

**Evaluation of the
interlaboratory test 2010-2011
on the method DIN EN15188:2007
“Determination of the
spontaneous ignition behaviour
of dust accumulations”
Final Report**



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Report

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1 Glossary

Interlaboratory test steps

TS 1	Interlaboratory test step 1: determination of T_{SI} / checking the volume of the sample baskets by laboratories without any recommended method
TS 2	Interlaboratory test step 2: checking the volume of the sample baskets by BAM method: glass beads
TS 3	Interlaboratory test step 3: repetition of determination of T_{SI} / checking the volume of the sample baskets by laboratories (recommended method: glass beads)

Technical terms

Mesh wire screen	Additional screen installed into the laboratory oven
Sample basket.....	Double walled mesh wired baskets (cubes) of different volumes

Volume

Nominal volume.....	Projected target volume of the sample baskets (intended edge length: 5 cm, 6 cm, 8.5 cm and 10 cm) manufactured by BAM (125 mL, 216 mL, 614 mL, 1000 mL)
TS 1 volume	Volume of the sample basket checked by the laboratory in TS 1 by own methods
Reference volume (TS 2 volume).....	Volume of the sample basket determined by BAM with glass beads in TS 2
TS 3 volume	Volume of the sample basket determined by the laboratory in TS 3; recommended method: glass beads
Storage volume	Practice-related volume considered for extrapolation of T_{SI} measurements

Temperature

Oven temperature.....	Arithmetic mean of the measured values of two thermocouples, both freely installed in an oven (inside the mesh wire screen)
“go”- ignition temperature	Lowest oven temperature at which a given volume of dust has ignited
“No go”- no ignition	Highest oven temperature at which a given volume of dust just did not ignite, T_{SI}

T_{SI} Self-ignition temperatures in the sense of DIN EN 15188:2007

extrapolated T_{SI} Self-ignition temperatures of practice-related volumes (storage volumes) extrapolated by the aid of the curves of the Pseudo-Arrhenius plot

Statistics

Robust mean value mean value calculated by applying the so-called Hampel estimator as described e.g. in DIN 38402-45 (=ISO/TS 20612) which requires no outlier examination.

Total robust mean value robust mean value according to DIN 38402-45 of the modified method DIN EN 15188

Repeatability standard deviation Precision under repeatability conditions, i.e. same laboratory, same operator and same apparatus

Reproducibility standard deviation Precision under reproducibility conditions, i.e. different laboratories, different operators, different apparatuses

Robust reproducibility / repeatability standard deviation Calculated by using the Q method as described DIN 38402-45 (=ISO/TS 20612) which requires no outlier examination.

Coverage factor k Multiplier to estimate the (expanded) measurement uncertainty U of a result y [4]. The value of the coverage factor k is chosen on the basis of the level of confidence required of the interval $y - U$ to $y + U$ (→ Table 1-1)

Table 1-1: Value of the coverage factor k that produces an interval having level of confidence assuming a normal distribution (source [4])

Coverage factor k	Level of confidence [%]
1	68,27
1,645	90
1,960	95
2	95,45
2,576	99
3	99,73

Measurement uncertainty (U) is about twice the robust reproducibility standard deviation s_R (k=2) in this report; also referred to as expanded uncertainty

Expanded uncertainty of mean value is about twice the standard deviation of the robust mean value (k=2) in this report

95 % tolerance interval for

extrapolated T_{SI} Range, where in 95 of 100 laboratory tests the extrapolated T_{SI} will be in as expected (range between the curve of the lower tolerance limit values and the curve of the upper tolerance limit values). The interval has been calculated on the basis of the measurement uncertainty with $k=2$.

Lower/upper tolerance limit of the

95 % tolerance interval = 'Robust mean value' \pm 'measurement uncertainty'

2 Introduction

For the classification and safe handling and use of the chemicals, special standardized testing procedures have been developed and are used world-wide. Safety experts must be able to fully rely on the precise execution of the respective laboratory tests and assessments. In this context interlaboratory tests (round robin tests, interlaboratory comparisons / intercomparisons) are a crucial element of a laboratory's quality system. Participation in interlaboratory tests is explicitly recommended by the standard ISO/IEC 17025.

The present document reports on the results of the interlaboratory test 2010/2011 on the test method DIN EN 15188:2007 "Determination of the spontaneous ignition behaviour of dust accumulations" [1] which was organized by the Center for Quality Assurance for Testing of Dangerous Goods and Hazardous Substances.

The test method DIN EN 15188:2007 is applied to characterize the self-ignition behaviour of combustible dusts. The experimental basis for describing the self-ignition behaviour of a given dust is the determination of the self-ignition temperatures (T_{Si}) of differently-sized volumes of the dust sample by isoperibolic hot storage experiments (storage at constant oven temperatures) in commercially available ovens. The results thus measured reflect the dependence of self-ignition temperatures upon dust volume [1].

Several internal investigations and interlaboratory comparisons in the past have shown significant differences between the lab-specific results of hot storage tests.

Figure 2-1 shows the Pseudo-Arrhenius plot of hot storage tests of eight different laboratories (Round Robin Test 2002, BAM). The dust under this investigation was Lycopodium powder (spores). The participants of this interlaboratory test used different laboratory ovens (size, ventilation) as well as different sample baskets (shape, mesh size, single- and double-walled).

Figure 2-1 shows clearly that this test failed to produce reasonable reproducibility of the T_{Si} between the different laboratories. As possible reasons for the deviations have been identified lab-specific differences, e.g.:

- oven ventilation (enforced, natural convection),
- oven size,
- sample baskets,
- radiation effects,
- measuring precision (temperature difference between tests with ignition and no ignition),
- minimum sample size.

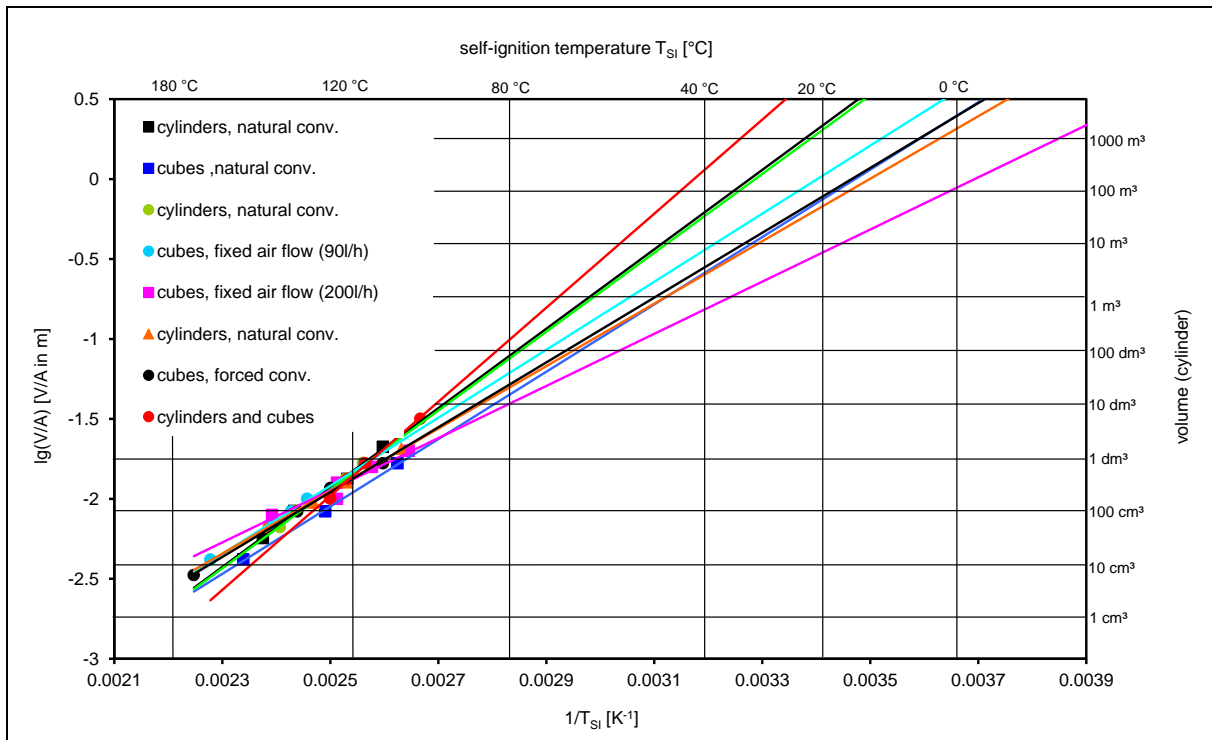


Figure 2-1: Pseudo-Arrhenius plot of self-ignition temperatures of Lycopodium powder, (Round Robin Test 2002, BAM)

To reduce the differences between the labs it was necessary to ameliorate the testing method and to improve the execution of the method by the lab. From there, the installation of an inner chamber into the laboratory oven was suggested as experimental set-up in EN 15188:2007 to provide more reproducible test conditions. The appropriateness of this set-up has not been verified yet.

The current interlaboratory test 2010-2011 focuses on the use of a special mesh wire screen and special volumes of the sample baskets (cubes) to normalise/harmonise the test conditions in the different labs. In preparation for the interlaboratory test a joint program between Syngenta and BAM has been initiated in 2009. As a result of these investigations a modified set-up (→ chapter 3) has been identified to be probably more appropriate than the suggested set-up in DIN EN-15188:2007.

Due to the time-consuming test procedure and to optimize the workflow for the laboratories this interlaboratory test should be performed stepwise as a multi-level test (→ chapter 5.4) on one typical test sample.

3 Aim

The aims of this interlaboratory test 2010/2011 on the method DIN EN 15188:2007 “Determination of the spontaneous ignition behaviour of dust accumulations” are:

- (1) Assessment of the performance of the modified method DIN EN 15188, by means of the investigation of a suitable, typical test sample
- (2) Assessment of other influence (disturbing) factors
- (3) Recommendations for the participants of the interlaboratory test to improve the execution of the method

In the following, these three aims will be described in detail.

3.1 Assessment of the performance of the modified method DIN EN 15188

The following modifications of the DIN EN 15188:2007 were suggested for this interlaboratory test:

Mesh wire screen

The current practical application of the modified method in different laboratories was assessed in the first step of the interlaboratory test. For this purpose, specific precision indicators (e.g. reproducibility, repeatability etc.) were generated. The use of a mesh wire screen and the volumes of the sample baskets (double-walled cubes, hanging in oven) in this test series were standardized (→ Figure 3-1). The modification of the test method was based on proposals by CEN/TC 305/ WG 01 and by BAM [9], [10], [12].



a) sample holder

b) mesh wire screen with sample holder

Figure 3-1: Suggested set-up

Volumes ratios of sample baskets of 1 : 1.7 : 5 : 8

DIN EN 15188:2007 recommends the usage of mesh wire cylinders with a height to diameter ratio of 1 or cubes as sample baskets.

At least three mesh wire baskets of different volumes have to be used for the tests; the smallest volume should normally be in the order of 10 cm³ and the largest should normally not be smaller than approximately 1 L. It has to be assured that the volume of a basket exceeds that of the previous one in the series by a factor of 2 at a minimum. VDI guideline 2263, part 1 recommends sample baskets with volumes of 100 cm³, 1000 cm³ and 1600 cm³.

Figure 3-2 shows a pseudo-Arrhenius plot for four cylindrical samples of 100 mL, 400 mL, 800 mL and 1600 mL.

The smallest and the largest volume are the determining factors for the slope of the regression line. Changes in the T_{SI} of the volumes between will only have a marginal effect on the regression line.

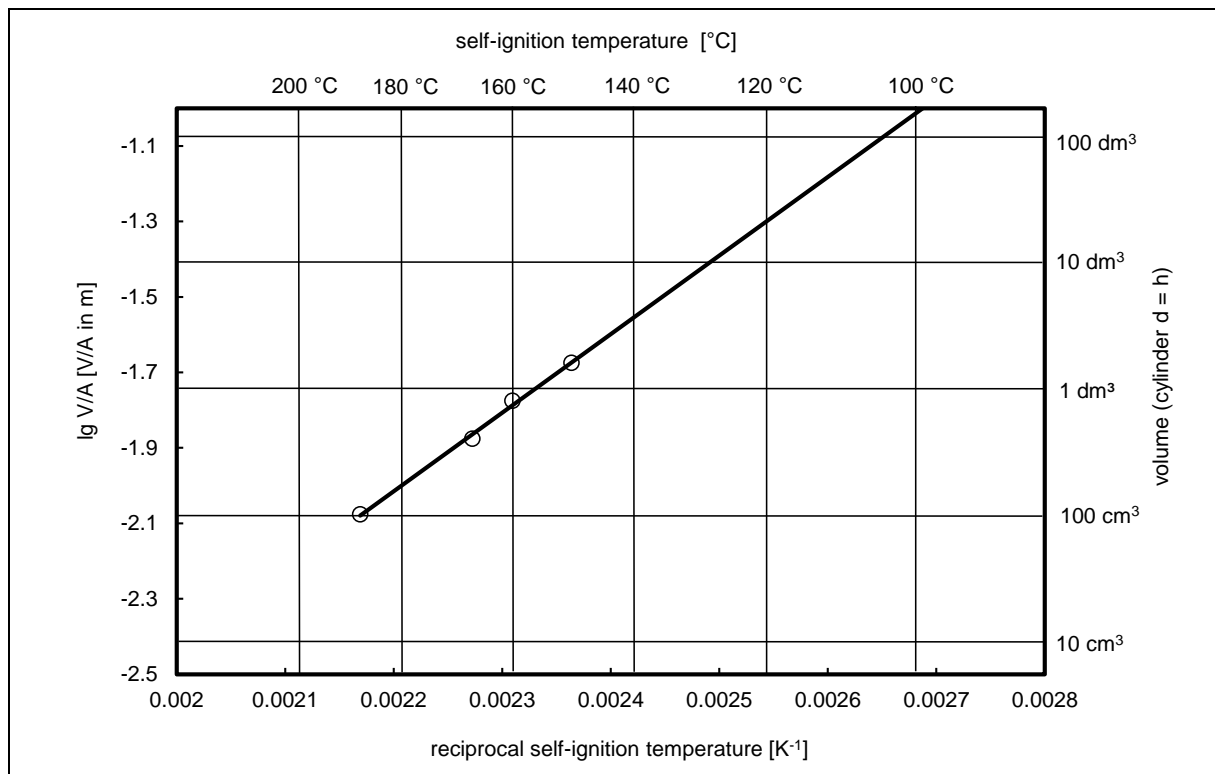


Figure 3-2: Pseudo-Arrhenius plot of cylindrical samples

Four different sample baskets with a volume ratio of 1 : 1.7 : 5 : 8 have been used in this interlaboratory test. The example of the Pseudo-Arrhenius plot for four sample volumes of 125 mL, 216 mL, 614 mL and 1000 mL (cubes of 5 cm, 6 cm, 8.5 cm and 10 cm) is demonstrated in Figure 3-3. The sample volumes of 125 mL, 216 mL, 614 mL and 1000 mL correspond to the volume ratio volume ratio of approx. 1 : 1.7 : 5 : 8.

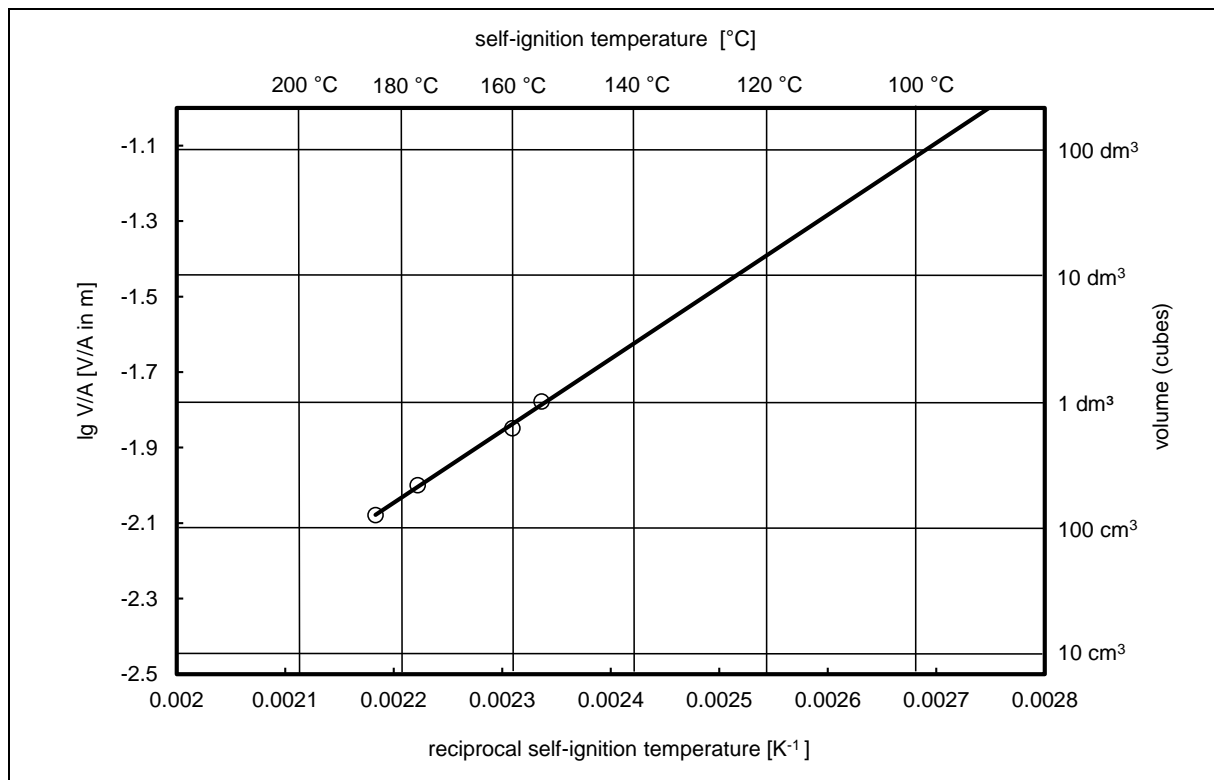


Figure 3-3: Pseudo-Arrhenius plot of cubic samples as used as for the interlaboratory test

Using sample baskets of volume ratios of 1 : 1.7 : 5 : 8 should lead to a better defined regression line since there are two volumes tested in the lower and upper section of the regression line, respectively. This mathematical problem of the regression line and the extrapolation is comparable to the problem of the distance between iron sights (notch and bead sight) for firearms. Furthermore by application of these ratios significant and sufficiently large differences between the T_{Si} of the different volumes should be ensured.

3.2 Assessment of other influence (disturbing) factors

Other laboratory specific factors which possibly may have an influence on the test result (T_{Si}) were evaluated with the aid of a further exploratory analysis.

3.3 Recommendations for the participants of the interlaboratory test to improve the execution of the modified method DIN EN 15188

In view of the results of the interlaboratory test, it was assessed which recommendations could be given to the participating laboratories to improve the execution of the modified method DIN EN 15188.

4 Interlaboratory test sample

Activated carbon powder “Carbon Black, Norit CN4” (Norit (U.K.) Limited, Glasgow, United Kingdom) was chosen as interlaboratory test sample.

4.1 Manufacturing and delivery of the interlaboratory test sample

The interlaboratory test sample “Carbon Black, Norit CN4” (total amount 960 kg, batch number 0002.1) was manufactured by Norit (U.K.) Limited, Glasgow, on 04/01/2011. BAM received the test sample on 24/01/2011 (48 20kg-bags on 1 pallet, → Figure 4-1).

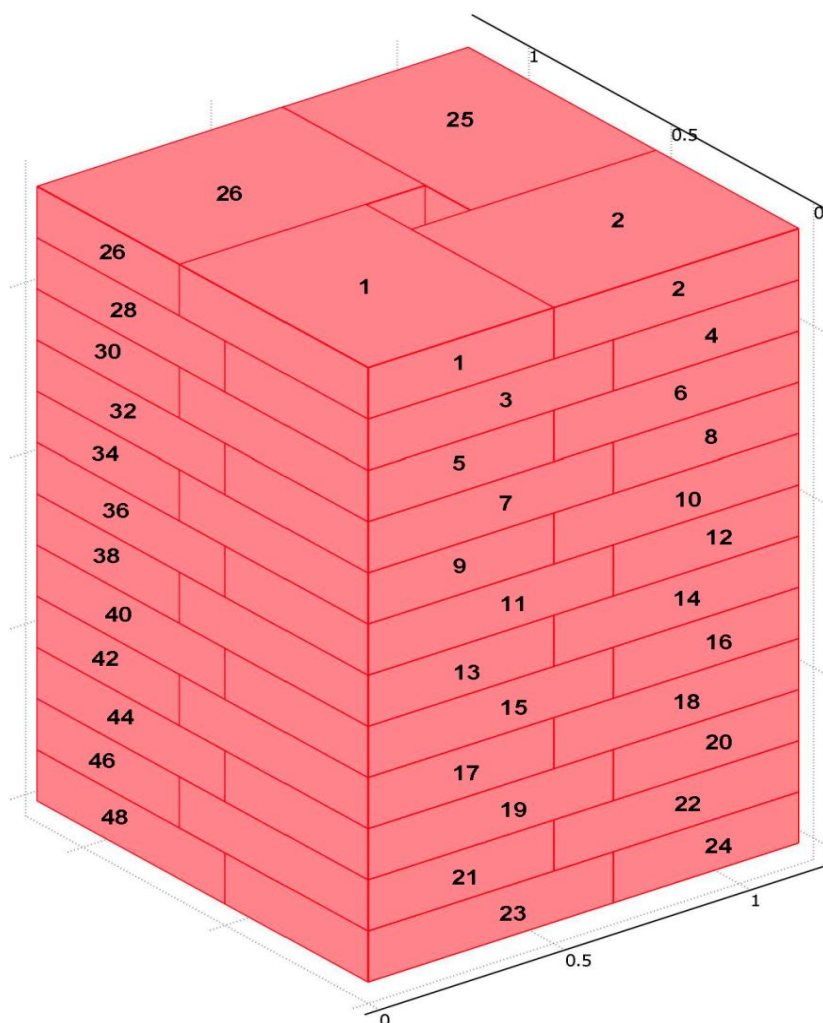


Figure 4-1: Position and numeration of the bags on the pallet

Interlaboratory test samples were taken directly from the bags manufactured “Carbon Black, Norit CN4” without any additional homogenization, i.e. the bag number was equivalent to the interlaboratory test sample number for the respective lab.

In June 2011 the interlaboratory test samples (bags) were packed into transport containers (two hob-bucks for each lab, → Figure 4-2) and sent to the participants of the interlaboratory test.



Figure 4-2: Interlaboratory test sample “Carbon Black, Norit CN4”:
bag from the supplier and 2 hobo-bucks (20 l, Dosen-Zentrale Züchner GmbH, Köln)
for one participant of the interlaboratory test

4.2 Homogeneity and stability of the interlaboratory test sample

Based on experience with activated carbon and with the comparable substance carbon black, the interlaboratory test sample “Carbon Black, Norit CN4” is known to be sufficiently homogeneous and stable within the testing time frame of the interlaboratory test (July 2011 to November 2012).

In addition, tests on homogeneity and stability were performed before sending out the test samples, during the testing period.

For the test on homogeneity the parameters

1. particle size (in μm)
2. mass content water (in %)
3. caloric value (in J/g)
4. relative self-ignition temperature according to Test EC A.16 (in $^{\circ}\text{C}$)

were considered.

For the test on stability, which was carried out from June 2011 to January 2012 monthly and then up to November 2012 every other month (in total 15 times), only the relative self-ignition temperature was analysed continuously; the other three parameters were only analysed once in June 2011.

The test results and reports are listed in Annex 9.1 (Test of the homogeneity at the beginning of TS 1) and Annex 9.2 (Test on stability during TS 1, TS 2 and TS 3).

Conclusion: The result of the test on homogeneity was that the sample material can be considered as suitably homogenous for the interlaboratory test regarding the analysed parameters.

However, contrary to our expectations the stability of the interlaboratory sample material regarding the relative self-ignition temperature is questionable because of the notable shift of the relative self-ignition temperature during the testing period. It could be observed that the relative self-ignition temperature of the sample material decreased from September 2011 to January 2012 and then notably increased from March 2012 on.

5 Procedure of the interlaboratory test

5.1 Organisation

The interlaboratory test was organized by the BAM Federal Institute for Materials Research and Testing, Berlin, in the frame of the interlaboratory test programme within the Center for Quality Assurance for Testing of Dangerous Goods and Hazardous Substances.

5.2 Participating laboratories

The interlaboratory test sample “Carbon Black, Norit CN4” together with test instructions and the laboratory data input form (→ Appendix 9.3.1 and 9.3.2), were distributed to 18 participating laboratories (→ Table 5-1).

Table 5-1: List of all 18 participating laboratories (17 laboratories submitted data)

Laboratory / Agency	Country
AQura GmbH	Germany
BAM Bundesanstalt für Materialforschung und –prüfung	Germany
BASF AG	Germany
Bayer Technology Services GmbH	Germany
Berufsgenossenschaft Nahrungsmittel und Gastgewerbe	Germany
Consilab Gesellschaft für Anlagensicherheit mbH	Germany
DEKRA EXAM GmbH	Germany
DMT GmbH & Co. KG	Germany
Dr. Krause GmbH	Germany
Fire Technical Institute Prague	Czech Republic
IBExU Institut für Sicherheitstechnik GmbH	Germany
INERIS	France
Institut für Arbeitsschutz der Deutschen Gesetzlichen Unfallversicherung (IFA)	Germany
Intertek Safety Testing Laboratory *	United Kingdom
Laboratorio Oficial J.M. Madariaga (LOM)	Spain
Siemens AG	Germany
Syngenta UK Ltd	United Kingdom
TNO Defence, Security and Safety	The Netherlands

*... Laboratory did not submit data during the testing period.

5.3 Test method

The interlaboratory test was performed with the test method DIN EN 15188:2007 “Determination of the spontaneous ignition behaviour of dust accumulations” (→ Table 5-2) with consideration of additional conditions / modifications (→ chapter 3).

Table 5-2: Test method of the interlaboratory test

Test method	Source
DIN EN 15188:2007 “Determination of the spontaneous ignition behaviour of dust accumulations”	Beuth Verlag, Berlin [1]

5.4 Steps of the interlaboratory test

The interlaboratory test was performed stepwise as a multi-level test (→ Table 5-3) on one interlaboratory test sample.

Test step 1 (TS 1) was obligatory for all participating laboratories.

Test steps 2 and 3 (TS 2 and TS 3) were optional and had depended on the results of previous test steps (TS 1 and/or TS 2).

Table 5-3: Scheduled interlaboratory test steps

Test step TS	Additional measures	Remark
1	No	Obligatory for all: Standard sample baskets with volumes ratios of 1 : 1.7 : 5 : 8 and optional, additional: sample basket with other test volumes
2	Exchange of equipment (e.g. ovens, temperature sensors) between laboratories	Optional, depending on results of test step 1 and if necessary for only some laboratories
3	Repeat tests under the conditions of test step 1	Optional in dependence on results of test steps 1 + 2

5.4.1 TS 1: Interlaboratory test instructions and laboratory data input

The test instructions for the interlaboratory test (→ Appendix 9.3.1 and 9.3.3) which were distributed together with the test sample focus on the specifics of the special conditions / modifications. The test instructions are more detailed than the current description of the method DIN EN 15188:2007 and included the following information:

1. The tests should always be performed by one and the same laboratory assistant, otherwise it shall be noted.
2. The sample shall be tested as delivered (do not dry, sieve, grind etc.).
3. The sample shall be mixed by hand-shaking the closed container (hobcock) for at least 10 second before filling the sample basket.
4. The sample shall be stored in the container which you have received from BAM over the whole testing period. The container shall keep close during storage.
5. The sample shall be shielded by using an additional screen installed in the oven. Use the test screen manufactured by BAM (if you have received these from BAM).
6. The temperature difference between „go“ and „no go“ shall be 2 K maximum.
7. The required standardized defined bulk density is: 460 kg/m³ (range 455...465 kg/m³)

5 cm sample basket (cube), 125 mL:	57.5 g (56.2 g ... 58.7 g)
6 cm sample basket (cube), 216 mL:	99.3 g (97.2 ... 101.5 g)
8.5 cm sample basket (cube), 614 mL:	282.5 g (276.3 ... 282.5 g)
10 cm sample basket (cube), 1000 mL:	460 g (450 ... 470 g).
8. **Standard test volumes:** 4 volumes to be tested (volumes = 5 cm, 6 cm, 8.5 cm and 10 cm cubes, which were manufactured / distributed by BAM). The sample baskets shall be double-walled. The minimum volume of the smallest basket is 100 mL; the minimum volume of the largest basket is 1000 mL.
9. **Additional test volumes** (optional): If possible please perform additional test with volumes smaller 100 mL and/or volumes larger 1000 mL
10. The oven should be pre-heated, if possible. If not possible, please make a note of this (data input form).
11. **Standardized temperature gradient inside the oven:**

Determine the temperature gradient within the shielding in a standard way.

 - 11.1 The oven shall be equipped with the shielding screen and a 1000 mL sample basket (empty, without sample).
 - 11.2 Adjust the oven temperature to 120 °C (temperature inside the screen as measured during the hot storage tests; arithmetic mean of both temperature sensors installed). Do not change the temperature setting of the oven for the whole test (step 11.3).
 - 11.3 Place a temperature sensor between the sample basket and the wall of the screen at a distance of 5 cm from the sample basket. Carry out this step using the same temperature sensor for the following positions:
 - (1) to the right of the sample basket
 - (2) to the left of the sample basket
 - (3) in front of the sample basket
 - (4) behind the sample basket
 - (5) above the sample basket
 - (6) below the sample basket

Determine the respective temperature when the temperature becomes stable.
12. **Moisture of sample (gravimetric test):** The moisture of the sample shall be determined at the beginning (first day) and the end (last day) of the whole hot storage tests. A sample shall be dried in an oven at a temperature of 105 °C for 24 hours. The sample mass shall be about 50 g. The sample shall be dried as a layer of a thickness of ≤ 5 mm.

Apart from this, the other details of the procedure were supposed to be applied as usual in the laboratory and in accordance with DIN EN 15188:2007 “Determination of the spontaneous ignition behaviour of dust accumulations”.

Laboratory specific parameters and test conditions were collected by means of laboratory data input forms (→ Appendix 9.3.2 and 9.3.4).

5.4.2 TS 2: Interlaboratory test instructions and laboratory data input (checking the volume of the sample baskets by BAM)

The pre-analysis of the results of TS 1 shows clearly (→ chapter 0) that it was necessary to check the sample baskets which the participants had used for the tests the previous year (TS 1).

For this purpose the participants were asked to send back the four sample baskets which they have used during TS 1, by March 2nd 2012 to BAM. After this BAM checked the volumes of all laboratories' sample baskets (TS 2) by the aid of glass beads (diameter ~ 0.3 mm). The sample baskets were sent back to the participants after this check.

5.4.3 TS 3: Interlaboratory test instructions and laboratory data input (repetition of TS 1)

On the basis of an analysis of the results of TS 1 and TS 2 it was decided to perform a 3rd interlaboratory test step (TS 3). The determination of the Tsi in TS 3 should be performed in the same way as in TS 1 (repetition).

The test instructions for the interlaboratory test (→ Appendix 9.5.1) were distributed together with the baskets which were measured by BAM in TS 2 (volumetry by glass beads by BAM). The test instructions included the following information:

1. The tests should always be performed by one and the same laboratory assistant as in the 1st test step, otherwise it shall be noted.
2. The sample shall be tested as delivered (do not dry, sieve, grind etc.).
3. The sample shall be mixed by hand-shaking the closed container (hobcock) for at least 10 seconds before filling the sample basket.
4. The sample shall be stored in the container which you have received from BAM over the whole testing period. The container shall keep close during storage.
5. The sample shall be shielded by using an additional screen installed in the oven in the same way as in the 1st test step.
6. **The temperature difference between „go“ and „no go“ shall be 2 K maximum.**
7. The required **standardized defined bulk density** is 450 kg/m³ to 470 kg/m³:

5 cm sample basket (cube, approx. 125 mL):	from 56.2 g to 58.7 g
6 cm sample basket (cube), approx. 216 mL):	from 97.2 g to 101.5 g
8,5 cm sample basket (cube), approx. 614 mL):	from 276.3 g to 288.6 g
10 cm sample basket (cube), approx. 1000 mL):	from 450 g to 470 g
8. **Standard test volumes:** 4 volumes to be tested (volumes = 5 cm, 6 cm, 8.5 cm and 10 cm cubes). Use the sample baskets which we have sent together with this instruction (which were checked by BAM in March 2012).
9. **Additional test volumes** (optional): If possible please perform additional test with volumes smaller 100 mL and/or volumes larger 1000 mL.
10. **Effective volume of the sample baskets:** Due to the manufacturing tolerances sample baskets and differences the volumetric the effective volume of each basket shall be determined by using **glass beads (diameter ~ 0.3 mm)**¹. Otherwise it shall be noted what material you have used.
11. The oven should be pre-heated, if possible. If not possible, please remark (data input form).
12. **Moisture of sample (gravimetric test):** The moisture of the sample shall be determined at the beginning (first day) and the end (last day) of the whole hot storage tests. A sample shall be dried in an oven at a temperature of 105 °C for 24 hours. The sample mass shall be about 50 g. The sample shall be dried as a layer of a thickness of ≤ 5 mm.

Apart from this, the other details of the procedure were supposed to be applied as usual in the laboratory and in accordance with DIN EN 15188:2007.

¹ The following suppliers were recommended in the interlaboratory test instruction of the TS 3 (→ Appendix 9.5.1):
<http://www.edmund-buehler.de/english/i-homogenisatoren-und-zellmuehlen.pml>
<http://www.sartorius-mechatronics.com/DE/en/index.htm> (product number: BBI-8541604)

Laboratory specific parameters and test conditions were collected with the aid of the laboratory data input forms (→ Appendix 9.5.2).

5.5 Time schedule

The following Table 5-4 shows the time schedule of the study.

Table 5-4: Time schedule of the interlaboratory test

Interlaboratory test step		Time period
Conceptual design		February 2010 to October 2010
Pre-announcement		September 2010 to October 2010
Test sample preparation, tests on homogeneity and stability		November 2010 to June 2011
Public announcement		May 2011
Order and registration		May 2011
Distribution of the test sample and the test instruction		June 2011
TS 1	Laboratory testing period	July 2011 to 16th January 2012 *
	Statistical evaluation	January 2012 to February 2012 **
TS 2	Laboratory testing period	February 2012 to March 2012
	Statistical evaluation	April 2012
TS 3	Laboratory testing period	April 2012 to 9 th November 2012 ***
	Statistical evaluation	November 2012 **
Final statistical evaluation and draft report		November 2012 **

*The testing period was prolonged from 5th September 2011 to 16th January 2012 because not all laboratories were able to perform the tests before 5th September 2011 (as originally arranged in June 2011).

**The statistical evaluation was postponed due to the prolonged testing period.

*** ... The testing period was prolonged from 3rd October 2012 to 9th November 2012 because not all laboratories were able to perform the tests before 3rd October 2012 (as originally arranged in April 2012).

6 Evaluation

6.1 Test results

6.1.1 Quantity of test results

Measurements were conducted by 17 out of 18 labs of the interlaboratory test.

3 of these 17 labs also submitted results of optional tests with additional sample baskets with other volumes than the standard nominal volumes of the baskets manufactured by BAM (125 mL, 216 mL, 614 mL, 1000 mL). This concerns the laboratories 228 and 840 in TS 1 and the laboratories 238 and 840 in TS 3.

Conclusion: Due to the high number of participating labs the quantity of the test results can be assumed to be reliable for the statistically evaluation.

6.1.2 Data check (inspection upon receipt)

The check of the submitted data (inspection upon receipt) includes the check of:

- **Completeness** of the data
 - e.g. missing data
- **Conformity**, check of irregular deviations from
 - the testing method DIN EN 15188:2007
 - the interlaboratory test instruction(s)
- **Plausibility**, check of the obvious incorrectness of the values of the submitted data
 - e.g. distorted data
- **Consistency**, check of real incorrectness of the values in the submitted data input form by means of the additionally submitted raw data
 - e.g. check of the T_{Si} measurements against the original temperature vs. time plots of the tests

Independent and stepwise data check

The data check was performed directly after the different interlaboratory test steps and before starting the statistical analysis by different experts independently of one another:

- **1. phase of data check**
by Peter Lüth (BAM) and by Martin Schmidt (BAM) (independently of one another) directly after the submission(s) of the data from the labs (TS 1, TS 3)
- **2. phase of data check**
by Kirstin Kunath (QuoData) directly before starting the statistical analysis (TS 1, TS 2, TS 3).

Improving the data quality

If necessary and possible, faulty data were corrected after consultation and in agreement with the respective lab, or, in case of missing data, the labs were asked to complete their data.

Important deviations

Individual laboratory deviations from the requirements of the method DIN EN 15188:2007 / interlaboratory test instructions, which may result in an incorrect statistical evaluation and thus in incorrect conclusions, are shown in Table 6-1.

Table 6-1: Result of the data check – important deviations from the method DIN EN 15188:2007 / interlaboratory test instructions

“0” + blue field = no deviation, “1”+ red field = deviation, “ + white field = no data
 (Note: The lab-no was not specified and the ranking of the column was changed for the sake of anonymity.)

Parameter	Sum	Lab-No.																			
		XXX XXX	XXX XXX	XXX XXX	XXX XXX	XXX XXX	XXX XXX	XXX XXX	XXX XXX	XXX XXX	XXX XXX										
Differences of the test setup inside the oven between TS 1 and TS 3	2	1																			
Oven not pre-heatet - TS 1	6	0	0	1	0	0	0	1	1	1	0	0	0	0	0	0	0	0	0	0	0
Oven not pre-heatet - TS 3	6	0	0	1	0	0	0	1	1	1	0	0	0	0	0	0	0	0	0	0	0
Oven temperature not stable (standard: required 1%) - TS 1	2	0	0	1	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
Oven temperature not stable (standard: required 1%) - TS 3	2	0	0	1	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
Different laboratory assistants within TS 1	1	0	1	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
Different laboratory assistants within TS 3	1	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	1
Different laboratory assistants between in TS 1 and TS 3	5	1	1	1	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	1
Wrong quantity of the test sample in the sample basket - TS 1	4	0	0	1	0	1	0	0	1	0	0	0	0	0	0	0	0	0	0	0	0
Wrong quantity of the test sample in the sample basket - TS 3	2	0	0	0	0	0	1	0	0	0	0	0	0	0	0	0	0	0	0	0	0
Use of only one thermocouple to detect the oven temperature (instead of two) - TS 1	1	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
Use of only one thermocouple to detect the oven temperature (instead of two) - TS 3	1	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
Unclear position of the thermocouples - TS 1	3	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
Unclear position of the thermocouples - TS 3	1	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
Position of the thermocouples (one/two) outside the screen - TS 1	2	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
Position of the thermocouples (one/two) outside the screen - TS 3	1	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
Temperature difference criterion (<2K) between „go“ and „no go“ exceeded - TS 1	3	0	0	1	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
Temperature difference criterion (<2K) between „go“ and „no go“ exceeded - TS 3	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
Stipulated rounding down procedure neglected - TS 1	12	1	1	1	0	0	1	0	1	0	0	1	1	1	1	1	1	1	1	1	1
Stipulated rounding down procedure neglected - TS 3	7	1	1	0	0	1	0	1	0	1	0	1	0	0	0	0	0	0	0	0	0
Incorrect Tsi calculation (Mean between „go“ „no go“, wrong calibration, by „go“-value or other mistake) - TS 1	6	0	1	0	0	0	0	1	0	0	0	1	0	1	1	1	1	1	1	1	0
Incorrect Tsi calculation (Mean between „go“ „no go“, wrong calibration, by „go“-value or other mistake) - TS 3	3	0	1	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
Unclear raw data (temperature curves) - TS 1	3	0	1	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
Unclear raw data (temperature curves) - TS 3	2	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
Wrong data input form - TS 3	6	0	1	1	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
Sum	3	8	11	0	3	2	3	2	5	4	8	6	10	4	8	3	2				

The following important deviations have been observed.

- **Differences of the test setup inside the oven between TS 1 and TS 3**
 - It was stipulated in the instruction of TS 3 (→ appendix 9.5.1) that TS 3 should be performed in the same way as TS 1 (repetition).
 - However, two laboratories have changed the test setup inside the oven in TS 3 and thus not performed the tests of TS 3 in the same way (conditions) as in TS 1.
- **Pre-heating of the oven**
 - It was stipulated in the instruction of TS 1 (→ appendix 9.3.1) that the oven should be pre-heated, if possible. However, six laboratories have not pre-heated the oven.
- **Stability of the oven temperature**
 - The method DIN EN 15188:2007, chapter 3.2 requires that the oven temperature shall be stable within a range of ± 1 % of the respective oven temperature. Two laboratories have not reached the required stability level.
- **Changes of the laboratory assistant within TS 1 and within TS 3 and between TS 1 and TS 3**
 - It was stipulated in the instruction of TS 3 (→ appendix 9.5.1) that TS 3 should always be performed by one and the same laboratory assistant as in TS 1, otherwise it shall be noted. However, five laboratories have changed the laboratory assistant test setup between TS 1 and TS 3. Furthermore one laboratory has noted that more than one assistant performed tests in TS 1. A further laboratory has noted that more than one assistant have performed the tests in TS 3.
- **Quantity of the test sample in the sample basket**
 - Special ranges of the quantity of the test sample in the sample baskets were stipulated in the instructions of TS 1 (→ appendix 9.3.1 and 9.3.3). Four laboratories in TS 1 and two laboratories in TS 3 have sent results with quantities outside these ranges.
- **Use of only one thermocouple to detect the oven temperature**
 - The method DIN EN 15188:2007, chapter 2.2 requires the use of two thermocouples to determine the temperature of the oven. One laboratory has used only 1 thermocouple.
- **Unclear position of the thermocouples**
 - The position of the thermocouples was unclear in the data of three laboratories in the TS 1 and in one laboratory in TS 3.
- **Position of the thermocouples outside the screen**
 - The position of the thermocouples to detect the oven temperature should be between the sample basket and the screen. However, the position of one or both thermocouples were outside the screen in two laboratories in TS 1 and in one laboratory in TS 3.
- **Temperature difference between „go“ and „no go“**
 - It was stipulated in the instruction of TS 1 (→ appendix 9.3.1) that the temperature difference between „go“ and „no go“ shall be 2 K maximum. It could be observed that almost all laboratories have not reached this level. Three laboratories were requested to reduce the difference between „go“ and „no go“ due to a considerable deviation.

- **T_{SI} measurements – rounding down to the nearest degree**
 - The method DIN EN 15188:2007, chapter 5.2 requires that the result of T_{SI} measurements should be rounded down to the nearest degree. Twelve laboratories in TS 1 and seven laboratories in TS 3 have neglected this special rule and have not sent correct values. Five of these laboratories have neglected the required rounding procedure in both steps (TS 1 and TS 3).
- **T_{SI} calculation**
 - Six laboratories in TS 1 and three laboratories in TS 3 have not calculated the T_{SI} measurements in a correct way. Three of these laboratories have done the same calculating mistake in both steps (TS 1 and TS 3).
- **Unclear raw data (temperature curves)**
 - The raw data of the temperature curves was not clear in the data of three laboratories in TS 1 and in two laboratories in TS 3.

Conclusion: Deviations from the requirements of the method DIN EN 15188:2007 and / or the interlaboratory test instructions, which may result in an incorrect statistical evaluation and thus in incorrect conclusions, were identified. These deviations must be taken into account in the following statistical evaluation. The checked test results can be assessed as a sufficient basis for the statistical evaluation and for reliable conclusions.

6.1.3 Specifics and limitations of the usability of the test results for the statistical evaluation

In TS 3, not all labs determined their sample baskets' volumes by using glass beads. This concerns the following four labs:

- 118 (tap water)
- 229 (coal of known density was used to calculate the volume of sample baskets)
- 233 (Pulverulent alumina)
- 908 (quartz sand).

It has to be noted that laboratory 277 carried out their analyses twice in each test step. The conditions between the replicates within one test step are not comparable to the conditions between the two test steps (TS 1 and TS 3). In order to obtain a repeatability standard deviation for the extrapolated self-ignition temperature T_{SI} of a certain storage volume between both test steps consistently across all laboratories, the test step-specific extrapolated T_{SI} of laboratory 277 used for the following statistical analysis is given by the laboratory's mean value of both available T_{SI} per test step.

Furthermore it has to be noted that the laboratories 154, 238 and 251 do not exhibit acceptable repeatability conditions between TS 1 and TS 3:

- In laboratory 154 the sheet metal of the mesh wire screen (→ Figure 3-1 and Test instruction; chapter 9.3.1) was removed to enhance the heat transport. In addition, the bottom plate of the sample holder was replaced by two flat bars. Hence, the air flow was changed in TS 3 compared to TS 1.
- In laboratory 238, the sample baskets used in TS 1 were replaced by new sample baskets in TS 3.
- In laboratory 251, the positions of the sensors differ in TS 3 compared to TS 1.

Conclusion:

Specifics and limitations concerning the usability of the submitted test results were identified and must be taken into account when performing the statistical analysis. The quantity of the submitted results can be assessed as sufficient for a statistical analysis.

6.2 Scientific-methodological basis for the statistical evaluation

Scientific basis of T_{SI} measurements

According to the modified method DIN EN 15188:2007 the labs determine T_{SI} measurements for the differently-sized sample baskets. On this basis a functional relationship between the T_{SI} measurement and the samples volumes will be calculated (Pseudo-Arrhenius Plot, → DIN EN 15188:2007, chapter 5.2). In this Pseudo-Arrhenius Plot the x value is given by the reciprocal self-ignition temperature (in 1/K), i.e. $x = 1/T_{SI}$, and the y value is given by $\lg(V/A)$, where V is the volume and A the surface of the sample basket. A linear relationship can be assumed between x and y. Thus the T_{SI} of large storage volumes can be calculated by extrapolation.

Assessment of the performance of the modified method on basis of the extrapolated T_{SI} for storage volumes (27 m³, 100 m³, 500 m³, 1000 m³)

The performance of the modified method DIN EN 15188 was assessed on basis of the extrapolated T_{SI} for storage volumes (27 m³, 100 m³, 500 m³, 1000 m³).

For this purpose the T_{SI} of TS 1 and TS 3 of four different typically large storage volumes (27 m³, 100 m³, 500 m³, 1000 m³) were calculated by extrapolation. These extrapolated T_{SI} were used for the further statistical evaluation and to assess the modified method 15188.

The extrapolated T_{SI} of TS 1 and the extrapolated T_{SI} of TS 3 will be regarded as replicates. Thus it is possible to determine not only a reproducibility precision but also a repeatability precision of the modified method.

6.3 Evaluation steps

Pre-analysis for preparing a suitable data basis for calculation of the precision parameters of the modified method DIN EN 15188

In order to get a suitable data basis the following steps are carried out before calculating the final precision parameters of the modified method DIN EN 15188 (→ chapter 6.8)

- (1) *Quality of the regression curves of Pseudo-Arrhenius plot and compliance to the method and to repeatability conditions (→ chapter 6.4)*

The compliance to the test method (requirements) and to the repeatability conditions of the labs during the tests of TS 1 and TS 3 is of crucial importance to the statistical evaluation. Based on the T_{Si} obtained for the four sample baskets, the quality of the linear regression should be assessed and compared between TS 1 and TS 3. If there are no noticeable differences within one laboratory (at least for the majority of laboratories), the present data can be used for obtaining statistically reliable results.

- (2) *Determining a suitable correction of data due to instability of sample material (→ chapter 6.5)*

Due to the identified instability of sample material (→ chapter 4.2), a correction of the provided T_{Si} measurements is necessary. This correction is necessary to ensure comparability between laboratories as well between TS 1 and TS 3.

- (3) *Checking the effect of rounding and non-rounding of the oven temperature on the extrapolated T_{Si} regarding DIN EN 15188:2007 (→ chapter 6.6)*

In method DIN EN 15188:2007, for obtaining the actual T_{Si} measurements the oven temperature should be rounded down to the nearest degree, i.e. measurements equal to e.g. 142.01 and 142.99 will be rounded likewise to 142. It will be discussed whether there are significant differences regarding extrapolated T_{Si} for large volumes.

- (4) *Checking the effect of volumetry (→ chapter 6.7)*

According to method DIN EN 15188:2007 it is sufficient to use the given nominal volumes of the sample baskets to derive a functional relationship of the T_{Si} measurements. However, in this interlaboratory test, the nominal volumes are often smaller than the actual effective volume. Furthermore, there are different methods to determine the effective volume precisely. So, it is recommended to use a “standardized” method for volumetry.

- (5) *Checking other influence (disturbing) factors (→ chapter 6.8)*

To be sure that no other factors influence the accuracy and precision of the extrapolated T_{Si} for large volumes, selected lab-specific method settings were analysed by an additional exploratory data analysis.

Calculation of the precision parameters of the modified method DIN EN 15188

Based on the pre-analysis for preparing a suitable data basis, as described above, the mean across laboratories as well as the precision data of the modified method DIN EN 15188 will be derived in chapter 6.9.

Although the extrapolated T_{Si} cannot be included for calculating the final precision data of the modified method DIN EN 15188 for all laboratories, the assessment of laboratory performance is carried out for all laboratories (→ chapter 6.10).

6.4 Quality of the regression curves of Pseudo-Arrhenius plot and compliance to the method and to repeatability conditions

The curves of the Pseudo-Arrhenius plot of all labs are shown in Figure 6-1 (separately for TS 1 and TS 3). For the sake of overall clarity, the curves of the labs are not differentiated by colours.

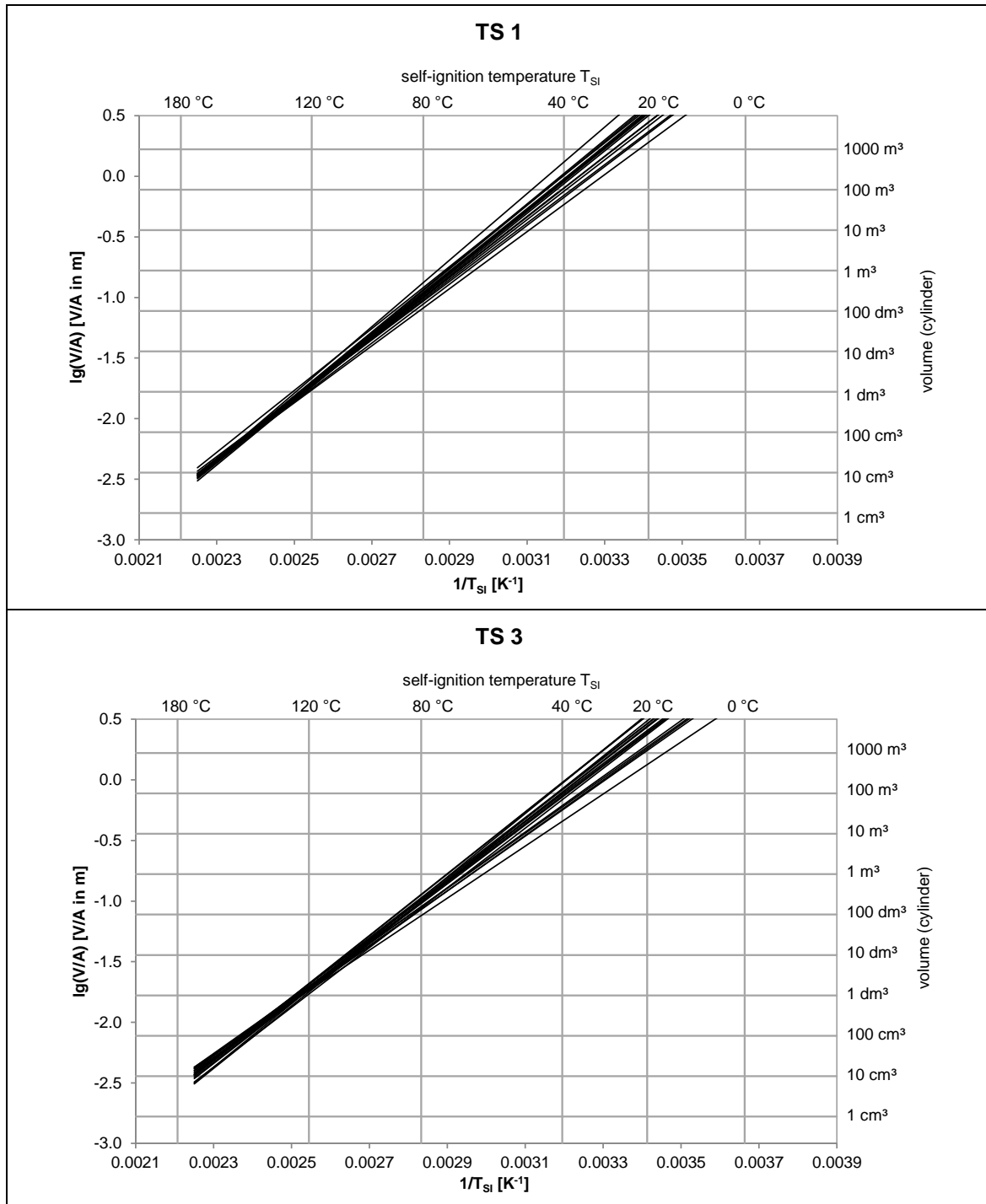


Figure 6-1: Pseudo-Arrhenius plot of self-ignition temperatures of “Carbon Black, Norit CN4” of TS 1 and TS 3 based on original T_{si} measurements and the reference volume

Residual standard deviation (RSD) of the curves of the Pseudo-Arrhenius plot of self-ignition temperatures

The compliance to the test method (requirements) and to the repeatability conditions of the labs during the tests of TS 1 and TS 3 is of crucial importance to the statistical evaluation.

For this purpose the residual standard deviation (RSD)² of the curves of the Pseudo-Arrhenius plot of self-ignition temperatures was determined for each lab. The RSD can be used as an indicator for the quality level of the compliance to the method and to repeatability conditions.

Please note: The T_{SI} measurements of laboratory 238 cannot be used for this analysis, because the reference volume is not known for all used sample baskets in TS 1 and TS 3.

The RSD of the lab of TS 1 and TS 3 is shown in the Figure 6-2.

In case of identical RSD in TS 1 and TS 3, a data point is located directly on the grey bisecting line. Identical RSD in TS 1 and TS 3 can be interpreted as high repeatability compliance. This is very nearly the case for laboratories 177 and 840.

If the lab-specific data point lies in the yellow segment in Figure 6-2 then there is a significant difference of the residual standard deviations between TS 1 and TS 3 to the significance level of 5 %. This is the case for the three laboratories 118, 201 and 233: their Pseudo-Arrhenius plot is significantly poorer in TS 3 than in TS 1.

The data point of laboratory 233 lies very close to the red segment, which indicates a significant difference between TS 1 and TS 3 regarding the RSD to the 1 % significance level.

² By the so-called residual standard deviation (RSD), the quality of the linear regression can be assessed, similar to the coefficient of determination.

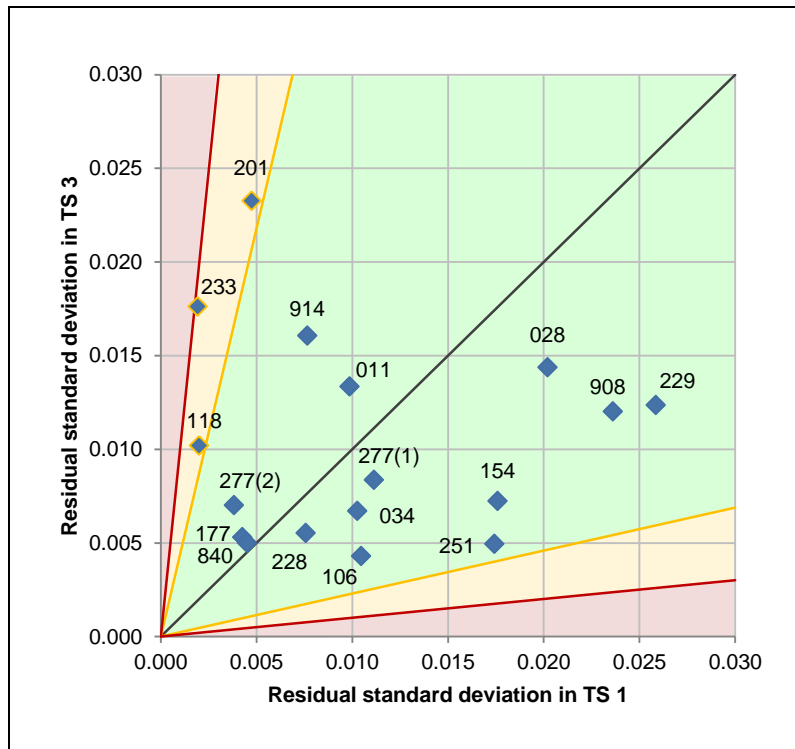


Figure 6-2: Residual standard deviations RSD of TS 1 and TS 3 based on original T_{Si} measurements and the reference volume of the lab (lab number) and area of significant differences

Conclusion: The majority of laboratories exhibit a constant performance in TS 1 and TS 3 regarding the quality of the curves of the Pseudo-Arrhenius plot of self-ignition temperatures. Thus a sufficiently high level of compliance to the method and to repeatability conditions can be concluded. Summarizing: the majority of the data can be used for obtaining reliable statistically results. However, there is a high variability between laboratories regarding the quality of the curves of the Pseudo-Arrhenius plot.

Lab-specific slopes of the curves of the Pseudo-Arrhenius plot of self-ignition temperatures

The lab-specific slopes of the curves of the Pseudo-Arrhenius plot together with their expanded uncertainty for both TS 1 and TS 3 are shown in Figure 6-3.

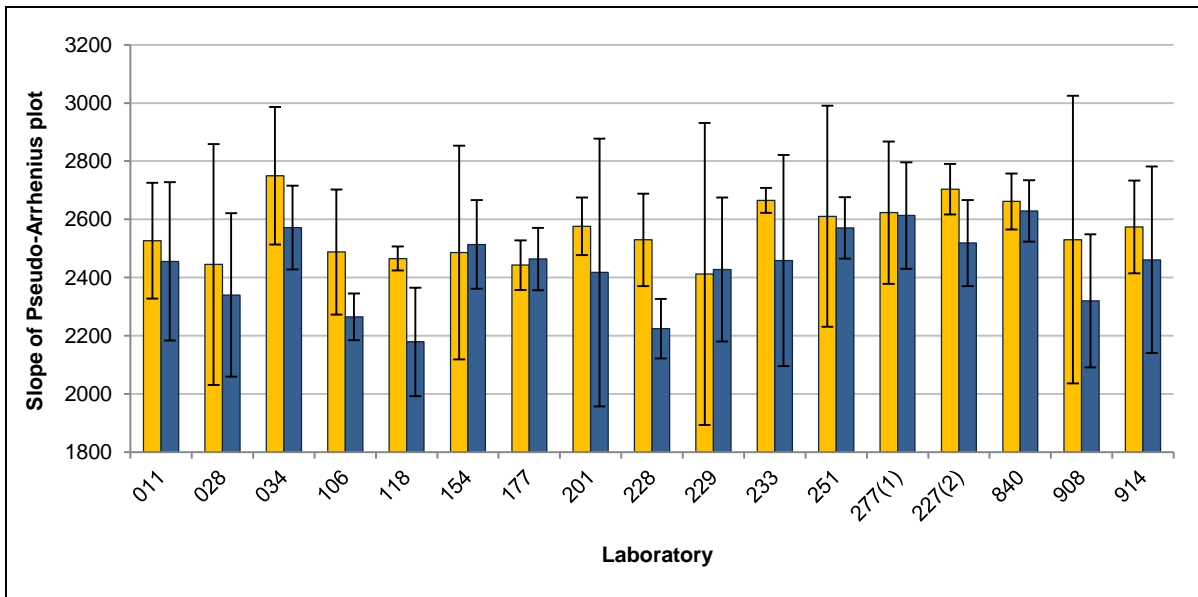


Figure 6-3: Comparison of slopes of lab-specific curves of the Pseudo-Arrhenius plot between TS 1 (yellow bars) and TS 3 (blue bars) based on original T_{Si} measurements and the reference volume

It is noticeable that the slope for almost all laboratories is higher in TS 1 than in TS 3. This fact would imply that the older the sample material the lower the apparent activation energy E and, in consequence, the self-ignition temperature T_{Si} for large volumes. This is not what one would have expected. Even if this time-dependent effect is not significant, it can be assumed that the substance is not completely stable over the whole period of the interlaboratory test.

Conclusion: It can be assumed that the interlaboratory test sample was not sufficiently stable regarding the self-ignition temperature during the whole period of the interlaboratory test.

6.5 Correction of T_{SI} measurements due to instability of the interlaboratory test sample

The results of the test on stability indicate that the interlaboratory test sample cannot be considered as sufficiently stable regarding the *relative* self-ignition temperature (\rightarrow chapter 4.2). Furthermore the slopes of lab-specific curves of the Pseudo Arrhenius plot between TS 1 (yellow bars) and TS 3 (blue bars) indicate the instability of the interlaboratory test sample regarding the self-ignition behavior / T_{SI} measurements (\rightarrow chapter 6.4).

Conclusion: Thus it is necessary to correct the T_{SI} measurements by means of a statistical-computational elimination of the effect of the instability of the interlaboratory test sample. In order to derive a time-dependent and lab-comprehensive correction factor for the T_{SI} measurements, the lab-specific increases of T_{SI} measurements from TS 1 to TS 3 – depending on the date of analysis – are considered.

6.5.1 Preconditions

The relative deviations of the lab-specific T_{SI} measurements from TS 3 compared to TS 1 ($=\Delta T_{SI}$ [%]) depending on the reference volume, but for each nominal volume separately, are displayed in Figure 6-4. It can be seen that for a given nominal volume of the sample basket the effective volume determined in TS 2 does not affect the T_{SI} measurements (neither downwards nor upwards). Therefore, the time-dependent correction factor will be determined on the basis of the nominal volumes.

A single correction factor for all nominal volumes cannot be recommended because the mean relative deviations behave differently between different nominal volumes (also \rightarrow Table 6-2).

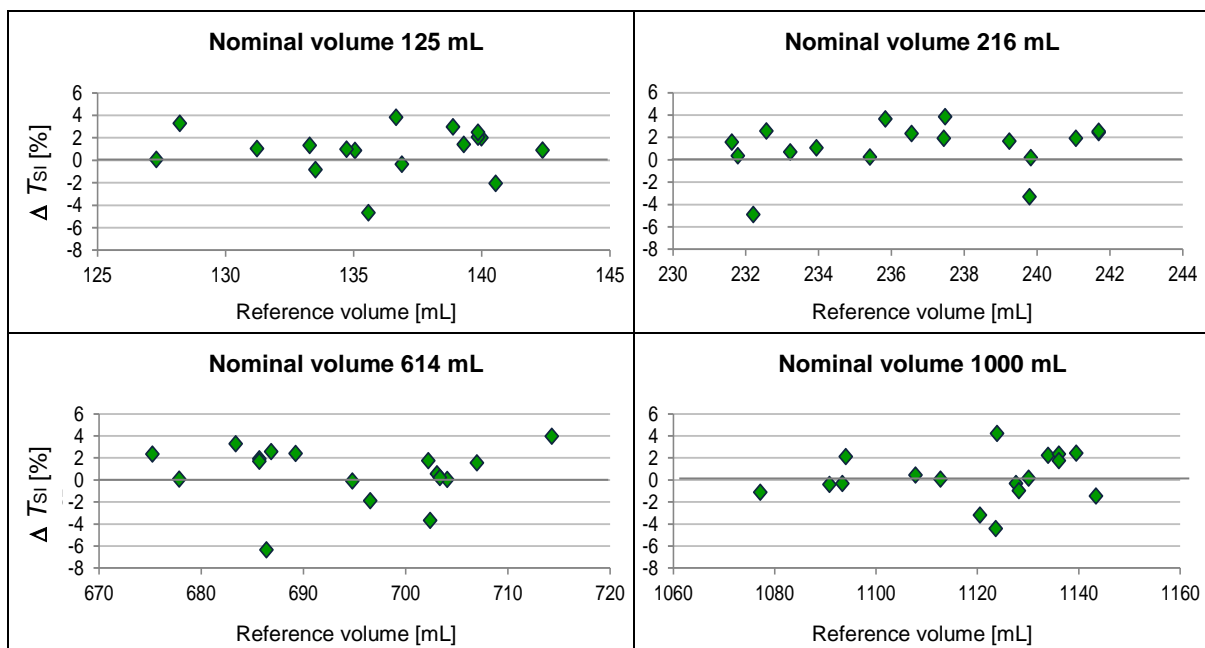


Figure 6-4: Relative deviations of the lab-specific T_{SI} measurements from TS 3 compared to TS 1 ($=\Delta T_{SI}$ [%]) for different nominal volumes

For the determination of the time-dependent correction factor only data from laboratories were considered if the following conditions were fulfilled:

- (A) Labs which performed their analyses under acceptable repeatability conditions (between TS 1 and TS 3)
- (B) Labs where the results exhibit a maximum temperature difference between ignition and no ignition of 3 K.

Condition (A):

Condition (A) is not fulfilled by the laboratories 154, 238 and 251. In lab 154 the sheet metal of the mesh wire screen (→ Figure 3-1 and Test instructions in chapter 9.3.1) was removed to enhance the heat transport. In addition, the bottom plate of the sample holder was replaced by two flat bars. Hence, the air flow was changed in TS 3 compared to TS 1. In laboratory 238, the sample baskets used in TS 1 were replaced by new sample baskets in TS 3. In laboratory 251, the positions of the sensors differ in TS 3 compared to TS 1.

Condition (B):

The test instructions stipulated that the temperature difference between ignition (“go”) and no ignition (“no go”) should not be higher than 2 K. In total – both TS 1 and TS 3 considered together – in approximately 45 % of all determined T_{SI} the limit of 2 K was exceeded. To ensure an acceptable accuracy of the obtained self-ignition temperatures it was decided to eliminate data with a temperature difference of more than 3 K. By this procedure only 5 % of all determined T_{SI} (laboratories 154, 238 and 251 unconsidered) have been eliminated from the data set, i.e. the following data have been eliminated (also → Figure 6-5):

- TS 1:
 - Lab 034, nominal volume 125 mL
 - Lab 908, nominal volume 216 mL
 - Lab 229, nominal volume 1000 mL
- TS 3:
 - Lab 229, nominal volume 125 mL
 - Lab 908, nominal volume 125 mL
 - Lab 908, nominal volume 1000 mL.

In order to determine the time-dependent correction factor for the T_{SI} measurements, the data of one laboratory and one nominal volume can only be considered, if valid” T_{SI} measurements are available for both TS 1 and TS 3 “. So, if condition (B) is not fulfilled in TS 1 the respective value of TS 3 cannot be considered and vice versa.

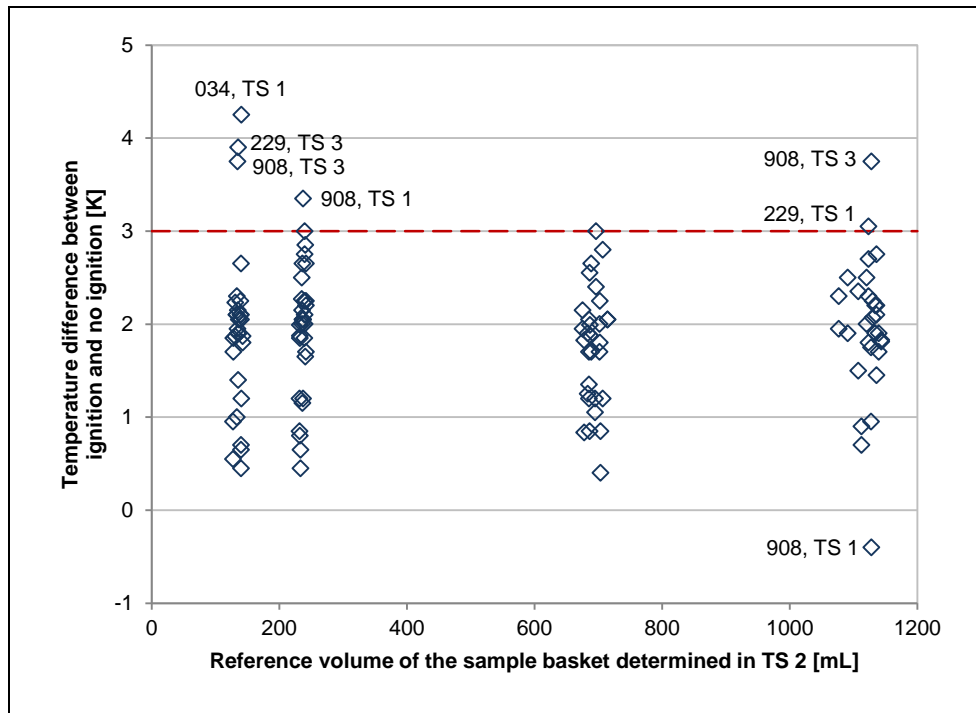


Figure 6-5: Temperature difference between ignition and no ignition of the T_{Si} determined in TS 1 and TS 3

Note for Figure 6-5: In one case the temperature difference exhibits a negative value: laboratory 908 in TS 1 for the sample basket of 1000 mL (nominal volume). This negative value was rechecked and confirmed by laboratory 908. However, a comparatively low negative temperature difference is not unreasonable, because each measurand is generally affected by random errors.

6.5.2 Calculating the time-dependent and lab-comprehensive correction factor for the T_{Si} measurements

The lab-specific intercepts (represents the level in vertical direction) and slopes regarding the change of T_{Si} from TS 1 to TS 3 depending on the date of analysis were determined by the data pairs which fulfil conditions (A) and (B) (→ chapter 6.5.1.). Then the lab-comprehensive function of the change of T_{Si} depending on the date of analysis was calculated. For this purpose a robust method according to DIN 38402-45 [3] (=ISO/TS 20612) was applied.³

In Table 6-2, the lab-comprehensive robust mean changes are given separately for each nominal volume. The right column contains the absolute and relative change of T_{Si} from TS 1 (earliest date of analysis; 21.07.2011) to TS 3 (latest date of analysis; 05.11.2012). It can be seen that for the nominal volumes 125 mL, 216 mL and 614 mL the rate of change is almost the same with approximately 2.5 %. For the nominal volume of 1000 mL, a lab-comprehensive “robust mean” change of 0.8 % is stated.

³ This statistical method doesn't require a special outlier examination. The robust mean values of the lab-specific intercepts as well of the slopes were determined by the method of the so-called Hampel estimator.

Table 6-2: Lab-comprehensive “robust mean” changes of the T_{Si} depending on the date of analysis and depending on the nominal volume of sample baskets

Nominal volume of sample baskets	lab-comprehensive “robust mean” change = <i>intercept</i> + <i>slope</i> *Date		ΔT_{Si} from 21.07.2011 to 05.11.2012
	intercept	slope	
125 mL	-158	0.0074	3.5 K (= 2.4 %)
216 mL	-175	0.0077	3.6 K (= 2.7 %)
614 mL	-145	0.0067	3.1 K (= 2.5 %)
1000 mL	40	0.0020	1.0 K (= 0.8 %)

Conclusion: For the further statistical evaluation it is mandatory that all T_{Si} measurements obtained in TS 1 and TS 3 by the labs must be corrected by the statistical operator (QuoData GmbH) using the slope depending on the nominal volume given in Table 6-2 according to the following equation:

$$\text{'corrected } T_{Si} \text{ measurement'} = \text{'original } T_{Si} \text{ measurement'} - \text{'slope'} \cdot (\text{'date of analysis'} - \text{'reference date'})$$

As “reference date” the earliest date of laboratory’s analyses was chosen: 21.07.2011.

6.5.3 Time-dependent corrected T_{Si} measurements

The lab-specific changes of uncorrected (“original”) and time-dependent corrected T_{Si} measurements from TS 1 and TS 3 as well as the lab-comprehensive “robust mean” change are displayed in the following Figure 6-6.

Laboratories which do not fulfil conditions (A) or (B) (→ chapter 6.5.1) are mentioned under the nominal volume. (Note that the T_{Si} measurements of these laboratories are not involved for determining the time-dependent correction factor and are also not displayed in the respective figures.)

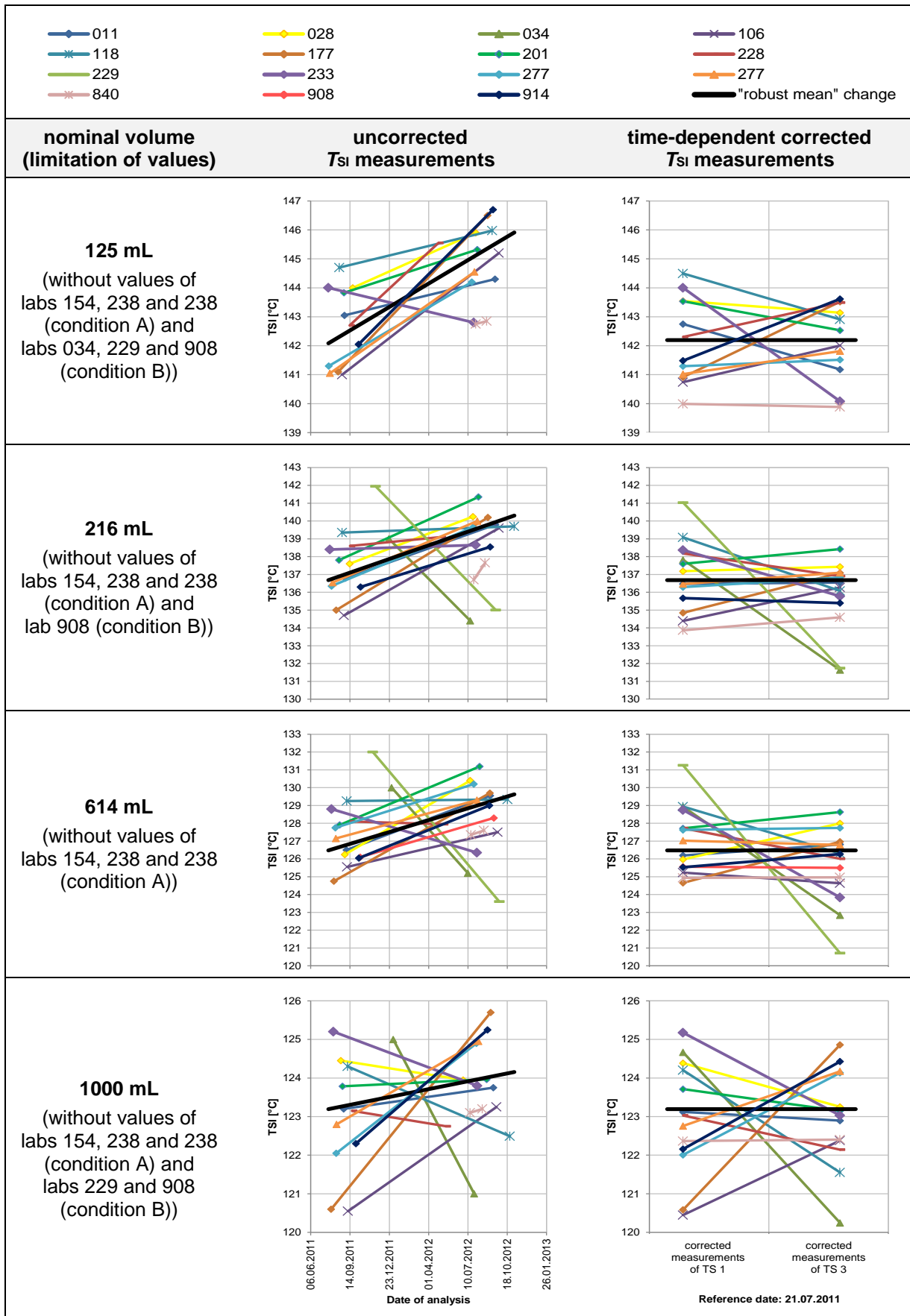


Figure 6-6: Change of self-ignition temperatures depending on the date of analysis
 left charts: uncorrected T_{Si}
 right charts: time-dependent corrected T_{Si}

Conclusion: Figure 6-6 demonstrates that applying a robust method to obtain lab-comprehensive “robust mean” change of the T_{Si} is quite appropriate for the time-dependent correction of the results (because of the instability of the sample) even if some outlying lab-specific changes, e.g. laboratory 233 for the nominal volume of 125 mL, can be observed.

The lab-specific slopes of the Pseudo-Arrhenius plots obtained by time-dependent corrected T_{Si} depending on the reference volume are demonstrated in Figure 6-7.

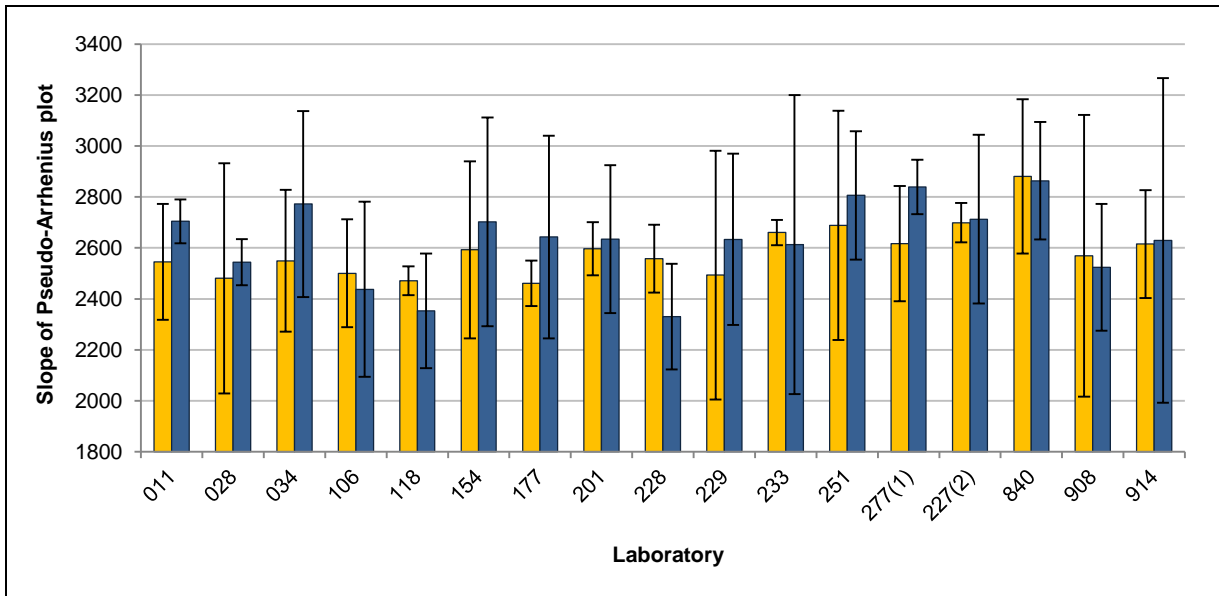


Figure 6-7: Comparison of slopes of lab-specific curves of the Pseudo-Arrhenius plot between TS 1 (yellow) and TS 3 (blue) based on time-dependent corrected T_{Si} measurements (“reference date” 21.07.2011) and the reference volume

Conclusion: Figure 6-7 demonstrates that the slopes of the lab-specific curves of the Pseudo-Arrhenius plot are no longer always higher in TS 1 as in TS 3, once the time-dependent correction of T_{Si} measurements has been applied (→ Figure 6-3). The time-dependent correction procedure of T_{Si} measurements can be assessed as suitable.

The curves of the Pseudo-Arrhenius plot of all laboratories based on time-dependent corrected T_{Si} measurements and the reference volume are shown in Figure 6-8, in which – for the sake of clarity – the laboratories are not differentiated by colour.

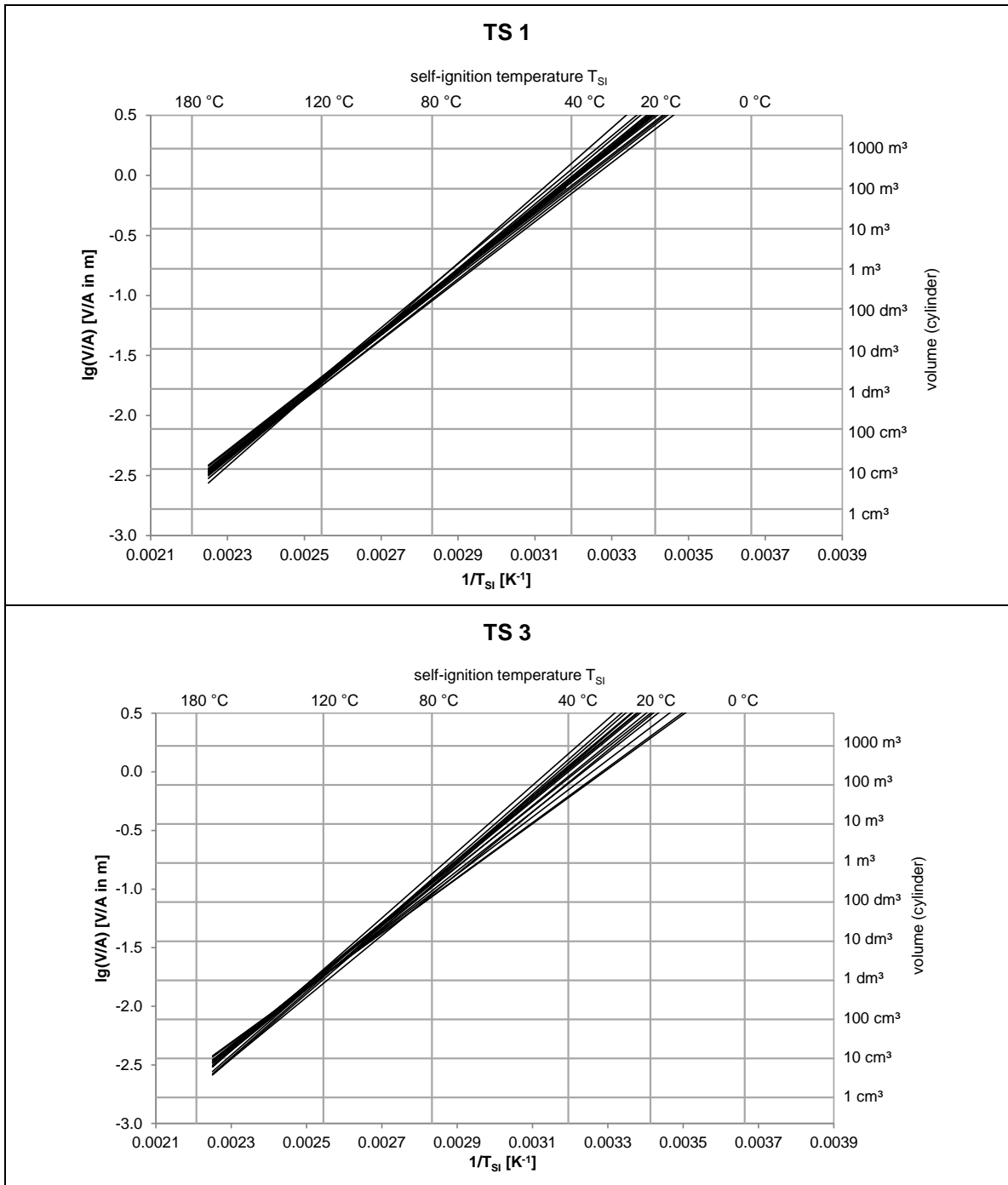


Figure 6-8: Pseudo-Arrhenius plot of self-ignition temperatures of “Carbon Black, Norit CN4” based on time-dependent corrected T_{Si} measurements (“reference date” 21.07.2011) and the reference volume (plot above: test results of TS 1; plot below: test results of TS 3)

Conclusion: It can be seen in Figure 6-8 that the regression curves of the labs scatter slightly more after the time-dependent correction than before the correction (→ Figure 6-1). Nevertheless the time-dependent correction procedure can be assessed as suitable because the effect of the increasing of the scattering is not significant and can be assessed as negligible.

6.6 Effect of rounding down and non-rounding of the oven temperature on the extrapolated T_{SI}

The following special rounding procedure is stipulated in method DIN EN 15188:2007:

- “The T_{SI} measurements should be rounded down to the nearest degree, i.e. measurements equal to e.g. 142.01 and 142.99 will be rounded likewise to 142.”

The effect of the rounding in comparison to non-rounding of the laboratory's T_{SI} measurements on the extrapolation to larger storage volumes (Pseudo-Arrhenius plot) was established (→ Table 6-3) on the basis of the following values / under the following conditions:

- (1) The time-dependent corrected T_{SI} in accordance with chapter 6.5 used as oven temperature (i.e. the “original” oven temperatures submitted by the labs were not used).
- (2) The linear regression for extrapolating the T_{SI} for large storage volumes based on the reference volume.
- (3) Only T_{SI} measurements of laboratories with acceptable repeatability conditions were involved.
- (4) Only T_{SI} measurements based on a temperature difference between ignition and no ignition of 3 K maximum were considered.
- (5) In order to establish a statistically balanced data base, the data of one laboratory were considered only if for both TS 1 and TS 3 and all four nominal volumes, in each case “valid” T_{SI} measurements are available. As an example: if condition (4) is not fulfilled in TS 1 the respective value of TS 3 cannot be considered and vice versa.

Conditions (3) and (4) are equivalent to conditions (A) and (B), resp., in chapter 6.5, i.e. the T_{SI} measurements of the following laboratories cannot be involved for determining the effect of rounding vs. non-rounding of oven temperature:

- corresponding to condition (3): lab 154, lab 238 and lab 251
- corresponding to condition (4) and (5): lab 034, lab 229 and lab 908.

Hence, time-dependent corrected T_{SI} measurements (“reference date” 21.07.2011) of 11 laboratories can be used for the effect analysis in this chapter. Therefore, the robust mean values using the Hampel estimator were determined for both extrapolated T_{SI} based on rounded oven temperature and extrapolated T_{SI} based on non-rounded oven temperature. These values together with the expanded uncertainty of the respective robust mean value are given in the following Table 6-3.

Table 6-3: Extrapolated T_{SI} of different storage volumes in dependence of the rounding procedure

Storage volume	Robust mean ± expanded uncertainty of extrapolated T_{SI} based on ...	
	... rounded down oven temperature	... non-rounded oven temperature
27 m ³	49.8 ± 1.9	50.3 ± 2.2
100 m ³	42.2 ± 1.9	42.8 ± 2.3
500 m ³	33.4 ± 2.0	34.0 ± 2.4
1000 m ³	29.8 ± 2.1	30.4 ± 2.6

Based on the comparison of the robust mean values of extrapolated T_{SI} and their expanded uncertainty ($= 2 \times$ standard error of robust mean value) in Table 6-6, no significant differences of the extrapolated T_{SI} for large volumes between rounded and non-rounded oven temperatures can be identified.

As can be demonstrated by Figure 6-9, if extrapolating from the laboratory's sample baskets to larger volumes the usage of non-rounded oven temperatures in relation to the rounded (down) values can lead to both lower and higher extrapolated T_{SI} values.

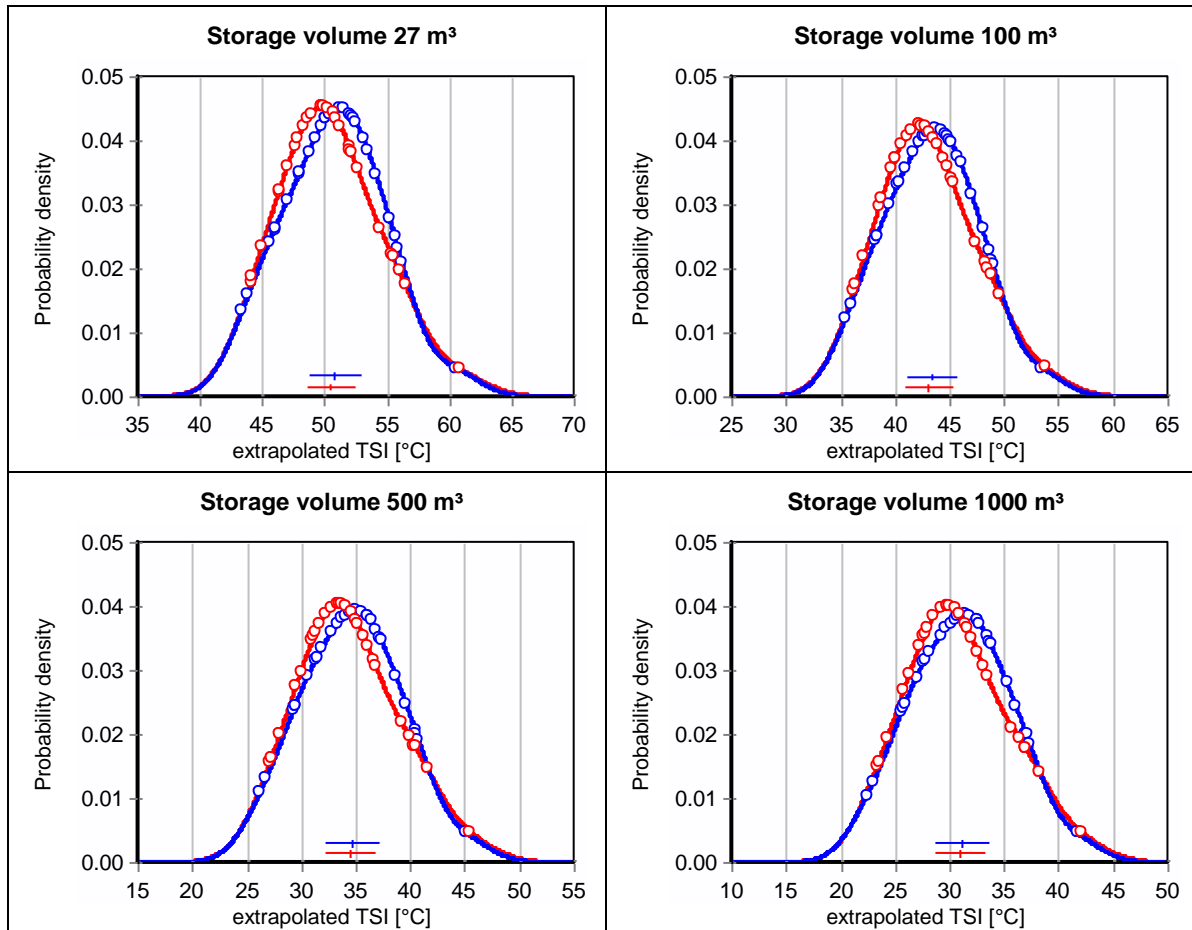


Figure 6-9: Comparison of kernel density estimations of extrapolated T_{SI} for storage volumes of 27 m³, 100 m³, 500 m³ and 1000 m³ based on rounded down (red) and non-rounded (blue) oven temperatures

Conclusion: The stipulated rounding down procedure as required in DIN EN 15188:2007 would not be appropriate for example to get “safer or better” extrapolated T_{SI} values for larger volumes. To avoid mistakes and misinterpretations it is recommended to use only the non-rounded oven temperature T_{SI} measurements in order to calculate extrapolated T_{SI} for large volumes.

6.7 Effect of volumetry

BAM manufactured sample baskets with nominal volumes of 125 mL, 216 mL, 614 mL and 1000 mL, which were made available to the laboratories during TS 1 of the interlaboratory test. Since the effective volumes of the sample baskets differ more or less from the respective nominal volume due to manufacturing tolerances, the laboratories were asked in TS 1 to determine the effective volumes of each basket by their volumetric methods (in the following, the results of the volumetry by the lab will be referred to as “TS 1 volume”, → chapter 1 Glossary). For this purpose it was recommended to use material of an invariant bulk density to do the volumetric analyses (not dependent on degree of compaction); for example table salt or fine sand (→ appendix 9.3.3 Additionally test instructions).

The deviations of the “TS 1 volume” from the nominal volume of each sample basket (provided by BAM) are demonstrated in Figure 6-10.

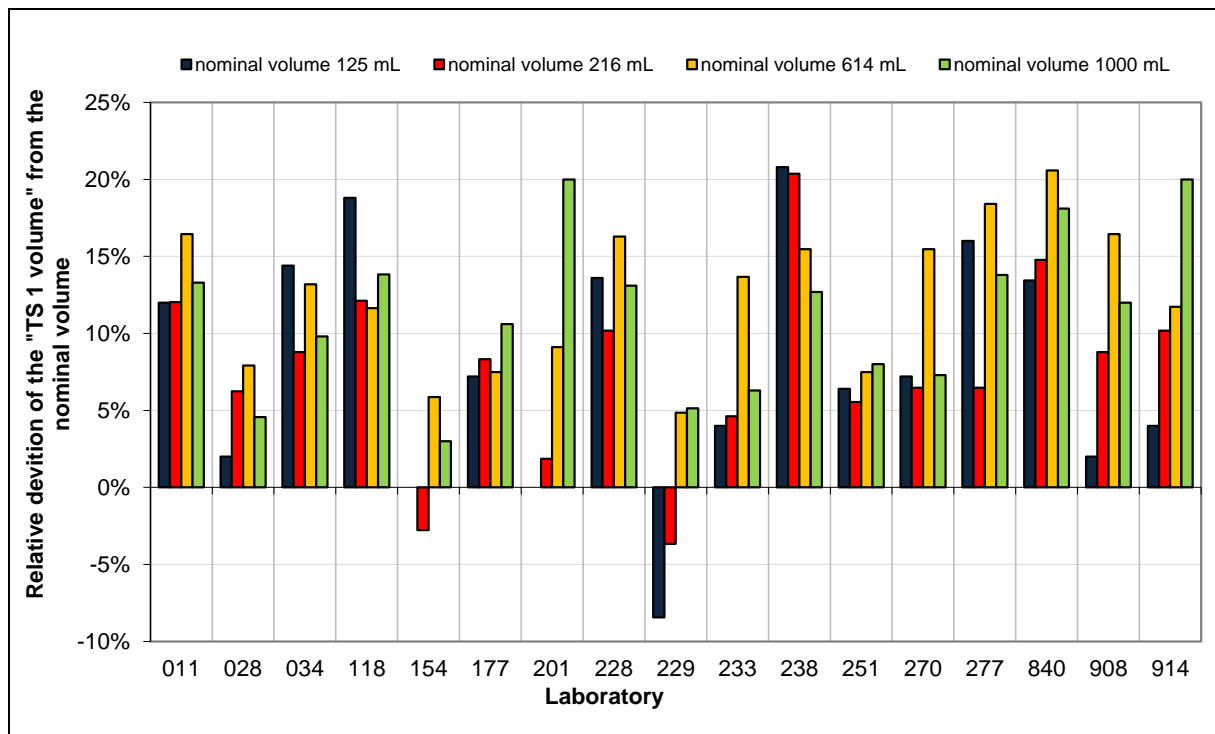


Figure 6-10: Relative deviation of the effective volume obtained by the labs in TS 1 (“TS 1 volume”) from the nominal volume of the sample baskets

In Figure 6-10, it can be seen that the “TS 1 volume” in general is higher than the nominal volume. This may have an effect on the extrapolated T_{Si} .

The “TS 1 volume” is determined – as any measure – with some degree of uncertainty, which can also affect the extrapolated T_{Si} . This uncertainty has two components: the uncertainty of the determination of the “TS 1 volume” within a laboratory and the uncertainty of the determination of the “TS 1 volume” between laboratories. Even if in TS 1 it was not asked in the data input form B (→ Appendix 9.3.4) to describe the volumetric method, some labs sent information about the bulk material used (→ Table 6-4).

Table 6-4: Bulk material used at volumetry in TS 1 and TS 3

Laboratory	Bulk material used for the volumetry of the sample baskets	
	TS 1	TS 3
011	salt	glass beads
028	n/a	glass beads
034	n/a	glass beads
034	n/a	glass beads
118	n/a	water
154	n/a	glass beads
177	sea-sand, (sieved (homogenized) to < 500 µm: mean bulk density in the 1000 ml-basket: 1.76 g/cm ³)	glass beads
201	n/a	glass beads
228	n/a	glass beads
228	n/a	glass beads
229	n/a	coal of known density
233	n/a	pulverulent alumina
238	n/a	glass beads
251	n/a	glass beads
270	n/a	glass beads
277	powder Neutrex ABC-70. (The density of the powder was determined by weighting t1 l of this powder (average of the five measurements).)	glass beads
840	n/a	glass beads
908	n/a	quartz sand
914	table salt	glass beads

Conclusion: The volumetric results of TS 1 scatter in a wide range. The reason for this scattering may result from the differences by manufactory tolerances of the sample baskets and from the lab specific measurement uncertainty but also from different volumetric method used by the different labs in TS 1.

6.7.1 Standardized volumetric method with glass beads with a diameter of about 0.3 mm

To reduce the uncertainty resulting from the different volumetric methods to determine the sample basket volumes, BAM has proposed a volumetric method as standard, where glass beads⁴ with a diameter of about 0.3 mm (→ Figure 6-11) should be used.

⁴ The following suppliers were recommended in the interlaboratory test instructions of the TS 3 (→ Appendix 9.5.1):
<http://www.edmund-buehler.de/english/i-homogenisatoren-und-zellmuehlen.pml>
<http://www.sartorius-mechatronics.com/DE/en/index.htm> (product number: BBI-8541604)

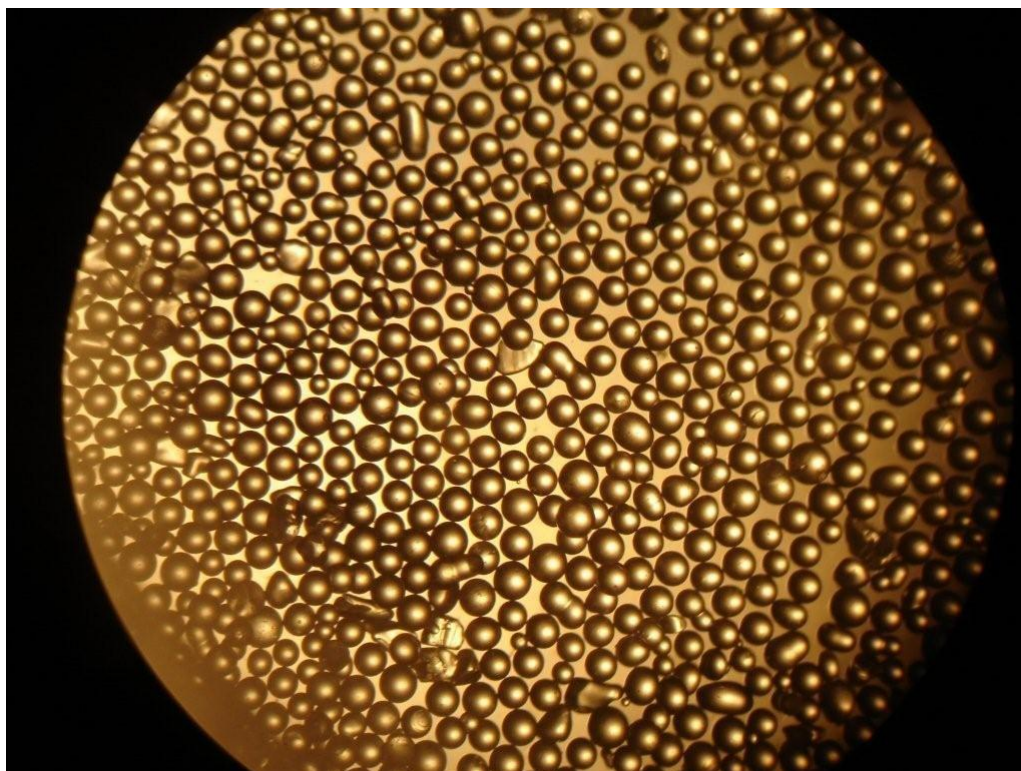


Figure 6-11: Enlarged photo of commercial available glass beads of about 0.3 mm (Edmund Bühler GmbH, Hechingen, Germany) examined under light microscope

For this purpose BAM has determined the volumes of all sample baskets (originally provided by BAM) of all laboratories in TS 2 by this glass bead method. The volume of the sample baskets determined by BAM in TS 2 using the glass bead method is hereinafter referred to as “reference volume”.

In order to check the effect of volumetry, time-dependent corrected T_{SI} measurements (“reference date” 21.07.2011) of the same 11 labs as in the previous chapter 6.6. have been considered. Here, the robust mean values were determined for both extrapolated T_{SI} based on nominal volumes and extrapolated T_{SI} based reference volumes. The robust mean values together with the expanded uncertainty of the respective robust mean value are given in the following Table 6-5.

Table 6-5: Extrapolated T_{SI} of different storage volumes on the basis of the nominal volume and reference volume of the sample baskets

Storage volume	Robust mean value of the extrapolated T_{SI} ± expanded uncertainty [°C]	
	based on nominal volumes	based on reference volumes
27 m ³	48.4 ± 1.6	50.3 ± 2.2
100 m ³	40.8 ± 1.7	42.8 ± 2.3
500 m ³	32.0 ± 1.9	34.0 ± 2.4
1000 m ³	28.4 ± 2.0	30.4 ± 2.6

Conclusion: Due to manufacture tolerances the effective volume of sample baskets may differ more or less from the projected nominal volume. Even if the effect of different volumetry on the extrapolated T_{SI} is not statistically significant for a given storage volume in this interlaboratory test, the effect may exist and may have been masked by other disturbing factors.

In TS 3 the labs were asked to determine the effective volumes of their sample baskets again (in the following referred as to “TS 3 volume”). But unlike in TS 1, the volumes in TS 3 should be determined by the method recommended by BAM with glass beads with a diameter of 0.3 mm. In order to find out the random errors regarding the determination of the effective volume, the laboratories should have performed this volumetry procedure three times.

The following figures (Figure 6-12 to Figure 6-14) show the relative deviations of

- nominal volumes
- the “TS 1 volumes” determined by the different laboratories and
- reference volumes (measured centrally by BAM in TS 2)

from the “TS 3 volumes”.

It has to be noted that laboratory 238 was not considered for the determination of the relative deviations since the sample baskets used at TS 1 and TS 2 were replaced in TS 3 by new sample baskets.

Labs which have not used glass beads to determine the volume of the sample baskets in TS 3 (→ Table 6-4) are marked in red. This concerns the following four laboratories:

- laboratory 118 (used water)
- laboratory 229 (used coal of known density)
- laboratory 233 (used pulverulent alumina)
- laboratory 908 (used quartz sand)

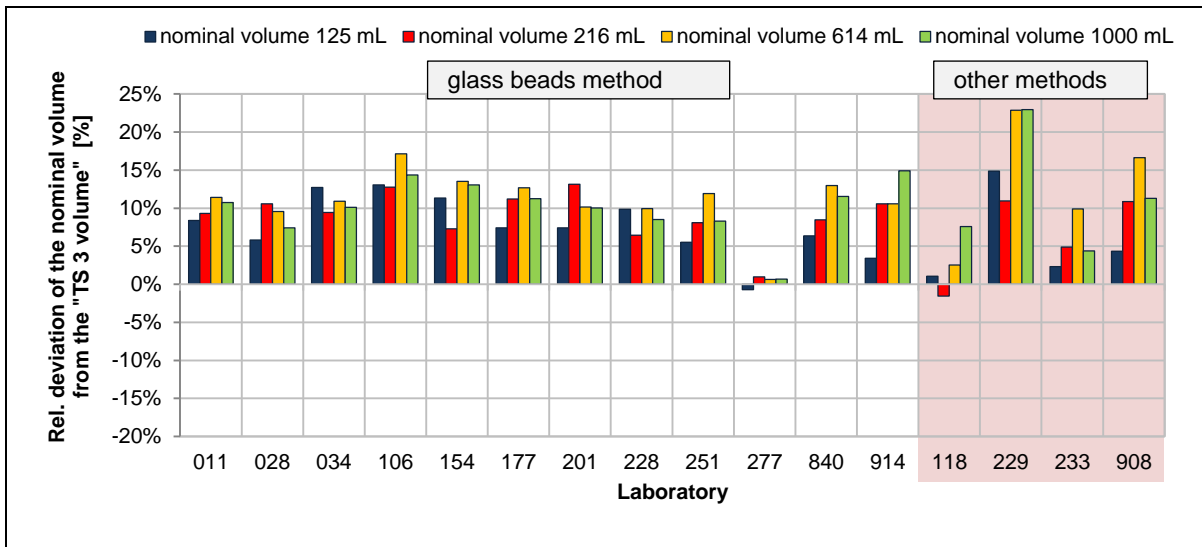


Figure 6-12: Relative Deviation of the nominal volume from the “TS 3 volume“

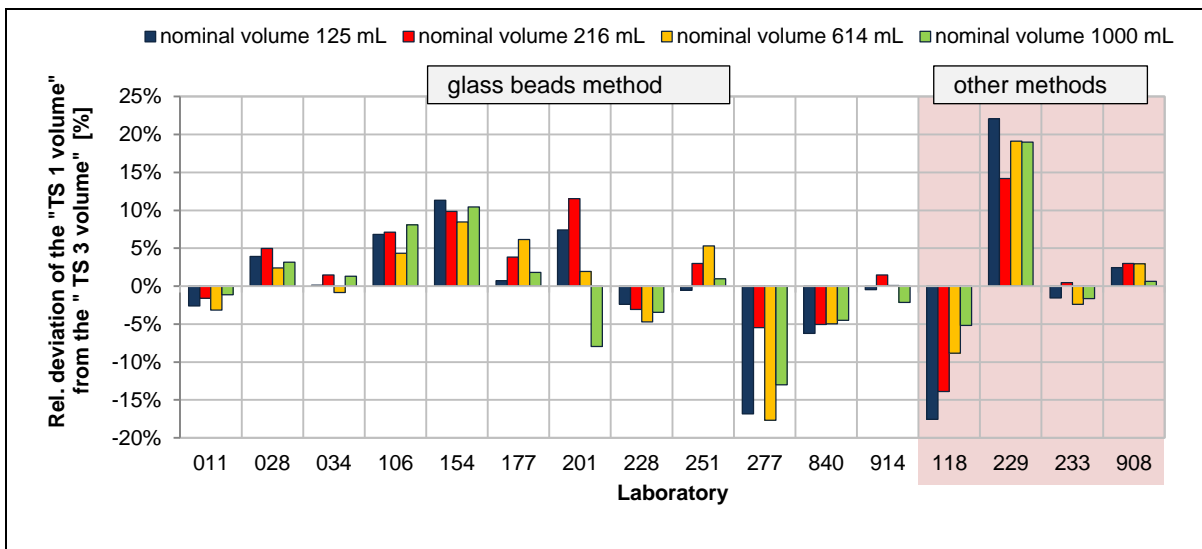


Figure 6-13: Relative Deviation of the “TS 1 volume” from the “TS 3 volume“

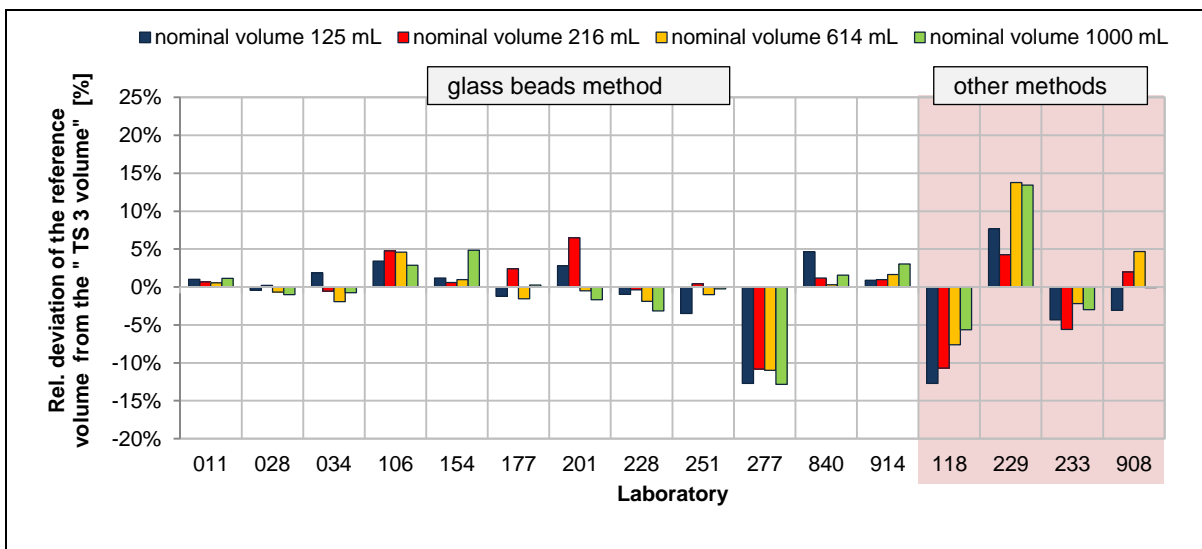


Figure 6-14: Relative Deviation of the reference volume from the ”TS 3 volume“

Figure 6-12, Figure 6-13 and Figure 6-14 show that the suggested standardized method for determining the volumes of the sample basket (by means of glass beads) leads to comparable results between the laboratories. Only the test results of laboratory 277 are not plausible and will be regarded as exceptional (→ Figure 6-14).

The “TS 3 volumes” of laboratory 233, which used pulverulent alumina instead of glass beads and laboratory 118, which used tap water, were determined relatively precisely. However, in laboratory 229 (coal of known density was used to calculate the volume of the sample baskets) and in laboratory 908 (quartz sand) the precision is much lower.

Conclusion: Due to manufacture tolerances the effective volume of sample baskets may differ more or less from the projected nominal volume. Since the extrapolated T_{Si} for larger storage volumes are based on the volumes of the sample baskets used in the interlaboratory test, it is essential to check their effective volume by a reliable volumetric method.

It was shown that the results of other volumetric methods (“water” by lab 118 and “coal of known density” by lab 229) significantly deviate from the results which were analyzed by the aid of the recommended glass beads. However, it can also be assumed that, by other methods (e.g. with “pulverulent alumina” by lab 233 and with “quartz sand” by lab 908), it may be possible to achieve sufficiently comparable results to the “glass beads” volumetric method. In order to limit deviations between the results of different labs and as long as no other method is sufficiently validated it is recommended to apply the volumetric method with glass beads as standard (reference).

6.8 Other influencing (disturbing) factors

The reason for the spread of the results and the deviations between the laboratories was analysed by an additional exploratory data analysis. However, the conclusiveness of these findings is limited because the aim of this interlaboratory test was mainly focused on the modification of the method (use of a mesh wire screen and standardized sample baskets) and not on other factors.

In general, the following parameters may vary from laboratory to laboratory:

- laboratory oven (type/supplier)
- laboratory oven convection (natural vs. forced)
- laboratory oven size
- screen volume
- calibration of sensor (yes vs. no)
- stability of oven temperature [K]
- noise of temperature signal [K].

Based on the time-dependent corrected T_{Si} measurements and the “TS 3 volume” (→ chapter 0) the extrapolated T_{Si} for a storage volume of 1000 m³ were calculated as described in chapter 6.1.2 – separately for TS 1 and TS 3.

The effects of the different laboratory-specific parameters are shown in the following figures (Figure 6-15 and Figure 6-16).

It has to be noted that these figures provide only a rough orientation and no statistically firm conclusions can be derived from them.

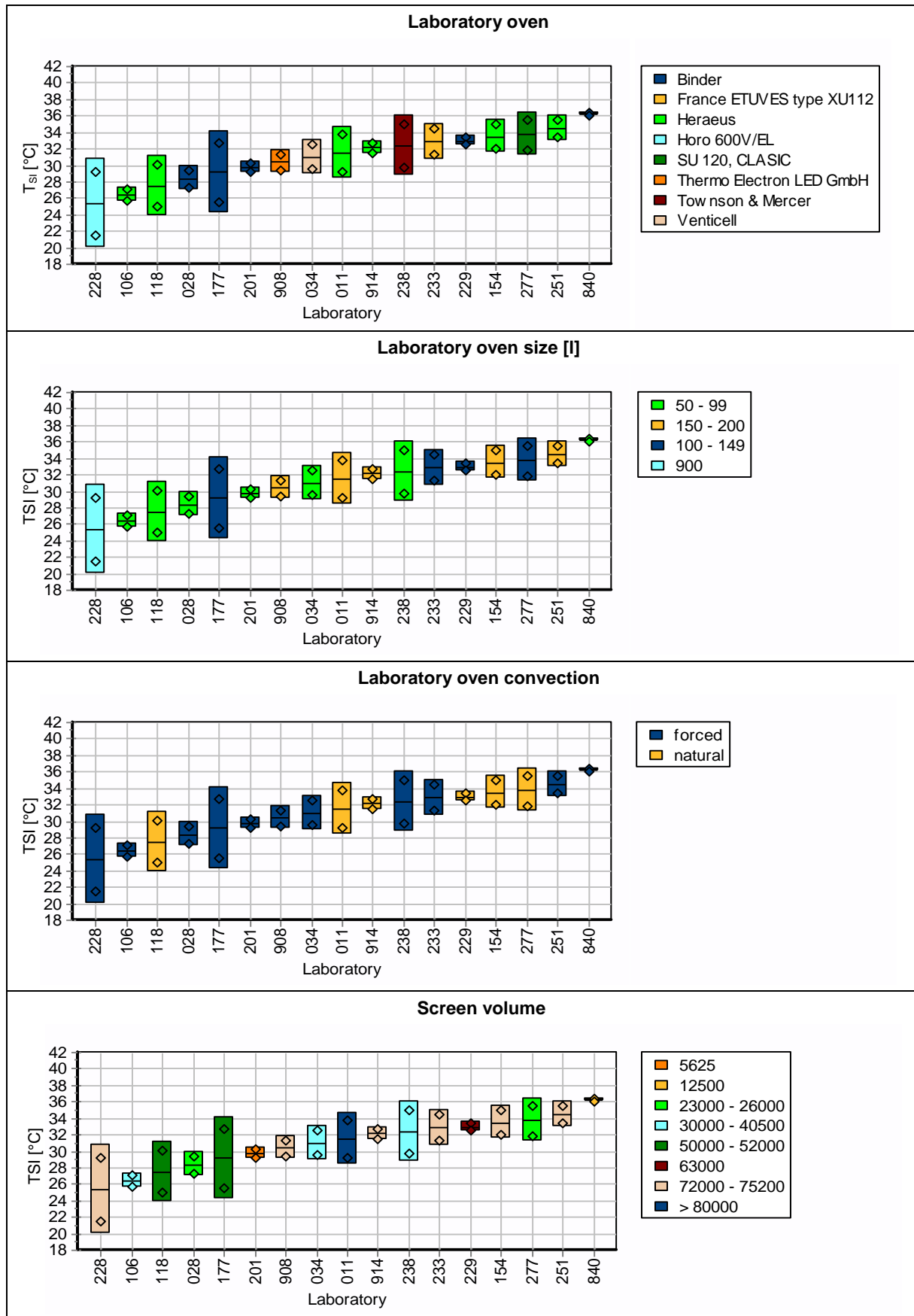


Figure 6-15: Influence of lab-specific method parameters regarding the extrapolated T_{si} for a storage volume of 1000 m^3 (continued in Figure 6-16)

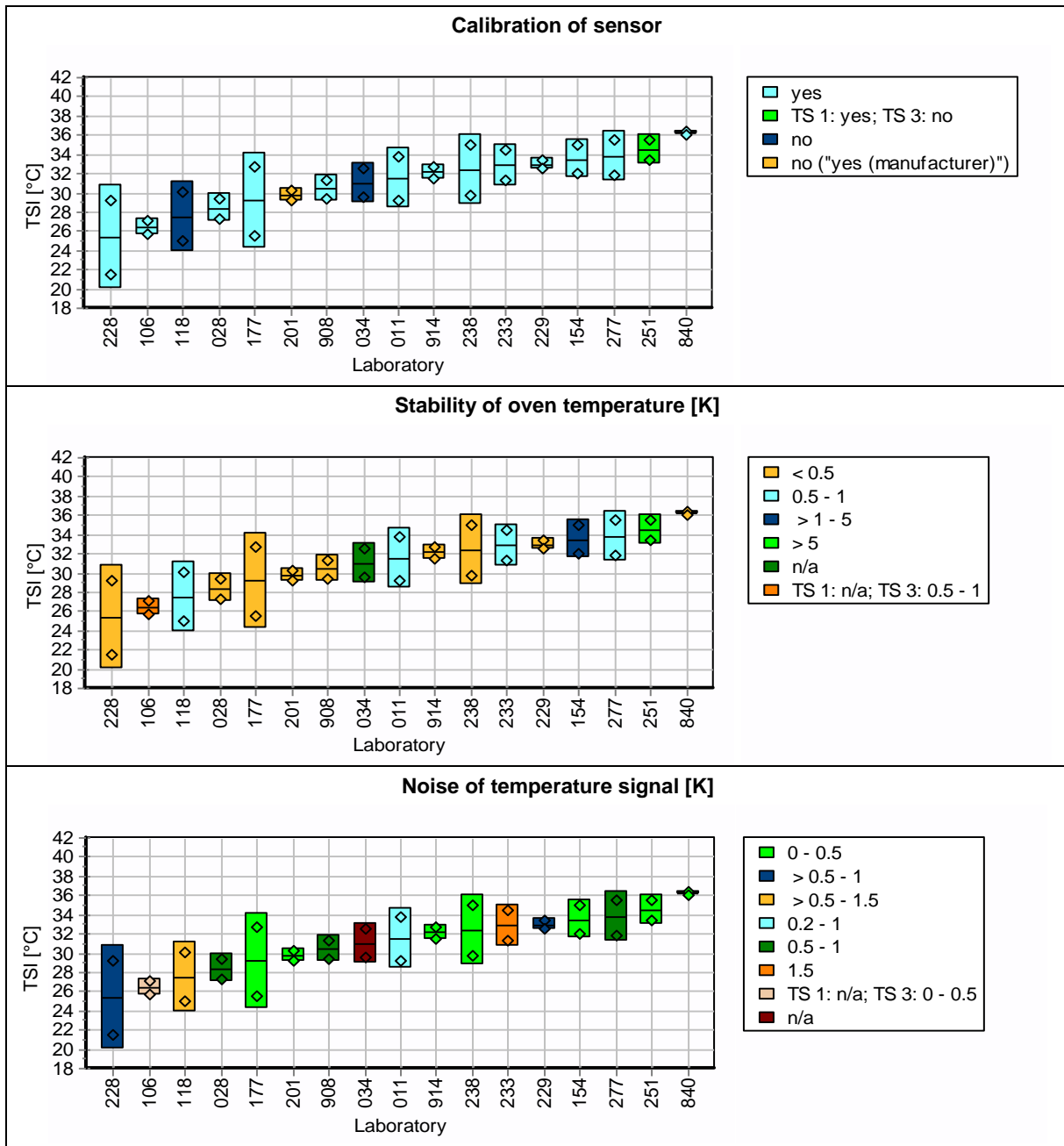


Figure 6-16: Influence of lab-specific method parameters regarding the extrapolated T_{SI} for a storage volume of 1000 m³ (continuation of Figure 6-15)

Conclusion: An influence of the listed influencing factors on the extrapolated T_{SI} cannot be observed in this interlaboratory test. However, the conclusiveness of these findings is limited because the aim and the testing concept of this interlaboratory test were mainly focused on the above mentioned modification of DIN EN 15188:2007. These effects could be checked by the aid of more specific investigations.

6.9 Precision parameters of the modified method DIN EN 15188

6.9.1 Statistical method

The method according to DIN 38402-45 (=ISO/TS 20612) was applied to calculate the mean extrapolated T_{SI} across all “valid” labs (→ chapter 6.9.2) as well as the corresponding repeatability and reproducibility standard deviations for storage volumes of 27 m³, 100 m³, 500 m³ and 1000 m³. This method is a robust method and no outlier examination is required.

The evaluation of the data was performed using a specially modified version⁵ of the software package PROLab Plus 2012 [8]. PROLab Plus is widely employed for the evaluation of interlaboratory tests and laboratory proficiency tests.

6.9.2 Data base

The precision parameters of the modified method DIN EN 15188 by using “Carbon Black, Norit CN4” as sample material were calculated on the basis of the corresponding non-rounded results (i.e. the rounded down oven temperatures as stipulated in DIN EN 15188:2007 were not used as T_{SI} measurements, → chapter 6.6).

First of all it has to be noted that the extrapolated T_{SI} are based on

- time-dependent corrected T_{SI} measurements of the considered sample nominal volumes of 125 mL, 216 mL, 614 mL and 1000 mL corresponding to the “reference date” 21.07.2011
- the effective volumes obtained by the labs in TS 3 (“TS 3 volumes”).

Due to the fact that the volumetric method recommended by BAM, i.e. using glass beads with a diameter of 0.3 mm, will be prescribed in the modified DIN EN 15188 (→ chapter 0), only the extrapolated T_{SI} of those laboratories will be included in the following statistical analysis, which actually applied the “glass bead” method in TS 3. Furthermore it is also mandatory that the included data be based on acceptable repeatability conditions. This is essential to determine statistically reliable precision data of the method.

In accordance with these conditions, the extrapolated T_{SI} values of the following laboratories cannot be included for determining the final precision data of the modified method DIN EN 15188:

Laboratories with unacceptable repeatability conditions:

- 154
- 238
- 251

Laboratories, which did not use the “glass bead” method in TS 3

- 118 (tap water)
- 229 (coal of known density was used to calculate the volume of sample baskets)
- 233 (Pulverulent alumina)
- 908 (quartz sand).

⁵ The basic PROLab Plus version has been extended by additional tools taking into account the specific design of the intercomparison. These additional tools are in-house tools only.

Conclusion: Not all data of all labs can be included in the statistical procedure to generate reliable precision data of the modified method. Nevertheless it can be assumed that the number of valid results from 10 labs is sufficient to generate reliable precision data of the modified method.

6.9.3 Kernel density estimation of extrapolated T_{SI} values for storage volumes of 27 m³, 100 m³, 500 m³ and 1000 m³

An analysis of the underlying distribution of the extrapolated T_{SI} values for a given storage volume was carried out by the so-called kernel density estimation in order to check the homogeneity.

Figure 6-17 shows the result of the kernel density estimation of extrapolated T_{SI} values for storage volumes of 27 m³, 100 m³, 500 m³ and 1000 m³.

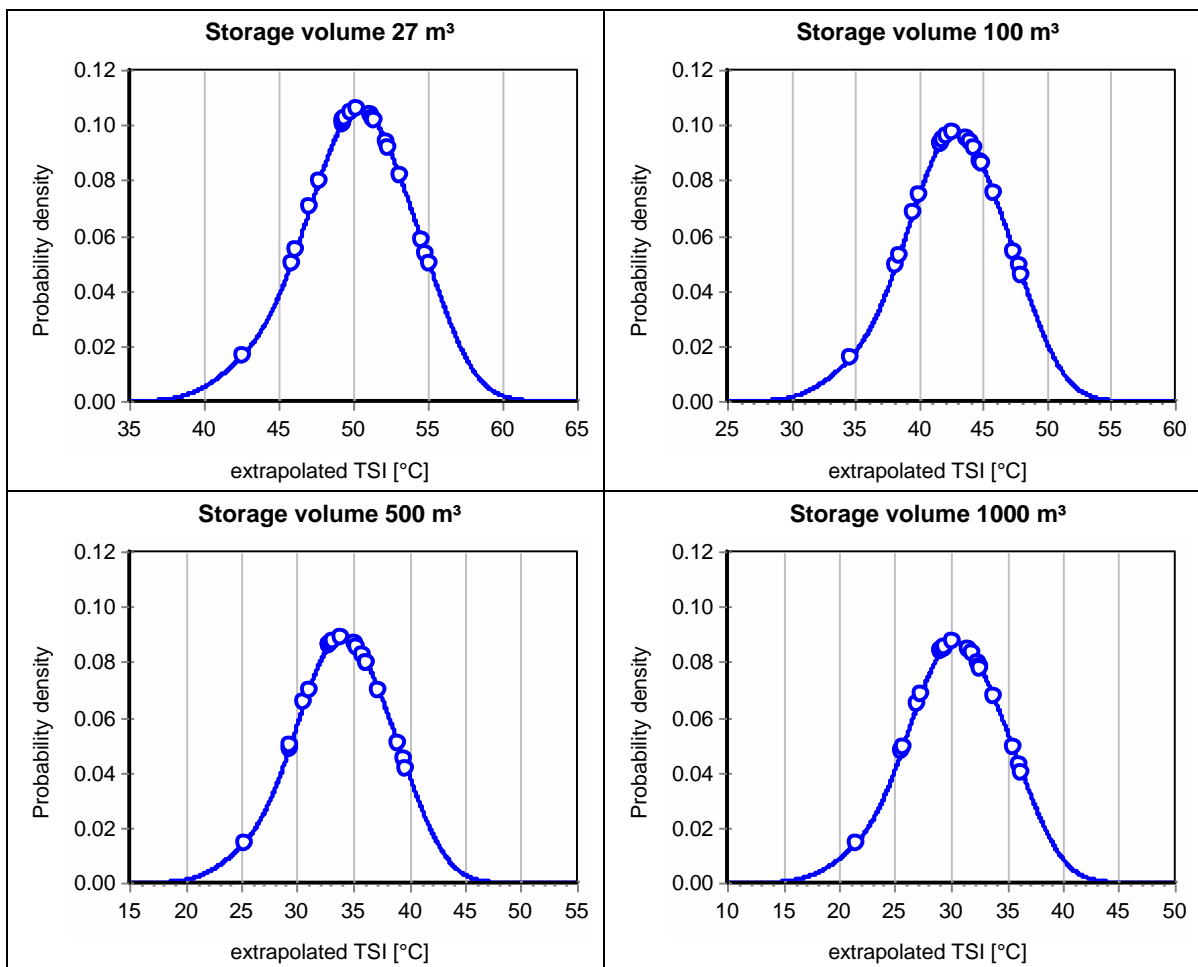


Figure 6-17: Kernel density estimation for extrapolated T_{SI} values for storage volumes of 27 m³, 100 m³, 500 m³ and 1000 m³

In each figure, the blue curve characterizes the distribution of extrapolated T_{SI} values for the respective storage volume obtained by the kernel density estimation, where single (from TS 1 and TS 3 separately) extrapolated T_{SI} values of the labs are marked as small blue circles.

In general, distributions with only one mode are called unimodal, while distributions with two or more modes are called bimodal or multimodal. Multimodal distributions indicate that there might be two or more groups of participants with clearly differing results. However, only a mode which is based on at least 25 % of the measurement values by one group of labs should be considered as forming a sub-group.

The left axis of the kernel density plot shows the probability density. This probability density is neither the probability nor the frequency. It indicates the relative frequency of values occurring at different points along the x-axis. It is not the values on the left axis which are of interest, but the shape of the curve.

Conclusion: The distribution of the extrapolated T_{Si} values can be assumed to be normal for all four considered storage volumes. This is confirmed by the Shapiro-Wilk test [13] (at the significance level of 5 %). Therefore it can be assumed that the data are sufficient to generate reliable precision parameters of the method.

6.9.4 Total robust mean value, reproducibility and repeatability of the modified method DIN EN 15188

A summary of the obtained total robust mean values, reproducibility and repeatability standard deviations (→ chapter 6.9.1) of the extrapolated T_{SI} values for the storage volumes 27 m³, 100 m³, 500 m³ and 1000 m³ of the sample material “Carbon Black, Norit CN4” across laboratories are given in the following Table 6-6.

The measurement uncertainty ($k=2$) and also the 95 % tolerance interval with the lower and upper tolerance limits for the extrapolated T_{SI} of the sample material “Carbon Black, Norit CN4” according the modified method DIN EN 15188 can be derived from the reproducibility standard deviation s_R . These values are also given in Table 6-6.

Table 6-6: Precision parameters of the modified method DIN EN 15188 obtained by the interlaboratory test 2010 - 2011

Precision parameter according to DIN 38402-45 (→ chapter 6.9.1)		Self-ignition temperature (T_{SI}) for a storage volume of			
		27 m ³	100 m ³	500 m ³	1000 m ³
Number of involved laboratories		10	10	10	10
robust	Total robust mean value⁶ ± expanded uncertainty⁷ ($k=2$) [°C]	50.2 ± 2.4	42.7 ± 2.6	34.0 ± 2.9	30.4 ± 2.9
	Repeatability s.d. s_r [°C]	1.8	2.1	2.5	2.6
	Reproducibility s.d. s_R⁸ [°C]	4.0	4.4	4.8	4.9
	Measurement uncertainty⁹ ($k=2$) [°C]	8.0	8.8	9.6	9.8
	Lower tolerance limit of the 95 % tolerance interval [°C]	42.2	33.9	24.4	20.6
	Upper tolerance limit of the 95 % tolerance interval [°C]	58.2	51.5	43.6	40.2

Conclusion: The aim of this interlaboratory test was met. A clear picture concerning precision parameters of the suggested modifications of the method DIN EN 15188 (→ chapter 3) could be determined. The precision of the modified method DIN EN 15188 can be assessed as acceptable. The rate between repeatability s.d. s_r and reproducibility s.d. s_R is approximately 1:2. Thus considerable systematic deviations / errors by using the modified method cannot be assumed. The performance of the modified method DIN EN 15188 can be assessed as sufficient to determine the self-ignition temperature.

⁶ These values have been used as target value in chapter 6.10 Assessment of laboratories.

⁷ The 95 % confidence intervals of the total robust mean value in Figure 6-19, Figure 6-20, Figure 6-21 and Figure 6-22 has been calculated on the basis of the expanded uncertainty.

⁸ These values have been used as target standard deviation in chapter 6.10 Assessment of laboratories.

⁹ Validity of the values of the measurement uncertainty → chapter 6.9.5.

The 95 % tolerance interval for the extrapolated T_{SI} – i.e. the range between the curve of the lower tolerance limit values and the curve of the upper tolerance limit values – is graphically displayed in the Pseudo-Arrhenius plots in Figure 6-18. Here, the lower and upper 95 % tolerance limit values of extrapolated T_{SI} for the four considered storage volumes are given as red diamonds. The continuous red lines characterize the interpolated tolerance limit values, and the dashed red lines characterize the extrapolated tolerance limit values.

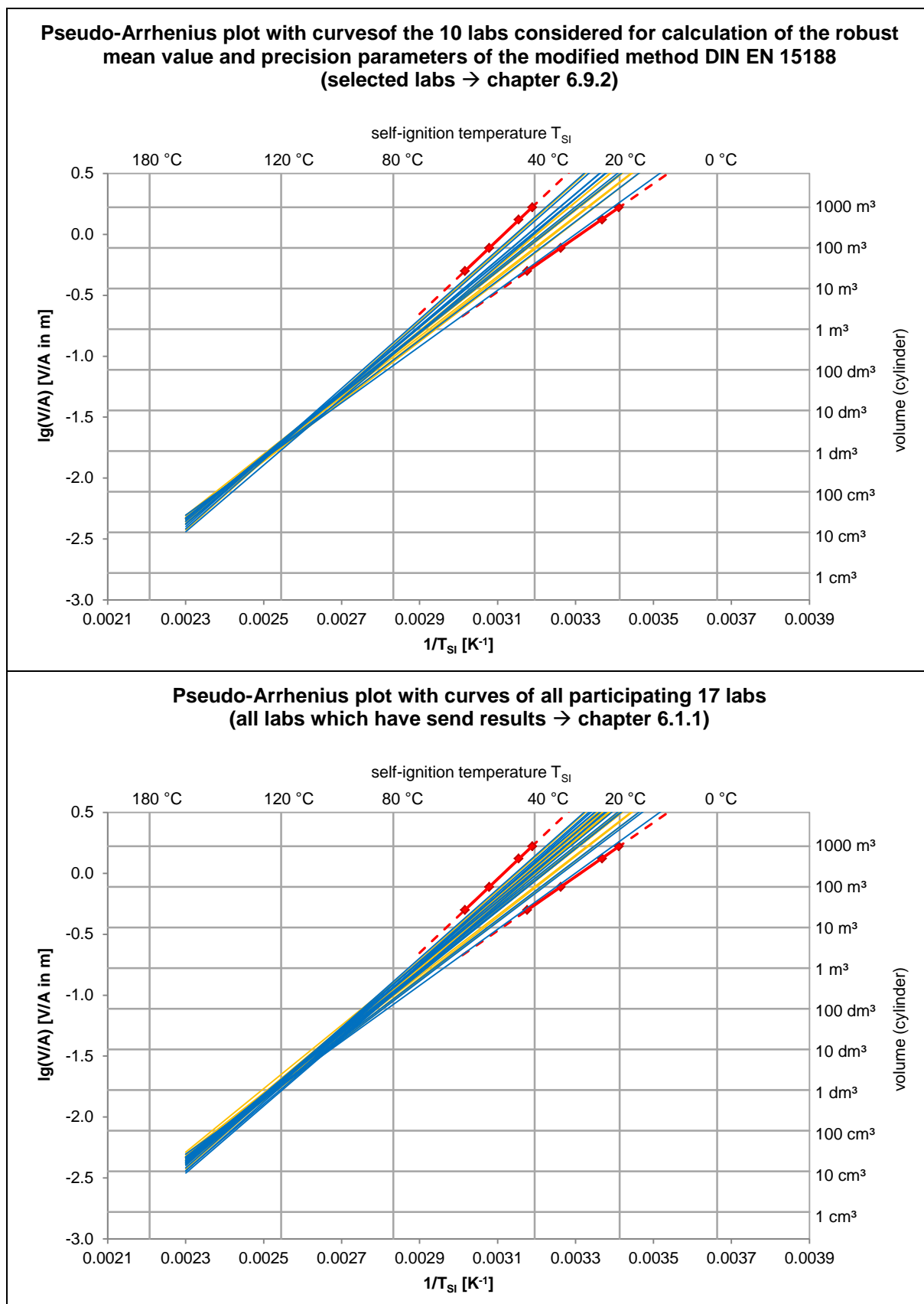


Figure 6-18: Pseudo-Arrhenius plots of self-ignition temperatures of “Carbon Black, Norit CN4” based on time-dependent corrected T_{SI} measurements and the “TS 3 volume” (yellow lines: Pseudo-Arrhenius plot corresponding to TS 1; blue lines: Pseudo-Arrhenius plots corresponding to TS 3)

Conclusion: The aim of this interlaboratory test was met. The suggested modifications of the method DIN EN 15188 (→ chapter 3) can be assessed as sufficient. The scattering of slopes of the curves in the Pseudo-Arrhenius plots of all labs can be assessed as reduced in comparison to the results of the non-modified method (→ chapter 2, Figure 2-1: Pseudo-Arrhenius plot of self-ignition temperatures of Lycopodium of the Round Robin Test 2002 (BAM)).

The laboratory's extrapolated T_{SI} values of TS 1 and TS 3 for the storage volumes 27 m³ (Figure 6-19), 100 m³ (Figure 6-20), 500 m³ (Figure 6-21) and 1000 m³ (Figure 6-22) are shown in the following figures.

In these figures the extrapolated T_{SI} values of all 17 participating laboratories are displayed in two types of coloured boxes:

- **10 labs with boxes coloured in medium blue:**

The boxes of the 10 labs from which the extrapolated T_{SI} values were included for determining the precision parameters of the modified method DIN EN 15188 are coloured in medium blue.

These labs are referred to in the following as “**considered labs**”

- **7 labs with boxes coloured in light blue:**

The boxes of the 7 labs from which the extrapolated T_{SI} values could not be included are coloured in light blue.

These labs are referred to in the following as “**unconsidered labs**”.

The size of the medium blue and light blue boxes symbolizes the laboratory's repeatability standard deviation of the extrapolated T_{SI} values for the respective storage volume corresponding to TS 1 and TS 3. The larger the box, the higher the variability of the extrapolated T_{SI} values for the corresponding laboratory. The horizontal line in the middle of the medium blue and light blue boxes indicates the laboratory mean value, while the small diamonds indicate the individual extrapolated T_{SI} values of TS 1 (yellow) and TS 3 (blue), respectively.

The figures also include the precision parameters according to DIN 38402-45 of the modified method DIN EN 15188 (→ Glossary 1 and Table 6-6):

- the total robust mean value (→ chapter 1 Glossary) across laboratories as a dark blue horizontal line, together with the 95 % confidence interval of the total robust mean value (green strip) and the repeatability standard deviation s_r (right grey box) and
- the reproducibility standard deviation s_R (left grey box).
- the lower and upper limits of the 95 % tolerance interval (→ chapter 1 Glossary) for the laboratory mean values (red lines)

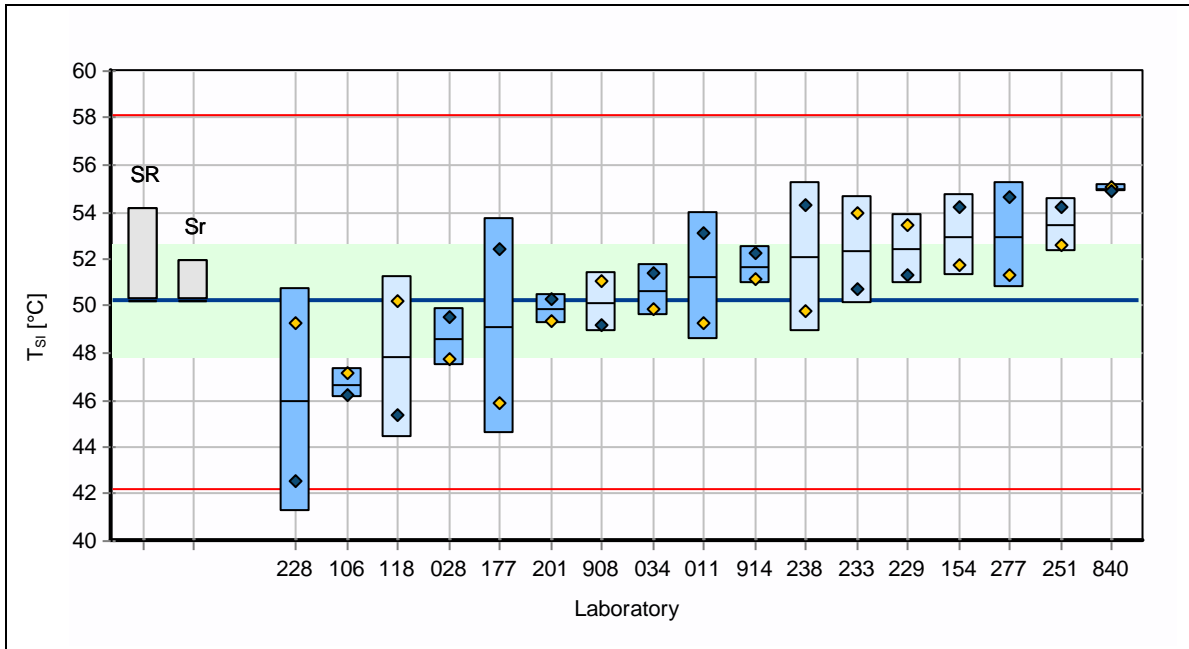


Figure 6-19: Results for the self-ignition temperature for a storage volume of 27 m³ (yellow diamonds: extrapolated T_{SI} of TS 1; blue diamonds: extrapolated T_{SI} of TS 3; medium blue boxes: considered lab; light blue boxes: unconsidered lab SR...reproducibility s.d. s_R , Sr...repeatability s.d. s_r)

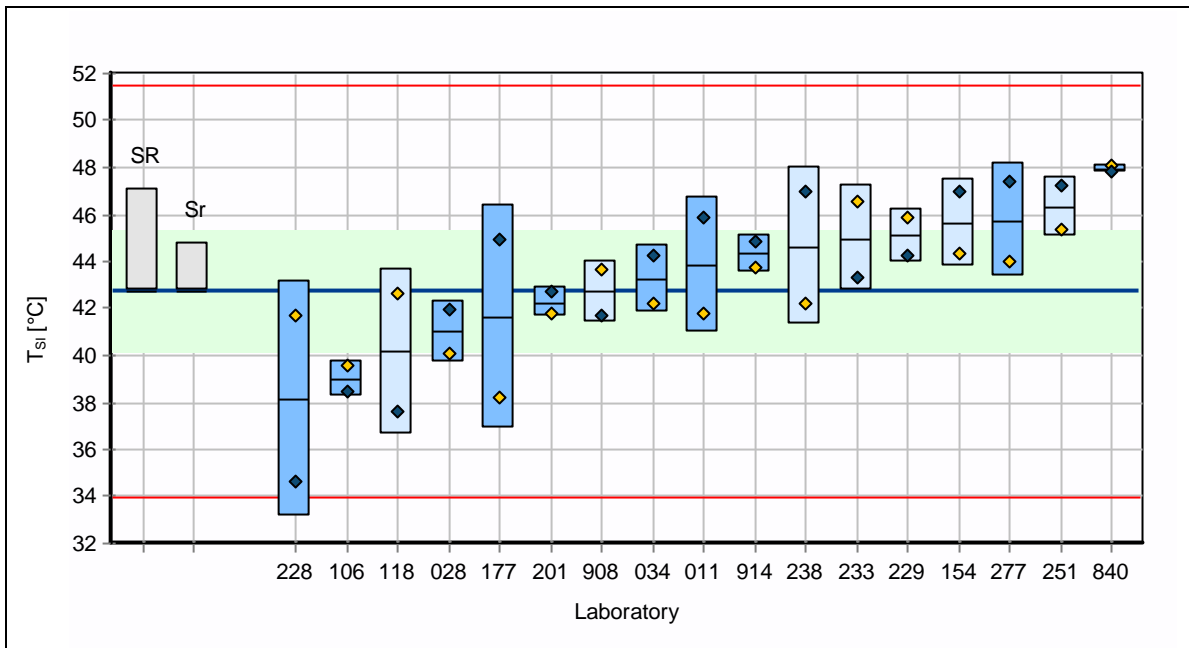


Figure 6-20: Results for the self-ignition temperature for a storage volume of 100 m³ (yellow diamonds: extrapolated T_{SI} of TS 1; blue diamonds: extrapolated T_{SI} of TS 3; medium blue boxes: considered lab; light blue boxes: unconsidered lab SR...reproducibility s.d. s_R , Sr...repeatability s.d. s_r)

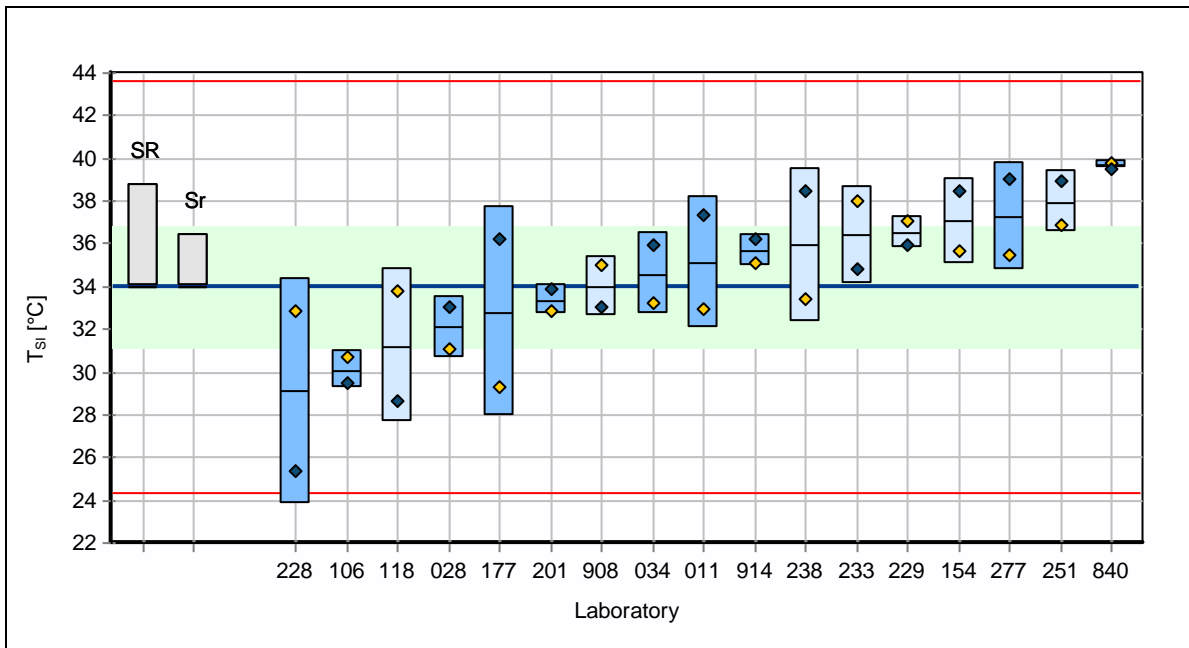


Figure 6-21: Results for the self-ignition temperature for a storage volume of 500 m³ (yellow diamonds: extrapolated T_{SI} of TS 1; blue diamonds: extrapolated T_{SI} of TS 3; medium blue boxes: considered lab; light blue boxes: unconsidered lab SR...reproducibility s.d. s_R , Sr...repeatability s.d. s_r)

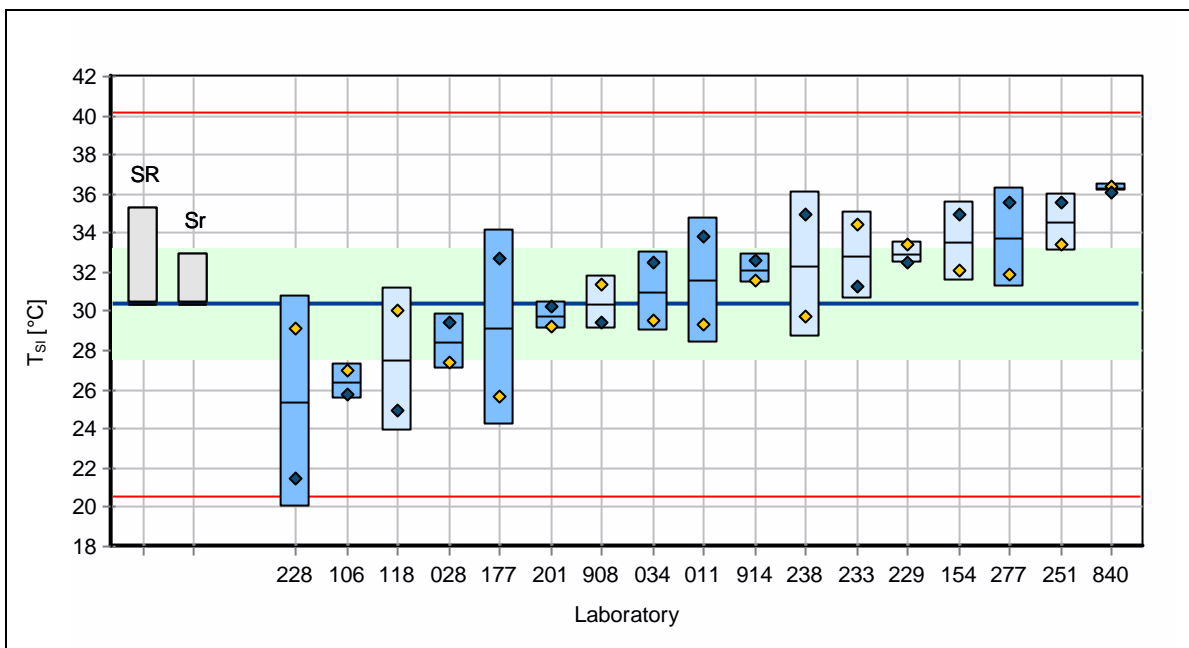


Figure 6-22: Results for the self-ignition temperature for a storage volume of 1000 m³ (yellow diamonds: extrapolated T_{SI} of TS 1; blue diamonds: extrapolated T_{SI} of TS 3; medium blue boxes: considered lab; light blue boxes: unconsidered lab SR...reproducibility s.d. s_R , Sr...repeatability s.d. s_r)

Conclusion: The figures show that the precision of the modified method DIN EN 15188 can be assessed as acceptable for the considered labs as well as for the unconsidered labs. This indicates that the two suggested modifications (→ chapter 3.1 / modifications: mesh wire screen and volumes ratios of sample baskets of 1 : 1.7 : 5 : 8) are highly effective and should be implemented.

6.9.5 Measurement uncertainty of the lab based on the precision parameters of the modified method DIN EN 15188 and validity for other test substances

Extrapolated T_{SI} determined by the modified method DIN EN 15188 are generally affected by the measurement uncertainty of a laboratory.

Measurement uncertainty on the basis of the reproducibility s.d. s_R of an interlaboratory test

Results and specially the reproducibility s.d. s_R obtained in interlaboratory tests are a valid basis for measurement uncertainty evaluation ([6], [7]).

The following equation applies

$$\text{'Laboratory's result'} = \text{'Extrapolated } T_{SI}' \pm 'U' [^{\circ}\text{C}],$$

at which U denotes the measurement uncertainty at $k=2$ by

$$U = 2 \cdot \text{'reproducibility s.d. } s_R \text{ (depending on the storage volume)'} \rightarrow [6], [7]$$

Furthermore the measurement uncertainty of the extrapolated T_{SI} depends on the storage volume (\rightarrow Table 6-6). For the four storage volumes considered in this interlaboratory study the measurement uncertainties ($k=2$) are given as follows, where the reproducibility s. d. s_R (depending on the storage volume) were taken from Table 6-6:

- **Storage volume 27 m³**
'Laboratory's result' = 'Extrapolated T_{SI} ' $\pm 2 \cdot 4.0$ °C = **'Extrapolated T_{SI} ' ± 8.0 °C ($k=2$)**
- **Storage volume 100 m³**
'Laboratory's result' = 'Extrapolated T_{SI} ' $\pm 2 \cdot 4.4$ °C = **'Extrapolated T_{SI} ' ± 8.8 °C ($k=2$)**
- **Storage volume 500 m³**
'Laboratory's result' = 'Extrapolated T_{SI} ' $\pm 2 \cdot 4.8$ °C = **'Extrapolated T_{SI} ' ± 9.6 °C ($k=2$)**
- **Storage volume 1000 m³**
'Laboratory's result' = 'Extrapolated T_{SI} ' $\pm 2 \cdot 4.9$ °C = **'Extrapolated T_{SI} ' ± 9.8 °C ($k=2$)**

Conclusion: It can be generalized that the measurement uncertainty U (for $k=2$) for extrapolated T_{SI} is not higher than 10 °C for storage volumes up to 1000 m³ maximum. The measurement uncertainty cannot be ignored and must be considered if T_{SI} results should be used in the practice.

Validity of the measurement uncertainty for other test substances

Please note! It must be considered that the measurement uncertainty U ($k=2$) calculated by the equations above can only be used by the lab, if the following criteria are fulfilled (\rightarrow Table 6-7), [6]:

Table 6-7: Criteria to express the measurement uncertainty U by the aid of the reproducibility s.d. s_R obtained by the interlaboratory test [6]

Criterion	Content
Criteria 1	The modified method DIN EN 15188 must be applied.
Criteria 2	The test conditions must be comparable to those of the interlaboratory test and the tested sample substance must be comparable to the interlaboratory test sample "Carbon Black, Norit CN4".
Criteria 3	The trueness of the results of the lab must be established.
Criteria 4	The near agreement between the lab-specific repeatability standard deviation for a certain storage volume and the repeatability standard deviation obtained in this interlaboratory must be established.

Conclusion: The aim of this interlaboratory test was to assess the suggested modifications of the method (→ chapter 3) by the investigation of one typical test sample material. "Carbon Black, Norit CN4" was chosen as a typical sample. The determined measurement uncertainties can be assessed as highly acceptable for this interlaboratory test sample substance.

Nevertheless to avoid any doubts and to prevent any difficulties regarding the practical usage of the precision parameters (including the measurement uncertainty), the validity of the precision parameters must be ensured by additional tests with other, different substances which will cover a sufficiently wide range of self-ignition behaviours.

6.10 Assessment of laboratories

The assessment of the performance of the labs in determining the extrapolated T_{SI} for storage volumes of 27 m³, 100 m³, 500 m³ and 1000 m³ was carried out using z scores according to DIN 38402-45 / ISO/IEC 17043.

In general, z scores describe the standardised deviation of laboratory mean values from the total mean under consideration that the lower limit of tolerance does not fall below zero. Under a normal distribution, z scores lie within the limits -2 and 2 with probability 95 % and therefore if $|z \text{ score}| > 2$ holds, the quality criterion is not fulfilled.

Summarised, a laboratory's result is

- satisfactory if..... $|z \text{ score}| \leq 2$;
- questionable if $2 < |z \text{ score}| < 3$;
- unsatisfactory if..... $|z \text{ score}| \geq 3$.

In general, a z score less (or greater) than zero means that the laboratory's mean is less (or greater) than the total mean over all considered labs.

The assessment of the labs is carried out for each laboratory which participated in this interlaboratory test. However, the z scores considered in this chapter are based on the following target values of the modified method DIN EN 15188 (as derived in chapter 6.9.4 → Table 6-6):

- the total robust mean value of the modified method DIN EN 15188 of the extrapolated T_{SI} for the respective storage volume and
- the corresponding robust reproducibility s.d. s_R (as target standard deviation).

Note that these target values are based on labs which met the repeatability conditions and used the glass bead method for determining the "TS 3 volume".

The quality criterion ($-2 \leq z \leq +2$) is fulfilled by all labs for all four storage volumes as shown in Figure 5-12.

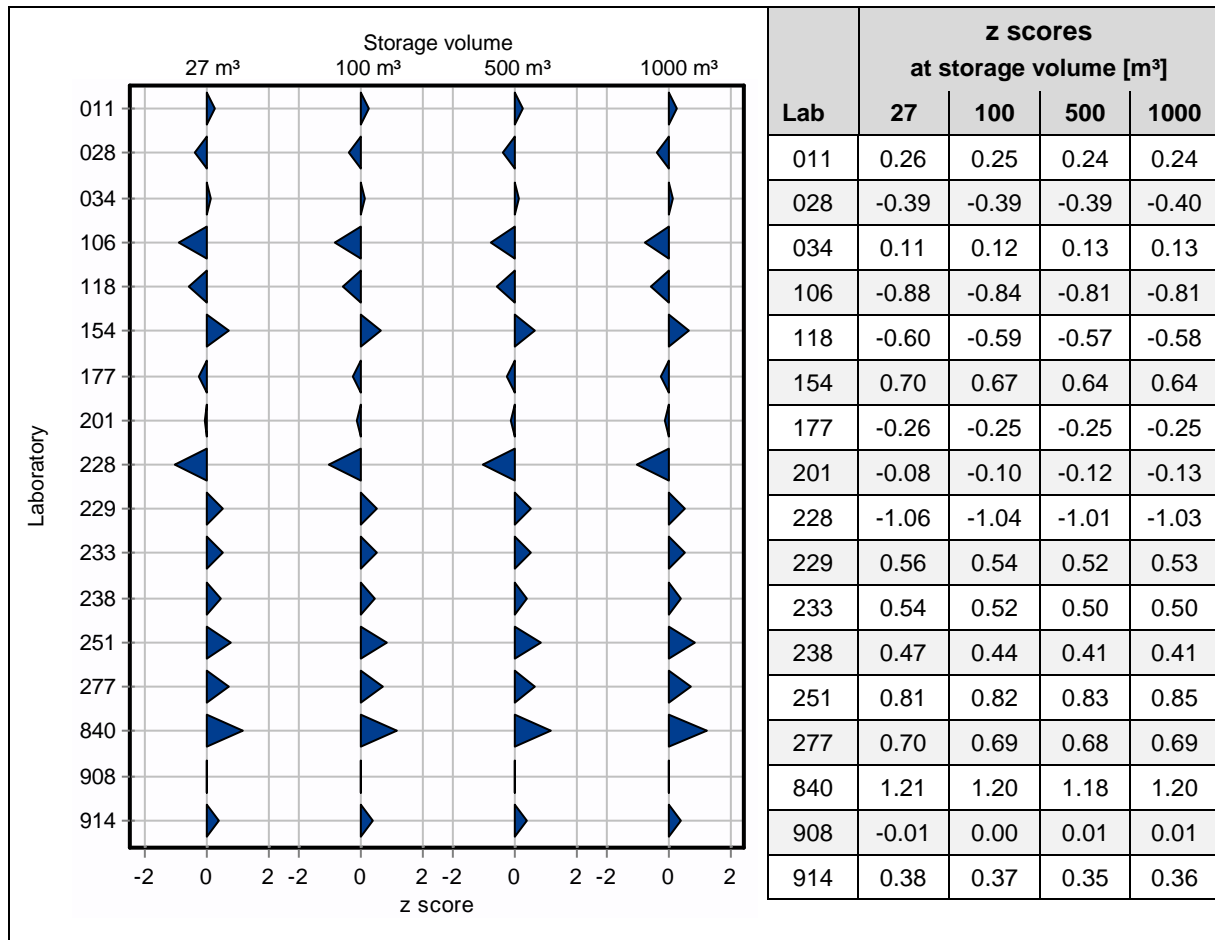


Figure 6-23: z scores based on the laboratory's mean value of T_{SI} for the storage volumes 27 m³, 100 m³, 500 m³ and 1000 m³

Conclusion: The z scores of all laboratories can be considered as satisfactory for all considered storage volumes. Considered labs as well as unconsidered labs (→ chapter 6.9.4) and laboratories which did not use the “glass bead” method in TS 3 as well as laboratories with unacceptable repeatability conditions exhibit a satisfactory performance. This indicates that the two suggested modifications (→ chapter 3.1 / modifications: mesh wire screen and volumes ratios of sample baskets of 1 : 1.7 : 5 : 8) are highly effective and should be implemented.

7 Summary and conclusions

Quality of the data

Due to the high number of participants, the accuracy of data check and the accurately performed data selection during the different steps of the statistical evaluation, the resulting conclusions can be assessed as highly safe.

Deviations from the requirements of the method DIN EN 15188:2007 and / or the interlaboratory test instructions, which may result in an incorrect statistical evaluation and thus in incorrect conclusions, were identified. These deviations have been taken into account in the statistical evaluation. The checked test results can be assessed as a sufficient basis for the statistical evaluation and for reliable conclusions (→ chapter 6.1.2).

Specifics and limitations concerning the usability of the submitted test results were identified and have been taken into account when performing the statistical analysis. The quantity of the submitted results can be assessed as sufficient for a statistical analysis (→ chapter 6.1.3).

The majority of laboratories exhibit a constant performance in TS 1 and TS 3 regarding the quality of the curves of the Pseudo-Arrhenius plot of self-ignition temperatures. Thus a sufficiently high level of compliance to the method and to repeatability conditions can be concluded. Summarizing: the majority of the data can be used, thus making it possible to obtain statistically reliable results. However, a high variability between laboratories regarding the quality of the curves of the Pseudo-Arrhenius plot can be observed (→ chapter 6.4).

Homogeneity and stability of the interlaboratory sample material “Carbon Black, Norit CN4”

By means of the test on homogeneity, it was shown that the interlaboratory sample material “Carbon Black, Norit CN4” can be considered as suitably homogenous for the interlaboratory test regarding the analysed parameters (→ chapter 4.2).

However, contrary to our expectations, the stability of the interlaboratory sample material “Carbon Black, Norit CN4” regarding the relative self-ignition temperature is questionable because of the notable shift of the relative self-ignition temperature during the testing period. It could be observed that the relative self-ignition temperature of the sample material decreased from September 2011 to January 2012 and then notably increased from March 2012 on (→ chapter 4.2). Furthermore the slopes of lab-specific curves of the Pseudo Arrhenius plot between TS 1 (yellow bars) and TS 3 (blue bars) and the significant changes in the lab-comprehensive “robust mean” values of the T_{SI} depending on the date of analysis (→ Figure 6-6) are indications of the instability of the interlaboratory test sample regarding the self-ignition behavior / T_{SI} measurements (→ chapter 6.4).

Thus it was necessary to correct the T_{SI} measurements by means of a statistical-computational elimination of the effect of the instability of the interlaboratory test sample (→ chapter 6.5). In order to derive a time-dependent and lab-comprehensive correction factor for the T_{SI} measurements, the lab-specific increases of T_{SI} measurements from TS 1 to TS 3 – depending on the date of analysis – were considered. For the time-dependent correction of the T_{SI} measurements the earliest date of laboratory’s analyses was chosen: 21.07.2011 as “reference date”. The time-dependent correction procedure of T_{SI} measurements can be assessed as suitable (→ chapter 6.5.3).

Aims of this interlaboratory test

The aims of this interlaboratory test were met and a clear picture concerning the suggested modifications of the method (→ chapter 3) can be shown by the results of this interlaboratory test.

The following conclusions can be drawn concerning the aim of this interlaboratory test.

7.1 Performance of the modified method DIN EN 15188

Rounding down procedure

It was demonstrated that the stipulated rounding down procedure as required in DIN EN 15188:2007 is not appropriate for example to get “safer or better” extrapolated T_{SI} values for larger volumes. To avoid mistakes and misinterpretations it is recommended to use only the non-rounded oven temperature T_{SI} measurements in order to calculate extrapolated T_{SI} for large volumes (→ chapter 6.6).

Volumetry

It was observed that the volumetric results of TS 1 (effective volume of sample baskets) scatter in a wide range.

The reason for this scattering may be due to manufacture tolerances of the sample baskets, to the lab specific measurement uncertainty, but also to different volumetric methods used by the different labs in TS 1 (→ chapter 6.7).

To reduce the uncertainty resulting from the different volumetric methods to determine the sample basket volumes, BAM has proposed a volumetric method as standard, where glass beads¹⁰ with a diameter of about 0.3 mm (→ Figure 6-11) should be used. It was shown that the results of other volumetric methods (“water” by lab 118 and “coal of known density” by lab 229) significantly deviate from the results which were analyzed by the aid of the recommended glass beads (→ chapter 6.7.1). However, it can be assumed that also by other methods (e.g. with “pulverulent alumina” by lab 233 and with “quartz sand” by lab 908) it may be possible to achieve sufficiently comparable results to the “glass beads” volumetric method. In order to limit deviations between the results of different labs and as long as no other method is sufficiently validated it is recommended to apply the volumetric method with glass beads as standard (reference).

Even if the effect by different volumetry on the extrapolated T_{SI} is not statistically significant for a given storage volume in this interlaboratory test, the effect may exist and may have been masked by other disturbing factors in this interlaboratory test.

Due to manufacture tolerances the effective volume of sample baskets may differ more or less from the projected nominal volume. Since the extrapolated T_{SI} for larger storage volumes are based on the volumes of the sample baskets used in the interlaboratory test, it is essential to check their effective volume by a reliable volumetric method.

Precision parameters of the modified method DIN EN 15188

Several internal investigations and interlaboratory comparisons in the past have shown significant differences between the results of hot storage tests by the method DIN EN 15188:2007.

¹⁰ The following suppliers were recommended in the interlaboratory test instruction of the TS 3 (→ Appendix 9.5.1):
<http://www.edmund-buehler.de/english/i-homogenisatoren-und-zellmuehlen.pml>
<http://www.sartorius-mechatronics.com/DE/en/index.htm> (product number: BBI-8541604)

The aim of this interlaboratory test 2010-2011 was to assess the suggested modifications (→ chapter 3 / modifications: mesh wire screen and volumes ratios of sample baskets of 1 : 1.7 : 5 : 8) by the investigation of one typical test sample material. “Carbon Black, Norit CN4” was chosen as a typical sample.

The comparison between the Pseudo-Arrhenius plot of self-ignition temperatures of Lycopodium powder (→ Figure 2-1) and the Pseudo-Arrhenius plot of self-ignition temperatures of “Carbon Black, Norit CN4” (→ e.g. Figure 6-8) demonstrates clearly the high relevance of the suggested modifications. The precision of the modified method DIN EN 15188 in this interlaboratory test 2010-2011 (i.e. under the suggested modifications of the method) where activated carbon powder “Carbon Black, Norit CN4” was used as test sample material, has clearly increased compared to a former interlaboratory test 2002 in accordance to VDI Guideline 2263 part 1 [15] where Lycopodium powder (spores) was used as test sample substance.

Not all data of all labs can be included in the statistical procedure to generate reliable precision data of the modified method. Nevertheless it can be assumed that the number of valid results from 10 labs is sufficient to generate reliable precision data of the modified method. (→ chapter 6.9.2).

The distribution of the extrapolated T_{SI} values can be assumed to be normal for all four considered storage volumes (→ chapter 6.9.3). Therefore it can be assumed that the data are sufficient to generate reliable precision parameters of the method.

The precision of the modified method DIN EN 15188 can be assessed as acceptable (→ Table 6-6). The ratio between repeatability s.d. s_r and reproducibility s.d. s_R is approximately 1:2. Thus considerable systematic deviations / errors by using the modified method cannot be assumed. The performance of the modified method DIN EN 15188 can be assessed as sufficient to determine the self-ignition temperature. (→ chapter 6.9.4)

The scattering of slopes of the curves Pseudo-Arrhenius plots of all labs (→ Figure 6-18) can be assessed as reduced in this interlaboratory test in comparison to the results of the non-modified method (→ chapter 2, Figure 2-1: Pseudo-Arrhenius plot of self-ignition temperatures of Lycopodium of the Round Robin Test 2002 (BAM)). Furthermore the z scores of all laboratories can be considered as satisfactory for all considered storage volumes. Considered labs as well as unconsidered labs (→ chapter 6.9.4) and laboratories which did not use the “glass bead” method in TS 3, as well as laboratories with unacceptable repeatability conditions, all exhibit a satisfactory performance. This indicates that the two suggested modifications (→ chapter 3.1 / modifications: mesh wire screen and volumes ratios of sample baskets of 1 : 1.7 : 5 : 8) are highly effective and should be implemented.

Measurement uncertainty on the basis of the reproducibility s.d. s_R of an interlaboratory test

Results and specially the reproducibility s.d. s_R obtained in interlaboratory tests are a valid basis for measurement uncertainty evaluation ([6], [7]). For example the expected self-ignition temperature T_{SI} for a volume of 27 m³ based on the current statistical evaluation of the laboratory's test results is equal to 50.2 °C ± 8°C.

It can be generalized that the measurement uncertainty U (for $k=2$) for extrapolated T_{SI} is not higher than 10 °C for storage volumes up to 1000 m^3 maximum. The measurement uncertainty cannot be ignored and must be considered if T_{SI} results should be used in the practice.

Validity of the measurement uncertainty for other test substances

It must be considered that the measurement uncertainty U ($k=2$) calculated by the equations in chapter 6.9.5 can only be used by the lab, if special criteria are fulfilled (→Table 6-7).

To avoid any doubts and to prevent any difficulties regarding the practical usage of the precision parameter (including the measurement uncertainty) the validity of the precision parameters must be ensured by additional tests with other, different substances which will cover a sufficiently wide range of self-ignition behaviour.

7.2 Other influencing (disturbing) factors

An influence of other factors (→ chapter 6.8) on the extrapolated T_{SI} cannot be observed in this interlaboratory test. However, the conclusiveness of these findings is limited because the aim and the testing concept of this interlaboratory test were mainly focused on the above mentioned modification of DIN EN 15188:2007. These effects could be checked by the aid of more specific investigations.

7.3 Recommendations for the participants of the interlaboratory test to improve the execution of the method

In view of the results of the interlaboratory test, the following recommendations for improving execution of the method can be given to the participating laboratories (→ Table 7-1).

Table 7-1: Recommendations to improve the execution of the method

Laboratory	Recommendation
011	No additional recommendations
028	No additional recommendations
034	<ul style="list-style-type: none"> • Improve/check the spatial temperature deviation inside the screen • Calibrate the whole measuring chain (sensors, compensating cable, logger, computer)
106	No additional recommendations
118	<ul style="list-style-type: none"> • Use glass beads for determining the volume of sample baskets • Calibrate the whole measuring chain (sensors, compensating cable, logger, computer) • Reduce noise in temperature signals (→Figure 7-1)
154	<ul style="list-style-type: none"> • Use recommended set-up (screen including sheet metal for additional shielding) • Improve stability of oven temperature (→Figure 7-1)
177	No additional recommendations
201	<ul style="list-style-type: none"> • Place both thermocouples for measuring the oven temperature inside the screen • If suggested set-up is used: Install an inflector plate in front of the fan to reduce the air flow in the centre of the oven

228	<ul style="list-style-type: none"> Reduce noise of temperature signal (→Figure 7-1)
229	<ul style="list-style-type: none"> Use glass beads for determining the volume of sample baskets
233	<ul style="list-style-type: none"> Improve/check the spatial temperature deviation inside the screen Reduce noise in temperature signals (→Figure 7-1) Use glass beads for determining the volume of sample baskets.
238	<ul style="list-style-type: none"> Use two thermocouples for measuring the oven temperature
251	<ul style="list-style-type: none"> Improve stability of oven temperature (→Figure 7-1) Calibrate the whole measuring chain (sensors, compensating cable, logger, computer)
277	<ul style="list-style-type: none"> Check procedure for determining the volume of sample baskets
840	<ul style="list-style-type: none"> Keep the inflector plate in front of the fan to reduce the air flow in the centre of the oven. Reduce noise in temperature signals (→Figure 7-1)
908	<ul style="list-style-type: none"> Calibrate the whole measuring chain (sensors, compensating cable, logger, computer)
914	No additional recommendations

The examples of temperature-time curves in Figure 7-1 illustrate sufficient and insufficient stability of oven temperatures and noise of temperature signals respectively.

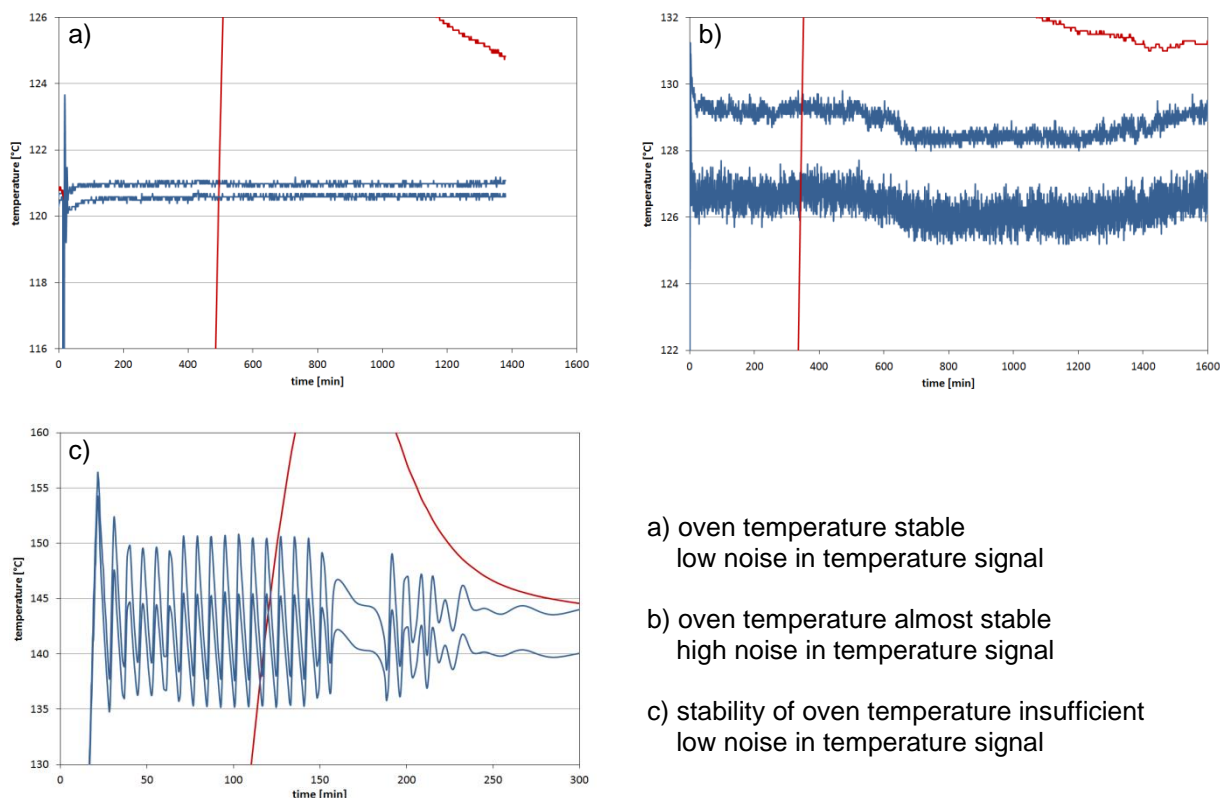


Figure 7-1: Temperature-time curves of different labs
blue curves- oven temperatures, red curves- sample temperatures

7.4 Recommendations to improve execution of the method

Based on the interlaboratory test, the gained experience and the actual results, the following measures / actions are recommended:

1. Training of personnel:

- Special samples should be manufactured and distributed centrally for use by the laboratories for their internal training / quality control (e.g. RM (reference material) or CRM (certified reference material)).
- An appropriate proficiency test scheme should be developed for regular external quality control.
- A scheme of so-called reference laboratories should be developed e.g. in order to compensate the lack of reference material.

2. Development of the method

- The description of the method should be revised in order to give a clear and unmistakable description of the method.
- The suggested modifications (→ chapter 3.1: mesh wire screen and volumes ratios of sample baskets of 1 : 1.7 : 5 : 8) should be implemented.
- The non-rounding procedure of the oven temperature on the extrapolated T_{SI} (→ chapter 6.6) should be implemented.
- A standardized volumetric method to determine the sample basket volumes (→ chapter 6.7) should be implemented.
- The range of the validity of the measurement uncertainty should be enlarged for other test substances (→ chapter 6.9.5).

8 References

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9 Appendix

9.1 Test of the homogeneity at the beginning of TS 1

Interlaboratory test 2010-2011
on the method DIN EN
15188:2007
Test on homogeneity



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Report

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04/18/2011



Interlaboratory test 2010-2011 on the method DIN EN 15188:2007 – Test on homogeneity

Inhalt

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RV 2010/11 „DIN EN 15188:2007 – Self-ignition temperature“ – Homogeneity test

1 Data basis

Based on experiences of former interlaboratory tests regarding homogeneity of the interesting parameter self-ignition temperature, the samples for the current interlaboratory test are tested for homogeneity on the basis of the following parameters:

- Particle size [μm]
- mass content water [%]
- caloric value [J/g]
- relative Self-ignition temperature acc. to Test EC A. 16 [$^{\circ}\text{C}$]

Here the median of the particle size is being used to prove homogeneity.

The sample material is Carbon Black, Type Norit CN4 on a pallet with altogether 48 bags at 20 kg (total: 960 kg).

For the test on homogeneity 12 bags were chosen randomly and the four interesting parameters were determined in duplicate. The obtained results are being displayed in Table 1 for the caloric value and the mass content of water and in Table 2 for the particle size and the relative self-ignition temperature.

Interlaboratory test 2010-2011 on the method DIN EN 15188:2007 – Test on homogeneity

Table 1: Obtained results in the test on homogeneity for the parameters *caloric value and mass content of water*

Parameter	Test portion	Bag no.	Date meas. value 1	Date meas. value 2	Meas. value 1	Meas. value 2	Measuring unit	Remarks
Caloric value	1	35	02/14/2011	03/02/2011	30063	30057	J/g	Determination of caloric value acc. to DIN 51900-3 For each sample 3 to 6 measurements have been performed. The mean value of the individual measurements is being listed.
	2	28	02/15/2011	03/02/2011	29954	29912	J/g	
	3	25	02/11/2011	03/02/2011	29883	29871	J/g	
	4	32	02/14/2011	03/07/2011	29999	29930	J/g	
	5	29	02/14/2011	03/07/2011	29991	29943	J/g	
	6	10	02/11/2011	03/07/2011	29976	29896	J/g	
	7	48	02/09/2011	03/08/2011	30116	30000	J/g	
	8	6	02/09/2011	03/08/2011	30025	29916	J/g	
	9	31	02/15/2011	03/08/2011	29985	29935	J/g	
	10	16	02/11/2011	03/09/2011	30120	29969	J/g	
	11	9	02/11/2011	03/09/2011	30095	29873	J/g	
	12	5	02/09/2011	03/09/2011	30026	29825	J/g	
Mass content water	1	35	02/08-02/10/2011	03/04-03/07/2011	9.238	8.435	%	Coulometric determination of water acc. to Karl-Fischer For each sample 2 to 3 measurements have been performed on various days. The mean value of the individual measurements is being listed.
	2	28	02/08-02/10/2011	03/04-03/07/2011	9.586	8.987	%	
	3	25	02/08-02/10/2011	03/04-03/07/2011	9.453	8.095	%	
	4	32	02/08-02/10/2011	03/04-03/07/2011	9.236	8.639	%	
	5	29	02/08-02/10/2011	03/04-03/07/2011	9.455	8.180	%	
	6	10	02/08-02/10/2011	03/04-03/07/2011	9.273	8.861	%	
	7	48	02/08-02/10/2011	03/04-03/07/2011	9.142	8.019	%	
	8	6	02/08-02/10/2011	03/04-03/07/2011	9.125	8.737	%	
	9	31	02/08-02/10/2011	03/04-03/07/2011	9.156	8.366	%	
	10	16	02/08-02/10/2011	03/04-03/07/2011	9.220	8.065	%	
	11	9	02/08-02/10/2011	03/04-03/07/2011	9.174	8.160	%	
	12	5	02/08-02/10/2011	03/04-03/07/2011	9.554	8.688	%	

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RV 2010/11 „DIN EN 15188:2007 – Self-ignition temperature“ – Homogeneity test

Table 2: Obtained results in the test on homogeneity for the parameters Particle size and relative self-ignition temperature

Parameter	Test portion	Bag no.	Date meas. value 1	Date meas. value 2	Meas. value 1	Meas. value 2	Measuring unit	Remarks
Particle size (median d(0.5))	1	35	02/09/2011	03/02/2011	31.580	31.213	µm	Measurement with laser diffraction spectrometer Order of the measurements Particle size: measurement series 1 sorted by bag number in ascending order measurement series 2 sorted by test portion in ascending order
	2	28	02/09/2011	03/02/2011	31.950	30.871	µm	
	3	25	02/09/2011	03/02/2011	32.599	30.857	µm	
	4	32	02/09/2011	03/02/2011	30.847	30.809	µm	
	5	29	02/09/2011	03/02/2011	31.424	30.805	µm	
	6	10	02/09/2011	03/02/2011	33.133	31.071	µm	
	7	48	02/09/2011	03/02/2011	30.010	31.129	µm	
	8	6	02/09/2011	03/02/2011	33.046	30.662	µm	
	9	31	02/09/2011	03/02/2011	30.886	30.863	µm	
	10	16	02/09/2011	03/02/2011	34.627	32.278	µm	
	11	9	02/09/2011	03/02/2011	34.083	30.637	µm	
	12	5	02/09/2011	03/02/2011	33.466	30.853	µm	
rel. self-ignition temperature A.16 (300 °C value)	1	35	02/25/2011	03/03/2011	219.7	218.7	°C	Temperature of the oven (temperature in surroundings of the sample) at the time when the centre of the sample reaches 300 °C
	2	28	02/24/2011	03/04/2011	218.3	220.1	°C	
	3	26	02/22/2011	03/07/2011	220.5	219.6	°C	
	4	32	02/23/2011	03/08/2011	219.9	219.8	°C	
	5	29	03/01/2011	03/08/2011	219.9	219.7	°C	
	6	10	02/14/2011	04/01/2011	220.5	221.2	°C	
	7	48	02/16/2011	04/04/2011	221.6	220.0	°C	
	8	6	02/17/2011	04/06/2011	221.4	221.1	°C	
	9	31	02/28/2011	04/06/2011	219.6	221.3	°C	
	10	16	02/14/2011	04/07/2011	220.5	220.9	°C	
	11	9	02/15/2011	04/08/2011	219.5	220.1	°C	
	12	5	02/18/2011	04/11/2011	219.4	221.3	°C	

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2 Results

There are temporal effects for the mass content of water as can be seen in Table 1: the longer the sample is being stored the lower its water content is. For the test on homogeneity this effect is being considered in so far as all measured values of the 2nd day of the measurement are being multiplied with the correcting factor 1.104, for all measured values of the 1st day of the measurement are on average 1.104 times higher. The corrected data basis for the mass content of water is given in the following Table 3.

Table 3: Corrected Data basis (regarding Measured value 2) for the parameter *Mass content of water*

Test portion	Measured value 1 [%]	Measured value 2 [%]
1	9.238	9.312
2	9.586	9.921
3	9.453	8.936
4	9.236	9.537
5	9.455	9.030
6	9.273	9.782
7	9.142	8.852
8	9.125	9.645
9	9.156	9.235
10	9.220	8.903
11	9.174	9.008
12	9.554	9.591

In the following Table 4 the achieved characteristic values of the homogeneity test for all four parameters are listed: mean, analytical precision (standard deviation within the test portions), heterogeneity standard deviation (standard deviation between the test portions) as well as the assigned standard deviation, which is set on twice the analytical precision in all four cases.

RV 2010/11 „DIN EN 15188:2007 – Self-ignition temperature“ – Homogeneity test

Table 4: Characteristic values of the homogeneity test

Characteristic	Measuring unit	Mean	Analytical precision	Heterogeneity s.d.	Assigned s.d.	ISO 13528 verification for sufficient homogeneity	Harmonized protocol verification for significant heterogeneity
Caloric value	J/g	29973	81	0	200	OK	OK
Mass content of water	%	9.31	0.24	0.15	0.5	OK	OK
Particle size	µm	31.6	1.3	0	3	OK	OK
Rel. self-ignition temperature	°C	220.2	0.8	0.33	2	OK	OK

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8

3 Conclusions

All four parameters fulfill the criteria for homogeneity according to ISO 13528 as well as according to the Harmonized protocol, that means the sample material which has been filled in various bags can be considered as homogenous.

4 References

- [1] ISO 13528
- [2] The International Harmonized Protocol for the Proficiency Testing of Analytical Chemistry Laboratories; Pure Appl. Chem., Vol. 78, No. 1, pp. 145–196, 2006.
- [3] A new test for 'sufficient homogeneity'; Tom Fearn and Michael Thompson; Analyst, 2001, 126, 1414–1417

9.2 Test on stability during TS 1, TS 2 and TS 3

Interlaboratory test 2010-2011
on the method DIN EN
15188:2007
Test on stability



Kirstin Kunath
Kirsten Simon
Steffen Uhlig

Report

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11/26/2011



Interlaboratory test 2010-2011 on the method DIN EN 15188:2007 – Test on stability

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1 Data basis

BAM (Federal Institute for Materials Research and Testing) already proved sufficient stability for material comparable to Carbon black (Co. Norit) within a former proficiency test, that means there were no crucial changes of the self-ignition temperature in the sample according to EN 15188:2007 (Self-ignition temperature) then over a period of more than 20 months.

The current interlaboratory test is being performed with comparable material of the same producer wherefore only a checkup was performed in order to prove the main parameter *self-ignition temperature*. With regard to a close-to-DIN- and – over the period of performance of the current proficiency test – regular test on stability the relative self-ignition temperature is used as proof of stability. This parameter already fulfilled the criteria of homogeneity within the test of homogeneity according to ISO 13528 as well as according to the Harmonized protocol. Hence, the sample material which has been filled in various bags may be considered sufficiently homogenous.

For the test on stability those samples were chosen, which already have been considered in the test on homogeneity. The studies were randomized and performed in duplicate under repeatability conditions (same person for the respective parameter).

The temporal order of the tests on homogeneity and stability as well as the respective evaluated parameters are being displayed in the following Table 1.

Interlaboratory test 2010-2011 on the method DIN EN 15188:2007 – Test on stability

Table 1: Temporal order of the tests on homogeneity (H) and stability (S) with specification of the respective evaluated parameters in TS 1

	Month of study	Year	Bag no.										Evaluated parameters							
			35	28	25	32	29	10	48	6	31	16	9	5	rel. self-ignition temperature	Particle size	Mass content	Caloric value		
H	February	2011															x	x	x	x
S	June	2011															x	x	x	x
	July	2011															x			
	August	2011															x			
	September	2011															x			
	October	2011															x			
	November	2011															x			
	December	2011															x			
	January	2012															x			
	March	2012															x			
	May	2012															x			
	July	2012															x			
	September	2012															x			
	November	2012															x			

Interlaboratory test 2010-2011 on the method DIN EN 15188:2007 – Test on stability

Table 2: Obtained test results within the test on stability for the parameter *relative self-ignition temperature*

Period of study	Bag no.	Date Meas. value 1	Date Meas. value 2	Meas. value 1 [°C]	Meas. value 2 [°C]	Remarks
June 2011	35	06/08/2011	06/10/2011	218.2	218.1	
	28	06/09/2011	06/13/2011	218.3	218.9	
	35	06/22/2011	06/23/2011	220.3	220.0	Additional test in June: test for the aliquots used for the test on homogeneity – which have been stored in tightly closed cases – measured again
	28	06/24/2011	06/27/2011	219.9	219.2	
	35	06/28/2011	06/29/2011	218.1	219.3	Repetition with new aliquots
	28	06/30/2011	07/01/2011	218.0	218.2	
	35	06/22/2011	06/23/2011	220.3	220.0	Additional test in June: test for the samples used for the test on homogeneity (test on stability 1) – which have been stored in tightly closed cases
	28	06/24/2011	06/27/2011	219.9	219.2	
	35	06/28/2011	06/29/2011	218.1	219.3	Repetition with new samples
	28	06/30/2011	07/01/2011	218.0	218.2	
July 2011	25	07/12/2011	07/13/2011	219.4	219.0	
	32	07/14/2011	07/15/2011	218.1	217.9	Aliquot taken from already existing opening of the paper bag
August 2011	29	08/08/2011	08/09/2011	221.2	219.5	
	10	08/10/2011	08/11/2011	218.8	219.9	
September 2011	48	09/07/2011	09/08/2011	218.9	219.1	
	6	09/09/2011	09/12/2011	219.9	220.6	
October 2011	31	07/10/2011	10/10/2011	218.6	219.2	
	16	10/13/2011	10/19/2011	218.2	219.2	
November 2011	9	11/07/2011	11/08/2011	218.0	217.5	
	5	11/09/2011	11/10/2011	217.3	217.6	
December 2011	35	12/07/2011	12/08/2011	219.1	216.5	
	28	12/09/2011	12/12/2011	216.6	218.6	
January 2012	25	01/05/2012	01/06/2012	219.1	217.9	Aliquot transferred into hobbocks at the end of December 2011, before: storage in original container (foil-wrapped paper bag)
	32	01/09/2012	01/10/2012	216.8	218.2	

Interlaboratory test 2010-2011 on the method DIN EN 15188:2007 – Test on stability

Period of study	Bag no.	Date Meas. value 1	Date Meas. value 2	Meas. value 1 [°C]	Meas. value 2 [°C]	Remarks
March 2012	29	03/19/2012	03/13/2011	220.9	221.0	
	10	03/20/2012	03/15/2012	222.4	223.8	
May 2012	48	05/14/2012	05/15/2012	223.9	223.8	
	6	05/16/2012	05/21/2012	224.2	225.9	
July 2012	31	07/06/2012	07/09/2012	225.9	225.8	
	16	07/10/2012	07/11/2012	225.5	226.1	
September 2012	9	09/14/2012	09/19/2012	222.5	221.1	
	5	09/12/2012	09/13/2012	222.6	220.8	
November 2012	35	10/30/2012	10/31/2012	224.3	223.2	
	28	11/01/2012	11/02/2012	223.4	224.4	

2 Results

In the following Figure 1, the results of the test on stability for the parameter *relative self-ignition temperature* are shown graphically. Here, the blue horizontal lines characterize the mean value of the two replicates and the vertical black line the respective standard deviation (in each direction).

It can be seen that the relative self-ignition temperature seems to be increasing up to January 2012. From March 2012 on, there is a highly increase. However, in both years 2011 and 2012 there is a local peak in July/August. But while in November 2011 the relative self-ignition temperature decreases, an obvious increase in November 2012 is visible.

Interlaboratory test 2010-2011 on the method DIN EN 15188:2007 – Test on stability

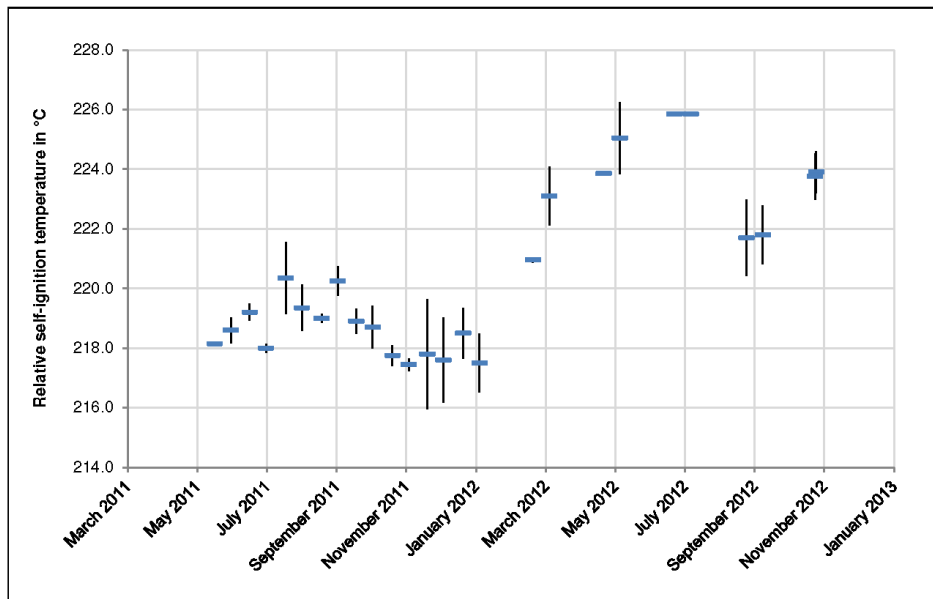


Figure 1: Results of the test on stability regarding the parameter relative self-ignition temperature (blue = mean value; black vertical line = \pm standard deviation)

3 Conclusions

The stability of the sample material regarding the relative self-ignition temperature is questionable due to the highly irregular trend. However, in order to evaluate the interlaboratory test's results, there is a correction based on the stability data results recommended.

Interlaboratory test 2010-2011 on the method DIN EN 15188:2007 – Test on stability

4 References

[1] ISO 13528

9.3 Instructions and forms of TS 1

9.3.1 Test instruction

BAM Federal Institute for Materials Research and Testing

Department 2 "Chemical Safety Engineering"
Working group "Flammable Bulk Materials and Dusts, Solid Fuels"
in co-operation with

Center for quality assurance for testing of dangerous goods and hazardous substances



BAM Federal Institute for Materials
Research and Testing



Quality Management and Statistics
GmbH



Physikalisch-Technische
Bundesanstalt (PTB)

Interlaboratory test 2010/11 „DIN EN 15188:2007 – Self-ignition temperature“

Instruction to perform the Interlaboratory Test

2011-06-21

Dear colleagues,

As announced in September 2010 the BAM in co-operation with the *Center for quality assurance for testing of dangerous goods and hazardous substances* agreed to perform a standardized Interlaboratory test on the method „EN 15188:2007 Determination of the spontaneous ignition behaviour of dust accumulations“¹.

18 laboratories did confirm to participate in this Interlaboratory test. Each participant will get a report and a certificate of participation² which allows him to identify his laboratory's results by the lab-code. All data and the evaluation will be dealt with anonymously. No other participant will be able to assign other results to other laboratory.

For this Interlaboratory test the substance "Carbon Black, Norit CN4" was chosen as test substance.

Together with the test substance "Carbon Black, Norit CN4" (approx. 30 l) you receive from BAM a prepared data input form (xls-file) for the acquisition of your measurement data. Some laboratories have ordered and received standardized test baskets for the samples and test screens.

The interlaboratory test will be performed stepwise as a multi-level test (see Table 1). Test step 1 is obligate for all participating laboratories. Test step 2 and 3 will be executed in dependence on the results of previous test steps (step 1 and/or 2).

¹ DIN EN 15188:2007 Determination of the spontaneous ignition behaviour of dust accumulations, Beuth Verlag, Berlin

² Note: The interlaboratory test is not a proficiency test.

Continuation of the test instruction on the next page.

Continuation of the test instruction:

Interlaboratory test DIN EN 15188:2007 - Instruction**Page 2****Table 1: Interlaboratory test steps**

Test step TS	Additional measures	Remark
1	No	Standard test volumes for all and additional test volumes (optional)
2	exchange of equipment (ovens, temperature sensors) between laboratories	Optional in dependence on results of test step 1 and if necessary for only some laboratories
3	repeat of tests under the conditions of test step 1	Optional in dependence on results of test step 1 + 2

Therefore we ask the participating laboratories to perform the DIN EN 15188:2007 test with this substance submitted to you by BAM.

We ask you to perform the tests of step 1 by end of August 2011 and to send back the duly completed data file and the raw data files of the tests (see data input file) by September 5th 2011 to Peter Lueth via email: peter.lueth@bam.de.

1st Interlaboratory test step

Please perform the 1st Interlaboratory test step under the following special conditions:

- The tests should always be performed by one and the same laboratory assistant, otherwise it shall be noted.
- The sample shall be tested as delivered (do not dry, sieve, grind etc.).
- The sample shall be mixed by hand-shaking the closed container (hobcock) for at least 10 second before filling the sample basket.
- The sample shall be stored in the container which you have received from BAM over the whole testing period. The container shall keep close during storage.
- The sample shall be shielded by using an additional screen installed in the oven, see Figure 1. Use the test screen manufactured by BAM (if you have received these from BAM).
- The temperature difference between „go“ and „no go“ shall be 2 K maximum.
- The required standardized defined bulk density is: 460 kg/m³ (range 455...465 kg/m³)
5 cm sample basket (cube), 125 mL: 57.5 g (56,2 g ... 58,7 g)
6 cm sample basket (cube), 216 mL: 99.3 g (97,2 ...101,5 g)
8,5 cm sample basket (cube), 614 mL: 282,5 g (276,3 ... 282,5 g)
10 cm sample basket (cube), 1000 mL: 460 g (450 ... 470 g).
- Standard test volumes:** 4 volumes to be tested (volumes = 5 cm, 6 cm, 8,5 cm and 10 cm cubes, which were manufactured / distributed by BAM). The samples baskets shall be double-walled. The minimum volume of the smallest basket is 100 ml; the minimum volume of the largest basket is 1000 ml.
- Additional test volumes** (optional): If possible please perform additional test with volumes smaller 100 mL and/ore volumes larger 1000 mL
- The oven should be pre-heated, if possible. If not possible, please remark (data input form).

Continuation of the test instruction on the next page.

Continuation of the test instruction:

Interlaboratory test DIN EN 15188:2007 - Instruction

Page 3

11. Standardized temperature gradient inside the oven:

Determine the temperature gradient within the shielding in a standard way.

11.1 The oven shall be equipped with the shielding screen and a 1000 ml sample basket (empty, without sample).

11.2 Adjust the oven temperature to 120 °C (temperature inside the screen as measured during the hot storage tests; arithmetic mean of both temperature sensors installed).

Do not change the temperature setting of the oven for the whole test (step 11.3).

11.3 Place a temperature sensor between the sample basket and the wall of the screen at a distance of 5 cm from the sample basket. Carry out this step using the same temperature sensor for the following positions:

- to the right of the sample basket
- to the left of the sample basket
- in front of the sample basket
- behind the sample basket
- above the sample basket
- below the sample basket

Determine the respective temperature when the temperature became stable.

12. Moisture of sample (gravimetric test): The moisture of the sample shall be determined at the beginning (first day) and the end (last day) of the whole hot storage tests. A sample shall be dried in an oven at a temperature of 105 °C for 24 hours. The sample mass shall be about 50 g. The sample shall be dried as a layer of a thickness of ≤ 5 mm.

All the other procedures as prescribed in DIN EN 15188:2007 "Determination of the spontaneous ignition behaviour of dust accumulations" should be applied as usual in your laboratory.

Important:

Do not remove / dispose the test sample after test step 1. It is possible that additional tests are necessary (2nd and 3rd Interlaboratory test steps (see Table 1)).

2nd and 3rd Interlaboratory test steps

The investigations will be performed in dependence on the results of the 1st step and/or 2nd step. On the basis of a pre-analysis of the results it has to decide whether this test steps are necessary or not. We will inform you about the next test steps 2 and 3 as soon as possible after the pre-analysis of the results of test step 1.

The tests of the 2nd test step should be performed in the same way as in the 1st test step. Only the equipment (e.g. oven, temperature sensors) should be changed.

The test of the 3rd test step should be performed in the same way as in the 1st test step (repetition).

Contact:

If you have any questions or difficulties with the schedule, please do not hesitate to contact:
Dr. Martin Schmidt via Email (martin.schmidt@bam.de) or telephone +49+030-8104 4443 or
Dr. Peter Lueth via Email (peter.lueth@bam.de) or telephone +49+030-81041201.

We thank you for participating in this interlaboratory test and wish you a successful performance.

Dr. Martin Schmidt and Dr. Peter Lueth (BAM)
June 2011

Continuation of the test instruction on the next page.

Continuation of the test instruction:

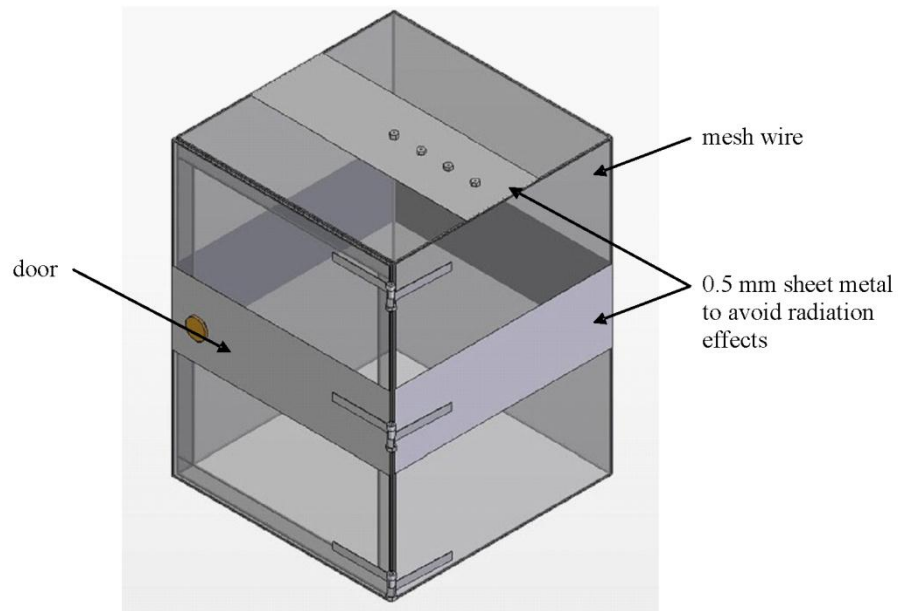


Figure 1: Mesh wire screen to be installed into the hot storage oven

9.3.2 Laboratory data input form

BAM, Department 2 "Chemical Safety Engineering", Working group "Flammable Bulk Materials and Dusts, Solid Fuels"
 in co-operation with
 Center for quality assurance for testing of dangerous goods and hazardous substances (BAM, qou data GmbH, PTB)

Interlaboratory test 2010/11 „DIN EN 15188:2007 – Self-ignition temperature“ - 1st step

Data input form

Please complete the grey fields.

Laboratory-No:

Description of the test equipment:

Laboratory oven - type, manufacturer:	<input type="text"/>
Laboratory oven - size [l]:	<input type="text"/>
Laboratory oven - convection (natural/forced):	<input type="text"/>
Manufacturer/type of basket and screen (e.g. manufactured by BAM):	<input type="text"/>
Screen size (length,depth,height) [cm]:	<input type="text"/>
Type of temperature sensor (thermocouple):	<input type="text"/>
Calibration of the test sensor [yes/no]:	<input type="text"/>

Continuation of the laboratory data input form on the next page.

Continuation of the laboratory data input form:

<p>(Please fill out) Further description of the used test equipment: Kind of screen and sample baskets (cubes) (mesh size, form / profile...)</p>	<p>Description of the storage conditions of the test sample during the interlaboratory test:</p> <p>(Please fill out) Description of the storage conditions of the test sample during the interlaboratory test: (room, temperature, moisture ...):</p>	<p>Description of your procedure to fill the sample basket:</p> <p>(Please fill out) Description of your procedure of filling the sample baskets (number of portions, intensity of tapping, equipment (eg. funnel, rod, brush), duration...):</p>
--	--	---

Continuation of the laboratory data input form on the next page.

Continuation of the laboratory data input form:

Standardized temperature gradient inside the oven	
Position in the oven*	Temperature [°C] (1 decimal, e.g. 120,1)
left	
right	
at the head	
on the bottom	
in front	
back	
* Position see instruction	

Moisture of the sample at the beginning and at the end of the testing period				
Time	Testing date	sample mass before drying [g] (2 decimals, e.g. 49,67)	sample mass after drying [g] (2 decimals, e.g. 48,99)	Moisture of the sample* [%] (1 decimal, e.g. 48,9)
at the beginning of the whole test (first day)	xx.xx.2011			#DIV/0!
at the end of the whole test (last day)	xx.xx.2011			#DIV/0!
* Moisture of the sample see instruction				

Continuation of the laboratory data input form on the next page.

Continuation of the laboratory data input form:

Air inlet and air outlet openings of the oven open? *** [yes/no]	Oven temperature of 1st sensor *** [°C]; (calibrated, 1 decimal), e.g. 153,3	Position of 1st sensor in the oven space (description of the distance to the sample basket - free text)	Oven temperature of and sensor *** [°C]; (calibrated, 1 decimal), e.g. 153,3	Position of and sensor in the oven space (description of the distance to the sample basket - free text)	Oven temperature - Mean value of 1st and 2nd sensor *** [°C]	Oven temperature T _{is} *** (rounded off result of the laboratory) [°C] (no decimal, e.g. 153)	Self-ignition [yes / no]	max. Temperature of the sample [°C]	Lab. ID-N. of the temperature sensor (if available)	Remarks	File name of test (raw data) [xxx.xls]
					#DIV/0!		yes				
					#DIV/0!		no				
					#DIV/0!		yes				
					#DIV/0!		no				
					#DIV/0!		yes				
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9.3.3 Additionally test instruction

BAM Federal Institute for Materials Research and Testing

Department 2 "Chemical Safety Engineering"
Working group "Flammable Bulk Materials and Dusts, Solid Fuels"
in co-operation with

**Center for quality assurance for testing
of dangerous goods and hazardous substances**



BAM Federal Institute for Materials
Research and Testing



Quality Management and Statistics
GmbH



Physikalisch-Technische
Bundesanstalt (PTB)

Interlaboratory test 2010/11

„DIN EN 15188:2007 – Self-ignition temperature“

Important information to the “Instruction to perform the Interlaboratory Test” and additional data input form B

2011-08-15

Dear colleagues,
Please consider the following important information.

1. Quantity of the test sample:

After reviewing the first results of two laboratories, unfortunately, we found that there are some misunderstandings concerning the required standardized filling quantities of the test sample baskets. To avoid further misunderstandings we like to draw your attention to the special condition 7 of the “Instruction to perform the Interlaboratory test”. The range of the quantity of the test sample to fill in the test sample basket should be:

5 cm sample basket (cube), **125 mL: from 56,2 g to 58,7 g**
6 cm sample basket (cube), **216 mL: from 97,2 g to 101,5 g**
8,5 cm sample basket (cube), **614 mL: from 276,3 g to 282,5 g**
10 cm sample basket (cube), **1000 mL: from 450 g to 470 g**

2. Volume of test baskets and additional data input form B:

Due to the fact that the delivered test baskets have manufacturing tolerances we would like to ask you to determine the effective volume of each basket.

It is recommended to use material of an invariant bulk density to do the volumetric analyses (not dependent on degree of compaction); for example table salt or fine sand. The bulk density of the used material has to be determined by your own laboratory. Please do not use the carbon black since the bulk density of this material strongly depends on the degree of compaction.

Determining the volume by measuring the dimensions will also lead to inexact results.
Please use the data input form B to transmit the data.

Contact:

If you have any questions, please do not hesitate to contact:

Dr. Martin Schmidt via Email (martin.schmidt@bam.de) or telephone +49+030-8104 4443 or

Dr. Peter Lueth via Email (peter.lueth@bam.de) or telephone +49+030-81041201.

Continuation of the test instruction on the next page.

Continuation of the test instruction:

Interlaboratory test DIN EN 15188:2007 - Instruction

Page 2

Best regards,

Dr. Martin Schmidt and Dr. Peter Lueth (BAM)

Dr. Peter Lüth

BAM Bundesanstalt für Materialforschung und -prüfung
Abteilung 2 "Chemische Sicherheitstechnik"
Arbeitsgruppe "Rechtsfortentwicklung in der chemischen Sicherheitstechnik"

BAM Federal Institute for Materials Research and Testing
Department 2 "Chemical Safety Engineering"
Working group "Legislation in Chemical Safety Engineering"

Unter den Eichen 87
D-12205 Berlin

Telefon: +49 (0)30 8104-1201
Telefax: +49 (0)30 8104-1207
e-mail: peter.lueth@bam.de
www: <http://www.bam.de>

9.3.4 Additionally laboratory data input form B

BAM, Department 2 "Chemical Safety Engineering", Working group "Flammable Bulk Materials and Dusts, Solid Fuels"
 in co-operation with
 Center for quality assurance for testing of dangerous goods and hazardous substances (BAM, qou data GmbH, PTB)

Interlaboratory test 2010/11 „DIN EN 15188:2007 – Self-ignition temperature“ - 1st step

Data input form B (additional)

Please complete the grey fields.

Laboratory-No:

Description of the test equipment - volume of the test sample baskets / cubes:

Results

Test sample basket no. (nominal volume)	Effective (real) volume of the sample basket [ml]
1 (125 ml *)	<input type="text"/>
2 (216 ml *)	<input type="text"/>
3 (614 ml *)	<input type="text"/>
4 (1000 ml *)	<input type="text"/>

* ... recommended by the test instruction (= 5 cm, 6 cm, 8,5 cm and 10 cm cubes, which were manufactured / distributed by BAM)

Please send back this completed data form by September 5th 2011 to Peter Lueth (peter.lueth@bam.de)

9.4 Instruction of TS 2

9.4.1 Test instruction

Email

Von: Lüth, Peter
Gesendet: Montag, 20. Februar 2012 11:37
An: Lüth, Peter; Schmidt, Martin
Betreff: Interlaboratory test on the method „DIN EN 15188:2007 - Self-ignition temperature" - Start of Step 2 - Check of 4 sample baskets
Anlagen: 20110621_RR Interlaboratory Test Instruction.pdf; 20110621 RR Data Input Form Lab xxx.xls
Wichtigkeit: Hoch

Dear colleagues,
Thank you very much for participating in the interlaboratory test on the method „DIN EN 15188:2007 - Self-ignition temperature“.

As described in the interlaboratory test instruction of 2011-06-21 the interlaboratory test will be performed stepwise as a multi-level test. The tests of step 1 of the interlaboratory test were performed in the last year.

The pre-analysis of the results of test step 1 shows clearly that it is necessary to check the sample baskets which you have used during the tests last year (at the 1st step of the interlaboratory test).

For this purpose we ask you to **send the four sample baskets**, which you have used at the 1st step, **by March 2nd 2012** to the following address:

Dr. Peter Lüth
BAM Bundesanstalt für Materialforschung und -prüfung
Abteilung 2 "Chemische Sicherheitstechnik"
Unter den Eichen 87
D-12205 Berlin
Alemagne

After this check we will send back to you the baskets.

If you have any questions or difficulties with the schedule, please do not hesitate to contact:
Dr. Martin Schmidt via Email (martin.schmidt@bam.de) or telephone +49+030-8104 4443 or
Dr. Peter Lueth via Email (peter.lueth@bam.de) or telephone +49+030-81041201.

Best regards

Peter Lüth and Martin Schmidt (BAM)

Dr. Peter Lüth
BAM Bundesanstalt für Materialforschung und -prüfung
Abteilung 2 "Chemische Sicherheitstechnik"
BAM Federal Institute for Materials Research and Testing
Department 2 "Chemical Safety Engineering"
Unter den Eichen 87
D-12205 Berlin
Telefon: +49 (0)30 8104-1201
Telefax: +49 (0)30 8104-1207
e-mail: peter.lueth@bam.de
www: <http://www.bam.de>

9.5 Instruction and form of TS 3

9.5.1 Test instruction

BAM Federal Institute for Materials Research and Testing

Department 2 "Chemical Safety Engineering"
in co-operation with

Center for quality assurance for testing
of dangerous goods and hazardous substances



BAM Federal Institute for Materials
Research and Testing



Quality Management and Statistics
GmbH



Physikalisch-Technische
Bundesanstalt (PTB)

Interlaboratory test 2010/11 „DIN EN 15188:2007 – Self-ignition temperature“ Instruction to perform the interlaboratory test Step 3

2012-04-04

Dear colleagues,

As described in the interlaboratory test instruction of 2011-06-21 the interlaboratory test will be performed stepwise as a multi-level test (see Table 1). The tests of step 1 and 2 were performed during the last 12 months.

Table 1: Interlaboratory test steps

Test step	Additional measures	Remark	Status
1	No	Standard test volumes for all and additional test volumes (optional)	✓
2	Exchange of equipment between laboratories	Result of the pre-analysis: a. Check the sample baskets: (all laboratories) b. Repeat of step 1 using a smaller screen (lab no 840)	✓
3	Repeat of tests under the conditions of test step 1	Decision after analysis of the results of test step 1 and 2	Opened (period: April – September 2012)

On the basis of an analysis of the results of test step 1 and 2 it was decided to perform the next test step 3. The test of the 3rd test step should be performed in the same way as in the 1st test step (repetition).

We ask you to perform the tests of step 3 by end of September 2012 and to **send back the duly completed data input file by October 1st 2012** to Peter Lueth via email: peter.lueth@bam.de.

Continuation of the test instruction on the next page.

Continuation of the test instruction:

Interlaboratory test DIN EN 15188:2007 – Instruction 3rd step**Page 2****3rd Interlaboratory test step**Please perform the 3rd Interlaboratory test step under the following special conditions:

1. The tests should always be performed by one and the same laboratory assistant as in the 1st test step, otherwise it shall be noted.
2. The sample shall be tested as delivered (do not dry, sieve, grind etc.).
3. The sample shall be mixed by hand-shaking the closed container (hobcock) for at least 10 second before filling the sample basket.
4. The sample shall be stored in the container which you have received from BAM over the whole testing period. The container shall keep close during storage.
5. The sample shall be shielded by using an additional screen installed in the oven in the same way as in the 1st test step.
6. The temperature difference between „go“ and „no go“ shall be 2 K maximum.
7. The required **standardized defined bulk density** is 450 kg/m³ to 470 kg/m³:

5 cm sample basket (cube, approx. 125 mL):	from 56,2 g to 58,7 g
6 cm sample basket (cube), approx.216 mL):	from 97,2 g to 101,5 g
8,5 cm sample basket (cube), approx.614 mL):	from 276,3 g to 288,6 g
10 cm sample basket (cube), approx.1000 mL):	from 450 g to 470 g
8. **Standard test volumes:** 4 volumes to be tested (volumes = 5 cm, 6 cm, 8,5 cm and 10 cm cubes. **Use the sample baskets which we have sent together with this instruction (which were checked by BAM in March 2012).**
9. **Additional test volumes** (optional): If possible please perform additional test with volumes smaller 100 mL and/or volumes larger 1000 mL.
10. **Effective volume of the test baskets:** Due to the manufacturing tolerances test baskets and differences the volumetric the effective volume of each basket shall be determined by using **glass beads (diameter ~ 0.3 mm)**¹. Otherwise it shall be noted what material you have used.
11. The oven should be pre-heated, if possible. If not possible, please remark (data input form).
12. **Moisture of sample (gravimetric test):** The moisture of the sample shall be determined at the beginning (first day) and the end (last day) of the whole hot storage tests. A sample shall be dried in an oven at a temperature of 105 °C for 24 hours. The sample mass shall be about 50 g. The sample shall be dried as a layer of a thickness of ≤ 5 mm.

Important:

Do not remove / dispose the test sample. It will be needed, if additional interlaboratory tests are necessary.

Contact:

If you have any questions or difficulties with the schedule or if you need additional test sample material or if you require any further details about the 3rd step of interlaboratory test, please do not hesitate to contact:

Dr. Martin Schmidt via Email (martin.schmidt@bam.de) or telephone +49+030-8104 4443 or
 Dr. Peter Lueth via Email (peter.lueth@bam.de) or telephone +49+030-81041201.

We thank you for participating in this 3rd step of the interlaboratory test and wish you a successful performance.

Dr. Martin Schmidt and Dr. Peter Lueth (BAM)
 April 2012

¹ Possible suppliers:

<http://www.edmund-buehler.de/english/i-homogenisatoren-und-zellmuehlen.pml>

<http://www.sartorius-mechatronics.com/DE/en/index.htm> (product number: BBI-8541604)

9.5.2 Laboratory data input form

BAM, Department 2 "Chemical Safety Engineering"
 in co-operation with
 Center for quality assurance for testing of dangerous goods and hazardous substances (BAM, qou data GmbH, PTB)

Interlaboratory test 2010/11 „DIN EN 15188:2007 – Self-ignition temperature“ - 3rd step

Data input form C

Please complete the grey fields.

Laboratory-No:

Description of the test equipment:

Laboratory oven - type, manufacturer	
Laboratory oven - size [l]	
Laboratory oven - convection (natural/forced):	
Manufacturer / type of basket and screen (e.g. manufactured by BAM):	
Screen size (length,depth,height) [cm]:	
Type of temperature sensor (thermocouple):	

Calibration of the test sensor [yes/no]:	
Calibration document available (e.g. certificate, protokoll, report ...): [yes/no]	

If yes, please, send us a copy if it is possible.

Calibration of the whole temperature test chain [yes/no]:	
Calibration document available (e.g. certificate, protokoll, report ...): [yes/no]	

"whole temperature test chain", including sensor, compensating cable, data logger, computer etc.
If yes, please, send us a copy if it is possible.

Continuation of the laboratory data input form on the next page.

Continuation of the laboratory data input form:

<p>(Please fill out) Further description of the used test equipment: Kind of screen and sample baskets (cubes) (mesh size, form / profile...)</p>
<p>Description of the storage conditions of the test sample during the interlaboratory test:</p>
<p>(Please fill out) Description of the storage conditions of the test sample during the interlaboratory test: (room, temperature, moisture ...):</p>
<p>Description of your procedure to fill the sample basket with test sample:</p>
<p>(Please fill out) Description of your procedure of filling the sample baskets (number of portions, intensity of tapping, equipment (e.g. funnel, rod, brush), duration...):</p>

Continuation of the laboratory data input form on the next page.

Continuation of the laboratory data input form:

Determination of the effective volume of the test sample baskets / cubes:

Material used for volumetry (type, manufacturer):

(Please fill out)
Description of your procedure of volumetry
 (e.g. filling the baskets with material for volumetry, intensity of tapping,...):

Test sample basket no. (nominal volume)	Effective (real) volume of the sample basket [ml]			Mean value
	Trial 1	Trial 2 (1 decimal, e.g. 128,1)	Trial 3	
1 (125 ml*)				#DIV/0!
2 (216 ml*)				#DIV/0!
3 (614 ml*)				#DIV/0!
4 (1000 ml*)				#DIV/0!

* ... recommended by the test instruction (= 5 cm, 6 cm, 8,5 cm and 10 cm cubes, which were manufactured / distributed by BAM)

Moisture of the sample at the beginning and at the end of the testing period

Time	Testing date	sample mass before drying [g] (2 decimals, e.g. 49,67)	sample mass after drying [g] (2 decimals, e.g. 48,99)	Moisture of the sample [%] (1 decimal, e.g. 48,9)
at the beginning of the whole test (first day)	xx.xx.2012			#DIV/0!
at the end of the whole test (last day)	xx.xx.2012			#DIV/0!

*...Moisture of the sample see instruction

Continuation of the laboratory data input form on the next page.

Continuation of the laboratory data input form:

Air inlet and air outlet openings of the oven open? [yes/no]	Oven temperature of 1st sensor [°C] (calibrated, 1 decimal, e.g. 153.2)	Position of 1st sensor in the oven space (description of the distance to the sample, basket-free test)	Oven temperature of 2nd sensor [°C] (calibrated, 1 decimal, e.g. 153.2)	Position of 2nd sensor in the oven space (description of the distance to the sample, basket-free test)	Oven temperature - Mean value of 1st and 2nd sensor [°C]	Oven temperature T _o [°C] (rounded off result of the laboratory) (no decimal, e.g. 153)	Self ignition [yes / no]	max. Temperature of the sample [°C]	Lab ID-Nr. of the temperature sensor (if available)	Remarks	File name of test (raw data) [xxx.xls]
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					#DIV/0!		yes				
					#DIV/0!		no				
					#DIV/0!		yes				
					#DIV/0!		no				
					#DIV/0!		yes				
					#DIV/0!		no				
					#DIV/0!		yes				
					#DIV/0!		no				
					#DIV/0!		yes				
					#DIV/0!		no				
					#DIV/0!		yes				
					#DIV/0!		no				
					#DIV/0!		yes				
					#DIV/0!		no				
					#DIV/0!		yes				
					#DIV/0!		no				
					#DIV/0!		yes				
					#DIV/0!		no				
					#DIV/0!		yes				
					#DIV/0!		no				
					#DIV/0!		yes				
					#DIV/0!		no				
					#DIV/0!		yes				
					#DIV/0!		no				
					#DIV/0!		yes				
					#DIV/0!		no				
					#DIV/0!		yes				
					#DIV/0!		no				
					#DIV/0!		yes				
					#DIV/0!		no				
					#DIV/0!		yes				
					#DIV/0!		no				
					#DIV/0!		yes				
					#DIV/0!		no				
					#DIV/0!		yes				
					#DIV/0!		no				

9.6 Measured values

Table 9-1: Raw data of laboratories 106, 118, 154, 177 and 201

Lab	TS	Volume of the sample basket [ml]				Date	Difference between "Go" and "No Go"	Oven temperature [°C]	
		nominal	TS 1	Reference (TS 2)	TS 3			Non-rounded mean value of 1 st and 2 nd sensor	original
106	1	125	134	138.9	143.8	25/08/2011	2.25	141.00	140.74
		216	230	235.8	247.6	30/08/2011	2.00	134.70	134.39
		614	709	707.0	741.1	07/09/2011	1.20	125.55	125.23
		1000	1073	1133.9	1167.7	09/09/2011	2.20	120.55	120.45
	3	125	134	138.9	143.8	27/09/2012	2.10	145.20	142.01
		216	230	235.8	247.6	27/09/2012	2.15	139.60	136.28
		614	709	707.0	741.1	24/09/2012	2.80	127.50	124.63
		1000	1073	1133.9	1167.7	21/09/2012	1.90	123.25	122.38
118	1	125	148.51	142.4	126.3	18/08/2011	1.80	144.70	144.49
		216	242.17	235.4	212.7	25/08/2011	2.50	139.35	139.08
		614	685.52	677.9	630.0	06/09/2011	1.82	129.25	128.94
		1000	1138.27	1143.4	1082.3	08/09/2011	1.83	124.30	124.20
	3	125	148.51	142.4	126.3	10/09/2012	1.87	145.98	142.91
		216	242.17	235.4	212.7	05/11/2012	2.27	139.70	136.08
		614	685.52	677.9	630.0	19/10/2012	0.84	129.34	126.31
		1000	1138.27	1143.4	1082.3	25/10/2012	1.81	122.49	121.55
154	1	125	125	139.3	141.0	22/12/2011	0.50	143.00	141.87
		216	210	231.6	233.0	01/09/2011	3.25	136.85	136.53
		614	650	703.1	710.0	05/09/2011	1.50	128.30	127.99
		1000	1030	1094.0	1150.0	08/09/2011	1.45	122.40	122.30
	3	125	125	139.3	141.0	13/08/2012	2.00	145.00	142.14
		216	210	231.6	233.0	15/08/2012	2.00	139.00	136.00
		614	650	703.1	710.0	17/08/2012	2.50	129.00	126.39
		1000	1030	1094.0	1150.0	21/08/2012	2.20	125.00	124.19
177	1	125	134	136.7	135.0	15/08/2011	2.05	141.10	140.92
		216	234	237.5	243.3	10/08/2011	2.05	135.00	134.85
		614	660	714.3	703.3	04/08/2011	2.05	124.75	124.66
		1000	1106	1123.9	1126.7	28/07/2011	2.30	120.60	120.59
	3	125	134	136.7	135.0	29/08/2012	1.90	146.50	143.52
		216	234	237.5	243.3	30/08/2012	2.05	140.20	137.09
		614	660	714.3	703.3	04/09/2012	2.05	129.70	126.97
		1000	1106	1123.9	1126.7	06/09/2012	1.80	125.70	124.86
201	1	125	125	131.2	135.0	30/08/2011	2.23	143.83	143.54
		216	220	232.6	248.7	17/08/2011	1.88	137.81	137.60
		614	670	686.9	683.3	19/08/2011	1.99	127.91	127.72
		1000	1200	1130.2	1111.7	26/08/2011	2.07	123.79	123.71
	3	125	125	131.2	135.0	03/08/2012	1.87	145.32	142.53
		216	220	232.6	248.7	06/08/2012	1.99	141.35	138.42
		614	670	686.9	683.3	09/08/2012	1.88	131.19	128.63
		1000	1200	1130.2	1111.7	27/08/2012	2.25	123.97	123.15

Table 9-2: Raw data of the laboratories 228, 229, 233, 238 and 251

Lab	TS	Volume of the sample basket [ml]				Date	Difference between "Go" and "No Go"	Oven temperature [°C]	
		nominal	TS 1	Reference (TS 2)	TS 3			Non-rounded mean value of 1 st and 2 nd sensor	original
228	1	125	142	140.0	138.7	13/09/2011	0.65	142.70	142.30
		216	238	231.8	230.9	14/09/2011	0.85	138.60	138.18
		614	714	694.8	681.8	16/09/2011	1.20	128.10	127.72
		1000	1131	1127.6	1093.3	19/09/2011	0.95	123.15	123.03
	n/a	3225	---	---	05/12/2011	0.85	114.00	---	
	3	125	142	140.0	138.7	26/04/2012	0.70	145.55	143.49
		216	238	231.8	230.9	02/05/2012	1.20	139.10	136.91
		614	714	694.8	681.8	08/05/2012	1.05	127.95	126.01
1000		1131	1127.6	1093.3	15/05/2012	1.75	122.75	122.14	
229	1	125	114.463	135.6	146.9	28/11/2011	1.40	145.50	144.54
		216	208.075	232.2	242.5	18/11/2011	1.85	141.95	141.03
		614	643.76	686.4	796.1	11/11/2011	0.85	132.00	131.25
		1000	1051.444	1123.6	1298.2	04/11/2011	3.05	124.45	124.23
	3	125	114.463	135.6	146.9	10/09/2012	3.90	138.70	135.63
		216	208.075	232.2	242.5	19/09/2012	0.80	135.00	131.74
		614	643.76	686.4	796.1	28/09/2012	2.55	123.60	120.71
		1000	1051.444	1123.6	1298.2	10/10/2012	2.70	118.95	118.04
233	1	125	130	133.5	128.0	21/07/2011	2.30	144.00	144.00
		216	226	239.8	227.1	25/07/2011	2.10	138.40	138.37
		614	698	696.6	681.6	29/07/2011	3.00	128.80	128.75
		1000	1063	1077.2	1045.9	03/08/2011	2.30	125.20	125.17
	3	125	130	133.5	128.0	25/07/2012	1.00	142.80	140.08
		216	226	239.8	227.1	30/07/2012	2.00	138.65	135.78
		614	698	696.6	681.6	02/08/2012	2.40	126.35	123.84
		1000	1063	1077.2	1045.9	03/08/2012	1.95	123.80	123.03
238	1	125	151	159.7	151.0	18/08/2011	1.90	142.30	142.09
		216	260	257.0	260.0	07/09/2011	1.60	137.40	137.03
		614	709	670.2	709.0	09/09/2011	2.10	127.20	126.87
		1000	1127	1289.8	1127.0	25/08/2011	1.90	123.80	123.73
	3	125	151	159.7	154.1	28/06/2012	1.80	145.00	142.48
		216	260	257.0	254.7	05/07/2012	1.80	137.50	134.82
		614	709	670.2	730.0	09/07/2012	1.70	129.90	127.55
		1000	1127	1289.8	1292.3	18/07/2012	2.00	124.20	123.46
3375	---	---	4083.7	07/08/2012	1.80	113.00	---		
251	1	125	133	136.9	132.3	03/12/2011	1.38	142.05	141.06
		216	228	233.9	235.0	07/12/2011	2.94	134.91	133.85
		614	660	704.1	697.0	30/11/2011	1.75	126.60	125.72
		1000	1080	1093.4	1090.7	10/11/2011	1.60	122.55	122.32
	3	125	133	136.9	132.3	03/09/2012	2.05	141.55	138.53
		216	228	233.9	235.0	14/09/2012	2.10	136.35	133.12
		614	660	704.1	697.0	06/09/2012	2.00	126.65	123.90
		1000	1080	1093.4	1090.7	11/09/2012	2.35	122.15	121.30

Table 9-3: Raw data of the laboratories 277.1, 277.2, 908, 914 and 011

Lab	TS	Volume of the sample basket [ml]				Date	Difference between "Go" and "No Go"	Oven temperature [°C]	
		nominal	TS 1	Reference (TS 2)	TS 3			Non-rounded mean value of 1 st and 2 nd sensor	original
277.1	1	125	125	139.9	124.1	22/07/2011	2.05	141.30	141.29
		216	216	241.7	218.1	29/07/2011	2.25	136.35	136.29
		614	614	685.7	617.9	08/08/2011	1.35	127.75	127.63
		1000	1000	1136.1	1007.0	10/08/2011	2.75	122.05	122.01
	3	125	125	139.9	124.1	20/07/2012	2.10	144.20	141.51
		216	216	241.7	218.1	23/07/2012	1.70	139.65	136.83
		614	614	685.7	617.9	25/07/2012	2.05	130.20	127.74
		1000	1000	1136.1	1007.0	01/08/2012	1.45	124.90	124.13
277.2	1	125	125	139.9	124.1	25/07/2011	2.65	141.05	141.02
		216	216	241.7	218.1	01/08/2011	2.20	136.55	136.47
		614	614	685.7	617.9	09/08/2011	1.20	127.15	127.02
		1000	1000	1136.1	1007.0	11/08/2011	2.20	122.80	122.76
	3	125	125	139.9	124.1	27/07/2012	0.45	144.55	141.81
		216	216	241.7	218.1	03/08/2012	2.65	140.00	137.10
		614	614	685.7	617.9	02/08/2012	1.70	129.30	126.79
		1000	1000	1136.1	1007.0	06/08/2012	2.10	124.95	124.17
908	1	125	127.5	134.7	130.7	18/10/2011	1.95	143.40	142.75
		216	235	237.4	242.3	13/10/2011	3.35	135.35	134.71
		614	715	702.2	736.7	10/10/2011	2.00	126.10	125.56
		1000	1120	1128.2	1127.3	17/10/2011	-0.40	123.45	123.27
	3	125	127.5	134.7	130.7	20/09/2012	3.75	144.80	141.66
		216	235	237.4	242.3	26/09/2012	1.20	137.95	134.63
		614	715	702.2	736.7	14/09/2012	1.70	128.30	125.50
		1000	1120	1128.2	1127.3	21/09/2012	3.75	122.25	121.38
914	1	125	130	128.2	129.4	06/10/2011	1.70	142.05	141.48
		216	238	239.2	241.6	11/10/2011	1.85	136.30	135.67
		614	686	675.2	686.7	07/10/2011	2.15	126.05	125.53
		1000	1200	1139.5	1175.1	29/09/2011	1.90	122.30	122.16
	3	125	130	128.2	129.4	12/09/2012	1.85	146.70	143.62
		216	238	239.2	241.6	05/09/2012	2.25	138.55	135.39
		614	686	675.2	686.7	03/09/2012	1.95	129.00	126.27
		1000	1200	1139.5	1175.1	29/08/2012	1.70	125.25	124.43
011	1	125	140	135.1	136.5	31/08/2011	1.90	143.05	142.75
		216	242	236.6	238.2	02/09/2011	2.65	136.40	136.07
		216	242	236.6	238.2	02/09/2011	1.15	136.80	136.47
		614	715	689.2	693.2	05/09/2011	2.65	126.50	126.19
	3	1000	1133	1107.8	1120.5	29/08/2011	2.35	123.20	123.12
		125	140	135.1	136.5	17/09/2012	2.15	144.30	141.18
		216	242	236.6	238.2	18/09/2012	2.05	139.80	136.54
		614	715	689.2	693.2	05/09/2012	1.70	129.55	126.81
		1000	1133	1107.8	1120.5	13/09/2012	1.50	123.75	122.90

Table 9-4: Raw Data of the laboratories 028, 034 and 840

Lab	TS	Volume of the sample basket [ml]				Date	Difference between "Go" and "No Go"	Oven temperature [°C]	
		nominal	TS 1	Reference (TS 2)	TS 3			Non-rounded mean value of 1 st and 2 nd sensor	original
028	1	125	127.5	133.3	132.7	21/09/2011	2.10	144.00	143.54
		216	229.5	241.1	241.6	13/09/2011	2.85	137.60	137.19
		614	662.6	683.4	679.0	01/09/2011	1.25	126.25	125.97
		1000	1045.7	1090.8	1080.0	22/08/2011	1.90	124.45	124.38
	3	125	127.5	133.3	132.7	30/07/2012	2.10	145.90	143.14
		216	229.5	241.1	241.6	23/07/2012	1.65	140.25	137.43
		614	662.6	683.4	679.0	16/07/2012	1.90	130.40	128.00
		1000	1045.7	1090.8	1080.0	28/06/2012	2.50	123.95	123.25
034	1	125	143	140.5	143.2	06/01/2011	4.25	143.25	144.69
		216	235	239.8	238.5	22/12/2011	3.00	139.00	137.82
		614	695	702.4	689.2	29/12/2011	2.25	130.00	128.93
		1000	1098	1120.5	1112.4	02/01/2012	2.50	125.00	124.66
	3	125	143	140.5	143.2	19/07/2012	1.20	140.30	137.62
		216	235	239.8	238.5	16/07/2012	2.75	134.40	131.63
		614	695	702.4	689.2	10/07/2012	1.80	125.20	122.84
		1000	1098	1120.5	1112.4	26/07/2012	2.00	121.00	120.25
840	1	15.625	---	---	12.3	09/08/2012	1.30	169.30	---
		125	141.8	127.3	133.5	30/07/2012	0.95	142.75	139.99
		216	247.9	233.2	236.0	25/07/2012	0.65	136.70	133.87
		400	---	---	398.8	03/08/2012	0.70	131.70	---
		614	740.4	703.4	705.5	18/07/2012	0.85	127.35	124.94
		1000	1181	1112.7	1130.3	16/07/2012	0.90	123.10	122.37
	3	15.625	---	---	12.3	31/08/2012	0.80	169.40	---
		125	141.8	127.3	133.5	27/08/2012	0.55	142.85	139.88
		216	247.9	233.2	236.0	23/08/2012	0.45	137.65	134.59
		400	---	---	398.8	29/08/2012	0.55	132.20	---
		614	740.4	703.4	705.5	20/08/2012	0.40	127.60	124.97
		1000	1181	1112.7	1130.3	16/08/2012	0.70	123.20	122.40