LABORATÓRIO NACIONAL
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INTER-LABORATORY TESTS OF ANODIZED ALUMINIUM TESTING METHODS

Statistical analysis – determination of repeatability and reproducibility

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INTER-LABORATORY TEST OF ANODIZED ALUMINIUM TESTING METHODS

STATISTICAL ANALYSIS - DETERMINATION OF REPEATABILITY AND REPRODUCIBILITY

ENSAIOS INTERLABORATORIAIS DE MÉTODOS DE ENSAIO DE ALUMÍNIO ANODIZADO

ANÁLISE ESTATÍSTICA - DETERMINAÇÃO DA REPETIBILIDADE E DA REPRODUTIBILIDADE

ESSAIS INTER-LABORATOIRES DE METHODES D’ESSAI DE L’ALUMINIUM ANODISÉ

ANALYSE STATISTIQUE – DÉTERMINATION DE LA RÉPÉTABILITÉ ET DE LA REPRODUCTIBILITÉ

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INTER-LABORATORY TEST OF ANODIZED ALUMINIUM TESTING METHODS.

STATISTICAL ANALYSIS - DETERMINATION OF REPEATABILITY AND REPRODUCIBILITY

1. INTRODUCTION

The objective of this Round Robin Test (RRT) on anodized aluminium was to produce precision data (reproducibility and repeatability) for the most used methods to control the quality of anodized aluminium.

The methods selected are: thickness measurement (EN ISO 2360) and sealing quality assessment by mass loss (EN 12373-7), by admittance measurement (EN 12373-5), by dye spot test (EN 12373-4), determination of surface abrasion resistance (BS 6161-18) and also measurement of wear resistance and wear index using an abrasive wheel wear test apparatus (EN 12373-9). Four types of anodized aluminium specimens were prepared to be tested by these methods.

The statistical analysis of the results obtained will be done following the ISO 5725 procedures [1].

The inter-laboratory test program was planned and coordinated by Laboratório Nacional de Engenharia Civil, I.P. (LNEC). The testing specimens were produced by EXTRUSAL - Companhia Portuguesa de Extrusão, S.A., a portuguese company specialised in the extrusion and in the surface treatment of aluminium, whose anodizing process is certified by QUALANOD (EURAS/EWAA) since 1983.

Four types of anodized aluminium specimens with different anodizing and sealing time were produced for this inter-laboratory test. The aim was to obtain testing material with different thickness classes and sealing quality of the anodic coating.

Thirteen laboratories from QUALANOD inspection bodies have participated in the inter-laboratory test.

2. PARTICIPATING LABORATORIES

The fourteen Institutions/laboratories that intended to participate in this inter-laboratory test are listed in Table 1. All of them have received the test specimens and the instructions how to perform the tests.

Table 1 – Participating institution/laboratories for various test methods

Responsible person	**Country**	**Name**	**Laboratory**
MEISSNER Herbert	AUSTRIA	Aluminium Ranshofen Service GmbH	AMAG
VONCLEX Sophye	BELGIUM	CORI - Coatings Research Institute	CoRI
JOSEPH Jean-Paul	FRANCE	TESTAL	TESTAL
HOLZ Marc	GERMANY	IFO GmbH - Institut für Oberflächentechnik	IFO GmbH
VGONTZAS Manolis	GREECE	AAG Quality – EKANAL for Aluminium Ass. Of Greece	EKANAL
BOI Riccardo	ITALY	QUALITAL	QUALITAL
WALRAVE	NETHERLANDS	COT bv – Centrum voor Onderzoek en Technisch Advies bv	COT
TOMASSI Piotr	POLAND	Instytut Mechaniki Precyzyjnej	IMP
MOZARYN Teresa	POLAND	Instytut Techniki Budowlanej	ITB
SALTA Manuela	PORTUGAL	LNEC - Laboratório Nacional de Engenharia Civil	LNEC
GIL-DELGADO Vicente	SPAIN	QUALESPAÑA - Ministerio de Vivienda Subdirección General de Innovación y Calidad de la Edificación	CEDEX
WERNER René	SWITZERLAND	EMPA, Abtl. Korrosion	EMPA
SIPS Hubertus CARTER John	UK & IRELAND	Bodycote Materials Testing	Bodycote
VILLAR René	CUBA	-	-

3. ANODIZED ALUMINIUM

Aluminium sheets with 2 m × 1 m × 1 mm of alloy type 6060 were anodized in the anodizing plant of EXTRUSAL following QUALANOD requirements. This plant has the facility to semi pre-cut the test specimens in the aluminium sheets previously to anodization and sealing, the separation of these specimens was only made after anodizing and sealing treatments. This cutting system was used to avoid the cracking of the anodic coating due to cutting.

Four different process conditions were applied to produce four groups of testing specimens, corresponding to four types of anodic coating and surface finishing, whose characteristics are presented in Table 2. Details of the different process conditions are listed in Annex A.

Table 2 – General characteristics of the testing specimens

ALUMINIUM	**Alloy Type**	**Type of product**	**Dimensions of testing specimens**	
	6060	Sheet with 1 mm of thickness	70mmx140 mm	
ANODIC COATING	**Type**	**Thickness class**	**Sealing**	**Colour**
Polished surface	**A**	15 µm	3 min/µm[a]	natural
	C	25 µm	3 min/µm[b]	bronze
	D	25 µm	1 min/µm[a]	bronze
Satined surface	**B**	20 µm	3 min/µm[a]	natural

[a] done in the anodization plant on 2006 [b] done in laboratory on 2008 (without additive)

Fifty specimens of each coating type were produced. During the QUALANOD inspectors meeting on 2008, three specimens of each coating type were delivered to each one of the laboratories willing to participate in this inter-laboratory test.

To select the 42 needed specimens from the total of 50 produced for each coating type, given to the testing laboratories, it was decided by LNEC to measure the coating thickness in all the specimens. Those with the highest and the lowest thickness values were repeatedly rejected till reach the number of necessary specimens. This procedure allowed to reduce the natural widespread of the anodic coating thickness due to the production process within the test specimens groups.

4. TEST METHODS AND INSTRUCTIONS

The methods selected to be subjected to this evaluation are described in the following standards:

- EN ISO 2360:2003 – Non-conductive coatings on non-magnetic electrically conductive basis materials. Measurement of coating thickness. Amplitude-sensitive eddy current method (ISO 2360:2003).
- EN 12373-7:2002 (2ndEd.) – Aluminium and aluminium alloys. Anodizing. Part 7: Assessment of quality of sealed anodic oxidation coatings by measurement of the loss of mass after immersion in phosphoric acid/chromic acid solution with prior acid treatment.
- EN 12373-5:1998 – Aluminium and aluminium alloys. Anodizing. Part 5: Assessment of quality of sealed anodic oxidation coatings by measurement of admittance.

- EN 12373-4:1998 – Aluminium and aluminium alloys. Anodizing. Part 4: Estimation of loss absorptive power of anodic oxidation coatings after sealing by dye spot test with prior acid treatment.
- BS 6161-18:1991 – Anodic oxidation coatings and its alloys. Part 18. Determination of surface abrasion resistance.
- EN 12373-9:1998 – Aluminium and aluminium alloys. Anodizing. Part 9: Measurement of wear resistance and wear index of anodic oxidation coatings using an abrasive wheel wear test apparatus.

All the laboratories received additional instructions concerning the laboratory code number and location of the measuring points in the specimens to assure uniform distribution of the testing zones among the several laboratories. This was done using a plastic sheet of the same dimensions of the test specimens with the localization of the measurement points (

Figure 1). Additionally, previously formatted Excel worksheets were also sent to all the participating laboratories to uniform the presentation of test results (Annex B).

The thickness measurements on testing specimens of coating types A, C and D are to be done only on the front surface (polished), and on testing specimens of coating type B (satined) in both front and back surfaces. In each surface, the local thickness should be measured on three points, following a scheme like the one indicated in Figure 1. The local thickness value is given by the average of three individual measures at the same point. The coating thickness of each specimen is given by the average of the three local thicknesses.

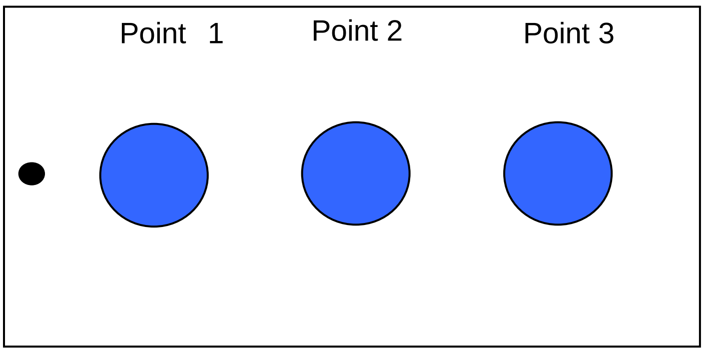


Figure 1 - Scheme of the testing zones for thickness measurement.

The order for carrying the tests was: first the thickness measurements on the 3 holes marked in the plastic sheet, second cutting of the specimen for the sealing test by mass loss and in the remaining part of the specimen, measure the admittance and abrasion resistance, respectively, on points 1 and 2, like in Figure 2, using the same plastic sheet of the thickness measurements. The measurement of wear resistance and wear index was to be carried on B sample back side.

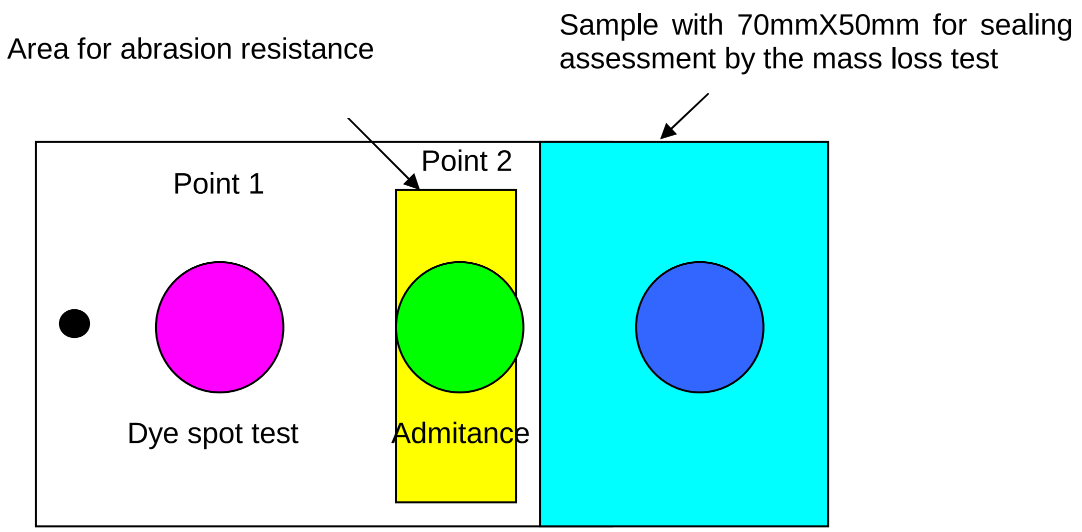


Figure 2 – Scheme of testing zones for sealing quality assessment

Following this instructions, according to the number of test specimens distributed, the number of measurements to be done by testing method for each coating type is presented in the next table.

Table 3 – Number of measurements by testing method for each coating type

Coating type	**Thickness** (EN ISO 2360)	**Mass loss** (EN 12373-7)	**Admittance** (EN 12373-5)	**Dye Spot** (EN 12373-4)	**Abrasion** (BS 6161-18)	**Wear resis.** (EN 12373-9)
A	3×3	3	3	3	3	n. a.
B	3×3 + 3×3	3	3	3	3	3
C	3×3	3	3	n. a.	3	n. a.
D	3×3	3	3	n. a.	3	n. a.
Total	15	12	12	6	12	3

n. a. – not applied

5. TEST RESULTS AND STATISTICAL ANALYSIS

The test specimens were delivered to all the laboratories listed in Table 1 on October of 2008. The participating laboratories have done the tests and reported the results obtained during December of 2008 and February of 2009.

For each anodic coating type under testing (4), according to the scheme of measurements proposed and number of specimens distributed by anodic coating type (3), each laboratory should have reported 15 average thickness results, 12 loss of mass, 12 admittance, 6 dye spot, 12 abrasion resistance and 3 wear resistance and wear index tests results. Some laboratories didn't carried out all the tests requested and one laboratory didn't report any result at all. The next table resumes the number of reported results by method, for all the laboratories. All the reported results (original data) are presented in Annex C.

Table 4 – Number of test results reported by the participating laboratories by testing method

Laboratory	**Thickness** (EN ISO 2360)	**Mass loss** (EN 12373-7)	**Admittance** (EN 12373-5)	**Dye Spot** (EN 12373-4)	**Abrasion** (BS 6161-18)
1	15	12	12	6	12
2	15	12	12	6	12
3	15	12	11	6	12
4	15	12	12	6	12
5	15	12	12	6	12
6	Any results reported				
7	15	12	12	6	12
8	15	12	Any results reported		
9	15	12	12	6	12
10	15	12	12	6	12
11	15	12	12	6	12
12	15	12	12	6	12
13	15	12	12	6	12
14	15	12	12	6	12

The statistical analysis of the reported results for methods EN ISO 2360, EN 12373-7 and EN 12373-5 was carried out according to the criteria of ISO 5725-2:1994 [1]. This part of ISO 5725 is concerned exclusively with measurement methods which yield measurements on a continuous scale and give a single value as the test result, although this single value may be the outcome of a calculation from a set of observations. It is also assumed that the test results have a distribution which is approximately normal.

The dye spot (EN 12373-4) and the abrasion resistance (BS 6161-18) tests give qualitative results that aren't eligible to do this treatment. So, the results of these methods are going to be analyzed using statistical parameters appropriated to qualitative data. The dye spot test results are also going to be analysed in terms of their coherence with the result of the mass loss test (reference method), what can be a cause of rejection of some of these results.

The wear resistance and wear index (EN 12373-9) test results, although of eligible nature for the statistical analysis, were not considered because only one laboratory has performed the test.

5.1. Erroneous data

Obviously erroneous data should be investigated and corrected or discharged. A visual examination of the reported data revealed some test results that seem abnormal, like mass loss values reported by Lab 4 for the coating types A and B, that are around the double of the values reported by all the other participating laboratories (see Annex C).

However, because these mass loss results, although high, are still in agreement with the expecting sealing quality of the coatings under test, it was decided to include these results in the statistical analysis of the respective method.

5.2. Consistency test according to ISO 5725-2

When the test results aren't obviously wrong, the rejection of data can only be possible after doing a statistical analysis of all the results.

In ISO 5725-2:1994[1] two methods are described to do the scrutiny of results for consistency and outliers, that are: Mandel's *k*-statistic (graphical) and Cochran's test (numerical) for within-laboratory consistency, and Mandel's *h*-statistic (graphical) and Grubbs' tests - one and two outlying observations - (numerical) for between-laboratory consistency. According to the standard, the significant level $\alpha = 0,01$ was used for the criteria of outlier and $\alpha = 0,05$ for the criteria of straggler.

According to ISO 5725-2:1994[1] practice, the test results classified as stragglers are retained as correct and only should be discarded those classified as outliers, unless there's a good reason to retain them.

To assure proper rejection of data, the consistency test results from both methods (graphical and numerical) should be consistent. Thus, to reject one set of data, it should be classified as an outlier by both Mandel's *k*-statistic and Cochran's tests or by both Mandel's *h*-statistic and Grubbs' (one outlying observation) tests.

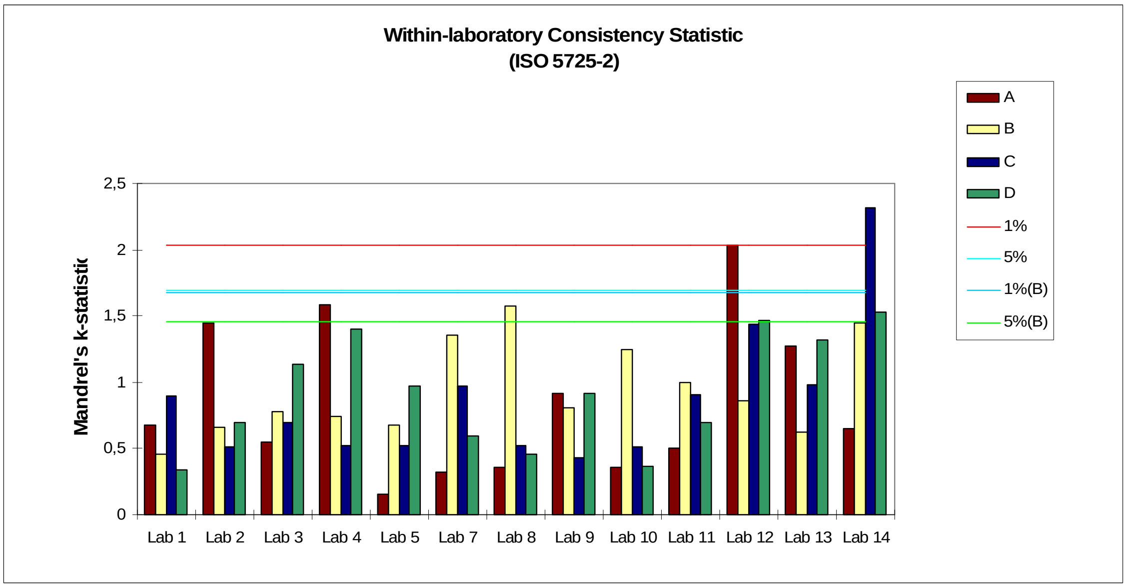
The mathematical expressions of ISO 5725-2:1994[1] that were used for the calculation of these statistics and respective critical limits are listed in Annex F.

5.3. Precision analysis according to ISO 5725-2

5.3.1 Thickness measurement (EN ISO 2360)

The results of the consistency test are shown in Figure 3 and Tables 5, 6 and 7. The precision results are shown in Table 8. In Figure 3, the α values for the rejection criteria of Mandel's k-statistic for the results of coating type B are different from the other coatings, because the number of individual thickness measurements done in these specimens (n=6) was higher than for the other coating types (n=3).

The Mandel's statistics *k* and *h* plots identified four sets of results as stragglers and three set of results as outliers (Lab 12, coating type A and Lab 14, coating type C). The Cochran's test didn't indicate any outlier and identified one set as straggler. The Grubs' test for one outlying observation (single) and the Grubbs' test for two outlying observations (double) didn't indicate any outlier or straggler.



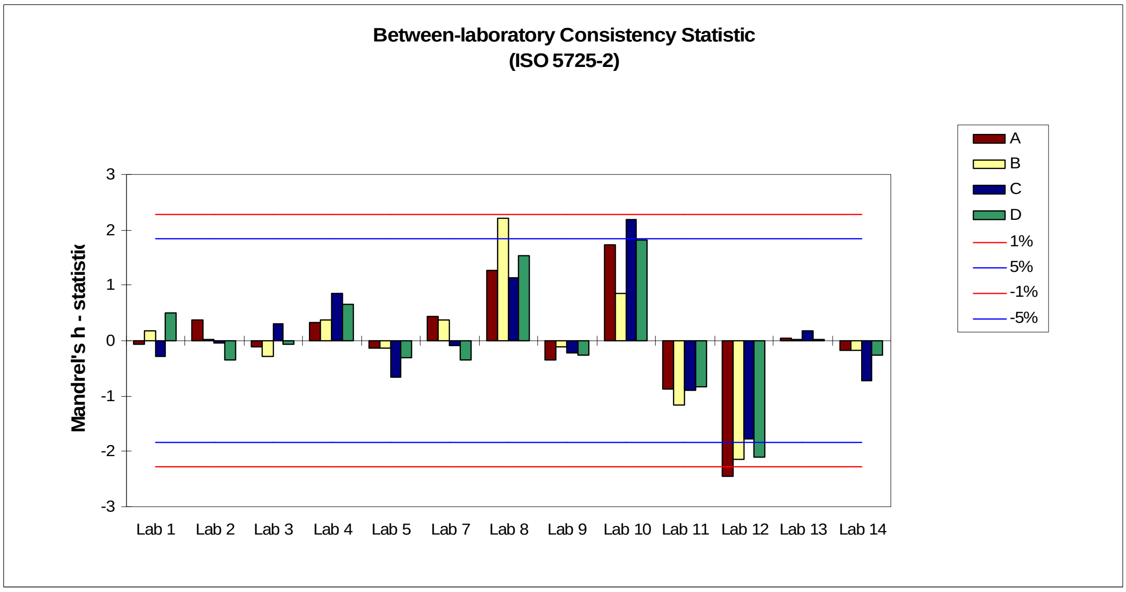


Figure 3 – Mandel's *k*-statistic and *h*-statistic of the results of thickness measurement by EN ISO 2360

Table 5 – Laboratories outside critical value lines of Mandel's statistics for thickness measurement method (EN ISO 2360)

Level	A	B	C	D
Mandel's k-plot	Lab 12	Lab 8	Lab 14	-
Classification	*Outlier*	*Straggler*	*Outlier*	-
Mandel´s h-plot	Lab 12	Lab 8; Lab 12	Lab 10	Lab 12
Classification	*Outlier*	*Straggler*	*Straggler*	*Straggler*

Table 6 – Cochran's test results for thickness measurement (EN ISO 2360)

Level	A	B	C	D
Valid laboratories *p*	13	13	13	13
Number of replicates *n*	3	6	3	3
1% Critical value $C_{Cr\ (1\%)}$	0,450	0,291	0,450	0,450
5% Critical value $C_{Cr\ (5\%)}$	0,371	0,243	0,371	0,371
Cochran's test statistic ***C***	0,319	0,192	0,413	0,179
Classification	*Correct*	*Correct*	*Straggler*	*Correct*
Straggler Lab (***C*** $>C_{Cr\ (1\%)}$)	-	-	Lab 14	-
Outlier Lab (***C*** $>C_{Cr\ (1\%)}$)	-	-	-	-

Table 7 – Grubbs' test results for thickness measurement (EN ISO 2360)

Level	A	B	C	D
Valid laboratories *p*	13	13	13	13
Single G_{Cr} (1%)	2,699	2,699	2,699	2,699
Single G_{Cr} (5%)	2,462	2,462	2,462	2,462
Single high G_p	1,729	2,205	2,199	1,820
Single low G_1	2,448	2,141	1,768	2,110
Classification (low)	*Correct*	*Correct*	*Correct*	*Correct*
Outlier Lab ($G_p > G_{Cr}$ (1%))	-	-	-	-
Classification (low)	*Correct*	*Correct*	*Correct*	*Correct*
Outlier Lab ($G_1 > G_{Cr}$ (1%))	-	-	-	-
Double G_{Cr} (1%)	0,2016	0,2016	0,2016	0,2016
Double G_{Cr} (5%)	0,2836	0,2836	0,2836	0,2836
Double high $G_{largest}$	0,5490	0,4612	0,4037	0,4414
Double low $G_{smalest}$	0,3510	0,4214	0,6198	0,5042
Classification (two largest)	*Correct*	*Correct*	*Correct*	*Correct*
Outlier Lab($G_{largest} < G_{Cr}$ (1%))	-	-	-	-
Classification (two smallest)	*Correct*	*Correct*	*Correct*	*Correct*
Outlier Lab($G_{smalest} < G_{Cr}$ (1%))	-	-	-	-

Single: test for one outlying observation; Double: test for two outlying observations

According to the rejection criteria established in 5.2, none of these outlying results indicated by the consistency tests should be discarded, therefore they have been considered in the precision analysis done, which results are reported in the next table. All the stragglers were also retained.

Table 8 – Results of precision analysis for thickness measurement (EN ISO 2360)

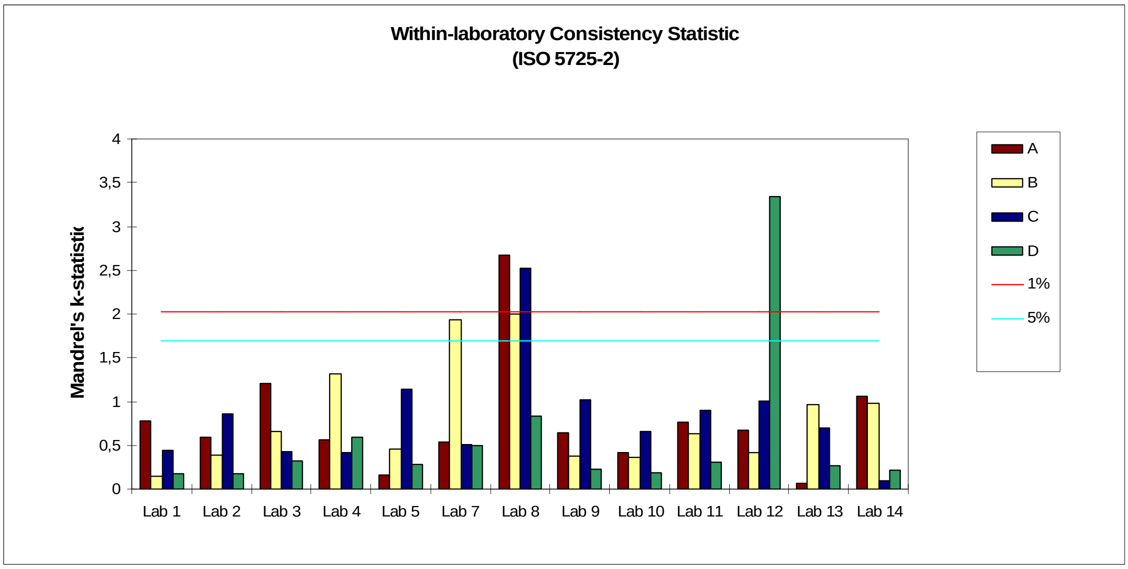
Level	A	B	C	D
Number of replicates *n*	3	6	3	3
Valid laboratories *p*	13	13	13	13
General mean *m* / µm	**18,98**	**21,32**	**29,29**	**28,26**
Repeatability variance s_r^2	0,412	0,361	0,761	0,625
Between-lab variance s_L^2	1,555	2,472	2,542	2,527
Reproducibility variance s_R^2	1,967	2,833	3,303	3,152
Repeatability std. dev. s_r	**0,64**	**0,60**	**0,87**	**0,79**
Reproducibility std. dev. s_R	**1,40**	**1,68**	**1,82**	**1,78**
Repeatability COV (s_r/m)	3,4%	2,8%	3,0%	2,8%
Reproducibility COV (s_R/m)	7,4%	7,9%	6,2%	6,3%
Number of outliers	1	0	1	0
Number of excluded outliers	0	0	0	0
Outlier type	[1]M*h*, [2]M*k*	-	M*k*	-
Outlier laboratories	Lab 12 [1,2]	-	Lab 14	-

Outlier type: M*h* – Mandel's *h* ;M*k* – Mandel's *k* ; C - Cochran's ; G(I) – Grubbs (one outlying observation) ; G(II) – Grubbs (two outlying observations)

5.3.2 Sealing quality assessment by mass loss (EN 12373-7)

Analysing the results sent by the different laboratories for the sealing quality assessment by the mass loss method, it can be noticed a trend in laboratory 4 results: except for the specimens of coating type C, Lab 4 always report the highest results of this testing method.

The results of the consistency test are shown in Figure 4 and Tables 9, 10 and 11. The precision results are shown in Table 12. The Mandel's statistics *k* and *h* plots identified four sets of results as stragglers and five sets of results as outliers (Lab 4, coating types A and B; Lab 8, coating types A and C; Lab 12, coating type D). The Cochran's test also indicates the results reported by Lab 8 and Lab 12, for the same coating types as the Mandel's statistics *k*, as outliers. The Grubbs' tests identified two stragglers and two outliers for the same coatings and laboratories as reported by Mandel's statistics *h*.



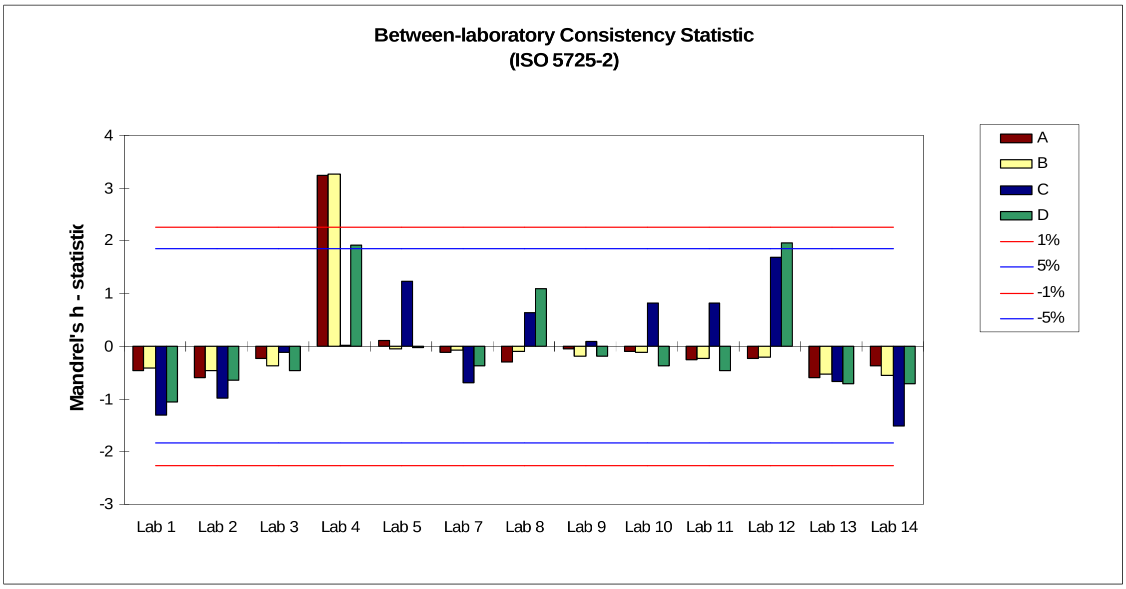


Figure 4 – Mandel's *k*-statistic and *h*-statistic of the results of sealing quality assessment by mass loss (EN ISO 12373-7).

Table 9 – Laboratories outside critical value lines of Mandel's statistics for the sealing quality assessment by mass loss method (EN ISO 12373-7)

Level	A	B	C	D
Mandel's k-plot	Lab 8	Lab 7, Lab 8	Lab 8	Lab 12
Classification	*Outlier*	*Straggler*	*Outlier*	*Outlier*
Mandel´s h-plot	Lab 4	Lab 4	-	Lab 4, Lab 12
Classification	*Outlier*	*Outlier*	-	*Straggler*

Table 10 – Cochran's test results for sealing quality assessment by mass loss (EN ISO 12373-7)

Level	A	B	C	D
Valid laboratories *p*	13	13	13	13
Number of replicates *n*	3	3	3	3
1% Critical value $C_{Cr\ (1\%)}$	0,450	0,450	0,450	0,450
5% Critical value $C_{Cr\ (5\%)}$	0,371	0,371	0,371	0,371
Cochran's test statistic ***C***	0,551	0,307	0,491	0,859
Classification	*Outlier*	*Correct*	*Outlier*	*Outlier*
Outlier Lab (***C*** >$C_{Cr\ (1\%)}$)	Lab 8	-	Lab 8	Lab 12

Table 11 – Grubbs' test results for sealing quality assessment by mass loss (EN ISO 12373-7)

Level	A	B	C	D
Valid laboratories *p*	13	13	13	13
Single G_{Cr} (1%)	2,699	2,699	2,699	2,699
Single G_{Cr} (5%)	2,462	2,462	2,462	2,462
Single high G_p	3,256	3,278	1,688	1,969
Single low G_1	0,605	0,548	1,504	1,049
Classification (high)	*Outlier*	*Outlier*	*Correct*	*Correct*
Outlier Lab (G_p >G_{Cr} (1%))	Lab 4	Lab 4	-	-
Classification (low)	*Correct*	*Correct*	*Correct*	*Correct*
Outlier Lab (G_1 >G_{Cr} (1%))	-	-	-	-
Double G_{Cr} (1%)	0,2016	0,2016	0,2016	0,2016
Double G_{Cr} (5%)	0,2836	0,2836	0,2836	0,2836
Double high $G_{largest}$	-	-	0,5709	0,2568
Double low $G_{smalest}$	0,9281	0,9430	0,6092	0,8423
Classification (two largest)	-	-	*Correct*	*Straggler*
Outlier Lab($G_{largest}$<G_{Cr} (1%))	-	-	-	Lab 12, Lab 4
Classification (two smallest)	*Correct*	*Correct*	*Correct*	*Correct*
Outlier Lab($G_{smalest}$<G_{Cr} (1%))	-	-	-	-

Single: test for one outlying observation; Double: test for two outlying observations

According to the rejection criteria established in 5.2, five of the outlying results should be discarded: results from Lab 4 for anodic coating types A and B; from Lab 8 for types A and C and from Lab 12 for coating type D. The stragglers were retained. The excluded laboratories are the ones that have presented either relative higher mean or higher dispersion of the mass loss measured values within each coating type. Therefore, they haven't been considered in the precision analysis done, which results are shown in the next table.

Table 12 – Results of precision analysis for sealing quality assessment by mass loss (EN ISO 12373-7)

Level	A	B	C	D
Number of replicates *n*	3	3	3	3
Valid laboratories *p*	11	12	12	12
General mean *m* / g.dm^{-2}	**11,69**	**9,61**	**29,81**	**13,88**
Repeatability variance s_r^2	0,217	0,295	11,361	0,962
Between-lab variance s_L^2	0,622	0,788	25,225	15,415
Reproducibility variance s_R^2	0,839	1,083	36,586	16,377
Repeatability std. dev. s_r	**0,46**	**0,54**	**3,37**	**0,98**
Reproducibility std. dev. s_R	**0,92**	**1,04**	**6,05**	**4,05**
Repeatability COV (s_r/m)	4,0%	5,7%	11,3%	7,1%
Reproducibility COV (s_R/m)	7,8%	10,8%	20,3%	29,2%
Number of outliers	2	1	1	1
Number of excluded outliers	2	1	1	1
Outlier type	[1]M*h*, [2]G(I), [3]M*k*, [4]C	M*h*, G(I)	M*k*, C	M*k*, C
Outlier laboratories	Lab 4 [1,2] Lab 8 [3,4]	Lab 4	Lab 8	Lab 12

Outlier type: M*h* – Mandel's *h* ;M*k* – Mandel's *k* ; C - Cochran's ; G(I) – Grubbs (one outlying observation) ; G(II) – Grubbs (two outlying observations)

5.3.3 Sealing quality assessment by measurement of admittance (EN 12373-5)

The results of the consistency test for this method are shown in Figure 5 and Tables 13, 14 and 15. The precision results are shown in Table 16. One of the previous participating laboratory (Lab 8) hasn't reported any result at all.

The Mandel's statistics *h* and *k* plots identified, respectively, five and three sets of results as outliers involving the following: Lab 4, anodic coating type C; Lab 7, anodic coating types A, B and D; Lab 12, anodic coating type D. These same results from Lab 7 were also classified as outliers by the Grubbs' test. The Cochran's test outlied only results from Lab 7 for anodic coating type A.

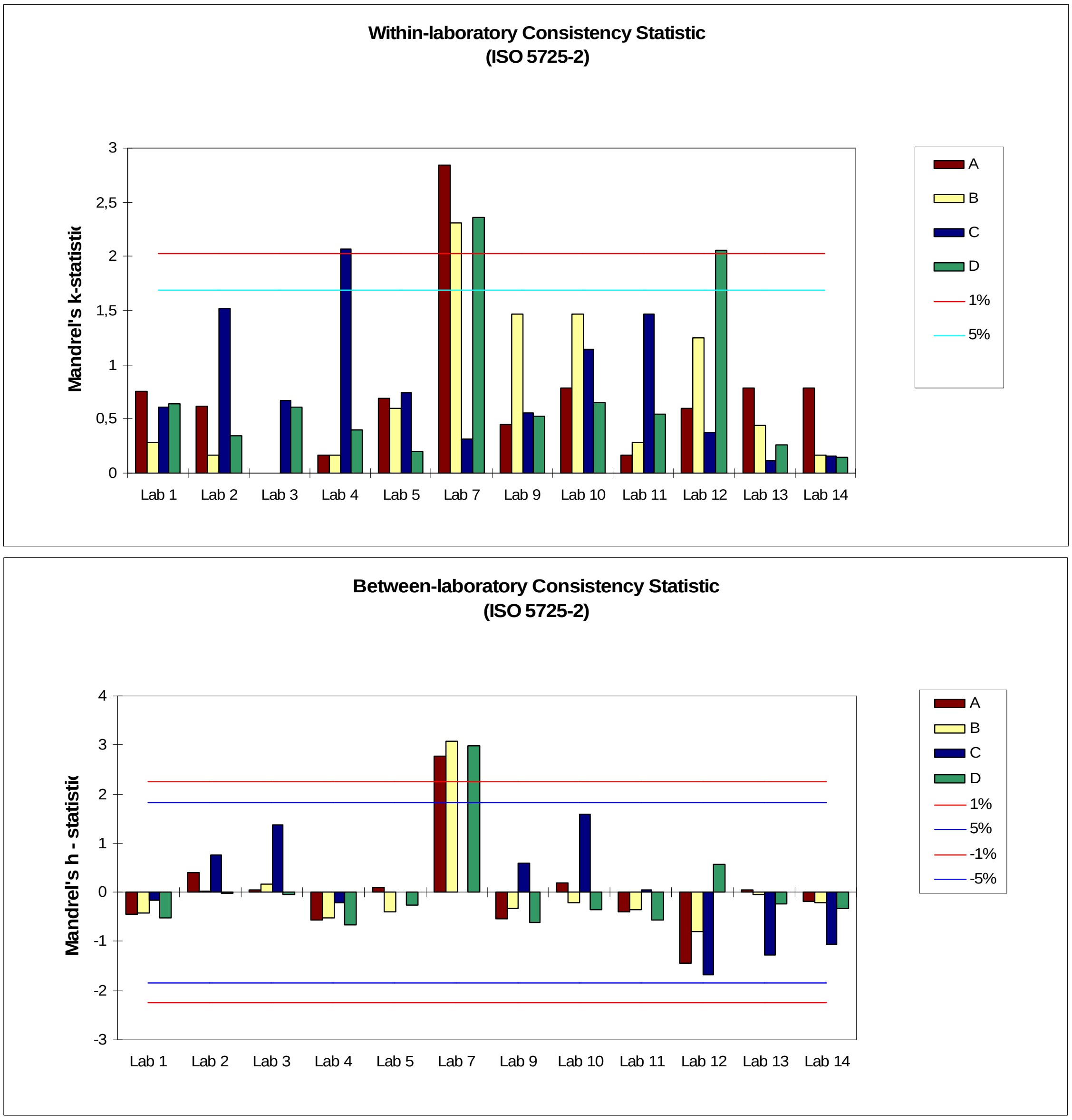


Figure 5 – Mandel's *k*-statistic and *h*-statistic of the results of sealing quality assessment by measurement of admittance (EN ISO 12373-5).

Table 13 – Laboratories outside critical value lines of Mandel's statistics for the sealing quality assessment by measurement of admittance (EN ISO 12373-5)

Level	A	B	C	D
Mandel's k-plot	Lab 7	Lab 7	Lab 4	Lab 7, Lab 12
Classification	*Outlier*	*Outlier*	*Outlier*	*Outlier*
Mandel´s h-plot	Lab 7	Lab 7	-	Lab 7
Classification	*Outlier*	*Outlier*	-	*Outlier*

Table 14 – Cochran's test results for sealing quality assessment by measurement of admittance (EN ISO 12373-5)

Level	A	B	C	D
Valid laboratories *p*	12	12	12	12
Number of replicates *n*	3	3	3	3
1% Critical value $C_{Cr\ (1\%)}$	0,475	0,475	0,475	0,475
5% Critical value $C_{Cr\ (5\%)}$	0,392	0,392	0,392	0,392
Cochran's test statistic ***C***	0,674	0,445	0,355	0,466
Classification	*Outlier*	*Straggler*	*Correct*	*Straggler*
Outlier Lab ($\boldsymbol{C} > C_{Cr\ (1\%)}$)	Lab 7	Lab 7	-	Lab 7

Table 15 – Grubbs' test results for sealing quality assessment by measurement of admittance (EN ISO 12373-5)

Level	A	B	C	D
Valid laboratories *p*	12	12	12	12
Single G_{Cr} (1%)	2,636	2,636	2,636	2,636
Single G_{Cr} (5%)	2,412	2,412	2,412	2,412
Single high G_p	2,778	3,068	1,587	2,993
Single low G_1	1,440	0,804	1,687	0,649
Classification (high)	*Outlier*	*Outlier*	*correct*	*Outlier*
Outlier Lab ($G_p > G_{Cr}$ (1%))	Lab 7	Lab 7	-	Lab 7
Classification (low)	*correct*	*correct*	*correct*	*correct*
Outlier Lab ($G_1 > G_{Cr}$ (1%))	-	-	-	-
Double G_{Cr} (1%)	0,1738	0,1738	0,1738	0,1738
Double G_{Cr} (5%)	0,2537	0,2537	0,2537	0,2537
Double high $G_{largest}$	-	-	0,5166	-
Double low $G_{smalest}$	0,7469	0,9004	0,5147	0,9136
Classification (two largest)	-	-	*correct*	-
Outlier Lab($G_{largest} < G_{Cr}$ (1%))	-	-	-	-
Classification (two smallest)	*correct*	*correct*	*correct*	*correct*
Outlier Lab($G_{smalest} < G_{Cr}$ (1%))	-	-	-	-

Single: test for one outlying observation; Double: test for two outlying observations

According to the rejection criteria established in 5.2, only the results from Lab 7 for anodic coating types A, B and D should be discarded from this precision analysis. These three sets of results are characterized for having both a relative higher mean and higher dispersion of the admittance measured values within each anodic coating type than those reported by the other laboratories. The other outliers and the stragglers were retained. The results of the precision analysis done are presented in the next table.

Table 16 – Results of precision analysis for sealing quality assessment by measurement of admittance (EN ISO 12373-5)

Level	A	B	C	D
Number of replicates *n*	3	3	3	3
Valid laboratories *p*	11	11	12	11
General mean *m* / Y	**5,62**	**5,71**	**36,63**	**9,38**
Repeatability variance s_r^2	0,040	0,074	41,067	0,341
Between-lab variance s_L^2	0,410	0,149	99,675	0,662
Reproducibility variance s_R^2	0,450	0,223	140,742	1,003
Repeatability std. dev. s_r	**0,20**	**0,27**	**6,41**	**0,58**
Reproducibility std. dev. s_R	**0,67**	**0,47**	**11,86**	**1,00**
Repeatability COV (s_r/m)	3,6%	4,8%	17,5%	6,2%
Reproducibility COV (s_R/m)	11,9%	8,3%	32,4%	10,7%
Number of outliers	1	1	1	2
Number of excluded outliers	1	1	0	1
Outlier type	M*h*, G(I), M*k*, C	M*h*, G(I), M*k*,	M*k*	M*h*[1], G(I)[2], M*k*[3]
Outlier laboratories	Lab 7	Lab 7	Lab 4	Lab 7[1,2,3] Lab 12[3]

Outlier type: M*h* – Mandel's *h* ;M*k* – Mandel's *k* ; C - Cochran's ; G(I) – Grubbs (one outlying observation) ; G(II) – Grubbs (two outlying observations)

5.3.4 Dependency analysis

According to ISO 5725-2 it should be investigated whether the precision depends upon mean values and if so, the functional relation ship should be determined. A plot of the repeatability and reproducibility standard deviations *vs* mean values was done in Figure 6. This procedure helps to visualize what kind of functional relationship would better apply to data, it was decided to use a linear relationship to describe the dependency. In some cases, other relationships than the linear could give a little better fitting but the improvement was not significant. The linear regression analysis was done by the least square method and its results were included in the plots shown in Figure 6.

Both methods of sealing quality assessment (mass loss and admittance) show dependency of the precision values on the mean. For the thickness measurement method, this dependency is less accentuated, therefore is probably more correct to assume reproducibility and repeatability as constants independent of mean values, and calculate it by averaging the values obtained for each anodic coating type or majoring it, taking the highest value obtained.

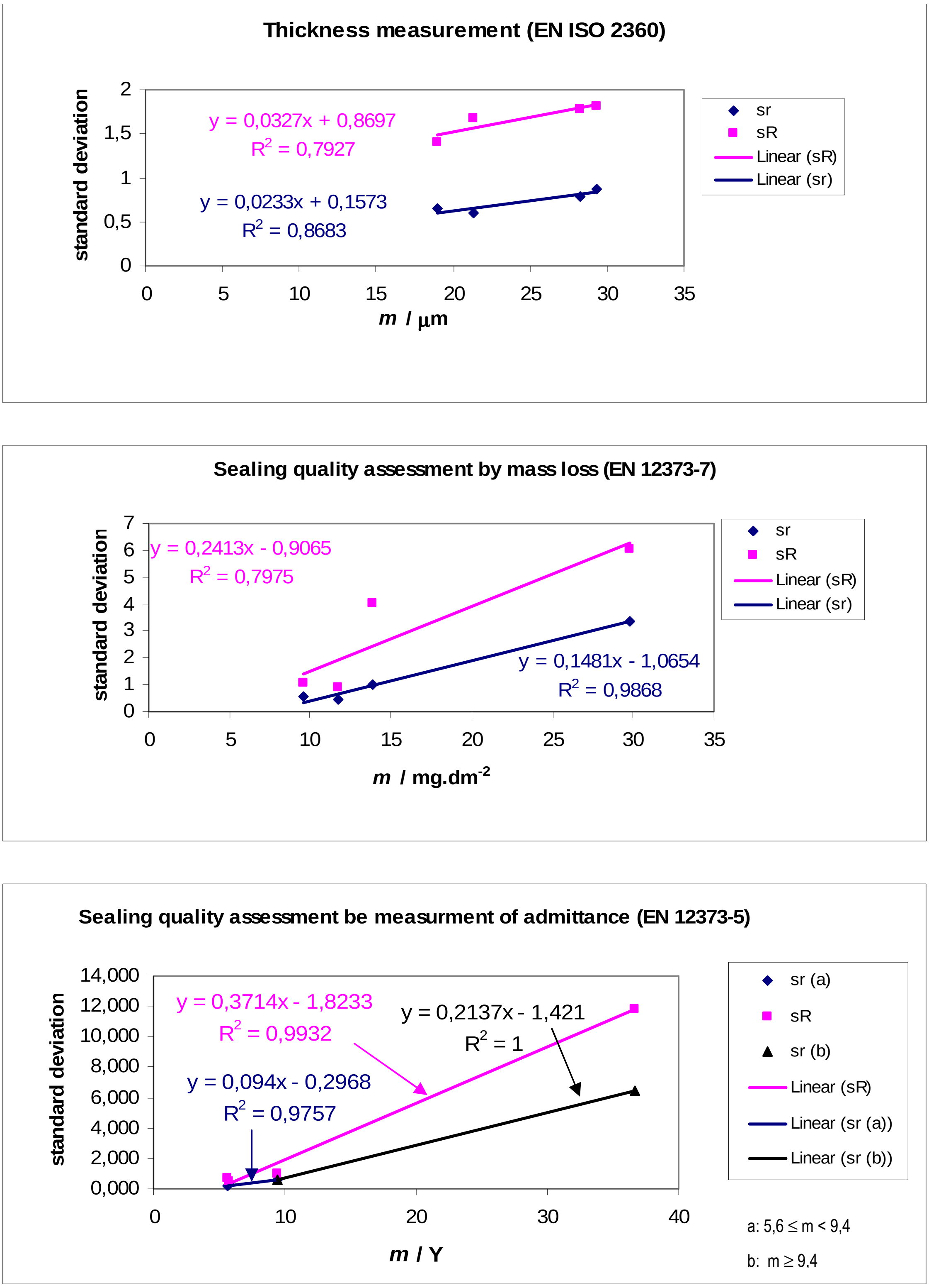


Figure 6 – Dependency of repeatability/reproducibility standard deviations on mean value for the three methods.

5.4 Analysis of precision results

The results of the precision analysis done are resumed in Table 17. The precision data (repeatability and reproducibility) is presented in the individual and global forms.

Table 17 – Results of precision analysis for each method by anodic coating type

EN ISO 2360 – Thickness measurement	Anodic coating type				Excluded data lab:anodic coating type
	A	B	C	D	
General mean m / µm	19,0	21,3	29,3	28,3	None
Repeatability std. dev. s_r	0,64	0,60	0,87	0,79	
Reproducibility std. dev. s_R	1,40	1,68	1,82	1,78	
Global repeatability std. dev.	$S_R = 0,0233\ m + 0,1573$ ($R^2 \approx 0,9$)				
Global reproducibility std. dev.	$S_R = 0,0327\ m + 0,8697$ ($R^2 \approx 0,8$)				
EN 12373-7 - Sealing quality assessment by mass loss	Anodic coating type				Excluded data lab: anodic coating type
	A	B	C	D	
General mean m / g.dm^{-2}	11,7	9,6	29,8	13,9	Lab 4 and 8: A Lab 4: B Lab 8: C Lab 12: D
Repeatability std. dev. s_r	0,46	0,54	3,37	0,98	
Reproducibility std. dev. s_R	0,92	1,04	6,05	4,05	
Global repeatability std. dev.	$S_R = 0,148\ m – 1,0654$ ($R^2 \approx 1$)				
Global reproducibility std. dev.	$S_R = 0,2413\ m – 0,9065$ ($R^2 \approx 0,8$)				
EN 12373-5 - Sealing quality assess. by admittance	Anodic coating type				Excluded data lab:anodic coating type
	A	B	C	D	
General mean m / Y	5,6	5,7	36,6	9,4	Lab 7: A Lab 7: B Lab 7: D
Repeatability std. dev. s_r	0,20	0,27	6,41	0,58	
Reproducibility std. dev. s_R	0,67	0,47	11,86	1,00	
Global repeatability std. dev.	$S_R = 0,094\ m – 0,2968$ ($R^2 \approx 1$)[a] $S_R = 0,2137\ m – 1,421$ ($R^2 = 1$)[b]				
Global reproducibility std. dev.	$S_R = 0,3714\ m – 1,8233$ ($R^2 \approx 1$)				

[a] valid for m values between $5,6 \leq m < 9,4$ [b] valid for m values $\geq 9,4$

The repeatability and reproducibility standard deviations of thickness measurement (EN ISO 2360) method are slightly dependent on mean values, thus they are represented by the linear relationships showed above. However some simplification could be assumed when measured thickness values are below 30 µm: those two statistical parameters can be represented by the respective highest standard deviations obtained, which are 0,87 and 1,82. These repeatability and reproducibility standard deviations values, although higher are not very different from the values obtained for this method in the past analysis [2] .

In the case of the sealing quality assessment methods (by mass loss and by admittance) an higher dependency of the repeatability and reproducibility standard deviations on mean was found, being incorrect the use of averaged values or other simplifications and is more appropriate to use the linear relationships presented. This has to do with the fact that the material was subjected to different sealing conditions (Table 2): anodic coating types A and B were fully sealed in the anodization plant, while anodic coating type D was only partially sealed and C was not sealed. Samples of the anodic coating type D, meanwhile, have achieved a more complete sealing state due to natural ageing, and the sealing of samples of anodic coating type C was carried later on the laboratory and was not complete (Annex C). All this naturally enlarges the range of measurable values and also their dispersion, causing the repeatability and reproducibility standard deviations to be more dependent on the measured value (mean). However, if all the anodic coating types tested had been correctly sealed, repeatability and reproducibility standard deviations probably would be lower in average and practically constants (independent of the mean). This was also noticed in the in the statistical analysis done in previous interlaboratory tests [2].

5.5 Analysis of qualitative results

The dye spot test (EN 12373-4) and abrasion resistance test (BS 6161-18) give qualitative results. These results aren't eligible to do the statistical analysis according to ISO 5725-2. The calculation of mean values and standard deviation parameter doesn't apply and would lead to values without physical meaning. Therefore, these results are going to be treated using statistical parameters adequate to discrete data.

5.5.1 Estimation of loss of absorptive power of anodic oxidation coatings after sealing by dye spot test (EN 12373-4)

The range of test results reported for the dye spot test can be well visualized through its frequency distribution (Figure 7). Using this graphical representation of data it's possible to determine two statistical parameters that characterize the results of this testing method reported by the different laboratories for each anodic coating type: the *mode* and the *median*. The first gives the most frequent colour absorption rating attributed and the second represents the value that divides the group of results at the middle: 50% are inferior or equal to the median value and the other 50% are equal or superior. As a measure of dispersion of the results, the range of values that represent 90% of the reported test results for each anodic coating type It is indicated, and also if any laboratory had reported results 95% out of that range. The results of this statistical analysis are presented in Table 18.

It should be noticed that one laboratory (Lab 08) from the participating group didn't reported any results for this method.

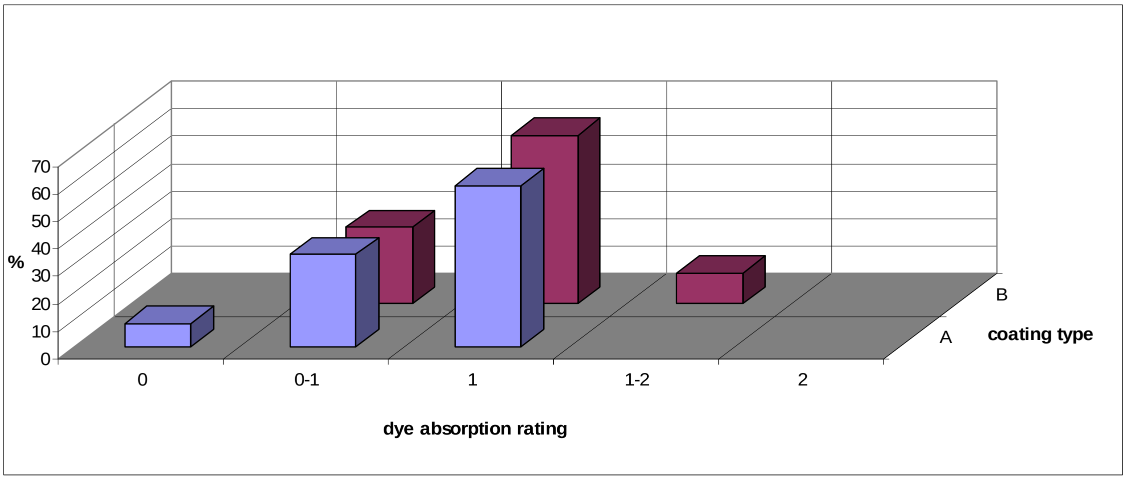


Figure 7 – Frequency distribution of the results of estimation of loss of absorptive power of anodic oxidation coatings after sealing by dye spot test (EN 12373-4), for each anodic coating type.

Table 18 – Statistical analysis of the results of estimation of loss of absorptive power of anodic oxidation coatings after sealing by dye spot test (EN 12373-4)

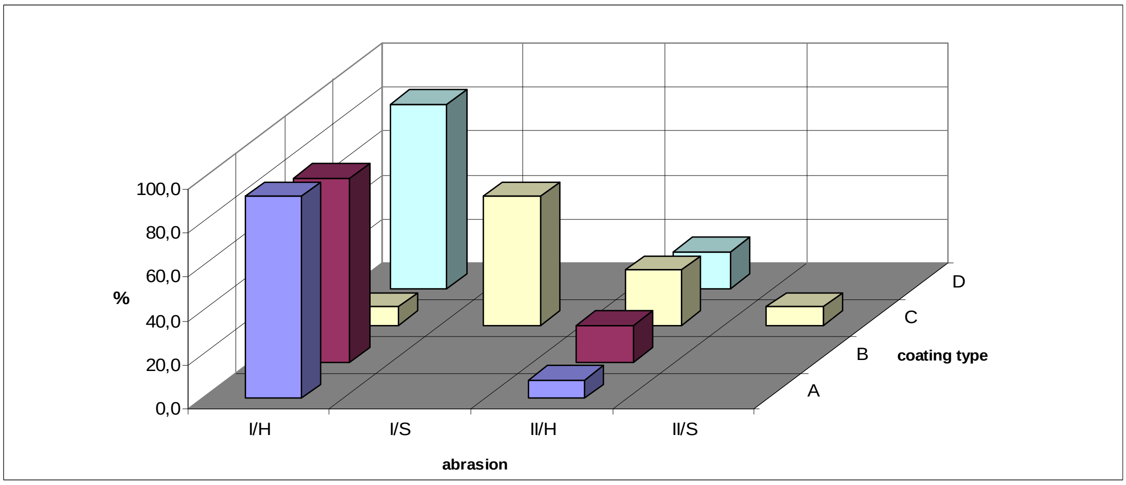
	Dye absorption rating				Laboratory with results outside the range of 95%
Anodic coating type	Mode	Median	Range of 90% of results	Labs outside 90% range	
A	1	1	0-1 to 1	Lab 1	None
B	1	1	0-1 to 1	Lab 5	None

5.5.2 Determination of surface abrasion resistance (BS 6161-18)

This method defines two experimental procedures (method I, method II) to reach the final result, which is expressed by two categories: H – harder than the abrasive and S –

softer than the abrasive. The range of test results reported can be visualized through its frequency distribution (Figure 8). It should be noticed that again one laboratory (Lab 08) from the participating group didn't reported any results for this method.

For the group of results yielded by method there will be no statistical treatment beyond the frequency distribution presented, due to the limited number of classes possible (two). They will only be analysed simply in terms of coherence within each anodic coating type. From this point of view, the results reported for anodic coating types A, B and D are coherent, but in the case of anodic coating type C, around one third of the results reported are in disagreement.



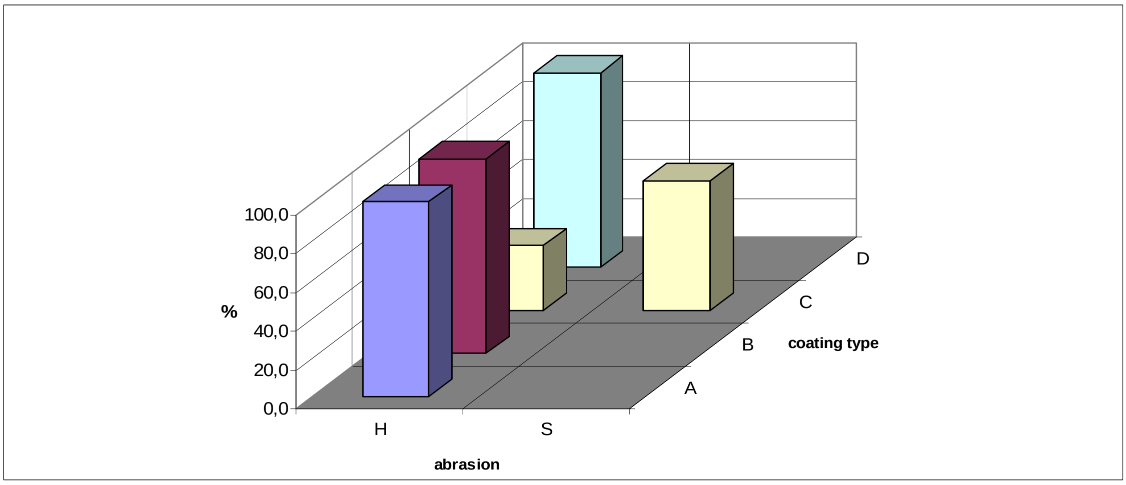


Figure 8 – Frequency distribution of the results of abrasion (BS6161-18), for each anodic coating type.

6. DISCUSSION

The preliminary analysis of the reported data from revealed, only in the case of method EN 12373-7, some possibly abnormal results from the Lab 4, relative to the anodic coating types A, B and D. Specially for the first two anodic coating types, where the results reported are around the double of the values reported by all the other participating laboratories, while in the case of anodic coating type D, two more laboratories (Lab 8 and 12) have also reported relative high values. However, because none of these results reported from Lab 4 are physically wrong, they could not be discarded without further analysis, and so were included in the precision analysis.

After the precision analysis done to the results of the thickness measurement method (EN ISO 2360) none of total of thirteen participating laboratories in this inter-laboratory test was considered as outlier. However for the methods of sealing quality assessment by mass loss (EN 12373-7) and by admittance measurement (EN 12373-5), four laboratories reported results that were definitively considered as outliers and have been excluded from the final precision analysis, three in the case of the mass loss method: Lab 4 (anodic coating types A and B) as it was expected, also Lab 8 (anodic coating types A and C) and Lab 12 (anodic coating type D), and one in the case of the admittance measurement method: Lab 7 (anodic coating types A, B and D).

The statistical outliers found for methods EN 12373-7 and EN 12373-5 resulted from a relative high dispersion of the results within each laboratory in the case of Labs 8 and 12, and in the case of Labs 4 and 7, from a relative high deviations of the results between laboratories. Aspects like heterogeneity of the material tested, change of operator or variations in the experimental procedure could have contributed in part to the dispersion of results.

The precision analysis done to the results reported for the three methods above showed dependency of the precision parameters (repeatability and reproducibility) on the mean values. Therefore, although some simplifications could be done in the case of the thickness measurement method (EN ISO 2360), globally the calculation of both precision parameters for these three methods should be done by the mathematical expressions given in Table 17.

Concerning the dye spot test method (EN 12373-4), the statistical analysis done to the results of this method, of qualitative nature and applicable only to anodic coating types A and B, showed a great coherence among the results reported and revealed two laboratories with results less than 10% frequent: Lab 1 (anodic coating type A) and Lab 5 (anodic coating type B), but none with results less than 5% frequent.

In relation to the method for the determination of surface abrasion resistance, only a simple analysis could be done to its results. It was found that, when the anodic coating is well sealed results are very consistent within the anodic coating type (A, B and D). However, in the case of anodic coating type C, not completed sealed, around one third of the results reported are in opposition. This derives from the material characteristics due to the conditions in which it was sealed (in water without additives), that might have led to the formation of a very thin superficial layer of *boehmite* crystals, more or less extent, with different hardness (softer) from the oxide underneath [3], causing the misleading of the surface abrasion resistance determinations.

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Annex A – Production of test specimens

Table A.1 indicates anodizing and sealing conditions used for the production of test specimens. Fifty specimens of each anodic coating type were produced.

Table A.1 – Anodizing and sealing conditions used for the production of test specimens of each anodic coating type

Anodic coating type	**Anodizing bath**		**Sealing bath**	
A	*Free H_2SO_4* -	184,7 g/l	*Demineralised water*	
	Al content -	12,8 g/l	*pH* -	5,70
	Temperature -	18,0 ºC	*Additive* -	Anodal SH1
	Current density -	1,6 A/dm^2	*Time* -	3 min/µm[a]
B	*Free H_2SO_4* -	184,7 g/l	*Demineralised water*	
	Al content -	12,8 g/l	*pH* -	5,70
	Temperature -	19,0 ºC	*Additive* -	Anodal SH1
	Current density -	1,7 A/dm^2	*Time* -	3 min/µm[a]
C	*Free H_2SO_4* -	182,1 g/l	*Demineralised water*	
	Al content -	11,4 g/l	-	
	Temperature -	19,0 ºC	*Additive*	No additive
	Current density -	1,7 A/dm^2	*Time*	3 min/µm[b]
D	*Free H_2SO_4* -	182,1 g/l	*Demineralised water*	
	Al content -	11,4 g/l	*pH* -	5,75
	Temperature -	19,0 ºC	*Additive* -	Anodal SH1
	Current density -	1,7 A/dm^2	*Time* -	1 min/µm[a]

[a] done in anodization plant on 2006

[b] done in laboratory on 2008

Annex B – EXCEL Worksheets for data registration

Protocol

Test laboratory:	LNEC
Sample type:	A
Tested by:	N Garcia
Date of report:	

Test conditions

Temperature:	21	±	2	℃
Date of the test:	17-07-2008			yyyy-mm-dd
Zero base	0,0	±		µm
Calibration standard 1	23,5	±	0,5	µm
Calibration standard 2		±		µm
Calibration standard 3		±		µm

NOTES H +- 70 mm L +- 50 mm T +- 1 mm

Test results

Thickness results (ISO 2360)

Sample No.	Front side			
A15	1	2	3	
measure 1	21,6			
measure 2	22,0			
measure 3	21,3			
Average thickness	21,6			21,6

Admittance results (EN 12373-5)

Sample No.	Measurement	Thickness	Temperature	Admittance
A15	Ym	(µm)	(℃)	Y
	10,5	24,6	21,2	8,0

Surface abrasion resistance results (BS 6161-18)

Harder or Softer	H	Method I or II	I

Dye spot results (EN 12373-4)

Dye spot	0-1

Weight loss results (EN 12373-7)

Sample No.	Height H	Lenght L	Thickness T	Weight_0	Weight_1	Weigth loss
A15	(mm)	(mm)	(mm)	(g)	(g)	(g/dm^2)
measure 1	71,59	50,29	1,04	9,4563	9,4444	-
measure 2	71,34	50,86	1,05	-	-	-
measure 3	71,83	51,08	1,03	-	-	-
Average	71,59	50,74	1,04	9,4563	9,4444	15,98

Sample No.	Front side			
	1	2	3	
measure 1				
measure 2				
measure 3				
Average thickness				

Sample No.	Measurement	Thickness	Temperature	Admittance
	Ym	(µm)	(℃)	Y

Harder or Softer	H	Method I or II	I

Dye spot	0-1

Sample No.	Height H	Lenght L	Thickness T	Weight_0	Weight_1	Weigth loss
	(mm)	(mm)	(mm)	(g)	(g)	(g/dm^2)
measure 1						-
measure 2				-	-	-
measure 3				-	-	-
Average						

Sample No.	Front side			
	1	2	3	
measure 1				
measure 2				
measure 3				
Average thickness				

Sample No.	Measurement	Thickness	Temperature	Admittance
	Ym	(µm)	(℃)	Y

Harder or Softer	H	Method I or II	I

Dye spot	0-1

Sample No.	Height H	Lenght L	Thickness T	Weight_0	Weight_1	Weigth loss
A15	(mm)	(mm)	(mm)	(g)	(g)	(g/dm^2)
measure 1						-
measure 2				-	-	-
measure 3				-	-	-
Average						

Figure B.1 – Example of EXCEL worksheet for the registration of tests results for specimens of coating type A

Protocol

Test laboratory:	LNEC
Sample type:	B
Tested by:	N Garcia
Date of report:	

Test conditions

Temperature:	21	±	2	℃
Date of the test:	17-07-2008			yyyy-mm-dd
Zero base	0,0	±		µm
Calibration standard 1	23,5	±	0,5	µm
Calibration standard 2		±		µm
Calibration standard 3		±		µm

NOTES H +- 70 mm L +- 50 mm T +- 1 mm

Test results

Thickness results (ISO 2360)

Sample No.	Front side			
B02	1	2	3	
measure 1	25,6			
measure 2	25,4			
measure 3	25,3			
Average thickness	25,4			25,4

Sample No.	Back side			
B02	1	2	3	
measure 1	23,8			
measure 2	23,4			
measure 3	23,5			
Average thickness	23,6			23,6

Admittance results (EN 12373-5)

Sample No.	Measurement	Thickness	Temperature	Admittance
B02	Ym	(µm)	(℃)	Y
	10,5	24,6	21,2	8,0

Surface abrasion resistance results (BS 6161-18)

Harder or Softer	H	Method I or II	I

Dye spot results (EN 12373-4)

Dye spot	0-1

Weight loss results (EN 12373-7)

Sample No.	Height H	Lenght L	Thickness T	Weight_0	Weight_1	Weigth loss
A15	(mm)	(mm)	(mm)	(g)	(g)	(g/dm^2)
measure 1	71,59	50,29	1,04	9,4563	9,4444	-
measure 2	71,34	50,86	1,05	-	-	-
measure 3	71,83	51,08	1,03	-	-	-
Average	71,59	50,74	1,04	9,4563	9,4444	15,98

Sample No.	Front side			
	1	2	3	
measure 1				
measure 2				
measure 3				
Average thickness				

Sample No.	Back side			
	1	2	3	
measure 1				
measure 2				
measure 3				
Average thickness				

Sample No.	Measurement	Thickness	Temperature	Admittance
	Ym	(µm)	(℃)	Y

Harder or Softer	H	Method I or II	I

Dye spot	0-1

Sample No.	Height H	Lenght L	Thickness T	Weight_0	Weight_1	Weigth loss
	(mm)	(mm)	(mm)	(g)	(g)	(g/dm^2)
measure 1						-
measure 2				-	-	-
measure 3				-	-	-
Average						

Sample No.	Front side			
	1	2	3	
measure 1				
measure 2				
measure 3				
Average thickness				

Sample No.	Back side			
	1	2	3	
measure 1				
measure 2				
measure 3				
Average thickness				

Sample No.	Measurement	Thickness	Temperature	Admittance
	Ym	(µm)	(℃)	Y

Harder or Softer	H	Method I or II	I

Dye spot	0-1

Sample No.	Height H	Lenght L	Thickness T	Weight_0	Weight_1	Weigth loss
A15	(mm)	(mm)	(mm)	(g)	(g)	(g/dm^2)
measure 1						-
measure 2				-	-	-
measure 3				-	-	-
Average						

Test results

Wear resistance and wear index test (EN 12373-9) - Thickness measurements (ISO 2360)

Standard	Befor abrasion (d1s)		
	1	2	3
measure 1	25,6		
measure 2	25,4		
measure 3	25,3		
Average thickness	25,4		

Standard	After abrasion (d2s)		
	1	2	3
measure 1	20,2		
measure 2	20,2		
measure 3	20,2		
Average thickness	20,2		

Sample No.	Befor abrasion ($d1_r$)		
B02	1	2	3
measure 1	24,5		
measure 2	24,6		
measure 3	24,5		
Average thickness	24,5		

Sample No.	Befor abrasion ($d2_r$)		
B02	1	2	3
measure 1	19,4		
measure 2	19,4		
measure 3	19,4		
Average thickness	19,4		

Final results summary

d1s-d2s	5,2
$d1_r$-$d2_r$	5,1
WR	78,4
WRC	1,02
WI	0,98
CWR	102

Standard	Befor abrasion (d1s)		
	1	2	3
measure 1			
measure 2			
measure 3			
Average thickness			

Standard	After abrasion (d2s)		
	1	2	3
measure 1			
measure 2			
measure 3			
Average thickness			

Sample No.	Befor abrasion ($d1_r$)		
	1	2	3
measure 1			
measure 2			
measure 3			
Average thickness			

Sample No.	Back side - after abrasion		
	1	2	3
measure 1			
measure 2			
measure 3			
Average thickness			

d1p-d2p	
d1-d2	
WR	
WRC	
WI	
CWR	

Standard	Befor abrasion (d1s)		
	1	2	3
measure 1			
measure 2			
measure 3			
Average thickness			

Standard	After abrasion (d2s)		
	1	2	3
measure 1			
measure 2			
measure 3			
Average thickness			

Sample No.	Befor abrasion ($d1_r$)		
	1	2	3
measure 1			
measure 2			
measure 3			
Average thickness			

Sample No.	Back side - after abrasion		
	1	2	3
measure 1			
measure 2			
measure 3			
Average thickness			

d1p-d2p	
d1-d2	
WR	
WRC	
WI	
CWR	

Figure B.2 – Example of EXCEL worksheet for the registration of tests results for specimens of coating type B

Protocol

Test laboratory:	LNEC
Sample type:	C
Tested by:	N Garcia
Date of report:	

Test conditions

Temperature:	21	±	2	℃
Date of the test:	17-07-2008			yyyy-mm-dd
Zero base	0,0	±		µm
Calibration standard 1	23,5	±	0,5	µm
Calibration standard 2		±		µm
Calibration standard 3		±		µm

NOTES H +- 70 mm L +- 50 mm T +- 1 mm

Test results

Thickness results (ISO 2360)

Sample No.	Front side			
C15	1	2	3	
measure 1	21,6			
measure 2	22,0			
measure 3	21,3			
Average thickness	21,6			21,6

Admittance results (EN 12373-5)

Sample No.	Measurement	Thickness	Temperature	Admittance
C15	Ym	(µm)	(℃)	Y
	10,5	24,6	21,2	8,0

Surface abrasion resistance results (BS 6161-18)

Harder or Softer	H	Method I or II	I

Weight loss results (EN 12373-7)

Sample No.	Height H	Lenght L	Thickness T	Weight_0	Weight_1	Weigth loss
A15	(mm)	(mm)	(mm)	(g)	(g)	(g/dm^2)
measure 1	71,59	50,29	1,04	9,4563	9,4444	-
measure 2	71,34	50,86	1,05	-	-	-
measure 3	71,83	51,08	1,03	-	-	-
Average	71,59	50,74	1,04	9,4563	9,4444	15,98

Sample No.	Front side			
	1	2	3	
measure 1				
measure 2				
measure 3				
Average thickness				

Sample No.	Measurement	Thickness	Temperature	Admittance
	Ym	(µm)	(℃)	Y

Harder or Softer		Method I or II	

Sample No.	Height H	Lenght L	Thickness T	Weight_0	Weight_1	Weigth loss
	(mm)	(mm)	(mm)	(g)	(g)	(g/dm^2)
measure 1						-
measure 2				-	-	-
measure 3				-	-	-
Average						

Sample No.	Front side			
	1	2	3	
measure 1				
measure 2				
measure 3				
Average thickness				

Sample No.	Measurement	Thickness	Temperature	Admittance
	Ym	(µm)	(℃)	Y

Harder or Softer		Method I or II	

Sample No.	Height H	Lenght L	Thickness T	Weight_0	Weight_1	Weigth loss
A15	(mm)	(mm)	(mm)	(g)	(g)	(g/dm^2)
measure 1						-
measure 2				-	-	-
measure 3				-	-	-
Average						

Figure B.3 – Example of EXCEL worksheet for the registration of tests results for specimens of coating type C

Protocol

Test laboratory:	LNEC
Sample type:	D
Tested by:	N Garcia
Date of report:	

Test conditions

Temperature:	21	± 2		℃
Date of the test:	17-07-2008			yyyy-mm-dd
Zero base	0,0	±		µm
Calibration standard 1	23,5	± 0,5		µm
Calibration standard 2		±		µm
Calibration standard 3		±		µm

NOTES H +- 70 mm L +- 50 mm T +- 1 mm

Test results

Thickness results (ISO 2360)

Sample No.	Front side			
D48	1	2	3	
measure 1	21,6			
measure 2	22,0			
measure 3	21,3			
Average thickness	21,6			21,6

Admittance results (EN 12373-5)

Sample No.	Measurement	Thickness	Temperature	Admittance
D48	Ym	(µm)	(℃)	Y
	10,5	24,6	21,2	8,0

Surface abrasion resistance results (BS 6161-18)

Harder or Softer	H	Method I or II	I

Weight loss results (EN 12373-7)

Sample No.	Height H	Lenght L	Thickness T	Weight_0	Weight_1	Weigth loss
A15	(mm)	(mm)	(mm)	(g)	(g)	(g/dm^2)
measure 1	71,59	50,29	1,04	9,4563	9,4444	-
measure 2	71,34	50,86	1,05	-	-	-
measure 3	71,83	51,08	1,03	-	-	-
Average	71,59	50,74	1,04	9,4563	9,4444	15,98

Sample No.	Front side			
	1	2	3	
measure 1				
measure 2				
measure 3				
Average thickness				

Sample No.	Measurement	Thickness	Temperature	Admittance
	Ym	(µm)	(℃)	Y

Harder or Softer		Method I or II	

Sample No.	Height H	Lenght L	Thickness T	Weight_0	Weight_1	Weigth loss
	(mm)	(mm)	(mm)	(g)	(g)	(g/dm^2)
measure 1						-
measure 2				-	-	-
measure 3				-	-	-
Average						

Sample No.	Front side			
	1	2	3	
measure 1				
measure 2				
measure 3				
Average thickness				

Sample No.	Measurement	Thickness	Temperature	Admittance
	Ym	(µm)	(℃)	Y

Harder or Softer		Method I or II	

Sample No.	Height H	Lenght L	Thickness T	Weight_0	Weight_1	Weigth loss
A15	(mm)	(mm)	(mm)	(g)	(g)	(g/dm^2)
measure 1						-
measure 2				-	-	-
measure 3				-	-	-
Average						

Figure B.4 – Example of EXCEL worksheet for the registration of tests results for specimens of coating type D

Annex C – Collation of the original data

Test method – Thickness measurement (ISO 2360)					/µm
Laboratory	*Level* - Sample type				
	A	B-front	B-back	C	D
Lab 01	19,2	21,2	21,9	29,7	29,4
	18,4	21,8	21,5	28,3	28,9
	19,1	21,4	21,8	28,4	29,0
Lab 02	19,9	21,5	21,6	29,7	27,4
	18,4	21,1	20,8	29,2	28,3
	20,1	21,9	21,2	28,8	27,3
Lab 03	18,5	21,5	21,4	30,5	27,1
	18,8	20,7	20,7	29,4	28,7
	19,2	20,4	20,5	29,5	28,6
Lab 04	20,5	22,2	22,4	31,1	28,3
	19,2	21,1	21,9	30,2	30,5
	18,5	21,8	22,0	30,8	29,2
Lab 05	18,9	21,3	21,5	27,8	27,6
	18,8	20,6	21,6	28,7	28,6
	18,7	20,8	20,9	28,1	27,1
Lab 07	19,4	22,4	23,0	30,0	28,2
	19,8	20,6	22,0	29,1	27,5
	19,5	21,5	22,0	28,3	27,3
Lab 08	20,5	25,3	23,3	31,1	30,4
	20,9	24,6	24,8	30,8	30,9
	20,5	26,2	24,8	31,7	31,1
Lab 09	18,3	21,2	22,1	28,5	27,0
	19,2	20,7	21,0	29,2	28,2
	18,1	21,0	21,0	29,1	28,3
Lab 10	21,5	22,6	23,1	33,4	31,1
	21,1	21,6	22,1	33,0	31,6
	21,1	23,2	23,6	32,5	31,1
Lab 11	17,7	20,4	20,0	28,7	26,3
	17,6	19,2	19,0	27,5	27,4
	18,2	18,9	19,3	27,2	26,9
Lab 12	17,3	17,9	18,5	24,9	23,7
	15,2	17,0	18,3	27,2	26,0
	14,9	17,9	17,9	26,9	24,6
Lab 13	19,4	21,6	21,4	30,5	27,1
	18,1	21,0	21,3	29,5	28,9
	19,6	20,9	21,9	28,8	28,9
Lab 14	18,3	21,0	20,4	26,9	26,7
	18,9	22,1	22,1	26,9	27,7
	19,1	20,3	20,3	30,4	29,1

Test method - Sealing quality assessment by mass loss measurement (EN 12373-7)				/mg.dm^{-2}
Laboratory	***Level*** **- Sample type**			
	A	B	C	D
Lab 01	10,45	8,91	20,89	9,85
	11,48	8,88	24,48	10,08
	10,93	8,75	24,28	9,21
Lab 02	10,01	8,63	22,83	11,70
	10,72	8,90	22,47	12,01
	10,64	8,47	29,35	11,16
Lab 03	12,69	8,76	27,37	12,43
	11,57	9,19	29,76	13,27
	11,15	9,49	31,19	11,64
Lab 04	25,03	28,00	29,25	21,97
	24,32	27,58	32,35	24,65
	24,48	29,01	28,87	24,41
Lab 05	12,95	10,66	40,53	13,82
	13,13	10,57	30,70	14,71
	13,13	11,05	38,48	15,21
Lab 07	11,84	9,45	25,10	11,78
	12,26	10,87	25,24	12,75
	12,54	11,57	29,14	14,28
Lab 08	11,04	11,8	28,26	17,43
	10,14	9,71	25,49	20,75
	13,54	10,06	46,60	21,23
Lab 09	12,55	10,04	28,03	13,95
	11,99	9,90	27,62	14,23
	12,83	10,32	35,84	13,11
Lab 10	12,60	10,58	32,05	13,20
	12,05	10,17	33,37	13,15
	12,29	10,42	37,71	12,37
Lab 11	11,64	9,41	29,93	11,81
	11,29	10,10	37,92	13,34
	12,29	9,91	35,20	12,35
Lab 12	11,44	10,15	35,33	17,96
	11,67	10,07	37,45	20,32
	12,30	9,71	44,10	33,50
Lab 13	10,41	7,82	22,98	10,69
	10,45	8,13	28,90	12,05
	10,50	8,87	27,78	11,22
Lab 14	11,97	7,62	21,75	10,66
	11,28	8,17	22,17	11,60
	10,58	8,72	22,63	11,60

Test method - Sealing quality assessment by admittance measurement (EN 12373-5)				/Y_{20}
Laboratory	***Level* - Sample type**			
	A	B	C	D
Lab 01	5,1	5,5	39,4	9,0
	5,6	5,4	32,6	9,1
	5,4	5,6	32,8	8,2
Lab 02	6,7	6,2	33,8	10,2
	6,4	6,1	52,7	9,7
	6,3	6,2	47,5	10,1
Lab 03	6,0	6,4	56,3	10,2
	6,0	6,4	48,8	10,2
	n.r.	6,4	49,0	9,4
Lab 04	5,3	5,4	25,1	8,1
	5,2	5,3	28,7	8,7
	5,2	5,3	49,6	8,5
Lab 05	6,2	5,7	39,5	9,6
	5,8	5,6	31,2	9,3
	6,2	5,3	39,5	9,4
Lab 07	8,6	11,6	34,4	19,3
	9,4	11,0	36,9	17,8
	10,5	10,0	38,4	15,7
Lab 08	n.r.	n.r.	n.r.	n.r.
	n.r.	n.r.	n.r.	n.r.
	n.r.	n.r.	n.r.	n.r.
Lab 09	5,1	5,5	38,9	8,3
	5,4	6,2	44,1	9,0
	5,3	5,2	45,8	8,3
Lab 10	6,5	5,7	49,6	9,7
	6,1	5,4	49,0	8,7
	6,0	6,4	62,0	9,2
Lab 11	5,5	5,6	26,7	8,2
	5,4	5,7	44,7	8,8
	5,4	5,5	40,3	9,0
Lab 12	4,3	5,2	16,0	10,8
	3,9	5,1	20,7	13,3
	4,1	4,4	19,3	10,4
Lab 13	6,2	6,2	23,9	9,3
	5,7	6,1	23,0	9,7
	6,1	5,9	22,4	9,5
Lab 14	5,6	5,8	25,1	9,3
	6,0	5,8	24,6	9,1
	5,5	5,9	26,5	9,3

n.r. – Didn’t report any results

Test method - Estimation of loss of absorptive power of anodic oxidation coatings after sealing by dye spot test (EN 12373-4) /dye absorption rating				
Laboratory	***Level*** **- Sample type**			
	A	B	C	D
Lab 01	0	0-1	n.a.	n.a.
	0	0-1	n.a.	n.a.
	0	0-1	n.a.	n.a.
Lab 02	0-1	1	n.a.	n.a.
	0-1	1	n.a.	n.a.
	0-1	1	n.a.	n.a.
Lab 03	0-1	1	n.a.	n.a.
	0-1	1	n.a.	n.a.
	0-1	1	n.a.	n.a.
Lab 04	1	1	n.a.	n.a.
	1	1	n.a.	n.a.
	1	1	n.a.	n.a.
Lab 05	1	1-2	n.a.	n.a.
	1	1-2	n.a.	n.a.
	1	1-2	n.a.	n.a.
Lab 07	1	1	n.a.	n.a.
	1	1	n.a.	n.a.
	1	1	n.a.	n.a.
Lab 08	n.r.	n.r.	n.a.	n.a.
	n.r.	n.r.	n.a.	n.a.
	n.r.	n.r.	n.a.	n.a.
Lab 09	1	1	n.a.	n.a.
	1	1	n.a.	n.a.
	1	1	n.a.	n.a.
Lab 10	1	0-1	n.a.	n.a.
	1	0-1	n.a.	n.a.
	1	0-1	n.a.	n.a.
Lab 11	1	1	n.a.	n.a.
	1	1	n.a.	n.a.
	1	1-2	n.a.	n.a.
Lab 12	1	1	n.a.	n.a.
	1	1	n.a.	n.a.
	1	0-1	n.a.	n.a.
Lab 13	0-1	1	n.a.	n.a.
	0-1	1	n.a.	n.a.
	0-1	1	n.a.	n.a.
Lab 14	0-1	0-1	n.a.	n.a.
	0-1	0-1	n.a.	n.a.
	0-1	0-1	n.a.	n.a.

n.a. – not applicable
n.r. – Didn't report any results

Test method - Anodic oxidation coatings and its alloys. Part 18. Determination of surface abrasion resistance. (BS 6161-18) /surface abrasion resistance				
Laboratory	***Level* - Sample type**			
	A	B	C	D
Lab 01	I/H I/H I/H	I/H I/H I/H	I/S I/S I/S	I/H I/H I/H
Lab 02	I/H I/H I/H	I/H I/H I/H	I/S I/S I/S	I/H I/H I/H
Lab 03	I/H I/H I/H	I/H I/H I/H	I/S I/S I/S	I/H I/H I/H
Lab 04	I/H I/H I/H	I/H I/H I/H	I/S I/S I/S	I/H I/H I/H
Lab 05	II/H II/H II/H	II/H II/H II/H	II/H II/H II/H	II/H II/H II/H
Lab 07	I/H I/H I/H	II/H II/H II/H	II/H II/H II/H	I/H I/H I/H
Lab 08	n.r. n.r. n.r.	n.r. n.r. n.r.	n.r. n.r. n.r.	n.r. n.r. n.r.
Lab 09	I/H I/H I/H	I/H I/H I/H	II/S II/S II/S	II/H II/H II/H
Lab 10	I/H I/H I/H	I/H I/H I/H	I/S I/S I/S	I/H I/H I/H
Lab 11	I/H I/H I/H	I/H I/H I/H	I/S I/S I/S	I/H I/H I/H
Lab 12	I/H I/H I/H	I/H I/H I/H	I/H I/H I/H	I/H I/H I/H
Lab 13	I/H I/H I/H	I/H I/H I/H	I/S I/S I/S	I/H I/H I/H
Lab 14	I/H I/H I/H	I/H I/H I/H	II/H II/H II/H	I/H I/H I/H

n.r. – Didn't report any results

Legend: I – Method I H – Hard than the abrasive

II – Method II S – Soft than the abrasive

Test method - Aluminium and aluminium alloys. Anodizing. Part 9: Measurement of wear resistance and wear index of anodic oxidation coatings using an abrasive wheel wear test apparatus. (EN 12373-9) /wear resistance and wear index				
Laboratory	***Level*** - **Sample type A**			
	WR	WRC	WI	CWR
Lab XX	59,4	0,68	1,46	68,3
	70,6	0,81	1,23	81,2
	75,0	0,86	1,16	86,3

Legend: WR – Wear resistance WRC – Hard than the abrasive

WI – Wear index CWR – Soft than the abrasive

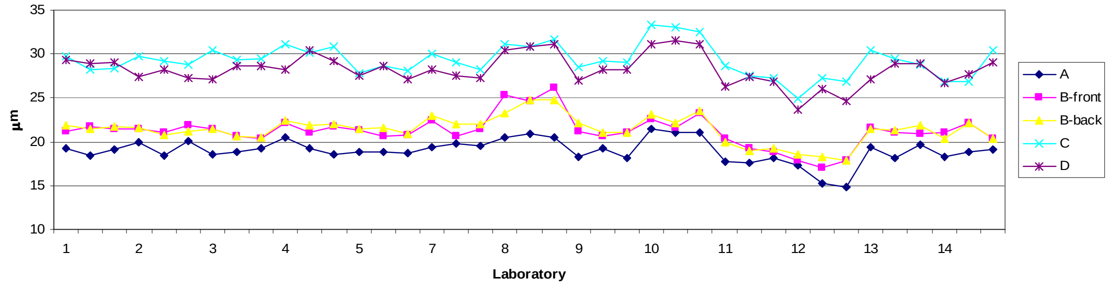

Figure B.1 – Test results of method EN ISO 2360

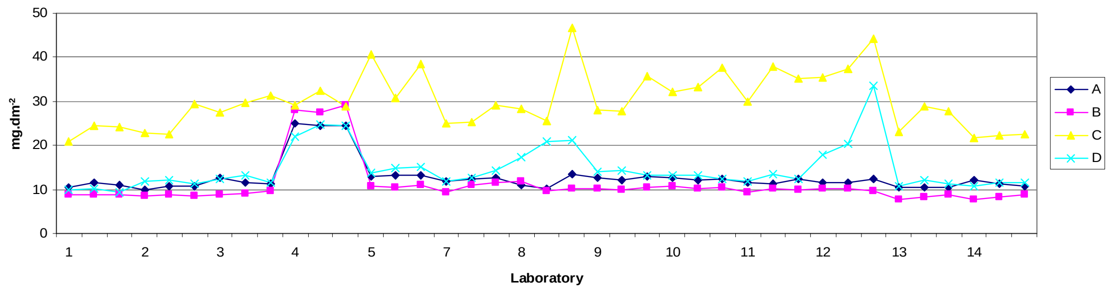

Figure B.2 – Test results of method EN 12373-7

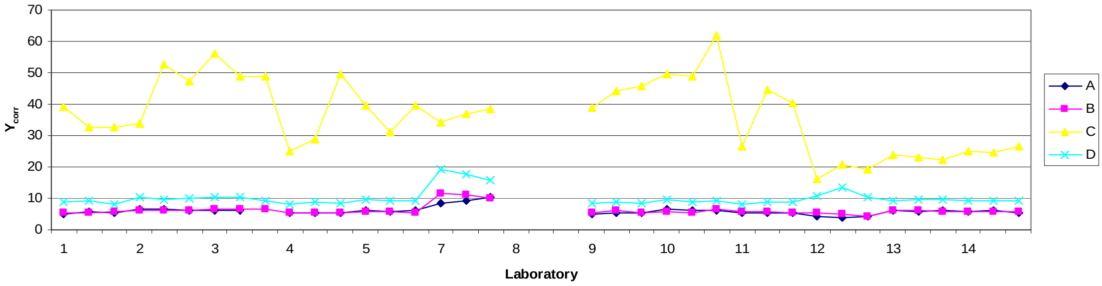

Figure B.3 – Test results of method EN 12373-5

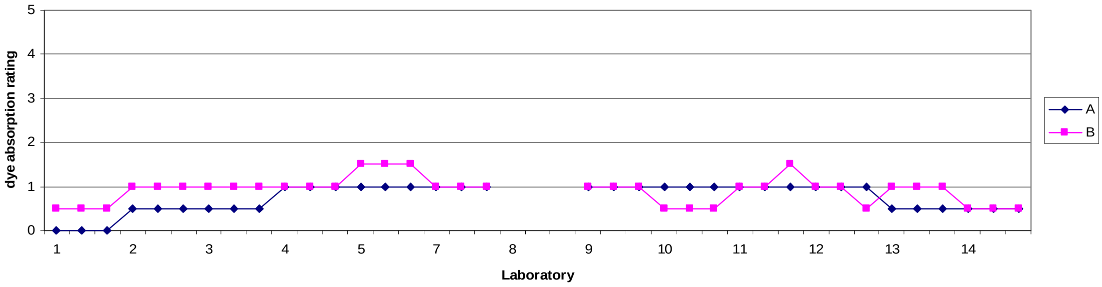

Figure B.4 – Test results of method EN 12373-4

Annex D – Collation of the means

Method EN ISO 2360				/µm
Laboratory	*Level* - **Sample type**			
	A	B	C	D
01	18,9	21,6	28,8	29,1
02	19,5	21,4	29,2	27,7
03	18,8	20,9	29,8	28,1
04	19,4	21,9	30,7	29,3
05	18,8	21,1	28,2	27,8
07	19,6	21,9	29,1	27,7
08	20,6	24,8	31,2	30,8
09	18,5	21,2	28,9	27,8
10	21,2	22,7	33,0	31,3
11	17,8	19,5	27,8	26,9
12	15,8	17,9	26,3	24,8
13	19,0	21,4	29,6	28,3
14	18,8	21,0	28,1	27,8

Method EN ISO 12373-7				/mg.dm^{-2}
Laboratory	*Level* - **Sample type**			
	A	B	C	D
01	10,95	8,85	23,22	9,71
02	10,46	8,67	24,88	11,62
03	11,80	9,15	29,44	12,45
04	24,61	28,20	30,16	23,68
05	13,07	10,76	36,57	14,58
07	12,21	10,63	26,49	12,94
08	11,57	10,52	33,45	19,80
09	12,46	10,09	30,50	13,76
10	12,31	10,39	34,38	12,91
11	11,74	9,81	34,35	12,50
12	11,80	9,98	38,96	23,93
13	10,45	8,27	26,55	11,32
14	11,28	8,17	22,18	11,29

Method EN ISO 12373-5				/Y_{corr}
Laboratory	*Level* - **Sample type**			
	A	B	C	D
01	5,4	5,5	34,9	8,8
02	6,5	6,2	44,7	10,0
03	6,0	6,4	51,4	9,9
04	5,2	5,3	34,5	8,4
05	6,1	5,5	36,7	9,4
07	9,5	10,9	36,6	17,6
08	-	-	-	-
09	5,3	5,6	42,9	8,5
10	6,2	5,8	53,5	9,2
11	5,4	5,6	37,2	8,7
12	4,1	4,9	18,7	11,5
13	6,0	6,1	23,1	9,5
14	5,7	5,8	25,4	9,2

Annex E – Collation of the measures of spread within cells

Method EN ISO 2360				/µm
Laboratory	*Level* - **Sample type**			
	A	B	C	D
01	0,44	0,28	0,78	0,26
02	0,93	0,39	0,45	0,55
03	0,35	0,47	0,61	0,90
04	1,01	0,45	0,46	1,11
05	0,10	0,41	0,46	0,76
07	0,21	0,82	0,85	0,47
08	0,23	0,95	0,46	0,36
09	0,59	0,48	0,38	0,72
10	0,23	0,75	0,45	0,29
11	0,32	0,60	0,79	0,55
12	1,31	0,52	1,25	1,16
13	0,81	0,37	0,85	1,04
14	0,42	0,87	2,02	1,21

Method EN ISO 12373-7				/mg.dm^{-2}
Laboratory	*Level* - **Sample type**			
	A	B	C	D
01	0,52	0,09	2,02	0,45
02	0,39	0,22	3,87	0,43
03	0,80	0,37	1,93	0,82
04	0,37	0,74	1,91	1,48
05	0,10	0,26	5,19	0,70
07	0,35	1,08	2,29	1,26
08	1,76	1,12	11,47	2,07
09	0,43	0,21	4,63	0,58
10	0,28	0,21	2,96	0,47
11	0,51	0,36	4,06	0,78
12	0,45	0,23	4,58	8,37
13	0,05	0,54	3,14	0,69
14	0,70	0,55	0,44	0,54

Method EN ISO 12373-5				/Y_{corr}
Laboratory	*Level* - **Sample type**			
	A	B	C	D
01	0,25	0,10	3,87	0,49
02	0,21	0,06	9,76	0,26
03	0,00	0,00	4,27	0,46
04	0,06	0,06	13,23	0,31
05	0,23	0,21	4,79	0,15
07	0,95	0,81	2,02	1,81
08	-	-	-	-
09	0,15	0,51	3,59	0,40
10	0,26	0,51	7,34	0,50
11	0,06	0,10	9,38	0,42
12	0,20	0,44	2,41	1,57
13	0,26	0,15	0,75	0,20
14	0,26	0,06	0,98	0,12

Annex F – Mathematical expressions and critical limits of ISO 5725-2

In this type of experiments (balanced uniform-level test results) there are: *p* laboratories called *i* (*i* = 1,2,…, *p*), each testing *q* levels called *j* (*j* = 1, 2, …, *q*) with *n* replicates at each level (each *ij* combination), giving, in the ideal case, a total of *pqn* results. In the case of the inter-laboratory test object of this report, this means that there are *p* = 13 participating laboratories (*p* = 12 for admittance), *q* =4 levels corresponding to the four anodic coating types prepared and *n* = 3, the number of specimens tested by each laboratory in each method, except in the case of thickness measurement of anodic coating type B, where *n* = 6.

However, because missing, or deviating test results, or outlying laboratories, or erroneous data, the ideal situation is not always attained.

For the scrutiny of results for consistency and outliers two approaches are introduced by ISO 5725-2:1994 [1].

- **Graphical consistency technique**

Two measures called Mandel's *k* and *h* statistics are used. To calculate the between-laboratory consistency statistic, *h*, use the following expression for each laboratory within each level:

$$h_{ij} = \frac{\bar{y}_{ij} - \bar{\bar{y}}_j}{\sqrt{\frac{1}{(p_j - 1)} \sum (\bar{y}_{ij} - \bar{\bar{y}}_j)^2}} \qquad (1)$$

where $\bar{y}_{ij}$ is the mean of test results reported by each laboratory *i* for level *j* (Annex D) and $\bar{\bar{y}}_j$ is the general mean for level *j*.

$$\bar{y}_{ij} = \frac{1}{n} \sum_{k=1}^{n_{ij}} y_{ijk} \qquad (2) \qquad\qquad \bar{\bar{y}}_j = \frac{\sum_{i=1}^{p} n_{ij} \bar{y}_{ij}}{\sum_{i=1}^{p} n_{ij}} \qquad (3)$$

n_{ij} is the number of test results reported by laboratory *i* for level *l*, y_{ijk} is any one of these test results and p_j is the number of laboratories reporting at least one test result for level *j* (excluding test results designated as outliers or as erroneous).

To calculate the within-laboratory consistency statistic, *k*, use the following expression for each laboratory within each level:

$$k_{ij} = \frac{s_{ij}\sqrt{p_j}}{\sqrt{\sum s_{ij}^2}} \qquad (4)$$

where s_{ij} means the standard deviation for each laboratory *i* at level *j* (Annex E):

$$s_{ij} = \sqrt{\frac{1}{n_{ij}-1}\sum_{k=1}^{n_j}(y_{ijk}-\overline{y}_{ij})^2} \qquad (5)$$

Examination of *h* and *k* plots may indicate that specific laboratories exhibit patterns of results markedly different from the others. Indicator lines serve as guides for this examination. Excessive *h* or *k* values near or outside these critical value lines may point out a straggler or an outlying result.

Table F.1 – Indicators for Mandel's *h* and *k* statistics critical value lines [1]

Significance level	*p*	*h*	*k*	
			n = 3	*n = 6*
1%	13	2,27	2,03	1,68
5%	13	1,84	1,69	1,46
1%	12[1]	2,25	2,02	-
5%	12[1]	1,83	1,69	-

1) For admittance analyses

- **Numerical outlier technique**

Two tests called Cochran's test and Grubb's test are applied to identify stragglers or outliers. The following practice is used:

- if the test statistic is less than or equal to its 5% critical value, the item tested is accepted as correct;
- if the test statistic is greater than its 5% critical value and less than or equal to its 1% critical value, the item tested is called a straggler;
- if the test statistic is greater than its 1% critical value, the item tested is called a statistical outlier.

Cochran's test is a test of the within-laboratory variabilities and should be applied first. This test is applied to the values of Annex E (measures of spread within cells) at each level separately. The Cochran's test statistic, C, is calculated using the following expression:

$$C = \frac{s_{max}^2}{\sum_{i=1}^{p} s_i^2} \qquad (6)$$

where s_{max} is the highest standard deviation in the set of results. If the highest standard deviation is classed as an outlier, then the values should be omitted and Cochran's test repeated on the remaining values.

Grubb's test is primarily a test of between-laboratory variability. It's applied to the highest and to the lowest mean values of the test results (Annex D). It implies a rearrangement of means data of each level in ascending order:

$x_i = \overline{y}_{ij}$, where i = 1, 2, …, p and $p = p_j$ where j is fixed

Grubb's test for one outlying observation (*single test*). To determine whether the largest or the smallest mean is an outlier, compute for each level the Grubb's statistic, G_p or G_1, respectively.

$$G_p = (x_p - \overline{x}) / s \qquad (7) \qquad\qquad G_1 = (\overline{x} - x_1) / s \qquad (8)$$

where

$$\overline{x} = \frac{1}{p}\sum_{i=1}^{p} x_i \qquad (9) \qquad\qquad s = \sqrt{\frac{1}{p-1}\sum_{i=1}^{p}(x_i - \overline{x})^2} \qquad (10)$$

If any of these means is shown to be an outlier by this test, exclude it, and repeat the test at the other extreme mean, but do not apply the Grubb's test for two outlying observations. If the Grubb's test (single) does not show any mean to be an outlier, apply the double-Grubb's test described below.

Grubb's test for two outlying observations (*double test*). To test whether the two largest observations are outliers, compute the Grubb's statistic G:

$$G = s_{p-1,p}^2 / s_0^2 \qquad (11)$$

where

$$s_0^2 = \sum_{i=1}^{p}(x_i - \overline{x})^2 \qquad (12) \qquad\qquad s_{p-1,p}^2 = \sum_{i=1}^{p-2}(x_i - \overline{x}_{p-1,p})^2 \qquad (13)$$

$$\bar{x}_{p-1,p} = \frac{1}{p-2}\sum_{i=1}^{p-2} x_i \quad (14)$$

Alternatively, to test the two smallest observations, compute the following Grubb's statistic *G*:

$$G = s_{1,2}^2 / s_0^2 \quad (15)$$

where

$$s_{1,2}^2 = \sum_{i=3}^{p} (x_i - \bar{x}_{1,2})^2 \quad (16) \qquad \bar{x}_{1,2} = \frac{1}{p-2}\sum_{i=3}^{p} x_i \quad (17)$$

Critical values for Cochran's test and Grubb's test are given in the next table. While for the Cochran's test and for the "single" Grubb's test outliers and stragglers give rise to values which are, respectively, larger than the tabulated 1% and 5% critical values, for the "double" Grubb's test, outliers and stragglers give rise to values which are smaller than the respective tabulated critical values.

Table F.2 - Critical values for Cochran's test and Grubb's test [1]

Significance level	*p*	*Cochran's test*		*Grubb's test*	
		n = 3	*n = 6*	*upper limit (single)*	*lower limit (double)*
1%	13	0,450	0,291	2,699	0,2016
5%	13	0,371	0,243	2,462	0,2836
1%	12[1]	0,475	-	2,636	0,1738
5%	12[1]	0,392	-	2,412	0,2537

1) For admittance analyses

After concluding the consistency tests and discarding the eventual outliers according to ISO 5725-2:1994[1], the calculation of the general means and variances can be done by applying the following expressions:

- **General means**

For level *j*, the general means is

$$\hat{m}_j = \bar{\bar{y}}_j = \frac{\sum_{i=1}^{p} n_{ij}\bar{y}_{ij}}{\sum_{i=1}^{p} n_{ij}} \quad (18)$$

- **Variances**

Three variances are calculated for each level. They are the repeatability variance, the between-laboratory variance and the reproducibility variance.

The repeatability variances is

$$s_{rj}^2 = \frac{\sum_{i=1}^{p}(n_{ij}-1)s_{ij}^2}{\sum_{i=1}^{p}(n_{ij}-1)} \qquad (19)$$

The between-laboratory variance is

$$s_{Lj}^2 = \frac{s_{dj}^2 - s_{rj}^2}{\overline{\overline{n}}_j} \qquad (20)$$

where

$$s_{dj}^2 = \frac{1}{p-1}\sum_{i=1}^{p} n_{ij}(\overline{y}_{ij} - \overline{\overline{y}}_j)^2 = \frac{1}{p-1}\left[\sum_{i=1}^{p} n_{ij}(\overline{y}_{ij})^2 - (\overline{\overline{y}}_j)^2 \sum_{i=1}^{p} n_{ij}\right] \qquad (21)$$

$$\overline{\overline{n}}_j = \frac{1}{p-1}\left[\sum_{i=1}^{p} n_{ij} - \frac{\sum_{i=1}^{p} n_{ij}^2}{\sum_{i=1}^{p} n_{ij}}\right] \qquad (22)$$

The reproducibility variance is

$$s_{Rj}^2 = s_{rj}^2 + s_{Lj}^2 \qquad (22)$$

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