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).

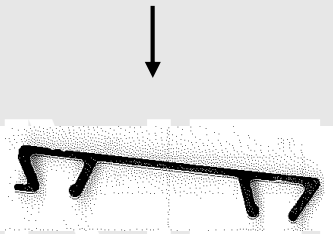
• **EN ISO 2931: 2010 –** Anodizing of aluminium and its alloys – Assessment of quality of sealed anodic oxidation coatings by ***measurement of admittance*** (ISO 2931: 2010)

• **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<br>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)

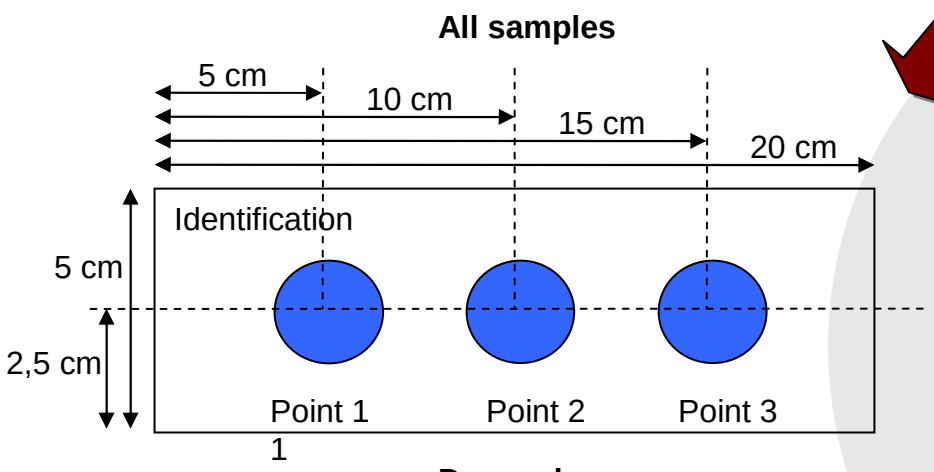
| Anodic coating type | Anodizing | | Hot water sealing | |
|---|---|---|---|---|
| D 15 | *Free $H_2SO_4$* | 180,3 g/l | *Demineralised water* | |
| | *Al content* | 12,6 g/l | *pH* | 5,7 |
| | *Temperature* | 25 ºC | *Additive* | P3 Almeco Seal |
| | *Current density* | 1,35 A/dm$^2$ | *Time* - | 3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

M. Salta, R. Fontinha, N. Garcia

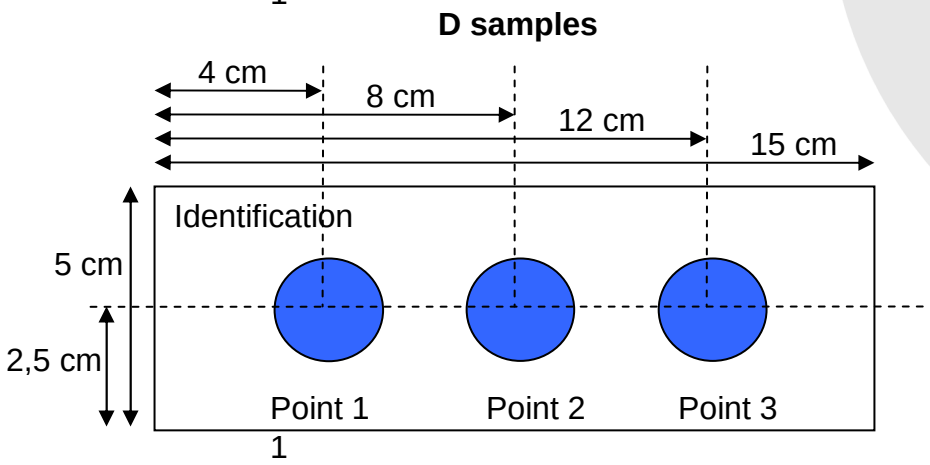
# Instructions

- Laboratory code number
- Measuring points

Scheme of the testing zones for thickness measurement




a)



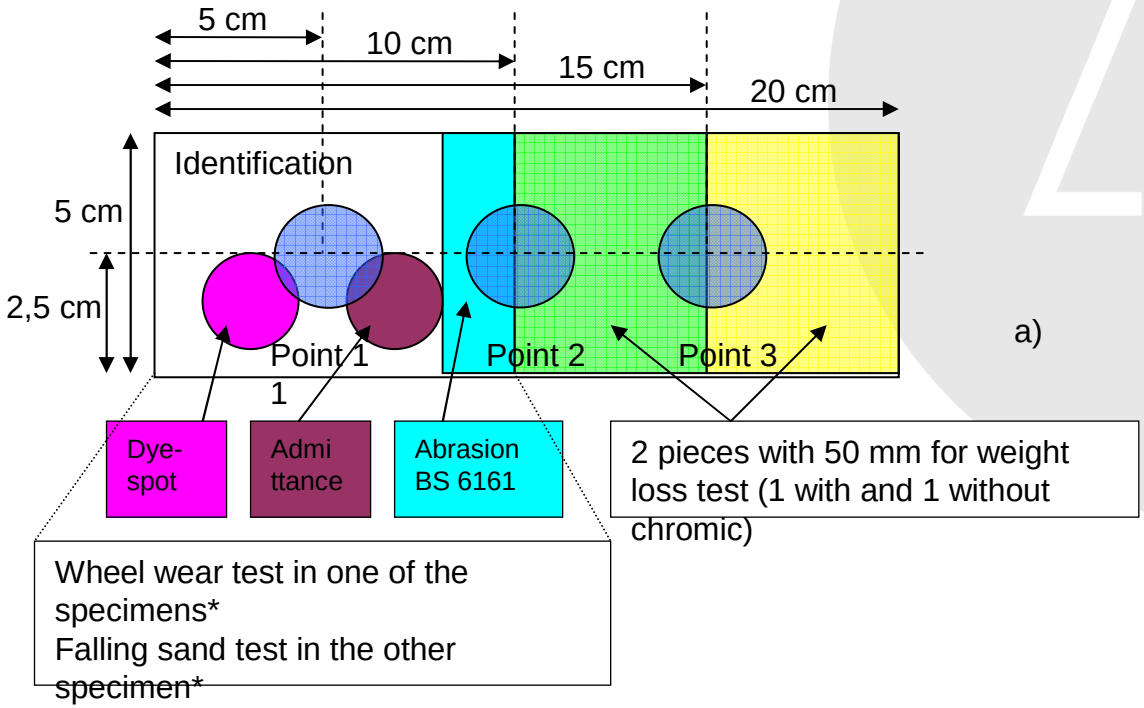

b)

M. Salta, R. Fontinha, N. Garcia

# Instructions

Scheme of testing zones for sealing quality assessment

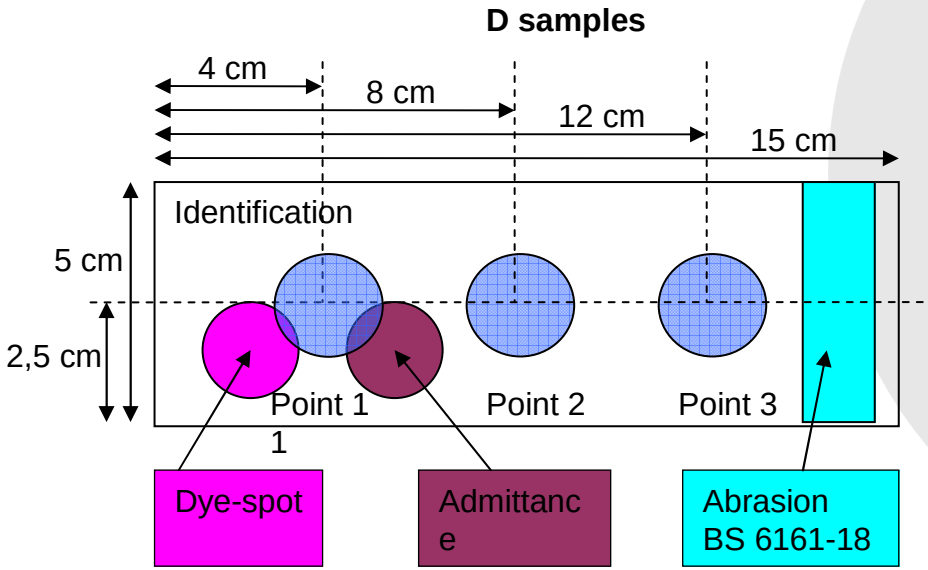
**All samples**




M. Salta, R. Fontinha, N. Garcia

# 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

M. Salta, R. Fontinha, N. Garcia

LABORATÓRIO NACIONAL DE ENGENHARIA CIVIL

# 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 | |

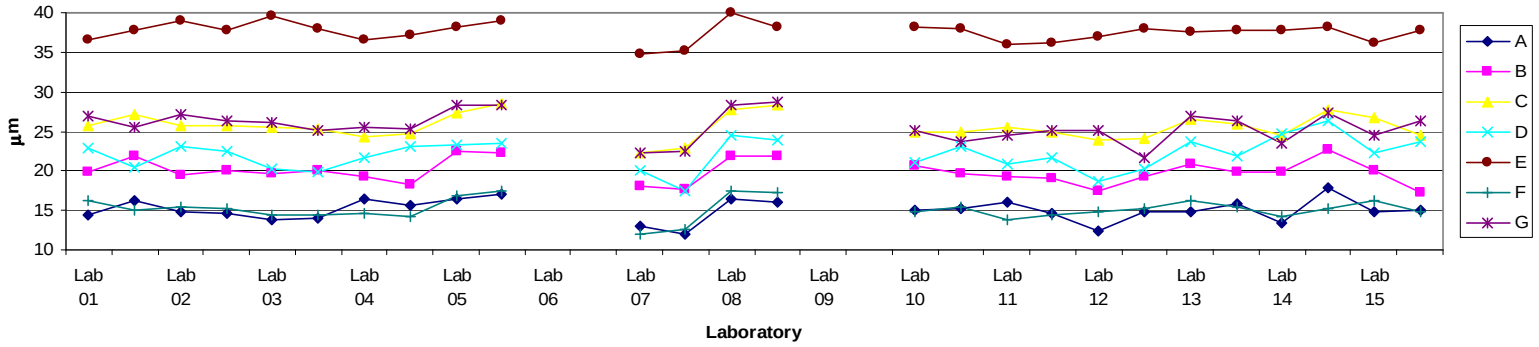
*Example for the registration of tests results for specimens of coating type A*

M. Salta, R. Fontinha, N. Garcia

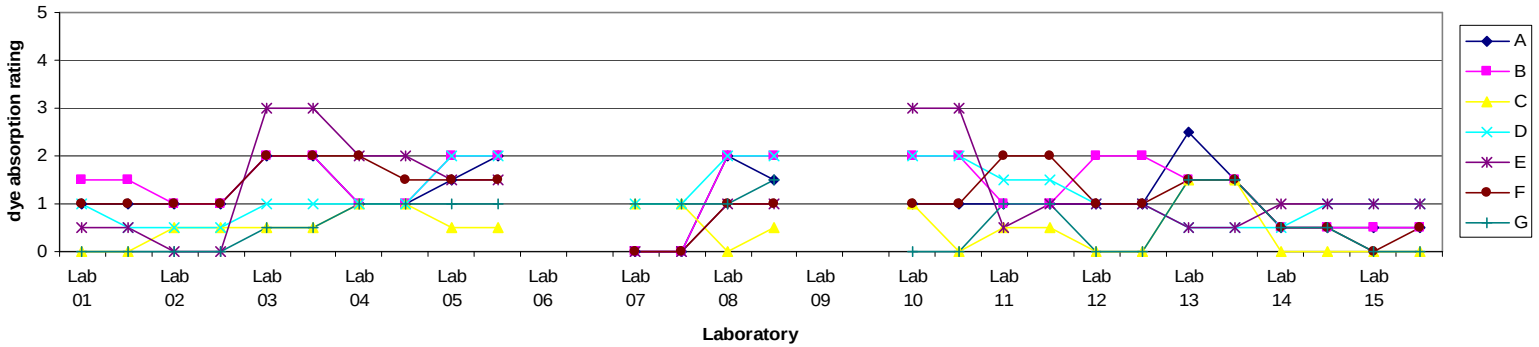
LABORATÓRIO NACIONAL DE ENGENHARIA CIVIL

# Results reported

## Test results of method EN ISO 2360



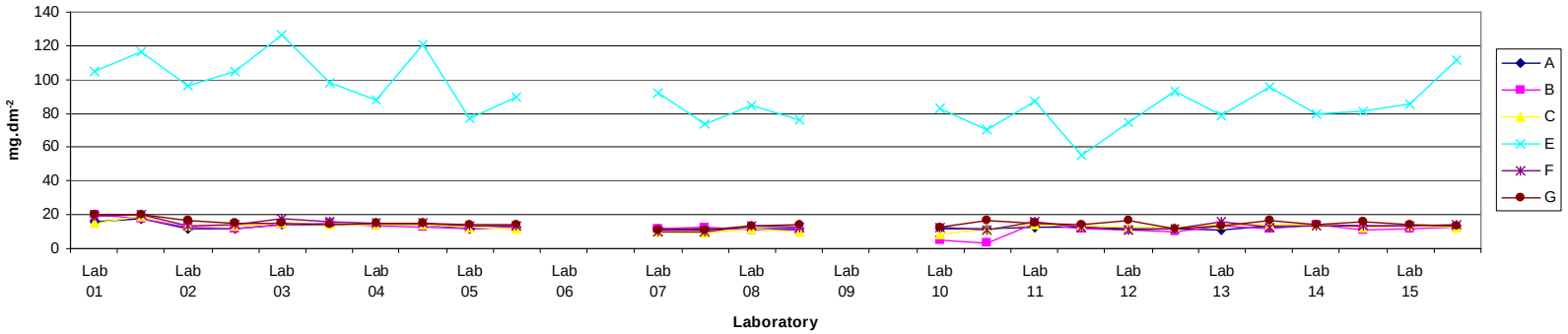

## Test results of method EN ISO 2143

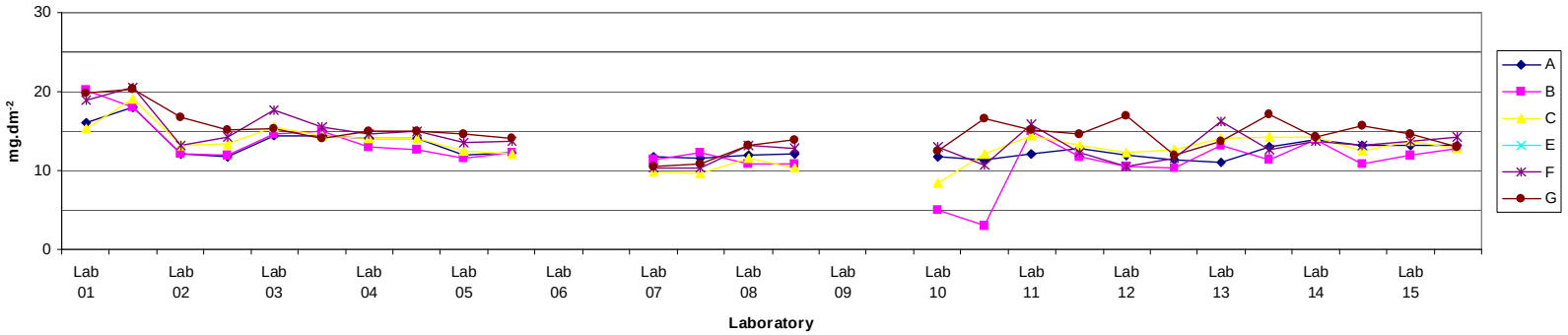


LABORATÓRIO NACIONAL DE ENGENHARIA CIVIL

# Results reported

Test results of method EN ISO 3210

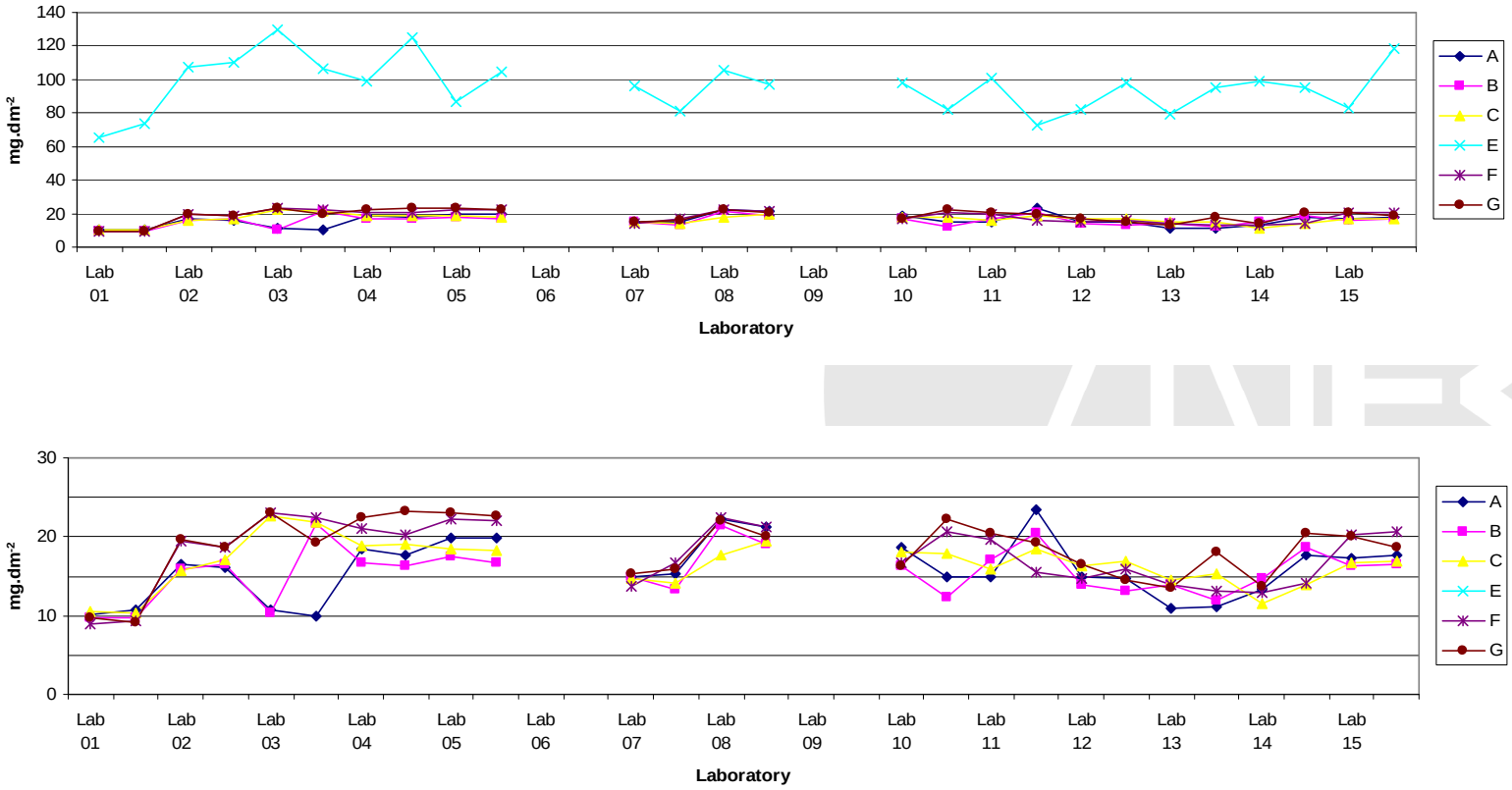



M. Salta, R. Fontinha, N. Garcia

# Results reported

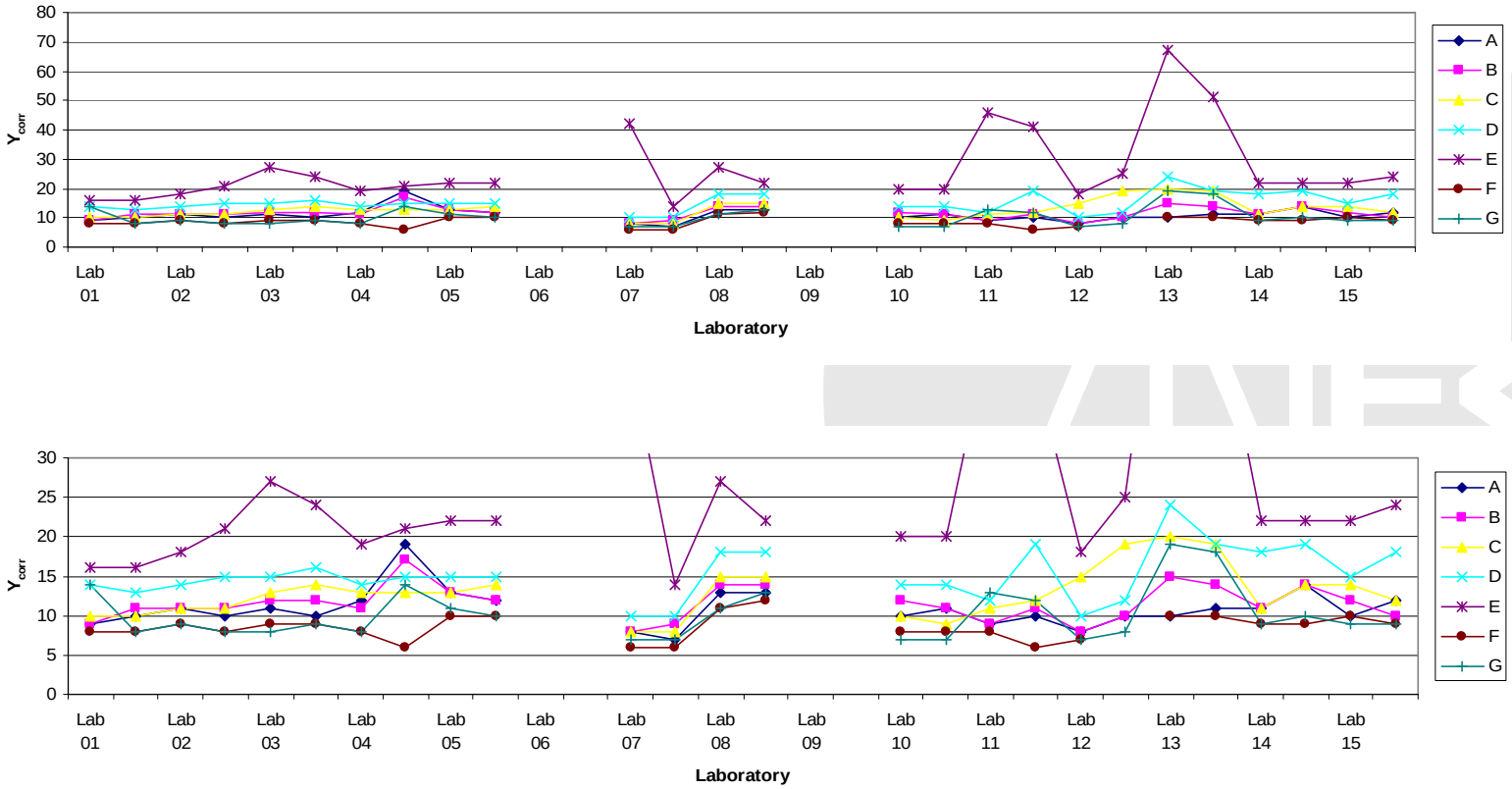
Test results of method Chromium free test




M. Salta, R. Fontinha, N. Garcia

# Results reported

Test results of method EN ISO 2931




M. Salta, R. Fontinha, N. Garcia

# 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*

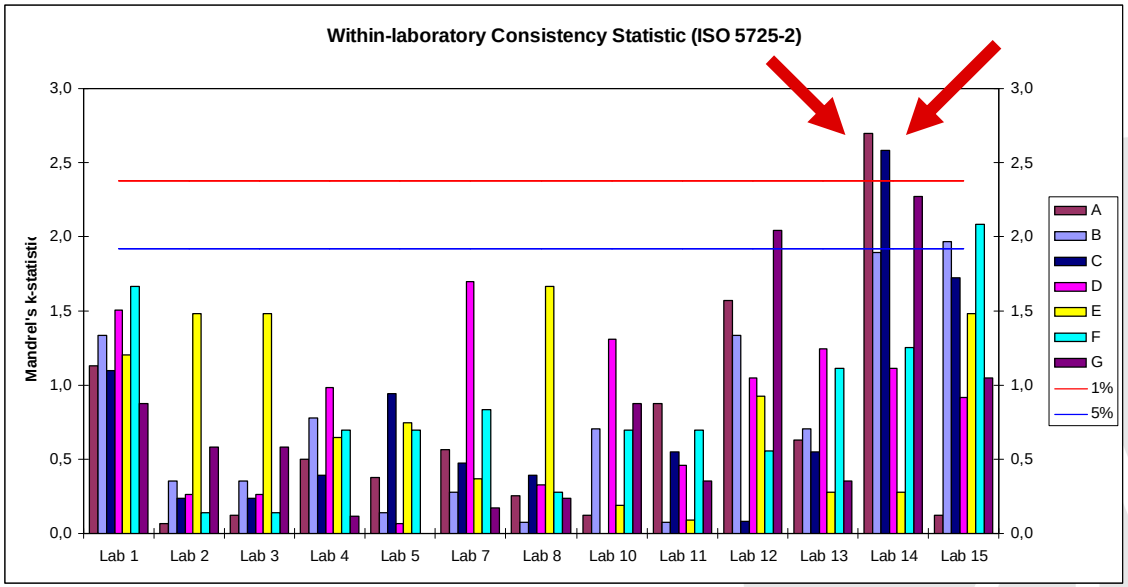
$$s_{Rj}^2 = s_{rj}^2 + s_{Lj}^2$$

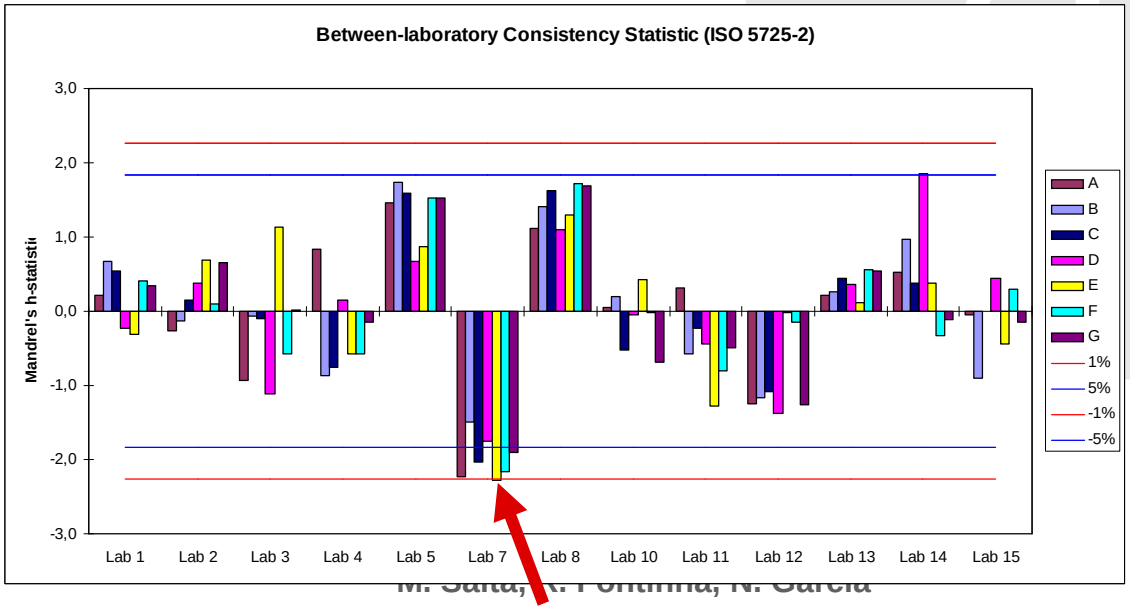
*Reproducibility variance*

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

LABORATÓRIO NACIONAL DE ENGENHARIA CIVIL

# Thickness measurement (EN ISO 2360)




M. Salta, K. Pontinha, N. García

# Thickness measurement (EN ISO 2360)

LABORATÓRIO NACIONAL DE ENGENHARIA CIVIL

## 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*** >$C_{Cr\ (1\%)}$) | - | - | - | - | - | - | - |

M. Salta, R. Fontinha, N. Garcia

LABORATÓRIO NACIONAL DE ENGENHARIA CIVIL

# 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

M. Salta, R. Fontinha, N. Garcia

# Thickness measurement (EN ISO 2360)

LABORATÓRIO NACIONAL DE ENGENHARIA CIVIL

## Results of precision analysis

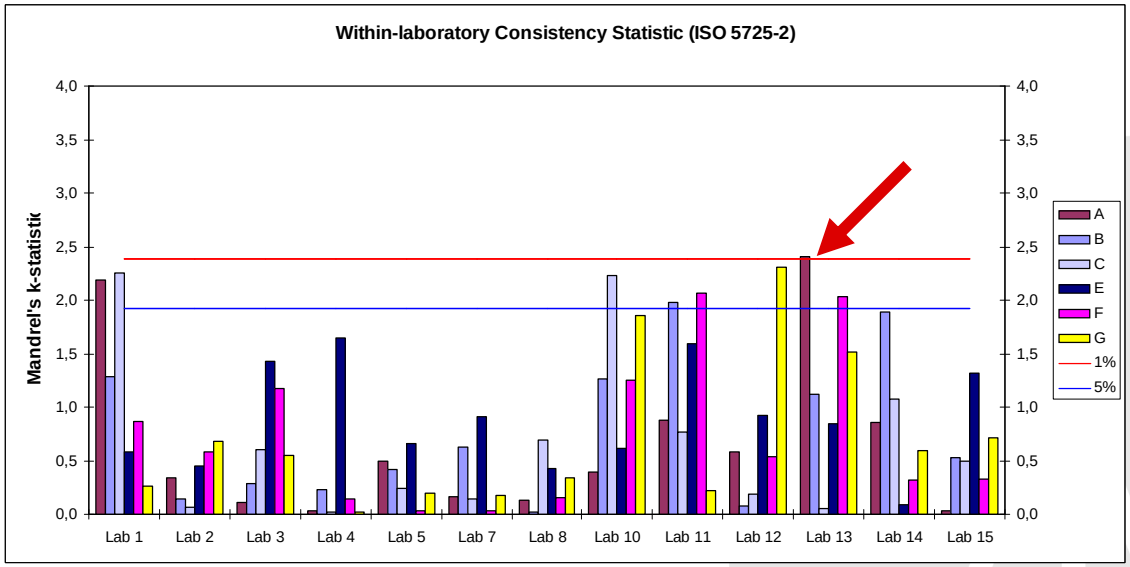
| Level | A | B | C | D | E | F | G |
|---|---|---|---|---|---|---|---|
| Number of replicates *n* | 2 | 2 | 2 | 2 | 2 | 2 | 2 |
| Valid laboratories *p* | 13 | 13 | 13 | 13 | 13 | 13 | 13 |
| **General mean *m* / µm** | **15,05** | **19,94** | **25,59** | **22,08** | **37,51** | **15,18** | **25,66** |
| 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 | - | - |

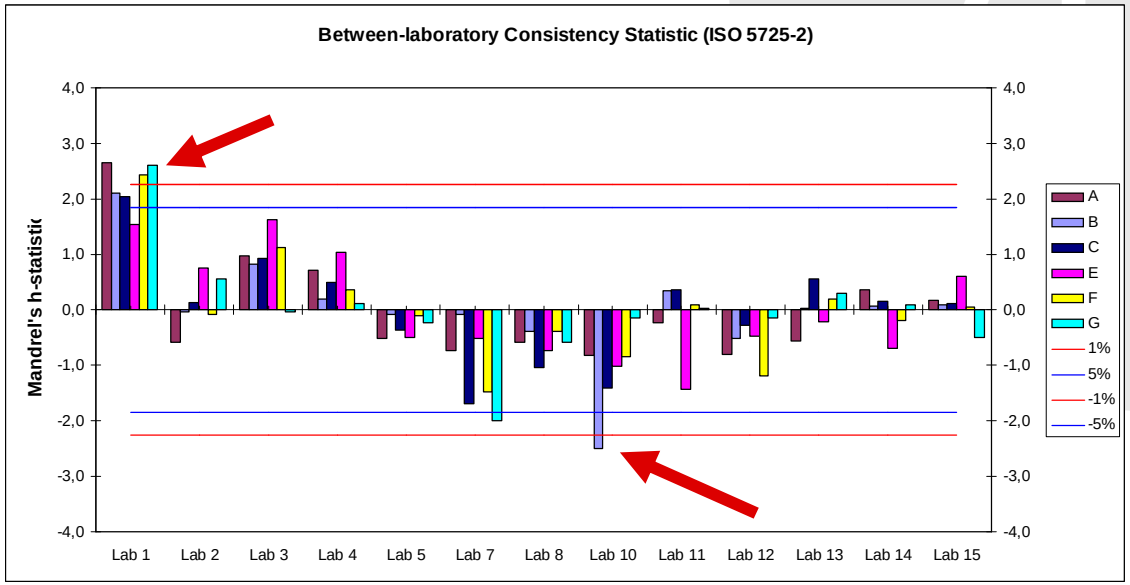
Outlier type: M*h* – Mandel's *h* ;M*k* – Mandel's *k* ; C - Cochran's ; G(I) – Grubs (one outlying observation) ; G(II) – Grubs (two outlying observations)

M. Salta, R. Fontinha, N. Garcia

LABORATÓRIO NACIONAL DE ENGENHARIA CIVIL

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




M. Salta, R. Fontinha, N. Garcia

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*** >$C_{Cr\ (1\%)}$) | - | - | - | - | - | - |

M. Salta, R. Fontinha, N. Garcia

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

**M. Salta, R. Fontinha, N. Garcia**

LABORATÓRIO NACIONAL DE ENGENHARIA CIVIL

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

## Results of precision analysis

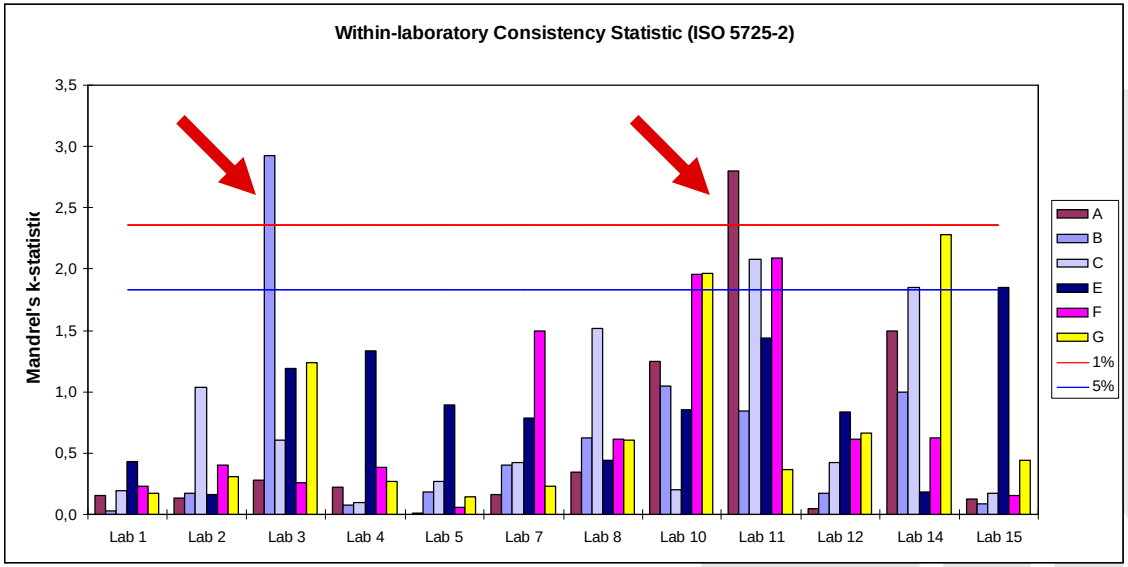
| Level | A | B | C | E | F | G |
|---|---|---|---|---|---|---|
| Number of replicates *n* | 2 | 2 | 2 | 2 | 2 | 2 |
| Valid laboratories *p* | 13 | 13 | 13 | 13 | 13 | 13 |
| **General mean *m* / g.dm$^{-2}$** | **12,92** | 12,19 | **13,10** | **90,23** | **13,93** | **14,83** |
| Repeatability variance $s_r^2$ | 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]<br>Lab 13 [2] | Lab 1 | - | - | Lab 1 | Lab 1 |

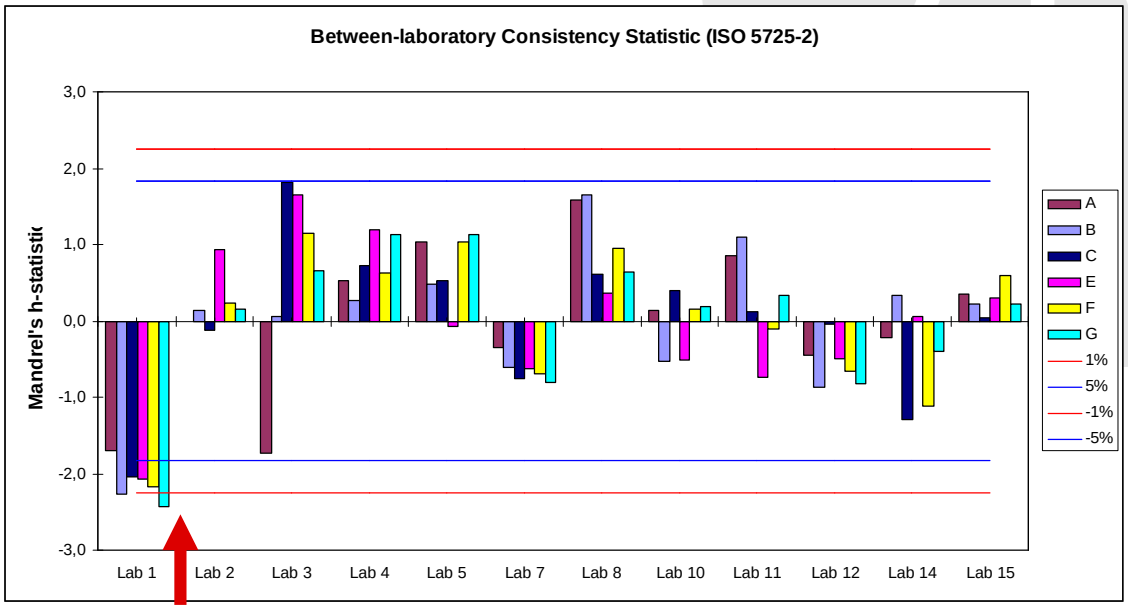
Outlier type: M*h* – Mandel's *h* ;M*k* – Mandel's *k* ; C - Cochran's ; G(I) – Grubs (one outlying observation) ; G(II) – Grubs (two outlying observations)

M. Salta, R. Fontinha, N. Garcia

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 | - | - | - | - |

M. Salta, R. Fontinha, N. Garcia

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

M. Salta, R. Fontinha, N. Garcia

LABORATÓRIO NACIONAL DE ENGENHARIA CIVIL

# Sealing quality assessment by mass loss (Chromium free)

## Results of precision analysis

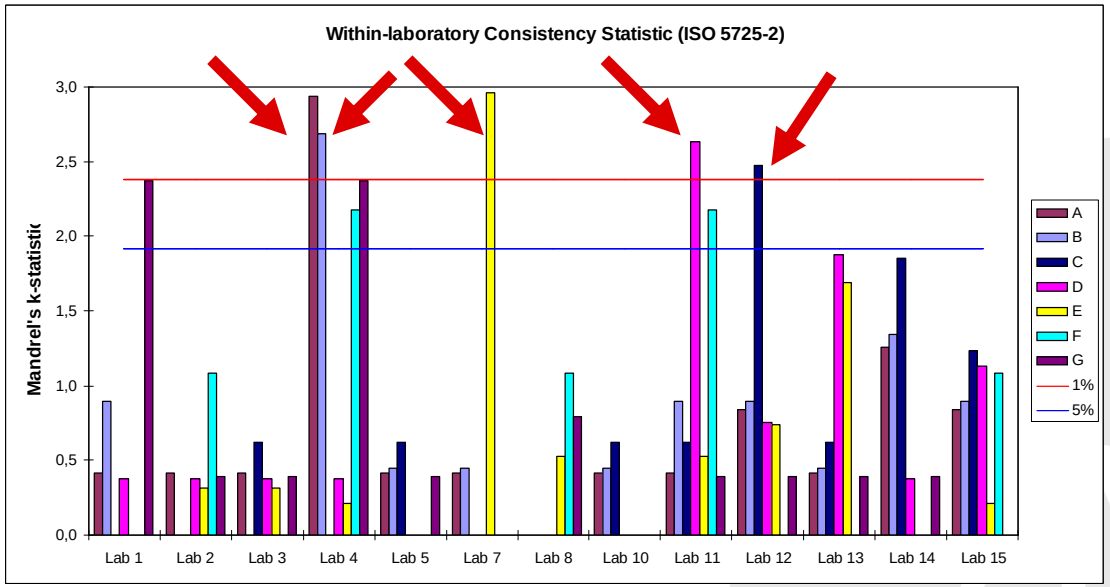
| Level | A | B | C | E | F | G |
|---|---|---|---|---|---|---|
| Number of replicates *n* | 2 | 2 | 2 | 2 | 2 | 2 |
| Valid laboratories *p* | 11 | 11 | 12 | 12 | 12 | 12 |
| **General mean *m* / g.dm$^{-2}$** | **16,00** | **15,79** | **16,74** | **96,57** | **18,02** | **18,61** |
| Repeatability 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 |

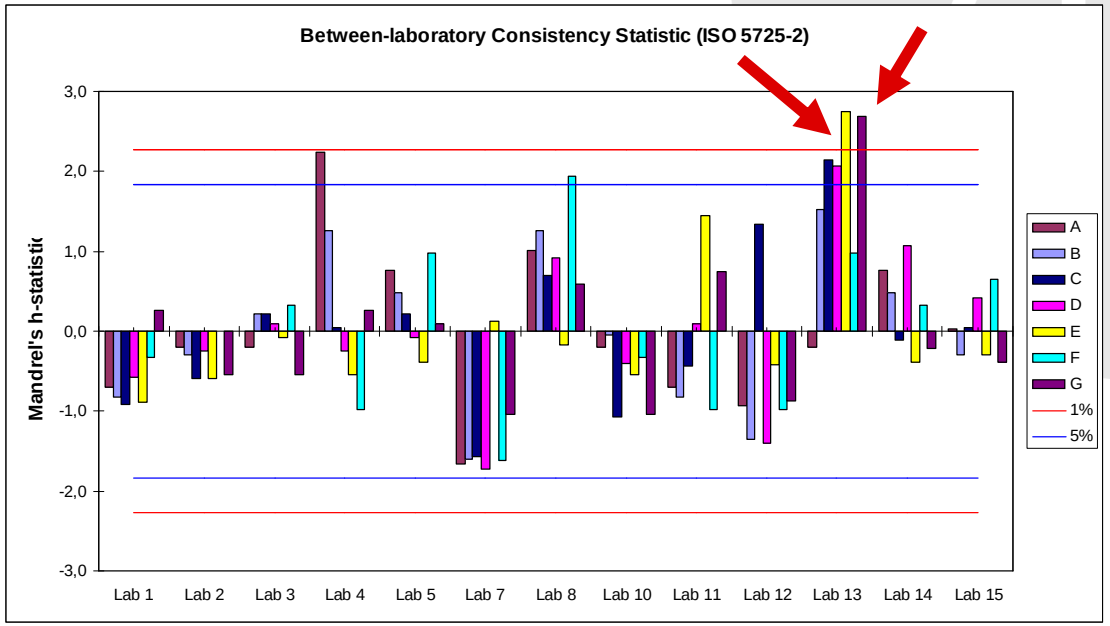
Outlier type: M*h* – Mandel's *h* ;M*k* – Mandel's *k* ; C - Cochran's ; G(I) – Grubs (one outlying observation) ; G(II) – Grubs (two outlying observations)

M. Salta, R. Fontinha, N. Garcia

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 | - | - |

M. Salta, R. Fontinha, N. Garcia

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

M. Salta, R. Fontinha, N. Garcia

LABORATÓRIO NACIONAL DE ENGENHARIA CIVIL

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

## Results of precision analysis

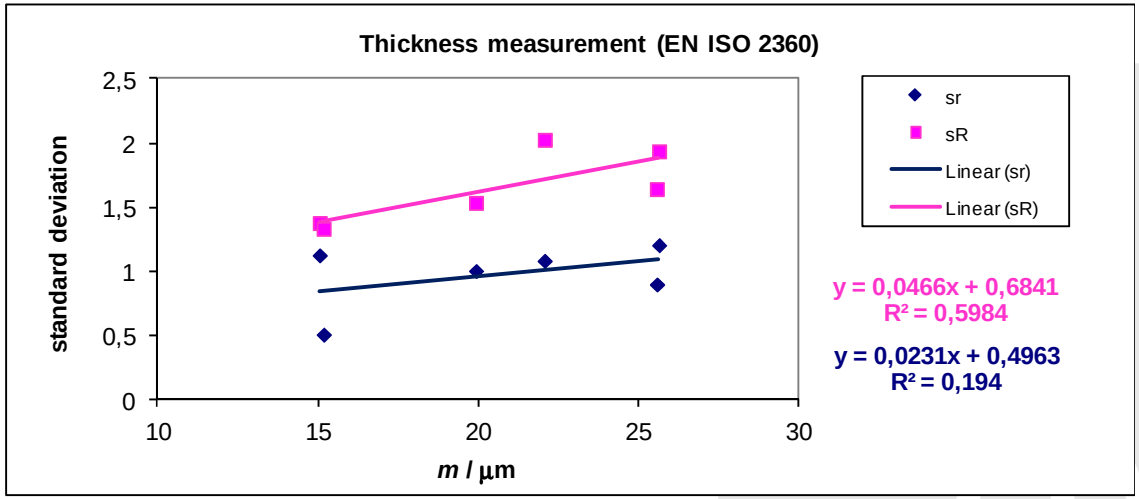
| Level | A | B | C | D | E | F | G |
|---|---|---|---|---|---|---|---|
| Number of replicates *n* | 2 | 2 | 2 | 2 | 2 | 2 | 2 |
| Valid laboratories *p* | 12 | 13 | 13 | 13 | 11 | 13 | 13 |
| **General mean *m* / Y** | **10,54** | **11,58** | **12,85** | **15,23** | **23,41** | **8,50** | **10,19** |
| 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 |

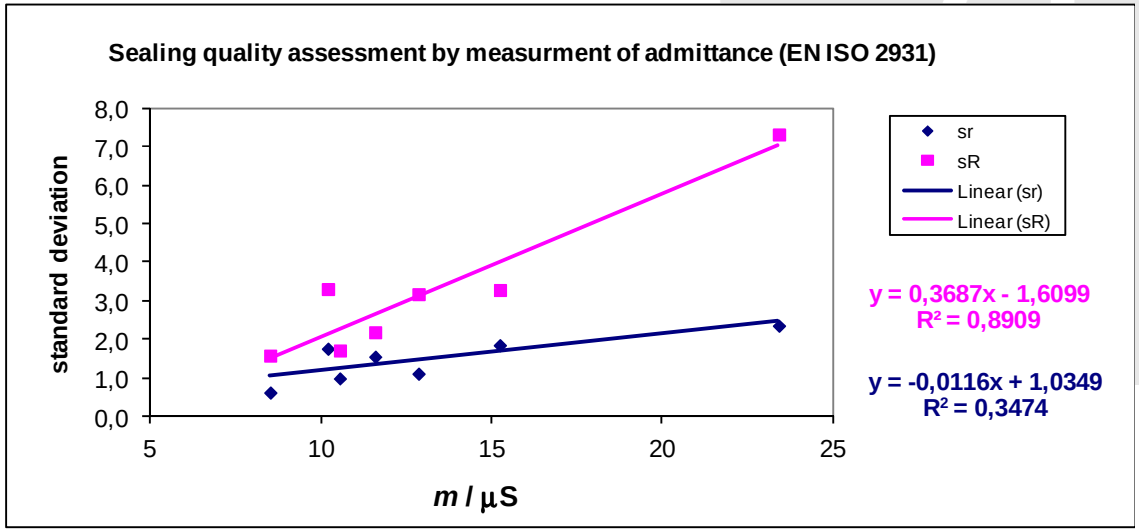
Outlier type: M*h* – Mandel's *h* ;M*k* – Mandel's *k* ; C - Cochran's ; G(I) – Grubs (one outlying observation) ; G(II) – Grubs (two outlying observations)

**M. Salta, R. Fontinha, N. Garcia**

# Dependency analysis of

## precision (repeatability and reproducibility) with the mean

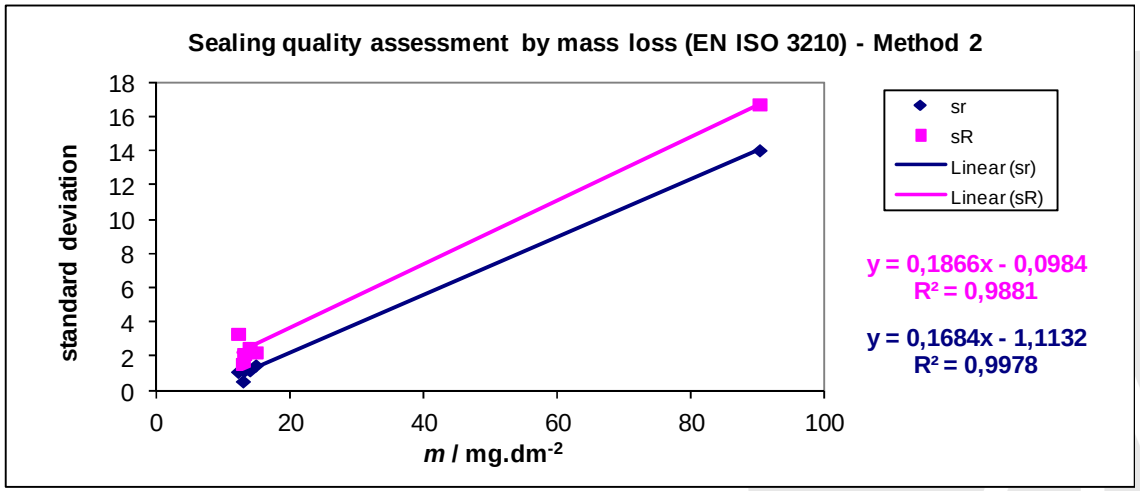


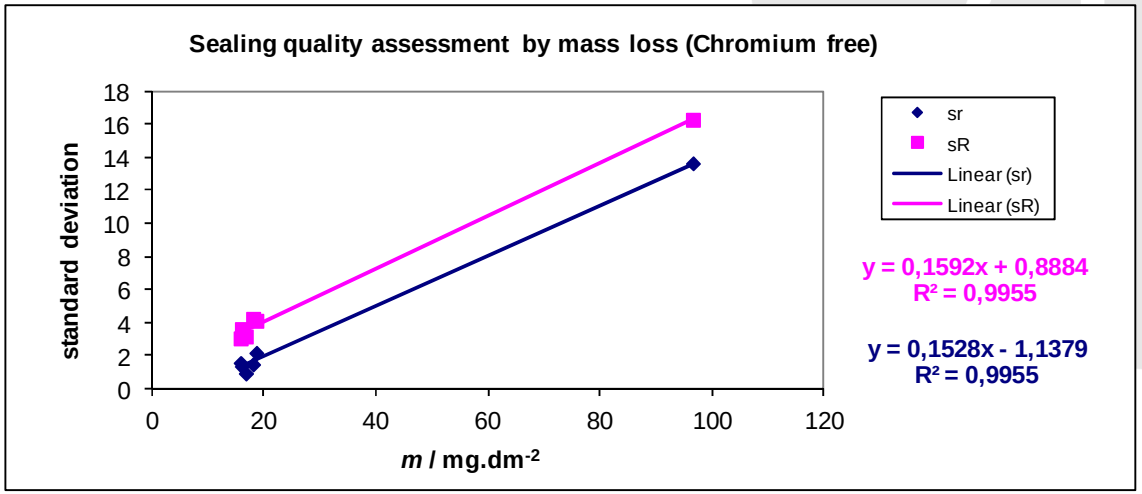




M. Salta, R. Fontinha, N. Garcia

# Dependency analysis of

## precision (repeatability and reproducibility) with the mean




M. Salta, R. Fontinha, N. Garcia

# 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<br>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<br>Lab 7: E<br>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

M.Salta, R. Fontinha, N. Garcia

# 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<br>Lab 4: B<br>Lab 8: C<br>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<br>Lab 7: B<br>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]<br>$S_R = 0{,}2137\ m - 1{,}421$ ($R^2 = 1$)[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*

M. Salta, R. Fontinha, N. Garcia

# Precision analysis results 2006

LABORATÓRIO NACIONAL DE ENGENHARIA CIVIL

| **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<br>Lab 8:I<br>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<br>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

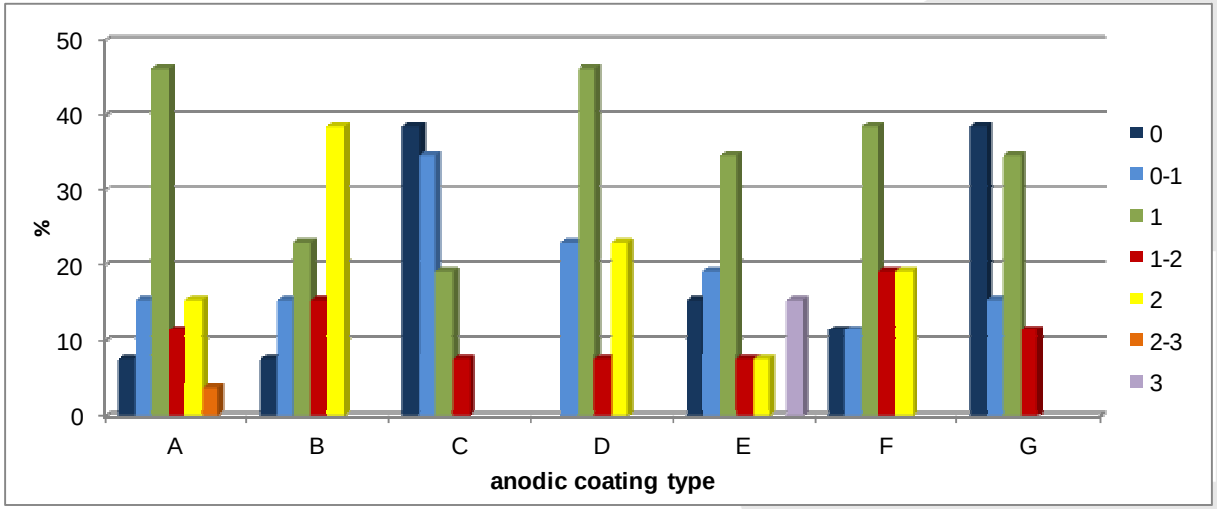
M. Salta, R. Fontinha, N. Garcia

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 |

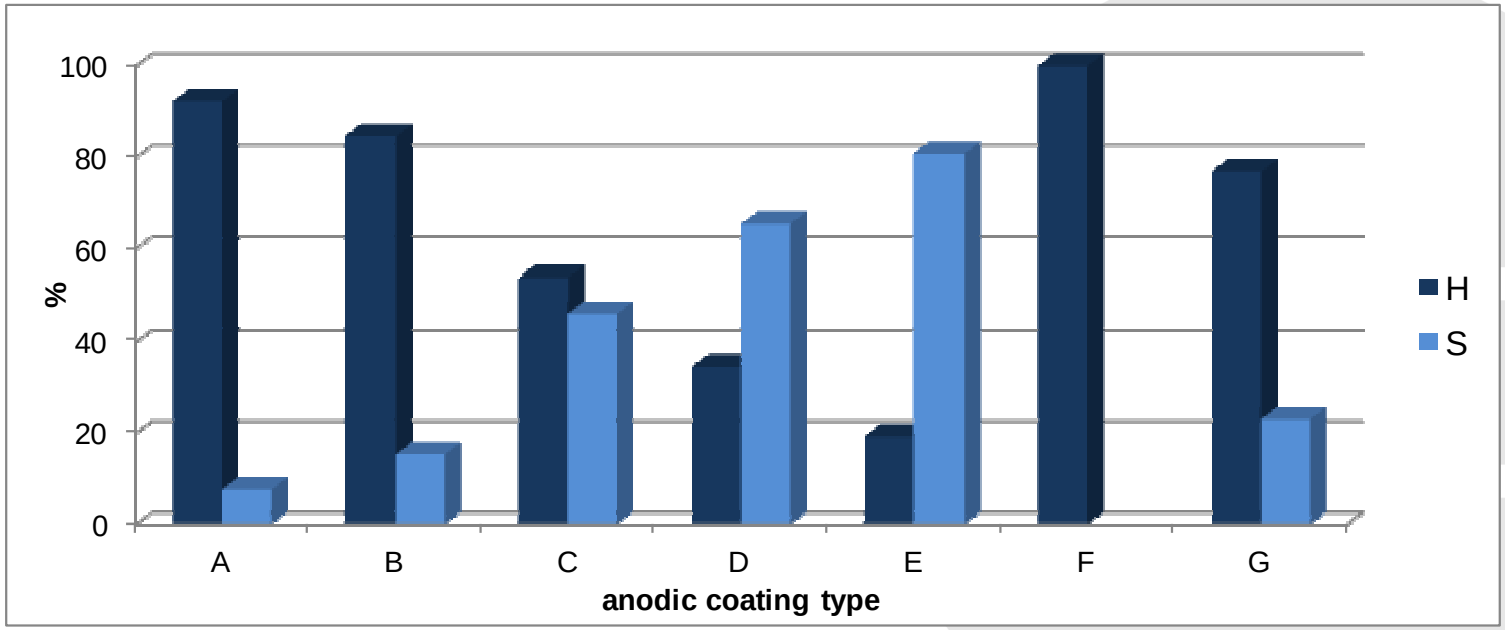
M. Salta, R. Fontinha, N. Garcia

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 |

M. Salta, R. Fontinha, N. Garcia

LABORATÓRIO NACIONAL DE ENGENHARIA CIVIL

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

LABORATÓRIO NACIONAL
DE ENGENHARIA CIVIL

M. Salta, R. Fontinha, N. Garcia