IFA
Institut für Arbeitsschutz der
Deutschen Gesetzlichen Unfallversicherung

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

Proficiency testing scheme “Inorganic acids 2022”

March 2022

- **Volatile inorganic acids: hydrochloric acid, HCl and nitric acid, HNO_3**

Institute for Occupationial Safety and Health of the
German Social Accident Insurance (IFA)
B. Maybaum, K. Gusbeth, F. Nürnberger, Prof. Dr. D. Breuer
Alte Heerstraße 111, 53757 Sankt Augustin, Germany
Phone.: +49 30 13001 3299
Email: proficiency-testing@dguv.de

Summary of laboratory test results

Measurand Hydrochloric acid

Laboratory	Sample 1	Z score	Outlier type	Sample 2	Z score	Outlier type	Sample 3	Z score	Outlier type
Unit	mg/m³			mg/m³			mg/m³		
13	1.730	-0.16		0.835	1.10		3.330	0.08	
33	1.800	0.24		0.793	0.54		3.430	0.38	
68	1.778	0.11		0.758	0.07		3.065	-0.72	
83	1.960	1.14		0.850	1.30		3.657	1.07	
110	1.570	-1.07		0.670	-1.09		2.420	-2.68	E
120	1.830	0.41		0.808	0.74		3.490	0.56	
138	1.770	0.06		0.550	-2.69	E	2.790	-1.56	
151	1.700	-0.33		0.730	-0.30		3.230	-0.22	
163	1.260	-2.84	BE	0.750	-0.03		3.020	-0.86	
177	1.534	-1.28		0.628	-1.65		3.163	-0.43	
178	2.985	6.97	BE	1.702	12.62	BE	4.035	2.21	E
188	1.809	0.29		0.806	0.71		3.457	0.46	
248	1.720	-0.22		0.750	-0.03		3.141	-0.49	
266	1.760	0.01		0.790	0.50		3.440	0.41	
269	1.840	0.46		1.210	6.08	BE	3.630	0.99	
272	1.820	0.35		0.815	0.83		3.564	0.79	
–	–	– –		–	– –		–	– –	
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	16			16			16		
Mean	1.759			0.752			3.304		
Reproducibility s.d.	0.108			0.085			0.380		
Rel. reproducibility s.d.	6.16 %			11.25 %			11.49 %		
Reference value	1.754			0.799			3.473		
Target s.d.	0.176			0.075			0.330		
Rel. target s.d.	10.00 %			10.00 %			10.00 %		
Low er limit of tolerance	1.407			0.602			2.643		
Upper limit of tolerance	2.110			0.903			3.965		
Type B outliers	2			2					

Laboratory	Sample 1	Z score	Outlier type	Sample 2	Z score	Outlier type	Sample 3	Z score	Outlier type
Type E outliers	2			3			2		
No. of laboratories after elimination of outliers type A-D and F (w ithout laboratories that only gave states but no measured values)	14			14			16		

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	

Summary of laboratory test results

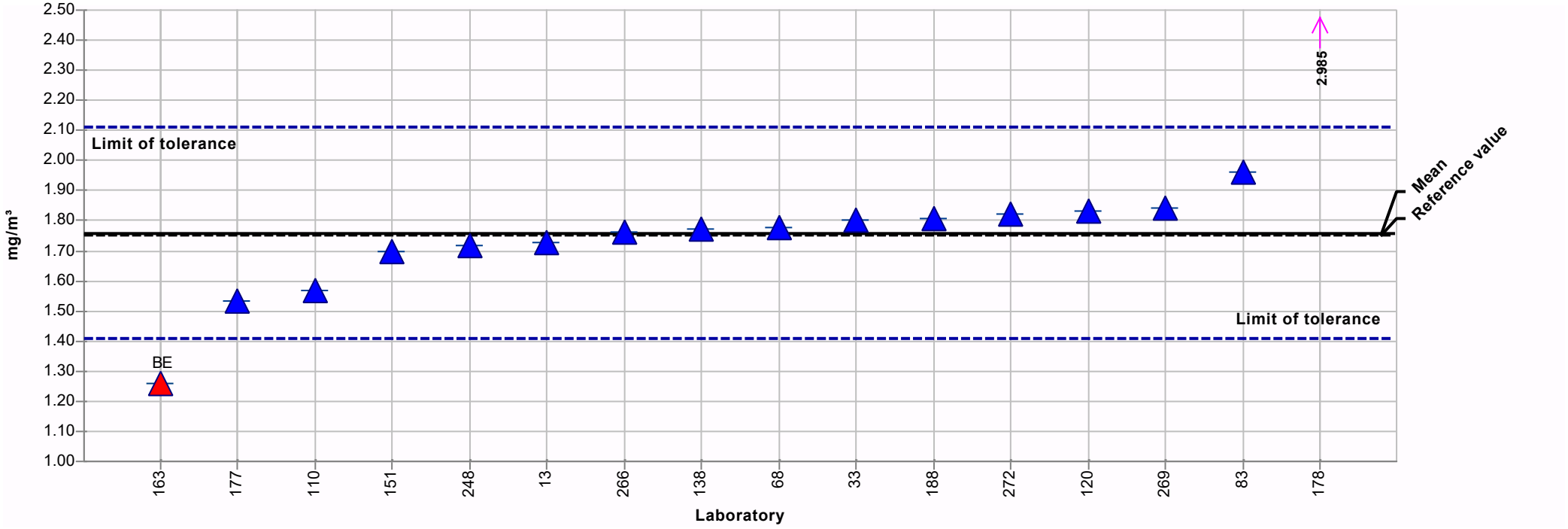
Measurand Nitric acid

Laboratory	Sample 1	Z score	Outlier type	Sample 2	Z score	Outlier type	Sample 3	Z score	Outlier type
Unit	mg/m³			mg/m³			mg/m³		
13	0.673	-0.22		1.300	0.06		1.410	0.49	
33	0.699	0.16		1.280	-0.09		1.420	0.57	
68	0.658	-0.44		1.210	-0.63		1.310	-0.25	
83	0.705	0.24		1.323	0.24		1.208	-1.01	
120	0.726	0.55		1.333	0.32		1.367	0.17	
138	0.670	-0.27		0.930	-2.80	BE	1.030	-2.33	E
151	0.670	-0.27		1.250	-0.32		1.390	0.34	
163	0.560	-1.86		1.120	-1.33		4.000	19.77	BE
177	0.606	-1.20		1.280	-0.09		1.302	-0.31	
178	0.933	3.56	BE	2.180	6.88	BE	2.120	5.78	BE
188	0.767	1.14		1.330	0.30		1.318	-0.19	
248	0.685	-0.05		1.299	0.06		1.305	-0.29	
266	0.690	0.03		1.300	0.06		1.360	0.12	
269	0.820	1.91		1.460	1.30		1.650	2.28	E
272	0.707	0.27		1.307	0.12		1.399	0.41	
–	–	– –		–	– –		–	– –	
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	15			15			15		
Mean	0.688			1.292			1.344		
Reproducibility s.d.	0.062			0.077			0.139		
Rel. reproducibility s.d.	9.08 %			5.96 %			10.35 %		
Reference value	0.725			1.286			1.677		
Target s.d.	0.069			0.129			0.134		
Rel. target s.d.	10.00 %			10.00 %			10.00 %		
Low er limit of tolerance	0.551			1.033			1.075		
Upper limit of tolerance	0.826			1.550			1.613		
Type B outliers	1			2			2		
Type E outliers	1			2			4		

Laboratory	Sample 1	Z score	Outlier type	Sample 2	Z score	Outlier type	Sample 3	Z score	Outlier type
No. of laboratories after elimination of outliers type A-D and F (w ithout 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: mean outside tolerance limits									
F: |Z-Score|>3.5									

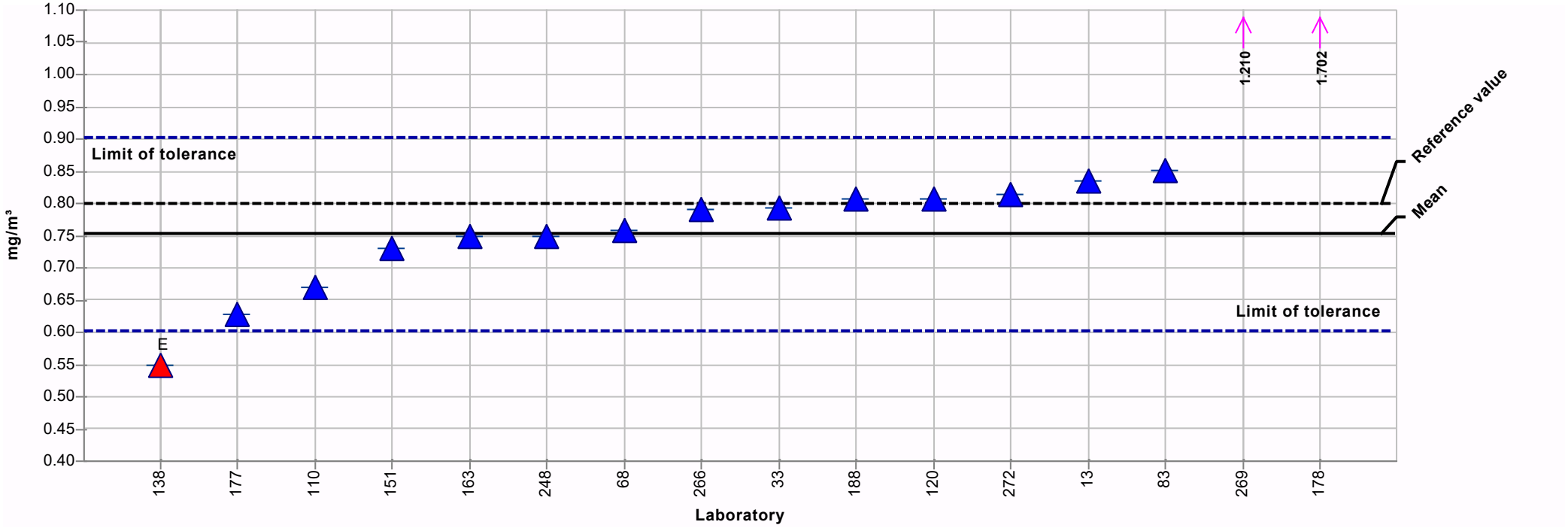
Summary results

Sample:	**1**	**Mean:**	**1.759 mg/m³**
Measurand:	**Hydrochloric acid**	**Reproducibility s.d.:**	**0.108 mg/m³**
Method:	**ISO 5725-2**	**Rel. reproducibility s.d.:**	**6.16%**
Rel. target s.d.:	**10.00% (Limited)**	**Reference value:**	**1.754 mg/m³**
Number of laboratories in calculation + outliers:	**16**	**Range of tolerance:**	**1.407 - 2.110 mg/m³ (|Z-Score| <= 2.00)**



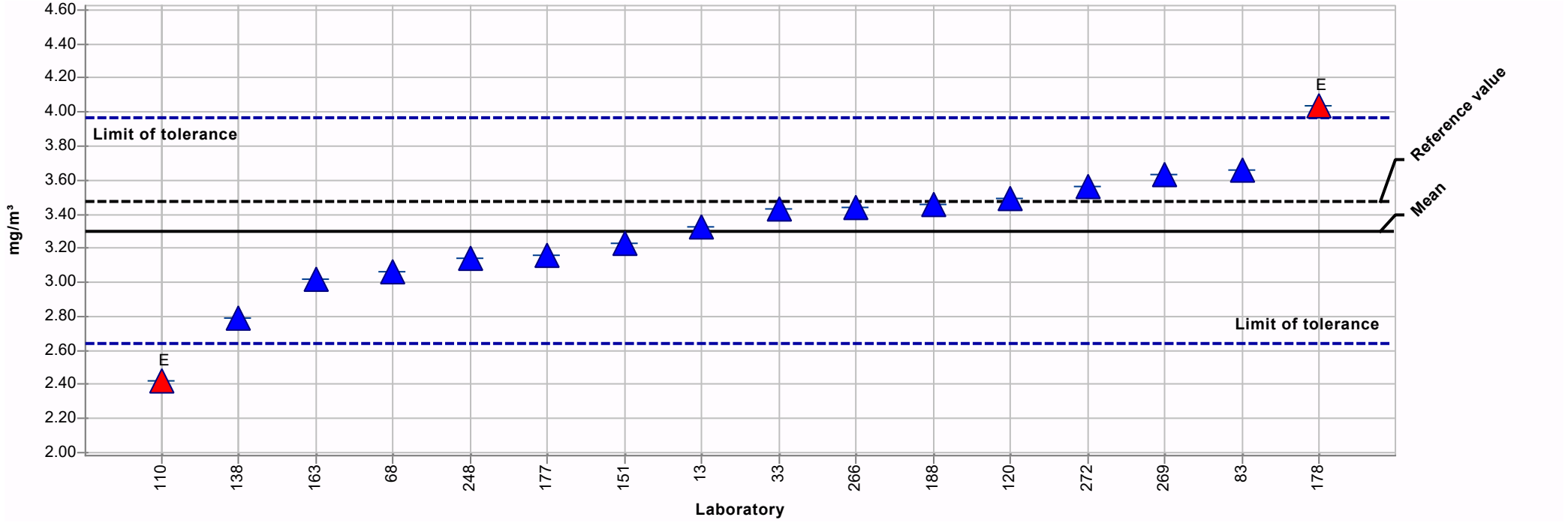
Summary results

Sample:	**2**	**Mean:**	**0.752 mg/m³**
Measurand:	**Hydrochloric acid**	**Reproducibility s.d.:**	**0.085 mg/m³**
Method:	**ISO 5725-2**	**Rel. reproducibility s.d.:**	**11.25%**
Rel. target s.d.:	**10.00% (Limited)**	**Reference value:**	**0.799 mg/m³**
Number of laboratories in calculation + outliers:	**16**	**Range of tolerance:**	**0.602 - 0.903 mg/m³ (|Z-Score| <= 2.00)**



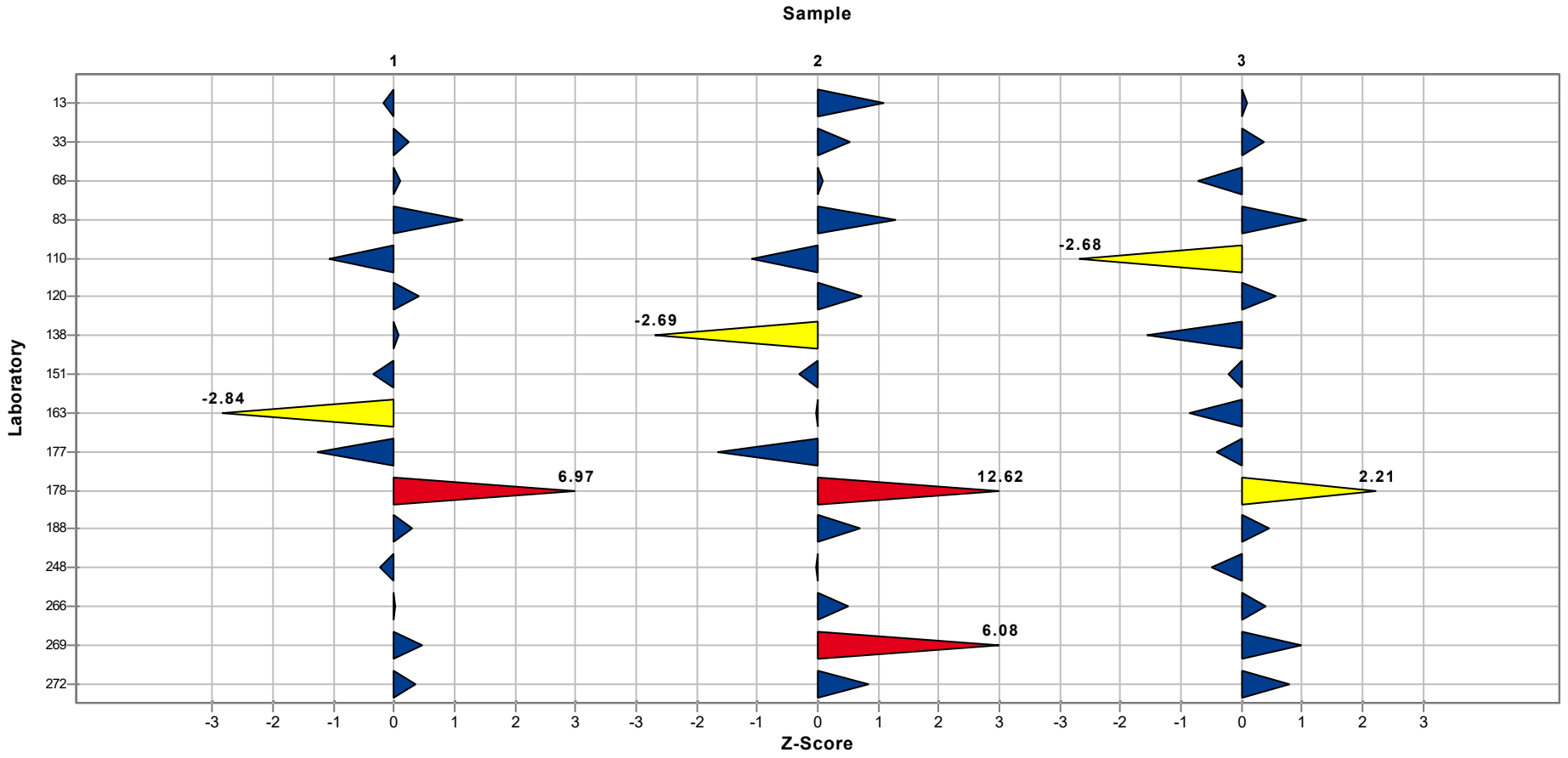
Summary results

Sample:	**3**	**Mean:**	**3.304 mg/m³**
Measurand:	**Hydrochloric acid**	**Reproducibility s.d.:**	**0.380 mg/m³**
Method:	**ISO 5725-2**	**Rel. reproducibility s.d.:**	**11.49%**
Rel. target s.d.:	**10.00% (Limited)**	**Reference value:**	**3.473 mg/m³**
Number of laboratories in calculation:	**16**	**Range of tolerance:**	**2.643 - 3.965 mg/m³ (|Z-Score| <= 2.00)**



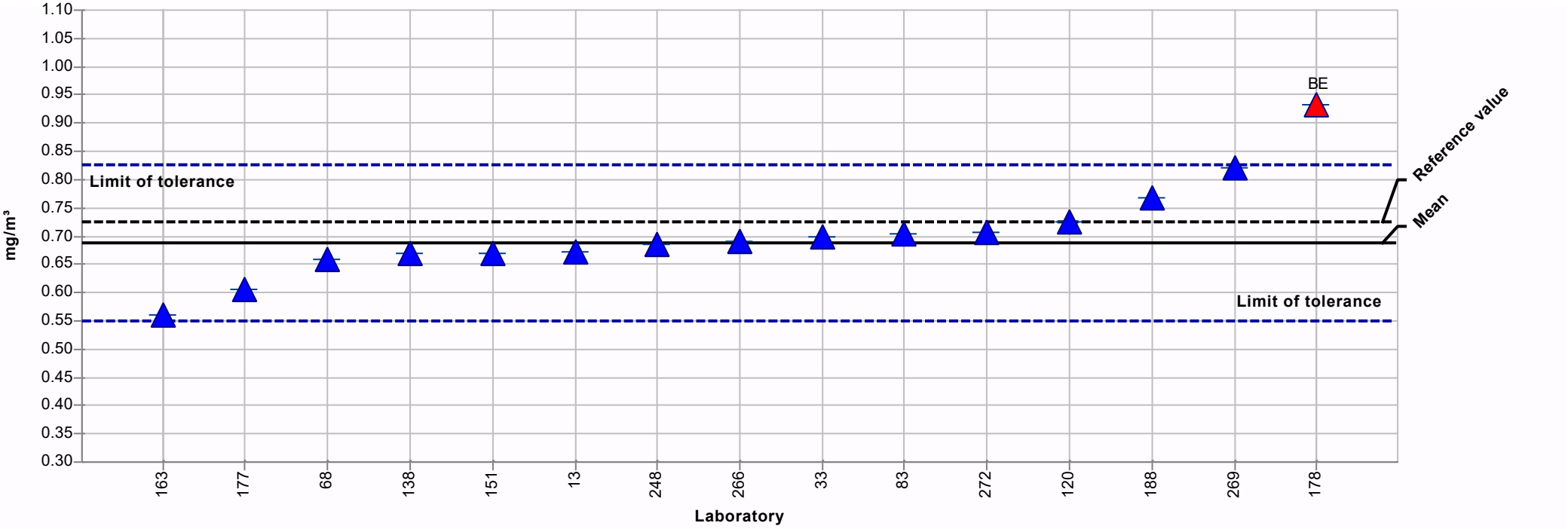
Analyte chart of Z-Scores

Measurand: **Hydrochloric acid**



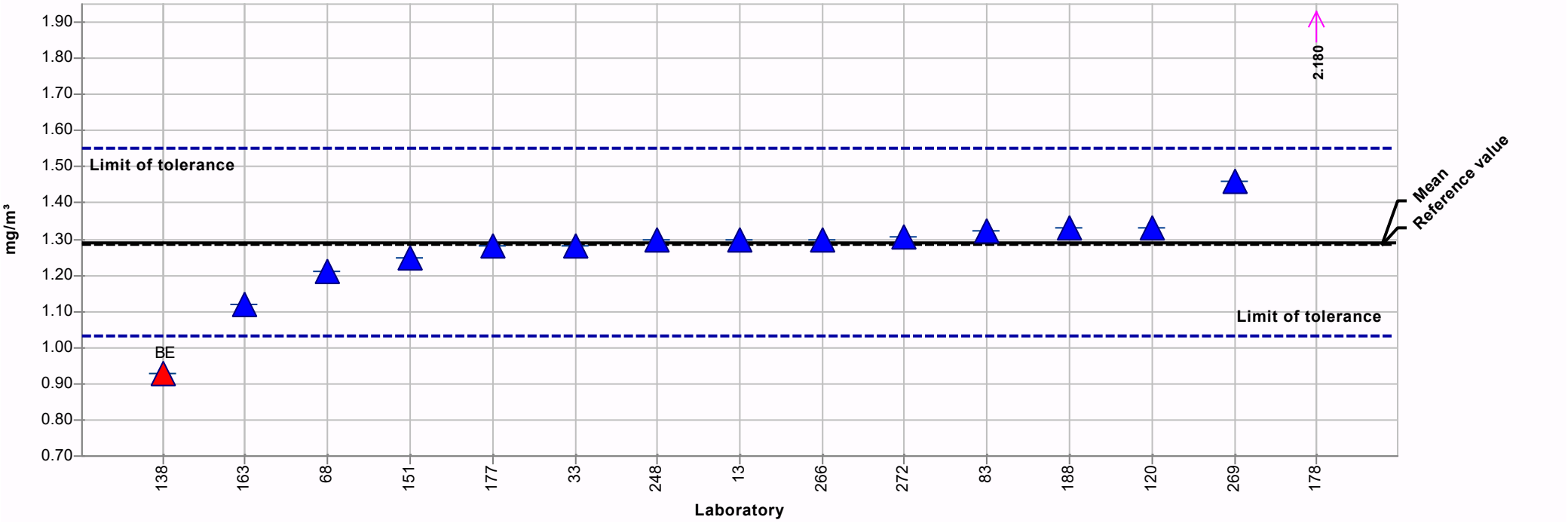
Summary results

Sample:	**1**	**Mean:**	**0.688 mg/m³**
Measurand:	**Nitric acid**	**Reproducibility s.d.:**	**0.062 mg/m³**
Method:	**ISO 5725-2**	**Rel. reproducibility s.d.:**	**9.08%**
Rel. target s.d.:	**10.00% (Limited)**	**Reference value:**	**0.725 mg/m³**
Number of laboratories in calculation + outliers:	**15**	**Range of tolerance:**	**0.551 - 0.826 mg/m³ (|Z-Score| <= 2.00)**



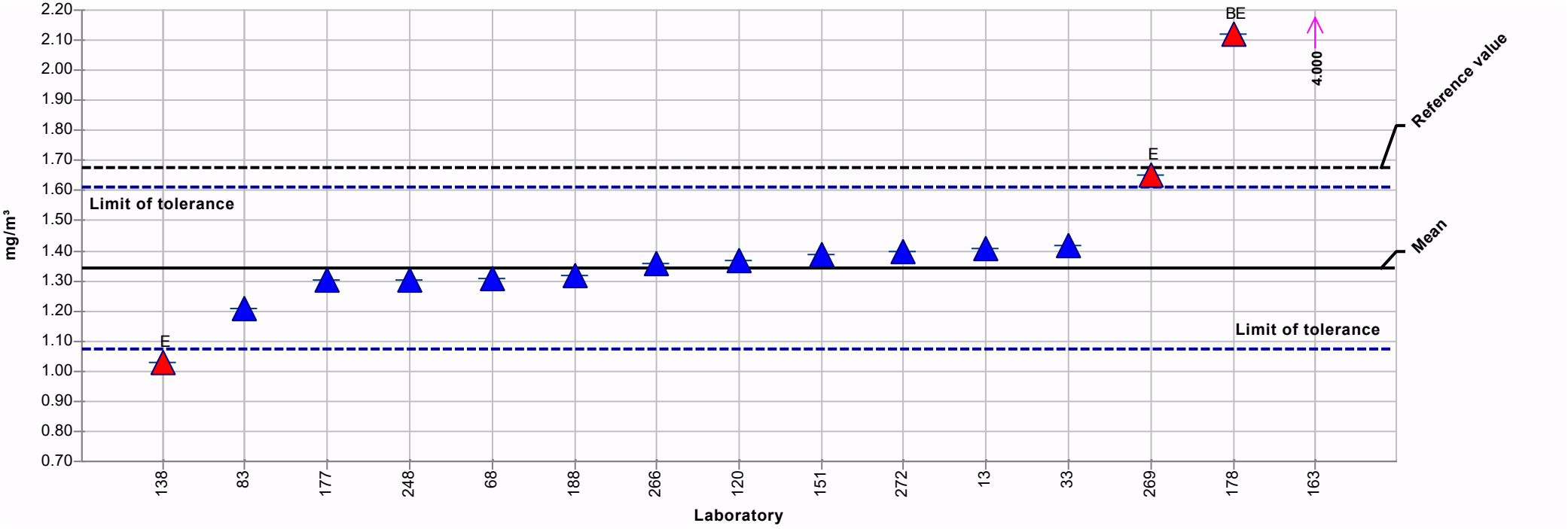
Summary results

Sample:	**2**	**Mean:**	**1.292 mg/m³**
Measurand:	**Nitric acid**	**Reproducibility s.d.:**	**0.077 mg/m³**
Method:	**ISO 5725-2**	**Rel. reproducibility s.d.:**	**5.96%**
Rel. target s.d.:	**10.00% (Limited)**	**Reference value:**	**1.286 mg/m³**
Number of laboratories in calculation + outliers:	**15**	**Range of tolerance:**	**1.033 - 1.550 mg/m³ (|Z-Score| <= 2.00)**



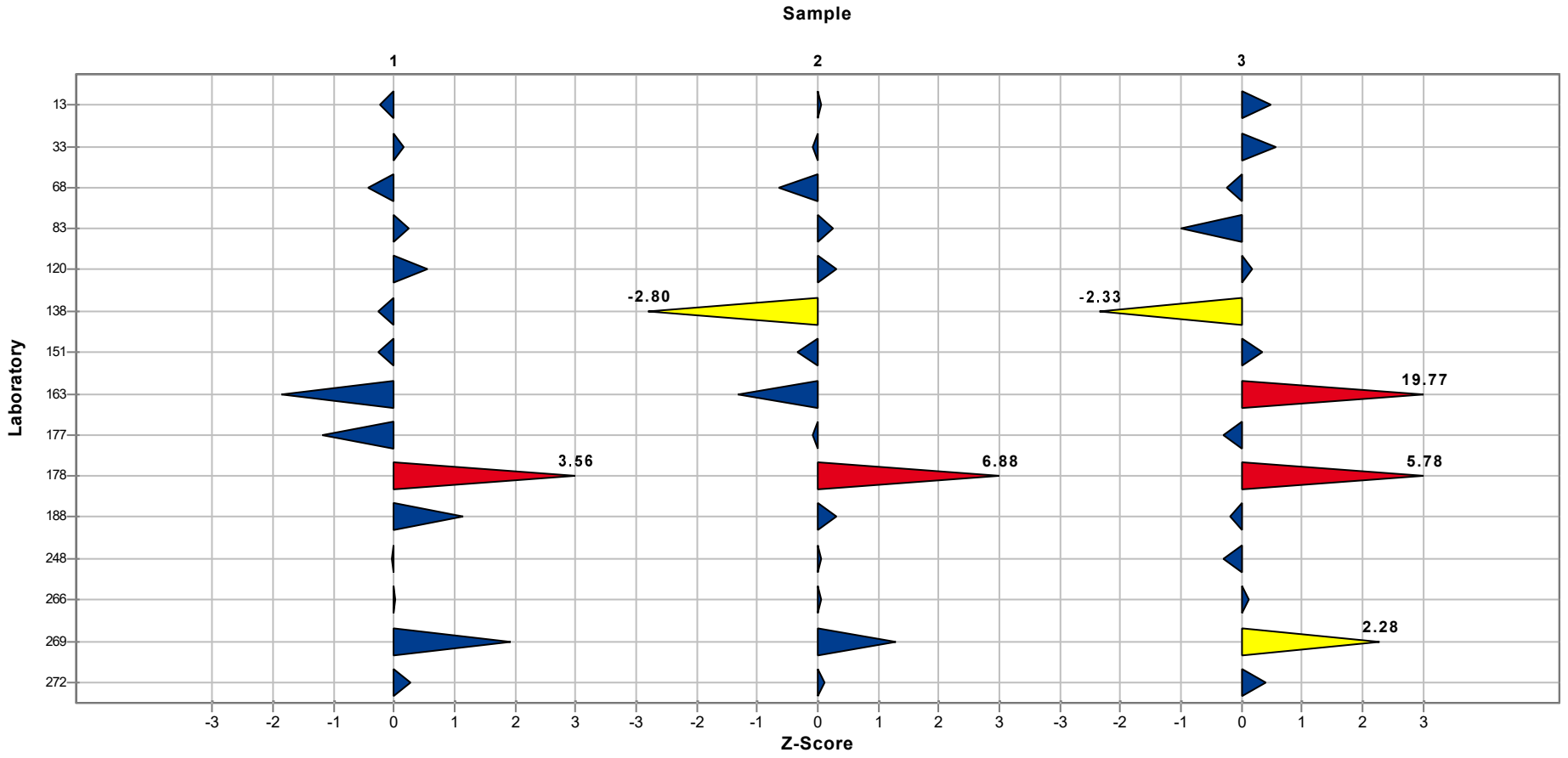
Summary results

Sample:	**3**	**Mean:**	**1.344 mg/m³**
Measurand:	**Nitric acid**	**Reproducibility s.d.:**	**0.139 mg/m³**
Method:	**ISO 5725-2**	**Rel. reproducibility s.d.:**	**10.35%**
Rel. target s.d.:	**10.00% (Limited)**	**Reference value:**	**1.677 mg/m³**
Number of laboratories in calculation + outliers:	**15**	**Range of tolerance:**	**1.075 - 1.613 mg/m³ (|Z-Score| <= 2.00)**



Analyte chart of Z-Scores

Measurand: **Nitric acid**



Questions and Answers

Participant	Analytical method
13	SOP-LCA-36
33	IFA 6172
68	IFA-Arbeitsmappe
83	Ion Chromatography
110	nach IFA Arbeitsmappe
120	IFA 6172
138	DGUV 6172, IC
151	Ion Chromatography
163	NIOSH 7907 (Modified)
177	IFA-Arbeitsmappe
178	NIOSH 7907
188	IFA 6172 von 2007 - Anorganische Säuren, flüchtig: Bromw asserstoff, Chlorw asserstoff, Salpetersäure
248	IFA-Arbeitsmappe 6172.
266	NIOSH 7908 modified (IRSST MA-211)
269	IFA 6172
272	IFA 6172:2007-04

Participant	Desorption solution	Volume of desorption solution
13	Ultrapure w ater	10 ml
33	Reinstw asser	10 mL
68	Reinstw asser	10 ml
83	Water	30 ml
110	8,0mmol Na2CO3 + 1mmol NaHCO3	50 ml
120	Wasser	10 mL
138	Reinstw asser	10 ml
151	DI w ater	10
163	Carbonate (2.7 mM) / Bicarbonate (0.3 mM)	10 ml
177	Reinstw asser	50 ml bzw . 20 ml
178	Deionzied w ater to each sample	10 mL
188	destilliertes Wasser	20 mL

Participant	Desorption solution	Volume of desorption solution
248	Bidest.	10
266	Water type 1	20mL
272	Wasser	10 mL

Participant	Time of desorption
13	15 minutes ultrasonic and 30 minutes cool dow n
33	15 min im Ultraschall behandelt und 30 min stehen lassen. Anschließend über einen Spritzenvorsatzfilter filtriert
68	15 Min. Ultraschallbad, anschl. mind. 30 Min. stehen lassen
83	30 minutes
110	15 min, ja
120	15 min im Ultraschallbad
138	15 Minuten im Ultraschallbad, 30 Minuten stehen lassen
151	60 minutes, orbital shaker
163	10 min in Shaker
177	30 Minuten im Ultraschallbad
178	15 min ultrasonic
188	nach Vorschrift 15 min Ultraschallbad und 30 min Stehen
248	15 min Ultraschallbad, 30 min ruhen lassen
266	30 minutes, ultrasonic bath
272	Extraktion 15 min Ultraschallbad, anschließend w eitere 30 min stehen gelassen

Participant	Ion Chromatographic System
13	Gilair plus
33	Peristaltikpumpe, Leitfähigkeitsdetektor, Metrohm-Autosampler
68	930 Compact IC Flex von Metrohm
83	ICS 5000 - ASDV Dionex
110	Thermo Scientific, Dionex, Integrion, AS/AP Autosampler
120	Metrohm IC 850 Professional IC
138	Dionex ICS 1100
163	Quaternary pump. Conductivity detector.
177	Thermo Scientific ICS-6000 mit Autosampler AS-AP
178	Cromatografo iónico Methrom 883 Basic IC Plus/Detector Conductividad
188	930 Compact IC Flex

Participant	Ion Chromatographic System
248	ICS-1100, DS6 Heated Conductivity Cell, AS-AP Thermo Scientific
266	Thermo Dionex ICS-Integrion
272	Metrohm: 881 Compact IC pro mit Professional IC Detector MF und 858 Professional Sample Processor

Participant	Analytical column
13	Dionex IonPac AS22 RFIC w ith AG22 RFIC Guard column
33	Metrosep A Supp 5 150/4.0 Vorsäule: Metrosep A Supp 5 Guar 4.0
68	Metrosep C 6 - 250/4.0 von Metrohm
83	AS 11-HC 4mm
110	AG18/ AS18
120	Metrosep A Supp 5 - 150/4.0
138	Dionex IonPac AS22 4 * 250 mm
151	Dionex AS22
163	Dionex IonPac As11-HC 4 x 250 mm
177	AS 15
178	Metrosep A Supp 5-150/4,0
188	Metrosep A Supp 4-250/4.0
248	AS21
266	AS22 FAST
272	Metrosep A Supp 5-150 (Trennsäule) plus Metrosep A Supp 4/5 Guard (Vorsäule)

Participant	Mobile phase	Flow rate	Recovery rate	Date of analysis
13	1,4 mM NaHCO3 and 4,5 mM Na2CO3	0.3	Yes (98,5%)	14.04.2022
33	3,2 mmol/L Na2CO3+ 1 mmol/L NaHCO3	0,7	ja	05.04.2022
68	3.6 mM Natriumcarbonat-Lsg.	0.7 ml/min.	Keine	12. - 14.04.2022
83	KOH	1	No	07.04.2022
110	23mmol KOH	0,25 ml/min		05.05.2022
120	1 mmol/L Natriumhydrogencarbonat + 3,2 mmol/L Natriumcarbonat	0,7	Nein	05. Apr 22
138	Na2CO3 / NaHCO3	1,2		Mai 22
151	1.4mM bicarbonate + 4.5mM carbonate	0.3		
163	KOH 20 mM	01. Jan	no	17.05.2022
177	KOH 12-40 mmol/l	0,3		30.05.2022
178	carbonato sódico/bicarbonato sódico (3,2mM/1mM)	0,8 ml/min	NO	17.05.2022

Participant	Mobile phase	Flow rate	Recovery rate	Date of analysis
188	3,6 ml Na2CO3 (1M) und 3,4 ml NaHCO3 (1M) auf 2 l aufgefüllt	1 ml/min	nein	06.04.2022
248	Na2CO3 / NaHCO3	1,3 ml/min	nein	09.04.2022
266	Carbonate/Bicarbonate : 1,5 / 0,5 (mM)	1,2	No	12. Apr 22
272	3,2 mM Na2CO3 / 1,0 mM NaHCO3	0,7 mL/min	Nein	21.04.2022