

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

Proficiency testing scheme aldehydes with own sampling

25 - 26 October 2023

Summary of laboratory test results

Sample 1

	Acetaldehyde	Z score	Butyraldehyde	Z score	Formaldehyde	Z score	Propionaldehyde	Z score
Unit	mg/m ³		mg/m ³		mg/m ³		mg/m ³	
23	0,257	0,26	0,658	-1,00	0,101	0,09	0,540	0,10
33	0,254	0,16	0,694	-0,51	0,104	0,36	0,573	0,72
36	0,252	0,06	0,752	0,29	0,099	-0,11	0,541	0,12
61	0,240	-0,42	0,790	0,81	0,096	-0,38	0,490	-0,83
134	0,258	0,30	0,806	1,03	0,106	0,59	0,576	0,78
158	0,249	-0,06	0,709	-0,30	0,098	-0,21	0,528	-0,12
222	0,251	0,02	0,746	0,21	0,100	-0,01	0,527	-0,14
236	0,230	-0,82	0,690	-0,56	0,080	-2,01 BE	0,510	-0,46
267	0,256	0,22	0,759	0,39	0,096	-0,37	0,537	0,04
297	0,258	0,28	0,704	-0,37	0,100	0,02	0,523	-0,21
–	–	--	–	--	–	--	–	--
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	10		10		10		10	
Mean	0,250		0,731		0,100		0,534	
Reprod. s.d.	0,009		0,047		0,003		0,026	
Rel. reproducibility s.d.	3,59 %		6,48 %		3,21 %		4,87 %	
Reference value	0,223		0,655		0,086		0,488	
Target s.d.	0,025		0,073		0,010		0,053	
Rel. target s.d.	10,00 %		10,00 %		10,00 %		10,00 %	
Lower limit of tolerance	0,200		0,585		0,080		0,428	
Upper limit of tolerance	0,301		0,877		0,120		0,641	
Type B outliers					1			
No. of laboratories after elimination of outliers type A-D and F (without laboratories that only gave states but no measured values)	10		10		9		10	
Explanation of outlier types								

	Acetaldehyde	Z score	Butyraldehyde	Z score	Formaldehyde	Z score	Propionaldehyde	Z score
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 >3,50								

Summary of laboratory test results

Sample 2

	Acetaldehyde	Z score	Formaldehyde	Z score	Propionaldehyde	Z score
Unit	mg/m ³		mg/m ³		mg/m ³	
23	0,088	0,00	0,189	0,24	0,368	0,08
33	0,091	0,37	0,184	-0,03	0,369	0,12
36	0,087	-0,11	0,188	0,18	0,374	0,24
61	0,087	-0,15	0,177	-0,43	0,347	-0,50
134	0,089	0,12	0,196	0,61	0,392	0,74
158	0,088	0,00	0,184	-0,03	0,364	-0,03
222	0,084	-0,45	0,180	-0,25	0,359	-0,17
236	0,070	-2,04 BE	0,150	-1,88 B	0,350	-0,41
267	0,088	-0,01	0,178	-0,36	0,364	-0,02
297	0,090	0,23	0,186	0,07	0,363	-0,05
–	–	--	–	--	–	--
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	10		10		10	
Mean	0,088		0,185		0,365	
Reprod. s.d.	0,002		0,006		0,013	
Rel. reproducibility s.d.	2,36 %		3,26 %		3,46 %	
Reference value	0,080		0,162		0,338	
Target s.d.	0,009		0,018		0,037	
Rel. target s.d.	10,00 %		10,00 %		10,00 %	
Lower limit of tolerance	0,070		0,148		0,292	
Upper limit of tolerance	0,106		0,222		0,438	
Type B outliers	1		1			
No. of laboratories after elimination of outliers type A-D and F (without laboratories that only gave states but no measured values)	9		9		10	
Explanation of outlier types						

	Acetaldehyde	Z score	Formaldehyde	Z score	Propionaldehyde	Z score
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 >3,50						

Summary of laboratory test results

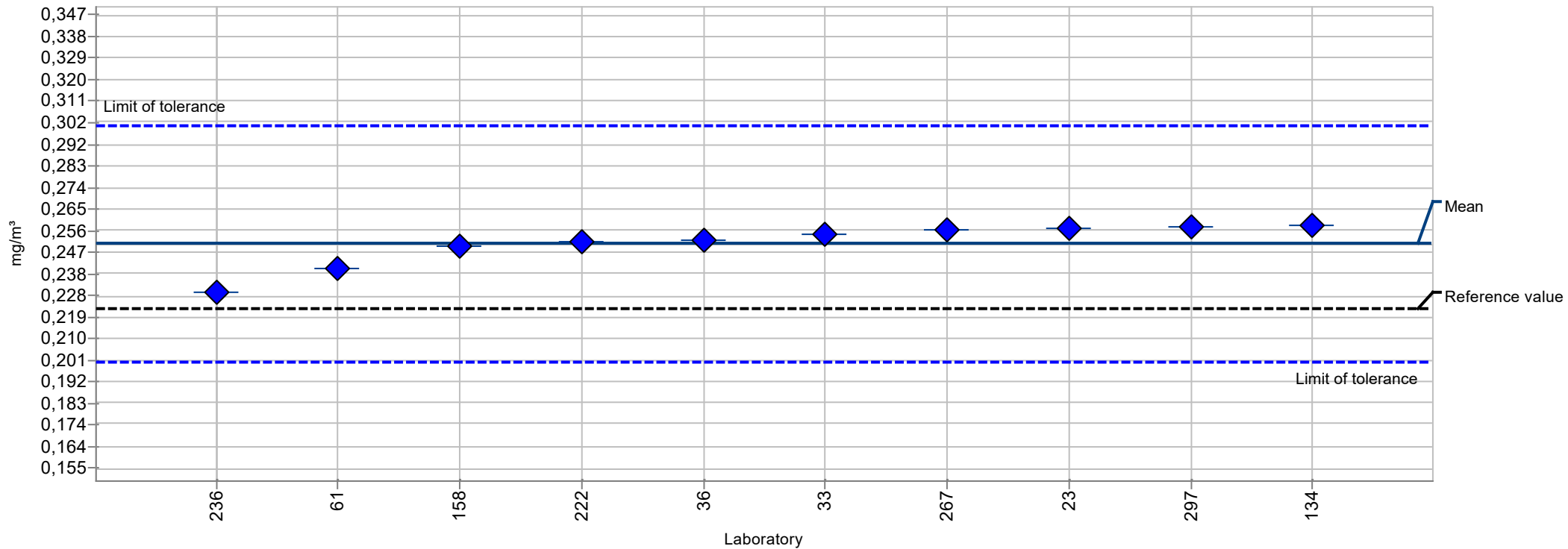
Sample 3

	Acetaldehyde	Z score	Butyraldehyde	Z score	Formaldehyde	Z score
Unit	mg/m ³		mg/m ³		mg/m ³	
23	0,154	0,19	0,953	-0,90	0,250	0,51
33	0,150	-0,08	0,994	-0,51	0,238	0,01
36	0,150	-0,07	1,089	0,40	0,248	0,43
61	0,140	-0,73 B	1,100	0,50	0,220	-0,75
134	0,154	0,19	1,165	1,12	0,260	0,93
158	0,150	-0,07	1,023	-0,23	0,242	0,18
222	0,149	-0,14	1,070	0,21	0,240	0,09
236	0,130	-1,40 B	1,020	-0,26	0,200	-1,59
267	0,149	-0,13	1,051	0,03	0,236	-0,09
297	0,153	0,11	1,011	-0,35	0,244	0,27
–	–	--	–	--	–	--
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	10		10		10	
Mean	0,151		1,048		0,238	
Reprod. s.d.	0,002		0,061		0,017	
Rel. reproducibility s.d.	1,41 %		5,81 %		7,09 %	
Reference value	0,137		0,941		0,205	
Target s.d.	0,015		0,105		0,024	
Rel. target s.d.	10,00 %		10,00 %		10,00 %	
Lower limit of tolerance	0,121		0,838		0,190	
Upper limit of tolerance	0,181		1,257		0,285	
Type B outliers	2					
No. of laboratories after elimination of outliers type A-D and F (without laboratories that only gave states but no measured values)	8		10		10	
Explanation of outlier types						

	Acetaldehyde	Z score	Butyraldehyde	Z score	Formaldehyde	Z score
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 >3,50						

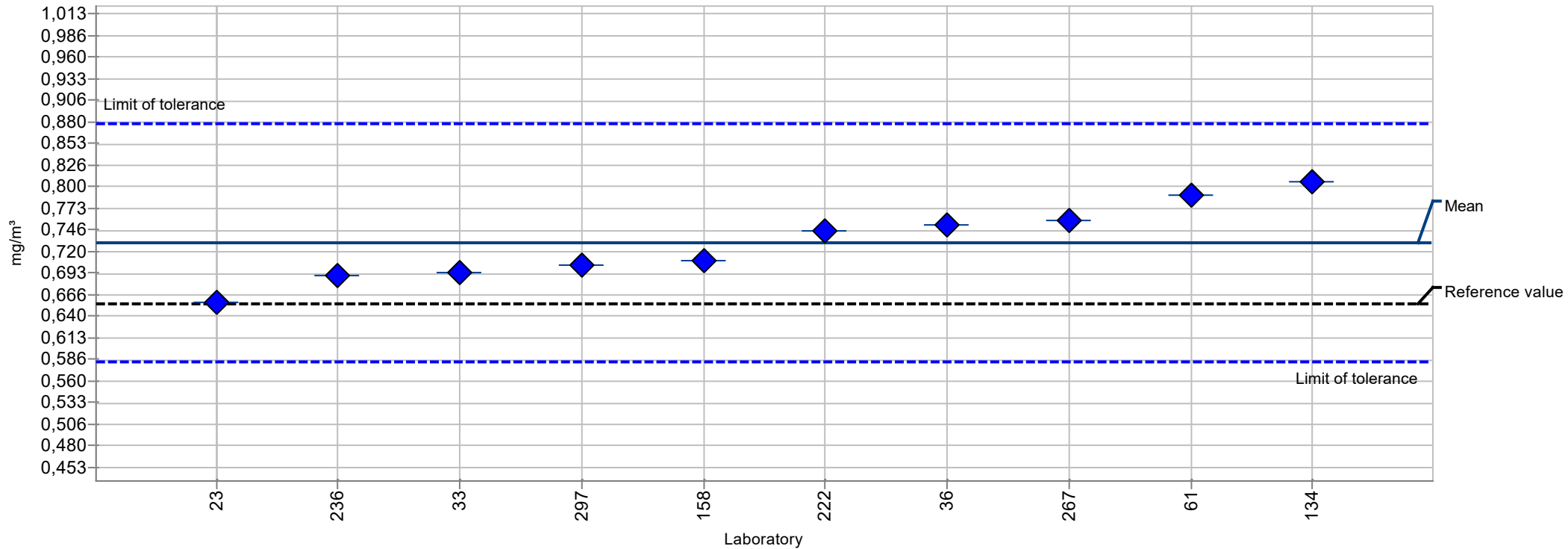
Summary results

Measurand:	Acetaldehyde	Mean:	0,250 mg/m ³
Sample:	Sample 1	Reprod. s.d.:	0,009 mg/m ³
Method:	ISO 5725-2	Rel.reprod. s.d.:	3,59%
Rel.target s.d.:	10,00%	Reference value:	0,223 mg/m ³
Number of laboratories in calculation:	10	Range of tolerance:	0,200 - 0,301 mg/m ³ (Z-Score <= 2,00)



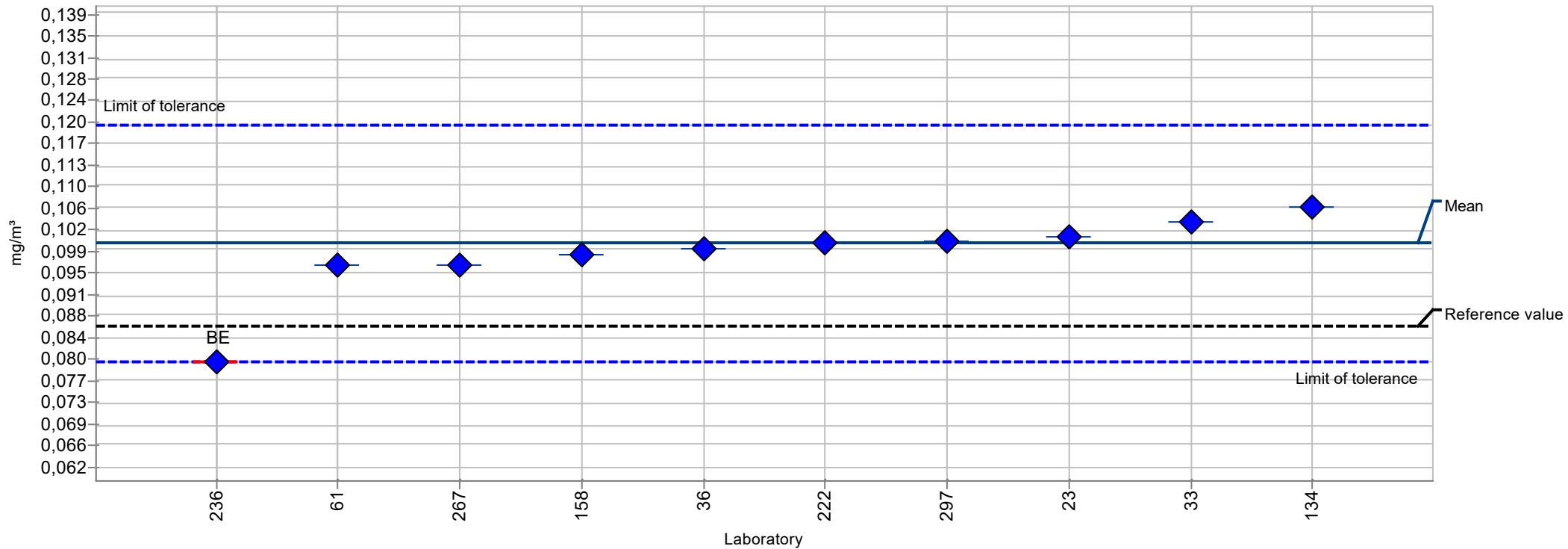
Summary results

Measurand:	Butyraldehyde	Mean:	0,731 mg/m ³
Sample:	Sample 1	Reprod. s.d.:	0,047 mg/m ³
Method:	ISO 5725-2	Rel.reprod. s.d.:	6,48%
Rel.target s.d.:	10,00%	Reference value:	0,655 mg/m ³
Number of laboratories in calculation:	10	Range of tolerance:	0,585 - 0,877 mg/m ³ (Z-Score <= 2,00)



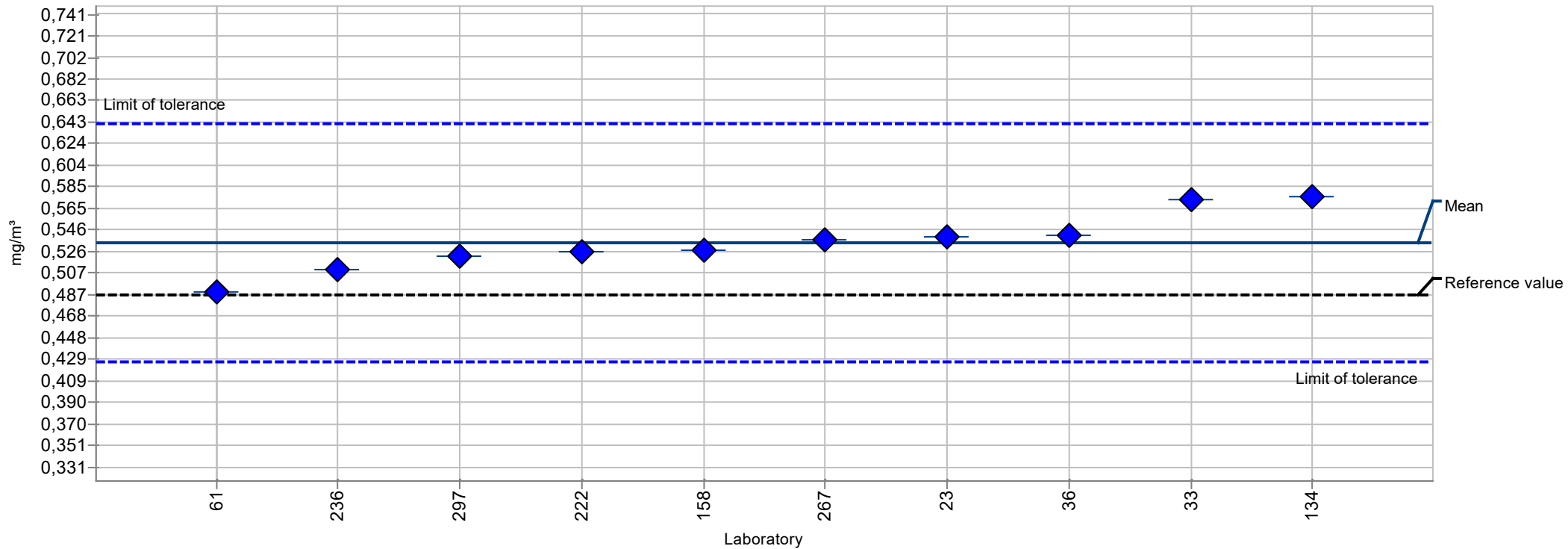
Summary results

Measurand:	Formaldehyde	Mean:	0,100 mg/m ³
Sample:	Sample 1	Reprod. s.d.:	0,003 mg/m ³
Method:	ISO 5725-2	Rel.reprod. s.d.:	3,21%
Rel.target s.d.:	10,00%	Reference value:	0,086 mg/m ³
Number of laboratories in calculation:	10	Range of tolerance:	0,080 - 0,120 mg/m ³ (Z-Score <= 2,00)



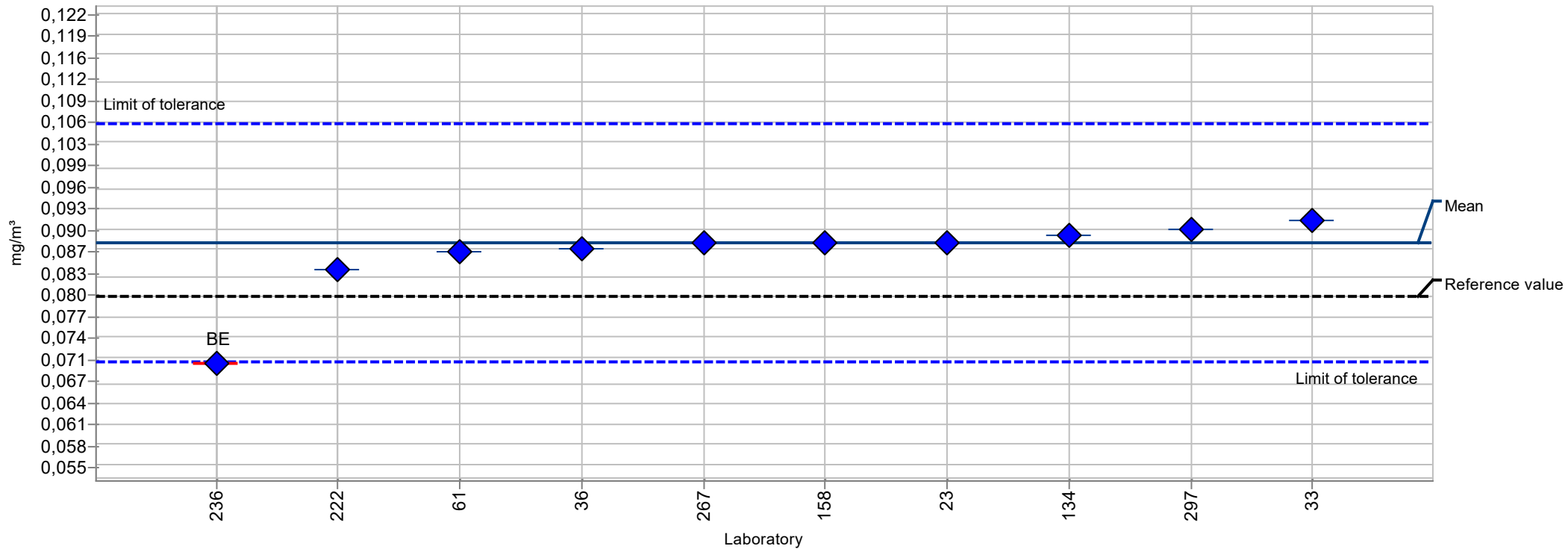
Summary results

Measurand:	Propionaldehyde	Mean:	0,534 mg/m ³
Sample:	Sample 1	Reprod. s.d.:	0,026 mg/m ³
Method:	ISO 5725-2	Rel.reprod. s.d.:	4,87%
Rel.target s.d.:	10,00%	Reference value:	0,488 mg/m ³
Number of laboratories in calculation:	10	Range of tolerance:	0,428 - 0,641 mg/m ³ (Z-Score <= 2,00)



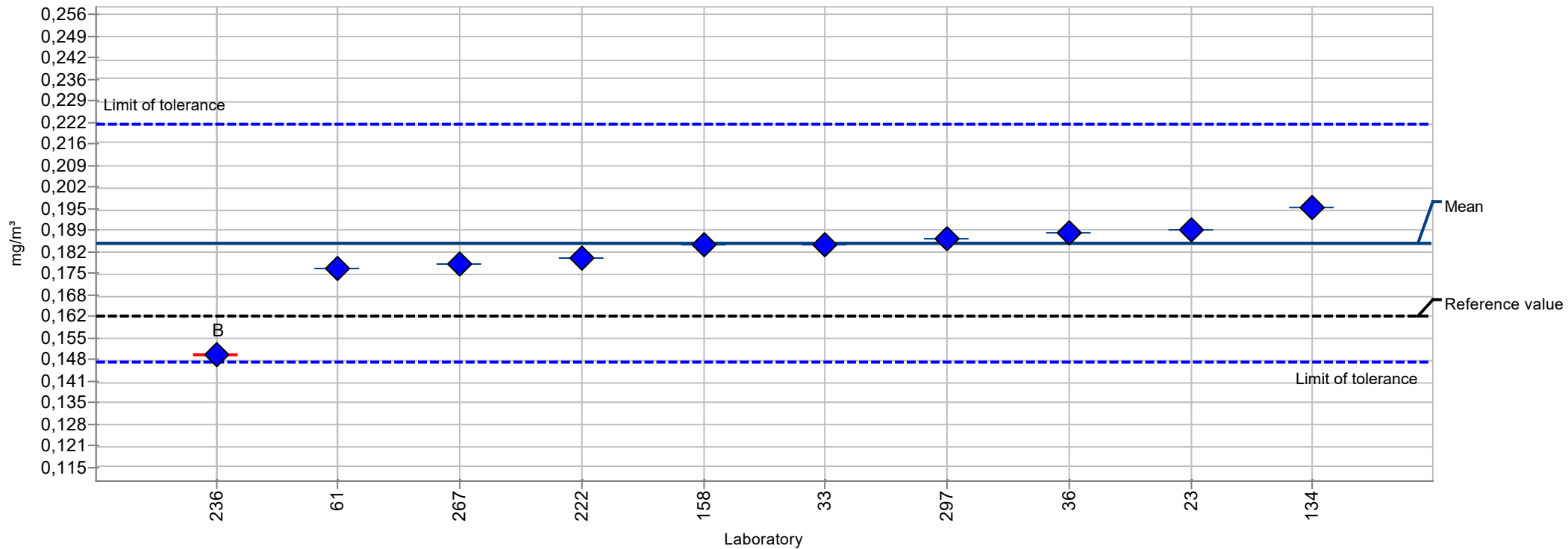
Summary results

Measurand:	Acetaldehyde	Mean:	0,088 mg/m ³
Sample:	Sample 2	Reprod. s.d.:	0,002 mg/m ³
Method:	ISO 5725-2	Rel.reprod. s.d.:	2,36%
Rel.target s.d.:	10,00%	Reference value:	0,080 mg/m ³
Number of laboratories in calculation:	10	Range of tolerance:	0,070 - 0,106 mg/m ³ (Z-Score <= 2,00)



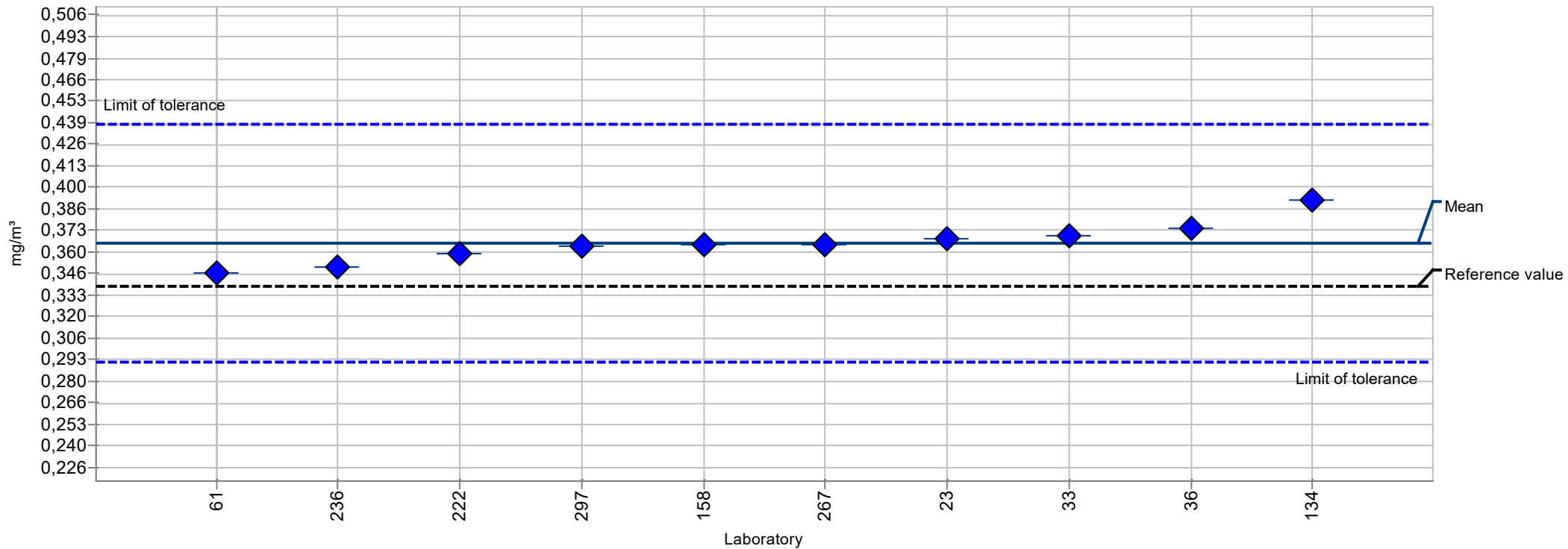
Summary results

Measurand:	Formaldehyde	Mean:	0,185 mg/m ³
Sample:	Sample 2	Reprod. s.d.:	0,006 mg/m ³
Method:	ISO 5725-2	Rel.reprod. s.d.:	3,26%
Rel.target s.d.:	10,00%	Reference value:	0,162 mg/m ³
Number of laboratories in calculation:	10	Range of tolerance:	0,148 - 0,222 mg/m ³ ($ Z\text{-Score} \leq 2,00$)



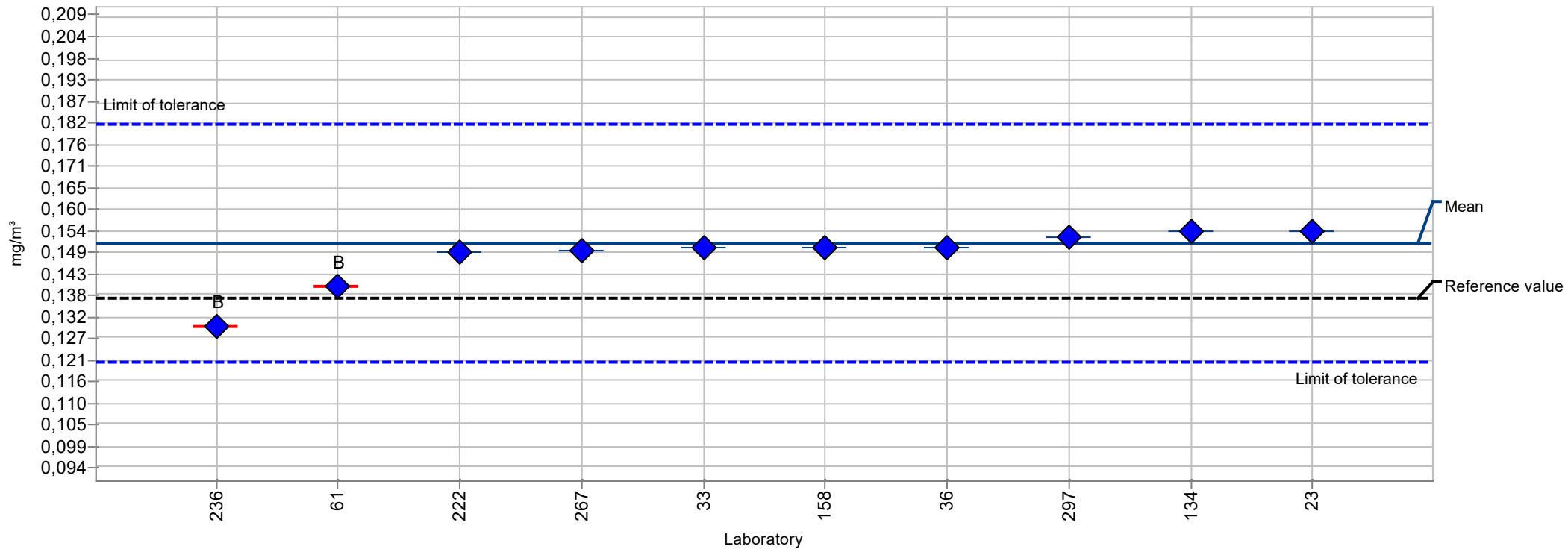
Summary results

Measurand:	Propionaldehyde	Mean:	0,365 mg/m ³
Sample:	Sample 2	Reprod. s.d.:	0,013 mg/m ³
Method:	ISO 5725-2	Rel.reprod. s.d.:	3,46%
Rel.target s.d.:	10,00%	Reference value:	0,338 mg/m ³
Number of laboratories in calculation:	10	Range of tolerance:	0,292 - 0,438 mg/m ³ ($ Z\text{-Score} \leq 2,00$)



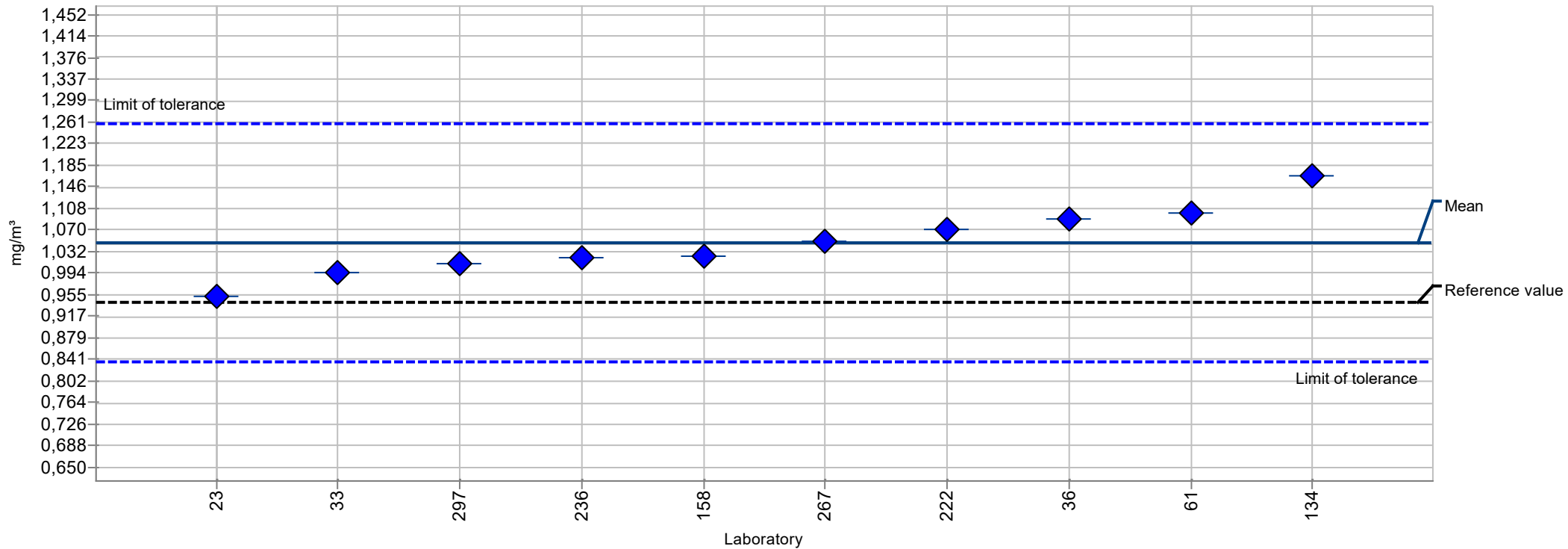
Summary results

Measurand:	Acetaldehyde	Mean:	0,151 mg/m ³
Sample:	Sample 3	Reprod. s.d.:	0,002 mg/m ³
Method:	ISO 5725-2	Rel.reprod. s.d.:	1,41%
Rel.target s.d.:	10,00%	Reference value:	0,137 mg/m ³
Number of laboratories in calculation:	10	Range of tolerance:	0,121 - 0,181 mg/m ³ (Z-Score <= 2,00)



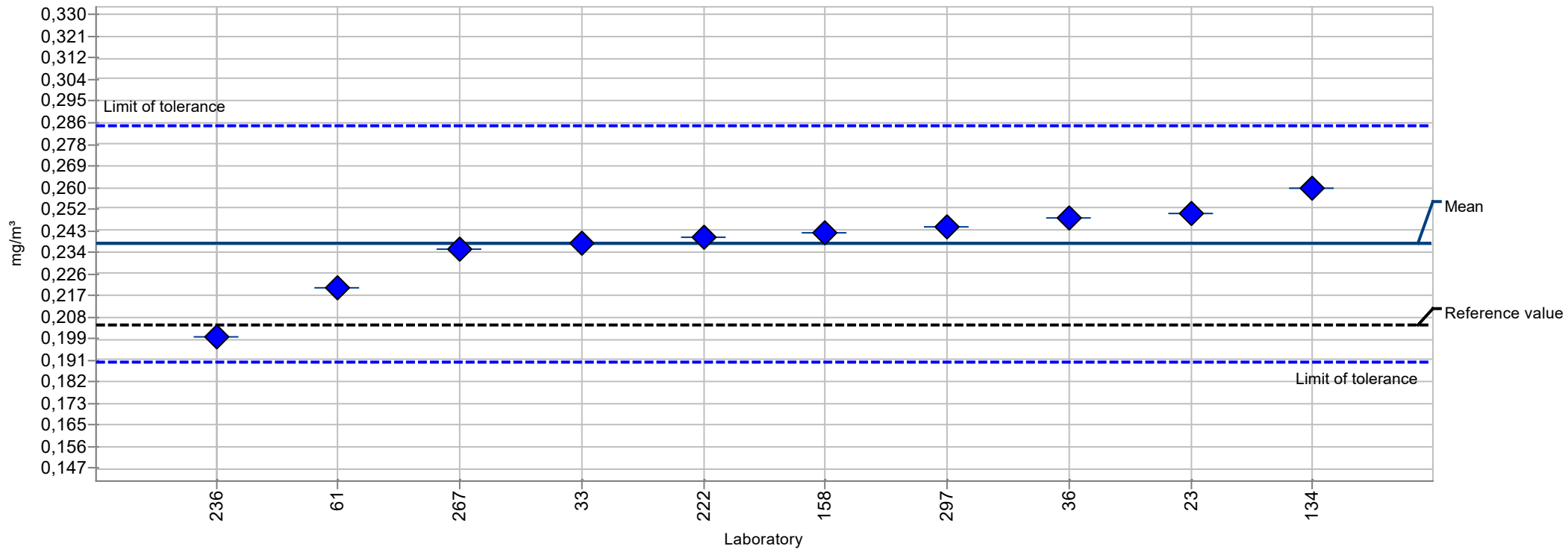
Summary results

Measurand:	Butyraldehyde	Mean:	1,048 mg/m ³
Sample:	Sample 3	Reprod. s.d.:	0,061 mg/m ³
Method:	ISO 5725-2	Rel.reprod. s.d.:	5,81%
Rel.target s.d.:	10,00%	Reference value:	0,941 mg/m ³
Number of laboratories in calculation:	10	Range of tolerance:	0,838 - 1,257 mg/m ³ (Z-Score <= 2,00)



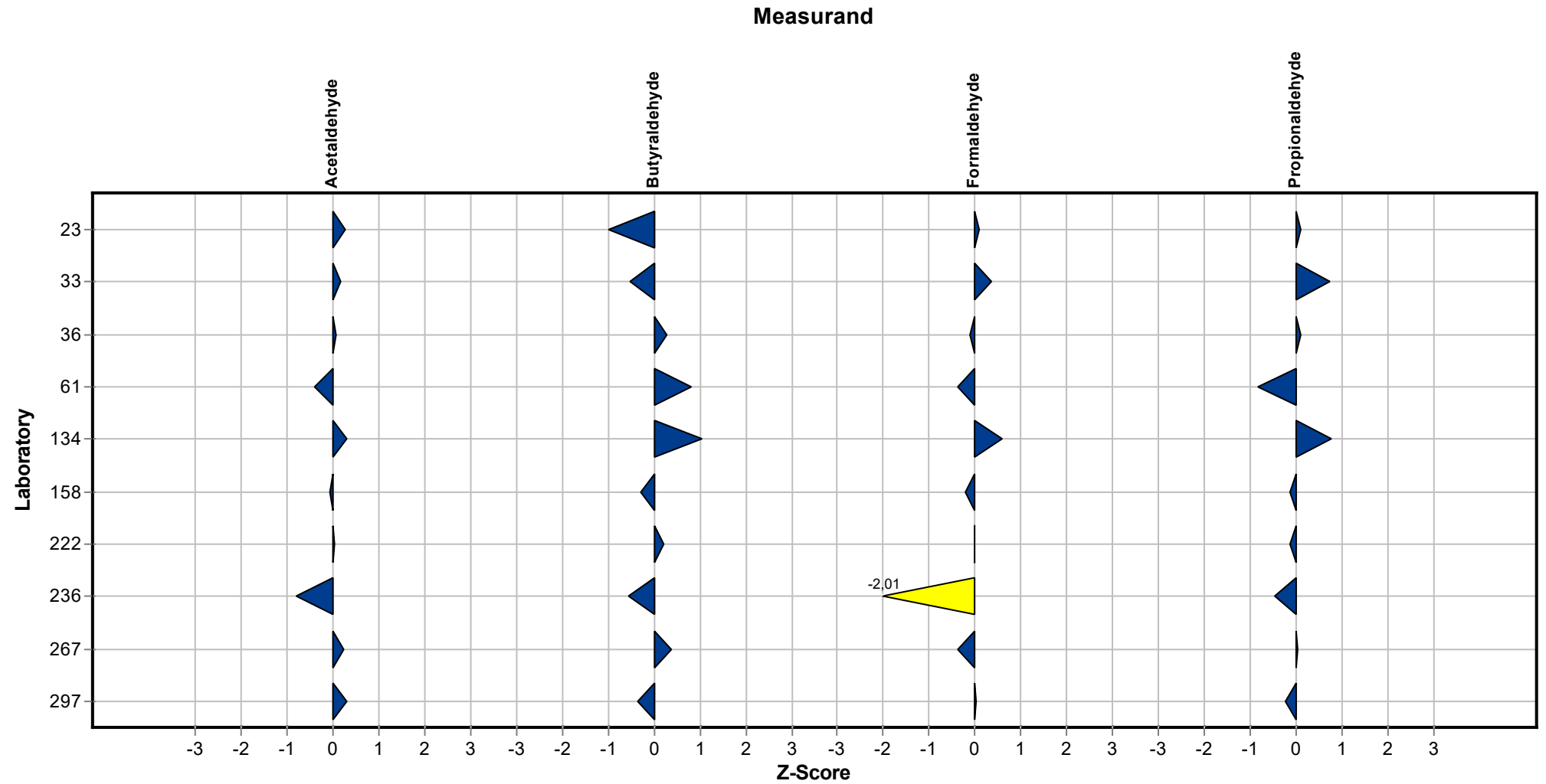
Summary results

Measurand:	Formaldehyde	Mean:	0,238 mg/m ³
Sample:	Sample 3	Reprod. s.d.:	0,017 mg/m ³
Method:	ISO 5725-2	Rel.reprod. s.d.:	7,09%
Rel.target s.d.:	10,00%	Reference value:	0,205 mg/m ³
Number of laboratories in calculation:	10	Range of tolerance:	0,190 - 0,285 mg/m ³ (Z-Score <= 2,00)



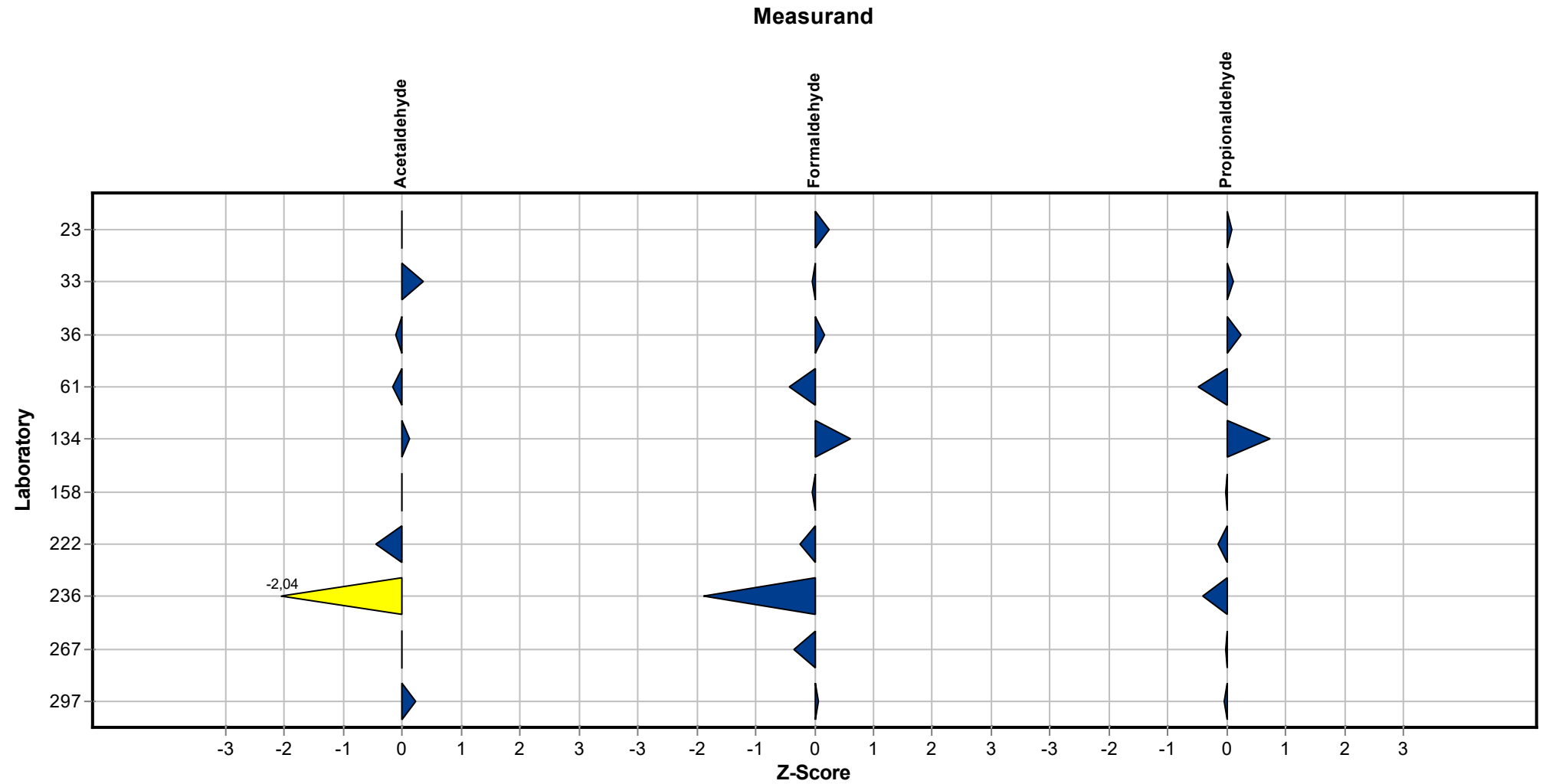
Sample chart of Z-scores

Sample Sample 1



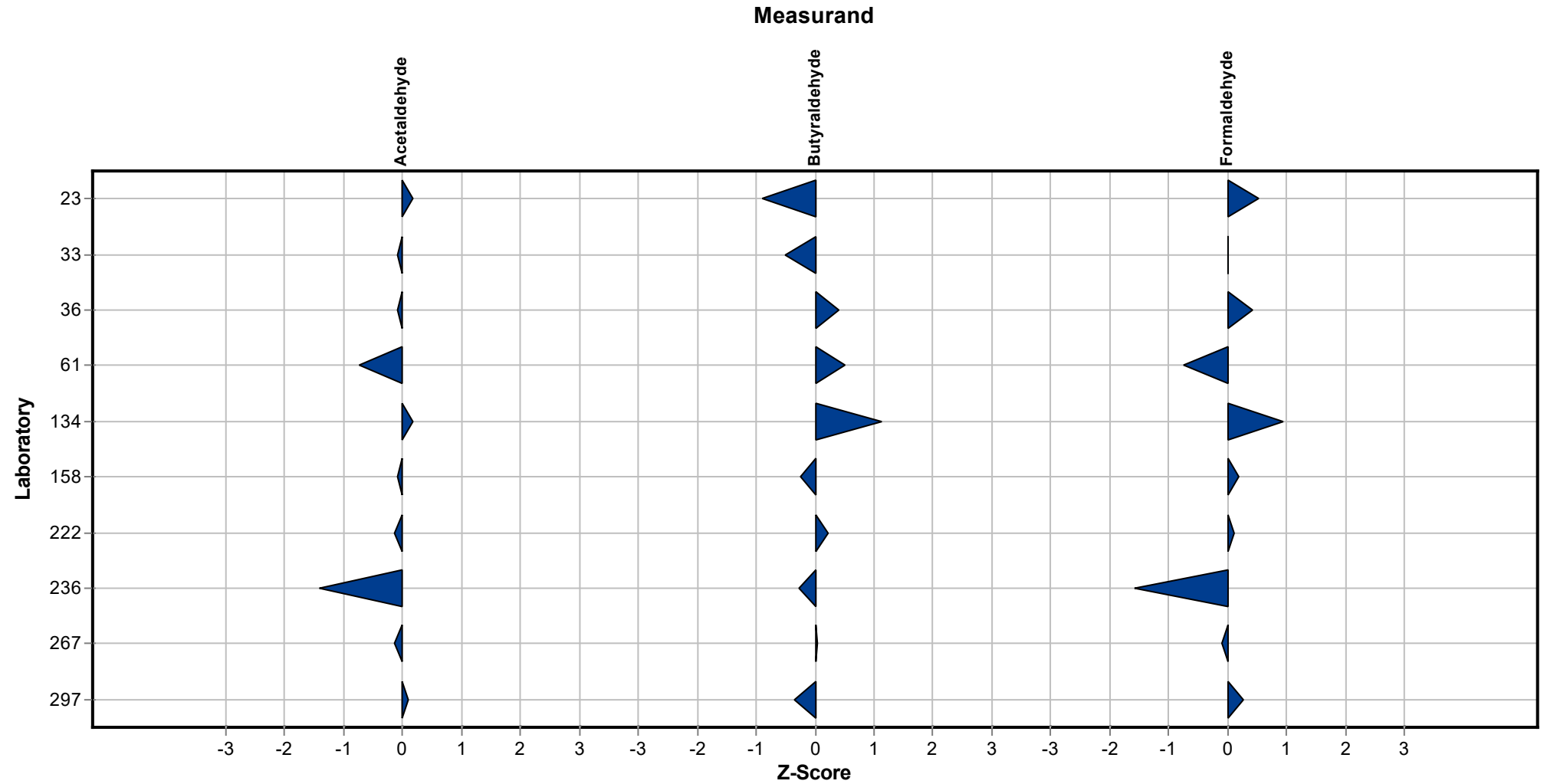
Sample chart of Z-scores

Sample Sample 2



Sample chart of Z-scores

Sample Sample 3



Questions and Answers

Participant	Type of sample carrier	Sampling pump	Volume flow
23	Supelco DNPH Kartuschen	Bivoc	800ml/min
33	Waters DNPH Siicagel WAT 039550	SKC Personal Air Sampler 224PCMTX8	0,1; 0,15; 0,2 L/min
36	SupelClean LPD DNPH Adsorbent Cartridge	Hohlbach BiVOC2 B2	1.2 l/min
61	LpDNPH S10x, 350 mg SPe Tube von Supelco	Bivoc 2	1.2 L/min
134	Supelco LpDNPH S10	SKC 224 PC XR8	0,7-0,8 l/min
158	Supelco LpDNPH S10	Gilian LFS-113DC	0,5 L/min
222	SKC Silicagel Typ DNPH	Sensidyne Gil Air Plus	0,500 l/min.
236	Silicagel (2,4-DNPH)	Sensidyne LFS113	100 mL/min
267	DNPH cartridge réf. 21014, Supelco	Gilian GilAir Plus	1L/min
297	DNPH-Kartusche	BIVOC2	0,16 L/min

Participant	Volume flow measurement	Sampling time
23	BIOS Definer 220	125 min
33	BIOS Defender 510	120 Minuten
36	Bios Definer 220	30:00 min
61	Bivoc 2	33.33 min
134	Flow meter 35810MLW Analyt MTC	40-60min
158	BIOS Defender 510	20 min, 30 min und 40 min
222	Mesa Labs Defender 530-L	45 Minuten
236	MesaLabs DryCal Definer 220	60 Minuten
267	Gilibrator 3 (GilAir)	25 min
297	Rückgeführt durch MesaLabs Defender 520M	125min

Participant	Analytical method
23	i.A. nach ISO 16000-3 (Aldehyde Ketone)
33	IFA 6045 (XI/09)
36	ISO 16000-3
61	Die Sorption erfolgte an Dinitrophenylhydrazin (DNPH)-Kartuschen mit anschließender HPLC-Auswertung der Hydrazonderivate.

Proficiency testing scheme Aldehydes with own sampling 2/2023

Participant	Analytical method
134	DIN ISO 16000-3
158	Bestimmung als Derivate von 2,4- Dinitrophenylhydrazin (2,4-DNPH) mittels Hochleistungs-Flüssigkeits-Chromatografie (HPLC) und UV-Absorption
222	IFA 7520 2007
236	ca. IFA 6045:2007
267	ISO 16000-3
297	DIN ISO 16000-3

Participant	Date start sample preparation
23	30.10.2023
33	30.10.2023
36	04.12.2023
61	30.10.2023
134	07.11.2023
158	27.10.2023
236	31.10.23
267	30/10/2023
297	08.11.2023

Participant	Storage time after desorption
23	nein
33	24 h im Kühlschrank
36	4°C, die Probenlösungen bleiben bis zu 4 Wochen nach Abschluss des Auftrags im Kühlschrank gelagert. Die Proben werden nach Eingang bis zur Entsorgung in einem Kühlschrank gelagert.
61	1 Tag im Kühlschrank bei 4°C
134	nein
158	Nein
236	im KS für 5 d
267	Analyse direkt nach der Desorption, Analyse der Verdünnungen 15 Tage später (Lagerung der Extrakte im Kühlschrank)
297	Raumtemperatur, ca. 22°C

Proficiency testing scheme Aldehydes with own sampling 2/2023

Participant	Date of analysis	Desorption solution	Volume of desorption solution
23	30.10.2023	Acetonitril HPLC	5 ml
33	31.10.23 - 01.11.2023	Acetonitril	5 ml
36	04.12.2023 - 05-12.2023	Acetonitril	5 mL bzw . +1mL auf 10 mL für die hohen Konzentrationen
61	Untersuchungsende 15.11.2023	Acetonitril eluent, verdünnt mit DNPH-Lösung	1 mL
134	08.11.2023	Acetonitril	2ml
158	27. und 28.10.2023	Acetonitril	2 x 2 ml und auf 5 aufgefüllt
222		Acetonitril	
236	31.10.23	Acetonitril	3 mL
267	30/10/2023 (dilutions on 15/11/2023)	Acetonitril	5 mL
297	09.11.2023		2,6

Participant	Chromatography system	Refrigerated autosampler
23	HPLC/DAD 20A Shimadzu System	nein
33	HPLC-Anlage von Agilent, System 1100, BinPumpe G1312A, ALS G1313A, DAD G1315A	nein
36	Binäre Pumpe, PDA Detektor , Thermo Vanquish Horizon Autosampler (mit Kühlfunktion)	Ja, 10°C
61	binäre Pumpe, DAD, Hip-sampler alles von Agilent	nein
134	HPLC System Dionex Ultimate 3000	nein
158	Agilent HPLC mit DAD-UV Detektor	Nein
222	HPLC mit UV-Detektion	
236	quaternäre Pumpe, DAD-Detektor, Standard-ALS	nein
267	Agilent HPLC-DAD 1260	Nein, Raumtemperatur

Participant	Analytical column	Mobile phase
23	Synergie 4µm Hydro-RP 80A LC Column 250x4,6mm	A: Wasser; B: Acetonitril mit 5% Wasser (V/V)
33	Multospher 120 RP 18 AQ-3, 125 * 4 mm, 3 µm	Acetonitril/Wasser
36	Dr.Maisch Grace Grom-Sil ods-5, GromSil 120, 200mm, 3.0mm, 3.0Å, GSOD50312s2002	Acetonitril: Wasser 6:4, Acetonitril: Wasser 95:5, gradient
61	Nucleodur C18 Isis 3 µm	ACN : H2O = 70 : 30; Gradient 95 : 5
134	Allure AK 5µm 200x4,6mm Restek	Acetonitril/ Wasser Gradient
158	Hypersil ODS 4.0 x 250mm 5 Micron	Von 60% Acetonitril bis 100% Acetonitril in 30 min
236	Pursuit 3 C18 100x2.0 mm	Wasser/Acetonitril
267	Waters Symmetry C18, 250 mm x 4.6 mm x 5 µm	Acetonitril/Wasser

Proficiency testing scheme Aldehydes with own sampling 2/2023

Participant	Flow rate HPLC	Wavelength	Column temperature
23	1ml/min	360nm	25 Grad
33	1,2 ml/min	365 nm	Raumtemperatur
36	0.5 mL/min	360 nm, + DAD Array	40°C
61	0.5 mL/min	sig = 365.4; Ref =590.10	30 °C
134	1,5 ml/min	360nm	30°C
158	1,000 ml/min	360 nm	30°C
236	0,2 mL/min	360 nm	33 °C
267	1.5 mL/min	365 nm	25 °C

Participant	Calibration standard
23	fertiger Mix Standard Neochema
33	gekaufte Einzelstandards, Fa. Neochema
36	Merck Supelco Standard Mix, zertifiziertes Referenzmaterial
61	fertiger Mix, Kalibration Accu Standard, Ktr. supelco
134	Fertiger Mix , Hersteller Supelco
158	Der Standard w urde aus Reinsubstanzen folgender Firmen hergestellt: Supelco, Restek, Dr. Ehrensdorfer, Sigma-Aldrich
236	Einzelstandards; Supelco
267	Fertigmischung von Supelco

Participant	Recovery rates
23	nein
33	nein
36	Nein. Es w urden QC Standard mit geführt um die Stabilität der Chormatografie sicher zu stellen. Mittels Blindw ertbestimmung w erden mögliche Kontaminationsquellen im Labor überw acht.
61	Nein
134	nein
158	Es w urde ein unabhängiger Kontrollstandard verw endet
236	ja
267	Nein