Reproducibility and uncertainty of wastewater turbidity measurements

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ABSTRACT

Turbidity monitoring is a valuable tool for operating sewer systems, but it is often considered as a somewhat tricky parameter for assessing water quality, because measured values depend on the model of sensor, and even on the operator. This paper details the main components of the uncertainty in turbidity measurements with a special focus on reproducibility, and provides guidelines for improving the reproducibility of measurements in wastewater relying on proper calibration procedures. Calibration appears to be the main source of uncertainties, and proper procedures must account for uncertainties in standard solutions as well as non linearity of the calibration curve. With such procedures, uncertainty and reproducibility of field measurement can be kept lower than 5% or 25 FAU. On the other hand, reproducibility has no meaning if different measuring principles (attenuation vs. nephelometry) or very different wavelengths are used.

Key words | calibration, reproducibility, turbidity, uncertainty, wastewater

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INTRODUCTION

Continuous *in situ* turbidity measurement in sewers appears as a fairly simple way for monitoring the dynamics of pollutants associated with suspended solids, especially during rainfall events. (Maréchal *et al.* 2001; Henckens *et al.* 2002; Veldkamp *et al.* 2002; Bertrand-Krajewski 2004; Langeveld *et al.* 2005; Aumond & Joannis 2006; Ruban *et al.* 2006). However, turbidity is also considered as a somewhat tricky parameter for assessing water quality, because values measured in a single sample may vary, depending on the model of sensor, and even on the operator.

Moreover turbidity is a parameter related to other water quality parameters, especially suspended solids concentration (SS), but it can hardly be considered as a surrogate measurement method for these parameters, because the relationship between turbidity and SS or COD do vary, especially with rain events. But many applications, e.g. real time control, can use turbidity on its own, or can cope with an average relationship for translating turbidity values into SS or COD values.

Thus improving the reproducibility of turbidity measurements is the key point for promoting a more common application of this type of measurements in sewer systems, and this is the topic of this paper.

Although turbidity is best used as a continuous monitoring tool, this paper will focus mainly on turbidity measurements performed on samples, in the laboratory conditions. One reason for this choice is that reproducibility has no meaning unless a measurand is precisely defined,

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and this is much easier to achieve with samples. Moreover, *in situ* monitoring does involve laboratory work for calibrations. Nevertheless, a few results related to *in situ* monitoring will be also presented.

TURBIDITY, REPRODUCIBILITY AND UNCERTAINTY

Definitions

Turbidity is defined by the ISO 7027 standard for turbidity measurement (NF EN ISO 7027 2000) as "the reduction of transparency of a liquid caused by the presence of undissolved matter". It is measured by comparing the intensity I_0 of an incident light beam, with the intensity I_1 of the same beam after its travel through a medium, for instance a wastewater sample.

The absorbance A is defined as

$$A = \log\left(\frac{I_0}{I_1}\right)$$

The turbidity T is defined as the absorbance per length unit. If l is the length of the optical path trough the medium then

$$T = \frac{A}{l}$$

ISO 7027 states that two measuring principles can be used by sensors, i.e. attenuation or scattering at a 90° angle, the latter being also called nephelometry. Details about optical features for each principle are given by the standard, especially regarding the wavelength (860 nm $\pm$30 nm). Formazine ($C_2H_4N_2$) standard suspensions to be used for calibration and units attached to both the calibration standard and the measuring principles (FAU and FNU respectively for attenuation and nephelometry) are also described. 1 FAU or 1 FNU are obtained with a concentration of 0.72 mg/L of formazine.

"Reproducibility" relates to the consistency of results obtained by measuring one single measurand under *varying* conditions (ISO TAG4 1993). The varying conditions may include the model of sensor, the standards used for calibration, the measurement principle, temperature, etc… Reproducibility must not be confused with "repeatability", which relates to the consistency of results obtained by measuring one single measurand under the *same* conditions. Thus repeatability is included in reproducibility. For turbidity, repeated measurements can be obtained by successive independent readings of the display (or recordings of the output).

In this paper, two types of reproducibility are worth considering.

(a) Reproducibility for *different individuals* (different serial numbers) of the same model (same reference number). This kind of reproducibility is best suited to applications on one particular site, which is the case of most operational situations, especially for monitoring and control purposes. Uncertainty is a measure of this kind of reproducibility, if all possible varying conditions are considered, including calibration, influence factors, operator, etc;
(b) Reproducibility for *different models* of sensors, usually supplied by different manufacturers. In this case, reproducibility must be limited to models based on the same measurement principle, with features fairly close to the ISO 7027 specifications. This kind of reproducibility is needed for comparing and extrapolating results from different sites: it is most useful for research purposes, but it would also contribute to promote turbidity as a regulatory water quality parameter. It may exceed the uncertainty obtained for each sensor.

Reproducibility of different models of sensors

In the case of turbidity, the model of sensor is a key aspect, which may go beyond the usual scope of reproducibility. Indeed, the model of sensor is included in the definition of the measurand, as turbidity is not defined on an absolute basis, but instead in relationship with a measurement principle (attenuation or scattering). This is the reason why two different units are used for expressing results. The effect of the measuring principle is huge: turbidity values commonly observed on wastewater during dry weather are around 100–200 FNU and 250–500 FAU. This means that the same wastewater sample measured with two sensors calibrated with the same formazine standard will provide completely different numerical values.

Even when the question of measuring principle is solved, by limiting reproducibility to sensors using the same principle, different sensor specifications may provide

different results. Among these specifications is the wavelength. ISO 7027 provides a reference value and a bandwidth, which allows some differences. Moreover, manufacturers supply only a limited number of turbidimeters suited for field measurement in sewer systems, some of them not strictly complying with the ISO standard: wavelength as high as 950 nm are used for technical reasons. Figure 1 shows the effect of the wavelength on the absorbance of different samples: wastewater from different origins (with different SS concentrations) and formazine. For formazine, the absorbance varies ±6.5% in the range of wavelength complying with the standard, and −16% in an "extended" range going up to 950 nm. For wastewaters the variation of absorbance is somewhat lower (respectively ±4% and −10%). As turbidity is the ratio between the absorbance of the sample to the absorbance of formazine, *two turbidimeters with different wavelengths, both calibrated with formazine, will provide different turbidity values for the same wastewater sample*. Tests performed on a few wastewater samples with a spectrometer (Figure 1) show that the reproducibility related to wavelength is expected to be 2–3% in the standard range of wavelengths, and as high as 6–9% in the "extended" range.

Reproducibility of different individuals of the same model of sensor

When a single type of sensor is used, factors affecting reproducibility can be sorted into two categories:

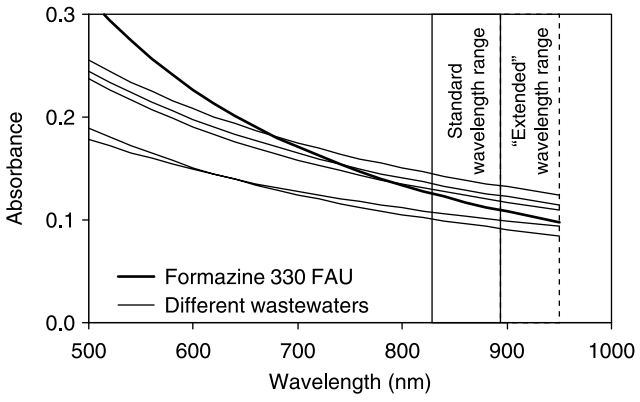

Figure 1 | Influence of wavelength on absorbance by formazine and waste water.

Factors related to the sensor:

- calibration
- intrinsic repeatability
- resolution

Factors related to both the measured medium and measuring procedure

- sample conditioning: sieving, homogeneizing.
- influential factors: ambient light, temperature, size of the measuring vessel…

Provided a few precautions (detailed in the next section) are taken, the influence of the procedure can be kept to a low level, and the main concern about the sensor will be its calibration.

A PROCEDURE FOR MEASURING THE TURBIDITY OF WASTEWATER SAMPLES

A common procedure has been set up by the authors for achieving a level of reproducibility which can be assessed on the same basis for the different laboratories. This procedure is divided into two parts. The first one is a set of actions to apply to a particular sensor. It includes checks and calibration of the sensor. This part is the most important as it must be applied for *in situ* monitoring as well as for turbidity measurement performed on samples. The second part of our procedure is dedicated to the latter kind of measurement. It is a set of actions related to a particular sample of wastewater.

Components of the procedure

Both parts quoted here above combine elementary procedures (items) from the following list:

a) Preparing a turbidimeter and laboratory environment
b) Preparing samples

- Reference for calibration and checks
- Wastewater

c) Performing a measurement/quantifying repeatability
d) Checking and adjusting a turbidimeter on the basis of a simplified calibration (including preparing of standard samples)

- Reference materials, and turbidity values to check
- Checking and adjusting zero value
- Checking and adjusting a reference value other than zero

e) Full calibration (may include preparing standard samples)
- Number and values of standard solutions (calibrators)
- Number of readings
- Calibration process for one set of standard solutions
- Calculation of calibration uncertainty, from one or several sets of standard solutions.

Part #1 (sensor related) of the general procedure includes:

- Preparing the measuring device and laboratory environment;
- Checking the sensor and adjusting it if needed on the basis of a simplified calibration;
- Initial full calibration, and assessment of calibration uncertainty (provided the uncertainty on standards is known);
- Checking the calibration curve ounce a year, and updating it when necessary, on the basis of a full calibration.

Part #2 (sample related) of the general procedure includes:

- Preparing the measuring device and laboratory environment;
- Assessing repeatability, either on the sample or on the basis of available experience;
- Checking the sensor and adjusting it if needed on the basis of a simplified calibration;
- Preparing samples;
- Performing the measurement.

Each item cannot be developed in the scope of this paper, so we will focus on two aspects: sample preparation and full calibration

Sample preparation

One key point in sample preparation is avoiding artefacts, like large particles and bubbles. Bubbles are obviously artefacts and can be easily avoided by limiting mixing energy. Large particles are artefacts regarding turbidity (although they are a full part of wastewater pollution) because they cannot be properly sampled with the narrow light beam (# 5 mm) used by turbidimeters: partial or complete occultation of the beam would rather result in particle counting than an actual turbidity measurement. So it is advisable to sieve the wastewater samples with a 2 mm mesh before performing any turbidity measurement. Of course this sieving is not handy for *in situ* monitoring. Thus turbidity measured on raw wastewater will result in a higher scattering of measured values, which can be somewhat reduced by a suitable signal processing, which discards short-duration peaks (Joannis & Aumond 2005).

The second point is the need for stirring the sample before and during the measurement. This is necessary to avoid any sedimentation of suspended solids and thus to obtain a homogenous sample. It is best performed mechanically (magnetic stirrer) and this kind of device should be used even for (offline) measurements performed on the field.

Other points are specific of the medium: wastewater needs much care when taking samples from a sewer, and transporting them to the laboratory. Formazine standard solutions must be prepared carefully, kept in the dark, for a period not exceeding 4 weeks, and additional care is needed to avoid spoiling, especially in the case of multiple uses of the same sample.

Sensor calibration

This is the major component of uncertainty, but it can be substantially reduced by properly addressing non-linearity. Another point is that the uncertainty in standard solutions must be assessed.

In order to lesser the *relative* uncertainty for the whole measurement range, we suggest using series of 6 standards (this is the minimum number required by ISO 7027) distributed on progressive scales, like 0, 100, 250, 500, 1000 and 2000 FAU or 0, 50, 125, 250, 500 and 1000 FNU. Five independent readings, obtained by rinsing and drying the sensor between each reading, enable a good assessment and a reduction of the experimental scattering s_{I0} (Equation 1).

An assessment of the effect of the uncertainty in standards, together with a reduction of this effect, can be obtained by repeating the whole process on different series

of standards. Five series should be sufficient for that purpose. The uncertainty on standards can also be assessed by regular uncertainty calibrations, or specified by a manufacturer. In any case a specific fitting method must be applied, known as "Williamson's method" (see Bertrand-Krajewski 2004 for the case of linear calibration curve). The calculation of uncertainties is also different.

Using a non-linear calibration curve may be needed for some sensors. This will result in more complicated calculations for uncertainties, and a general calculation method like Monte Carlo simulation may be preferred to dedicated analytical methods (Ruban & Joannis 2007).

UNCERTAINTY ON TURBIDITY MEASUREMENTS

Method

If x_0 is the measured value obtained by applying an inverse calibration curve to a displayed value y_0, a particular expression of the uncertainty on x_0, which implies some hypotheses on the distribution of errors is given by:

$$S_T^2(x_0) = \frac{1}{f'(x_0)^2}\left[\frac{S_{l1}^2}{n_0} + S_{l0}^2\left(\frac{1}{N} + A_{F5}\right)\right] \quad (1)$$

where $S^2T(x_0)$ is the standard deviation of the turbidity value derived from a reading y_0 by inverse application of a calibration curve $x_0 = f^{-1}(y_0)$.

In Equation (1) the first item between brackets expresses the effect of the scattering of readings for a given measurand (i.e. wastewater), and the second item represents the influence of the calibration process. The ultimate source of the uncertainty for this component is also the repeatability of readings, but in this case the readings are performed on standard solutions (formazine), and their effect on the calibration curve is fairly complex. This term can also be split into two components. The first component ($1/N$) is the uncertainty of the position of the calibration curve in relation to the ordinate axis. The second one (A_{F5}) is the additional uncertainty affecting other parameters (overall slope and shape) of the calibration curve.

A_{F5} is equal to $(x_0 - \bar{x})^2/\sum_{i=1,P} n_i(x_i - \bar{x})^2$ if the calibration curve is a straight line, and expresses the uncertainty in the slope. It has a more complex expression in the case of a polynomial, which can be found in (Bertrand Krajewski *et al.* 2000).

The scattering of readings (repeatability) has two different value s_{l0} and s_{l1}s, which are usually estimated by different means:

s_{l1} is derived from one specific repeatability experiment, and applied to any measurement performed in the same conditions (i.e., sieving of the sample, mixing energy, damping of the display…):

$s_{l1} = [\sum_{i=1,n_0}(y'_{0,i} - \bar{y}'_0)^2]/(N-1)$

$y'_{0,i}$ = displayed value for the reading i for a wastewater sample with a (real) value x_0

$n_{0=}$ number of independent readings for the value x_0

s_{l0} is estimated by the scattering of residuals between the calibration curve and actual readings:

$s_{l0} = [\sum_{i=1,P}\sum_{j=1,n_i}(y'_{i,j} - y_i)^2]/(N-2)$

P = number of different values of standard solutions used for calibration

N = total number of all independent readings for each standard solution: $N = \sum_{i=1,P} n_i$

n_i = number of independent readings for a standard solution with a (theoretical) value x_i

n_0 = number of independent readings for a sample with a "true" value x_o

y_i = ordinate of the calibration curve for the abscissa x_i

$y'_{i,j}$=displayed value for the reading j for a standard solution with a (theoretical) value x_i

f' = derivative of the calibration function, relating displayed values y with true values x.

Ideally s_{l0} should be equal to repeatability of readings for a particular reference sample, thus avoiding the need for a repeatability experiment for standard samples, and the standard deviation of repeatability be should constant over the measuring range in order to fulfil the hypothesis underlying Equation (1). In the real world, s_{l0} may vary and/or include effects due to an insufficient treatment of the non-linearity of the calibration curve, and/or include errors on the standard solution values (which is a question of reproducibility, not properly addressed by Equation 1). Another version of Equation 1 is available for varying S_{l0}, at least for calibration curves with a degree lower than 3.

For a non-linear calibration curve, Equation1 provides an approximation of the uncertainty obtained with a linear

fitting, but a non-linear fitting should be preferred when needed (usually a 2nd order polynomial is sufficient).

For the uncertainty in standard solutions, Equation 1 must be replaced by calculations defined in Williamson's method.

Anyway, the form of Equation1 remains relevant in any case, and is valuable to show the different contributions to the overall uncertainty.

RESULTS

Experiments performed by our teams provided a numerical assessment of several components of reproducibility and uncertainty. These values are given here as examples, but each operator should perform both i) repeatability and reproducibility experiments and ii) uncertainty calculations in order to derive values suited to his own application.

- Repeatability (standard deviation): 0.5–2 FAU (0.5%–0.1%) and 0.5–2 FNU (1%–0.1%) for formazine, 0.5–8 FAU (0.8–0.4% %) for wastewater samples.
- Reproducibility of standard solutions (standard deviation) 0.5–1% of the nominal value.
- Overall calibration uncertainty (95% confidence interval) (5 readings, 5 sets of standard solution, polynomial): 0.5–1%
- Overall calibration uncertainty (95% confidence interval) (5 readings, One set of standard solution, polynomial): 1–1.5%.

First results about drift and influence factors for *in situ* measuring suggest that they might be lower than 2.5%.

In situ signal dispersion, quantified by the standard deviation of the values recorded over one minute with a time step of one second is in a range from 5 to 20 FAU. The uncertainty related to this dispersion can easily be reduced by a factor larger than 5 by averaging.

CONCLUSION

Being able to measure turbidity of samples, either in laboratory or in the field, with immersion sensors suited for continuous monitoring is essential for:

- calibrating sensors with formazine, and getting reproducible results with different sensors using the same principle (attenuation or nephelometry) and preferably of the same model;
- if required by the application, setting up relationships between turbidity and regulatory parameters, like suspended solids concentrations. However, the accuracy of the estimates derived from these relationships will be limited by the variability of the characteristics of suspended solids during rain events, especially their size and density.

The procedures outlined in this paper enables measurements on wastewater samples with an uncertainty lower than 2% or 5 FAU, provided non-linear calibration curves are applied when needed. In some cases, the uncertainty in measurement standard solutions (formazine) is to be considered. Thus these procedures ensure a good reproducibility of results obtained with different sensors, provided they use the same optical principle (either attenuation or diffusion) and similar wavelengths. On the other hand, results obtained by attenuation are very different from those obtained by nephelometry for a given wastewater sample, even if when the sensors are calibrated with the same formazine standard solutions: turbidity values expressed as FAU are at least twice greater than the values expressed as FNU.

For *in situ* monitoring, the experimental uncertainty is higher than in the laboratory, and the effects of drift and influential factors like temperature must be accounted for. This should result in global uncertainties lower than 5% or 25 FAU.

Turbidity sensors designed for monitoring wastewaters quality are nowadays fairly reliable, and when proper calibration procedures are applied, they provide data with a good accuracy and reproducibility. Thus their use should spread in the years to come, as they are a very valuable tool for continuously assessing pollutant loads transported in sewers, and for real time control of facilities like combined sewers overflows and detention tanks.

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