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Legal metrology in tanning industry: statistical analysis and critical issues using pin-wheel machines

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Abstract

The publication of the "Measuring Instruments Directive - MID" (European Directive 2014/32/UE) has determined the inclusion of area measuring machines of leathers in the category of legal metrology devices. Finished leather is sold by area and, up to now, its measurement for verification purposes in commercial trades has been regulated by the International Contract No. 7 (1998) between the ICT and the ICHSLTA. That document provides not only specific indications on commercial issues but defines clear rules for area checking in case of legal disputes too. In general, for ordinary trades the contract allows the use of any agreed machine between the seller and the buyer (mechanical or optoelectronic), but, in the event of a dispute, it refers exclusively to pin-wheel machine according to ISO 11646 standard. This limitation is unique among the metrological devices reported in MID Directive and does not allow the application of modern technologies and the verifications by accredited laboratories according to ISO/IEC 17025. ICT, in fact, indicates only 5 measurement Institutes all over the world equipped with working machines. In this study 13 small batches of different leather articles have been subjected to an interlaboratory trial that has involved 8 participants including 4 European ICT Measurement Institutes. The scope is to verify the critical issues and the performances of measurement related to the use of pin-wheel machine with particular reference to leather characteristics (dimension, deformability, etc).

Introduction

Leather in the finished state is sold in terms of surface that can be assessed using different devices with different feeding systems (i.e. optoelectronic techniques according to ISO 19076 [6]). Until now, unless otherwise agreed or specified, the sales of finished leather are regulated by the indications of International Contract No. 7 [2] between ICHSLTA and ICT that is taken into account even with the entry into force of the MID directive (ref. MI-009) because there are no accredited bodies to perform the periodic verification of measuring instruments.

The paragraph 17 of this Contract indicates how the control of surface shall be made in routine or in case of dispute. Without an agreement, in the event of a dispute original leathers shall be conditioned and measured using the mechanical pin-wheel in accordance with ISO 11646 [3]. The total shall be compared with the invoiced measure and no adjustment shall be made for differences of area equal to or less than 2% for firm leather and 3% for softer, stretcher leather, but in the case of a greater discrepancy such adjustment to be made on the actual discrepancy.

The above requirements do not consider the variability of the measurements using different optoelectronic devices related to pin-wheel machine used for disputes or controls; furthermore, no technical definition of firm or flexible leathers is provided for a unique application of ICT requirements. Finally, there are many operative problems due to the presence of only five laboratories equipped with the devices between the Measurement Institutes [4] agreed in the International Contract No 7.

The scope of this study is to underline the criticalities deriving from measurement controls using pin-wheel machines through a statistical analysis in comparison with the ICT tolerances. The same leather articles involved in the CEN/TC 289 project [5], have been measured in accordance with ISO 11646 [3] in small batches of three leathers by several Measurement Institutes; the whole set of data obtained have been defined the statistical distribution for the leather article. For each sample, the conformity of the data set to the I. C. No 7 in terms of probability have been estimated to identify critical materials for which the control activities could be very uncertain.

Material and Methods

14 small batches of 3 leathers have been measured using calibrated pin-wheel machine. The same leather articles presented in XXXV IULTCS Congress [5] have been analyzed with the exclusion of sample N. 12 and N. 13 (for upholstery and automotive). These sample were too large to be measured by 4 of the laboratories involved into the project.

Each sample have been previously conditioned according toISO 2419:2012 [9]. Eight laboratories attended to the project (see Aknowledgements) using the following devices: N. 4 Turner - Mod. Primesma, N. 3 Tomboni / Tomboni Polvara, N. 1 Gozzini.

The reference test procedure was in accordance with ISO 11646:2014 [3]. For the statistical assessment each leather of the sample has been measured 10 time from each participant and the values provided have been verified using one sided Grubbs, two sided Grubbs and Dixon test for the identification of eventual outliers. After, the Shapiro-Wilks test has been carried out to check the normal distribution of the data with a significance level of 0,05 for the rejection of the null assumption of the normality.

The overall average was considered as the reference surface value of the sample from which the requirements of the ICT International Contract n. 7 were identified (3% and 2% of the reference value).

For verifying the correlation of the results with the mechanical properties of materials, the force-displacement diagram of leathers has been determined in accordance with ISO 13934-1:2013 [7] using a dynamometer class 0,5 according to ISO 7500-1:2018 [8] Acquati Giuseppe AG30 equipped with a 1.000 newton load cell.

ID.	ARTICLE	TYPE	TANNING	SHAPE	MEAN (DMQ)	DEV.ST (DMQ)	C.V. (%)	ICT 3% (%)	ICT 2% (%)	ε_{100N}	R (%)	r (%)
1	Washed	Bovine	Vegetable	Whole	**201**	*9,4*	*4,7*	**47,7**	**33,0**	20	12,1	1,9
2	Wrinkled	Bovine	Chrome	Whole	**210**	*3,6*	*1,7*	**90,4**	**73,3**	8	4,3	1,5
3	Calf	Bovine	Chrome	Side	**513**	*2,7*	*0,5*	**100,0**	**100,0**	3	2,4	0,7
4	Calf	Bovine	Vegetable	Whole	**265**	*2,8*	*1,1*	**99,6**	**92,6**	4	1,3	0,0
5	Goat	Caprine	Chrome	Whole	**161**	*1,8*	*1,1*	**99,5**	**90,4**	11	4,7	1,5
6	Lamb	Ovine	Chrome	Whole	**115**	*2,4*	*2,1*	**78,9**	**59,5**	28	4,4	2,0
7	Suede	Caprine	Chrome	Whole	**127**	*3,9*	*3,1*	**69,5**	**55,8**	18	8,3	1,9
8	Washed	Ovine	Chrome	Whole	**146**	*4,2*	*2,9*	**65,9**	**52,5**	13	12,0	2,5
9	Wrinkled	Ovine	Chrome	Whole	**214**	*4,3*	*2,0*	**83,7**	**64,8**	14	6,5	1,3
10	Calf	Bovine	Chrome	Whole	**174**	*5,6*	*3,2*	**62,8**	**40,8**	30	9,8	2,3
11	Calf	Bovine	Chrome	Side	**422**	*2,9*	*0,7*	**100,0**	**99,4**	10	2,9	0,9
14	Suede (split)	Ovine	Chrome	Whole	**156**	*5,4*	*3,5*	**64,6**	**42,1**	12	10,6	2,0
15	Ovine	Ovine	Chrome	Whole	**182**	*6,3*	*3,5*	**57,3**	**47,5**	18	8,6	1,7
16	Ovine with hairs	Ovine	Chrome	Whole	**98**	*2,2*	*2,2*	**82,7**	**63,7**	25	4,6	2,9

Table 1 - Leather samples, Robust Statistics and percentage compliance to ICT requirements

Results and Discussion

In Table 1 the description of sample with the relative overall average, standard deviation, coefficient of variation are reported. For each sample, Table 1 shows also the compliance theoretical data to the ICT 2% and 3% requirements, the robust statistics [5] and the mechanical properties of samples defined as the percentage strain at 100 newtons in accordance with ISO 13934-1:2013 [7].

For a visual representation, the results were plotted using the theoretical normal cumulative distributions and the experimental data of the surface measurements; in the same diagrams the compliance values to the ICT I.C. No 7 are highlighted.

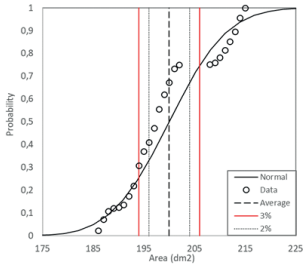


Figure 1 - Sample N. 1 data vs ICT requirements

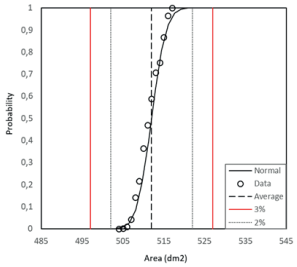


Figure 2 - Sample N. 3 data vs ICT requirements

Conclusion

The results show that the exclusive use of pin-wheel machine as reference could determine criticizes for the definition of the reference surface value of the batch.

The strong operator dependence (low reproducibility) of the measurements for many types of leather articles analysed could determine the not reciprocal compliance also between two ICT Measurement Institutes working with highly qualified personal, perfectly calibrated pin-wheel machines and after the stabilization in standard atmosphere in accordance with ISO 2419. This is due to the characteristics of the device that is devoid of a stopping mechanism of the wheels when a force is applied to leathers during the feeding into the machine, determining a significative overestimation of the results.

Only for leathers with a flat surface and relative flexibility a good fitting of results by different laboratories has been obtained. This effect could be more relevant for leather with surface values around 100 dmq and small samples (below 60 dmq), for which the only resolution error could contribute for a value within 1% and 3%.

Another criticize has been detected for large leather, that are those with a surface greater than 300 dmq (i.e. leather for automotive or upholstery); for these kind of leathers, in fact, only 1 or 2 devices are available all over Europe. For measuring large leathers, some measurement institutes determine the surface by splitting them in two halves, that is an operation not permitted by the standard.

Finally, shall be taken into account problems relative to the maintenance and the management of pin-wheel machines because do not exist any manufacturer of the device with the subsequential issues related to spare parts and maintenance costs.

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References

[1] Directive 2014/32/EU of the European Parliament and of the Council of 26 February 2014 on the harmonisation of the laws of the Member States relating to the making available on the market of measuring instruments (recast). [2] International Contract no. 7 — Finished leather. [3] ISO 11646:2014 [IULTCS/IUP 32] Leather - Measurement of area. [4] Measurement Institutes (ICT/ICHSLTA) for Arbitration Purpose. [5] Mascolo R., Bruno C., De Rosa P., Zorzi G., Pepe F.; Measurement of leather surface: variability in the measurement using electronic and pin wheel devices on different kind of leathers. XXXV IULTCS 2019 Dresden. [6] ISO 19076:2016 [IULTCS/IUP 58] Measurement of leather surface - Using electronic techniques. [7] ISO 13934-1:2013 "Determination of maximum force and elongation at maximum force using the strip method". [8] ISO 7500-1:2018 "Metallic materials - Calibration and verification of static uniaxial testing machines - Part 1: Tension/compression testing machines - Calibration and verification of the force-measuring system". [9] ISO 2419:2012 "Leather –Physical and mechanical tests – Sample preparation and conditioning"