AWARD OF THE EU ECOLABEL TO LAUNDRY DETERGENTS:

PERFORMANCE TEST OF HOUSEHOLD DETERGENTS

1. INTRODUCTION

1.1 Carrying out wash performance tests will ensure that those detergents that are subject to Ecolabelling will have a sufficient efficiency in cleaning textiles according to the current state of product efficiency.

It is important that consumers' expectations, of having a satisfactorily working detergent as well as an Ecolabelled product, are fulfilled.

In other words, an Ecolabelled product should be fit for use and not compromise in its washing performance.

Furthermore, it should be recognised that the dosage per wash of a detergent is one of the most important criteria for Ecolabelling. The necessity to have sufficient washing performance should avoid any deceit in Ecolabel application, e.g. recommending too low dosage in full knowledge that sufficient washing performance is not achievable and/or higher dosages will need to be used by the consumer in order to achieve the required sufficient performance.

1.2 The most important characteristics of washing performance in the interpretation of a basic fitness for use (e.g. soil removal and redeposition, incrustation, fibre damage) can be assessed if textiles are washed several times using special monitors to investigate the individual characteristics. The evaluation of cumulative effects which give information on smaller quality differences however needs a very time-consuming combination of wash and wear cycles.

The soil load of the liquor is introduced by using a soil ballast fabric of defined soil composition and content.

As for Ecolabelling the determination of fitness for use is asked for, multiple wash cycles excluding an intermediate wear with a separate determination of the characteristics mentioned using special monitors and based on the principles of an already accepted European and international standard for testing washing machines (IEC 60456) seem to be appropriate.

The scope of the described method is to find out whether a detergent to be labelled with an Ecolabel is fit for use to achieve a sufficient cleaning performance and acceptable conservation of the textile properties and colours. It shall exclude those products from be labelled which do not correspond to average consumers' expectations and/or reduce textile life more than can be expected from the state of the art. Beyond this aim it is not meant to differentiate between different qualities of detergents of the market.

This test method will be laid down in this guideline. The normalisation of the monitors will be carried out using the reference detergent according to IEC 60456.

2. RANGE Of APPLICATION

- 2.1. In the context of the Ecolabel, this test can be applied to the following types of laundry detergents for textiles (powder, liquid and tablets):
 - 1. Heavy duty detergent (contains bleach and sometimes optical whitener (FWA))
 - 2. Colour-Safe detergents (without bleach, contains sometimes PVP and FWA)
 - 3. Light duty detergents (without enzymes)

A further differentiation, considering different detergents' ingredients, e. g phosphate-containing or phosphate-free or such with or without enzymes or optical brighteners, may be meaningful under the specific market situation and for selecting the reference product. However, since for the present purpose the reference product is a standardised product this additional differentiation may not always be possible.

3. BASIS FOR TESTING

3.1. **Testing Principles**

It is the basic principle of this test method to compare heavy duty and colour-safe test detergents with the standard detergent as described in IEC 60456 type A* (Annex A) or modified parts of it and light duty test deter-

gents with the light duty reference detergent (Annex B). The wash test is carried out using 25 cycles for heavy duty and colour-safe detergents respectively 15 cycles for light duty detergents using special white clean, soiled and/or stained swatches as monitors for determining the performance. In the case of colour-safe detergents additional tests with selected coloured monitors are added to evaluate colour stability and transfer.

3.2. **Test Criteria**

Test criteria	Type of Monitors	Detection
Soil Removal	Uniformly Soiled Monitors	Reflectance Measurements
Stain Removal	Uniformly stained Monitors	Reflectance Measurements
Degree of Whiteness acc. to colour shift number, Basic degree of Whiteness	Standard cotton fabric acc. To ISO 2267	Reflectance Measurements
Fabric Damage Fabric Incrustation	Standard cotton fabric acc. To ISO 2267	Physical Measurement
Dye Transfer	Test Textiles	Physical Measurement

4. TESTING PROCEDURE

4.1. General Test Procedures

4.1.1. Washing Machines

Programmable electronic washing machines with drain closure, which are set to a medium performance programme as the modified Miele Novotronic W 375 (technical specifications see Annex C).

4.1.2. Wash Water

In order to get comparable results, the hardness of the water used shall be 2.5 ± 0.2 mmol/L calculated as CaCO₃. This corresponds to the medium of European hardness. The Ca/Mg ratio shall be 3 ± 0.5 . The temperature of the supply water shall be (15 ± 2) °C; the dynamic pressure of the water supply at each water inlet shall be within the range indicated by the manufacturer and shall be stated in the report in case that the detergent under test shows problems during transfer into the machine.

If needed, water hardening can be performed according to the principles of IEC 60734 for testing of wet appliances. Water softening can be achieved mixing hard water with softened water.

4.1.3. Wash Programmes and Temperatures

The wash programmes used for the different detergents are carried out without a pre-wash:

DETERGENT	P/L	PROGRAMME*	TEMPERATURE
Heavy Duty	Powder	Normal Cotton Wash	40 °C
	Liquid	Normal Cotton Wash	40 °C
Colour-Safe	Powder	Normal Cotton Wash	40 °C
	Liquid	Normal Cotton Wash	40 °C
Light Duty	Powder	Delicate Wash	40 °C
	Liquid	Delicate Wash	40 °C

^{*} The programmes used in the recommended washing machine and the description of programming the 40 °C cotton programme to a medium washing performance are given in Annex C. For the delicate wash the original programme is used without a special change of the programme and without using the water plus button. The final spin (600 min⁻¹) is switched of and at the end of the washing programme an extra spin with 1600 min⁻¹ is adjusted.

4.1.4. Wash Loads

Each test series has to be started with a new wash load. The wash load used is dependent on the type of detergent under test. It consists of

Heavy duty and colour-safe detergents:

- ♦ A clean all cotton ballast load ^[1] for the normal cotton wash programme as described below
- ♦ 2 standard cotton cloth according to ISO 2267
- 6 uniformly soiled soil removal and 12 stain removal monitors as described below included in the washes 6 to 15
- Two pieces of soil ballast load added to all washes

The total load per wash including ballast and monitors shall be (4.0 ± 0.2) kg for the cotton programme.

Light duty detergents:

- ♦ A clean knitted polyester load ^[2] for the delicate wash programmes as described below to reach a total weight of 2,5 kg load
- ♦ 2 standard cotton cloth according to ISO 2267
- 4 uniformly soiled soil removal and 14 stain removal monitors as described below included in the washes
 6 to 15
- Two pieces of soil ballast load added to all washes

The total load per wash including ballast and monitors shall be $(2,5 \pm 0,1)$ kg for the delicate programme. Therefore all test fabrics (soiled monitors, soil ballast, standard cotton fabrics) are filled up with polyester base load to 2,5 kg.

[1] The base load of cotton shall consist of sanforised cotton pillowcases and cotton huckaback hand-towels conforming with the following specifications. The values are for new (unwashed) textiles

PillowcasesBleached cotton 1/1 plain weaveMass per unit area (185 ± 5) g/m² (of finished fabric)Warp (23 ± 1) threads/cm of (36 ± 1) texWeft (23 ± 1) threads/cm of (36 ± 1) tex

Pieces of (1 600 mm x 800 mm) $\pm 2\%$ folded in half and sewn

Weft

Size

along the three open edges thus forming double thickness $(finished\ size = 800\ mm\ x\ 800\ mm) - the\ shrinkage\ shall\ be\ less$ than 2% in a test according to ISO 6330, programme 4.1

Hand-towels Mass per unit area Warp

 $(230 \pm 10) \text{ g/m}^2$ (of finished fabric) (19 ± 1) threads/cm of (36 ± 1) tex (13 ± 1) threads/cm of (90 ± 1) tex $(1\ 000\ mm\ x\ 460\ mm) \pm 10\ mm$

Proportion of textiles

Bleached cotton weave-huckaback

Rated capacity [kg]	Number of Pillowcases	Number of hand-towels
4	12	9 – 14

The base load shall consist of double knitted polyester in pieces conforming with the following specification.

Knitted polyester fabric

 $(35 \pm 1) g$ Mass: $(200 \pm 10) \text{ g/m}^2$ Mass per unit area:

 (30 ± 1) cm $X(30 \pm 1)$ cm, double layer sewn along all four Size:

At least every 3rd wash cycle of the 25 test cycles the cotton ballast (pillowcases and huckaback towels) must be dried. The polyester ballast load is not dried between the cycles. After each wash cycle the two standard cotton clothes has to be ironed (adjustment: 2 points without steam).

4.1.5. Pre-treatment of standard cotton fabric

Before starting the 25 respectively 15 test cycles the standard cotton clothes for all products including reference (5 pieces per product) and huckaback towels which should be used as carrier fabrics for the stains (4 pieces per product plus an additional set of 4 pieces per product to spend time during the test) are put together to a $(4.0 \pm$ 0,5) kg load. 3 washes at 95 °C cotton programme without pre wash with water plus button in the Miele Novotronic W 375 machine have to be carried out. The basic powder of ECE standard detergent [1] for colour fastness (free of optical brighteners) in a dosage of 91,0 g per 4,0 kg load is used. Only after the 3rd wash the standard cotton cloth will be ironed (adjustment: 2 points without steam).

[1] Detergent according. to ISO 6330 is used

4.1.6. **Dosage of the Detergent under Test**

The dosage (g/wash) is taken as recommended by the detergent producer or distributor for "normally soiled" textiles. The addition is carried out according to the manufacturer's dosage instructions for a water hardness of 2,5 mmol/L. If this water hardness is borderline in the water hardness categories given in the dosage instructions, the lower dosage will be used. If the dosage is expressed as a volume and the density of a product is not given on the package, the test organisation shall get the bulk density of the product from the manufacturer in order to calculate the dosage in grams. If the detergent producer recommends a dosing aid, it will be used.

4.1.7. **Reference Detergent**

Specification for the reference detergent for heavy duty and colour safe detergents are given in IEC 60456. It shall be run in parallel with a detergent to be tested in order to achieve reproducible results. The following dosage is used:

Detergent under Test	Ref	eference Detergents	
	Basic Detergent	Bleach	Additive
Heavy Duty	91 g (Basic Powder IEC A*	24 g (SPB4)	3,5 g (TAED)
Colour Safe	91 g (Basic Powder IEC A* ^[1])	None	2.5 ml (PVP ^[2])
Light Duty	100 ml/wash cycle ^[3]	None	None

- [1] Detergent A according. to IEC 60456 is used (see Annex A)
- [2] BASF Sokalan HP 56
- [3] Liquid References for light duty detergents is used (see Annex B)

4.1.8. **Test products**

The test samples are provided by the producer or distributor. Both reference detergents and PVP are available from **w**-Testgewebe GmbH.

The amount of products needed for the whole test (max. 30 wash cycles for heavy duty and colour-safe products respectively max. 20 cycles for light duty detergents) shall be thoroughly mixed in a container and stored, in individual dosage lots, in sealed bags or cans. Mixing shall be done in such a way that it does not destroy the structure of the powder or change its physical properties.

4.1.9. Number of Wash Cycles

Heavy duty and colour-safe detergents:

25 wash cycles are carried out consisting of:

- 5 cycles with ballast load, soil ballast fabric and standard cotton fabric acc. to ISO 2267.
- 10 cycles with ballast load, added soil and stain removal monitors, soil ballast fabric and standard cotton fabric acc. to ISO 2267.
- 10 cycles with ballast load, soil ballast fabric and standard cotton fabric acc. to ISO 2267.
- Extra cycles for determination of colour transfer in a launder-O-meter or linitest only for colour-safe detergents.

Light duty detergents:

15 wash cycles are carried out consisting of:

- 5 cycles with ballast load, soil ballast fabric and standard cotton fabric acc. to ISO 2267.
- 10 cycles with ballast load, added soil and stain removal monitors, soil ballast fabric and standard cotton fabric acc. to ISO 2267 wash test cloth.

4.2. General Test Procedures

4.2.1. Type and Application of Soil Removal Monitors

The type of soil removal monitors is independent of the detergent under test. They are prepared and used in a way that they reflect today's detergency problems as well as consumers' habits with frequent washes and short storage time of the dirty articles.

Criteria	Heavy Duty	Colour-Safe	Light Duty
General Detergency Soil Removal	Pigment/Sebum on cotton (w 10D) mineral oil/black ink (EMPA 106) vegetable oil/milk/ ink on cotton (w 10 PPM)	Pigment/Sebum on cotton (w 10D) mineral oil/black ink (EMPA 106) vegetable oil/milk/ ink on cotton (w 10 PPM)	Pigment/Sebum on wool (w 60D) vegetable oil/milk/ink on cotton (w 10PPM)

The monitors are cut into squares of $(12 \times 12) \text{ cm}^2$ and marked on the right side. They are fixed together with the uniformly stained wash monitors on four huckaback towels as shown under point 4.2.4 with the right side showing. The monitors used in the wash performance test shall be from the same production lot.

4.2.2. White Unsoiled Multiple Wash Monitors

White cotton fabric according to ISO 2267. Per test series and product under test 3 m of standard fabric (for heavy duty and colour-safe detergents) respectively 2 m of standard fabric for light duty detergents is prewashed according to ISO 2267 and pre-cut into pieces of a length of 60 cm (heavy duty and colour-safe detergents) respectively 40 cm (light duty detergents) giving the no. 1 to 5. For heavy duty and colour-safe detergents initial tensile strength, fluidity, ash content, colour shift number (FAZ-value) and basic degree of whiteness are determined before use taking the specimens 1 and 5. For light duty detergents only initial ash content and basic degree of whiteness are determined before use. Sample 3 has to be stored as a reserve. Samples 2 and 4 are added to the washes 1 to 25 respectively 1 to 15 drying and ironing them cautiously between the individual washes (adjustment: 2 points, without steam).

3 pre-washes of standard cotton cloth: See point 4.1.5

A scheme how to manage the samples for determination of secondary washing effects is given in Annex D.

4.2.3. Type and Application Soil Ballast Load Fabric

In order to get information about the anti-redeposition capacity of the detergent under test and its elasticity against soil loads of the textiles washed two ballast soil cloths (24 x 34) cm² shall be added. It shall represent the kind (pigments, fat, proteins, starch, hardness etc.) and amount of soiling usually being present in a "normally soiled wash load. They shall be added to every wash repetition. A commercially available ballast soil is **W** 10SBL (24 x 34) cm² which consists of a mixture of pigment and fatty soil, proteins and starch as well as hardness salts on cotton.

4.2.4. Uniformly Stained Wash Monitors

As it is the main objective of this test method to decide whether a product is fit for use or not it is reasonable to substitute the hand made stains by a more reproducible production technique which may be padding, spraying or printing of stain materials uniformly onto fabrics.

Criteria	Heavy Duty	Colour-Safe	Light Duty
Fatty Stains	Used motor oil on cotton (w 10 GM)	Used motor oil on polyester/cotton (w 20 GM)	Make up on cotton (w 70 MU)
Bleaching	Red wine (EMPA 114) tomato ketchup (w 10T) on cotton grass (EMPA 164) on cotton	Red wine (w 20L) tomato ketchup (w 20T) on polyester/cotton	Tea on polyester/cotton (W 20 J) red wine on polyes- ter/cotton (W 20 L or EMPA 114) tomato ketchup on polyes- ter/cotton (W 20 T)
Enzymatic Soil Removal	Pre-aged egg yolk on cotton (w 10EG) milk cacao on cotton (EMPA 112)	Pre-aged egg yolk on cotton (w 10EG) milk cacao on cotton (EMPA 112) grass (EMPA 164) on cotton	Pre-aged egg yolk on polyester/cotton (w 20 EG) milk cacao on cotton (EMPA 112) grass on cotton (EMPA 164)

All stains are commercially available. As in practical applications stains shall not be aged, only dried at low temperature. The prepared stain fabrics are stored in the dark at low temperatures (according to the suppliers recommendations) excluding oxygen.

The chosen monitors are cut into squares of (12 x 12) cm² and fixed as described below on four huckaback towels for each wash cycle.

FIGURE 1 Example for heavy duty detergents how to fix 18 stains on four carrier fabrics

1.	10 D 2.	10 D		
	Empa 106 10 PPM 10 GM		Empa 106 10 GM	10 PPM

3.	Empa 114		4.	Empa 114	
	10 T	10 EG		10 T	10 EG
	Empa 112	Empa 164		Empa 112	Empa 164

<u>FIGURE 2</u> Example for light duty detergents how to fix 18 stains on four carrier fabrics

1.	60 D 10 PPM	20J 70 MU	2.	60 D 10 PPM	20J 70 MU	
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- 1	20 L 20 EG Empa 164	20 T EMPA 112	4.	20 L 20 EG Empa 164	20 T EMPA 112
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One further set of huckaback towels (each 4 pieces) must be prepared to spend time between two washes.

5 Washing Performance

Depending on the type of detergent, the following criteria will be evaluated:

Test criteria	Heavy Duty Detergent	Colour-Safe Detergent	Light Duty Detergent
Soil Removal	from cycle 6 to 15	from cycle 6 to 15	from cycle 6 to 15
Stain Removal	from cycle 6 to 15	+ from cycle 6 to 15	from cycle 6 to 15
Colour shift number	+ after 25 cycles	-	-
Basic degree of Whiteness (Y-Value)	+ after 25 cycles	+ after 25 cycles	+ after 15 cycles
Fabric Damage (Loss in Tensile Strength and Chemical Fibre Damage)	+ after 25 cycles	+ after 25 cycles	-
Fabric Incrustation (Organic and Inorganic Incrustation)	+ after 25 cycles	+ after 25 cycles	+ after 15 cycles
Dye Transfer	-	+ after one cycle	-

5.1 Test Procedure

The monitors used for the evaluation must be from the same production lot. The appropriate amount is stored at low temperatures (according to the suppliers recommendations) under exclusion of light and oxygen. The material is cut into specimens of 12 x 12 cm and stored until ready for use in the dark and cold.

2 test monitors of each kind are used for every single wash and fixed on different huckaback towel carrier fabrics with the marked right side upwards. An extra set of four carrier fabrics will be used for the next wash cycle

in order to dry the first set in the meantime.

The prepared carrier fabric with the test swatches are evenly distributed in the wash load and washed in the respective programme parallel to washes at the same conditions using the reference detergent. After one wash they are removed from the machine. Afterwards the monitors are removed from the carrier and dried in the dark at ambient conditions lying flat on a sieve.

The whole procedure is repeated 10 times (washes 6 to 15).

5.2 Evaluation

The determination of the cleaning efficiency is carried out using a spectrophotometer which allows the measurement of the total reflectance over the wavelength of visual light filtering the UV part of the incoming spectrum by a filter. The Y-Values are determined under the following measuring conditions:

Measuring equipment: Datacolor Spectraflash 500 or 600

Measuring geometry: d / 8°

D 65 / 10 ° observer

with UV-filter (420 nm cut off)

Measuring diameter: minimum 20 mm, the bigger the better

Gloss: without

Calibration: Measurements shall be carried out at the latest 8 hours after calibration with white tile and black trap.

For each soil monitor the mean of the 80 measurements (2 samples per soil x 4 readings x 10 wash cycles) are calculated. Standard deviation ought to be calculated from 10 washes and not from 80 measurements owing to the statistical dependence of the eight measurements in each wash.

The samples have to be measured at minimum fourfold on the marked right side.

The further evaluation of the results is performed considering the following remarks.

5.2.1 Evaluation of Reference Detergents

The heavy duty reference detergent is according to dosage and high activity of bleach system evaluated as good/very good performing detergent.

The colour-safe reference detergent is classified as a good performing detergent.

The light duty reference detergent is evaluated as a good performing detergent.

5.2.2 "Fit for use criterion"

The "Fit for use criterion" is defined with sufficient cleaning performance.

5.2.3 Final Evaluation procedure for cleaning performance

Cleaning performance as essential criterion includes General detergency, soil removal and stain removal.

The rounded difference of the Y-values between reference detergent and detergent under test are evaluated for each test fabric and classified in steps of 2 discriminating units according to the following table:

Δ to reference value	Discriminating units
≤ 0,99	0
1,00 to 2,99	2
3,00 to 4,99	4
5,00 to 6,99	6
≥ 7,00	8

Performance results of detergent under test better than the results of reference detergent are calculated with 0 units. That means, that bad results with one test fabric cannot be compensated by extra good results with other test fabrics.

Performance results on single soilings worse than 6 units (Heavy duty detergent), 4 units (Colour-safe detergent and Light duty detergent) according to the above mentioned table are not excepted, but one failure in this rule is allowed. In this case the maximal agreed number of units is charged as result. Heavy duty liquid detergents without bleach components will be charged with not more than this maximal unit number, even if calculation according the above table exceeds this number. This exception is foreseen for the test fabrics soiled with red wine, ketchup and grass. For heavy duty liquid detergent a maximum of 3 failures are allowed. The failures accepted are red wine, grass and another one. If failures occur these should be charged 6 CPUs for each soils.

Summarisation of the units for all test fabrics respecting the above mentioned exceptions shall not exceed the following number of discriminating **Cleaning Performance Units** (**CPU**):

Heavy duty powder detergent	24
Heavy duty liquid detergent	36
Colour-safe detergent	10
Light duty detergent	18

6 Determination of Secondary Washing Effects

In Annex D a scheme is given how to manage the samples of standard cotton cloth for determination of secondary washing effects.

6.1. Loss in Tensile Strength

Loss in tensile strength (TSL) is determined according to ISO 2267. It is carried out on 10 wet test specimens. The calculation of the tensile strength loss is related to the pre-washed fabric (initial value). TSL-value gives a quick information about possible damage caused by cellulase.

6.2. Chemical Fibre Damage

The chemical fibre damage (characterised by damage factor s) is determined by comparing the fluidity of a dissolved cotton sample in a solution of iron tartrate (EWNN_{mod(NaCl)}-procedure as in DIN 54270 Part 3) or couoxam (as in DIN 54270 Part 2 or ISO 4312).

6.3. Inorganic Incrustation

The inorganic incrustation is determined by igniting a standard cotton sample after the agreed washes and weighing the residual ash according to DIN 53 919 part 2 with reference to the initial value of the pre-washed sample.

6.4. Organic Incrustation

The organic incrustation (calcium soap, surfactants etc.) is determined by extraction of a sample of the standard cotton fabric with methanol after the 3 pre-washes and after the agreed number of washes using a soxhlet type extraction according to ISO 4312. After evaporating the solvent the residue is weighed and calculated as % of the textile extracted.

The determination is carried out according to ISO 4312 with the following exceptions:

amount of test sample: 8 g Extraction solvent: methanol Test cloth is not cut into small pieces

No use of an extract hull

Duration of extraction: about 4 hours with 5 - 6 fillings per hour

Drying temperature of the residue 80 °C Duration of drying the residue: at least 12 hours

6.5. Basic Degree of Whiteness (Y-Value) and Colour Shift Number (FAZ-value)

The Y-values and the colour shift numbers (FAZ-values) of the unsoiled standard cotton fabrics are determined after three pre-washes. The effect of the detergent under test is determined after 15 and 25 washes. From both standard cotton samples (1 and 5 for initial values respectively 2 and 4 for values after 15 and 25 washes) 8 measurements were taken. The samples have to be measured at minimum fourfold. Averages are calculated out of 16 single measurements.

Conditions of measurement:

Colour Shift Number (FAZ-Value)

Measuring equipment: Datacolor Spectraflash 500 or 600

Measuring geometry: $d / 8^{\circ}$ D 65/ 10 °-observer

with UV-light, that means without UV filter Calibration with "datacolor flourescent standard"

Measuring diameter: minimum 20 mm, the bigger the better

Gloss: without

Calibration: for each set of testfabrics, - measurements shall be carried out at the latest 8 hours after calibration with white tile (e.g. datacolor international white calibration standard or equivalent) and black trap.

The colour shift number (FAZ-value) is not required when testing detergents free of optical brightener.

The measurement of the <u>Basic Degree of Whiteness</u> excludes the influence of UV-light which is excluded by an appropriate filter.

Measuring equipment: Datacolor Spectraflash 500 or 600

Measuring geometry: $d / 8^{\circ}$ D 65 / 10 $^{\circ}$ observer

with UV-filter (420 nm cut off)

Measuring diameter: minimum 20 mm, the bigger the better

Gloss: without

Calibration: for each set of test fabrics, -- measurements shall be carried out at the latest 8 hours after calibration with white tile (e.g. datacolor international white calibration standard or equivalent) and black trap.

6.6. Determination of Dye Transfer

6.6.1 Dye Donators and Dye Acceptors

In case of testing colour-safe detergents, fabrics, that have been dyed with the dyes listed below, are used as dye donators.

They are typical dyestuffs for the respective fabrics and/or are used in large quantities by the textile industry:

Kind of Dye Dye Acceptor

◆ C.I. Direct Blue 71 (wfk DB 71)
 ♦ C.I. Reactive Brown 7 (wfk RB 32)
 ♦ C.I. Sulphur Blue 19 (wfk SB 19)
 ♦ C.I. Acid Blue 113 (wfk AB 113)

Polyamide

The direct dye has a poor water fastness. The reactive dye is deliberately not fixed too strongly so that there is dye transfer in the wash liquor.

The selection of the dyes may be object of change due to the state of the art and their availability.

Dye Acceptors

- ♦ Standard Cotton according to DIN 53919, part 1 (size 5,5 x 16 cm)
- ♦ Polyamide according to ISO 105 F03 (size 6 x 16 cm)

6.6.2 Procedure

The washes are done in a suitable laundering device (e.g. Lini-Test equipment) consisting of a water bath containing a rotable shaft which supports, radially stainless steel containers (diameter 7,5 \pm 0,5 cm, height 12,0 \pm 0,5 cm) with 525 \pm 50 ml capacity each), the bottom of the containers being 4,5 \pm 1 cm from the centre of the shaft. The shaft/container assembly is rotated at a frequency of 40 \pm 2 1/min. The temperature of the water bath is thermostatically controlled to maintain the test solution at the prescribed temperature \pm 2°C.

For the preparation of the wash liquor the same liquor concentration and water hardness is used as in the washing machine. The product in test (amount for 1 l) is dispersed in 1 l of lukewarm water using a magnetic stirrer and then rapidly heated until the liquor reaches 40°C.

Dye donator (0,3 g) and dye acceptor are placed in the container (no addition of steel balls). Both textiles are not fixed to each other. The volume to give the correct liquor: fabric ratio 100:1 is added and the containers are placed in the preheated (40°C) machine. Temperature raises 2°C per minute up to 60°C and the wash is continued for 20 minutes at this temperature.

After the wash the textiles are removed and rinsed twice for 1 minute in warm running water, then in cold running water for 10 minutes. Textiles are dried hanging in the air (no direct sun).

6.6.3 Evaluation

In order to assess the dye transfer after one wash, colour differences ΔE between the standard cotton or polyamide piece washed without and with dye donator is determined. Measurements are taken at two defined areas of the dye acceptor using an appropriate device (e.g. datacolor). The instrumental assessments on colour fastness are done according to ISO 105-A04. They are based on ISO 105-A03 (Grey scale for assessing staining). The measurements for all products to be compared are performed using one common calibration and the same conditions.

Measuring equipment: Datacolor Spectraflash 500 or 600

Measuring geometry: d / 8° D 65 / 10 $^{\circ}$ observer

with UV-filter (420 nm cut off)

Measuring diameter: minimum 20 mm, the bigger the better

Gloss: without

Calibration: for each set of test fabrics, measurements shall be carried out at the latest 8 hours after calibration with white tile (e.g. datacolor international white calibration standard or equivalent) and black trap.

7. TEST REPORT AND EVALUATION OF RESULTS

7.1. **Test Report**

The test report must contain the following:

7.1.1. **Test Procedure**

- exact description of the test product
- origin of the product (trade name, code, and other relevant data)
- ♦ time frame of the test
- dosage and way of dosage
- detergent concentration in the laboratory washing device and kind of device (e.g. linitest)
- type of washing machine, selected programme, amount of water in main wash
- wash temperature
- water hardness (mmol/L) total and ratio Ca/Mg; way of producing the hardness
- load in kg
- composition of load (types, individual weights, fibre composition of the coloured and white textiles
- characteristics of soils and stains used including fibre substrates
- statistical details on the test design
- ♦ kind and type of used measuring devices

7.1.2. **Test Results**

The results of the test product P and the reference product R have to be correlated for the defined test criteria. The individual results are summarised for the different criteria and set into correlation to the results achieved with the reference product R.

Heavy duty and colour-safe detergents:

1. Cleaning and Stain Removal Performance on Single Wash Monitors

Cleaning Performance Units (CPU) achieved according to point 5.2

2. Secondary Washing Effects

Chemical damage factor after 25 washes in %

Loss of tensile strength after 25 washes in %

Organic incrustation after 25 washes in %

Inorganic incrustation after 25 washes in %

3. Colour Shift Number (FAZ-value) only for heavy duty detergents

FAZ-value after 15 and 25 washes

4. <u>Basic Degree of Whiteness</u>

Y-value after 15 and 25 washes

5. <u>Determination of Dye Transfer on white single-wash textiles only for colour-safe detergents</u>

Instrumental assessment (colour differences ΔE , grey scale for assessing staining) of the dye acceptors and average of the samples under test.

6. <u>Significant Observations that Require Noting</u>

Any points not covered by the test programme, or minimum requirements of washing performance, that would prevent granting of the ecolabel.

Light duty detergents:

1. Cleaning and Stain Removal Performance on Single Wash Monitors

Cleaning Performance Units (CPU) achieved according to point 5.2

2. Secondary Washing Effects

Organic incrustation after 15 washes in %

Inorganic incrustation after 15 washes in %

3. Basic Degree of Whiteness

Y-value after 15 washes

4. Significant Observations that Require Noting

Any points not covered by the test programme, or minimum requirements of washing performance, that would prevent granting of the ecolabel.

7.2. Evaluation of Results

The minimum requirements of washing performance which have to be fulfilled by the detergent product under test are expressed for the individual criteria in absolute terms or grades.

The test criteria are differentiated into criteria of lower priority for cleaning performance described in chapter 5.2 and for secondary washing effects listed in chapter 6. Criteria of lower priority are:

Heavy duty detergent: Inorganic incrustations

Colour-safe detergent: Basic degree of whiteness or inorganic incrustations

Light duty detergent: Basic degree of whiteness or inorganic incrustations

The limits of these criteria can be extended on the responsibility of the test institute to max. +25% of the limiting value. The exception has to be notes with comment in the test report.

For the above listed criteria of lower priority one exception is allowed. The extended limits in this care are

for colour – safe detergent: Basic degree of whiteness Ref – P < 3.0 for light duty detergents; Basic degree of whiteness P - Ref > 1.0

In any case, for light duty detergents, the basic degree of whiteness has to be equal to or better than the original unwashed fabric.

In case of alternative use of an exception for inorganic incrustations minimum requirement value of max. +50% is accepted, but this value should not exceed the value of reference detergent. Ring test has shown that the test results for inorganic incrustations seem to depend significantly on specific conditions of the method of determination.

TABLE 1 lists the minimum requirements for the different criteria for the different detergent classes.

The test products meets the requirements, if it shows better or equal results in all criteria (with the exception of one of the above mentioned criteria of lower priority) than the calculated value derived from results of the reference product in table 1.

Test Criteria	Test Monitors	Heavy Duty Detergents (containing FWA) Colour-Safe Deterg		Light Duty Detergents			
Cleaning and Stain Removal Efficiency	Single Wash Monitors and Uniformly Produced Stains (sum of grades)	Powder: CPU max. 24 Liquid: CPU max. 36 CPU max. 10		CPU max. 18			
Colour Shift Number	Standard fabric	Direction Red-Violet max. 1,0; Direction Blue-Green max. 3,0 No requirements		No requirements			
Basic Degree of Whiteness Y	Standard fabric	R – P <	P – R > 2,0 *				
Dye Transfer	Colour acceptors [1]	No requirements $R-P < 0.5$		No requirements			
Loss in Tensile Strength	Standard fabric	Max 12 %	Max 12 %	No requirements			
Chemical Fibre Damage	Standard fabric	Max. 0,25	Max. 0,1 %	No requirements			
Inorganic Incrustation	Standard fabric		Max 2,5 %				
Organic Incrustation	Standard fabric	Powders Max. 1,0 % Liquids Max. 1,5 %					

[1] Average over all items

R = **Reference**

P = Product

TABLE 1 Minimum Requirements for the Ecolabel

^{* =} In any case equal to or better than the original unwashed fabric

ANNEX A: Composition of IEC reference detergent A*

Ingredient	%	Tolerance (±)
Linear sodium alkyl benzéne sulfonate	8,8	0,5
Ethoxylated fatty alcohol C ₁₂₋₁₄ (7 EO)	4,7	0,3
Sodium soap (tallow soap)	3,2	0,2
Foam inhibitor concentrate, 12 % silicon on inorganic carrier)	3,9	0,3
Sodium aluminium silicate zeolite 4 A (80 % active substance)	28,3	1,0
Sodium carbonate	11,6	1,0
Sodium salt of a copolymer from acrylic and maleic acid (granules)	2,4	0,2
Sodium silicate (SiO_2 : $Na_2O = 3,3:1$)	3,0	0,2
Carboxymethylcellulose	1,2	0,1
Phosphonate (25% active acid)	2,8	0,2
Optical whitener for cotton (stilbene type)	0,2	0,02
Sodium sulfate	6,5	0,5
Protease (Savinase 8,0)	0,4	0,04
Basic Powder	77,0	
Bleach:		
Sodium perborate tetrahydrate (SPB4)	20,0	
Additive:		
Tetraacetylethylenediamine (TAED)	3,0	
	100,0	

NOTES

- 1 The reference detergent is distributed in three separate parts:
 - a) Basic powder;
 - b) Sodium perborate tetrahydrate;
 - c) Tetraacethylethylenediamine.

The proportions of ingredients of the ready detergent are: 77 % basic powder; 20 % sodium perborate tetrahydrate; 3 % tetraacethylethylenediamine.

- Due to the variability, which may result from the manufacturing procedure of the detergent or of its ageing, for comparative measurements, it is recommended to use a reference detergent supplied by one definite manufacturer from one definite production batch recently supplied. It is also recommended to keep the detergent and perborate separately in small quantities (e.g. 1 kg) and to use it within a limited time.
- 3 It is recommended that the detergent manufacturer should indicate the pH of the product supplied.

The ingredients shall be mixed prior to use. The maximum storage time after mixing is 7 days.

ANNEX B: Composition of Light Duty Reference Detergent

35 %	Fatty alcohol ethoxylate (EO = 7)
15 %	Low foaming fatty alcohol ethoxylate
	(ethylenoxide/higher alkylene oxide-co-polymer)
7,5 %	Sodium dodecyl sulfonate
15 %	Modified polycarboxylate
	(suitable for liquid detergents)
5 %	Ethanol
	Water add 100 %

NOTES

Due to the variability, which may result from the manufacturing procedure of the detergent or of its ageing, for comparative measurements, it is recommended to use a reference detergent supplied by one definite manufacturer from one definite production batch recently supplied.

The bottle shall be agitated lightly before use.

ANNEX C: Programmes and description of programming the 40 °C cotton programme to a medium washing performance (Miele Novotronic W 375)

The wash programmes used for the different type of detergents are carried out without a pre-wash:

DETERGENT	P/L	PROGRAMME*	PROGRAMMATION
Heavy Duty	Powder	40 Cotton Wash with water plus button	see (1)
	Liquid	40 Cotton Wash with water plus button	see (1)
Colour-Safe	Powder	40 °C Cotton Wash with water plus button	see (1)
	Liquid	40 °C Cotton Wash with water plus button	see (1)
Light Duty	Powder	40 °C Delicate Wash	No
	Liquid	40 °C Delicate Wash	No

(1) Cotton 40 °C with water plus button

<u>Programming modus</u>, disconnecting of "automatically adjustment of quantity of water proportional to quantity of laundry"

- 1) Door closed, machine off
- 2) Turn the Program choice position to "End"
- 3) Press buttons "delayed start" and "water plus" and meanwhile turn on the machine
- 4) Leave all buttons
- 5) LED's "Soak/prewash" and "Wash" are now illuminated and Display shows "P 0".
- 6) Choose program postion "Minimum iron 50 °C"
- 7) Display shows "P15 0"
- 8) Change to "P15 1" with short press on "Start"
- 9) Choose program position Cottons 75 °C
- 10) Display shows "P20 0"
- 11) Change to "P20 1" with short press on "Start"
- 12) Turn of the machine the changes are saved.

Use the program Cotton 40 °C with button "Water plus".

ANNEX D: Scheme how to manage the samples of standard cotton cloth for determination of secondary washing effects of a heavy duty and colour-safe detergent.

Samples of standard cotton cloth No 1 and 5 for determination of the initial values

Code for standard cotton cloth	TSL	LSL	TST	TST	TSL	TST	TSL	TSL	LSL	TST	LSL	60 cm
Wr Co Ba	niteness lor shift sic degr iteness	Ganz/	r/							Chemical damage Inorg. Incrust.	Inorganic Incrustation (single determination)	

80 cm

Samples of standard cotton cloth No 2 and 4 for determination of secondary washing effects after 15 and 25 wash cycles

Code for standard cotton	TST	ISL	191	TSL	LSL	LSL	TSL	TSL	TSL	ISL	TSL	LSL	ermination)	60 cm
	White Color numb degre White 15 wa	shift er/ Ba e of ness a	sic after	/		Color Bas White	teness r shift n sic degr eness a rash cyc	umber/ ree of after 25		chemical damage Inorg. Incrust.	Inorganic Incrustation (single determination)	Organic Incrustation	Organic Incrustation (single determination)	

80 cm

Scheme how to manage the samples of standard cotton cloth for determination of secondary washing effects of a light duty detergent.

Samples of standard cotton cloth No 1 and 5 for determination of the initial values

Organic Incrustation V-Value Code for standard cotton cloth

80 cm

Samples of standard cotton cloth No 2 and 4 for determination of secondary washing effects after 15 wash cycles

Inorganic Incrustation	Organic Incrustation			Y-Value			Code for standard cotton cloth	40 cn
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80 cm

ANNEX E: ECO-Label Performance Test for household laundry detergents Technical Data Test Protocol of modified Miele Novotronic W 375 washing machine

- 1. Buntwäsche 40°C, Programm 2
 - = Cotton 40°C programme with water plus button (if machine is prepared by described programming modus)
- 2. Feinwäsche 40°C (original programme with 1.0 kg load and 600 rpm)
 - = 40°C Delicate programme (for ECO-Label Performance Test use 2.5 kg load and 1600 rpm final spin)