

# 2013 QUALANOD INTER-LABORATORY TEST OF ANODIZED ALUMINIUM TESTING METHODS

## ***STATISTICAL ANALYSIS - REPEATIBILITY AND REPRODUCIBILITY DETERMINATION***

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## 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	
VGONTZAS Manolis	GREECE	AAG Quality – EKANAL for Aluminium Ass. Of Greece	AAG	No results
JUHASZ, Péter	HUNGARY	EMI – Institut für Qualitätskontrolle	EMI	3)
BOI, Riccardo	ITALY	QUALITAL	QUALITAL	
BRAKENHOFF, Rob	NETHERLANDS	COT bv – Centrum voor Onderzoek en Technisch Advies bv	COT	2)
TOMASSI, Piotr	POLAND	IMP – Instytut Mechaniki Precyzyjnej	IMP	
MOZARYN, Teresa	POLAND	ITB – Instytut Techniki Budowlanej	ITB	
SALTA, Manuela	PORTUGAL	LNEC – Laboratório Nacional de Engenharia Civil	LNEC	
PAZ, Angel	SPAIN	QUALESPAÑA - Ministerio de Vivienda Subdirección General de Innovación y Calidad de la Edificación	QUALESPAÑA	1), 3)
WERNER, René	SWITZERLAND	EMPA, Abtl. Korrosion	EMPA	
AVCI, Beyazit	TURKEY	TSI – Turkish Standards Institution	TSI	4)
BARRON, Lynda	UK & IRELAND	Bodycote Materials Testing	EXOVA	No results




1) No results for admittance; 2) No results for dye-spot ; 3) No results for abrasions; 4) No results for abrasion II



## 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**.*

## General characteristics of the test specimens

Anodic Coating	Profile type	Colour	Thickness class	Anodizing Temperature	Sealing	
					Process	Time/ $\mu\text{m}$
NB		Natural	20 $\mu\text{m}$	18 °C	Hot water sealing (T > 96 °C)	3 min/ $\mu\text{m}$
NE		Natural	25 $\mu\text{m}$	25 °C		2 min/ $\mu\text{m}$
BM		Bronze	15 $\mu\text{m}$	21 °C		0,6 min/ $\mu\text{m}$
BC		Bronze	15 $\mu\text{m}$	21 °C		5 min/ $\mu\text{m}$
FM		Natural	15 $\mu\text{m}$	18 °C	Cold sealing (T = 27 °C)	0,4 min/ $\mu\text{m}$
FC		Natural	15 $\mu\text{m}$	18 °C		0,8 min/ $\mu\text{m}$
XN		Natural	15 $\mu\text{m}$	17 °C	Hot water (T > 96 °C)	4 min/ $\mu\text{m}$

## Anodizing and sealing conditions used for the production of test specimens

Anodic coating type	Anodizing		Hot water sealing	
<b>NB</b>	<i>Free H<sub>2</sub>SO<sub>4</sub></i>	180,3 g/l	<i>Demineralised water</i>	
	<i>Al content</i>	12,6 g/l	<i>pH</i>	5,7
	<i>Temperature</i>	18 °C	<i>Additive</i>	P3 Almeco Seal
	<i>Current density</i>	1,35 A/dm <sup>2</sup>	<i>Time -</i>	3 min/μm
<b>NE</b>	<i>Free H<sub>2</sub>SO<sub>4</sub></i>	180,3 g/l	<i>Demineralised water</i>	
	<i>Al content</i>	12,6 g/l	<i>pH</i>	5,7
	<i>Temperature</i>	25 °C	<i>Additive</i>	P3 Almeco Seal
	<i>Current density</i>	1,35 A/dm <sup>2</sup>	<i>Time -</i>	3 min/μm
<b>BM</b>	<i>Free H<sub>2</sub>SO<sub>4</sub></i>	183,3 g/l	<i>Demineralised water</i>	
	<i>Al content</i>	13,1 g/l	<i>pH</i>	5,6
	<i>Temperature</i>	21 °C	<i>Additive</i>	P3 Almeco Seal
	<i>Current density</i>	1,28 A/dm <sup>2</sup>	<i>Time -</i>	5 min
<b>BC</b>	<i>Free H<sub>2</sub>SO<sub>4</sub></i>	183,3 g/l	<i>Demineralised water</i>	
	<i>Al content</i>	13,1 g/l	<i>pH</i>	5,6
	<i>Temperature</i>	21 °C	<i>Additive</i>	P3 Almeco Seal
	<i>Current density</i>	1,28 A/dm <sup>2</sup>	<i>Time -</i>	90 min

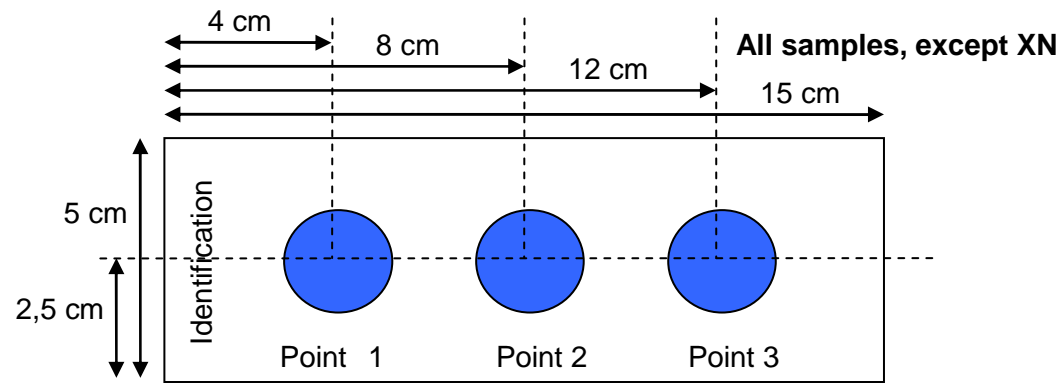
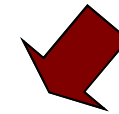
## Anodizing and sealing conditions used for the production of test specimens (cont)

Anodic coating type	Anodizing		Hot water sealing	
<b>FM</b>	<i>Free H<sub>2</sub>SO<sub>4</sub></i>	199 g/l	<i>Demineralised water</i>	
	<i>Al content</i>	12,6 g/l	<i>pH</i>	6,0
	<i>Temperature</i>	18 °C	<i>Additive</i>	Alfiseal 986 / 987
	<i>Current density</i>	1,35 A/dm <sup>2</sup>	<i>Time -</i>	5 min
<b>FC</b>	<i>Free H<sub>2</sub>SO<sub>4</sub></i>	199 g/l	<i>Demineralised water</i>	
	<i>Al content</i>	12,6 g/l	<i>pH</i>	6,0
	<i>Temperature</i>	18 °C	<i>Additive</i>	Alfiseal 986 / 987
	<i>Current density</i>	1,35 A/dm <sup>2</sup>	<i>Time -</i>	14 min
<b>XC</b>	<i>Free H<sub>2</sub>SO<sub>4</sub></i>	175,4 g/l	<i>Demineralised water</i>	
	<i>Al content</i>	11,7 g/l	<i>pH</i>	5,7
	<i>Temperature</i>	17 °C	<i>Additive</i>	GARDO TP 10779
	<i>Current density</i>	1,12 A/dm <sup>2</sup>	<i>Time -</i>	80 min

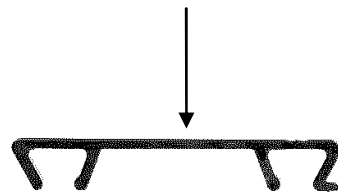
## Instructions

- Laboratory code number
- Measuring points

Scheme of the testing zones for thickness measurement

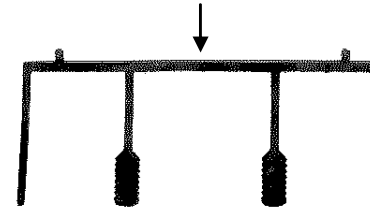


Surface for tests



NB, NE, BM and BC samples

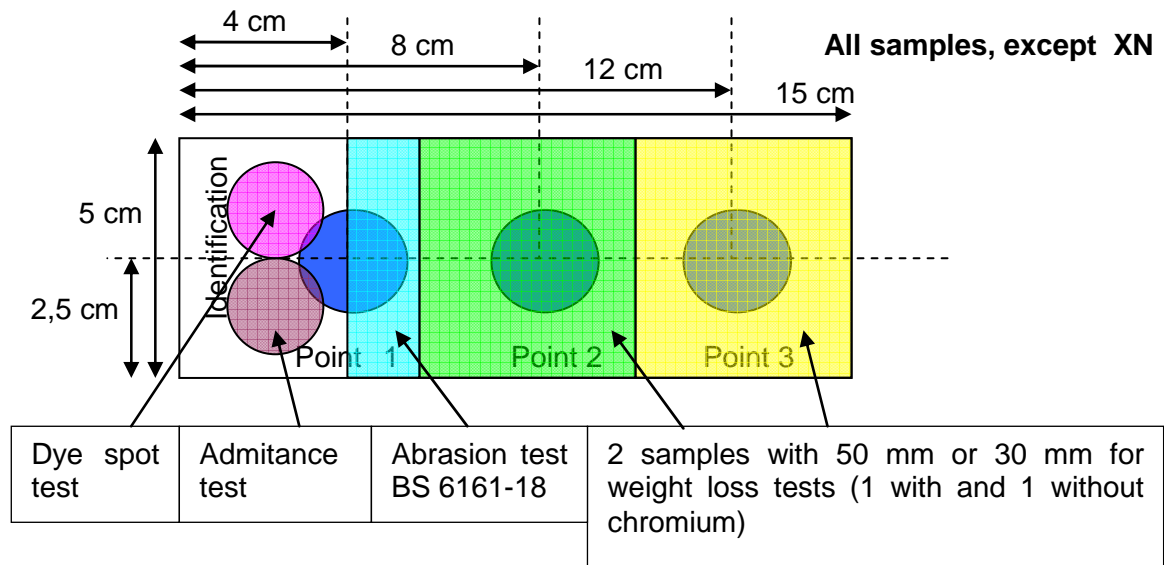
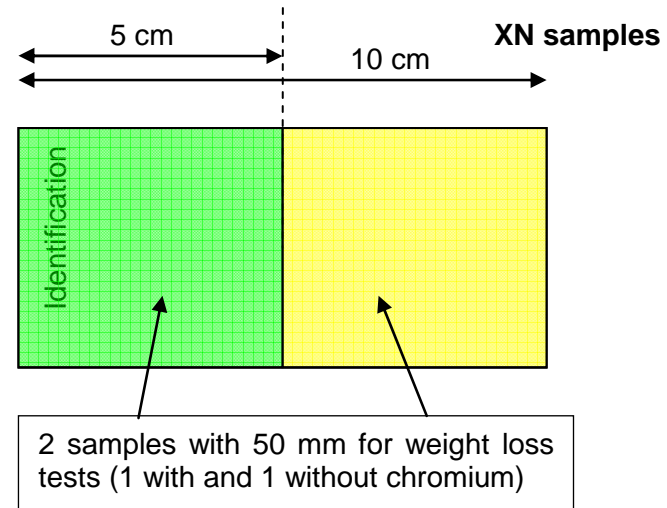
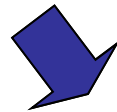
Surface for tests



FM and FC samples

# Instructions

Scheme of testing zones for sealing quality assessment





# EXCEL Worksheets for data registration

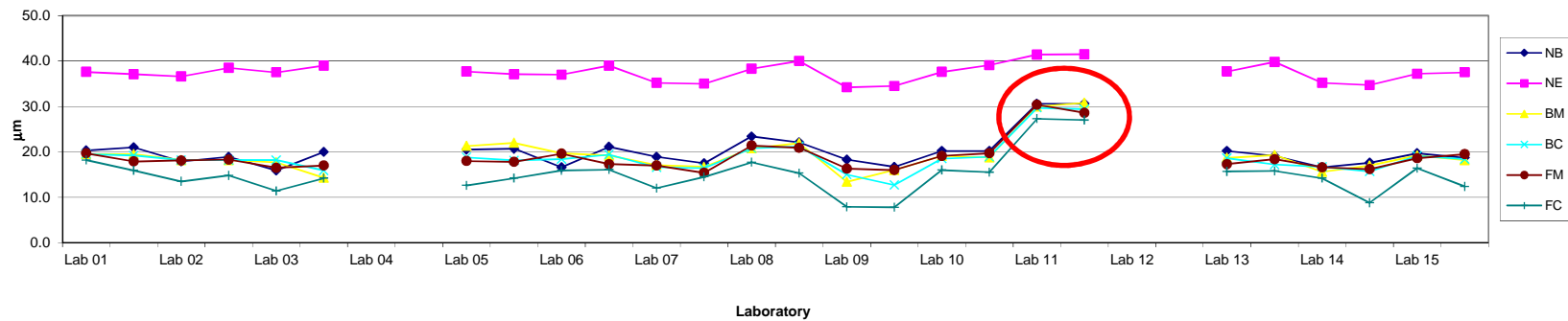
Protocol for NB sample									
Laboratory identification				Test conditions					
Test laboratory				Temperature					
Sample type: NB				Date of the test					
Tested by				Zero base					
Date of report				Calibration standard 1					
				Calibration standard 2					
				Calibration standard 3					
Test results									
Sample No. NB??									
Thickness results (EN ISO 2360)				Admittance results (EN ISO 2931)					
Sample No.	Front side			Sample No.	Measurement	Thickness	Temperature	Admittance	
NB??	1	2	3	NB??	Ymes	( $\mu\text{m}$ )	( $^{\circ}\text{C}$ )	Y20	
measure 1	21.6				150	24.8	21.2	20	
measure 2	22.0								
measure 3	21.3								
Average thickness	21.8								
Dye spot results (EN ISO 2143)				Surface abrasion resistance results (BS 6161-18)					
Dye spot	0-1	Color	Red	Method I	H	Harder or softer			
Dye spot	0-1	Color	Blue	Method II	H	Harder or softer			
REMARKS: Length $\pm$ 50 mm Perimeter: 0.155 m <sup>2</sup> /m									
Weight loss results (EN ISO 3210: method 2)				Weight loss results (chromic free test)					
Sample No.	Length L	Weight <sub>0</sub>	Weight <sub>1</sub>	Weight loss	Sample No.	Length L	Weight <sub>0</sub>	Weight <sub>1</sub>	Weight loss
NB??	(mm)	(g)	(g)	(mg/dm <sup>2</sup> )	NB??	(mm)	(g)	(g)	(mg/dm <sup>2</sup> )
measure 1	50.29	9.4863	9.4444	-	measure 1	50.29	9.4863	9.4444	-
measure 2	50.09	-	-	-	measure 2	50.09	-	-	-
measure 3	50.83	-	-	-	measure 3	50.83	-	-	-
Average	50.34	9.4863	9.4444	16.26	Average	50.34	9.4863	9.4444	16.26
REMARKS: Length $\pm$ 50 mm Perimeter: 0.155 m <sup>2</sup> /m									
Thickness results (EN ISO 2360)				Admittance results (EN ISO 2931)					
Sample No.	Front side			Sample No.	Measurement	Thickness	Temperature	Admittance	
NB??	1	2	3	NB??	Ymes	( $\mu\text{m}$ )	( $^{\circ}\text{C}$ )	Y20	
measure 1									
measure 2									
measure 3									
Average thickness									
Dye spot results (EN ISO 2143)				Surface abrasion resistance results (BS 6161-18)					
Dye spot		Color	Red	Method I		Harder or softer			
Dye spot		Color	Blue	Method II		Harder or softer			
REMARKS: Length $\pm$ 50 mm Perimeter: 0.155 m <sup>2</sup> /m									
Weight loss results (EN ISO 3210: method 2)				Weight loss results (chromic free test)					
Sample No.	Length L	Weight <sub>0</sub>	Weight <sub>1</sub>	Weight loss	Sample No.	Length L	Weight <sub>0</sub>	Weight <sub>1</sub>	Weight loss
NB??	(mm)	(g)	(g)	(mg/dm <sup>2</sup> )	NB??	(mm)	(g)	(g)	(mg/dm <sup>2</sup> )
measure 1					measure 1				
measure 2					measure 2				
measure 3					measure 3				
Average					Average				

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

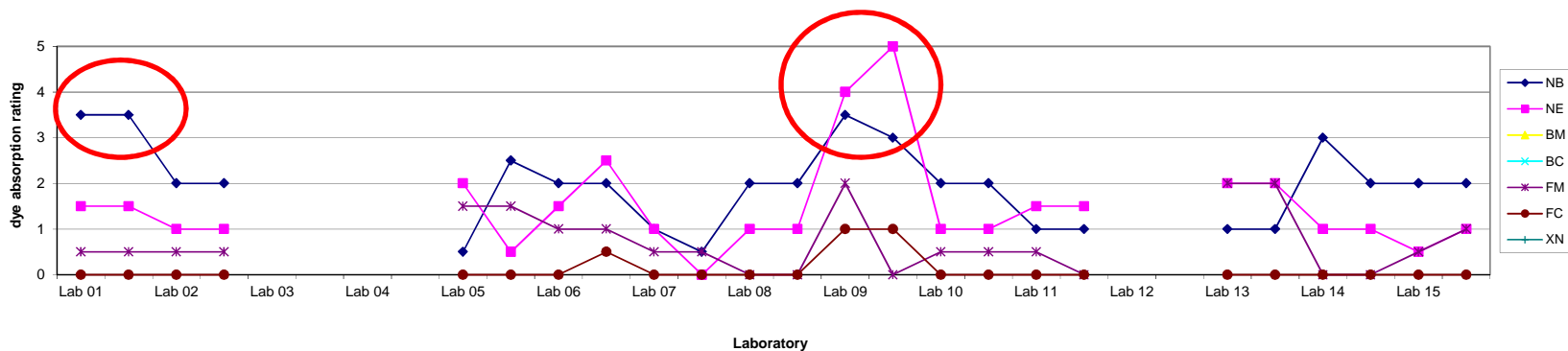


# Results reported

## Test results of method EN ISO 2360

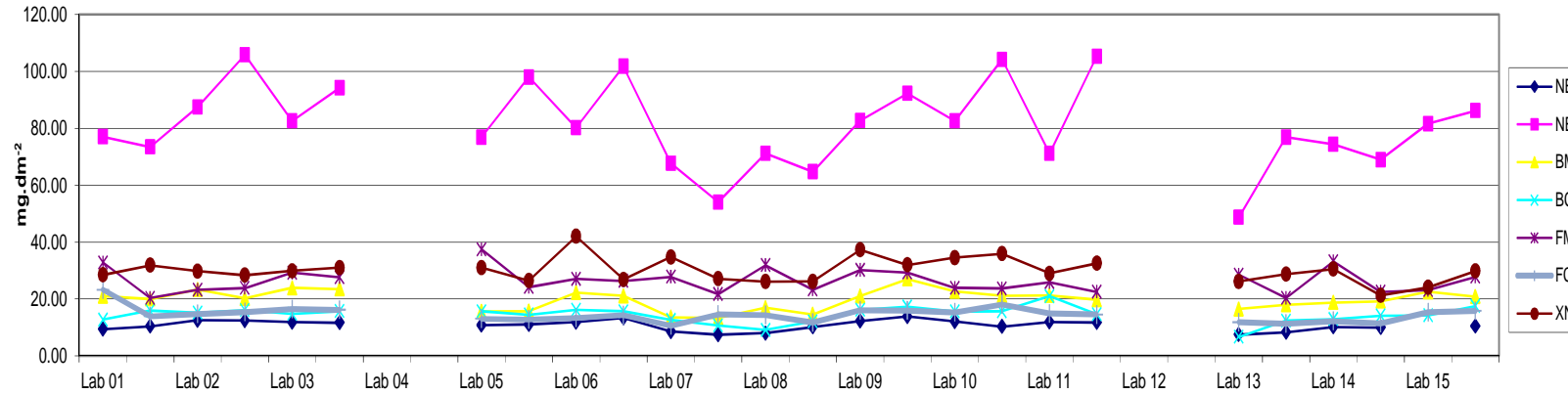


## Test results of method EN ISO 2143

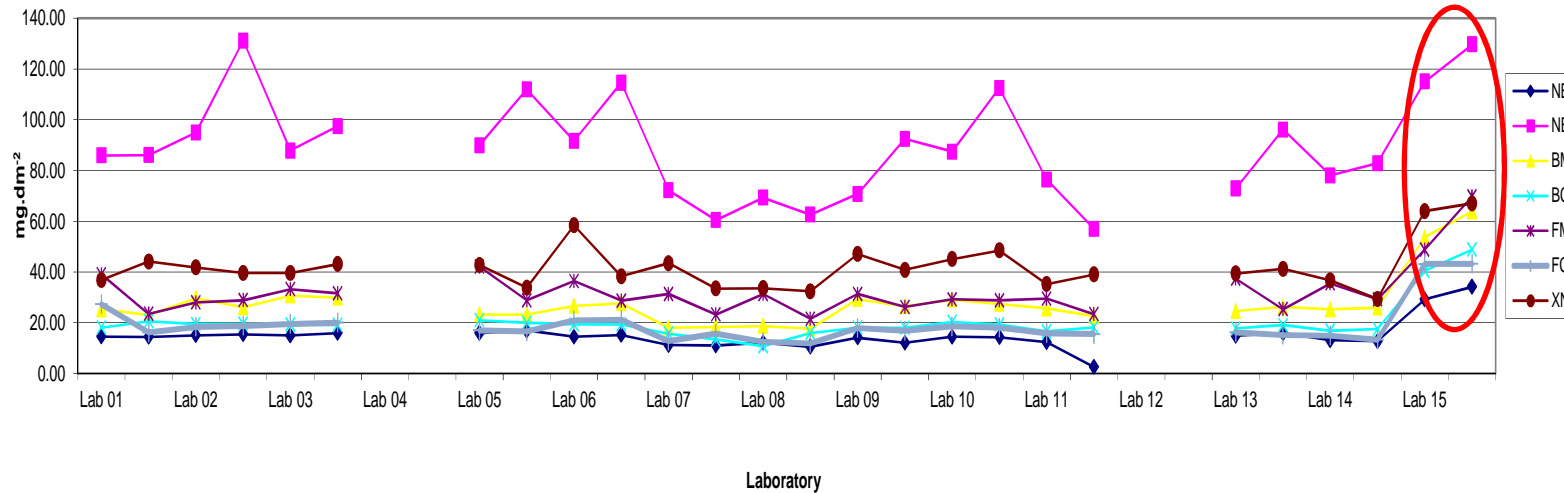


# Results reported

## Test results of method EN ISO 3210

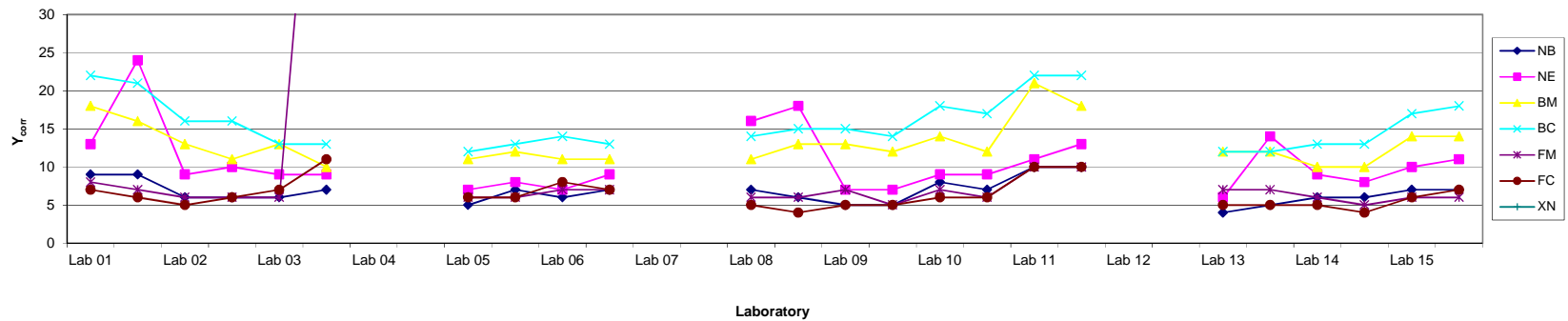
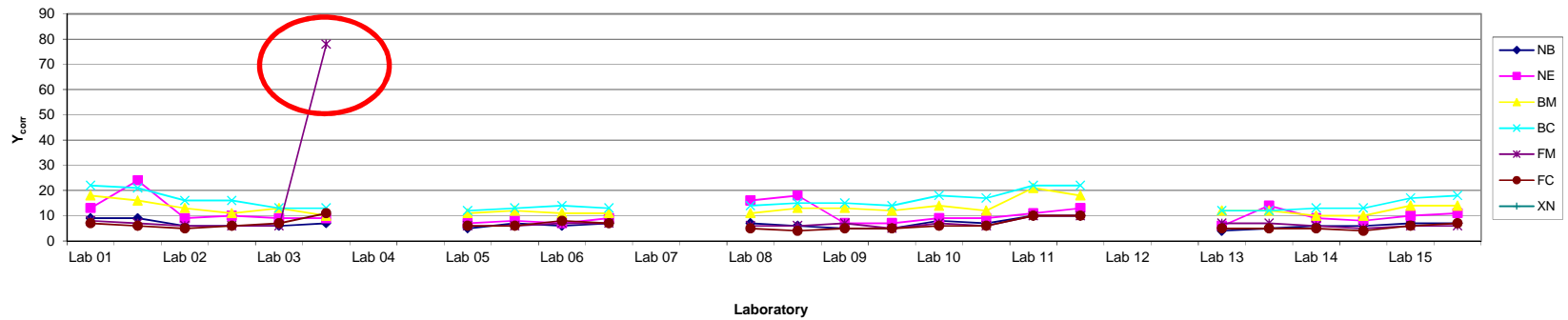


## Test results of method Chromium free test



# Results reported

## Test results of method EN ISO 2931



# Precision analysis according to ISO 5725-2

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

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

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

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

$$G_p = (x_p - \bar{x})/s$$

$$G_1 = (\bar{x} - x_1)/s$$

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

$$G = s_{1,2}^2 / s_0^2$$

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

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

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

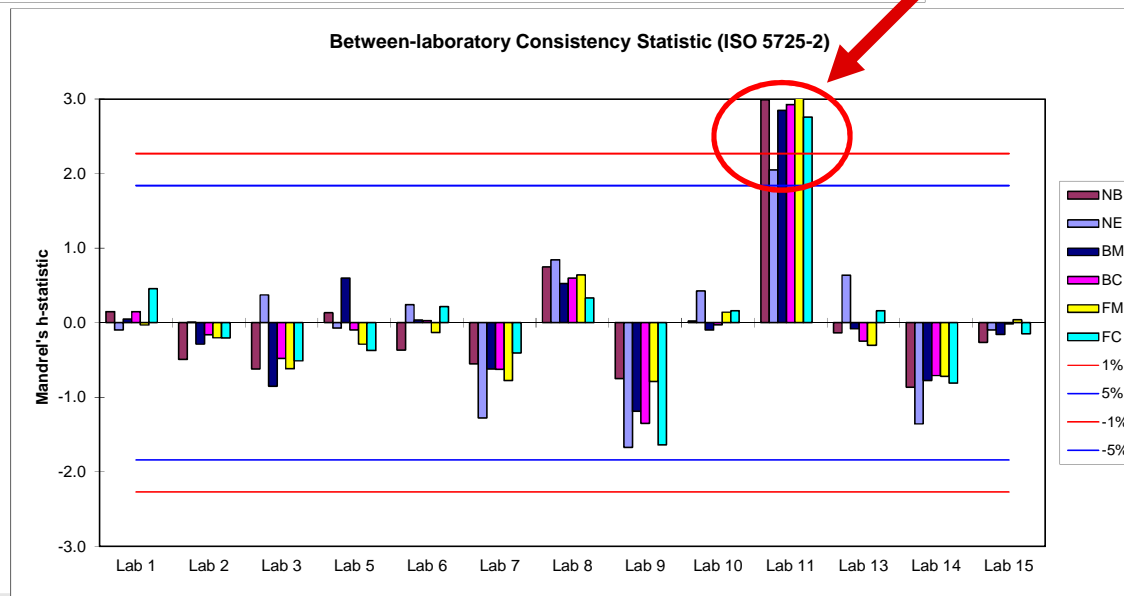
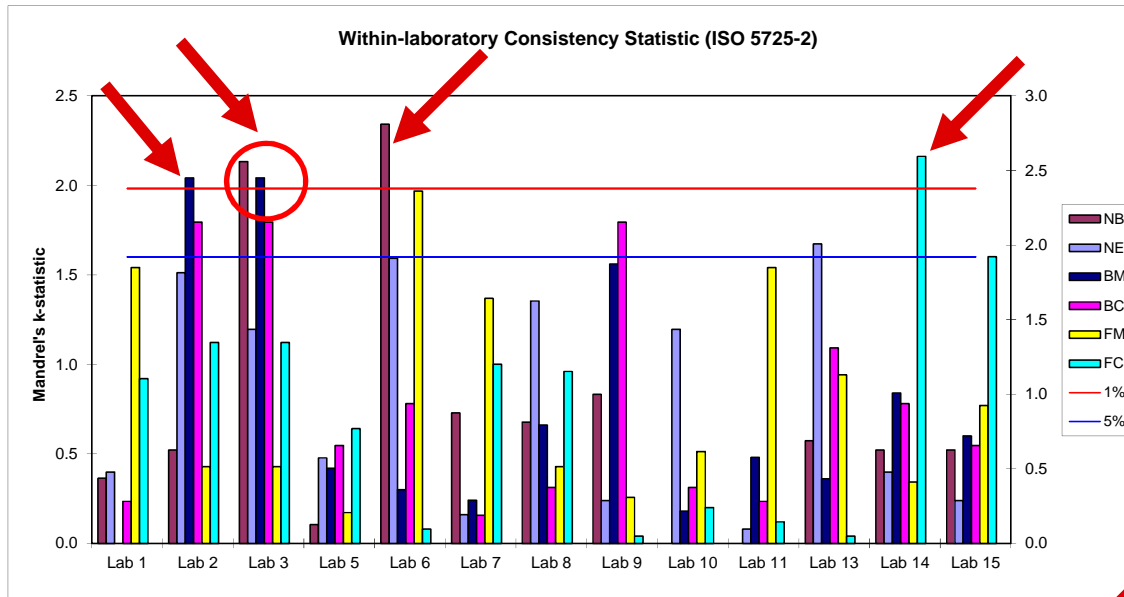
*Repeatability variance*

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

*Reproducibility variance*

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

# Thickness measurement (EN ISO 2360)



# Thickness measurement (EN ISO 2360)

## Laboratories outside critical value lines of Mandel's statistics

Level	NB	NE	BM	BC	FM	FC
Mandel's <i>k</i> -plot	Lab 3, 6	Lab 13	Lab 2, 3	Lab 2, 3, 9	Lab 6	Lab 14
Classification	<b>Outlier</b>	<i>Straggler</i>	<b>Outlier</b>	<i>Straggler</i>	<i>Straggler</i>	<b>Outlier</b>
Mandel's <i>h</i> -plot	Lab 11	Lab 11	Lab 11	Lab 11	Lab 11	Lab 11
Classification	<b>Outlier</b>	<i>Straggler</i>	<b>Outlier</b>	<b>Outlier</b>	<b>Outlier</b>	<b>Outlier</b>

## Cochran's test results

Level	NB	NE	BM	BC	FM	FC
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 $C$	0,422	0,215	0,321	0,248	0,298	0,360
Classification	<i>Correct</i>	<i>Correct</i>	<i>Correct</i>	<i>Correct</i>	<i>Correct</i>	<i>Correct</i>
Straggler Lab ( $C > C_{Cr(1\%)}$ )	-	-	-	-	-	-
Outlier Lab ( $C > C_{Cr(1\%)}$ )	-	-	-	-	-	-

# Thickness measurement (EN ISO 2360)

## Grubb's test results

Level	NB	NE	BM	BC	FM	FC
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 $G_p$	2,989	2,051	2,850	2,929	3,034	2,759
Single low $G_1$	0,864	1,672	1,186	1,349	0,790	1,639
Classification (low)	<b>Outlier</b>	<i>Correct</i>	<b>Outlier</b>	<b>Outlier</b>	<b>Outlier</b>	<b>Outlier</b>
Outlier Lab ( $G_p > G_{Cr}$ (1%))	Lab 11	-	Lab 11	Lab 11	Lab 11	Lab 11
Classification (low)	<i>Correct</i>	<i>Correct</i>	<i>Correct</i>	<i>Correct</i>	<i>Correct</i>	<i>Correct</i>
Outlier Lab ( $G_1 > G_{Cr}$ (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 $G_{largest}$	0,1027	0,5264	0,2032	0,1608	0,0959	0,2841
Double low $G_{smallest}$	0,8712	0,5440	0,7907	0,7745	0,8877	0,6765
Classification (two largest)	<i>Correct</i>	<i>Correct</i>	<i>Correct</i>	<i>Correct</i>	<i>Correct</i>	<i>Correct</i>
Outlier Lab ( $G_{largest} < G_{Cr}$ (1%))	-	-	-	-	-	-
Classification (two smallest)	<i>Correct</i>	<i>Correct</i>	<i>Correct</i>	<i>Correct</i>	<i>Correct</i>	<i>Correct</i>
Outlier Lab ( $G_{smallest} < G_{Cr}$ (1%))	-	-	-	-	-	-

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





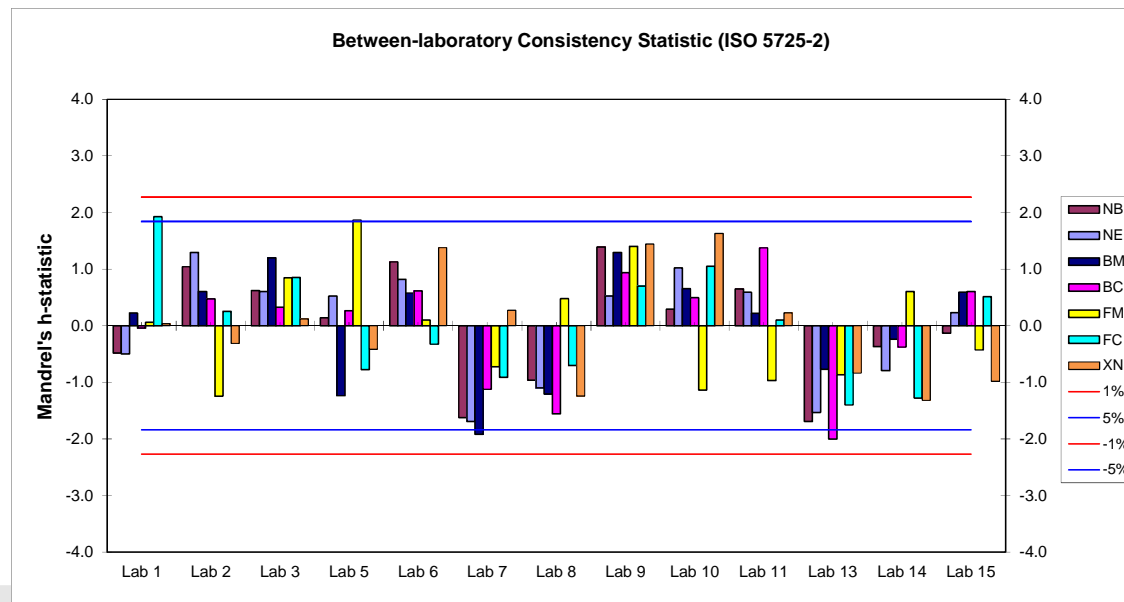
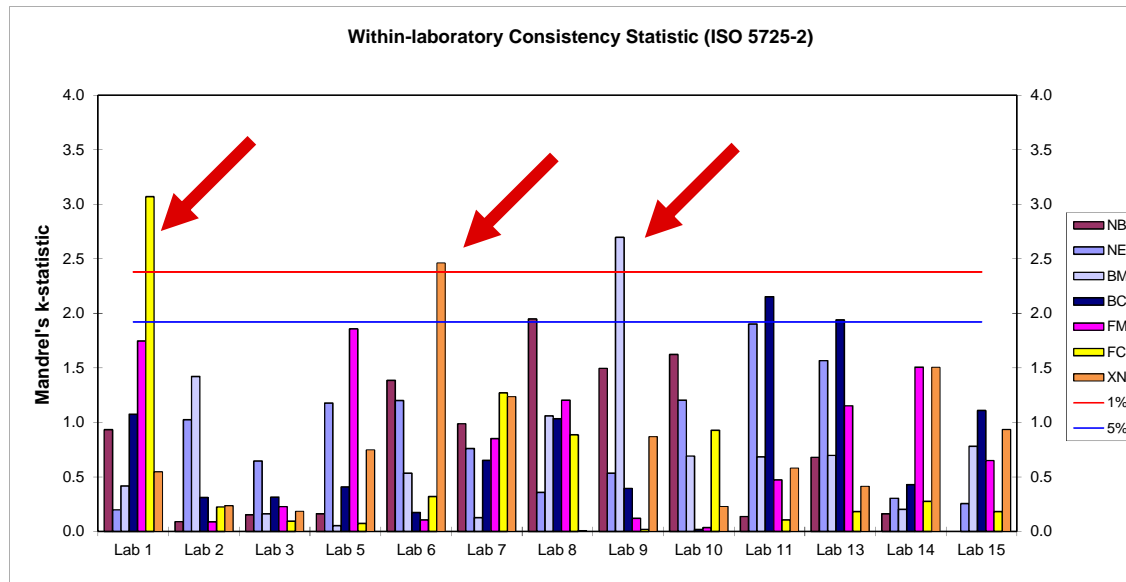
# Thickness measurement (EN ISO 2360)

## Results of precision analysis

Level	NB	NE	BM	BC	FM	FC
Number of replicates $n$	2	2	2	2	2	2
Valid laboratories $p$	12	13	12	12	12	12
<b>General mean <math>m / \mu\text{m}</math></b>	<b>19,25</b>	<b>37,54</b>	<b>18,39</b>	<b>17,90</b>	<b>18,03</b>	<b>14,03</b>
Repeatability variance $s_r^2$	2,000	0,854	0,993	0,665	0,596	3,119
Between-lab variance $s_L^2$	1,587	2,034	3,911	2,980	1,945	5,011
Reproducibility variance $s_R^2$	3,587	2,888	4,904	3,646	2,541	8,131
<b>Repeatability std. dev. <math>s_r</math></b>	<b>1,41</b>	<b>0,92</b>	<b>1,00</b>	<b>0,82</b>	<b>0,77</b>	<b>1,77</b>
<b>Reproducibility std. dev. <math>s_R</math></b>	<b>1,89</b>	<b>1,70</b>	<b>2,21</b>	<b>1,91</b>	<b>1,59</b>	<b>2,85</b>
Repeatability COV ( $s_r/m$ ), %	7,3	2,5	5,4	4,6	4,3	12,6
Reproducibility COV ( $s_R/m$ ), %	9,8	4,6	12,0	10,7	8,8	20,3
Number of outliers	3	0	3	1	1	2
Number of excluded outliers	1	0	1	1	1	1
Outlier type	<sup>1,2</sup> Mk, <sup>3</sup> Mh, <sup>3</sup> G(I)	-	<sup>1,2</sup> Mk, <sup>3</sup> Mh, <sup>3</sup> G(I)	Mh, G(I)	Mh, G(I)	<sup>1</sup> Mk, <sup>2</sup> Mh, <sup>2</sup> G(I)
Outlier laboratories	Lab 3 <sup>1</sup> Lab 6 <sup>2</sup> Lab 11 <sup>3</sup>	-	Lab 2 <sup>1</sup> Lab 3 <sup>2</sup> Lab 11 <sup>3</sup>	Lab 11	Lab 11	Lab 14 <sup>1</sup> Lab 11 <sup>2</sup>

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

# Sealing quality assessment by mass loss (EN ISO 3210)



## Sealing quality assessment by mass loss (EN ISO 3210)

### Laboratories outside critical value lines of **Mandel's** statistics

Level	NB	NE	BM	BC	FM	FC	XN
<i>Mandel's k</i> -plot	Lab 8	-	Lab 9	Lab 11, 13	-	Lab 1	Lab 6
Classification	<i>Straggler</i>	-	<b>Outlier</b>	<i>Straggler</i>	-	<b>Outlier</b>	<b>Outlier</b>
<i>Mandel's h</i> -plot	-	-	Lab 7	Lab 13	Lab 5	Lab 1	-
Classification	-	-	<i>Straggler</i>	<i>Straggler</i>	<i>Straggler</i>	<i>Straggler</i>	-

### **Cochran's** test results

Level	NB	NE	BM	BC	FM	FC	XN
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 $C$	0,292	0,278	0,559	0,356	0,265	0,724	0,466
Classification	<i>Correct</i>	<i>Correct</i>	<i>Straggler</i>	<i>Correct</i>	<i>Correct</i>	<b>Outlier</b>	<i>Correct</i>
Outlier Lab ( $C > C_{Cr (1\%)}$ )	-	-	Lab 9	-	-	Lab 1	-

# Sealing quality assessment by mass loss (EN ISO 3210)

## Grubb's test results

Level	NB	NE	BM	BC	FM	FC	XN
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 $G_p$	1,391	1,293	1,294	1,378	1,866	1,926	1,627
Single low $G_1$	1,690	1,692	1,919	1,999	1,243	1,400	1,320
Classification (high)	<i>Correct</i>	<i>Correct</i>	<i>Correct</i>	<i>Correct</i>	<i>Correct</i>	<i>Correct</i>	<i>Correct</i>
Outlier Lab ( $G_p > G_{Cr}$ (1%))	-	-	-	-	-	-	-
Classification (low)	<i>Correct</i>	<i>Correct</i>	<i>Correct</i>	<i>Correct</i>	<i>Correct</i>	<i>Correct</i>	<i>Correct</i>
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 $G_{largest}$	0,6847	0,7330	0,6933	0,7277	0,4643	0,5320	0,5344
Double low $G_{smallest}$	0,4591	0,4873	0,4900	0,3691	0,7206	0,6463	0,6761
Classification (two largest)	<i>Correct</i>	<i>Correct</i>	<i>Correct</i>	<i>Correct</i>	<i>Correct</i>	<i>Correct</i>	<i>Correct</i>
Outlier Lab ( $G_{largest} < G_{Cr}$ (1%))	-	-	-	-	-	-	-
Classification (two smallest)	<i>Correct</i>	<i>Correct</i>	<i>Correct</i>	<i>Correct</i>	<i>Correct</i>	<i>Correct</i>	<i>Correct</i>
Outlier Lab ( $G_{smallest} < G_{Cr}$ (1%))	-	-	-	-	-	-	-

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



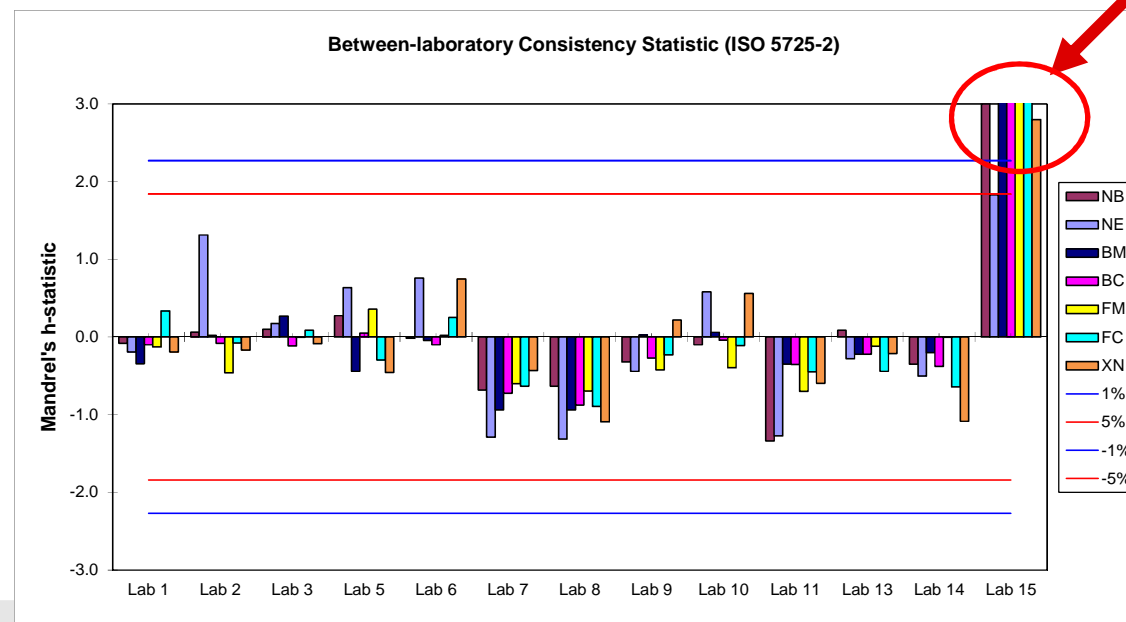
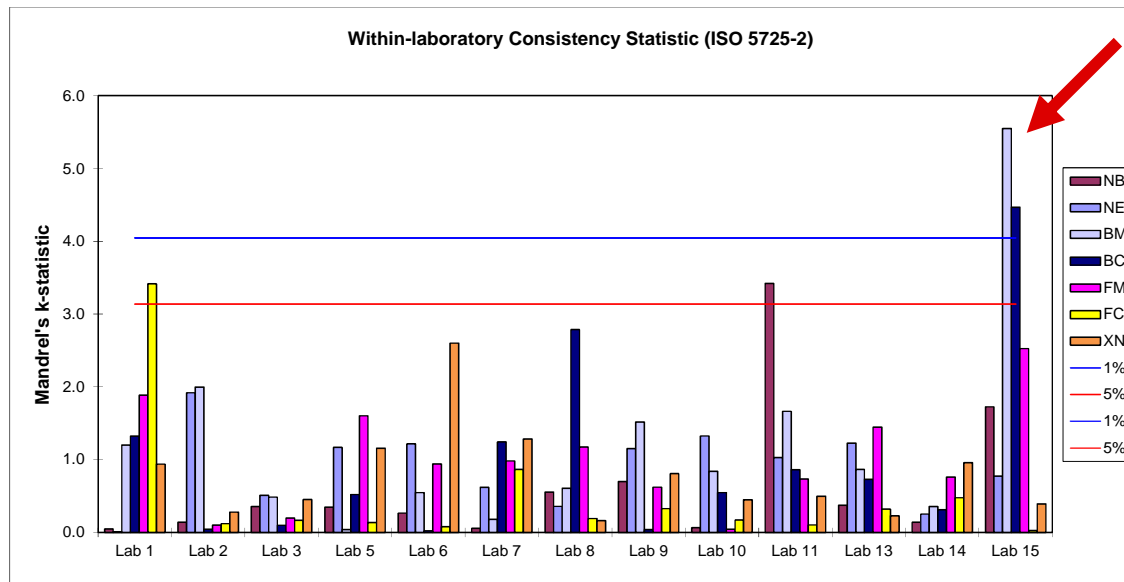
## Sealing quality assessment by mass loss (EN ISO 3210)

### Results of precision analysis

Level	NB	NE	BM	BC	FM	FC	XN
Number of replicates $n$	2	2	2	2	2	2	2
Valid laboratories $p$	13	13	13	13	13	12	13
<b>General mean <math>m</math> / mg.dm<sup>-2</sup></b>	<b>10,72</b>	<b>81,15</b>	<b>19,77</b>	<b>14,45</b>	<b>26,47</b>	<b>14,19</b>	<b>30,08</b>
Repeatability variance $s_r^2$	0,609	161,480	2,476	4,301	25,493	1,377	18,901
Between-lab variance $s_L^2$	2,490	62,775	9,585	3,849	0	2,502	0,605
Reproducibility variance $s_R^2$	3,099	224,255	12,061	8,150	25,493	3,879	19,505
<b>Repeatability std. dev. <math>s_r</math></b>	<b>0,78</b>	<b>12,71</b>	<b>1,57</b>	<b>2,07</b>	<b>5,05</b>	<b>1,17</b>	<b>4,35</b>
<b>Reproducibility std. dev. <math>s_R</math></b>	<b>1,76</b>	<b>14,98</b>	<b>3,47</b>	<b>2,85</b>	<b>5,05</b>	<b>1,97</b>	<b>4,42</b>
Repeatability COV ( $s_r/m$ ) / %	7,3	15,7	8,0	14,4	19,1	8,3	14,5
Reproducibility COV ( $s_R/m$ ) / %	16,4	18,5	17,6	19,8	19,1	13,9	14,7
Number of outliers	0	0	1	0	0	1	1
Number of excluded outliers	0	0	0	0	0	1	0
Outlier type	-	-	<i>Mh</i>	-	-	<i>C, Mk</i>	<i>Mh</i>
Outlier laboratories	-	-	Lab 9	-	-	Lab 1	Lab 6

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

# Sealing quality assessment by mass loss (Chromium free)



# Sealing quality assessment by mass loss (Chromium free)

## Laboratories outside critical value lines of **Mandel's** statistics

Level	NB	NE	BM	BC	FM	FC	XN
Mandel's <i>k</i> -plot	Lab 11	-	Lab 15	Lab 15	-	Lab 1	-
Classification	<i>Straggler</i>	-	<b>Outlier</b>	<b>Outlier</b>	-	<i>Straggler</i>	-
Mandel's <i>h</i> -plot	Lab 15	-	Lab 15	Lab 15	Lab 15	Lab 15	Lab 15
Classification	<b>Outlier</b>	-	<b>Outlier</b>	<b>Outlier</b>	<b>Outlier</b>	<b>Outlier</b>	<b>Outlier</b>

## Cochran's test results

Level	NB	NE	BM	BC	FM	FC	XN
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 $C$	0,732	0,270	0,703	0,605	0,329	0,898	0,514
Classification	<b>Outlier</b>	<i>Correct</i>	<b>Outlier</b>	<i>Straggler</i>	<i>Correct</i>	<b>Outlier</b>	<i>Correct</i>
Straggler Lab ( $C > C_{Cr (1\%)}$ )	-	-	-	Lab 15	-	-	-
Outlier Lab ( $C > C_{Cr (1\%)}$ )	Lab 11 Lab 15	-	Lab 15	-	-	Lab 1	-

# Sealing quality assessment by mass loss (Chromium free)

## Grubb's test results

Level	NB	NE	BM	BC	FM	FC	XN
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 $G_p$	3,001	1,830	3,104	3,206	3,155	3,106	2,800
Single low $G_1$	1,337	1,313	0,939	0,876	0,700	0,894	1,092
Classification (high)	<b>Outlier</b>	<i>Correct</i>	<b>Outlier</b>	<b>Outlier</b>	<b>Outlier</b>	<b>Outlier</b>	<b>Outlier</b>
Outlier Lab ( $G_p > G_{Cr}$ (1%))	Lab 15	-	Lab 15	Lab 15	Lab 15	Lab 15	Lab 15
Classification (low)	<i>Correct</i>	<i>Correct</i>	<i>Correct</i>	<i>Correct</i>	<i>Correct</i>	<i>Correct</i>	<i>Correct</i>
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 $G_{largest}$	0,1621	0,5023	0,1051	0,0630	0,0662	0,0971	0,2049
Double low $G_{smallest}$	0,7814	0,6663	0,8267	0,8731	0,9165	0,8809	0,7668
Classification (two largest)	<b>Outlier</b>	<i>Correct</i>	<b>Outlier</b>	<b>Outlier</b>	<b>Outlier</b>	<b>Outlier</b>	<b>Outlier</b>
Outlier Lab ( $G_{largest} < G_{Cr}$ (1%))	Lab 15	-	Lab 15	Lab 15	Lab 15	Lab 15	Lab 15
Classification (two smallest)	<i>Correct</i>	<i>Correct</i>	<i>Correct</i>	<i>Correct</i>	<i>Correct</i>	<i>Correct</i>	<i>Correct</i>
Outlier Lab ( $G_{smallest} < G_{Cr}$ (1%))	-	-	-	-	-	-	-

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





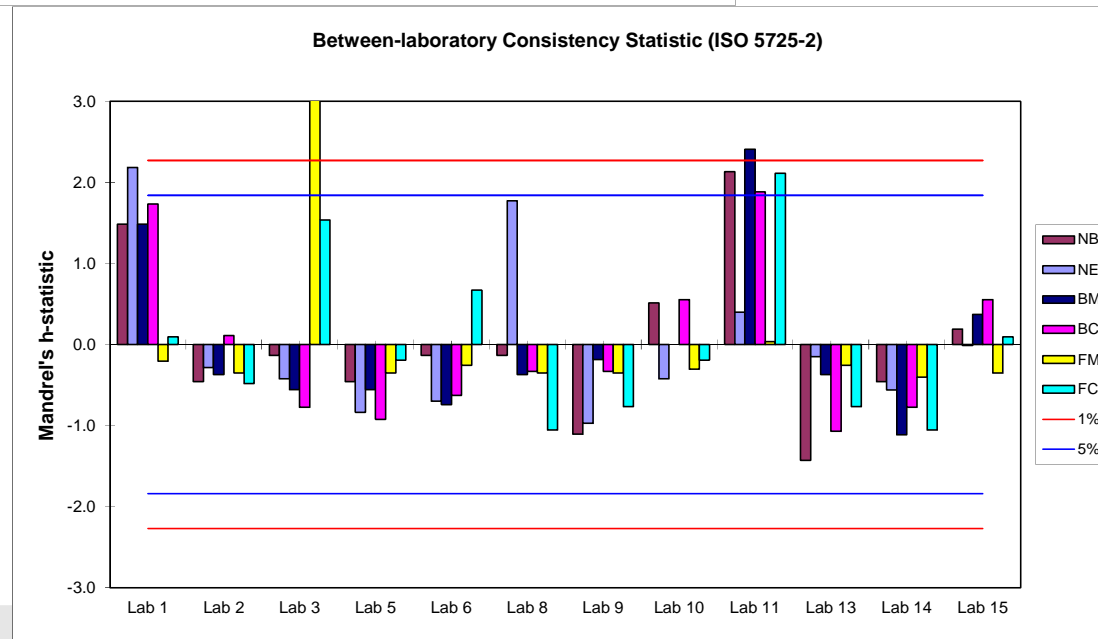
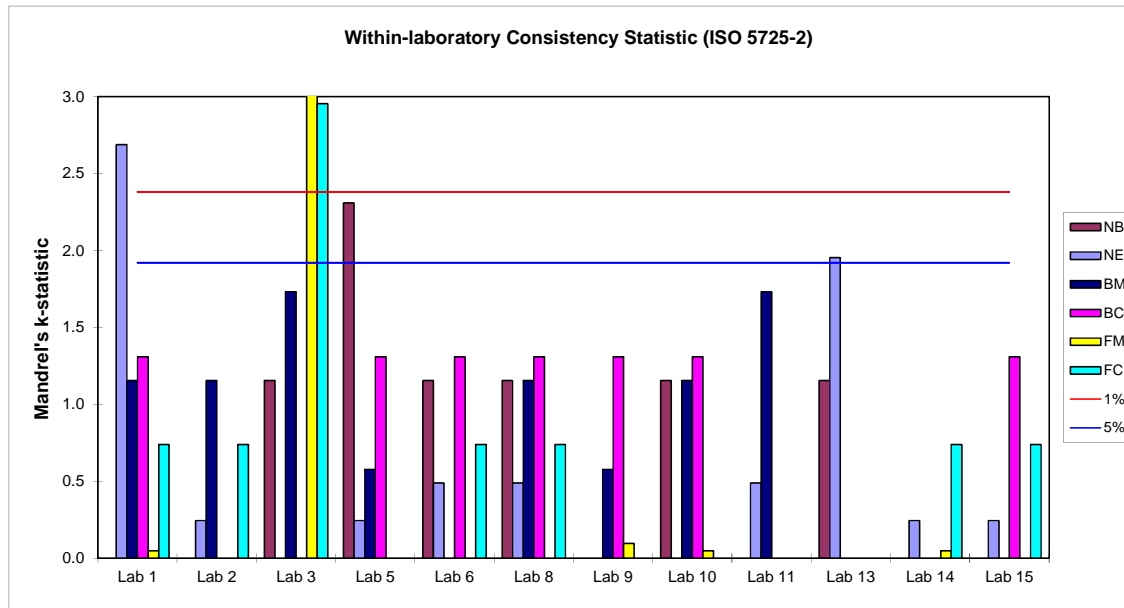
## Sealing quality assessment by mass loss (Chromium free)

### Results of precision analysis

Level	NB	NE	BM	BC	FM	FC	XN
Number of replicates $n$	2	2	2	2	2	2	2
Valid laboratories $p$	12	13	12	12	12	12	12
<b>General mean <math>m</math> / mg.dm<sup>-2</sup></b>	<b>13,53</b>	<b>89,51</b>	<b>25,04</b>	<b>18,08</b>	<b>30,17</b>	<b>17,14</b>	<b>40,18</b>
Repeatability variance $s_r^2$	4,455	186,573	1,822	1,889	37,184	5,738	32,392
Between-lab variance $s_L^2$	4,138	228,218	13,379	3,668	0	5,606	6,051
Reproducibility variance $s_R^2$	8,592	414,791	15,201	5,557	37,184	11,344	38,443
<b>Repeatability std. dev. <math>s_r</math></b>	<b>2,11</b>	<b>13,66</b>	<b>1,35</b>	<b>1,37</b>	<b>6,10</b>	<b>2,40</b>	<b>5,69</b>
<b>Reproducibility std. dev. <math>s_R</math></b>	<b>2,93</b>	<b>20,37</b>	<b>3,90</b>	<b>2,36</b>	<b>6,10</b>	<b>3,37</b>	<b>6,20</b>
Repeatability COV ( $s_r/m$ ) / %	15,6	15,3	5,4	7,6	20,2	14,0	14,2
Reproducibility COV ( $s_R/m$ ) / %	21,7	22,8	15,6	13,0	20,2	19,6	15,4
Number of outliers	2	0	1	1	1	2	1
Number of excluded outliers	1	0	1	1	1	1	1
Outlier type	<sup>1</sup> Mh, <sup>1</sup> Mk, <sup>2</sup> C, <sup>1</sup> G(I), <sup>1</sup> G(II)	-	Mh, Mk, C, G(I), G(II)	Mh, Mk, G(I), G(II)	Mh, G(I), G(II)	<sup>1</sup> Mh, <sup>1</sup> Mk, <sup>2</sup> C, <sup>1</sup> G(I), <sup>1</sup> G(II)	Mh, G(I), G(II)
Outlier laboratories	Lab 15 <sup>1</sup> Lab 11 <sup>2</sup>	-	Lab 15	Lab 15	Lab 15	Lab 15 <sup>1</sup> Lab 1 <sup>2</sup>	Lab 15

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

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



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

## Laboratories outside critical value lines of **Mandel's** statistics

Level	NB	NE	BM	BC	FM	FC
<i>Mandel's k-plot</i>	Lab 5	Lab 15 <sup>1</sup> Lab 13 <sup>2</sup>	-	-	Lab 3	Lab 3
Classification	<i>Straggler</i>	<sup>1</sup> <b>Outlier</b> <sup>2</sup> <i>Straggler</i>	-	-	<b>Outlier</b>	<b>Outlier</b>
<i>Mandel's h-plot</i>	Lab 13	Lab 1	Lab 11	Lab 11	Lab 3	Lab 11
Classification	<i>Straggler</i>	<i>Straggler</i>	<b>Outlier</b>	<i>Straggler</i>	<b>Outlier</b>	<b>Outlier</b>

## **Cochran's** test results

Level	NB	NE	BM	BC	FM	FC
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 $C$	0,444	0,602	0,250	0,143	0,999	0,727
Classification	<i>Correct</i>	<i>Straggler</i>	<i>Correct</i>	<i>Correct</i>	<b>Outlier</b>	<b>Outlier</b>
Straggler Lab ( $C > C_{Cr} (1\%)$ )	-	Lab 1	-	-	-	-
Outlier Lab ( $C > C_{Cr} (1\%)$ )	-	-	-	-	Lab 3	Lab 3

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

## Grubb's test results

Level	NB	NE	BM	BC	FM	FC
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 $G_p$	2,132	2,186	2,410	1,883	3,154	2,112
Single low $G_1$	1,430	0,973	1,112	1,071	0,402	1,056
Classification (low)	<i>Correct</i>	<i>Correct</i>	<i>Correct</i>	<i>Correct</i>	<b>Outlier</b>	<i>Correct</i>
Outlier Lab ( $G_p > G_{Cr}$ (1%))	-	-	-	-	Lab 3, 11	-
Classification (low)	<i>Correct</i>	<i>Correct</i>	<i>Correct</i>	<i>Correct</i>	<i>Correct</i>	<i>Correct</i>
Outlier Lab ( $G_1 > G_{Cr}$ (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 $G_{largest}$	0,2678	0,1366	0,1341	0,2848	0,0030	0,2593
Double low $G_{smallest}$	0,6443	0,8207	0,8063	0,7822	0,9688	0,7568
Classification (two largest)	<i>Correct</i>	<b>Outlier</b>	<b>Outlier</b>	<i>Correct</i>	<b>Outlier</b>	<i>Correct</i>
Outlier Lab ( $G_{largest} < G_{Cr}$ (1%))	-	Lab 1	Lab 11	-	Lab 3	-
Classification (two smallest)	<i>Correct</i>	<i>Correct</i>	<i>Correct</i>	<i>Correct</i>	<i>Correct</i>	<i>Correct</i>
Outlier Lab ( $G_{smallest} < G_{Cr}$ (1%))	-	-	-	-	-	-

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

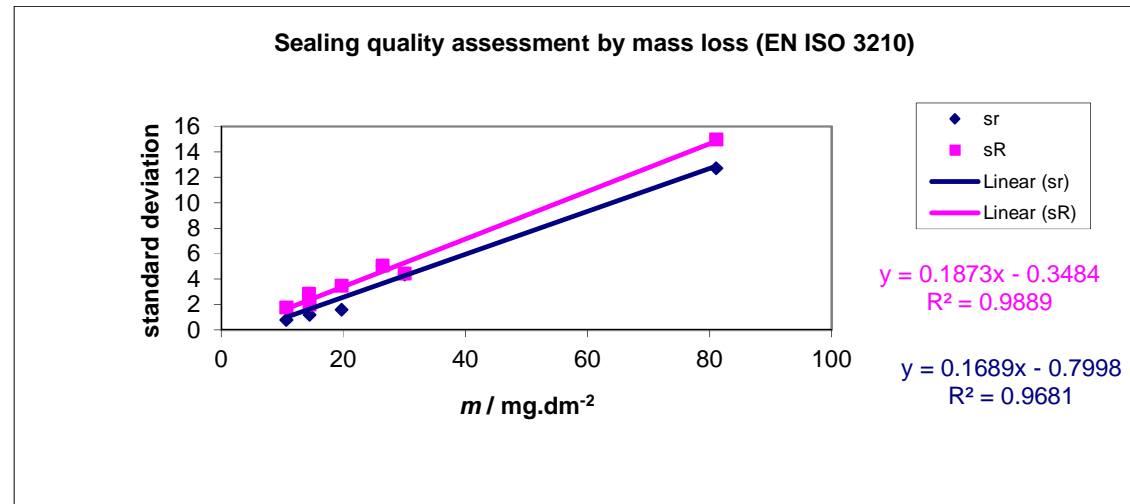
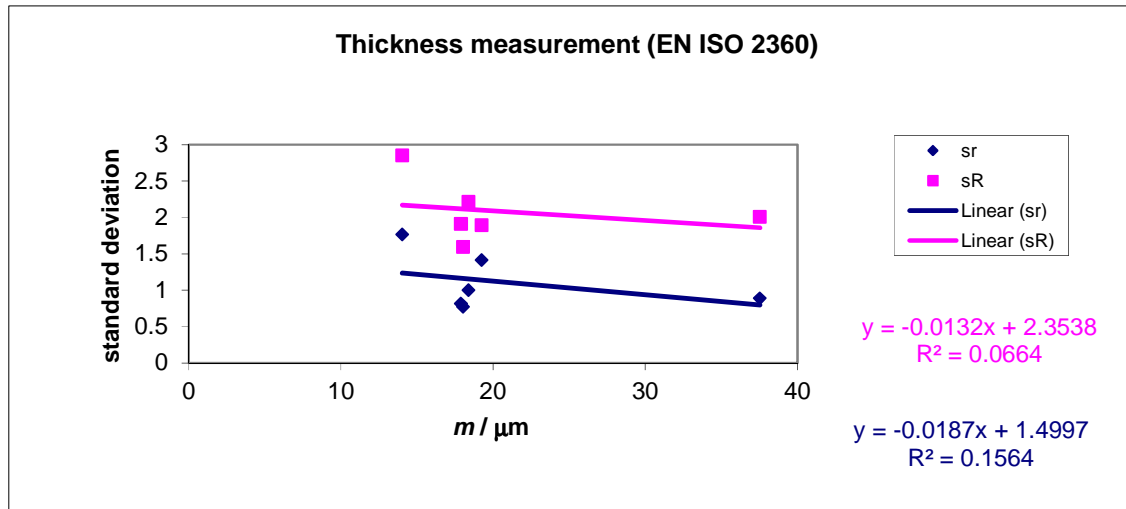
## Sealing quality assessment by measurement of admittance (EN ISO 2931)

### Results of precision analysis

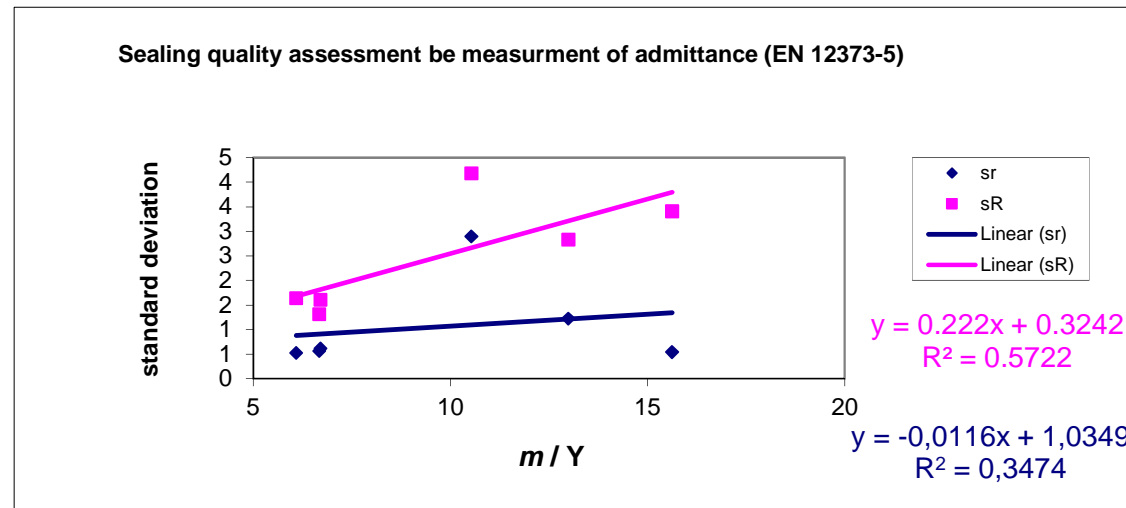
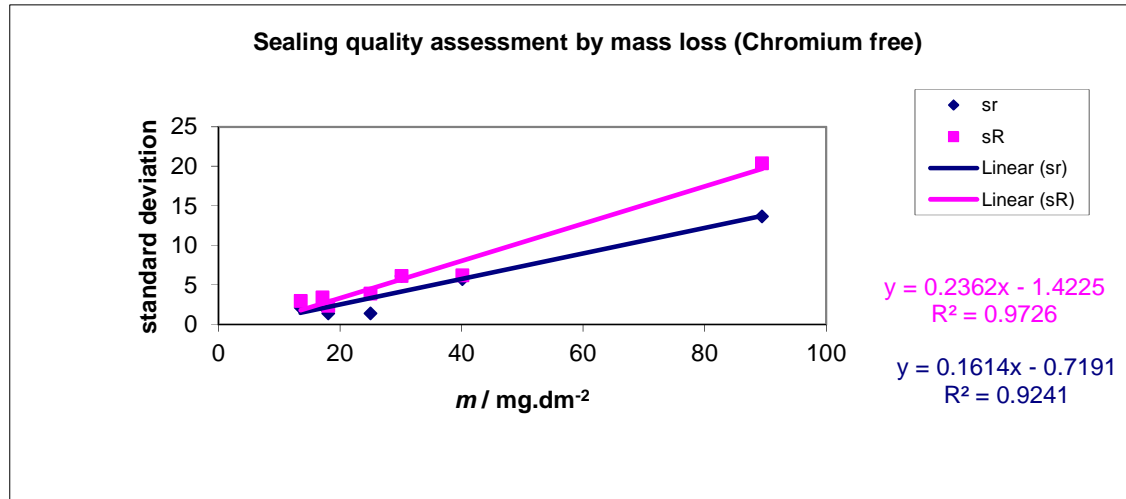
Level	NB	NE	BM	BC	FM	FC
Number of replicates $n$	2	2	2	2	2	2
Valid laboratories $p$	12	12	12	12	11	11
<b>General mean <math>m / \mu\text{m}</math></b>	<b>6,71</b>	<b>10,54</b>	<b>13,00</b>	<b>15,63</b>	<b>6,68</b>	<b>6,09</b>
Repeatability variance $s_r^2$	0,375	8,375	1,500	0,292	0,318	0,273
Between-lab variance $s_L^2$	2,197	9,061	6,523	11,314	1,405	2,405
Reproducibility variance $s_R^2$	2,572	17,436	8,023	11,606	1,723	2,677
<b>Repeatability std. dev. <math>s_r</math></b>	<b>0,61</b>	<b>2,89</b>	<b>1,22</b>	<b>0,54</b>	<b>0,56</b>	<b>0,52</b>
<b>Reproducibility std. dev. <math>s_R</math></b>	<b>1,60</b>	<b>4,18</b>	<b>2,83</b>	<b>3,41</b>	<b>1,31</b>	<b>1,64</b>
Repeatability COV ( $s_r/m$ ), %	9,1	27,5	9,4	3,5	8,4	8,6
Reproducibility COV ( $s_R/m$ ), %	23,9	39,6	21,8	21,8	19,6	26,9
Number of outliers	0	1	1	0	2	1
Number of excluded outliers	0	0	0	0	1	1
Outlier type	-	$Mk, G(II)$	$Mh, G(II)$	-	$Mk^1, Mh^1, C^1, G(I)^{1,2}, G(II)^1$	$Mk, C$
Outlier laboratories	-	Lab 1	Lab 11	-	<sup>1</sup> Lab 3 <sup>2</sup> Lab 11	Lab 3

Outlier type:  $Mh$  – Mandel's  $h$ ;  $Mk$  – Mandel's  $k$ ;  $C$  – Cochran's;  $G(I)$  – Grubs (one outlying observation);  $G(II)$  – Grubs (two outlying observations)

# Dependency analysis of precision (repeatability and reproducibility) with the mean



# Dependency analysis of precision (repeatability and reproducibility) with the mean



# Precision analysis results 2013

EN ISO 2360 – Thickness measurement	Anodic coating type							Excluded data lab: anodic coating type
	NB	NE	BM	BC	FM	FC	XN	
General mean $m / \mu\text{m}$	19,3	37,5	18,4	17,9	18,0	14,0	-	Lab 11: NB, BM, BC, FM, FC
Repeatability std. dev. $s_r$	1,41	0,92	1,00	0,82	0,77	1,77	-	
Reproducibility std. dev. $s_R$	1,89	1,70	2,21	1,91	1,59	2,85	-	
Global repeatability std. dev.	$s_r = 1,1$							
Global reproducibility std. dev.	$s_R = 2,1$							
EN ISO 3210 - Sealing quality by mass loss	Anodic coating type							Excluded data lab: anodic coating type
	NB	NE	BM	BC	FM	FC	XN	
General mean $m / \text{g.dm}^{-2}$	10,7	81,2	19,8	14,5	26,5	14,5	30,1	None
Repeatability std. dev. $s_r$	0,78	12,71	1,57	2,07	5,05	1,17	4,35	
Reproducibility std. dev. $s_R$	1,76	14,98	3,47	2,85	5,05	1,97	4,42	
Global repeatability std. dev.	$s_r = 0,1689 m - 0,7998$ ( $R^2 \approx 1$ ) or $s_r = 1,4$ (if $m < 25 \text{ mg/dm}^2$ )							
Global reproducibility std. dev.	$s_R = 0,1873 m - 0,3484$ ( $R^2 \approx 1$ ) or $s_R = 2,5$ (if $m < 25 \text{ mg/dm}^2$ )							
Chromium free test - Sealing quality by mass loss	Anodic coating type							Excluded data lab: anodic coating type
	NB	NE	BM	BC	FM	FC	XN	
General mean $m / \text{g.dm}^{-2}$	13,5	89,5	25,0	18,1	30,2	17,1	40,2	Lab 15: NB, BM, BC, FM, FC
Repeatability std. dev. $s_r$	2,11	13,66	1,35	1,37	6,10	2,40	5,69	
Reproducibility std. dev. $s_R$	2,93	20,37	3,90	2,36	6,10	3,37	6,20	
Global repeatability std. dev.	$s_r = 0,1614 m - 0,7191$ ( $R^2 \approx 0,9$ ) or $s_r = 1,8$ (if $m < 30 \text{ mg/dm}^2$ )							
Global reproducibility std. dev.	$s_R = 0,2362 m - 1,4225$ ( $R^2 \approx 1$ ) or $s_R = 3,1$ (if $m < 30 \text{ mg/dm}^2$ )							
EN 2931 - Sealing quality by admittance	Anodic coating type							Excluded data lab: anodic coating type
	NB	NE	BM	BC	FM	FC	XN	
General mean $m / Y$	6,7	10,5	13,0	15,6	6,7	6,1	-	Lab 3: FM, FC
Repeatability std. dev. $s_r$	0,61	2,89	1,22	0,54	0,56	0,52	-	
Reproducibility std. dev. $s_R$	1,60	4,18	2,83	3,41	1,31	1,64	-	
Global repeatability std. dev.	$s_r = 1,1$							
Global reproducibility std. dev.	$s_R = 2,5$							

**2013**  
**Thickness**  
 Repeatability=1,1  
 Reproducibility=2,1  
 (standard :  
 1  $\mu\text{m}$  (until 10  $\mu\text{m}$  or 10%))

**Sealing/mass loss**  
 Repeatability=1,4  
 Reproducibility=2,5

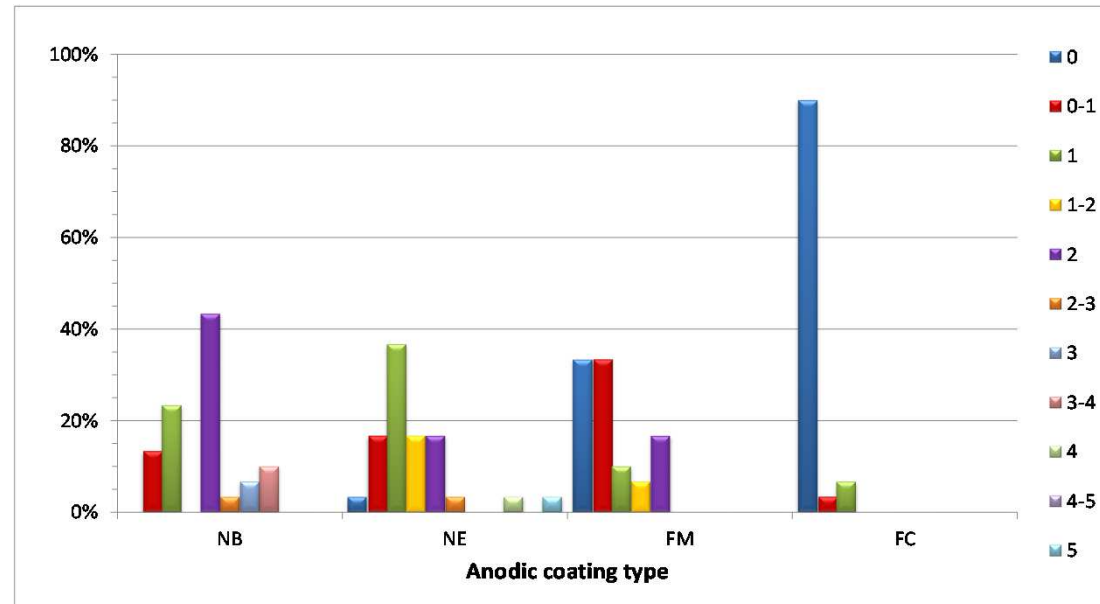
**Sealing/mass loss Cr free**  
 Repeatability= 1,8  
 Reproducibility=3,1

**Admittance**  
 Repeatability=1,1  
 Reproducibility=2,5



# 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

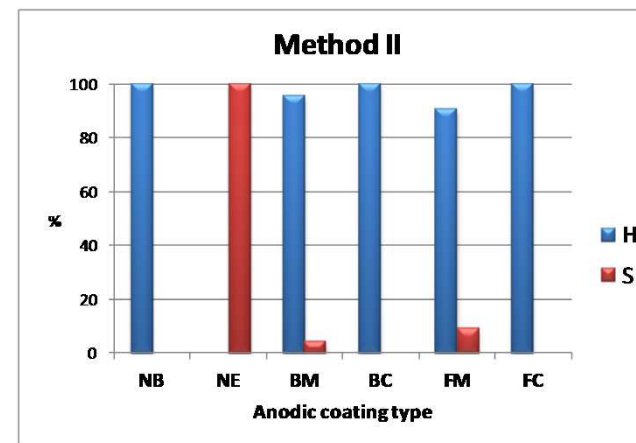
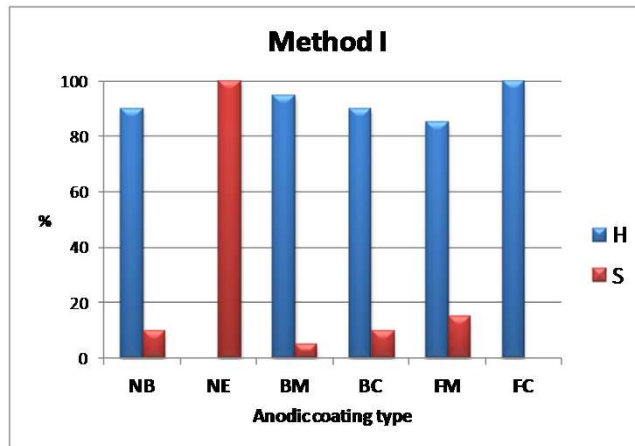


Anodic coating type	Dye absorption rating			Labs with results less than 10% frequent	Labs with results less than 5% frequent
	Mode	Median	Range of results more than 10% frequent		
NB	2	2	0-1 to 1, 2 and 3-4	Lab 3,5,9,14	Lab 5
NE	1	1	0-1 to 2	Lab 6, 7, 9	Lab 6, 7, 9
FM	0-1	0-1	0 to 1 and 2	Lab 5	None
FC	0	0	0	Lab 6, 9	Lab 6

# 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		NB	NE	BM	BC	FM	FC
Method I	Hard (%)	90	0	95	90	85	100
	Soft (%)	10	100	5	10	15	0
	Laboratories with results 10% or less frequent	Lab 15	none	Lab 8	Lab 11	Lab 8, 11	none
Method II	Hard (%)	100	0	95	100	91	100
	Soft (%)	0	100	5	0	9	0
	Laboratories with results 10% or less frequent	none	none	Lab 8	none	Lab 8	none

2006

### Thickness

Repeatability = 0.56  
Reproducibility = 1.51

(standard :  
1  $\mu\text{m}$  (until 10  $\mu\text{m}$  or 10 %

2009

### Thickness

Repeatability = 0,87  
Reproducibility = 1,82

(standard :  
1  $\mu\text{m}$  (until 10  $\mu\text{m}$  or 10 %

2011

### Thickness

Repeatability = 1  
Reproducibility = 2

(standard :  
1  $\mu\text{m}$  (until 10  $\mu\text{m}$  or 10 %)

2013

### Thickness

Repeatability=1,1  
Reproducibility=2,1

(standard :  
1  $\mu\text{m}$  (until 10  $\mu\text{m}$  or 10%)

### Sealing

#### mass loss

Repeatability = 1  
Reproducibility = 3

### Sealing

#### mass loss

Repeatability = 1  
Reproducibility = 4

### Sealing

#### mass loss

Repeatability = 1  
Reproducibility = 2

#### mass loss Cr free

Repeatability = 2  
Reproducibility = 4

### Sealing

#### mass loss

Repeatability=1,4  
Reproducibility=2,5

#### mass loss Cr free

Repeatability=1,8  
Reproducibility=3,1

### Sealing

#### admittance

Repeatability = 0.51  
Reproducibility = 3.12

### Sealing

#### admittance

Repeatability < 0.5  
Reproducibility = 1

### Sealing

#### admittance

Repeatability = 2  
Reproducibility = 3

### Sealing

#### admittance

Repeatability = 1,1  
Reproducibility = 2,5

