score  <= 2,00)



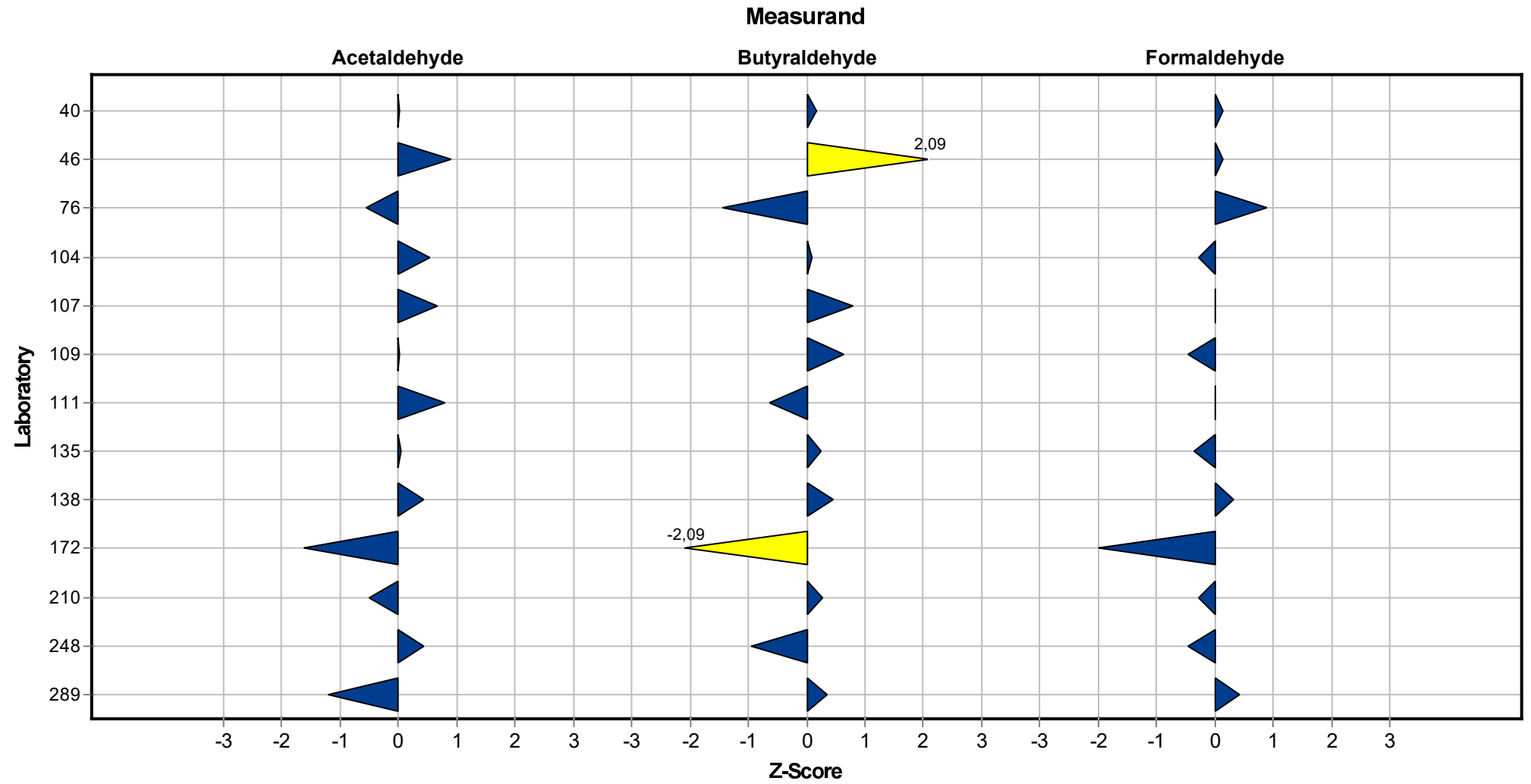
## Summary results

Measurand:	Formaldehyde	Mean:	0,165 mg/m <sup>3</sup>
Sample:	1	Reprod. s.d.:	0,007 mg/m <sup>3</sup>
Method:	ISO 5725-2	Rel.reprod. s.d.:	4,12%
Rel.target s.d.:	10,00% (Limited)	Reference value:	0,150 mg/m <sup>3</sup>
No. of laboratories:	12	Range of tolerance:	0,132 - 0,198 mg/m <sup>3</sup> ( Z-Score  <= 2,00)



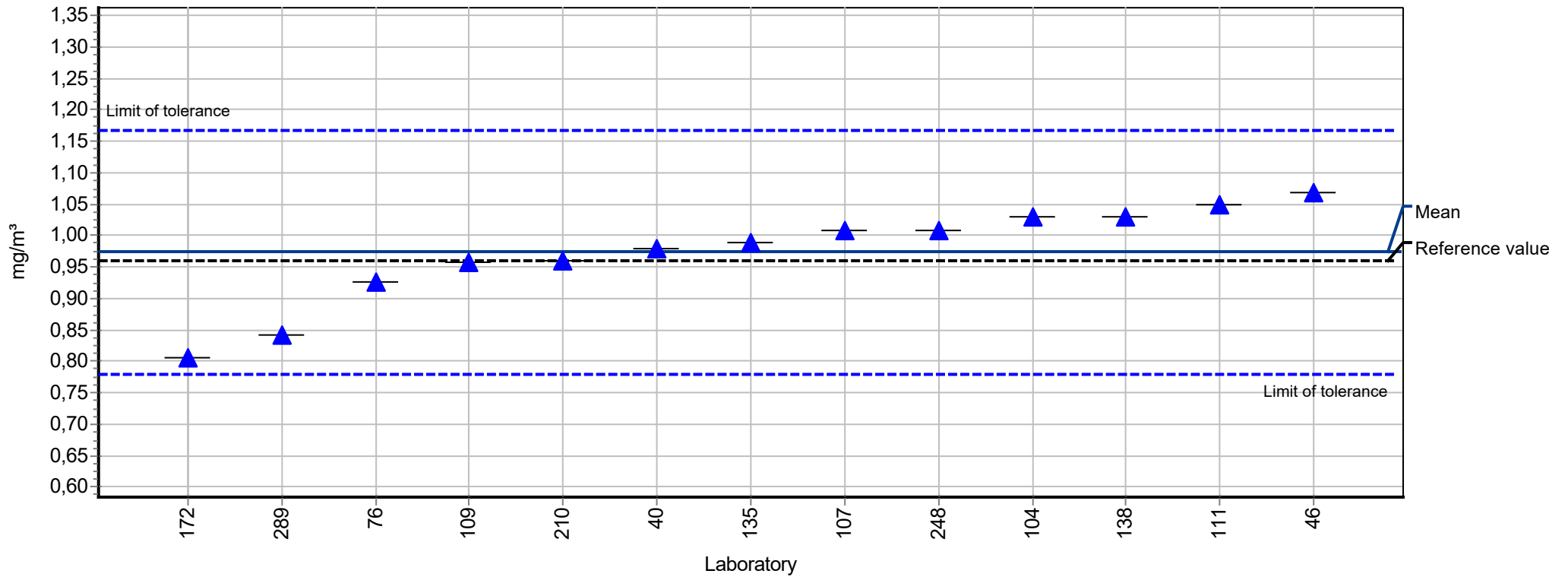
# Sample chart of Z-scores

Sample 1



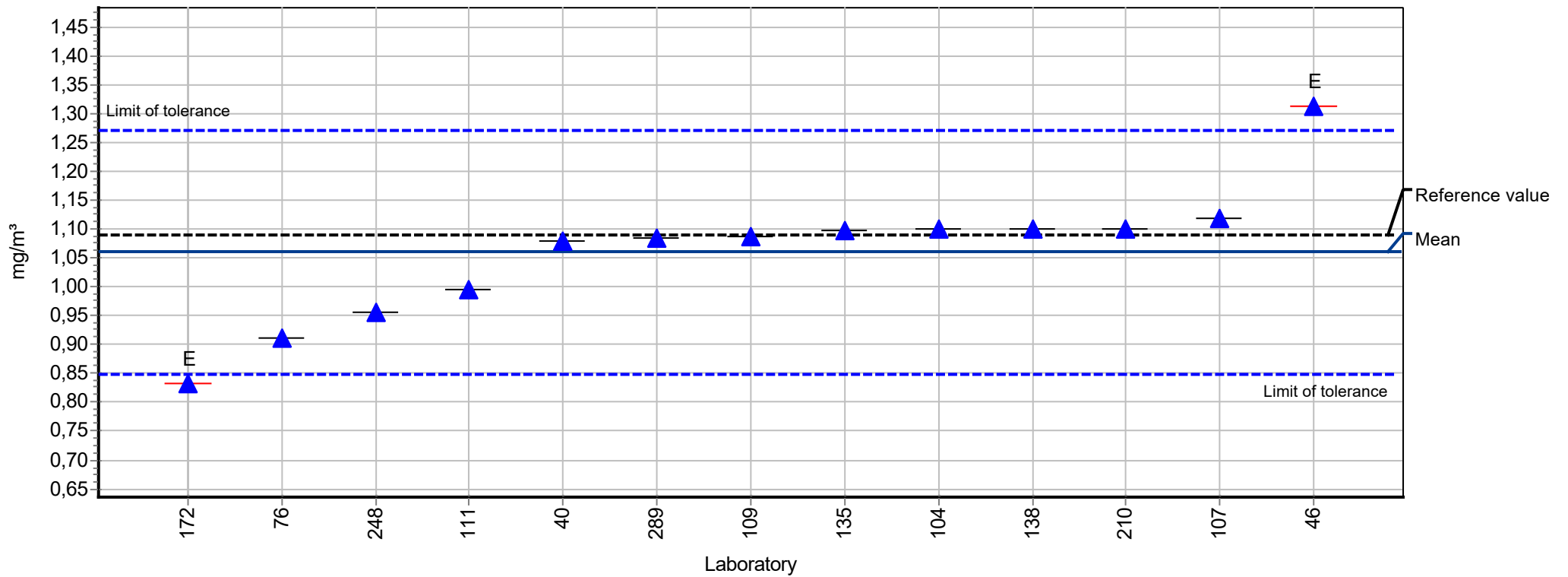
## Summary results

Measurand:	Acetaldehyde	Mean:	0,973 mg/m <sup>3</sup>
Sample:	2	Reprod. s.d.:	0,077 mg/m <sup>3</sup>
Method:	ISO 5725-2	Rel.reprod. s.d.:	7,95%
Rel.target s.d.:	10,00% (Limited)	Reference value:	0,960 mg/m <sup>3</sup>
No. of laboratories:	13	Range of tolerance:	0,778 - 1,168 mg/m <sup>3</sup> ( Z-Score  <= 2,00)



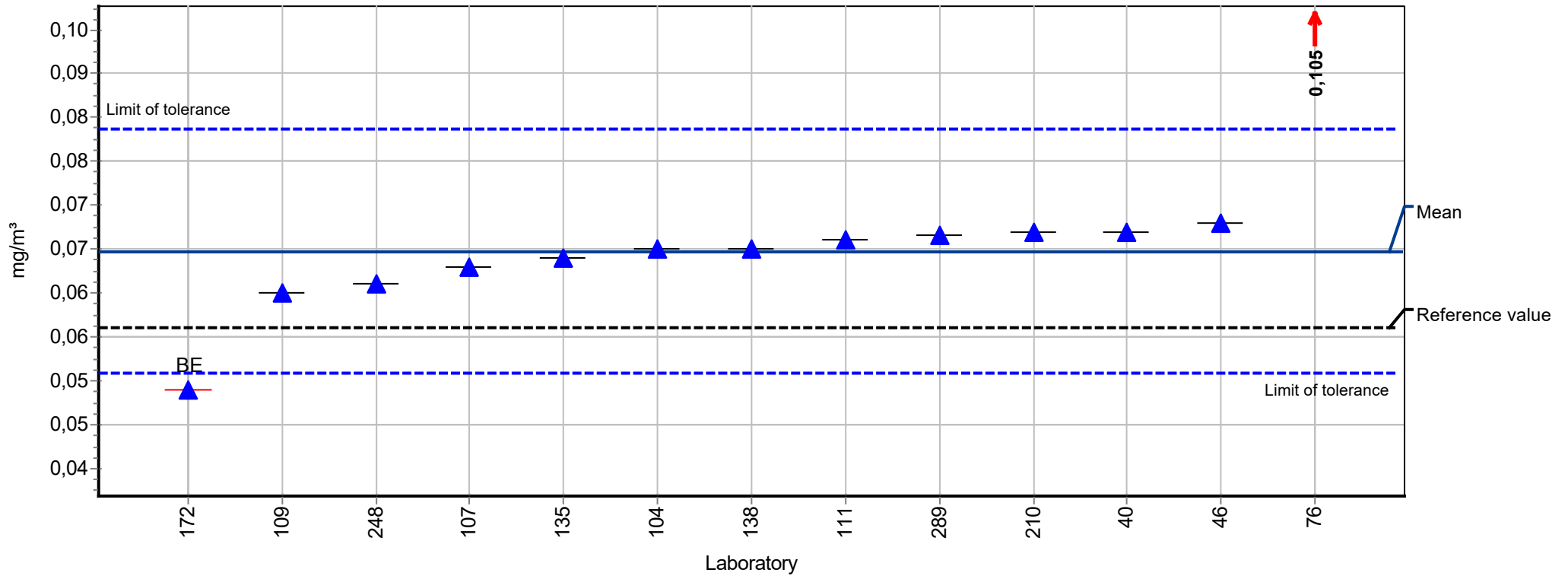
## Summary results

Measurand:	Butyraldehyde	Mean:	1,060 mg/m <sup>3</sup>
Sample:	2	Reprod. s.d.:	0,117 mg/m <sup>3</sup>
Method:	ISO 5725-2	Rel.reprod. s.d.:	11,07%
Rel.target s.d.:	10,00% (Limited)	Reference value:	1,090 mg/m <sup>3</sup>
No. of laboratories:	13	Range of tolerance:	0,848 - 1,272 mg/m <sup>3</sup> ( Z-Score  <= 2,00)



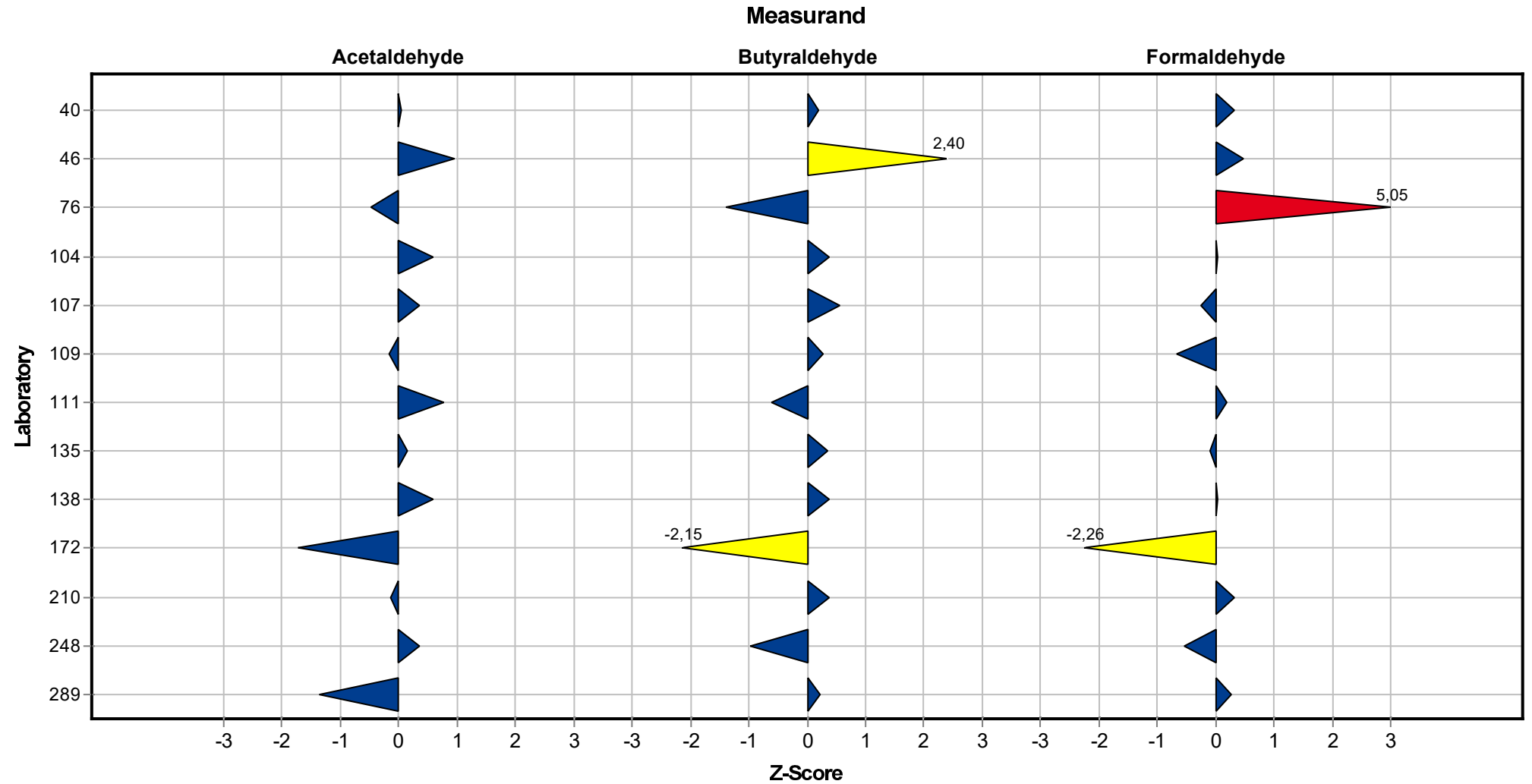
## Summary results

Measurand:	Formaldehyde	Mean:	0,070 mg/m <sup>3</sup>
Sample:	2	Reprod. s.d.:	0,003 mg/m <sup>3</sup>
Method:	ISO 5725-2	Rel.reprod. s.d.:	3,68%
Rel.target s.d.:	10,00% (Limited)	Reference value:	0,061 mg/m <sup>3</sup>
No. of laboratories:	11	Range of tolerance:	0,056 - 0,084 mg/m <sup>3</sup> ( Z-Score  <= 2,00)



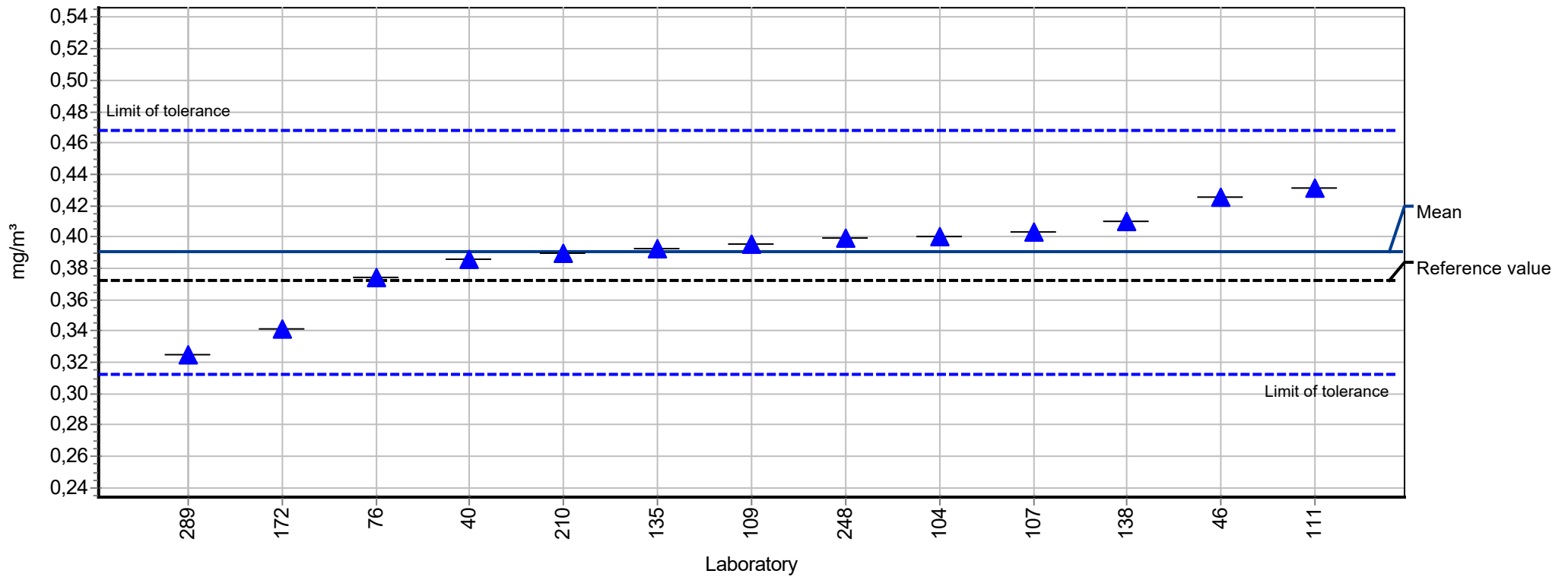
# Sample chart of Z-scores

Sample 2



## Summary results

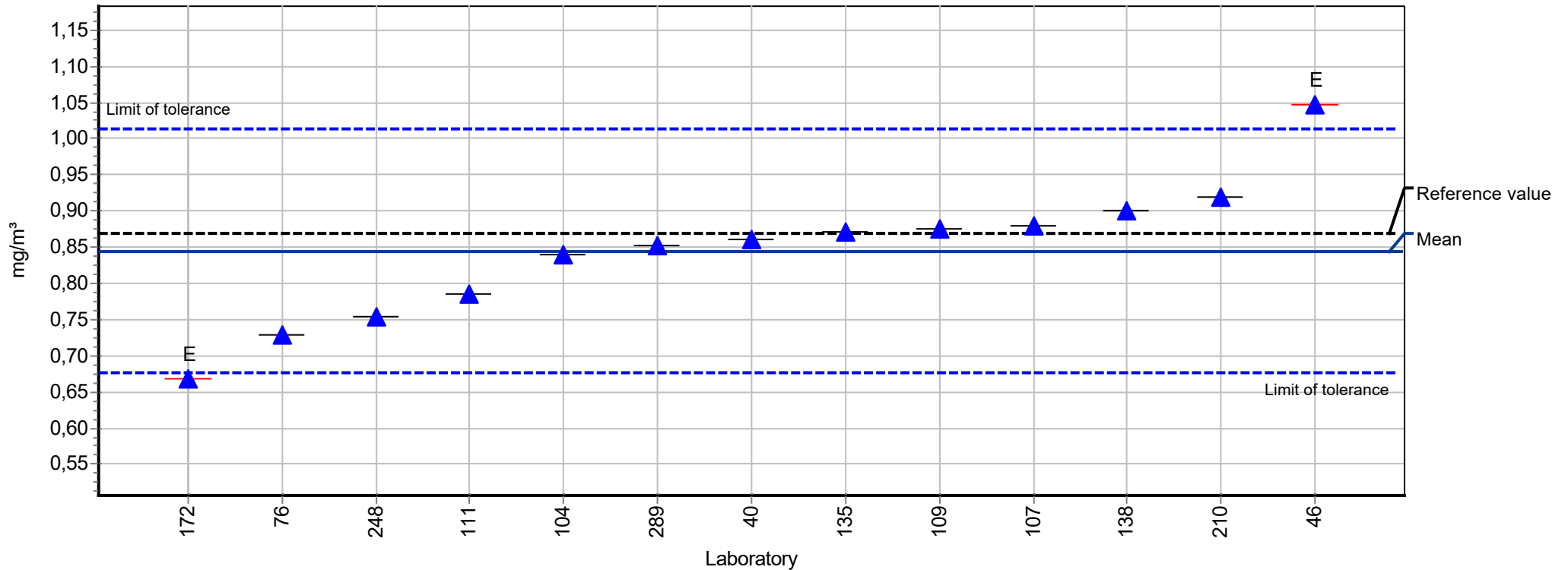
Measurand:	Acetaldehyde	Mean:	0,390 mg/m <sup>3</sup>
Sample:	3	Reprod. s.d.:	0,030 mg/m <sup>3</sup>
Method:	ISO 5725-2	Rel.reprod. s.d.:	7,61%
Rel.target s.d.:	10,00% (Limited)	Reference value:	0,372 mg/m <sup>3</sup>
No. of laboratories:	13	Range of tolerance:	0,312 - 0,468 mg/m <sup>3</sup> ( Z-Score  <= 2,00)





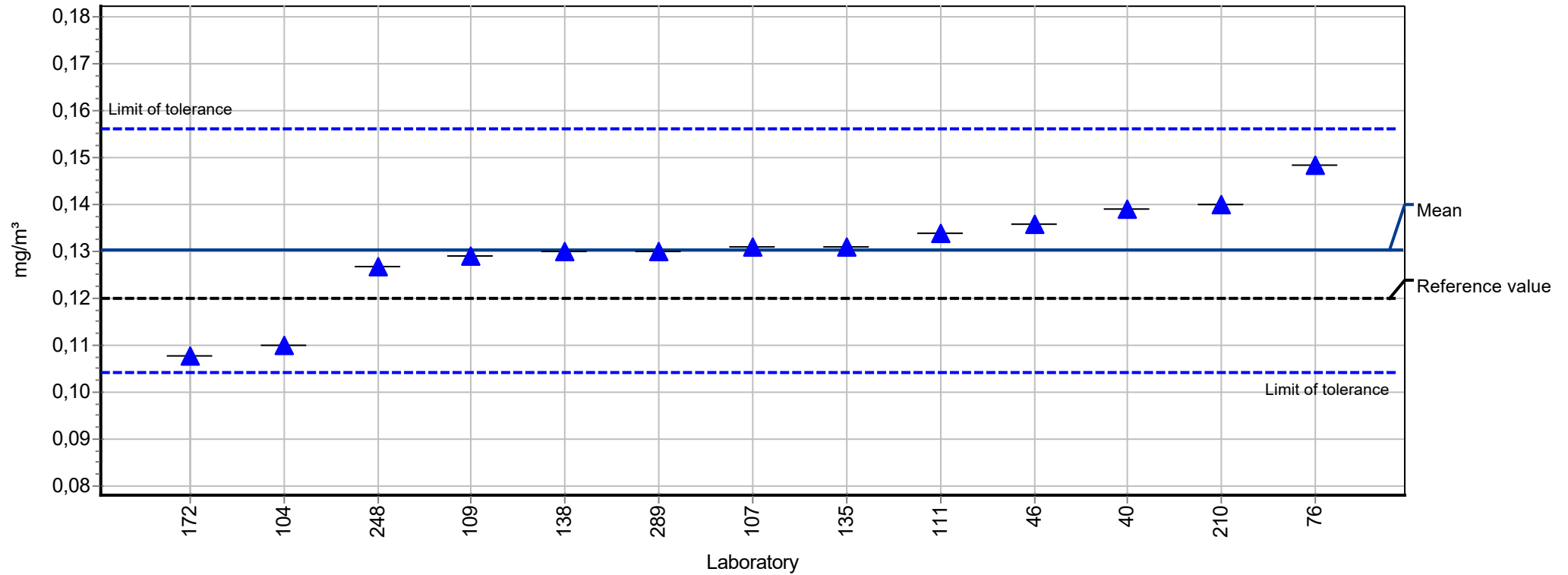
## Summary results

Measurand:	Butyraldehyde	Mean:	0,845 mg/m <sup>3</sup>
Sample:	3	Reprod. s.d.:	0,095 mg/m <sup>3</sup>
Method:	ISO 5725-2	Rel.reprod. s.d.:	11,27%
Rel.target s.d.:	10,00% (Limited)	Reference value:	0,870 mg/m <sup>3</sup>
No. of laboratories:	13	Range of tolerance:	0,676 - 1,014 mg/m <sup>3</sup> ( Z-Score  <= 2,00)



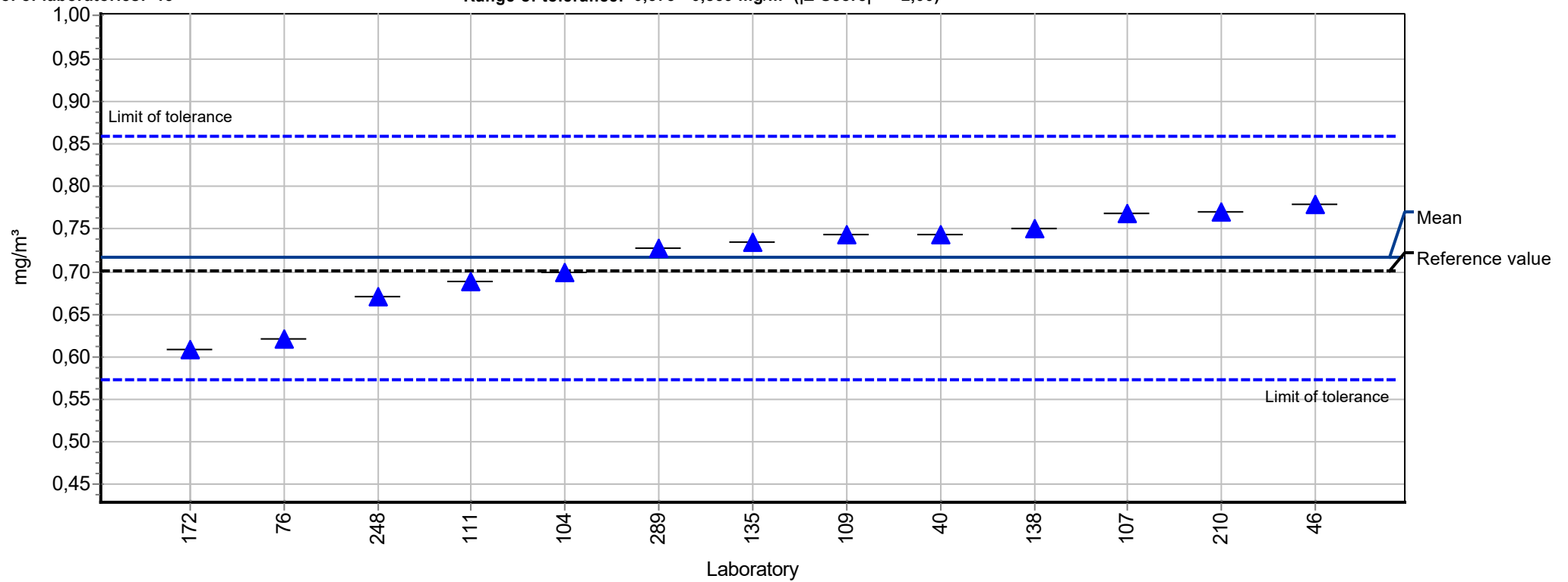
## Summary results

Measurand:	Formaldehyde	Mean:	0,130 mg/m <sup>3</sup>
Sample:	3	Reprod. s.d.:	0,011 mg/m <sup>3</sup>
Method:	ISO 5725-2	Rel.reprod. s.d.:	8,51%
Rel.target s.d.:	10,00% (Limited)	Reference value:	0,120 mg/m <sup>3</sup>
No. of laboratories:	13	Range of tolerance:	0,104 - 0,156 mg/m <sup>3</sup> ( Z-Score  <= 2,00)



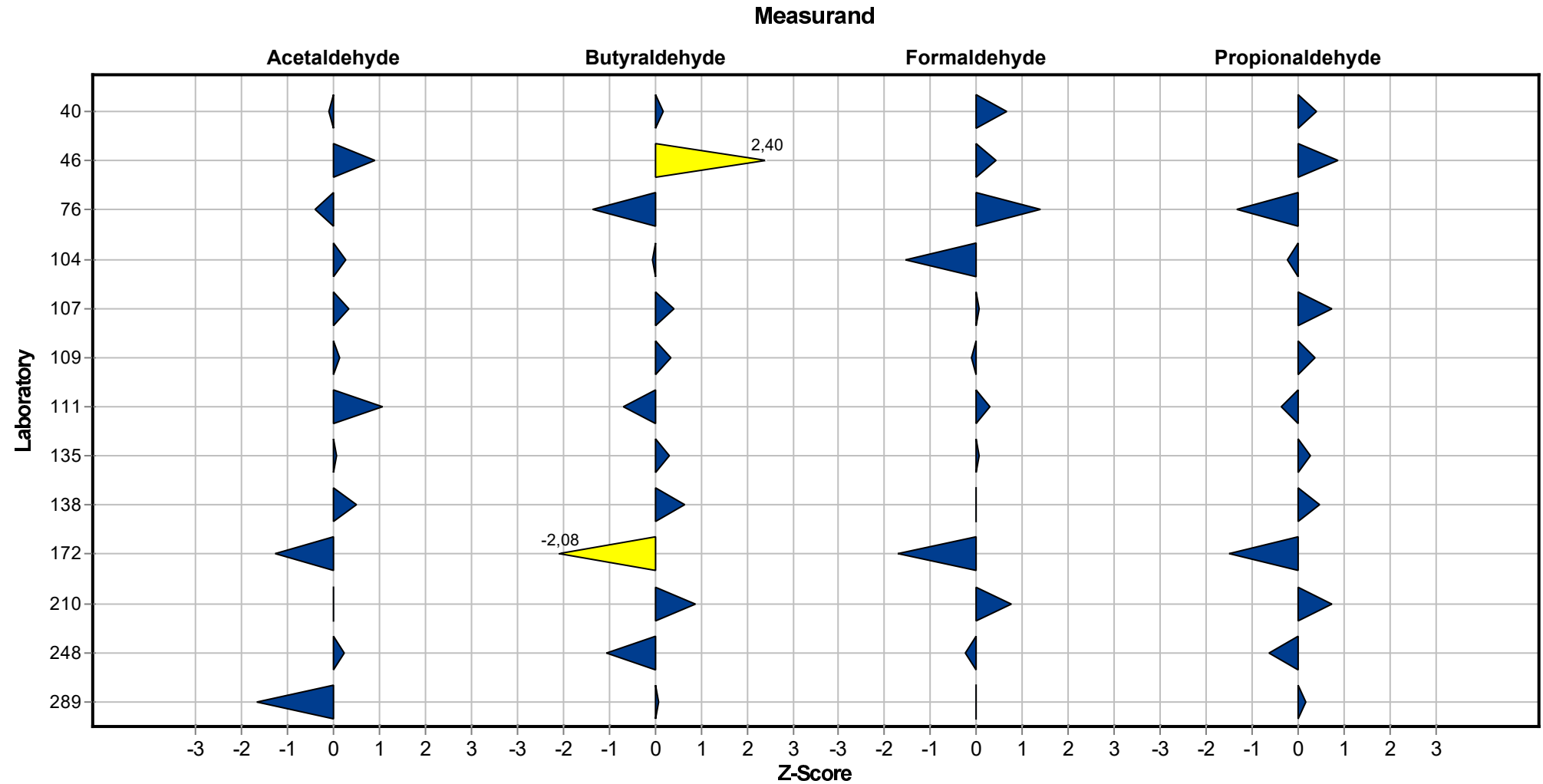
## Summary results

Measurand:	Propionaldehyde	Mean:	0,716 mg/m <sup>3</sup>
Sample:	3	Reprod. s.d.:	0,055 mg/m <sup>3</sup>
Method:	ISO 5725-2	Rel.reprod. s.d.:	7,70%
Rel.target s.d.:	10,00% (Limited)	Reference value:	0,701 mg/m <sup>3</sup>
No. of laboratories:	13	Range of tolerance:	0,573 - 0,859 mg/m <sup>3</sup> ( $ Z\text{-Score}  \leq 2,00$ )



# Sample chart of Z-scores

Sample 3



## Questions and Answers

Participant	Type of sample carrier	Sampling pump
40	SEP-Pak DNPH-Silica Kartusche Fa. Watersn WAT037500	Gilian LFS-111 DC
46	DNPH	Desaga und Hohlbach
76	SKC 226-119 DNPH-impr. Silicagel	Gilian LFS-113DC bzw . Buchholz-ISM-Absaugung mit Gasuhr
104	DNPH-Kartuschen von Supelco LpDNPH S 10	Bravo M2 bzw . Gilian GilAirPlus
107	Supelco-DNPH Kartuschen	Dionex Ultimate 3000
109	Supelco LpDNPH S10	Gillian Gilair Plus
111	Supelco DNPH-Kartusche	Sensidyne Gilair Plus
135	SUPELCO Lp DNPH S10	Holbach BiVOC2
138	SKC 226-118	SKC PCXR-8
172	DNPH	BiVOC2
210	Supelco S10	SKC PCXR8
248	Waters Sep-Pak Xposure	GSA, Typ SG 350
289	DNPH SKC 226-120	Gilian, GilAir Plus

Participant	Volume flow	Volume flow measurement	Sampling time
40	190 ml/Minute	Bios Defender 510	90 Minuten bis 120 Minuten
46	1,0 L/min	Blasenflow meter	20 Minuten
76	0,3 l/min und 0,8 l/min	DryCal Lite bzw . kalibrierte Gasuhr	2,0 h
104	1,5 l (Bravo) bzw . 1,5 l (Gilian)	Drycal DC-LITE KIT	10 - 55 min
107	0,5l/min ; 0,7L/min	Meslabs Definer 220 Series	60 min, 30 min
109	0,35 L/Min	MesaLabs Defender 530	114 Minuten
111	0,3 l/min und 0,8 l/min	Bios Drycal DC Lite	60 min
135	1,0 l/min bzw . 0,5 l/min	Interner Massenstromsensor + externe Kontrolle mit Sensidyne Gilibrator 2	60 Minuten
138	0,5 - 1 l/min	Massendurchflussmesser Analyt	120 Minuten
172	2 l/min		30 min
210	1,0 Liter pro Minute	TSI 4146	10 - 40 Minuten
248	ca. 0,2 l/min	Defender, Typ 530 (0,05 - 5 l/min)	45 Minuten
289	1L/min	Gilian, Gilibrator 2	60 min

**Proficiency testing scheme Aldehydes with sampling 1/2018**

Participant	Analytical method	Date start sample preparation	Storage time after desorption
40	HPLC	12.10.2018	2 Tage im Kühlschrank
46		17.10.2018	Nein
76	IFA 6045 XI/07 bzw . VDI 3862 Blatt 2	16.10.2018	keine Lagerung
104	DIN EN ISO 16000-3:2013	22.10.2018	Nach Desorption 1 Tag im Kühlschrank gelagert
107	Hausmethode in Anlehnung an IFA Methode 6045	12.10.2018	nein
109	Hausmethode in Anlehnung an IFA Arbeitsmappe 6045	11.10.2018	1 Tag im Kühlschrank, 5 °C
111	IFA 6045	16.10.2018	sofort gemessen bzw im Kühlschrank für Nachmessungen
135	HPLC-DAD IFA Arbeitsmappe 6045	12.10.2018	nein
138	BGIA 6045	18.10.2018	Kühlschrank
172	i.A. DIN ISO 16000-3	11.10.2018	nein
210	DIN ISO 16000-3 (2013-1)	18.10.18	Keine Lagerung, direkte Analyse im Anschluss
248	IFA 6045	15.10.2018	Kühlschrank
289	DIN ISO 16000-3	17/10/2018	keine Lagerung

Participant	Date of analysis	Desorption solution	Volume of desorption solution
40	12.10.2018	Acetonitril	2
46	17.10.2018	ACN	2 mL
76	16.10.2018	Acetonitril	10 ml
104	23.10.2018	Acetonitril	10 ml
107	12.10.2018	Acetonitril	5ml
109	12.10.2018	Acetonitril	10 mL
111	16.10 - 07.11.2018	Acetonitril	5 ml
135	12.10.2018	Acetonitril	2 ml
138	05.11.2018	Acetonitril	10 ml
172	11.10.2018	Acetonitril	5 ml
210	18.10.18	Acetonitril	2 ml
248	26.-29.10.2018	Acetonitril	5 ml
289	17/10/2018	Acetonitril	3 mL

Participant	Chromatography system	Autosampler
40	Agilent HP 1200	Raumtemperatur

**Proficiency testing scheme Aldehydes with sampling 1/2018**

Participant	Chromatography system	Autosampler
46	Quat. Pumpe, DAD, Autosampler	Nein
76	HPLC Gradientensystem mit DAD-Detektor Typ 1200 Series (Agilent) DAD- Bereich 200-450 nm	nicht temperierter Autosampler
104	HP Series 1090 LC-DAD	kein gekühlter Autosampler
107	Dionex, Ultimate 3000	nein
109	Pumpe: Shimadzu LC20-AD, Detektor: SPD-M20A	ohne Kühlung
111	HPLC Thermo UltiMate 3000 / Photodiodenarraydetektor Thermo DAD-3000	Nein
135	Agilent 1290 Series	Ja, 10 °C
138	ThermoFisher	
172	HPLC/DAD 20 A Shimadzu-System	nein
210	HPLC Dionex U-3000, Pumpe: LPG-3400 SD, Detektor: DAD-3000, Autosampler: WPS-3000SL	kein gekühlter Autosampler
248	Chromaster	nein, klimatisierter Laborraum 23°C
289	quadratische Pumpe, UV Detektor	Ja/nein Temperaturregler

Participant	Analytical column	Flow rate HPLC
40	LiChrospher 100 RP 18 e, 125 mm * 4 mm, 5 µm, Fa. Merck	1 ml/min
46	C18	1 mL/min
76	Eclipse XDB-C18, 4,6 x 50 nm, 1,8 µm, 600 bar, Agilent	2,0 ml/min
104	Reprosil-Pur C18-AQ, 5 µ, 250*4,6 mm	0,6 ml/min
107	Supelcosil LC 18; 25x 4,6 mm	0,6ml/min
109	Kinetex RP18 5µm 100Å 250*4,6 mm	1,0 mL/Min
111	Phenomenex Synergy Max-RP80A 250x4,6mm 4µm	0,8 ml/min
135	M&N EC 250/4.6 Nucleodur 100-5 C18ec	2,25 ml/min
172		1 ml/min
210	Restek Allure AK 5µm, 200x4.6mm	1,5 ml/min
248	Purospher Star RP-18, 5 im	0,65 ml/min
289	C18 5 µm 150*4,6 mm	1 ml/min

Participant	Mobile phase	Wavelength	Column temperature
40	Acetonitril/Wasser (Gradient)	365 nm	40 °C
46	ACN und Wasser	265 nm	40 °C

**Proficiency testing scheme Aldehydes with sampling 1/2018**

Participant	Mobile phase	Wavelength	Column temperature
76	Eluent A: Wasser / Eluent B: Acetonitril:THF (80/20)	DAD- Bereich 200-450 nm	32 °C
104	Eluent A: MeOH/H2O/Acetonitril 52/30/18; Eluent B: MeOH/H2O/Acetonitril 52/15/33	365,8 nm	Raumtemperatur
107	Wasser /ACN	365nm	30 °C
109	Acetonitril / Wasser (75:25)	365 nm	40 °C
111	Isokratisch 65 % Acetonitril, 35 % Wasser	365 nm	20 °C
135	Wasser-Acetonitril-Tetrahydrofuran	365 nm	45 °C
172		360 nm	25 °C
210	Acetonitril/ Wasser	360 nm	30 Grad
248	Acetonitril, Wasser, THF	360 nm	50°C
289	Wasser (30) / ACN (70)	360 nm	20°C

Participant	Recovery rate
40	ja
46	Nein
76	ja
104	nein
107	nein
109	nein
111	nein
135	nein
172	nein
210	Nein, Wiederfindungsraten wurden nicht berücksichtigt
248	ja
289	Nein