

## Influence of Raw Materials on the Formulation of Interior Emulsion Paints from the Point of View of EN 13300

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**Summary:** The prior standard for interior emulsion paints, DIN 53778, was replaced by EN 13300 in April 2001. The new standard includes test standards ISO 11998 (Wet scrub resistance) and ISO 6504-3 (Contrast ratio). The new procedures differ greatly from the old test standards. This paper describes the influence of binders, film-forming agents, dispersants, extenders and pigments on the classification of interior emulsion paints in relation to EN 13300 compared to the prior standard.

The polymer dispersion tests showed that both the old and new test standards indicate similar influences. However, this is not the case with additives. Extenders also have varying effects that are linked to their hardness or other characteristics. The opacity to ISO 6504-3 is affected in various ways by the extenders and the selection of a suitable titanium dioxide pigment.

**Keywords:** new standards; indoor emulsion paints; wet scrub resistance; wash and scrub resistance; contrast ratio

### Introduction

Just under 2.4 million metric tons of emulsion paint were manufactured in Europe in 2001, with Germany contributing roughly 830,000 tons to this total, including 580,000 tons of emulsion paint for indoor use.

Minimum requirements for interior emulsion paints were formerly specified in DIN 53778 Part 1. These essentially provided for data on the assessment of the wash and scrub resistance, and of the opacity as expressed by the contrast ratio. The test specifications were described in standards DIN 53778 Parts 2 and 3. This German Industrial Standard (**Deutsche Industrie Norm**) was replaced by European Standard EN 13300 in April 2001. The methods in the new test specifications, including ISO 11998 (Wet scrub resistance) and ISO 6504-3 (Contrast ratio), differ greatly from DIN 53778 Parts 2 and 3. This paper describes the influence of raw materials on the classification of interior emulsion paints in relation to EN 13300 compared to the prior standard.

### 1. Comparison of the test methods

#### *1.1. Determination of wash and scrub resistance vs. wet scrub resistance (DIN 53778 Part 2 vs. ISO 11998)*

DIN 53778 Part 2 specifies the application of a uniform coating with a dry film thickness of  $100 (\pm 5) \mu\text{m}$ . After 28 days of storage in a standard climate, the coating is scrubbed down to the substrate using a conditioned brush with a known abrasion factor. According to DIN 53778 Part 1, an interior emulsion paint was classified as wash-resistant if it withstood 1,000 scrub cycles, and as scrub-resistant if it withstood 5,000 scrub cycles, without being worn down to the substrate. One weak point was considered to be the visual estimation of when the

coating was worn through, which differed from person to person. Consequently, transfer in an ISO standard was rejected.

To determine the wet abrasion according to ISO 11998, the coating material is preferably applied with a 400 µm doctor blade. Again after 28 days in storage, the wetted film is scrubbed with an abrasive pad using a maximum of 200 scrubs. After drying, the difference in weight is determined and the wet abrasion calculated in µm based on the dry film density. The advantages of this method are the shorter operating time of the scrubbing device (roughly 5 minutes compared to over 2 hours for testing scrub-resistant coatings according to the prior standard) and the more precise determination that results from using a prescribed number of scrubs and calculating the weight difference.

EN 13300 divides paints into the following five classes based on their wet abrasion:

Classification	Wet abrasion	
Class 1	< 5 µm	at 200 scrubs
Class 2	5µm and < 20 µm	at 200 scrubs
Class 3	20 µm and < 70 µm	at 200 scrubs
Class 4	< 70 µm	at 40 scrubs
Class 5	70 µm	at 40 scrubs

*1.2. Comparison of contrast ratio determination according to the old and new standards (DIN 53778 Part 3 and ISO 6504-3)*

The weakness of DIN 53778-3 was that the determination of the contrast ratio with a spreading weight of 300 g/m<sup>2</sup> resulted in virtually no differentiation between the emulsion paints. In other words, virtually every emulsion paint achieved a contrast ratio of at least 98% at this high spreading weight and could thus be classified as opaque. For this reason, emulsion paints were applied to Morest cards with a doctor blade gap of 100 or 150 µm in order to allow differentiation. However, different solids contents, rheological properties and film application speeds resulted in different dry film thicknesses at the same blade gap, meaning that a comparison based on contrast ratio or opacity was subject to error. Therefore, a more precisely defined method was required to determine opacity.

The two methods described in ISO 6504-3 are based on the observation that the contrast ratio is a roughly linear function of the reciprocal spreading rate over a limited film thickness range. If the contrast ratio is determined for a given spreading rate, which EN 13300 requires manufacturers to state as an average value in m<sup>2</sup>/l, reproducible results can be obtained for multiple film thicknesses by interpolating the measured values [1].

Because the wet film thickness usually cannot be determined precisely, the mass per unit area of the respective coating is determined in the methods described and the corresponding wet film thickness calculated accordingly. The density and solids content of the coating must be known for this calculation. Although determining these values means additional laboratory work, being able to calculate the wet film thicknesses is a great advantage compared according to DIN 53778-3.

EN 13300 divides paints into the following four classes based on their contrast ratio:

Classification	Contrast ratio
Class 1	99.5%
Class 2	98.0% and < 99.5%
Class 3	95.0% and < 98.0%
Class 4	< 95.0%

## 2. Influence of binders

The binder plays a key role when formulating emulsion paints. It ensures that the pigments and extenders are bound together and that the emulsion paint adheres to the substrate. Thirty-seven polymer dispersions were tested in a series of trials according to DIN 53778 Part 2, ISO 11998 and other standards.

The formulation shown in Table 1 was used as the base formulation. The pigment volume concentration (PVC) was 78%. Paints containing polymer dispersions with a minimum film-forming temperature (MFT) higher than 5 °C had 1% Texanol as the film-forming agent (FFA). The solids content of the paints was held constant when switching polymer dispersions.

**Table 1. Base formulation**

Formulation	Parts by weight
Water	179.5
In-can preservative	2.0
Dispersant	0.5
Dispersant (45%)	3.0
Defoamer	2.0
Film-forming agent *	10.0
Cellulose paste (3%)	90.0
Titanium dioxide	220.0
CCP 0.3 µm	20.0
CCN 2.5 µm	118.0
CCN 5.0 µm	155.0
Talcum	70.0
Diatomaceous earth	20.0
Polymer dispersion (54.4%)	110.0
Total	1000.0
PVC (%)	78.0

\*The film-forming agent was replaced with water in formulations whose binder displayed an MFT < 5 °C

CCP- calcium carbonate precipitated

CCN- calcium carbonate natural

according to EN 13300. However, there are seven paints that do not fulfil the minimum requirement of 1,000 scrubs, but are still assigned to Class 3 according to EN 13300 because their abrasion is less than 70 µm. Three paints yielded abrasion values > 70 µm.

Triple determinations were carried out. The range of variation in determining the wet abrasion was ± 2.5 µm for the paints assigned to Class 2, and deteriorated to ± 5 µm for the paints in Class 3.

ISO 11998 and DIN 53778 Part 2 yielded matching results for 85% of the paints tested (Fig. 1), in that wash-resistant formulations can be assigned to Class 3 and scrub-resistant ones to Class 2 according to the new standard.

Analysis of the chemical binder composition showed that emulsion paints containing styrene acrylics have the lowest abrasion at an average of 17.6 µm, while those with pure acrylics

Every binder was tested with a special emulsion-paint pigment and a general-purpose pigment. Both TiO<sub>2</sub> pigments were produced by the sulphate process and had different surface treatments and oil absorption values. The oil absorption of the special emulsion-paint pigment is 34 g/100 g pigment, while that of the general-purpose pigment is 18 g (Table 9, Pigments 1 and 4).

Figure 1 compares the wet abrasion test according to ISO 11998 with the wash and scrub resistance test according to DIN 53778 Part 2. Thirty-two paints were classified as scrub-resistant, 30 (94%) of which displayed a wet abrasion of < 20 µm. Two paints, each containing special emulsion-paint pigment 1, were assigned to EN 13300 Class 3 based on their wet abrasion values of 21.3 µm and 23.4 µm.

On the other hand, of the 32 wash-resistant formulations, two paints also containing special emulsion-paint pigment 1 were assigned to Class 2 with wet abrasion values of 19.6 µm and 18.6 µm. The remaining 30 wash-resistant formulations range between 20 µm and 70 µm wet abrasion and are assigned to Class 3

have a mean wet abrasion of 24.1  $\mu\text{m}$  and those with terpolymers based on vinyl acetate have a mean wet abrasion of 35.3  $\mu\text{m}$  (Figure 2). Consequently, the good pigment-extender binding capacity of styrene acrylics is characterised by comparatively low wet abrasion values. Among the terpolymers based on vinyl acetate, a polymer dispersion gave outstanding results with being on the same level as the styrene acrylics and pure acrylics: One difference compared to the other vinyl acetates is its rather broad particle size distribution. This seems to result in a coating film of unusual compactness.

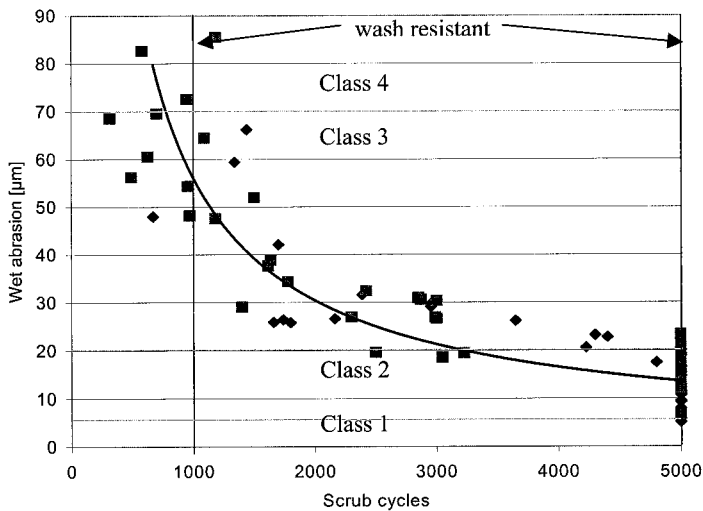


Figure 1. Correlation between of ISO 11998 and DIN 53778 based on a general-purpose pigment  $\blacklozenge$  and a special emulsion-paint pigment  $\circ$  in different polymer dispersions

Twenty-one binders with an MFT below 5  $^{\circ}\text{C}$  were tested. They yielded a mean wet abrasion of 44.9  $\mu\text{m}$  with the special emulsion-paint pigment and 27.5  $\mu\text{m}$  with the general-purpose pigment. The lower wet abrasion values with the general-purpose pigment can be attributed to the lower oil absorption and the associated lower binder demand. The wet abrasion of the 16 binders with an MFT above 5  $^{\circ}\text{C}$  averaged 24.4  $\mu\text{m}$  with the special emulsion-paint pigment and 13.7  $\mu\text{m}$  with the general-purpose pigment.

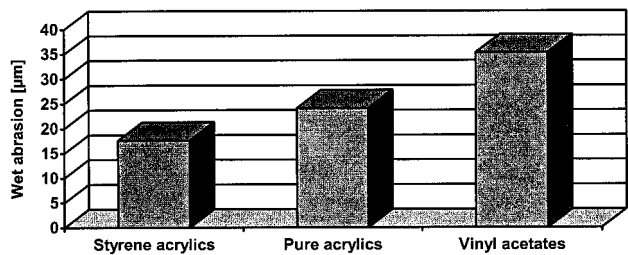


Figure 2. Mean wet abrasion with various polymer dispersions

Polymer dispersions with an MFT above 5 °C generally have a higher glass transition temperature and better mechanical properties. This results in harder films with lower wet abrasion (Figure 3).

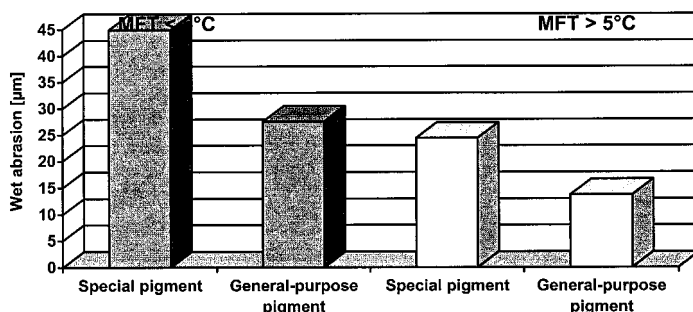


Figure 3. Mean wet abrasion of polymer dispersions with MFT < 5 °C and MFT > 5 °C

Because there is a growing trend towards VOC-free coatings in interior emulsion paints, the mechanical superiority of binders with an MFT above 5 °C can only be exploited if some VOC content is accepted.

### 3. Influence of film-forming agents

The lowest temperature at which a binder can form a film is decisive for the formulation of coatings. If the MFT of the polymer dispersion is higher than 5 °C, substances must be added to lower the MFT. This task is handled by film-forming agents (FFA). The higher the MFT, the greater the quantity of FFA that must be added.

A well spread styrene acrylic with a MFT of 20 °C was used as the polymer dispersion in the base formulation. Texanol, Lusolvan FBH and Kristallöl 30 were added at 1% each as FFAs.

#### 3.1. Comparison of film-forming agents

The scrub resistance increases (DIN 53778) in the order Kristallöl 30, Lusolvan FBH and Texanol (Table 2). The selected products also displayed significantly different abrasion values in previous in-house tests conducted by KRONOS. Upon an exchange of FBH the performance of a coating can be improved by some 500 cycles.

**Table 2. Test results for film-forming agents**

	DIN 53778	ISO 11998	ISO 6504-3
Film-forming agent	Scrub cycles	Wet abrasion [µm]	Spreading rate [m <sup>2</sup> /l] for Class 1
Texanol	2,590	12.5	< 10.00
Kristallöl 30	2,060	14.0	< 10.00
Lusolvan FBH	2,300	14.4	< 10.00

The wet abrasion (ISO 11998) of the test formulation containing 1% FFA by weight varies only by 2  $\mu\text{m}$ . That the difference is not significant unlike the results after the old standard may be due to the much shorter test time (5 minutes instead of more than 2 hours). The wash liquid thus leads to considerably less swelling of the coating film. As a result, the water sensitivity of the coating film is of less importance in the wet abrasion test according to ISO 11998 than in the scrub resistance test according to DIN 53778 Part 2. There is no evidence of an influence on the opacity according to ISO 6504-3.

#### 4. Influence of dispersants

The mere objectives of dispersants are to facilitate the wetting of the pigments and extenders and to stabilize the state of dispersion achieved. How excessive dispersant can have a negative effect on wash and scrub resistance is shown in [2]. Other in-house tests conducted by KRONOS have indicated that especially sodium polyphosphate lowers this resistance.

The base formulation containing a styrene acrylic was used to test the influence of dispersants. The first paint contained a combination of sodium polyphosphate and the sodium salt of a polymeric carboxylic acid. One of the dispersants was used alone in each of the other two paints. The total concentration of the solids content of the dispersant was 0.3% by weight referred to the proportion of pigment and extender, or 0.185% w/w for the overall formulation.

##### *Comparison of dispersants*

The best abrasion values are obtained with the paint containing the sodium salt of a polymeric carboxylic acid. The abrasion values deteriorate as the quantity of sodium polyphosphate increases. As in the FFA test, however, the wet abrasion varies 2  $\mu\text{m}$  only and no differentiation is possible. This can probably also be attributed to the shorter action time of the wash liquid. There is no evidence of an influence on the opacity according to ISO 6504-3.

**Table 3. Test results for dispersants**

	DIN 53778 Part 2	ISO 11998	ISO 6504-3
Dispersant	Scrub cycles	Wet abrasion [ $\mu\text{m}$ ]	Spreading rate [ $\text{m}^2/\text{L}$ ] for Class 1
Carboxylic acid	2,500	13.0	< 10.00
Polyphosphate/carboxylic acid	2,290	13.8	< 10.00
Polyphosphate	2,100	15.1	< 10.00

#### 5. Influence of extenders

In terms of quantity, extenders are the main constituents of interior emulsion paints. Thus, they also influence the optical properties of the paint directly - via their brightness and tone - and indirectly - via their effect on the distribution of the titanium dioxide pigment. They also affect the other properties, such as the ease of application, storage stability, mud cracking, sheen, wet abrasion and scrub resistance.

The styrene acrylic selected resulted in scrub-resistant paints, i.e. differentiation was not possible using DIN 53778 Part 2. For this reason, the PVC of the base formulation was increased from 78% to 80% by reducing the binder quantity, in order to allow differentiation.

### 5.1. Comparison of extenders

The extenders were compared to one another in the following groups, depending on their particle shape and size: fine extenders (with a separate investigation comparing different fine calcium carbonates), coarse extenders, coarse lamellar extenders and matting agents.

#### 5.1.1. Influence of very fine extenders

The quantity of very fine extenders added was only 2%, as higher concentrations can lead to an increase in internal film tension and thus also to an increase in mud cracking. Furthermore, the high binder demand can result in a decrease in the number of scrubs. Precipitated calcium carbonate, precipitated aluminium silicate, natural kaolin and a calcined kaolin were tested.

Despite the low concentration used, the above tested extenders have a marked effect on the test results. The number of scrub cycles varies between 1,580 and 2,590 (Table 4). The good performance of precipitated calcium carbonate compared to the other extenders could be explained by its lower oil absorption and the associated lower binder demand. The emulsion paints with natural kaolin and those with calcined kaolin withstood fewer scrub cycles than the paints with precipitated aluminium silicate. This is a remarkable result because the oil absorption of the aluminium silicate is by approx. 3-fold of the kaolines.

Comparing the kaolins to one another shows that the formulation with the calcined kaolin achieves more scrub cycles than that containing natural kaolin. Since the wet abrasion varies by a maximum of only 2.5  $\mu\text{m}$ , the results do not reveal a clear trend.

**Table 4. Test results of very fine extenders**

	Precipitated aluminium silicate	Precipitated calcium carbonate	Natural kaolin	Calcined kaolin
Particle shape	Roundish	Cigar-shaped	Fine-lamellar	Amorphous
Mean particle size [ $\mu\text{m}$ ]	0.035	0.3	0.5	1.4
Oil absorption [g/100 g]	150	26	48	55
Whiteness	95	97	88	91
Brightness L*	97.5	97.4	97.4	97.3
Tone (white) b*	2.25	2.31	2.39	2.38
Tinting strength L *	57.1	56.2	57.2	56.3
Tone (grey) b*	-2.32	-2.59	-2.50	-2.54
Porosity $\Delta L^*$	5.7	5.1	4.4	5.6
Sheen (85° gloss)	1.6	1.4	1.5	1.3
Scrub cycles, DIN 53778	2,370	2,590	1,580	2,100
Wet abrasion [ $\mu\text{m}$ ], ISO 11998	13.2	12.5	14.3	15.0
Spreading rate [ $\text{m}^2/\text{l}$ ] for Class 1, ISO 6504-3	< 10.80	< 10.00	< 10.00	< 9.30

According to ISO 6504-3, the reproducibility of the opacity test is 4%. Consequently, a formulation with precipitated aluminium silicate has an advantage over paint containing precipitated calcium carbonate or natural kaolin in terms of the spreading rate, which is 0.8 m<sup>2</sup>/l higher for Class 1. In contrast, a slight disadvantage results with calcined kaolin. This could be attributed to the calcination process, in which the natural kaolin loses its lamellar structure and becomes amorphous. Experience has shown that the distribution of titanium dioxide pigment in the coating film is somewhat less uniform as a result.

5.1.2. Influence of different fine calcium carbonates

The following calcium carbonates were tested separately in the base formulation: 11.8% limestone powder with a mean particle size of 0.7 µm, marble powder with 0.9 µm and 2.5 µm, and chalk with 3.0 µm. The emulsion paint containing chalk withstands the most scrub cycles, followed by that containing the coarser marble powder and that with limestone powder. The wet abrasion was not clearly different. It is conceivable that the chalk generates lubricious abraded material and the scrub brush therefore removes less of the coating film. This results in comparatively good abrasion values. In contrast, the coarse abrasive pad with the 135 g mount is much more abrasive than the scrub brush. The other calcium carbonates display higher wet abrasion in keeping with the low abrasion values.

**Table 5. Test results of fine calcium carbonates (CCN)**

	Limestone	Marble	Marble	Chalk
Particle shape	Trigonal rhombohedral	Trigonal rhombohedral	Trigonal rhombohedral	Amorphous
Mean particle size [µm]	0.7	0.9	2.5	3.0
Oil absorption [g/100 g]	20	20	19	17
Whiteness	93.5	95.0	94.5	84.5
Brightness L*	97.5	97.3	97.4	96.2
Tone (white) b*	2.48	2.17	2.31	3.07
Tinting strength L *	58.2	58.3	56.2	55.8
Tone (grey) b*	-2.53	-2.30	-2.59	-2.61
Porosity Δ L*	3.6	4.4	5.1	4.4
Sheen (85° gloss)	1.8	1.7	1.5	1.5
Scrub cycles, DIN 53778	1,950	1,690	2,590	3,120
Wet abrasion [µm], ISO 11998	13.4	14.4	12.5	13.2
Spreading rate [m <sup>2</sup> /l] for Class 1, ISO 6504-3	< 8.40	< 10.00	< 10.00	< 10.20

As regards the opacity according to ISO 6504-3, only the formulation containing limestone powder shows a somewhat poorer spreading rate, this probably being attributable to the relatively low porosity of the coating surface and the lower dry-hiding effect associated with it.

5.1.3. Influence of relatively coarse extenders

The following were tested separately as relatively coarse extenders: 15.5% marble powder with a mean particle diameter of 7 µm and 5 µm, and cristobalite with 5 µm. The paint with the coarser marble powder displayed the highest scrub values and the lowest wet abrasion due to its low oil absorption. It was followed by the 5 µm marble powder and, after a fairly large interval, by the cristobalite. Although the mean particle size of this marble powder and the



cristobalite are the same, the upper particle size range of the cristobalite is lower. This is manifested in a higher oil absorption and thus explains the lower scrub value. However, the advantage of the 5  $\mu\text{m}$  marble powder is no longer apparent when it comes to wet abrasion. It can be assumed that this is due to the greater hardness of the cristobalite (Mohs hardness 6-7; marble: Mohs hardness 3).

As regards the opacity according to ISO 6504-3, both 5  $\mu\text{m}$  extenders are on the same level. However, the formulation with the 7  $\mu\text{m}$  calcium carbonate has a much lower spreading rate. In addition to the poorer titanium dioxide dispersion, this can also be attributed to an effect best described by the term "optical window". The 7  $\mu\text{m}$  extender has an upper particle size range of 30  $\mu\text{m}$ . If two or more of these coarse extender grains are on top of one another they extend from the coating surface all the way to the substrate. Because there are virtually no differences in the refractive indices of the extenders and the polymer dispersion, light rays are not scattered at their boundary surfaces, but rather pass through them, as through a window. The result is lower opacity. On the other hand, the upper particle size range produces a somewhat rougher surface, so that the sheen can be reduced.

**Table 6. Test results of relatively coarse extenders**

	Cristobalite	CCN, marble	CCN, marble
Particle shape	Trigonal rhombohedral	Trigonal rhombohedral	Trigonal rhombohedral
Mean particle size [ $\mu\text{m}$ ]	5	5	7
Oil absorption [g/100 g]	27	16	15
Whiteness	94	94	93.5
Brightness L*	97.4	97.4	97.3
Tone (white) b*	2.25	2.31	2.34
Tinting strength L *	56.9	56.2	55.6
Tone (grey) b*	-2.24	-2.59	-2.72
Porosity $\Delta L^*$	5.9	5.1	5.0
Sheen (85° gloss)	1.3	1.5	1.1
Scrub cycles, DIN 53778	990	2,590	2,750
Wet abrasion [ $\mu\text{m}$ ], ISO 11998	12.7	12.5	9.6
Spreading rate [ $\text{m}^2/\text{l}$ ] for Class 1, ISO 6504-3	< 10.00	< 10.00	< 7.70

#### 5.1.4. Coarse lamellar extenders

Talcum, mica and an intergrowth of mica, quartz and chlorite were tested as lamellar extenders at a proportion of 7% (w/w) referred to the base formulation. The number of scrub cycles increases and the wet abrasion decreases in the order mica, talcum and mineral intergrowth. While talcum and mica are relatively soft, with Mohs hardness values of 1 and 3, respectively, the mineral intergrowth has a higher overall hardness due to the quartz content (Mohs hardness: 7).

As a result of their brightness, the extenders influence the optical properties of the paints. The dark mica produces a brightness value in the emulsion paint that is a good unit lower than that of the talcum. This higher light absorption contributes to the elevated opacity of the paint containing mica, as determined according to ISO 6504-3. In contrast, the formulations

containing talcum and the mineral intergrowth require lower spreading rates to achieve the highest opacity class.

**Table 7. Test results of coarse lamellar extenders**

	Talcum	Intergrowth	Mica
Particle shape	Coarse lamellar	Coarse lamellar	Coarse lamellar
Mean particle size [μm]	13	25	27
Oil absorption [g/100 g]	32	22	37
Whiteness	91.0	77.5	Not available
Brightness L*	97.4	96.9	96.3
Tone (white) b*	2.31	2.41	2.70
Tinting strength L *	56.2	55.5	56.2
Tone (grey) b*	-2.59	-2.69	-2.29
Porosity Δ L*	5.1	5.0	6.2
Sheen (85° gloss)	1.4	1.5	1.5
Scrub cycles, DIN 53778	2,590	4,630	750
Wet abrasion [μm], ISO 11998	12.5	7.3	22.9
Spreading rate [m <sup>2</sup> /l] for Class 1, ISO 6504-3	< 10.00	< 7.80	< 12.00

5.1.5. Influence of diatomaceous earth and cellulose fibre

On account of the irregular particle shape of diatomaceous earth and the fibrous structure of cellulose fibres, both have a matting effect. Diatomaceous earth is more effective in reducing sheen.

**Table 8. Test results of diatomaceous earth and cellulose fibre**

	Diatomaceous earth	Cellulose fibre
Particle shape	Chalk-like	Fibrous
Mean particle size [μm]	6	40 x 20
Oil absorption [g/100 g]	110	120
Whiteness	88	84
Brightness L*	97.4	97.3
Tone (white) b*	2.31	2.38
Tinting strength L *	56.2	56.0
Tone (grey) b*	-2.59	-2.68
Porosity Δ L*	5.1	4.0
Sheen (85° gloss)	1.5	1.8
Scrub cycles, DIN 53778	2,590	3,200
Wet abrasion [μm], ISO 11998	12.5	14.0
Spreading rate [m <sup>2</sup> /l] for Class 1, ISO 6504-3	< 10.00	< 6.40

The cellulose fibre leads to the higher scrub values. However, lower wet abrasion is achieved with diatomaceous earth. It cannot be ruled out that the soft cellulose fibres are virtually plucked out of the coating surface by the abrasive pad, whereas the scrub brush does not work the coating as hard. The decrease in opacity when using cellulose fibre is pronounced. This may be due to the fibre size, which could cause the "optical window" effect as experienced with the coarse calcium carbonates.

## 6. Influence of titanium dioxide pigments

No other raw material lends coatings such high opacity and tinting strength as titanium dioxide. This is due to the high refractive index of the white pigment and the great difference in refractive index compared to the polymer dispersions.

The five titanium dioxide pigments characterised in Table 9 were tested with four selected polymer dispersions in the base formulation with a PVC of 80%. Pigments 1 and 2 are highly surface-treated special emulsion-paint pigments, while  $\text{TiO}_2$  grades 4 and 5 can be characterised as general-purpose pigments with less surface treatment. Pigment 3 is an intermediate grade, as indicated by the inorganic surface treatment and oil absorption.

Each of the four binders forms a film at temperatures below 5 °C and is thus suitable for VOC-free coatings.

**Table 9. Characterisation of the titanium dioxide pigments**

	$\text{Al}_2\text{O}_3$ [%]	$\text{SiO}_2$ [%]	$\text{ZrO}_2$ [%]	Oil absorp. [g/100 g]	Density [g/cm <sup>3</sup> ]	C [%]
Pigment 1	4.0	10.0	-	34	3.7	-
Pigment 2	4.4	9.4	-	41	3.6	-
Pigment 3	4.7	3.4	-	27	3.8	0.10
Pigment 4	3.1	-	0.4	18	4.1	0.19
Pigment 5	3.9	-	-	16	4.1	0.19

### 6.1. Analysis according to DIN 53778-2 and ISO 11998

The scrub resistance test according to DIN 53778 Part 2 and the wet abrasion test according to ISO 11998 yield matching results. As a rule, the paints with more scrub cycles also have lower wet abrasion (Figures 4 and 5). The best values are achieved with general-purpose pigments (paint-grade pigments) 4 and 5. Special emulsion-paint pigments 1 and 2 result in the lowest scrub values and the highest wet abrasion. Pigment 3 occupies the middle field among the styrene acrylics.

The polymer dispersions used have a very strong influence on the differentiability of the titanium dioxide pigments tested. The pigments tested with the vinyl acetate-ethylene polymer dispersions having a fairly broad particle size distribution (0.1 to 0.55  $\mu\text{m}$ ) display only slight differences of approximately 300 scrub cycles and 3  $\mu\text{m}$  for wet abrasion. A much stronger pigment influence is observed with the styrene acrylics. Differences of up to 3,000 scrub cycles and up to 17  $\mu\text{m}$  for wet abrasion are found between the special emulsion-paint pigments and the paint-grade pigments in this case.

The vinyl acetate-ethylene benefits pigments 1 and 2, while pigments 4 and 5 do not display any advantages at all in this formulation. In contrast, pigment 3 behaves indifferently. This binder achieves higher scrub values and lower wet abrasion with pigments 1 and 2 than the three styrene acrylics. The wash-resistant coatings (approx. 1,700 scrub cycles) display a wet

abrasion of only approx. 19  $\mu\text{m}$  and are thus assigned to Class 2 to EN 13300. In contrast, the styrene acrylics offer advantages in combination with general-purpose titanium dioxide pigments 4 and 5. These formulations achieve more scrub cycles than paints formulated with vinyl acetate-ethylene. Styrene acrylic 1 yields particularly positive results in terms of wet abrasion and the number of scrub cycles.

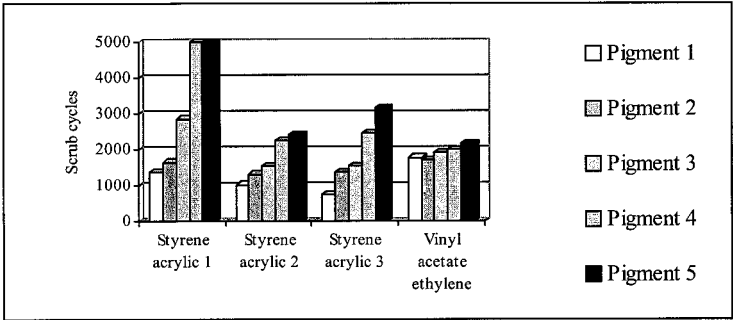


Figure 4. Wash and scrub resistance according to DIN 53778 Part 2

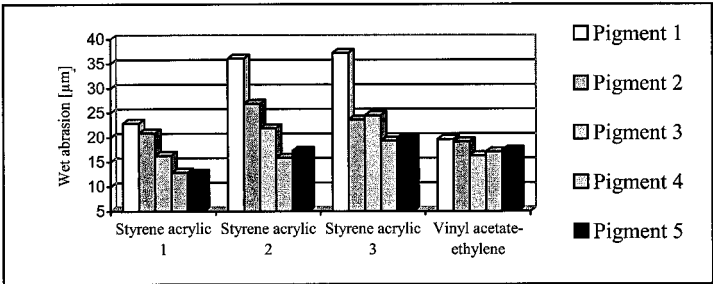


Figure 5. Wet abrasion according to ISO 11998

6.2. Analysis of opacity according to ISO 6504-3

The larger the surface area that can be coated opaquely with one litre of coating material, the more economic the coating is. Titanium dioxide pigments with a fairly high oil absorption value can be expected to achieve greater opacity due to the higher resulting porosity and the associated stronger dry-hiding effect. This is confirmed in some cases by the considerably higher spreading rates of the paints pigmented with pigments 1, 2 and 3 (Figure 6). Comparing general-purpose titanium dioxide pigments 4 and 5 shows that pigment 4, which was produced by the sulphate process, leads to a higher spreading rate in every paint tested. This is an often observed phenomenon which is commonly explained by the lower brightness and less pronounced bluish undertone of this kind of pigments. The following argument may explain the good results of the combination of the vinyl acetate-ethylene polymer dispersion with pigments 1, 2 and 3: the pigments are very highly surface-treated with  $\text{Al}_2\text{O}_3$  and  $\text{SiO}_2$  (less in pigment 3). In emulsion paints with a high extender content, this surface treatment acts like a "spacer" between the pigment particles and thus prevents excessively dense packing of the titanium dioxide particles, which would otherwise lead to a decline in the optical performance of the pigment. In conjunction with the broader particle size distribution (0.1 to 0.55  $\mu\text{m}$ ) of the vinyl acetate-ethylene polymer dispersion, it would appear that this results in a better packing density for the interior emulsion paint produced.

Pigment 3 particularly recommends itself as a good compromise between low wet abrasion values and high opacity.

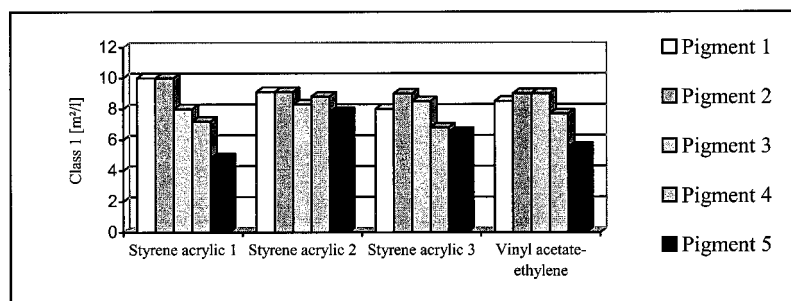


Figure 6. Spreading rate [ $\text{m}^2/\text{l}$ ] with different  $\text{TiO}_2$  pigments

## Summary

Thirty-seven polymer dispersions were tested in accordance with DIN 53778 Part 2, and ISO 11998. The results indicated that the old and new standards are comparable. Styrene acrylics displayed the lowest wet abrasion, followed by pure acrylics and terpolymers based on vinyl acetate. One dispersion in the latter group with a fairly broad particle size distribution had a particularly low wet abrasion value. Polymer dispersions with a minimum film-forming temperature (MFT) above  $5^\circ\text{C}$  display lower wet abrasion than those with an MFT below  $5^\circ\text{C}$ .

The results of tests with film-forming agents (FFA) and dispersants reveal distinct differences between the standards. The influence of raw materials on the scrub values determined by the old standard is much stronger than in the wet abrasion test according to the new standard. While there are only signs of a trend in the new standard, the water swellability and sensitivity of the raw materials in the coating hardly play a role any more due to the shorter action time of the wash liquid.

Extenders affect the results of tests according to EN 13300 in a variety of ways. The particle shape and particle size of extenders influence the dispersion of titanium dioxide pigments and thus play a part in the opacity according to ISO 6504-3, which is also affected by their brightness and tone, as well as dry-hiding. The higher the oil absorption or binder demand of the extenders, the greater the dry-hiding effect and thus the opacity caused by them, while the scrub and wet abrasion resistance deteriorate at the same time. Greater opacity can also be caused by stronger light absorption combined with lower extender brightness, as is the case with mica.

Relatively hard extenders apparently result in lower wet abrasion than the number of scrub cycles would suggest. This may be attributable to the greater abrasiveness of the abrasive pad, which is much harder than the scrub brush on fairly soft extenders in the coating film, such as chalk and cellulose fibres. The lowest wet abrasion value was achieved with a mineral intergrowth.

Different interior emulsion paint formulations are possible as regards the titanium dioxide pigment and the binder. In combination with styrene acrylics, general-purpose titanium dioxide pigments are the recommended choice because the low binder demand results in low wet abrasion values. Of the two general-purpose pigments, titanium dioxide 4 produced by the sulphate process displays definite advantages in terms of opacity according to ISO 6504-3. Another option is to combine a vinyl acetate-ethylene polymer dispersion having a fairly broad particle size distribution with a highly surface-treated pigment. The wet abrasion of this

combination is far lower than with the styrene acrylics tested. As the high binder demand of these pigments brings about a marked dry-hiding effect, very high opacity according to ISO 6504-3 is achieved in this way. When using this binder, however, the best results for wet abrasion and opacity are obtained with pigment 3, which is less surface-treated than pigments 1 and 2. Good compromises can also be achieved with this pigment in combination with the styrene acrylics.

## References

- [1] pr DIN ISO 6504-3, November 2001
- [2] KRONOS Titandioxid in Dispersionsfarben, 1989