DLA Dienstleistung Lebensmittel Analytik GbR

Evaluation Report proficiency test

DLA 43/2016

16-O-Methylcefestol in 3 Coffee Blends

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1. Introduction

The participation in proficiency testing schemes is an essential element of the quality-management-system of every laboratory testing food and feed, cosmetics and food contact materials. The implementation of proficiency tests enables the participating laboratories to prove their own analytical competence under realistic conditions. At the same time they receive valuable data regarding the validity of the particular testing method.

The purpose of DLA is to offer proficiency tests for selected parameters in concentrations with practical relevance.

Realisation and evaluation of the present proficiency test follows the technical requirements of DIN EN ISO/IEC 17043 (2010) and DIN ISO 13528:2009.

2. Realisation

2.1 Test material

The test material are homogeneous blends with different rations of ground Arabica and Robusta beans:

Blend A (7,5% Robusta):

Ingredient	percentage
Roasted coffee 100% Robusta	7,5 %
Roasted coffee 100% Arabica	91,9 %
NaCl	0,6 %

Blend B (15% + unknown proportion of Robusta):

Ingredient	percentage
Roasted coffee 100% Robusta	15,0 %
Roasted coffee from the market "classic selection",	84,2 %
no details of the type of coffee	
NaCl	0,8 %

Blend C (20 % Robusta)*:

Ingredient	percentage
Roasted coffee from the market	100 %
"Espresso", Ingredients: 20% Robusta/ 80% Arabica	
* on the package	

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Samples A and B were admixed with 0,6 % and 0,8 % table salt (NaCl) to determine the homogeneity.

Approximately 1 kg of the material was homogenized and then packaged lightproof in portions to approximately 20 g. The portions were numbered chronologically. The material was checked for homogeneity.

2.1.1 Homogeneity

To verify the homogeneity of the test material table salt (NaCl) was added before homogenisation. The homogeneity was examined in sample A and B by ICP-OES according to VDLUFA iii, 10.8.2. The homogeneity is considered verified with a standard deviation of 4,8 % resp., see in the documentation.

Additionally in the documentation the portion numbers are graphically assigned to the results of 16-O-Methylcafestol for the blends A and B. There is no trend recognizable in the results which could suggest inhomogeneity.

2.2 Test

One portions of test samples A, B and C were sent to every participating laboratory in the 4th week of 2016. The testing method was optional. The tests should be finished at 11^{th} march 2016 the latest.

2.3 Results

The participants submitted their results in standard forms, which have been handed out with the samples (by email). For statistical evaluation the final results for the numbered samples were used.

Queried and documented were single results for 16-O-Methylcafestol, Methylcafestol and Kahweol, recovery and the used testing method.

In case participants submitted several results for the same parameter obtained by different methods these results were evaluated with the same evaluation number with a letter as a suffix and indication of the related method.

Of the 10 participants, all participants have submitted their results in time. One participant sent us 2 results for each blend obtained with different methods.

3. Evaluation

3.1 Consensus values from participants (Assigned value)

Because the analysed material was no certified reference material the robust mean of the submitted results was used as assigned value X (6).

The distribution of submitted results showed no hint for bimodal distribution or other reasons for a higher variability (s. 4.1 - 4.3: Results Kernel density plots).

The statistical evaluation is carried out for all the parameters for which a minimum of 7 values were submitted.

The actual results were taken. Single results giving values outside the measuring range of the participating laboratory or given as "0" are not considered for statistical evaluation (e.g. results given as > 25 mg/kg and < 2,5 mg/kg, respectively). If a negative result is observed, the actual negative value must be specified (6).

3.2 Standard deviation

For comparison to the target standard deviation a robust standard deviation (S_x) was calculated (6).

3.3 Exclusion of results and outliers

Before statistical evaluation obvious blunders, such as those with incorrect units, decimal point errors, and results for another proficiency test item can be removed from the data set (1/6).

Results obtained by different analytical methods causing an increased variability and/or a bi- or multimodal distribution of results, are treated separately or could be excluded in case of too few numbers of results. For this results are checked by kernel density estimation (13).

Statistical outliers were determined by Mandel's-H-Statistic (significance level: 5%) (5). Detected outliers were stated for information only, when z-score simultaneously was < -2 or > 2. Due to the use of robust statistics outliers are not excluded, provided that no other reasons are present (6).

3.4 Target standard deviation

The target standard deviation of the consensus value is determined according to the following methods.

3.4.1 General model (Horwitz)

The relative target standard deviation in % of the consensus value is calculated according to the following equation.

$$\hat{\sigma}$$
 (%) = 2^(1-0, 5logX)

Out of this is calculated the target standard deviation in mg/kg

$$\hat{\sigma}$$
 = X * $\hat{\sigma}$ (%) / 100.

3.4.2 Precision experiment

Using the reproducibility standard deviation σ_R and the repeatability standard deviation σ_r of a precision experiment the between-laboratories standard deviation (σ_L) can be calculated:

$$\sigma_L = \sqrt{(\sigma_R^2 - \sigma_r^2)}$$
.

And then, using the number of replicate measurements n, each participant is to perform, the standard deviation for proficiency assessment is calculated:

$$\hat{\sigma} = \sqrt{(\sigma_L^2 + (\sigma_r^2/n))}$$
.

The statistical evaluation was realised with the target standard deviation according to ASU §64 LFGB L 46.02-4 (or DIN 10779/2011) because almost all of the participants have used this method.

The precision data of the ASU §64 LFGB L 46.02-4 (determination with HPLC-analysis) are: The repeatability standard deviation σ_r for the determination of 16-O-Methylcafestol is 4,5% and the reproducibility standard deviation σ_R is 11,6% for roasted coffee blends (portion Robusta = 20%).

The target standard deviation according to ASU § 64 LFGB L 46.02-4 $\,$ (13) was used for the evaluation.

The target standard deviations according to Horwitz are listed for information additionally in this evaluation.

3.4.3 Value by perception

The target standard deviation for proficiency assessment can be set at a value that corresponds to the level of performance that the coordinator would wish laboratories to be able to achieve (6).

3.5 z-Score

To assess the results of the participants the z-score is used. It indicates about which multiple of the target standard deviation ($\hat{\sigma}$) the result (x) of the participant is deviating from the consensus value (X)(6).

Participants' z-scores were derived as:

$$z = (x - X) / \hat{\sigma}$$
;

the requirements for the analytical performance are generally considered as fulfilled if

 $-2 \leq z \leq 2$.

In accordance with the norm DIN ISO 13528:2009 (6) it is recommended that a result that gives rise to a z-score above 3,0 or below -3,0, shall be considered to give an "action signal". Likewise, a z-score above 2,0 or below -2,0 shall be considered to give a "warning signal". A single "action signal", or "warning signals" in two successive PT-rounds, shall be taken as evidence that an anomaly has occurred which requires investigation. For example a fault isolation or a root cause analysis through the examination of transmission error or an error in the calculation, in the trueness and precision must be performed and if necessary appropriate corrective measures should be applied (6).

In the figures of z-scores DLA gives the limits of warning and action signals as yellow and red lines respectively.

According to ISO 13528:2009 the signals are valid only in case of a number of \geq 10 results (6).

3.6 z'-Score

The z'-score can be used for the valuation of the results of the participants, in cases the standard uncertainty has to be considered (s. 3.8). The z'-score represents the relation of the deviation of the result (x) of the participant from the respective consensus value (X) to the square root of quadrat sum of the target standard deviation ($\hat{\sigma}$) and the standard uncertainty (Ux) (6).

Participants' z'-scores are derived as:

$$z' = (x - X)/\sqrt{\hat{\sigma}^2 + u_X^2}$$

In the following we define the denominator $\sqrt{\hat{\sigma}^2 + u_X^2}$ as the target standard deviation $\hat{\sigma}$ '.

The requirements for the analytical performance are generally considered as fulfilled if

$$-2 \leq z' \leq 2$$
.

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3.7 Precision and coefficient of variation (V_{K})

Precision describes the random deviation of values around the mean, given as standard deviation S^{\star} or as coefficient of variation V_{K} (relative standard deviation).

The coefficient of variation $(V_{\mbox{\tiny K}})$ is calculated from the standard deviation S^* and the mean:

$$V_{\rm K} = \frac{S_{\rm R}}{X} \times \frac{100}{X}$$

The $V_{\mbox{\tiny K}}$ is used it to demonstrate the variability. The higher the $V_{\mbox{\tiny K}}$, the greater is the divergence. In contrast to the standard deviation as a measure of the absolute variability, the $V_{\mbox{\tiny K}}$ shows the relative variability within a range of data.

A $V_{\mbox{\tiny K}}$ of more than 50% suggest a "strong inhomogeneity of statistical mass".

3.8 Quotient $S^x/\hat{\sigma}$

Following the Horrat-value the results of a proficiency-test (PT) can be considered convincing, if the quotient of robust standard deviation and target standard deviation does not exceed the value of 2. A value > 2 means an insufficient precision, i.e. the analytical method is too variable, or the variation between the test participants is higher than estimated. Thus the comparability of the results is not given (11).

3.9 Standard uncertainty

The consensus value X has a standard uncertainty $u_{\rm X}$ that depends on the

analytical method, differences between the analytical methods used, the test material, the number of participant laboratories and perhaps on other factors. The standard uncertainty (u_X) for this PT is calculated as follows (6).

$$u_x = 1,25 * S^x / \sqrt{(p)}$$

If $u_x \leq 0.3 * \hat{\sigma}$ the standard uncertainty of the consensus value needs not to be included in the interpretation of the results of the PT (6).

A clear exceeded the value of 0.3 is an indication that the target standard deviation was possibly set too low for the standard uncertainty of the consensuf value.

The quotient $u_{x}/\hat{\sigma}$ is reported in the characteristics of the test.

4. Results

All following tables are anonymized. With the delivering of the evaluation-report the participants are informed about their individual evaluation-number.

From ASU §64 LFGB L 46.02-4 (13) and DIN 10779 (March 2011) resp. with 8 participating laboratories follows the correlation between the 16-0-Methylcafestol concentration and the Robusta rate in Arabica roasted coffee:



In the first table the characteristics are listed:

Statistic Data
Number of results
Number of outliers
Mean
Median
Robust mean (X)
Robust standard deviation (S ^x)
Target range:
Target standard deviation $\hat{\sigma}$
Target standard deviation for information
lower limit of target range (X - 2 $\hat{\sigma}$)
upper limit of target range (X + 2 $\hat{\sigma}$)
Coefficient of variation (V_{K}) in %
Quotient $S^{\times}/\hat{\sigma}$
Standard uncertainty u_x
Quotient $u_X/\hat{\sigma}$
Number of results in the target range
Percent in the target range

In the second table the individual results of the participating laboratories are listed:

Auswerte-	_	Abweichung	z-Score	z-Score	Hinweis
nummer	Parameter		$\hat{\sigma}$	(Info)	
Evaluation number	[Einheit / Unit]	Deviation			Remark

4.1 16-0-Methylcafestol (16-0-MC) in sample A (mg/kg)

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	11
Number of outliers	0
Mean	104
Median	110
Robust Mean (X)	104
Robust standard deviation (S^{x})	21,6
Target range:	
Target standard deviation ASU ($\hat{\sigma}$)	11,4
Target standard deviation Horwitz (for Information)	8,28
lower limit of target range	81,4
upper limit of target range	127
coefficient of variation ($V_{_{ m K}}$) in %	20,7
Quotient S*/ \hat{g}	1,9
Standard uncertainty u _x	8,1
Quotient $u_x / \hat{\sigma}$	0,72
Results in the target range	8
Percent in the target range	73

Notes to the statistic data:

The target standard deviation was calculated according to precision data from ASU $\$ LFGB L 46.02-4.

The evaluation of the results shows an acceptable variability of results. The quotient $S^x/\hat{\sigma}$ was below 2,0. The quotient $u_x/\hat{\sigma}$ of 0,72 is above 0,3.

The robust standard deviation shows an increased variability of the results and is relatively high compared to the reproducibility standard deviations of the ASU § 64 LMBG L 46.02-4.

For sample A, there was a roast coffee blend with Robusta content of 7.5%.

From the robust mean and the values specified in the ASU § 64 LFGB L 46.02-4 a proportion of Robusta coffee of 8,0% can be calculated.



Abb. 1: Ergebnisse 16-0-MC, Probe A Fig. 1: Results 16-0-MC, sample A

Ergebnisse der teilnehmenden Institute: Results of Participants:

Auswerte- nummer	16-OMC A (mg/kg)	Abweichung [mg/kg]	$egin{array}{c} \mathbf{z} extsf{-Score} \ \hat{\sigma} \end{array}$	z-Score (Info)	Hinweis
Evaluation number	-	Deviation [mg/kg]			Remark
1	129	24,9	2,2	3,0	
2	116	11,8	1,0	1,4	
3	115	10,9	1,0	1,3	
4a	126	21,9	1,9	2,6	
4b	118	13,9	1,2	1,7	
5	79	-25,1	-2,2	-3,0	
6	98	-6,15	-0,5	-0,7	
7	110	5 , 85	0,5	0,7	
8	94	-10,1	-0,9	-1,2	
9	89	-15,1	-1,3	-1,8	
10	70	-34,1	-3,0	-4,1	



Abb. 2: Z-Scores 16-OMC Probe A Fig. 2: Z-Scores 16-OMC sample A



Abb. 3: Kern Dichte Plot aller Probe A-Ergebnisse (16-OMC) mit h = ASU Zielstandardabweichung (11,38 mg/kg) Fig. 3: Kernel density plot of all sample A (16-OMC) results with h = ASU target standard deviation (11,38 mg/kg)

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4.2 16-O-Methylcafestol in sample B (mg/kg)

Statistic Data		
Number of results	11	
Number of outliers	1	
Mean	198	
Median	200	
Robust Mean (X)	200	
Robust standard deviation (S^{x})	35,6	
Target range:		
Target standard deviation ASU ($\hat{\sigma}$)	21,8	
Target standard deviation Horwitz (for Information)	14,4	
lower limit of target range	156	
upper limit of target range	243	
coefficient of variation $(V_{_{\rm K}})$ in %	17,8	
Quotient $S^{x}/\hat{\mathbf{G}}$	1,6	
Standard uncertainty u _x	13,4	
Quotient $u_x / \hat{\sigma}$	0,61	
Results in the target range	9	
Percent in the target range	82	

Notes to the statistic data:

The target standard deviation was calculated according to precision data from ASU § LFGB L 46.02-4.

The evaluation of the results shows an acceptable variability of results. The quotient $S^x/\hat{\sigma}$ was below 2,0. The quotient $u_x/\hat{\sigma}$ of 0,61 is above 0,3.

The robust standard deviation shows an increased variability of the results and is relatively high compared to the reproducibility standard deviations of the ASU § 64 LMBG L 46.02-4.

For sample A, there was a roast coffee blend with Robusta content of 15%.

From the robust mean and the values specified in the ASU § 64 LFGB L 46.02-4 a proportion of Robusta coffee of 15,5% can be calculated.



Abb. 4: Ergebnisse 16-OMC Probe B Fig. 4: Results 16-OMC sample B

Ergebnisse der teilnehmenden Institute: Results of Participants:

Auswerte- nummer	16-OMC B (mg/kg)	Abweichung [mg/kg]	z-Score ô	z-Score (Info)	Hinweis
Evaluation number		Deviation [mg/kg]	1		Remark
1	196	-3,80	-0,2	-0,3	
2	241	41,4	1,9	2,9	
3	221	21,2	1,0	1,5	
4a	273	73,2	3,4	5,1	
4b	201	1,20	0,1	0,1	
5	109	-90,8	-4,2	-6,3	Ausreisser / Outlier
6	193	-6,80	-0,3	-0,5	
7	204	4,20	0,2	0,3	
8	200	0,20	0,0	0,0	
9	182	-17,8	-0,8	-1,2	
10	160	-39,8	-1,8	-2,8	



Abb. 5: Z-Scores 16-OMC Probe B Fig. 5: Z-Scores 16-OMC sample B



Abb. 6: Kern Dichte Plot aller Probe B-Ergebnisse (16-OMC) mit h = ASU Zielstandardabweichung (21,8 mg/kg) Fig. 6: Kernel density plot of all sample B (16-OMC) results with h = ASU target standard deviation (21,8 mg/kg)

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4.3 16-O-Methylcafestol in sample C (mg/kg)

Statistic Data	
Number of results	11
Number of outliers	1
Mean	242
Median	250
Robust Mean (X)	247
Robust standard deviation (S $^{\times}$)	54,9
Target range:	
Target standard deviation ASU ($\hat{\sigma}$)	27,0
Target standard deviation Horwitz (for Information)	17,3
lower limit of target range	193
upper limit of target range	301
coefficient of variation ($V_{_{\rm K}}$) in %	22,2
Quotient S ^x / $\hat{\sigma}$	2,0
Standard uncertainty u_x	20,7
Quotient $u_x / \hat{\sigma}$	0,77
Results in the target range	9
Percent in the target range	82

Notes to the statistic data:

The target standard deviation was calculated according to precision data from ASU § LFGB L 46.02-4.

The evaluation of the results shows an acceptable variability of results. The quotient S^x/ $\hat{\sigma}$ was below 2,0. The quotient u_x/ $\hat{\sigma}$ of 0,77 is above 0,3.

The robust standard deviation shows an increased variability of the results and is relatively high compared to the reproducibility standard deviations of the ASU § 64 LMBG L 46.02-4.

For sample A, there was a roast coffee blend with Robusta content of 20%.

From the robust mean and the values specified in the ASU § 64 LFGB L 46.02-4 a proportion of Robusta coffee of 19,2% can be calculated.



Fig. 7: Results 16-OMC sample c

Ergebnisse der teilnehmenden Institute: Results of Participants:

Auswerte- nummer	16-OMC C (mg/kg)	Abweichung [mg/kg]	z-Score <i>ô</i>	z-Score (Info)	Hinweis
Evaluation number		Deviation [mg/kg]			Remark
1	243	-4,2	-0,2	-0,2	
2	298	51,1	1,9	3,0	
3	267	19,8	0,7	1,1	
4a	299	51,8	1,9	3,0	
4b	300	52 , 8	2,0	3,1	
5	153	-94,2	-3,5	-5,5	
6	250	2,82	0,1	0,2	
7	272	24,82	0,9	1,4	
8	221	-26,2	-1,0	-1,5	
9	239	-8,2	-0,3	-0,5	
10	120	-127,2	-4,7	-7,4	Ausreisser / Outlier



Abb. 8: Z-Scores 16-OMC Probe C Fig. 8: Z-Scores 16-OMC sample C



Abb. 9: Kern Dichte Plot aller Probe C-Ergebnisse (16-OMC) mit h = ASU Zielstandardabweichung (27,0 mg/kg) Fig. 9: Kernel density plot of all sample C (16-OMC) results with h = ASU target standard deviation (27,0 mg/kg)

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5. Documentation

5.1 Primary data

5.1.1 16-0-Methylcafestol

Teilnehmer/	Ergebnis A/	Ergebnis B/	Ergebnis C/	Wiederfin-
participants	result A	result B	result C	dungsrate/
				recovery
	mg/kg	mg/kg	mg/kg	in %
1	129	196	243	100
2	115,9	241,2	298,3	75 , 3
3	115	221	267	100,6
4a	126	273	299	-
4b	118	201	300	-
5	79	109	153	77 , 5
6	98	193	250	103
7	110	204	272	95
8	94	200	221	88,1
9	89	182	239	
10	70	160	120	100

5.1.2 Kahweol (1.2-Dehydrocafestol)

Teilnehmer/ participants	Ergebnis A/ results A	Ergebnis B/ results B	Ergebnis C/ results C	Wiederfin- dungsrate/ recovery
	mg/kg	mg/kg	mg/kg	in %
9	5322	4870	3458	
10	4740	4430	1880	100

5.1.3 Cafestol

Teilnehmer/ paticipants	Ergebnis A/ result A	Ergebnis B/ result B	Ergebnis C/ result C	Wiederfin- dungsrate/ recovery
	mg/kg	mg/kg	mg/kg	in %
9	5212	4993	3092	
10	4850	4620	1570	100

5.2 Homogeneity

5.2.1 Homogeneity testing before PT

To verify the homogeneity of the test material the content of sodium (Na) was determined in a 5-fold determination in sample A and B by ICP-OES (according to VDLUFA iii, 10.8.2).

Sample A	Sodium		
1	219	g/100g	
13	246	g/100g	
22	235	g/100g	
35	230	g/100g	
43	245	g/100g	
Mean	235	g/100g	
Standard deviation	11,2	4,8	olo

Sample B	Sodium		
6	305	g/100g]
17	343	g/100g	
24	333	g/100g	
32	343	g/100g	
41	326	g/100g	
Mean	330,0	g/100g	
Standard			
deviation	15 , 72	4,8	00

5.2.3 Comparison of sample number/test result

The comparison of the increasing sample-numbers and measured 16-OMC-results shows a sufficient homogeneity.



5.3 Analytical methods

Details by the participants:

5.3.1 16-0-Methylcafestol, Kahweol a. Cafestol

Teilnehmer/ paticipant	Methode/ method	Wiederfindung mit gleicher Matrix/ recovery with the same matrix	Akkreditiert/ accrededed	Sonstige Hinweise/ remarks
		ja / nein	ja / nein	
1	ASU L46.02-4		yes	After ASU no recovery correction is applied
2	\$64-Method L64.024	yes	yes	
3	DIN 10779	yes	yes	
4a	16-O-Methylcafestol, LC-MS/MS, DIN 10779 mod., roasted coffee	_	yes	
4b	16-O-Methylcafestol, NMR, roasted coffee	_	no	
5	NMR	yes	no	
6	16-OMC in coffee with HPLC-DAD	yes	yes	
7	\$64 46.02-4 HPLC method	yes	yes	
8	§ 64 LFGB Nr. L46.02-4 (mod.)	yes	yes	Sample C was coarser than Sample A and B
9	Determination of 16-OMC, Kahweol a. Cafestol in roasted coffee with 1H-NMR		16-OMC:yes Kahweol: no Cafestol: no	
10	NMR (16-OMC, Kahweol a. Cafestol)	yes	yes	Determination Robusta Anteil: 7%/ 12%/ 19%

6. Index of participant laboratories

<u>Teilnehmer/ participant</u>	<u>Ort/ town</u>	Land/ country
		Germany
		Germany
		Germany
		France
		Germany
		Switzerland
		Germany
		Germany
		Germany
		Germany

[Die Adressdaten der Teilnehmer wurden für die allgemeine Veröffentlichung des Auswerte-Berichts nicht angegeben.]

 $[\ensuremath{\textit{The}}\xspace$ address data of the participants were deleted for publication of the evaluation report.]

7. Index of literature

- DIN EN ISO/IEC 17043:2010; Konformitätsbewertung Allgemeine Anforderungen an Eignungsprüfungen / Conformity assessment - General requirements for proficiency testing
- Verordnung / Regulation 882/2004/EU; Verordnung über amtliche Kontrollen / Regulation on official controls
- 3. DIN EN ISO/IEC 17025:2005; Allgemeine Anforderungen an die Kompetenz von Prüf- und Kalibrierlaboratorien / General requirements for the competence of testing and calibration laboratories
- Richtlinie / Directive 1993/99/EU; über zusätzliche Maßnahmen im Bereich der amtlichen Lebensmittelüberwachung / on additional measures concerning the official control of foodstuffs
- 5. ASU §64 LFGB : Planung und statistische Auswertung von Ringversuchen zur Methodenvalidierung
- 6. DIN ISO 13528:2009; Statistische Verfahren für Eignungsprüfungen durch Ringversuche / Statistical methods for use in proficiency testing by interlaboratory comparisons
- 7. The International Harmonised Protocol for the Proficiency Testing of Ananlytical Laboratories ; J.AOAC Int., 76(4), 926 - 940 (1993)
- The International Harmonised Protocol for the Proficiency Testing of Ananlytical Chemistry Laboratories ; Pure Appl Chem, 78, 145 - 196 (2006)
- 9. Evaluation of analytical methods used for regulation of food and drugs; W. Horwitz; Analytical Chemistry, 54, 67-76 (1982)
- 10.A Horwitz-like funktion describes precision in proficiency test; M. Thompson, P.J. Lowthian; Analyst, 120, 271-272 (1995)
- 11.Protocol for the design, conduct and interpretation of method performance studies; W. Horwitz; Pure & Applied Chemistry, 67, 331-343 (1995)
- 12.Recent trends in inter-laboratory precision at ppb and sub-ppb concentrations in relation to fitness for purpose criteria in proficiency testing; M. Thompson; Analyst, 125, 385-386 (2000)
- 13.AMC Kernel Density Representing data distributions with kernel density estimates, amc technical brief, Editor M Thompson, Analytical Methods Committee, AMCTB No 4, Revised March 2006 and Excel Add-in Kernel.xla 1.0e by Royal Society of Chemistry
- 14.ASU §64 LFGB L46.02-4; Bestimmung des Gehaltes an 16-0-Methylcafestol in Röstkaffee, HPLC-Verfahren (Januar 2012) (Übernahme der gleichnamigen Norm DIN 10779, Ausgabe März 2011)