Proficiency Tests

DLA

food
cosmetics
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Evaluation Report
proficiency test

DLA 39/2016

Food ingredient:
Ethanol in Marzipan

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Allgemeine Informationen zur Eignungsprüfung (EP)
General Information on the proficiency test (PT)

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EP-Bericht Freigabe *PT-Report Authorization*	Dr. Matthias Besler (Technischer Leiter / Technical Manager) *- gezeichnet / signed M. Besler* Dr. Gerhard Wichmann (QM-Beauftragter / Quality Manager) *- gezeichnet / signed G. Wichmann* Datum / Date: 26 February 2017
Unteraufträge *Subcontractors*	Die Prüfung der Gehalte, Homogenität und Stabilität von EP-Parametern wird von DLA im Unterauftrag vergeben. The analysis of the content, homogeneity and stability of PT-parameters are subcontracted by DLA.

Inhalt / Content

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 verification and/or validation of the particular testing method [1, 5].
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 / ISO 13528:2015 [2, 3].

2. Realisation

2.1 Test material

The test material is a commercial food from a pre-packed product (Marzipan praline with dark chocolate and marzipan filling) from a production containing ethanol as a declared ingredient.

According to the ingredients list, the food is composed as follows:

Almonds, sugar, cocoa mass, cocoa butter, whole milk powder, invert sugar syrup, emulsifier: soya lecithins, vanilla extract, alcohol; the chocolate content is 29%.

Two pralines were welded together in a PE bag under vacuum. The portions were numbered chronologically.

The determination of the content of ethanol was carried out in preliminary investigations of the material.

The material was checked for homogeneity.

2.1.1 Homogeneity

The ethanol content was determined before welding the samples to check the homogeneity in 10 different samples in the marzipan portion by means of an enzymatic UV test according to ASU § 64 LFGB L 40.00-12. With a standard deviation of 2,2%, the homogeneity can be considered as sufficiently assured, see documentation.

The calculation of the **variation coefficient** of the repeatability standard deviation (CV_r/6,4%) of the participants was used as an indicator of homogeneity. The variation coefficient VK_r is similar to the precision data of the ASU § 64 LFGB L 40.00-12 (see 3.6.2/ Table 1).

Furthermore, the homogeneity was characterized by the **trend line function of participants' results for the packaged single samples.** The maximum deviations from the mean value of the trend line was in the range of 30% of the target standard deviation *σpt* (s. 5.2 homogeneity) and is to be judged as sufficient.

If the criteria for sufficient homogeneity of the test material are not fulfilled on a particular parameter, the impact on the target standard deviation is checked and optionally the evaluation of the results of the participants will be done using the z´-score considering the standard uncertainty of the assigned value (see 3.8 and 3.11) [3].

2.1.2 Stability

The storage of the vacuum-sealed samples in a gas-tight film shows good stability of the sample (spoilage) and the content of ethanol at a temperature of -18 ° C. The sample material is therefore stable against microbial spoilage and loss of test parameters when frozen (- 18°C).

2.2 Sample shipment and information to the test

Two portions of test material were sent to every participating laboratory in the 50th week of 2016. The testing method was optional. The tests should be finished at January 27th 2017 the latest.

With the cover letter along with the sample shipment the following information was given to participants:

The two samples are identical samples marzipan (with chocolate coating) to perform a complete duplicate determination. **The ethanol content must be determined only in the marzipan portion.**
In general we recommend to homogenize a representative sample amount before analysis according to good laboratory practice, especially in case of low sample weights.

2.3 Results

The participants submitted their results in standard forms, which have been handed out with the samples (by email).

The finally calculated concentrations as average of duplicate determinations of both numbered samples was used for the statistical evaluation. For the calculation of the Repeatability- and Reproducibility standard deviation the single values of the double determination were used.

Queried and documented were single results, recovery and the used testing method, information on the limit of quantification, the date of the analysis and general points to the method.

All 14 participants submitted at least one result in time. One participant delivered the results separately for Samples A and B, as the nature of the samples was apparently differed. These results were evaluated separately and the evaluation number was extended with the suffix a or b.

3. Evaluation

3.1 Consensus values from participants (Assigned value)

The robust mean of the submitted results was used as assigned value (X) („consensus value from participants") providing a normal distribution. The calculation was done according to algorithm A as described in annex C of ISO 13528 [3].

The condition is that the majority of the participants' results show a normal distribution or are distributed unimodal and symmetrically. To this end, an examination of the distribution is carried out, inter alia, using the kernel density estimate [3, 12].

In case there are indications for sources of higher variability such as a bimodal distribution of results, a cause analysis is performed. Frequently different analytical methods may cause an anomaly in results' distribution. If this is the case, separate evaluations with own assigned values (X_{pti}) are made whenever possible.

The statistical evaluation is carried out for all the parameters for a minimum of 7 values are present.

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. For the method description, see documentation 5.1.

The actual measurement results will be drafted. Individual results, which are outside the specified measurement range of the participating laboratory (for example with the result > 25 mg/kg or < 2,5 mg/kg) or the indicating "0" will not be considered for the statistic evaluation [3].

3.2 Robust standard deviation

For comparison to the target standard deviation σ_{pt} (standard deviation for proficiency assessment) a robust standard deviation (S^x) was calculated. The calculation was done according to algorithm A as described in annex C of ISO 13528 [3].

3.3 Repeatability standard deviation

The repeatability standard deviation S_r is based on the laboratory´s standard deviation of (outlier free) individual participant results, each under repeatability conditions, that means analyses was performed on the same sample by the same operator using the same equipment in the same laboratory within a short time. It characterizes the mean deviation of the results within the laboratories [3] and is used by DLA as an indication of the homogeneity of the sample material.

In case single results from participants are available the calculation of the repeatability standard deviation S_r, also known as standard deviation within laboratories S_w, is performed by: [3, 4].

The relative repeatability standard deviation as a percentage of the mean value is indicated as coefficient of variation CV_r in the table of statistical characteristics in the results section in case single results from participants are available.

3.4 Reproducibility standard deviation

The reproducibility standard deviation S_R represents a inter-laboratory estimate of the standard deviation for the determination of each parameter on the bases of (outlier free) individual participant results. It takes into account both the repeatability standard deviation S_r and the within-laboratory standard deviation S_S. Reproducibility standard deviations of PT´s may differ from reproducibility standard deviations of ring trials, because the participating laboratories of a PT generally use different internal conditions and methods for determining the measured values.

In the present evaluation, the specification of the reproducibility standard deviation, therefore, does not refer to a specific method, but characterizes approximately the comparability of results between the laboratories, assumed the effect of homogeneity and stability of the sample are negligible.

In case single results from participants are available the calculation of the reproducibility standard deviation S_R is performed by: [3, 4].

The relative reproducibility standard deviation as a percentage of the mean value is indicated as coefficient of variation CV_R in the table of statistical characteristics in the results section in case single results from participants are available. Its meaning is explained in more detail in 3.9.

3.5 Exclusion of results and outliers

Before statistical evaluation obvious blunders, such as those with incorrect units, decimal point errors, and results for a another proficiency test item can be removed from the data set [2]. Even if a result is e.g. with a factor >10 deviates significantly from the mean value and has an influence on the robust statistics, a result can be excluded from the statistical evaluation [3].

All results should be given at least with 2 significant digits. Specifying 3 significant digits is usually sufficient.

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 [3, 12].

Results are identified as outliers by the use of robust statistics. If a value deviates from the robust mean by more than 3 times the robust standard deviation, it is classified as an outlier [3]. Detected outliers are stated for information only, when z-score are < -2 or > 2. Due to the use of robust statistics outliers are not excluded, provided that no other reasons are present [3].

3.6 Target standard deviation (for proficiency assessment)

The target standard deviation of the assigned value σ_{pt} (= standard deviation for proficiency assessment) can be determined according to the following methods.

If an acceptable quotient S^*/σ_{pt} is present, the target standard deviation of the general model by Horwitz is preferably used for the proficiency assessment. It is usually suitable for evaluation of interlaboratory studies, where different methods are applied by the participants. On the other hand the target standard deviation from the evaluation of precision data of an precision experiment is derived from collaborative studies with specified analytical methods.

In cases where both above-mentioned models are not suitable, the target standard deviation is determined based on values by perception, see under 3.6.3.

For information, the z-scores of both models are given in the evaluation, if available.

The target standard deviation according to Horwitz was used (see 3.6.1). It was evaluated using the z'-score, taking into account the increased variability. The reason for the relatively high statistical uncertainty could be the use of different methods (enzymatic methods, GC/MS, GC/FID).

For the purpose of information, the target standard deviation of a precision experiment is also given (ASU §64 Method: [16]), see 3.6.2 / Table 1.

3.6.1 General model (Horwitz)

Based on statistical characteristics obtained in numerous PTs for different parameters and methods Horwitz has derived a general model for estimating the reproducibility standard deviation σ_R [6]. Later the model was modified by Thompson for certain concentration ranges [10]. The reproducibility standard deviation σ_R can be applied as the relative target standard deviation σ_{pt} in % of the assigned values and calculated according to the following equations [3]. For this the assigned value X_{pt} is used for the concentration c.

Equations	**Range of concentrations**	**corresponds to**
$\sigma_R = 0{,}22c$	$c < 1{,}2 \times 10^{-7}$	< 120 µg/kg
$\sigma_R = 0{,}02c^{0{,}8495}$	$1{,}2 \times 10^{-7} \leq c \leq 0{,}138$	≥ 120 µg/kg
$\sigma_R = 0{,}01c^{0{,}5}$	$c > 0{,}138$	> 13,8 g/100g

with c = mass content of analyte (as relative size, e.g. 1 mg/kg = 1 ppm = 10^{-6} kg/kg)

3.6.2 Precision experiment

Using the reproducibility standard deviation σ_R and the repeatability standard deviation σ_r of a precision experiment (collaborative trial or proficiency test) the target standard deviation σ_{pt} can be derived considering the number of replicate measurements m of participants in the present PT [3]:

$$\sigma_{pt} = \sqrt{\sigma_R^2 - \sigma_r^2\left(m - 1/m\right)}$$

From the precision data of the relevant official method the target standard deviation for the corresponding parameters are calculated, if available, and used for the evaluation.

The relative repeatability standard deviations (RSD_r) and relative reproducibility standard deviations (RSD_R) given in Table 1 were determined in collaborative trials using the specified methods. The target standard deviation is given for information in the evaluation.

Table 2: *Relative repeatability standard deviations (RSD_r) and relative reproducibility standard deviations (RSD_R) from selected precision experiments and resulting target standard deviations σ_{pt}* [16]

Parameter	Matrix	Mean (g/100g)	RSD_r	RSD_R	σ_{pt} (g/100g)	Method/ Literature
Ethanol	Honey	0,00046	10,8%	13,2%	0,072	Enzymatic/ 16
Ethanol	Honey	0,0035	2,4%	5,8%	0,037	Enzymatic/ 16
Ethanol	Honey	0,0151	1,9%	7,8%	0,051[1]	Enzymatic/ 16

[1] *Value used in the evaluation (see 4.1)*

3.6.3 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 [3].

For the present evaluation the target standard deviation according to 3.6.1 were regarded suitable.

3.7 z-Score

To assess the results of the participants the z-score is used. It indicates about which multiple of the target standard deviation (σ_{pt}) the result (x_i) of the participant is deviating from the assigned value (X_{pt}) [3].
Participants' z-scores are derived from:

$$z_i = \frac{\left(x_i - x_{pt}\right)}{\sigma_{pt}}$$

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

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

The z-score valid for the PT evaluation is designated z-score (σ_{pt}), while the value of z-score (Info) is for information only. The two z-scores are calculated using the different target standard deviations according to 3.6.

3.7.1 Warning and action signals

In accordance with the norm ISO 13528 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" [3]. 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 signal" 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 [3].

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 the signals are valid only in case of a number of ≥ 10 results [3].

3.8 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_{pt}) [3].

The calculation is performed by:

$$z_i' = \frac{x_i - x_{pt}}{\sqrt{\sigma_{pt}^2 + u_{(x_{pt})}^2}}$$

If carried out an evaluation of the results by means of z 'score, we have defined below the expression in the denominator as a target standard deviation σ_{pt}'.

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

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

For warning- and action-signals see 3.7.1.

3.9 Reproducibility coefficient of variation (CV)

The variation coefficient (CV) of the reproducibility (= *relative reproducibility standard deviation)* is calculated from the standard deviation and the mean as follows [4, 13]:

$$CV_R = \frac{S_R * 100}{X}$$

In contrast to the standard deviation as a measure of the absolute variability the CV gives the relative variability within a data region. While a low CV, e.g. <5-10% can be taken as evidence for a homogeneous set of results, a CV of more than 50% indicates a "strong inhomogeneity of statistical mass", so that the suitability for certain applications such as the assessment of exceeded maximum levels or the performance evaluation of the participating laboratories possibly can not be done [3].

3.10 Quotient S^*/σ_{pt}

Following the Horrat-value the results of a proficiency-test (PT) can be considered convincing, if the quotient of robust standard deviation S* and target standard deviation σ_{pt} 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 [3].

3.11 Standard uncertainty

The consensus value has a standard uncertainty $U(X_{pt})$ that depends on the analytical method, differences between the analytical methods used, the test material, the number of participant laboratories (P) and perhaps on other factors. The standard uncertainty of the assigned value ($U_{(Xpt)}$) for this PT is calculated as follows [3]:

$$u_{(x_{pt})} = 1{,}25 \times \frac{s^*}{\sqrt{p}}$$

If $U_{(Xpt)} \leq 0{,}3\ \sigma_{pt}$ the standard uncertainty of the consensus value needs not to be included in the interpretation of the results of the PT [3]. 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 assigned value.

The quotient $U(X_{pt})/\sigma_{pt}$ 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.

In the first table the characteristics are listed:

Statistic Data
Number of results
Number of outliers
Mean
Median
Robust mean(X_{pt})
Robust standard deviation (S^*)
Number with 2 replicates
repeatability standard deviation (S_r)
Repeatability (CV_r) in %
reproducibility standard deviation (S_R)
Reproducibility (CV_R) in %
Target range:
Target standard deviation σ_{pt} or σ_{pt}'
Target standard deviation (for information)
lower limit of target range ($X_{pt} - 2\sigma_{pt}$) or ($X_{pt} - 2\sigma_{pt}$') *
upper limit of target range ($X_{pt} + 2\sigma_{pt}$) or ($X_{pt} + 2\sigma_{pt}$´) *
Quotient S/σ_{pt} or S*/σ_{pt}'*
Standard uncertainty $U(X_{pt})$
Quotient $U(X_{pt})/\sigma_{pt}$ or $U(X_{pt})/\sigma_{pt}$'
Results in the target range
Percent in the target range

* Target range is calculated with z-score or z'-score

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

Auswerte-nummer	**Parameter [Einheit/ Unit]**	**Abweichung**	**Z´-Score** σ_{pt}´	**z-Score** (Info)	**Hinweis**
Evaluation number		**Deviation**			**Remark**

4.1 Ethanol in g/100g

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	15
Number of outliers	0
Mean	0,659
Median	0,677
Robust Mean (X)	**0,667**
Robust standard deviation (S*)	**0,112**
Number with 2 replicates	14
Repeatability SD (S_r)	0,0424
Repeatability (CV_r)	6,4%
Reproducibility SD (S_R)	0,129
Reproducibility (CV_R)	19,6%
Target range:	
Target standard deviation σ_{pt}´	**0,0460**
Target standard deviation (for Information)	0,0513
lower limit of target range	**0,575**
upper limit of target range	**0,759**
Quotient S/σ_{pt}´*	*2,4*
Standard uncertainty $U_{(Xpt)}$	*0,0362*
Quotient $U_{(Xpt)}/\sigma_{pt}$´	*0,79*
Results in the target range	*9*
Percent in the target range	*60%*

Anmerkungen zu den Kenndaten:

The standard target deviation was evaluated using the model of Horwitz. It was evaluated using the z'-score, taking into account the standard uncertainty. The target standard deviation "for information" was calculated from values by precision experiments, see 3.6.2.

The distribution of the results showed an increased variability. The quotient S*/σpt was over 2,0. The variation coefficient (with respect to the repeatability standard deviation, CV_r) is in the range of established values for the methods used (see 3.6.2). The comparability of results is given.

The quotient $U(X_{pt})/\sigma_{pt}$ (0,64) is increased.

60% of the results were in the target range.

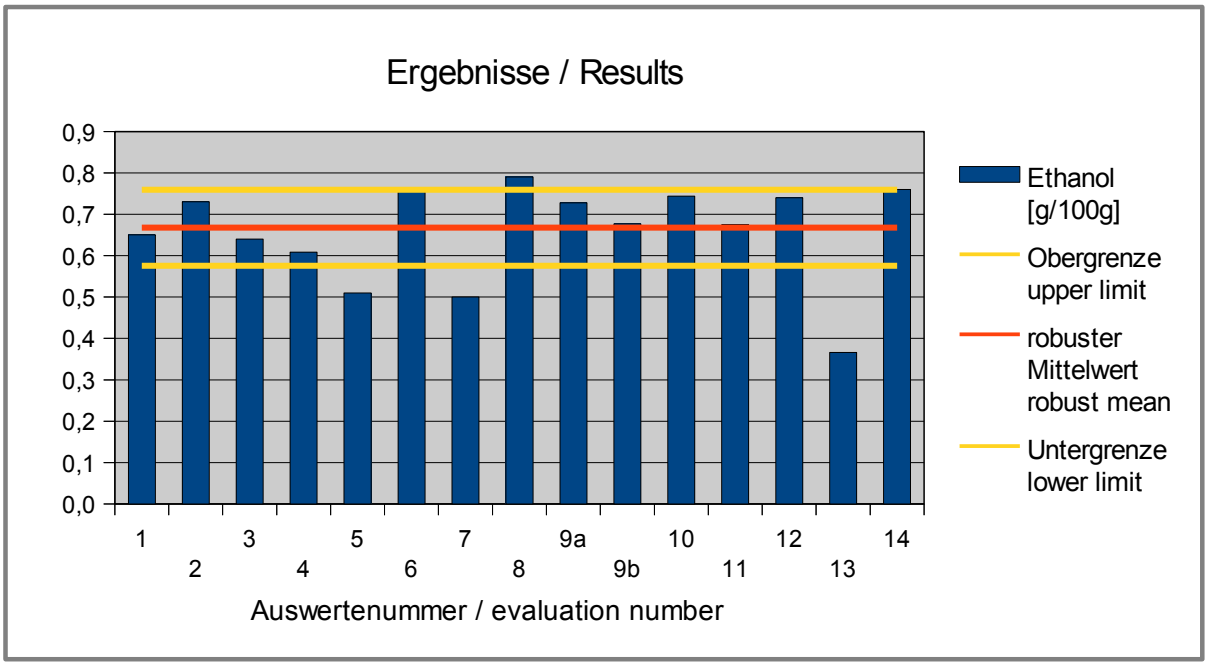

Abb. / Fig. 1: Ergebnisse Ethanol/ Results Ethanol

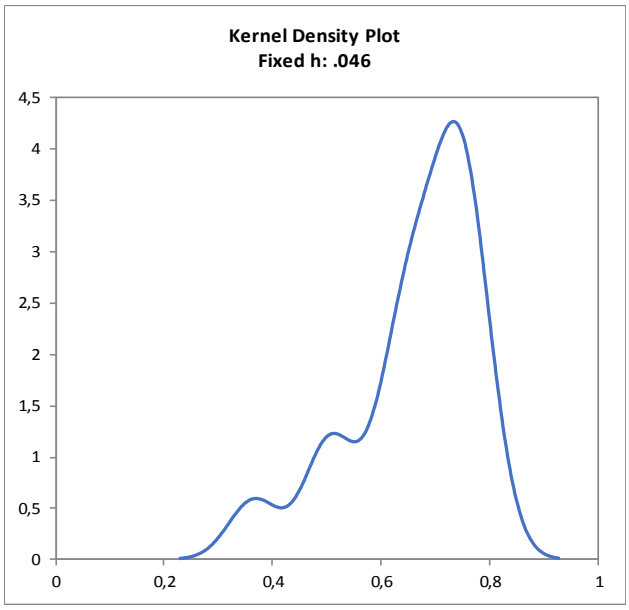

Abb. / Fig. 2:
Kerndichte-Schätzung der Ergebnisse (mit h = σ_{pt}´ von X_{pt} = 0,046 g/100g)

Kernel density plot of results (with h= σ_{pt}´ of X_{pt} = 0,046 g/100g)

Comment:
The kernel density shows a normal distribution of results with two side-peaks at 0,37 g/100g and 0,5 g/100g.

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer Evaluation number	Ethanol [g/100g]	Abweichung [µg/kg] Deviation [µg/kg]	z´-Score (σ_{pt})	z-Score (Info)	Hinweis Remark
1	0,650	-0,0174	-0,38	-0,34	
2	0,730	0,0626	1,4	1,2	
3	0,640	-0,0274	-0,60	-0,53	
4	0,608*	-0,0594	-1,3	-1,2	
5	0,510	-0,157	-3,4	-3,1	
6	0,760	0,0926	2,0	1,8	
7	0,495	-0,167	-3,6	-3,3	
8	0,790	0,123	2,7	2,4	
9a	0,728	0,0606	1,3	1,2	
9b	0,677	0,00958	0,21	0,19	
10	0,744	0,0766	1,7	1,5	
11	0,675**	0,00758	0,16	0,15	
12	0,740	0,0726	1,6	1,4	
13	0,366*	-0,301	-6,6	-5,9	
14	0,760	0,0926	2,0	1,8	

* By DLA converted from total sample to marzipan part
** Mean calculated by DLA

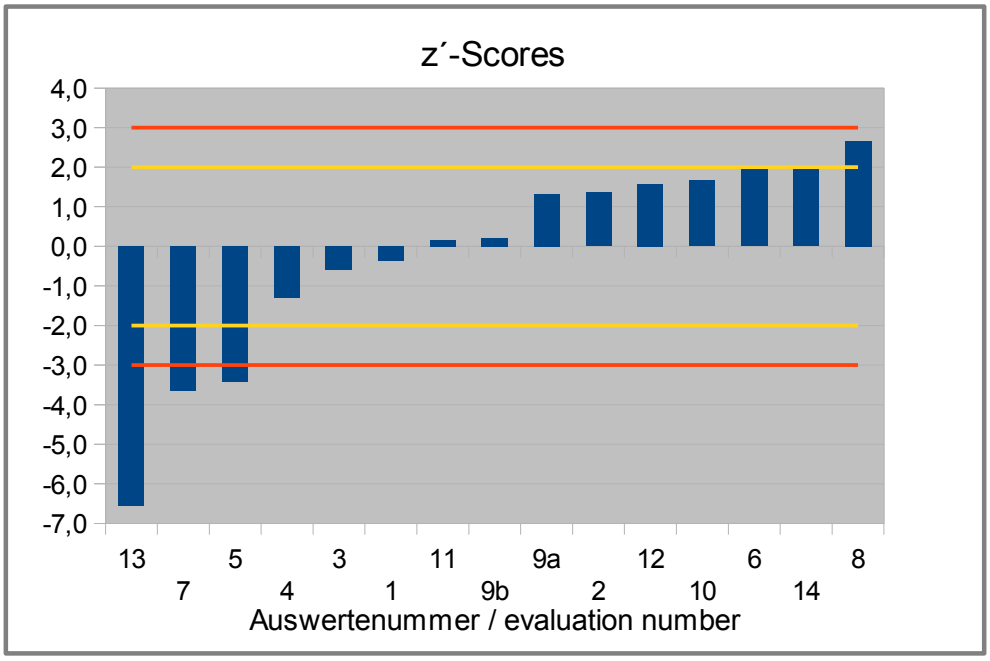

Abb. / Fig. 3: Z´-Scores Ethanol

5. Documentation

5.1 Details by participants

5.1.1 Primary data

5.1.1.1 Ethanol

Teilnehmer/ participant	Ergebnis/ result	DLA-Nr Probe A/ sampel A	DLA-Nr Probe B/ sample B	Datum der Analyse/ date of the analysis	Ergebnis A/ result A	Ergebnis B/ result B	Bestimmungs-grenze/ Limit of determina-tion	Inkl. Wiederfind ung/ incl. recovery	Wiederfin-dungsrate/ recovery	Wiederfindung mit gleicher Matrix/ recovery with the same matrix
	g/100g			day/month	g/100g	g/100g	g/100g	yes/ no	in %	yes/no
1	0,65			25.01.17						
2	0,73	4	50	18.01.17	0,72	0,73	0,01	no	n.d.	no
3	0,64	17	63	29.12.16	0,60	0,68	0,0005	no		no
4	0,432*1	46	69	14.12.16	0,420+ 0,458	0,39+ 0,458	10 mg/kg	no	no	
5	0,51	43	67	22.12.16	0,46	0,56	0,02	no	no	no
6	0,76	15	66	12.01.17	0,75	0,77	0,01	no		
7	0,50	18	30	12.01.17	0,50	0,49	0,05	no	95,8	no
8	0,79	24	35	16.12.16	0,78	0,80	0,07	yes	99,45	no
9a	0,728*3	10		10.01.17	0,728		0,05	no	90-110	yes
9b	0,677*3		54	10.01.17		0,677	0,05	no	90-110	yes
10	0,744	7	38	03.01.17	0,698	0,789	0,01	no	102	no
11	0,675*2	20	41	06.01.17	0,62	0,73	<10 mg/100g	no		
12	0,740	29	62	19.12.16	0,726	0,746				
13	0,26*1	11	60	14.12.2016/ 29.12.2016	0,26	0,25	0,02	yes	99,9	no
14	0,76	6	51	15.12.16	0,74	0,77		yes	98,5	no

*1 Values given based on the total sample (for the evaluation converted by DLA with the known chocolate content of 29%)
*2 Mean calculated by DLA
*3 Values not averaged as the optical properties of sample A and sample B differ

5.1.2 Analytical methods

5.1.2.1 Ethanol

Teilnehmer/ Participant	Methode/ Method	Methode ist akkreditiert/ Method is accredited	Sonstige Hinweise/ further remarks
		ja / nein	
1		no	
2	Residual solvents according to THV	yes	The chocolate content was removed before weighing in / analysis, the content refers to the marzipan content (without chocolate)
3	GC/MS	no	
4	Internal method, HS-GC-FID	yes	* per total sample
5	004 MPP Amb061 Rev1 2010	no	no
6	enzymatic	yes	Data without recovery
7		yes	
8	enzymatic, analog ASU L36.00-12 (2002-12)	yes	
9a	Enzymatic detemination according to method no. 307.1 SLMB	yes	
9b	Enzymatic detemination according to method no. 307.1 SLMB	yes	Sample B (54) with very moist surface
10	GC-FID, internal method	yes	
11	according to r-biopharm	yes	
12		no	
13	Ethanol, enzymatic (r-biopharm)	yes	Result total sample (Marzipan + chocolate)
14	By enzymatic UV-test (Megazyme K-ETOH 01/14) determined.	yes	

5.2 Homogeneity

5.2.1 Homogeneity testing before PT

The **homogeneity was tested** in 10-fold analysis before packing.

Probe/ sample	**Ethanol**		
1	0,74	g/100g	
2	0,79	g/100g	
3	0,78	g/100g	
4	0,78	g/100g	
5	0,79	g/100g	
6	0,80	g/100g	
7	0,80	g/100g	
8	0,81	g/100g	
9	0,80	g/100g	
10	0,89	g/100g	
Rob. Mittelwert/ Rob. Mean	**0,794**		
Rob. Standardabw./ Rob. Standard deviation	**0,0178**	**2,2**	**%**

5.2.2 Comparison of sample number/test results and trend line

By comparison of the **increasing sample numbers** and the measurement results, the homogeneity of the PT material can be characterized with the help of the trend line function:

Ethanol				
Target standard deviation σ_{pt}	0,0460			g/100g
Sample numbers	1 - 69			
Total numbers of samples	26			
Slope	-0,00100			
Trend line range	0,667	-	0,641	g/100g
Deviation trend line	0,654	±	0,013	g/100g
Precent of σ_{pt}	28,3	%		

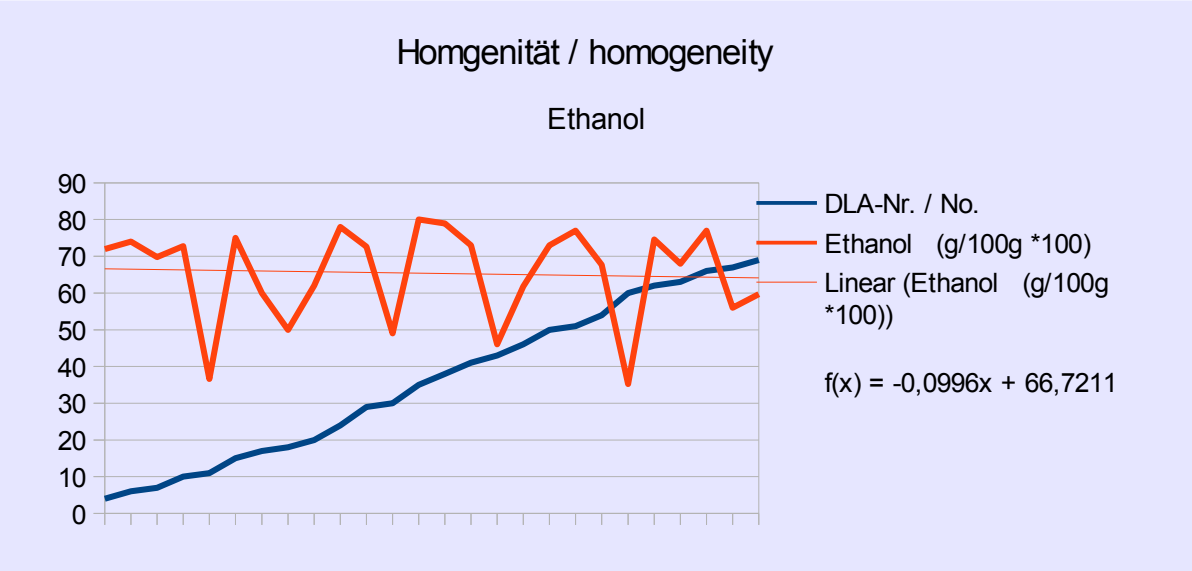

Abb./Fig. 4:
Trendfunktion Probennummern vs. Ergebnisse
trend line function sample number vs. results

6. Index of participant laboratories

Teilnehmer/ Participant	Ort/ Town	Land/ Country
		Italy
		Germany
		Germany
		Germany
		Germany
		Germany
		Germany
		Germany
		Germany
		Great Britain
		Netherlands
		France
		THAILAND
		Germany

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

[The address data of the participants were deleted for publication of the evaluation report.]

7. Index of literature

1. 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
2. DIN EN ISO/IEC 17043:2010; Konformitätsbewertung – Allgemeine Anforderungen an Eignungsprüfungen / Conformity assessment – General requirements for proficiency testing
3. ISO 13528:2015 & DIN ISO 13528:2009; Statistische Verfahren für Eignungsprüfungen durch Ringversuche / Statistical methods for use in proficiency testing by interlaboratory comparisons
4. ASU §64 LFGB: Planung und statistische Auswertung von Ringversuchen zur Methodenvalidierung / DIN ISO 5725 series part 1, 2 and 6 Accuracy (trueness and precision) of measurement methods and results
5. Verordnung / Regulation 882/2004/EU; Verordnung über über amtliche Kontrollen zur Überprüfung der Einhaltung des Lebensmittel- und Futtermittelrechts sowie der Bestimmungen über Tiergesundheit und Tierschutz / Regulation on official controls performed to ensure the verification of compliance with feed and food law, animal health and animal welfare rules
6. Evaluation of analytical methods used for regulation of food and drugs; W. Horwitz; Analytical Chemistry, 54, 67-76 (1982)
7. The International Harmonised Protocol for the Proficiency Testing of Ananlytical Laboratories ; J.AOAC Int., 76(4), 926 – 940 (1993)
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14. GMP+ Feed Certification scheme, Module: Feed Safety Assurance, chapter 5.7 Checking procedure for the process accuracy of compound feed with micro tracers in GMP+ BA2 Control of residues, Version: 1st of January 2015 GMP+ International B.V.
15. MTSE SOP No. 010.01 (2014): Quantitative measurement of mixing uniformity and carry-over in powder mixtures with the rotary detector technique, MTSE Micro Tracers Services Europe GmbH
16. ASU § 64 LFGB L 40.00-12 Untersuchung von Honig. Bestimmung des Gehaltes an Ethanol; enzymatisches Verfahren, nach DIN 10762; September 2006 (Ethanol in honey, UV-Test)
17. ASU § 64 LFGB L 36.00-13 Bestimmung von Ethanol in Bier mit geringem Alkoholgehalt; Dezember 1992