



Influence of humidity and temperature on leather

The determination of dimensional stability (linear shrinkage)

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Evaluation on behalf of the VGCT-Commission "Leather Testing and Evaluation"

The effect of change in the dimensional stability of leather can be significant when subjected to changes in temperature and relative humidity. This is especially important when leather is used in automotive interiors where leathers can be subjected to considerable changes in these conditions.

However, the results of the determination of these dimensional changes under specified conditions of temperature and air humidity can vary from one laboratory to another for the same sample of leather. These changes can be considerable.

In order to limit possible errors in the future, the causes attributable to the testing procedure were investigated within the framework of inter-laboratory trials.

It is well known that leather reacts to changes in ambient conditions (*temperature, air humidity*) by alterations of its properties, in particular of its dimensions. This is especially significant when leather is used in automotive interiors – such as leather-covered dashboards – where pronounced climate changes (*day/night*) can provoke effects such as delamination and mechanical deterioration.

Leather suitability for uses where dimensional stability is essential is determined by two different test procedures:

- The alternating climate test
- The heat ageing test

The inter-laboratory trials

In order to analyse the efficiency of these test procedures and to determine their performance characteristics quantitatively, the VGCT-Commission "Leather Testing and Evaluation" initiated a series of inter-laboratory trials.

Ring trials for this test were designed to take place in nine different test laboratories. Seven different leathers were tested: four were chrome-tanned and three chrome-free tanned automotive leathers.

In addition, a cotton fabric of the type "Cotton Duck" was also tested.

THE ALTERNATING CLIMATE TEST

The alternating climate test was subjected to three different inter-laboratory trials:

- Trial A (2004) – Leather samples 1 to 4
- Trial B (2005) – Leather samples I to III
- Trial C (2006) – Cotton fabric

The sample materials were tested according to two different procedures (*DBL 5310 and PA-N 123*), and the association between leather samples and the procedures can be seen in the

section entitled "Findings from the inter-laboratory trials", *Panel 2*.

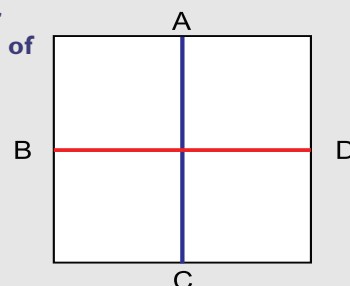
A comprehensive questionnaire for evaluation purposes was also used within these studies to detect any error sources independent from the test material. This included details concerning both technical equipment and handling details of the participants.

A high variation in results was experienced in the first two ring trials (*Trial A and Trial B*), so a reference material was sought. A cotton fabric that appeared appropriate was selected and used. This was dimensionally stable and independent of structure influence, but at the same time showed a distinct (*measurable*) shrinkage. In a new inter-lab trial (*Trial C*) every participant received six cotton test specimens (*quality "Cotton Duck", 200 mm x 200 mm*) which had to be tested as follows:

- 2 samples by alternating climate test PA-N 123
- 2 samples by alternating climate test DBL 5310

The procedures for sample handling were set down in detail. The samples were first conditioned at 23°C and 50% relative humidity (*RH*) for 24 hours. This was followed by plotting a measuring field according to the pattern set down in *Panel 1*:

Panel 1: Pattern for the measuring field of the cotton fabric



(Remeasured lines: **Blue line = E, Red line = F**)



During the test, samples were laid on a glass plate. After being exposed to the climate stress according to DBL 5310 or PA-N 123 respectively, samples were conditioned again and subsequently remeasured. The dimensional variation of the lines AC, BD, E and F was then taken into account in the test evaluation.

THE HEAT AGEING TEST

The dimensional alterations (*linear shrinkage*) in dry heat (*heat ageing test, 120°C, 168 hours*) were examined in detail within two round robin tests. The following materials were investigated:

- Inter-laboratory Trial C (2006)
 - cotton fabric
- Inter-laboratory Trial D (2007)
 - Leather E, chrome-free tanned, brown
 - Leather H, chrome-free tanned, black
 - Leather board
 - Cotton fabric

In these trials, particular attention was paid to the technical conditions, in particular, the volume of the drying oven and the methods used for sample handling in the participant laboratories. However, in inter-laboratory Trial C the cotton fabric failed to meet the degree of reproducibility that was expected.

Accordingly, the following compulsory criteria were set down for cotton fabric in a subsequent investigation as Inter-laboratory Trial D:

- Three specimens had to be tested together in one operation.
- Performance of the test with complete air circulation in the drying oven.
- Measuring lines marked parallel to warp and weft, with master plate of 200mm x 200mm according to the procedure used in Trial C (*AC, F perpendicular and BD, E parallel to the warp*)
- Measurement on the inner edge of the lines.
- Measuring with metal rule and with calliper gauge, if available.
- Storage lying on a glass plate.
- Conditioning prior to testing for at least 48 hours and afterwards for minimum 24 hours (*23°C / RH 50%*).

For leather (*and leather board*) the following additional specifications were agreed upon:

- Sample dimensions should be 250 mm x 250 mm, with a recorded square of 200 mm x 200 mm.
- The size of the drying oven should be stated: the test volume should be 64l if available.
- Two different chrome-free leathers, with two specimens per laboratory, with repeat determinations from one side of the same hide.
- All four specimens to be tested in one operation, with the flesh side to the glass plate.

In this inter-laboratory trial, drying ovens with different volumes were included.

The findings from the inter-laboratory trials

THE ALTERNATING CLIMATE TEST

In part, some of the problems relating to the test procedures were covered by:

- A statistical analysis of the measured values.
- The evaluation of questionnaires on technical conditions and lab-specific performance of the tests.
- Accompanying discussions in the Commission "Leather Testing and Evaluation".

Some critical points that are possibly responsible for the strong scatter of measured values that were experienced were detected using this approach; these issues are set down as appropriate in the following text.

In addition, errors of measurement (*type of measuring device, handling mode*), differences in laboratory equipment (*type of drying ovens, software*) and sample handling (*storage mode, conditioning*) can have a substantial effect on the results.

SAMPLE HANDLING

Conditioning of samples prior to testing was carried out using different time periods, and also to some extent slightly deviating conditions. One of the laboratories conditioned the samples at 20°C and RH 58% instead of at the conventional standard climate conditions of 23°C/RH 50% according to ISO 2419.

The storing of samples in drying ovens also differed in part. The majority of laboratories placed the samples on glass plates. However, examples of samples placed on grids, on non-woven fibre, and hanging were also observed.

After testing the samples were removed immediately from the climate chamber, and then conditioned for at least 24 hours in the standard climate 23°C/RH 50%. Only one of the participants subsequently carried out a programmed conditioning in the climate chamber.

DIMENSIONS OF CLIMATE CHAMBERS

Most of the climate cabinets came from the same provider, but differed in the size of the testing volume. The amount of leather tested in relation to the volume of the chamber (*gram of samples / testing volume*) did not appear to have any influence on the test results.

PROGRAMMING – TEMPERATURE AND HUMIDITY PROGRESSION

Differences in the programming of the testing course (*temperature and relative humidity progression, velocities of heating and cooling*) were observed.

In addition, humidification/dehumidification systems were partly operated at the performance limits. It became apparent that programming of humidity control has to be adjusted individually for every climate chamber.

MEASURING OF SAMPLES

Sample measuring was carried out with different measuring devices. These types included metal rule, aluminium rule, ruler, and calliper gauge, whereas measurement should only be made using a calliper gauge.



All inter-lab trials results related to the alternating climate test procedures are summarised in *Panel 2*:

**Panel 2: The Alternative climate test:
Performance characteristics of the test procedures
DBL 5310 and PA-N 123 for different materials**

Samples	Linear shrinkage (%)	Repeatability conditions				Reproducibility conditions		
	Mean value	s_r	V_r	r		s_R	V_R	R
DBL 5310								
Leather 1, chrome-tanned	4.96	0.38	7.6	1.07	1.05	21.1	2.97	
Leather 2, chrome-tanned	3.95	0.31	7.8	0.87	0.91	23.1	2.58	
Leather III, chrome-free tanned	1.68	0.36	21.3	1.02	1.05	62.5	2.98	
Cotton fabric (A, C, F)	0.77	0.10	12.9	0.28	0.24	30.9	0.68	
(B, D, E)	4.96	0.18	3.6	0.51	0.60	12.0	1.69	
PA-N 123								
Leather I, chrome-tanned	7.74	0.50	6.4	1.41	1.28	16.6	3.63	
Leather II, chrome-tanned	10.46	0.71	6.8	2.01	1.36	13.0	3.85	
Leather 3, chrome-free tanned	2.81	0.35	12.5	0.99	1.23	43.6	3.47	
Leather 4, chrome-free tanned	3.20	0.40	12.5	1.13	1.35	42.2	3.82	
Cotton fabric (A, C, F)	0.86	0.22	25.4	0.61	0.42	49.1	1.19	
(B, D, E)	4.92	0.33	6.6	0.93	1.05	21.4	2.97	

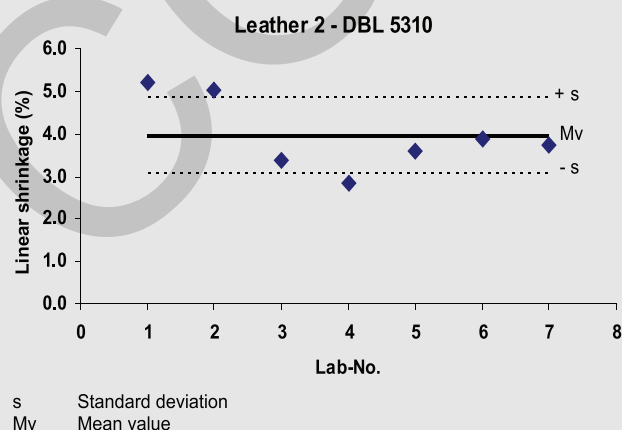
s_r Standard deviation under repeatability conditions in %
 V_r Variation coefficient under repeatability conditions in %
 r Repeatability in %
 s_R Standard deviation under reproducibility conditions in %
 V_R Variation coefficient under reproducibility conditions in %
 R Reproducibility in %

The variation coefficients under repeatability conditions vary for leather from 13% to 62% and for the tested cotton fabric between 12% and 49%. The cotton fabric was intended as a potential reference material, and showed a better reproducibility than leather for a comparable shrinkage (*DBL 5310, leather 1, cotton fabric B, D, E*).

When comparing the two alternating climate test procedures, the method DBL 5310 proved to be more precise. This was because the data of the participants scattered less than experienced using the method PA-N123 (*compare cotton fabric B, D, E for both procedures: also, see Panel 5*).

The variation of laboratory mean values for both test procedures based on Leather 2 and 4 is given in *Panel 3*: (*DBL 5310*) and *Panel 4* (*PA-N 123*) respectively.

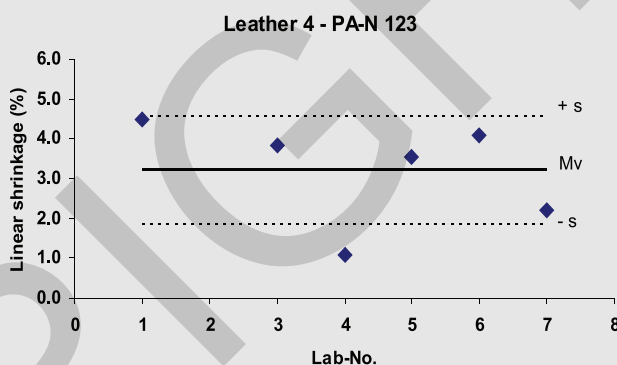
**Panel 3: Alternating climate test:
Variation of laboratory mean values for chrome
tanned Leather 2**



With reference to *Panel 4*, it is noted that the data from laboratory No. 2 were eliminated from the data set on the basis of statistical calculations.

This shows that although preparation of the samples and guidelines for the procedures were precisely followed, the evaluation of the alternating climate test findings showed that the scatter of the results under reproducibility conditions was relatively high.

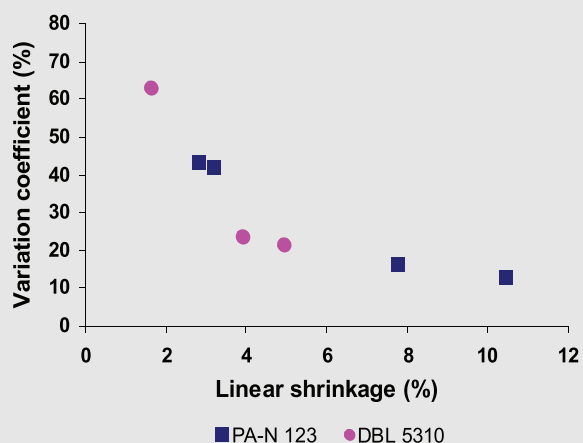
**Panel 4: Alternating climate test:
Variation of laboratory mean values for
chrome-free tanned Leather 4**



The variation coefficient under the test conditions as a function of the magnitude of the values for linear shrinkage in the alternating climate test is given in *Panel 5*.

With reference to the findings set down in *Panel 5*, it must be taken into account that when measured data decrease in value, then the precision of every test procedure will also lessen. In this situation, only values of the same order of magnitude should be compared. For this reason, a well-founded comparison of the different test materials in relation to heat ageing cannot be carried out.

**Panel 5: Linear shrinkage in the alternating
climate test. Variation coefficient under
reproducibility conditions as a function of the
magnitude of measured values**



**THE HEAT AGEING TEST**

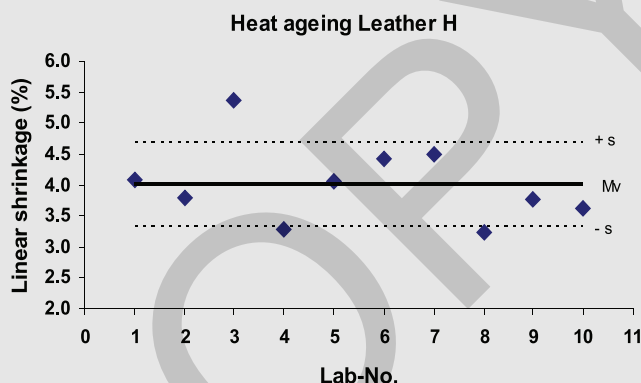
The performance characteristics for the determination of linear shrinkage due to heat ageing as revealed by inter-laboratory trials are given in *Panel 6*:

Panel 6: Performance characteristics of the heat ageing test for different materials

Samples	Direction	Linear shrinkage (%)	Repeatability conditions			Reproducibility conditions		
		Mean value	s _r	V _r	r	s _R	V _R	R
Leather E, FOC, brown	AB, DC, GH (perpendicular to backbone line)	2.86	0.21	7.2	0.58	0.59	20.7	1.66
	AD, BC, EF (parallel to backbone line)	2.79	0.27	9.6	0.75	0.61	21.7	1.70
Leather H, FOC, black	AB, DC, GH (perpendicular to backbone line)	3.99	0.27	6.6	0.74	0.69	17.2	1.93
	AD, BC, EF (parallel to backbone line)	4.04	0.27	6.8	0.77	0.69	17.0	1.92
Leather board	AB, DC, GH	6.28	0.24	3.9	0.68	0.57	9.1	1.61
	AD, BC, EF	8.03	0.29	3.6	0.81	0.69	8.6	1.93
Textile 1, cotton	A, C, F	0.15	0.06	40.9	0.17	0.23	157.8	0.66
	B, D, E	0.89	0.19	21.1	0.53	0.58	65.0	1.64
Textile 2, cotton	A, C, F	0.09	0.08	89.0	0.21	0.18	214.2	0.51
	B, D, E	0.66	0.15	23.4	0.43	0.20	30.9	0.57

The variations in mean values between the laboratories for Leather H in this series are shown in *Panel 7*:

Panel 7: Variation of laboratory mean values for leather H

**DIMENSIONS OF DRYING OVENS**

On closer inspection of the values, and of the corresponding volumes of drying cabinets, a certain trend can be distinguished. In high-volume drying ovens lower shrinkage values were obtained, as shown in *Panel 8*:

THE ALTERNATIVE CLIMATE TEST AND THE HEAT AGEING TEST

The low volume of data from these investigations does not allow a clear statement. However, comparison of the alternating climate test procedure with the heat ageing test shows a tendency that was expected when the experimental detail and the complexity of the test procedures were taken into account. For test leathers, the alternating climate tests

Panel 8: Heat ageing test: Relationship between testing room volume and linear shrinkage

Drying oven volume (l)	Leather E		Leather H		Leather board	
	AB	AD	AB	AD	AB	AD
	Linear shrinkage (%)					
53 (Lab 4)	2.4	2.3	3.3	3.3	5.6	7.2
53 (Lab 7)	3.5	3.3	4.5	4.5	6.7	8.8
64 (Lab 1)	3.1	3.0	4.1	4.1	6.2	8.1
64 (Lab 3)	4.0	4.0	5.3	5.4	7.2	8.8
64 (Lab 6)	3.2	3.1	4.4	4.4	6.7	8.5
115	2.7	2.7	4.0	4.1	6.5	8.5
150	2.5	2.6	3.9	3.7	6.2	7.7
161	2.4	2.3	3.5	3.8	6.2	7.7
190	2.3	2.2	3.2	3.2	5.4	7.1
256	2.6	2.5	3.7	3.9	6.2	7.8

showed higher variations than found with heat ageing (*Leather 3 and Leather E*).

Conclusions

An evaluation of inter-laboratory trials, and the accompanying discussions in VGCT-Commission "Leather Testing and Evaluation" result in the following conclusions:

- Alternating climate tests are complex and problematic procedures whose results are subjected to high variations.
- The determination of linear shrinkage at heat ageing is unproblematic, but potentially dependent on instrument conditions, such as the test room volume.
- As expected, the sample matrix has an influence on the precision of the test procedure.
- In the alternating climate test, the tested cotton fabric showed lower variation of measured values than leathers.
- The scatter of results, when subjected to inter-laboratory evaluation and cause analysis, has shown that - in part - the test instructions for the alternating climate test are not sufficiently detailed, in particular, the programming of humidity control.
- High variation coefficients were achieved. This was in spite of thorough preparation, precise specification of the procedure, and experience from previous ring trials under reproducibility conditions.

For 5% shrinkage values as required in specifications, a variation coefficient of 20 to 30% has to be taken into account.

The findings resulting from these inter-laboratory trials should be taken into close account in the revision of the test instructions and standards for the Alternating Climate and Heat Ageing tests respectively. ☺

Acknowledgment:

The VGCT Commission thanks all participants of the inter-laboratory trials for very dedicated cooperation and stimulating discussions as well as the supply of sample materials.