

Communication

Methodological Approach for an Online Water Quality Monitoring System in an Iron Ore Tailing Dam

Renato Oliveira da Silva Júnior ^{1,*}, Helena Pereira Almeida ¹, Marcio Sousa da Silva ¹, Adriano Cuenya França ², Eduardo Balleroni ², Nailson dos Santos ², Paulo Henrique Vilela ², Adayana Maria Queiroz de Melo ¹ 
and José Tasso Felix Guimarães ¹ 

¹ Vale Institute of Technology, Rua Boaventura da Silva 955, Nazaré, Belém 66055-090, PA, Brazil; helena.pereira.almeida@pq.itv.org (H.P.A.); marcio.sousa.silva@itv.org (M.S.d.S.); adayana.melo@pq.itv.org (A.M.Q.d.M.); tasso.guimaraes@itv.org (J.T.F.G.)

² Metrohm Brazil Ltd.a, R. Minerva, 161-Perdizes, São Paulo 05007-030, SP, Brazil; adriano.franca@metrohm.com.br (A.C.F.); eduardo.balleroni@metrohm.com.br (E.B.); nailson.dossantos@metrohm.com.br (N.d.S.); paulo.vilella@metrohm.com.br (P.H.V.)

* Correspondence: renato1965@gmail.com or renato.silva.junior@itv.org

Abstract: Monitoring the concentration of potentially toxic elements (PTEs) in the aquatic ecosystems of the Amazon is critical to guarantee the maintenance of the ecological balance and the life quality of human populations that reside in or use these environments for survival. In this sense, many rivers in the region are dammed to form lakes for depositing mining tailings. Among these, the Gelado Project has the largest iron ore dam in the Amazon that occupies about 13.5 km² of surface area with 142 million m³ of water and tailings volume, which are currently being mined for exploration, and its upstream waters and downstream are historically used by traditional populations. Based on this, to monitor the impacts of this activity, an online system for sampling and automatic analysis of water quality, composed of three process analyzers monitoring more than 20 parameters, including the PTEs (Cd, Pb, Fe, Mn, Ni, and Cu), was installed downstream of this dam. Therefore, this short communication describes this system's development, installation, operation, and main advantage over conventional methods.

Keywords: iron ore dam; water quality; potentially toxic elements; Amazonia; Serra dos Carajás



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1. The Gelado Project in Carajás

Iron ore production operations in Carajás, southeast of the Amazon, began in 1985 with the completion of the Igarapé Gelado Dam (IGD) to dispose of tailings produced by ore processing and recirculating water for the industrial plant (Figure 1). The dam was raised in 1995 and 2002 to guarantee more space for the accommodation of tailings and to ensure the expansion of production in Carajás. Nowadays, it is measuring 28 m high, 13.5 km² of surface area (where 4.5 km² of water and 9 km² of tailings) and it presents a volume of 142 million m³ (water plus tailings). Possibility studies of reusing waste began to be developed in 2008, which results have already seen the production of pellet feed with a content of 63% Fe [1]. The Gelado Project consists of dredging the tailings deposited over the years at the IGD and processing them at the ore processing plant. At the dam, sediment containment barriers are used to ensure the maintenance of the water supply in quantity and quality for the area downstream of Igarapé Gelado. The ore is subjected to a magnetic concentration process at the processing plant that separates ferrous particles from other impurities. The final product is a pellet feed that will nurture the company's pelletizing plant in São Luís (MA) city. These studies demonstrate that 14.2 million tons per year of tailings from the Gelado Dam will be recovered to produce 10.6 million tons per year of pellet feed [2]. The Brazilian Government granted the Installation License in 2010, but it only effectively started in 2018. The expected operating time is ten years. However,

this control system is located upstream of traditional communities that use the natural resources provided by the Igarapé Gelado, and any error resulting from the activities of dredging the tailings could have a profound impact on the waters 'quality.

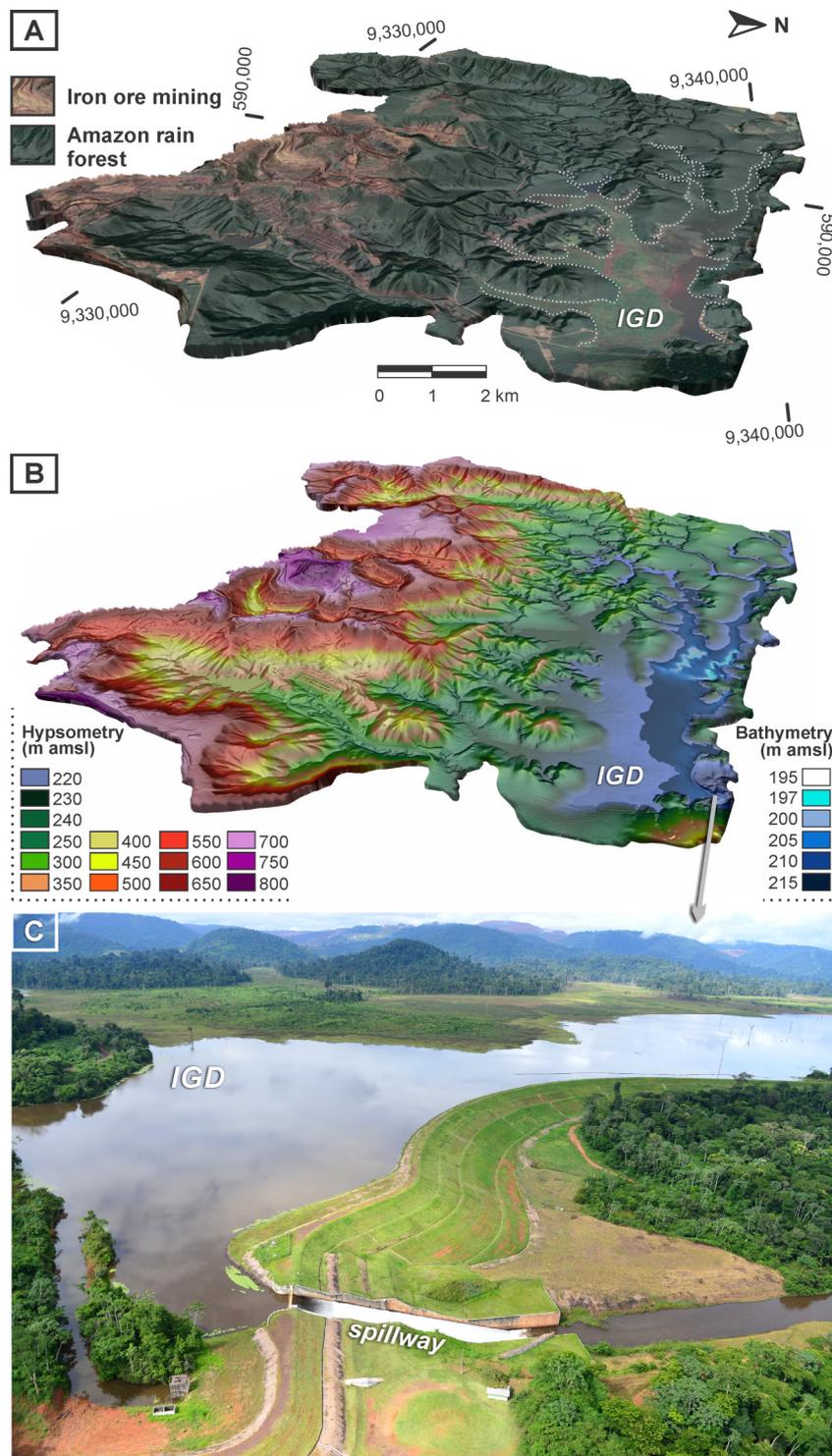


Figure 1. Igarapé Gelado Dam (IGD). (A) Iron ore mining and Amazon rain forest. (B) Hypsometry and bathymetry map. (C) Area, surroundings and lake of the Gelado dam in Carajás, Eastern Amazon.

The ruptures of the Mariana and Brumadinho iron tailings dam in 2015 and 2019, respectively, are considered the worst disasters related to mining activities in the world. Mariana's disaster released an estimated volume of 34 million m³ of tailings, resulting an

intense destruction in several villages close to the downstream of the mining company and several other impacts that extended for 650 km. Brumadinho's disaster resulted in a volume of tailings lower than Mariana in a total of 270 victims. These accidents reinforce the urgent need for measures to assess the environmental impacts caused by activities with tailings dams [3].

2. The System for Sampling and Automatic Analysis of IGD's Water Quality (IGD-WQ)

2.1. Process Analyzers

The IGD-WQ is installed after the dam spillway and hosts the online chemical analyzers PROCESS IC TWO (ion chromatography), ADI 2045VA (voltammetry) and 2045TI (colorimetry) manufactured in Schiedam, Netherlands, as well as oximeters, turbidimeters and electrical conductivity meters. The water is collected automatically every two hours by a pump submerged within the IGD. After the collection, the sample enters the IGD-WQ. It is analyzed by three analyzers that send the data to the ECU (Unit Control Electronic) manufactured in Taipei, Market by Advantech in Brazil and are compiled in open-source software, which is then accessed via internet by the control and monitoring room (Figure 2).

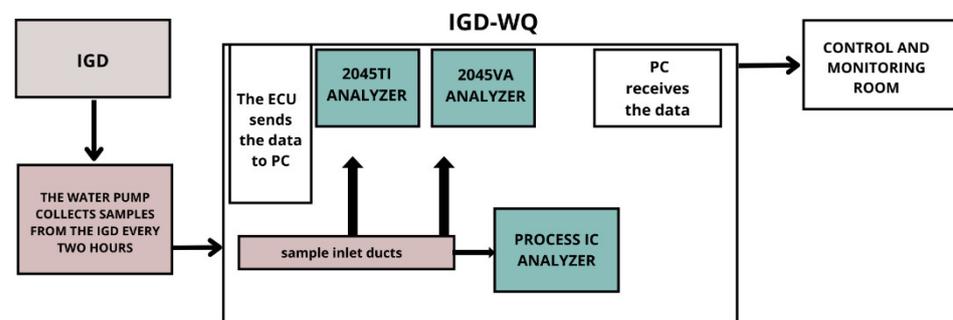


Figure 2. Schematic diagram of the IGD-WQ project: water is collected every two hours, the sample is analyzed by the system, data is collected by ECU (Unit Control Electronic) and transmitted to a control and monitoring room.

The PROCESS IC TWO: it is equipped with two chromatographic columns, one for cation analysis (Metrosep C supp 2 250/4.0, composed of polystyrene-divinylbenzene copolymer with carboxyl groups) and another for anion analysis (Metrosep A supp 5-250/4.0), in addition to a guard column (Metrosep C Supp 2 Guard/4.0) and three detectors; two conductivity and one visible (UV/VIS). It has an Industrial Computer with the software MagIC Net 3.2 and PROCESS LAB Manager version 2.8.6064 and a touchscreen monitor for viewing results [4]. Process IC Two is programmed to perform automatic water collection, filters (0.45 μm) and analysis of 20 chemical parameters: Dissolved Cadmium (Cd^{2+}), Dissolved Lead (Pb^{2+}), Dissolved Iron (Fe^{2+}), Dissolved Manganese (Mn^{2+}), Dissolved Nickel (Ni^{2+}), Ammonium (NH_4^+), Dissolved Copper (Cu^{2+}), Fluoride (F^-), Nitrate (NO_3^-), Nitrite (NO_2^-), Phosphate (PO_4^{2-}), Chloride (Cl^-), Dissolved Sulphate (SO_4^{2-}), Dissolved Magnesium (Mg^{2+}), Dissolved Potassium (K^+), Dissolved Sodium (Na^+), Calcium (Ca^{2+}), Dissolved Cobalt (Co^{2+}), Dissolved Lithium (Li^+) (Table 1).

The ADI 2045VA: this is a classical voltammetry instrumentation composed of a working electrode (WE), whose potential will vary according to a reference electrode (RE), and a third electrode called counter electrode (CE). The measured signal to determine the analyte concentration is the variable potential excitation expressed in the WE in an electrochemical cell. The voltammetric excitation signal is the linear scan; in this case, the voltage applied to the cell linearly increases according to time. The voltammetry applied to the 2045VA analyzer for analysis of Total Hg in IGD's water is square wave voltammetry, a type of pulse voltammetry [5]. In pulse voltammetry, currents are measured at various times during the lifetime of the pulses. Square wave voltammetry offers the advantages of high speed and high sensitivity. The instrument consists of a wet part where the devices

responsible for the analysis flow are inserted (Dosinos, Peristaltic Pumps, Injector, Peek Valve and Digestor), a touchscreen monitor and a reagent area. It can have three working electrodes: the Multi-Mode Electrode Pro (MME), the Rotating Disk Electrodes (RDE) and the scTrace Gold Sensor. The latter is the most suitable for detecting Mercury [5], precisely is the one in operation at IGD-WQ.

Table 1. Parameters and Limits of Detection (LOD).

Parameters	Limits of Detection (mg/L)
Dissolved Copper (Cu)	0.01
Dissolved Nickel (Ni)	0.003
Dissolved Iron (Fe)	0.04
Dissolved Cobalt (Co)	0.003
Dissolved Manganese (Mn)	0.19
Dissolved Cadmium (Cd)	0.009
Dissolved Lead (Pb)	0.006
Dissolved Lithium (Li)	0.06
Dissolved Sodium (Na)	0.04
Dissolved Ammonium (NH ₄ ⁺)	0.18
Dissolved Potassium (K)	0.05
Dissolved Magnesium (Mg)	0.32
Dissolved Calcium (Ca)	0.06
Fluoride (F ⁻)	0.02
Chloride (Cl ⁻)	0.26
Nitrite (NO ₂ ⁻)	0.21
Phosphate (PO ₄ ⁻)	0.006
Sulfate (SO ₄ ²⁻)	1.63
Total Aluminum (Al ³⁺)	0.0001
Total Phosphorus (P)	0.0001

The 2045TI: it works to determine total Al³⁺, total P using colorimetry and pH using potentiometry. Colorimetry is a spectro-analytical technique that involves determining analytes based on a sample's color characteristics (transparency or saturation) [6]. The colorimetric analysis basis consists the variation of color's intensity system with changes in one of the components' concentrations, which can be divided into visual or photometric colorimetry. The technique applied to the 2045TI is photometric colorimetry. According to APHA [7], the determination of phosphorus by photometric colorimetry has been used for decades in laboratories specialized in water quality control worldwide. The method uses acid digestion and conversion of phosphorus into a chromophore compound. In the 2045TI analyzer the reaction occurs similarly to the bench method. However, the automation of the process analyzer makes it more agile. The procedures occur as follows: Ammonium Molybdate Potassium and Antimony Tartrate react in an acid medium with orthophosphate (an organic form of phosphorus present in the sample) to form phosphomolybdic acid, which is reduced to molybdenum blue (chromophore compound) by acid ascorbic. Possible interferences in the reaction are: As (0.1 mg/L); Cr⁶⁺ and NO₂⁻ (3–15% interference); Na₂S and Silicate (between 1 and 10 mg/L) [8]. The determination of aluminum occurs in the same analyzer using the Eriochromocyanine R method. The analyte reacts with Eriochromocyanine to form a reddish-colored complex (chromophore). The two analyses occur in a consecutive cycle that lasts an average of 40 min per sample.

A submersible water pump carries out the water sampling from the river. Through ducts, the sample enters the chemical analyzers to conduct the analyses and determine the levels identified in that sample (Figure 2).

2.2. Software for Communication between Analyzers

The software will be installed to communicate between the analyzers and send the data to a free database software. It will be responsible for storing all the results obtained from all analyzers mentioned in this document, each one in the respective database. The

configuration of the communication parameters will be done directly in this software. The ECU (Electronic Control Unit) device will receive all signals via Modbus TCP from Process IC and 2045TI, or converted from analog to digital from 2045VA and pH, conductivity and dissolved oxygen sensors. The analog/digital converter is necessary because the ECU device only receives digital signals. Remote access to the results will be possible, as the ECU device can connect to an internet signal available in situ.

2.3. Laboratory Support

To facilitate maintenance, cleaning, preparation, reagent exchange, and calibration of the various sensors, crucial for the proper functioning and quality assurance of the analyses within the three implemented systems, a team of three chemistry technicians was contacted. This team was sourced from a specialized and accredited local laboratory compliant with legislation [9], called LIMNOS.

3. Problems during System Operation

Some technical and analytical problems were identified in the operation of the analyzers. The ADI 2045VA was delayed due to the very rapid degradation of a working electrode component. Some measures were taken to solve this problem, such as the installing of a vacuum pump for filtration in the electrolytic cell to remove the steam generated by the electrolytic solution during the analysis, since the generated vapors would interact with the electrode causing its oxidation. In addition, a new analysis method is being developed with the testing of less aggressive reagents.

In the ADI 2045TI analyzer, incorrect pH results were observed due to the resting solution of the electrode containing sulfuric acid, which would directly interfere the pH measurements. Additionally, incorrect results for Total Phosphorus and Aluminum were noted, culminating in the inadequacy of the calibration curve and the analysis method initially proposed for application. The solutions found were a review of the procedure and removal of sulfuric acid from the composition, resting with the sample itself and redefining the working range by the analytical curve.

4. First Results and Quality Control

4.1. Calibration of Analyzers

Table 2 shows the correlation coefficients (R^2) obtained after the first calibration of the parameters measured by PROCESS IC. Next, Table 3 presents the first results obtained after the calibration process. These preliminary findings will provide valuable insights into the capabilities and potential improvements of the online monitoring system, paving the way for further refinement and optimization, resulting in a more reliable and accurate water quality assessment. The analytes Cu, Ni, Co, Cd, Li, Fluoride (F^-), Nitrate (NO_3^-), Nitrite (NO_2^-) by chromatography, Al^{3+} and Total Phosphorus by photometry showed measurements below the limits of detection (LD) (Table 3). All analyzed parameters are in accordance with the legislation applicable 430 CONAMA resolution to effluents [10]. It is important to emphasize that the monitoring of water resources in the region is already carried out by a conventional commercial laboratory accredited to the ISO/IEC 17025 standard called "SGS-GEOSOL". This laboratory conducts periodic collections in the area and uses validated analytical methods explicitly developed to comply with local legislation regarding water quality. Laboratory analyzes found the following concentrations in mg/L at the same sampling point as IGD-WQ during the rainy season: Fe < 0.1, Co < 0.001, Mn = 0.36, Cd > 0.0003, Li > 0.001, Na = 1.01, Ca = 3.31, F^- < 0.05, Cl^- = 1.4, NO_3^- = 0.26.

Table 2. Correlation coefficient (R^2) and type of function used to calibrate the parameters measured by the IGD-WQ.

Parameter	R^2	Curve Type
Cu	0.99947	Quadratic throughout 0
Ni	0.99957	
Co	0.99999	
Mn	0.99670	
Cd	0.99827	
Pb	0.99506	
Li	0.99591	
Na	0.99721	
Ca	0.99637	
F ⁻	0.99991	
Cl ⁻	0.99963	
NO ²⁻	0.99986	
NO ³⁻	0.99996	
Fe	0.99870	Linear
Al ³⁺	0.99450	
P	0.99958	

Table 3. Descriptive statistics of measured parameters with numbers of samples (N), mean, standard deviation (SD), minimum (Min.), maximum (Max.), median, limits of detection (LD) and limits allowed by legislations 430 CONAMA (effluents [10]) and 357 CONAMA (class II surface water [11]).

Parameters	N	mg/L						CONAMA 430	CONAMA 357 (Class II)
		LD	Mean	SD	Min.	Median	Max.		
Cu	15	0.01	<LD	-	-	-	-	1	0.009
Ni	15	0.003	<LD	-	-	-	-	2	0.02
Fe	15	0.15	0.23	0.029	0.199	0.250	0.273	15	0.3
Co	15	0.003	<LD	-	-	-	-	-	0.05
Mn	15	0.19	0.02	0.012	0.002	0.015	0.035	-	0.1
Cd	15	0.009	<LD	-	-	-	-	0.2	0.001
Li	15	0.06	<LD	-	-	-	-	-	2.5
Na	15	0.04	1.03	0.058	0.963	1.02	1.141	-	-
Ca	15	0.06	6.92	0.131	6.724	6.949	7.09	-	-
F ⁻	15	0.02	<LD	-	-	-	-	10	1.4
Cl ⁻	15	0.26	2.03	0.234	1.638	1.952	2.49	-	250
NO ²⁻	15	0.21	<LD	-	-	-	-	-	1
NO ³⁻	15	0.19	<LD	-	-	-	-	-	10
Al ³⁺	14	0.0001	<LD	-	-	-	-	-	0.1
P	14	0.0001	<LD	-	-	-	-	-	0.1

The LD for Al³⁺ and P were calculated based on the simple estimation method from the analytical curve described by INMETRO [11] expressed by Equation (1). This method uses the standard deviation of the blank replicas of the analytical curve and its slope to determine LD:

$$LD = 3.3 \times S/s \quad (1)$$

Being,

S: blank standard deviation;

s: angular coefficient of the analytical curve.

The cations and anions determined, by chromatography LD (Table 1), were estimated based on the sample blank method with the addition of the lowest analyte acceptable contraction defined by INMETRO [12]. The method involves analyzing 7 aliquots, resulting in 6 degrees of freedom for a sample blank matrix with an added lowest acceptable analyte concentration. With these degrees of freedom, the one-sided t-value for a 99% confidence level is 3.143. The LD is calculated as 3.143 times the standard deviation of the sampling pattern. Equation (2) used in this calculation is as follows:

$$LD = 0 + t.s \quad (2)$$

Being,

t: abscissa of Student's distribution, dependent on sample size and confidence level;

s: sample standard deviation of sample blanks with addition.

For 2045TI analyzer quality control, a test was carried out in duplicate with the addition of 1 mg/L of P and Al^{3+} to the sample blank (deionized water), obtaining concentrations of 0.99 (99%), 0.98 mg/L (98%), 1.02 mg/L (102%) and 1.03 mg/L (103%), respectively. These results confirm good recovery of the investigated analytes. For PROCESS IC, during the standard verification test the following recoveries were obtained after 7 repetitions: Cu (80%), Ni (100%), Co (99%), Mn (97%), Pb (104%), Li (83%), Na (93%), NH_4^+ (91%), K (107%), Mg (105%), Ca (105%), F^- (98%), Cl^- (83%), NO_2^- (143%), NO_3^- (105%), PO_4^- (113%), SO_4^{2-} (103%). The elements Fe and Cd showed recovery below expectations; therefore, new tests and methods' adaptations are planned.

4.2. Key Advantages of an Online Water Quality Monitoring

Maintaining an online water quality analysis system offers several significant advantages. Firstly, real-time monitoring provides timely and accurate data on various water parameters, enabling prompt detection of deviations from the desired water quality standards [13,14]. This immediate feedback allows swift and targeted responses to potential issues, ensuring timely interventions to maintain water quality at desired levels. Additionally, continuous online monitoring significantly reduces the need for manual sampling and laboratory testing, saving time and resources. Automated monitoring systems can operate 24/7, providing a comprehensive and uninterrupted view of water quality, which is essential for industries, municipalities and environmental agencies to take action based on measured data and take proactive measures to safeguard public health and the environment [15].

Furthermore, this system allows long-term data collection and trend analysis, providing insights into seasonal and long-term variations in water quality. Groups of researchers and technicians around the world have been dedicated to implementing online water quality monitoring systems. That is the case of a system implemented in Malaysia which utilizes the Internet of Things, low-cost sensors and 3D printing to monitor water quality in near real-time [16]. These historical data can assist in the development of effective water management strategies and policies. The ability to access and analyze these historical data supports evidence-based decision-making and proactive planning for sustainable water resource management, ultimately contributing to preserving and protecting precious water sources [17].

5. Conclusions

The challenges faced from the installation to the system's operation were considerable, primarily due to the remote location of the IG in the Eastern Amazon. Furthermore, the proximity to a tailings dam increases complexity, requiring additional safety measures and meticulous planning to prevent environmental risks and ensure the system's operational safety. Despite the obstacles, the potential benefits for the region and the company will be remarkable when the analytical system is fully functional. Access to near real-time water

quality data and analysis in the Amazon can accelerate decision-making, especially in the environmental and sustainability context. These can improve management practices and strategies to preserve the local ecosystem.

The analytical system can potentially monitor, almost in real-time, the concentration of potentially toxic elements (PTEs) with analytical routines of quality control. The IGD-WQ aims to ensure good water quality and maintain the population's health around the dam, following the legal norms and limits established by the Brazilian National Council for the Environment (CONAMA).

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