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Evaluation of samples from a gold leaching plant and surrounding groundwater: Application of a potentiometric ion-selective electrode for cyanide

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Abstract

This paper presents the evaluation of physicochemical parameters of samples from a gold leaching plant and surrounding waters (groundwater and a river). A potentiometric selective electrode was calibrated in the concentration range of 1.99×10^{-5} to 7.9×10^{-3} mol/L for cyanide quantification. The calibration showed a Nernstian response with a slope of –61.8 mV/dec ($R^2 = 0.9938$), the practical detection limit was 1.45×10^{-6} mol/L, while the lower limit of linear response was 3.17×10^{-6} mol/L. The use of this sensor provides advantages such as shorter analysis time, less operational difficulty and fewer interferences compared to traditional cyanide methods such as volumetry or spectrophotometry. Various samples from gold leaching stages at a plant in Santiago de Cuba (Cuba) were analyzed. No cyanide contamination was detected in a river and two wells surrounding the plant, which are used as a source of drinking water. Other water quality parameters were analyzed in the two wells that showed elevated levels of nitrite and nitrate compared to quality standards, indicating contamination of biological origin or due to agricultural activities that do not allow the use of these groundwaters for drinking.

Keywords: groundwater, well, river, mining industry, water quality.

Evaluación de muestras de una planta de lixiviación de oro y de las aguas subterráneas aledañas: Aplicación de un electrodo potenciométrico selectivo a iones cianuro

Resumen

En este trabajo se presenta la evaluación de los parámetros fisicoquímicos de muestras procedentes de una planta de lixiviación de oro y de las aguas aledañas (subterráneas y de un río). Para la determinación del cianuro se calibró un electrodo selectivo potenciométrico en el rango de concentraciones de 1.99×10^{-5} a 7.9×10^{-3} mol/L. La calibración mostró una respuesta nernstiana con una pendiente de –61.8 mV/dec ($R^2 = 0.9938$), el límite práctico de detección fue de 1.45×10^{-6} mol/L, mientras que el límite inferior de respuesta lineal fue de 3.17×10^{-6} mol/L. El uso de este sensor proporciona las ventajas de un menor tiempo de análisis, menos complejidad operativa y menos interferencia en comparación con los métodos tradicionales para cianuro, como la volumetría o la espectrofotometría. Se analizaron varias muestras procedentes de etapas de lixiviación de oro en una planta de Santiago de Cuba (Cuba). No se detectó contaminación por cianuro en un río, ni en dos pozos que rodean la planta y que se utilizan como fuente de agua potable. Se analizaron otros parámetros de calidad del agua en los dos pozos, los cuales mostraron niveles elevados de nitrito y nitrato en comparación con las normas de calidad, lo que indica una contaminación de origen biológico o debida a las actividades agrícolas que no permiten el uso de estas aguas subterráneas como fuente potable.

Palabras clave: agua subterránea, pozo, río, industria minera, calidad del agua.

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INTRODUCTION

The extraction and processing of precious metal ores is one of the most important areas of mining due to their high commercial value and their applications in fields such as technology and medicine. Gold is well known as a precious metal, widely used in jewelry because of its resistance to corrosion, which makes it possible to create durable and eye-catching pieces thanks to its golden luster, but its applications go far beyond this. It is used in electronic circuits because of its high electrical conductivity and inertness to chemical reactions. Gold compounds are used in medicine to treat certain diseases such as rheumatoid arthritis and some cancers. It is also used in space satellites for its good electrical conductivity and low infrared emissivity, among many other applications (Huy Do, Tien Nguyen, Dong Thach, Lee & Huu Bui, 2023; Mertens, Gukathasan, Arojojoye, Olelewe & Awuah, 2023; Sharma, 1989).

Due to its low reactivity, gold is commonly found in nature as a native metal. However, it also occurs as an element in some ores. The processing of precious minerals such as gold and silver includes sodium cyanide leaching stages where the anion acts as a leaching agent by forming soluble complexes (Surimbayev *et al.*, 2024; Zounia, Yazdi, Hakimi, Zare & Amiri, 2024). Although alternatives to cyanide ion have been sought for the mining industry, the fact remains that it is virtually irreplaceable (Gui *et al.*, 2023; Zhang, Cui, Wang, Liu & Lyu, 2022).

Cyanide ion is well known for its toxicity and lethality at low concentrations. It is therefore a chemical species of high concern for industrial and environmental control. It is of particular concern in the mining industry, where the quantities required to leach precious metals can be as high as 4.5 g/L NaCN (Deschênes, McMullen, Ellis, Fulton & Atkin, 2005). Therefore, when residues are generated, they must be strictly controlled to avoid pollution episodes resulting in toxicity to fauna and flora, including humans. Cyanide species depend on pH, redox potential, and the presence of other ions with which they can form complexes (Bahrami, Hosseini & Razmi, 2007). This is critical in determining the mobility of cyanide in contaminated soil and waters. All this means that, under certain conditions, cyanide is less hazardous in ecosystems, while in others it is very easily incorporated into drinking water, especially in wells near mining areas. In Santiago de Cuba, there is a copper mine that is currently closed, but in the same place gold deposits have been found that are mined and processed using sodium cyanide leaching.

Groundwater quality in mining areas is particularly important due to the mobility of contaminants released during mineral processing. As a result, they can be easily incorporated into human and animal consumption, with adverse health effects. In the case of gold processing plants, the cyanide ion is of great toxicological importance in groundwater (Abdalla, Suliman, Al-Ajmi, Al-Hosni & Rollinson, 2010). However, it is worth noting that the quality of groundwater cannot be based solely on the determination of a single species or chemical.

This work presents a study of the quality of groundwater surrounding a mining plant in Santiago de Cuba, Cuba. Several physicochemical parameters of water quality were determined, and a potentiometric ion-selective electrode (ISE) was used to quantify cyanide in samples from the gold leaching plant and the groundwater studied. For this purpose, the electrode was calibrated, and the response parameters of the sensor were determined.

The novelty of this work lies in the fact that, for the first time, the groundwater surrounding the gold leaching plant in Santiago de Cuba has been studied and, in addition, the levels of cyanide handled at the plant and its impact on the waters have been reported.

MATERIALS AND METHODS

In this work, samples from a gold leaching plant were analyzed (Figure 1), a river, as well as groundwater from two wells (Well I and Well II) near the mining area that are used by the surrounding population as a source of drinking water.

All the experiments in this work were carried out using the materials and equipment available at the laboratory of the "Elio Trincado" Geominera Oriente Company and the Universidad de Oriente, Santiago de Cuba. A Mettler AE 160 digital balance, a Memmert 100-800 oven, and a Genesis 10S UV-Vis spectrophotometer were used. The chemical reagents used were analytical grade (purity $\geq$ 95%). The physicochemical indicators determined were selected considering the methods described in Cuban and international standards.

Temperature was measured *in situ*. Sampling was performed at 30 cm from the surface of the water column. Triplicate samples were taken at each sampling station. Samples were collected in 1.5 L plastic containers, previously washed with 10% HNO_3 and distilled water.

The parameters analyzed for groundwater from two wells near the gold leaching plant were: temperature, pH (potentiometric method), electrical conductivity (conductimetric method), settleable solids (gravimetry), chloride (titration), total hardness (titration), calcium hardness (titration), magnesium hardness (titration), sulfate (turbidimetry), phosphate, nitrate, nitrite, ammonium (all by the UV-vis spectrophotometric method), total dissolved solids (gravimetry), sodium and potassium (flame photometry). The determinations were conducted in accordance with the procedures outlined in the Standard Methods for the Examination of Water and Wastewater, as established by the American Public Health Association, the American Water Works

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Figure 1. Satellite image of the location of the gold-barite mine studied in this work.

Association, and the Water Environment Federation (American Public Health Association, American Water Works Association & Water Environment Federation, 2023).

Cyanide was determined by the potentiometric method with an Orion ion-selective electrode (Thermo Scientific, 9606BNWP). Sodium cyanide (NaCN) was used to calibrate the electrode, which was periodically standardized with $AgNO_3$ solution. For the ionic strength adjustment, a 0.01 mol/L NaOH solution was used. A Thermo Scientific Orion VersaStar multichannel potentiometer was used for potential measurements. The measured solutions were stirred with a Jenway-1000 magnetic stirrer.

The results obtained in the physicochemical characterization of the waters of the two wells were compared with the values established by the Cuban Standard 827 of 2017: "Potable Water - Sanitary Requirements", referred to as NC 827:2017 (Oficina Nacional de Normalización, 2017). In addition, some values established by international regulatory agencies such as the World Health Organization (WHO) (WHO, 2022), the Environmental Protection Agency (EPA) (U.S. EPA, 2024) and the European Union regulation for drinking water (European Commission, 2020) were considered.

RESULTS AND DISCUSSION

Cyanide quantification with the potentiometric ion-selective electrode (ISE)

Figure 2 shows the ISE calibration curve for cyanide using the standard addition method. It can be observed that the electrode responds linearly over the entire activity range of cyanide ion. The regression parameters are shown in Table I, where it can be confirmed that the response is linear with 99.69% direct correlation, while the mathematical model explains 99.38% of the linear relationship. The p-value of the analysis of variance of the regression is so small that it is close to zero and therefore lower than 0.05, which demonstrates the validity of the linear relationship between the potential difference and the logarithm of the cyanide ion activity with 95% confidence. On the other hand, Table II shows the ISE calibration parameters for cyanide. One of the most important parameters is the slope, which is very close to the theoretical Nernstian value of −59.16 mV/dec for a monoelectronic transfer (Manjakkal, Szwagierczak & Dahiya, 2020). This indicates that the potentiometric response is adequate as theoretically expected. On the other hand, the practical detection limit (PDL) is very low, corresponding to 37.73 ppb. This limit should be understood as the lowest concentration of cyanide that can be reliably detected by the sensor. The lower limit of linear response (LLLR) is 82.48 ppb. The LLLR is the lowest cyanide concentration at which the potentiometric sensor exhibits a linear response, ensuring that the measurement is quantitatively reliable. The detection and quantitation limits obtained in this study enable cyanide ions to be determined in natural and industrial waters.

Table III shows cyanide concentrations at various sampling points associated with the gold recovery process at the plant. The emergency lagoon is a reservoir into which the leach liquor is evacuated when the main lagoon is full or there is a situation that requires it to be maintained at a certain level. In the emergency lagoon, the cyanide undergoes a degradation process using hydrogen peroxide, a known oxidizing agent. Therefore, the concentration is expected to be lower than that used in the process. However, it should be noted that concentration

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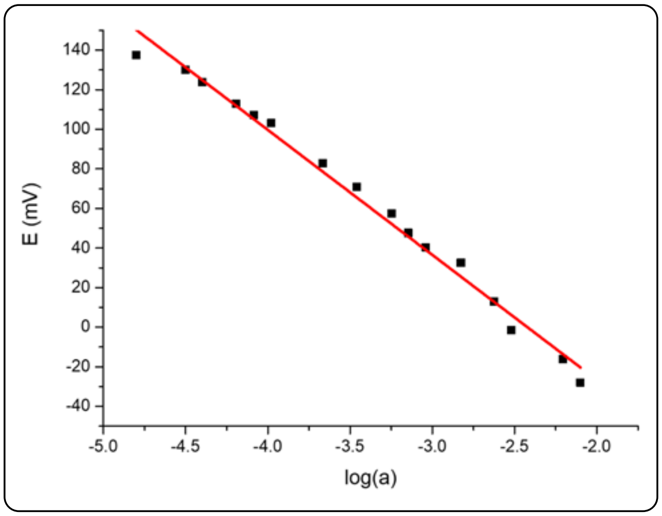

Figure 2. ISE calibration curve for cyanide using the standard addition method.

Table I. Regression parameters for the ISE calibration curve.

Regression parameter	Value
N	16
Correlation coefficient (r)	0.9969
Determination coefficient (R^2)	0.9938
$R^2_{adjusted}$	0.9925
p-value	0.0000

Table II. Calibration parameters of the ISE for cyanide.

Calibration parameter	Value
S (mV/dec)	–61.8±1.9
PDL (mol/L)	1.45×10^{-6} (0.038 ppm)
LLLR (mol/L)	3.17×10^{-6} (0.082 ppm)

Table III. Cyanide content in gold leaching plant samples.

Source	CN^- (ppm)
Emergency Lagoon	1.500 ± 0.300
River to tailings tank	0.020 ± 0.004 < PDL
Tailings tank	0.020 ± 0.006 < PDL
Mine tailings (1)	0.022 ± 0.003 < PDL
Mine tailings (2)	0.220 ± 0.020
< PDL: Value lower than the practical detection limit of the ISE (0.038 ppm)	

is still high, although this does not pose an environmental or toxicological risk, since it is an unexposed industrial disposal site, unless there is an exceptional event. The river to the tailings tank is a water channel that contains the residue in suspension and leads it to the tailings tank. In both cases, the cyanide levels are below even the levels set for drinking water by the WHO (0.07 ppm) (WHO, 2022) and other international agencies such as the EPA (0.2 ppm) (U.S. EPA, 2024). The same occurs with the concentration of cyanide in mine tailings (1), but in the case of mine tailings (2), the concentration is higher.

In the case of the river leading to the tailings tank, the tailings tank, and the mine tailings (1), it is important to note that the quantified cyanide value is lower than the PDL of the ISE (see Table III). However, the PDL is lower than the maximum limit established by the standards (0.07 and 0.2 ppm for WHO and EPA, respectively). This indicates that, although the cyanide concentration lacks statistical reliability, all evidence confirms that the cyanide concentration in these samples is well below the permitted limits for such controlled samples as drinking water, thus validating the use of the ISE. Tailings are very important because they are materials that remain exposed for long periods of time and, if not properly managed, can lead to contamination of soil and groundwater through mechanisms such as runoff and leaching. In addition, if there is no properly prepared structure for waste storage, cyanide can percolate into the soil and reach the groundwater.

Figure 3 shows the concentration of cyanide in the water of a river near the gold leaching plant and the two community wells nearby. In none of the cases did the cyanide level exceed the limit for drinking water set by both the WHO and the EPA (U.S. EPA, 2024; WHO, 2022), as well as the Cuban national standard for drinking water (0.05 ppm) (Oficina Nacional de Normalización, 2017). Note that here, the quantified values are also lower than the PDL of the ISE, but they are a good indication that the actual values are well below the permissible levels. This indicates that waste control at the leaching plant is adequate to ensure no contamination of the surrounding groundwater or surface water.

Quality parameters of groundwater

It is important to keep in mind that cyanide concentration is not the only indicator of contamination of the groundwater analyzed. Other variables may be important for the evaluation of water quality, whether natural or waste. Table IV shows some physicochemical parameters that can normally indicate pollution. Temperature is a physical parameter that influences the state of the water since it regulates the solubility of certain species that can be toxic depending on whether they are ionized or not. In addition, certain substances such as dissolved oxygen depend on temperature and directly impact life in aquatic ecosystems (Hussain, Lazem, Resan, Taher & Sabbar, 2010). Moreover, biological processes such as photosynthesis or the growth of microorganisms depend on temperature (Cruz-Paredes, Tájmel & Rousk, 2021; Lin, Medlyn & Ellsworth, 2012; Yamasaki,

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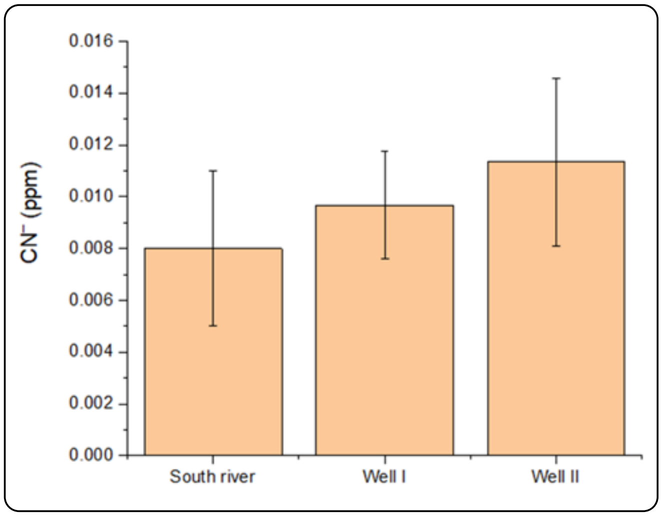

Figure 3. Cyanide concentrations from a river and the two wells (I and II) near the gold leaching plant.

Yamakawa, Yamane, Koike, Satoh & Katoh, 2002). Table IV shows that the temperature of the waters of both wells is normal for the Cuban tropical climate.

The pH is a chemical property of significant importance for aquatic ecosystems. The concentration of hydronium ions regulates the solubility of metals; at low pH values, metals are more soluble, which increases bioaccumulation and toxicity (Wang, Meador & Leung, 2016). The pH also plays an essential role in aquatic life. In these wells, the pH is within both international and Cuban standards for drinking water quality (EPA and NC 827:2017 state the same range: 6.5 − 8.5). On the other hand, electrical conductivity is normally related to the amount of ions dissolved in the water. Conductivity is not regulated in the Cuban standard nor in many international standards such as WHO or EPA. However, the European Union establishes an upper limit of 2500 µS/cm (20°C) for drinking water (European Commission, 2020), which is much higher than the conductivity of the water from both wells.

Hardness is an important quality parameter, which describes the concentration of mineral salts in the water, mainly calcium and magnesium (Orellana, Darder & Quílez-Alburquerque, 2023). According to the WHO, the consumption of hard water does not harm human health (WHO, 2003); however, this type of water does have an effect on industrial and domestic water flow systems due to the accumulation of insoluble salt incrustations. The waters analyzed in both wells can be classified as soft because the content is less than 100 ppm. The quantified value does not exceed the upper limit established by the Cuban standard (Oficina Nacional de Normalización, 2017).

Calcium and magnesium contents are higher in Well I than in Well II, the difference being more significant for the first element. The variation in calcium and magnesium contents is usually associated with differences in the type of soil rock where the groundwater is located or some source of contamination. The former means that soil geochemistry plays a fundamental role in the chemical composition of groundwater (Mather, 2020). In this case, for Well I, the Ca content is higher than the limit established by NC 827:2017 (Oficina Nacional de Normalización, 2017). This may indicate contamination. The sodium content is within the permissible level, and also higher for Well I than for Well II; whereas the potassium content is not regulated by national or international standards.

In the case of solids, it can be observed that only the maximum permissible limit for total dissolved solids is regulated by NC 827:2017 (Oficina Nacional de Normalización, 2017). In this case, Well I presents a concentration of dissolved solids higher than the 1000 ppm limit established by the standard, which has to be related to the higher amount of solubilized salts in these waters according to Table IV. This indicates also contamination of Well I that makes it unsuitable as a source of drinking water. In contrast, in the case of Well II, the dissolved solids content does not exceed the permissible limit.

Total suspended solids are non-sedimentable solid particles found in water. Whereas settleable solids are suspended particles that, depending on their size, can settle. They are important because suspended solids decrease the clarity of the water, which causes blockage of sunlight, preventing photosynthesis of aquatic plants and therefore decreasing the dissolved oxygen in the water (Mitchell, West & Guymer, 1999). It also decreases the growth of photosynthetic plankton, which is the base of the aquatic food chain. Suspended solids are related to eutrophication, as solid particles can contain nutrients that promote algal blooms (Fernández del Castillo, Garibay, Senés-Guerrero, Orozco-Nunnelly, de Anda & Gradilla-Hernández, 2022). For Wells I and II, both suspended and settleable solids levels are not elevated.

Ammonium, nitrite, and nitrate are chemical species involved in the natural nitrification process, where ammonium is oxidized by microorganisms to form nitrite, and this is subsequently oxidized by microorganisms to form nitrate (Bernhard, 2010). The relationship between concentrations of these species indicates whether there is recent contamination of the waters, mainly of fecal, agricultural, or industrial origin. Table V shows the ammonium, nitrite, and nitrate values for Wells I and II analyzed in this work. It can be observed that in both cases the ammonium and nitrite concentrations are low; however, the nitrate concentration is very high for both wells. The Cuban standard for drinking water (Oficina Nacional de Normalización, 2017) does not establish control over ammonium concentration, however, it does for nitrite and nitrate. For both wells, the nitrite content is slightly higher than the maximum permissible limit of 0.01 ppm established by this standard. On the other hand,

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the nitrate content is significantly higher than the maximum limit of 45 ppm established by this standard. This indicates biological contamination in advanced stages of mineralization during the nitrification process and may also be an indicator of contamination from agricultural processes where fertilizers are used. The use of this type of product in agricultural activities to increase crop production causes nitrogen species pollution, as fertilizers not absorbed by plants are either lost as gases (e.g., ammonia or nitrous oxide) or leach from the soil-plant system into water bodies (Bijay-Singh & Craswell, 2021; Wick, Heumesser & Schmid, 2012). Nitrate-based fertilizers, such as ammonium nitrate, sodium nitrate, and potassium nitrate, contribute directly to groundwater contamination through leaching. In contrast, non-nitrate fertilizers, such as urea, lead to nitrite and nitrate contamination through microbial transformation processes. Urea is subject to hydrolysis by microbial urease, which generates ammonia that can be lost through volatilization or be oxidized by microbial nitrifiers to form nitrate. The stages of nitrification include the microbial oxidation of ammonia to hydroxylamine, which is converted to nitrite; and nitrite is then further oxidized to nitrate (Fu *et al.*, 2020). It is important to consider that the very high concentration of nitrate leads to an accelerated development of algae that eventually leads to a decrease of dissolved oxygen in the water, affecting the development of aquatic life and the potability of the natural resource (Akinnawo, 2023; Mishra, 2023). All these elements lead to state that the waters of Wells I and II are contaminated and they are not suitable for human consumption as a drinking source. However, it should be noted that nearby mining activity is not responsible for the contamination of these waters.

Conclusions

The calibration of commercial potentiometric selective electrodes for cyanide by the method of standard addition allows a reliable determination in natural and industrial samples with a response close to the theoretical one for a monoelectronic transfer according to the Nernst equation. In the case of the electrode used in this work, a slope of –61.8 mV/dec ($R^2 = 0.9938$) was obtained.

The well and river waters analyzed in this work do not show cyanide contamination due to nearby gold leaching activities. Most of the other physicochemical quality parameters are within the limits established by Cuban and international regulations. However, the calcium concentration and total dissolved solids in one of the wells (249.78 and 1567.62 ppm, respectively, for Well I) exceed the Cuban standard. On the other hand, nitrite and nitrate contents are higher than the Cuban standard for

Table IV. Physicochemical parameters for the waters of Well I and Well II, and maximum permissible limit or range stablished by the Cuban standard for drinking water.

Parameter	Well I	Well II	NC 827:2017
T (°C)	30	28	–
pH	7.19	6.54	6.5 – 8.5
Conductivity (µS/cm)	532.37 ± 1.25	622.71 ± 0.41	–
Total hardness (ppm)	17.5 ± 0.54	16.57 ± 0.35	400
Ca (ppm)	249.78 ± 0.05*	33.47 ± 0.05	200
Mg (ppm)	68 ± 0.42	39.64 ± 0.4	150
Na (ppm)	32.87 ± 0.15	6.56 ± 0.21	200
K (ppm)	7.02 ± 0.5	6.56 ± 0.41	–
Total dissolved solids (ppm)	1567.62 ± 0.45*	420.71 ± 0.85	1000
Total suspended solids (ppm)	23.75 ± 0.45	32.71 ± 0.45	–
Settleable solids (ppm)	25.25 ± 0.45	25.28 ± 0.45	–
*Value exceeding the permissible limit			

Table V. Ammonium, nitrite, and nitrate values for Wells I and II.

Parameter (ppm)	Well I	Well II	NC 827:2017
Ammonium	0.02 ± 0.01	0.01 ± 0.01	–
Nitrite	0.02 ± 0.02*	0.02 ± 0.01*	0.01
Nitrate	53.76 ± 0.15*	59.33 ± 0.05*	45
*Value exceeding the permissible limit			

drinking water with values of 0.02 ppm nitrite for both wells, 53.76 ppm nitrate for Well I, and 59.33 ppm nitrate for Well II. The water in the wells analyzed is not contaminated by mining activities but may be contaminated by biological sources or agricultural activities, making it unfit for human consumption.

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