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INFLUENCE OF THE AGE OF EPOXY DISPERSION ON THE EFFECTIVENESS OF PROTECTION OF CONCRETE SURFACES

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Abstract. The scope of this paper is to test stability and mechanical and chemical properties of a modern freshly mixed epoxy dispersion, and as a variant, of a dispersion which is two years old. During ageing, the dispersion particles probably agglomerate, which is tested by laser diffraction and by sedimentation in a cylinder. Physical-mechanical tests assess the appearance, drying speed and mechanical properties of the film. Chemical tests focus on assessing the resistance of films of dispersions of different ages to common chemicals and to chemical defrosting agents. It is possible to say that two years of dispersion storage lead to a considerable increase in the diameter of a certain number of dispersion particles with the subsequent effect of increasing the sedimentation speed. The hardened film of a two-year old dispersion dries faster, has a rougher surface, is softer, but on the other hand, is less elastic and its adhesive capacity to the substrate is worse. Its chemical resistance is decreased but its defrosting agent resistance remains good. Due to the two-year storage of the epoxy dispersion, most of the monitored parameters decrease, nevertheless, certain protection of concrete structures by a hardened film of such epoxy dispersions is possible.

Keywords: epoxy dispersion, size distribution, mechanical stability, chemical stability, protection of concrete.

1. Introduction

In the last decades there has been a visible trend to evaluate the quality of building structures according to their anticipated life, or as the case may be, according to the durability of its components. In the case of concrete structures widespread nowadays, their long-term durability is determined by the quality of execution and subsequent protection of the 20–50 mm thin surface layer of the structure, the so-called ‘covercrete’. Building material engineers try hard to draw up optimum formulas of durable concrete mixtures; the already existing older or newer structures can subsequently be protected by impregnation or paint (Hošek 1996). In the second half of the 20th century, various types of resins and of their water dispersions began to appear and spread quickly in the building practice (Padget 1994).

These were first of all resins based on acrylates or their copolymers with styrene or vinyl acetate (Alyamac, Soucek 2011; Okamoto *et al.* 1996; van de Wiel, Zom 1981). They are mainly used in the modification of cement concretes and in the preparation of refurbishment mixes (Hwang *et al.* 2007; Dumitru, Jitaru 2011). Two-part resins on the basis of polyesters, epoxides, polyurethanes and other substances have been successfully used for pressure grouting, for the preparation of polymer concretes and for the surface protection of concrete and other building structures (Sebök 1985; Schulze *et al.* 1990). Water-based resin dispersions were put on the market in the CR (former CSSR) rather late, e.g. acetate and acrylic dispersions in the 1970s, epoxy dispersions in the 1980s and 1990s (Richardson 1988). Although belonging to relatively new products, epoxy dispersions have undergone a certain development in the course of their short existence (Ohama 1995; Zhen-zhong 2007; Oosterlinck *et al.* 2009). Type I dispersions are based on waterborne low-molecular resins. Their drawback is, however, that they are too fragile. Type II dispersions are based on the dispergation of medium- or high-molecular epoxy resin in water but contain a certain amount of auxiliary solvent for the purpose of achieving the necessary qualities. Type III dispersions are epoxy dispersions containing either no or only a tiny amount of auxiliary organic solvents (Michalski 2006). These binders are, together with the 'high solids' systems and powdered or radiation-hardened binders, included in the so-called eco-friendly coatings (Novák 2006, 2008, 2009; Shoaf 2010).

The development of these modern coating compositions was brought about by the development of new processing technologies on the one side, and by the changing legislation focused on air protection on the other. It was European Directive 1999/13/EC and subsequently Directive 2004/42/EC of the European Parliament and of the Council on the limitation of emissions of volatile organic compounds due to the use of organic solvents in certain paints and varnishes that had a great impact on the structural changes in the area of production and processing of coatings. The directive anticipated the reduction of emissions in two stages, namely by 2007 and by 2010. On the grounds of their current wide usage, representatives of type III dispersions were chosen for testing.

The research presented in this paper aims to clarify possible changes in the functional properties of epoxy dispersions in the course of their storage at room temperature. The decision about the practical use of an epoxy dispersion at the end of or after the storage (usable) life declared by the manufacturer for the protection of ceramic or metal surfaces is neither easy nor unambiguous. These considerations can perhaps be facilitated by comparing the mechanical properties and chemical resistance of dry films formed by the tested dispersions with markedly different storage times.

In the past, this issue was addressed by several authors. For example Michalski (2006) and Novák (2009) evaluated the change in stability of liquid dispersion due to ageing by means of comparing the viscosities. Wegmann (1995, 1997) examined a similar area focusing on changes in dispersion stability at temperatures around the freezing point. Zubielewicz and Królikowska (2009) assessed the ageing of epoxy dispersion in relation to adhesion and protective properties of the hardened paint film. Most authors attribute the change in dispersion stability caused by ageing to a change in contents and composition of solids and to the amount and type of emulsifiers and solvents. The current research described in this paper further develops the theory of the influence of a change in dispersion solids due to ageing on the properties of both liquid and hardened dispersions. The laser diffraction method, carried out in cooperation between Brno University of Technology and Resin Research Institute SYNPO Pardubice, was used to explain the change in the distribution of particles in both old and new dispersion. The results of the physical-mechanical and chemical tests of a hardened paint film of both old and new dispersion will supplement the theoretical knowledge derived from the laboratory assessment of a change in dispersion particles distribution.

2. Materials and methods

2.1. Materials

In order to compare the stability of fresh dispersions with that of dispersions which were stored for a period of two years, two batches of type III epoxy dispersion CHS EPOXY 160V55, hereinafter referred to as E 160V55 (Novák 2006, 2008, 2009), containing no auxiliary organic solvents were chosen. The dispersion's viscosity is 400 mPa.s/25°C, the content of nonvolatile components is 57%, the content of volatile organic components, the so-called 'VOC' (volatile organic components) is 0 g/l. As a hardener component, we used Telalit 1040 dosed in the mass ratio of 100:10. The viscosity of the hardener is 25 mPa.s/25°C.

2.2. Methods

2.2.1. Laser diffraction and sedimentation in a cylinder

A change in dispersion stability can be caused by many processes which manifest themselves by a change in particle size and thus by a change in viscosity and in sedimentation velocity (Loria *et al.* 2011; Overbeek 2011). Two methods of testing the stability of the new and old dispersion were chosen – laser diffraction and sedimentation in a cylinder. Laser diffraction determines the size of dispersion particles in a liquid. Laser beam passes through a cuvette containing the measured sample dispersed in water. Due to particles present the laser beam bends (diffracts) at an angle which is inversely proportional to their size. The size of particles is subsequently determined on the basis of the Fraunhofer diffraction image. In the case of 'sedimentation cylinder' method, the measured values indicate the number of millilitres (divisions on the graduated cylinder) of the settling dispersion remaining from the original 100 ml of the white coloured column. The sedimentation of dispersion particles was determined in two dilutions within one month.

2.2.2. Subjective appearance, drying speed and mechanical properties of paint film

Similarly as in the case of a liquid dispersion, the ageing of the original dispersion also manifests itself in the properties of hardened films. The following two methods were used for mechanical testing of hardened paint films created on standard surfaces. The first method determined the subjective appearance and the drying speed of paint film with the subsequent derivation of the degree of drying out. The degree of drying out 1–5 is characterized by a type of weight from the mass scale 1 to 5 which does not imprint into the paint any more. The second method determined the selected mechanical properties of hardened dispersion films by means of several standard techniques. In the pendulum damping test (ISO 1522) the hardness is expressed by the number of oscillations made by the pendulum within the specified limits of amplitude. The measured percentage of the film hardness is related to the 100% hardness of the etalon, i.e. of the glass slide. Cylindrical mandrel bend test (ISO 1519) is based on determining the resistance of coatings to cracking or detachment from a metal substrate when subjected to bending round a cylindrical mandrel. The result is given by the diameter (mm) of the first mandrel in descending order over which the coating cracks. Impact resistance (ISO 6272) is expressed as the height (cm) of a weight which, after a fall on the testing panel, does not cause damage to the paint in the convex/concave of the indentation. The cupping test (ČSN 67 3081) in the Erichsen apparatus is based on determining the depth (mm) of indentation of the spherical nose punch with a diameter of 20 mm into the substrate sheet metal with a coating at the moment of the film cracking on the convex part of the indentation. Cross-cut test (ISO 2409) consists in making 6 parallel cuts and 6 perpendicular cuts into the coating through to the substrate metal. During cutting the appearance of the right-angle lattice pattern is observed and the adhesive capacity is assessed in grades 1 (lattice squares adhere strongly to the substrate, traces of the cut are sharp and smooth) to 5 (almost all or all squares of the lattice are separated from the substrate, the coating is raised and peels off during the first parallel cut already).

2.2.3. Chemical resistance using both coating and building testing methods

The chemical resistance of hardened dispersion films of different ages was tested by means of two methods – the coating testing method and the building testing method. In the case of the coating method of testing the chemical resistance of the paint film, we chose one of the standardized tests included in Czech standard ČSN EN ISO 2812-2 Determination of resistance to liquids – Part 1: General testing methods – Method 2 (Method using an absorbent medium). The principle of this method is a description and classification of the reaction of a liquid absorbed in an absorbent disc with a film of the tested dispersion on a glass substrate. The chemical resistance of dispersion films was evaluated from the point of view of the chemicals' influence time: after 2 h (short-term resistance), after 1 day (medium-term resistance) and after 7 days (long-term resistance). To carry out a more substantial and perhaps also more objective evaluation of the resistance of paints beyond the scope of the above mentioned standard ČSN EN ISO 2812-2, we used our own scale evaluating the following types of defects of the dried paint:

- no change observed in any parameter (marked 0);
- bubbles (0 – no bubbles, B2 – isolated bubbles, B3 – bubbles all over the surface).

The second test of chemical resistance of dispersion films of various ages was basically carried out in accordance with Czech standard ČSN 73 1326 Change Z1 Determination of cement concrete surface resistance to water and chemical deicing agents – Part C: Automatic cycling method. Unlike in the above mentioned standard, however, we did not test the chemical resistance of the concrete itself but that of the dispersion paint applied to its surface. Two triplets of standard concrete cubes with an average compressive strength of 20 MPa and approximately one year old were provided with two coats of paint on one demolded area at an interval of 24 hours. The evaluation was carried out after one month from the application of paint. As a hardener we used Telalit 1040. Subsequently the cubes were fitted with a sheet metal frame delimiting the painted area proper. The frame made it possible to constantly keep a min. 5 mm water column of a 3% solution of NaCl in water during the cycles of freezing and thawing. As it was necessary to visually check the possible damage to the dispersion film protecting the concrete surface, we chose a combination of manual and automatic cycling for the test. One cycle took exactly 1 day, i.e. 24 hours: 3 samples painted with the old dispersion and three samples painted with the new dispersion including the 5 mm column of a 3% solution of NaCl were placed in the freezing box at a temperature of −18°C, and then transferred to an environment with a temperature of +20°C for 5 hours. The cycling was ended after 60 days.

3. Results and discussion

3.1. Change in the stability of a liquid dispersion

In the course of ageing, dispersions undergo a whole range of processes which manifest themselves externally by, among others, a change in particle size. The newly created larger agglomerates have then a greater tendency towards sedimentation (Ganguly, Chakraborty 2011). As a result of this process, also viscosity (Novák 2006; Ehrlich *et al.* 1973), which is generally dependent on the number and size of particles in a unit of capacity, is changed. The smaller the particles and the higher their number, the higher the viscosity of the dispersion (Michalski 2006). The following figure (Fig. 1), a laser diffraction apparatus record, captures the changes in particle distribution caused by a two-year ageing period of the dispersion in the laboratory environment at a temperature of 20°C. Columns (frequency histogram) show the occurrence frequency and curves show the cumulative distribution function of particles with regard to their size.

From the comparison of histograms and cumulative distribution functions it is apparent that the fresh dispersion shows greater stability. This is in accordance with one peak of the left frequency histogram with a corresponding particle diameter of approx. 2 µm. The first/left cumulative distribution function determines the maximum diameter of particles existing in the fresh dispersion to be approximately 5 µm. In the case of the two-year old dispersion, it comes to a shift in the frequency histogram of particle diameters and to its division (Fig. 1, peaks 2 and 3 from the left), and the dispersion becomes unstable. Approximately 77% of particles remain slightly increased in size with a diameter of 3 µm, 23% of particles is made by large agglomerates with a diameter of approx. 90 µm (Fig. 1, the second/right derived curve). One of the causes of the change in dispersion stability after two years of storage is therefore an increase in the dimensions of approximately 1/4 of particles with a diameter of 2 µm to the new 90 µm, which is 45 times as much as regards dimensions. The above given values of particle diameters were read from the x-axis of Fig. 1 for the peaks of histograms or for the ends of curve bends of cumulative distribution functions. The change in dispersion stability due to ageing is naturally accompanied by a decrease in viscosity, acceleration of sedimentation

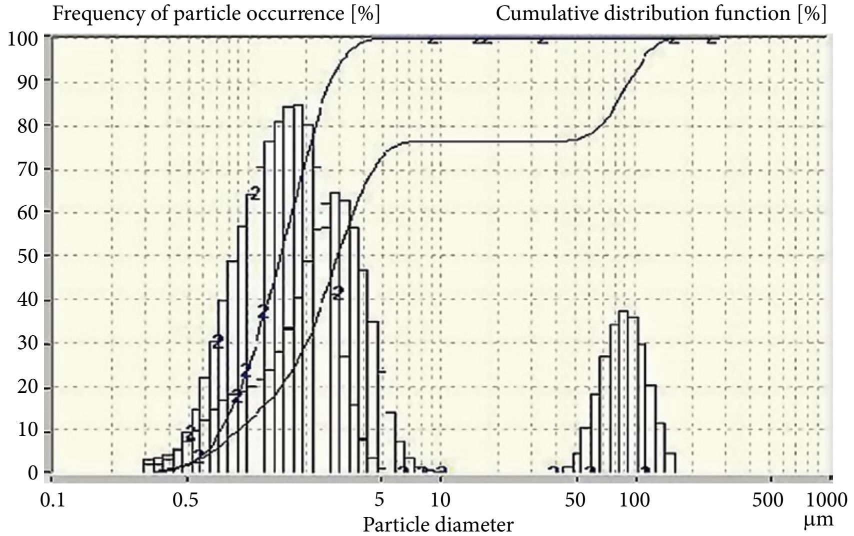

Fig. 1. Comparison of the frequency histograms and cumulative distribution functions of type E 160V55 epoxy dispersion particles, the first higher histogram and the first distribution function corresponding to the new dispersion

(Tyson *et al.* 2011) and a change in mechanical and chemical properties of hardened dispersion paint films.

The second test of the change in stability of both new and old dispersion E 160V55 was the cylinder sedimentation test. After the evaluation of data from Table 1 it is apparent that the older (two-year old) dispersion settles faster in both the 5% and 10% water solution. It is undoubtedly caused by a certain amount of the many times larger and therefore heavier agglomerated particles originated due to the two years of storage. The term 'separated', used in the evaluation of the extent of sedimentation of the old dispersion after 30 days, describes the end of sedimentation on the grounds of creating a solid dispersion cake in the lower part of the graduated cylinder.

Table 1. Sedimentation of dispersions after dilution to 5% and 10%

Time	E 160V55 old		E 160V55 new	
[days]	5% [ml]	10% [ml]	5% [ml]	10% [ml]
1	97	98	99	99
3	96	96	98	98
7	95	96	97	98
14	87	92	92	96
30	Separated		75	86

3.2. Change in the physical-mechanical properties of a dried dispersion film

Similarly as in the case of a liquid dispersion, the ageing of the original dispersion also manifests itself in the properties of hardened films. The following part of this paper focuses on the description and evaluation of the properties of hardened paint films created on standard surfaces using both new and old dispersion E 160V55 hardened by Telalit 1040 hardener. The first physical-mechanical test focuses on the evaluation of the appearance of paints (see Figs. 2 and 3) and the drying speed of paint films – see Table 2.

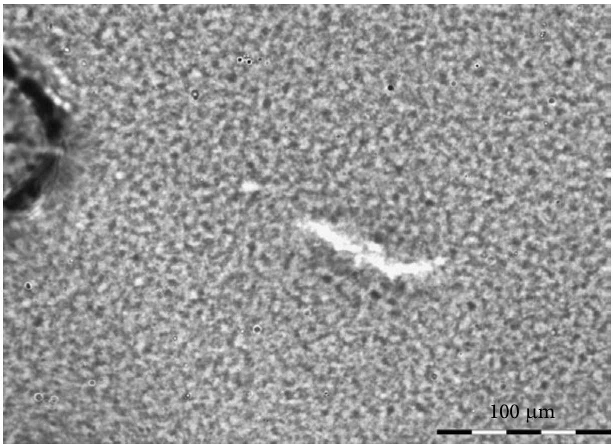

Fig. 2. Photographic recording of the E 160V55 film – rough surface of the hardened old dispersion film (50-fold magnification)

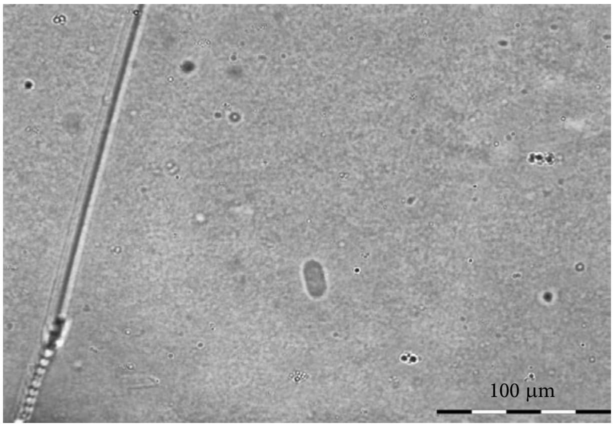

Fig. 3. Photographic recording of the E 160V55 film – smooth surface of the hardened new dispersion film (50-fold magnification)

From the results in Table 3 it is apparent that the dried film of the new dispersion E 160 is harder (pendulum damping test 62.2% compared to 50% in the old dispersion) but more elastic (bend test result, impact resistance, cupping test) and better adhering (grade 4 – approximately half of the squares are separated from the substrate), compared to the older dispersion.

Table 2. The appearance of paints and the drying speeds of paint films

Sample	Appearance	Drying out to degree [hrs]					Thickness of the film [µm]
		1	2	3	4	5	
E 160V55 old	rough surface	1	3	5	7	9	60
E 160V55 new	smooth surface	3	5	12	19	25	60

Table 3. Properties of paint films

Dispersion	Time [days]	Pendulum damping test [%]	Bend test [mm]	Impact resistance [cm]	Cupping test [mm]	Cross-cut test [grade]	Thickness of the film [µm]
E 160V55 old	3	42.6	<3	100/100	10	2	65
	10	49.0	5	100/100	7	5	
	20	52.0	4	100/90	7	5	
	30	50.0	5	90/100	8	5	
E 160V55 new	3	47.2	<3	100/100	8	1	65
	10	62.8	<3	100/100	8	2	
	20	66.8	<3	100/100	8	3	
	30	62.2	<3	100/100	10	4	

3.3. Change in the chemical properties of a dried dispersion film

Samples with two coats were first tested using the coating testing method. The thickness of two-coat paint films ranged from 110 to 120 µm.

The results of the chemical resistance of films of the old (see Table 4) and new dispersions (see Table 5) determined using the ČSN EN ISO 2812-2 coating testing method can be summarized as follows: the new dispersion better resists acids and alkalis, and partly also organic volatile matters. The results of the second test of chemical resistance of hardened new and old dispersion films to water and chemical deicing agents are illustrated by Fig. 4.

It is apparent that in both cases, i.e. in both the old and the new dispersion, the dispersion film itself resisted 60 freezing cycles with a 5 mm column of the 3% NaCl deicing solution. In neither case any macroscopic damage to the paint film, e.g. blisters or cracks, was apparent.

There was, however, a change in the colour especially of the old dispersion (a change in the colour of dispersion film is more noticeable in reality than in the photograph in Fig. 4). The milky colour of the upper three samples was a consequence of penetration of the saline solution (Šauman 1965; Hill *et al.* 2001) into the structure of the film and subsequently into the base concrete, which manifested itself by a soaking and optical darkening of the base. The relatively massive penetration of the saline solution into the old dispersion film was probably caused by the lowered diffusion resistance of the film (Henning, Lach 1983; Matoušek, Drochytka 1998) due to greater dimensions of the agglomerated dispersion particles (Tyson *et al.* 2011; Pedraza, Soucek 2007). A certain penetration of the deicing solution under the layer of the dispersion film

Table 4. Chemical resistance of the old dispersion film, two coats

Dispersion E 160V55 old	Chemical resistance after 2 hrs	24 hrs	7 days
H_2O	0	0	0
10% H_2SO_4	0	0	B2
10% HCl	0	0	B3
10% NaOH	0	0	0
"Natural" petrol	0	0	0
Ethanol	0	B3	---
Xylene	0	B3	---
Motor oil	0	0	0
10% NaCl	0	0	0

Table 5. Chemical resistance of the new dispersion film, two coats

Dispersion E 160V55 new	Chemical resistance after 2 hrs	24 hrs	7 days
H_2O	0	0	0
10% H_2SO_4	0	0	0
10% HCl	0	0	0
10% NaOH	0	0	0
"Natural" petrol	0	0	0
Ethanol	0	0	B2
Xylene	0	0	B2
Motor oil	0	0	0
10% NaCl	0	0	0

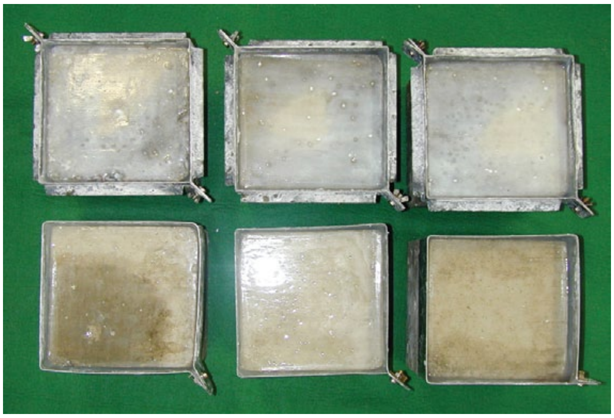

Fig. 4. Change in appearance of a film (areal dimensions 150×150 mm) of the old (upper three samples) and new (lower three samples) dispersions after 60 freezing cycles in an environment of the 3% NaCl solution. The substrate of the dispersion film is ordinary cement concrete with a 28-day compressive strength of 30 MPa

is also apparent in the new dispersion, specifically on the first testing cube (see Fig. 4, bottom left sample). Here, however, was a local failure of the silicone sealing of the sheet metal frame holding the 5 mm column of the solution above the tested film.

On the basis of the results of all tests on both liquid and hardened dispersions of two ages (old and new), the following conclusions can be drawn: as regards the change in the microstructure of dispersions due to ageing, it can be said that fine particles gradually agglomerate into formations that are approximately 45 times as large as the original particles. This fact results, in the case of coating films, in the changes to the film structure, changes to the dimensions of capillaries and probably in the insufficient penetration of the hardener into great micelles. That leads subsequently to decreasing the hardness of the film and to increasing the absorption capacity and permeability of the film for water solutions - it is possible to speak about the transition of the dispersion - due to ageing - into the composite of type III, i.e. into an open system of capillaries (Novák 2006).

4. Conclusion

The degradation of macrostructural properties of dispersions due to ageing evaluated on the changes in mechanical and chemical properties of the films formed by them was apparent in almost all the standard tests carried out including the test for chemical resistance and for chemical defrosting agents resistance. In spite of that it is possible to say that the protective film made of an old epoxy dispersion, often expired, can meet certain requirements on the effective protection of concrete and steel structures against the effects of weak alkalis, motor oils or chemical defrosting agents, naturally with an impaired appearance (matting and milky colouring) of the epoxy dispersion film surface.

Considering the increasing requirements on the service life and durability of both the existing and new building structures in which single- and multi-component polymer dispersions have increasingly been used, the future research should aim at a detailed assessment of the changes in the diffusion properties of the dried films of dispersions of various ages. It is only a high-quality barrier created on the surface of the structure that can stop the penetration of aggressive acid gases from the atmosphere and simultaneously allow for a regressive release of the water vapour overpressure. Another direction of research could lead to an alternative assessment of the influence of the dispersion age on the physical-mechanical properties of the dispersion-modified PCC and concretes made of recycled concrete (Cui, Xiang 2011), or possibly to an assessment of the influence of the dispersion age on the effectiveness of the protection of fresh concretes, the so-called green concretes (Stehlík 2011).

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