



Studies on the determination of soiling and cleaning behaviour of leather.

Part 1: Inter-laboratory test on soiling and cleaning behaviour of leather

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(On behalf of the VGCT Commission "Leather Testing and Assessment")

A main task of the VGCT Commission "Leather Testing and Assessment" is the detailed examination for the determination of physical and colour fastness properties of leather, especially in terms of procedures. The intention is to detect performance characteristics of the procedures and to reveal and eliminate possible sources of error.

Due to the trend for light-coloured leather for upholstery in the automotive and furniture sector, the soiling and cleaning behaviour of leather and its determination gain in importance.

From the multiplicity of available test procedures for the characterisation of soiling and cleaning behaviour, the procedure VDA 230-212 was chosen and considered closely by means of an inter-laboratory test programme. In the context each procedural step was examined closely with regard to their impact on the final result of the test.

Part 1 presents the results of the inter-laboratory test on soiling and cleaning behaviour of leather.

Part 2 (to be published in World Leather later in 2016) will deal with the validation of an optimised mechanical cleaning step.

The investigation

Testing institutes and companies within the leather and chemical industry – ten in total – took part in this series of inter-laboratory tests to assess the soiling and cleaning behaviour of leather. The determination of these tests was performed according to VDA 230-212.

Seven automotive leathers in light colour shades with differently structured surfaces were used, namely:

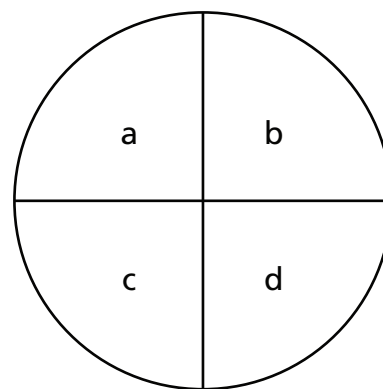
- Sample 1: beige, smooth,
- Sample 2: grey, smooth,
- Sample 3: beige, embossed,
- Sample 4: grey, embossed,
- Sample 5: beige, smooth, with anti-soiling treatment,
- Sample 6: beige, embossed, with anti-soiling treatment,
- Sample 7: grey, embossed, with anti-soiling treatment.

Procedures used in the investigation

Specimens were cut from butt and shoulder areas. Bellies were not included to minimise structural effects of the material on the findings. The specimens were then distributed to the participants of the inter-laboratory test (*identified as laboratory 1, 2-8, and 10-12 in the study*).

The samples were soiled using a standard soiling cloth EMPA 128 (*now SwissaTest 128/1, cotton jeans, indigo/sulphur black, soiled with carbon black/olive oil*) using the Martindale abrasion tester. For the soiling step the soiling cloth was fixed (foam backed) on the pilling specimen holder head and variants of 128 as well as 1,000 abrasion rubs (cycles) were performed. After soiling the specimens were cut into halves as a+b and c+d, as shown in *Panel 1*.

Panel 1: Partition of the soiled specimen



One half (a + b) was stored for 24 hours in the dark under defined climate conditions (23°C, 50% relative humidity), and the second half (c + d) for 24 hours at 80°C in a drying oven using full supply air. Subsequently both halves were stored for one hour in a standard atmosphere in the dark.

In a next step, the two halves were bisected and one part from each half cleaned manually with a 0.5% sodium lauryl ether sulphate solution (*standard cleaning agent Texapon, CAS-no. 9004-82-4*) as described in VDA 230-212. Meanwhile the remaining parts of the bisected specimens were kept covered. The specimen quarters were classified as follows:

- a) soiled, stored in standard atmosphere,
- b) soiled, stored in standard atmosphere, cleaned,
- c) soiled, stored for 24 hours at 80°C,
- d) soiled, stored for 24 hours at 80°C, cleaned.



Assessment of results

The assessment was performed according to VDA 230-212 using the corresponding original leather as reference. In the case of embossed leather only the tips of the profile of the surface are assessed.

Changes in colour and staining of the specimens both visually (*ISO 105-A02 and -A03*) and instrumentally (*ISO 105-A05 and -A04*) were assessed as a deviation from VDA 230-212. The evaluations were performed after the cleaning step on the dried and conditioned specimens (*at least 16 hours in the dark under standard climate conditions*).

Furthermore, laboratory 5 visually and instrumentally assessed (*two different apertures – 5mm and 15mm were used*) the following samples from all of the participating laboratories:

Sample 5: soiled with 128 abrasion rubs,

Sample 7: soiled with 1,000 abrasion rubs.

The data generated in all of the inter-laboratory tests together with observations were fully verified, and statements can be made on the influence of:

- Item 1] The number of cycles in the soiling stage.
- Item 2] The surface structure of leather.
- Item 3] The storage conditions of soiled specimens on the cleaning result.
- Item 4] The type of assessment applied.
- Item 5] The differences between laboratories in steps of test procedure.

These are set down as follows.

ITEM 1] THE INFLUENCE OF THE NUMBER OF CYCLES IN THE SOILING STAGE

VDA 230-212 includes two test variants, namely using 128 and 1,000 cycles (*abrasion rubs*). In general, the differences in visual assessment between the soiling variants were perceived as low, and the colorimetric measurements resulted in differences of a half grey scale rate. These findings are shown in *Panel 2*.

Based on available data and practical experience with the test procedure, it can be recommended to focus on only one of the test variants.

ITEM 2] THE INFLUENCE OF THE SURFACE STRUCTURE OF THE LEATHER

The influence of surface structure of leather on soiling and cleaning results is well-known from testing practice. Embossing leads to an increased soiling of the tips of the profiles, and the inter-laboratory test results confirm this empirical knowledge. The effect of embossing on soiling with smooth leather (*Sample 2*) and embossed leather (*Sample 4*) for test variants with 128 and 1,000 abrasion cycles is given in *Panel 3*.

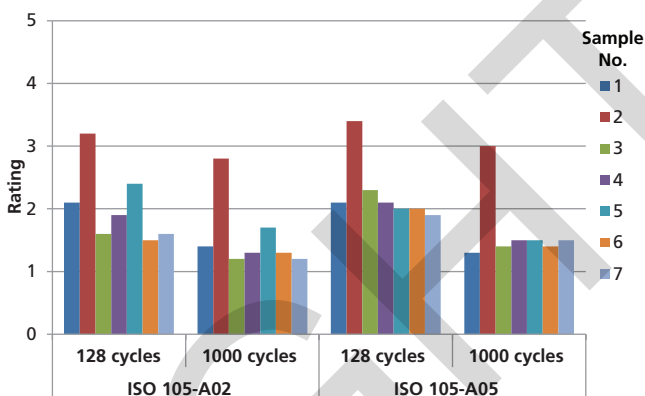
However, the tendency was similar for the assessment of staining.

ITEM 3] THE INFLUENCE OF STORAGE CONDITIONS OF SOILED SPECIMENS ON THE CLEANING RESULT

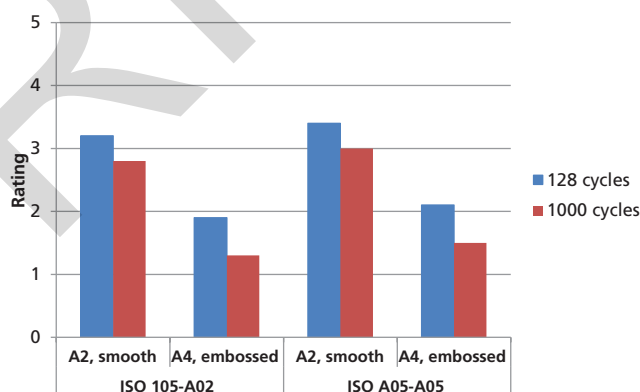
Heat storage after soiling leads in part to an increase of the grey scale rate (*note the assessment of Sample 2*) for several of the leathers. This is shown in *Panel 4* for the assessment according to ISO 105-A02.

This effect could be due to change of the indigo dye from the EMPA 128 soiling cloth. The result of the subsequent cleaning was either not affected by heat storage or impaired.

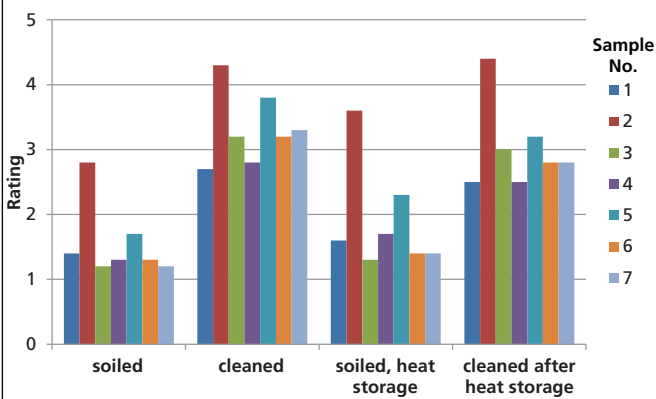
Panel 2: Comparison of mean values of the inter-laboratory test of soiled specimens (EMPA 128) for each leather and testing variant (128 and 1,000 cycles): (Assessment visually (ISO 105-A02) and instrumentally (ISO 105-A05) for change in colour).



Panel 3: Comparison of mean values of the inter-laboratory test of smooth leather (Sample 2) with embossed leather (Sample 4), soiled with 128 and 1,000 cycles respectively. (Assessment as ISO 105-A02 and ISO 105-A05).



Panel 4: Comparison of mean values of the inter-laboratory test for each leather after soiling (1,000 cycles) and cleaning. (Assessment as ISO 105-A02).

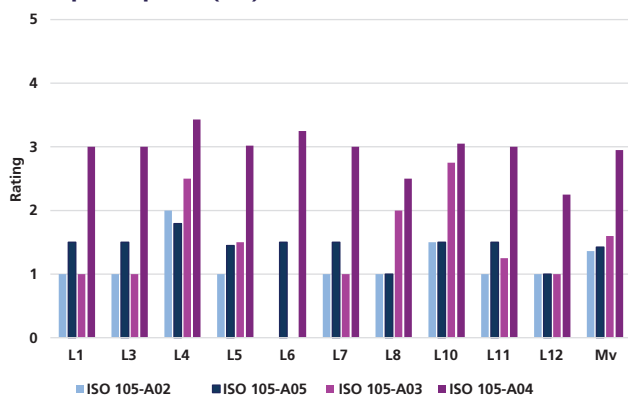


ITEM 4] THE INFLUENCE OF THE TYPE OF ASSEMENT APPLIED

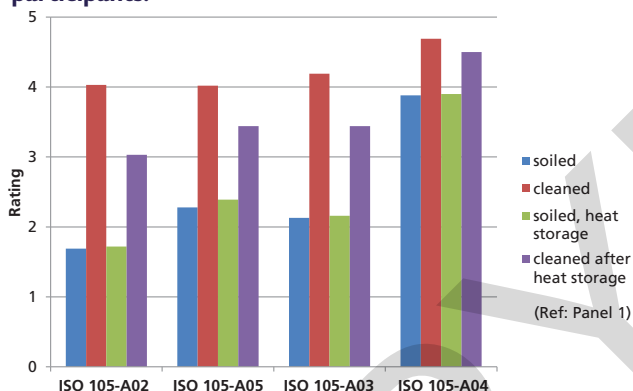
In testing practice, changes of specimen surfaces are considered and assessed both as change in colour (*ISO 26082-1, assessment according to ISO 105-A02 and ISO 105-A05 respectively*) and as



Panel 5: Comparison of all assessment variants using the example of leather Sample 3, soiled with 1,000 cycles. Mean values of the duplicate determinations for each participating laboratory as well as the mean value of all participants (Mv).



Panel 6: Comparison of all assessment variants using the example of leather Sample 5. Mean values of all participants.



staining (VDA 230-212, assessment according to ISO 105-A03 and ISO 105-A04 respectively).

In this inter-laboratory test, all the assessment possibilities were applied concurrently. The findings can be decisive for the application of a procedure and therefore should be taken into consideration in future standardisation activities.

When comparing the types of assessment the colorimetric determination of staining (ISO 105-A04) showed a significant deviation (see Panels 5 and 6). When compared to the other three variables the rates of this assessment type were distinctly higher. Instrumentally determined values for colour change (ISO 105-A05) were 0.5 to 1.0 rates lower than those for staining.

Also, in comparison with the visual assessment variants (ISO 105-A02 and ISO 105-A03) an obvious deviation of colorimetric measurements according to ISO 105-A04 was observed.

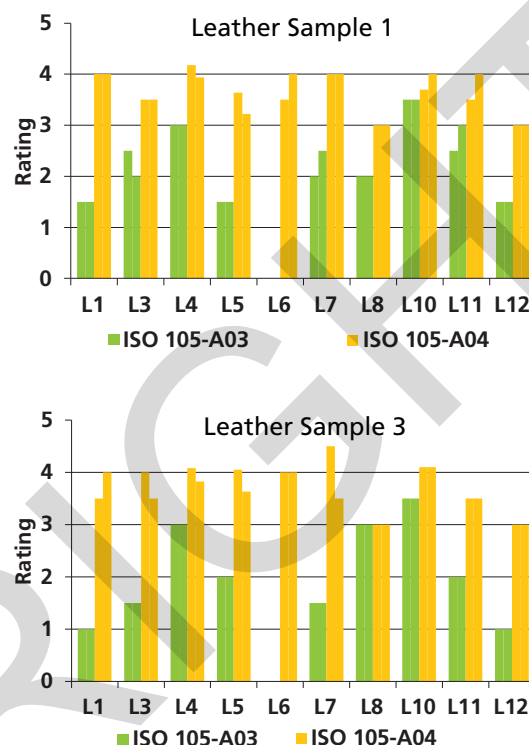
Moreover, the colorimetric measurement of change in colour was more sensitive in the upper range (from rate 3-4 upwards) than that of staining.

Considering these results, the staining assessment specified by VDA 230-212 should be critically reviewed.

ITEM 5] THE INFLUENCE OF THE DIFFERENCES BETWEEN LABORATORIES IN STEPS OF TEST PROCEDURE

The evaluation of the inter-laboratory test results showed that the visual assessment of staining was clearly more difficult than

Panel 7: Illustration of the assessment pattern using as example leather Sample 1 and Sample 3. (Soiled with 128 cycles, assessment of staining, duplicate determinations per laboratory).



that of a change in colour. Here, the variations observed between participating laboratories were altogether higher.

Using the examples of leather Sample 1 and leather Sample 3 soiled with 128 cycles, shows that laboratories always assessed similarly bad or good respectively (assessment with ISO 105-A03). These findings are given as Panel 7.

This shows that large differences in visual assessment from one laboratory to the other can be found (up to 2 rates). Differences in colorimetric measurements are considerably smaller. Overall, the visual assessments presented greater differences between the laboratories in comparison to the colorimetric measurements.

In order to determine which proportion of the differences has to be assigned solely to the assessment step, the specimens of leathers Sample 5 (smooth) and Sample 7 (embossed) of all participants were additionally assessed by laboratory 5.

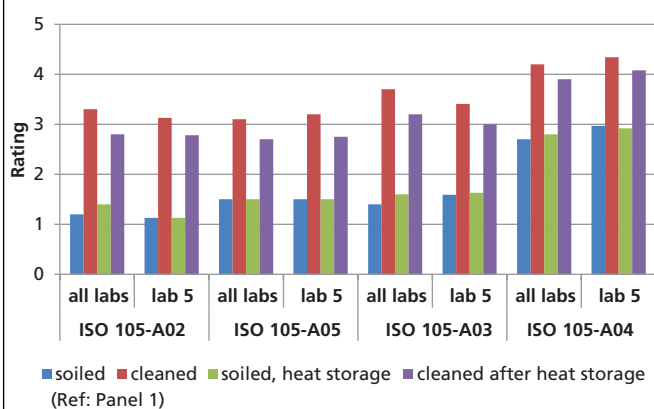
In colorimetric measurements, different aperture sizes (5 and 15mm) were used, but had no influence on the results. Trends already observed with mean values of all the participants (see Item 4) were confirmed by the measurements in this laboratory. These findings are shown in Panel 8.

It is noted that the visual assessment made by laboratory 5 was slightly more severe than the overall mean value of the inter-laboratory test. Through this approach, the effect of the assessment step could be minimised. Hence, evidence on differences in conducting procedural steps in the participating labs were carved out.

Accordingly, the soiling step has been generally mastered and from this point only a small difference ($< \pm 0.5$) between the participants occurred (not illustrated).

However, the intensity of cleaning differed very much in individual laboratories. The manual cleaning stated by VDA 230-212 is difficult to specify and leaves much leeway when to

Panel 8: Comparison of the assessment variants: inter-laboratory test mean values and assessment of laboratory 5 (repeatability conditions) for leather Sample 7, soiled with 1,000 cycles.



perform the cleaning stage. As a consequence the cleaning step was revised and better defined.

Findings from the investigation

Inter-laboratory tests enabled a closer view on the test procedures on soiling and cleaning behaviour of leather and their impact on the final result. Thereby, some of the experience of testing practice was now scientifically supported. These evaluations allow the following conclusions:

- When soiling, one of the cycle number variants could be renounced because the difference between 128 and 1,000 cycles lies nearly constant at a half grey scale rate.
- The effect of embossing observed in practice was also confirmed where a higher soiling is created at the tips of the profiles.
- Heat storage after soiling generally slightly impaired the cleaning result.
- Cleaning intensity differed in the participating laboratories. This caused a careful revision of this procedural step (*this will be detailed in Part 2 of this paper*).
- The colorimetric assessment of staining (ISO 105-A04) deviated systematically and significantly from the other assessment variants. In comparison, the colorimetric assessment of change in colour (ISO 105-A05) lay about 0.5 – 1.0 rates lower.
- The assessment of change in colour in the upper range (starting from rate 3 – 4) proved to be more sensitive than that of staining.
- The visual assessment of staining was subject to greater variations than that of change in colour.
- The comparison of all assessment variants shows the necessity to critically examine procedural steps described in particular testing procedures. These should be revised where required.

Part 2 of this paper will be published in *World Leather* later in 2016. This will refer to the development and validation of the cleaning step that was identified as being problematic in Part 1 of this paper.

Acknowledgements

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