IFA
Institut für Arbeitsschutz der
Deutschen Gesetzlichen Unfallversicherung

Round-robin tests for

in-house measuring laboratories

Institut für Arbeitsschutz der Deutschen Gesetzlichen Unfallversicherung (IFA)
Institute for Occupational Safety and Health of the German Social Accident Insurance

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Results and Evaluation

Organic solvents with sampling

03/04 November 2015

Summary of laboratory test results

Sample 1

	4-Methyl-2-Pentanone	Z score	2-Butanone	Z score	Cycclopentanone	Z score
Unit	mg/m³		mg/m³		mg/m³	
5	64,56	0,65	134,09	0,50	36,08	-1,97
18	62,10	0,24	125,50	-0,17	48,70	0,84
37	66,30	0,94	128,40	0,05	25,30	-4,37 FE
40	65,05	0,73	127,40	-0,03	47,84	0,65
72	67,70	1,17	143,40	1,23	23,60	-4,74 FE
78	58,45	-0,36	118,79	-0,70	46,29	0,31
111	58,20	-0,40	119,10	-0,68	46,20	0,29
146	54,00	-1,09	125,00	-0,21	23,30	-4,81 FE
158	59,80	-0,14	135,30	0,59	48,70	0,84
236	60,04	-0,10	131,98	0,33	45,56	0,14
271	50,70	-1,64	116,00	-0,92	39,90	-1,12
281			160,00	2,53 BE		
–	–	– –	–	– –	–	– –
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	11		12		11	
Mean	60,63		127,72		44,91	
Reproducibility s.d.	5,23		8,16		4,54	
Rel. reproducibility s.d.	8,63 %		6,39 %		10,11 %	
Reference value	60,90		122,90		47,20	
Target s.d.	6,06		12,77		4,49	
Rel. SDPA	10,00 %		10,00 %		10,00 %	
Lower limit of tolerance	48,50		102,18		35,93	
Upper limit of tolerance	72,75		153,27		53,89	
Type B outliers			1			
Type F outliers					3	
No. of laboratories after elimination of outliers type A-D and F (without laboratories that only gave states but no measured values)	11		11		8	

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	
L: Differing laboratory mean (Grubbs II)	Grubbs II

Summary of laboratory test results

Sample 2

	n-Butyl acetate	Z score	Toluene	Z score	m-Xylene	Z score	n-Hexane	Z score	n-Octane	Z score	Ethylbenzene	Z score
Unit	mg/m³		mg/m³		mg/m³		mg/m³		mg/m³		mg/m³	
5	76,63	0,51	64,96	0,42	60,40	-4,26 BE	58,85	0,90	131,76	0,42	94,10	8,62 BE
18	80,30	1,01	59,20	-0,50	100,90	-0,41	52,60	-0,26	134,00	0,59	50,10	-0,09
37	78,30	0,74	69,70	1,18	112,50	0,70	71,90	3,32 E	152,10	2,02 E	59,80	1,83
40	77,29	0,60	65,42	0,50	110,71	0,53	49,66	-0,80	131,36	0,38	52,63	0,41
72	71,90	-0,14	64,30	0,32	103,30	-0,18	53,70	-0,05	133,40	0,55	50,40	-0,03
78	73,56	0,09	61,36	-0,15	106,83	0,16	48,35	-1,04	127,08	0,05	50,82	0,06
111	66,60	-0,87	56,60	-0,92	96,20	-0,85	43,00	-2,03 E	113,60	-1,02	45,10	-1,08
146	73,00	0,01	63,30	0,16	106,00	0,08	47,30	-1,24	127,00	0,04	49,80	-0,15
158	60,30	-1,73	57,80	-0,72	97,50	-0,73	53,10	-0,16	105,70	-1,64	45,70	-0,96
236	66,32	-0,91	56,46	-0,94	111,81	0,63	60,26	1,16	117,53	-0,71	45,59	-0,98
271	68,00	-0,68	55,70	-1,06	89,10	-1,53	51,00	-0,55	118,00	-0,67	46,00	-0,90
281	83,00	1,38	73,00	1,71	122,00	1,60	58,00	0,75	179,00	4,15 BE	60,00	1,87
–	–	– –	–	– –	–	– –	–	– –	–	– –	–	– –
Method	ISO 5725-2		ISO 5725-2		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		|Z|<=2,00		|Z|<=2,00	
No. of laboratories that submitted results	12		12		12		12		12		12	
Mean	72,93		62,32		105,17		53,98		126,50		50,54	
Reproducibility s.d.	6,65		5,49		9,13		7,55		12,50		5,28	
Rel. reproducibility s.d.	9,12 %		8,82 %		8,69 %		13,99 %		9,88 %		10,44 %	
Reference value	70,50		61,30		90,60		50,20		128,80		45,30	
Target s.d.	7,29		6,23		10,52		5,40		12,65		5,05	
Rel. SDPA	10,00 %		10,00 %		10,00 %		10,00 %		10,00 %		10,00 %	
Lower limit of tolerance	58,35		49,85		84,13		43,18		101,20		40,43	
Upper limit of tolerance	87,52		74,78		126,20		64,77		151,80		60,65	
Type B outliers					1				1		1	
No. of laboratories after elimination of outliers type A-D and F (without laboratories that only gave states	12		12		11		12		11		11	

	n-Butyl acetate	Z score	Toluene	Z score	m-Xylene	Z score	n-Hexane	Z score	n-Octane	Z score	Ethylbenzene	Z score

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-Score|>3,5	
L: Differing laboratory mean (Grubbs II)	Grubbs II

	1-Methoxy-2-propanole	Z score		1-methoxy-2-propyl acetate	Z score	
Unit	mg/m³			mg/m³		
5	137,03	1,91		114,04	5,87	BE
18	109,60	-0,47		77,20	0,75	
37	59,90	-4,79	BE	73,90	0,29	
40	121,10	0,52		78,04	0,86	
72	123,60	0,74		87,20	2,14	E
78	114,30	-0,07		73,32	0,21	
111	114,10	-0,08		64,40	-1,04	
146	102,00	-1,14		73,90	0,29	
158	126,20	0,97		62,50	-1,30	
236	107,72	-0,64		56,90	-2,08	E
271	95,00	-1,74		71,00	-0,12	
–	–	– –		–	– –	
Method	ISO 5725-2			ISO 5725-2		
Assessment	|Z|<=2,00			|Z|<=2,00		
No. of laboratories that submitted results	11			11		
Mean	115,06			71,84		
Reproducibility s.d.	12,36			8,69		

	1-Methoxy-2-propanole	Z score	1-methoxy-2-propyl acetate	Z score
Rel. reproducibility s.d.	10,74 %		12,10 %	
Reference value	104,60		69,30	
Target s.d.	11,51		7,18	
Rel. SDPA	10,00 %		10,00 %	
Lower limit of tolerance	92,05		57,47	
Upper limit of tolerance	138,08		86,20	
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)	10		10	

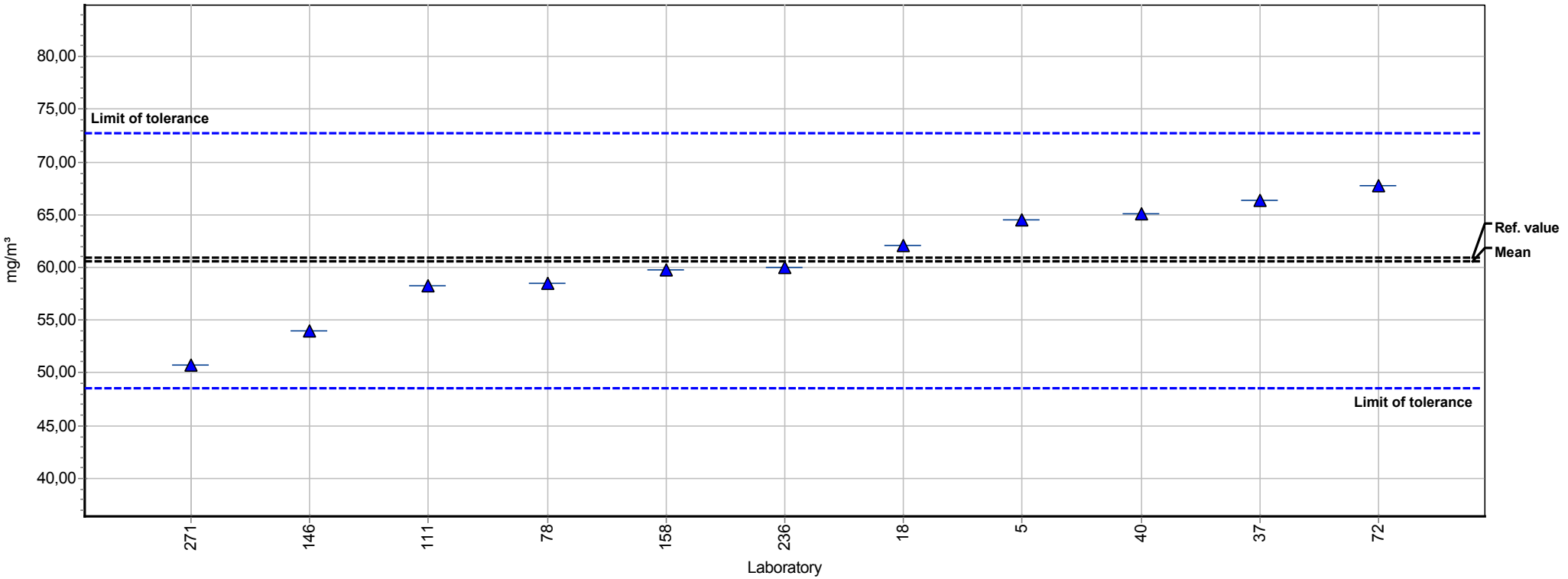
Summary of laboratory test results

Sample 3

	Benzene	Z score
Unit	µg/m³	
5	140,3000	1,16
18	121,1000	-0,36
37	< 1000,0000	
40	124,0500	-0,13
72	130,0000	0,34
78	132,8900	0,57
111	128,0000	0,18
146	117,0000	-0,69
158	118,4000	-0,58
236	111,0200	-1,17
271	134,0000	0,66
–	–	– –
Method	ISO 5725-2	
Assessment	|Z|<=2,00	
No. of laboratories that submitted results	11	
Mean	125,6760	
Reproducibility s.d.	8,9810	
Rel. reproducibility s.d.	7,15 %	
Reference value	127,4000	
Target s.d.	12,5676	
Rel. SDPA	10,00 %	
Lower limit of tolerance	100,5408	
Upper limit of tolerance	150,8112	
No. of laboratories after elimination of outliers type A-D and F (without laboratories that only gave states but no measured values)	10	
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		
L: Differing laboratory mean (Grubbs II)	Grubbs II	

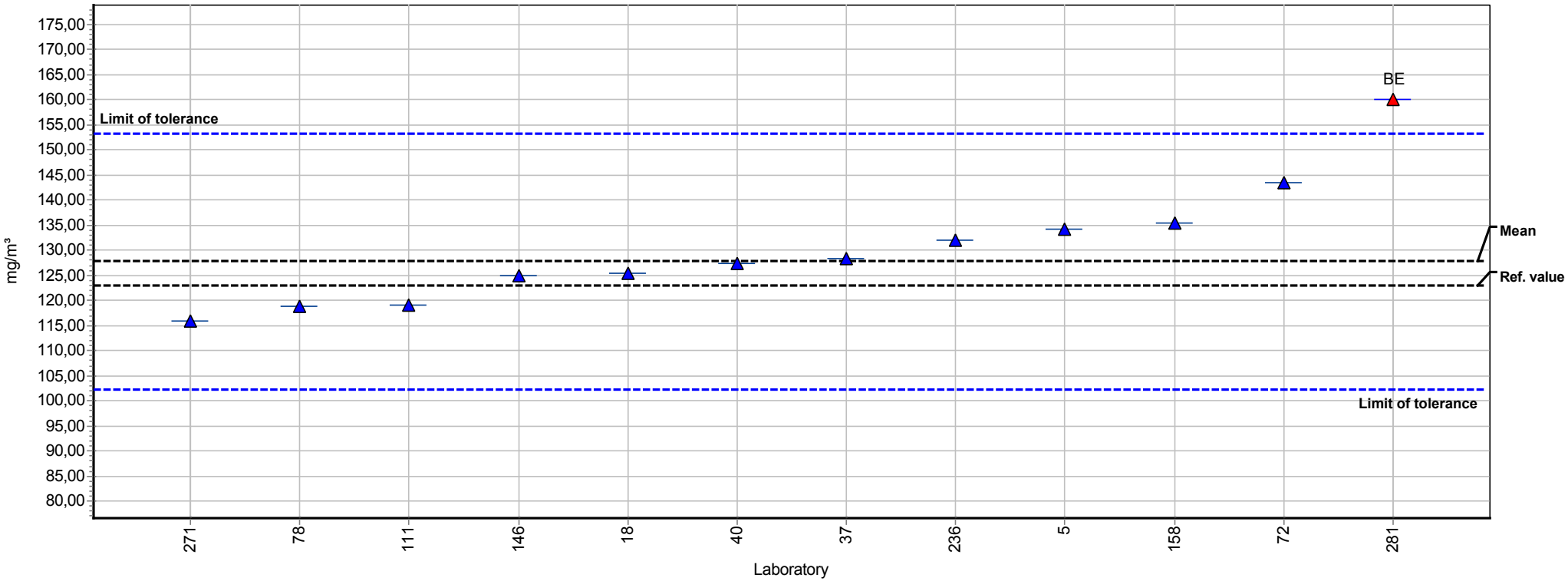
Summary results

Measurand:	**4-Methyl-2-Pentanone**	**Mean:**	**60,63 mg/m³**
Sample:	**1**	**Reproducibility s.d.:**	**5,23 mg/m³**
Method:	**ISO 5725-2**	**Relative reproducibility s.d.:**	**8,63%**
No. of laboratories:	**11**	**Reference value:**	**60,90 mg/m³**
Relative target s.d.:	**10,00% (Limited)**	**Range of tolerance:**	**48,50 - 72,75 mg/m³ (|Z-Score| <= 2,00)**



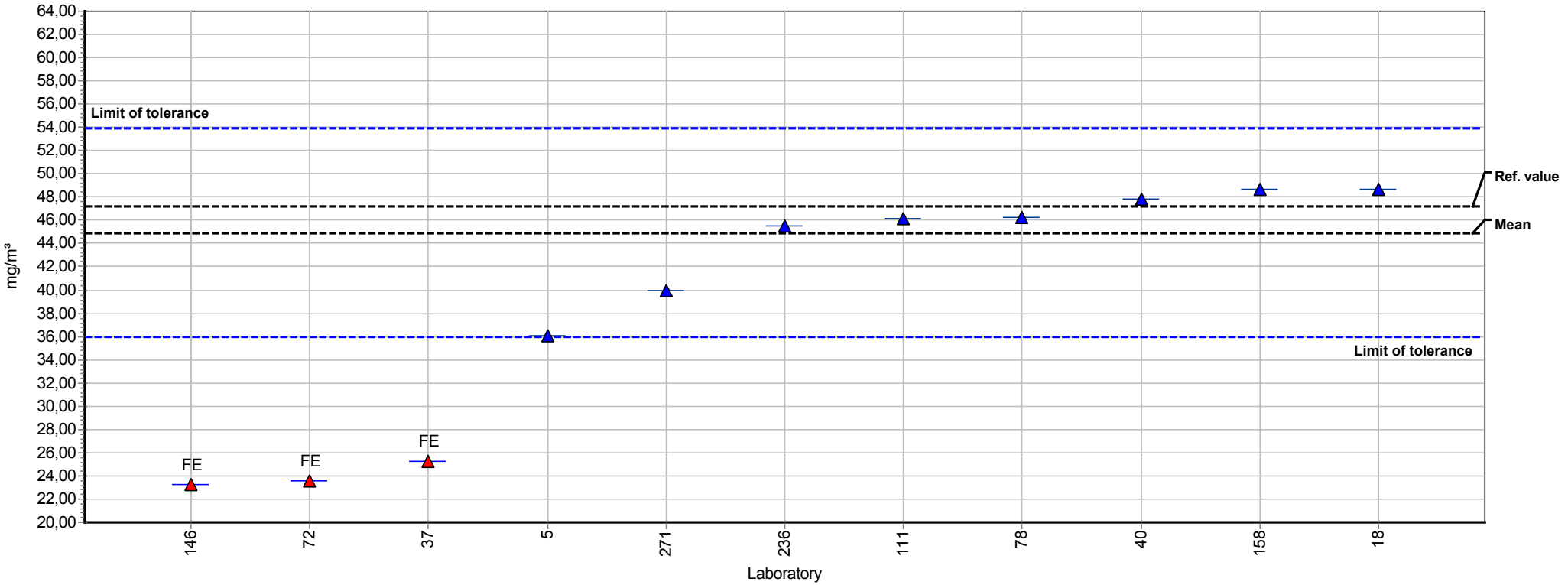
Summary results

Measurand:	**2-Butanone**	**Mean:**	**127,72 mg/m³**
Sample:	**1**	**Reproducibility s.d.:**	**8,16 mg/m³**
Method:	**ISO 5725-2**	**Relative reproducibility s.d.:**	**6,39%**
No. of laboratories:	**11**	**Reference value:**	**122,90 mg/m³**
Relative target s.d.:	**10,00% (Limited)**	**Range of tolerance:**	**102,18 - 153,27 mg/m³ (|Z-Score| <= 2,00)**



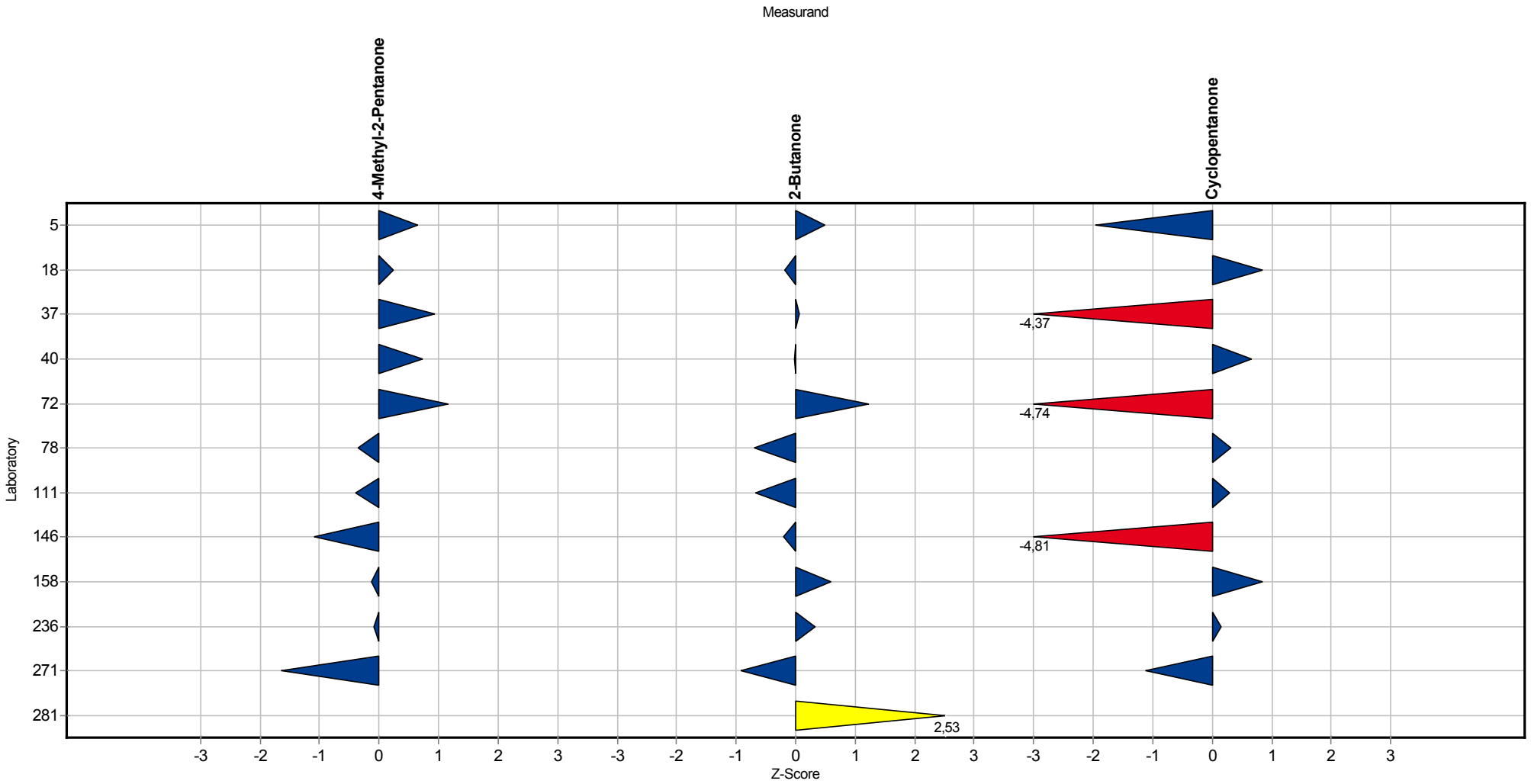
Summary results

Measurand:	**Cyclopentanone**	**Mean:**	**44,91 mg/m³**
Sample:	**1**	**Reproducibility s.d.:**	**4,54 mg/m³**
Method:	**ISO 5725-2**	**Relative reproducibility s.d.:**	**10,11%**
No. of laboratories:	**8**	**Reference value:**	**47,20 mg/m³**
Relative target s.d.:	**10,00% (Limited)**	**Range of tolerance:**	**35,93 - 53,89 mg/m³ (|Z-Score| <= 2,00)**



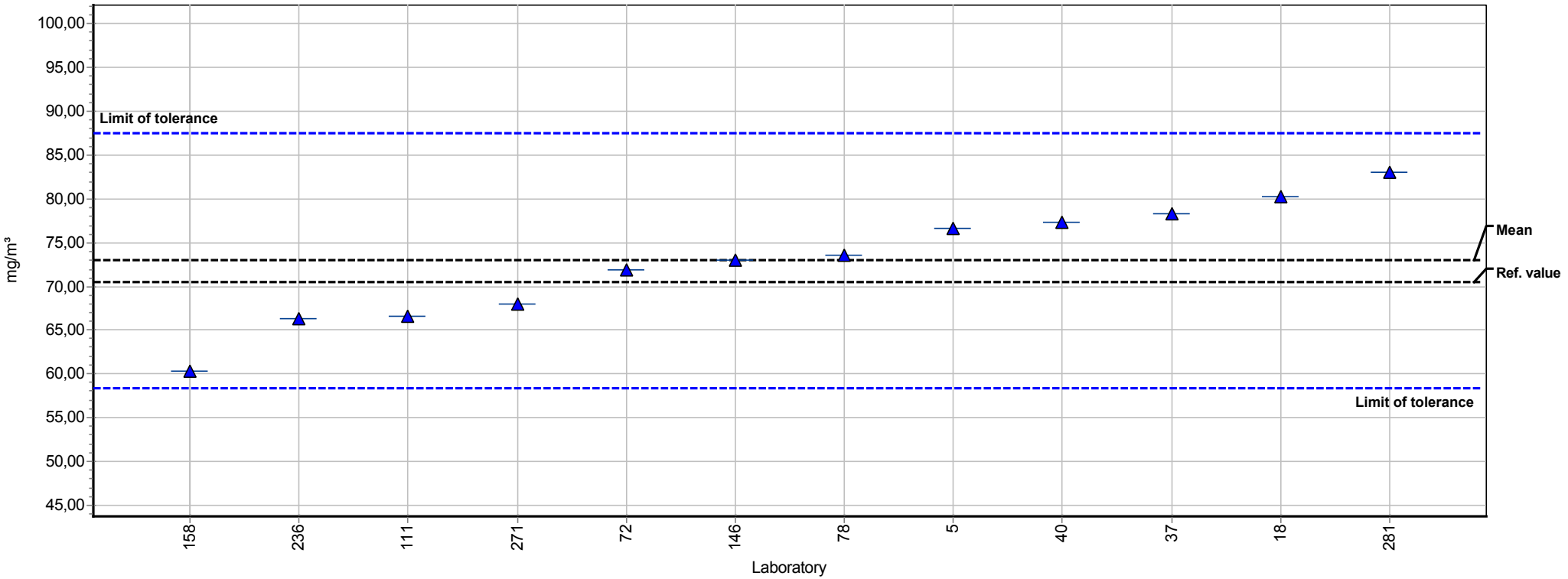
Sample chart of Z-scores

Sample 1



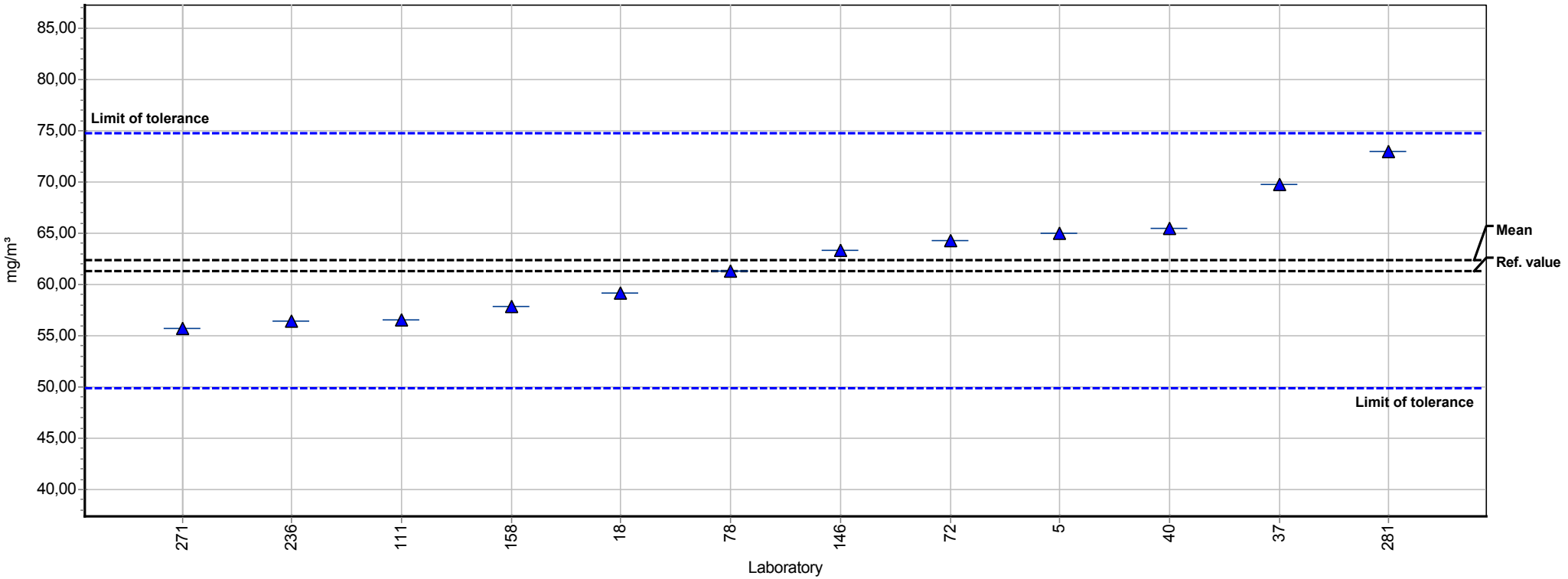
Summary results

Measurand:	**n-Butyl acetate**	**Mean:**	**72,93 mg/m³**
Sample:	**2**	**Reproducibility s.d.:**	**6,65 mg/m³**
Method:	**ISO 5725-2**	**Relative reproducibility s.d.:**	**9,12%**
No. of laboratories:	**12**	**Reference value:**	**70,50 mg/m³**
Relative target s.d.:	**10,00% (Limited)**	**Range of tolerance:**	**58,35 - 87,52 mg/m³ (|Z-Score| <= 2,00)**



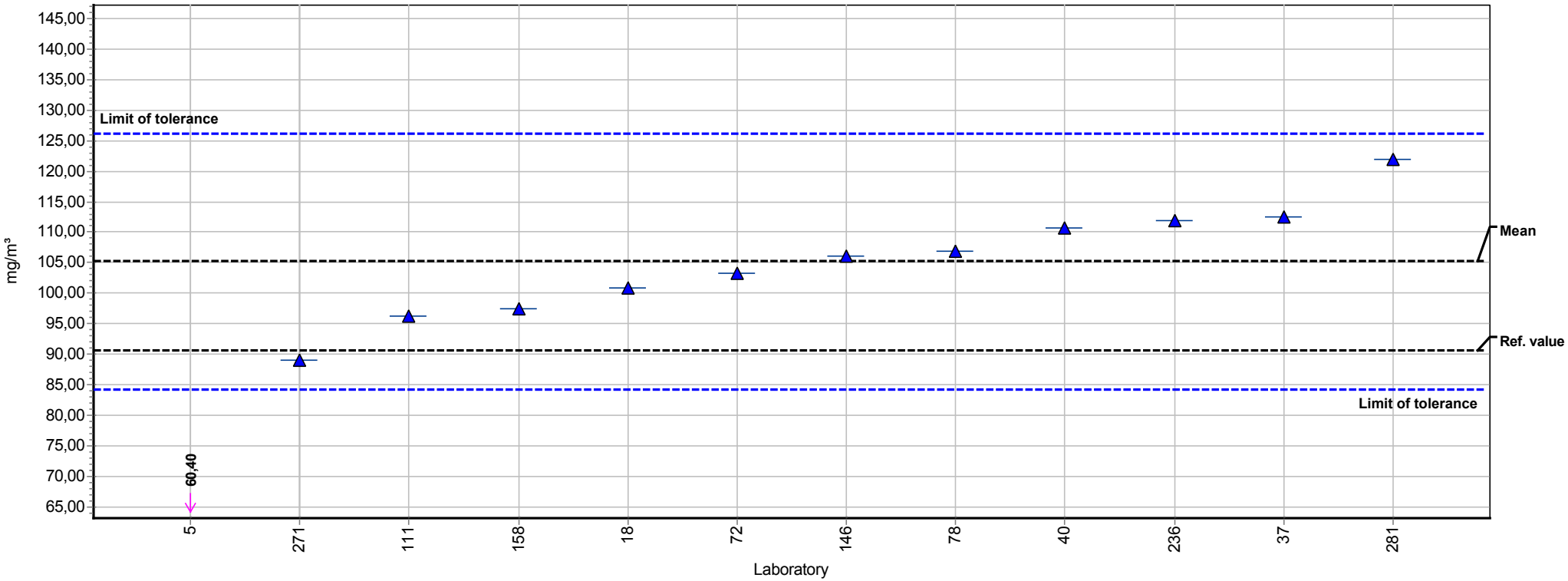
Summary results

Measurand:	**Toluene**	**Mean:**	**62,32 mg/m³**
Sample:	**2**	**Reproducibility s.d.:**	**5,49 mg/m³**
Method:	**ISO 5725-2**	**Relative reproducibility s.d.:**	**8,82%**
No. of laboratories:	**12**	**Reference value:**	**61,30 mg/m³**
Relative target s.d.:	**10,00% (Limited)**	**Range of tolerance:**	**49,85 - 74,78 mg/m³ (|Z-Score| <= 2,00)**



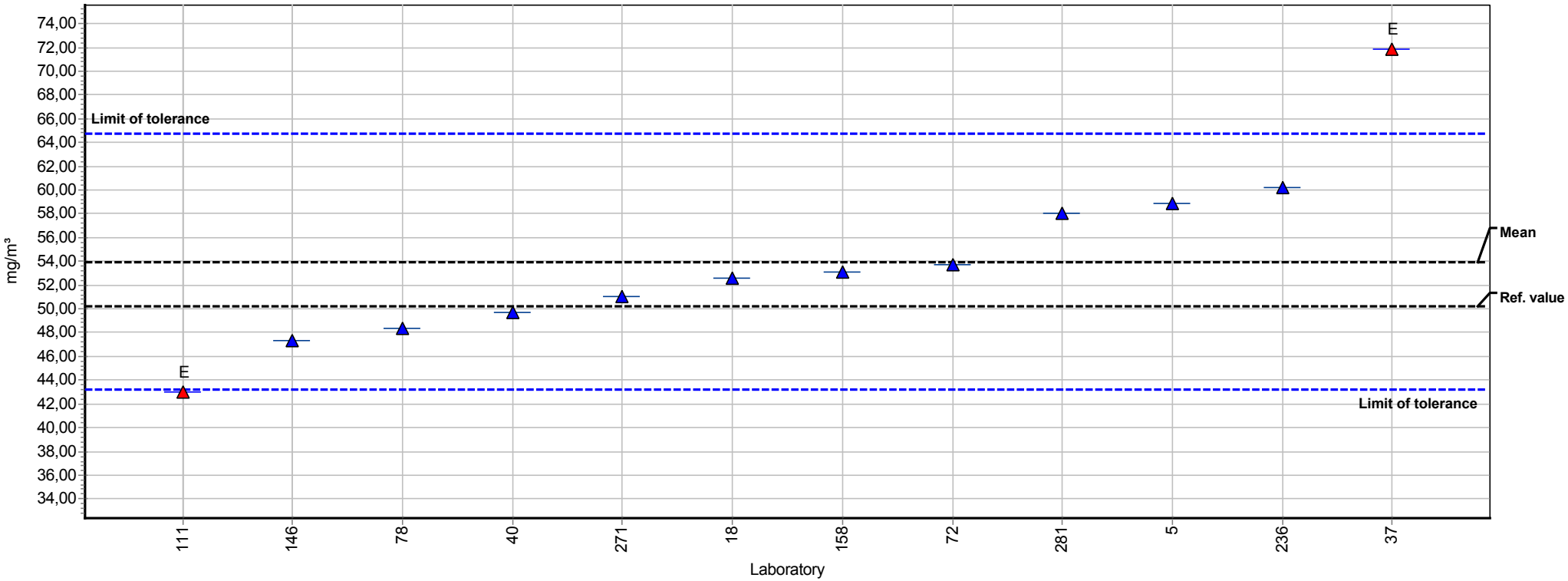
Summary results

Measurand:	**m-Xylene**	**Mean:**	**105,17 mg/m³**
Sample:	**2**	**Reproducibility s.d.:**	**9,13 mg/m³**
Method:	**ISO 5725-2**	**Relative reproducibility s.d.:**	**8,69%**
No. of laboratories:	**11**	**Reference value:**	**90,60 mg/m³**
Relative target s.d.:	**10,00% (Limited)**	**Range of tolerance:**	**84,13 - 126,20 mg/m³ (|Z-Score| <= 2,00)**



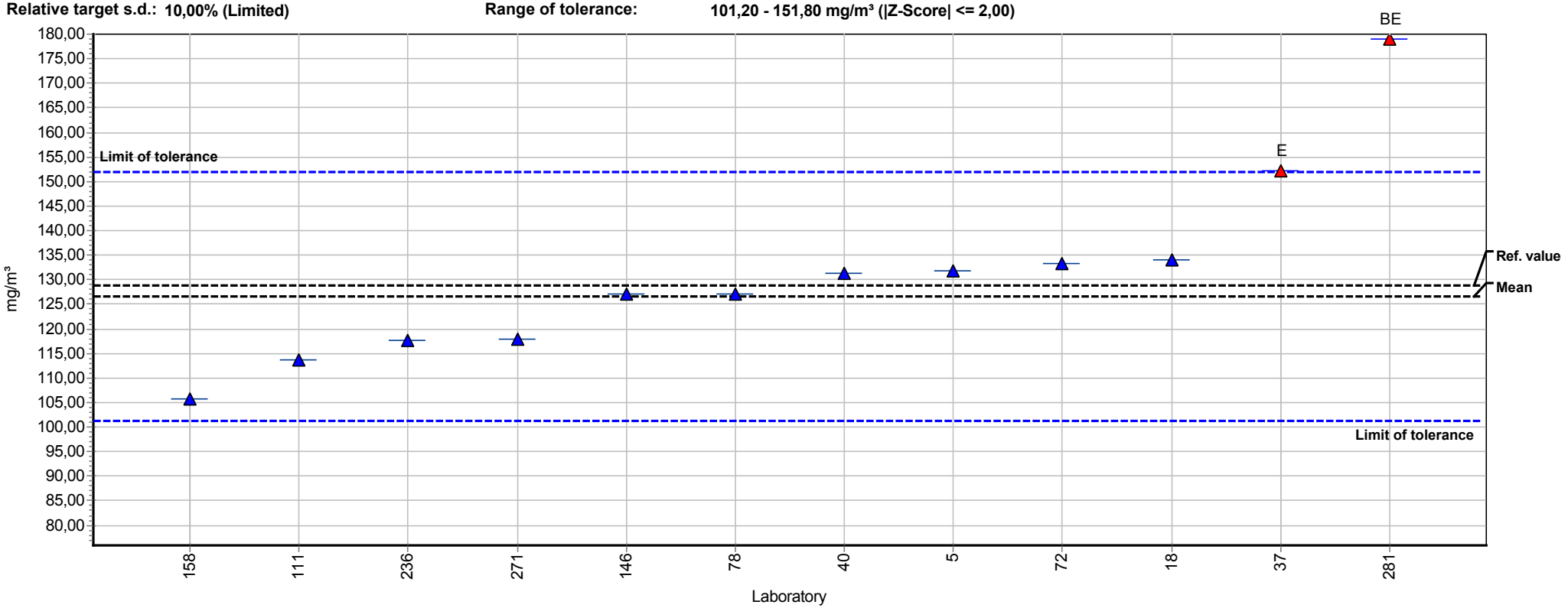
Summary results

Measurand:	**n-Hexane**	**Mean:**	**53,98 mg/m³**
Sample:	**2**	**Reproducibility s.d.:**	**7,55 mg/m³**
Method:	**ISO 5725-2**	**Relative reproducibility s.d.:**	**13,99%**
No. of laboratories:	**12**	**Reference value:**	**50,20 mg/m³**
Relative target s.d.:	**10,00% (Limited)**	**Range of tolerance:**	**43,18 - 64,77 mg/m³ (|Z-Score| <= 2,00)**



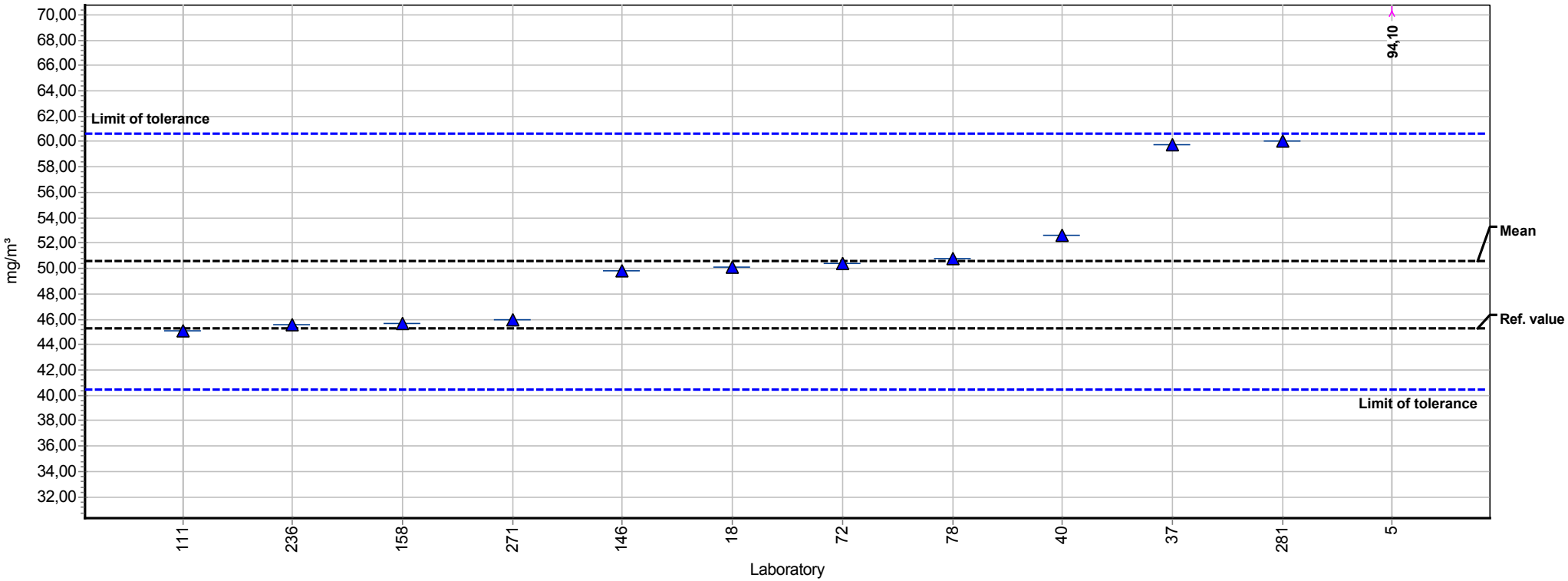
Summary results

Measurand:	**n-Octane**	**Mean:**	**126,50 mg/m³**
Sample:	**2**	**Reproducibility s.d.:**	**12,50 mg/m³**
Method:	**ISO 5725-2**	**Relative reproducibility s.d.:**	**9,88%**
No. of laboratories:	**11**	**Reference value:**	**128,80 mg/m³**
Relative target s.d.:	**10,00% (Limited)**	**Range of tolerance:**	**101,20 - 151,80 mg/m³ (|Z-Score| <= 2,00)**



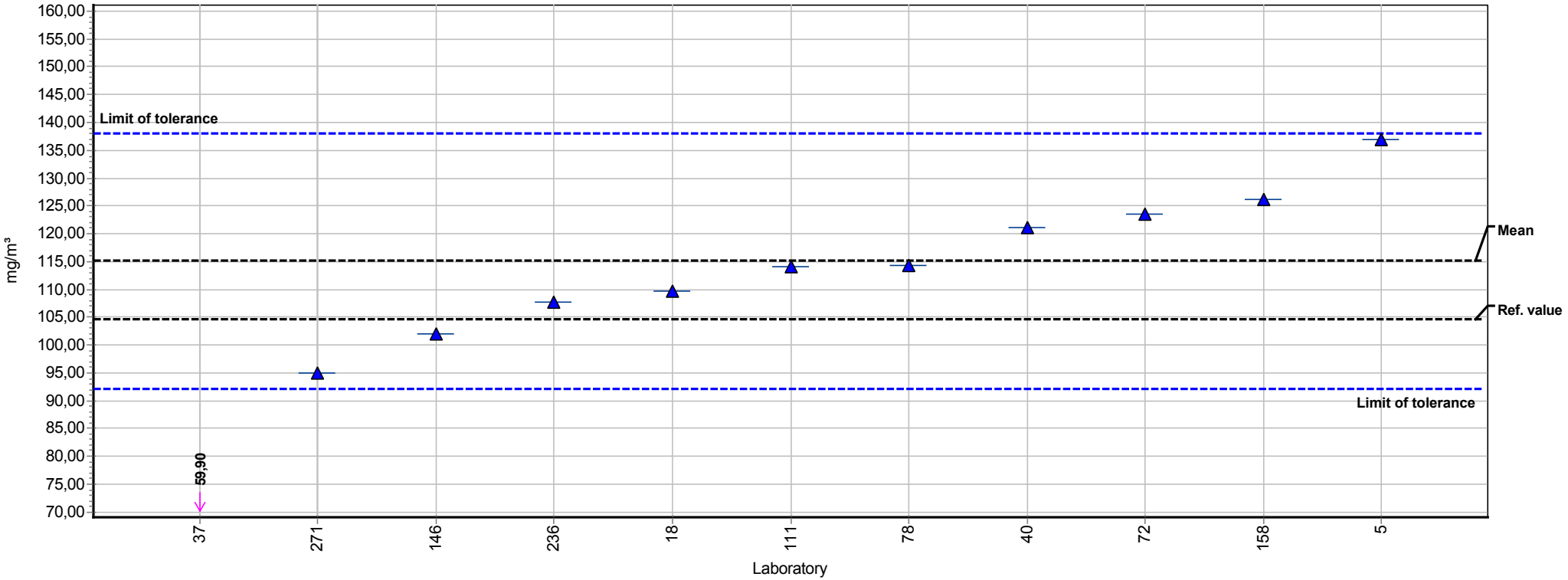
Summary results

Measurand:	**Ethylbenzene**	**Mean:**	**50,54 mg/m³**
Sample:	**2**	**Reproducibility s.d.:**	**5,28 mg/m³**
Method:	**ISO 5725-2**	**Relative reproducibility s.d.:**	**10,44%**
No. of laboratories:	**11**	**Reference value:**	**45,30 mg/m³**
Relative target s.d.:	**10,00% (Limited)**	**Range of tolerance:**	**40,43 - 60,65 mg/m³ (|Z-Score| <= 2,00)**



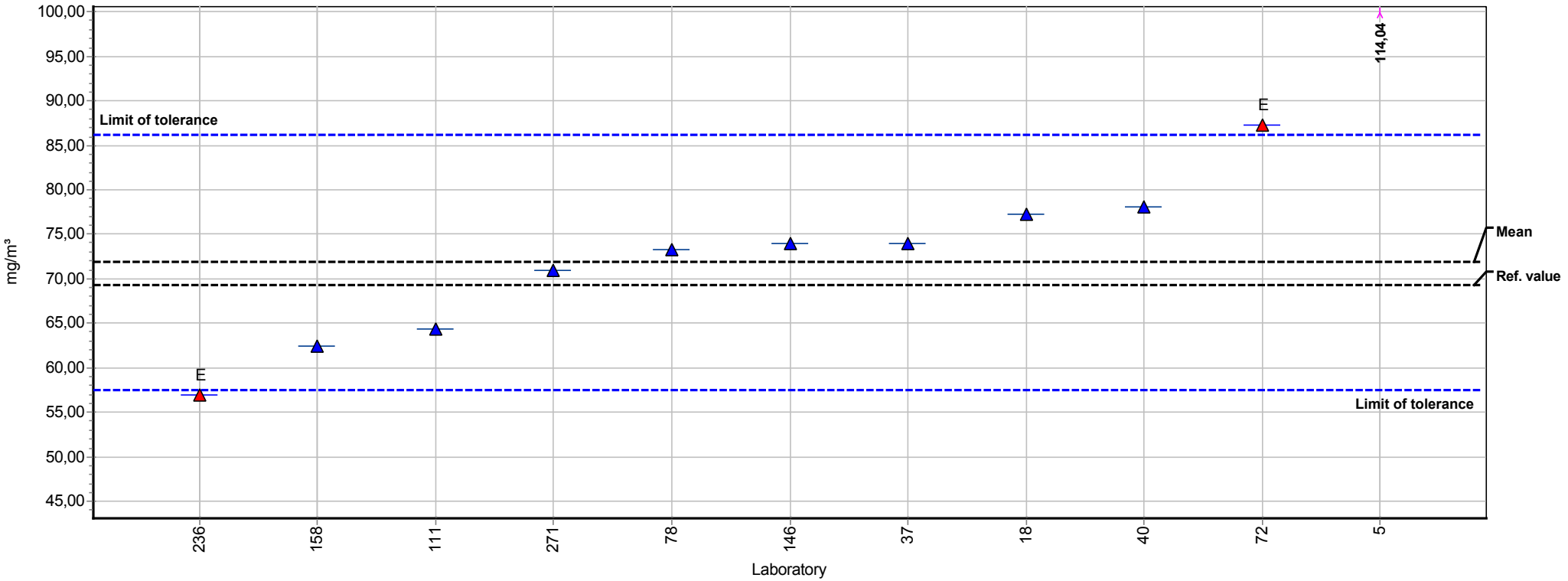
Summary results

Measurand:	**1-Methoxy-2-propanol**	**Mean:**	**115,06 mg/m³**
Sample:	**2**	**Reproducibility s.d.:**	**12,36 mg/m³**
Method:	**ISO 5725-2**	**Relative reproducibility s.d.:**	**10,74%**
No. of laboratories:	**10**	**Reference value:**	**104,60 mg/m³**
Relative target s.d.:	**10,00% (Limited)**	**Range of tolerance:**	**92,05 - 138,08 mg/m³ (|Z-Score| <= 2,00)**



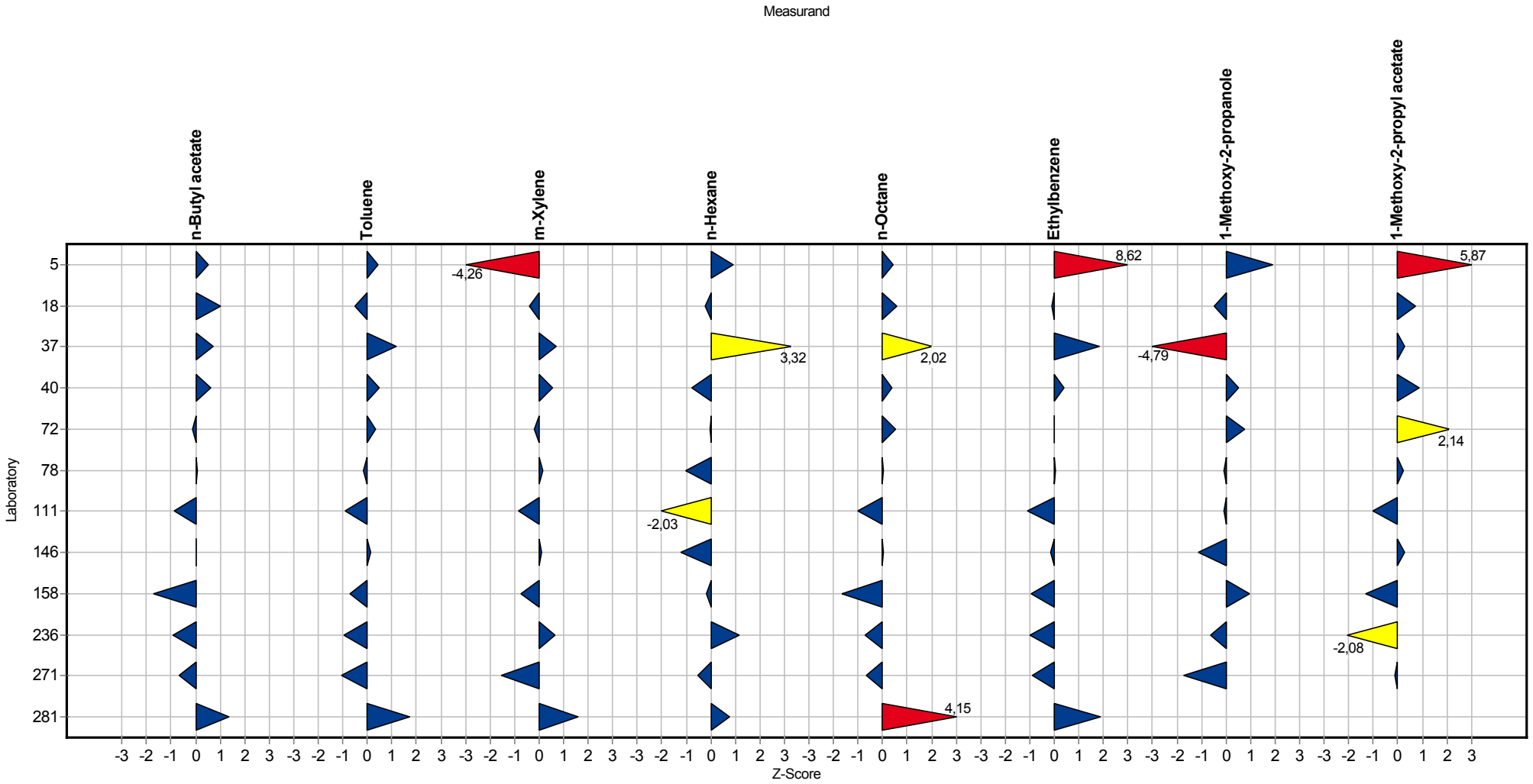
Summary results

Measurand:	**1-Methoxy-2-propyl acetate**	**Mean:**	**71,84 mg/m³**
Sample:	**2**	**Reproducibility s.d.:**	**8,69 mg/m³**
Method:	**ISO 5725-2**	**Relative reproducibility s.d.:**	**12,10%**
No. of laboratories:	**10**	**Reference value:**	**69,30 mg/m³**
Relative target s.d.:	**10,00% (Limited)**	**Range of tolerance:**	**57,47 - 86,20 mg/m³ (|Z-Score| <= 2,00)**



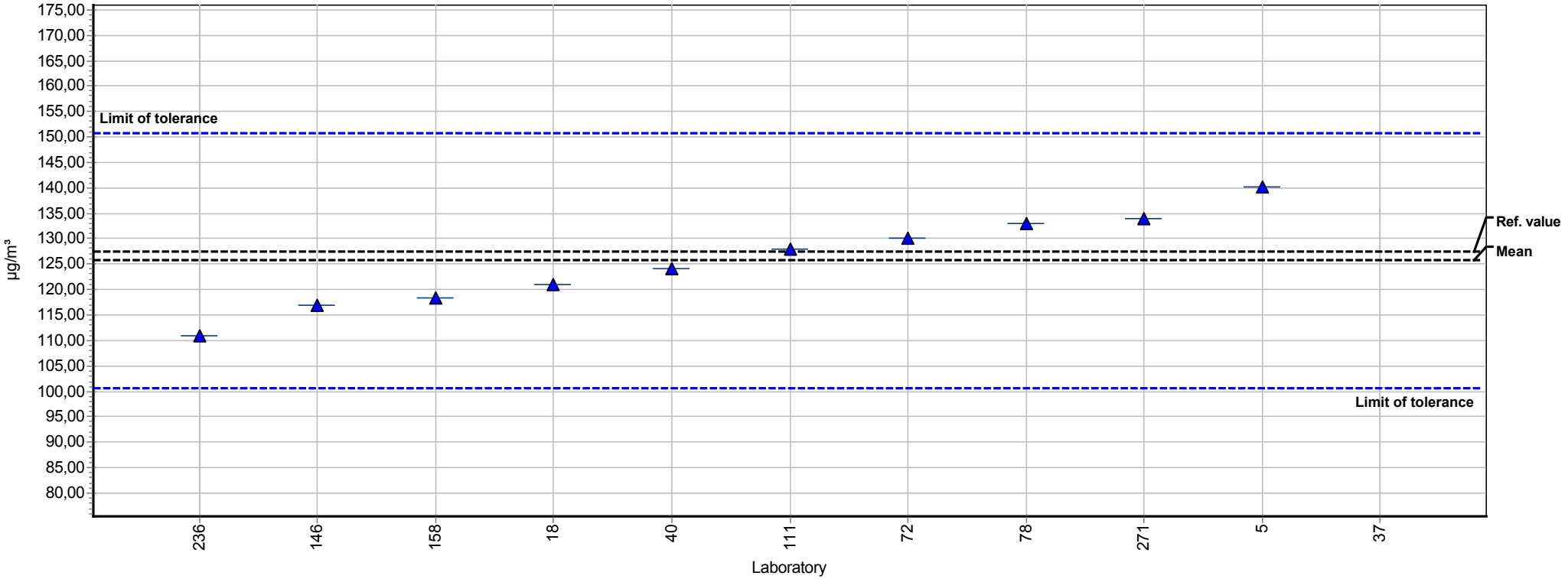
Sample chart of Z-scores

Sample 2



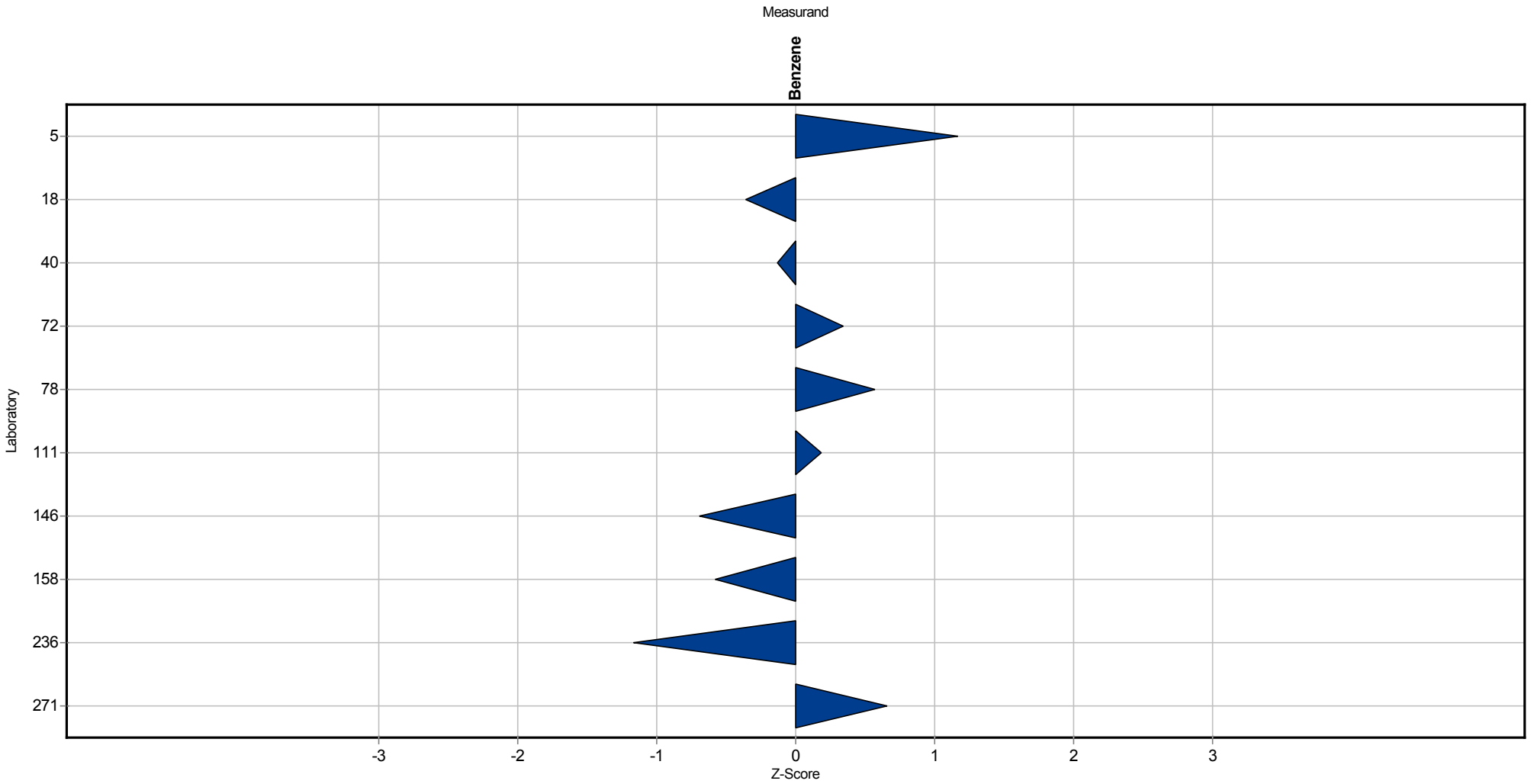
Summary results

Measurand:	**Benzene**	**Mean:**	**125,68 µg/m³**
Sample:	**3**	**Reproducibility s.d.:**	**8,98 µg/m³**
Method:	**ISO 5725-2**	**Relative reproducibility s.d.:**	**7,15%**
No. of laboratories:	**10**	**Reference value:**	**127,40 µg/m³**
Relative target s.d.:	**10,00% (Limited)**	**Range of tolerance:**	**100,54 - 150,81 µg/m³ (|Z-Score| <= 2,00)**



Sample chart of Z-scores

Sample 3



Questions and Answers

Participant	Sample carrier	Kind of pump	Flow rate
5	Aktickohle	Fa. SKC, PocketPump	70 und 250 ml/min
18	Silicagel Typ BIA und Aktivkohle Typ BIA	SG350 und SG4000ex, GSA Messgerätebau GmbH	ca. 0,083 l/min und ca. 0,333 l/min
37	A-Kohle Typ BIA und Silicagel Typ BIA, Fa. Dräger	Gilian PP1EX, LFS-113 DC	300 - 330 ml/min
40	Aktivkohle, Silicagel	Gilian LFS-113DC	15 bis 200 ml/min
72	Aktivkohle	SG 350 / SG 4000	200 bzw. 333 bzw. 1000 ml/min
78	Aktivkohle und Silicagel	Gilair und GSA (DEHA)	Gas1+2: 0,3 L/Min. und Gas 3 1,0 L/Min.
111	PG1=Silica, PG2+3=AK	SG-350	PG1=75ml/min, PG2+3=350ml/min
146	Aktivkohle Typ BIA, Silicagel Typ BIA (2-Butanon)	GilAir 5	333 mL/min. 83 mL/min.
158	Tenax TA	Gilian LFS-113 DC Low Flow Sampler	0,010 L/min und
236	Tenax TA	PSA LFS113, Gilian	10 mL/min
271	A-Kohle + Silicagel (Ketone)	GSA SG 350 + GSA SG 4000	0,33 l/min + 0,083 l/min (Ketone) + 1 l/min (Benzol)
281		SKC Delux	

Participant	Volume flow measurement	Sampling time
5	Fa. DEHA, Defender	2 h
18	Digitaler Durchflussmesser, Flowmeter 4140, TSI GmbH	120 min, gemäß IFA-Arbeitsmappe 6265, 7708, 7322, 7569, 7732, 7733
37	Analyt Massenflussmesser GFM 17, 0 - 500 ml/min, geeicht auf Luft	30 min und 60 min
40	DryCal DC-Lite	140 min
72	Defender 520 (50-5000 ml/min)	1 h bzw . 2 h
78	Gilibrator	Gas 1+2: 60 Min. und Gas 3: 120 Min.
111	Bios Defender 510M	120 min.
146	Gilibrator	90 Minuten
158	Bios Int. Corp. Defender 510-L Rev C1	60 min und 30 min
236	Gilibrator	15, 30, 60 min
271	DryCal DC Lite	2 h

Participant	Analytical method	Sample preparation
5	Hausmethode	Desorption mit CS2

Round-robin test Organic solvents with sampling 1/2015

Participant	Analytical method	Sample preparation
18	GCMS / IFA Arbeitsmappe 6265, 7708, 7322, 7569, 7732, 7733, 7735	-
37	Hausmethode	AK + 3ml Benzylalkohol, Headspace, Silicagel + 5ml Aceton , 30min Ultraschall, Direktinj.
40	validierte Hausmethoden	Schwefelkohlenstoff 1,5 ml; Dichlormethan/Methanol (9:1) 5 ml; Dichlormethan/Methanol/Wasser (65/35/2) 5 ml
72	validierte eigene SOP in Anlehnung an IFA Arbeitsmappe	Benzylalkohol und Gemisch Dichlormethan - Schwefelkohlenstoff - Methanol
78	IFA-Arbeitsmappe	Gas 1+2: 20 mL; Gas 3: 5 mL
111	PG1 = IFA7708, PG2 = IFA7322,7732, PG3 = IFA6265	PG1+PG2 = 2ml ternäres Gemisch, PG3 = 2ml CS2
146	2-Butanon DFG Methode Nr. 6 bzw. IFA Methode 7708	
158	VOC in Luft mittels Thermodesorption	Thermodesorption
236	Hausmethode	Thermodesorption
271	IFA 7733, IFA 6265, IFA 7569, IFA 7322, IFA 7732, IFA 7708	terneres Gemisch (65% Dichlormethan, 30% Schwefelkohlenstoff und 5 % Methanol) bzw. 100% Schwefelkohlenstoff

Participant	Carrier gas	Injection	Analytical column
5	Helium	split	HP1
18	-	-	-
37	Helium 5.0	Split	60m Rtx Volatile, 60m Stabilwax DA
40	Helium	split	DBWAX; DB1; HP5-MS; RTx-35
72	Helium 6.0	split	Phenomenex Zebron ZB-WAX, 30 m x 0,25 mm x0,25 mm
78	Stickstoff	split	DB-1
111	Helium	split	SE54
158	Helium	Inletsplit und Outletsplit	Restek RTX-1 60m, ID 0,25 x 1 µm
236	He	split/split	HP-5 50m ID=0.32mm Film=1.05µm
271	Stickstoff bzw. Helium (Ketone)	Split	Macherey Nagel Optima 1; 30 m * 0,32 mm ID; 5 µm Filmdicke bzw. Thermo TG-1301 MS; 30 m * 0,25 mm ID; 0,25 µm Filmdicke (Ketone)

Participant	Detector	Data evaluation	Recovery rate	Date of analysis
5	FID	ISTD	Ja	05.11.2015
18	-	-	-	05.11.-25.11.2015
37	FID	externer Standard	nein	19.11.2015
40	FID; massenslektiver Detektor	interner Standard	Ja	05.11.2015;
72	FID	externer Standard, 4 und 6 Pkt. Kalibrierung	ja (0,7 bis 0,95)	9.11. - 17.11.2015
78	FID	interner Standard	Ja	47. und 48. KW
111	PG1 = FID, PG2+PG3 = MS	interner Standard	nein	10.-27.11.15

Round-robin test Organic solvents with sampling 1/2015

Participant	Detector	Data evaluation	Recovery rate	Date of analysis
146				25.11.2015
158	MSD Agilent 5975C	Interner Standard	Es wurde ein Kontrollstandard eingesetzt	09.11.2015 - 20.11.2015
236	FID	externe Kalibration	nein	11. - 18.11.2015
271	FID, Range = 1; At = -4 bzw. MS (Ketone)	Interner Standard (Schwefelkohlenstoff mit Chlorbenzol, tern. Gemisch mit Undecan)	nein	10.11.2015 - 24.11.2015