

Ch. Nitsch*, G. Hüttmann**

Recommendation for the Quality Assessment of the Cleaning Performance of Hand Dishwashing Detergents

Recommendation elaborated in an IKW Working Group by Colgate-Palmolive GmbH, Dalli Werke Wäsche- und Körperpflege GmbH & Co. KG, fit GmbH, Henkel KGaA, Luhns GmbH, Reckitt Benckiser plc., Werner & Mertz GmbH, published by The German Cosmetic, Toiletry, Perfumery and Detergent Association e.V. (IKW), 2002

Preamble

The member companies of IKW, the German Cosmetic, Toiletry, Perfumery and Detergent Association, make their expert knowledge about the products manufactured by them available to the general public in the form of quality recommendations. The quality recommendations aim to allow the qualified testing of the relevant products by the companies themselves, by consumers and by the test institutes. Quality standards are established which must be met by the products concerned in order to achieve the effects expected by consumers and manufacturers.

The member companies of IKW strive to achieve the optimum quality standards for their products. Through a consistent orientation towards the sustainability model, they have set themselves the goal of safeguarding their future viability in a constantly changing world. This commitment to the sustainability model draws on experience which has manifested itself in numerous, exemplary initiatives. Based on the Rio Declaration of 1992, Johannesburg »92 plus10« and the Agenda 21, sustainability is understood to mean the balanced linking of economic and social aspects with ecological ones in order to meet the needs of people today whilst, at the same time, securing all possibilities for future generations. Against this backdrop the »Recommendations for quality assessment« help both the employees in companies to adopt a more responsible attitude towards man and the environment when developing and manufacturing products and also the consumer who can expect effective, safe and environmentally compatible products. The quality standards lay down which qualities are relevant for a specific

product and to what degree they must be met. Here, it should be borne in mind that each finished product has an agreed range of action which is oriented above all towards the ideas of the consumer about each individual quality characteristic. It should also, therefore, be remembered that for an individual article some individual properties will be more consciously stressed than others. The desired combination of the individual properties is subject to constant change and is, in turn, dependent on new technical developments and new consumer habits. Quality recommendations may not impede developments of that kind. Consequently, it can only be determined in a general manner whether an article corresponds or not to the quality recommendations. The emphasis on isolated test characteristics is inadmissible and can be misleading.

1. Introduction

Hand dishwashing detergents are still of considerable importance on the market and are used in almost every household. The products are aqueous solutions of different surfactant mixtures and contain additives like perfumes, colouring agents and auxiliary agents, for instance for the adjustment of viscosity. In this product category, too, attention has been paid to ecological aspects through the development of concentrated and innovative formulations. Today, the market offers a wide range of hand dishwashing products with different requirement profiles, including special formulations with specific claims. Both the users and the suppliers of these products are, therefore, interested in reliable and practice-related test methods for the quality assessment, mainly of cleaning performance.

A working group was set in 1998 within the German Cosmetic, Toiletry, Perfumery and Detergent Association (IKW) consisting of experts from dishwashing detergent manufacturers to develop just such a method.

2. Goal

The task of the working group was to elaborate a recommendation for the quality assessment of the cleaning performance of hand dishwashing detergents. This recommendation was to make possible the qualified testing by manufacturers themselves and by independent test institutes. It can also serve as a contribution to the discussion about test methods for hand dishwashing detergents on the European level and aims to meet the following criteria:

- simple, no major outlay in equipment
- relevant for daily practice
- reproducible
- differentiating.

3. Procedure of the working group

After examining and comparing the methods used by the manufacturers and existing experience, the test method used in the last hand dishwashing detergent test in 1994 by the »Stiftung Warentest« was selected as the starting point for the joint development. Several elements of this test were revised, described in greater detail, amended and examined in numerous interlaboratory tests. The recommendation for quality assessment presented here was coordinated with the leading European test institutes.

4. Test principle

In a dishwashing solution soiled plates are washed by hand until the foam layer collapses. Besides the dishwashing detergents for testing, a control is always conducted with a standard detergent. Details of the test set-up and the necessary equipment are given in Annex 1.

5. Preparation of the dishwashing solution and foam generation

Hot water flows freely from reservoir into a washing up basin and foams the dishwashing detergent placed there (Fig. 1). The dosage of the dishwashing detergent complies with the manufacturer's instructions. The volume of the dishwashing solution is 5 litres. The dishwashing detergent is dosed directly into the washing up basin with an accuracy of 1/10 g, half way between the centre and the wall of the basin. This can either be done gravimetrically or volumetrically bearing in mind the density of the product.

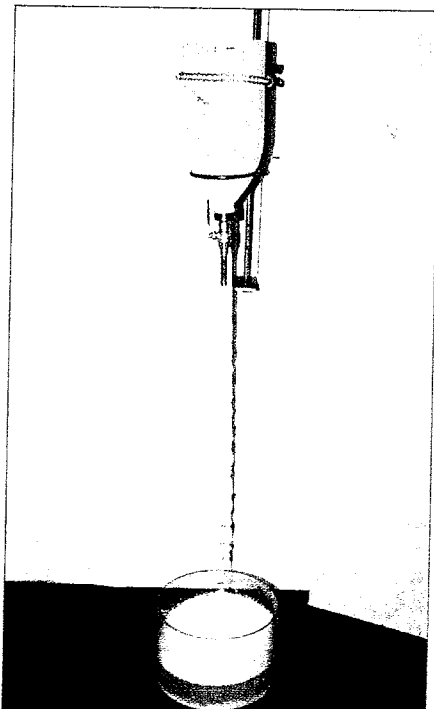
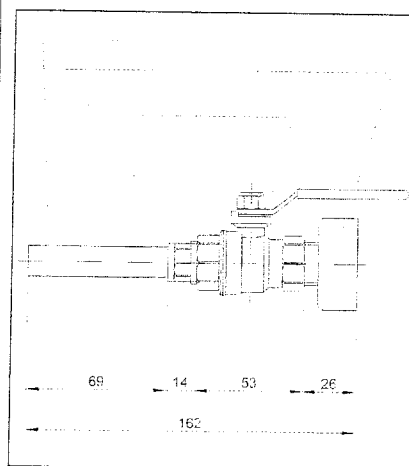
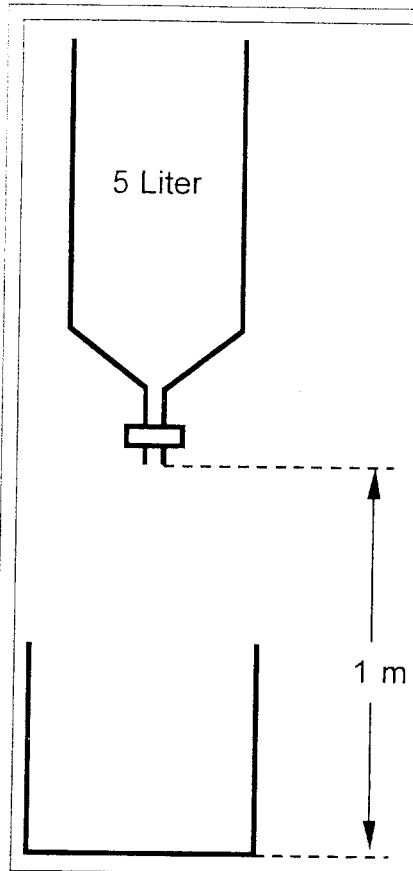
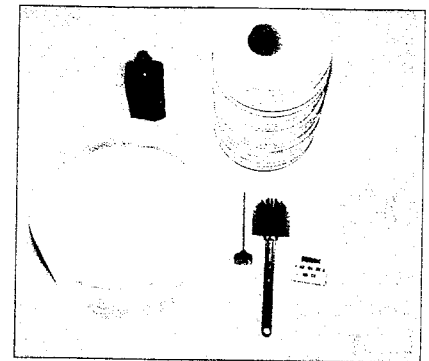


Fig. 1 Foam generation



Outlet tap



Equipment needed

Washing up basin

Cylindrical Plexiglas bowl
Inner diameter 29 cm, inner height 19 cm
Volume 12.5 litres, individually manufactured by:
Carl Delhas GmbH
Märkische Str. 54, 42281 Wuppertal, Germany
Tel.: +49 (202) 5288-166, Fax: +49 (202) 5288-865

Water reservoir

Narrow-mouthed bottle made of polyethylene with self-separated bottom, Content 5 litres.

Plastic washing up brushes

Manufacturer CORONET-Werke GmbH
Neustadt 2, 69483 Wald-Michelbach, Germany
Tel. + 49 (6207) 601-0, Fax + 49 (6207) 601-260
(Article No. 1165215, as per February 2002)

Plates

Breakfast plates, diameter 19 cm, e.g. Arzberg
Manufacturer: Winterling Porzellan AG
Jakobsburg 1-7, 95659 Arzberg/Oberfranken, Germany; Sales: Tel. +49 (9233) 403-0, Fax +49 (9233) 403-156
Breakfast plates model »Arzberg City-white«, diameter 19 cm, weight approx. 230 g (Art. No. 0219100, as per February 2002)

Outlet tap

Frank Wondrej
Heating-Sanitary Facilities-Plumbing
Zittauer Straße 40, 02788 Hirschfelde, Germany
Tel./Fax +49 (35843) 25233

Technical drawing of the tap

Source: Werner & Mertz GmbH

Photos

Anne Wolf, Reckitt Benckiser

Annex 1 Test set up, equipment used and sources

The reservoir is placed in such a way that its outlet tube points toward the centre of the basin and the distance between its lower edge and the basin bottom is 1 metre. To generate foam, 5 l of water are run into the basin by fully opening the tap (running time: approx. 16-21 seconds).

6. Water hardness

The water used for the dishwashing solution must have a hardness of 16 ± 2 °d

(corresponding tap water, hardened tap water or synthetic hard water may be used). The water hardness is to be measured and recorded before conducting the tests.

7. Water temperature

The soak temperature in the basin at the beginning of the test is $45 \pm 1^\circ\text{C}$. To reach this temperature, the water in the reservoir must be about 47°C warm. At the end of the washing test the temperature of the soak should not be less than approximately 35°C . The soak temperature is recorded at the beginning and end of the test.

Test soil

Common components are used to produce the test soil. Two soil formulations are used: Soil 1 (low fat) and Soil 2 (normal) (Table 1).

The maximum stability of the food stored is the same as the best-by date when stored in line with the manufacturer's recommendations. The manufacture and storage of the test soil is undertaken in both cases in line with the following provision:

8. Preparation of the test soil

All components are separately weighed. Water with a hardness of $16 \pm 2^\circ\text{d}$ is used as described in section 6. The fat components are carefully melted in a beaker in the microwave or in a water bath. In order to avoid splattering, the container is covered. Colouring agent, milk powder, flour and water are stirred into the melted fat mixture ($50 - 60^\circ\text{C}$). The mixture is then stirred thoroughly (for 1 kg batch e.g. Braun Electronic Mixer, 5 minutes) in order to obtain homogeneous test soil.

After preparation the test soil is divided into portions if required, frozen for at least 12 hours in the deep freezer (-18°C or colder) and then defrosted either over night in the fridge (approx. 4°C) or directly in the water bath. The test soil should be used portion by portion; a mixing of various portions is not admissible. The test soil can be stored for 12 weeks in the deep freezer. Thawed test soil may not be refrozen.

Testsoil:			1 (low fat)	2 (normal)
Components	Specification	Supplier	%weight	%weight
Beef tallow	Not specified	local source	1.2	4.7
Vegetable fat	Palmin soft	Dr. Schlinck	1.2	4.7
Margarine	80 % fat content	e.g. Unilever	1.2	4.7
Butter	Sweet-cream butter	local source	1.2	4.7
Lard	Not specified	e.g. Laru	1.2	4.7
Creme fraîche	30 % fat content	Dr. Oetker	1.2	4.7
Sunflower oil	Not specified	e.g. Thomy	1.2	4.7
Olive oil	Cold pressed extra virgin	e.g. Dante	1.2	4.7
Skim-milk powder	1 % fat content	e.g. Frema	9.6	6.3
Flour	Wheat flour Type 405	local source	28.8	18.8
Water	$16 \pm 2^\circ\text{d}$	see section 6	51.8	37.1
Colouring agent	Duasyn RotR-F 3 B fl. C.I. reactive red 180	Simon & Werner	0.2	0.2

Components	Percent in the dry matter	Percent in the dry matter
Fat	20	60
Carbohydrates	60	30
Proteins	20	10

Table 1

9. Preparation of the plates

The plates are washed in a common household dishwasher (normal programme, dishwashing temperature $50 - 65^\circ\text{C}$). A low alkaline dishwasher detergent and standard machine soft-

ened water without any rinse aid are used. The plates are visually checked for particle or film residues and can then be stored.

Prior to applying the soil, the test soil is heated in a water bath to 40°C , thoroughly homogenised (e.g. Braun Electronic Mixer, 5 minutes) and applied using a pipette, a dosimat or the like as »spots« (Fig. 2) to the plates at room temperature ($22 \pm 2^\circ\text{C}$). The soil stock in the storage container must be constantly stirred during application to the plates

Dosage:

Test soil 1: 6.7 g per plate
 Test soil 2: 5.0 g per plate

The soiled plates are stacked and must be used up, after a waiting period of at least 10 minutes at room temperature ($22^\circ\text{C} \pm 2^\circ\text{C}$), within three hours of soiling. To prevent the test soil drying out, the stack of plates is to be covered with a clean plate.

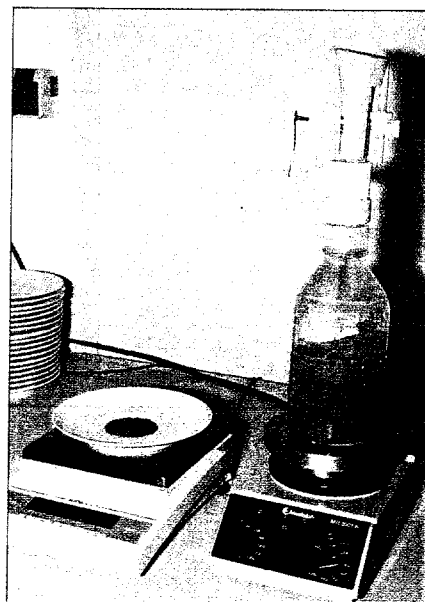


Fig. 2 Test soil in the dosimat and on a plate

10. Dishwashing auxiliary

The washing up basin is to be thoroughly cleaned with a hand dishwashing detergent, well rinsed with tap water and dried after each test. Before the start of the experiment a visual check is made for dirt.

DETERGENTS

Values obtained (Number of washed plates) for test soil 1

Number experiments	5	5	5	5	5	5	5	5
Product	Product A	Product B	Product C	Product D	Product E	Product F	Product G	Product H
Dosage	4 ml	4 ml	4 ml	4 ml	4 ml	4 ml	4 ml	4 ml
Dishwashing test 1	18	16	15	17	16	15	18	17
Dishwashing test 2	17	16	15	17	16	15	17	14
Dishwashing test 3	17	16	16	16	16	15	16	16
Dishwashing test 4	17	17	16	16	16	15	16	16
Dishwashing test 5	16	15	15	16	15	16	16	15
Mean value (plates)	17.0	16.0	15.4	16.4	15.8	15.2	16.6	15.6

Single factor analysis of variance (ANOVA)

ANOVA (calculated with Microsoft Excel 97[™]):
selected significance level: 95 % ($\alpha = 0,05$)

SUMMARY

Groups	Number	Sum	Mean value	Variance
Column 1	5	85	17	0,5
Column 2	5	80	16	0,5
Column 3	5	77	15,4	0,3
Column 4	5	82	16,4	0,3
Column 5	5	79	15,8	0,2
Column 6	5	76	15,2	0,2
Column 7	5	83	16,6	0,8
Column 8	5	78	15,6	1,3

ANOVA

Reason for scattering	Sum of squares (SS)	Degree of freedom (df)	Mean sum of square (MS)	Test parameter (F)	P-value	Crit. F value
Differences between groups	13.6	7	1.94286	3.79094	0.004197	2.31274
Within groups ($MS_i = MQ_i$)	16.4	32	0,5125			
Total	30	39				
Total	18.375	39				

ANOVA: $H_0: \mu_{(A)} = \mu_{(B)} = \mu_{(C)} = \mu_{(D)} \dots$ $F = 3,79 > 2,31 = F_{crit} \implies H_0$: rejected

Assessment: At least two of the dishwashing detergents have a significantly different cleaning performance

Duncan-Test

Duncan-Test: $d_{crit} = \gamma * q_{rit(p,f)}$	$\alpha = 0.05$	Significance level	$MS_i = MQ_i = 0.51$	Mean sum of squares (within groups)
	$P = 8$	Number of products	$q_{rit} = 3.32$	Highest score for this test design ex Table (see source)
	$f = 32$	Degree of freedom	$\gamma = 0.32$	calculated from Duncan Test
	$n_1 = 5$	Number of dishwashing tests	$d_{crit} = 1.06$	Highest calculated critical difference for this test design
	$n_2 = 5$	Number of dishwashing tests		

Significant distance for mean values (number of washed plates) at $\alpha = 0.05$, when $d_{1,2} > d_{crit} = 1.06$ (highest critical difference)

Difference plates $d_{i,j}$	Product A	Product B	Product C	Product D	Product E	Product F	Product G	Product H
Product A	-	1.0	1.6	0.6	1.2	1.8	0.4	1.4
Product B	1.0	-	0.6	0.4	0.2	0.8	0.6	0.4
Product C	1.6	0.6	-	1.0	0.4	0.2	1.2	0.2
Product D	0.6	0.4	1.0	-	0.6	1.2	0.2	0.8
Product E	1.2	0.2	0.4	0.6	-	0.6	0.8	0.2
Product F	1.8	0.8	0.2	1.2	0.6	-	1.4	0.4
Product G	0.4	0.6	1.2	0.2	0.8	1.4	-	1.0
Product H	1.4	0.4	0.2	0.8	0.2	0.4	1.0	-

Source: *Clauß, F.-R. Finze und L. Partsch: »Statistik für Soziologen, Pädagogen, Psychologen und Mediziner. Band I«. Publisher: Verlag Harri Deutsch, Frankfurt 1995, Germany*

Result of study for test soil 1

Sequence	Product A	Product G	Product D	Product B	Product E	Product H	Product C	Product F
Cleaning performance	17.0	16.6	16.4	16.0	15.8	15.6	15.4	15.2

Products joined with a line are not significantly different at a significance level of 95%.

Annex 3 Example of a significance test using single factor analysis of variance (ANOVA) and the Duncan Test

Plastic washing up brushes are used in the dishwashing tests. The brushes may be used several times but have to be thoroughly cleaned after every dishwashing process.

11. Dishwashing process

The tests must be conducted by one or more well trained persons and they are, in principle, to wear washing up gloves. The time specifications must be exactly complied with and the exact holding of the plates and washing up brush during the dishwashing process must also be practiced (Table 2).

The soiled plates are cleaned using circular movements of the brushes on the front and back sides; the plates and the hand are completely immersed in the dishwashing soak (Fig. 3). Then the foam is allowed to run off, any larger amounts of clinging foam is returned with the brush to the dishwashing soak. The washed plate is placed on the draining board and the next plate is picked up. If the foam collects at one

point on the water surface, it should be evenly distributed with the brush over the soak.

The total test length from turning on the tap up to the last plate (including 15 seconds waiting time after the last plate in order to determine whether the foam layer has broken up permanently) is recorded for each test.

The sequence of the dishwashing tests for the test products in a study must be undertaken in a random manner. With a larger number of test products, it may make sense to exchange the test persons within a dishwashing test.

12. End point identification

When the foam layer has broken up permanently on the surface of the dishwashing soak (i.e. at least 15 sec), the dishwashing soak is deemed to be exhausted and the end point has been reached (Fig. 4). The number of washed plates per washing test is recorded in the test protocol.

13. Validation and Documentation

Besides the dishwashing detergents for testing, a standard must also be tested in order to examine reproducibility (standard used to develop this method: Annex 2). If the study is extended over several days, the day results of the standard may not significantly deviate from each other. For each type of soil, five washing tests are to be conducted for each test product. The results are to be presented separately for each type of soil; the presentation of a mean value calculated for both types of soil is not admissible.

A significance test is necessary for a comparative presentation of the dishwashing results. This must be done according to a recognised statistical method, for instance the combination of ANOVA and Duncan Tests (Annex 3). The results for the standard are not included in the significance test.

	Time	Number of circular movements
Cleaning the front side	10 sec	20
Turning the plate over	2 sec	
Cleaning the back side	3 sec	6
Wiping off foam, changing plates	15 sec	

Table 2

Standard dishwashing detergent	(%-data as active content):
Sec. Sodium alkane sulfonate (ex 60 %)	10.8 %
Sodium lauryl ether sulfate 2 EO (ex 70 %)	2.8 %
Cocamidopropyl betaine (ex 30 %)	1.2 %
Kathon CG™ (as received)	0.08 %
Water	ad 100
Mean value from 5 dishwashing tests (dosage 4 ml):	
Indicative value for soil 1: 11 – 15 plates, range ± 10%	
Indicative value for soil 2: 15 – 20 plates, range ± 10%	

Annex 2



Fig. 3 Conduct of a dishwashing test

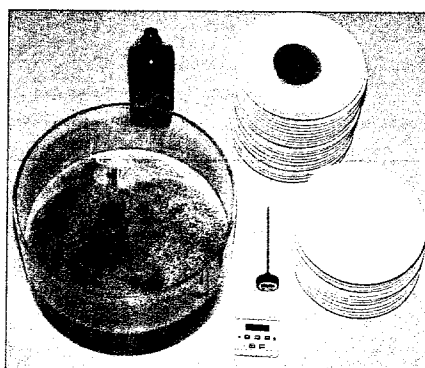


Fig. 4 End point identification

Correspondence addresses:

* Dr. Christian Nitsch
c/o Henkel KGaA
WEH-Dishwashing Detergents
Henkelstraße 67
40191 Düsseldorf
Germany

** Dr. Gerd Hüttmann
c/o Reckitt Benckiser
RSE Dishwashing
Benckiserplatz 1
67059 Ludwigshafen/Rhein
Germany

