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**Measurement of water flow in closed conduits —
Tracer methods —
Part II : Constant rate injection method using non-radioactive
tracers**

*Mesure de débit de l'eau dans les conduites fermées — Méthodes par traceurs —
Partie II : Méthode d'injection à débit constant, utilisant des traceurs non radio-actifs*

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FOREWORD

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Draft International Standards adopted by the Technical Committees are circulated to the Member Bodies for approval before their acceptance as International Standards by the ISO Council.

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It has been approved by the Member Bodies of the following countries :

Australia	Ireland	Thailand
Belgium	Mexico	Turkey
Bulgaria	Netherlands	United Kingdom
Czechoslovakia	Romania	U.S.A.
France	Spain	U.S.S.R.
Germany	Switzerland	Yugoslavia

No Member Body expressed disapproval of the document.

Measurement of water flow in closed conduits — Tracer methods — Part II : Constant rate injection method using non-radioactive tracers

0 INTRODUCTION

This International Standard is the second of a series of standards covering tracer methods of water flow measurement in closed conduits. The complete series of standards will be as follows :

- Part I : General.
- Part II : Constant rate injection method using non-radioactive tracers.
- Part III : Constant rate injection method using radioactive tracers.
- Part IV : Integration (sudden injection) method using non-radioactive tracers.
- Part V : Integration (sudden injection) method using radioactive tracers.
- Part VI : Transit time method using non-radioactive tracers.
- Part VII : Transit time method using radioactive tracers.

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies the constant rate injection method using non-radioactive tracers for the measurement of water flow rate in closed conduits.

2 PRINCIPLE

Flow rate measurement by a constant rate injection method is based on a comparison between the concentration C_1 of a tracer, continuously introduced with a known volume rate of flow q , and the concentration of samples C_2 taken at some place beyond the mixing distance. The mixing distance is defined as the shortest distance at which the variation in concentration of the tracer over the cross-section is less than some pre-determined value (for example : 0,5 %). (See clause 6 of part I.)

The tracer rate at the injection point is equal to the rate at which it passes the sampling point :

$$q C_1 + Q C_0 = (Q + q) C_2$$

where C_0 is the initial concentration in the stream which is flowing at the rate Q .

Hence :

$$Q = q \frac{(C_1 - C_2)}{(C_2 - C_0)}$$

Generally C_1 is much greater than C_2 which leads to a simplification of equation (1) as follows :

$$Q = q \frac{C_1}{C_2 - C_0}$$

When C_2 is much greater than C_0 , the equation can be reduced still further to

$$Q = q \left(\frac{C_1}{C_2} \right)$$

Flow rate Q can thus be determined by comparing the concentration of the injected solution with the concentration of samples removed from the conduit.

In order to increase the accuracy it is recommended that the standard solution be prepared to a given dilution ratio N_{st} which shall be approximately equivalent to the dilution ratio N which is expected in the sample taken from the measuring cross-section.

The standard solutions and the samples taken from the measuring cross-section shall be compared using identical methods, and the following formula is applicable :

$$m = \frac{N}{N_{st}}$$

The flow rate to be determined can be found from the formula

$$Q = q m \bar{N}_{st}$$

3 REQUIRED CONDITIONS

3.1 Tracer

For the constant rate injection method the tracer shall meet the general requirements defined in 5.1 of part I. A list of generally used tracers is given in 5.1.1 of part I and their advantages with respect to the radioactive tracers are stated in 5.2.1.3 and with respect to other non-radioactive tracers in 5.2.2 of part I.

3.2 Duration of injection

The duration of injection shall be such that stable concentration conditions are established at all points of the measuring cross-section over a sufficient period of time.

Constant concentration shall be obtained for a period of at least several minutes.

A suitable duration of injection may be determined by a preliminary investigation involving the instantaneous injection of a dye such as fluorescein or a radioactive tracer. Curves 1 and 2 of the figure may be plotted for a given flow rate from the observation of the moment when the tracer (dye or activity) appears and disappears in each cross-section.

If it is required to achieve steady conditions for a period of time Δt in a selected measuring cross-section S, it is sufficient to add this period of time Δt to the time t_1 corresponding to the disappearance of the tracer at this point (i.e. on curve 2) and to plot through the resulting point C a curve 1' parallel to curve 1 characteristic of the appearance of the dye (or activity). The ordinate at the origin of this curve gives the duration of the injection to obtain a concentration level of duration Δt at cross-section S.

In practice, to obtain a concentration level of duration Δt at cross-section S, a calculation is made of the minimum required period of injection by adding to the value of Δt the time $t_2 = AB$ taken by the instantaneously injected tracer to pass through S.

The time interval t_1 between the beginning of the injection and the establishment of stable conditions can be read from curve 2 (ordinate of point B) and is directly obtained by measuring the time interval between the instantaneous injection and the tracer disappearance in cross-section S.

In the case of central injection, straight measuring length and turbulent flow, time $t_2 = AB$ may be estimated from the relationship

$$t_2 = \frac{6}{U} \sqrt{\frac{DX}{2}}$$

where

- U is the mean velocity of the flow;
- X is the distance to the injection point;
- D is the conduit diameter.

The fraction of maximum concentration remaining after time t_2 as given by this formula is 0,3 %.

It is possible, when no preliminary injection is made, to check the appearance of the concentration level, and therefore the minimum injection time, by taking samples and analysing them during the measurement, with the apparatus used for the measurement. This procedure enables the concentration variation to be determined as a function of time.

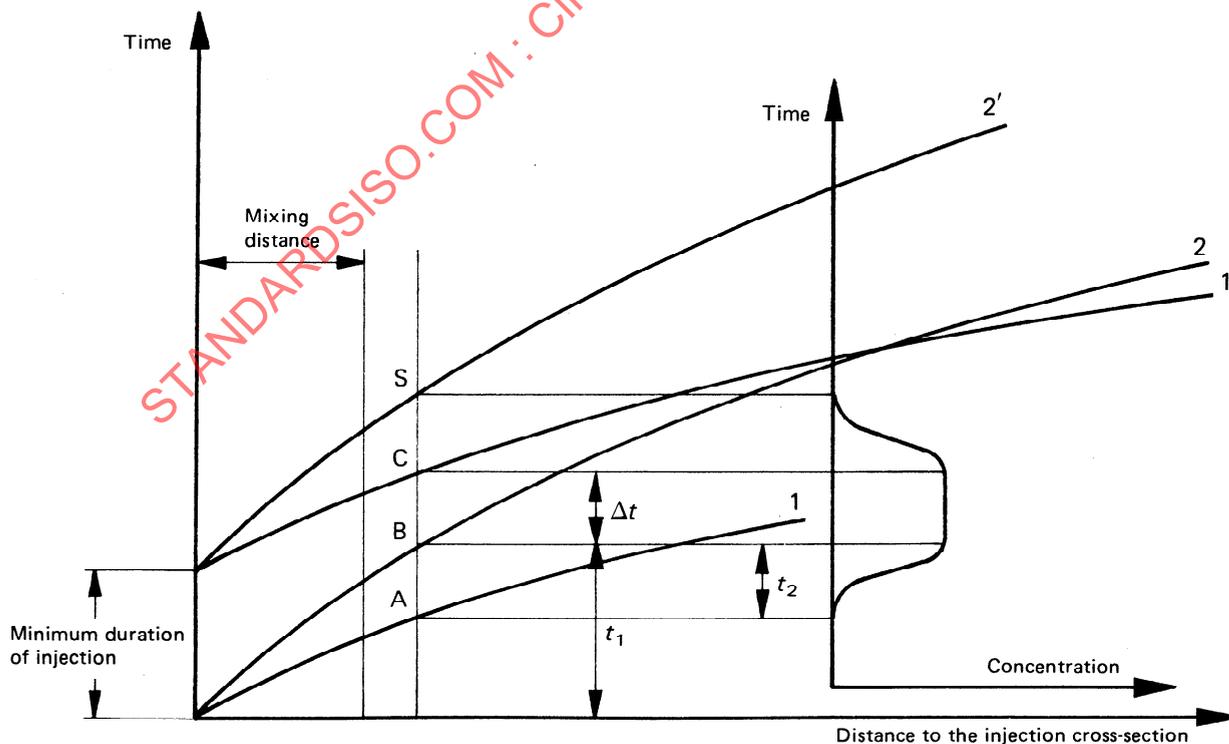


FIGURE – Determination of the duration of injection

4 CHOICE OF MEASURING LENGTH

4.1 Mixing distance

The mixing distance is defined in 6.1 of part I and may be calculated theoretically according to 6.2.1 of part I. Figure 3 of part I indicates the measured variation of the actual mixing distance according to the required accuracy in the case of a central injection, and in the case of three other injection methods. Methods for reducing the mixing distance are described in 6.3 of part I.

4.2 Experimental checking

It is seldom possible in conduits to check experimentally the homogeneity of the mixture and the duration of the tracer passage resulting from an instantaneous injection in a large number of cross-sections.

In practice, therefore, the experimentation consists of

- a) determining the minimum period of injection in the measuring length chosen by a preliminary test (see 3.2);
- b) checking, where possible, at the time of the measurement, that the mixture is homogeneous, by taking samples from at least two points of the measuring cross-section. In the case of high accuracy measurements (for example 0,2%), it is necessary to check the homogeneity of the mixture, prior to the measurement itself, when a doubt exists on the quality of this mixture;
- c) studying, whenever possible by taking samples from another cross-section further downstream, whether there are any systematic differences between the mean concentrations at the two measuring cross-sections. In particular, this method permits verification that the injected tracer is not absorbed in the measuring length either by entrained products in the liquid or by the conduit walls.¹⁾

4.3 Losses and additions

The losses and additions of the same liquid as that in the measuring length do not affect the results provided certain conditions are met as described in 6.5.1 of part I.

5 PROCEDURE

5.1 Preparation of the concentrated solution

It is essential for the injected solution to be homogeneous. The homogeneity of the solution can be obtained by vigorous mixing, by means of a mechanical stirrer or a closed circuit pump. It is advisable to prepare the injection solution in a separate container from the supply container with water filtered using an appropriate procedure.

However, if mixing is carried out in the supply container, the latter shall have sufficient capacity so that it is not necessary to add liquid or tracer during the injection. The solution shall be taken at a certain level above the bottom of the container, and every precaution shall be taken so that undissolved particles of the tracer are not carried in the solution injected. In the case of an injection of long duration, provision shall be made as required to avoid a variation of the solution concentration with time (for example by evaporation under the influence of ambient temperature).

5.2 Injection of the concentrated solution

The concentrated solution shall be injected into the conduit at a constant rate and for a sufficient duration to ensure a satisfactory period of constant concentration at the measuring cross-section (see 3.2 and 4.2). Several devices may be used for the injection of the concentrated solution. For all devices it must be possible to check the following :

- that the injection system is always free from leaks;
- that the injection rate is constant over the whole injection duration. To this end, any entrained impurities which may be contained in the solution shall be eliminated as they could partly or totally block the injection circuits.

One of the possible devices is a positive displacement pump driven by a constant speed motor.

5.3 Measurement of injection flow rate

The accuracy with which the injection rate can be measured depends on the measuring instruments used. The accuracy of the device shall be taken into account in the estimation of the total error on flow measurement.

Various measuring devices can be used provided they comply with one of the following requirements :

- a) they satisfy a principle based on the measurement of the basic quantities of mass, length, time, directly involved in the definition of the quantity of flow;
- b) they are calibrated in the conditions of use by measuring basic quantities involved in the flow rate definition (for example calibrated capacity method – weighing method, etc.);
- c) they are installed and used in conformity with the requirements of a Standard making it possible to calculate the accuracy obtained.

In the case of high accuracy measurements (for example 0,2%), it is necessary to measure the injection rate in accordance with items a) or b).

1) See 5.1 of part I.

The various devices may either give instantaneous values of the injection rate or allow the computation of one or several average values during the injection period.

5.3.1 Among the devices satisfying the first requirement, positive displacement pumps or calibrated tanks can be mentioned.

The former shall preferably be driven by an electric synchronous motor and the constancy of discharge checked by measuring the frequency of the electrical supply.

The use of the level drop in a tank as a measurement of the average flow rate requires that the tank be calibrated according to the permissible rules provided for this type of operation. If the dimensions of the tank are measured to estimate the capacity, care shall be taken that no deformation occurs during filling and that horizontal areas are measured over a sufficient number of sections to determine the relationship between height and volume with an error consistent with the required accuracy of flow rate.

Another method of calibration consists of determining the relationship between the height and the volume by means of a calibrated container the contents of which are transferred to the main container, and in measuring the heights of liquid reached each time.

An example for the computation of the error is given in 5.5.

5.3.2 Among the devices satisfying the second requirement can be mentioned propeller flowmeters and volumetric meters used under special conditions (for example lack of straight lengths and of straightener for a propeller flowmeter) and therefore needing calibration on site. In this case the mean of two calibrations made before and after testing shall be taken for the value of the injection flow rate, provided the calibrations do not differ by more than a value consistent with the required accuracy, for example 1 %.

5.3.3 Among the devices satisfying the third requirement, propeller flowmeters and volumetric meters can again be mentioned, when they are installed and used according to the accepted rules and therefore do not require calibration.

5.4 Samples

Samples shall be taken :

- from the conduit, to verify that the background concentration of tracer in the flow system is constant;
- from the conduit, to determine the tracer concentration in the measuring cross-section, to check that the tracer concentration is homogeneous in the sampling cross-section and to check the concentration level (see 4.2);

- from the injected solution, to check the homogeneity of the tracer concentration (see 5.1);
- from the injected solution, to compare the concentration of tracer in the injected solution with the concentration of tracer in the samples taken from the conduit.

In practice samples shall be taken under the following conditions :

- two or three samples of the liquid flowing in the conduit upstream from the injection section during injection or, when variations of the background concentration are suspected, along the measuring length, in the sampling cross-section before and after the passage of the solution;
- three to five samples of the injected solution at the outlet of the injection equipment immediately before and after the injection period;
- at least five samples of the diluted solution in the conduit, if possible from at least two points in the measuring cross-section (at least three at the same point), distributed in time during the passage of the solution.

5.5 Example of error computation in injection flow rate

In this example, it is assumed that the tracer is injected by means of a positive displacement pump driven by a synchronous motor and the frequency of the electrical supply is measured during the injection duration.

The injection rate of the pump, 2,09 cm³/s at 50 Hz, is determined before and after the flow tests by comparison with a calibrated capacity which determines this flow rate to within ± 2 σ_d = 0,005 cm³/s (for a 95 % confidence level).

During testing, the measured frequency is 49,9 Hz with a tolerance of ± 2 σ_f = 0,1 Hz.

The tolerance on flow rate is then :

$$\frac{2 \sigma_q}{q} = 2 \sqrt{\left(\frac{0,0025}{2,09}\right)^2 + \left(\frac{0,05}{49,9}\right)^2} = 0,3 \%$$

6 ERRORS IN FLOW RATE MEASUREMENT

For the determination of errors, reference shall be made to clause 7 of part I.

The application of the specifications described in this International Standard enable an accuracy of flow measurement of about 1 % to be obtained provided the mixture of the tracer in the flow is of equivalent accuracy and the injection rate is measured with a better accuracy.

The use of this method also enables higher accuracies to be obtained in the best conditions.

The estimation of errors as described in clause 7 of part I enables the accuracy of the measurement to be defined for each case.

7 ANALYSIS METHODS PRESENTLY USED FOR WATER FLOW RATE MEASUREMENT

7.1 Colorimetric method of analysis

7.1.1 Principle

Colorimetric analysis is based on measuring the transmission efficiency of monochromatic light passing through a glass cell containing the sample to be measured and comparing the transmission with that obtained through samples of known dilution ratio (i.e. control solutions). The wavelength of the monochromatic light normally used corresponds to that for the maximum absorption of light in the particular tracer solution used.

The phenomenon is governed by the law of Beer-Lambert :

$$\frac{I}{I_0} = 10^{-\epsilon lc} \text{ or } D = \log \frac{I_0}{I} = \epsilon lc$$

where

I is the luminous intensity transmitted after passage through the sample;

I_0 is the reference luminous intensity (intensity of the incident light);

ϵ is the extinction coefficient; this depends on the wavelength of the incident light and on the temperature and nature of the absorbent;

c is the concentration of the molecules or ions absorbing the light in the sample;

l is the thickness of the absorbing substance;

D is the optical density.

7.1.2 Calibration of the colorimeter

Using one of the samples taken at the outlet of the injection apparatus, a set of control solutions (at least four) is made having a known dilution, similar to the dilution obtained in the conduit.

For this purpose, a sample of the concentrated injection solution is diluted by a volumetric or gravimetric method with water taken from the conduit upstream of the injection cross-section.

These samples are then tested in the colorimeter, the indications of which are plotted on a graph as a function of the dilution ratio.

It is advisable to use a colorimeter of which the indication is proportional to the optical density.

The homogeneity of the solution injected shall be verified by diluting different samples in an identical manner and analysing them in turn.

7.1.3 Use of sodium dichromate

Sodium dichromate ($\text{Cr}_2\text{O}_7\text{Na}_2 \cdot 2\text{H}_2\text{O}$) is commonly used for the application of dilution methods. It has a high

solubility in water (at least 600 g/l) which is a function of the temperature. It complies with most of the conditions specified in clause 5 of part I.

7.1.3.1 ANALYSIS

Colorimetric analysis permits the measurement of concentrations of the sodium dichromate which may reach 0,2 mg/l using a reagent of the ion Cr^{6+} which is diphenylcarbazide. This reagent may be used in the following form :

- a) diphenylcarbazide (crystalline) $(\text{C}_6\text{H}_5\text{NH.NH})_2\text{CO}$: 0,25 g
+ high purity acetone (CH_3COCH_3) : 100 ml
- b) diphenylcarbazide (crystalline) $(\text{C}_6\text{H}_5\text{NH.NH})_2\text{CO}$: 0,25 g
+ phthalic anhydride (crystalline) $(\text{C}_6\text{H}_4\text{CO})_2\text{O}$: 4,0 g
+ 95 % (V/V) ethanol ($\text{C}_2\text{H}_5\text{OH}$) : 100 ml

To 50 ml of the sample for analysis, sufficient sulphuric acid (H_2SO_4) is added to obtain a solution with a pH of approximately 2,2, followed by the selected reagent.

The action of this reagent is sufficiently rapid in an acid medium for the colorimetric measurement to be possible approximately 10 min after its introduction.

The time lag to be observed between the introduction of the reagent and of the acid, and the point at which the colorimetric analysis is carried out, shall be the same for all the samples and all the control solutions.

The colorimeter shall be adjusted for a maximum extinction to a wavelength of 540 nm. The optical density is a linear function of the concentration. Since the dilute sodium dichromate solutions change over a period of time, the analysis shall be carried out within a few hours after the test.

Should a delay of several days be unavoidable between taking the samples and preparing the control dilutions and analysing them, it is advisable to take the following precautions bearing in mind the risk of instability of sodium dichromate solutions :

- the sample shall be kept away from light,
- at least three control solutions of a dilution approximately equal to that of the samples taken shall be made on the spot and then kept under exactly the same conditions as all the other samples.

Any error arising from variations of the samples over a period of time can be evaluated by comparing the control solutions of the same concentrated solution prepared at different times. This precaution is particularly recommended when the water contains organic matter likely to reduce the sodium dichromate.

7.1.3.2 EXAMPLE FOR CALCULATING THE ERROR ON THE DILUTION RATIO

In this example, it is assumed that the measurement is carried out by injecting a flow rate $q = 20,73 \text{ cm}^3/\text{s}$ of a solution of sodium dichromate with a concentration approximating 125 g/l. Ten samples are taken downstream in a cross-section of conduit where homogeneity of tracer is assumed to be better than 1 %. For each sample the ratio of the concentration to the concentration of the injected solution is determined by colorimetric analysis according to the following procedure :

- dilution with water from the conduit, of a sample of the injected solution, collected at the time of measurement, to obtain seven control solutions, the concentrations of which are respectively 5, 6, 7, 8, 9, 10 and 11×10^{-6} times the concentration of the injected solution,
- treatment of the collected samples and control solutions with diphenylcarbazide reagent and passage through a colorimeter strictly according to the above-defined method. The readings with the colorimeter (measurement of the displacement of a neutral optical density glass wedge interposed across the light path through the solution to be analysed and illuminating a photocell to maintain the zero setting of the point of a galvanometer connected differentially between the photocell and a reference cell) were the following :

Control solutions :

Dilution ratio $n \times 10^{-6}$	Reading graduation
0	80,5
5	115
6	122
7	129
8	136
9	142,5
10	149,5
11	157

Collected samples :

No.	Reading graduation
1	130,75
2	131
3	131
4	132
5	132,5
6	132,5
7	132
8	132,5
9	132,5
10	132,5

The analysis of the control solutions makes it possible to plot the response curve of the colorimeter to dilution, from which the required concentration ratio is obtained by plotting the readings obtained by the analysis of the collected samples.

To determine the tolerance of the result obtained it is assumed that the method of analysis is strictly abided by and that the only errors to be considered are those affecting the determination of the dilution ratios of the control solutions and the colorimeter readings.

a) *Error on the response curve of the colorimeter*

The theoretical distribution of the variations of the measured points with respect to the theoretical curve may be derived from the theoretical distribution of the errors on the dilution ratio and colorimeter readings.

- Error on dilution ratios n

The control solutions were obtained by diluting by means of 25 and 20 cm^3 pipettes and 500 cm^3 flasks to ratios of 25/500, then 20/500 and by delivering, with a microburette, adequate volumes of this first solution into samples of 20 cm^3 of conduit water.

- Standard deviation on the volumes measured with 20 cm^3 pipette : 0,07 %
- Standard deviation on the volumes measured with 25 cm^3 pipette : 0,06 %
- Standard deviation on the volumes measured with 500 cm^3 flask : 0,06 %
- Standard deviation on the volumes delivered by the microburette : 0,25 %

Hence, by quadratic combination, the tolerance on the dilution ratios n :

$$\frac{2 \sigma_n}{n} = 2 \left[\left(\frac{0,07}{100} \right)^2 + \left(\frac{0,06}{100} \right)^2 + \left(\frac{0,06}{100} \right)^2 + \left(\frac{0,06}{100} \right)^2 + \left(\frac{0,25}{100} \right)^2 \right]^{1/2}$$

$$= 0,56 \%$$

- Error on colorimeter readings c

The judicious use of the "zero method" eliminates systematic precision errors and takes account of only random sensitivity and reading errors which, for normal equipment, are of about 1 graduation of the displacement knob of the glass wedge.

The corresponding tolerance, expressed in terms of dilution ratio, is :

$$2 \sigma_c = \frac{11}{76,5} \times 10^{-6} = 0,15 \times 10^{-6}, \text{ i.e. } 1,25 \text{ to } 3 \%$$

in relative value according to the value of the achieved dilution ratio.

– Tolerance on response curve

The scattering about the theoretical curve is the combination of the scatter due to the dilution processes and colorimeter readings.

If a very great number of measurements is carried out, 95 % of the points observed will be at a distance from the theoretical curve (in terms of dilution ratio) less than :

$$2 \sigma_r = 2 \sqrt{\sigma_n^2 + \sigma_c^2}$$

i.e. for a dilution ratio of $7,5 \times 10^{-6}$, in an interval of :

$$7,5 \times 10^{-6} \pm 2 \sigma_r \text{ or } \boxed{(7,5 \pm 0,16) 10^{-6}}$$

– Validity test of the assumptions made for experimental points

The straight line of the "least squares" of the eight experimental values has the following equation :

$$n = 0,144 c - 11,53$$

where

n is the dilution ratio expressed in 10^{-6} ;

c is the colorimeter reading.

An estimation of the "linked standard deviation" of the distribution of experimental points about the straight length may be made from the deviations between experimental values of n and calculated values of equation (2) for experimental values of c , i.e. $0,183 \times 10^{-6} = s_{nc}$.

For a colorimeter reading $c_0 = 132,5$ (No. 5 sample), the 95 % confidence interval of the dilution ratio is therefore :

$$n_0 \pm s_{nc} \frac{t}{\sqrt{N-2}} \sqrt{1 + \frac{(c_0 - c)^2}{\sigma_c^2}}$$

where

$$n_0 = 0,144 c_0 - 11,53$$

t is the value of the Student variable for $(N-2)$ degrees of freedom;

c and σ_c^2 are respectively the mean and variance of N colorimeter readings used for calibrating the colorimeter, i.e.

$$7,55 \pm 0,183 \frac{2,44}{\sqrt{6}} \times \sqrt{1,025} \text{ or } \boxed{(7,55 \pm 0,184) 10^{-6}}$$

a value very near that found above, which justifies the assumptions and constitutes a strong presumption for the good performance of the colorimetric analysis.

b) Tolerance on the value of the dilution ratio of a collected sample

This tolerance results from the combination of the colorimeter reading relating to the sample and the error due to the plotting on the experimental curve, i.e. :

$$2 \sigma_n = 2 \sqrt{(0,092 \times 10^{-6})^2 + (0,075 \times 10^{-6})^2} \\ = 0,24 \times 10^{-6}$$

$$\text{that is } \frac{0,24}{7,5} = 3,2 \%$$

c) Tolerance on the mean value of the dilution ratio of ten collected samples

The tolerance found above is that which corresponds to one single sample. The tolerance on the mean dilution as obtained from several samples will be less. However, it should be noted that variations in the colorimeter reading for the ten collected samples can be due not only to the reading error of the device but also to deviations of the mixture quality in the conduit, of the injection rate constancy, of the stability of the various collected samples between sampling and analysis periods, etc. When these are negligible, the tolerance on the mean dilution ratio of ten collected samples will be :

$$2 \sigma = 2 \sqrt{(0,092 \times 10^{-6})^2 + \frac{(0,075 \times 10^{-6})^2}{10}} \\ = 0,18 \times 10^{-6}$$

$$\text{that is } \frac{0,18}{7,5} = 2,4 \%$$

7.1.3.3 REOXIDATION METHOD

While the risk of tracer reduction shall always be recognized, it does not occur frequently in practice. Previous chemical analysis of the water allows this possibility to be evaluated.

When reduction is possible, the reoxidation method is recommended, since it eliminates this source of systematic error.

7.1.3.4 WHEN THE WATER CONTAINS MATTER IN SUSPENSION

If the water is not perfectly clear and contains solid matter in suspension, several cases have to be taken into consideration :

a) If simple decanting is sufficient to clarify the samples, the control dilutions made with the water from the conduit and the samples taken from identical containers are allowed to settle for equal periods of time until satisfactory clarity is obtained;

b) if decanting is not sufficient to clarify the water, it is then possible to filter the samples and control dilutions under vacuum using plastic filters with a porosity of $1 \mu\text{m}$. It is then necessary to use one filter per sample