International Association for Soaps, Detergents and Maintenance Products

A.I.S.E. Laundry Detergent Testing Guidelines

Minimum requirements for comparative detergents testing

v.7. – October 2020

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IMPORTANT NOTICE

The users of these Guidelines shall only quote the A.I.S.E. Guidelines as and when the test has been run fully in line with the A.I.S.E. Guidelines and the fixed 14 stain set.

These guidelines are for comparative performance testing of detergents, NOT washing conditions. This means that identical wash conditions are to be used for all detergents tested.

A.I.S.E. Working Group “Laundry Detergent Testing”

Test Protocol

- Principle based
- Includes **minimum** requirements – free to exceed those but not lower them
- Can be adapted to different countries/regions, differences in wash habits, wash cycles and temperatures, recommended dosages, etc.*

Scope

- Logistics: communication, selection, pick-up, sampling
- Test execution
- Results: calculation, evaluation, communication

Categories

- Heavy Duty Detergents, Light Duty Detergents, Laundry Additives

Countries

- Should apply to all A.I.S.E. member countries

Benefits

- More realistic and more reliable comparison of product qualities for consumers
- Improvement in test quality
- Common approach

Main Topics of the Guidelines

- Information about start of test
- Quality assurance in test lab
- Washing machine, cycle, temperature
- Number of replicates
- Dosage
- Soil Donator
- Stain Set
- Dyes for dye transfer inhibition *
- Dyes for color maintenance
- Test execution
- Statistical evaluation
- Communication of results prior to publication
- Future improvements of protocol

*** further development ongoing**

Information/Communication prior to testing

ISO/IEC GUIDE 46-1985

"Every reasonable effort should be made to reflect what is available on the market …"

This could include consulting manufacturers or checking current literature and catalogues.

It is in the interest of consumers and manufacturers that such information should be made

available.

(excerpt from ISO/IEC GUIDE 46-1985 section 2.1)

Process followed for Test Protocol

Review existing test protocols in Western Europe, e.g.

- Que Choisir, StiWa, Which? Consumentenbond, Test Achat, AFISE
- Terpstra soil workshop

Review and sharing of internal company data

- on consumer habits (stains/soils, wash temperature)
- textile/dye markets/trends
- internal & external testing experience

Develop “consensus” proposal

Key Learnings and Principles (1/2)

- Only a representative set of stains can give a consumer relevant assessment of a product's performance – drive towards use of stains that represent consumer problems in the most realistic way.

- Stains should be commercially available.

- Include (body) soil source since testing WITHOUT soil can lead to seriously misleading results as (body) soil has a significant impact on the absolute level of performance and can also impact the relative performance and ranking of products (ref. Soil Workshop Wageningen).

Key Learnings and Principles (2/2)

- Select most consumer/market relevant dyes for Dye Transfer and Color Maintenance.
- Select the most consumer relevant wash temperature for the product categories that are tested.
- For additives, select detergents as “base” that are representative of additives user habits.

Quality Assurance

- Reliable washing machines, representative for local market
- Fuzzy logic disabled to ensure equal program length/rinse cycles for all test products (to avoid suds interference with washing cycle length)
- Calibration of machines and yearly validation
- Purchase of stains and other test materials from same production lots
- Adherence to expiry date/recommended storage conditions
- NO drying of test fabrics in tumble dryer and ensure constant exposure conditions to light

Washing Cycle and Temperature

- Select the washing cycle and temperature that are most relevant in the country.
- Different washing cycles/temperatures may be used if justified by product category.
- In most countries this is 40°C for generalist detergents.

Dosage/Usage

- **Dosages should be based on manufacturers' recommendation:**
  - Generalists: normal soil/medium water hardness.
  - Additives: a harmonized dosage (when not clear from the instructions); mechanical action (rubbing) and exposure time should be selected according to test objective
- **In case the manufacturers' dosage instructions lead to large differences between products, it should be highlighted.**
- **Dosage of powders is according to the <u>declared</u> grams on the package. For liquids it is ml.**

Soil

- **Addition of 4 SBL 2004 sheets, introducing about 32 grams of ballast soil.**

- **SBL 2004 is an improved version of SBL (with increased sebum/oil load and a “bleach consuming agent”) and is currently the best option to simulate normally soiled laundry (in the absence of normally soiled consumer bundles or wash & wear tests).**

Stain Set – Principles

- A representative set of stains should typically comprise a minimum of about 15 stains. Size of stain should permit accurate reading (minimum diameter about 50 mm).
- The total amount of soil coming from ballast soil and stains should be consistent with consumer habits.
- Stains should cover all consumer relevant stain categories for the products that are tested.
- Stains should be produced in a reproducible quality.
- Select suitable stain candidates from available suppliers (Warwick Equest, WFK, EMPA, CFT) - use mix of “natural” and “standard” stains.
- Remove stains with artifacts e.g. high T aging or presence of foreign pigments/carbon black, soot etc. which can alter the chemical behaviour of the “stain”
- Low variability (before and after wash)
- High discrimination

A.I.S.E. Stain Set – *v.7. October 2020*

Stains	Standard Stains			Hand-made Stains* (ex Warwick-Equest)	Stain classes Consumer denomination/Chemical nature
Tea		WFK 10J	CFT CS-97	WE5LTWKC	Drink / Bleachable
Coffee			CFT KC-H109	WE5ECWKC	Drink / Bleachable
Red wine			CFT KC-H026	WE5RWWKC	Drink / Bleachable
Fruit juice			CFT CS-15		Drink / Bleachable
Tomato puree				WE5TPWKC	Food / Bleachable
Salad Dressing Balsamico			CFT CS-406		Food / Bleachable Enzymatic
French Squeezy Mustard				WE5FSMWKC	Food / Bleachable Enzymatic
Chocolate		WFK 10Z	CFT CS-44		Food / Enzymatic
Grass	EMPA 164		CFT CS-07	WE5SGWKC	General soil / Bleachable Enzymatic
Grass/Mud				WE5GMWKC	General soil / Bleachable Enzymatic Particulate
Blood				WE5DASBWKC	General soil / Enzymatic
Unused motor oil	EMPA 106	WFK 10 RM	CFT C-01s		Grease, Oil / Greasy Particulate
Cooked Beef Fat				WE5BBPC2 (on polyester/cotton)	Grease, Oil / Greasy Enzymatic
Make up	EMPA 143/2	WFK 10MU	CFT CS-17	WE5FM2WKC	Cosmetics / Greasy Particulate

Dyes (1/2)

Color Maintenance

- Adopt A.I.S.E. 14 monitor dye set as common, most consumer/market relevant dye set.
- Add local consumer/market relevant dyes/shades (pastel, unbrightened) if needed/desired.

A.I.S.E. 14 Dye Set

Fabric number of A.I.S.E. (14) Monitor Dye Set	Fabric number of A.I.S.E. (14) Dye Set	Dye Class
1	A.I.S.E. 1	Sulphur Black
2	A.I.S.E. 3	Vat Green
3	A.I.S.E. 5	Vat Blue
4	A.I.S.E. 8	Direct Yellow + cationic after-treatment (Tinofix ECO)
5	A.I.S.E. 16	Reactive Red
6	A.I.S.E. 20	Reactive Black (pale shade)
7	A.I.S.E. 21	Reactive Black (heavy shade)
8	A.I.S.E. 22	Reactive Orange
9	A.I.S.E. 24	Reactive Blue
10	A.I.S.E. 26	Reactive Violet
11	A.I.S.E. 27	Reactive trichromatic combination
12	A.I.S.E. 29	Reactive trichromatic combination
13	A.I.S.E. 33	Disperse Navy + heat set
14	A.I.S.E. 39	Acid Red + syntan

Dyes (2/2)

Dye Transfer

- **The current StiWa protocol focuses on the following unfinished dyes:**
  - Direct Black 22
  - Direct Orange 39
  - Direct Red 83.1
  - Acid Blue 113
- **Market reality is that most direct dyes will have some sort of finish. Thus dye transfer is dependent on the durability of that finish.**
- **Further development work is underway to optimize this dye set.**

Execution of the Tests

Test Execution for Heavy Duty Detergents (Stain Removal and Whiteness)

Number of cycles	minimum of 6, ideally 8
# Stain set Replicates	1 internal
Standard white fabrics	Cotton, polyester/cotton, polyester and polyamide to measure whiteness
Ballast load	3 kg, clean white ballast load, normalized with 3 washes at 60C with ECE (88031 ex WFK) detergent w/o brightener and bleach to have comparable low optical brightener levels for ALL test products
Ballast soil	4 SBL 2004 sheets for HDD
Dosage	recommended dosage for normal soil/medium hardness for generalists
Temperature	40C (or locally adjusted) for generalist detergents

Test execution – SR and Whiteness

Evaluation

Stain removal:

- The evaluation of the degree of stain removal can either be assessed via suitable instrumental measurements such as reflectance (Y-value, SRI) or image analysis, as long as these methods are fully validated.
- Important is a statistical evaluation to arrive at meaningful conclusions.
- Reflectance via spectrophotometer, using the Y-value of the Y, x, y colour coordinates measurement, light source D65 with a UV cut-off filter at 420 nm. Aperture used for real stains 15 mm (minimum 12 mm). Stains are measured unfolded, 2 measurements per stain (in the center of the circular area, or closest homogenous area).
- Measurements are taken on each stain BEFORE wash (to verify quality of stains) unless there is a quality control in place and AFTER the wash and to evaluate standard deviations and are reported.
- Rank products based on statistic evaluation (95% confidence level). An approach for how to do this for a multitude of test products is recommended.
- Rank products on all stains, and if desired per consumer relevant stain classes. An approach for how to do this for a multitude of test products is recommended.

Test execution – SR and Whiteness

Evaluation

Whiteness:

- Basic whiteness as Y-value and Visual whiteness simulated as Ganz-Griesser value on 4 standard fabrics (cotton, polyester/cotton, polyester and polyamide) after 6 (8) cumulative washes

Test Execution – Color Maintenance

Colored Fabrics	14 A.I.S.E. Monitor Dye set (plus additional “local fabrics” if needed/desired)
Temperature	40°C for generalists (or adjusted based on consumer habits) Reference to Tenside 2004, 156-162.
Dosage	Recommended Dosage for normally soiled laundry/medium water hardness (15l water / machine) or harmonized dosage
Ballast	3.0 kg (normalised as in SR testing) Addition of 2 SBL 2004 to suppress excessive foam formation
Number of washes	20
Evaluation	Before and After the wash : Measurement of colour difference delta E expressed in Grey Scale units (ISO 105 A 05)

Test Execution – Dye Transfer

Equipment	Linitester
Color Donator	Direct Orange 39 Direct Black 22 Acid Blue 113 Direct Red 83.1
Color Acceptor	Cotton and polyamide (6x16cm)
Temperature	60°C
Time	30 minutes
Dosage	See dosage/usage (slide 11)
Water volume	100 ml
Number of replicates (internal/external)	1 fabric per cyclinder / 2 external repetitions
Evaluation	Measurement of color difference delta E, expressed as Grey Scale units (ISO 105 A 04)

Test Execution for Light Duty Detergents (Stain Removal and Whiteness)

Wash Program	Delicate cycle (NOT cotton or wool), high water level
Ballast load	2.5 kg (normalised as in SR testing for generalists)
Ballast soil	2 SBL 2004 sheets
Dosage	Recommended Dosage for lightly soiled laundry/medium water hardness (15l water / machine)
Temperature	30°C

Communication of results prior to publication

- **According to ISO/IEC GUIDE 46-1985**

"…. inform the manufacturer (agent/representative/importer) of test results on his own product and to invite his comments in sufficient time before publication. The test results submitted to the manufacturer (agent/ representative/importer) should b accompanied by the list of charactersitics tested and the test methods used. If the manufacturer (agent/ representative/importer) disagrees with the test results, it is recommended that he speedily supplies data to demonstrate that the test results are wrong or exceptional, or that the test methods used were unsuitable. In the case of exceptional results, it may be appropriate to take further samples."

(excerpt from ISO/IEC GUIDE 46-1985 section 3.2)

Future Improvements

- **Future improvements to increase consumer relevance as far as stain removal, ballast soil, whiteness maintenance, color maintenance or dye transfer testing is concerned will be evaluated when they become available.**

- **We will review this on a yearly basis.**

Acronyms

A.I.S.E.	International Association for Soaps, Detergents and Maintenance Products
ISO	International Standardisation Organisation
IEC	International Electrotechnical Commission
SBL	Soil Ballast Load
ECE	ECE Standard Detergent
HDD	Heavy Duty Detergent
SRI	Stain Removal Index
UV	Ultra violet
SR	Stain Removal