IFA
Institut für Arbeitsschutz der
Deutschen Gesetzlichen Unfallversicherung

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

Proficiency testing scheme aldehydes

October 2018

Institute for Occupationial Safety and Health of the
German Social Accident Insurance (IFA)
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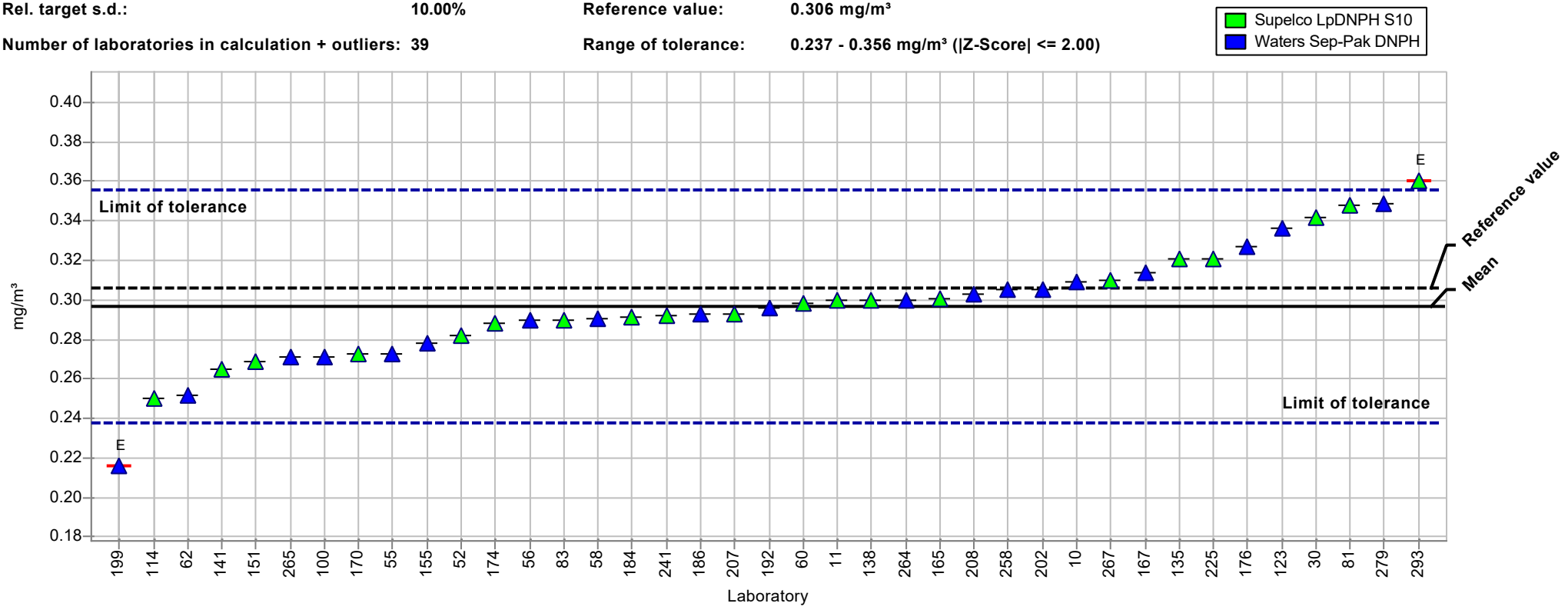
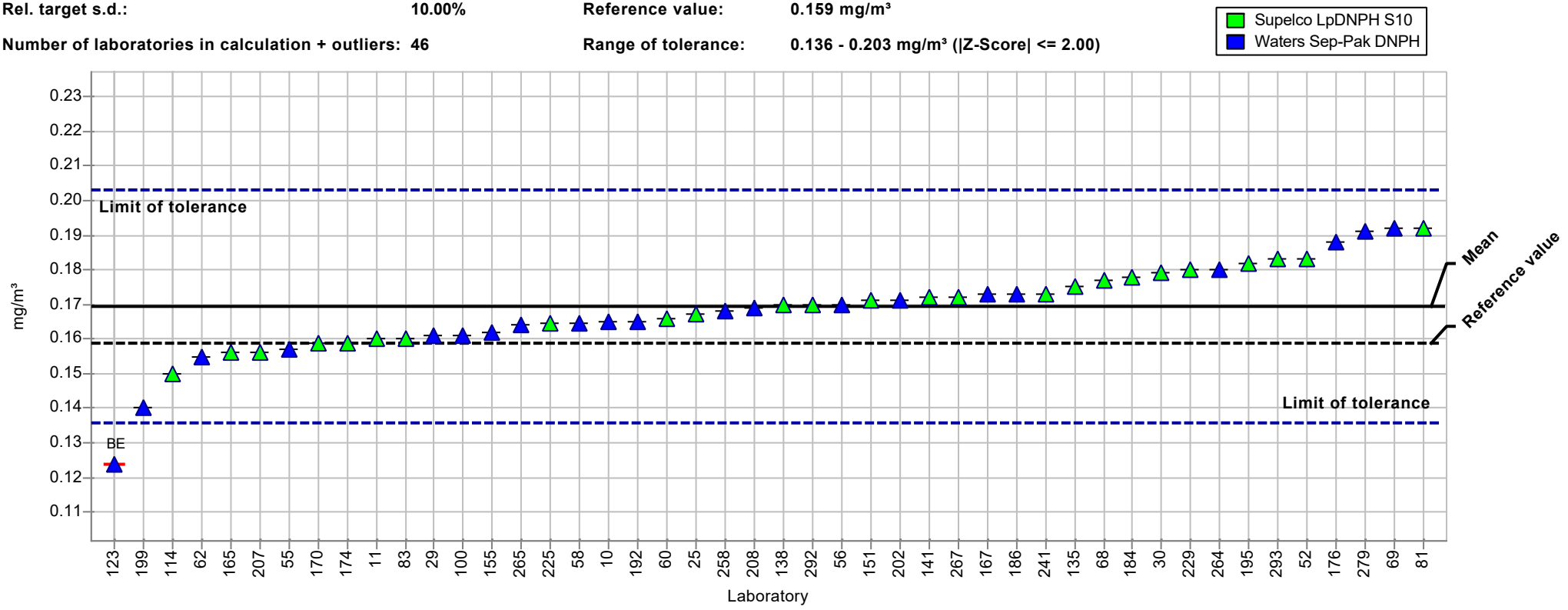
Summary results

Measurand:	**Butyraldehyde**	**Mean:**	**0.297 mg/m³**
Sample:	**1**	**Reproducibility s.d.:**	**0.029 mg/m³**
Method:	**ISO 5725-2**	**Rel. reproducibility s.d.:**	**9.73%**
Rel. target s.d.:	**10.00%**	**Reference value:**	**0.306 mg/m³**
Number of laboratories in calculation + outliers:	**39**	**Range of tolerance:**	**0.237 - 0.356 mg/m³ (|Z-Score| <= 2.00)**



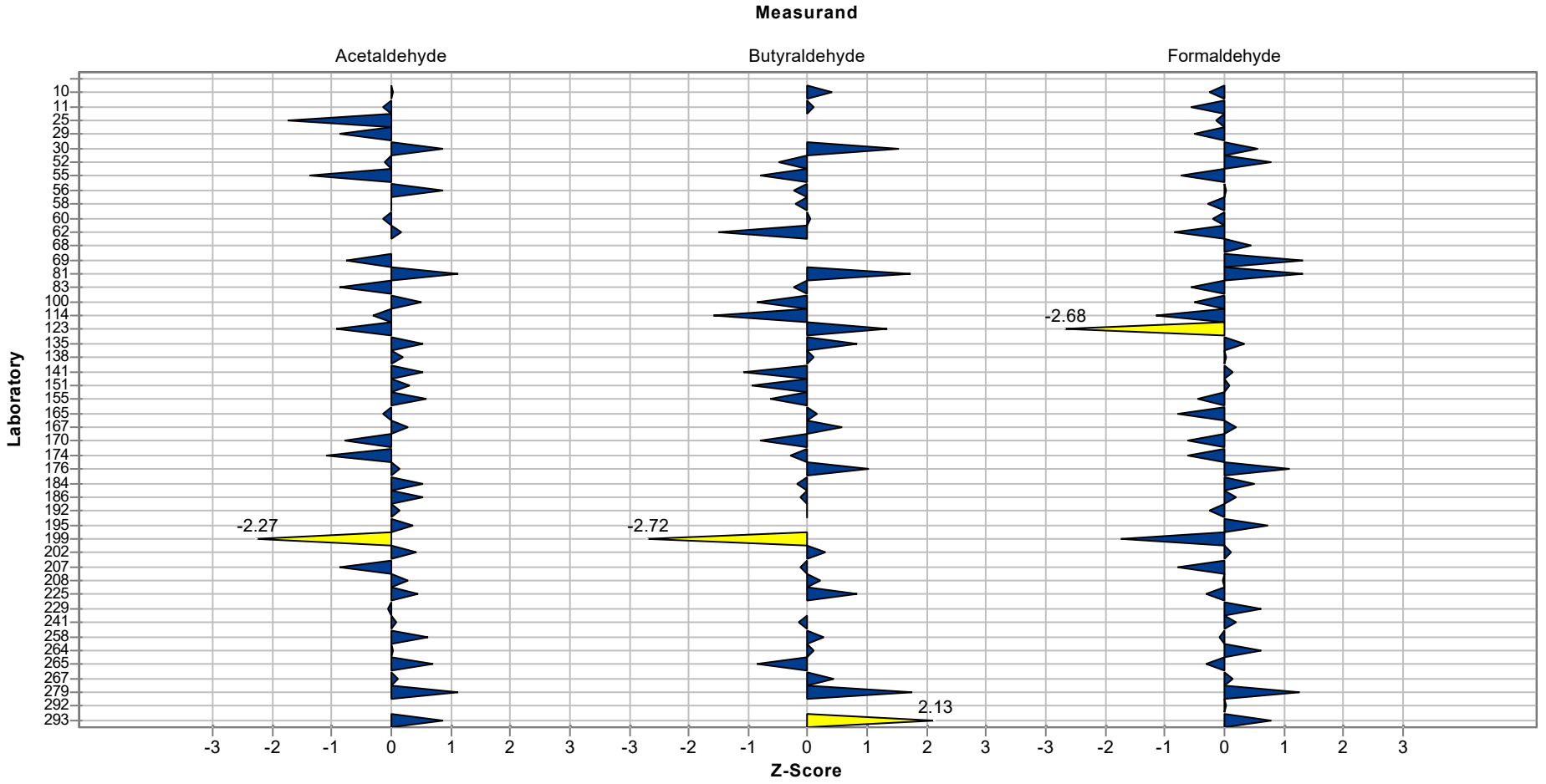
Summary results

Measurand:	**Formaldehyde**	**Mean:**	**0.169 mg/m³**
Sample:	**1**	**Reproducibility s.d.:**	**0.011 mg/m³**
Method:	**ISO 5725-2**	**Rel. reproducibility s.d.:**	**6.65%**
Rel. target s.d.:	**10.00%**	**Reference value:**	**0.159 mg/m³**
Number of laboratories in calculation + outliers:	**46**	**Range of tolerance:**	**0.136 - 0.203 mg/m³ (|Z-Score| <= 2.00)**



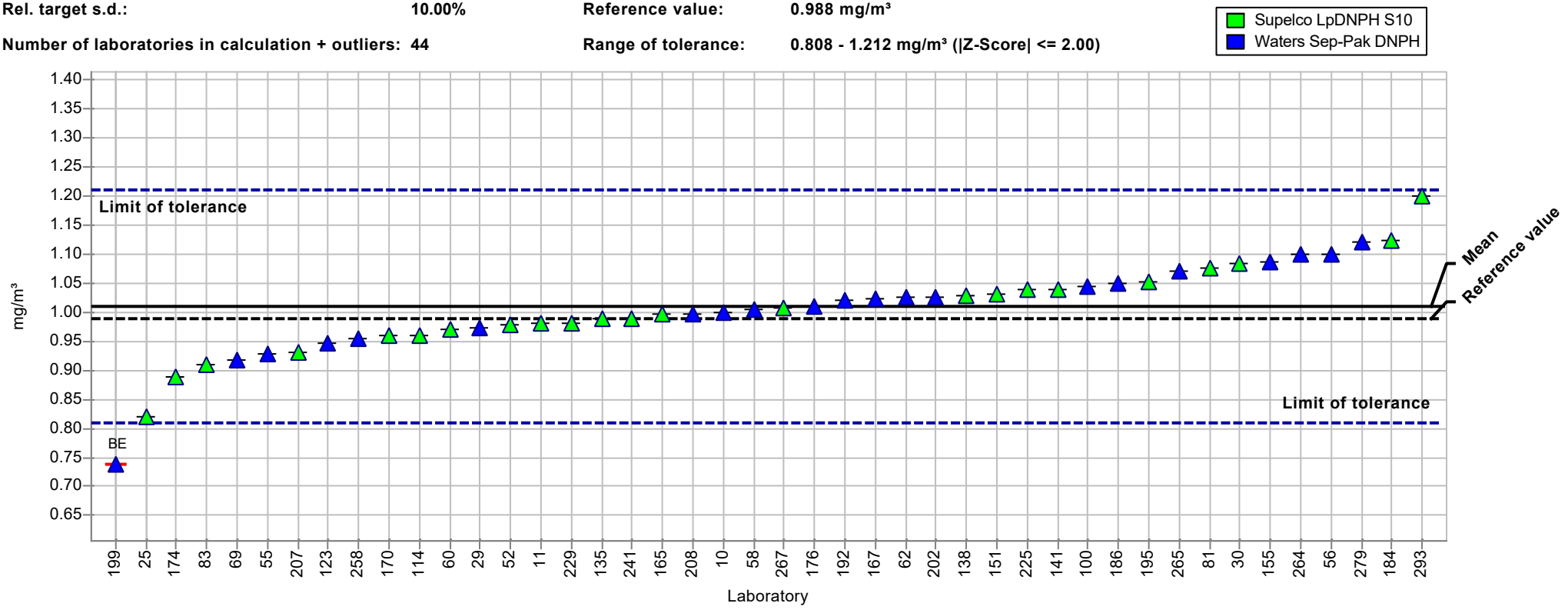
Sample chart of *Z-Scores*

Sample: 1



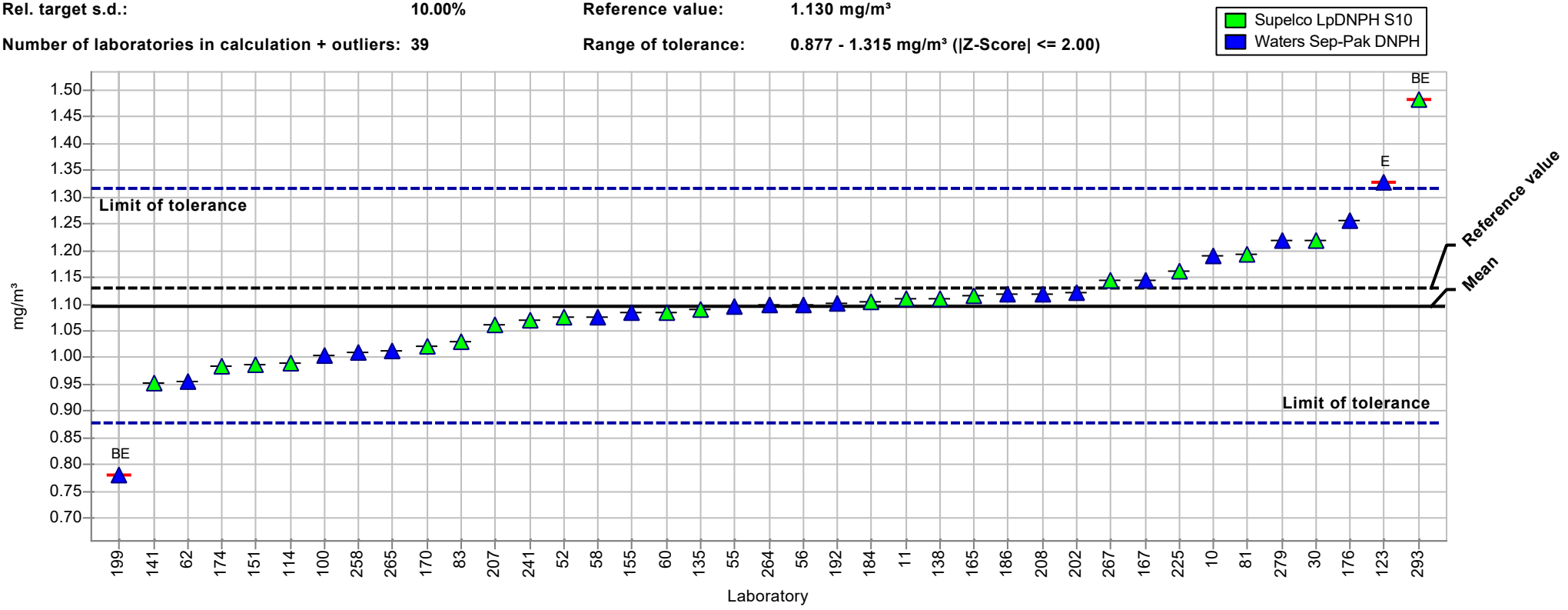
Summary results

Measurand:	**Acetaldehyde**	**Mean:**	**1.010 mg/m³**
Sample:	**2**	**Reproducibility s.d.:**	**0.070 mg/m³**
Method:	**ISO 5725-2**	**Rel. reproducibility s.d.:**	**6.93%**
Rel. target s.d.:	**10.00%**	**Reference value:**	**0.988 mg/m³**
Number of laboratories in calculation + outliers:	**44**	**Range of tolerance:**	**0.808 - 1.212 mg/m³ (|Z-Score| <= 2.00)**



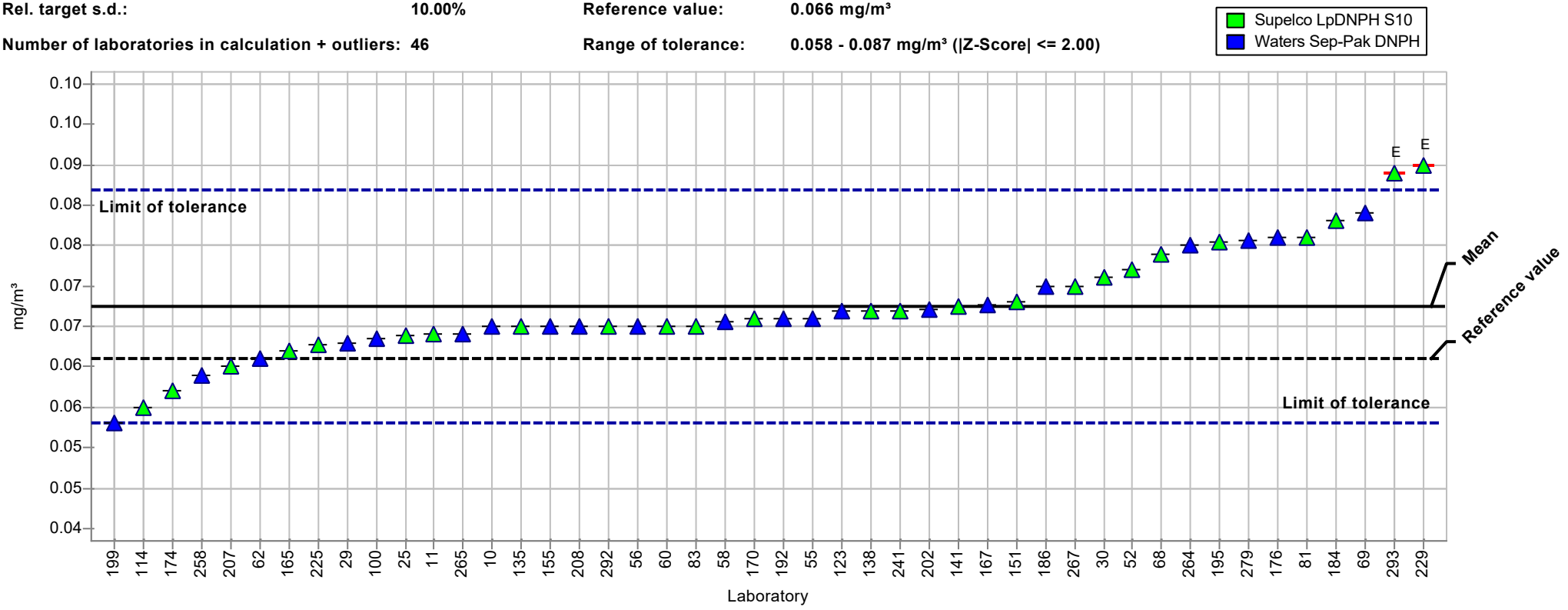
Summary results

Measurand:	**Butyraldehyde**	**Mean:**	**1.096 mg/m³**
Sample:	**2**	**Reproducibility s.d.:**	**0.084 mg/m³**
Method:	**ISO 5725-2**	**Rel. reproducibility s.d.:**	**7.64%**
Rel. target s.d.:	**10.00%**	**Reference value:**	**1.130 mg/m³**
Number of laboratories in calculation + outliers:	**39**	**Range of tolerance:**	**0.877 - 1.315 mg/m³ (|Z-Score| <= 2.00)**



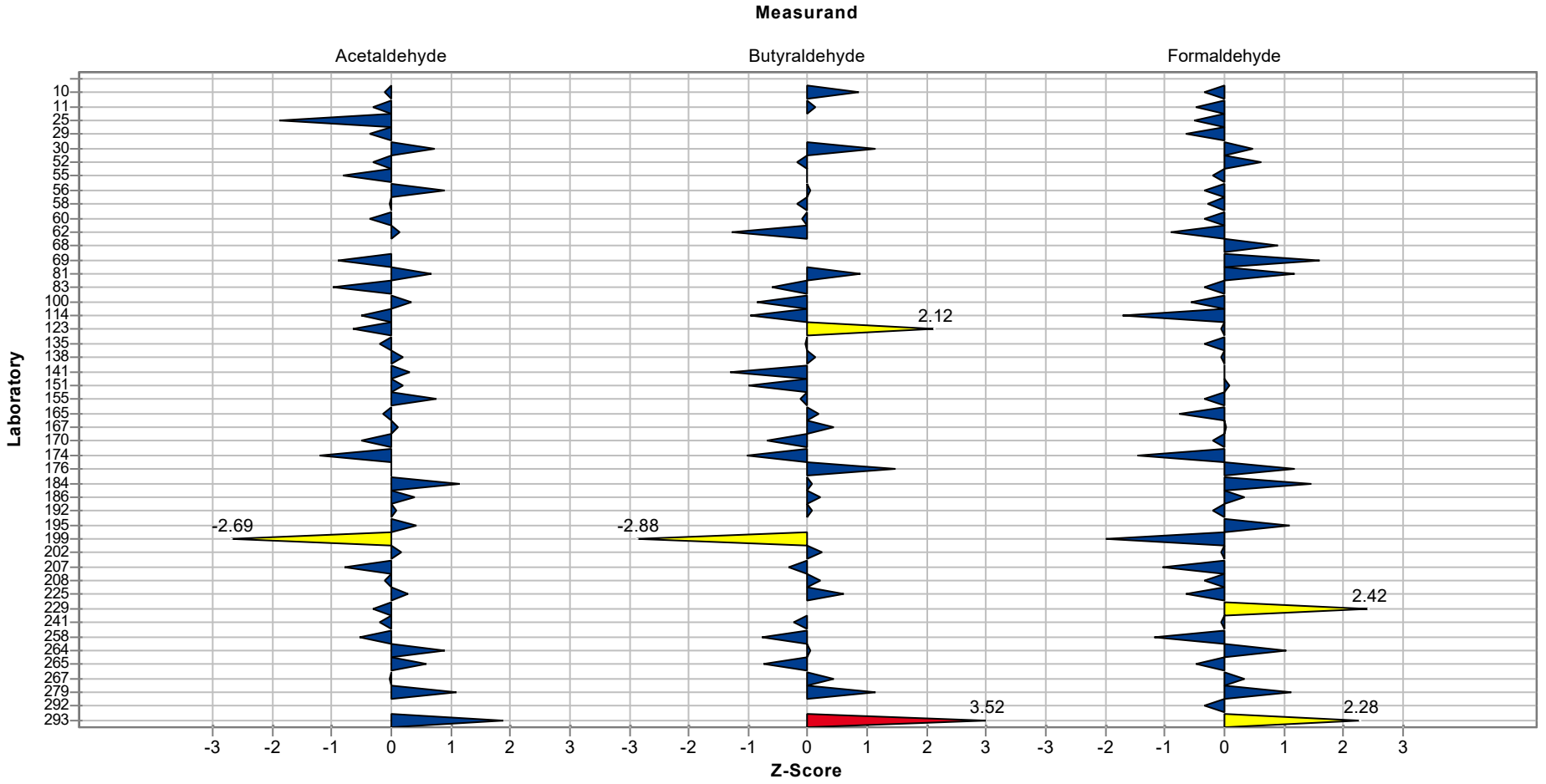
Summary results

Measurand:	**Formaldehyde**	**Mean:**	**0.072 mg/m³**
Sample:	**2**	**Reproducibility s.d.:**	**0.007 mg/m³**
Method:	**ISO 5725-2**	**Rel. reproducibility s.d.:**	**9.37%**
Rel. target s.d.:	**10.00%**	**Reference value:**	**0.066 mg/m³**
Number of laboratories in calculation + outliers:	**46**	**Range of tolerance:**	**0.058 - 0.087 mg/m³ (|Z-Score| <= 2.00)**



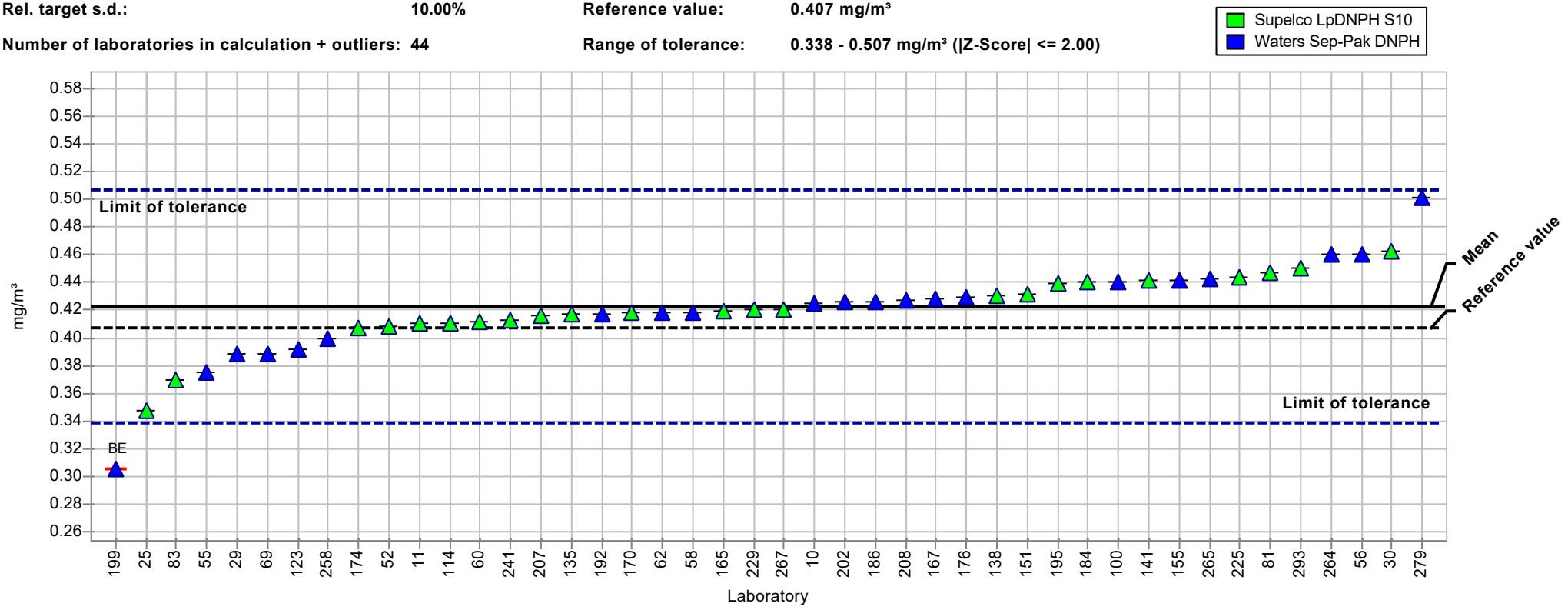
Sample chart of *Z-Scores*

Sample: 2



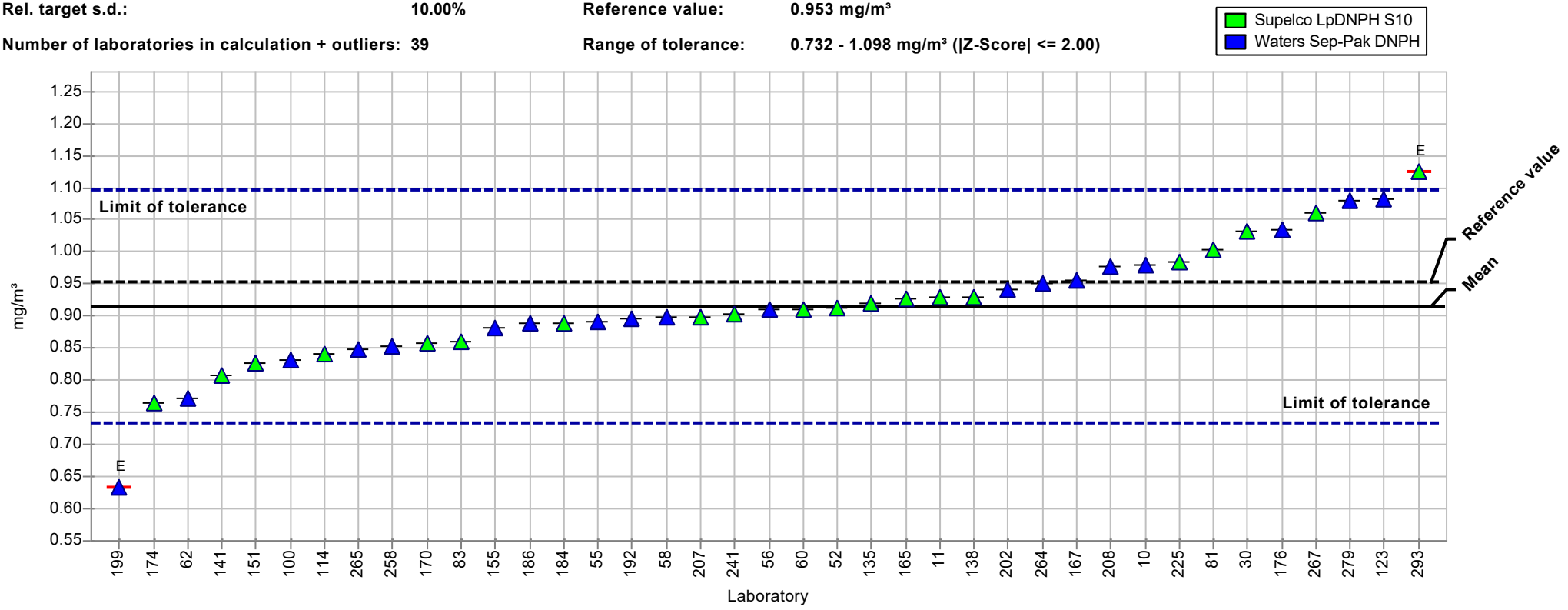
Summary results

Measurand:	**Acetaldehyde**	**Mean:**	**0.423 mg/m³**
Sample:	**3**	**Reproducibility s.d.:**	**0.027 mg/m³**
Method:	**ISO 5725-2**	**Rel. reproducibility s.d.:**	**6.36%**
Rel. target s.d.:	**10.00%**	**Reference value:**	**0.407 mg/m³**
Number of laboratories in calculation + outliers:	**44**	**Range of tolerance:**	**0.338 - 0.507 mg/m³ (|Z-Score| <= 2.00)**



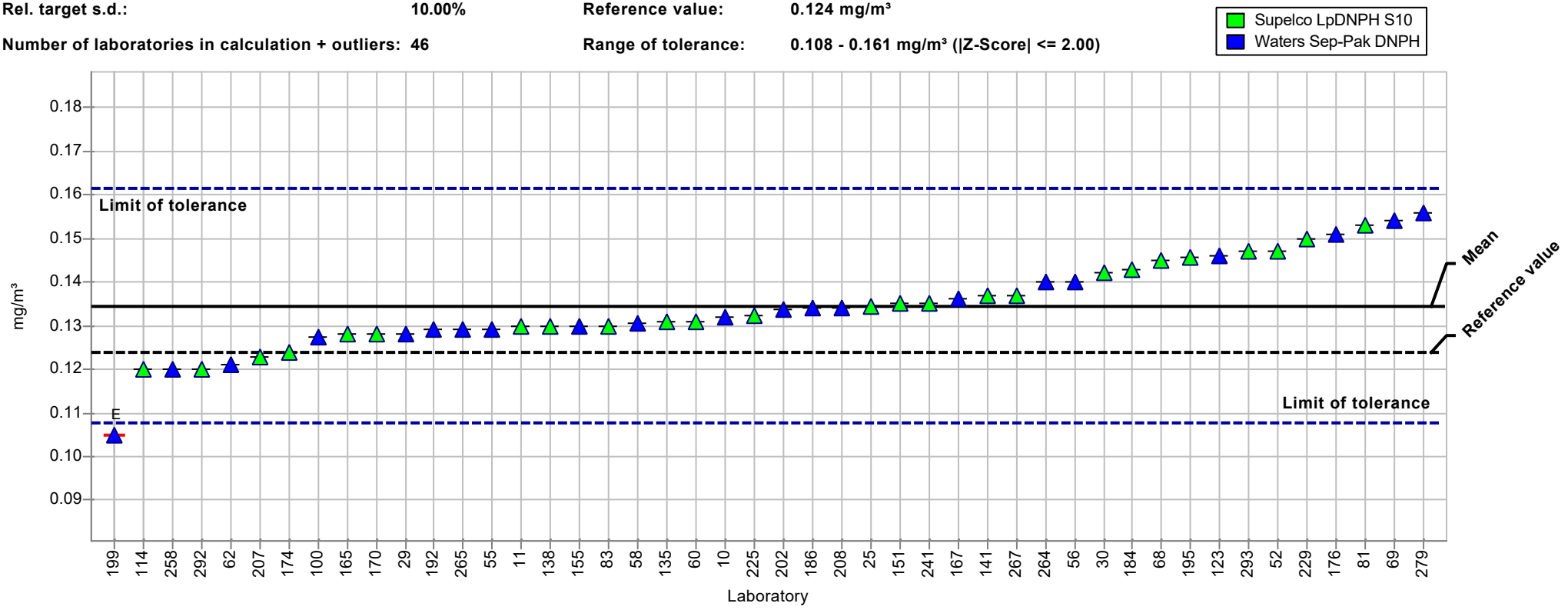
Summary results

Measurand:	**Butyraldehyde**	**Mean:**	**0.915 mg/m³**
Sample:	**3**	**Reproducibility s.d.:**	**0.096 mg/m³**
Method:	**ISO 5725-2**	**Rel. reproducibility s.d.:**	**10.46%**
Rel. target s.d.:	**10.00%**	**Reference value:**	**0.953 mg/m³**
Number of laboratories in calculation + outliers:	**39**	**Range of tolerance:**	**0.732 - 1.098 mg/m³ (|Z-Score| <= 2.00)**



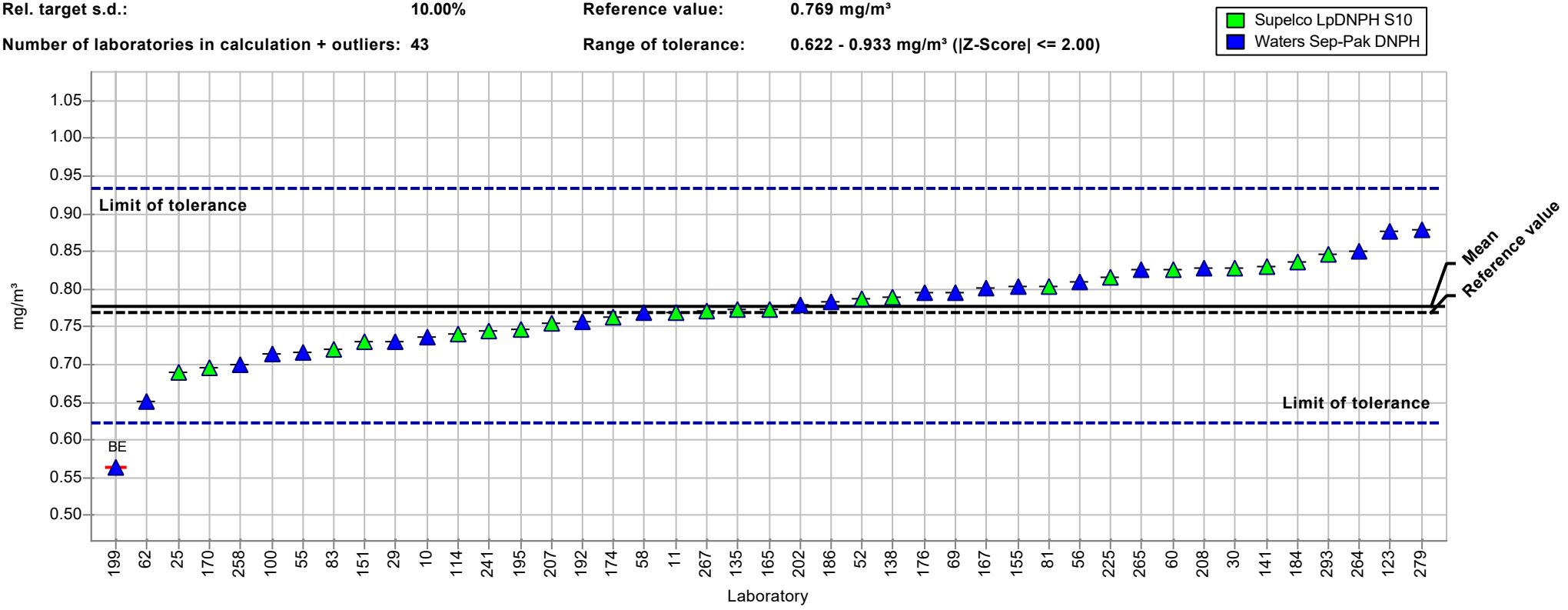
Summary results

Measurand:	**Formaldehyde**	**Mean:**	**0.134 mg/m³**
Sample:	**3**	**Reproducibility s.d.:**	**0.010 mg/m³**
Method:	**ISO 5725-2**	**Rel. reproducibility s.d.:**	**7.77%**
Rel. target s.d.:	**10.00%**	**Reference value:**	**0.124 mg/m³**
Number of laboratories in calculation + outliers:	**46**	**Range of tolerance:**	**0.108 - 0.161 mg/m³ (|Z-Score| <= 2.00)**



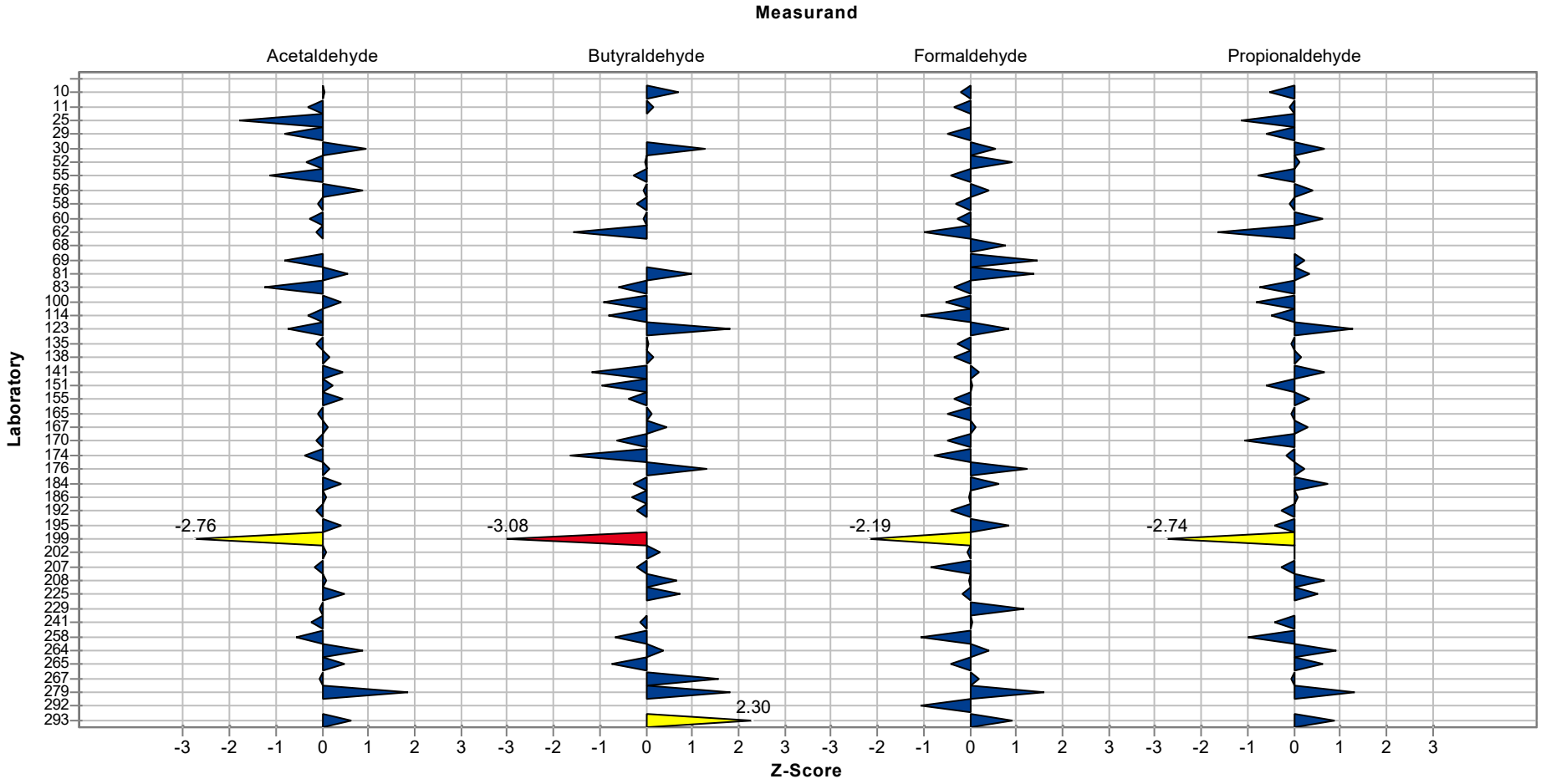
Summary results

Measurand:	**Propionaldehyde**	**Mean:**	**0.777 mg/m³**
Sample:	**3**	**Reproducibility s.d.:**	**0.052 mg/m³**
Method:	**ISO 5725-2**	**Rel. reproducibility s.d.:**	**6.66%**
Rel. target s.d.:	**10.00%**	**Reference value:**	**0.769 mg/m³**
Number of laboratories in calculation + outliers:	**43**	**Range of tolerance:**	**0.622 - 0.933 mg/m³ (|Z-Score| <= 2.00)**



Sample chart of *Z-Scores*

Sample: 3



Questions and Answers

Participant	Analytical method	Date start sample preparation
10	HPLC-DAD-NF-X-43-264	17/10/2018
11	ISO 16000-3	17/10/2018
25	eigene Prüfmethode	16.10.2018
29	Arbeitsmappe 6045, ISO 16000-3	11.10.18
30	ISO 16000-3	22/10/2018
52	Hausverfahren	30.10.2018
55	HPLC - Photodiode array detector	18/10/2018
56	HPLC-UV	10/10/18
58	LC-UV	16 october 2018
60	NF ISO 16000-3	12/10/2018
62	NF X43-264	19/10/2018
68	interne Arbeitsanw eisung	16.10.18
69	HPLC	19/10/2018
81	LC-MS	16/10/2018
83	NF ISO 16000-3	11/10/18
100	NF X43-264	10/11/2018
114	HPLC/UV based on NF X43-264 and metropol M-4	16/10/18
123	NF ISO 16000-3	19/10/18
135	HPLC-DAD IFA Arbeitsmappe 6045	12.10.2018
138	BGIA 6045	18.10.2018
141	HPLC	23-10-2018
151	HPLC UV	12/10/2018
155	EN 16516, DIN ISO 16000-3	15.10.2018
165	ISO 16000-3	12/10/2018
167	HPLC	15.10.2018
170	DIN ISO 16000-3	16. Oktober 2018
174	HPLC-UV	30/10/2018
176	Instituts-Methode	11.10.2018

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Participant	Analytical method	Date start sample preparation
184	QMA Nummer: MS-0023257	19.10.18
186	N EN ISO 16000-3	16/10/18
192	ISO16000-3	2018/10/11
195	NIOSH 2016	24-10-18
199	in Anlehnung an DIN 16000-3:2013-01	02.11.2018
202	in Anl. 16000-3	18.10.2018
207	DIN ISO 16000-3	18.10.2018
208	ISO 16000-3	10.10.2018
225	DFG-Luftanalysen, Aldehyde Nr.2	10.10.2018
229	HPLC	11-10-2018
241	ISO 16000-3	10.10.18
258	ISO16000-3	15-10-2018
264	HPLC/UV	11/10/2018
265	DIN ISO 1600-3	10.10.2018
267	interne Methode	22/10/2018
279	IFA 6045	19. Oktober 2018
292	HPLC w ith UV/Vis Detection	10/10/2018
293	NIOSH 2016:2003	October, 12 2018

Participant	Storage time after desorption	Date of analysis
10	1 day refrigerator	18/10/2018
11	no storage after desorption	17/10/2018
25	nein, Analyse direkt nach Desorption	16.10.2018
29	nein	11.10.18
30	< 4h at room temperature	22/10/18
52	keine Lagerung	30.10.2018
55	7 days - refrigerator	18/10/2018
56	1 day in refrigerator	10/10/18
58	as soon as the samples w ere desorbed they w ere placed in the autosampler	19 october 2018
60	0	12/10/2018

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Participant	Storage time after desorption	Date of analysis
62	10 days in fridge	29/10/2018
68	7 Tage im Kühlschrank	23.10.18
69	Three w eeks after desorption in refrigerator	14/11/2018
81	1 day at room temperature	16/10/2018
83	3h	11/10/18
100	1 day at 5°C	10/12/2018
114	some hours in the autosampler (10°C)	17/10/18
123	no storage	19/10/18
135	nein	12.10.2018
138	Kühlschrank	05.11.2018
141	Right after preparation, roomtemp.	23-10-2018
151	1Day RT 1 Day Freezer	12/10/2013
155	Analyse direkt im Anschluss der Desorption, Rückstellproben Lagerung im Kühlschrank,	15.10.2018
165	nein	12/10/2018
167	30 min for first sample	15.10.2018
170	RT	17. Oktober 2018
174	6 days	6 november
176	bis 22.10.2018 im Kühlschrank bei 2° C	23.10.2018
184	Nein	19.10.18
186	No storage after desorption	16/10/18
192	Set in the Analyzer immediately after desoeptiion. Roomtemp 21.7 degC	2018/10/12,13
195	1 DAY AT REFRIGERATOR	25-10-18
199	Tiefkühlschrank, 24Tage	02.11.2018
202	-18°C, 10 Tage	18.10.2018
207		18.10.2018
208	-	10.10.2018
225	Analyse direkt nach der Aufarbeitung	11.10.2018
229	2 days, in freezer about -18C	11-10-2018
241	2-5 Tage im Kühlschrank	11.10.18 bis 16.10.18
258	4 hours at roomtemperature in vial	15-10-2018
264	<1 day in refrigerator	11/10/2018

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Participant	Storage time after desorption	Date of analysis
265	nein	10.10.2018
267	0 Tage	22/10/2018
279	Kühlschrank bei 8°C	ab 19. Oktober 2018
292	2 h at RT	10/10/2018
293	less than 8 h	October, 12 2018

Participant	Desorption solution	Volume of desorption solution
10	Acetonitrile	5 ml
11	acetonitrile	5 mL
25	Acetonitril	10 ml
29	Acetonitril	5
30	acetonitrile	5 ml
52	Acetonitril	30 ml
55	acetonitrile	3
56	acéonitrile	5
58	acetonitril	10 ml
60	ACN	3
62	Acetonitrile	5 mL
68	Acetonitril	10mL
69	Acetonitrile	5 ml
81	Acetonitrile	10 mL
83	ACN	10ml
100	acetonitrile	4 ml
114	Desorption w ith acetonitrile	2 mL
123	acetonitrile	5
135	Acetonitril	2 ml
138	Acetonitril	10 ml
141	Acetonitril	10
151	Acetonitrile	5 ml
155	Acetonitril	5 ml

Participant	Desorption solution	Volume of desorption solution
165	Acetonitril	3 ml
167	Acetonitrile (AcN)	8 mL (filled to 10 mL w ith distilled w ater)
170	Acetonitril	
174	acn	6 ml
176	Acetonitril	5ml
184	Acetonitril	5ml
186	acetonitrile	10 mL
192	Acetonitrile	5mL
195	ACETONITRILE	5 mL
199	Acetonitril	3mL
202	ACN	10
207	ACN/H2O	5
208	Acetonitrile	3 ml
225	Acetonitril	in 5 mL
229	acetonitrile	5 ml
241	Acetonitril	2
258	Acetonitrile	5.00
264	Acetonitrile	5mL
265	Acetonitril	10 ml
267	Acetonitril	5 ml
279	Acetonitril	5 ml
292	Acetonitrile	5 mL
293	acetonitryl:w ater 80:20 solution w ith DNPH	10

Participant	Chromatography system (HPLC)
10	Perkin Elmer, Series 200-DAD
11	HPLC-UV
25	Agilent Infinity 1260: Quad Pump, 1260 MWD VL, 1260 ALS
29	Niederdruckpumpe, DAD, Autosampler
30	Waters Alliance 2695 + PDA 2996

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Participant	Chromatography system (HPLC)
52	Perkin Elmer Flexa HPLC mit PDA
55	Acquity Waters UPLC system
56	Thermo
58	HPLC agilent 1100
60	Agilent
62	Agilent 1200 series w ith UV detector
68	Agilent HPLC 1260 Infinity
69	Elite La Chrom Merck Hitachi, Pump: L-2130; Autosampler: L-2200; Detector: Uv-visibleL-2420
81	Pump Waters Alliance system. Detector QDa Waters
83	LaChromElite VWR
100	LC-UV Agilent
114	Thermoscientific HPLC U3000, UV detector
123	HPLC/DAD Agilent technologies 1260 infinity
135	Agilent 1290 Series
138	Thermofisher
141	Pump: Quarternair DEAB703330, Detector: LC-DAD-UV/VIS DEAAX 02347, Autosampler Agilent DEAAC08258
151	Waters Acquity H classs
155	Agilent 1200 LC System DAD-Detektor
165	HPLC Agilent 1100-DAD
167	Waters e2695 w ith Waters 2487 detector
170	Gradientenpumpe / DAD / Detektor
174	Shimadzu SIL20AD; UV
176	Ultimate 3000/UV_VIS
184	Agilent 1100 Serie
186	HPLC-PAD, quaternary pump
192	Agilent 1260,Agilent1260‡ (Agilent Technorogies)
195	Infinity 1290 Agilent
199	Agilent
202	Agilent G1312B-Pump, G4212B-DAD, G1329A-Autosampler
207	Agilent 1260 Infinity LC-System mit DAD
208	Acquity H-class, PDA-detector

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Participant	Chromatography system (HPLC)
225	quat. Pumpe Ser. 200, DAD Ser. 200 EP, Ser. 200 Autosampler (alle PerkinElmer)
229	HPLC system, Nexera, Shimadzu. Detector -DAD.
241	HP 1100
258	Waters Acquity HClass w ith UV detector
264	Shimadzu Nexera i-series
265	Shimadzu
267	Agilent (Quat Pump) 1260, DAD
279	Modell: SD 200, Modell: UV-1 beides Fa. Rainin; Basic Marathon, Fa. Spark Holland
292	Waters 1515/2489/2707
293	Perkin Elmer Flexar

Participant	Refrigerated Autosampler	Analytical column
10	No	Brow lee C18 150x4,6mm, 5µm
11	4 °C	C18
25	20 °C	Zorbax Eclipse Plus C18, 4.6 x 100mm, 3.5-Micron
29	nein	Waters XBridge Phenyl 3,5µm, 4,6x150mm
30	No	Allure AK 200 4.6 mm 5 µm
52	nein	Kromasil 100 C18 von MZ
55	yes/20°C	Waters Acquity UPLC BEH Phenyl
56	yes, 15°C	RSCL Carbonyl (100*2.1mm, 2.2µm)
58	no	zorbax ODS 1.6 nm id * 25 cm (5 µm)
60	no	C18
62	no	Ascentis RP amide
68	Nein	Poroshell 120 EC-C18 4,6x50mm, 2,7um
69	No	Ascentis RP-Amide 25 cmx 4,6 mm
81	Not used a refrigerated autosampler	BEH C18 Waters
83	yes, 15°C	Kinetex C18
100	no	RAPTOR ARC-18 100*3*2.7mm
114	refrigerated autosampler, 10°C	Acclaim 120, C18, 5µm, 250 mm x 4.6 mm
123	yes, 4°C	supelco discovery C 18 (250*4,6 mm, 5µm)

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Participant	Refrigerated Autosampler	Analytical column
135	Ja, 10°C	M&N EC 250/4.6 Nucleodur 100-5 C18ec
141	No	Formaldehyde: Agilent Poroshell 120 EC-C18, Other components: Supelcosil LC-18
151	20 C	UHPLC BEH C18 2 1.7µm 100 mm
155	gekühlter Autosampler; 20°C	Agilent Zorbax Eclipse XDB-C18; 4,6 mm x 150 mm x 5 µ
165	nein	LC-18
167	Waters e2695	Waters Symmetry C18, 3,5 µm, 4,6 x x100 mm
170	20°C	C18
174	4 °C	C18 250mm*4.6*5µm
176	nein	2 C18 Säulen 5µm Dionex
184	Ungekühlt	EC 50/3 NUCLEODUR Sphinx RP, 1,8µm
186	Yes, 4°C	WATERS Nova-Pak C18/ 150nm*3.9nm*4µm
192	Cooling function OFF (Roomtemp)	Formaldehyde,Acetaldehyde InertSustain C18 Propionaidehyde,Butyraldehyde Inertsil ODS-HL
195	no	Zorbax eclipse plus C18 4.6 x 100 mm x 1.8 um
199	Nein	C18
202	nein	Phenomenex Kinetex EVO C18, 3.0x50mm, 2.6um
207		Phenomenex Kinetex 2,6µ 100*4,6 mm
208	yes 15 C	BEH C18 2,1x 100mm 1,7µm
225	nein, Raumtemperatur	Kromasil 100 C18, 250 mm Länge, 3 mm ID, 5 µm Partikelgröße
229	Autosampler w as termostated at 28C	C18, RP, 200 mm x 4,6 mm, 5 micrometer (Restek)
241	nein	ULTRASEP ES ALD 125 mm x 2 mm
258	No	Waters Acquity UPLC BEH C18 1.7µm 2.1 x 50 mm
264	no	Acclaim Carbonyl
265	ja, 15°C	Agilent Zorbax, RHHD Eclipse Plus C18, 2,1x150mm, 1,8µm
267	nein, bei 25 °C	Symmetry C18, 250 mm x 4.6 mm x 5 µm (Waters)
279	nein	Luna 5µm C18(2), (150x4,6) mm Fa. Phenomenex
292	no	Supelco C18
293	No	Perkin Elmer Brow nlee SPP 2.7 um C18 4.6x100 mm

Participant	Mobile phase	Flow rate HPLC	Wavelength
10	Acetonitrile/Water (60/40)	1 ml/min	365 nm

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Participant	Mobile phase	Flow rate HPLC	Wavelength
11	acetonitrile/w ater/methanol	0,8 mL/min	360 nm
25	60/30/10 Wasser/Acetonitril/Tetrahydrofuran, 60/40 Acetonitril/Wasser	1,5 ml/min	360 nm
29	A: H2O, ACN, THF B: ACN	1,5	365
30	acetonitrile / w ater	1.2	360 nm
52	Acetoniril Methanol/Wasser	1ml/min.	365nm
55	gradient elution of 10%THF in w ater and acetonitrile	0.5	360
56	Water/acetonitrile	0.4 mL/min	360nm
58		1 ml/min	360 nm
60	H2O / ACN	1	360
62	Acetonitrile/w ater gradient	1 mL/min	360 nm
68	Gradient: Acetonitril, Methanol, Wasser	1	365nm
69	Acetonitrile-w ater	1,5 ml/min	UV-visible
81	Acetonitrile and Water w ith acid acetic + ammonium bicarbonate	0.3 mL/min	not applicable because use un mass detector
83	H2O/ACN/THF	1.2	
100	w ater/THF/ACN	0.6ml/min	360 nm
114	ACN/w ater	1 mL/min	360 nm
123	Acetonitrile/w ater	1	365.4 nm
135	Wasser-Acetonitril-Tetrahydrofuran	2,25 ml/min	365 nm
141	Acetonitril / w ater	1	360 nm
151	60/40 w ater/acetonitrile	0.5	360
155	Gradient Start ACN 30%/THF 10%/H2O 60%	1 ml	360 nm, 365 nm, 380 nm
165	Wasser/Acetonitril (40/60)	1,3 ml/min	360nm
167	AcN w ith 0,1 % Phosphoric Acid	1,5 mL/min	360 nm
170	Acetonitril/Wasser	0,8	365nm
174	ACN/H2O (60/40) gradient	1	360 nm
176	Acetonitril/Wasser 40_60	1,1ml/min	360nm
184	H2O+10% THF / ACN	0,9 ml/min	360nm
186	acetonitrile/w aterTHF	1.5	360 nm
192	Water/acetonitrile	1.2mL/min	360nm
195	ACN:H2O 40:60	1.8	365
199	Acetonitril/Wasser	0,8mL/min.	370nm

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Participant	Mobile phase	Flow rate HPLC	Wavelength
202	Gradient mit Wasser und ACN/MeOH/THF	0.8	365nm
207	ACN/THF/H2O	1,5	360 nm
208	ACN/THF/w ater	0,42 ml/min	360 nm
225	CH3CH 50-H2O 50, 2 min, dann auf 97,5 % CH3CN innerhalb von 10 min	0,60	360 nm
229	60% acetonitrile/40 % w ater	1 ml/min	360 nm
241	Acetonitril/Wasser/THF	0,5	365 nm
258	Water/acetonitrile gradient	0.8	367 nm
264	ACN/Buffer		
265	Wasser/Acetonitril 55:45	0,25 ml/min	356 nm
267	Acetonitrile/Wasser	1,5 ml/min	365 nm
279	A= ACN:H2O = 40%:60%; B= 100% ACN	1,0	365 nm
292	60% MeOH in w ater	1.5 mL/min	365 nm
293	Acetonitrile/w ater	1	360 nm

Participant	Column temperature	Recovery rate
10	25°C	No
11	30 °C	-
25	40°C	nein
29	27	nein
30	30	no
52	25°C	nein
55	40°C	yes
56	28°C	no
58	35°C	no
60	20°C	no
62	30 °C	no
68	Raumtemperatur	Ja, Formaldehyd 94%
69	40 ºC	No
81	22°C	NO
83	40°C	No

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Participant	Column temperature	Recovery rate
100	40°C	no
114	40°C	No
123	25°C	no
135	45 °C	nein
141	30 degrees C	No
151	50 C	No
155	35°C	nein
165	25°C	nein
167	40 °C	Yes
170	30°C	ja
174	no	
176	24°C	bei Propanal
184	40°C	Nein
186	35°C	No
192	40degC	No
195	30 ° C	NO
199	30°C	Nein
202	40°C	nein
207	30°C	
208	40 C	No
225	30	nein
229	35C	no
241	25°C	nein
258	40°C	No
264		Yes
265	45	nein
267	25°C	nein
279	22°C	nein
292	38oC	no, recovery is very high (>95%)
293	30	no