Friction Reduction Degradation in Dilute Poly(ethylene Oxide) Solutions

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Synopsis

Degradation of friction-reducing effectiveness of dilute poly(ethylene oxide) solutions subjected to continuous agitation is examined in disk flow. A residual or steady-state effectiveness is observed which disappears only at a sufficiently high Reynolds number. The onset of friction reduction appears to occur within the transition region and seems to be independent of solution concentration. The onset of degradation also appears to be independent of concentration. Prior shear working is shown to have a significant effect on residual friction-reducing effectiveness only when the shear working is performed in a more intense shear field than that in which the friction-reducing effectiveness is evaluated. Degradation of friction-reducing effectiveness is compared to corresponding changes in viscosity-average molecular weight. The experimental results are interpreted in terms of a model of the interaction between polymer molecules and the turbulent flow field.

INTRODUCTION

Certain long-chain polymer molecules in very dilute solutions on the order of parts per million are known to cause drastic reductions in the frictional drag characteristics of the solvent in turbulent flow. This behavior was first noted well over 20 years ago. Since that time, the rheological behavior of such systems has come under the increasingly intense scrutiny of a number of fundamental and applied research efforts.

One polymer/solvent pair of particular practical interest is that of poly-(ethylene oxide) molecular-weight homologs (MW > 10⁵) in water. Systems composed of PEO with an average molecular weight of approximately 4×10^6 and water have been shown to exhibit a reduction in frictional drag of upwards of 75% in turbulent pipe flow over that of pure water; and this at concentrations of less than 100 weight parts per million! Moreover, significant reductions are observed at concentrations of less than 1 weight parts per million.

Turbulent flow fields subject the polymer chains to intense shearing forces which tend to rupture the longer chains. This mechanical degradation, leading to a reduced average molecular weight, is accompanied by a diminuation of the capability of the solution to reduce friction. Studies

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of the turbulent pipe flow characteristics of various polymer solutions have indicated a noticeable decrease in friction-reducing effectiveness with increased exposure to turbulent flow. Ripkin and Pilch¹ subjected 100 and 1000 weight parts per million (wppm) solutions of PEO (WSR-301) to repeated passes through a 0.054×54.0 in. capillary apparatus. Thev observed a decrease in the friction-reducing effectiveness with successive passes at 100 wppm. An initial improvement in the frictional performance at 1000 wppm was attributed to the concentration "effectiveness plateau." Similar results were obtained when fluid samples were sheared in a rotary mechanical mixer prior to measuring the friction-reducing characteristics. McNally² reported reduced frictional performance resulting from multiple passes of 20 wppm PEO (WSR-301) solutions through $^{3}/_{4} \times 100$ in. smooth and rough pipes. Kenis,³ in a much more extensive study of degradation effects in a 0.117×41.0 cm capillary apparatus, noted similar results for successive passes of PEO (WSR-301) at concentrations of 5, 10, 20, and 40 wppm in water.

Patterson⁴ measured pipe friction in a 1/4-in.-diameter pipe using differential transducers located 1327 diameters apart, with the pressure taps spread 10 diameters apart at each station. He recorded higher pressure drops at the downstream station. These measurements were accompanied by the simultaneous determination of the intrinsic viscosity of samples of solution withdrawn from the apparatus at both stations. For those Reynolds numbers for which a significant degradation in friction-reducing effectiveness was observed, Patterson consistently found a lower intrinsic viscosity at the downstream station. Fabula⁵ also noted increased friction along the direction of flow for some PEO solutions. In these experiments, friction increased over the first third of the pipe, reaching a steady level which exhibited no further change.

Gilbert and Ripkin⁶ measured the drag reduction on a rotating $19^{5}/_{8}$ -in. enclosed disk using the natural plant derivative guar gum. They recorded the torque required to rotate the disk at a constant speed in 100 and 500 wppm solutions as a function of time. They observed a gradual increase in the required torque which appeared to be approaching a steady value after some hours of continuous operation. Degradation of the frictionreducing characteristics of PEO solutions in disk flow was also noted by Hoyt and Fabula.⁷ Degradation of PEO solutions in turbulent flow in a Couette viscometer was reported by Shin.⁸ Frictional drag was observed to increase with continuous agitation for dilute solutions. For concentrations beyond the "effectiveness plateau," however, the drag initially decreased, reached some minimum value, and then increased. Shin's experiments were terminated shortly after the increase in drag was noted. Gadd⁹ studied the degradation of 15 wppm PEO solutions in a concentric cylinder apparatus with the inner cylinder rotating at 1000 rpm by recording the drag on the rotating cylinder as a function of time. He observed a rapid initial increase in the drag, which after about 1/2 hr became close to the drag exerted by pure water.

The observed decrease in friction reduction capability is usually attributable to actual molecular scission induced by the intense shear environments encountered in the turbulent flow fields. As the relative concentration of higher molecular weight components is reduced by the scission process, the friction-reducing effectiveness of the solution is correspondingly reduced. Simultaneously, however, the solution viscosity also decreases as a result of molecular scission. It is this factor which leads to the observed initial increase in friction-reducing effectiveness for concentrations beyond the "effectiveness plateau."

The studies just described were not directed primarily at an investigation of mechanical degradation processes. Rather, they were concerned with various aspects of friction reduction phenomena in which the observations of degradation form an incidental part or were used to compare the performance of various polymer/solvent pairs. Hence, understanding of the nature of the degradation process, the rate at which it occurs, and the extent to which it proceeds in a given hydrodynamic environment is far from adequate.

The objective of the experiments reported here was to focus attention on the degradation in friction-reducing effectiveness of PEO solutions in turbulent flow fields. The procedures utilized and the results of these experiments are described in the following paragraphs.

EXPERIMENTAL

Materials

Poly(ethylene oxide) was obtained from Union Carbide Chemicals Co., New York (Polyox WSR 205, WSR 301, and Coagulant Grade) as unfractionated samples and used as received. Intrinsic viscosity and viscosity-average molecular weight of the three grades was found to be, for WSR 205: $[\eta] = 4.5$, MW = 8.9×10^5 ; for WSR-301: $[\eta] = 12.4$, MW = 3.2×10^6 ; for Coagulant Grade: $[\eta] = 15.5$, MW = 4.2×10^6 .

Solutions were initially prepared in 1% (by weight) concentrations using distilled water. Isopropyl alcohol (0.5%) was added to inhibit chemical degradation.¹⁰ The "master" solutions were then diluted to the appropriate concentration as required. Friction reduction effectiveness of dilute PEO solutions is subject to mechanical degradation during the solution preparation process, the extent of which depends upon the intensity of agitation used.¹¹ In addition, this degradation is influenced by the concentration at which the solution is initially mixed as well as the concentration at which the corresponding friction-reducing effectiveness is determined. It was found that a moderate amount of mechanical agitation during the preparation of the 1% solutions had no deleterious effects on the friction-reducing effectiveness of subsequently diluted forms, at least over the ranges of concentration and flow field intensity encountered. Consequently, the "boiling water" technique described by the manufacturer was adopted for these studies.¹² This method is particularly simple, requiring only 1 or 2 hr to produce a homogeneous 1% solution.

Each "master" solution was used in the preparation of dilute test solutions for a period of about one week, during which time no effects on friction reduction were evidenced. (One concentrated solution stored for over 9 months exhibited no loss in friction reduction effectiveness at 50 wppm.) Dilute solutions were prepared by injecting carefully measured quantities of "master" solution directly into the turbulent flow field.

Measurements

Friction reduction measurements were performed by injecting carefully measured quantities of "master" solution directly into the turbulent flow field of a rotating disk apparatus. Preliminary flow visualization using dyed "master" solutions indicated that complete mixing was achieved within several seconds. Confidence in the accuracy with which homogeneous dilute solutions could be so produced was further bolstered by the reproducibility of the results.

Two turbulent flow rheometers based on rotating disk flow were used to measure the friction reduction effectiveness and to mechanically degrade the polymer solutions examined. The smaller scale of the two consists of a 12.7-cm-diameter polished brass disk enclosed in a thermostated housing. The principal element of this system is a Master Servodyne laboratory mixer (Cole-Parmer Instrument Company, Chicago). The mixer consists of a d.c.-motor generator and a control unit with the built-in capacity to deliver a variable torque equal to the demand rate of the load while maintaining a preset speed. The control unit includes a calibrated torque sensor.

The second rheometer consists of a 25.4-cm-diameter polished brass disk, also enclosed in a thermostated housing. Power is supplied by a Reeves variable speed motor with a maximum deliverable output of 30 hp at the top operating speed. The torque required to turn the disk at a fixed speed is measured with a Lebow type A shaft torque and rpm pickup in conjunction with a Daytronic strain gauge-type torque indicator and a Hewlett-Packard electronic rpm counter.

Friction reduction characteristics were obtained in all cases by first measuring the torque required to rotate the disk at a given speed in distilled water. The corresponding torque required at the same speed with a dilute PEO solution then yielded the per cent friction reduction defined as

$$f_R = 100 \left[\frac{T_0 - T}{T_0} \right] \tag{1}$$

where T_0 is the torque measured with distilled water, and T is the torque measured with PEO solution.

Because of the length of some of the friction reduction decay experiments (up to several hours), temperatures were controlled to $30^{\circ} \pm 0.5^{\circ}$ C in the small disk apparatus and to $40^{\circ} \pm 1^{\circ}$ C in the large disk apparatus.

The principal series of friction reduction experiments was conducted in the large disk rheometer. This series consisted of the determination of the per cent friction reduction as a function of time for PEO (WSR-301) concentrations of 10, 25, 50, 100, and 150 wppm at disk speeds of 500, 1000, 1500, and 2000 rpm. Each test was allowed to continue until an apparent steady state had been obtained, that is, until no further change in friction reduction was observed for at least 10 min. All of the measurements were conducted in the turbulent flow regime.

A second, "step sequence," series of experiments was also conducted using the large disk rheometer. In this series, a dilute PEO (WSR-301) solution was allowed to come to a steady state in the turbulent flow field at 500 rpm. The disk speed was increased to 1000 rpm and the solution allowed to come to another steady state at this speed. This procedure was then repeated by increasing the speed to 1500 rpm and, subsequently, to 2000 rpm. After a steady friction reduction was observed at 2000 rpm, the disk speed was reduced in rapid steps to 500 rpm. A measure of the friction reduction was obtained at each of the previous intermediate disk speeds. The entire procedure was repeated for polymer concentrations of 10, 25, 50, 100, and 150 wppm.

The small disk rheometer was utilized to compare the friction reduction degradation characteristics of Polyox grades WSR-205, WSR-301, and Coagulant Grade at 2, 50, 100, and 200 wppm. Concurrently, a Cannon-Fenske size 50 routine-type capillary viscometer was used to obtain an approximate measure of the intrinsic viscosity of some of the solutions studied. Samples of polymer solution were withdrawn from the small disk rheometer periodically during the degradation test. Measurements of solution viscosities were obtained at 25° C and the reduced viscosity calculated according to the relation

$$\eta_r = (\eta - \eta_0)/c\eta_0 \tag{2}$$

where η_{τ} is the reduced viscosity, η is the solution viscosity, η_0 is the solvent viscosity, and *c* is the polymer concentration in g/dl.

Intrinsic viscosity is defined as

$$[\eta] = \lim_{\substack{D \to 0 \\ c \to 0}} \eta_{\tau} \tag{3}$$

where D is the shear rate at which the viscosity measurement is obtained. The extrapolation to zero concentration was made, using Huggins' relation⁴:

$$(\eta_r / [\eta]) - 1 = 0.4c \ [\eta]. \tag{4}$$

The shear rate dependence of the reduced viscosity of PEO solutions is still subject to a great deal of controversy.⁴ The approximate shear rate in the capillary instrument used here is 1200 sec^{-1} , and no effort to extrapolate to zero shear rate was made. Rather, reduced viscosities extrapolated to zero concentration as above were taken as roughly approximate to the intrinsic viscosity, and polymer molecular weights were estimated using Shin's relation⁸:

$$\eta = 1.03 \times 10^{-4} \, \bar{M}_{p}^{0.78} \, (\text{at } 25^{\circ}\text{C})$$
 (5)

where \overline{M}_{v} is the viscosity-average molecular weight.

RESULTS AND DISCUSSION

The results of the turbulent shear degradation tests conducted with the 25.4-cm-diameter disk are summarized in Figures 1 through 5. Figures 1 through 4 depict the per cent friction reduction obtained with various concentrations as a function of time for disk speeds of 500, 1000, 1500, and 2000 rpm, respectively. The friction reduction characteristics of the 10-wppm solution are shown in Figure 5 for comparision crossplotted as a function of time at various disk speeds.

With several exceptions, the friction reduction characteristics display a more-or-less exponential decay. It is noteworthy to observe that the decay does not proceed to the extent that the friction reduction effect disappears entirely. Rather, a steady state which exhibits a residual friction reduction effectiveness is achieved after a sufficiently long time. Presumably, the solution has reached a state of dynamic equilibrium with the applied shear field in which it can exist without additional degradation. The exceptions to this behavior occur only for the higher concentrations at the lower disk speeds. In these few cases, friction reduction initially



Fig. 1. Initial friction reduction and mechanical degradation of WSR-301 at 500 rpm.



Fig. 2. Initial friction reduction and mechanical degradation of WSR-301 at 1000 rpm.

increases, reaches a maximum value, and then follows the usual pattern of decay to a residual value.

This apparently anomalous characteristic is easily rationalized by reference to Figure 6, which typifies the effect of concentration on the initial and residual friction reduction effectiveness. The behavior of these curves is typical of the concentration effect.⁷ That is, friction reduction initially increases with concentration because of the availability of additional friction-reducing molecules. As concentration is further increased, however,



Fig. 3. Initial friction reduction and mechanical degradation of WSR-301 at 1500 rpm.



Fig. 4. Initial friction reduction and mechanical degradation of WSR-301 at 2000 rpm.

solution viscosity also is increased. This leads to an increase in the frictional drag. Eventually, the increased viscosity becomes the dominant factor, and friction reducing effectiveness reaches a maximum called the "effectiveness plateau" and decreases. The maximum in disk flow is typically fairly broad,⁷ and the friction reduction effectiveness remains more or less independent of concentration over a wide range of concentrations.

Mechanical degradation leads to a reduction in the solution viscosity as well as to a reduction in the concentration of the most effective friction-



Fig. 5. Initial friction reduction and mechanical degradation of WSR-301 at 10 wppm.



Fig. 6. Initial and residual friction reduction of WSR-301 at 1500 rpm.

reducing molecules. Consequently, solutions whose initial concentrations place them past the "effectiveness plateau" display an initially increasing friction-reducing effectiveness as a result of mechanical degradation. The predominance of this effect at lower disk speeds can be partially explained on the basis of the normally pseudoplastic viscous characteristics of PEO solutions. That is, a given solution exhibits a higher effective viscosity at lower shear rates. Similar behavior at higher disk speeds would be expected at higher polymer concentrations.

It has been suggested^{2,5,7} that friction reduction could be a direct consequence of energy-absorbing molecular scission processes induced by intense turbulent shear fields. Although it is plausible that molecular scission could be a contributing factor, the existence of a significant residual friction-reducing effectiveness after long periods of exposure to a steady-state shear environment seems to rule out such an explanation¹³

Whatever the exact mechanism, it is clear that the friction-reducing effect involves some mode of interaction between the energy-dissipating vortices encountered in the turbulent flow field and polymer molecules or clusters of molecules. (It is probable that a significant clustering of polymer chains can persist even at high dilutions.¹⁴) If the decay of friction-reducing effectiveness in a turbulent shear field may be associated with the breakup of these microscopic units, then the residual friction-reducing effect corresponds to a state in which the units are small enough to be stable. This steady state is still characterized by a condition in which molecular dimensions are large enough to permit interaction with the turbulent vortices and, therefore, displays a friction reduction effect.

These observations are reinforced by the results of the "step sequence" experiments which were described above. These results are depicted in



Fig. 7. Influence of shear working on friction reduction effectiveness of WSR-301 at 10 wppm.

Figures 7 and 8 for the 10- and 50-wppm solutions, respectively, and summarized in detail for all concentrations in Table I. It is interesting to note that the initial friction reduction at any disk speed is affected by the solution having been agitated at lower speeds, while the residual friction reduction at any speed is independent of any previous agitation at lower



Fig. 8. Influence of shear working on friction reduction effectiveness of WSR-301 at 50 wppm.

Concen- tration, wppm	Shear Worked at			
	500 rpm	1000 rpm	1500 rpm	2000 rpm
		Measured at 500	rpm	
10	25.0	6.3	0	0
25	29.4	12.5	0	0
50	31.3	12.5	2.1	0
100	29.2	16.5	4.1	0
150	29.2	16.3	4.2	0
		Measured at 1000	rpm	
10	10.6	10.0	3.7	1.3
25	15.6	14.4	5.6	4.4
50	19.4	17.5	6.3	5.0
100	23.1	20.0	6.8	5.6
150	23.8	23.8	6.3	6.4
		Measured at 1500	rpm	
10		7.0	6.7	1.5
25	_	12.1	9.7	4.6
50		13.0	11.8	7.7
100		14.2	13.7	7.9
150		14.9	14.6	6.4
		Measured at 2000	rpm	
10			4.8	4.8
25	<u></u>	—	9.0	8.9
50			13.0	13.2
100		—	13.2	13.6
150		_	13.4	12.9

 TABLE I

 Effect of Shear Working on Per Cent Residual Friction Reduction

speeds (compare Figs. 1 through 4, for example). On the other hand, the residual friction reduction effectiveness at lower disk speeds is drastically diminished by agitation at higher speeds. Indeed, virtually no friction reduction is observed at 500 rpm after shearing to steady state at 2000 rpm for all concentrations. This corresponds to a shift of the friction reduction onset point to higher wall shear stresses. The residual effectiveness is also shown in Figure 7 and 8 (dashed line). The time scale does not apply to these points.

These characteristics combine to suggest that the residual friction reduction effect can be associated with the existence of stable polymer molecules or clusters whose relative size decreases with increasing shear stress.

A direct comparison of initial and residual friction reduction effectiveness is shown in Figures 9 and 10, where per cent friction reduction is plotted as a function of disk speed for the 10- and 50-wppm solutions, respectively. Each initial value corresponds to the behavior of undegraded polymer, hence initial friction reduction increases with disk speed as expected. On



Fig. 9. Initial and residual friction reduction of WSR-301 at 10 wppm.

the other hand, each residual value corresponds to degraded polymer. Since the extent of degradation is greater at higher disk speeds, residual friction reduction decreases as disk speed is increased.

Figures 11 and 12 illustrate the effect of mechanical degradation on the disk flow friction coefficient itself. The factor T/N^2 , which is proportional to the friction coefficient, is plotted logarithmically as a function of disk speed, which is proportional to the water-based Reynolds number. These figures depict the initial and residual frictional characteristics (corresponding to Figs. 9 and 10) for the 10- and 50-wppm solutions, respectively. The corresponding frictional characteristics of pure distilled water are included for comparison. The onset of friction reduction appears to lie within the transition region and seems to be essentially independent of concentration. Since a logarithmic extrapolation of the initial T/N^2 curve is somewhat speculative, these observations remain open to some question. The concentration independence of onset has been exhibited in pipe flow by solutions of random-coiling polymers of the type studied here.¹⁶ Another feature is the existence of an onset Reynolds number below which no degrada-This degradation onset point also appears to be indetion is observed. pendent of concentration.



Fig. 10. Initial and residual friction reduction of WSR-301 at 50 wppm.

It has already been observed that the residual friction reduction decreases with increasing disk speed. Figures 11 and 12 show that in a sufficiently intense shear field, the friction reduction vanishes completely. As might be expected, higher concentrations require higher disk speeds to achieve this effect.

The effects of prior shear working on residual friction reduction are omitted from Figures 11 and 12 to preserve clarity. However, solutions which have been degraded by prior shear working display a shift of friction reduction onset to higher Reynolds numbers and a general decrease in friction reduction effectiveness.

The final series of degradation studies was conducted with the small disk apparatus. The per cent friction reduction was observed as a function of time for 2, 50, 100, and 200 wppm of PEO (WSR-205, WSR-301, and Coagulant Grade) at a disk speed of 1800 rpm. The general features of these curves are similar to Figures 1 through 7. The differences between polymer grades are illustrated in Figure 13.

Simultaneous determinations of the degradation of per cent friction reduction and intrinsic viscosity (viscosity-average molecular weight) were made for PEO (WSR-205 and Coagulant Grade). Measurements were made at



Fig. 11. Onset and extent of friction reduction of WSR-301 to its vanishing residual effectiveness at 10 wppm.

200 wppm, and intrinsic viscosity was approximated as described above. These results are summarized in Figure 14.

It is clear from these data that, in terms of friction-reducing effectiveness, WSR-205 degrades more rapidly than the higher molecular weight homologs. On the other hand, WSR-205 exhibits no corresponding change in average molecular weight, while that of the sample of Coagulant Grade polymer tested drops significantly.

Poly(ethylene oxide) resin, like most synthetic polymers, consists of molecules of various sizes. This effect arises out of the statistical nature of the polymerization process. The viscosity-average molecular weight in itself does not represent an adequate characterization of the actual distribution of molecular weights in a given sample. The molecular weight



Fig. 12. Onset and extent of friction reduction of WSR-301 to its vanishing residual effectiveness at 50 wppm.

distributions of the series are still somewhat uncertain, although recent investigations indicate that they are relatively broad.^{13,17} Since it is reasonable to expect the preferential degradation of the high molecular weight portion of the distribution,¹⁸ the overall effect of mechanical degradation is nonuniformly felt throughout the distribution.

The results depicted in Figure 14 can be interpreted in the light of these considerations. The significant degradation of friction-reducing effectiveness while the viscosity-average molecular weight remains relatively constant implies that friction reduction is largely dependent upon the contributions of the higher molecular weight species in the distribution.^{4,18} Coagulant Grade Polyox has a greater proportion of high molecular weight species in its distribution. Consequently, the decay of friction reduction



Fig. 13. Initial friction reduction and mechanical degradation of WSR-205, WRS-301, and Coagulant Grade at 50 wppm.

effectiveness is slower than observed in WSR-205. The same factor accounts for the behavior of the viscosity-average molecular weight. Presumably, WSR-205 does not contain a sufficient proportion of high molecular weight species to affect the average molecular weight when they are degraded. On the other hand, Coagulant Grade has a significant number of high molecular weight species, and their degradation materially influences the average molecular weight.

CONCLUSIONS

The decrease in friction-reducing effectiveness observed in a turbulent flow environment is primarily due to molecular scission of the larger-chain components of the molecular weight distribution. This degradation does not always proceed to the complete destruction of friction-reducing effectiveness. Rather, a steady state or residual friction reduction effectiveness is observed if the shear environment is not too intense. It is clear that this state is characterized by the attainment of a stable molecular structure.

The residual friction-reducing effectiveness and, therefore, the significant steady-state molecular dimensions in a given shear environment are not affected by previous agitation of the solution in a less intense shear field. On the other hand, the friction-reducing effectiveness can be diminished, or even completely destroyed, if it is agitated for a sufficiently long time in a more intense field.

From a practical point of view, it is important to provide a more quantitative interpretation of the influence of the significant hydrodynamic, molecular, and solution parameters upon friction reduction effectiveness and degradation. Unfortunately, the rotating disk shear field is nonuniform. The use of an inhomogeneous flow field makes it difficult to develop a quan-



Fig. 14. Friction reduction and viscosity decay of WSR-205 and Coagulant Grade.

titative specification of shear field intensity which can be used to correlate degradation. Ideally, friction reduction degradation studies should be conducted in a uniform flow field wherein each fluid element is subjected to the identical shear history. Efforts are currently under way in this laboratory to develop a turbulent flow rheometer capable of subjecting a polymer fluid to a continuous, uniform shear field of various intensities.

It is clear that viscosity-average molecular weight is not an adequate molecular parameter for the correlation of mechanical degradation effects. Evidence points to the conclusion that friction reduction is largely dependent upon the contributions of the higher molecular weight species in the PEO distribution. In order to develop an understanding of the influence of shear working on friction-reducing effectiveness, it is necessary to accompany these investigations with a thorough analysis of the PEO molecular weight distribution, including any such changes as may be caused by the shear working.

Nomenclature

- c polymer concentration, grams/deciliter (g/dl)
- D shear rate
- f_{τ} per cent friction reduction defined by eq. (1)
- \overline{M}_{v} viscosity-average molecular weight
- N disk speed
- T disk torque
- T_0 disk torque measured using water
- η solution viscosity
- $[\eta]$ intrinsic viscosity defined by eq. (3)
- η_0 water viscosity
- η_r reduced viscosity defined by eq. (2)

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