

2013 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	
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/ μm
NB		Natural	20 μm	18 °C	Hot water sealing (T > 96 °C)	3 min/ μm
NE		Natural	25 μm	25 °C		2 min/ μm
BM		Bronze	15 μm	21 °C		0,6 min/ μm
BC		Bronze	15 μm	21 °C		5 min/ μm
FM		Natural	15 μm	18 °C	Cold sealing (T = 27 °C)	0,4 min/ μm
FC		Natural	15 μm	18 °C		0,8 min/ μm
XN		Natural	15 μm	17 °C	Hot water (T > 96 °C)	4 min/ μm

Anodizing and sealing conditions used for the production of test specimens

Anodic coating type	Anodizing		Hot water sealing	
NB	<i>Free H₂SO₄</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 ²	<i>Time -</i>	3 min/μm
NE	<i>Free H₂SO₄</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 ²	<i>Time -</i>	3 min/μm
BM	<i>Free H₂SO₄</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 ²	<i>Time -</i>	5 min
BC	<i>Free H₂SO₄</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 ²	<i>Time -</i>	90 min

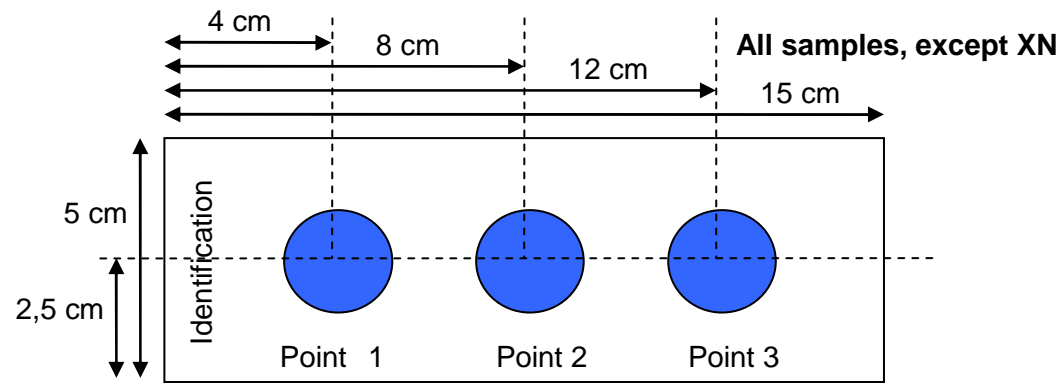
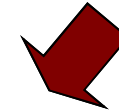
Anodizing and sealing conditions used for the production of test specimens (cont)

Anodic coating type	Anodizing		Hot water sealing	
FM	<i>Free H₂SO₄</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 ²	<i>Time -</i>	5 min
FC	<i>Free H₂SO₄</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 ²	<i>Time -</i>	14 min
XC	<i>Free H₂SO₄</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 ²	<i>Time -</i>	80 min

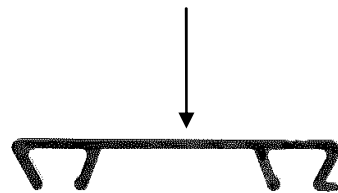
Instructions

- Laboratory code number
- Measuring points

Scheme of the testing zones for thickness measurement

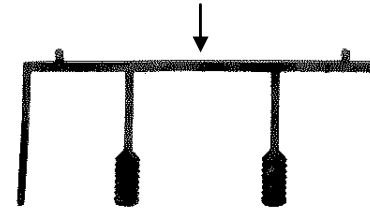


Surface for tests



NB, NE, BM and BC samples

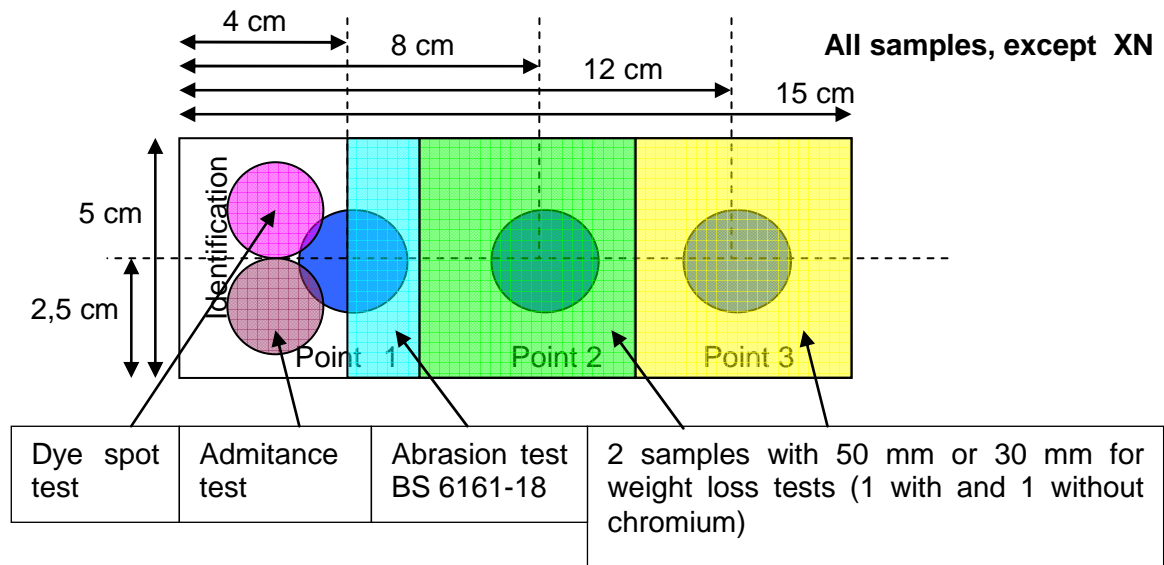
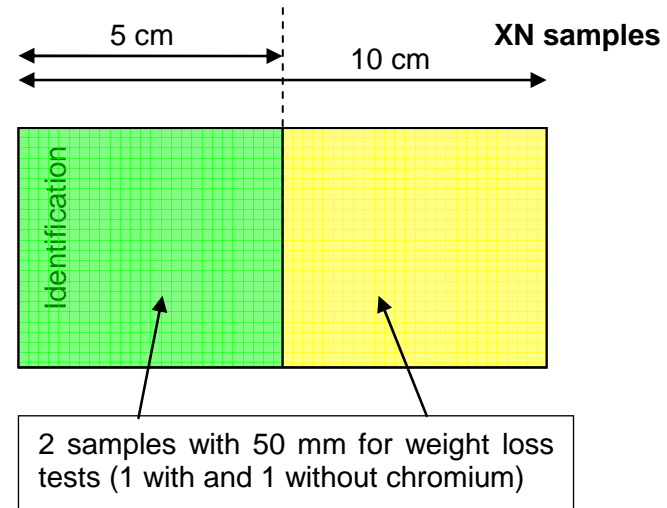
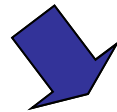
Surface for tests



FM and FC samples

Instructions

Scheme of testing zones for sealing quality assessment



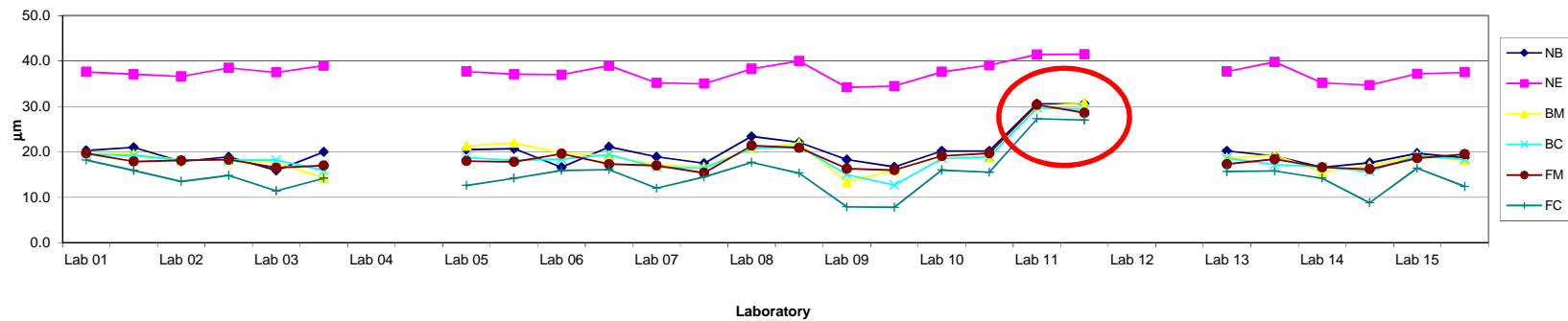
EXCEL Worksheets for data registration

Protocol for NB sample									
Laboratory identification					Test conditions				
Test laboratory					Temperature				
Sample type	NB				Date of the test			//// -mm-aa	
Tested by					Zero base			µm	
Date of report					Calibration standard 1			µm	
					Calibration standard 2			µm	
					Calibration standard 3			µm	
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	µm	(°C)	Y20
measure 1	21.6					150	24.6	21.2	20
measure 2	22.0								
measure 3	21.3								
Average thickness	21.8			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 = 50 mm Perimeter: 0.155 m ² /m									
Weight loss results (EN ISO 3210: method 2)					Weight loss results (chromic free test)				
Sample No.	Length L	Weight ₀	Weight ₁	Weight loss	Sample No.	Length L	Weight ₀	Weight ₁	Weight loss
NB??	(mm)	(g)	(g)	(mg/dm ²)	NB??	(mm)	(g)	(g)	(mg/dm ²)
measure 1	50.29	9.4683	9.4444	-	measure 1	50.29	9.4683	9.4444	-
measure 2	50.09	-	-	-	measure 2	50.09	-	-	-
measure 3	50.83	-	-	-	measure 3	50.83	-	-	-
Average	50.34	9.4683	9.4444	16.26	Average	50.34	9.4683	9.4444	16.26
REMARKS: Length = 50 mm Perimeter: 0.155 m ² /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	µm	(°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 = 50 mm Perimeter: 0.155 m ² /m									
Weight loss results (EN ISO 3210: method 2)					Weight loss results (chromic free test)				
Sample No.	Length L	Weight ₀	Weight ₁	Weight loss	Sample No.	Length L	Weight ₀	Weight ₁	Weight loss
NB??	(mm)	(g)	(g)	(mg/dm ²)	NB??	(mm)	(g)	(g)	(mg/dm ²)
measure 1		-	-	-	measure 1		-	-	-
measure 2		-	-	-	measure 2		-	-	-
measure 3		-	-	-	measure 3		-	-	-
Average					Average				

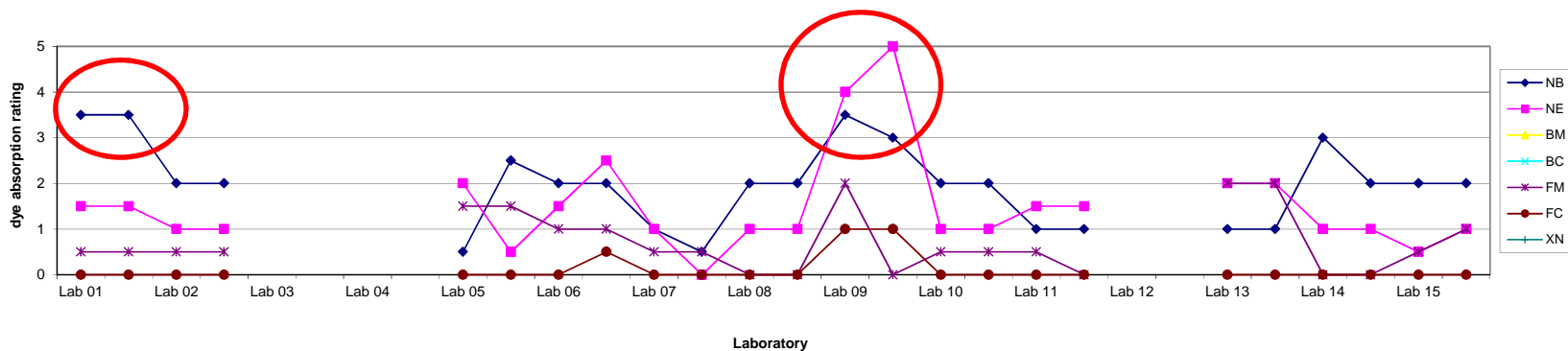
Example for the registration of tests results for specimens of coating type NB

Results reported

Test results of method EN ISO 2360

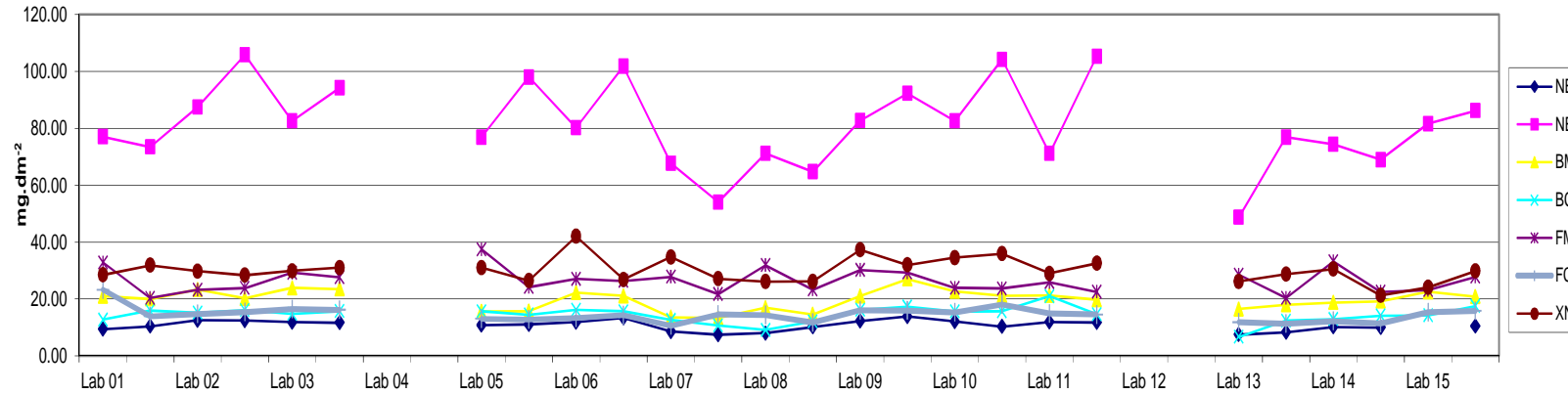


Test results of method EN ISO 2143

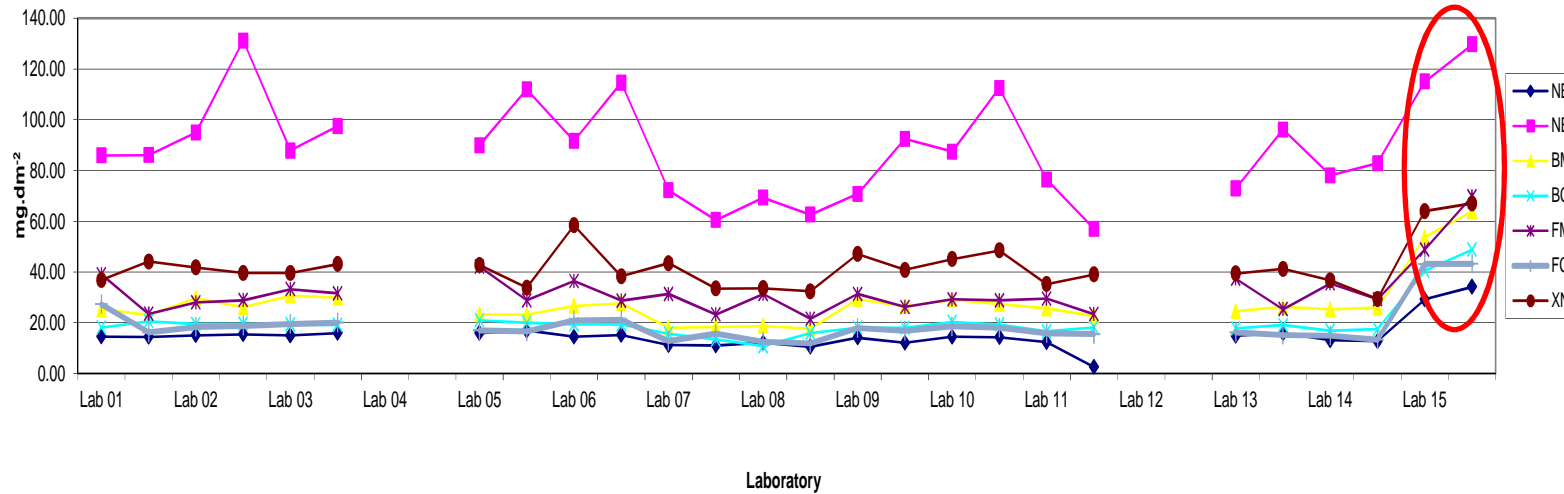


Results reported

Test results of method EN ISO 3210

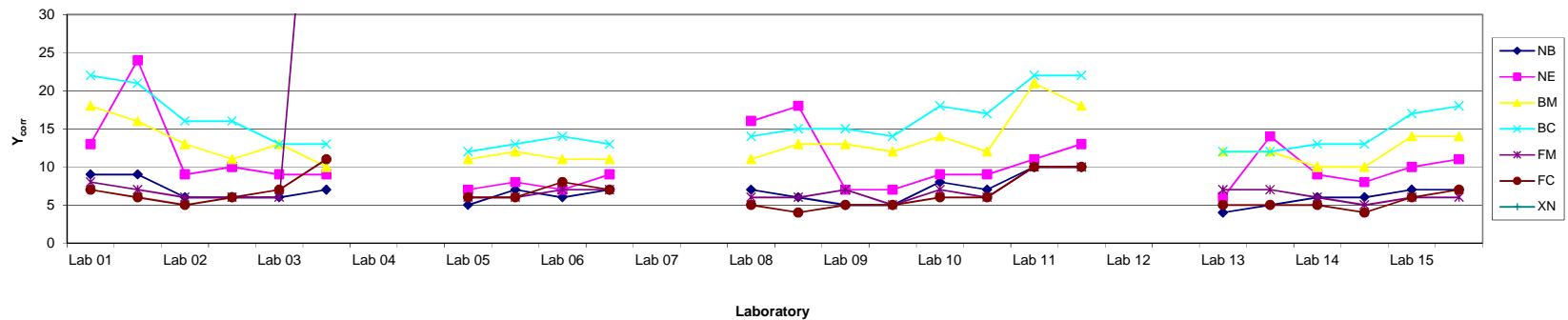
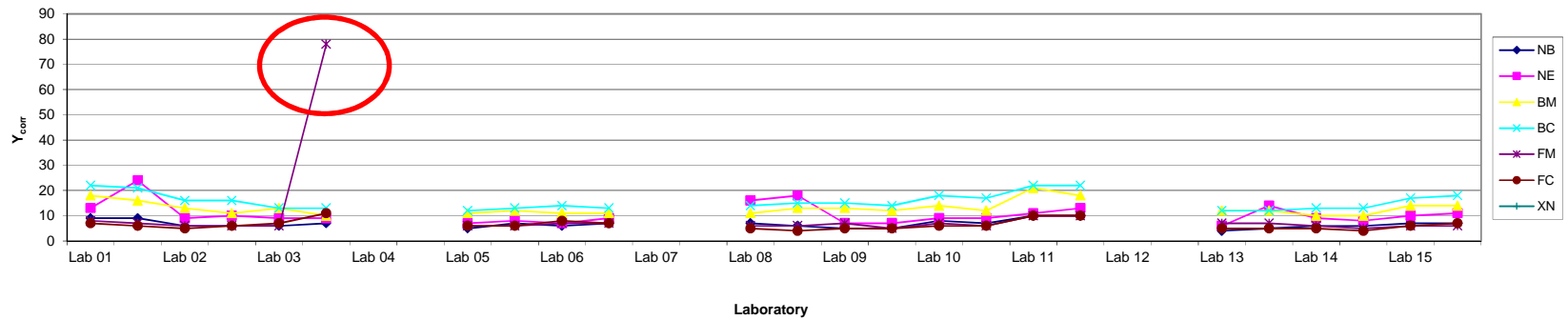


Test results of method Chromium free test



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**

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

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

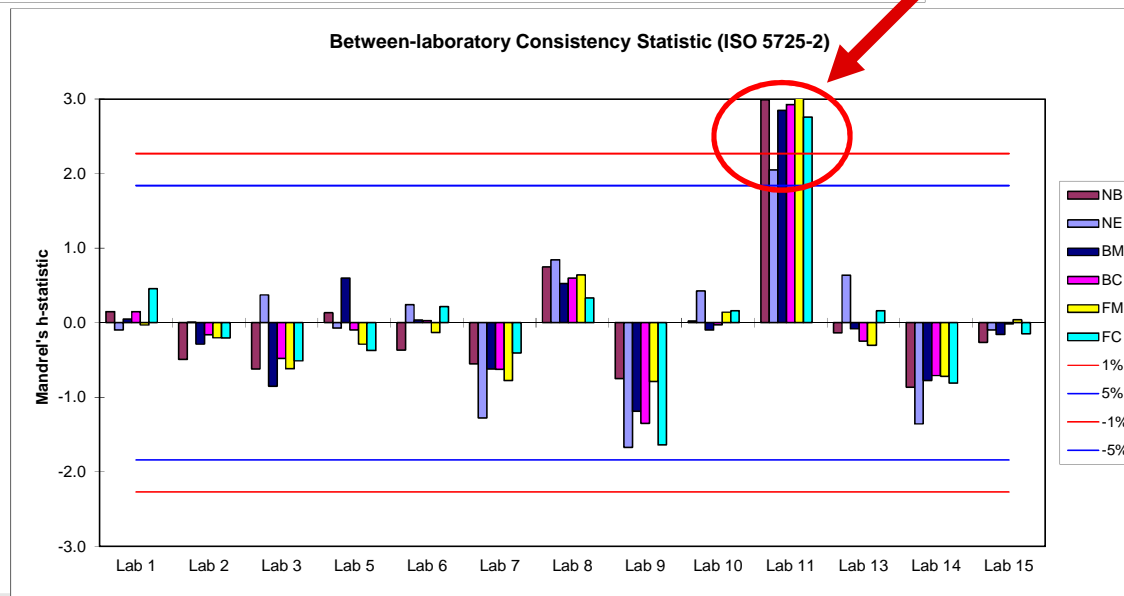
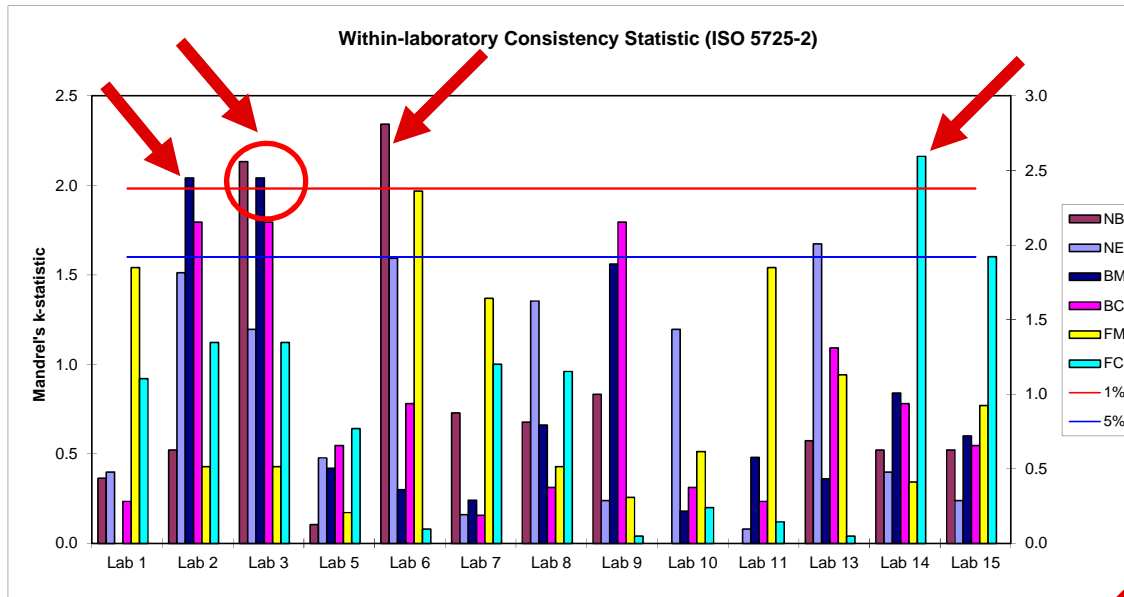
Repeatability variance

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

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	Outlier	<i>Straggler</i>	Outlier	<i>Straggler</i>	<i>Straggler</i>	Outlier
Mandel's <i>h</i> -plot	Lab 11	Lab 11	Lab 11	Lab 11	Lab 11	Lab 11
Classification	Outlier	<i>Straggler</i>	Outlier	Outlier	Outlier	Outlier

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)	Outlier	<i>Correct</i>	Outlier	Outlier	Outlier	Outlier
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



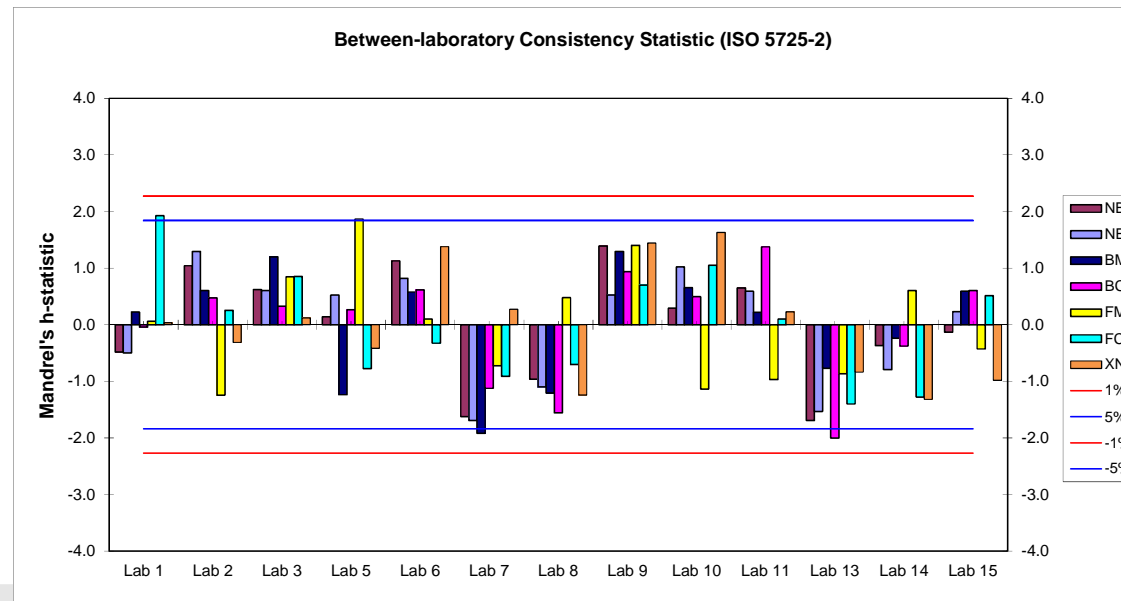
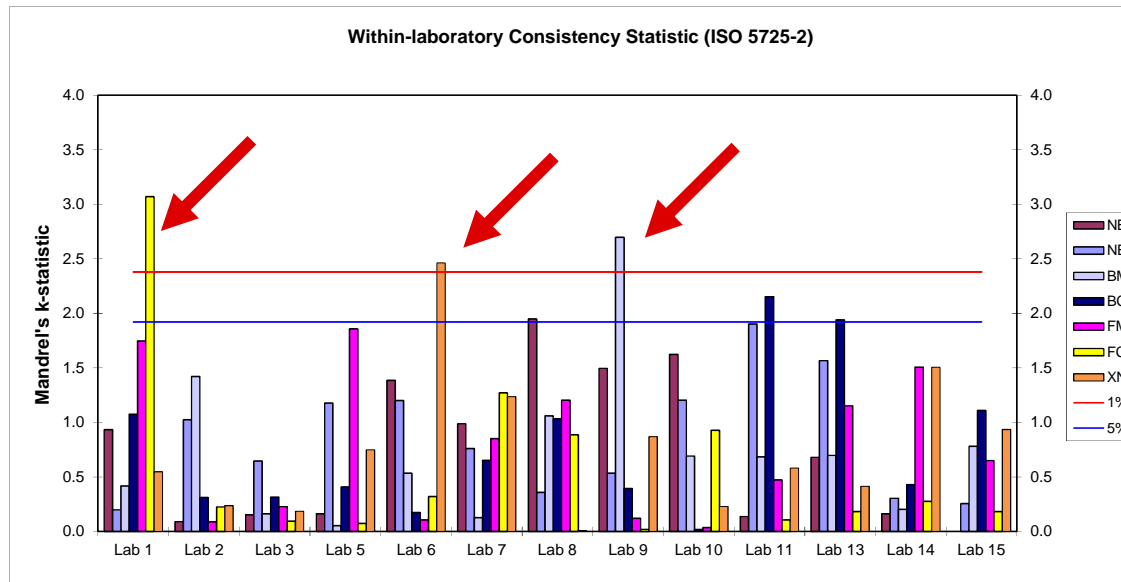
Thickness measurement (EN ISO 2360)

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
General mean $m / \mu\text{m}$	19,25	37,54	18,39	17,90	18,03	14,03
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
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
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	^{1,2} Mk, ³ Mh, ³ G(I)	-	^{1,2} Mk, ³ Mh, ³ G(I)	Mh, G(I)	Mh, G(I)	¹ Mk, ² Mh, ² G(I)
Outlier laboratories	Lab 3 ¹ Lab 6 ² Lab 11 ³	-	Lab 2 ¹ Lab 3 ² Lab 11 ³	Lab 11	Lab 11	Lab 14 ¹ Lab 11 ²

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>	-	Outlier	<i>Straggler</i>	-	Outlier	Outlier
<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>	Outlier	<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



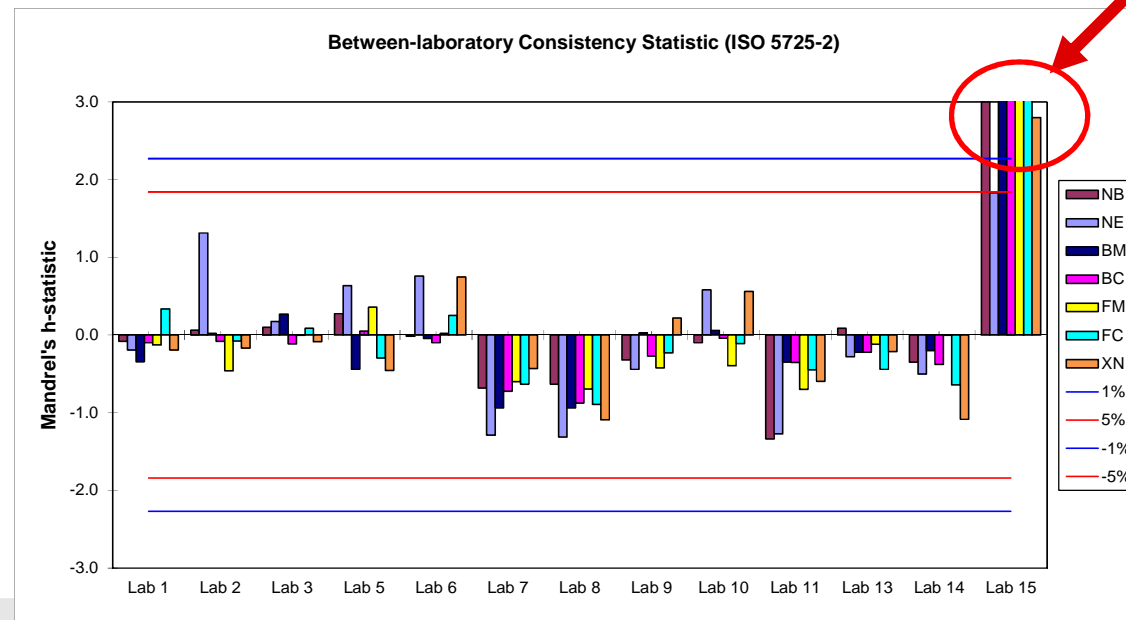
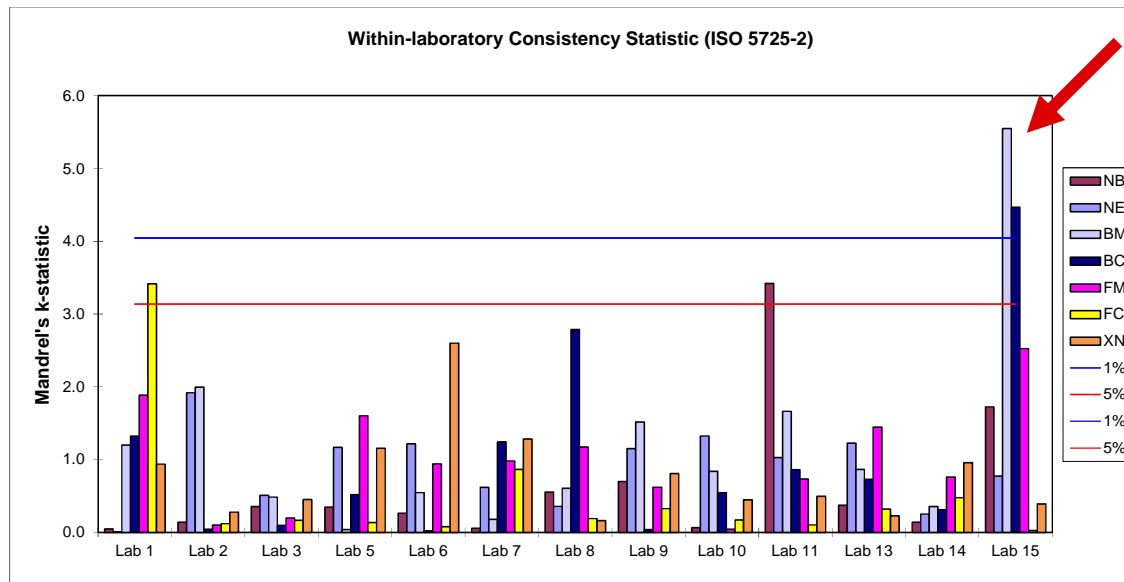
Sealing quality assessment by mass loss (EN ISO 3210)

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
General mean m / mg.dm⁻²	10,72	81,15	19,77	14,45	26,47	14,19	30,08
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
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
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	-	-	Mh	-	-	C, Mk	Mh
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>	-	Outlier	Outlier	-	<i>Straggler</i>	-
Mandel's <i>h</i> -plot	Lab 15	-	Lab 15	Lab 15	Lab 15	Lab 15	Lab 15
Classification	Outlier	-	Outlier	Outlier	Outlier	Outlier	Outlier

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	Outlier	<i>Correct</i>	Outlier	<i>Straggler</i>	<i>Correct</i>	Outlier	<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)	Outlier	<i>Correct</i>	Outlier	Outlier	Outlier	Outlier	Outlier
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)	Outlier	<i>Correct</i>	Outlier	Outlier	Outlier	Outlier	Outlier
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



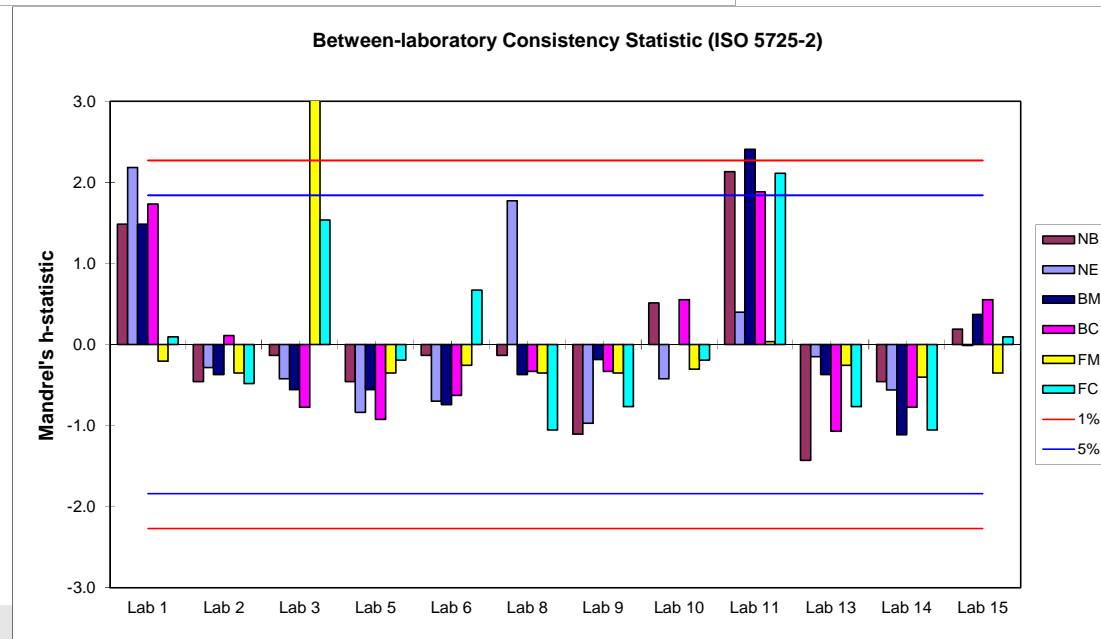
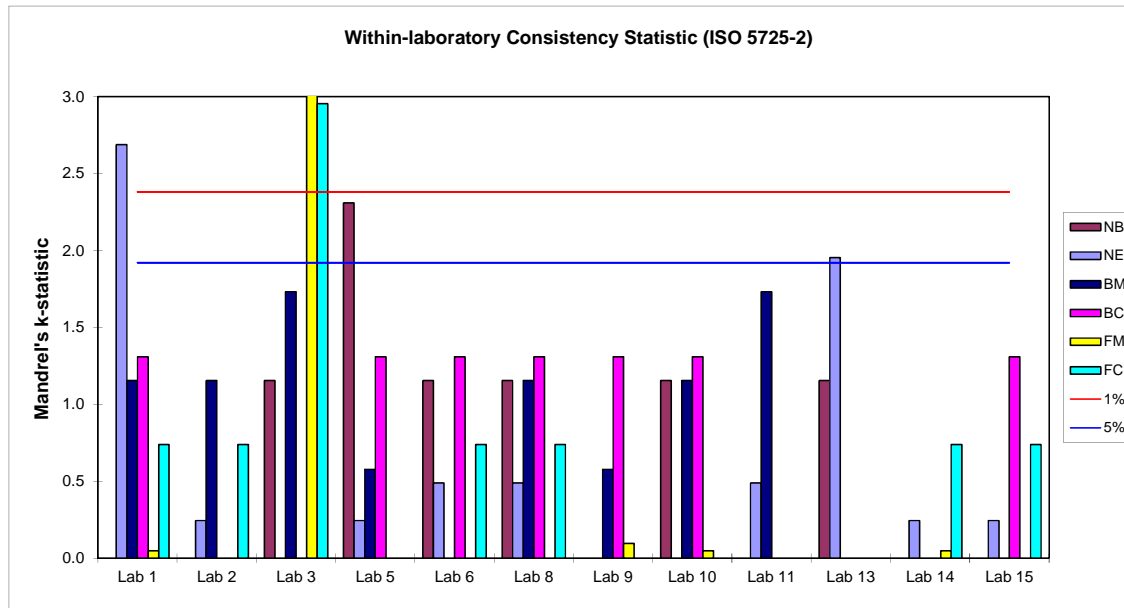
Sealing quality assessment by mass loss (Chromium free)

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
General mean m / mg.dm⁻²	13,53	89,51	25,04	18,08	30,17	17,14	40,18
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
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
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	¹ Mh, ¹ Mk, ² C, ¹ G(I), ¹ G(II)	-	Mh, Mk, C, G(I), G(II)	Mh, Mk, G(I), G(II)	Mh, G(I), G(II)	¹ Mh, ¹ Mk, ² C, ¹ G(I), ¹ G(II)	Mh, G(I), G(II)
Outlier laboratories	Lab 15 ¹ Lab 11 ²	-	Lab 15	Lab 15	Lab 15	Lab 15 ¹ Lab 1 ²	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 ¹ Lab 13 ²	-	-	Lab 3	Lab 3
Classification	<i>Straggler</i>	¹ Outlier ² <i>Straggler</i>	-	-	Outlier	Outlier
<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>	Outlier	<i>Straggler</i>	Outlier	Outlier

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>	Outlier	Outlier
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>	Outlier	<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>	Outlier	Outlier	<i>Correct</i>	Outlier	<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



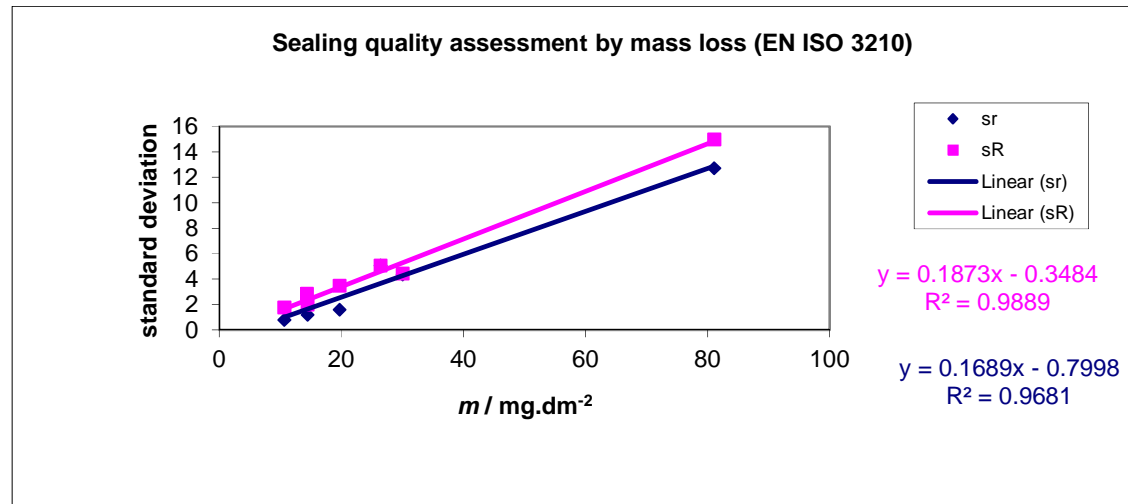
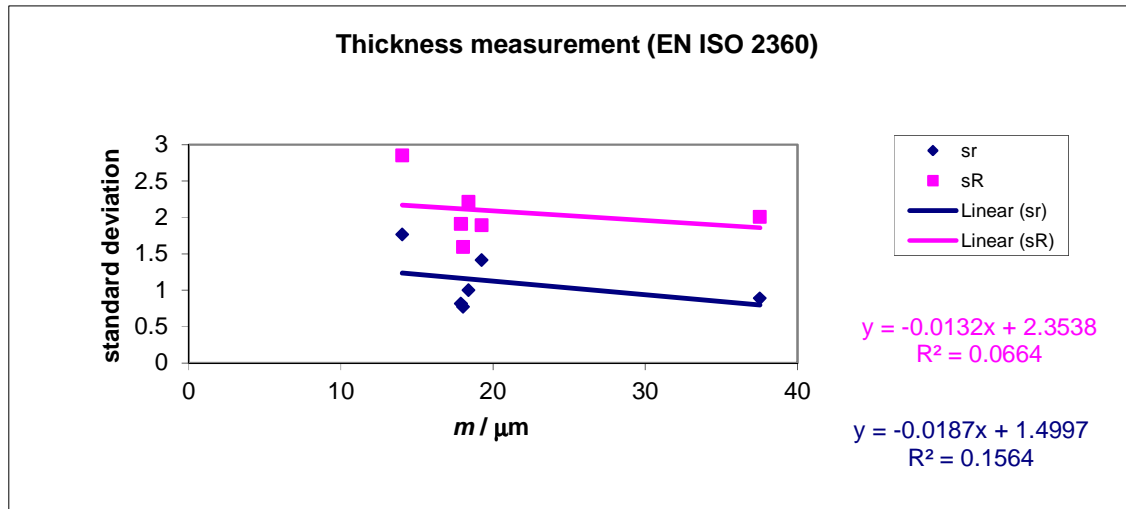
Sealing quality assessment by measurement of admittance (EN ISO 2931)

Results of precision analysis

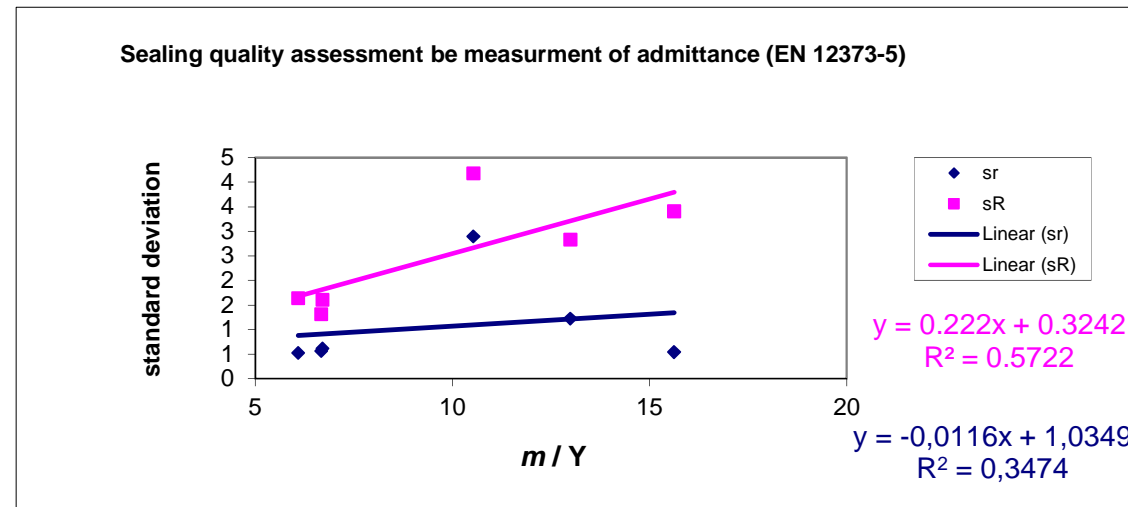
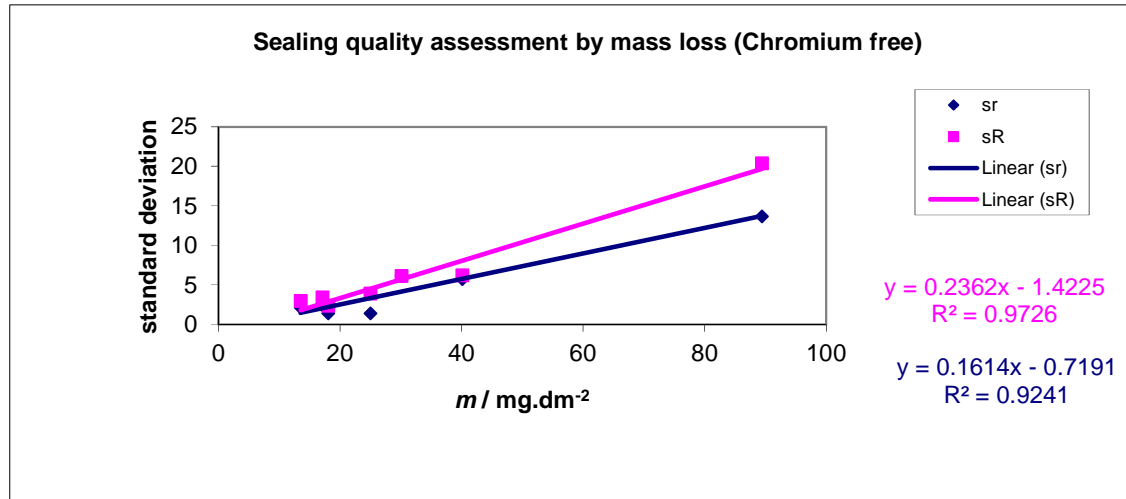
Level	NB	NE	BM	BC	FM	FC
Number of replicates n	2	2	2	2	2	2
Valid laboratories p	12	12	12	12	11	11
General mean $m / \mu\text{m}$	6,71	10,54	13,00	15,63	6,68	6,09
Repeatability variance s_r^2	0,375	8,375	1,500	0,292	0,318	0,273
Between-lab variance s_L^2	2,197	9,061	6,523	11,314	1,405	2,405
Reproducibility variance s_R^2	2,572	17,436	8,023	11,606	1,723	2,677
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
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	-	¹ Lab 3 ² 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 μm (until 10 μm or 10%))

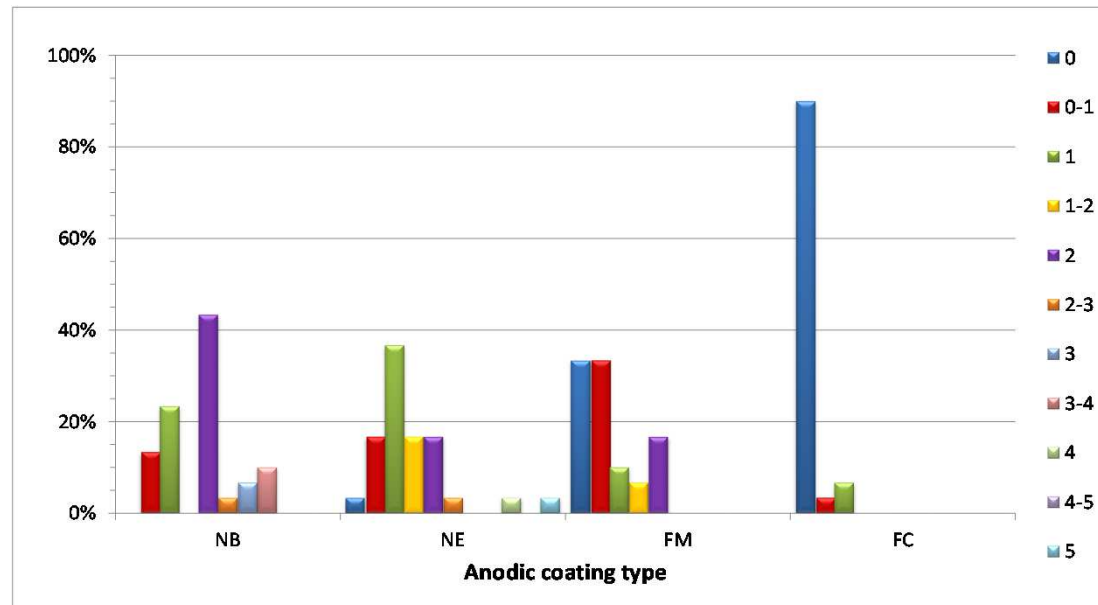
Sealing/mass loss
 Repeatability=1,4
 Reproducibility=2,5

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

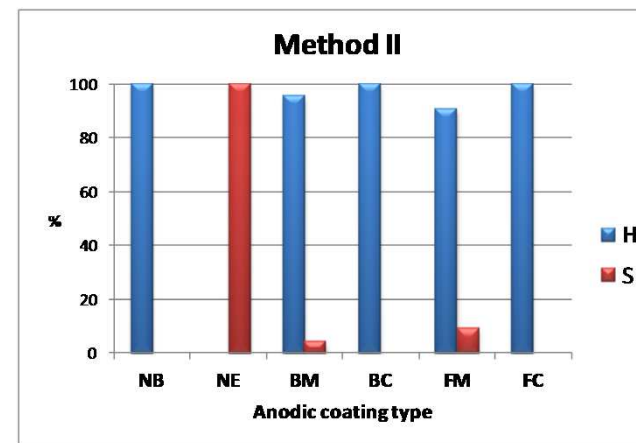
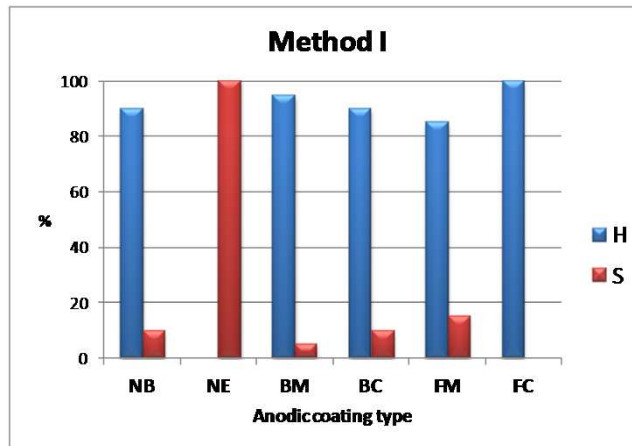


Anodic coating type	Dye absorption rating			Labs with results less than 10% frequent	Labs with results less than 5% frequent
	Mode	Median	Range of results more than 10% frequent		
NB	2	2	0-1 to 1, 2 and 3-4	Lab 3,5,9,14	Lab 5
NE	1	1	0-1 to 2	Lab 6, 7, 9	Lab 6, 7, 9
FM	0-1	0-1	0 to 1 and 2	Lab 5	None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

2009

Thickness

Repeatability = 0,87
Reproducibility = 1,82

(standard :
1 μm (until 10 μm or 10 %

2011

Thickness

Repeatability = 1
Reproducibility = 2

(standard :
1 μm (until 10 μm or 10 %)

2013

Thickness

Repeatability=1,1
Reproducibility=2,1

(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

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

