

Particle characterization of urban wet-weather discharges: methods and related uncertainties

Caractérisation des particules des rejets urbains de temps de pluie : méthodes et incertitudes associées

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RÉSUMÉ

Les particules des eaux résiduaires urbaines jouent un rôle majeur dans l'impact des rejets urbains de temps de pluie sur l'environnement. Il est ainsi particulièrement intéressant de les caractériser pour évaluer leur impact et comprendre leur comportement en réseau. La mesure de la masse volumique humide et sèche avec un pycnomètre à eau ne demande, pour une précision de 5%, qu'une prise d'essai d'environ 1.4 g de masse sèche. On peut faire ainsi plusieurs mesures de masses volumiques avec un préleveur classique (24 bouteilles de 1 L). La mesure de granulométrie par diffraction laser des particules dont la taille est inférieure à 560 μm permet d'appréhender les variations de granulométrie entre le temps sec et le temps de pluie. La méthode de détermination des incertitudes par simulation de Monte Carlo présente plusieurs avantages par rapport à la méthode classique de composition des incertitudes : conditions d'utilisation plus larges et aisées, distribution « réelle » des valeurs de la variable de sortie. Cependant la méthode expérimentale d'essais de répétabilité et de reproductibilité est complémentaire car elle permet notamment de vérifier si des sources d'incertitudes n'ont pas été oubliées ou sous-estimées. Pour déterminer une incertitude de mesure en fonction de la valeur mesurée, il est intéressant d'estimer l'écart type à partir de la médiane sur des séries de duplicats. Cette méthode permet notamment de détecter des valeurs atypiques et des changements dans les conditions de mesure, d'où la possibilité d'améliorer la méthode de mesure.

ABSTRACT

Urban wastewater particles play a major role on the impact of wet weather discharges in the environment. So particle characterization is of particular interest to assess their impacts and understand their behaviour in sewers. For a 5% uncertainty, wet and dry density measurements with a water pycnometer only require a test portion of about 1.4 g dry mass. It is, therefore, given the wastewater concentrations, easy to proceed to several density measurements with a classical 24 1 L-bottles sampler. The particle size distribution (PSD) measurement by laser diffraction of particles the size of which is smaller than 560 μm , allows to evaluate the PSD variations between dry and wet weather. The method for determining measurement uncertainties with the Monte Carlo simulation presents several advantages in comparison with the classical method of uncertainty combination: wider and easier use conditions, "real" distribution of the output quantity values. However, the experimental method with repeatability and reproducibility tests is complementary as it allows particularly to check if uncertainty causes were not forgotten or under-estimated. To determine the measurement uncertainty as a function of the measured value, it is interesting to evaluate the standard deviation from the median of series of duplicates. This method allows particularly to detect atypical values and changes in the measurement conditions on the long term, thus the possibility of improving the measurement method.

KEYWORDS

Density, measurement uncertainty, size analysis, suspended solids, urban discharges

1 INTRODUCTION

Urban wastewater particles play a major role on the impact of wet weather discharges in the environment. Indeed pollutants are mainly in particular form or bound to particles (Chebbo, 1992), and moreover the erosion of deposits in combined sewers often accounts for 30 to 40% of urban storm discharges (Ashley et al., 2004). So particle characterization is of particular interest to assess their impacts and understand their behaviour in sewers, especially their deposition and erosion. Standardized or non standardized methods exist to measure their characteristics, the uncertainties of which must be known to assess their impacts and model performances correctly.

This paper deals with methods for measuring classical particle characteristics and evaluating their uncertainties:

- Wet and dry density with a water pycnometer;
- Particle size with a laser granulometer (Malvern Mastersizer MS-1000).

The pros and cons of the evaluation of uncertainties using repeatability and reproducibility experiments compared with the classical method of uncertainty combination are discussed. Finally a method for evaluating the uncertainty related to the measured value using reproducibility experiments is presented, applied to the standardized methods for global pollution parameters (Suspended Solids and Chemical Oxygen Demand).

The described methods were applied to combined sewer particles in suspension, a representative collection of which can be made using an automated sampler. A representative collection of the full cross-sectionnal particles including the coarse ones near the sewer bed is more difficult (Kim, 2008).

2 MEASUREMENT OF WET AND DRY PARTICLE DENSITIES

Particle density is classically measured using gaz pycnometers (e.g. ASTM D5550 in Lin, 2009). Chebbo (1992) also selected an air pycnometer because some floating particles blocked the water pycnometer capillary. Confronted with the same difficulty for the dry density measurement, we first measure the wet density using a water pycnometer, then the water content, from which we deduce the dry density. Wet density is relevant to the hydraulic behaviour of the particle and the dry density is representative of its more or less mineral nature.

2.1 Method description

The method includes the following steps:

- Sample centrifugation at 4000g (g: gravity acceleration) during 20 minutes;
- Dripping of the centrifuge cake and volume determination with a water pycnometer (50 mL);
- Water content determination of the centrifuge cake after drying in an oven at 105 °C during 24 hours.

The formulas expressing the wet density ρ_w and the dry one ρ_d are the following (in metrologic terms, those formulas correspond to the model of the measurement process):

$$\rho_w = \frac{m_w}{V - \left(\frac{m_f}{\rho_{f\theta}} \right)} \quad \text{and} \quad \rho_d = \frac{\rho_w (1 - w_c)}{1 - \left(\frac{\rho_w}{\rho_{f\theta}} w_c \right)} \quad (1)$$

Meaning of the input quantities symbols:

- m_w : wet sample mass; m_f : fluid (water) mass in the pycnometer;
- V : calibrated volume of the pycnometer;
- $\rho_{f\theta}$: fluid density at θ temperature; w_c : sample water content.

2.2 Uncertainty evaluation by combination of the input variable uncertainties

2.2.1 Classical method combining the standard uncertainties

Considering a quantity y as a function f of N independent quantities x_i , the standard uncertainties of

which are $u(x_i)$, the method combining the uncertainties stands that the standard uncertainty u_c for y is the following (ISO/CEI GUIDE 98-3:2008, Guide to the expression of uncertainty in measurement – GUM):

$$u_c(y) = \sqrt{\sum_{i=1}^N \left[\frac{\partial f}{\partial x_i} \right]^2 u^2(x_i)} \quad (2)$$

Table 1 shows for the different quantities of equations (1): their value, standard uncertainty $u(x_i)$, partial derivative $\partial f/\partial x_i$, product $\partial f/\partial x_i u(x_i)$ called component of standard uncertainty (squared).

$\rho_w = f1(m_w, V, m_f, \rho_{f0})$				
m_w (g) =	4.04331			
V (cm ³) =	48.917276			
m_f (g) =	44.98658			
ρ_{f0} (g.cm ⁻³) =	0.998185	ρ_w (g.cm ⁻³) =	1.0505	
quantity	$u(x_i)$	$\partial f/\partial x_i$	$(u(x_i) \partial f/\partial x_i)^2$	
m_w (g) =	8.00E-04	0.259815	4.32E-08	
V (cm ³) =	0.0074	0.272939	4.08E-06	
m_{f0} (g) =	8.00E-04	0.273435	4.79E-08	
ρ_{f0} (g.cm ⁻³) =	2.00E-04	12.323271	6.07E-06	$u(\rho_w)$ (g.cm ⁻³) = 0.0032
$\rho_d = f2(\rho_w, w_c, \rho_{f0})$				
ρ_w (g.cm ⁻³) =	1.0505			
w_c =	0.87720			
ρ_{f0} (g.cm ⁻³) =	0.998185	ρ_d (g.cm ⁻³) =	1.6794	
quantity	$u(x_i)$	$\partial f/\partial x_i$	$(u(x_i) \partial f/\partial x_i)^2$	
ρ_w (g.cm ⁻³) =	0.0032	19.279172	3.81E-03	
w_c =	8.00E-04	26.766185	4.59E-04	
ρ_{f0} (g.cm ⁻³) =	2.00E-04	20.219950	1.64E-05	$u(\rho_d)$ (g.cm ⁻³) = 0.0654

Table 1. Balance sheet of the components of the standard and relative uncertainties on wet and dry densities

The squared component of standard uncertainty represents the weight of the considered quantity in the uncertainty combination. Those of the volume V of the pycnometer and of the water density ρ_{f0} are about a hundred times larger than the others. Then, in order to master the uncertainty of the wet density, the volume V of the pycnometer must be carefully calibrated, and the influence of the water temperature θ on its density ρ_{f0} must be taken into account. Concerning the dry density, besides the uncertainty of the wet density, the water content w_c determination by weighing must particularly be controlled.

In order to interpret the standard uncertainty in terms of probability, one considers that the output quantity y is normally distributed. Then the confidence level of the standard uncertainty is 68% and has to be multiplied by the Student coefficient $t_{95}(v_{eff})$ to reach the 95% usual confidence level. The Student coefficient is found in tables as a function of the degrees of freedom v_{eff} , which in our case is the number of samples used in the standard uncertainty calculation minus the number of estimated parameters, i.e. one because the population mean is estimated from the sample mean. Finally the expression of the relative uncertainties $U_{r,p}$ on the densities at a 95% confidence level is:

$$U_{r,p} = t_{95}(v_{eff}) u_p/\rho \quad (3)$$

The relative uncertainties for the mean values of the repeatability experiment (cf. §2.3.1) amount to 0.67% for the wet density and to 8.6% for the dry density.

It must be noted that this method of uncertainty evaluation includes the deviations of the measurement devices with the standards, recorded during the metrological controls (balance and thermometer in our case). The deviations for all the device checks are treated as random errors, without considering a systematic error or bias.

2.2.2 Uncertainty evaluation with the Monte Carlo method

The GUM has been recently complemented with the Monte Carlo method (ISO/CEI GUIDE 98-3/S1:2008). This method takes advantage of the present computer facilities to make numerous calculations, particularly with spreadsheets.

It consists in calculating the distribution of the output quantity values with the process model, by drawing the input quantity values randomly according to their own statistical distributions. This method shows the following advantages in comparison with the classical method combining the standard uncertainties:

- No partial derivative calculation, the expression of which may be complicated;
- No validity limit for the method in relation with the non-linearity of the model or with the variation range of the input quantities;
- The output quantity distribution is no longer supposed to be normal, but on the contrary the real distribution is obtained.

Then the Monte Carlo method was applied to the models of wet and dry densities, with the same values of the input quantities and associated standard uncertainties as those of §2.3.1. One thousand sets of values were simulated by random drawing following the normal distribution.

The relative uncertainties at a 95% confidence level amount to 0.60% for the wet density and to 7.9% for the dry density. They are then slightly smaller than those obtained with the classical method. It is due to the fact that the density distribution is more compact than the normal one, hence a narrower confidence interval and a smaller uncertainty.

2.3 Uncertainty evaluation by experimental repeatability

The above uncertainty evaluation by model analysis (B type) is attractive as it allows to identify the influence of the various uncertainty causes. The measurement uncertainty can also be evaluated by experimental repeatability. Investigating all the possible cases with such an experimental method (A type) would require too many experiments, especially for the influence quantities. But it allows to quantify the uncertainties in “real” conditions, and particularly to check if uncertainty causes were not forgotten or under-estimated.

2.3.1 Comparison of uncertainty evaluation by uncertainty combination and experimental repeatability

A repeatability experiment was carried out with twice 40 liters of sample which were pre-concentrated in the fridge for 24 hours, then distributed into 12 sub-samples. Then wet and dry densities were determined for those 12 sub-samples using the method described in §2.1. Figure 1 shows the successive values obtained. The fact that no trend arises (slope equal to zero with the Student test) means that results are homogeneous and random.

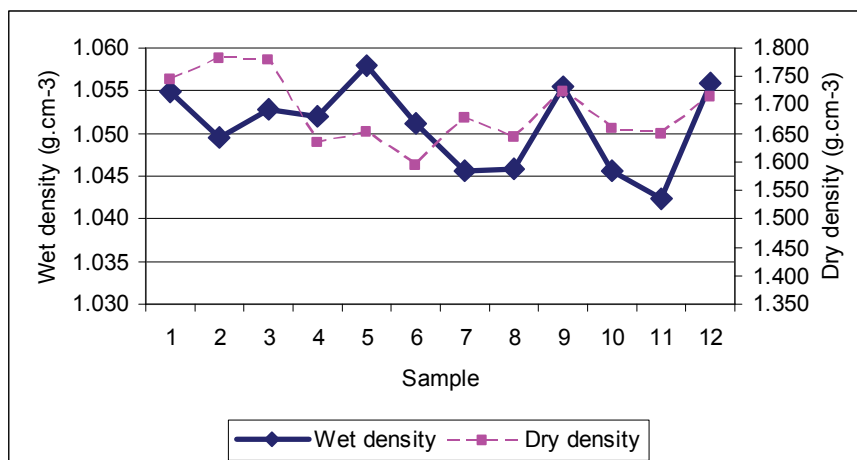


Figure 1. Wet and dry densities of the 12 sub-samples

The relative uncertainty amounts to 0.96% for the wet density and to 8.1% for the dry density, to be compared to the respective values of 0.60% and 7.9% obtained with the Monte-Carlo method. Those

values are within the confidence interval of the uncertainties at a 95% confidence level for 12 samples. It can, therefore, be concluded that they are equivalent and that the uncertainty causes were correctly evaluated.

2.3.2 Sample volume determination to obtain a satisfactory uncertainty

It is necessary to know in advance the sample mass to be collected in order to obtain a satisfactory uncertainty on wet and dry densities. Indeed it can be shown with the Monte Carlo method that the relative uncertainty is inversely proportional to the dry mass of the sample, or which is the same, that the product of the relative uncertainty by the sample dry mass is constant.

As an example we simulated, for the mean values of wet density (1.05 g.cm^{-3}) and of water content (86%) recorded on our runoff samples, the product of the relative uncertainty on the dry density, by the dry mass for values between 0.5 and 5 g. This simulation showed that this product is effectively nearly constant (about 0.04 g.cm^{-3}) for dry masses larger than 0.5 g.

But as the density relative uncertainties determined by experimental repeatability are nevertheless larger than those determined with the Monte Carlo method, and in order to take into account the operator effect, it was decided to use the experimental method to determine the constant equivalent to the product of the relative uncertainty by the sample dry mass.

Figure 2 shows the results of the repeatability experiment on 12 samples described in §2.3.1, just as those of 4 complementary experiments carried out by several operators with the same procedure with 8 samples each. The products of the relative uncertainty by the dry mass for wet and dry densities seem effectively constant (slope equal to zero with the Student test). However, the larger value of 0.07 was selected for the dry density constant.

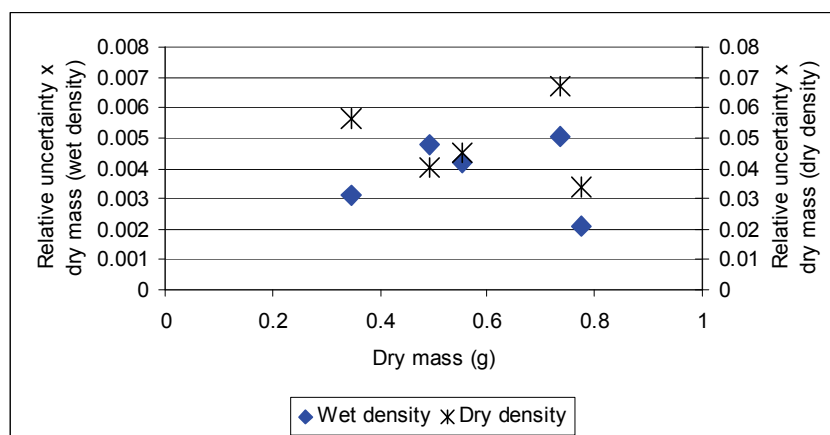


Figure 2. Product of the relative uncertainty by the dry mass vs dry mass

Then, to reach a relative uncertainty $U_{r,p}$ of 5% at the 95% confidence level, the sample dry mass must amount to:

$$U_{r,p} \ m_d = 0.07 \quad \text{then} \quad m_d = 0.07/5\% = 1.4 \text{ g} \quad (4)$$

This method is attractive as it necessitates a sample mass really smaller than the gaz pycnometer one (about ten times smaller). As an example, for samples with a suspended solid concentration of about 250 mg.L^{-1} , only 6 litres are needed to reach the required mass, which is easy with classical samplers equipped with 24 1L-bottles.

Besides, the suspended solid concentrations can be evaluated before analysis by turbidimetry. Indeed during wet weather (WW), the ratio suspended solid concentration (SS) versus turbidity T is larger than the dry weather (DW) one (Ruban in Rodriguez, 2005):

$$\frac{SS_{WW}}{T_{WW}} > \frac{SS_{DW}}{T_{DW}} = K_{DW} \quad (5)$$

Hence when applying the dry weather ratio to the turbidities, one is certain that the analysed concentrations will be larger than the estimated ones SS_E :

$$SS_{WW} > SS_E = K_{DW} T_{WW} \quad (6)$$

So the sample volumes required to reach a satisfactory relative uncertainty for the density determination can be evaluated before the SS analysis.

3 MEASUREMENT OF WASTEWATER PARTICLE SIZE DISTRIBUTION BY LASER DIFFRACTION

The measurement of wastewater particle size distribution (PSD) by laser diffraction combined with sieves is common for runoff waters (Anta, 2006; Kim et al., 2008) and for combined waters as well (Chebbo et al., 1992; Verbanck et al., 1993). Kim (2008) mentions Memon and Butler (2005) who note a reasonable agreement between laser diffraction and sieving observations for the size range 50 μm – 564 μm . The NF ISO 13320-1 standard (2000) deals with the particle sizing by laser diffraction and describes the general principles of the measurement process and of the quality control of the analysis.

Our measurement procedure of particle sizing is based on the sieving of particles larger than 560 μm and on laser diffraction measurement of the smaller particles. PSD measurement by sieving is a classical method and its uncertainties are known (e.g. Chebbo, 1992). Then we shall consider in this paper the particle sizing by laser diffraction.

The settings of the Malvern Mastersizer MS-1000 granulometer (homogeneization and circulation speeds) were determined so that the particles be kept in suspension without being altered. As the objective is to measure the particle size distribution in their original state, no ultrasonics were applied, which according to Chebbo (1992) desagglomerate them. The PSD measurements were made generally on the day of sample collection to prevent the alteration of organic matter.

3.1 Evaluation of the measurement reproducibility

The NF ISO 13320-1 standard (2000) specifies that calibration is not strictly speaking imposed, but the good working of the device must be confirmed using a reference material. So the measurement reproducibility on the long term can be controlled. Before each measurement series, the device adjustment is validated by an Internal Quality Control using a specific lot of kaolinite.

The check deviations between the device indications and the kaolinite values for 15 measurement series between July 2007 and May 2009 were examined. Figure 3 shows the mean volumetric percentages for 27 size fractions between 1.6 and 560 μm , so that the relative standard deviation (dotted lines). Here the term standard deviation is used as it is evaluated statistically from experimental results (type A method), but it is equivalent to the term standard uncertainty. The relative standard deviation s_{r_repro} varies from about 0.01 to 0.06. As its value is no longer representative for the fractions above 80 μm (percent of particle volume too small), it was linearly extrapolated between 100 and 560 μm .

3.2 Evaluation of the measurement uncertainty in wastewaters

The relative standard deviation s_{r_repro} determined above expresses the long term stability of the device in relation to the reference material, but is not representative of the random measurement errors in wastewaters. Then we proceeded to a repeatability experiment with 4 dry weather samples collected on the 18-19/08/2009 at 1h00, 10h00, 15h00 and 21h00.

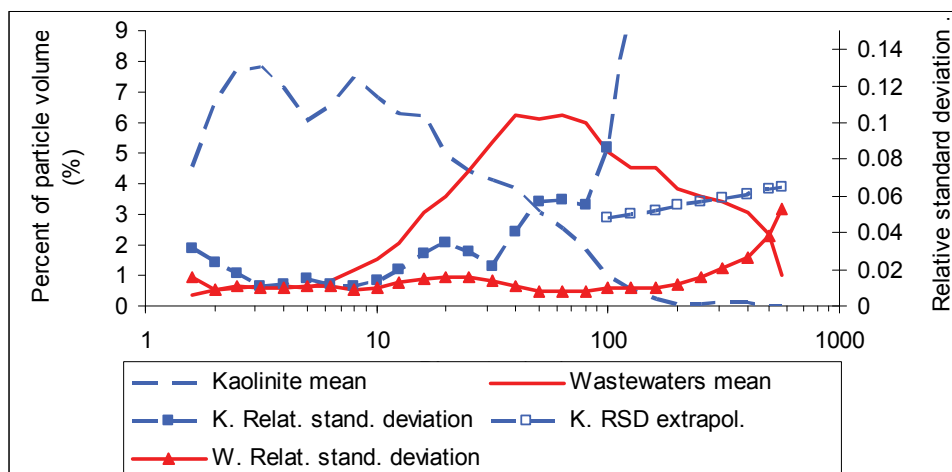


Figure 3. Size distributions and associated relative standard deviations for kaolinite and wastewaters

Each PSD measurement was repeated 3 times. As the relative standard deviations of the 4 samples were statistically similar, the mean relative standard deviation was calculated from the 12 repetitions (4 samples x 3 repetitions). Figure 3 shows that the repeatability standard deviations s_{r_repeat} vary from about 0.01 to 0.05 (continuous line), and are smaller than the reproducibility standard deviations s_{r_repro} with kaolinite.

Considering that the relative standard deviations s_{r_repeat} and s_{r_repro} are representative components of the measurement uncertainty for the measured value, the total relative standard deviation of the measurement results from their quadratic combination, and the measurement uncertainty at a 95% confidence level is:

$$U_{r_total}(95\%) = t_{95}(v_{total}) \sqrt{s_{r_repro}^2 + s_{r_repeat}^2} = t_{95}(v_{total}) s_{r_total} \quad \text{with} \quad v_{total} = \frac{s_{total}^4}{\frac{s_{r_repro}^4}{15-1} + \frac{s_{r_repeat}^4}{12-1}} \quad (7)$$

The degrees of freedom v_{total} are calculated with the Welch formula as the number of samples used to calculate s_{r_repeat} and s_{r_repro} are different. U_{r_total} varies from about 3% for the fine particles to 15% for the large ones. Figure 4 shows that this relative uncertainty allows to evaluate the PSD variations between dry and wet weather for the fractions of particles smaller than 200 μm , which accounts for about 80% of the particle mass in combined sewers (Chebbo, 1992).

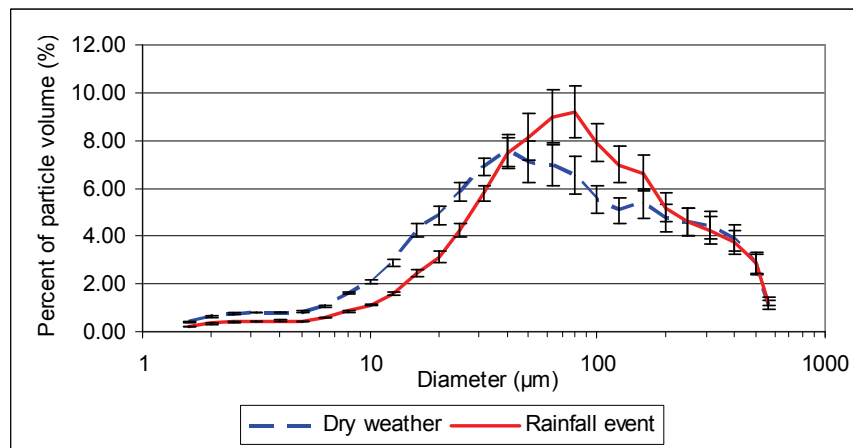


Figure 4. Size distributions and associated total relative standard deviations for dry and wet weather wastewaters

4 UNCERTAINTIES ON SUSPENDED SOLIDS AND CHEMICAL OXYGEN DEMAND ANALYSIS

4.1 Method

The uncertainties of measurement results often vary with the measured values. A method consists in evaluating the standard deviation by repeating the measurement on the same sample, for example three replicates (Bertrand-Krajewski, 2007). This method allows to evaluate the standard deviation in short term measurement conditions and to detect outliers, but the uncertainty on the standard deviation is high.

The method described below consists in evaluating the standard deviation as a function of the measured value on series of duplicates (2 replicates). This method allows to evaluate the standard deviation on the long term (change in measurement conditions) and to detect outliers, and the uncertainty on the standard deviation is moderate as the sample number is high.

This method proposed in « Statistique appliquée à l'exploitation des mesures » (Anonymous, 1978), first consists in calculating the differences d_i between the duplicates. The variance of those differences ($d_i = x_i' - x_i''$) is twice the variance of the results x_i . Then, if the result distribution is normal, 50% of the absolute values of d_i are lower than (σ being the standard deviation of the population):

$$d_{75} = t_{75} \sqrt{2} \sigma = 0.954 \sigma \quad (8)$$

d_{75} is the median of the population of the absolute values of the differences d_i , i.e. of the ranges $w_i = |x_i' - x_i''|$. It is estimated with the median M_w of the measured ranges and σ can then be estimated with :

$$s = Mw/0.954 \tag{9}$$

This s estimation is in relation with the mean value $(x_i' + x_i'')/2$. Those pairs are then classified in the mean increasing order. The result pairs are then grouped in series of 11 consecutive pairs in which the standard deviation is supposed to remain constant. The median which is the 6th value of each series, is used to estimate its standard deviation and to observe its variation with the measured value.

4.2 Uncertainties on suspended solids and chemical oxygen demand analysis

The analysis were made according to NF EN 872 standard (2005) for the Suspended Solids (SS) and ISO 15705 for the Chemical Oxygen Demand (Small-scaled sealed-tube method, 2002).

We first studied the distributions of $d_i (= x_i' - x_i'')$ deviations which are symmetrical and centered on zero. They are, therefore, compatible with the normality hypothesis, the more or less narrow look of the histogram being the consequence of the distribution of the results versus the values. We also verified there was no noticeable variation in deviation during the study period (2004-2007). We will then suppose that the distribution is normal as it is usual for analysis results.

We then applied the above described method to the 560 SS samples and to the 172 COD samples. Figure 5 shows that a linear adjustment is sufficient to describe the median variation with the mean of analysis duplicates. We can then deduce the standard deviation by dividing the median by 0.954 as indicated above.

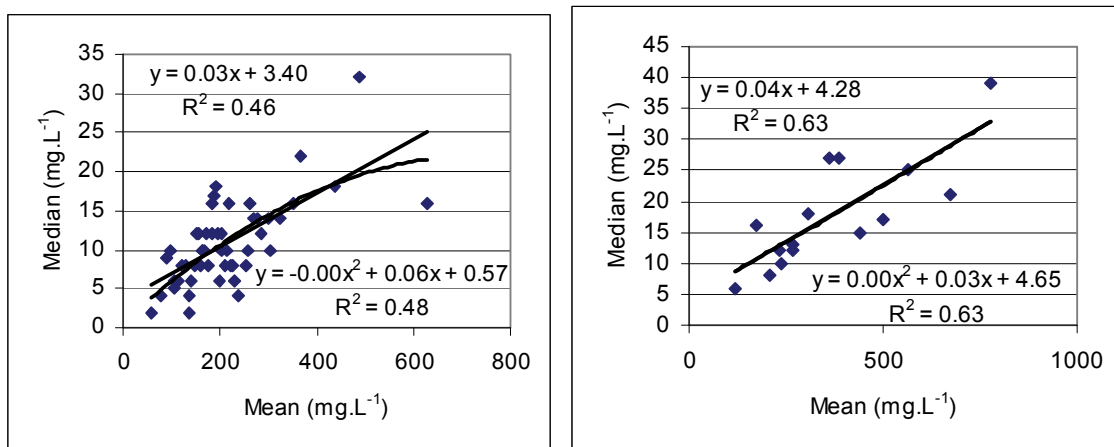


Figure 5 : medians of increasing 11-sample series versus the means $(x_i' + x_i'')/2$

If we suppose that the distributions of the results, then of the ranges are normal, those results for which the range is larger than 3.64 times the standard deviation have a probability less or equal to 1%. Figure 6 shows the time distribution of these ratios for SS. We have 45 ratios > 3.64, i.e. 8% (the normal value should be 1%).

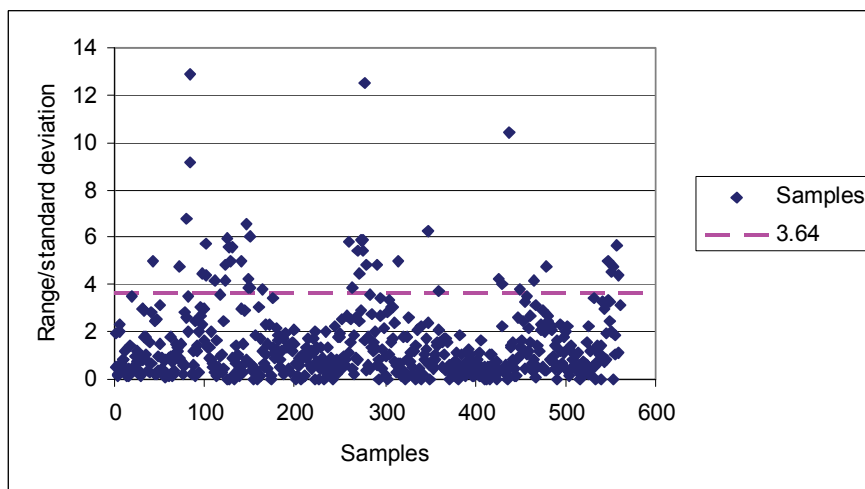


Figure 6 : position of duplicates in relation with the threshold for atypical values (3.64)

Ten of them are scattered on the 34 series and can be considered as « normal » atypical values. Five correspond to concentrations of about 100 mg.L⁻¹ (possible under-estimation of standard deviation for low values). However, the 30 remaining values are distributed in groups in certain series. This allows to think there is an heterogeneity of the results depending on the operators.

Results are better for COD : there are nevertheless 9 ratios > 3.64, i.e. 5% instead of 1 %. Six of them are scattered on the 20 series and can be considered as “normal” atypical values, but the last 3 are grouped in the penultimate series: they are diluted samples.

These results are expressed as relative uncertainties in order to compare them to other sources of uncertainty evaluation: twice the coefficient of variation (i.e. relative uncertainty at a 95% level of confidence for a normal distribution), and relative uncertainty on 95% of the samples (with outliers).

Concentrations mg.L ⁻¹	Relative uncertainty % (2CV) without outliers				Relative uncertainty % with outliers	
	LCPC	ASTEE	BKJL et al.	AGLAE	LCPC (95% samples)	
Suspended Solids						
100-600	14-8	15	18 ± 9		34-20	
100-150				25 (5)		
Chemical Oxygen Demand						
	LCPC	ASTEE	BKJL et al.	AGLAE	LCPC (95% samples)	BKJL et al. (100% samples)
80-800	19-9	10	13-19		35-16	14-29
300-350				11 (4)		

Table 2 : comparison of the LCPC results with other sources of uncertainty evaluation

The following comments can be made concerning the results in table 2 :

- for SS the results expressed as 2CV without outliers seem homogenous, but the ASTEE (Association Scientifique et Technique pour l'Eau et l'Environnement – working group « Méthodes d'analyses alternatives ») and AGLAE (Association Générale des Laboratoires d'Analyse de l'Environnement) results were obtained in inter-laboratories comparisons when the LCPC and Bertrand-Krajewski (BKJL: 2007) ones are intra-laboratory comparisons. However, the LCPC analyses were made by 4 different operators and the BKJL results include sub-sampling;

- for COD the LCPC and BKJL results expressed as 2CV without outliers are higher than the ASTEE and AGLAE ones, which can be explained by the same comment as above;

- the figures in brackets refer to the LCPC intra-results for the inter-laboratories comparison. This shows that a well experienced operator obtains good results. The reproducibility of the wastewater sample used is also perhaps better than that of an ordinary sample as it has to remain stable between the operator laboratory and the participating laboratories;

- the LCPC results expressed as 95% of the samples are much higher than the others, because of the numerous atypical results. Those atypical results probably originate from an heterogeneity due to certain less experienced operators. Bertrand-Krajewski et al. (2007) found that sub-sampling is the major source of analysis uncertainty. Then sub-sampling during homogenization of the sample will be tested instead of sub-sampling after homogenization by reversing the sampling bottle.

5 CONCLUSIONS

With regard to methods for characterizing the particles of urban wet-weather discharges, wet and dry density measurements with a water pycnometer only require a test portion of about 1.4 g dry mass for a 5% uncertainty. It is, therefore, given the wastewater concentrations, easy to proceed to several density measurements with a classical 24 L-bottles sampler. The particle size distribution (PSD) measurement by laser diffraction of particles the size of which is smaller than 560 µm, allows to evaluate the PSD variations between dry and wet weather.

The method for determining measurement uncertainties with the Monte Carlo simulation presents

several advantages in comparison with the classical method of uncertainty combination: wider and easier use conditions, “real” distribution of the output quantity values. However, the experimental method with repeatability and reproducibility tests is complementary as it allows particularly to check if uncertainty causes were not forgotten or under-estimated.

To determine the measurement uncertainty as a function of the measured value, it is interesting to evaluate the standard deviation from the median of series of duplicates (2 replicates). This method allows particularly to detect changes in the measurement conditions on the long term.

It is, therefore, necessary to evaluate the uncertainties of measurement methods, not only to specify their use conditions (e.g. test portion mass or volume) and use possibilities (e.g. for modeling), but also to improve them.

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