Proficiency Tests

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Evaluation Report
proficiency test

DLA 53/2018

Cosmetic Products III:

Allergenic Fragrances

in Skin Cream

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

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Unteraufträge *Subcontractors*	Falls im Rahmen der Eignungsprüfung eine Prüfung der Gehalte, Homogenität und Stabilität von EP-Parametern durchgeführt wurde, hat DLA diese im Unterauftrag vergeben. In case the analysis of the content, homogeneity and stability of PT-parameters was part of the proficiency test, the determinations were subcontracted by DLA.
Vertraulichkeit *Confidentiality*	Die Teilnehmerergebnisse sind im EP-Bericht in anonymisierter Form mit Auswertenummern benannt. Daten einzelner Teilnehmer werden ausschließlich nach vorheriger Zustimmung des Teilnehmers an Dritte weitergegeben. Participant result are named anonymously with evaluation numbers in the PT report. Data of individual participants will be passed on to third parties only with prior consent of the participant.

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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 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 mixture of common in commerce skin creams or body lotions from European Suppliers with added essential oils.

The materials were mixed and homogenized. The composition of the PT samples (list of ingredients) is shown in table 1.

Afterwards the samples were portioned to approximately 25 g into 28 ml plastic containers, sealed in metallised PET film bags and chronologic-ally numbered.

Table 1: Composition of DLA-Samples

PT-Samples Skin Cream

Skin cream / Body lotion 1
Ingredients: Aqua, Glycerin, Cetearyl Alcohol, C15-19 Alkane, Isopropyl Palmitate, Paraffinum Liquidum, Glyceryl Stearate SE, Butyrospermum Parkii Butter, Dimethicone, Glyceryl Stearate, Sodium Cetearyl Sulfate, Sodium hydroxide, Carbomer, Phenoxyethanol, Ethylhexylglycerin, Linalool, Limonene, Butylphenyl Methylpropional, Benzyl Alcohol, Citronellol, Alpha-Isomethyl Ionone, Parfum

Skin cream / Body lotion 2
Ingredients: Aqua, Glycerin, Paraffinum Liquidum, Stearic Acid, Sorbitol, Glycol Stearate, Caprylic/Capric Triglyceride, Dimethicone, Octyldodecanol, Carbomer, Cetyl Alcohol, Cyclopentasiloxane, Dimethiconol, Disodium EDTA, Glyceryl Stearate, Helianthus Annuus Hybrid Oil, Isohexadecane, Isomerized Linoleic Acid, Magnesium Aluminum Silicate, Methylparaben, Parfum, Phenoxyethanol, Propylparaben, Polysorbate 80, Sodium Acrylate/Sodium Acryloyldimethyl Taurate Copolymer, Sorbitan Oleate, Stearamide AMP, Tocopheryl Acetate, Triethanolamine, Alpha-Isomethyl Ionone, Benzyl Alcohol, Citronellol, Coumarin, Geraniol, Hexyl Cinnamal, Limonene, Linalool

Skin cream / Body lotion 3
Ingredients: Aqua, Hydrogenated Polydecene, Propylene Glycol, Dimethicone, Cyclopentasiloxane, Parfum, Squalane, Aloe Barbadensis Leaf Juice, Benzyl Benzoate, BHT, Butylphenyl Methylpropional, Carbomer, Citral, Citric Acid, Citronello, Coumarin, Dimethiconol, Geraniol, Helianthus Annuus Seed Oil, Limonene, Linalool, Polyglcderyl-3 Ricinolate, Polyquaternium-51, Sodium Hyaluronate, Sorbitan Oleate, Tetrasodium EDTA, Tocopherol, Tromethamine, Butylparaben, Chlorphenesin, Ethylparaben, O-Cymen-5-ol, Phenoxyethanol, Potassium Sorbate, Propylparaben, Sodium Benzoate

Skin cream / Body lotion 4
Ingredients: Aqua, Glycerin, Caprylic/Capric Triglyceride, Dicaprylyl Ether, Cetearyl Alcohol, Dicaprylyl Carbonate, Glyceryl Stearate, Pentaerythrityl Distearate, Parfum, Butyrospermum Parkii Butter, Dimethicone, Sodium Stearoyl Glutamate, Carbomer, Sodium Polyacrylate, Phenoxyethanol, Benzyl Alcohol, Limonene, Linalool, Citral, Geraniol, Citronellol, Sodium Hydroxide

Further ingredients (essential oils)
Ingredients: Cymbopogon Flexuosus Herb Oil, Amyris Balsamifera Bark Oil, Cananga Odorata Flower Oil, Cinnamomum Cassia Bark Oil

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Note: The metrological traceability of temperature, mass and volume during production of the PT samples is ensured by DAkkS calibrated reference materials.

2.1.1 Homogeneity

The calculation of the **repeatability standard deviations S_r of the participants** was used as an indicator of homogeneity. For all parameters the repeatability standard deviation was < 8,0% (2,48% - 7,82%) (see Table 2). Thus the repeatability standard deviations are comparable to the precision data from a method development (2D GC-MS method) which gave relative repeatability standard deviations of < 10% (Belhassen et. al. 2017) [18-20]. The repeatability standard deviations of the participants' results are given in the documentation in the statistic data (see 4.1 to 4.14).

Table 2: Repeatability standard deviation S_r of double determinations of the participants (coefficient of variation CV_r in %)

Parameter	CV_r
Alpha-Isomethyl Ionone	7,82 %
Benzyl Alcohol	3,29 %
Benzyl Benzoate	2,62 %
Benzyl Salicylate	5,51 %
Butylphenyl Methylpropional	3,89 %
Cinnamal	2,48 %
Citral	4,79 %
Citronellol	4,12 %
Coumarin	5,15 %
Eugenol	3,81 %
Geraniol	6,88 %
Hexylcinnamaldehyd	5,00 %
Limonene	3,34 %
Linalool	2,77 %

Furthermore, the homogeneity was graphically characterized for information by the **trend line function of participants' results for chronological bottled single samples** for the parameters Benzyl Benzoate and Butylphenyl Methylpropional (s. 5.2.1).

In case the criterion for sufficient homogeneity of the test items is not fulfilled the impact on the target standard deviation will be verified. If necessary the evaluation of results will be done considering the standard uncertainty of the assigned value by z'-scores (s. 3.8 and 3.11) [3].

2.1.2 Stability

Experience has shown that unopened preserved skin creams are stable for several years. For the products, the manufacturer gave a shelf life of 12 months after opening. The stability of the sample material was thus ensured during the investigation period under the specified storage conditions.

2.2 Sample shipment and information to the test

Two portions of test material were sent to every participating laboratory in the 34th week of 2018. The testing method was optional. The tests should be finished at 19th October 2018 the latest.

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

The two portions contain identical samples of a mixture of common in commerce skin creams and body lotions with volatile oils with the parameters allergenic fragrances to be determined (substances to be labelled according to Cosmetics Regulation EC 1223/2009 Art.19 g + Annex III).

Note: When a parameter is a mixture of isomers the final amount is calculated according to method EN 16274 section 8.2.

Please note the attached information on the proficiency test.
(see documentation, section 5.4 Information on the PT)

2.3 Submission of results

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

The finally calculated concentrations of the parameter as average of duplicate determinations of both numbered samples were 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 methods. 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.

Out of 14 participants, 12 participants submitted their results in time. Two participants have not submitted any results.

3. Evaluation

3.1 Consensus value from participants (assigned value)

The robust mean of the submitted results was used as assigned value (X_{pt}) („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]. If there are < 12 quantitative results and an increased difference between robust mean and median, the median may be used as the assigned value (criterion: Δ median - rob. mean > 0,3 σ_{pt}) [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 justified cases, an evaluation may also be carried out from 5 results onwards.

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 stat-

istical 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 CV_R in percent of the mean is given as variation coefficient in the statistical data of participant for each parameter. The significance of CV_R is further explained in section 3.9.

3.5 Exclusion of results and outliers

Before statistical evaluation obvious blunders, such as those with incorrect units, decimal point errors, too few significant digits (valid digits) or results for another proficiency test item can be removed from the data set [2]. Even if a result e.g. with a factor >10 deviates significantly from the mean and has an influence on the robust statistics, a result of the statistical evaluation can be excluded [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 tested for outliers by the use of robust statistics (algorithm A): If a value deviates from the robust mean by more than 3 times the robust standard deviation, it can be classified as an outlier (see above) [3]. Due to the use of robust statistics outliers are not excluded, provided that no other reasons are present [3]. Detected outliers are only mentioned in the results section, if they have been excluded from the statistical evaluation.

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.

*For valuation of all following parameters in the present PT the target standard deviation according to the **general model of Horwitz** was applied (see 3.6.1): Alpha-Isomethyl Ionone, Benzyl Alcohol, Benzyl Benzoate, Benzyl Salicylate, Butylphenyl Methylpropional, Cinnamal, Citral, Citronellol, Coumarin, Eugenol, Geraniol, Hexylcinnamal, Limonene, Linalool.*

__Additionally__ for all parameters except Benzyl Salicylate the standard uncertainty was considered by evaluation using z'-scores (see 3.6.8).

Wherein the results of the following parameters were not evaluated due to the number of < 7: Amyl Cinnamal, Amylcinamyl Alcohol, Anise Alcohol, Benzyl Cinnamate, Cinnamyl Alcohol, Evernia Furfuracea Extract, Evernia Puranstri Extract, Farnesol, Hydroxycitronellal, Hydroxyisohexyl 3-Cyclohexene, Isoeugenol, Methyl 2-Octynoate.

The corresponding participant results can be found in the documentation section.

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
$\sigma_R = 0{,}02c^{0,8495}$	$1{,}2 \times 10^{-7} \le c \le 0{,}138$	≥ 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 Value by 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]:

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

For the determination of allergenic fragrances in cosmetic products to our knowledge currently there are no sufficient data available on relative repeatability standard deviations (RSD$_r$) and relative reproducibility standard deviations (RSD$_R$) from interlaboratory comparisons or ring trials. A standardized method (EN 16274 / IRFA) and an extensive method development (Belhassen et al., 2017) have been published [18-20].

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 was regarded suitable partly using the z'-scores.

Table 3 shows selected statistic data of participants results of present PT compared to PT results of previous years.

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 valid z-Score for each parameter is indicated as z-Score (σ_{pt}). The value indicated as z-Score (Info) only obtains a informative character. The both z-Scores were calculated with the different target standard deviations in accordance with 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].

<u>Table 3:</u> Characteristics of the present PT (on dark gray) in comparison to previous PTs since 2014 (SD = standard deviation, CV = coefficient of variation)

Parameter	Matrix	robust Mean [mg/kg]	rob. SD (S*) [mg/kg]	rel. SD (VK$_{S*}$) [%]	Quotient S*/σ$_{pt'}$	DLA-Report
Citral	After shave	83,1	45,1	54,3	6,6	DLA 34/2014
Citral	Skin cream	531	71,8	13,5	1,6	DLA 53/2018
Benzyl Salicyl-ate	After shave	425	64,0	15,1	2,3	DLA 34/2014
Benzyl Salicyl-ate	Skin cream	102	14,4	14,1	1,8*	DLA 53/2018
Coumarin	After shave	438	58,3	13,3	2,1	DLA 34/2014
Coumarin	Skin cream	63,3	16,2	25,6	2,0	DLA 53/2018
Geraniol	After shave	37,3	14,5	38,9	4,2	DLA 34/2014
Geraniol	Skin cream	86,2	20,0	23,1	1,9	DLA 53/2018
Butylphenyl Methylpropional	After shave	224	90,0	40,1	5,7	DLA 34/2014
Butylphenyl Methylpropional	Skin cream	306	83,3	27,2	2,3	DLA 53/2018
Linalool	After shave	946	112	12,2	2,1	DLA 34/2014
Linalool	Skin cream	509	119	23,3	2,2	DLA 53/2018
Limonene	After shave	2980	1080	36,2	7,5	DLA 34/2014
Limonene	Skin cream	250	106	42,5	2,5	DLA 53/2018
Alpha-Isomethyl Ionone	After shave	152	44,5	29,2	3,9	DLA 34/2014
Alpha-Isomethyl Ionone	Skin cream	17,1	5,75	33,6	2,0	DLA 53/2018

with target standard deviation σ$_{pt}$

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 (σ_{pt}) 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 cofficient of variation (CV_R)

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 varia- bility 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 evalu- ation 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 of the assigned value

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

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

If $U_{(Xpt)}$ ≤ 0,3 σ_{pt} the standard uncertainty of the assigned value needs not to be included in the interpretation of the results of the PT [3]. Values exceeding 0,3 imply, that the target standard deviation could be too low with respect to the standard uncertainty of the assigned value.

The traceability of the assigned value is ensured on the basis of the consensus value as a robust mean of the participant results.

4. Results

<u>Comments to the distribution of the results:</u>

The kernel density plots showed for all parameters nearly a symmetrical distribution of results (figures see documentation 5.3). Partly slight shoulders and separated smaller peaks can be seen, which are due to individual results, outliers or values outside the target range.

<u>Comments to the statistic data:</u>

For Amyl Cinnamal, Amylcinamyl Alcohol, Anise Alcohol, Benzyl Cinnamate, Cinnamyl Alcohol, Evernia Furfuracea Extract, Evernia Puranstri Extract, Farnesol, Hydroxycitronellal, Hydroxyisohexyl 3-Cyclohexene, Isoeugenol, Methyl 2-Octynoate there were < 7 results, therefore no statistical evaluation could be done.

For all other parameters the target standard deviation was calculated according to the general model of Horwitz.

For Benzyl Salicylate the distribution of results showed a normal variability. The quotients S^*/σ_{pt} was 1,8 (see table 4).

For the other parameters Alpha-Isomethyl Ionone, Benzyl Alcohol, Benzyl Benzoate, Butylphenyl Methylpropional, Cinnamal, Citral, Citronellol, Coumarin, Eugenol, Geraniol, Hexylcinnamal, Limonene and Linalool the distribution of results showed an increased variability with quotients above 2,0. These parameters were evaluated considering the standard uncertainty by z´-scores. The quotients S^*/σ_{pt}' were then in the range of 2,0 (1,6-2,5) (see table 3).

The robust standard deviation and the repeatability and reproducibility standard deviations were in the expected range of usual values for the used determination methods (s. 3.6.3) [20].

The comparability of results is given.

64% to 90% of results were in the respective target range.

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 m replicate measurements
Repeatability standard deviation (S_r)
Coefficient of Variation (CV_r)in %
Reproducibility standard deviation (S_R)
Coefficient of Variation (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})
Number of results in the target range
Percent in the target range

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

In the table below, the results of the participating laboratories are formatted in 3 valid digits**:

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

*** In the documentation part, the results are given as they were transmitted by the participants.*

4.1 Alpha-Isomethyl Ionone in mg/kg

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	11
Number of outliers	0
Mean	17,1
Median	17,0
Robust Mean (X_{pt})	**17,1**
Robust standard deviation (S*)	**5,75**
Number with 2 replicates	11
Repeatability SD (S_r)	1,34
Repeatability (CV_r)	7,82%
Reproducibility SD (S_R)	5,14
Reproducibility (CV_R)	30,1%
Target range:	
Target standard deviation σ_{pt}'	**2,81**
lower limit of target range	**11,5**
upper limit of target range	**22,7**
Quotient S*/σ_{pt}'	*2,0*
Standard uncertainty $U_{(Xpt)}$	*2,17*
Results in the target range	*8*
Percent in the target range	*73%*
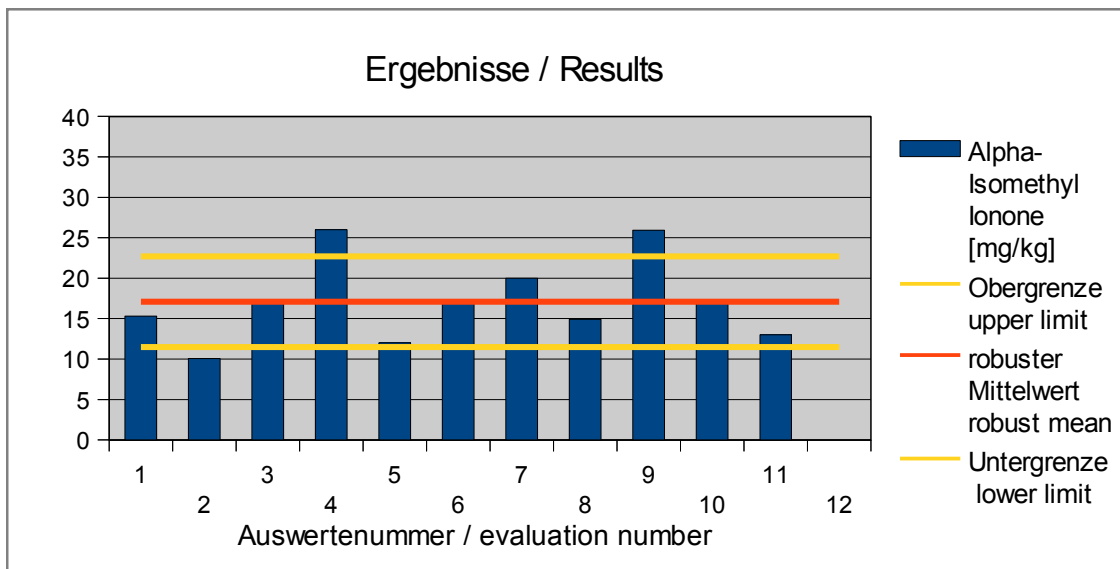
Abb. / Fig. 1: Ergebnisse / Results Alpha-Isomethyl Ionone

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer / Evaluation number	Alpha-Isomethyl Ionone [mg/kg]	Abweichung [mg/kg] / Deviation [mg/kg]	z'-Score (σ_{pt})	Hinweis / Remark
1	15,3	−1,79	−0,64	
2	10,1	−7,01	−2,5	
3	17,0	−0,09	−0,03	
4	26,0	8,91	3,2	
5	12,0	−5,09	−1,8	
6	17,0	−0,09	−0,03	
7	20,0	2,91	1,0	
8	14,9	−2,19	−0,78	
9	25,9	8,84	3,1	
10	17,3	0,21	0,07	
11	13,0	−4,09	−1,5	
12	< 1			
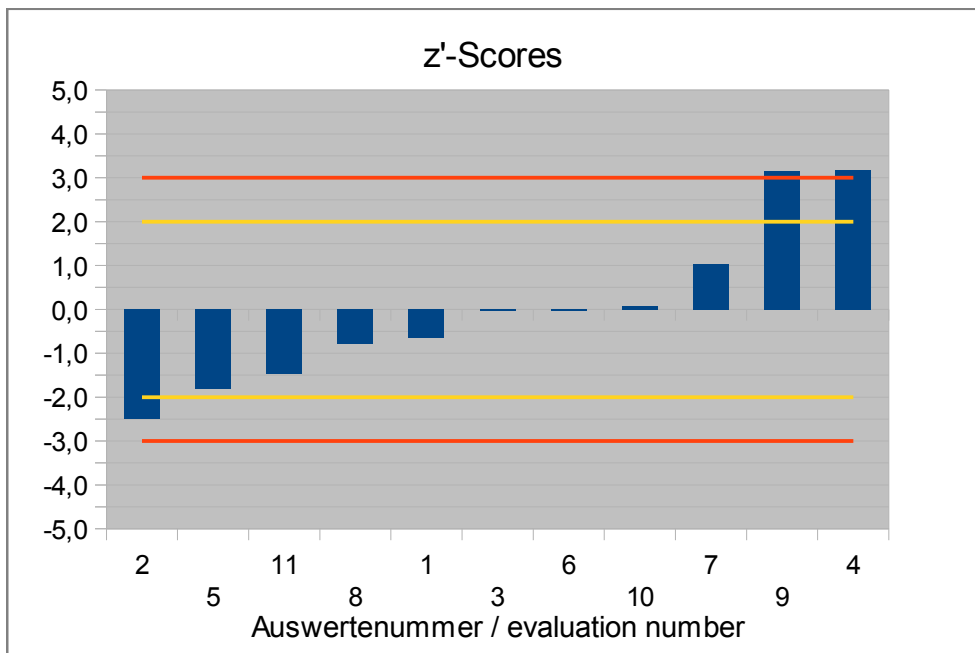
Abb. / Fig. 2:   z'-Scores Alpha-Isomethyl Ionone

4.2 Benzyl Alcohol in mg/kg

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	11
Number of outliers	0
Mean	463
Median	499
Robust Mean (X_{pt})	**464**
Robust standard deviation (S*)	**71,7**
Number with 2 replicates	11
Repeatability SD (S_r)	15,2
Repeatability (CV_r)	3,29%
Reproducibility SD (S_R)	66,4
Reproducibility (CV_R)	14,3%
Target range:	
Target standard deviation σ_{pt}'	**40,0**
lower limit of target range	**384**
upper limit of target range	**544**
Quotient S*/σ_{pt}'	*1,8*
Standard uncertainty U(X_{pt})	*27,0*
Results in the target range	*9*
Percent in the target range	*82%*
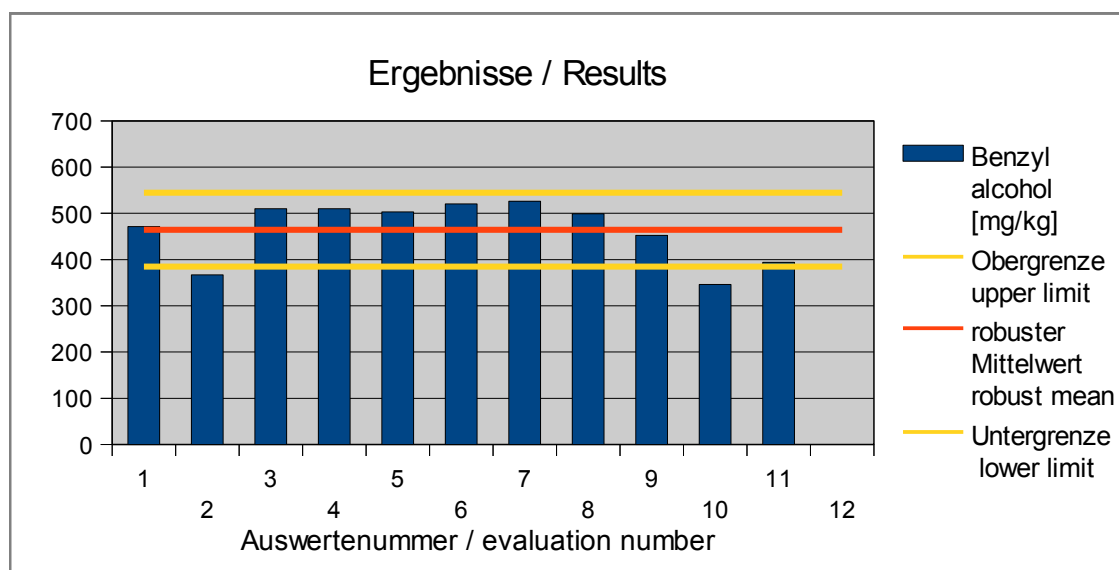
Abb. / Fig. 3: Ergebnisse **/** Results Benzyl alcohol

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte- nummer Evaluation number	Benzyl alcohol [mg/kg]	Abweichung [mg/kg] Deviation [mg/kg]	z'-Score (σ_{pt})	Hinweis Remark
1	471	6,6	0,16	
2	367	−97,8	−2,4	
3	510	45,6	1,1	
4	510	45,6	1,1	
5	503	38,6	1,0	
6	520	55,6	1,4	
7	526	61,6	1,5	
8	499	34,6	0,86	
9	452	−12,0	−0,3	
10	346	−118	−3,0	
11	394	−70,4	−1,8	
12	< 1			
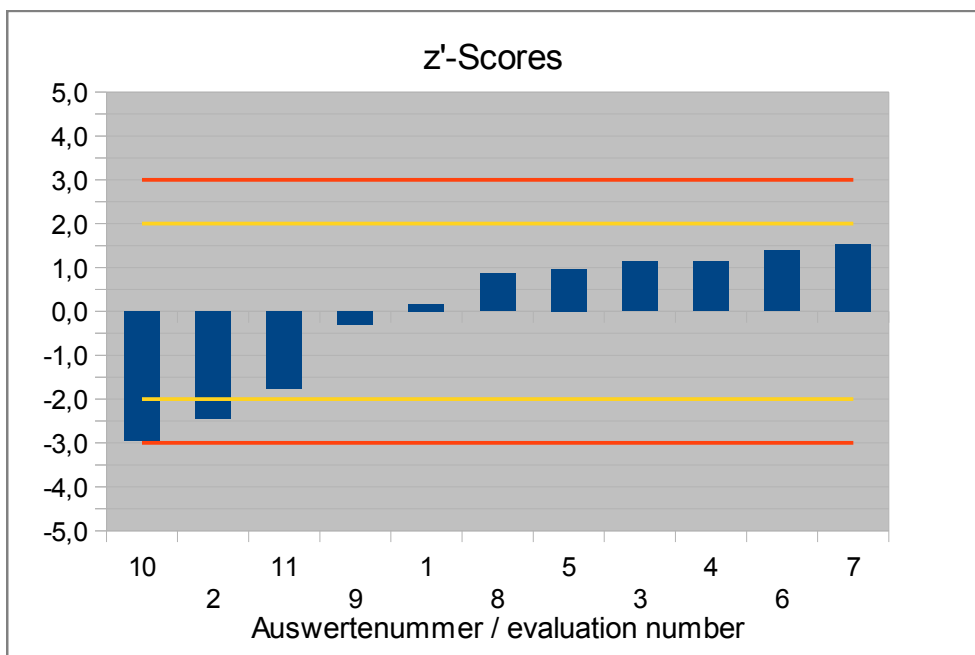
Abb. / Fig. 4:   z'-Scores Benzyl alcohol

4.3 Benzyl Benzoate in mg/kg

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	12
Number of outliers	0
Mean	195
Median	194
Robust Mean (X_{pt})	**198**
Robust standard deviation (S*)	**52,3**
Number with 2 replicates	12
Repeatability SD (S_r)	5,13
Repeatability (CV_r)	2,62%
Reproducibility SD (S_R)	51,2
Reproducibility (CV_R)	26,2%
Target range:	
Target standard deviation σ_{pt}'	**23,7**
lower limit of target range	**151**
upper limit of target range	**245**
Quotient S*/σ_{pt}'	*2,2*
Standard uncertainty $U_{(Xpt)}$	*18,9*
Results in the target range	*8*
Percent in the target range	*67%*
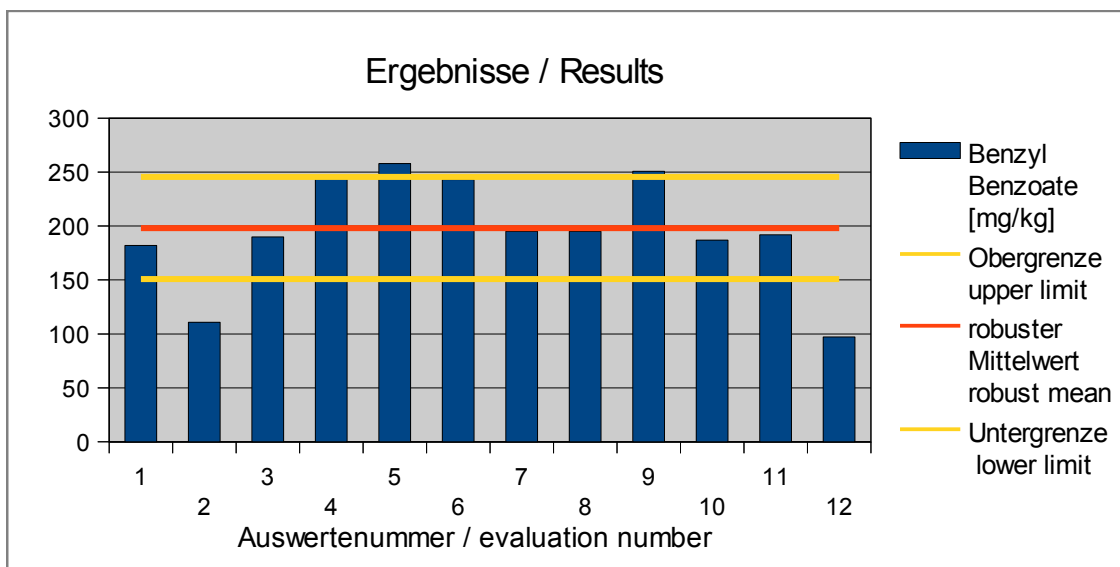
Abb. / Fig. 5: Ergebnisse **/** Results Benzyl Benzoate

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer Evaluation number	Benzyl Benzoate [mg/kg]	Abweichung [mg/kg] Deviation [mg/kg]	z'-Score (σ_{pt})	Hinweis Remark
1	182	−16,1	−0,68	
2	111	−87,3	−3,7	
3	190	−8,1	−0,34	
4	245	46,9	2,0	
5	258	59,9	2,5	
6	243	44,9	1,9	
7	195	−3,1	−0,13	
8	195	−3,1	−0,13	
9	251	52,6	2,2	
10	187	−11,1	−0,47	
11	192	−6,1	−0,26	
12	97,1	−101,0	−4,3	
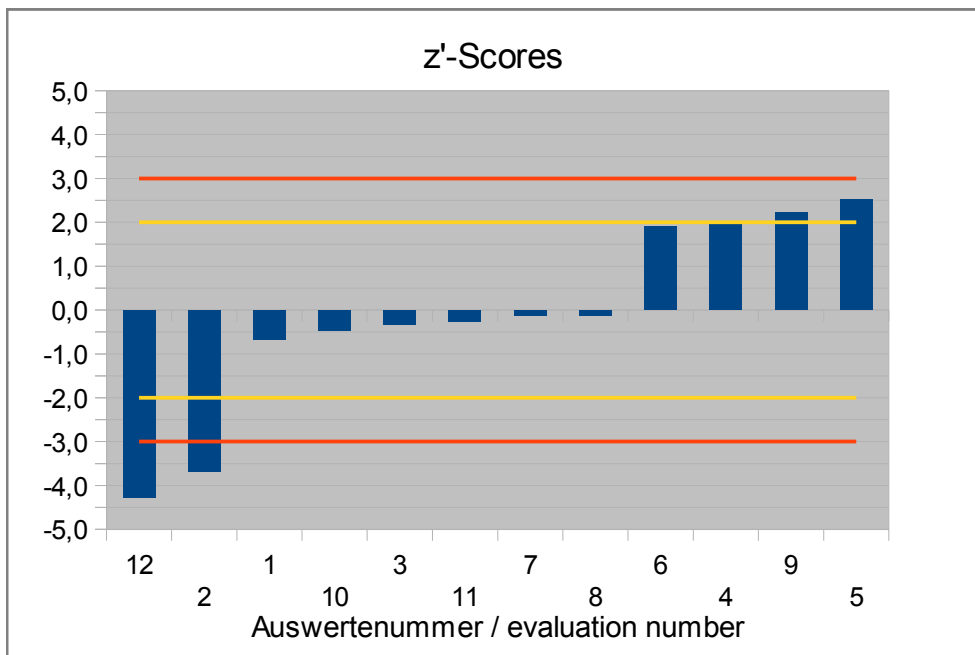
Abb. / Fig. 6:   z'-Scores Benzyl Benzoate

4.4 Benzyl Salicylate in mg/kg

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	10
Number of outliers	0
Mean	104
Median	102
Robust Mean (X_{pt})	**102**
Robust standard deviation (S*)	**14,4**
Number with 2 replicates	10
Repeatability SD (S_r)	5,73
Repeatability (CV_r)	5,51%
Reproducibility SD (S_R)	17,3
Reproducibility (CV_R)	16,6%
Target range:	
Target standard deviation σ_{pt}	**8,15**
lower limit of target range	**86,0**
upper limit of target range	**119**
Quotient S*/σ_{pt}	*1,8*
Standard uncertainty $U_{(X_{pt})}$	*5,70*
Results in the target range	*9*
Percent in the target range	*90%*
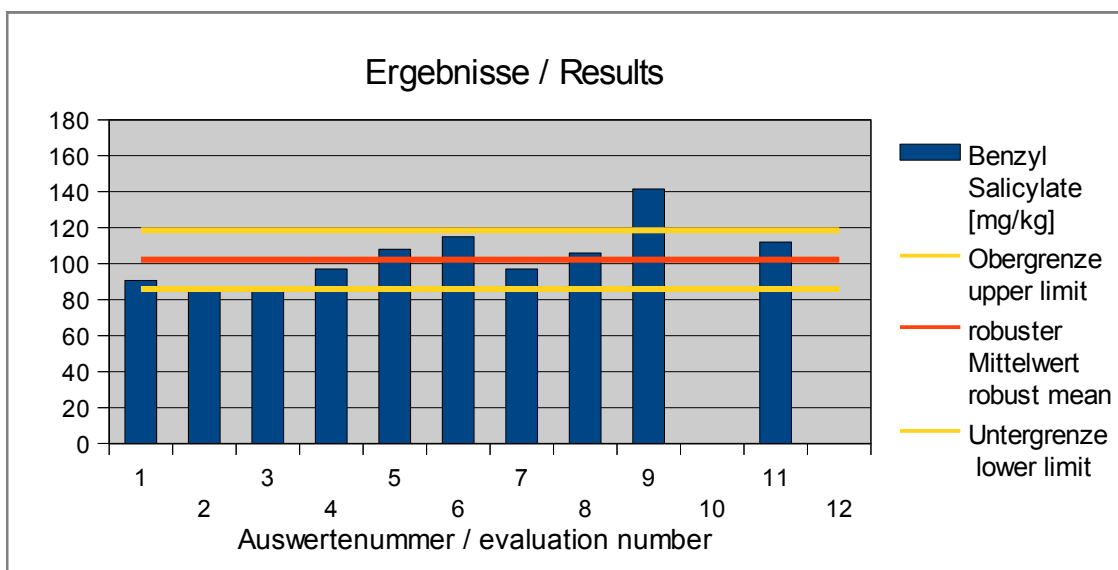
Abb. / Fig. 7: Ergebnisse **/** Results Benzyl Salicylate

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte- nummer Evaluation number	Benzyl Salicylate [mg/kg]	Abweichung [mg/kg] Deviation [mg/kg]	z-Score (σ_{pt})	Hinweis Remark
1	90,7	−11,6	−1,4	
2	87,1	−15,2	−1,9	
3	86,0	−16,3	−2,0	
4	97,0	−5,3	−0,65	
5	108	5,7	0,70	
6	115	12,7	1,6	
7	97,0	−5,3	−0,65	
8	106	3,7	0,46	
9	141	39,2	4,8	
10	< 10			
11	112	9,7	1,2	
12	< 1			
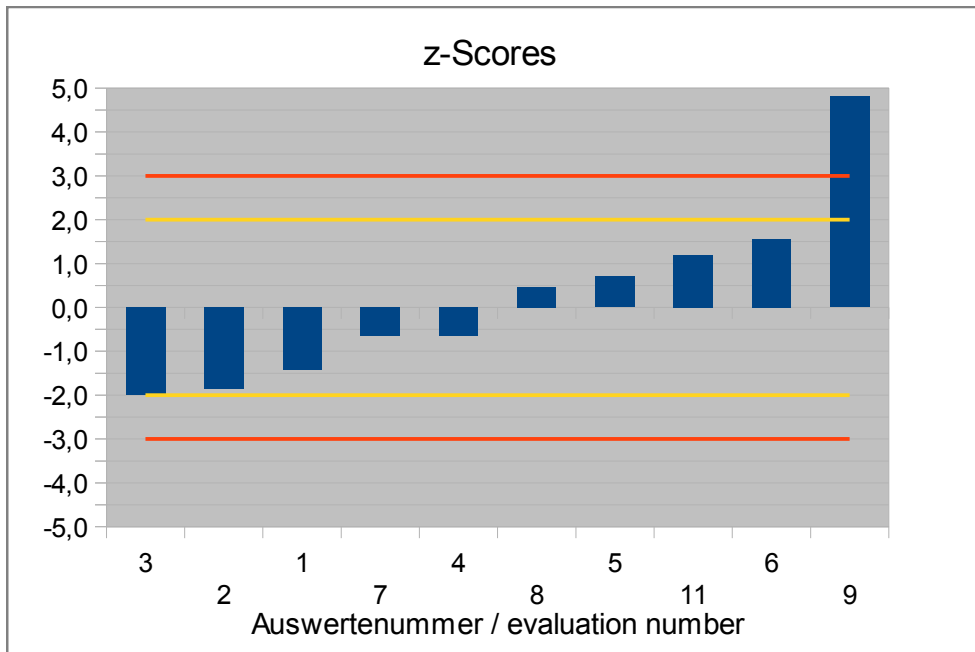
Abb. / Fig. 8:   z-Scores Benzyl Salicylate

4.5 Butylphenyl Methylpropional in mg/kg

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	12
Number of outliers	0
Mean	299
Median	312
Robust Mean (X_{pt})	**306**
Robust standard deviation (S*)	**83,3**
Number with 2 replicates	12
Repeatability SD (S_r)	11,6
Repeatability (CV_r)	3,89%
Reproducibility SD (S_R)	88,0
Reproducibility (CV_R)	29,4%
Target range:	
Target standard deviation $\sigma_{pt'}$	**36,5**
lower limit of target range	**233**
upper limit of target range	**379**
Quotient S*/$\sigma_{pt'}$	*2,3*
Standard uncertainty $U_{(Xpt)}$	*30,1*
Results in the target range	*8*
Percent in the target range	*67%*
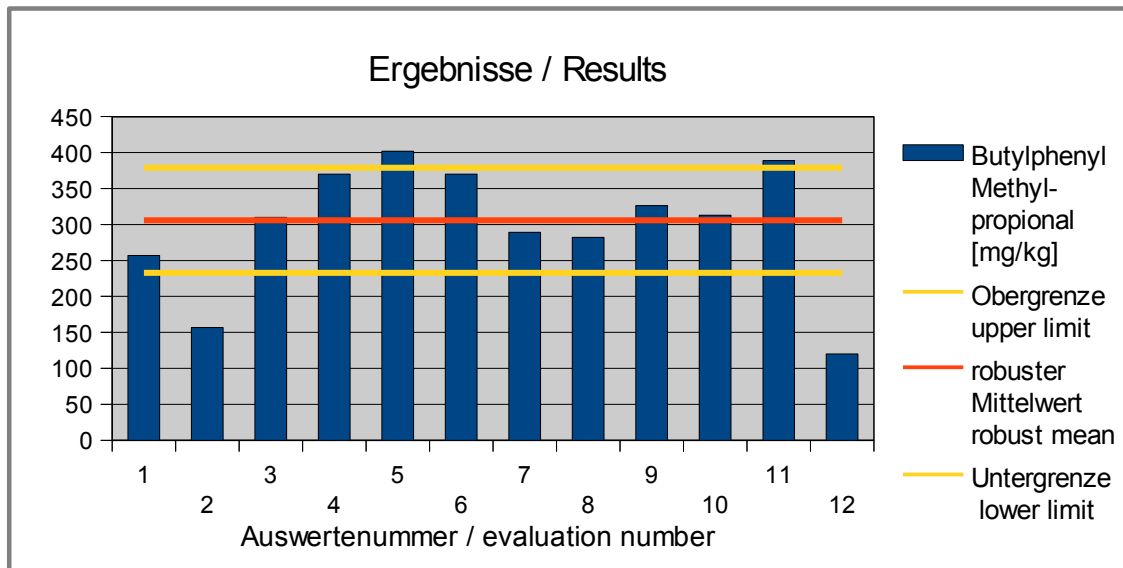
Abb. / Fig. 9: Ergebnisse / Results Butylphenyl Methylpropional

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer Evaluation number	Butylphenyl Methyl-propional [mg/kg]	Abweichung [mg/kg] Deviation [mg/kg]	z'-Score (σ_{pt})	Hinweis Remark
1	257	−48,8	−1,3	
2	157	−149	−4,1	
3	310	4,2	0,11	
4	370	64,2	1,8	
5	402	96,2	2,6	
6	370	64,2	1,8	
7	289	−16,8	−0,46	
8	282	−23,8	−0,65	
9	326	20,3	0,56	
10	313	7,2	0,20	
11	389	83,2	2,3	
12	120	−186	−5,1	
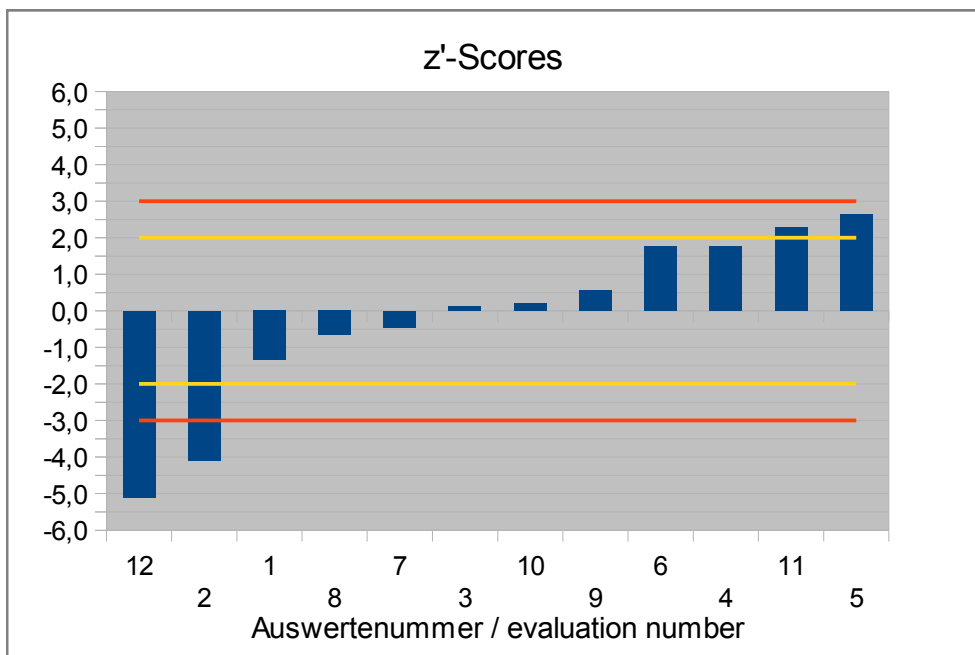
Abb. / Fig. 10: z'-Scores Butylphenyl Methylpropional

4.6 Cinnamal in mg/kg

Vergleichsuntersuchung / **Proficiency Test**

Statistic Data	
Number of results	12
Number of outliers	0
Mean	414
Robust Mean	418
Median (X_{pt})	**447**
Robust standard deviation (S*)	**120**
Number with 2 replicates	12
Repeatability SD (S$_r$)	10,3
Repeatability (CV$_r$)	2,48%
Reproducibility SD (S$_R$)	114
Reproducibility (CV$_R$)	27,40%
Target range:	
Target standard deviation σ_{pt}'	**51,9**
lower limit of target range	**343**
upper limit of target range	**551**
Quotient S*/σ_{pt}'	*2,3*
Standard uncertainty U$_{(Xpt)}$	*43*
Results in the target range	*9*
Percent in the target range	*75%*
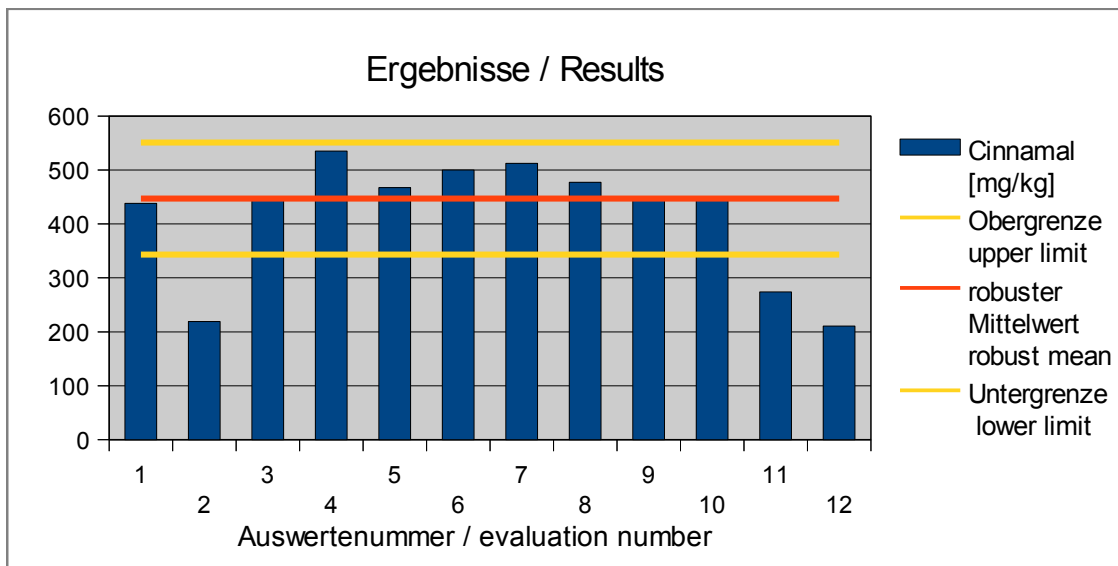
Abb. / Fig. 11: Ergebnisse / Results Cinnamal

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer Evaluation number	Cinnamal [mg/kg]	Abweichung [mg/kg] Deviation [mg/kg]	z'-Score (σ_{pt})	Hinweis Remark
1	438	−9,2	−0,18	
2	219	−228	−4,4	
3	450	2,8	0,05	
4	535	87,8	1,7	
5	467	19,8	0,38	
6	500	52,8	1,0	
7	512	64,8	1,2	
8	477	29,8	0,57	
9	444	−2,8	−0,05	
10	444	−3,2	−0,06	
11	274	−173	−3,3	
12	210	−237	−4,6	
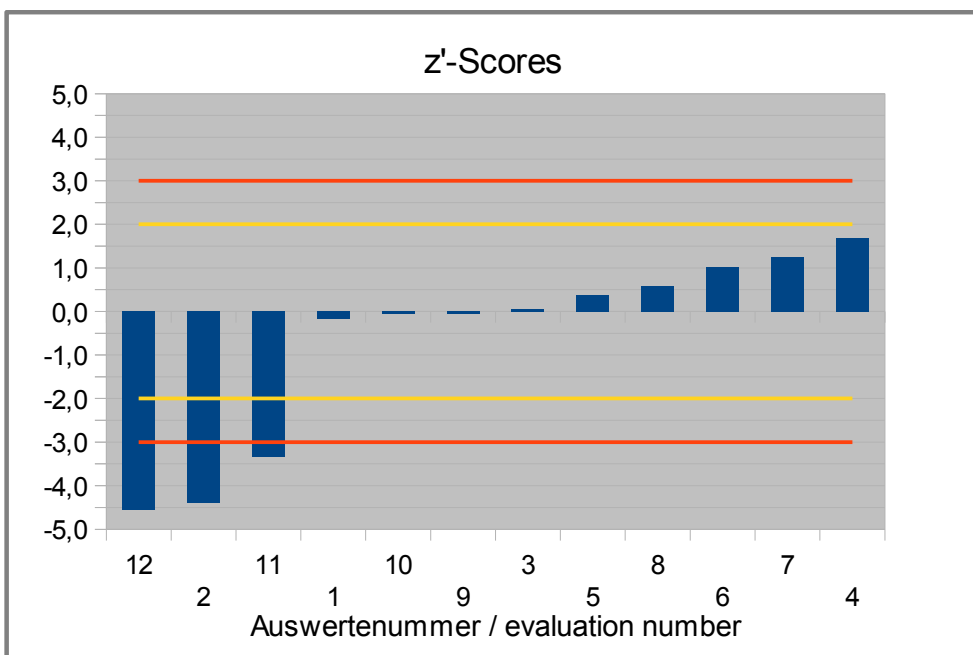
Abb. / Fig. 12: z'-Scores Cinnamal

4.7 Citral in mg/kg

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results*	10
Number of outliers	2
Mean	533
Median	537
Robust Mean (X_{pt})	**531**
Robust standard deviation (S*)	**71,8**
Number with 2 replicates	10
Repeatability SD (S_r)	26,5
Repeatability (CV_r)	4,97%
Reproducibility SD (S_R)	70,0
Reproducibility (CV_R)	13,1%
Target range:	
Target standard deviation $\sigma_{pt'}$	**43,5**
lower limit of target range	**444**
upper limit of target range	**618**
Quotient S*/$\sigma_{pt'}$	*1,6*
Standard uncertainty $U_{(Xpt)}$	*28,4*
Results in the target range	*8*
Percent in the target range	*80%*

° *without outliers (results no. 2 and 12)*
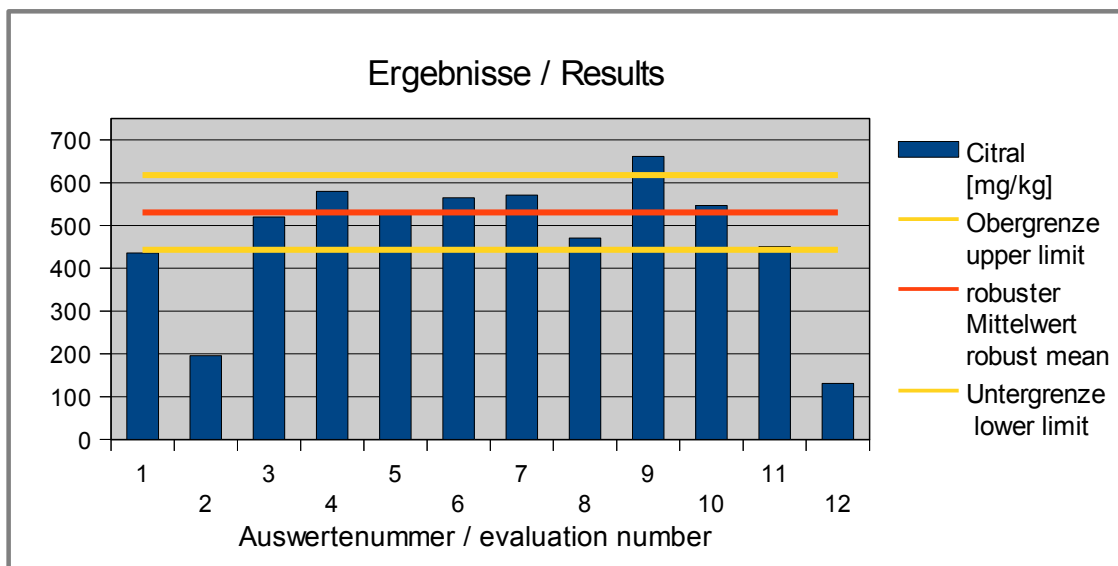
Abb. / Fig. 13: Ergebnisse / Results Citral

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer Evaluation number	Citral [mg/kg]	Abweichung [mg/kg] Deviation [mg/kg]	z'-Score (σ_{pt})	Hinweis Remark
1	436	−94,6	−2,2	
2	196			Ausreißer ausgeschlossen / Outlier excluded
3	520	−10,6	−0,24	
4	580	49,4	1,1	
5	527	−3,6	−0,08	
6	565	34,4	0,79	
7	571	40,4	0,93	
8	471	−59,6	−1,4	
9	662	131,0	3,0	
10	547	16,4	0,38	
11	451	−79,6	−1,8	
12	131			Ausreißer ausgeschlossen / Outlier excluded
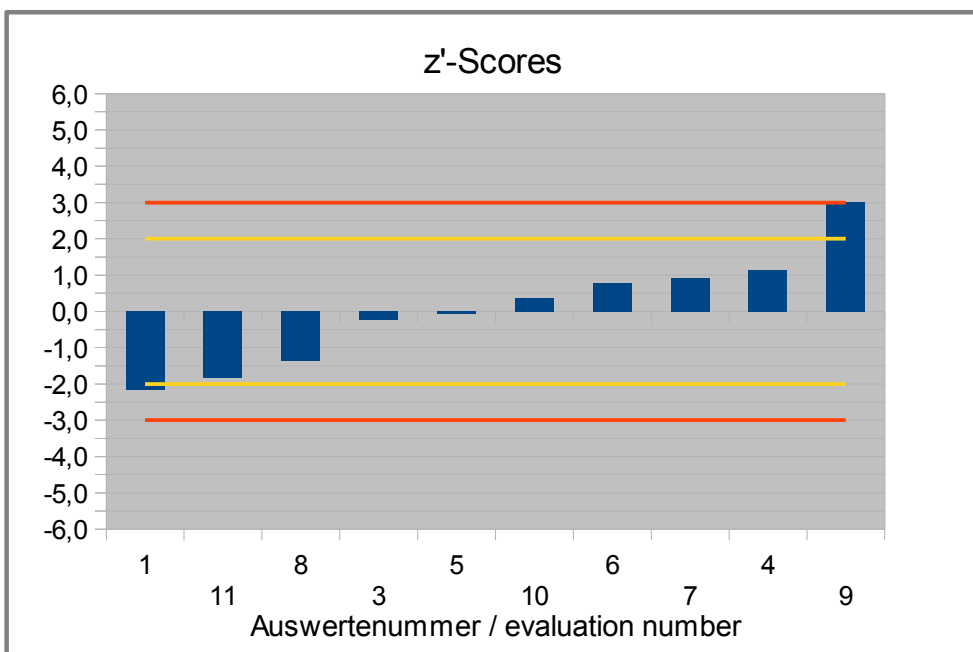
Abb. / Fig. 14: z'-Scores Citral

4.8 Citronellol in mg/kg

Vergleichsuntersuchung / Proficiency Test

Number of results*	8
Number of outliers	2
Mean	177
Median	161
Robust Mean (X_{pt})	**177**
Robust standard deviation (S*)	**46,5**
Number with 2 replicates	8
Repeatability SD (S_r)	7,31
Repeatability (CV_r)	4,12%
Reproducibility SD (S_R)	41,4
Reproducibility (CV_R)	23,3%
Target range:	
Target standard deviation σ_{pt}'	**24,3**
lower limit of target range	**129**
upper limit of target range	**226**
Quotient S*/σ_{pt}'	*1,9*
Standard uncertainty U$_{(Xpt)}$	*20,6*
Results in the target range	*6*
Percent in the target range	*75%*

° without outliers (results no. 2 and 9)
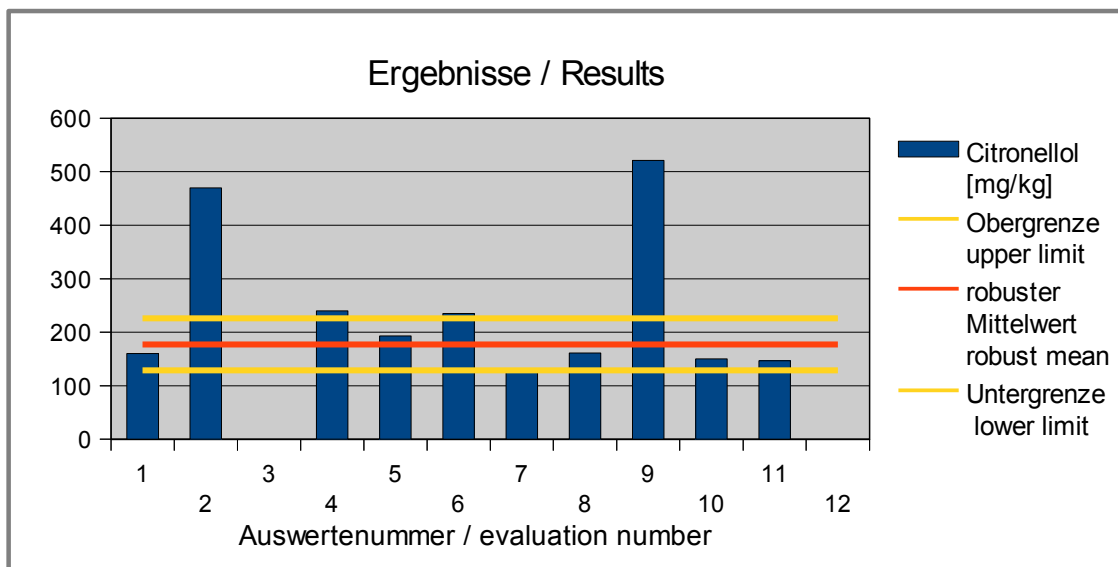
Abb. / Fig. 15: Ergebnisse / Results Citronellol

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer / Evaluation number	Citronellol [mg/kg]	Abweichung [mg/kg] / Deviation [mg/kg]	z'-Score (σpt)	Hinweis / Remark
1	160	−17,3	−0,71	
2	470			Ausreißer ausgeschlossen / Outlier excluded
3				
4	240	62,8	2,6	
5	193	15,8	0,65	
6	235	57,8	2,4	
7	132	−45,3	−1,9	
8	161	−16,3	−0,67	
9	521			Ausreißer ausgeschlossen / Outlier excluded
10	150	−27,3	−1,1	
11	147	−30,3	−1,2	
12	< 1			
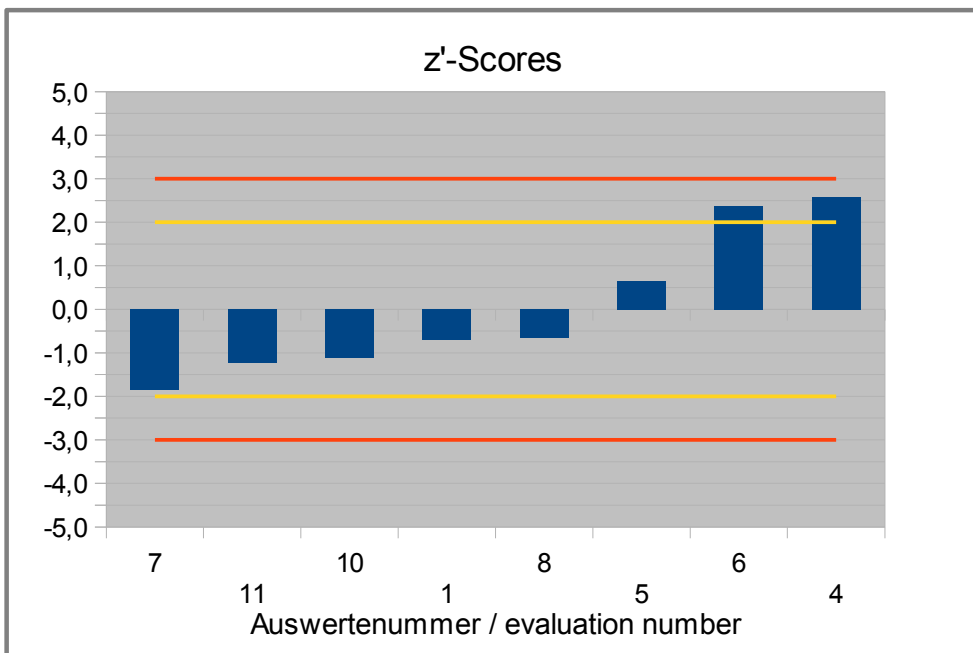
Abb. / Fig. 16: z'-Scores Citronellol

4.9 Coumarin in mg/kg

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	12
Number of outliers	0
Mean	61,7
Median	63,4
Robust Mean (X_{pt})	**63,3**
Robust standard deviation (S*)	**16,2**
Number with 2 replicates	12
Repeatability SD (S_r)	3,18
Repeatability (CV_r)	5,15%
Reproducibility SD (S_R)	18,3
Reproducibility (CV_R)	29,7%
Target range:	
Target standard deviation $\sigma_{pt'}$	**7,98**
lower limit of target range	**47,3**
upper limit of target range	**79,3**
Quotient S*/$\sigma_{pt'}$	*2,0*
Standard uncertainty $U_{(Xpt)}$	*5,86*
Results in the target range	*8*
Percent in the target range	*67%*
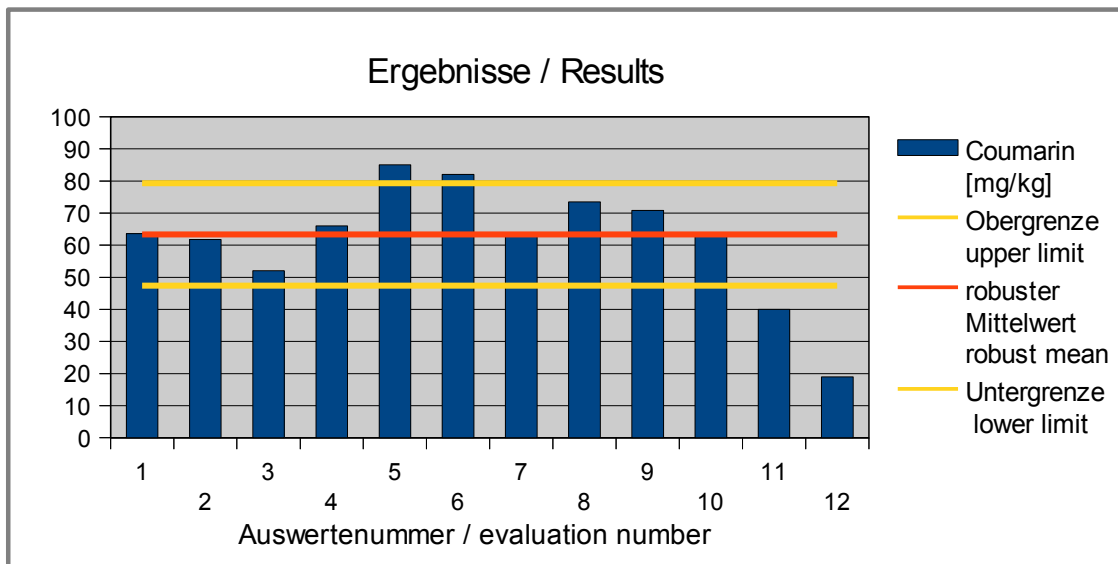
Abb. / Fig. 17: Ergebnisse / Results Coumarin

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer Evaluation number	Coumarin [mg/kg]	Abweichung [mg/kg] Deviation [mg/kg]	z'-Score (σ_{pt})	Hinweis Remark
1	63,6	0,3	0,04	
2	61,7	−1,6	−0,20	
3	52,0	−11,3	−1,4	
4	66,0	2,7	0,34	
5	85,0	21,7	2,7	
6	82,0	18,7	2,3	
7	63,0	−0,3	−0,04	
8	73,5	10,2	1,3	
9	70,8	7,5	0,94	
10	63,2	−0,1	−0,01	
11	40,0	−23,3	−2,9	
12	19,0	−44,3	−5,6	
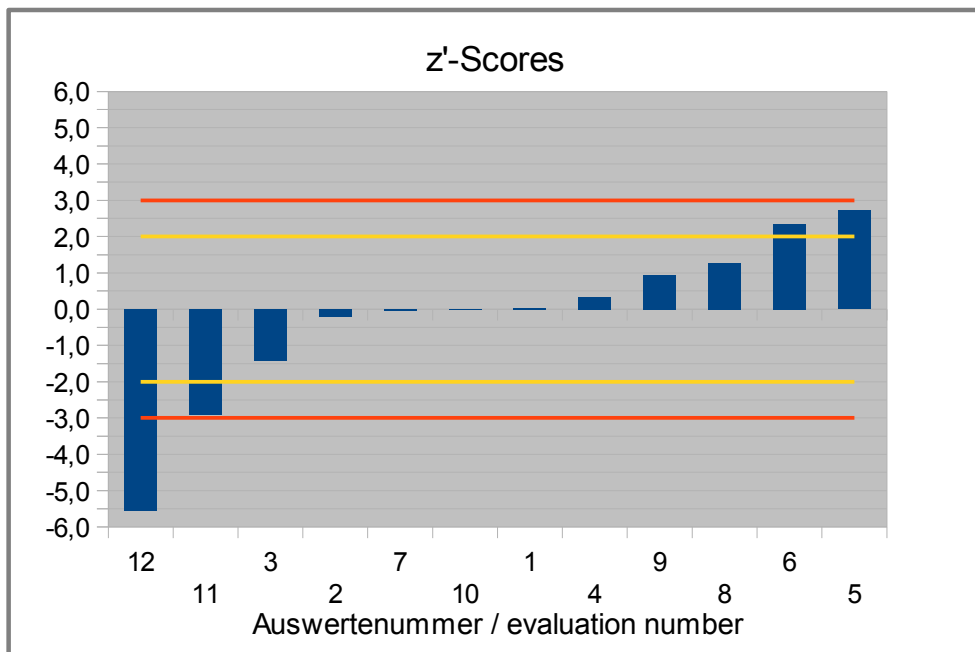
Abb. / Fig. 18: z'-Scores Coumarin

4.10 Eugenol in mg/kg

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	10
Number of outliers	0
Mean	161
Median	160
Robust Mean (X_{pt})	**161**
Robust standard deviation (S*)	**34,0**
Number with 2 replicates	10
Repeatability SD (S_r)	6,17
Repeatability (CV_r)	3,81%
Reproducibility SD (S_R)	29,8
Reproducibility (CV_R)	18,4%
Target range:	
Target standard deviation $\sigma_{pt'}$	**18,0**
lower limit of target range	**125**
upper limit of target range	**197**
Quotient S*/$\sigma_{pt'}$	*1,9*
Standard uncertainty $U_{(Xpt)}$	*13,5*
Results in the target range	*8*
Percent in the target range	*80%*
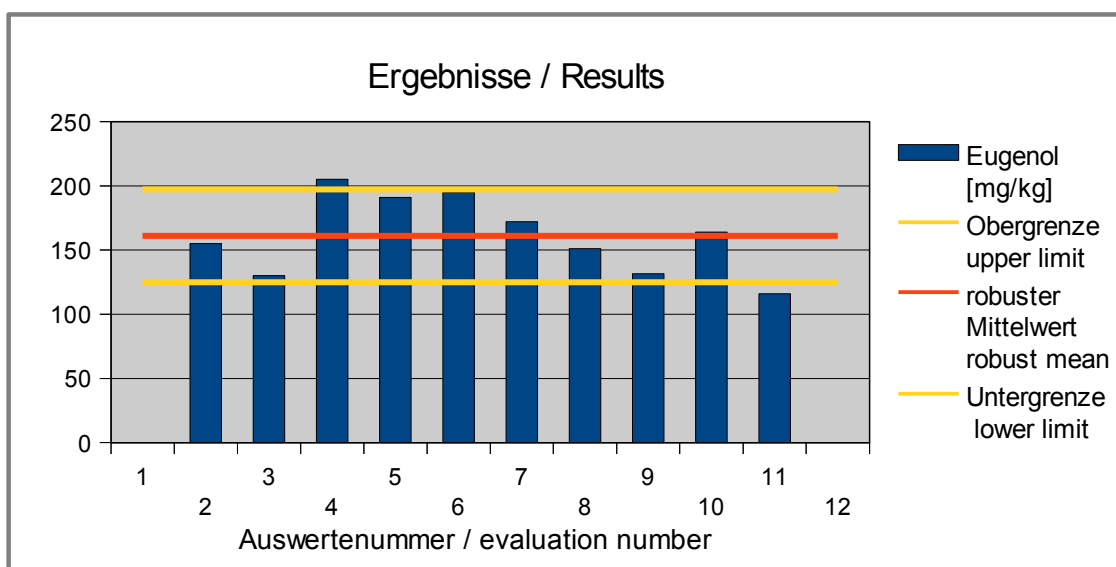
Abb. / Fig. 19: Ergebnisse / Results Eugenol

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer Evaluation number	Eugenol [mg/kg]	Abweichung [mg/kg] Deviation [mg/kg]	z'-Score (σ_{pt})	Hinweis Remark
1	< 5			
2	155	−5,9	−0,33	
3	130	−31,1	−1,7	
4	205	43,9	2,4	
5	191	29,9	1,7	
6	195	33,9	1,9	
7	172	10,9	0,61	
8	151	−10,1	−0,56	
9	131	−29,6	−1,6	
10	164	2,9	0,16	
11	116	−45,1	−2,5	
12	< 1			
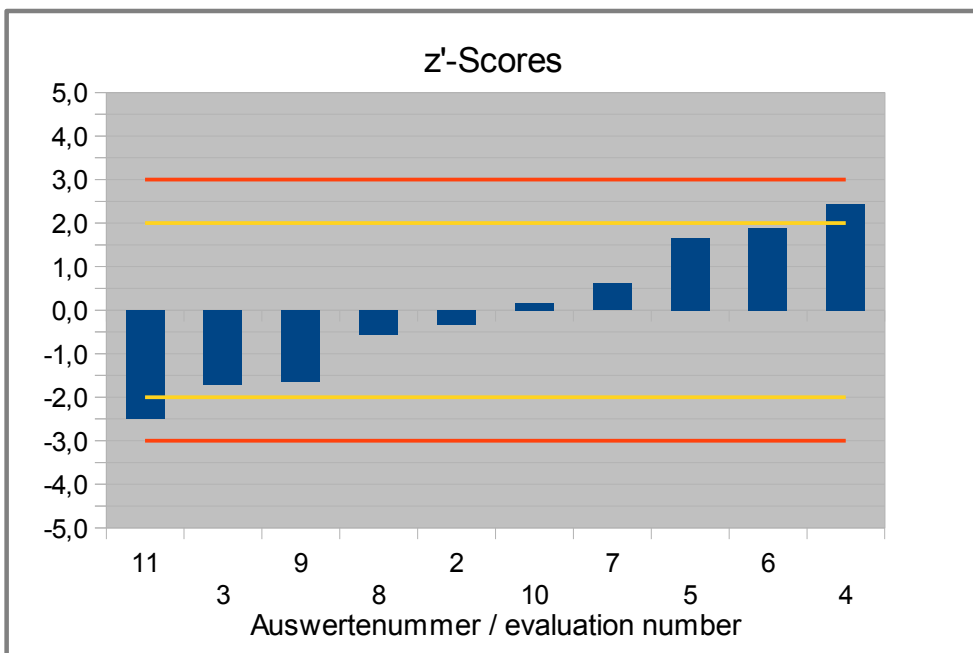
Abb. / Fig. 20:   z'-Scores Eugenol

4.11 Geraniol in mg/kg

Vergleichsuntersuchung / Proficiency Test

| Statistic Data	
Number of results	11
Number of outliers	0
Mean	87,1
Median	79,0
Robust Mean (X_{pt})	**86,2**
Robust standard deviation (S*)	**20,0**
Number with 2 replicates	11
Repeatability SD (S_r)	6,00
Repeatability (CV_r)	6,88%
Reproducibility SD (S_R)	20,0
Reproducibility (CV_R)	22,9%
Target range:	
Target standard deviation $\sigma_{pt'}$	**10,3**
lower limit of target range	**65,6**
upper limit of target range	**107**
Quotient S*/$\sigma_{pt'}$	*1,9*
Standard uncertainty $U_{(Xpt)}$	*7,52*
Results in the target range	*8*
Percent in the target range	*73%*
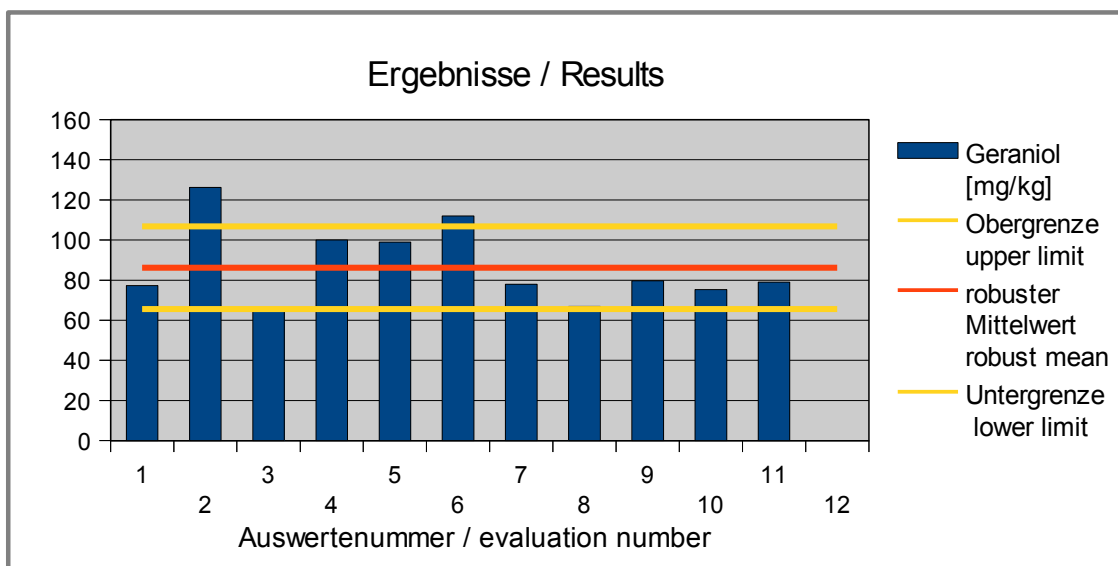
Abb. / Fig. 21: Ergebnisse / Results Geraniol

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer / Evaluation number	Geraniol [mg/kg]	Abweichung [mg/kg] / Deviation [mg/kg]	z'-Score (σ_{pt})	Hinweis / Remark
1	77,3	−8,9	−0,86	
2	126	40,0	3,9	
3	65,0	−21,2	−2,1	
4	100	13,8	1,3	
5	99,0	12,8	1,2	
6	112	25,8	2,5	
7	78,0	−8,2	−0,80	
8	66,9	−19,3	−1,9	
9	79,7	−6,5	−0,63	
10	75,3	−10,9	−1,1	
11	79,0	−7,2	−0,70	
12	< 1			
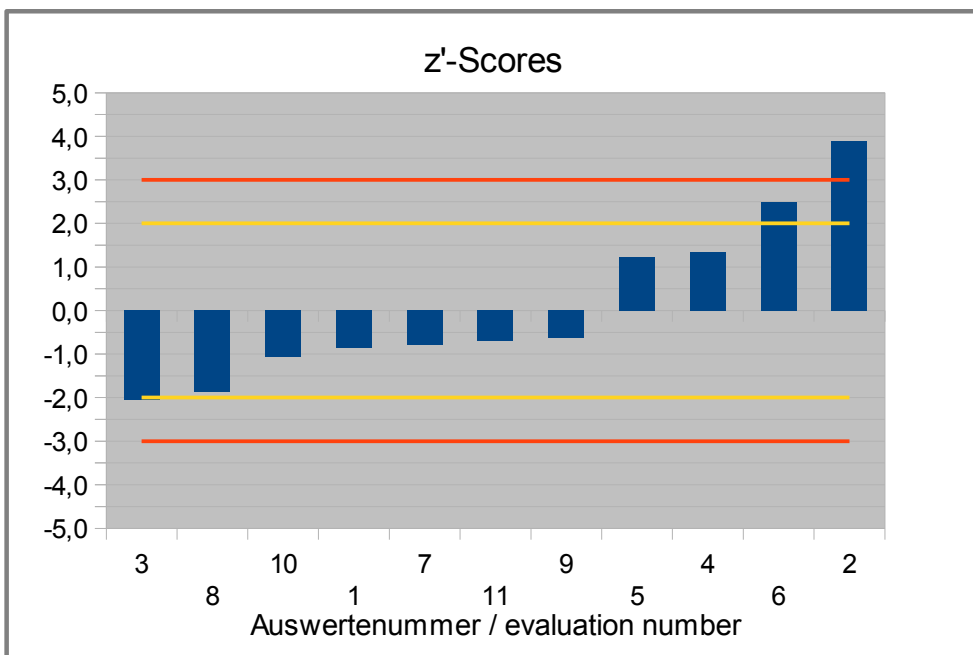
Abb. / Fig. 22: z'-Scores Geraniol

4.12 Hexylcinnamaldehyd in mg/kg

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	12
Number of outliers	0
Mean	97,7
Median	94,5
Robust Mean (X_{pt})	**95,8**
Robust standard deviation (S*)	**37,5**
Number with 2 replicates	12
Repeatability SD (S_r)	4,89
Repeatability (CV_r)	5,00%
Reproducibility SD (S_R)	37,0
Reproducibility (CV_R)	37,9%
Target range:	
Target standard deviation σ_{pt}'	**15,6**
lower limit of target range	**64,7**
upper limit of target range	**127**
Quotient S*/σ_{pt}'	*2,4*
Standard uncertainty $U_{(X_{pt})}$	*13,5*
Results in the target range	*8*
Percent in the target range	*67%*
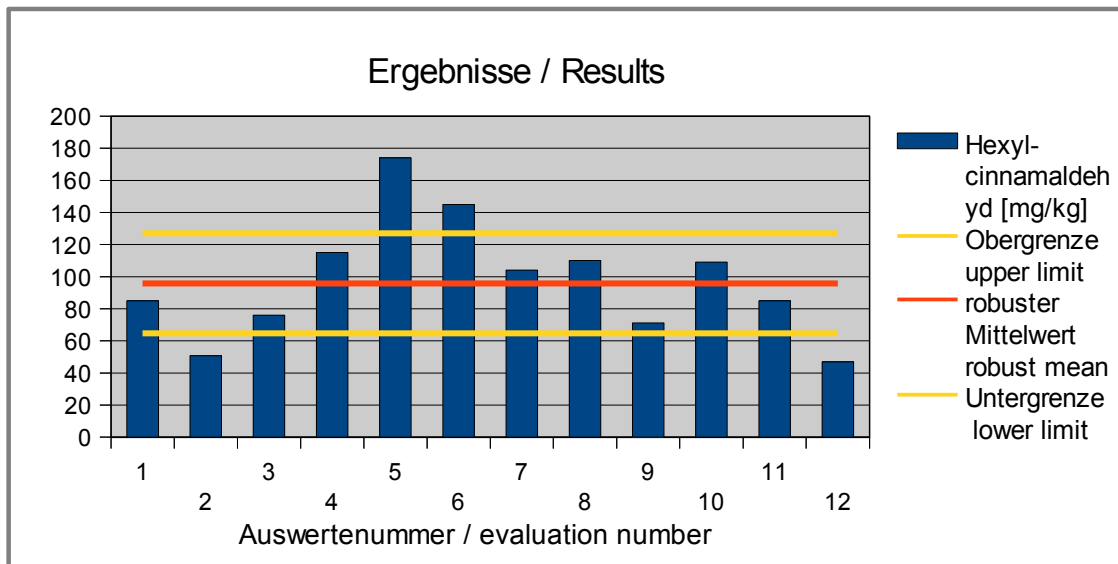
Abb. / Fig. 23: Ergebnisse / Results Hexylcinnamaldehyd

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer Evaluation number	Hexyl-cinnamaldehyd [mg/kg]	Abweichung [mg/kg] Deviation [mg/kg]	z'-Score (σ_{pt})	Hinweis Remark
1	85,0	–10,8	–0,70	
2	50,8	–45,1	–2,9	
3	76,0	–19,8	–1,3	
4	115	19,2	1,2	
5	174	78,2	5,0	
6	145	49,2	3,2	
7	104	8,2	0,52	
8	110	14,2	0,91	
9	71,2	–24,6	–1,6	
10	109	13,2	0,84	
11	85,0	–10,8	–0,70	
12	46,9	–48,9	–3,1	
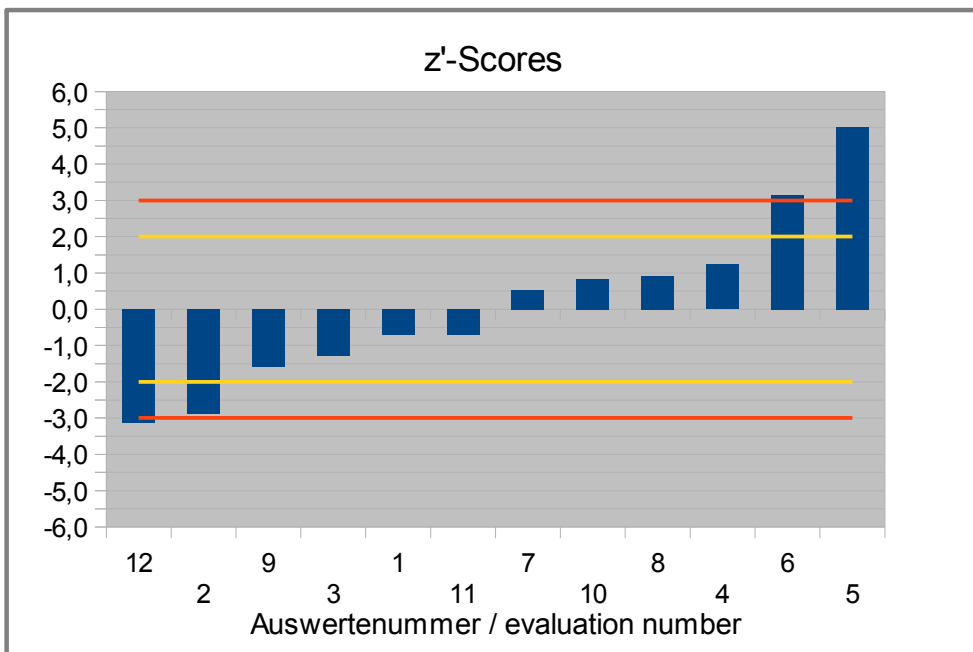
Abb. / Fig. 24: z'-Scores Hexylcinnamaldehyd

4.13 Limonene in mg/kg

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	12
Number of outliers	0
Mean	254
Median	255
Robust Mean (X_{pt})	**250**
Robust standard deviation (S*)	**106**
Number with 2 replicates	12
Repeatability SD (S_r)	8,48
Repeatability (CV_r)	3,34%
Reproducibility SD (S_R)	102
Reproducibility (CV_R)	40,0%
Target range:	
Target standard deviation $\sigma_{pt'}$	**42,2**
lower limit of target range	**166**
upper limit of target range	**335**
Quotient S*/$\sigma_{pt'}$	*2,5*
Standard uncertainty $U_{(Xpt)}$	*38,4*
Results in the target range	*8*
Percent in the target range	*67%*
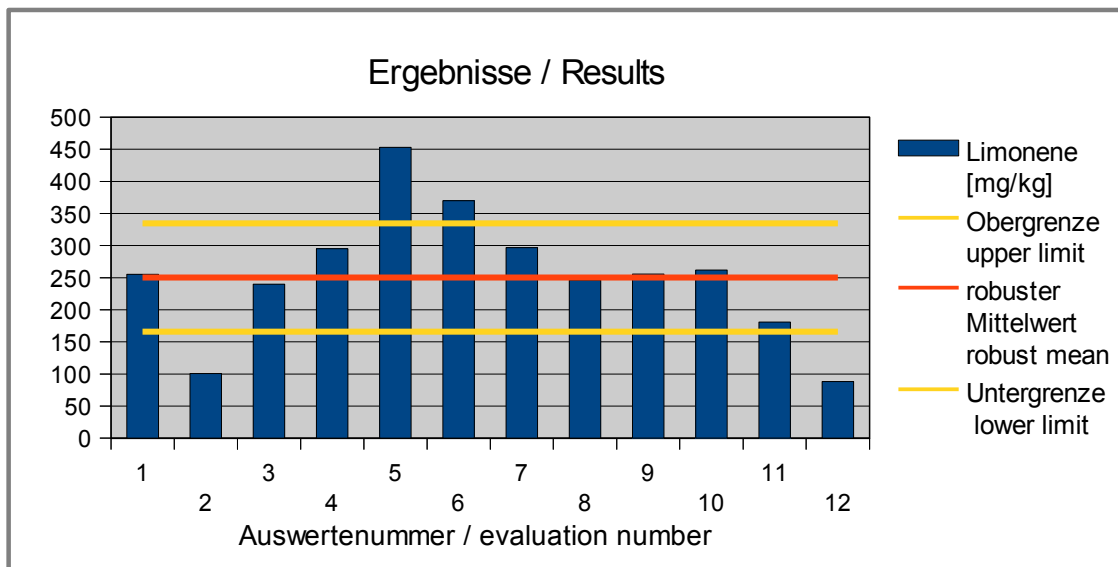
Abb. / Fig. 25: Ergebnisse / Results Limonene

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer / Evaluation number	Limonene [mg/kg]	Abweichung [mg/kg] / Deviation [mg/kg]	z'-Score (σ_{pt})	Hinweis / Remark
1	255	4,7	0,11	
2	101	−149	−3,5	
3	240	−10	−0,24	
4	295	45	1,1	
5	453	203	4,8	
6	370	120	2,8	
7	297	47	1,1	
8	246	−4,3	−0,10	
9	256	5,6	0,13	
10	262	12	0,28	
11	181	−69	−1,6	
12	88,3	−162	−3,8	
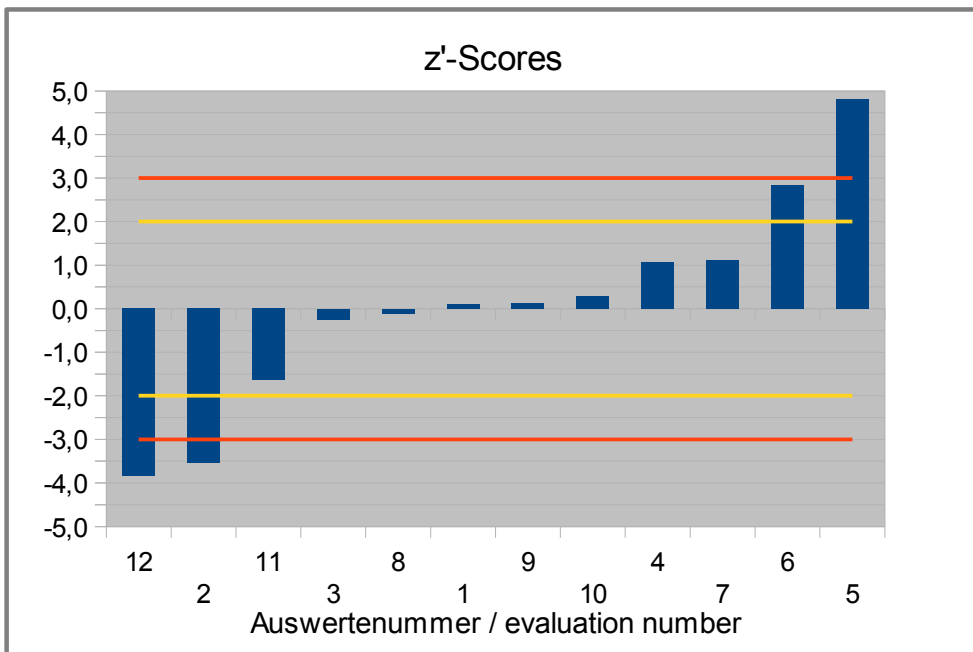
Abb. / Fig. 26: z'-Scores Limonene

4.14 Linalool in mg/kg

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	11
Number of outliers	0
Mean	505
Median	504
Robust Mean (X_{pt})	**509**
Robust standard deviation (S*)	**119**
Number with 2 replicates	11
Repeatability SD (S_r)	14,0
Repeatability (CV_r)	2,77%
Reproducibility SD (S_R)	114
Reproducibility (CV_R)	22,6%
Target range:	
Target standard deviation $\sigma_{pt'}$	**55,0**
lower limit of target range	**399**
upper limit of target range	**619**
Quotient S*/$\sigma_{pt'}$	2,2
Standard uncertainty $U_{(Xpt)}$	44,8
Results in the target range	7
Percent in the target range	64%
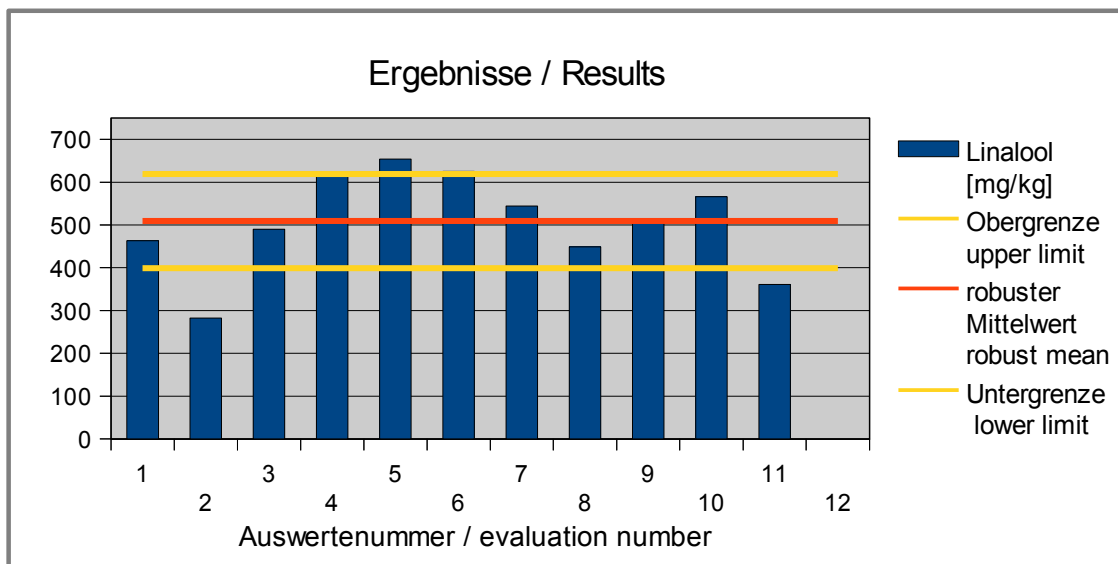
Abb. / Fig. 27: Ergebnisse / Results Linalool

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer / Evaluation number	Linalool [mg/kg]	Abweichung [mg/kg] / Deviation [mg/kg]	z'-Score (σ_{pt})	Hinweis / Remark
1	463	−46	−0,84	
2	283	−227	−4,1	
3	490	−19	−0,35	
4	615	106	1,9	
5	654	145	2,6	
6	625	116	2,1	
7	544	35	0,63	
8	449	−60	−1,1	
9	504	−5,4	−0,1	
10	566	57	1,0	
11	361	−148	−2,7	
12	< 1			
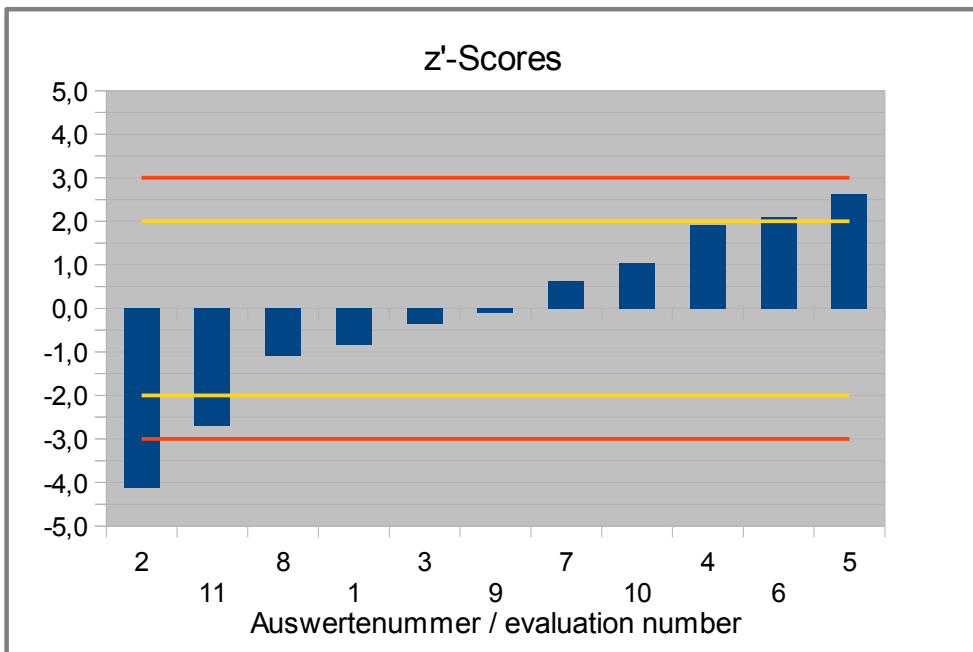
Abb. / Fig. 28: z'-Scores Linalool

5.   Documentation

5.1 Details by the participants

Note: Information given in German were translated by DLA to the best of our knowledge (without guarantee of correctness).

5.1.1 Primary Data

Parameter	Parti-cipant	Ann. III cons. no.	Unit	Sample I DLA No.	Sample II DLA No.	Date of ana-lysis	Result (Mean)	Result Sample I	Result Sample II	Limit of quantifi-cation	Incl. RR	Recovery rate
						day/month					yes/no	in %
Alpha-Isomethyl Ionone	1	90	mg/kg	8	64	17/10	15,3	17,6	13,1	5	yes	
	2	90	mg/kg	13	59	08.-16.18	10,08	9,27	10,89		no	
	3	90	mg/kg	34	38	17.10.18	17	16	17	5	no	
	4	90	mg/kg	31	41	16.10.18	26	26	25	5	yes	Standard solutions for external calibration prepared like samples, recovery in calibration included
	5	90	mg/kg	12	60	27.08.	12	11,4	12,3	10	no	
	6	90	mg/kg	7	65	18.09.18	17	16	18	30	no	88
	7	90	mg/kg	24	48	07.09.18	20	20	20	5 mg/kg	no	102
	8	90	mg/kg	66	6	10.10.18	14,9	14,8	14,9	4	no	
	9	90	mg/kg	4	68	12.09.18	25,93	27,38	24,48	5	no	
	10	90	mg/kg	1	71	22.08.18	17,3	17,5	17,1	10	no	-
	11	90	mg/kg	18	54	16.10.18	13	13	14	10	no	100
	12	90	mg/kg	28	44	24.10.18	<1					

Parameter	Parti-cipant	Ann. III cons. no.	Unit	Sample I DLA No.	Sample II DLA No.	Date of ana-lysis	Result (Mean)	Result Sample I	Result Sample II	Limit of quantifi-cation	Incl. RR	Recovery rate
						day/month					yes/no	in %
Amyl Cinnamal	1	67	mg/kg	8	64	17/10	<5	<5	<5	5	yes	
	2	67	mg/kg	13	59	08.-16.18	4,81	4,15	5,47		no	
	3	67	mg/kg	34	38	17.10.18	<5	<5	<5	5	no	
	4	67	mg/kg	31	41	16.10.18	n.n.	n.n.	n.n.	5	yes	
	5	67	mg/kg	12	60	27.08.	n.n.	n.n.	n.n.	10	no	
	6	67	mg/kg	7	65		n.n.	n.n.	n.n.	23	no	110
	7	67	mg/kg	24	48	07.09.18	< 5	< 5	< 5	5 mg/kg	no	103
	8	67	mg/kg	66	6	10.10.18	n.n.	n.n.	n.n.	6	no	
	9	67	mg/kg	4	68	12.09.18	<BG			5	no	
	10	67	mg/kg	1	71	22.08.18	< 10	< 10	< 10	10	no	-
	11	67	mg/kg	18	54	16.10.18	<LOQ	<LOQ	<LOQ	10	no	100
	12	67	mg/kg	28	44	24.10.18	<1					

Parameter	Parti-cipant	Ann. III cons. no.	Unit	Sample I DLA No.	Sample II DLA No.	Date of ana-lysis	Result (Mean)	Result Sample I	Result Sample II	Limit of quantifi-cation	Incl. RR	Recovery rate
						day/month					yes/no	in %
Amylcinnamyl Alcohol	1	74	mg/kg	8	64	17/10	<5	<5	<5	5	yes	
	2	74	mg/kg	13	59	08.-16.18	-	-	-		no	
	3	74	mg/kg	34	38	17.10.18	<5	<5	<5	5	no	
	4	74	mg/kg	31	41	16.10.18	n.n.	n.n.	n.n.	5	yes	
	5	74	mg/kg	12	60	27.08.	n.n.	n.n.	n.n.	10	no	
	6	74	mg/kg	7	65		n.n.	n.n.	n.n.	23	no	80
	7	74	mg/kg	24	48	07.09.18	< 5	< 5	< 5	5 mg/kg	no	103
	8	74	mg/kg	66	6	10.10.18	n.n.	n.n.	n.n.	6	no	
	9	74	mg/kg	4	68	12.09.18	<BG			5	no	
	10	74	mg/kg	1	71	22.08.18	< 10	< 10	< 10	10	no	-
	11	74	mg/kg	18	54	16.10.18	<LOQ	<LOQ	<LOQ	10	no	100
	12	74	mg/kg	28	44	24.10.18	<1					

Parameter	Parti-cipant	Ann. III cons. no.	Unit	Sample I DLA No.	Sample II DLA No.	Date of ana-lysis	Result (Mean)	Result Sample I	Result Sample II	Limit of quantifi-cation	Incl. RR	Recovery rate
						day/month					yes/no	in %
Anise Alcohol	1	80	mg/kg	8	64	17/10	<5	<5	<5	5	yes	
	2	80	mg/kg	13	59	08.-16.18	-	-	-		no	
	3	80	mg/kg	34	38	17.10.18	<5	<5	<5	5	no	
	4	80	mg/kg	31	41	16.10.18	n.n.	n.n.	n.n.	5	yes	
	5	80	mg/kg	12	60	27.08.	n.n.	n.n.	n.n.	10	no	
	6	80	mg/kg	7	65		n.n.	n.n.	n.n.	22	no	85
	7	80	mg/kg	24	48	07.09.18	< 5	< 5	< 5	5 mg/kg	no	101
	8	80	mg/kg	66	6	10.10.18	n.n.	n.n.	n.n.	11	no	
	9	80	mg/kg	4	68	12.09.18	<BG			5	no	
	10	80	mg/kg	1	71	22.08.18	< 10	< 10	< 10	10	no	-
	11	80	mg/kg	18	54	16.10.18	<LOQ	<LOQ	<LOQ	10	no	100
	12	80	mg/kg	28	44							

Parameter	Parti-cipant	Ann. III cons. no.	Unit	Sample I DLA No.	Sample II DLA No.	Date of ana-lysis	Result (Mean)	Result Sample I	Result Sample II	Limit of quantifi-cation	Incl. RR	Recovery rate
						day/month					yes/no	in %
Benzyl alcohol	1	45	mg/kg	8	64	17/10	471	460	481	5	yes	
	2	45	mg/kg	13	59	08.-16.18	366,64	359,18	374,09		no	
	3	45	mg/kg	34	38	17.10.18	510	510	520	5	no	
	4	45	mg/kg	31	41	16.10.18	510	530	490	5	yes	
	5	45	mg/kg	12	60	10.10.	503	514	493	10	no	
	6	45	mg/kg	7	65		520	510	530	12	no	130
	7	45	mg/kg	24	48	07.09.18	526	545	507	5 mg/kg	no	100
	8	45	mg/kg	66	6	10.10.18	499	494	504	6	no	
	9	45	mg/kg	4	68	12.09.18	452,4	444,87	459,93	5	no	
	10	45	mg/kg	1	71	22.08.18	346	349	343	10	no	-
	11	45	mg/kg	18	54	16.10.18	394	389	399	10	no	100
	12	45	mg/kg	28	44	24.10.18	<1					

Parameter	Parti-cipant	Ann. III cons. no.	Unit	Sample I DLA No.	Sample II DLA No.	Date of ana-lysis	Result (Mean)	Result Sample I	Result Sample II	Limit of quantifi-cation	Incl. RR	Recovery rate
						day/month					yes/no	in %
Benzyl Benzoate	1	85	mg/kg	8	64	17/10	182	178	187	5	yes	
	2	85	mg/kg	13	59	08.-16.18	110,82	105,15	116,5		no	
	3	85	mg/kg	34	38	17.10.18	190	190	190	5	no	
	4	85	mg/kg	31	41	16.10.18	245	240	250	5	yes	
	5	85	mg/kg	12	60	10.10.	258	254	262	10	no	
	6	85	mg/kg	7	65		243	240	245	35	no	101
	7	85	mg/kg	24	48	07.09.18	195	196	195	5 mg/kg	no	99
	8	85	mg/kg	66	6	10.10.18	195	191	199	5	no	
	9	85	mg/kg	4	68	12.09.18	250,66	255,54	245,78	5	no	
	10	85	mg/kg	1	71	22.08.18	187	188	187	10	no	-
	11	85	mg/kg	18	54	16.10.18	192	189	196	10	no	100
	12	85	mg/kg	28	44	24.10.18	97,12	94,73	99,52	1		78

Parameter	Parti-cipant	Ann. III cons. no.	Unit	Sample I DLA No.	Sample II DLA No.	Date of ana-lysis	Result (Mean)	Result Sample I	Result Sample II	Limit of quantifi-cation	Incl. RR	Recovery rate
						day/month					yes/no	in %
Benzyl Cinnamate	1	81	mg/kg	8	64	17/10	<5	<5	<5	5	yes	
	2	81	mg/kg	13	59	08.-16.18	-	-	-		no	
	3	81	mg/kg	34	38	17.10.18	<5	<5	<5	5	no	
	4	81	mg/kg	31	41	16.10.18	n.n.	n.n.	n.n.	5	yes	
	5	81	mg/kg	12	60	10.10.	n.n.	n.n.	n.n.	10	no	
	6	81	mg/kg	7	65		n.n.	n.n.	n.n.	42	no	85
	7	81	mg/kg	24	48	07.09.18	< 5	< 5	< 5	5 mg/kg	no	107
	8	81	mg/kg	66	6	10.10.18	n.n.	n.n.	n.n.	7	no	
	9	81	mg/kg	4	68	12.09.18	<BG			5	no	
	10	81	mg/kg	1	71	22.08.18	< 10	< 10	< 10	10	no	-
	11	81	mg/kg	18	54	16.10.18	<LOQ	<LOQ	<LOQ	10	no	100
	12	81	mg/kg	28	44	24.10.18	<1			1		

Parameter	Parti-cipant	Ann. III cons. no.	Unit	Sample I DLA No.	Sample II DLA No.	Date of ana-lysis	Result (Mean)	Result Sample I	Result Sample II	Limit of quantifi-cation	Incl. RR	Recovery rate
						day/month					yes/no	in %
Benzyl Salicylate	1	75	mg/kg	8	64	17/10	90,7	91,2	90,2	5	yes	
	2	75	mg/kg	13	59	08.-16.18	87,07	85,38	88,77		no	
	3	75	mg/kg	34	38	17.10.18	86	84	87	5	no	
	4	75	mg/kg	31	41	16.10.18	97	94	100	5	yes	
	5	75	mg/kg	12	60	27.08.	108	112	105	10	no	
	6	75	mg/kg	7	65		115	120	110	62	no	110
	7	75	mg/kg	24	48	07.09.18	97	97	96	5 mg/kg	no	200
	8	75	mg/kg	66	6	10.10.18	106	104	108	8	no	
	9	75	mg/kg	4	68	12.09.18	141,47	150,85	132,09	5	no	
	10	75	mg/kg	1	71	22.08.18	< 10	< 10	< 10	10	no	-
	11	75	mg/kg	18	54	16.10.18	112	117	108	10	no	100
	12	75	mg/kg	28	44	24.10.18	<1			1		

Parameter	Parti-cipant	Ann. III cons. no.	Unit	Sample I DLA No.	Sample II DLA No.	Date of ana-lysis	Result (Mean)	Result Sample I	Result Sample II	Limit of quantifi-cation	Incl. RR	Recovery rate
						day/month					yes/no	in %
Butylphenyl Methylpropional	1	83	mg/kg	8	64	17/10	257	250	263	5	yes	
	2	83	mg/kg	13	59	08.-16.18	156,68	153,13	160,22		no	
	3	83	mg/kg	34	38	17.10.18	310	310	320	5	no	
	4	83	mg/kg	31	41	16.10.18	370	360	380	5	yes	
	5	83	mg/kg	12	60	10.10.	402	400	404	10	no	
	6	83	mg/kg	7	65		370	360	380	42	no	88
	7	83	mg/kg	24	48	07.09.18	289	285	294	5 mg/kg	no	100
	8	83	mg/kg	66	6	10.10.18	282	277	287	6	no	
	9	83	mg/kg	4	68	12.09.18	326,12	347,2	305,04	5	no	
	10	83	mg/kg	1	71	22.08.18	313	317	310	10	no	-
	11	83	mg/kg	18	54	16.10.18	389	392	385	10	no	100
	12	83	mg/kg	28	44	24.10.18	119,85	116	123,7	1		92

Parameter	Parti-cipant	Ann. III cons. no.	Unit	Sample I DLA No.	Sample II DLA No.	Date of ana-lysis	Result (Mean)	Result Sample I	Result Sample II	Limit of quantifi-cation	Incl. RR	Recovery rate
						day/month					yes/no	in %
Cinnamal	1	76	mg/kg	8	64	17/10	438	429	447	5	yes	
	2	76	mg/kg	13	59	08.-16.18	219,29	219,9	218,68		no	
	3	76	mg/kg	34	38	17.10.18	450	440	460	5	no	
	4	76	mg/kg	31	41	16.10.18	535	530	540	5	yes	
	5	76	mg/kg	12	60	10.10.	467	478	457	10	no	
	6	76	mg/kg	7	65		500	510	490	22	no	105
	7	76	mg/kg	24	48	07.09.18	512	520	505	5 mg/kg	no	101
	8	76	mg/kg	66	6	10.10.18	477	469	485	7	no	
	9	76	mg/kg	4	68	12.09.18	444,37	451,08	437,67	5	no	
	10	76	mg/kg	1	71	22.08.18	444	449	439	10	no	-
	11	76	mg/kg	18	54	16.10.18	274	275	273	10	no	100
	12	76	mg/kg	28	44	24.10.18	210,45	205,6	215,3	1		84

Parameter	Parti-cipant	Ann. III cons. no.	Unit	Sample I DLA No.	Sample II DLA No.	Date of ana-lysis	Result (Mean)	Result Sample I	Result Sample II	Limit of quantifi-cation	Incl. RR	Recovery rate
						day/month					yes/no	in %
Cinnamyl alcohol	1	69	mg/kg	8	64	17/10	<5	<5	<5	5	yes	
	2	69	mg/kg	13	59	08.-16.18	11,97	12,14	11,79		no	
	3	69	mg/kg	34	38	17.10.18	<5	<5	<5	5	no	
	4	69	mg/kg	31	41	16.10.18	n.n.	n.n.	n.n.	5	yes	
	5	69	mg/kg	12	60	10.10.	not evaluable	not evaluable	not evaluable	10	no	
	6	69	mg/kg	7	65		n.n.	n.n.	n.n.	70	no	105
	7	69	mg/kg	24	48	07.09.18	< 5	< 5	< 5	5 mg/kg	no	99
	8	69	mg/kg	66	6	10.10.18	n.n.	n.n.	n.n.	12	no	
	9	69	mg/kg	4	68	12.09.18	<BG			5	no	
	10	69	mg/kg	1	71	22.08.18	< 10	< 10	< 10	10	no	-
	11	69	mg/kg	18	54	16.10.18	<LOQ	<LOQ	<LOQ	10	no	100
	12	69	mg/kg	28	44	24.10.18	<1					

Parameter	Parti-cipant	Ann. III cons. no.	Unit	Sample I DLA No.	Sample II DLA No.	Date of ana-lysis	Result (Mean)	Result Sample I	Result Sample II	Limit of quantifi-cation	Incl. RR	Recovery rate
						day/month					yes/no	in %
Citral	1	70	mg/kg	8	64	17/10	436	427	446	5	yes	
	2	70	mg/kg	13	59	08.-16.18	195,77	194,35	197,19		no	
	3	70	mg/kg	34	38	17.10.18	520	510	530	5	no	
	4	70	mg/kg	31	41	16.10.18	580	570	590	5	yes	
	5	70	mg/kg	12	60	10.10.	527	578	477	10	no	
	6	70	mg/kg	7	65		565	570	560	14	no	121
	7	70	mg/kg	24	48	07.09.18	571	567	576	5 mg/kg	no	101
	8	70	mg/kg	66	6	10.10.18	471	457	494	6	no	
	9	70	mg/kg	4	68	12.09.18	661,64	659,3	663,98	5	no	
	10	70	mg/kg	1	71	22.08.18	547	552	542	10	no	-
	11	70	mg/kg	18	54	16.10.18	451	467	435	10	no	100
	12	70	mg/kg	28	44	24.10.18	131,19	133,65	128,74	1		79

Parameter	Parti-cipant	Ann. III cons. no.	Unit	Sample I DLA No.	Sample II DLA No.	Date of ana-lysis	Result (Mean)	Result Sample I	Result Sample II	Limit of quantifi-cation	Incl. RR	Recovery rate
						day/month					yes/no	in %
Citronellol	1	86	mg/kg	8	64	17/10	160	153	167	5	yes	
	2	86	mg/kg	13	59	08.-16.18	470,48	465,55	475,4		no	
	3	86	mg/kg	34	38	17.10.18	-	-	-	5	no	
	4	86	mg/kg	31	41	16.10.18	240	230	250	5	yes	
	5	86	mg/kg	12	60	10.10.	193	189	198	10	no	
	6	86	mg/kg	7	65		235	230	240	24	no	108
	7	86	mg/kg	24	48	07.09.18	132	136	128	5 mg/kg	no	105
	8	86	mg/kg	66	6	10.10.18	161	159	162	6	no	
	9	86	mg/kg	4	68	12.09.18	521,18	528,3	514,07	5	no	
	10	86	mg/kg	1	71	22.08.18	150	150	151	10	no	-
	11	86	mg/kg	18	54	16.10.18	147	146	148	10	no	100
	12	86	mg/kg	28	44	24.10.18	<1					

Parameter	Parti-cipant	Ann. III cons. no.	Unit	Sample I DLA No.	Sample II DLA No.	Date of ana-lysis	Result (Mean)	Result Sample I	Result Sample II	Limit of quantifi-cation	Incl. RR	Recovery rate
						day/month					yes/no	in %
Coumarin	1	77	mg/kg	8	64	17/10	63,6	63,3	64	5	yes	
	2	77	mg/kg	13	59	08.-16.18	61,73	61,15	62,3		no	
	3	77	mg/kg	34	38	17.10.18	52	51	53	5	no	
	4	77	mg/kg	31	41	16.10.18	66	67	65	5	yes	
	5	77	mg/kg	12	60	27.08.	85	88	83	10	no	
	6	77	mg/kg	7	65		82	80	84	24	no	112
	7	77	mg/kg	24	48	07.09.18	63	64	63	5 mg/kg	no	97
	8	77	mg/kg	66	6	10.10.18	73,5	73	74	8	no	
	9	77	mg/kg	4	68	12.09.18	70,8	77,44	64,17	5	no	
	10	77	mg/kg	1	71	22.08.18	63,2	64,2	62,2	10	no	-
	11	77	mg/kg	18	54	16.10.18	40	38	41	10	no	100
	12	77	mg/kg	28	44	24.10.18	18,99	18,73	19,25	1		77

Parameter	Parti-cipant	Ann. III cons. no.	Unit	Sample I DLA No.	Sample II DLA No.	Date of ana-lysis	Result (Mean)	Result Sample I	Result Sample II	Limit of quantifi-cation	Incl. RR	Recovery rate
						day/month					yes/no	in %
Eugenol	1	71	mg/kg	8	64	17/10	<5	<5	<5	5	yes	
	2	71	mg/kg	13	59	08.-16.18	155,15	146,27	164,03		no	
	3	71	mg/kg	34	38	17.10.18	130	130	140	5	no	
	4	71	mg/kg	31	41	16.10.18	205	200	210	5	yes	
	5	71	mg/kg	12	60	27.08.	191	187	196	10	no	
	6	71	mg/kg	7	65		195	200	190	29	no	105
	7	71	mg/kg	24	48	07.09.18	172	174	171	5 mg/kg	no	109
	8	71	mg/kg	66	6	10.10.18	151	149	152	6	no	
	9	71	mg/kg	4	68	12.09.18	131,49	133,73	129,25	5	no	
	10	71	mg/kg	1	71	22.08.18	164	165	164	10	no	-
	11	71	mg/kg	18	54	16.10.18	116	114	119	10	no	100
	12	71	mg/kg	28	44	24.10.18	<1					

Parameter	Parti-cipant	Ann. III cons. no.	Unit	Sample I DLA No.	Sample II DLA No.	Date of ana-lysis	Result (Mean)	Result Sample I	Result Sample II	Limit of quantifi-cation	Incl. RR	Recovery rate
						day/month					yes/no	in %
Evernia Furfuracea Extract	1	92	mg/kg	8	64	17/10	<5	<5	<5	5	yes	
	2	92	mg/kg	13	59	08.-16.18	-	-	-		no	
	3	92	mg/kg	34	38	17.10.18	negative	negative	negative		no	
	4	92	mg/kg	31	41							
	5	92	mg/kg	12	60							
	6	92	mg/kg	7	65		n.d.				no	
	7	92	mg/kg	24	48	07.09.18	n.d.	n.d.	n.d.		no	/
	8	92	mg/kg	66	6	10.10.18	n.d.	n.d.	n.d.		no	
	9	92	mg/kg	4	68	12.09.18	<LOQ			5	no	
	10	92	mg/kg	1	71		-					
	11	92	mg/kg	18	54		n.d.	n.d.	n.d.			
	12	92	mg/kg	28	44							

Parameter	Parti-cipant	Ann. III cons. no.	Unit	Sample I DLA No.	Sample II DLA No.	Date of ana-lysis	Result (Mean)	Result Sample I	Result Sample II	Limit of quantifi-cation	Incl. RR	Recovery rate
						day/month					yes/no	in %
Evernia Prunastri Extract	1	91	mg/kg	8	64	17/10	<5	<5	<5	5	yes	
	2	91	mg/kg	13	59	08.-16.18	-	-	-		no	
	3	91	mg/kg	34	38	17.10.18	negative	negative	negative		no	
	4	91	mg/kg	31	41							
	5	91	mg/kg	12	60							
	6	91	mg/kg	7	65		n.d.				no	
	7	91	mg/kg	24	48	07.09.18	n.d.	n.d.	n.d.		no	/
	8	91	mg/kg	66	6	10.10.18	n.d.	n.d.	n.d.		no	
	9	91	mg/kg	4	68	12.09.18	<LOQ			5	no	
	10	91	mg/kg	1	71		-					
	11	91	mg/kg	18	54		n.d.	n.d.	n.d.			
	12	91	mg/kg	28	44							

Parameter	Parti-cipant	Ann. III cons. no.	Unit	Sample I DLA No.	Sample II DLA No.	Date of ana-lysis	Result (Mean)	Result Sample I	Result Sample II	Limit of quantifi-cation	Incl. RR	Recovery rate
						day/month					yes/no	in %
Farnesol	1	82	mg/kg	8	64	17/10	28,7	24,1	33,4	5	yes	
	2	82	mg/kg	13	59	08.-16.18	59,11	54,75	63,47		no	
	3	82	mg/kg	34	38	17.10.18	<5	<5	<5	5	no	
	4	82	mg/kg	31	41	16.10.18	110	115	105	10	yes	
	5	82	mg/kg	12	60	10.10.	not evaluable	not evaluable	not evaluable	100	no	
	6	82	mg/kg	7	65		n.n.			28	no	112
	7	82	mg/kg	24	48	07.09.18	< 5	< 5	< 5	5 mg/kg	no	99
	8	82	mg/kg	66	6	10.10.18	n.d.	n.d.	n.d.	7	no	
	9	82	mg/kg	4	68	12.09.18	<LOQ			5	no	
	10	82	mg/kg	1	71	22.08.18	< 10	< 10	< 10	10	no	-
	11	82	mg/kg	18	54	16.10.18	46	47	45	10	no	100
	12	82	mg/kg	28	44							

Parameter	Parti-cipant	Ann. III cons. no.	Unit	Sample I DLA No.	Sample II DLA No.	Date of ana-lysis	Result (Mean)	Result Sample I	Result Sample II	Limit of quantifi-cation	Incl. RR	Recovery rate
						day/month					yes/no	in %
Geraniol	1	78	mg/kg	8	64	17/10	77,3	71,4	83,2	5	yes	
	2	78	mg/kg	13	59	08.-16.18	126,26	133,53	119		no	
	3	78	mg/kg	34	38	17.10.18	65	65	65	5	no	
	4	78	mg/kg	31	41	16.10.18	100	105	95	5	yes	
	5	78	mg/kg	12	60	27.08.	99	97	102	10	no	
	6	78	mg/kg	7	65		112	110	114	13	no	88
	7	78	mg/kg	24	48	07.09.18	78	74	82	5 mg/kg	no	107
	8	78	mg/kg	66	6	10.10.18	66,9	65,7	68,1	7	no	
	9	78	mg/kg	4	68	12.09.18	79,74	72,21	87,28	5	no	
	10	78	mg/kg	1	71	22.08.18	75,3	75,5	75,2	10	no	-
	11	78	mg/kg	18	54	16.10.18	79	80	78	10	no	100
	12	78	mg/kg	28	44	24.10.18	<1					

Parameter	Parti-cipant	Ann. III cons. no.	Unit	Sample I DLA No.	Sample II DLA No.	Date of ana-lysis	Result (Mean)	Result Sample I	Result Sample II	Limit of quantifica-tion	Incl. RR	Recovery rate
						day/month					yes/no	in %
Hydroxycitronellal	1	72	mg/kg			17/10	<5	<5	<5	5	yes	
	2	72	mg/kg			08.-16.18	-	-	-		no	
	3	72	mg/kg			17.10.18	<5	<5	<5	5	no	
	4	72	mg/kg			16.10.18	n.d.	n.d.	n.d.	5	yes	
	5	72	mg/kg			27.08.	n.d.	n.d.	n.d.	10	no	
	6	72	mg/kg				n.d.	n.d.	n.d.	31	no	125
	7	72	mg/kg			07.09.18	< 5	< 5	< 5	5 mg/kg	no	101
	8	72	mg/kg			10.10.18	n.d.	n.d.	n.d.	7	no	
	9	72	mg/kg			12.09.18	<LOQ			5	no	
	10	72	mg/kg			22.08.18	< 10	< 10	< 10	10	no	-
	11	72	mg/kg			16.10.18	<LOQ	<LOQ	<LOQ	10	no	100
	12	72	mg/kg			24.10.18	<1					

Parameter	Parti-cipant	Ann. III cons. no.	Unit	Sample I DLA No.	Sample II DLA No.	Date of ana-lysis	Result (Mean)	Result Sample I	Result Sample II	Limit of quantifica-tion	Incl. RR	Recovery rate
						day/month					yes/no	in %
Hexylcinnamaldehyd	1	87	mg/kg			17/10	85	84,4	85,7	5	yes	
	2	87	mg/kg			08.-16.18	50,76	47,82	53,69		no	
	3	87	mg/kg			17.10.18	76	75	77	5	no	
	4	87	mg/kg			16.10.18	115	120	110	5	yes	
	5	87	mg/kg			27.08.	174	169	179	10	no	
	6	87	mg/kg				145	140	150	42	no	101
	7	87	mg/kg			07.09.18	104	104	103	5 mg/kg	no	100
	8	87	mg/kg			10.10.18	110	108	112	7	no	
	9	87	mg/kg			12.09.18	71,24	78,35	64,13	5	no	
	10	87	mg/kg			22.08.18	109	109	109	10	no	-
	11	87	mg/kg			16.10.18	85	84	86	10	no	100
	12	87	mg/kg			24.10.18	46,94	45,39	48,49	1		91

Parameter	Parti-cipant	Ann. III cons. no.	Unit	Sample I DLA No.	Sample II DLA No.	Date of ana-lysis	Result (Mean)	Result Sample I	Result Sample II	Limit of quantifica-tion	Incl. RR	Recovery rate
%						day/month					yes/no	in %
Hydroxyisohexyl 3-Cyclohexene Carboxaldehyde	1	79	mg/kg			17/10	<5	<5	<5	5	yes	
	2	79	mg/kg			08.-16.18	18,09	19,44	16,73		no	
	3	79	mg/kg			17.10.18	<5	<5	<5	5	no	
	4	79	mg/kg			16.10.18	n.d.	n.d.	n.d.	10	yes	
	5	79	mg/kg			27.08.	n.d.	n.d.	n.d.	10	no	
	6	79	mg/kg				n.d.	n.d.	n.d.		no	106
	7	79	mg/kg			07.09.18	< 5	< 5	< 5	5 mg/kg	no	97
	8	79	mg/kg			10.10.18	n.d.	n.d.	n.d.	15	no	
	9	79	mg/kg			12.09.18	<LOQ			5	no	
	10	79	mg/kg			22.08.18	< 10	< 10	< 10	10	no	-
	11	79	mg/kg			16.10.18	<LOQ	<LOQ	<LOQ	10	no	100
	12	79	mg/kg									

Parameter	Parti-cipant	Ann. III cons. no.	Unit	Sample I DLA No.	Sample II DLA No.	Date of ana-lysis	Result (Mean)	Result Sample I	Result Sample II	Limit of quantifica-tion	Incl. RR	Recovery rate
%						day/month					yes/no	in %
Isoeugenol	1	73	mg/kg			17/10	144	138	151	5	yes	
	2	73	mg/kg			08.-16.18	-	-	-		no	
	3	73	mg/kg			17.10.18	<5	<5	<5	5	no	
	4	73	mg/kg			16.10.18	12	12	12	5	yes	
	5	73	mg/kg			27.08.	19	19,3	19,2	10	no	
	6	73	mg/kg				n.d.	n.d.	n.d.	39	no	110
	7	73	mg/kg			07.09.18	8	8	7	5 mg/kg	no	115
	8	73	mg/kg			10.10.18	n.d.	n.d.	n.d.	9	no	
	9	73	mg/kg			12.09.18	<LOQ			5	no	
	10	73	mg/kg			22.08.18	< 10	< 10	< 10	10	no	-
	11	73	mg/kg			16.10.18	<LOQ	<LOQ	<LOQ	10	no	100
	12	73	mg/kg			24.10.18	<1					

Parameter	Parti-cipant	Ann. III cons. no.	Unit	Sample I DLA No.	Sample II DLA No.	Date of ana-lysis	Result (Mean)	Result Sample I	Result Sample II	Limit of quantifica-tion	Incl. RR	Recovery rate
						day/month					yes/no	in %
	1	88	mg/kg			17/10	255	245	265	5	yes	
	2	88	mg/kg			08.-16.18	100,89	100,23	101,56		no	
	3	88	mg/kg			17.10.18	240	240	250	5	no	
	4	88	mg/kg			16.10.18	295	290	300	5	yes	
	5	88	mg/kg			10.10.	453	445	462	10	no	
Limonene	6	88	mg/kg				370	360	380	3	no	102
	7	88	mg/kg			07.09.18	297	300	294	5 mg/kg	no	95
	8	88	mg/kg			10.10.18	246	245	246	6	no	
	9	88	mg/kg			12.09.18	255,88	262,23	249,52	5	no	
	10	88	mg/kg			22.08.18	262	267	256	10	no	-
	11	88	mg/kg			16.10.18	181	176	186	10	no	100
	12	88	mg/kg			24.10.18	88,25	86,23	90,27	1	yes	81

Parameter	Parti-cipant	Ann. III cons. no.	Unit	Sample I DLA No.	Sample II DLA No.	Date of ana-lysis	Result (Mean)	Result Sample I	Result Sample II	Limit of quantifica-tion	Incl. RR	Recovery rate
						day/month					yes/no	in %
	1	84	mg/kg			17/10	463	451	475	5	yes	
	2	84	mg/kg			08.-16.18	282,74	283,77	281,71		no	
	3	84	mg/kg			17.10.18	490	490	500	5	no	
	4	84	mg/kg			16.10.18	615	600	630	5	yes	
	5	84	mg/kg			10.10.	654	634	675	10	no	
Linalool	6	84	mg/kg				625	620	630	23	no	110
	7	84	mg/kg			07.09.18	544	547	541	5 mg/kg	no	107
	8	84	mg/kg			10.10.18	449	445	452	5	no	
	9	84	mg/kg			12.09.18	503,9	510,23	497,57	5	no	
	10	84	mg/kg			22.08.18	566	571	560	10	no	-
	11	84	mg/kg			16.10.18	361	349	373	10	no	100
	12	84	mg/kg			24.10.18	<1					

Parameter	Parti-cipant	Ann. III cons. no.	Unit	Sample I DLA No.	Sample II DLA No.	Date of ana-lysis	Result (Mean)	Result Sample I	Result Sample II	Limit of quantifica-tion	Incl. RR	Recovery rate
						day/month					yes/no	in %
Methyl 2-Octynoate	1	89	mg/kg			17/10	<5	<5	<5	5	yes	
	2	89	mg/kg			08.-16.18	-	-	-		no	
	3	89	mg/kg			17.10.18	<5	<5	<5	5	no	
	4	89	mg/kg			16.10.18	n.d.	n.d.	n.d.	5	yes	
	5	89	mg/kg			10.10.	n.d.	n.d.	n.d.	10	no	
	6	89	mg/kg				n.d.			19	no	90
	7	89	mg/kg			07.09.18	< 5	< 5	< 5	5 mg/kg	no	104
	8	89	mg/kg			10.10.18	n.d.	n.d.	n.d.	6	no	
	9	89	mg/kg			12.09.18	<LOQ			5	no	
	10	89	mg/kg			22.08.18	< 10	< 10	< 10	10	no	-
	11	89	mg/kg			16.10.18	<LOQ	<LOQ	<LOQ	10	no	100
	12	89	mg/kg			24.10.18	<1					

5.1.2 Analytical Methods

* For application of the same method notes to the method could be given for Alpha-Isomethyl Ionone only.

Analyte	Parti-cipant	Method specification, as in test report / standard / literature *	Remarks about sample preparation*	Method description*	Calibration and reference material	Recovery with same matrix	Method accredited to ISO / IEC 17025	Further remarks
						yes / no	yes / no	
Alpha-Isomethyl Ionone	1	SM-SZ-249:2018	-	-		yes	no	-
	2	in-house method	liquid extraction with Cyclohexane/Ethylacetate	GC-MS			yes	
	3	LA-GC-604.05 according to DIN EN 16274:2012-12					yes	
	4	in-house method	Extraction with Cyclohexane/Ethylacetate with addition of silica and sodium sulfate	GC-MSD measurement and evaluation with SIM	External calibration with ISTD	no	yes	
	5	SPME-HS GC-MS/MS (in-house method)	Sample diluted with Water/MeOH, ISTD added and extraction with SPME	Evaluation with 16 isotopic labeled internal standards; two MS transitions used for evaluation	Internal labeled standard and fragrance mix from Restek as control		yes	
	6	in-house method	Extraction with org. solvent, measured by GC/Ms-MS/MS	Extraction with Sonotrode		yes	yes	
	7	allergenic fragraces by GC/MS	Extraction with MTBE		Restek Standard Kit allergenic fragrances	yes	yes	
	8	Quantitative determination of allergenic fragrances in cosmetic products	1 g sample mixed with silica gel and Na2SO4 + 20 ml TBME-solution + Int. Standard	GC-MS			area accredited	
	9	in-house method	sample+MTBE+IS	GC-MS	0,1µg/ml-10µg/ml		yes	
	10	in-house method	mixed with silica gel and Na2SO4, extracted with Cyclohexane/ Ethylacetate	GC-MS	calibration with standards, independant control standard	no	yes	
	11	MP 1197 rev 0 2004	The sample is extracted with organic solvent	The extract is analysed by GC/MS in MID-mode	Sigma Aldrich - CD: 93192	no	no	
	12							

Analyte	Parti-cipant	Method specification, as in test report / standard / literature *	Remarks about sample preparation*	Method description*	Calibration and reference material	Recovery with same matrix	Method accredited to ISO / IEC 17025	Further remarks
						yes / no	yes / no	
Amyl Cinnamal	1					yes	no	
	2							
	3	LA-GC-604.05 according to DIN EN 16274:2012-12					yes	
	4				External calibration with ISTD	no	yes	
	5						yes	n.d. = below LOD = 5mg/kg
	6							
	7				Restek Standard Kit allergenic fragrances	yes	yes	
	8							
	9	in-house method	sample+MTBE+IS	GC-MS	0,1µg/ml-10µg/ml		yes	
	10					no	yes	
	11				Sigma Aldrich - CD: 93192	no	no	
	12							

Analyte	Parti-cipant	Method specification, as in test report / standard / literature *	Remarks about sample preparation*	Method description*	Calibration and reference material	Recovery with same matrix	Method accredited to ISO / IEC 17025	Further remarks
						yes / no	yes / no	
Amylcinnamyl Alcohol	1					yes	no	
	2							
	3	LA-GC-604.05 according to DIN EN 16274:2012-12					yes	
	4				External calibration with ISTD	no	yes	
	5						yes	n.d. = below LOD = 5mg/kg
	6							
	7				Restek Standard Kit allergenic fragrances	yes	yes	
	8							
	9	in-house method	sample+MTBE+IS	GC-MS	0,1µg/ml-10µg/ml		yes	
	10					no	yes	
	11				Fluka - CD: 87894	no	no	
	12							

Analyte	Parti-cipant	Method specification, as in test re-port / standard / literature *	Remarks about sample preparati-on*	Method description*	Calibration and reference material	Recovery with same matrix	Method accre-dited to ISO / IEC 17025	Further remarks
						yes / no	yes / no	
Anise Alcohol	1					yes	no	
	2							
	3	LA-GC-604.05 according to DIN EN 16274:2012-12					yes	
	4				External calibration with ISTD	no	yes	
	5						yes	n.d. = below LOD = 5mg/kg
	6							
	7				Restek Standard Kit allergenic fragrances	yes	yes	
	8							
	9	in-house method	sample+MTBE+IS	GC-MS	0,1µg/ml-10µg/ml		yes	
	10					no	yes	
	11				Sigma Aldrich - CD: 136905	no	no	
	12							

Analyte	Parti-cipant	Method specification, as in test re-port / standard / literature *	Remarks about sample preparati-on*	Method description*	Calibration and reference material	Recovery with same matrix	Method accre-dited to ISO / IEC 17025	Further remarks
						yes / no	yes / no	
Benzyl alcohol	1					yes	no	
	2							
	3	LA-GC-604.05 according to DIN EN 16274:2012-12					yes	
	4				External calibration with ISTD	no	yes	
	5						yes	
	6							
	7				Restek Standard Kit allergenic fragrances	yes	yes	
	8							
	9	in-house method	sample+MTBE+IS	GC-MS	0,1µg/ml-10µg/ml		yes	
	10					no	yes	
	11				Sigma Aldrich - CD: 108006	no	no	
	12							

Analyte	Parti-cipant	Method specification, as in test report / standard / literature *	Remarks about sample preparation*	Method description*	Calibration and reference material	Recovery with same matrix	Method accredited to ISO / IEC 17025	Further remarks
						yes / no	yes / no	
Benzyl Benzoate	1					yes	no	
	2							
	3	LA-GC-604.05 according to DIN EN 16274:2012-12					yes	
	4				External calibration with ISTD	no	yes	
	5						yes	
	6							
	7				Restek Standard Kit allergenic fragrances	yes	yes	
	8							
	9	in-house method	sample+MTBE+IS	GC-MS	0,1µg/ml-10µg/ml		yes	
	10					no	yes	
	11				Sigma Aldrich - CD: B6630	no	no	
	12	IFRA Method November, 2016	Shaking and ultrasonic bath with solvent	GC/MS	0.1-1 mg/L, Restek Fragrance Allergen Standards Kit	yes	no	

Analyte	Parti-cipant	Method specification, as in test report / standard / literature *	Remarks about sample preparation*	Method description*	Calibration and reference material	Recovery with same matrix	Method accredited to ISO / IEC 17025	Further remarks
						yes / no	yes / no	
Benzyl Cinnamate	1					yes	no	
	2							
	3	LA-GC-604.05 according to DIN EN 16274:2012-12					yes	
	4				External calibration with ISTD	no	yes	
	5						yes	n.d. = below LOD = 5mg/kg
	6							
	7				Restek Standard Kit allergenic fragrances	yes	yes	
	8							
	9	in-house method	sample+MTBE+IS	GC-MS	0,1µg/ml-10µg/ml		yes	
	10					no	yes	
	11				Sigma Aldrich - CD: W214205	no	no	
	12							

Analyte	Parti-cipant	Method specification, as in test report / standard / literature *	Remarks about sample preparation*	Method description*	Calibration and reference material	Recovery with same matrix	Method accredited to ISO / IEC 17025	Further remarks
						yes / no	yes / no	
Butylphenyl Methylpropional	1					yes	no	
	2							
	3	LA-GC-604.05 according to DIN EN 16274:2012-12					yes	
	4				External calibration with ISTD	no	yes	
	5						yes	
	6							
	7				Restek Standard Kit allergenic fragrances	yes	yes	
	8							
	9	in-house method	sample+MTBE+IS	GC-MS	0,1µg/ml-10µg/ml		yes	
	10					no	yes	
	11				Sigma Aldrich - CD: 43884	no	no	
	12	IFRA Method November, 2016	Shaking and ultrasonic bath with solvent	GC/MS	0.1-1 mg/L, Restek Fragrance Allergen Standards Kit	yes	no	

Analyte	Parti-cipant	Method specification, as in test report / standard / literature *	Remarks about sample preparation*	Method description*	Calibration and reference material	Recovery with same matrix	Method accredited to ISO / IEC 17025	Further remarks
						yes / no	yes / no	
Benzyl Salicylate	1					yes	no	
	2							
	3	LA-GC-604.05 according to DIN EN 16274:2012-12					yes	
	4				External calibration with ISTD	no	yes	
	5						yes	
	6							
	7				Restek Standard Kit allergenic fragrances	yes	yes	
	8							
	9	in-house method	sample+MTBE+IS	GC-MS	0,1µg/ml-10µg/ml		yes	
	10					no	yes	
	11				Sigma Aldrich - CD: W215104	no	no	
	12							

Analyte	Parti-cipant	Method specification, as in test report / standard / literature *	Remarks about sample preparation*	Method description*	Calibration and reference material	Recovery with same matrix	Method accredited to ISO / IEC 17025	Further remarks
						yes / no	yes / no	
Cinnamal	1					yes	no	
	2							
	3	LA-GC-604.05 according to DIN EN 16274:2012-12					yes	
	4				External calibration with ISTD	no	yes	
	5						yes	
	6							
	7				Restek Standard Kit allergenic fragrances	yes	yes	
	8							
	9	in-house method	sample+MTBE+IS	GC-MS	0,1µg/ml-10µg/ml		yes	
	10					no	yes	
	11				Sigma Aldrich - CD: W228613	no	no	
	12	IFRA Method November, 2016	Shaking and ultrasonic bath with solvent	GC/MS	0.1-1 mg/L, Restek Fragrance Allergen Standards Kit	yes	no	

Analyte	Parti-cipant	Method specification, as in test report / standard / literature *	Remarks about sample preparation*	Method description*	Calibration and reference material	Recovery with same matrix	Method accredited to ISO / IEC 17025	Further remarks
						yes / no	yes / no	
Cinnamyl alcohol	1					yes	no	
	2							
	3	LA-GC-604.05 according to DIN EN 16274:2012-12					yes	
	4				External calibration with ISTD	no	yes	
	5						yes	
	6							
	7				Restek Standard Kit allergenic fragrances	yes	yes	
	8							
	9	in-house method	sample+MTBE+IS	GC-MS	0,1µg/ml-10µg/ml		yes	
	10					no	yes	
	11				Sigma Aldrich - CD: 108197	no	no	
	12							

Analyte	Parti-cipant	Method specification, as in test re-port / standard / literature *	Remarks about sample preparati-on*	Method description*	Calibration and reference material	Recovery with same matrix	Method accre-dited to ISO / IEC 17025	Further remarks
						yes / no	yes / no	
Citral	1					yes	no	
	2							
	3	LA-GC-604.05 according to DIN EN 16274:2012-12					yes	
	4				External calibration with ISTD	no	yes	
	5						yes	
	6							
	7				Restek Standard Kit allergenic fragrances	yes	yes	
	8							
	9	in-house method	sample+MTBE+IS	GC-MS	0,1µg/ml-10µg/ml		yes	
	10					no	yes	
	11				Sigma Aldrich - CD: C83007	no	no	
	12	IFRA Method November, 2016	Shaking and ultrasonic bath with solvent	GC/MS	0.1-1 mg/L, Restek Fragrance Allergen Standards Kit	yes	no	

Analyte	Parti-cipant	Method specification, as in test re-port / standard / literature *	Remarks about sample preparati-on*	Method description*	Calibration and reference material	Recovery with same matrix	Method accre-dited to ISO / IEC 17025	Further remarks
						yes / no	yes / no	
Citronellol	1					yes	no	
	2							
	3	LA-GC-604.05 according to DIN EN 16274:2012-12					yes	concealed by Phenoxyethanol
	4				External calibration with ISTD	no	yes	
	5						yes	
	6							
	7				Restek Standard Kit allergenic fragrances	yes	yes	
	8							
	9	in-house method	sample+MTBE+IS	GC-MS	0,1µg/ml-10µg/ml		yes	
	10					no	yes	
	11				Sigma Aldrich - CD: W230901	no	no	
	12							

Analyte	Parti-cipant	Method specification, as in test re-port / standard / literature *	Remarks about sample preparati-on*	Method description*	Calibration and reference material	Recovery with same matrix	Method accre-dited to ISO / IEC 17025	Further remarks
						yes / no	yes / no	
Coumarin	1					yes	no	
	2							
	3	LA-GC-604.05 according to DIN EN 16274:2012-12					yes	
	4				External calibration with ISTD	no	yes	
	5						yes	
	6							
	7				Restek Standard Kit allergenic fragrances	yes	yes	
	8							
	9	in-house method	sample+MTBE+IS	GC-MS	0,1µg/ml-10µg/ml		yes	
	10					no	yes	
	11				Sigma Aldrich - CD: C4261	no	no	
	12	IFRA Method November, 2016	Shaking and ultrasonic bath with solvent	GC/MS	0.1-1 mg/L, Restek Fragrance Allergen Standards Kit	yes	no	

Analyte	Parti-cipant	Method specification, as in test re-port / standard / literature *	Remarks about sample preparati-on*	Method description*	Calibration and reference material	Recovery with same matrix	Method accre-dited to ISO / IEC 17025	Further remarks
						yes / no	yes / no	
Eugenol	1					yes	no	
	2							
	3	LA-GC-604.05 according to DIN EN 16274:2012-12					yes	
	4				External calibration with ISTD	no	yes	
	5						yes	
	6							
	7				Restek Standard Kit allergenic fragrances	yes	yes	
	8							
	9	in-house method	sample+MTBE+IS	GC-MS	0,1µg/ml-10µg/ml		yes	
	10					no	yes	
	11				Sigma Aldrich - CD: E51791	no	no	
	12							

Analyte	Parti-cipant	Method specification, as in test report / standard / literature *	Remarks about sample preparation*	Method description*	Calibration and reference material	Recovery with same matrix	Method accredited to ISO / IEC 17025	Further remarks
						yes / no	yes / no	
Evernia Furfuracea Extract	1					yes	no	
	2							
	3	LA-GC-604.05 according to DIN EN 16274:2012-12					yes	
	4							
	5							
	6							
	7				Neochema	no	yes	
	8							
	9	in-house method	sample+MTBE+IS	GC-MS	0,1µg/ml-10µg/ml		yes	
	10							
	11							test not included in the method
	12							

Analyte	Parti-cipant	Method specification, as in test report / standard / literature *	Remarks about sample preparation*	Method description*	Calibration and reference material	Recovery with same matrix	Method accredited to ISO / IEC 17025	Further remarks
						yes / no	yes / no	
Evernia Prunastri Extract	1					yes	no	
	2							
	3	LA-GC-604.05 according to DIN EN 16274:2012-12					yes	
	4							
	5							
	6							
	7				Sigma Aldrich	no	yes	
	8							
	9	in-house method	sample+MTBE+IS	GC-MS	0,1µg/ml-10µg/ml		yes	
	10							
	11							test not included in the method
	12							

Analyte	Parti-cipant	Method specification, as in test re-port / standard / literature *	Remarks about sample preparati-on*	Method description*	Calibration and reference material	Recovery with same matrix	Method accre-dited to ISO / IEC 17025	Further remarks
						yes / no	yes / no	
Geraniol	1					yes	no	
	2							
	3	LA-GC-604.05 according to DIN EN 16274:2012-12					yes	
	4				External calibration with ISTD	no	yes	
	5						yes	
	6							
	7				Restek Standard Kit allergenic fragrances	yes	yes	
	8							
	9	in-house method	sample+MTBE+IS	GC-MS	0,1µg/ml-10µg/ml		yes	
	10					no	yes	
	11				Sigma Aldrich - CD: W250708	no	no	
	12							

Analyte	Parti-cipant	Method specification, as in test re-port / standard / literature *	Remarks about sample preparati-on*	Method description*	Calibration and reference material	Recovery with same matrix	Method accre-dited to ISO / IEC 17025	Further remarks
						yes / no	yes / no	
Farnesol	1					yes	no	
	2							
	3	LA-GC-604.05 according to DIN EN 16274:2012-12					yes	
	4				External calibration with ISTD	no	yes	
	5						yes	
	6							
	7				Restek Standard Kit allergenic fragrances	yes	yes	
	8							
	9	in-house method	sample+MTBE+IS	GC-MS	0,1µg/ml-10µg/ml		yes	
	10					no	yes	
	11				Sigma Aldrich - CD: F203	no	no	
	12							

Analyte	Parti-cipant	Method specification, as in test re-port / standard / literature *	Remarks about sample preparati-on*	Method description*	Calibration and reference material	Recovery with same matrix	Method accre-dited to ISO / IEC 17025	Further remarks
						yes / no	yes / no	
Hexylcinnamaldehyd	1					yes	no	
	2							
	3	LA-GC-604.05 according to DIN EN 16274:2012-12					yes	
	4				External calibration with ISTD	no	yes	
	5						yes	
	6							
	7				Restek Standard Kit allergenic fragrances	yes	yes	
	8							
	9	in-house method	sample+MTBE+IS	GC-MS	0,1µg/ml-10µg/ml		yes	
	10					no	yes	
	11				Sigma Aldrich - CD: 09178	no	no	
	12	IFRA Method November, 2016	Shaking and ultrasonic bath with solvent	GC/MS	0.1-1 mg/L, Restek Fragrance Allergen Standards Kit	yes	no	

Analyte	Parti-cipant	Method specification, as in test re-port / standard / literature *	Remarks about sample preparati-on*	Method description*	Calibration and reference material	Recovery with same matrix	Method accre-dited to ISO / IEC 17025	Further remarks
						yes / no	yes / no	
Hydroxycitronellal	1					yes	no	
	2							
	3	LA-GC-604.05 according to DIN EN 16274:2012-12					yes	
	4				External calibration with ISTD	no	yes	
	5						yes	n.d. = below LOD = 5mg/kg
	6							
	7				Restek Standard Kit allergenic fragrances	yes	yes	
	8							
	9	in-house method	sample+MTBE+IS	GC-MS	0,1µg/ml-10µg/ml		yes	
	10					no	yes	
	11				Sigma Aldrich - CD: 82934	no	no	
	12							

Analyte	Parti-cipant	Method specification, as in test report / standard / literature *	Remarks about sample preparation*	Method description*	Calibration and reference material	Recovery with same matrix	Method accredited to ISO / IEC 17025	Further remarks
						yes / no	yes / no	
Hydroxyisohexyl 3-Cyclohexene Carboxaldehyde	1					yes	no	
	2							
	3	LA-GC-604.05 according to DIN EN 16274:2012-12					yes	
	4				External calibration with ISTD	no	yes	
	5						yes	n.d. = below LOD = 5mg/kg
	6							
	7				Restek Standard Kit allergenic fragrances	yes	yes	
	8							
	9	in-house method	sample+MTBE+IS	GC-MS	0,1µg/ml-10µg/ml		yes	
	10					no	yes	
	11				Sigma Aldrich - CD: 95594	no	no	
	12							

Analyte	Parti-cipant	Method specification, as in test report / standard / literature *	Remarks about sample preparation*	Method description*	Calibration and reference material	Recovery with same matrix	Method accredited to ISO / IEC 17025	Further remarks
						yes / no	yes / no	
Isoeugenol	1					yes	no	
	2							
	3	LA-GC-604.05 according to DIN EN 16274:2012-12					yes	
	4				External calibration with ISTD	no	yes	
	5						yes	
	6							
	7				Restek Standard Kit allergenic fragrances	yes	yes	confirmed by GC-MSMS
	8							
	9	in-house method	sample+MTBE+IS	GC-MS	0,1µg/ml-10µg/ml		yes	
	10					no	yes	
	11				Sigma Aldrich - CD: I17206	no	no	
	12							

Analyte	Parti-cipant	Method specification, as in test report / standard / literature *	Remarks about sample preparation*	Method description*	Calibration and reference material	Recovery with same matrix	Method accredited to ISO / IEC 17025	Further remarks
						yes / no	yes / no	
Limonene	1					yes	no	
	2							
	3	LA-GC-604.05 according to DIN EN 16274:2012-12					yes	
	4				External calibration with ISTD	no	yes	
	5						yes	
	6							
	7				Restek Standard Kit allergenic fragrances	yes	yes	
	8							
	9	in-house method	sample+MTBE+IS	GC-MS	0,1µg/ml-10µg/ml		yes	
	10					no	yes	
	11				Sigma Aldrich - CD: 62118	no	no	
	12	IFRA Method November, 2016	Shaking and ultrasonic bath with solvent	GC/MS	0.1-1 mg/L, Restek Fragrance Allergen Standards Kit	yes	no	

Analyte	Parti-cipant	Method specification, as in test report / standard / literature *	Remarks about sample preparation*	Method description*	Calibration and reference material	Recovery with same matrix	Method accredited to ISO / IEC 17025	Further remarks
						yes / no	yes / no	
Linalool	1					yes	no	
	2							
	3	LA-GC-604.05 according to DIN EN 16274:2012-12					yes	
	4				External calibration with ISTD	no	yes	
	5						yes	
	6							
	7				Restek Standard Kit allergenic fragrances	yes	yes	
	8							
	9	in-house method	sample+MTBE+IS	GC-MS	0,1µg/ml-10µg/ml		yes	
	10					no	yes	
	11				Sigma Aldrich - CD: 8.18627.0000	no	no	
	12							

Analyte	Parti-cipant	Method specification, as in test re-port / standard / literature *	Remarks about sample preparati-on*	Method description*	Calibration and reference material	Recovery with same matrix	Method accre-dited to ISO / IEC 17025	Further remarks
						yes / no	yes / no	
Methyl 2-Octynoate	1					yes	no	
	2							
	3	LA-GC-604.05 according to DIN EN 16274:2012-12					yes	
	4				External calibration with ISTD	no	yes	
	5						yes	n.d. = below LOD = 5mg/kg
	6							
	7				Restek Standard Kit allergenic fragrances	yes	yes	
	8							
	9	in-house method	sample+MTBE+IS	GC-MS	0,1µg/ml-10µg/ml		yes	
	10					no	yes	
	11				Sigma Aldrich - CD: W272906	no	no	
	12							

5.2 Homogeneity

5.2.1 Comparision of sample numbers / test results and trend line

By comparison of the increasing sample numbers and the measurement results of participants, the homogeneity of the chronological bottled PT items can be shown by the trend line for information:
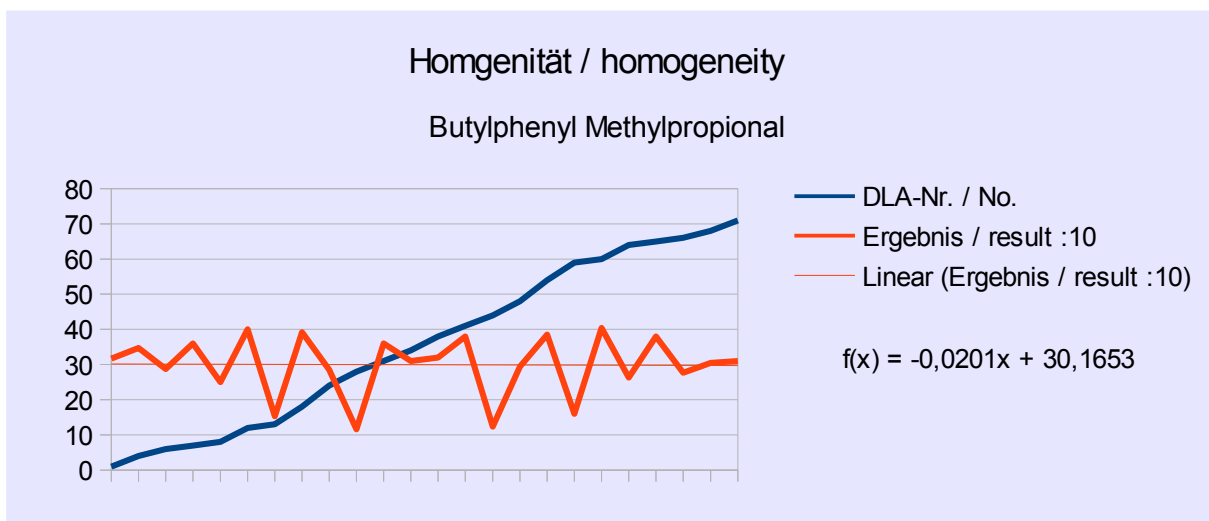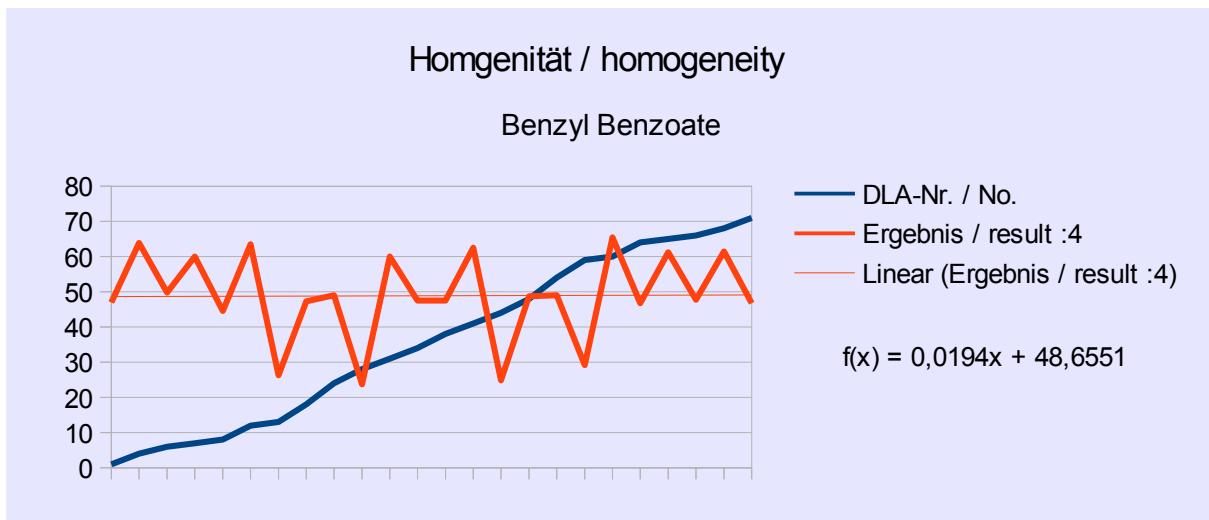
Abb./Fig. 29:
Trendfunktion Probennummern vs. Ergebnisse: Benzyl Benzoate und Butylphenyl Methylpropional (1/ 4 und 1/10 dargestellt)

Trend line function sample number vs. results: Benzyl Benzoate and Butylphenyl Methylpropional (1/ 4 and 1/10 shown)

5.3 Kernel Density Plots of Results

Abbildungen:
Kerndichte-Schätzungen
der Teilnehmerergebnisse
(mit h = 1 x σ_{pt} von X$_{pt}$)

Figures:
Kernel density plots
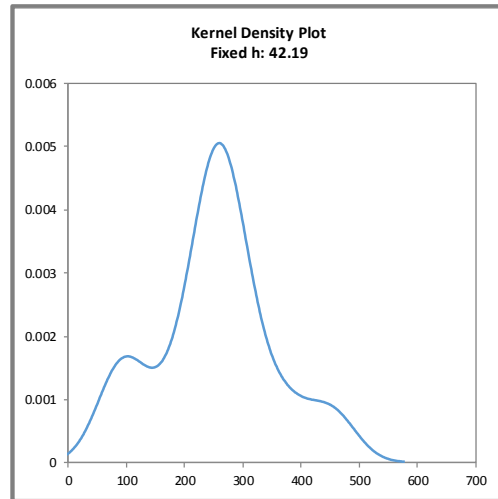of participants' results
(with h = 1 x σ_{pt} of X$_{pt}$)

Alpha Isomethyl Ionone
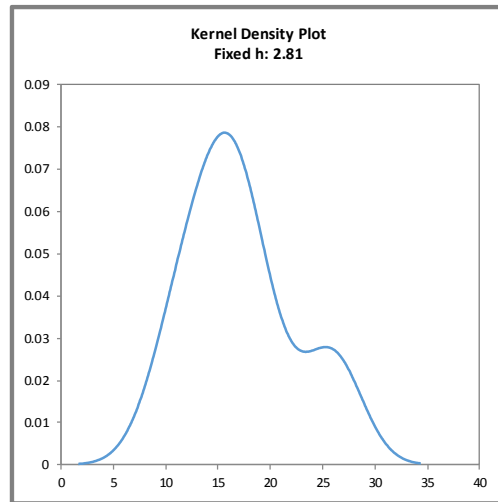
Benzyl Alcohol
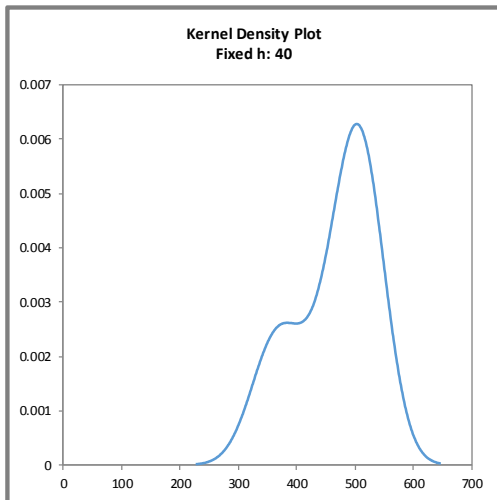
Benzyl Benzoate
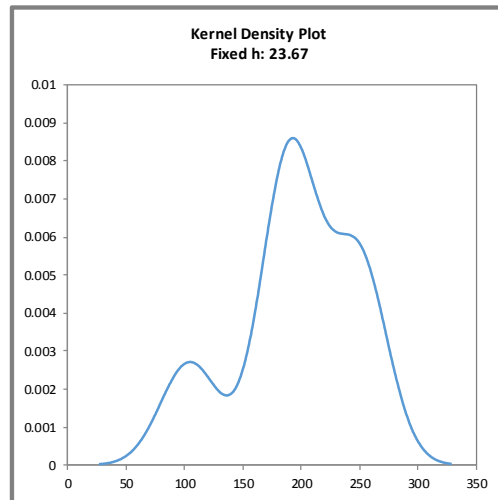
Benzyl Salicylate
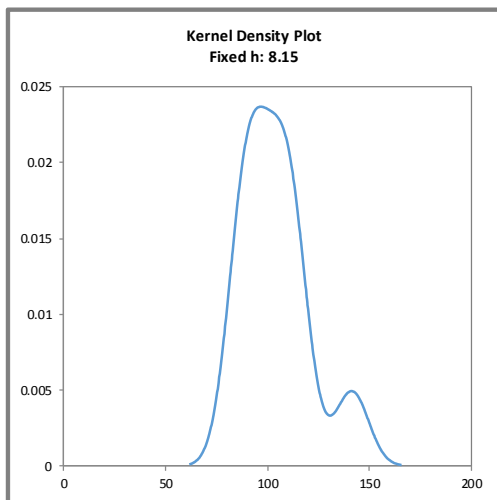
Butylphenyl Methylpropional

Abbildungen:
Kerndichte-Schätzungen
der Teilnehmerergebnisse
(mit h = 1 x σ_{pt} von X$_{pt}$)

Figures:
Kernel density plots
of participants' results
(with h = 1 x σ_{pt} of X$_{pt}$)
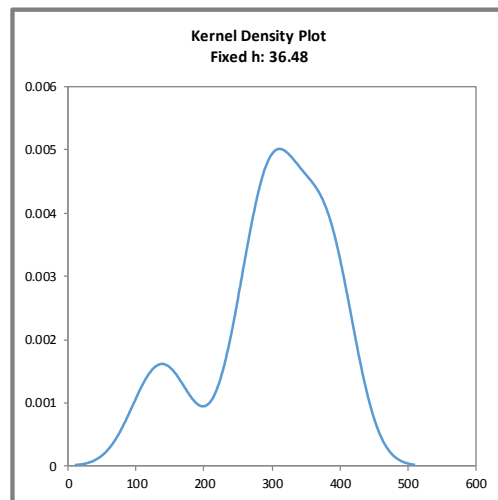
Cinnamal

Kernel Density Plot
Fixed h: 51.91

Citral

Kernel Density Plot
Fixed h: 43.55

Citronellol

Kernel Density Plot
Fixed h: 24.33

Coumarin

Kernel Density Plot
Fixed h: 7.98
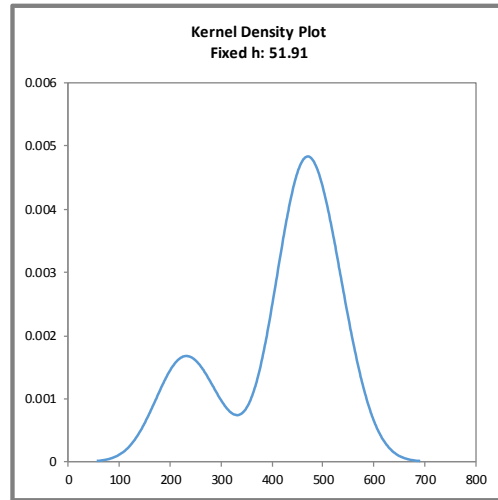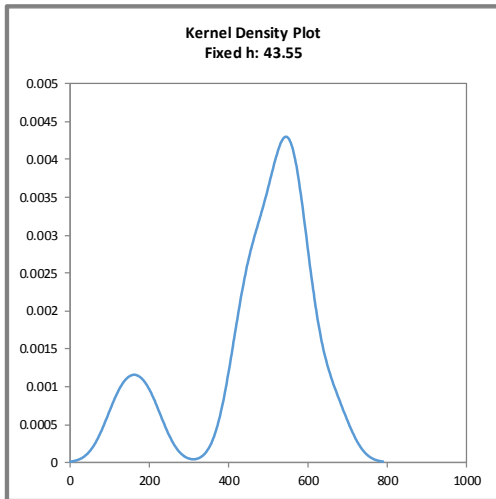
Eugenol
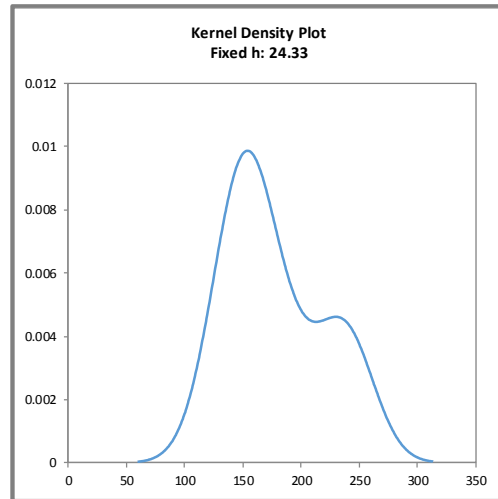
Kernel Density Plot
Fixed h: 18.03
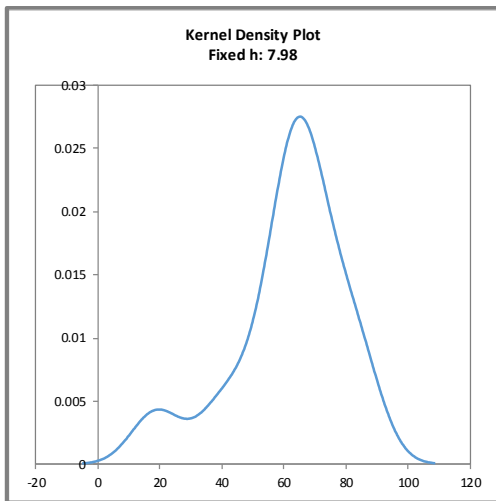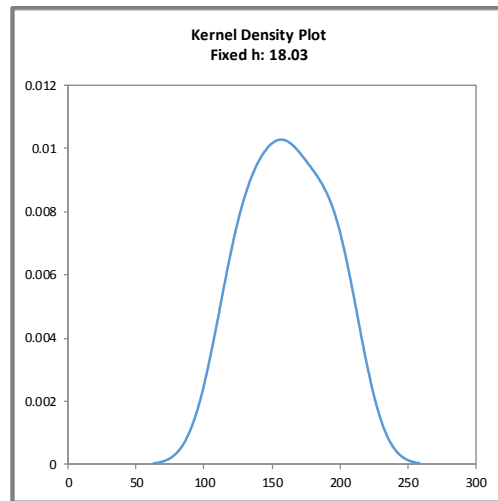
Abbildungen:
Kerndichte-Schätzungen
der Teilnehmerergebnisse
(mit h = 1 x σ_{pt} von X_{pt})

Figures:
Kernel density plots
of participants' results
(with h = 1 x σ_{pt} of X_{pt})

Geraniol
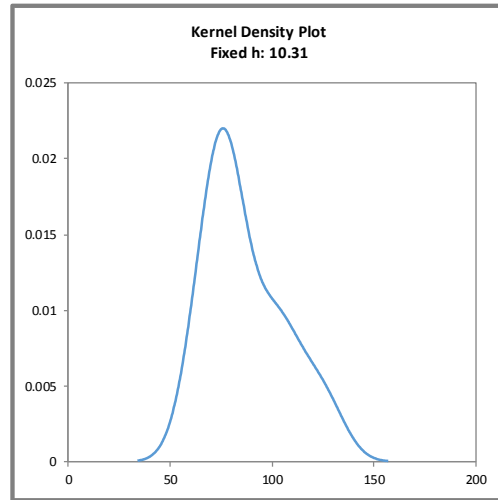
Hexylcinnamaldehyd
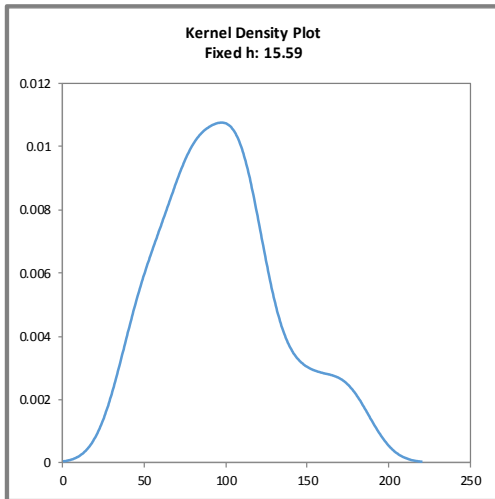
Limonene

Linalool
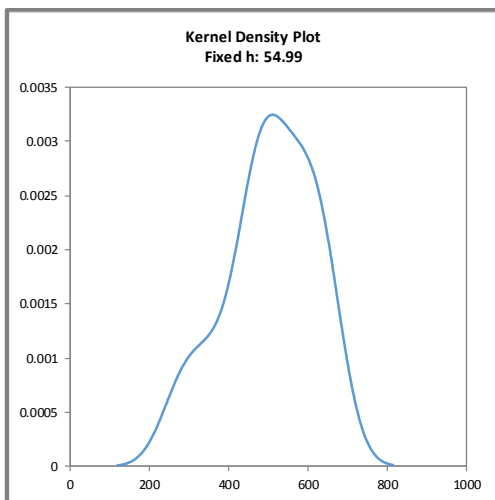
5.4 Information on the Proficiency Test (PT)

Before the PT the participants received the following information in the sample cover letter:

PT number	**DLA 53-2018**
PT name	**Cosmetic Products III: Allergenic Fragrances in Skin Cream**
Sample matrix*	Samples I + II: **Skin Cream/Bodylotion common in commerce ingredients**
Number of samples and sample amount	2 identical samples I + II, 25 g each.
Storage	Samples I + II: cooled 2 - 10°C (dry and dark)
Intentional use	Laboratory use only (quality control samples)
Parameter	quantitative: **Allergenic Fragrances** (substances to be labeled according to Cosmetics Regulation EC 1223/2009 Art.19 g + Annex III), see result submission file
Methods of analysis	Analytical methods are optional
Notes to analysis	The analysis of PT samples should be performed like a routine laboratory analysis. In general we recommend to homogenize a representative sample amount before analysis according to good laboratory practice, especially in case of low sample weights.
Result sheet	The results for sample I and II as well as the final results calculated as mean of the double determination (samples I and II) should be filled in the result submission file. The recovery rates, if carried out, has to be included in the calculation.
Units	mg/kg
Number of significant digits	at least 2
Further information	For information please specify: – Date of analysis – DLA-sample-numbers (for sample I and II) – Limit of detection – Assignment incl. Recovery – Recovery with the same matrix – Method is accredited
Result submission	The result submission file should be sent by e-mail to: **pt@dla-lvu.de**
Deadline	**the latest 19th October 2018**
Evaluation report	The evaluation report is expected to be completed 6 weeks after deadline of result submission and sent as PDF file by e-mail.
Coordinator and contact person of PT	Matthias Besler-Scharf, PhD

* Control of mixture homogeneity and qualitative testings are carried out by DLA. Testing of the content, homogeneity and stability of PT parameters is subcontracted by DLA.

6. Index of participant laboratories in alphabetical order

Teilnehmer / Participant	Ort / Town	Land / Country
		HUNGARY
		Germany
		ITALY
		Germany
		Germany
		Germany
		Germany
		Germany
		Germany
		Germany
		Germany
		Germany
		CROATIA
		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 references

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 inter-laboratory comparisons
4. ASU §64 LFGB: Planung und statistische Auswertung von Ringversuchen zur Methoden-validierung / DIN ISO 5725 series part 1, 2 and 6 Accuracy (trueness and preci-sion) 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. Hor-witz; 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)
8. A Horwitz-like funktion describes precision in proficiency test; M. Thompson, P.J. Lowthian; Analyst, 120, 271-272 (1995)
9. Protocol for the design, conduct and interpretation of method performance studies; W. Horwitz; Pure & Applied Chemistry, 67, 331-343 (1995)
10. Recent trends in inter-laboratory precision at ppb and sub-ppb concentrations in relation to fitness for purpose criteria in proficiency testing; M. Thompson; Ana-lyst, 125, 385-386 (2000)
11. The International Harmonised Protocol for the Proficiency Testing of Analytical Chemistry Laboratories; Pure Appl Chem, 78, 145 – 196 (2006)
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13. EURACHEM/CITAC Leitfaden, Ermittlung der Messunsicherheit bei analytischen Messun-gen (2003); Quantifying Uncertainty in Analytical Measurement (1999)
14. GMP+ Feed Certification scheme, Module: Feed Safety Assurance, chapter 5.7 Check-ing 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. Homogeneity and stability of reference materials; Linsinger et al.; Accred Qual Assur, 6, 20-25 (2001)
17. AOAC Official Methods of Analysis: Guidelines for Standard Method Performance Re-quirements, Appendix F, p. 2, AOAC Int (2016)

18. EN 16274:2012-12 Analyseverfahren für Allergene - Quantifizierung von mutmaßlichen Allergie auslösenden Duftstoffen in Verbrauchsgütern - Stufe 1: GC-Analyse von einspritzfertigen Proben / Methods for analysis of allergens - Quantification of suspected fragrance allergens in consumer products - Step 1: GC analysis of ready-to-inject sample
19. IFRA Method: Analytical method to quantify 57 suspected allergens (and isomers) in ready to inject fragrance materials by gas chromatography and mass spectometry, The International Fragrance Association, Analytical Working Group, November 15th, 2016
20. Belhassen et al. (2017): Routine quantification of 54 allergens in fragrances us-ing comprehensive two-dimensional gas chromatographyquadrupole mass spectrometry with dual parallel secondary columns. Part I: Method development, Flavour Fragr J. 2018;33:63-74