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Operationally stable pH measurement in wastewater treatment using a photocatalytic self-cleaning electrode: Extended field evidence and uncertainty-oriented performance indicators

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ABSTRACT
This study presents a 29-day field deployment of a HORIBA photocatalytic self-cleaning pH electrode for continuous wastewater monitoring in a full-scale treatment facility discharging into Laguna Lake, Philippines. The system generated 41,729 valid one-minute pH measurements between 11 December 2025 and 9 January 2026 with near-continuous uptime. The work focuses on uncertainty-oriented operational indicators derived from grouped observations, rather than on a complete formal Guide to the Expression of Uncertainty in Measurement (GUM) uncertainty budget. Statistical indicators included agreement with laboratory reference measurements, rolling median absolute deviation (MAD), grouped daily statistics, trend analysis, and distributional behavior. Laboratory reference measurements were obtained using a benchtop HORIBA pH meter, calibrated with buffer standards traceable to the National Institute of Standards and Technology (NIST). The results indicated low short-term variability, operationally stable variance behavior, preserved sensitivity to transient process events, and no evidence of uncontrolled sensor degradation during deployment. Type A and Type B uncertainty contributions were discussed qualitatively within a grouped-observation framework consistent with JCGM 100:2008 Appendix H.5. The observed temporal behavior was more consistent with gradual process evolution than with fouling-induced sensor instability. Future investigations include deployment in multiple scenarios for inter-electrode reproducibility, parallel experiments with controlled environments, and extended and multi-season temporal studies.

Section: RESEARCH PAPER

Keywords: self-cleaning pH electrode; wastewater monitoring; grouped observations; uncertainty-oriented analysis; photocatalytic TiO_2; continuous environmental sensing

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1. INTRODUCTION

Recent advances in wastewater monitoring have accelerated the transition from periodic grab sampling to high-frequency real-time sensing systems integrated into supervisory and Internet of Things (IoT)-based process architectures [1]–[5]. Continuous monitoring is increasingly important for process optimization, regulatory compliance, anomaly detection, and predictive process control [1]–[4]. Among routinely monitored variables, pH remains one of the most operationally important because it directly affects coagulation efficiency, nutrient removal, microbial activity, corrosion control, and disinfection performance [5], [6].

Long-term electrochemical pH monitoring in wastewater environments remains difficult because of membrane fouling, scaling, organic deposition, and biological film formation [7]–[12]. Several studies have shown that conventional ion-selective pH electrodes often experience drift, signal attenuation, increased variance, and maintenance-intensive recalibration during extended deployment [7], [8], [10], [12]. These limitations have motivated research into antifouling and self-cleaning sensing architectures.

Photocatalytic titanium dioxide (TiO_2)-based self-cleaning surfaces have recently emerged as promising approaches for improving operational sensor stability. Qi et al. reviewed anti-fouling membrane strategies for ion-selective electrodes and emphasized the importance of surface engineering for long-term environmental sensing [8]. Liu et al. subsequently demonstrated TiO_2-coated ion-selective electrodes with photocatalytic self-cleaning properties capable of reducing fouling accumulation and improving electrochemical stability in contaminated aqueous systems [11].

From a metrological perspective, continuous environmental monitoring introduces uncertainty challenges that differ from conventional laboratory measurements. In long-duration deployments, uncertainty becomes time-dependent because measurements are affected by calibration stability, sensor ageing, temporal correlation, environmental variability, and signal-processing choices [13]–[17]. Harris et al. emphasized that uncertainty evaluation for sensor-network metrology should explicitly consider temporal behaviour and correlated observations [15]. Razumić et al. likewise argued that modern measurement-system quality assessment must incorporate drift, repeatability, stability, and operational capability rather than relying solely on static calibration uncertainty [14].

Although the Guide to the Expression of Uncertainty in Measurement (GUM) remains the principal framework for uncertainty evaluation [12], continuous wastewater monitoring datasets are often more appropriately treated as grouped repeated observations rather than classical static measurements. However, an alternative reference employed by researchers that focus on grouped time-series observations is the Joint Committee for Guides in Metrology (JCGM) 100:2008 Appendix H.5 [12], [13].

The present paper reports a nearly one-month deployment of a HORIBA photocatalytic self-cleaning pH electrode, installed in a wastewater treatment plant discharging into Laguna Lake, Philippines. The work contributes to the literature by:

1. providing extended field evidence under realistic fouling conditions;
2. applying grouped-observation analysis to high-frequency pH data;
3. distinguishing between process evolution and possible sensor-related instability; and
4. interpreting measurement stability from both metrological and operations-management perspectives.

2. RELATED WORK

Recent wastewater-monitoring literature has emphasized the importance of reliable real-time sensing systems for operational control and environmental compliance [1]–[6]. Moretti et al. reviewed the feasibility of real-time wastewater characterization technologies and identified sensor reliability and maintenance burden as major barriers to large-scale deployment [1]. Marino et al. demonstrated that high-frequency fluorescence monitoring can support process optimization and contaminant tracking when measurement quality remains stable during continuous operation [2].

IoT-enabled monitoring systems are increasingly integrated into treatment facilities to support predictive maintenance, anomaly detection, and automated decision-making [3], [17]. Forhad et al. demonstrated cloud-connected water-quality monitoring architectures capable of continuous pH and dissolved oxygen monitoring [3]. Essamlali et al. further emphasized that machine-learning applications in water quality monitoring depend critically on sensor stability and trustworthy measurement quality [17].

Electrochemical sensing remains one of the dominant approaches for continuous environmental monitoring because of its compatibility with distributed low-power instrumentation [4], [6], [9], [18]. However, fouling remains one of the most significant operational limitations [7]–[12]. Delgado et al. reviewed antifouling approaches for water quality sensors, and concluded that biofilm accumulation and scaling are major contributors to drift and uncertainty growth [7]. Ohmura et al. characterized long-term wear and degradation of ion-selective pH sensors, and reported progressive changes in stability and electrochemical response during prolonged deployment [10].

Recent studies have therefore focused on anti-fouling and self-cleaning sensor architectures. Qi et al. reviewed polymeric membrane anti-fouling strategies for ion-selective electrodes [8], while Liu et al. demonstrated photocatalytic TiO_2-coated ion-selective electrodes capable of reducing surface contamination under ultraviolet illumination [11].

Parallel developments in metrology and measurement science have emphasized uncertainty evaluation in distributed and time-dependent sensor systems [12]–[16]. Harris et al. proposed uncertainty evaluation methods for sensor-network metrology that explicitly consider correlation structures and time-dependent behaviour [15]. Kok and van Dijk discussed differences between standardized uncertainty approaches and data-driven uncertainty interpretation in modern measurement systems [19]. Robust statistical approaches for non-Gaussian sensor systems have likewise gained attention in environmental sensing applications [20].

3. EXPERIMENTAL SETUP AND MEASUREMENT PROTOCOL

3.1. Wastewater treatment site

The experiment was conducted at a full-scale wastewater treatment facility discharging into Laguna Lake, Philippines. For confidentiality purposes, the name of the participating establishment is not disclosed. The monitored stream exhibited variable hydraulic loading and mixed domestic–commercial wastewater characteristics.

3.2. HORIBA self-cleaning electrode

The field system used an industrial HORIBA self-cleaning glass-membrane pH electrode, equipped with a photocatalytic TiO_2 cleaning mechanism activated by embedded ultraviolet illumination. No external mechanical cleaning devices or chemical cleaning loops were installed during the deployment.

The study used a single-field electrode because the objective was to evaluate operational stability during continuous deployment, rather than inter-electrode reproducibility. This limitation is acknowledged explicitly. However, the dataset contains more than 41,000 repeated observations across varying process conditions, allowing detailed time-dependent analysis of repeatability and drift-related indicators.

A separate, parallel pure-water experiment was not conducted because the deployment objective was evaluation under realistic wastewater conditions. Nevertheless, the interpretation of the results was constrained carefully to avoid attributing all observed changes solely to the self-cleaning mechanism. The observed trends are interpreted as the combined effects of process evolution and sensor behaviour.

3.3. Reference measurements and calibration

A laboratory benchtop HORIBA pH meter was used as the reference instrument for periodic validation measurements. Prior to each measurement session, the meter was calibrated using commercially manufactured HORIBA-certified pH standard solutions with certified values of pH 4.01, 6.86, and 10.02 at 25 °C, traceable to the National Institute of Standards and Technology (NIST).

A total of 18 laboratory reference measurements were obtained during the deployment period. Grab samples were collected manually from the same hydraulic location as the field probe. Samples were analysed within approximately 10 minutes of collection to minimize chemical change.

For comparison with the field sensor, the corresponding one-minute field measurement nearest to the grab sample timestamp was extracted. When short-term fluctuations exceeded ± 0.03 pH within a 5-minute interval around the sample time, a centred 5-minute median was used instead of a single-point comparison.

3.4. Data acquisition

The field sensor logged pH values at one-minute intervals from 11 December 2025 to 9 January 2026. The final dataset contained 41,729 valid records after filtering invalid status flags.

The acquisition chain consisted of:

- the HORIBA self-cleaning pH electrode,
- embedded signal conditioning electronics,
- a digital data logger,
- timestamp synchronization hardware, and
- a supervisory database for offline analysis.

No online filtering or smoothing was applied during acquisition.

4. DATA PROCESSING AND UNCERTAINTY-ORIENTED ANALYSIS

4.1. Scope of the uncertainty analysis

The present work does not claim to provide a complete formal dynamic uncertainty evaluation in the strict sense of GUM-based dynamic measurement theory. Instead, the study evaluates grouped observations collected continuously over time and derives uncertainty-oriented indicators relevant to operational monitoring.

The approach follows the grouped observation treatment discussed in Appendix H.5 of JCGM 100:2008, where repeated observations acquired under comparable conditions are analysed statistically [12].

The measured pH value $y(t)$ can be represented conceptually as:

$$y(t) = x(t) + b(t) + \varepsilon(t), \tag{1}$$

where:

- $x(t)$ is the true process pH,
- $b(t)$ represents systematic effects including calibration offset and slow drift,
- $\varepsilon(t)$ represents random effects.

The study focuses on the behaviour of:

- agreement with laboratory references,
- short-term variability,
- long-term stability,
- variance consistency across grouped observations, and
- preservation of process information.

4.2. Grouped observation statistics

The one-minute observations were grouped into daily datasets. For each group, the following statistics were calculated:

$$\bar{x}_\mathrm{i} = \frac{1}{n_\mathrm{i}}\sum_{j=1}^{n_\mathrm{i}} x_\mathrm{ij}, \tag{2}$$

$$s_\mathrm{i} = \sqrt{\frac{1}{n_\mathrm{i}-1}\sum_{j=1}^{n_i}(x_\mathrm{ij} - \bar{x}_\mathrm{i})^2}, \tag{3}$$

where:

- x_ij is the j-th observation in group i,
- n_i is the number of observations in group i,
- s_i is the within-group standard deviation.

As wastewater pH data exhibited non-Gaussian behavior and occasional extreme events, robust statistics were also evaluated using the median absolute deviation (MAD):

$$MAD = \mathrm{median}(|x_\mathrm{i} - \mathrm{median}(x)|). \tag{4}$$

4.3. Variance consistency across groups

To assess whether within-group dispersion could reasonably be compared across days, variance consistency was examined using a robust comparison of daily MAD and standard deviation values.

Although strict homoscedasticity was not observed because of process variability, no progressive inflation of variance over time was detected. Consequently, the grouped observations were considered sufficiently stable for comparative trend interpretation. As the wastewater process itself was nonstationary and strongly influenced by operational disturbances, strict variance homogeneity was not expected. Instead of formal pooled variance estimation for inferential statistics, the grouped observation framework was used primarily for comparative operational interpretation. The absence of progressive variance inflation across consecutive groups was considered sufficient to support the qualitative comparison of repeatability-related indicators over time. Consequently, the grouped statistics were interpreted operationally, rather than as the evidence of strict statistical stationarity. Formal inferential homogeneity testing was not emphasized because the primary objective was operational trend interpretation under nonstationary wastewater conditions.

4.4. Temporal correlation and effective observations

The one-minute measurements are temporally correlated and therefore do not represent fully independent observations. To avoid overstating statistical confidence, the interpretation was based primarily on grouped daily statistics and rolling-window indicators, rather than on the nominal sample size alone.

The temporal correlation of one-minute measurements implies that consecutive observations cannot be treated as fully independent samples. Consequently, the effective number of statistically independent observations is smaller than the nominal dataset size. The grouped observation framework adopted in this work therefore reduces the risk of overstating statistical confidence while preserving operational trend information. This treatment is consistent with modern sensor-network metrology recommendations for correlated environmental data streams.

4.5. Type A and Type B contributions

The uncertainty-oriented interpretation considered both Type A and Type B contributions.

Type A Contributions

Type A indicators were derived statistically from repeated observations and included:

- within-group standard deviation,
- rolling MAD,
- reference-agreement dispersion,
- day-to-day variability.

Type B Contributions

Indicative Type B contributions included:

- manufacturer-specified electrode accuracy,
- calibration-buffer uncertainty,
- analogue-to-digital conversion resolution,
- laboratory reference meter specification,
- temperature-compensation uncertainty.

As complete manufacturer internal design specifications were unavailable, the Type B analysis is presented qualitatively and semi-quantitatively, rather than as a complete traceable uncertainty budget.

4.6. Indicative combined uncertainty

An indicative combined standard uncertainty was estimated conceptually using:

$$u_{\mathrm{c}} = \sqrt{u_{\mathrm{A}}^2 + u_{\mathrm{B}}^2}, \tag{5}$$

where:

- u_{A} represents grouped Type A contributions,
- u_{B} represents aggregated Type B contributions.

The resulting expanded uncertainty may be expressed as:

$$U = k \cdot u_{\mathrm{c}}, \tag{6}$$

using coverage factor $k = 2$ for approximate 95 % coverage.

The manuscript emphasizes that this estimate is illustrative and it intended to support operational interpretation, rather than formal conformity assessment.

Table 1. Global statistics of HORIBA pH time series (one-minute data).

Metric	Value
Records (n)	41,729
Start (local)	2025-12-11 16:02
End (local)	2026-01-09 15:30
Duration (days)	28.9778
Mean pH	6.48
Median pH	6.36
Std. deviation (pH)	0.357
MAD (pH)	0.130
Minimum pH	5.89
Maximum pH	11.73
Skewness	2.83
Excess kurtosis	20.95

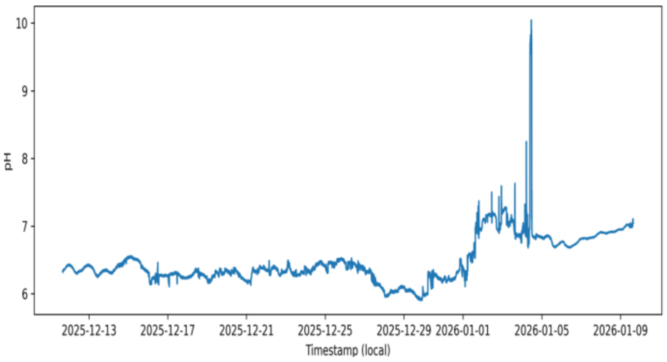

Figure 1. Five-minute median pH time series from the HORIBA self-cleaning electrode over the full deployment.

5. RESULTS

5.1. Dataset overview

The final dataset contained 41,729 valid one-minute pH observations, covering approximately 28.98 days. The global statistics are summarized in Table 1.

The dataset exhibited non-Gaussian behaviour with occasional extreme events (Figure 1). Most observations remained between pH 6.0 and 7.1.

The difference between the standard deviation (0.357 pH) and MAD (0.130 pH) indicates that the dataset contains transient extreme events and non-Gaussian tails. Because standard deviation is sensitive to extreme observations, its larger magnitude reflects occasional process excursions observed during wastewater operation. In contrast, the lower MAD value indicates that the majority of measurements remained tightly clustered around the median. This supports the interpretation that the sensor maintained stable short-term repeatability despite the presence of intermittent process disturbances.

The positive skewness and high excess kurtosis confirm that the pH distribution was strongly non-Gaussian, with infrequent but pronounced transient events. Such behaviour is expected in operational wastewater systems, where hydraulic fluctuations, intermittent chemical loading, and short-duration process disturbances can produce abrupt pH excursions. The presence of heavy tails further justifies the use of robust statistical indicators, such as MAD, instead of relying exclusively on Gaussian assumptions.

5.2. Reference agreement

The 18 laboratory reference comparisons showed mean absolute differences below 0.08 pH for most observation periods (Figure 2).

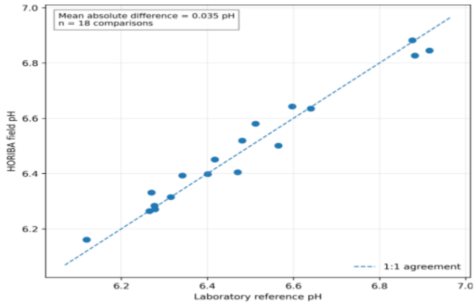

Figure 2. Comparison between laboratory reference measurements and corresponding HORIBA field measurements, showing agreement within operational wastewater tolerances.

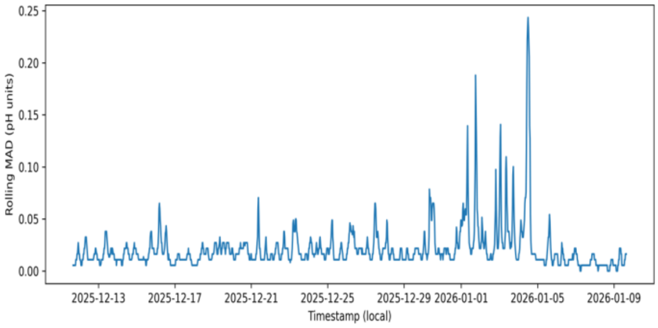

Figure 3. Rolling 60-minute median absolute deviation (MAD) of pH over the deployment.

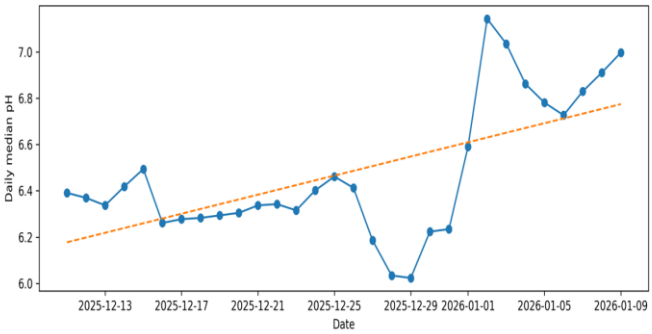

Figure 4. Daily median pH values with fitted linear trend over the deployment.

The laboratory reference comparisons provide additional evidence of operational stability. Mean absolute differences below 0.08 pH are within typical industrial process-monitoring tolerances for wastewater applications. More importantly, no monotonic increase in disagreement was observed over time. If progressive fouling or membrane degradation had occurred, increasing offset and widening dispersion relative to the laboratory reference would be expected. This behaviour was not observed in the present dataset.

5.3. Short-term variability

Rolling 60-minute MAD values remained low throughout the deployment, typically within a few hundredths of a pH unit (Figure 3).

Short-duration increases in MAD coincided with periods of increased process variability rather than sustained instability. Importantly, the dispersion indicators did not increase systematically over time.

The rolling MAD analysis demonstrates that local short-term repeatability remained operationally stable throughout the deployment period. Since MAD is less sensitive to isolated outliers than variance-based indicators, the consistently low MAD values indicate that the majority of one-minute observations remained internally consistent even during periods containing transient excursions.

5.4. Long-term stability

Daily median pH values exhibited a gradual monotonic increase during the deployment (Table 2).

The grouped daily statistics demonstrate that within-group dispersion remained operationally stable throughout the deployment period. Daily standard deviations generally remained below 0.08 pH despite evolving process conditions, indicating that measurement variability did not increase progressively over time. Likewise, MAD values remained relatively small and consistent across observation groups, suggesting that the electrode maintained stable local repeatability characteristics. The absence of systematic variance inflation supports the interpretation that fouling-related degradation remained limited during deployment.

Table 2. Daily statistics of pH (one-minute data; excerpt)

Date	N	Mean	Median	Std	Min	Max	MAD
2025-12-11	478	6.39	6.39	0.031	6.32	6.43	0.027
2025-12-12	1440	6.37	6.37	0.042	6.29	6.44	0.038
2025-12-13	1440	6.33	6.34	0.046	6.24	6.43	0.032
2025-12-14	1440	6.41	6.42	0.073	6.29	6.56	0.065
2025-12-15	1440	6.46	6.49	0.079	5.92	6.57	0.043
…	…	…	…	…	…	…	…
2026-01-05	1440	6.78	6.78	0.060	6.68	6.87	0.060
2026-01-06	1440	6.74	6.73	0.037	6.68	6.81	0.032
2026-01-07	1440	6.84	6.83	0.030	6.80	6.91	0.011
2026-01-08	1440	6.92	6.91	0.017	6.88	6.95	0.005
2026-01-09	931	7.00	7.00	0.033	6.95	7.29	0.022

The trend was smooth and approximately linear, with no abrupt offsets or instability episodes (Figure 4). The observed behaviour is more consistent with gradual process evolution than uncontrolled sensor drift.

5.5. Distributional behaviour

The pH distribution retained visible tails and transient extreme values throughout the monitoring period (Figure 5).

No evidence of progressive distribution compression or signal flattening was observed. This indicates the preservation of sensitivity to short-duration process excursions.

6. DISCUSSION

The present study evaluates uncertainty-oriented operational indicators, rather than formal dynamic measurement uncertainty.

The grouped observation approach is appropriate because the dataset consists of repeated observations acquired over time under continuously evolving process conditions. The use of robust statistics, such as MAD, was motivated by the strongly non-Gaussian distribution and the presence of occasional transient events.

The results indicate three important findings.

Firstly, short-term repeatability-like behaviour remained stable throughout the deployment. The absence of progressive

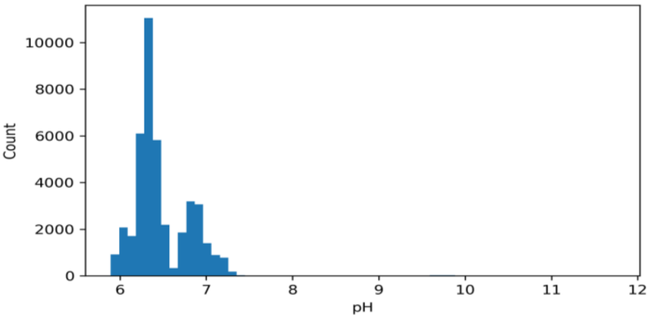

Figure 5. Histogram of one-minute pH measurements for the entire deployment.

variance inflation suggests that the self-cleaning mechanism successfully limited fouling-related degradation.

Secondly, the observed long-term trend appears process-driven, rather than sensor-driven. Conventional fouling-related degradation often manifests as erratic drift, variance growth, or abrupt signal instability [7], [10]. These characteristics were not observed.

Third, the sensor retained sensitivity to transient events and distribution tails. In practical wastewater operations, the preservation of rare-event information is important for anomaly detection and process diagnostics.

The study nevertheless has important limitations.

Only one field electrode was evaluated, and therefore inter-sensor reproducibility was not assessed directly. In addition, no parallel pure-water reference deployment was conducted. Consequently, the present work should be interpreted primarily as a field-stability case study rather than a complete validation of the photocatalytic mechanism in isolation.

Several important research directions emerge from the present study.

Firstly, future work should include the simultaneous deployment of multiple self-cleaning electrodes operating under identical environmental conditions. Such experiments would allow the direct estimation of inter-electrode reproducibility and between-sensor variance components, which could not be evaluated in the present single-electrode deployment.

Secondly, controlled parallel experiments using clean-water and wastewater environments are necessary to isolate the specific contribution of the photocatalytic self-cleaning mechanism from normal process variability. Such experiments would strengthen causal interpretation regarding fouling suppression.

Thirdly, longer multi-season deployments are required because wastewater composition, biological activity, and environmental conditions vary substantially across climatic periods. Extended monitoring would allow the evaluation of long-term ageing, seasonal drift, and maintenance intervals.

Fourthly, future studies should construct a more complete Type B uncertainty budget incorporating traceable calibration uncertainty, electronics characterization, temperature compensation uncertainty, and long-term drift components. This would enable more rigorous conformity-oriented uncertainty evaluation, aligned with formal GUM methodology.

Finally, future work should investigate explicit temporal-correlation modelling and effective degrees-of-freedom estimation for high-frequency environmental sensor networks. Such developments are important for uncertainty propagation in continuously sampled measurement systems, in which observations are strongly autocorrelated.

7. CONCLUSIONS

A HORIBA photocatalytic self-cleaning pH electrode was deployed continuously for approximately one month in a wastewater treatment plant, and produced more than 41,000 valid one-minute measurements.

The analysis demonstrates that:

- short-term variability remained low and stable,
- grouped observation variance remained operationally consistent,
- no evidence of uncontrolled sensor drift was observed,
- sensitivity to transient events was preserved,
- the observed behaviour was more consistent with process evolution than sensor degradation.

The manuscript explicitly clarifies that the work presents uncertainty-oriented performance indicators, rather than a full formal GUM uncertainty budget or strict dynamic uncertainty analysis.

Despite limitations associated with the single-electrode deployment and limited reference checks, the results suggest that photocatalytic self-cleaning pH electrodes can support stable long-term wastewater monitoring with reduced maintenance burden.

The study also demonstrates how grouped-observation analysis, robust statistics, and operational uncertainty indicators can complement conventional calibration-based assessment in continuous environmental sensing applications.

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