LABORATÓRIO NACIONAL
DE ENGENHARIA CIVIL

QUALANOD INTER-LABORATORY TEST OF ANODIZED ALUMINIUM TESTING METHODS

STATISTICAL ANALYSIS - REPEATIBILITY AND REPRODUCIBILITY DETERMINATION

M. Salta, R. Fontinha, N. Garcia

Institutions/laboratories that intended to participate in this inter-laboratory test

Responsible	Country	Name	Laboratory	Participation
MEISSNER, Herbert	AUSTRIA	Aluminium Ranshofen Service GmbH	ARS	
GROMMEN, Marc	BELGIUM	CORI - Coatings Research Institute	CORI	
JOSEPH, Jean-Paul	FRANCE	TESTAL	TESTAL	
HOLZ, Marc	GERMANY	IFO GmbH – Institut für Oberflächentechnik	IFO GmbH	2
VGONTZAS Manolis	GREECE*	AAG Quality – EKANAL for Aluminium Ass. Of Greece	AAG	No results
JUHASZ, Péter	HUNGARY	EMI – Institut für Qualitätskontrolle	EMI	
BOI, Riccardo	ITALY	QUALITAL	QUALITAL	
BOER, Albertus de	NETHERLANDS*	COT bv – Centrum voor Onderzoek en Technisch Advies bv	COT	No results
TOMASSI, Piotr	POLAND	IMP – Instytut Mechaniki Precyzyjnej	IMP	
MOZARYN, Teresa	POLAND	ITB – Instytut Techniki Budowlanej	ITB	
SALTA, Manuela	PORTUGAL	LNEC – Laboratório Nacional de Engenheira Civil	LNEC	2
PAZ, Angel	SPAIN	QUALESPAÑA - Ministerio de Vivienda Subdirección General de Innovación y Calidad de la Edificación	QUALESPAÑA	
WERNER, René	SWITZERLAND	EMPA, Abtl. Korrosion	EMPA	
AVCI, Beyazit	TURKEY	TSI – Turkish Standards Institution	TSI	1
BARRON, Lynda	UK & IRELAND	Bodycote Materials Testing	EXOVA	

1) No results for weight loss chromium free test
2) Present falling sand test results

Testing methods

• **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 ISO 3210: 2010** – Anodizing of aluminium and its alloys – Assessment of quality of sealed anodic oxidation coatings by measurement of the loss of mass after immersion in phosphoric acid/chromic acid solution (ISO 3210: 2010). (Method 2)

• **Chromic free test** – Assessment of quality of sealed anodic oxidation coatings by measurement of the loss of mass after immersion in phosphoric acid solution (no standard).

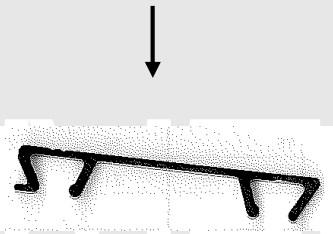
• **EN ISO 2931: 2010 –** Anodizing of aluminium and its alloys – Assessment of quality of sealed anodic oxidation coatings by ***measurement of admittance*** (ISO 2931: 2010)

• **EN ISO 2143: 2010** – Anodizing of aluminium and its alloys – Estimation of loss of absorptive power of anodic oxidation coatings after sealing – ***Dye-spot test*** with prior acid treatment (ISO 2143: 2010)

• **BS 6161-18:1991** – *Anodic oxidation coatings and its alloys. Part 18. Determination of surface* ***abrasion resistance****.*

• **EN ISO 8251: 2011** – Anodizing of aluminium and its alloys – Measurement of abrasion resistance of anodic oxidation coatings (ISO 8251: 2011). (Wheel wear test, falling sand)

General characteristics of the test specimens

SPECIMENS FOR TESTS	**Type of alloy**	6063	Surface for tests 
	Identification of samples	A, B, C, D, E, F and G	
	Samples	Two specimens by each sample	
	Dimensions	200 mm x 50 mm D 150 mm x 50 mm	
	Thickness classes	Three samples class 15, three samples class 25 and one sample class 20.	
STANDARD SPECIMEN	**Dimensions**	140 x 70 x 1 mm	

Anodizing and sealing conditions used for the production of test specimens

Anodic coating type	Anodizing		Hot water sealing	
A	*Free H_2SO_4*	180,3 g/l	*Demineralised water*	
	Al content	12,6 g/l	*pH*	5,7
	Temperature	18 ºC	*Additive*	P3 Almeco Seal
15	*Current density*	1,35 A/dm^2	*Time* -	3 min/µm
B	*Free H_2SO_4*	180,3 g/l	*Demineralised water*	
	Al content	12,6 g/l	*pH*	5,7
	Temperature	18 ºC	*Additive*	P3 Almeco Seal
20	*Current density*	1,35 A/dm^2	*Time* -	3 min/µm
C	*Free H_2SO_4*	180,3 g/l	*Demineralised water*	
	Al content	12,6 g/l	*pH*	5,7
	Temperature	18 ºC	*Additive*	P3 Almeco Seal
25	*Current density*	1,35 A/dm^2	*Time* -	3 min/µm

Anodizing and sealing conditions used for the production of test specimens (cont)

Anodic coating type	Anodizing		Hot water sealing	
D 15	*Free H_2SO_4*	180,3 g/l	*Demineralised water*	
	Al content	12,6 g/l	*pH*	5,7
	Temperature	25 ºC	*Additive*	P3 Almeco Seal
	Current density	1,35 A/dm^2	*Time* -	3 min/µm
E 25	*Free H_2SO_4*	180,3 g/l	*Demineralised water*	
	Al content	12,6 g/l	*pH*	5,7
	Temperature	25 ºC	*Additive*	P3 Almeco Seal
	Current density	1,35 A/dm^2	*Time* -	3 min/µm
F 15	*Free H_2SO_4*	180,3 g/l	*Demineralised water*	
	Al content	12,6 g/l	*pH*	5,7
	Temperature	18 ºC	*Additive*	P3 Almeco Seal
	Current density	1,35 A/dm^2	*Time* -	1 min/µm
G 25	*Free H_2SO_4*	180,3 g/l	*Demineralised water*	
	Al content	12,6 g/l	*pH*	5,7
	Temperature	18 ºC	*Additive*	P3 Almeco Seal
	Current density	1,35 A/dm^2	*Time* -	1 min/µm

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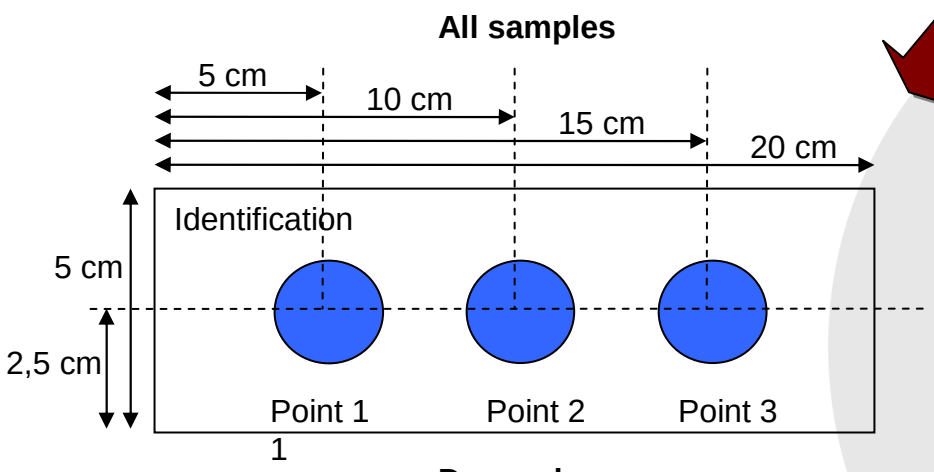
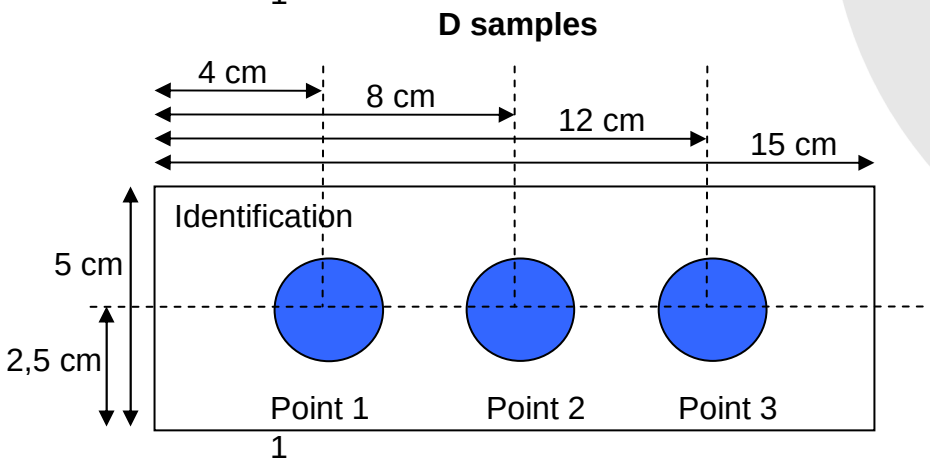
Instructions

- Laboratory code number
- Measuring points

Scheme of the testing zones for thickness measurement



a)



b)

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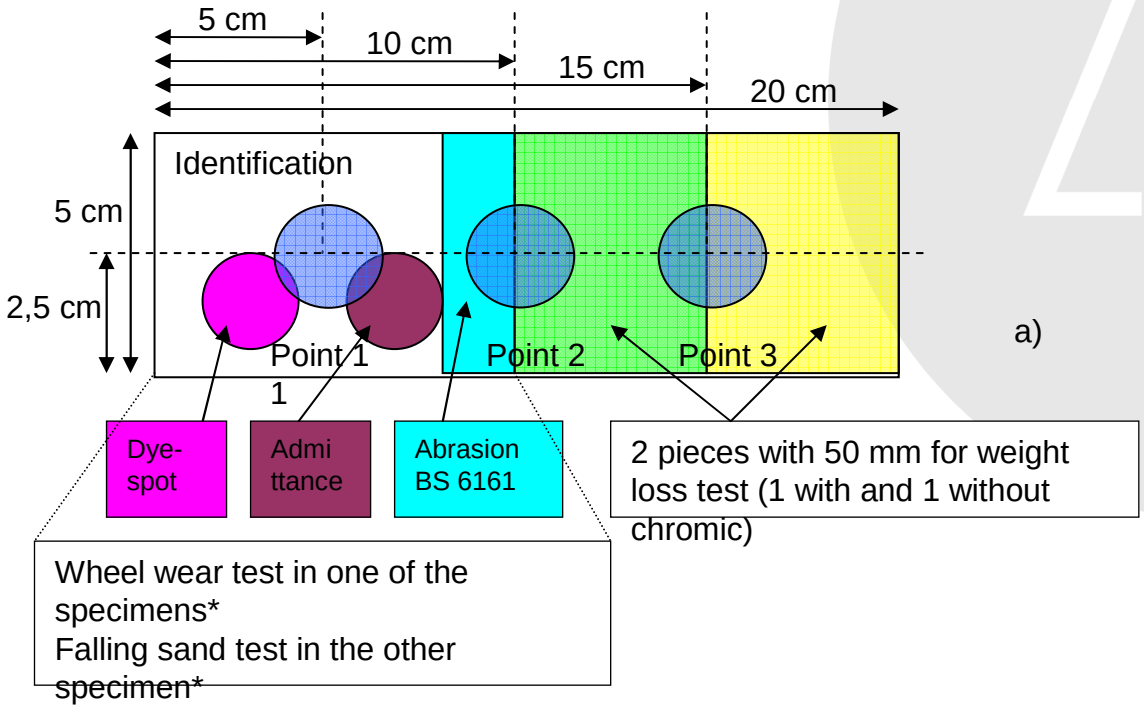
Instructions

Scheme of testing zones for sealing quality assessment

All samples



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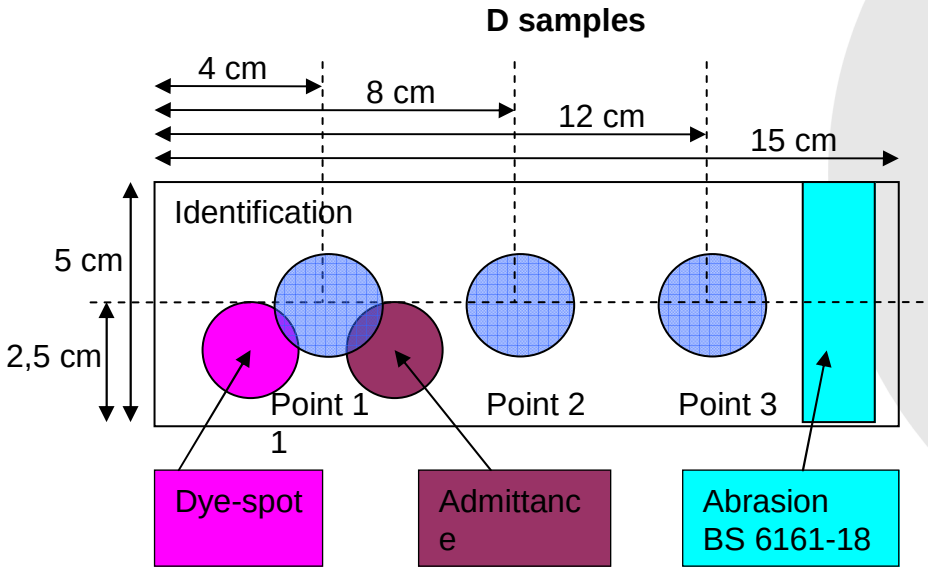
Instructions

Scheme of testing zones for sealing quality assessment



b)

Wheel wear test in one of the specimens*
Falling sand test in the other specimen*

* if only one test type is performed, please replicate in the 2 specimens of each sample

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EXCEL Worksheets for data registration

Protocol for A sample

Laboratory identification

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

Test conditions

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

Test results

Thickness results (EN 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 ISO 2931)

Sample No.	Measurement	Thickness	Temperature	Admittance
A15	Ymes	(nm)	(°C)	Y20
	15,0	24,6	21,2	20

Surface abrasion resistance results (BS 6161-18)

Harder or Softer	H	Method I or II	I

Dye spot results (EN ISO 2143)

Dye spot	0-1	Color	Red

Weight loss results (EN ISO 3210: method 2)

REMARKS: Length +- 50 mm Perimeter: 0,155 m^2/m

Sample No.	Length L	Weight_0	Weight_1	Weigth loss
A15	(mm)	(g)	(g)	(mg/dm^2)
measure 1	50,29	9,4563	9,4444	-
measure 2	50,09	-	-	-
measure 3	50,63	-	-	-
Average	50,34	9,4563	9,4444	15,25

Weight loss results (chromic free test)

REMARKS: Length +- 50 mm Perimeter: 0,155 m^2/m

Sample No.	Length L	Weight_0	Weight_1	Weigth loss
A15	(mm)	(g)	(g)	(mg/dm^2)
measure 1	50,29	9,4563	9,4444	-
measure 2	50,09	-	-	-
measure 3	50,63	-	-	-
Average	50,34	9,4563	9,4444	15,25

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

Sample No.	Measurement	Thickness	Temperature	Admittance
	Ymes	(nm)	(°C)	Y20

Harder or Softer	H	Method I or II	I

Dye spot	0-1	Color	Red

Sample No.	Length L	Weight_0	Weight_1	Weigth loss
	(mm)	(g)	(g)	(mg/dm^2)
measure 1				-
measure 2		-	-	-
measure 3		-	-	-
Average				

Sample No.	Length L	Weight_0	Weight_1	Weigth loss
	(mm)	(g)	(g)	(mg/dm^2)
measure 1				-
measure 2		-	-	-
measure 3		-	-	-
Average				

Abrasion test results

Wheel wear test (EN ISO 8251) - Thickness measurements (EN ISO 2360)

Standard	Before abrasion ($d1_s$)		
	1	2	3
measure 1	25,6		
measure 2	25,4		
measure 3	25,3		
Average thickness	25,4		

Standard	After abrasion ($d2_s$)		
	1	2	3
measure 1	20,2		
measure 2	20,2		
measure 3	20,2		
Average thickness	20,2		

Sample No.	Before abrasion ($d1_p$)		
A15	1	2	3
measure 1	24,5		
measure 2	24,6		
measure 3	24,5		
Average thickness	24,5		

Sample No.	After abrasion ($d2_p$)		
B02	1	2	3
measure 1	19,4		
measure 2	19,4		
measure 3	19,4		
Average thickness	19,4		

Final results summary

$d1_s$-$d2_s$	5,2
$d1_p$-$d2_p$	5,1
WR	78,4
WRC	1,02
WI	0,98
CWR	102

Standard	Before abrasion ($d1_s$)		
	1	2	3
measure 1			
measure 2			
measure 3			
Average thickness			

Standard	After abrasion ($d2_s$)		
	1	2	3
measure 1			
measure 2			
measure 3			
Average thickness			

Sample No.	Before abrasion ($d1_p$)		
	1	2	3
measure 1			
measure 2			
measure 3			
Average thickness			

Sample No.	After abrasion ($d2_p$)		
	1	2	3
measure 1			
measure 2			
measure 3			
Average thickness			

Final results summary	
$d1_p$-$d2_p$	
$d1_s$-$d2_s$	
WR	
WRC	
WI	
CWR	

Falling sand test (EN ISO 8251: spot diameter method) - Thickness measurements (EN ISO 2360)

Standard	Before abrasion ($d1_s$)		
	1	2	3
measure 1	25,6		
measure 2	25,4		
measure 3	25,3		
Average thickness	25,4		

Standard	After abrasion		
	1	2	3
Time (seconds)	68,0		
Average thickness	68,0		

Sample No.	Before abrasion		
A15	1	2	3
measure 1	24,5		
measure 2	24,6		
measure 3	24,5		
Average thickness	24,5		

Sample No.	After abrasion		
A15	1	2	3
Time (seconds)	75,0		
Time	75,0		

Final results summary	
Standard	
min	68
Sample	
min	75

Standard	Before abrasion ($d1_s$)		
	1	2	3
measure 1			
measure 2			
measure 3			
Average thickness			

Standard	After abrasion		
	1	2	3
Time (seconds)			
Average thickness			

Sample No.	Before abrasion		
	1	2	3
measure 1			
measure 2			
measure 3			
Average thickness			

Sample No.	After abrasion		
	1	2	3
Time (seconds)			
Time			

Final results summary	
Standard	
min	
Sample	
min	

Example for the registration of tests results for specimens of coating type A

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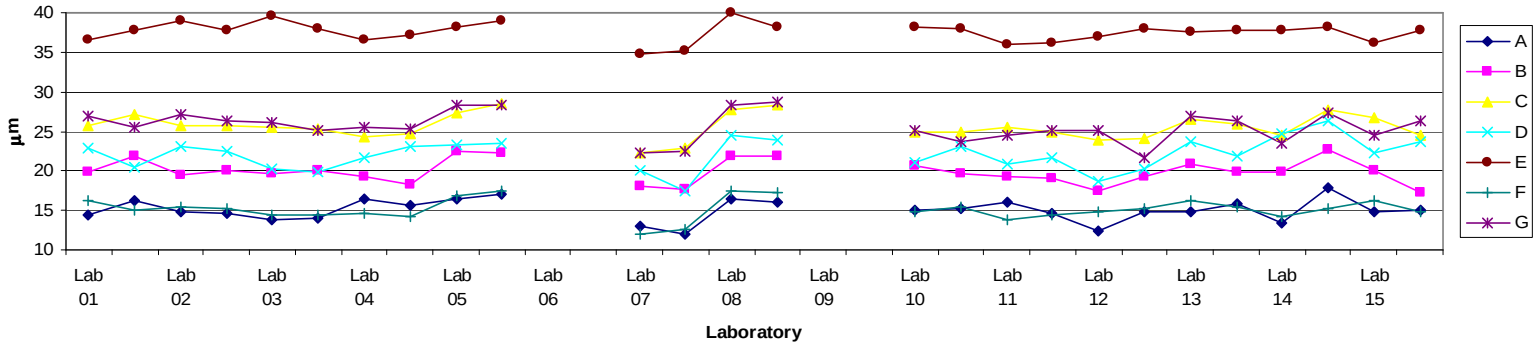
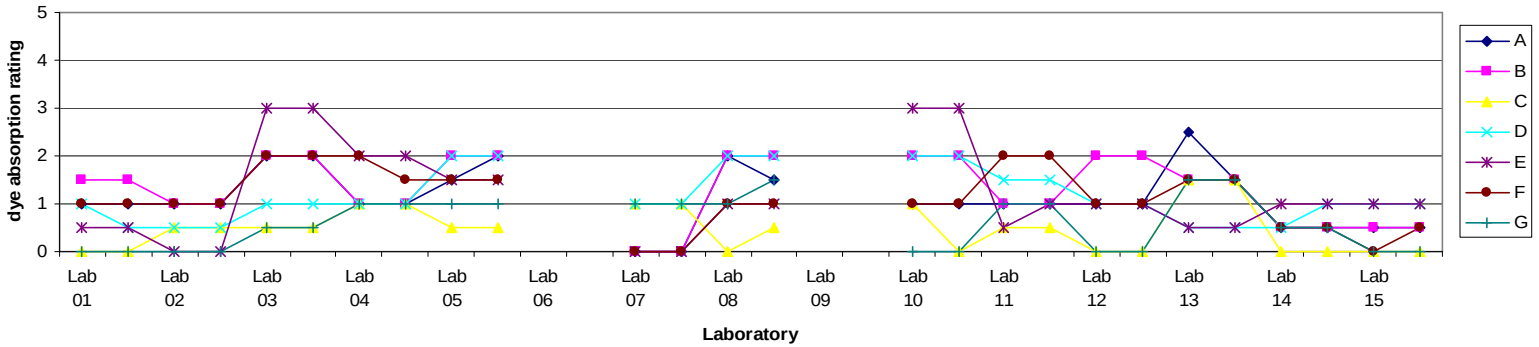
Results reported

Test results of method EN ISO 2360



Test results of method EN ISO 2143



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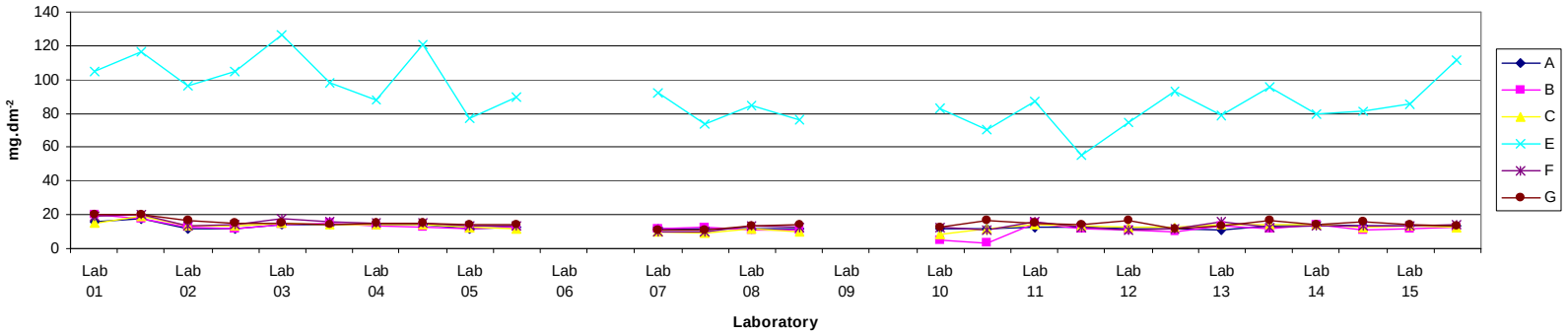
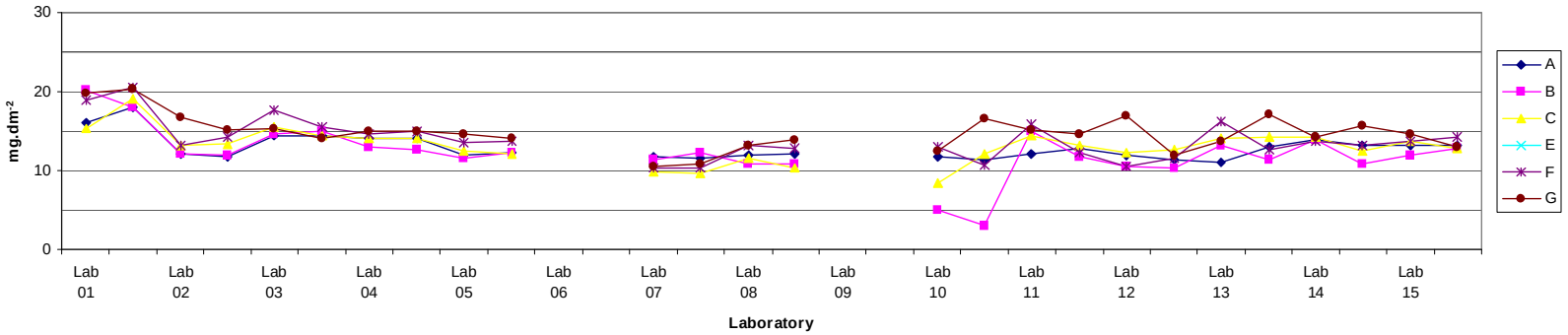
Results reported

Test results of method EN ISO 3210





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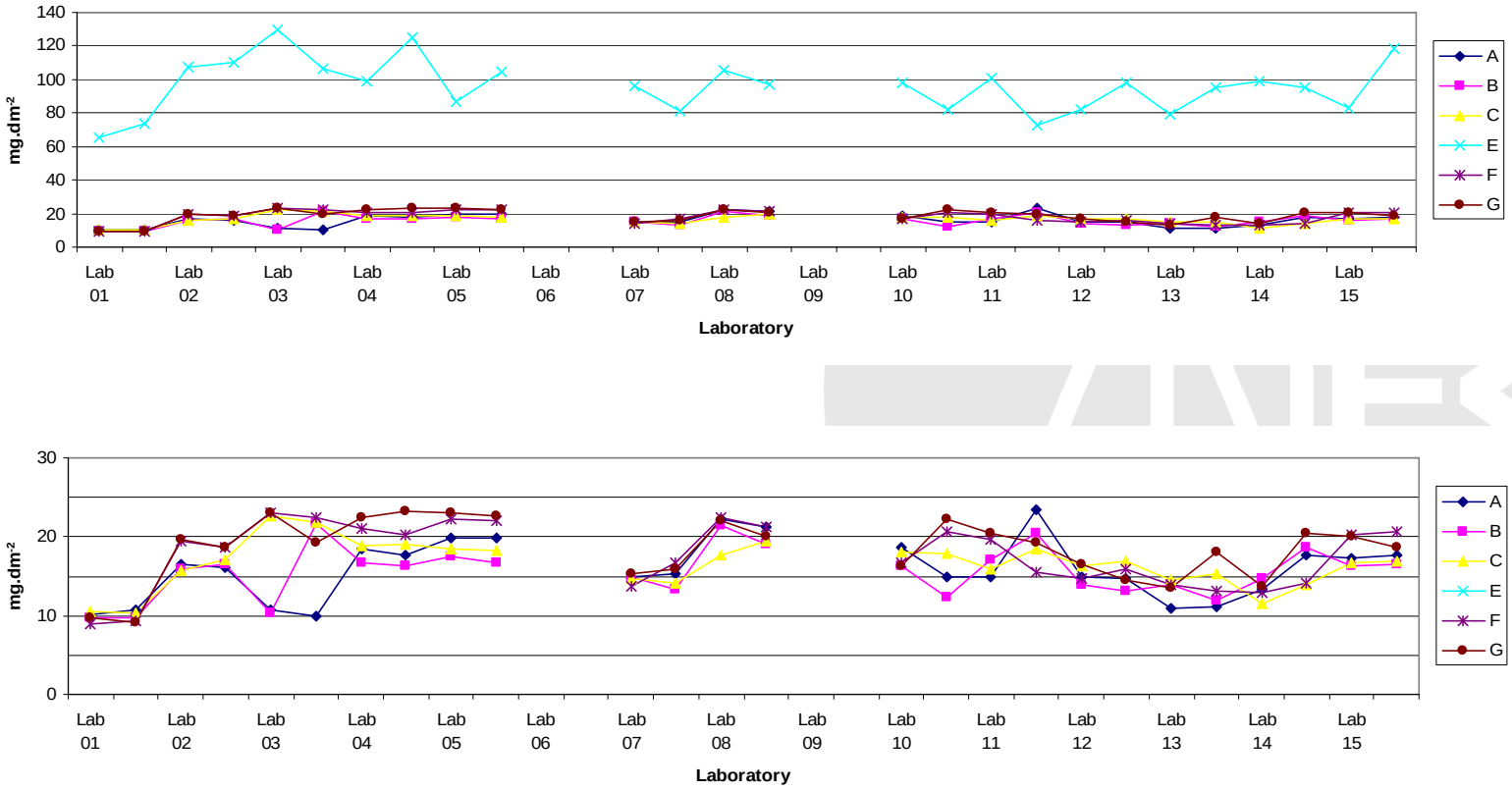
Results reported

Test results of method Chromium free test



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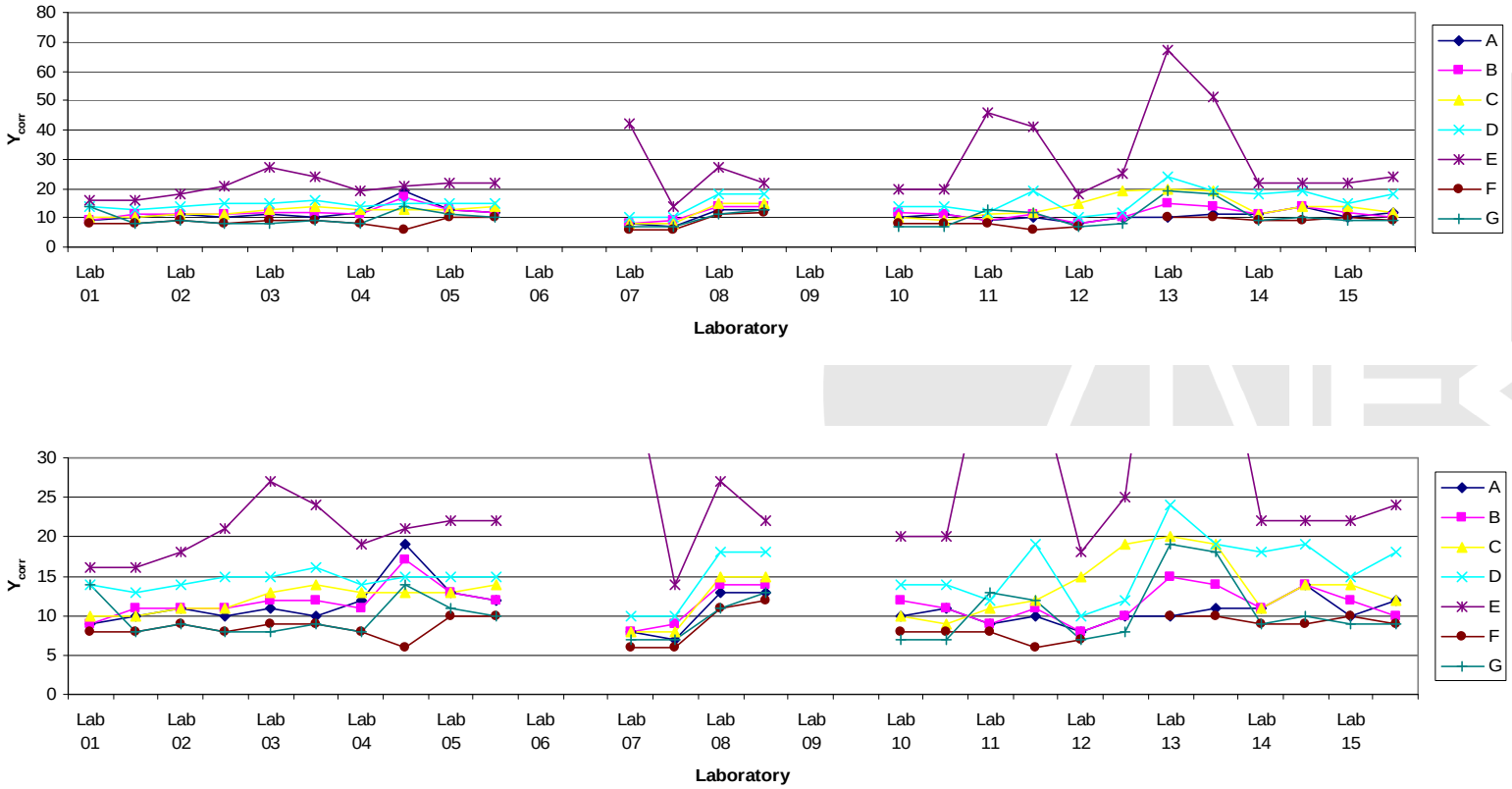
Results reported

Test results of method EN ISO 2931



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Precision analysis according to ISO 5725-2

- Consistency tests

  - **Graphical consistency technique** – Mandel's *h* and *k* statistics

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

  - **Numerical outlier technique** – Cochran's and Grubb's tests

$$C = \frac{s_{max}^2}{\sum_{i=1}^{p} s_i^2} \qquad G_p = (x_p - \bar{x})/s \qquad G_1 = (\bar{x} - x_1)/s$$

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

- Calculation of the **general mean** and **variances**

$$\hat{m}_j = \bar{\bar{y}}_j = \frac{\sum_{i=1}^{p} n_{ij} \bar{y}_{ij}}{\sum_{i=1}^{p} n_{ij}} \qquad s_{rj}^2 = \frac{\sum_{i=1}^{p} (n_{ij} - 1) s_{ij}^2}{\sum_{i=1}^{p} (n_{ij} - 1)}$$

Repeatability variance

$$s_{Rj}^2 = s_{rj}^2 + s_{Lj}^2$$

Reproducibility variance

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

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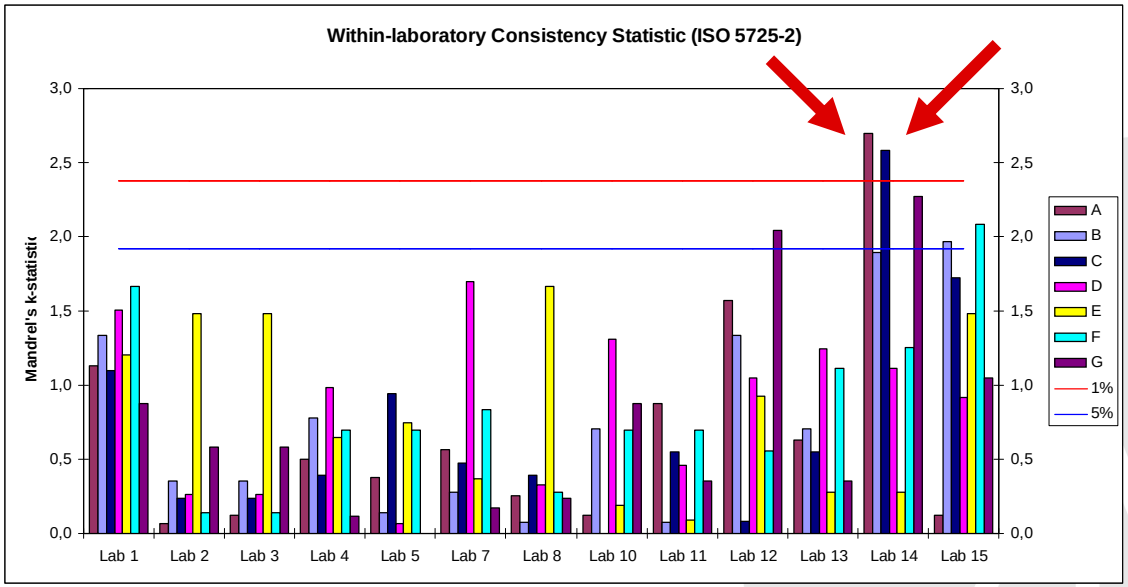
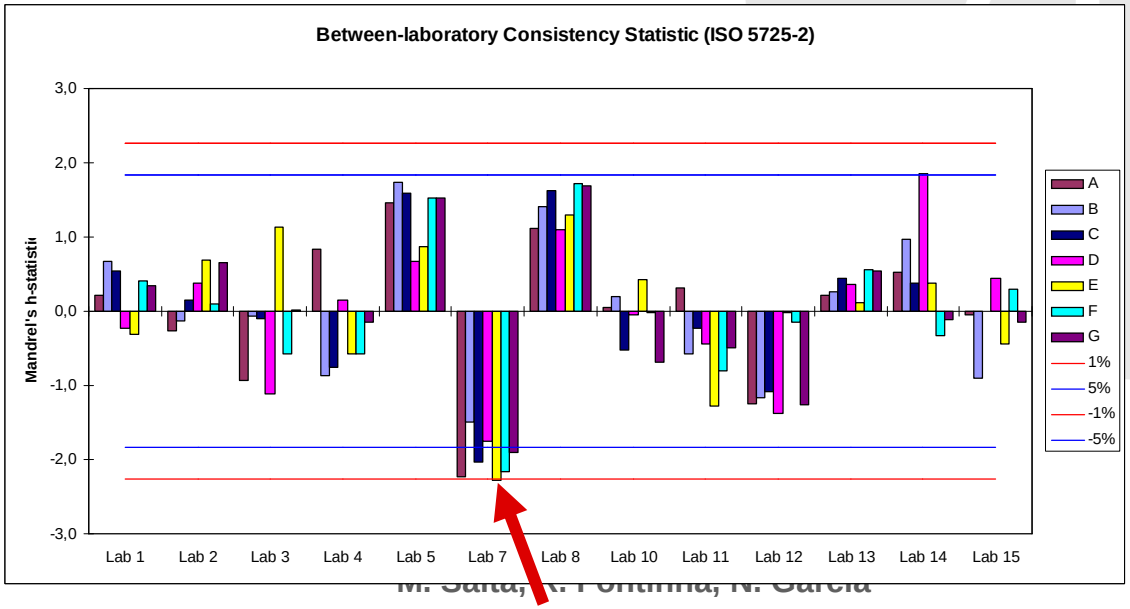
Thickness measurement (EN ISO 2360)





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Thickness measurement (EN ISO 2360)

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Laboratories outside critical value lines of Mandel's statistics

Level	A	B	C	D	E	F	G
Mandel's k-plot	Lab 14	Lab 15	Lab 14	-	-	Lab 15	Lab 12, Lab 14
Classification	*Outlier*	*Straggler*	*Outlier*	-	-	*Straggler*	*Straggler*
Mandel´s h-plot	Lab 7	-	Lab 7	Lab 14	Lab 7	Lab 7	Lab 7
Classification	*Straggler*	-	*Straggler*	*Straggler*	*Outlier*	*Straggler*	*Straggler*

Cochran's test results

Level	A	B	C	D	E	F	G
Valid laboratories *p*	13	13	13	13	13	13	13
Number of replicates *n*	2	2	2	2	2	2	2
1% Critical value $C_{Cr\ (1\%)}$	0,624	0,624	0,624	0,624	0,624	0,624	0,624
5% Critical value $C_{Cr\ (5\%)}$	0,515	0,515	0,515	0,515	0,515	0,515	0,515
Cochran's test statistic ***C***	0,560	0,298	0,515	0,222	0,214	0,335	0,398
Classification	*Straggler*	*Correct*	*Correct*	*Correct*	*Correct*	*Correct*	*Correct*
Straggler Lab (***C*** >$C_{Cr\ (1\%)}$)	Lab 14	-	-	-	-	-	-
Outlier Lab (***C*** >$C_{Cr\ (1\%)}$)	-	-	-	-	-	-	-

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Thickness measurement (EN ISO 2360)

Grubb's test results

Level	A	B	C	D	E	F	G
Valid laboratories *p*	13	13	13	13	13	13	13
Single G_{Cr} (1%)	2,699	2,699	2,699	2,699	2,699	2,699	2,699
Single G_{Cr} (5%)	2,462	2,462	2,462	2,462	2,462	2,462	2,462
Single high G_p	1,463	1,733	1,623	1,848	1,301	1,727	1,690
Single low G_1	2,225	1,496	2,036	1,748	2,282	2,164	1,900
Classification (low)	*Correct*	*Correct*	*Correct*	*Correct*	*Correct*	*Correct*	*Correct*
Outlier Lab ($G_p > G_{Cr}$ (1%))	-	-	-	-	-	-	-
Classification (low)	*Correct*	*Correct*	*Correct*	*Correct*	*Correct*	*Correct*	*Correct*
Outlier Lab ($G_1 > G_{Cr}$ (1%))	-	-	-	-	-	-	-
Double G_{Cr} (1%)	0,2016	0,2016	0,2016	0,2016	0,2016	0,2016	0,2016
Double G_{Cr} (5%)	0,2836	0,2836	0,2836	0,2836	0,2836	0,2836	0,2836
Double high $G_{largest}$	0,6696	0,5110	0,4916	0,5480	0,7087	0,4755	0,4920
Double low $G_{smalest}$	0,3667	0,6464	0,4836	0,5139	0,3339	0,4897	0,4891
Classification (two largest)	*Correct*	*Correct*	*Correct*	*Correct*	*Correct*	*Correct*	*Correct*
Outlier Lab($G_{largest} < G_{Cr}$ (1%))	-	-	-	-	-	-	-
Classification (two smallest)	*Correct*	*Correct*	*Correct*	*Correct*	*Correct*	*Correct*	*Correct*
Outlier Lab($G_{smalest} < G_{Cr}$ (1%))	-	-	-	-	-	-	-

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

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Thickness measurement (EN ISO 2360)

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Results of precision analysis

Level	A	B	C	D	E	F	G
Number of replicates *n*	2	2	2	2	2	2	2
Valid laboratories *p*	13	13	13	13	13	13	13
General mean *m* / µm	**15,05**	**19,94**	**25,59**	**22,08**	**37,51**	**15,18**	**25,66**
Repeatability variance s_r^2	1,271	1,011	0,810	1,177	0,539	0,260	1,456
Between-lab variance s_L^2	0,631	1,351	1,895	2,934	1,040	1,522	2,303
Reproducibility variance s_R^2	1,902	2,361	2,706	4,111	1,579	1,782	3,759
Repeatability std. dev. s_r	**1,13**	**1,01**	**0,90**	**1,08**	**0,73**	**0,51**	**1,21**
Reproducibility std. dev. s_R	**1,38**	**1,54**	**1,64**	**2,03**	**1,26**	**1,33**	**1,94**
Repeatability COV (s_r/m)	7,5 %	5,0 %	3,5 %	4,9 %	2,0 %	3,4 %	4,7 %
Reproducibility COV (s_R/m)	9,2 %	7,7 %	6,4 %	9,2 %	3,3 %	8,8 %	7,6 %
Number of outliers	1	0	1	0	1	0	0
Number of excluded outliers	0	0	0	0	0	0	0
Outlier type	M*k*	-	M*k*	-	M*h*	-	-
Outlier laboratories	Lab 14	-	Lab 14	-	Lab 7	-	-

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

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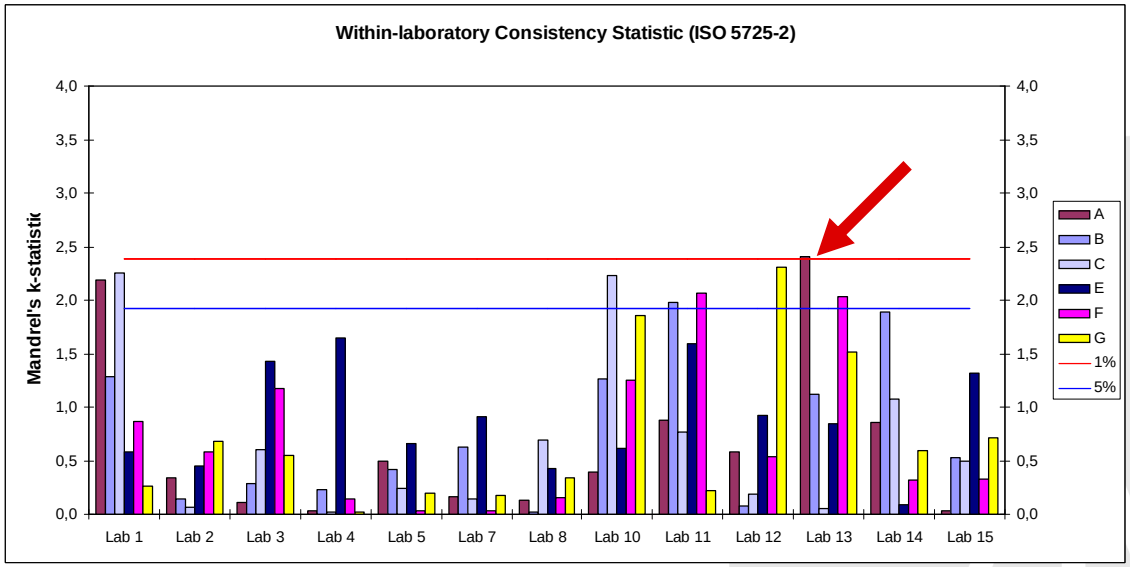
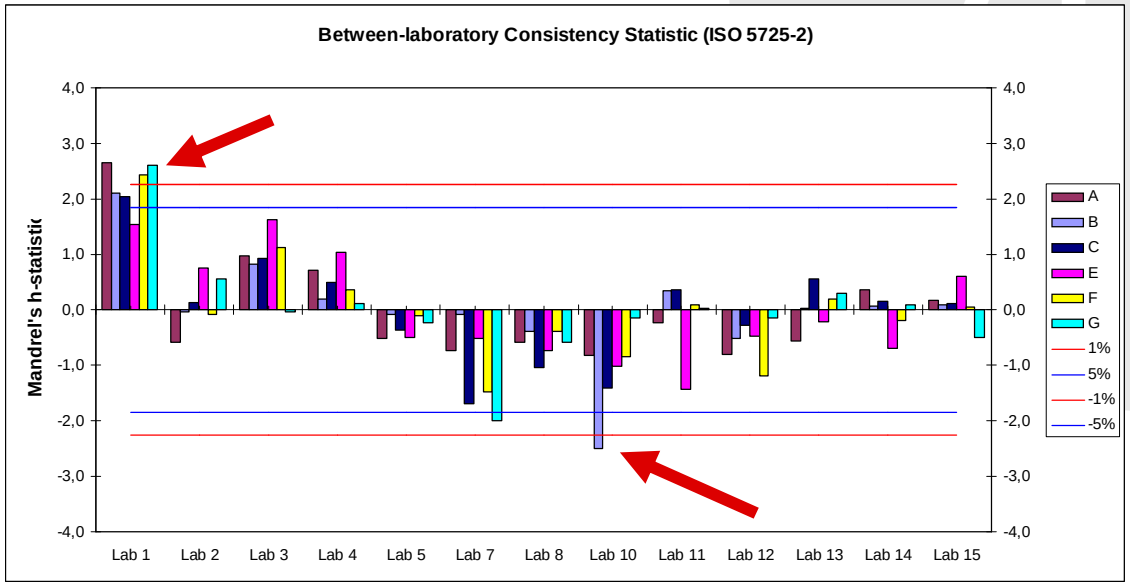
Sealing quality assessment by mass loss (EN ISO 3210)





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LABORATÓRIO NACIONAL DE ENGENHARIA CIVIL

Sealing quality assessment by mass loss (EN ISO 3210)

Laboratories outside critical value lines of Mandel's statistics

Level	A	B	C	E	F	G
Mandel's k-plot	Lab 13[1] Lab 1[2]	Lab 11	Lab 1, 10	-	Lab 11, 13	Lab 12
Classification	[1]***Outlier*** [2]*Straggler*	*Straggler*	*Straggler*	-	*Straggler*	*Straggler*
Mandel´s h-plot	Lab 1	Lab 1[1] Lab 10[2]	Lab 1	-	Lab 1	Lab 1[1] Lab 7[2]
Classification	***Outlier***	[1]*Straggler* [2]***Outlier***	*Straggler*	-	***Outlier***	[1]***Outlier*** [2]*Straggler*

Cochran's test results

Level	A	B	C	E	F	G
Valid laboratories *p*	13	13	13	13	13	13
Number of replicates *n*	2	2	2	2	2	2
1% Critical value $C_{Cr\ (1\%)}$	0,624	0,624	0,624	0,624	0,624	0,624
5% Critical value $C_{Cr\ (5\%)}$	0,515	0,515	0,515	0,515	0,515	0,515
Cochran's test statistic ***C***	0,446	0,301	0,390	0,210	0,328	0,410
Classification	*Correct*	*Correct*	*Correct*	*Correct*	*Correct*	*Correct*
Outlier Lab (***C*** >$C_{Cr\ (1\%)}$)	-	-	-	-	-	-

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LABORATÓRIO NACIONAL DE ENGENHARIA CIVIL

Sealing quality assessment by mass loss (EN ISO 3210)

Grubb's test results

Level	A	B	C	E	F	G
Valid laboratories *p*	13	13	13	13	13	13
Single G_{Cr} (1%)	2,699	2,699	2,699	2,699	2,699	2,699
Single G_{Cr} (5%)	2,462	2,462	2,462	2,462	2,462	2,462
Single high G_p	2,663	2,108	2,048	1,628	2,443	2,615
Single low G_1	0,836	2,505	1,694	1,428	1,481	2,010
Classification (high)	*Straggler*	*Correct*	*Correct*	*Correct*	*Correct*	*Straggler*
Outlier Lab ($G_p > G_{Cr}$ (1%))	Lab 1	-	-	-	-	Lab 1
Classification (low)	*Correct*	*Straggler*	*Correct*	*Correct*	*Correct*	*Correct*
Outlier Lab ($G_1 > G_{Cr}$ (1%))	-	Lab 1	-	-	-	-
Double G_{Cr} (1%)	0,2016	0,2016	0,2016	0,2016	0,2016	0,2016
Double G_{Cr} (5%)	0,2836	0,2836	0,2836	0,2836	0,2836	0,2836
Double high $G_{largest}$	0,2295	0,5094	0,5098	0,5056	0,2976	0,3273
Double low $G_{smalest}$	0,8660	0,3851	0,5193	0,6987	0,6454	0,5827
Classification (two largest)	*Straggler*	*Correct*	*Correct*	*Correct*	*Correct*	*Correct*
Outlier Lab($G_{largest} < G_{Cr}$ (1%))	Lab 1	-	-	-	-	-
Classification (two smallest)	*Correct*	*Correct*	*Correct*	*Correct*	*Correct*	*Correct*
Outlier Lab($G_{smalest} < G_{Cr}$ (1%))	-	-	-	-	-	-

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

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LABORATÓRIO NACIONAL DE ENGENHARIA CIVIL

Sealing quality assessment by mass loss (EN ISO 3210)

Results of precision analysis

Level	A	B	C	E	F	G
Number of replicates *n*	2	2	2	2	2	2
Valid laboratories *p*	13	13	13	13	13	13
General mean *m* / g.dm^{-2}	**12,92**	12,19	**13,10**	**90,23**	**13,93**	**14,83**
Repeatability variance s_r^2	0,345	1,319	1,471	198,16	1,529	2,329
Between-lab variance s_L^2	2,249	9,968	3,319	82,704	4,925	2,888
Reproducibility variance s_R^2	2,595	11,287	4,790	280,86	6,454	5,217
Repeatability std. dev. s_r	**0,59**	1,15	**1,21**	**14,08**	**1,24**	**1,53**
Reproducibility std. dev. s_R	**1,61**	3,36	**2,19**	**16,76**	**2,54**	**2,28**
Repeatability COV (s_r/m)	4,5 %	9,4%	9,3 %	15,6 %	8,9 %	10,3 %
Reproducibility COV (s_R/m)	12,5 %	27,6%	16,7 %	18,6 %	18,2 %	15,4 %
Number of outliers	2	1	0	0	1	1
Number of excluded outliers	0	0	0	0	0	0
Outlier type	[1]M*h*, [2]M*k*	M*h*	-	-	M*h*	M*h*
Outlier laboratories	Lab 1 [1] Lab 13 [2]	Lab 1	-	-	Lab 1	Lab 1

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

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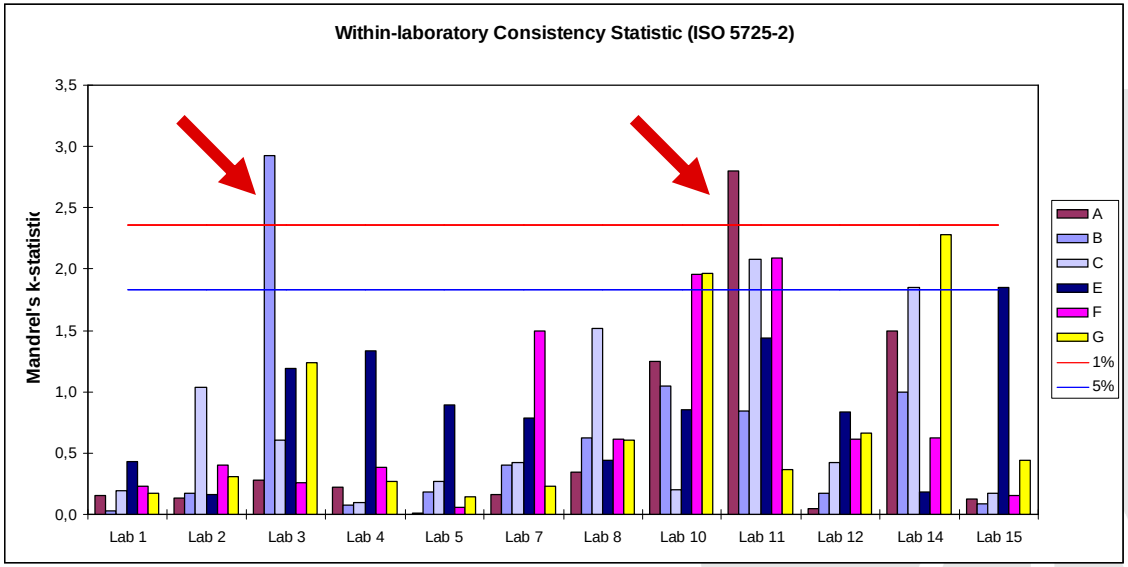
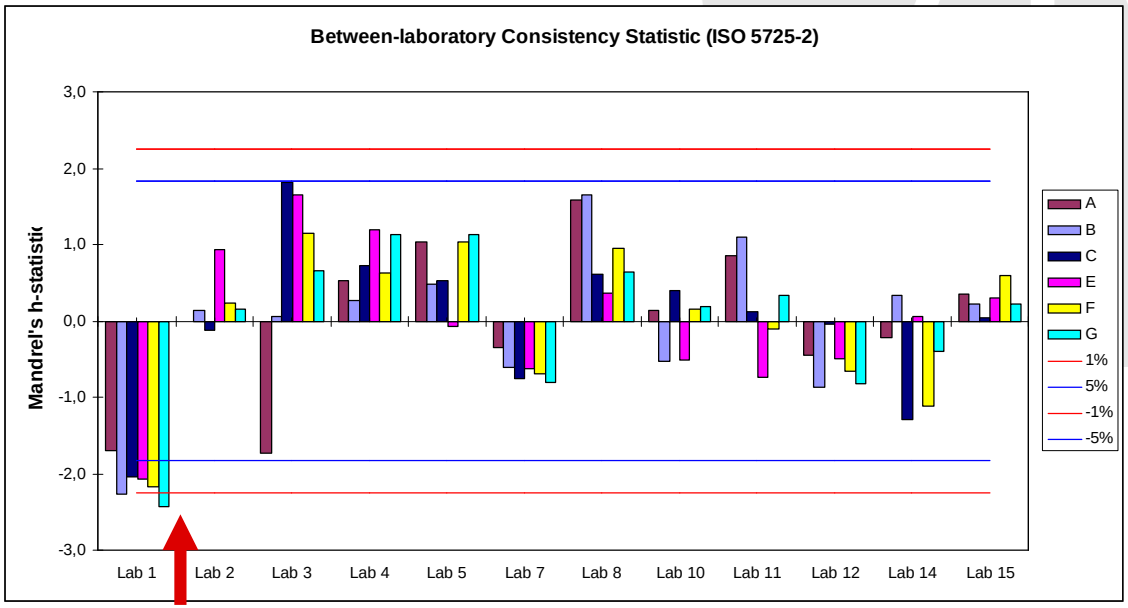
LABORATÓRIO NACIONAL DE ENGENHARIA CIVIL

Sealing quality assessment by mass loss (Chromium free)





LABORATÓRIO NACIONAL DE ENGENHARIA CIVIL

Sealing quality assessment by mass loss (Chromium free)

Laboratories outside critical value lines of Mandel's statistics

Level	A	B	C	E	F	G
Mandel's k-plot	Lab 11	Lab 3	Lab 11, 14	Lab 15	Lab 10, 11	Lab 10, 14
Classification	*Outlier*	*Outlier*	*Straggler*	*Straggler*	*Straggler*	*Straggler*
Mandel´s h-plot	-	Lab 1	Lab 1	Lab 1	Lab 1	Lab 1
Classification	-	*Outlier*	*Straggler*	*Straggler*	*Straggler*	*Outlier*

Cochran's test results

Level	A	B	C	E	F	G
Valid laboratories *p*	12	12	12	12	12	12
Number of replicates *n*	2	2	2	2	2	2
1% Critical value $C_{Cr\ (1\%)}$	0,653	0,653	0,653	0,653	0,653	0,653
5% Critical value $C_{Cr\ (5\%)}$	0,541	0,541	0,541	0,541	0,541	0,541
Cochran's test statistic ***C***	0,655	0,713	0,360	0,286	0,364	0,432
Classification	*Outlier*	*Outlier*	*Correct*	*Correct*	*Correct*	*Correct*
Outlier Lab (***C*** >$C_{Cr\ (1\%)}$)	Lab 11	Lab 3	-	-	-	-

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LABORATÓRIO NACIONAL DE ENGENHARIA CIVIL

Sealing quality assessment by mass loss (Chromium free)

Grubb's test results

Level	A	B	C	E	F	G
Valid laboratories *p*	12	12	12	12	12	12
Single G_{Cr} (1%)	2,636	2,636	2,636	2,636	2,636	2,636
Single G_{Cr} (5%)	2,412	2,412	2,412	2,412	2,412	2,412
Single high G_p	1,581	1,658	1,806	1,658	1,153	1,136
Single low G_1	1,735	2,274	2,036	2,073	2,175	2,428
Classification (high)	*Correct*	*Correct*	*Correct*	*Correct*	*Correct*	*Correct*
Outlier Lab ($G_p > G_{Cr}$ (1%))	-	-	-	-	-	-
Classification (low)	*Correct*	*Correct*	*Correct*	*Correct*	*Correct*	*Straggler*
Outlier Lab ($G_1 > G_{Cr}$ (1%))	-	-	-	-	-	Lab 1
Double G_{Cr} (1%)	0,1738	0,1738	0,1738	0,1738	0,1738	0,1738
Double G_{Cr} (5%)	0,2537	0,2537	0,2537	0,2537	0,2537	0,2537
Double high $G_{largest}$	0,6130	0,5726	0,5990	0,5469	0,7404	0,7219
Double low $G_{smalest}$	0,3540	0,3730	0,3700	0,4882	0,3606	0,3082
Classification (two largest)	*Correct*	*Correct*	*Correct*	*Correct*	*Correct*	*Correct*
Outlier Lab($G_{largest} < G_{Cr}$ (1%))	-	-	-	-	-	-
Classification (two smallest)	*Correct*	*Correct*	*Correct*	*Correct*	*Correct*	*Correct*
Outlier Lab($G_{smalest} < G_{Cr}$ (1%))	-	-	-	-	-	-

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

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LABORATÓRIO NACIONAL DE ENGENHARIA CIVIL

Sealing quality assessment by mass loss (Chromium free)

Results of precision analysis

Level	A	B	C	E	F	G
Number of replicates *n*	2	2	2	2	2	2
Valid laboratories *p*	11	11	12	12	12	12
General mean *m* / g.dm^{-2}	**16,00**	**15,79**	**16,74**	**96,57**	**18,02**	**18,61**
Repeatability variance s_r^2	1,740	2,345	0,802	185,272	2,087	4,560
Between-lab variance s_L^2	11,117	6,761	8,984	78,744	15,613	12,079
Reproducibility variance s_R^2	12,857	9,106	9,786	264,017	17,700	16,640
Repeatability std. dev. s_r	**1,32**	**1,53**	**0,90**	**13,61**	**1,44**	**2,14**
Reproducibility std. dev. s_R	**3,59**	**3,02**	**3,13**	**16,25**	**4,21**	**4,08**
Repeatability COV (s_r/m)	8,2	9,7	5,3	14,1	8,0	11,5
Reproducibility COV (s_R/m)	22,4	19,1	18,7	16,8	23,3	21,9
Number of outliers	1	1	0	0	0	1
Number of excluded outliers	1	1	0	0	0	0
Outlier type	M*k*, C	[1]M*h*, [3]M*k*, [4]C	-	-	-	M*h*
Outlier laboratories	Lab 11	Lab 1 [1] Lab 3 [3,4]	-	-	-	Lab 1

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

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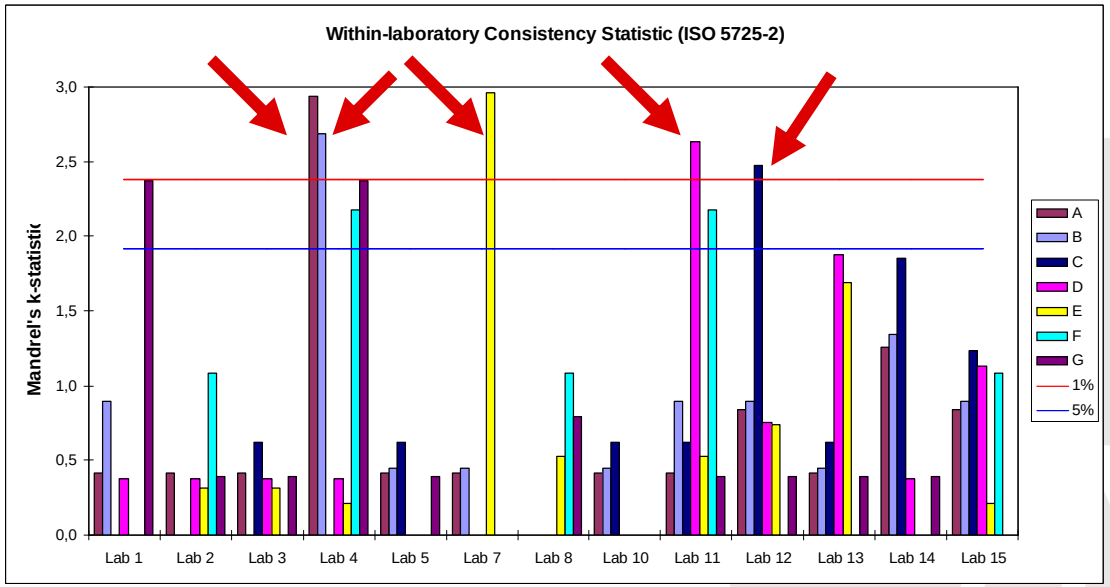
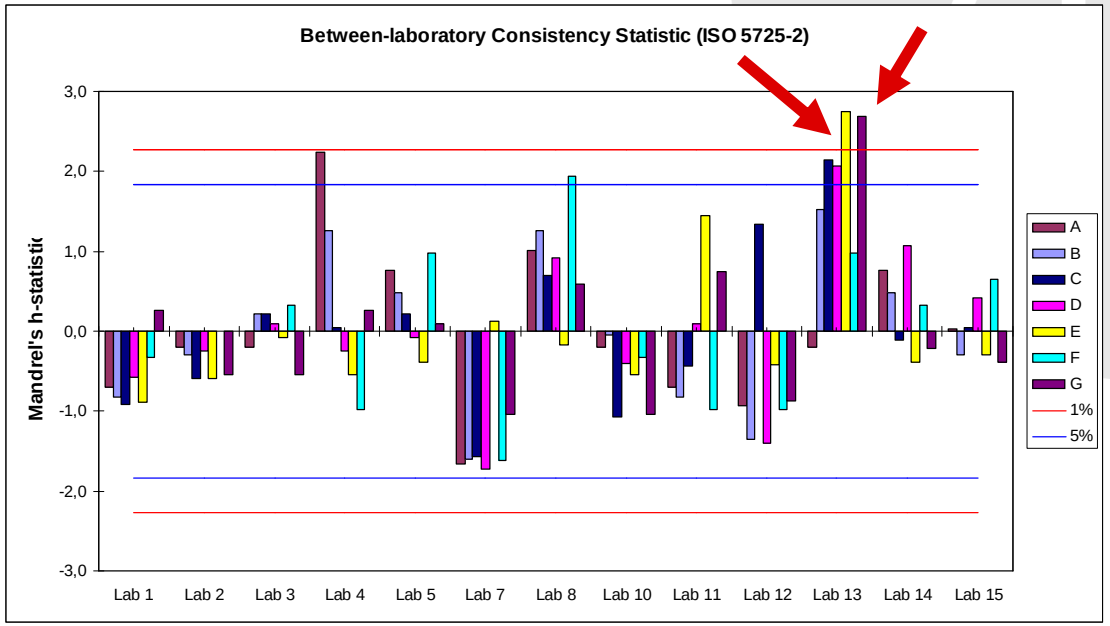
LABORATÓRIO NACIONAL DE ENGENHARIA CIVIL

Sealing quality assessment by measurement of admittance (EN ISO 2931)





LABORATÓRIO NACIONAL DE ENGENHARIA CIVIL

Sealing quality assessment by measurement of admittance (EN ISO 2931)

Laboratories outside critical value lines of Mandel's statistics

Level	A	B	C	D	E	F	G
Mandel's k-plot	Lab 4	Lab 4	Lab 12	Lab 11	Lab 7	Lab 4, 11	Lab 1, 4
Classification	*Outlier*	*Outlier*	*Outlier*	*Outlier*	*Outlier*	*Straggler*	*Straggler*
Mandel´s h-plot	Lab 4	-	Lab 13	Lab 13	Lab 13	Lab 8	Lab 13
Classification	*Straggler*	-	*Straggler*	*Straggler*	*Outlier*	*Straggler*	*Outlier*

Cochran's test results

Level	A	B	C	D	E	F	G
Valid laboratories *p*	13	13	13	13	13	13	13
Number of replicates *n*	2	2	2	2	2	2	2
1% Critical value $C_{Cr\ (1\%)}$	0,624	0,624	0,624	0,624	0,624	0,624	0,624
5% Critical value $C_{Cr\ (5\%)}$	0,515	0,515	0,515	0,515	0,515	0,515	0,515
Cochran's test statistic ***C***	0,662	0,554	0,471	0,533	0,673	0,364	0,434
Classification	*Outlier*	*Straggler*	*Correct*	*Straggler*	*Outlier*	*Correct*	*Correct*
Outlier Lab (***C*** >$C_{Cr\ (1\%)}$)	Lab 4	Lab 7	-	Lab 7	Lab 7, 13	-	-

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LABORATÓRIO NACIONAL DE ENGENHARIA CIVIL

Sealing quality assessment by measurement of admittance (EN ISO 2931)

Grubb's test results

Level	A	B	C	D	E	F	G
Valid laboratories *p*	13	13	13	13	13	13	13
Single G_{Cr} (1%)	2,699	2,699	2,699	2,699	2,699	2,699	2,699
Single G_{Cr} (5%)	2,462	2,462	2,462	2,462	2,462	2,462	2,462
Single high G_p	2,233	1,528	2,148	2,068	2,752	1,947	2,692
Single low G_1	1,670	1,608	1,564	1,725	0,889	1,622	1,035
Classification (high)	*correct*	*correct*	*correct*	*correct*	*Outlier*	*correct*	*Straggler*
Outlier Lab ($G_p > G_{Cr}$ (1%))	-	-	-	-	Lab 13	-	Lab 13
Classification (low)	*correct*	*correct*	*correct*	*correct*	*correct*	*correct*	*correct*
Outlier Lab ($G_1 > G_{Cr}$ (1%))	-	-	-	-	-	-	-
Double G_{Cr} (1%)	0,2016	0,2016	0,2016	0,2016	0,2016	0,2016	0,2016
Double G_{Cr} (5%)	0,2836	0,2836	0,2836	0,2836	0,2836	0,2836	0,2836
Double high $G_{largest}$	0,4192	0,6126	0,3737	0,4718	-	0,5407	0,2598
Double low $G_{smalest}$	0,6427	0,5671	0,6460	0,5159	0,8882	0,6507	0,7892
Classification (two largest)	*correct*	*correct*	*correct*	*correct*	-	*correct*	*Straggler*
Outlier Lab($G_{largest} < G_{Cr}$ (1%))	-	-	-	-	-	-	Lab 13
Classification (two smallest)	*correct*	*correct*	*correct*	*correct*	*correct*	*correct*	*correct*
Outlier Lab($G_{smalest} < G_{Cr}$ (1%))	-	-	-	-	-	-	-

Single: test for one outlying observation; Double: test for two outlying observations
n. a. – not applied

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LABORATÓRIO NACIONAL DE ENGENHARIA CIVIL

Sealing quality assessment by measurement of admittance (EN ISO 2931)

Results of precision analysis

Level	A	B	C	D	E	F	G
Number of replicates *n*	2	2	2	2	2	2	2
Valid laboratories *p*	12	13	13	13	11	13	13
General mean *m* / Y	**10,54**	**11,58**	**12,85**	**15,23**	**23,41**	**8,50**	**10,19**
Repeatability variance s_r^2	1,042	2,500	1,308	3,538	5,682	0,423	3,192
Between-lab variance s_L^2	2,000	2,410	8,946	7,423	48,200	2,163	7,926
Reproducibility variance s_R^2	3,042	4,910	10,253	10,962	53,882	2,587	11,119
Repeatability std. dev. s_r	**1,02**	**1,58**	**1,14**	**1,88**	**2,38**	**0,65**	**1,79**
Reproducibility std. dev. s_R	**1,74**	**2,22**	**3,20**	**3,31**	**7,34**	**1,61**	**3,33**
Repeatability COV (s_r/m)	9,7 %	13,7 %	8,9 %	12,4 %	10,2 %	7,7 %	17,5 %
Reproducibility COV (s_R/m)	16,5 %	19,1 %	24,9 %	21,7 %	31,4 %	18,9 %	32,7 %
Number of outliers	1	1	1	1	2	0	1
Number of excluded outliers	1	0	0	0	2	0	0
Outlier type	M*k*, C	M*k*	M*k*	M*k*	M*h*[1], G(I)[2], M*k*[3], C[4]	-	M*h*
Outlier laboratories	Lab 4	Lab 4	Lab 12	Lab 11	Lab 7[3,4] Lab 13[1,2,4]	-	Lab 13

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

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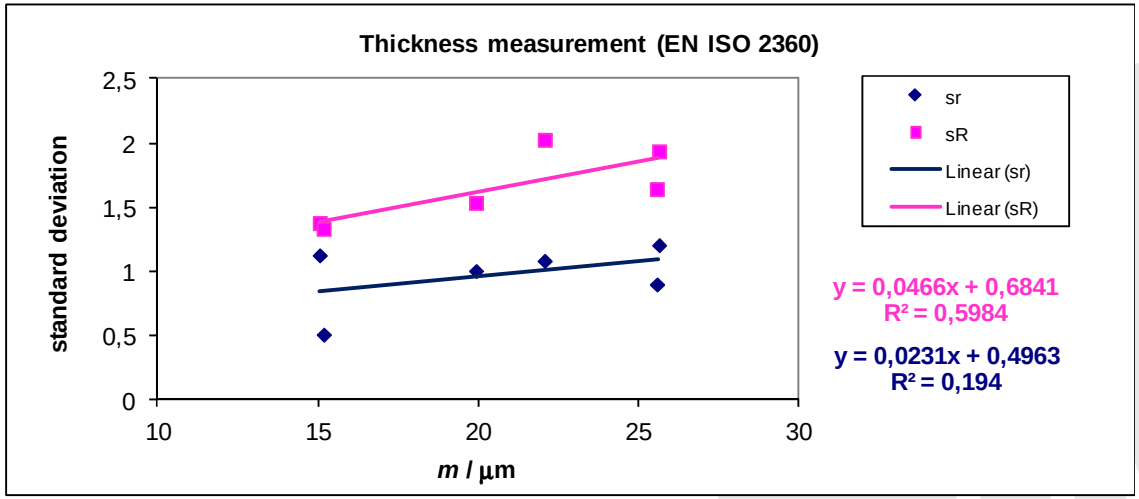
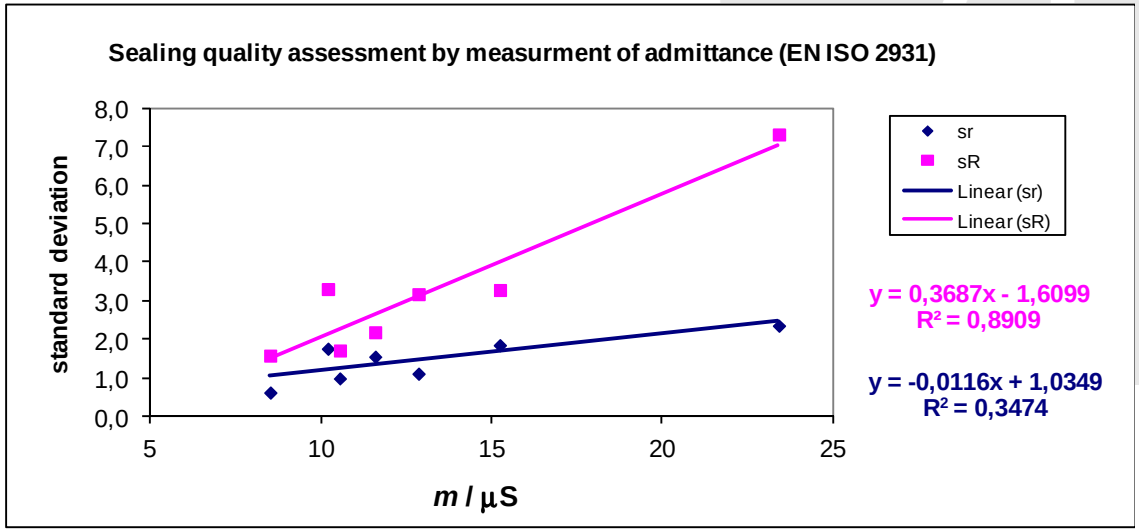
Dependency analysis of

precision (repeatability and reproducibility) with the mean





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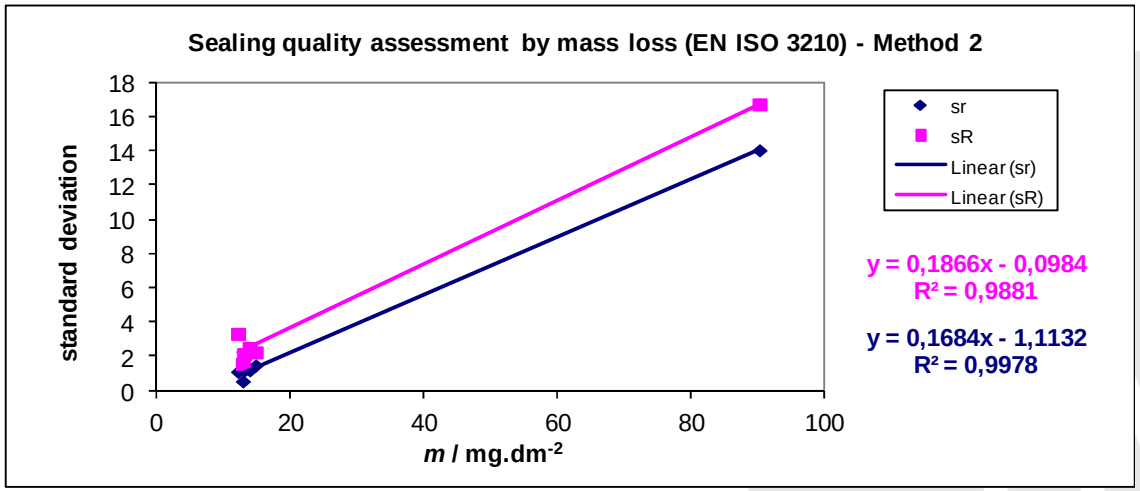
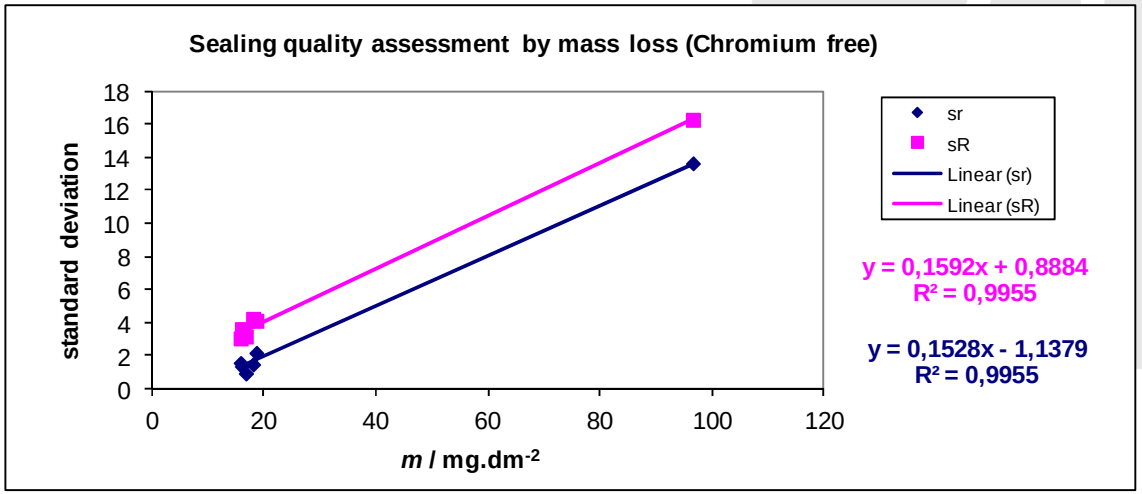
Dependency analysis of

precision (repeatability and reproducibility) with the mean





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Precision analysis results 2011

LABORATÓRIO NACIONAL DE ENGENHARIA CIVIL

EN ISO 2360 – Thickness measurement	Anodic coating type							Excluded data lab: anodic coating type
	A	B	C	D	E	F	G	
General mean *m* / µm	15,1	19,9	25,6	22,1	37,5	15,2	25,7	None
Repeatability std. dev. s_r	1,13	1,01	0,90	1,08	0,73	0,51	1,21	
Reproducibility std. dev. s_R	1,38	1,54	1,64	2,03	1,26	1,33	1,94	
Global repeatability std. dev.	$S_r = 1,2$							
Global reproducibility std. dev.	$S_R = 2,0$							

EN ISO 3210 - Sealing quality by mass loss	Anodic coating type							Excluded data lab: anodic coating type
	A	B	C	D	E	F	G	
General mean *m* / g.dm^{-2}	12,9	12,2	13,1	-	90,2	13,9	14,8	None
Repeatability std. dev. s_r	0,59	1,15	1,21	-	14,1	1,24	1,53	
Reproducibility std. dev. s_R	1,61	3,36	2,19	-	16,8	2,54	2,28	
Global repeatability std. dev.	$S_r = 0,1682\ m - 1,0969$ ($R^2 \approx 1$) or $S_r = 1,1$ (if $m < 30$ mg/dm^2)							
Global reproducibility std. dev.	$S_R = 0,1879\ m - 0,217$ ($R^2 \approx 1$) or $S_R = 2,3$ (if $m < 30$ mg/dm^2)							

Chromium free test - Sealing quality by mass loss	Anodic coating type							Excluded data lab: anodic coating type
	A	B	C	D	E	F	G	
General mean *m* / g.dm^{-2}	16,0	15,8	16,7	-	96,6	18,0	18,6	Lab 11: A Lab 3: B
Repeatability std. dev. s_r	1,32	1,53	0,90	-	13,6	1,44	2,14	
Reproducibility std. dev. s_R	3,59	3,02	3,13	-	16,3	4,21	4,08	
Global repeatability std. dev.	$S_r = 0,1528\ m - 1,1379$ ($R^2 \approx 1$) or $S_r = 1,5$ (if $m < 30$ mg/dm^2)							
Global reproducibility std. dev.	$S_R = 0,1592\ m - 0,8884$ ($R^2 \approx 1$) or $S_R = 3,6$ (if $m < 30$ mg/dm^2)							

EN 2931 - Sealing quality by admittance	Anodic coating type							Excluded data lab: anodic coating type
	A	B	C	D	E	F	G	
General mean *m* / Y	10,5	11,6	12,9	15,2	23,4	8,5	10,2	Lab 4: A Lab 7: E Lab 13: E
Repeatability std. dev. s_r	1,02	1,58	1,14	1,88	2,38	0,65	1,79	
Reproducibility std. dev. s_R	1,74	2,22	3,20	3,31	7,34	1,61	3,33	
Global repeatability std. dev.	$S_r = 1,5$							
Global reproducibility std. dev.	$S_R = 0,3687\ m - 1,6099$ ($R^2 \approx 0,9$) or $S_R = 2,6$ (if $m < 20$ µS)							

2011
Thickness
Repeatability=1
Reproducibility=2

(standard :
1 µm (until 10 µm or 10%)

Sealing/mass loss
Repeatability=1
Reproducibility=2

Sealing/mass loss Cr free
Repeatability=2
Reproducibility=4

Admittance
Repeatability=2
Reproducibility=3

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Precision analysis results 2009

EN ISO 2360 – Thickness measurement	Coating type				Excluded data lab: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	Coating type				Excluded data lab: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	Coating type				Excluded data lab: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.	$S_R = 0{,}3714\ m - 1{,}8233$ ($R^2 \approx 1$)				

2009

Thickness

Repeatability=0,87

Reproducibility=1,82

(standard :
1 µm (until 10 µm or 10%)

Sealing/mass loss

Repeatability=1

Reproducibility=4

admittance

Repeatability<0.5

Reproducibility=1

If the sealing is bad the repeat and reprod. In the 2 methods are very high and these statistical parameters must be calculated using the equations

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Precision analysis results 2006

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EN ISO 2360 – Thickness measurement	Coating type				Excluded data lab:coating type
	P	IP	I	A	
General mean *m* / µm	18,6	19,5	27,8	21,4	None
Repeatability std. dev. s_r	0,58	0,57	0,56	0,54	
Reproducibility std. dev. s_R	0,92	1,12	1,51	0,89	
Global repeatability std. dev.	0,56				
Global reproducibility std. dev.	S_R = 0,051 *m* (R^2=0,7338)				

EN 12373-7 - Sealing quality assessment by mass loss	Coating type				Excluded data lab:coating type
	P	IP	I	A	
General mean *m* / g.dm^{-2}	15,9	477,5	17,9	13,2	Lab 10:P Lab 8:I Lab 8:A
Repeatability std. dev. s_r	0,65	11,9	0,68	0,66	
Reproducibility std. dev. s_R	2,96	16,8	3,19	2,81	
Global repeatability std. dev.	S_R = 0,0234 *m* + 0,6738 (R^2≈1)				
Global reproducibility std. dev.	S_R = 0,0294 *m* + 2,7398 (R^2≈1)				

EN 12373-5 - Sealing quality assess. by admittance	Coating type				Excluded data lab:coating type
	P	IP	I	A	
General mean *m* / Y	8,98	n.a	15,29	9,88	Lab 12:P Lab 8:A
Repeatability std. dev. s_r	0,34	n.a	0,86	0,34	
Reproducibility std. dev. s_R	1,72	n.a	3,12	1,38	
Global repeatability std. dev.	0,51				
Global reproducibility std. dev.	S_R = 0,1932 *m* (R^2=0,8218)				

2006

Thickness
Repeatability=0.56
Reproducibility=1.51

(standard :
1 µm (until 10 µm or 10%

Sealing/mass loss < 30
Repeatability=1
Reproducibility=3

Sealing/admitance
Repeatability=0.51
Reproducibility=3.12

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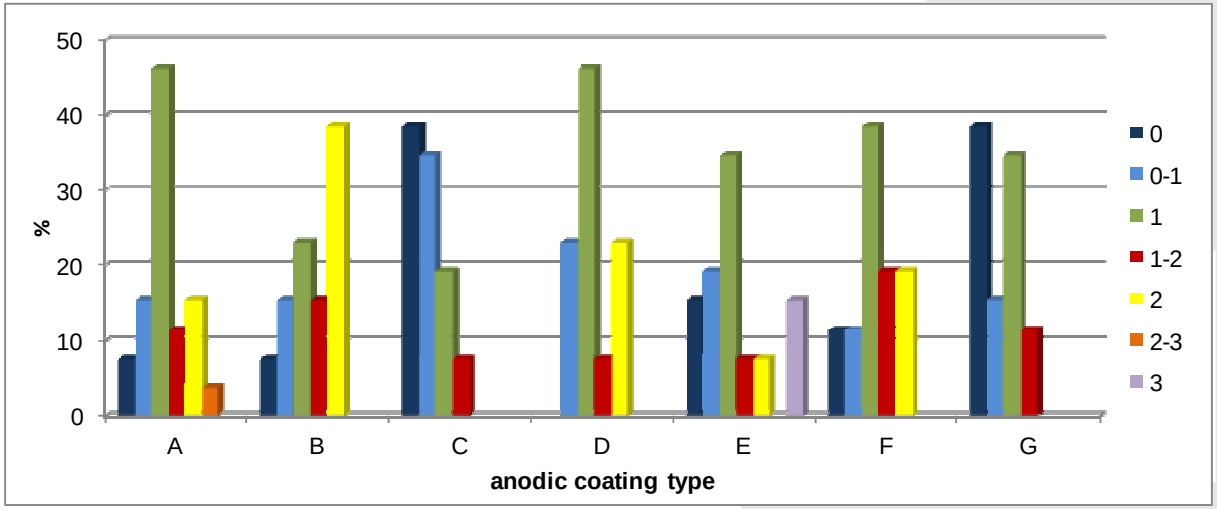
LABORATÓRIO NACIONAL DE ENGENHARIA CIVIL

Estimation of loss of absorptive power of anodic oxidation coatings after sealing by dye spot test (EN ISO 2143)

Analysis of qualitative results

Frequency distribution of the results



	Dye absorption rating				Labs with results less than 5% frequent
Anodic coating type	Mode	Median	Range of results more than 10% frequent	Labs with results less than 10% frequent	
A	1	1	0-1 to 2	Lab 7, Lab 13	Lab 13 (one result)
B	2	1-2	0-1 to 2	Lab 7	None
C	0	0-1	0 to 1	Lab 13	None
D	1	1	0-1 to 1 and 2	Lab 11	None
E	1	1	0 to 1 and 3	Lab 4, Lab 5	None
F	1	1	0 to 2	None	None
G	0	0-1	0 to 1-2	None	None

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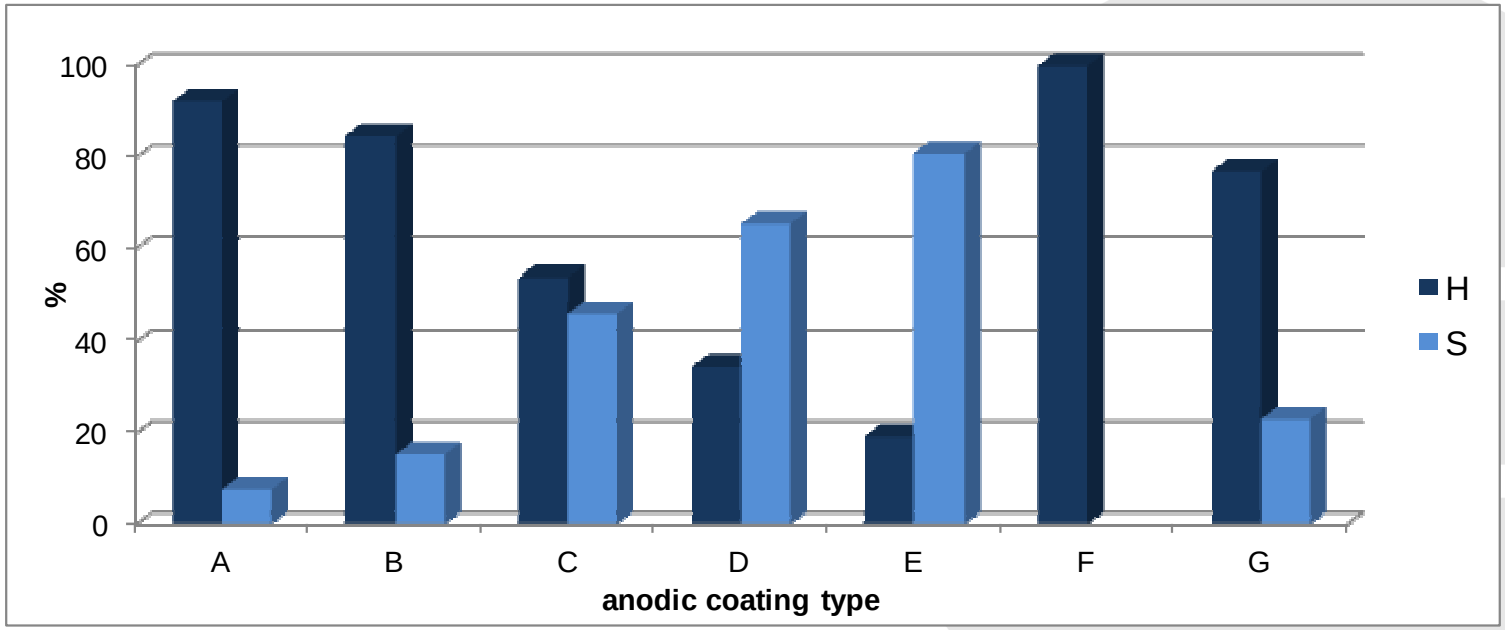
LABORATÓRIO NACIONAL DE ENGENHARIA CIVIL

Anodic oxidation coatings and its alloys. Part 18. Determination of surface abrasion resistance. (BS 6161-18)

Analysis of qualitative results

Frequency distribution of the results



	Anodic coating type						
Results	A	B	C	D	E	F	G
Hard (%)	**92**	**85**	54	35	19	**100**	**77**
Soft (%)	8	15	46	65	**81**	0	23
Laboratories with results 10% or less frequent	Lab 8	none	none	none	none	none	none

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2006	2009	2011
Thickness Repeatability = 0.56 Reproducibility =1.51 (standard : 1 µm (until 10 µm or 10 %	**Thickness** Repeatability = 0,87 Reproducibility = 1,82 (standard : 1 µm (until 10 µm or 10 %	**Thickness** Repeatability = 1 Reproducibility = 2 (standard : 1 µm (until 10 µm or 10 %)
Sealing/mass loss Repeatability = 1 Reproducibility = 3	**Sealing/mass loss** Repeatability = 1 Reproducibility = 4	**Sealing/mass loss** Repeatability = 1 Reproducibility = 2 **Sealing/mass loss Cr free** Repeatability = 2 Reproducibility = 4
Sealing/admittance Repeatability = 0.51 Reproducibility = 3.12	**Sealing/admittance** Repeatability < 0.5 Reproducibility = 1	**Sealing/admittance** Repeatability = 2 Reproducibility = 3

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