IGPG-03-13

3rd meeting of IGPG – 21 to 22 November 2011

Round Robin sand drop test:

Results and analysis

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Introduction

According to the 2nd meeting of IGPG, additionally to the Taber test other test methods for assessment of the abrasion resistance of plastic glazing should be investigated:

- Sand drop test

- Amtec-Kistler test (Car-wash test)

Points of interest:

Reproducibility of the test method
  - for different test samples of identical material within each laboratory
  - among all participating laboratories

Clarification of the test method
  - influences of test-method details

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Sand drop test

Standards:

- DIN 52348: "Testing of glass and plastics – Abrasion test – Sand trickling method"

- No equivalent ISO standard existing

- (EN 168: Personal eye-protection)

International application of sand drop test:

Within ECE-Regulation No. 22:

"Uniform provisions concerning the approval of protective helmets and their visors for drivers and passengers of motor cycles and mopeds"

for assessment of scratch resistance of helmet visors.

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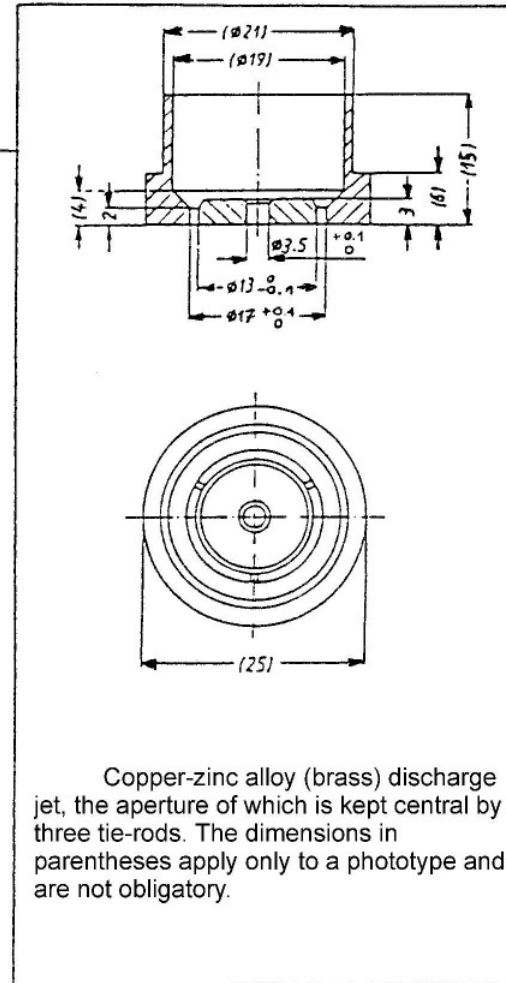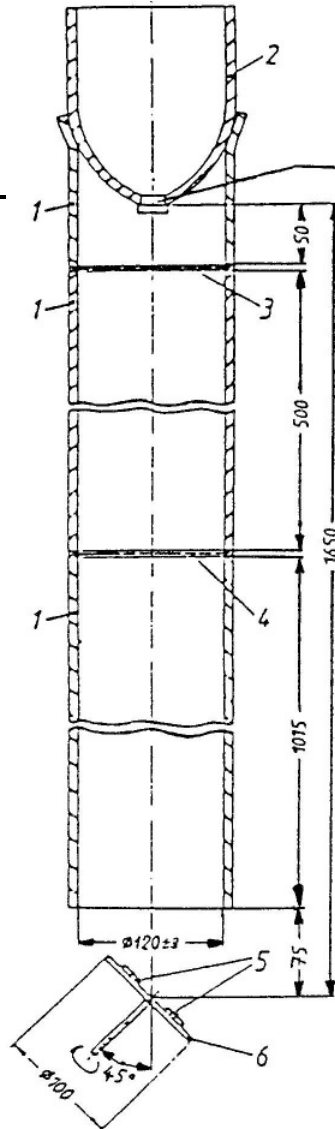PRÜFEN ▪ ÜBERWACHEN ▪ ZERTIFIZIEREN

Sand drop test

Basic test method:

- Abrasive particles: Three kilograms of natural quartz (silica) sand,
  grain size 0,5 / 0,7 mm, used up to ten times.

- The sample (flat or curved, min. 50 mm x 50 mm) is mounted on a turntable
  (250±10 rpm) which is inclined 45° to the vertical direction.

- Sand drops vertically through a gravity tube onto the sample (drop height 1650 mm).

- Depending on the sample size, more than one sample may be mounted on the
  turntable at the same time.

- Details of the sand drop equipment are given in ECE-R22 Annex 10, Figure 1.

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Test set-up

<u>ECE-R22 Annex 10, Fig.1</u>
Sand drop equipment

1.Gravity tube

2.Container w. discharge jet

3.Upper sieve

4.Lower sieve

5.Test samples

6.Turntable

Copper-zinc alloy (brass) discharge
jet, the aperture of which is kept central by
three tie-rods. The dimensions in
parentheses apply only to a phototype and
are not obligatory.

Dr. Matthias Dümmler,
21.11.2011

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Sand drop test

Modifications for Round Robin test:

- Three flat test pieces 50 mm x 50 mm of each type of glazing

- Conditioning (23±2)°C and (50±5)%rH for min. 48 hours (ref. ASTM D1044-08)

- Measurement of "initial haze" and "haze after abrasion" according to ECE-Reg. No. 43
  (According to ECE-R22, haze is measured alternatively as reduced light-density
  coefficient in cd/(m² lx) or as light diffusion in %.)

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Participating laboratories

- Three IGPG members

- Four further labs outside IGPG:

    - one vehicle manufacturer (via VDA)

    - three ECE-R22 Technical Services

    (two of them not able to measure haze according to ECE-R43)

Test samples

Each participating laboratory received three sample sets of following different materials, each set consisting of three samples:

- Monolithic float glass

- Polycarbonate (PC), hard-coated on both sides

- Polymethylmethacrylate (PMMA), hard-coated on one side

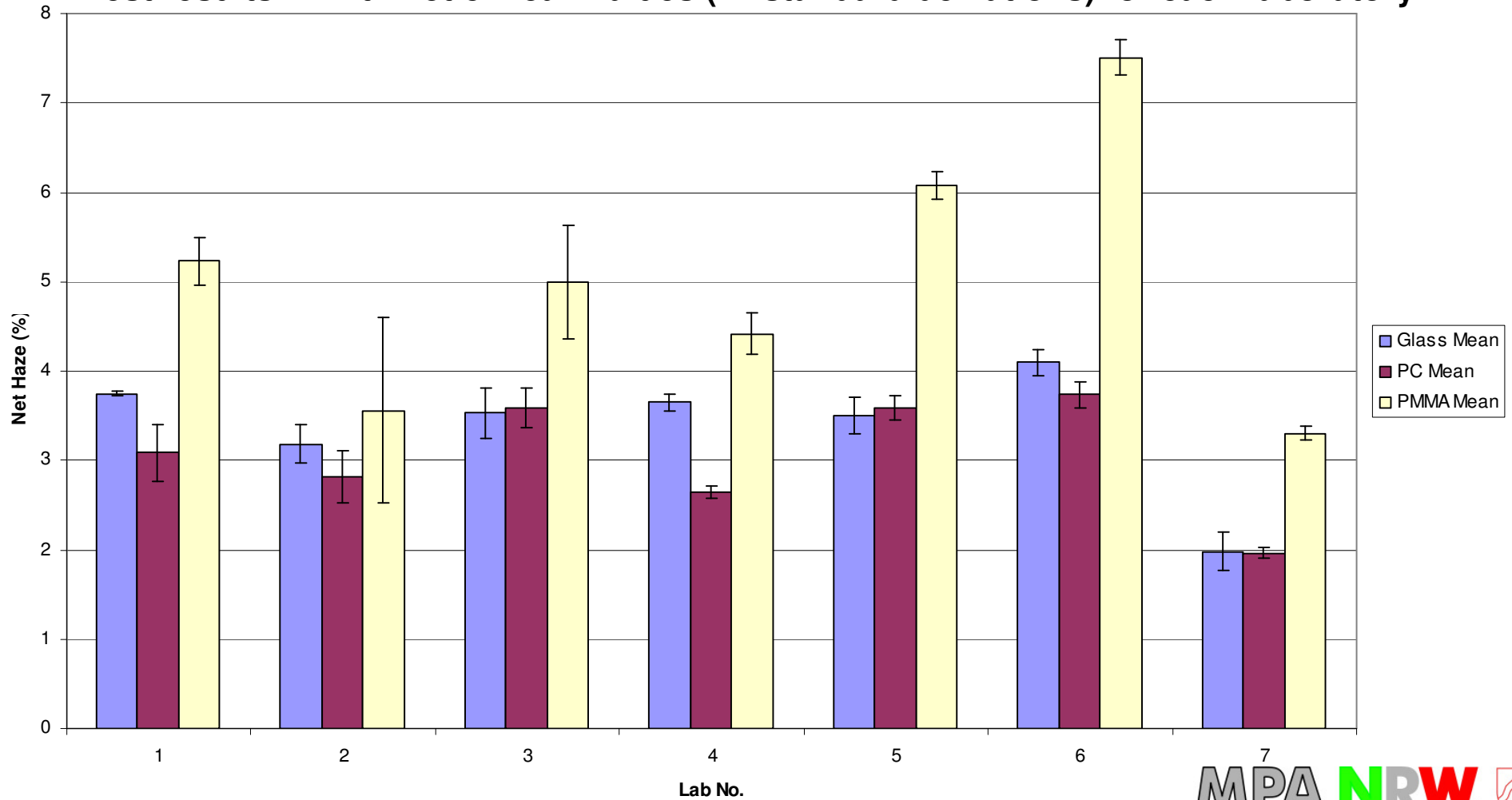**Sample delivery and control measurements**

Three IGPG members (producers of the different materials) delivered the test samples, two of them (glass, PC) measured "initial haze" before delivery and "haze after abrasion" after they had received the tested samples back (control measurements)
in order to get further information about the influence of the haze measurement on the total abrasion result.

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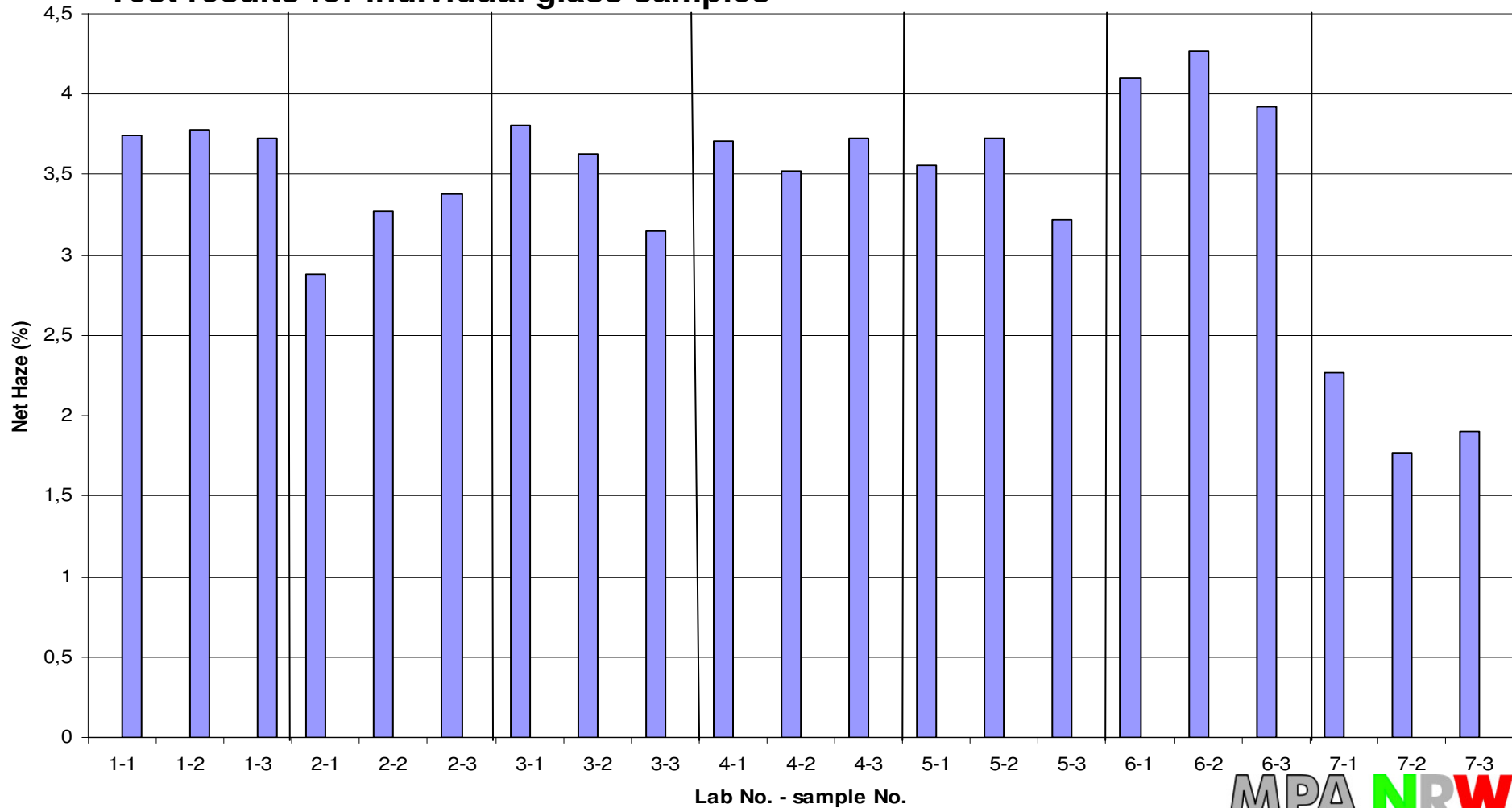**Further haze measurements**

Further haze measurements were carried out by the organizing laboratory, which distributed most sample sets among the participants, in order to get:

- haze values for two participating laboratories which are not able themselves to measure haze according to ECE-R43 (ECE-R22 labs)

- "control values" for PMMA

- further "control values" in case of two participating laboratories, which use Illuminant C (6774 K) instead of Illuminant A (2856 K) for haze measurement
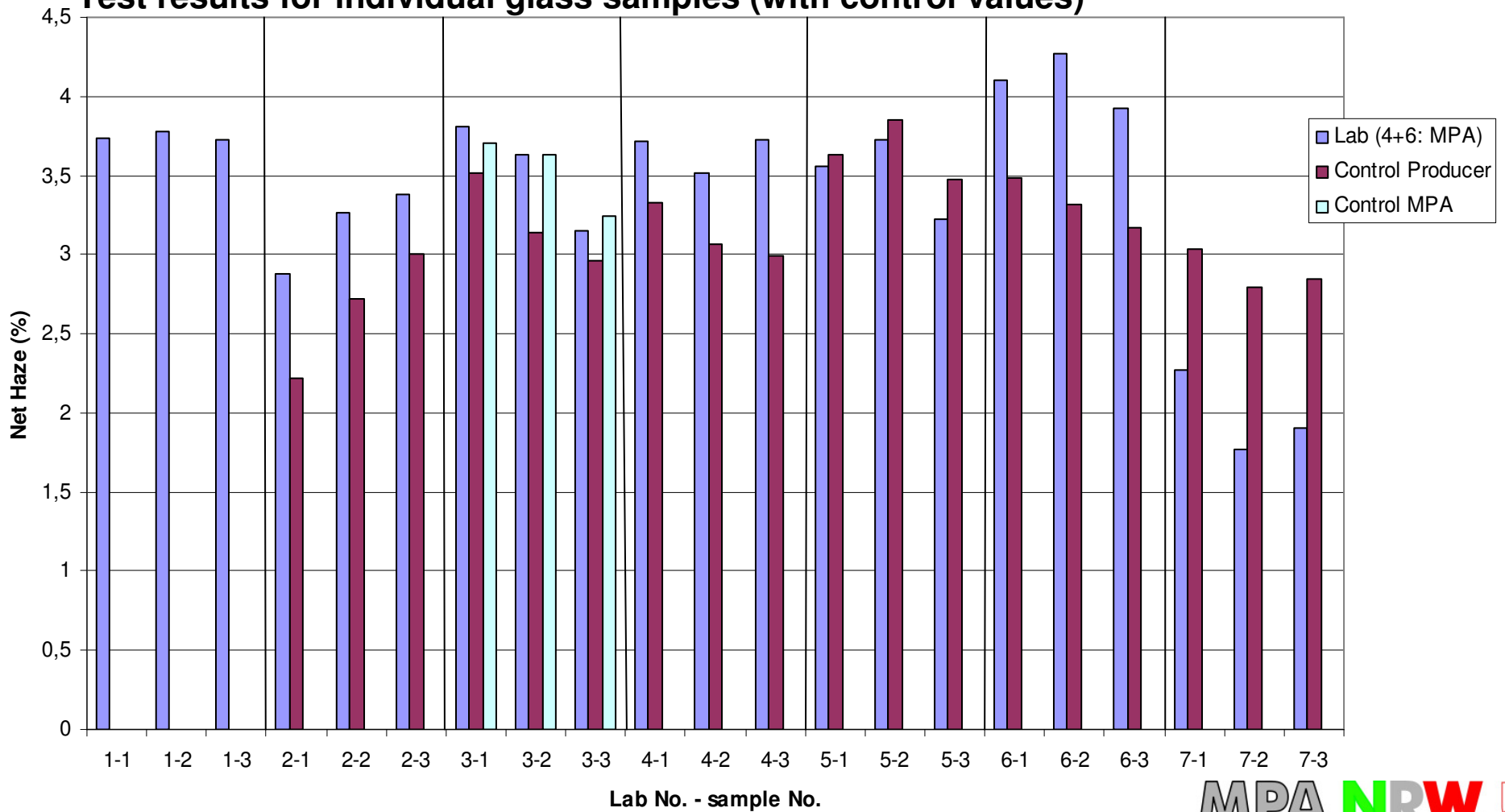
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Test results – Arithmetic mean values (w. standard deviations) for each laboratory

Test results for individual glass samples

Net Haze (%) vs Lab No. - sample No.

Test results for individual glass samples (with control values)

Legend:
- Lab (4+6: MPA)
- Control Producer
- Control MPA

Y-axis: Net Haze (%)

X-axis: Lab No. - sample No.

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Test results for individual PC samples
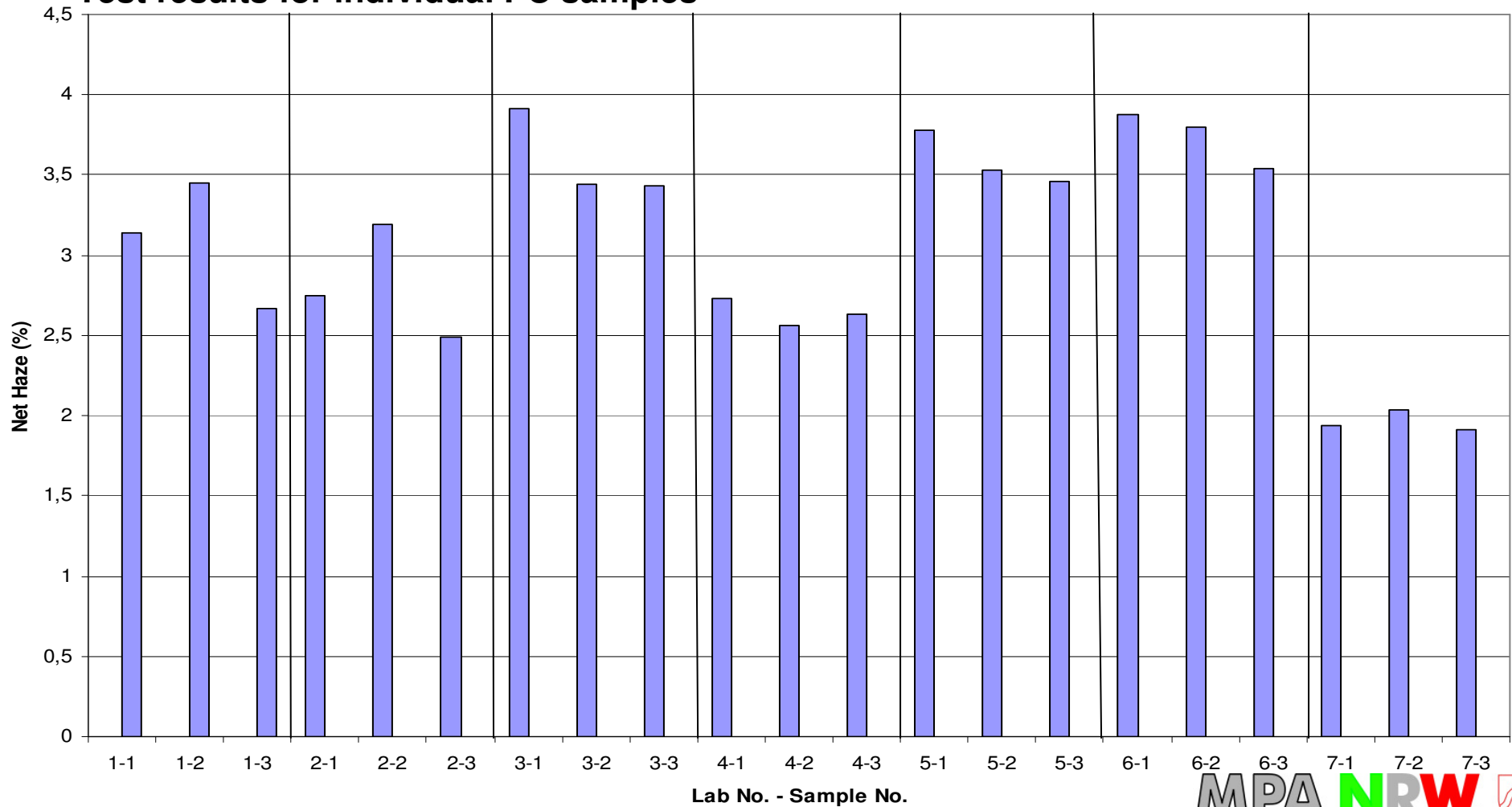
Test results for individual PC samples (with control values)

Legend:
- Lab (4+6: MPA)
- Control Producer
- Control MPA

Y-axis: Net Haze (%)

X-axis: Lab No. - Sample No.

Test results for individual PMMA samples

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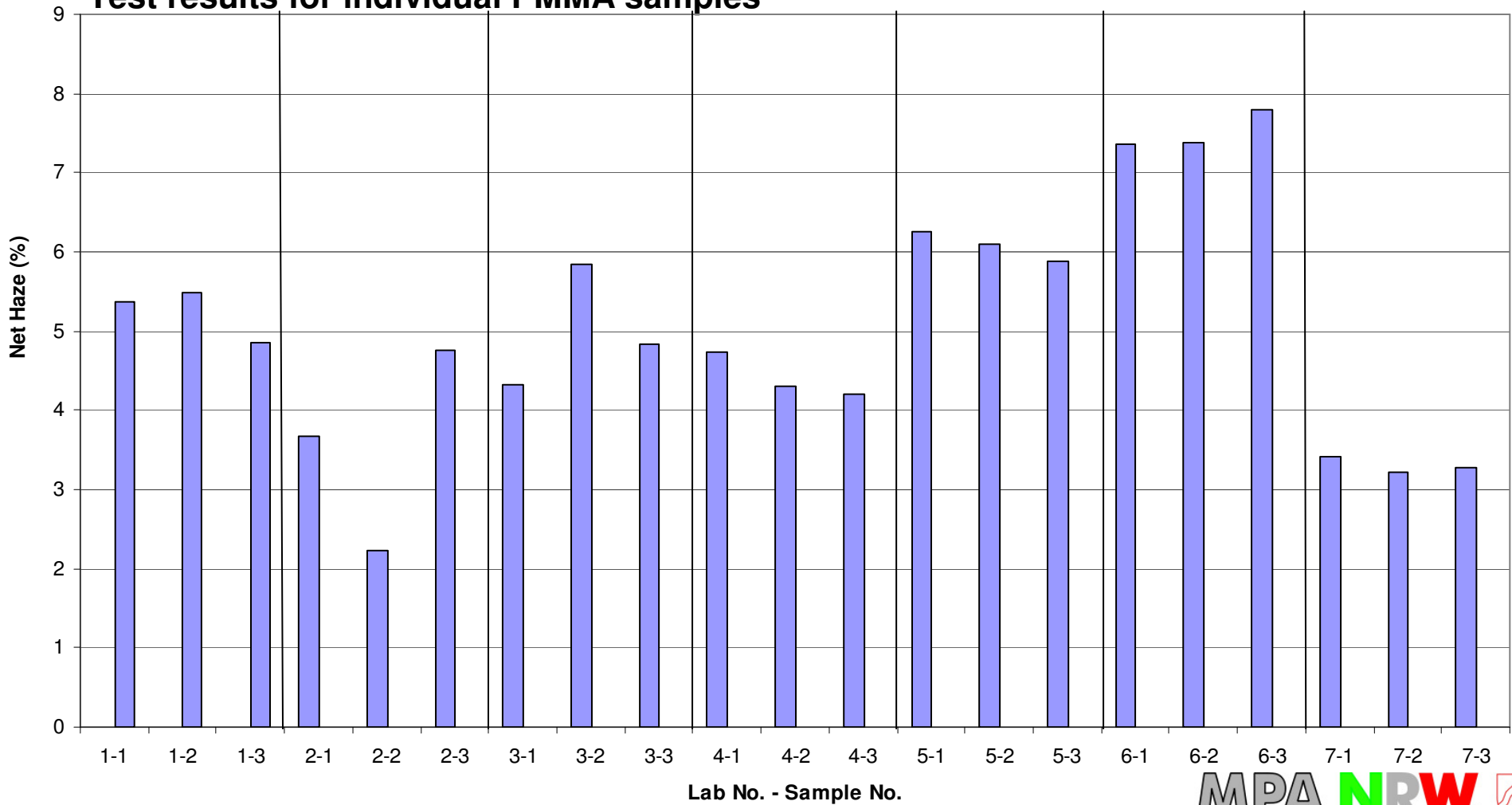
Test results for individual PMMA samples (with control values)
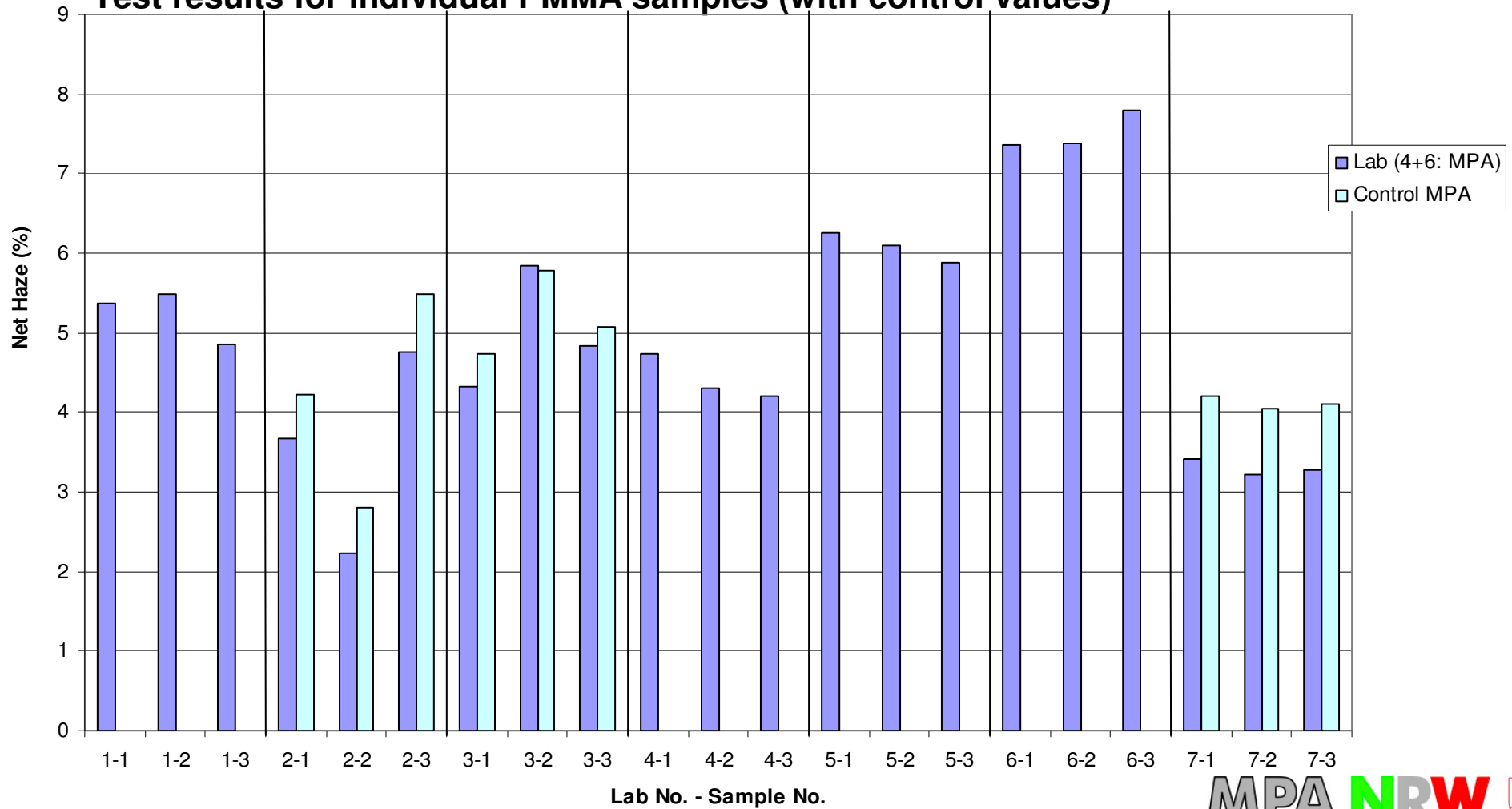
Evaluation of test results

Net haze (%)	Glass	PC	PMMA
Lowest measured value	1,77	1,91	2,23
Highest measured value	4,27	3,91	7,79

Statistical evaluation according to ISO 5725-2

"Accuracy (trueness and precision) of measurement methods and results – Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method"

<u>Definitions</u>:

Repeatability: Identical conditions (same lab, equipment, personnel,…)

Reproducibility : Different conditions (different labs, equipment, personnel,…)

$s_L{}^2$	Variance among labs
$s_W{}^2$	Variance within a lab
$s_r{}^2 = \text{mean } s_W{}^2$	Repeatability variance over all labs
$s_R{}^2 = s_L{}^2 + s_r{}^2$	Reproducibility variance

In the evaluation, these values are calculated separately for each characteristic investigated in the test (here: different materials - Glass, PC, PMMA).

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Statistical evaluation according to ISO 5725-2

Standard deviations are roots of the variances:

- Repeatability standard deviation s_r

- Reproducibility standard deviation s_R => suitable for evaluation of a Round Robin test.

Normal distribution of test results
=> 95% of the results lie within the interval   [Mean – 2x s_R , Mean + 2x s_R]

(P = 95% : Probability or Confidence interval)

Further definitions according to ISO 5725-2:

- Repeatability value $r = (2,8 \times s_r)$,

- Reproducibility value $R = (2,8 \times s_R)$

Max. absolute difference between two single results under repeatability resp. reproducibility conditions (P = 95%).

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Evaluation according to ISO 5725-2 (all lab results taken into account)

	Glass	PC	PMMA
Number of participating labs	7	7	7
Number of labs not eliminated	7	7	7
Number of individual values of not eliminated labs	21	21	21
Mean value of net haze, in % haze	**3,38**	**3,06**	**5,01**
Repeatability standard deviation s_r, in % haze	0,19	0,20	0,49
Repeatability value r = $(2,8 \times s_r)$, in % haze	0,52	0,56	1,37
Reproducibility standard deviation s_R , in % haze	**0,70**	**0,66**	**1,52**
Reproducibility value R = $(2,8 \times s_R)$, in % haze	1,95	1,85	4,24

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Evaluation according to ISO 5725-2

Remember:
95% of the results lie within the interval   [Mean – 2x s_R , Mean + 2x s_R],
where s_R is the reproducibility standard deviation.

$\Rightarrow$ For glass, results lie within interval   1,99 - 4,78 % haze

$\Rightarrow$ For PC, results lie within interval   1,74 - 4,39 % haze

$\Rightarrow$ For PMMA, results lie within interval   1,98 - 8,04 % haze

with a probability of 95%!

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Potential sources of influence on test results:

Influence of cycle number of sand use

The sand may be used up to ten times (10 cycles). Max. four samples can be fixed on the turntable at the same time.

The test laboratories used sand for all the nine individual samples differently, as follows:

mat. 1	mat. 2	mat. 3
1-1-1	2-2-2	3-3-3
1-2-3	1-2-3	1-2-3
1-4-7	2-5-8	3-6-9

and variations of these combinations.

Example: "1-4-7" means that for one sample set (e.g. material 1) consisting of three samples, sample No. 1 was impacted with sand used for the first time (= cycle No. 1), sample No. 2 with sand used for the fourth time (= cycle No. 4), and sample No. 3 with sand used for the seventh time (= cycle No. 7).

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Influence of cycle number of sand use

If the sand cycle influences haze, the results should show that:

- The individual haze values should be higher for "new" sand (sharper grains) and lower for "used" sand (grains getting blunt with each further cycle of use)
 => e.g. "1-4-7" should result in haze values decreasing from samples No. 1 to No. 3.

- The standard deviation within a sample set should be smaller in case of same sand cycle for all samples (1-1-1,…) and larger for different sand cycles (1-2-3, 1-4-7,…).

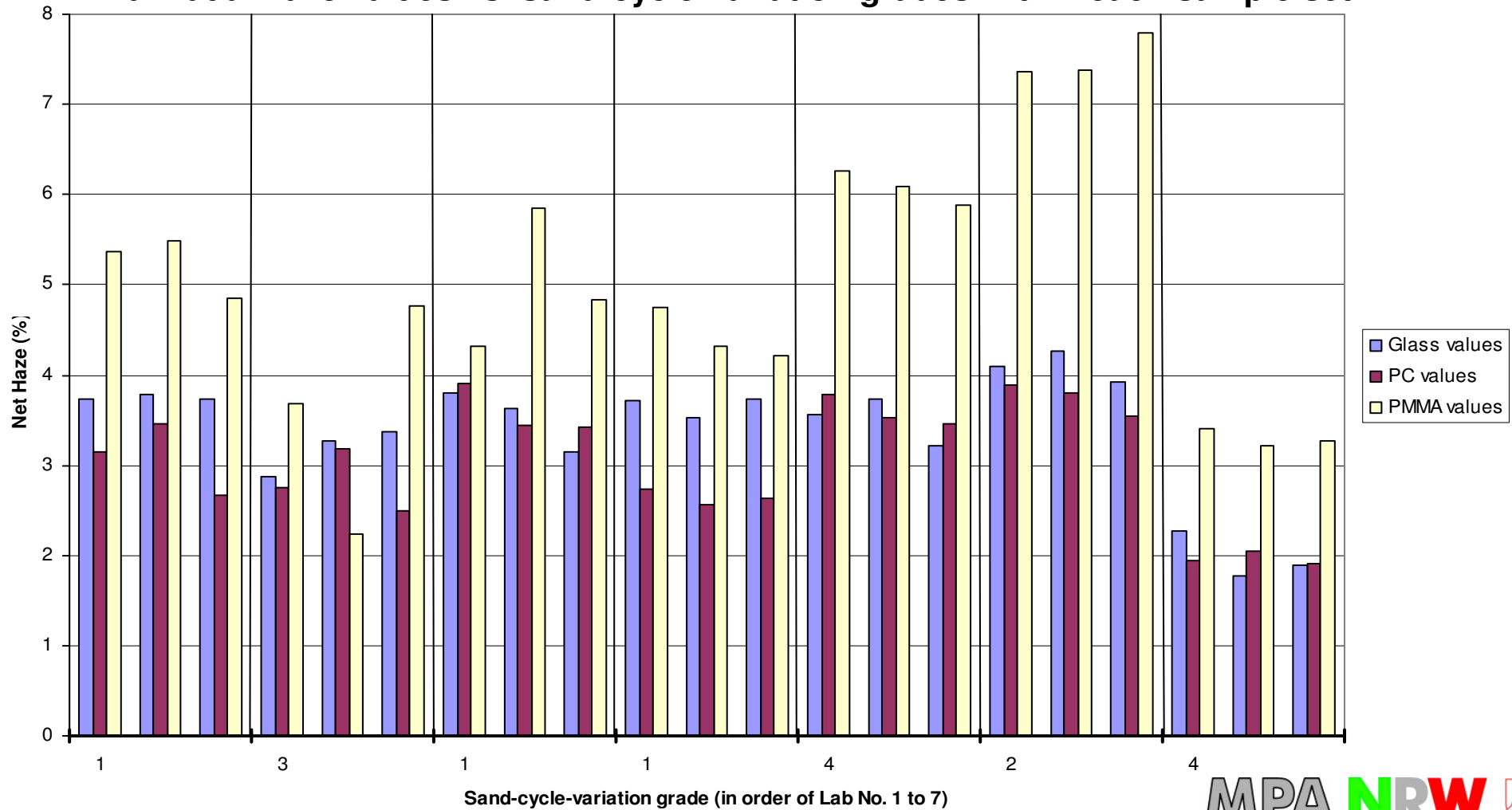Definition of grades of variation of sand cycle Nos. within each sample set:

Grade 1: 1-1-1, 2-2-2,… = same sand cycle for all samples of a set
Grade 2: 1-2-2, 2-2-3,… = partly different sand cycles within a set
Grade 3: 1-2-3, 1-2-3,… = completely different sand cycles within a set (successively)
Grade 4: 1-4-7, 2-5-8,… = compl. different sand cycles within a set (not successively)

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Individual haze values vs. sand-cycle-variation grades within each sample set

Standard deviations vs. sand-cycle-variation grades within each sample set
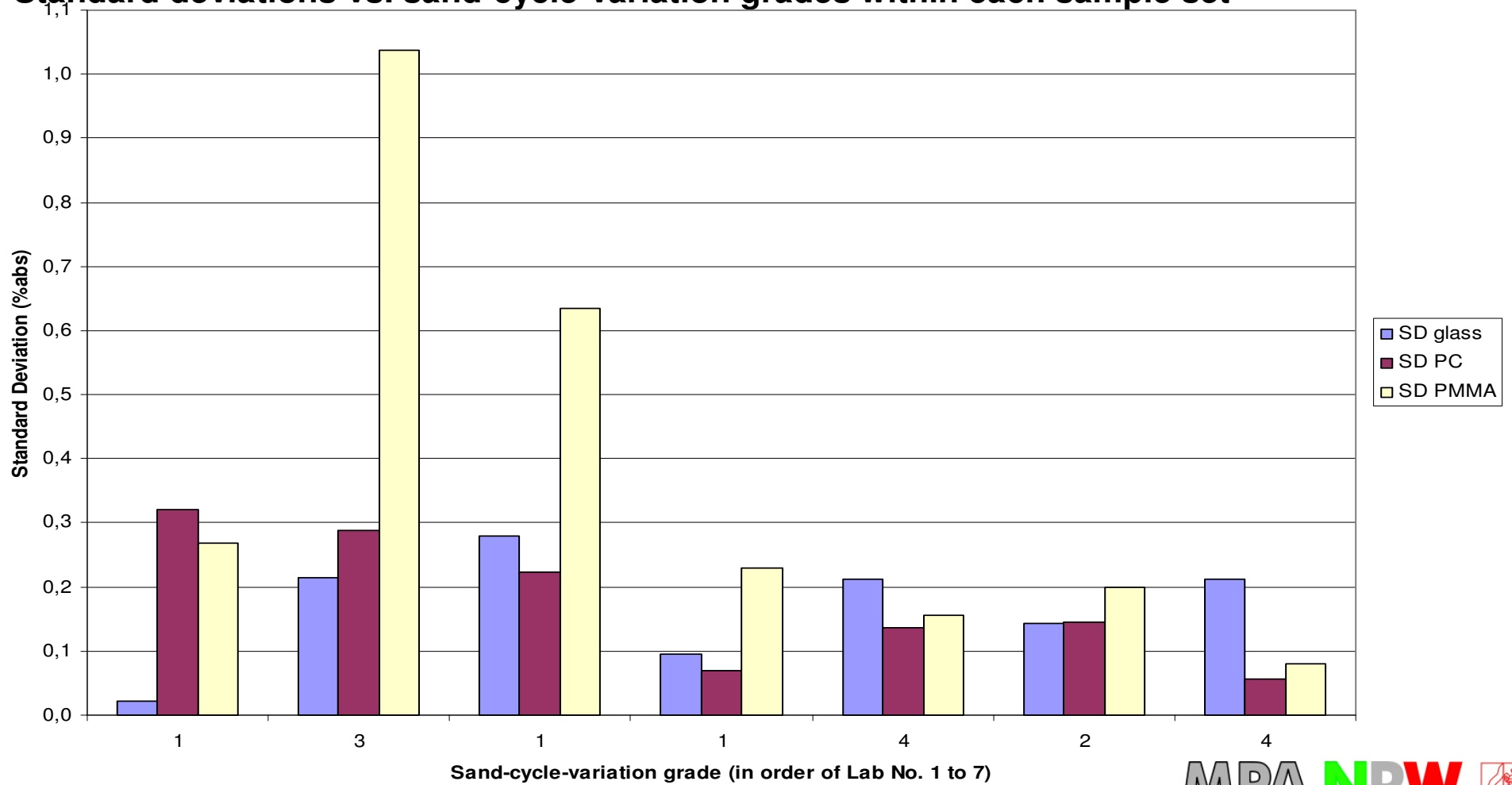
Potential sources of influence on test results

- <u>Haze measurement</u>
  - Different laboratories   => Variations observed
  - Illuminant A vs. Illuminant C   => No significant influence determined

- <u>Cycle number of sand use</u> (max. 10 cycles)   => No significant influence determined

- <u>Sand supplier</u>:
  Five of seven laboratories used same supplier
  (Busch Quarz, Schnaittenbach, Germany)   => No significant influence expected

- <u>Sample cleaning</u>
  - Type of detergent:
    All laboratories used a solution of 1% surfactants in water
    (e.g. commercial dish-washing detergent)   => No significant influence expected
  - Intensity of cleaning   => Indications of possible influence
    observed

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Summary

- Significant variations among individual samples of identical material per laboratory observed.

- Significant variations of mean values among all laboratories observed.

- The haze measurement may be a source of variations.

- The cleaning procedure may be a source of variations.

Statistical evaluation:

For glass, results lie within interval   1,99 - 4,78 % haze   (mean value 3,38 % haze),

for PC, results lie within interval   1,74 - 4,39 % haze   (mean value 3,06 % haze),

for PMMA, results lie within interval   1,98 - 8,04 % haze   (mean value 5,01 % haze)

with a probability of 95%.

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Conclusions

The reproducibility of the sand drop test (ECE-R22 modified) may be acceptable for glass and PC, but seems to be too bad for PMMA for approval testing of safety glazing materials according to ECE-R43.
The detailed assessment in comparison to other abrasion test methods should be done by comparing the statistical Round Robin results (means, reproducibility).

Potential steps for improvement:

- Reference materials to be defined (not included in ECE-R22, but in DIN 52348)
  (recommended also for other abrasion test methods).

- Cleaning procedure to be repeated until haze values do no more vary significantly
  (e.g. 10%rel).

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