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Electrochemical Impedance Spectroscopy (EIS) Characterization of Water/Sodium Bis(2-Ethylhexyl) Sulfosuccinate-HDEHP/n-Dodecane Reverse Micelles for Electroextraction of Neodymium

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Abstract: The extraction and separation of metal ions in the lanthanide series using the liquid-liquid extraction (LLX) technique poses a major challenge due to the chemical similarities of the metals and hence interest exists in devising a technique to improve the separation factor. In this work, sodium bis(2-ethylhexyl) sulfosuccinate (AOT) is explored for improved organic phase conductivity to aid the use of an imposed external field to improve the LLX. The electrochemical impedance spectroscopy (EIS) technique was used to determine the effect of molar water content, AOT and HDEHP (bis(2-ethylhexyl) phosphoric acid) concentration, and the temperature on the reverse micelle solution conductivity. Results showed that as AOT concentration and water content increases, conductivity increases until the reverse micelles collapse. The addition of HDEHP caused a significant drop in solution conductivity. For a mixed AOT and HDEHP system and at a small applied external field range of 0–1.4 kV m⁻¹ and 60 rpm stir rate, a significant improvement in Nd extraction was observed relative to the traditional LLX using HDEHP only. With AOT only, a 40% improvement in extraction was observed with applied field relative to the absence of field. Cost consideration favors the use of mixed AOT and HDEHP at a slow stir rate for improved Nd extraction.

Keywords: electrochemical impedance spectroscopy; reverse micelles; AOT; liquid-liquid extraction; neodymium; electroextraction

1. Introduction

The extraction and separation of lanthanides and actinides from nuclear waste provides multiple benefits. It has the potential to be a source of metals used in commercial and industrial applications, reduce the volume of waste disposed from a nuclear energy generation process, and may provide fuel for future nuclear reactors. Extracting the individual metals from aqueous solutions is not a trivial task as the similar chemistries between and within each series make separation difficult. Many investigators have developed techniques to partition lanthanides and actinides based on solvent extraction [1–7] and chromatography [8,9], with liquid-liquid extraction (LLX) making up a large percentage of the studies undertaken [10–14]. In traditional LLX, mechanical mixing increases the surface area between two phases where metal ion transfer can occur and is a large driving force for extraction and separation. Adding another driving force, such as an externally applied electric field, could enhance the extraction

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by increasing the amount of metal that is removed [15–18]. Electric fields are typically used in gel electrophoresis and to separate large molecules, such as proteins, thus should be sufficient in shifting metal ions in the solution [19]. Hence, we have chosen to modify the extraction of Nd from an aqueous phase into an organic phase containing bis(2-ethylhexyl) phosphoric acid (HDEHP) in n-dodecane.

It should come as no surprise that the above-mentioned organic phase is highly non-conductive and would inhibit the passage of an electric field. In order to pass an electric field and have an effective driving force, this solution needs to be more conductive than its current state. The use of sodium bis(2-ethylhexyl) sulfosuccinate (AOT) reverse micelles is proposed to increase the conductivity of the organic phase [20–22]. This work endeavors to show that the application of a micellar organic phase and electric field will increase the Nd extraction percent in a liquid-liquid extraction system [23–25]. In addition to increasing the conductivity of the organic phase, these reverse micelles may provide an added enhancement to the LLX system by reducing the surface tension between the aqueous and organic phases, allowing for increased Nd extraction [26]. The electrochemical characteristics (i.e., conductivity) of the reverse micelle-containing organic phase are proposed to be analyzed using the electrochemical impedance spectroscopy (EIS) [20,21,27] method.

One objective of this study is to identify suitable conditions for which water/AOT-HDEHP/n-dodecane reverse micelle systems can be conductive using EIS analysis. A simple mathematical correlation of the EIS experimental data can be used to approximate a resistance of the system, which in turn can be used to estimate the conductivity of the reverse micelle-containing organic phase. We also examine the effect of water content (W_0), AOT concentration, HDEHP concentration and temperature on the reverse micelle-containing LLX system. The study further aims to understand the effect an imposed electric field will have on Nd extraction by applying an electric field to the LLX system, as well as an AOT-containing system. Results of this work could be useful in the modification of industrial LLX operations to be less costly and increase the amount of metal extracted while using fewer separation stages.

2. Materials and Methods

HDEHP was purchased from Alfa Aesar (Haverhill, MA, USA) with a purity over 97%. This purity was determined through potentiometric titration [13]. AOT, n-dodecane and neodymium nitrate hexahydrate were purchased from Sigma Aldrich (St. Louis, MO, USA) and used as is. Degassed deionized water was used throughout the experiment.

2.1. Electrochemical Impedance Spectroscopy for Electrochemical Extraction

Impedance was measured for solutions of varying HDEHP and AOT concentrations in n-dodecane. Aliquots of water were added to 8 mL of the organic solution and the water content, W_0 , calculated by Equation (1) [20].

$$W_{o} = [water]/[AOT] \text{ or } [water]/[AOT + HDEHP]$$
 (1)

Experiments were conducted at room temperature and atmospheric pressure. Dependence of temperature on conductivity was investigated by heating the solution in a water bath within a range of 21 $^{\circ}$ C to 50 $^{\circ}$ C. Solutions temperatures were allowed to equilibrate for 10 min while stirring to eliminate any temperature differences.

Impedance data was collected using a Gamry Series G 300 or Gamry Series 1000 potentiostat (Gamry Instruments, Warminster, PA, USA) with two platinum foil electrodes as the counter/reference and working electrodes with a surface area of approx. 0.785 cm^2 . The distance between the electrodes was fixed at approx. 2.8 cm. The impedance measurements were performed at open circuit potential with an applied 10 mV AC potential in the frequency range of 10^{-1} – 10^5 Hz with 10 steps per decade. The platinum foil electrodes were rinsed with degassed DI (deionized) water between experimental runs. Results were modeled with Echem Analyst (Gamry Instruments), which estimated a resistance using an equivalent circuit model. A diagram of the cell is shown in Figure 1.

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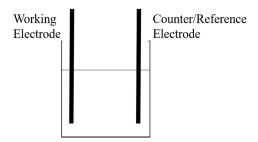


Figure 1. Experimental setup for electrochemical impedance spectroscopy (EIS) measurements.

The resistance obtained from the modeling software was used to calculate conductivity using Equation (2).

Conductivity =
$$\kappa \left(\frac{\mu S}{cm} \right) = \frac{10^6}{\text{Resistance} \times \text{Inter-electrode distance (cm)}}$$
 (2)

Results of this experiment would show the effect of water content, AOT concentration, HDEHP concentration and temperature on the conductivity of a water/AOT + HDEHP/n-dodecane reverse micelle system.

2.2. Effect of Electric Field Strength

The experimental setup used to test the effect of field strength is shown in Figure 2.

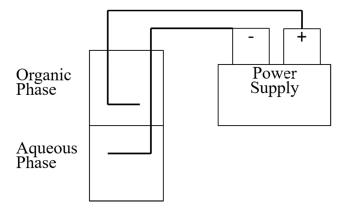


Figure 2. Electrochemical extraction setup.

A 7 V power supply was used to impose a field between two Pt wires. The distance between the wires was set at 5 mm. A 20 mL glass vial was used as the reactor vessel. The aqueous phase was charged to the vessel with a stir bar and the power supply set to a specific voltage. Then, the Pt wires were lowered into the vessel. Only the wire from the negative end of the power supply was submerged in the aqueous phase. The organic phase was gently pipetted in to limit splashing and unnecessary emulsion formation. When running an experiment, time began when the power supply was turned on. In this work, the power source used was limited to a 7 V power supply. The electric field was applied for 15 min while gently stirring to prevent emulsion formation and maintain a single plane where the field can be effective. Pre- and post-extraction Nd concentrations were measured via UV-Vis (ultraviolet visible) absorption spectroscopy. Equation (3) was used to determine the extraction percent of the extraction system.

Extraction % =
$$100 \times ([Nd]_{initial} - [Nd]_{final}) / [Nd]_{initial}$$
 (3)

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3. Results and Discussion

3.1. Electrochemical Impedance Spectroscopy for Electrochemical Extraction

3.1.1. Effect of AOT Concentration on EIS Analysis of Organic Solution Conductivity

To better pass an electric field in a LLX system, a conductive organic phase is necessary. In this work, the organic phase's conductivity was modified using AOT reverse micelles. Three organic solutions with varying initial AOT concentrations were prepared and their impedance (or resistance) measured using EIS. A specific volume of water was added and mixed into the solution and the impedance measured. Figure 3 shows the organic phase conductivity with respect to water content.

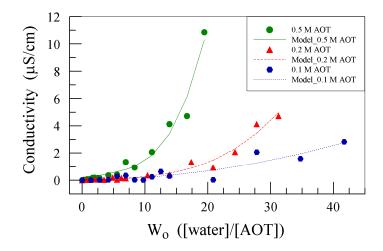


Figure 3. Effect of initial sodium bis(2-ethylhexyl) sulfosuccinate (AOT) concentration and water content (Wo) on conductivity.

Results show that with little water in the system, conductivity is low, essentially zero. This is due to the non-conductive nature of the n-dodecane matrix. However, as the water content increases, conductivity increases. We speculate that this is due to the expansion of the reverse micelles. As more water is added, the bulk water core that is present in the reverse micelles grows in size. This increase in size allows for larger and more conductive regions in the organic phase. The larger the conductive regions, the easier the signal from the frequency generator travels between the two electrodes, thus achieving a less resistive and more conductive system. Percolation conductivity, or the drastic increase in conductivity, occurs at lower water contents as the initial AOT concentration increases. The magnitude of the conductivity change due to percolation also increases as the initial AOT concentration increases. With more AOT monomers present and while waiting for the addition of water to form reverse micelles and aggregates, conductivity increased faster in the solutions with the higher AOT concentration.

Figure 3 also shows that at the same water content, conductivity increases as the initial AOT concentration of the organic phase increases. This can be understood in comparison to a pond filled with lily pads. The more lily pads present, the easier it is for frogs to hop across the pond. The more reverse micelles present, even though they are small in volume, the easier it is for the signal to pass between electrodes. A faster moving signal results from a less resistive, and thus, a more conductive solution. To calculate some comparative value that relates to the number of drops (number of reverse micelles) we use $Z = 100 \times (AOT)/(dodecane)$ which provides a ratio that can visually demonstrate the change in number of reverse micelles as the AOT concentration changes. An estimate of this parameter gave Z number of 4.35, 9.43 and 28.2 for the initial AOT concentration of 0.1, 0.2 and 0.5 M, respectively. The increase in the Z-value with increasing AOT concentration better shows how a small change in the AOT concentration affects the number of reverse micelles and thus the conductivity of the system.

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The fitted equations shown in Figure 3 are the model correlation equations given in Table 1 that show the relationship between the conductivity and molar water content at different AOT concentrations.

Table 1. Model correlation equations for conductivity (units in μ S/cm) of water/AOT/n-dodecane reverse micelles.

[AOT]	Equation	Correlation
0.5 M	$\sigma = 0.002680 \times W_0^3 - 0.03911 \times W_0^2 + 0.2870 \times W_0 - 0.2087$	98.67%
0.2 M	$\sigma = 0.0002087 \times W_o^3 - 0.002359 \times W_o^2 + 0.02918 \times W_o + 0.003058$	98.41%
0.1 M	$\sigma = -0.000001473 \times W_o^3 + 0.001628 \times W_o^2 + 0.0002050 \times W_o + 0.03963$	92.79%

3.1.2. Effect of AOT + HDEHP on EIS Analysis of the Conductivity of Organic Solution

HDEHP is one of many trivalent metal ion extracting ligands used in the LLX industry and research and is used here as the main extracting ligand. Figure 4 shows the effect of HDEHP on the organic phase conductivity.

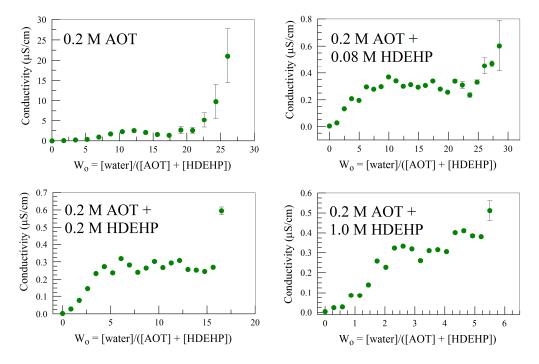


Figure 4. Effect of bis(2-ethylhexyl) phosphoric acid (HDEHP) on organic phase containing 0.2 M AOT.

For this experiment, water was added until the reverse micelles collapsed and formed a biphasic system. With our system, HDEHP has an estimated conductivity of $7.09 \times 10^{-2} \pm 0.0012~\mu S/cm$. As it was added to the AOT/n-dodecane solution, the conductivity of the organic phase plummeted. This indicates that the ligand is not adding to the conductivity and may disrupt the signal transfer between the water cores in the organic solution. We speculate that HDEHP is dispersed in and adds another form of resistance to the organic phase. Researchers have shown that metal salts of HDEHP can form aggregates as well as rod- and sphere-shaped reverse micelles [28,29]. These other reverse micelles or aggregates may be providing another set of landing pads for the signal to use to travel between the electrodes. While still being more conductive than the n-dodecane matrix, these HDEHP-based reverse micelles and aggregates are far less conductive than their AOT counterparts. Figure 4 also shows that as HDEHP concentration increases, less water can be added to the system. This shows that HDEHP may disrupt the AOT reverse micelle properties. A lower volume of the conductive water could result in reverse micelles with smaller bulk water contents, and thus the more difficult the signal

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transfer between electrodes. At lower HDEHP concentrations, there are enough HDEHP molecules to disrupt the AOT-based reverse micelle structure. At higher HDEHP concentrations, reverse micelles and aggregates based on HDEHP are also present. Both these events may lead to the decrease in conductivity. However, even at the two extreme ligand concentrations, the maximum conductivity remains relatively the same. This result shows that this range of HDEHP does not add to the resistance, but alters the AOT reverse micelle properties, making them less able to contain large volumes of water, thus reducing the conductivity of the organic phase.

3.1.3. Effect of Temperature on EIS Analysis of Organic Solution Conductivity

The effect of temperature on organic solution conductivity was tested by adding various volumes of water to three samples of differing initial AOT concentrations while keeping the HDEHP concentration constant. For the investigation of the temperature effects study, the volume of added water was used to calculate molar water content, and it ranged from 0 to ~3.5. This corresponds to the area in Figure 3 where the rate of change in conductivity with respect to water content is low. These experiments were conducted to test the effect of temperature in this low conductivity area. Figure 5 shows the results of these experiments.

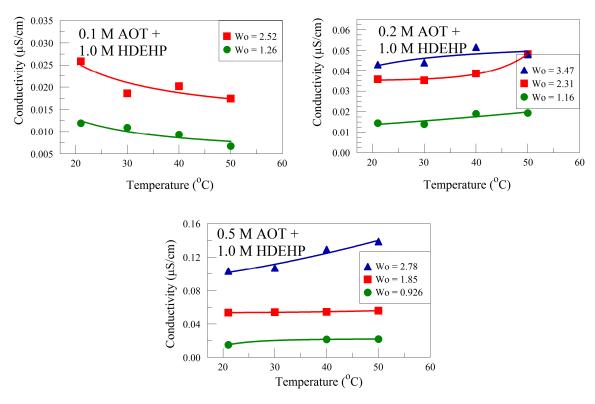


Figure 5. Effect of Temperature on water/AOT + HDEHP/n-dodecane reverse micelles at various volumes of added water.

Within each plot and at all the temperatures studied, an increased molar amount of water favors an increase in conductivity similar to the results in Figure 3. Across the plots, as the AOT concentration increases, the conductivity increases, which is also in accordance with the previous results. With 0.1 M AOT + 1 M HDEHP, an increase in temperature results in a decrease in conductivity, while at 0.2 M and 0.5 M AOT and 1 M HDEHP, the temperature increase results in increased conductivity. The difference between the findings is speculated to result from different amounts of reverse micelles present in the system. At a lower AOT concentration, there would be fewer reverse micelles present. At increased temperatures, Brownian motion would allow the few reverse micelles present, and thus the conductive water core, to quickly travel through the system, making it more difficult for the signal

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to pass between the electrodes, thereby reducing the conductivity. At increased AOT concentrations, more reverse micelles are present and they that can act as a transmission line for the signal between the electrodes. With an increase in temperature at larger AOT concentrations, increased Brownian motion reduced the time of the micelles are in a certain spot, spreading out the conductive regions and thus reducing the resistance of transfer, increasing conductivity. Moreover, it is theorized that beyond certain temperatures, the increased Brownian motion reduces the viscosity of the system, also increasing conductivity. Similar results were found by [20]. These results further show that AOT concentration affects conductivity more than molar water content. For subsequent results, the temperature was fixed at room temperature (approx. 21 °C).

Despite the negative effect of the HDEHP, these results show that a water/AOT + HDEHP/n-dodecane organic phase is conductive enough for the passage of small amount of electric current. Subsequently, the effect of varying field strengths on the extractability of Nd was examined.

3.2. Effect of Electric Field Strength

Electric fields of varying strengths were applied to three systems of differing organic phases: 0.2 M AOT only, 0.2 M HDEHP only and a mixture of 0.2 M AOT, and 0.2 M HDEHP all in n-dodecane. Based on EIS results, the concentration of AOT was chosen to provide a conductive solution while maintaining a lower viscosity. Experiments were stirred at 60 rpm to maintain a plane where the electric field can consistently be applied. Similar experiments at 60 rpm were carried out in absence of applied filed for the three organic phases. The results are presented in Figure 6 for all cases examined.

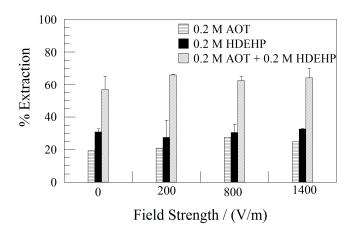


Figure 6. Effect of electric field strength (applied voltage) on Nd percent extraction with different organic phases at 60 rpm.

In organic solutions with 0.2~M AOT only, increasing the field strength up to 800~V/m gives a steady increase to percent extraction. In the case of 0.2~M HDEHP only organic phase, the effect of field strength on percent extraction does not appear to increase linearly. However, HDEHP extracts Nd better than AOT as HDEHP is selective towards trivalent metal ions. With a mixture of both AOT and HDEHP, there is an improvement in percent extraction when a field is applied in comparison to the absence of imposed field. The effect of mixed AOT and HDEHP is greater than the combined effect of the individual components at the stir rate of $60~\rm rpm$.

In order to quantify the relative effect of applied field on the extraction of Nd, a comparison is made between extraction in the presence and absence of applied fields. For instance, the relative effect of electric field on extraction of Nd when using only AOT is shown in Figure 7.

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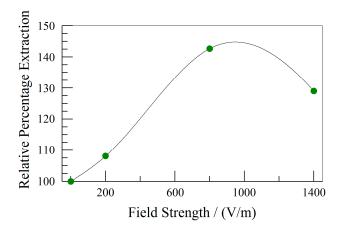


Figure 7. Relative extraction effect of an applied electric field on Nd extraction with AOT at 60 rpm.

The figure shows that an imposed electric field improves the percentage extraction of Nd relative to the results obtained in its absence to the extent that, within the low field range applied (0–1.4 kV/m), up to 40% improvement was observed. This improvement with the field could be a result of the reverse micelles increasing the conductivity of the organic phase. It can be argued that with an applied field, the charged particle experiences less resistance passing between the aqueous and organic phase as there is now present a more "conductive pathway" through the organic solution. The field direction can push more Nd toward the organic phase where the AOT reverse micelles can encapsulate it into their water cores. Similar results were obtained for the other organic phases (0.2 M HDEHP, and mixture of 0.2 M AOT and 0.2 M HDEHP) when a comparison is made between applied field facilitated extraction versus no imposed field extractions. However, only a slight improvement (less than 18%) was observed for these two cases containing HDEHP.

HDEHP is the more well-known and accepted LLX extractant for the lanthanides. In order to examine and quantify the applied field effects on the organic phases relative to traditional LLX HDEHP system, the results in Figure 6 are analyzed and compared to the percentage extraction obtained with HDEHP alone in a classic LLX system. Thus, in Figure 8, we compare relative extractions with applied field for AOT alone, HDEHP alone, and a mixture of AOT and HDEHP versus the 0.2 M HDEP LLX system. It is to be noted that the traditional LLX systems use stir rates much higher than the 60 rpm used for the present results.

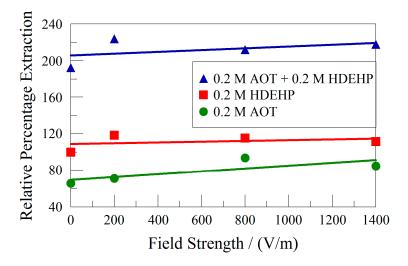


Figure 8. Electric field effects on Nd extraction compared to no field HDEHP LLX (liquid-liquid extraction) of Nd at 60 rpm.

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Figure 8 shows that both in the absence and presence of an applied field, the 0.2 M AOT organic phase extracts less Nd than 0.2 M HDEHP. For the 0.2 M HDEHP alone, the applied field led to a slight improvement relative to the LLX HDEHP system. A more interesting result is observed when the two—AOT and HDEHP—are mixed together, whereupon an improved extraction of almost 100% is observed. The application of the field showed little to no trend when using HDEHP for extractions. This was expected because HDEHP in n-dodecane was highly non-conductive, as the EIS measurements and conductivity calculations showed, which implies that the solution would resist the passage of the field, decreasing or eliminating its effect on Nd extraction. The effect of the field is more prominently displayed by the slope of the AOT and the AOT + HDEHP results. This slope, and thus the resulting trend, shows that the field had more of an effect in the systems where the AOT reverse micelles were present. The improved conductivity that the reverse micelles provide allows passage of the "field effect" and this ability to pass a field in combination with AOT's ability to extract Nd improved the overall percent extraction.

An electroextraction process involving AOT and HDEHP could reduce costs by using slower stir rates, mild electric field, less expensive ligand (AOT) along with the more expensive ligand that has a higher metal ion selectivity. Our results [30] demonstrate that the mixed AOT and HDEHP system at 60 rpm extracted as much as 96% of Nd relative to amount extracted by HDEHP LLX system stirred at 1000 rpm.

4. Conclusions

The goal of our work was to use AOT to increase the conductivity of the organic phase of a liquid-liquid extraction system in order to apply an electric field to aid in extraction. Our objectives were to identify suitable conditions where water/AOT + HDEHP/n-dodecane reverse micelles form a conductive organic phase and understand the effect of electric field on the Nd percent extraction in an electrically modulated LLX system. Impedance results showed that increasing AOT concentration and water content increased n-dodecane conductivity. This may be due to the increased number and size of the reverse micelles, respectively. HDEHP has a detrimental effect on conductivity, probably due to the ligand disrupting the properties of the reverse micelles. Temperature had a mild effect on conductivity. Results show that a limited field strength at a very low stir rate can be used in conjunction with mixed AOT and HDEHP to affect an improved extraction of Nd over the traditional LLX system in which HDEHP only is used. In the absence of a mixed organic phase, an applied field through this micellar organic phase had a mild effect on increasing percent extraction relative to the traditional liquid-liquid extraction experiment with HDEHP and without field. Cost and energy analysis favor a mixed organic phase at a low stir rate for Nd extraction.

Future works will include testing the electrically driven extraction at higher applied electrical fields and selectivity studies involving other Lanthanide series metals. Another future work could include testing the electrically driven extraction with increased AOT content in the organic solution.

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Author Contributions: Shannon Anderson, Egwu Eric Kalu and Mikael Nilsson conceived and designed the experiments. Shannon Anderson performed the experiments and wrote the paper. Egwu Eric Kalu advised the project. Shannon Anderson and Egwu Eric Kalu analyzed the results.

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