Rheology Modification of Hydrogen Peroxide-Based Applications Using Cross-Linked Polyacrylic Acid Polymers*

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1. Key Words:


2. Summary:

A fundamental study of the stability of hydrogen peroxide-based gels, thickened using cross-linked polyacrylic acid polymers, will be presented. Recent systematic experiments will demonstrate that excellent long-term stability of such gels can be achieved. The study evaluates different types of cross-linked polyacrylic acid polymers (as well as an associative acrylic polymer), several commercial sources of hydrogen peroxide, hydrogen peroxide concentrations and the effect of pH. Resulting viscosities from combinations of these variables will be presented, as well as the effects of accelerated aging on percent active peroxide and viscosity of the thickened peroxide gels. The results of this study will enable the formulator to determine the appropriate polymer and conditions necessary for optimal stability and rheological properties for hydrogen peroxide applications. This study will demonstrate that some cross-linked polyacrylic acid polymers are very compatible and stable with H₂O₂ and that these polymers do not affect the H₂O₂ stability under certain conditions. In addition, this study will show that certain cross-linked polyacrylic acid polymers can produce very viscous gels and yield higher viscosities in typical permanent hair color formulas compared to many commercially available products. Because of the excellent compatibility and stability demonstrated, cross-linked acrylic acid polymers are ideal rheological modifiers for hydrogen peroxide-based applications. The performance benefits of using a cross-linked acrylic acid polymer in two-part hair color formulations will be described in detail.

3. Introduction:

Hydrogen peroxide has been widely used as an oxidizer and bleach in hair care applications since the 1940s. Hydrogen peroxide is one of the most important ingredients in hair color and permanent wave products on the market today. The performance characteristics of numerous peroxide applications, including household, personal care, and pharmaceutical products could be potentially improved by the use of an appropriate rheological additive. Possible applications for gelled peroxide include hair bleach, topical gels, acne treatments, dental tooth whiteners, permanent waves and permanent hair color. Although the focus of this study will be on personal care applications, particularly hair bleach and permanent hair color, the results can be leveraged broadly for other applications.

Hydrogen peroxide, because of its reactive chemistry, is difficult to stabilize in many applications. There are many commonly-used formulation ingredients that are incompatible with hydrogen peroxide. Careful selection of formula ingredients, thickeners, and stabilizers, as well as the proper order of addition, pH and processing equipment are all essential to ensure acceptable long-term physical and chemical stability of a finished product.

Achieving both the desired viscosity and appropriate rheological properties can be a challenging task in peroxide-based formulations. Although cross-linked polyacrylic acid polymers are currently being used in some hydrogen peroxide-containing personal care and topical pharmaceutical applications, few systematic studies have been performed to evaluate different cross-linked acrylic acid polymers for specific hydrogen peroxide applications. This fundamental study demonstrates that excellent long-term stability of hydrogen peroxide-based gels thickened with particular cross-linked polyacrylic acid polymers can be achieved.

4. Cross-Linked Polyacrylic Acid Polymers

Cross-linked acrylic acid polymers are already well known for their unique rheological properties, such as the ability to impart vertical cling or a “no-drip” rheology. These unique rheological properties can enhance the performance characteristics of applications such as hair bleach, permanent waves and hair color, to name a few. One of the problems that can occur during the application of a permanent wave or hair color is the unpleasant experience of having the solution drip on the neck or face. The unique rheological behavior of cross-linked acrylic acid polymers enables the active material to adhere to the substrate for consistent coverage, minimizing the amount which may be lost due to runoff. Cross-linked acrylic acid polymers also provide a shear thinning rheology with the ability to recover viscosity immediately after the shearing has ceased, allowing hair color to be easily applied or squeezed out of a bottle. In addition, cross-linked acrylic acid polymers are odor free and have a high buffering capability due to the high number of carboxylic acids groups on the polymer, helping to maintain a stable pH in the finished product and during its use.

5. Fundamental Study:

5.1 Experimental Approach:

Hydrogen peroxide gel samples were made to determine the effects of different acrylic acid polymers and hydrogen peroxide grades. Physical properties and the stability of the gels were measured and observed. The material variables in the study include various acrylic acid polymers and hydrogen peroxide grades. Ammonium hydroxide was used to neutralize the gels where necessary. In order to minimize the introduction of any contaminants, particularly transition metals, special care was taken in this study. Samples were made using plastic beakers, agitators and spatulas, and stored in opaque polyethylene containers.

The test variables in the study include $H_2O_2$ concentration, which varied from 3 - 12 % active, and pH, from 2.7 - 4.5. This pH range was selected because of the inherent instability of hydrogen peroxide at neutral or high pH. All samples were neutralized using ammonium hydroxide to adjust the pH, except for the samples made at a pH of 2.7, which remained unneutralized. Samples were measured for percent hydrogen peroxide using a USP monograph assay for hydrogen peroxide. This method is a potassium permanganate titration, which utilizes the reduction of potassium...
permanganate by hydrogen peroxide in sulfuric acid. The samples were measured for viscosity, using a Brookfield viscometer, for pH, and were also visually observed. Teflon coated spindles were used with the Brookfield viscometer to avoid contaminating the gels. Triplicate samples of the peroxide gels were placed on stability for 12 weeks at room temperature (23 °C) and elevated temperature (45 °C). These samples were tested at intervals of 1, 2, 4, 8, and 12 weeks for the same properties as listed previously.

5.2 Acrylic Acid Polymers:

Five cross-linked acrylic acid polymers were used in the study, as well as a liquid associative polymer, and were labeled A through E (Figure 1). The results of this study will demonstrate that Polymers A and D have the most stable viscosity when used to gel hydrogen peroxide. Polymer A is a highly cross-linked homopolymer of acrylic acid, polymerized in benzene, and Polymer D is an easy dispersing cross-linked copolymer of acrylic acid, polymerized in ethyl acetate and cyclohexane.

<table>
<thead>
<tr>
<th>Polymer</th>
<th>INCI Name</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>Carbomer</td>
</tr>
<tr>
<td>B-1, B-2</td>
<td>Carbomer</td>
</tr>
<tr>
<td>C</td>
<td>Carbomer</td>
</tr>
<tr>
<td>D</td>
<td>Acrylates/ C10-30 Alkyl Acrylate Crosspolymer</td>
</tr>
<tr>
<td>E</td>
<td>Linear Acrylic Emulsion Polymer *</td>
</tr>
</tbody>
</table>

* Not INCI Name

figure 1

** The following is a legend of the polymer trade names featured in this study:

- Polymer A: Carbopol® 934
- Polymer B: Carbopol® 980
- Polymer C: Carbopol® Ultrez™ 10
- Polymer D: Carbopol® ETD™ 2020
- Polymer E: (Not supplied by BFGoodrich)
5.3 Hydrogen Peroxide:

Hydrogen peroxide is commercially available from several suppliers in a variety of grades. The grade of H$_2$O$_2$ will significantly impact the performance and physical properties of the application. Commercial grades of H$_2$O$_2$ contain stabilizers (chelants and sequestrants) to minimize decomposition during normal storage conditions. These stabilizers may improve the performance of H$_2$O$_2$ in an application. Typical stabilizers used in hydrogen peroxide may include: colloidal stannate, sodium pyrophosphate, organo-phosphonates, and colloidal silica. Four commercially available hydrogen peroxides were used in this study, and are referred to as: A, B, C, and D (figure 2). Hydrogen Peroxide A and C are both standard personal care grades, while B and D are stabilized grades. Hydrogen Peroxide B is a very highly stabilized grade.

<table>
<thead>
<tr>
<th>Commercial Hydrogen Peroxide Grades Used in Study</th>
<th>Hydrogen Peroxide Concentrations Used in Commercial Hair Care Applications</th>
</tr>
</thead>
<tbody>
<tr>
<td>H$_2$O$_2$</td>
<td>Description</td>
</tr>
<tr>
<td>A</td>
<td>Personal Care Grade (PC)</td>
</tr>
<tr>
<td>B</td>
<td>Highly Stabilized Grade (ST)</td>
</tr>
<tr>
<td>C</td>
<td>Personal Care Grade (PC)</td>
</tr>
<tr>
<td>D</td>
<td>Stabilized Grade (ST)</td>
</tr>
</tbody>
</table>

Traditionally, hydrogen peroxide solutions in the hair care industry are provided in different concentrations or “volumes” of H$_2$O$_2$. Figure 3 shows the relationship between volume and the percent by weight of H$_2$O$_2$. A concentration of 6% wt hydrogen peroxide was used in most of the study, since this is the most commonly used concentration in hair color developers.

*** The following is a legend of the hydrogen peroxide trade names featured in this study:

- Hydrogen Peroxide A: Albone® 50 CG (DuPont)
- Hydrogen Peroxide B: Ultra Cosmetic™ (Solvay Interox)
- Hydrogen Peroxide C: Cosmetic (Degussa)
- Hydrogen Peroxide D: Peralkali™ (Degussa)
5.4 Test Formulation and Procedure:

In order to evaluate the effects of several variables in this study (polymer type, pH, peroxide grade, and peroxide concentration) a standard test formula was used (figure 4). Using this test formula, the active polymer concentration was kept constant at 1.0% \text{ wt} as well as the hydrogen peroxide concentration at 6% \text{ active}. The H_2O_2 concentration was varied in a few experiments in order to determine the effect peroxide concentration has. The test formula was made using the following order of addition: deionized water, polymer, hydrogen peroxide, and ammonium hydroxide to adjust pH. It has been determined that this is the optimal order of addition to use in order to achieve the most stable peroxide gel system, based on previous internal studies. Samples were made in plastic beakers and mixed with plastic stirrers to avoid any contamination.

**Test Formulation Used in Current Study**

<table>
<thead>
<tr>
<th>Order of Addition</th>
<th>Ingredients</th>
<th>% wt</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.</td>
<td>Deionized Water</td>
<td>87.0</td>
</tr>
<tr>
<td>2.</td>
<td>Polymer (% Active)</td>
<td>1.0</td>
</tr>
<tr>
<td>3.</td>
<td>H_2O_2 (50%)</td>
<td>12.0</td>
</tr>
<tr>
<td>4.</td>
<td>NH_4OH (30%)</td>
<td>q.s. to desired pH</td>
</tr>
</tbody>
</table>

\( \text{H}_2\text{O}_2 \oplus 6\% \text{ wt active} \)

**Effect of Polymer Type and pH on Viscosity**

6. Results and Conclusions:

6.1 Hydrogen Peroxide Thickening

Before considering what affects the long-term stability of hydrogen peroxide gels thickened with acrylic acid polymers, it is necessary to first examine what affects the initial viscosity. The results of the study show how polymer type, pH, H_2O_2 grade and concentration affect the initial viscosity.

6.1.1 Effect of pH

The effect of pH on viscosity of a 6% \text{ wt} hydrogen peroxide solution is shown in figure 5. As noted earlier, all polymers are used in this study at 1% active. Unneutralized dispersions of cross-linked acrylic acid polymers, at pH 2.7, have a very low viscosity, ranging from 10 - 50 cP. Partially neutralized dispersions, at a pH of 3.6 and 4.0, produced viscosity ranges of 12,000 - 35,000 cP and 25,000 - 45,000 cP, respectively. As the pH of a dispersion increases, the resulting viscosity also increases. Low viscosity dispersions, at pH 2.7, may be useful in applications where two-part systems are desired, making it easy to mix the two parts together. It is well known in industry that cross-linked acrylic acid polymers cause an increase in viscosity as the pH is increased. The fact that these polymers can thicken at a low pH (3.6), as shown in figure 5 however, is not expected by many formulators.

6.1.2 Effect of Polymer Type

Figure 5 also shows the effect of polymer type on viscosity. Polymers A through D all follow a similar viscosity profile, with Polymer C providing the highest viscosity (45,000 cP), at a pH of 4.0. The cross-linked polymers, A through D, demonstrate very efficient thickening at pH 3.6 and 4.0, as compared to the liquid associative Polymer, E. Very little increase in viscosity occurs with Polymer E at a pH of 3.6 and 4.0. No dispersion was made with Polymer E, at pH 2.7, since the unneutralized dispersion pH for this polymer was 3.6.

6.1.3 Effect of Hydrogen Peroxide Grade
The grade of hydrogen peroxide used has a major influence on the initial viscosity. The effect of different peroxide grades, using Polymer C to thicken 6% \text{ wt} \% hydrogen peroxide solutions, is demonstrated in figure 6. This example shows that the personal care grades (A and C) of \( \text{H}_2\text{O}_2 \), result in comparable and significantly higher viscosities as compared to the stabilized grades (B and D), which differ significantly from each other. The viscosity properties in a given application are dependent on the type of \( \text{H}_2\text{O}_2 \) used.

![Effect of Peroxide Grades on Initial Viscosity](image1)

**Figure 6**

### 6.1.4 Effect of Hydrogen Peroxide Concentration

Peroxide concentration may have an effect on initial viscosity of hydrogen peroxide gels. Figure 7 shows a linear correlation between peroxide concentration and viscosity for Polymer A, but not for Polymer D. The viscosity of a peroxide gel may increase as the peroxide concentration increases, and is dependent on the type of polyacrylic acid polymer.

![Effect of H\textsubscript{2}O\textsubscript{2} Concentration on Viscosity](image2)

**Figure 7**

### 6.1.5 Conclusions

This fundamental study has determined significant effects of acrylic acid polymers on \( \text{H}_2\text{O}_2 \) gels. Some cross-linked polyacrylic acid polymers can be used to make low viscosity dispersions at pH 2.7, which may be useful in low-viscosity applications. Very high viscosity gels can be made at low pH's such as 3.6 and 4.0, where \( \text{H}_2\text{O}_2 \) is most stable. Cross-linked acrylic acid polymers yield significantly higher viscosity gels relative to the linear emulsion polymer, and as pH increases, the viscosity of the peroxide gel also increases. The grade of \( \text{H}_2\text{O}_2 \) affects viscosity, with personal care grades yielding comparable and significantly higher viscosities compared to stabilized grades. \( \text{H}_2\text{O}_2 \) concentration may also affect viscosity, but is polymer dependent.

### 6.2 Accelerated Aging

One of the most important steps in the formulating process is to perform stability testing to insure that the final product will perform consistently over its expected shelf life. The results of this study show how acrylic acid polymers affect the stability of hydrogen peroxide and the resulting viscosity.

### 6.2.1 Hydrogen Peroxide Stability

It is important to first understand what makes hydrogen peroxide inherently unstable. Decomposition of \( \text{H}_2\text{O}_2 \) is caused by elevated temperature, UV light, high pH, and contaminants, such as transition metals (copper, manganese and iron), as well as many common organic materials. Therefore, it is important to limit the potential environmental stresses on \( \text{H}_2\text{O}_2 \), such as heat, sunlight, and dust. It is also necessary to use passivated stainless steel equipment and high quality water, and
to formulate with other essential ingredients, such as rheology modifiers, that are compatible with hydrogen peroxide. Synthetic acrylic acid-based polymers are very compatible with H₂O₂, as compared to natural polymers. The following results show what effect polymer type, pH, and H₂O₂ concentration have on hydrogen peroxide stability when thickened with acrylic acid polymers.

No loss of hydrogen peroxide, at pH 4.0, for a 6.0 % w/w gel was observed after 12 weeks accelerated stability, at 45 °C, for Polymers A through D (figure 8). The cross-linked polymers have no effect on H₂O₂ stability, but the linear emulsion polymer causes about 7.0% H₂O₂ loss after 12 weeks. The accuracy of this assay is +/- 0.15 %. Figure 9 shows the effect of harsh conditions on H₂O₂ stability. The gels, thickened in this example by Polymer D, are put under very severe test conditions: a range of peroxide levels, a high pH (4.5), and accelerated stability at 45 °C for 24 weeks. The results show no loss of H₂O₂ over time, even at high pH and peroxide levels. The slight increase in H₂O₂ can be explained by the evaporation of water in the gel samples that may have occurred over time during the high temperature test.

From the results of this part of the study, it can be concluded that cross-linked acrylic acid polymers do not cause loss of hydrogen peroxide at elevated temperatures for 12 weeks, but that the linear emulsion polymer does cause the loss of about 7 % H₂O₂. Harsh conditions, such as high pH (4.5), high peroxide concentrations (12 % w/v), and elevated temperature (45 °C) for 24 weeks do not cause any loss in H₂O₂. Hydrogen peroxide decomposition, at higher pH ranges however, is likely to occur with any formula, due to the inherent instability of hydrogen peroxide at neutral or high pH.

6.2.2 Viscosity Stability

There are several factors which influence the viscosity stability of hydrogen peroxide gels thickened with acrylic acid polymers. The results will show that polymer selection, grade and concentration of H₂O₂, and pH affect the peroxide gel viscosity stability.
The effect of polymer type on viscosity stability at elevated temperature for 12 weeks @ pH 3.5 is shown in figure 10. It is evident from this graph that Polymer A maintains the most consistent viscosity profile at elevated temperature over a 12 week period as compared to the other polymers. Polymer D also shows a consistent viscosity profile for up to 8 weeks, after which about 20% loss of viscosity occurs. Polymers A and D were selected for additional experiments because of the stability data that was observed at 8 weeks.

![Effect of Polymer Type on Viscosity Stability](image10)

**figure 10**

It has been shown that the grade of hydrogen peroxide can impact the initial viscosity. The following results show how the H\textsubscript{2}O\textsubscript{2} grade can impact viscosity stability. Figure 11 shows the viscosity stability of a H\textsubscript{2}O\textsubscript{2} gel made with Polymer D at a pH of 3.6, using a stabilized and personal care grades of hydrogen peroxide (A and D). Although the gel made with the personal care grade (A) produces a higher initial viscosity (24,000 cP), than the viscosity (15,000 cP) produced using the stabilized grade (D), the viscosity loss using the stabilized grade is minimal compared to the personal care grade.

The results in this paper have already shown that Polymer A has a very stable viscosity profile as compared to the other polymers at H\textsubscript{2}O\textsubscript{2} concentration of 6 % active. The next graph, figure 12, illustrates the effect that high H\textsubscript{2}O\textsubscript{2} concentrations have on viscosity stability. This graph shows that even at very high H\textsubscript{2}O\textsubscript{2} concentrations (12 wt%,), the viscosity is very stable for up to 12 weeks. If the study is extended however, viscosity loss eventually occurs, as shown by the 9 and 12 wt % concentrations.

![Effect of H\textsubscript{2}O\textsubscript{2} Grade on Viscosity Stability](image11)

**figure 11**

![Effect of H\textsubscript{2}O\textsubscript{2} Concentration on Viscosity Stability](image12)

**figure 12**

![Effect of pH on Viscosity Stability](image13)

**figure 13**
The effect of pH on viscosity stability was the final factor that was studied. Figure 13 shows the percent viscosity stability at 45 °C using Polymer D at pH’s of 3.5, 4.0, and 4.5 at time intervals of 4, 8, and 12 weeks. No viscosity loss was observed at pH 3.5 after eight weeks, but about 15 - 35 % relative loss was observed at pH 4.0 and 4.5.

From these results, conclusions can be drawn about how the viscosity stability of hydrogen peroxide gels thickened with acrylic acid polymers is affected. Polymers A and D provide the most consistent viscosity stability over time for hydrogen peroxide gels. The grade of H$_2$O$_2$ has a significant impact on stability. Hydrogen Peroxide D, one of the stabilized grades, demonstrates superior viscosity stability compared to the personal care grade (A), although the personal care grade may give a higher viscosity response. As the concentration of H$_2$O$_2$ and pH is increased, the viscosity stability decreases.

### 7. Application of Results:

The results in this paper have demonstrated that excellent long-term stability of hydrogen peroxide-based gels, thickened with cross-linked polyacrylic acid polymers can be achieved. The next logical step in the study was to see how this information could be practically applied to hydrogen peroxide applications. Cross-linked acrylic acid polymers, due to their unique rheological properties, could enhance the performance characteristics of a broad range of applications. Additional experiments were completed to determine if the results obtained from this study could be used for the application of two-part permanent hair color.

#### 7.1 Permanent Hair Color:

Permanent hair color consists of two parts: a high pH color phase, and a low pH developer phase, that contains hydrogen peroxide. The two parts are mixed together prior to application on hair, where the H$_2$O$_2$ is activated at high pH. Figure 14 lists some of the key requirements of each phase as well as the mixture. Cross-linked acrylic acid polymers may offer several unique benefits to enhance the current performance properties as summarized in figure 14.

#### 7.1.1 Mock Hair Color Test

In order to evaluate the high pH thickening properties of the acrylic acid polymers with hydrogen peroxide, a mock hair color test was developed to screen potential polymers. The test formula consisted of:

<table>
<thead>
<tr>
<th>“Developer” :</th>
<th>Deionized Water (Q.S.)</th>
</tr>
</thead>
<tbody>
<tr>
<td>pH = 2.7</td>
<td>1 % active polymer</td>
</tr>
<tr>
<td></td>
<td>6 % active H$_2$O$_2$ (A)</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>“Dye” :</th>
<th>Deionized Water (Q.S.)</th>
</tr>
</thead>
<tbody>
<tr>
<td>pH = 12</td>
<td>8 % Ammonium Hydroxide</td>
</tr>
</tbody>
</table>

The two parts were mixed together (dye into the developer) using equal parts, and the viscosity and pH measured after 3 minutes. The final pH of the mixture was 10, and the active polymer concentration was 0.5% w/w.
Figure 15 shows the viscosity results of the mock hair color mixture at a pH of 10.0 (+/- 0.1). Viscosity results ranged from 85 - 6,200 cP for Polymers A through D, to 10 cP for Polymer E. The cross-linked acrylic acid polymers all show the ability to thicken hydrogen peroxide as compared to the liquid associative polymer. Associative polymers, unlike cross-linked acrylic acid polymers, require high amounts of surfactants to achieve thickening at high pH. What is interesting and unexpected is the unusually high viscosity response Polymer D gives in this test compared to the other polymers. Another unexpected result was the observation that Polymer D produces very clear gels in this test as compared to the other polymers which produced hazy gels.

### 7.1.2 Commercial Hair Color Thickening Test

The mock hair color formula, which contained a cross-linked acrylic acid polymer, demonstrated that a viscous hydrogen peroxide gel could be produced at high pH using a two-part system. In order to test if the cross-linked polymers would thicken in an actual two-part hair color application, commercial hair color was used to mix with a hydrogen peroxide dispersion, containing Polymer D. Five dark brown, commercially available, two-part permanent hair colors were selected and labeled V through Z.

For the first part of the test, each color was mixed with its given components according to the manufacturer’s directions. For the second test, the developer was replaced by the Polymer D Developer, which consisted of 1% w/v Polymer D, and 6% active H$_2$O$_2$ @ pH 2.7. Viscosity and pH of all mixtures were measured after three minutes. Polymer D was selected as the rheology modifier of choice because of the performance characteristics at high pH demonstrated in the mock hair color formula.

The results of the experiment show that the Polymer D developer produces much higher viscosities...
as compared to the commercial developer provided with each hair color. For example, figure 16 shows that the viscosity of the Hair Dye V mixture was 1,230 cP for the commercial developer, and 7,470 cP for the Polymer D Developer. The resulting pH’s of all mixtures, made with the experimental developer, were comparable to the commercial mixtures. From this experiment, it can be concluded that Polymer D performs in actual hair color formulas and yields higher viscosities compared to those produced by the commercial developer.

8. Final Conclusions:

The results of this study have demonstrated that certain cross-linked acrylic acid polymers offer key advantages in hydrogen peroxide applications. Some polymers are very stable in hydrogen peroxide and can provide the ability to produce viscous gels at low pH (3.6). In addition, stable low viscosity hydrogen peroxide solutions, containing a particular cross-linked acrylic acid polymer (D), can be made which will thicken when exposed to high pH. Unique clear gels can be produced by some of the cross-linked polymers.

One of the cross-linked acrylic acid polymers (D), is an ideal thickener for use in two-part permanent hair color applications due to the shear-thinning rheology and vertical cling, or “no drip” rheology. The results of this study show that Polymer D, which is an easy-dispersing acrylic acid copolymer, is an ideal choice for hair color applications. Polymer D produces a very low dispersion viscosity at low pH (2.7), which is necessary for a peroxide developer, and can also make a stable viscous gel at a pH of 3.6, which is optimal for a hair bleach gel. Polymer D is an excellent choice to thicken peroxide-containing systems, with or without surfactants, because of its efficient viscosity building characteristics and clarity when exposed to high pH environments.

9. Acknowledgments:

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10. References:


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