

REPORT



DRRR-Proficiency Testing

RVEP 200262

Total carbon emission VDA 277 - PV 3341

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Proficiency testing scheme accredited by A2LA according to ISO/IEC 17043:2010.




Proficiency testing scheme accredited by DAkkS according to DIN EN ISO/IEC 17043:2010. The accreditation is valid only for the scope listed in the annex of the accreditation certificate [D-EP-17063-01-00]. The Deutsche Akkreditierungsstelle GmbH is a signatory of the multilateral arrangements of EA, ILAC and IAF for mutual recognition.


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<u>1. timetable</u>		 Deutsches Referenzbüro für Ringversuche und Referenzmaterialien
announcement of the proficiency testing:		12.03.2020
shipment of samples:		23.03.-27.03.2020
final date for results:		29.05.2020
preparation and dispatch of report:		14.08.2020

<u>2. participants</u>		 Deutsches Referenzbüro für Ringversuche und Referenzmaterialien
national (Germany)		7
international		21
thereof from the EU		8
participants total		28

<u>3. subcontracting services</u>		 Deutsches Referenzbüro für Ringversuche und Referenzmaterialien	
For this proficiency test the following work was subcontracted			
	Sample production	Homogeneity test	Stability test
sample 1		x	x
sample 2		x	x

4. test items

sample 1: ABS (natural); RM CP W K GK 3

- Manufacturing process: Industrial manufacturing process
- Dimension: Plastic pellets
- Material: Non-reinforced ABS for general purpose, e.g. household appliances

sample 2: PP (black); RM CP W K GK 4

- Manufacturing process: Industrial manufacturing process
- Dimension: Plastic pellets
- Material: Talcum-reinforced PP for car industry, e.g. automotive interior parts.

5. requested test parameters

- 1) Total carbon emission

6. homogeneity and stability of the test items



homogeneity test according the post-hoc-system

The homogeneity testing in this proficiency testing was carried out according to the post-hoc homogeneity testing procedure. Therefore a two-way ANOVA with replicates was calculated for the following key parameters in this proficiency testing:

Sample 1: This procedure could not show, that the used material is not suitable.

Sample 2: Due to this statistical procedure the homogeneity for the samples was ensured. The used materials in this proficiency testing scheme meet the criteria for sufficient homogeneity and stability.

For additional information (e.g. calculation equations and evaluation criteria) we recommend our statistical protocol.

7. demonstration of the results



All individual results of the laboratories are shown in tables and diagrams and can include the following elements:

- Measurement results and average of the participants for each parameter
- Applied testing methods of the participants
- Performance evaluation of the participants by using the z'-score
- Precision data
- Graphic illustration (with the individual results in ascending order)
- If applicable, graphic illustration of further test methods
- Youden-Plot
- Graphic illustration of the z'-score of all participants

Interpretation possibilities of the graphics and additional information on the graphic illustrations can be found in our statistical protocol.

8. recommendations



In addition to the long-term monitoring of the own quality by means of reference material, control charts and proficiency testing, we have included a number of general recommendations in our statistical protocol.

9. determination of assigned value and SDPA



General information:

In principle the proficiency testings are evaluated by different statistical methods. This statistical methods can be adapted exactly to the available data or the available data set. The statistics used for the respective parameter to calculate the assigned value (x_{pt}) and the standard deviation for proficiency assessment (SDPA) can be found in the following table:

remark for the SDPA:

Usually the SDPA is calculated as the "Consensus value s_{best} " from the data of the participants. If this calculated value falls below or exceeds the defined minimum SDPA ($SDPA_{min}$) or maximum SDPA ($SDPA_{max}$) for the respective parameter, s_{best} is replaced by the defined $SDPA_{min}$ or $SDPA_{max}$.

Statistical background information (e.g. selection criteria for the different statistical methods, treatment of outliers, specialities of certain types of distributions and measurement of laboratory performance can be found in our statistical protocol.

sample	Parameter	Selection of SDPA	Applied statistic
sample 1: ABS (natural)	Total carbon emission	Consensus value s_{best}	Robust statistic (Hampel estimator, Q-method)
sample 2: PP (black)	Total carbon emission	Consensus value s_{best}	Conventional statistics (all values)

10. remarks and comments

This PT can be considered to be successful.

The relative SDPA of sample 1 is on a similar level as the mean relative standard deviation of the past years. The relative SDPA of sample 2 is below the mean relative standard deviation of the past years.

Comment on available precision data in current standards:

No precision data are available in the current edition of VDA 277 and PV 3341.

The following precision data could be calculated from our proficiency tests of the years 2017-2020, which we provide (informatively) below:

Total carbon emission:

Mean relative repeatability standard deviation (sr): 1.9 %

Mean relative reproducibility standard deviation (sR): 20.0 %

The participants were asked to specify the headspace sampling. The following systems were used: 5 x gas-tight syringe, 5 x balanced pressure system and 9 x pressure loop system, all other laboratories did not specify their headspace sampling.

No clear analysis of trends in measured values as a function of sampling can be made from the data. An assumption that the use of the gas-tight syringe leads to lower measured values can be made, but must first be confirmed in further proficiency tests.

For calculating the assigned value (x_{pt}) and the standard deviation (SDPA) the values of laboratory no. 2, 4, 16, 25 and 26 are not considered. The reason for this is that the laboratories have indicated an examination period outside the given period.

For calculating the assigned value (x_{pt}) and the standard deviation (SDPA) the values of laboratory no. 20,2 were not used at the customer's request.

10. remarks and comments



In some cases the participants don't report their applied test method/standard. For these laboratories it is assumed that they used the reference method, due to this Proficiency Test is performed in accordance with an international standard (VDA/PV).

results**Total carbon emission sample 1: ABS (natural)**assigned value x_{pt} [$\mu\text{g C/g}$] - VDA 277 - PV 3341

62,1

 \pm expanded uncertainty ($k=t$) of the assigned value $U(x_{pt})$

5,2



customer data							performance			
lab code no.	x single value 1	x single value 2	x single value 3	x single value 4	\bar{x} lab mean	standard / (test apparatus)	com-ments	z-score	z'-score	CRD-value
	[$\mu\text{g C/g}$]									
1	45,1	44,6	43,0	44,0	44,2	PV3341 - 1995		-1,34	-1,65	-0,67
2	64,1	63,9	64,4	64,7	64,3	VDA277-1995 , PV3341-1995		0,17	0,20	0,08
3	49,0	49,2	48,9	48,8	49,0	VDA 277- GC7890-Headspace		-0,98	-1,21	-0,49
4	58,8	58,4	59,0	58,8	58,8	VDA 277 / Agilent 7697A & GC MS		-0,25	-0,31	-0,12
5	48,6	48,7	48,9	48,6	48,7	VDA 277:1995-01 HS 110 Perkin Elmer mit Trace 1310 FID Thermo Fisher		-1,00	-1,23	-0,50
6	97,2	95,7	94,1	95,5	95,6	Agilent 7890B with PAL RSI 85 agitator		2,51	3,10	1,26
7	49,1	49,4	47,1	47,0	48,2	VDA 277: 01/1995, Shimadzu Nexis GC 2030, Shimadzu HS-20		-1,04	-1,29	-0,52
8	62,1	63,5	63,4	63,5	63,1	VDA 277		0,08	0,10	0,04
9	61,6	61,4	62,2	62,4	61,9	VDA 277		-0,01	-0,01	-0,01
10	63,7	64,1	65,0	63,7	64,1	VDA 277 / 1995-01 / GC-Agilent 7879B		0,15	0,19	0,08
11	59,0	59,0	59,0	59,0	59,0	VDA 277/ 1995/ HS7697A/ GC7890B		-0,23	-0,28	-0,11
12	69,8	69,0	68,7	69,7	69,3	VDA277-PV3341/1995/HS-GC-FID		0,54	0,67	0,27
13	73,0	73,4	72,4	74,3	73,3	VDA277-1995 HS-GC-FID		0,84	1,04	0,42
14	69,2	70,3	71,5	70,6	70,4	VDA277 : 1995/HS-GC-FID		0,62	0,77	0,31
15	n/a									
16	53,3	55,3	63,1	63,1	58,7	VDA277/ /Agilent		-0,25	-0,31	-0,13
17	62,7	61,5	62,3	62,6	62,3	VDA 277		0,02	0,02	0,01
18	36,1	36,1	36,5	36,3	36,3	VDA277/Perkin Elmer Clarus 580/ Perkin Elmer TurboMatrix40		-1,93	-2,38	-0,97
19	61,6	60,1	59,5	60,9	60,5	PV 3341		-0,11	-0,14	-0,06
20,1	70,2	70,2	72,1	72,1	71,2	VDA 277 (1995) & PV 3341 (1995) Agilent HSS 7697A/ GC 7820A equipment.		0,68	0,84	0,34
20,2	70,6	70,9	70,9	70,2	70,7	VDA 277 (1995) & PV 3341 (1995) Agilent HSS 7697A/ GC 7820A equipment.		0,64	0,79	0,32
21	69,9	69,6	68,0	69,0	69,1	PV3341		0,53	0,65	0,26

results**Total carbon emission sample 1: ABS (natural)**assigned value x_{pt} [$\mu\text{g C/g}$] - VDA 277 - PV 3341

62,1

 \pm expanded uncertainty ($k=t$) of the assigned value $U(x_{pt})$

5,2



customer data								performance		
lab code no.	x single value 1	x single value 2	x single value 3	x single value 4	\bar{x} lab mean	standard / (test apparatus)	com-ments	z-score	z'-score	CRD-value
	[$\mu\text{g C/g}$]									
22	73,6	74,9	75,1	72,2	74,0	VDA277/ none /HS-GC-FID		0,89	1,10	0,44
23	66,3	66,4	66,4	66,4	66,4	VDA277-PV3341/Agilent 7697A +7890B(HS-GCFID)		0,32	0,40	0,16
24	n/a									
25	65,1	64,2	64,6	65,0	64,7	Volkswagen AG PV 3341/Agilent 7890A (GC System) - Agilent 7697A (Headspace Standard) - Agilent J&W DB-WAX GC column (30-0.25-0.25)		0,20	0,25	0,10
26	68,6	69,7	69,8	70,3	69,6	VDA 277 - PV 3341 / 1st Revision 03.95/ HeadSpace-GC FID		0,56	0,70	0,28
27	n/a									
28	n/a									

summarized evaluation
Total carbon emission sample 1: ABS (natural)



assigned value x_{pt} [$\mu\text{g C/g}$] - VDA 277 - PV 3341	62,1
\pm expanded uncertainty ($k=t$) of the assigned value $U(x_{pt})$	5,2
standard deviation for proficiency assessment (SDPA)	10,8
no. of datasets excluding outlier	19
no. of outlier	0

performance rating of laboratory datasets:

As an additional service, the calculation of precision data according to ISO 5725-2 was carried out using the available proficiency testing results from the specified reference method(s). The calculated precision data are only valid for this evaluation and can be used in addition to the precision data of the literature, if available, or as orientation in case of missing or inapplicable precision data.

z-score

$z \leq 2$	$2 < z < 3$	$z \geq 3$
24	1	0

$$z = \frac{\bar{x} - x_{pt}}{s_R}$$

repeatability	
r	2,2
$r \text{ rel } (\%)$	3,61

reproducibility	
R	37,8
$R \text{ rel } (\%)$	60,98

z'-score

$z' \leq 2$	$2 < z' < 3$	$z' \geq 3$
23	1	1

$$z' = \frac{\bar{x} - x_{pt}}{\sqrt{SDPA^2 + s_M^2}}$$

repeatability standard deviation	
s_r	0,8

reproducibility standard deviation	
s_R	13,4

CRD-value

$CRD < 1$	$CRD > 1$
24	1

$$CRD = \frac{\bar{x} - x_{pt}}{CD}$$

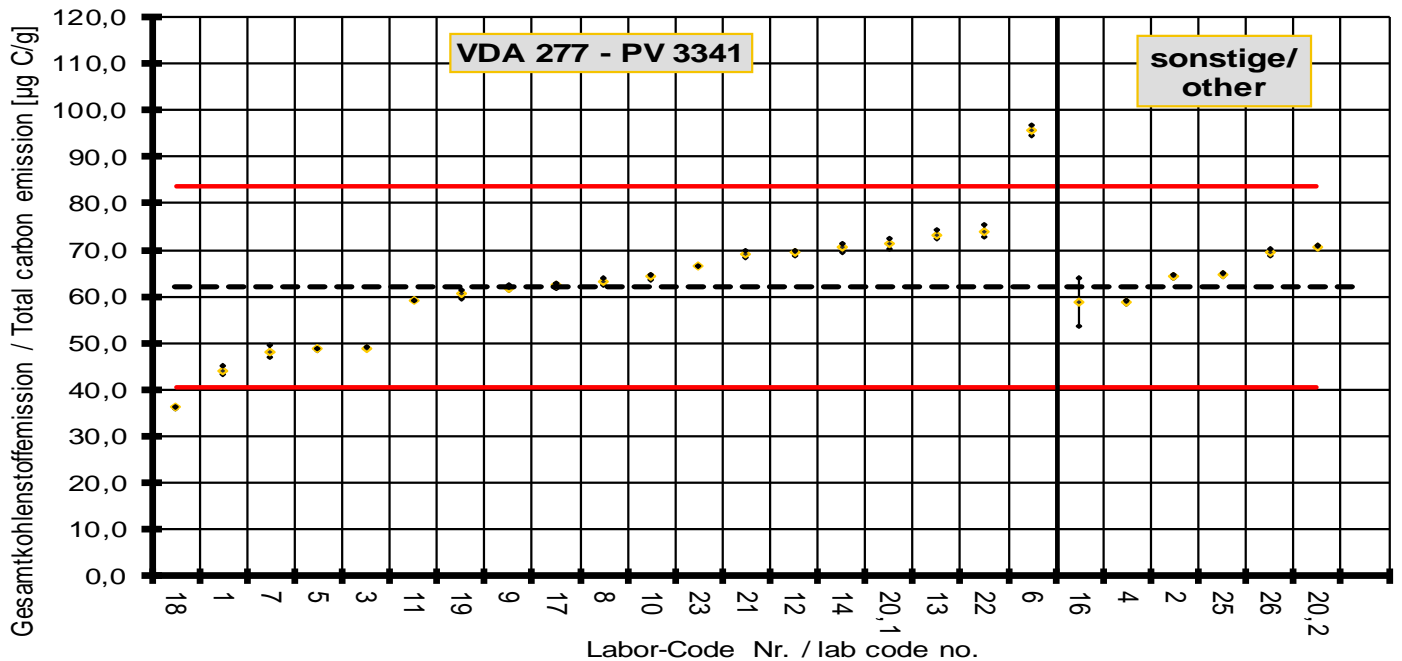
critical difference	
CD	26,7

material standard deviation from the homogeneity test	
s_M	0,6

Further information you will find in our statistical Protocol.
 As a registered customer you can download the statistical protocol on your personal home page in our Online-Data-Information-Network (ODIN).
 Alternatively, we also provide you with the statistical protocol on request.

\bar{x} = your lab mean value
 x_{pt} assigned value
 $U(x_{pt})$ expanded uncertainty ($k=t_{0.975, n-1}$) of the assigned value

results sorted according to methods
Total carbon emission sample 1: ABS (natural)
(error indicator = laboratory standard deviation)



- Labormittelwert \pm Laborstandardabweichung / lab mean value \pm lab standard deviation
- Labormittelwert / lab mean value
- Zugwiesener Wert / assigned value (VDA 277 - PV 3341) \pm U(xpt): 62,1 \pm 5,2
- z'-score \pm 2

results**Total carbon emission sample 2: PP (black)**assigned value x_{pt} [$\mu\text{g C/g}$] - VDA 277 - PV 3341

35,9

 \pm expanded uncertainty ($k=t$) of the assigned value $U(x_{pt})$

2,5



customer data								performance		
lab code no.	x single value 1	x single value 2	x single value 3	x single value 4	\bar{x} lab mean	standard / (test apparatus)	com-ments	z-score	z'-score	CRD-value
	[$\mu\text{g C/g}$]									
1	44,0	44,0	43,5	43,8	43,8	PV3341 - 1995		1,61	1,55	0,81
2	38,5	38,3	38,3	38,7	38,5	VDA277-1995 ,PV3341-1995		0,52	0,50	0,26
3	33,5	32,8	33,7	33,0	33,3	VDA 277- GC7890-Headspace		-0,53	-0,51	-0,27
4	29,1	28,8	28,3	28,4	28,7	VDA 277 / Agilent 7697A & GC MS		-1,46	-1,40	-0,73
5	25,9	26,7	26,6	26,2	26,4	VDA 277:1995-01 HS 110 Perkin Elmer mit Trace 1310 FID Thermo Fisher		-1,93	-1,85	-0,97
6	42,5	43,4	41,9	45,0	43,2	Agilent 7890B with PAL RSI 85 agitators		1,48	1,42	0,74
7	34,4	35,2	34,6	33,5	34,4	VDA 277: 01/1995, Shimadzu Nexis GC 2030, Shimadzu HS-20		-0,29	-0,28	-0,15
8	38,0	37,3	37,4	37,6	37,6	VDA 277		0,34	0,33	0,17
9	38,7	39,8	39,2	38,6	39,1	VDA 277		0,65	0,62	0,32
10	37,2	37,9	37,4	37,4	37,5	VDA 277 / 1995-01 / GC-Agilent 7879B		0,32	0,31	0,16
11	33,0	33,0	32,5	32,0	32,6	VDA 277/ 1995/ HS7697A/ GC7890B		-0,66	-0,63	-0,33
12	35,5	34,8	34,2	34,1	34,7	vda277-pv3341/1995/HS-GC-FID		-0,25	-0,24	-0,12
13	38,8	38,0	38,0	38,6	38,4	VDA277-1995 HS-GC-FID		0,50	0,48	0,25
14	34,8	34,7	35,2	35,3	35,0	VDA277 : 1995/HS-GC-FID		-0,18	-0,17	-0,09
15	n/a									
16	29,2	30,2	30,2	29,3	29,7	VDA277/ /Agilent		-1,25	-1,20	-0,62
17	33,9	34,1	34,0	34,3	34,1	VDA 277		-0,36	-0,35	-0,18
18	24,0	24,1	24,2	24,0	24,1	VDA277/Perkin Elmer Clarus 580/ Perkin Elmer TurboMatrix40		-2,39	-2,29	-1,20
19	37,2	35,7	36,5	36,1	36,4	PV 3341		0,10	0,10	0,05
20,1	32,3	31,6	31,1	30,1	31,3	VDA 277 (1995) & PV 3341 (1995) Agilent HSS 7697A/ GC 7820A equipment.		-0,93	-0,89	-0,47
20,2	31,5	31,6	31,2	30,5	31,2	VDA 277 (1995) & PV 3341 (1995) Agilent HSS 7697A/ GC 7820A equipment.		-0,95	-0,91	-0,47
21	40,2	39,7	39,6	38,5	39,5	PV3341		0,73	0,71	0,37

results**Total carbon emission sample 2: PP (black)**assigned value x_{pt} [$\mu\text{g C/g}$] - VDA 277 - PV 3341

35,9

 \pm expanded uncertainty ($k=t$) of the assigned value $U(x_{pt})$

2,5



customer data							performance			
lab code no.	x single value 1	x single value 2	x single value 3	x single value 4	\bar{x} lab mean	standard / (test apparatus)	com-ments	z-score	z'-score	CRD-value
	[$\mu\text{g C/g}$]									
22	41,4	40,9	41,1	41,1	41,1	VDA277/ none /HS-GC-FID		1,06	1,02	0,53
23	39,4	39,4	39,3	39,4	39,4			0,71	0,68	0,36
24	n/a									
25	38,0	37,8	37,3	37,4	37,6	Volkswagen AG PV 3341/Agilent 7890A (GC System) - Agilent 7697A (Headspace Standard) - Agilent J&W DB-WAX GC column (30-0.25-0.25)		0,35	0,34	0,18
26	38,7	38,4	38,8	38,6	38,6	VDA 277 - PV 3341 / 1st Revision 03.95/ HeadSpace-GC FID		0,56	0,54	0,28
27	n/a									
28	n/a									

summarized evaluation

Total carbon emission sample 2: PP (black)

assigned value x_{pt} [$\mu\text{g C/g}$] - VDA 277 - PV 3341	35,9
\pm expanded uncertainty ($k=t$) of the assigned value $U(x_{pt})$	2,5
standard deviation for proficiency assessment (SDPA)	5,1
no. of datasets excluding outlier	19
no. of outlier	0



performance rating of laboratory datasets:

As an additional service, the calculation of precision data according to ISO 5725-2 was carried out using the available proficiency testing results from the specified reference method(s). The calculated precision data are only valid for this evaluation and can be used in addition to the precision data of the literature, if available, or as orientation in case of missing or inapplicable precision data.

z-score

$z \leq 2$	$2 < z < 3$	$z \geq 3$
24	1	0

$$z = \frac{\bar{x} - x_{pt}}{s_R}$$

repeatability	
r	1,4
r rel (%)	3,77

reproducibility	
R	14,0
R rel (%)	38,95

z'-score

$z' \leq 2$	$2 < z' < 3$	$z' \geq 3$
24	1	0

$$z' = \frac{\bar{x} - x_{pt}}{\sqrt{SDPA^2 + s_M^2}}$$

repeatability standard deviation	
s_r	0,5

reproducibility standard deviation	
s_R	4,9

CRD-value

$CRD < 1$	$CRD > 1$
24	1

$$CRD = \frac{\bar{x} - x_{pt}}{CD}$$

critical difference	
CD	9,9

material standard deviation from the homogeneity test	
s_M	0,7

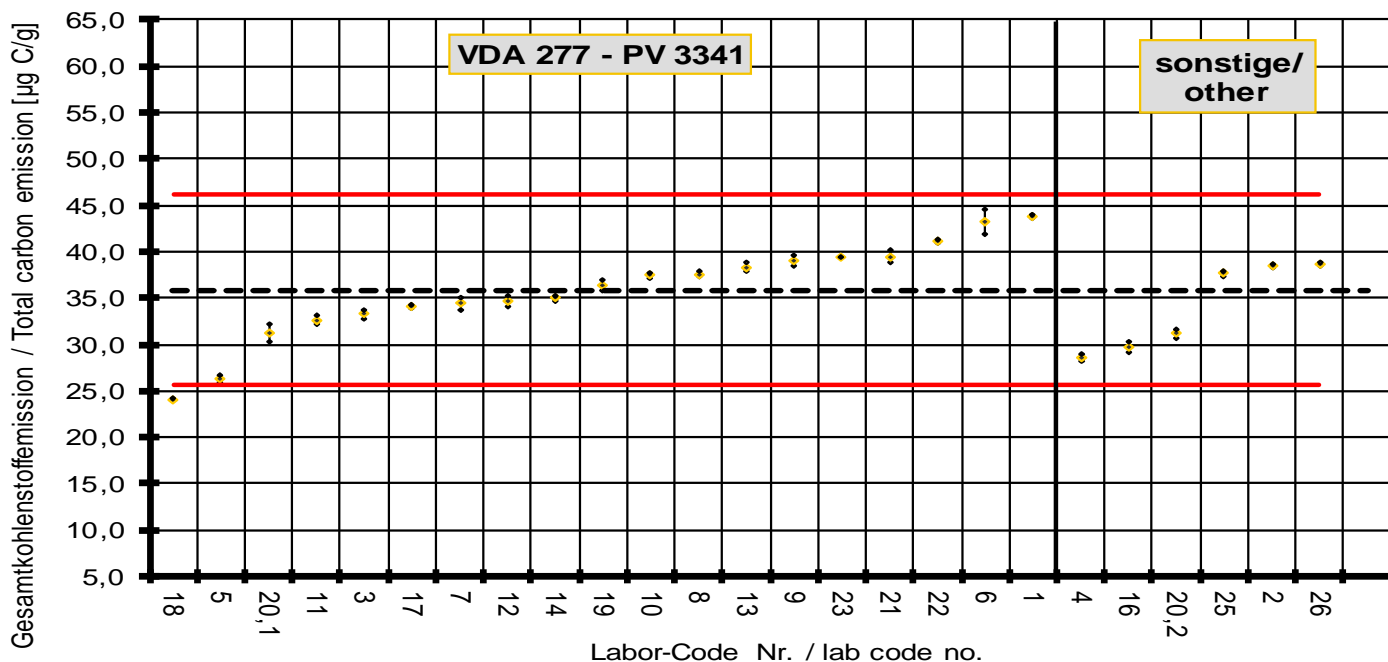
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\bar{x} = your lab mean value
 x_{pt} assigned value
 $U(x_{pt})$ expanded uncertainty ($k=t_{0.975, n-1}$) of the assigned value

results sorted according to methods

Total carbon emission sample 2: PP (black)

(error indicator = laboratory standard deviation)



- Labormittelwert ± Laborstandardabweichung / lab mean value ± lab standard deviation
- Labormittelwert / lab mean value
- Zugewiesener Wert / assigned value (VDA 277 - PV 3341) ± U(xpt): 35,9 ± 2,5
- z'-score ± 2

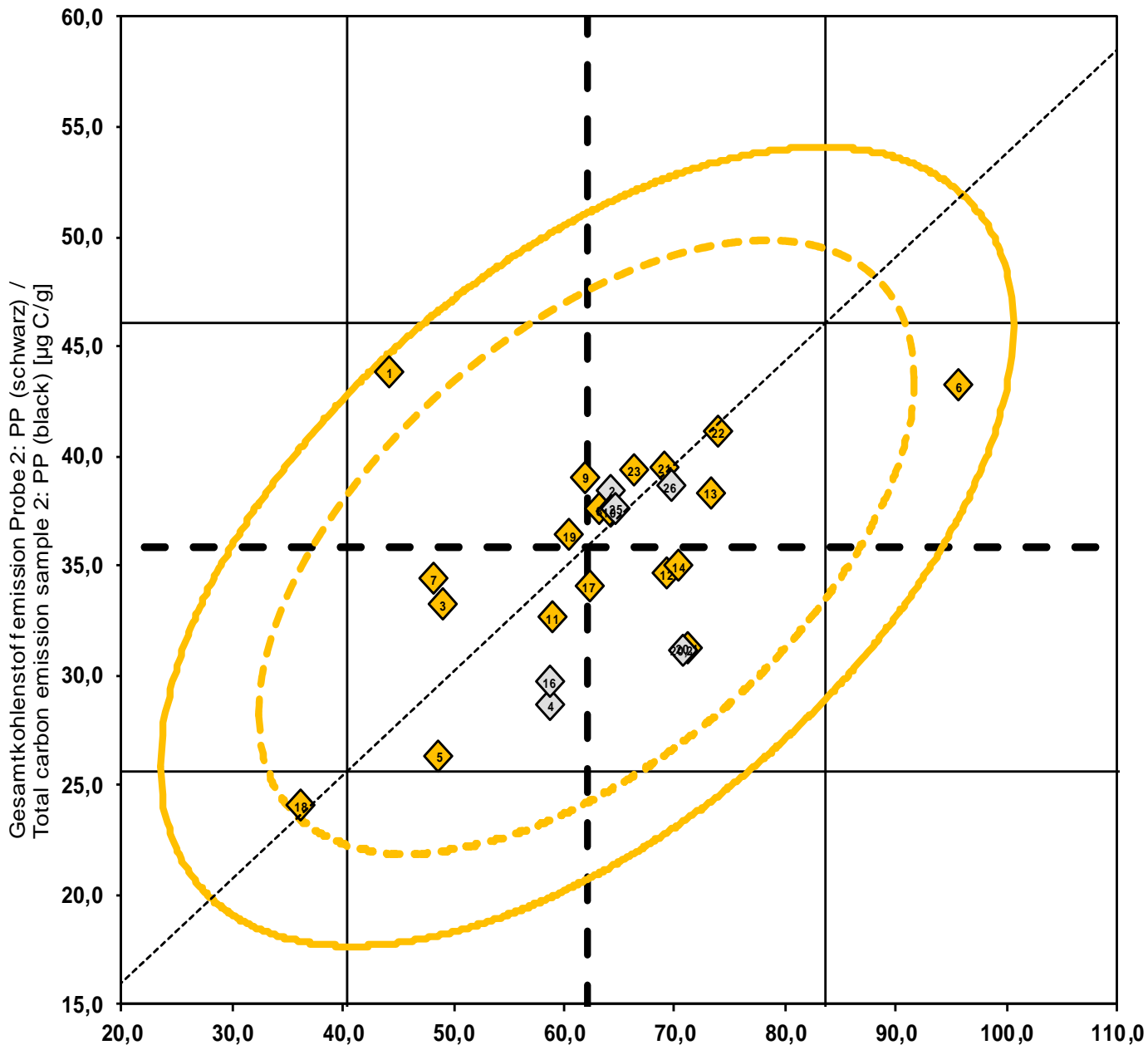
Youdenplot



zufällige Abweichung / random deviation

VDA 277 | PV 3341

systematisch zu hoch / systematically to high



systematisch zu niedrig / systematically to low

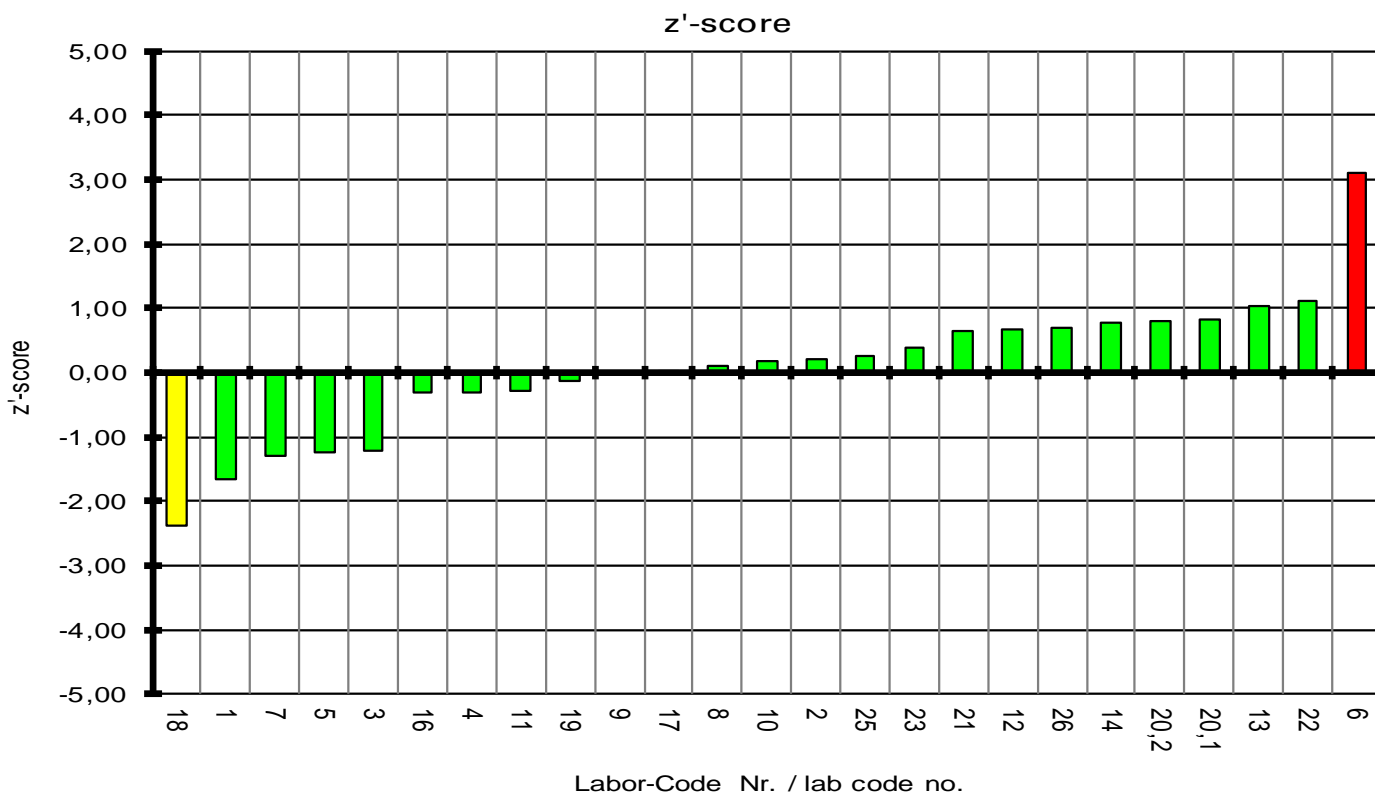
Gesamtkohlenstoffemission Probe 1: ABS (natur) / Total carbon emission sample 1: ABS (natural) [µg C/g]

zufällige Abweichung / random deviation



summary z'-score

Total carbon emission sample 1: ABS (natural)



summary z'-score

Total carbon emission sample 2: PP (black)

