

INTERNATIONAL  
STANDARD

**ISO**  
**9555-1**

First edition  
1994-08-15

---

---

**Measurement of liquid flow in open  
channels — Tracer dilution methods for  
the measurement of steady flow —**

**Part 1:**  
**(General)**

ISO 9555-1:1994

<https://standards.iteh.ai/catalog/standards/iso/1986-07-15/9555-1-1994>  
*Mesure de débit des liquides dans les canaux découverts — Méthodes  
de dilution en régime permanent utilisant des traceurs —*

*Partie 1: Généralités*



Reference number  
ISO 9555-1:1994(E)

## Contents

	Page
1 Scope .....	1
2 Normative reference .....	1
3 Definitions .....	1
4 Symbols and units .....	2
4.1 Symbols .....	2
4.2 Subscripts .....	3
4.3 Units of measurement .....	3
5 Principles of the different dilution methods .....	3
5.1 Constant rate injection method .....	3
5.2 Sudden injection method .....	4
6 Characteristics common to both methods .....	4
6.1 General formula for the calculation of discharge .....	4
6.2 Separate determination of tracer concentrations in the channel and in the injection solution .....	4
6.3 Direct determination of the dilution factor .....	5
6.4 Influence of background — Clarification .....	7
6.5 Accuracy and limitations of the dilution method .....	8
7 Choice of tracer .....	9
7.1 Characteristics required of a measuring tracer .....	9
7.2 Criteria for comparison .....	9
8 Choice of measuring reach .....	10
8.1 General considerations on the choice of a site .....	10
8.2 Preliminary tests and calculations .....	11
9 Procedure for the constant rate injection method .....	17
9.1 Preparation of the injection solution .....	17

© ISO 1994

All rights reserved. Unless otherwise specified, no part of this publication may be reproduced or utilized in any form or by any means, electronic or mechanical, including photocopying and microfilm, without permission in writing from the publisher.

International Organization for Standardization  
Case Postale 56 • CH-1211 Genève 20 • Switzerland

Printed in Switzerland

<b>9.2</b>	Injection of the solution .....	<b>17</b>
<b>9.3</b>	Measurement of injection rate .....	<b>17</b>
<b>9.4</b>	Sampling .....	<b>21</b>
<b>10</b>	Procedure for the sudden injection method .....	<b>21</b>
<b>10.1</b>	Preparation of the injection solution .....	<b>21</b>
<b>10.2</b>	Determination of the injected volume .....	<b>21</b>
<b>10.3</b>	Injection of the solution .....	<b>21</b>
<b>10.4</b>	Sampling .....	<b>22</b>
<b>11</b>	Comparison between the two dilution methods .....	<b>28</b>
<b>11.1</b>	General .....	<b>28</b>
<b>11.2</b>	Injection of tracer .....	<b>28</b>
<b>11.3</b>	Sampling and analysis of results .....	<b>28</b>
<b>12</b>	Estimation of uncertainties .....	<b>28</b>
<b>12.1</b>	General .....	<b>28</b>
<b>12.2</b>	Uncertainties due to different kinds of error .....	<b>29</b>
<b>12.3</b>	Sources of systematic error .....	<b>29</b>
<b>12.4</b>	Estimation of systematic uncertainty .....	<b>30</b>
<b>12.5</b>	Computation of random uncertainties .....	<b>30</b>
<b>12.6</b>	Computation of the total uncertainty .....	<b>32</b>
<b>12.7</b>	Working examples of computation of uncertainties .....	<b>32</b>
<b>Annexes</b>		
<b>A</b>	Standard dilutions using pipettes and flasks .....	<b>38</b>
<b>B</b>	Standard dilution ranges obtained with a micrometer-type microburette .....	<b>40</b>
<b>C</b>	Analysis for variance as a test of non-randomness .....	<b>42</b>
<b>D</b>	Bibliography .....	<b>44</b>

iTeh STANDARD PREVIEW  
(standards.iteh.ai)

<https://standards.iteh.ai/catalog/standards/sist/f60cb3f8-fb32-4074-91a5-710f4205aba3/iso-9555-1-1994>

## Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

International Standard ISO 9555-1 was prepared by Technical Committee ISO/TC 113, *Hydrometric determinations*, Subcommittee SC 4, *Dilution methods*.

ISO 9555 consists of the following parts, under the general title *Measurement of liquid flow in open channels — Tracer dilution methods for the measurement of steady flow*.

- Part 1: *General*
- Part 2: *Radioactive tracers*
- Part 3: *Chemical tracers*
- Part 4: *Fluorescent tracers*

Annexes A, B and C form an integral part of this part of ISO 9555. Annex D is for information only.

## Introduction

The former standard series ISO 555 was subdivided into parts on the basis of the method of field measurement, i.e. constant-rate injection method and integration (sudden injection) method. Since the choice of the type of tracer to be used in a field measurement will often depend on the expertise and the laboratory facilities available, this new series of standards ISO 9555 is divided into parts based on the type of tracer used. This revision has enabled the unnecessary repetition of text of the various parts to be avoided and will, it is hoped, prove to be a more convenient form of presentation for the user.

ISO 9555 deals with the measurement of steady flow in open channels by dilution methods using tracers. The methods described may also be applied to the measurement of slowly varying flow, but they may only be used when flow conditions ensure adequate mixing of the injected solution throughout the flow.

For the measurement of very large flows, tracer methods can be onerous in terms of tracer costs and measurement times. However, the use of tracers often reduces danger to personnel during flood periods.

ISO 9555-1 presents the general principles of the methods of constant-rate injection and integration (sudden injection). ISO 9555-2, ISO 9555-3 and ISO 9555-4 deal with the specific aspects of the use of radioactive, chemical and fluorescent tracers, respectively, as well as specific analytical procedures.

This approach has been adopted for the following reasons:

- to facilitate subsequent updating, additions or revisions which concern only ISO 9555-2, ISO 9555-3 or ISO 9555-4;
- to provide a more practical document for the user, who is often obliged to choose the tracer best suited to the available analytical equipment.

**iTeh STANDARD PREVIEW**  
**(standards.iteh.ai)**

This page intentionally left blank

[ISO 9555-1:1994](#)

<https://standards.iteh.ai/catalog/standards/sist/f60cb3f8-fb32-4074-91a5-7f0f4203aba3/iso-9555-1-1994>

# Measurement of liquid flow in open channels — Tracer dilution methods for the measurement of steady flow —

## Part 1: General

### 1 Scope

This part of ISO 9555 concerns principles of the constant rate injection method and the sudden injection method (integration method), and the principles of injection, sampling and analysis which apply for all types of tracer.

It is important to note that the tracers used and their concentrations should be compatible with the legislation of each country regarding the occasional and short-duration discharges of foreign substances into natural waters. They should also take into account possible short-term or long-term influences on flora and fauna, both natural and domestic. In the absence of appropriate regulations, the World Health Organization's (WHO) *Guidelines for drinking water quality* should serve as a guide, taking account of the brief nature of the measurement and of the use that is to be made of water receiving the injected tracer.

### 2 Normative reference

The following standard contains provisions which, through reference in this text, constitute provisions of this part of ISO 9555. At the time of publication, the edition indicated was valid. All standards are subject to revision, and parties to agreements based on this part of ISO 9555 are encouraged to investigate the possibility of applying the most recent edition of the standard indicated below. Members of IEC and ISO

maintain registers of currently valid International Standards.

ISO 772:1988, *Liquid flow measurement in open channels — Vocabulary and symbols*.

### 3 Definitions

For the purposes of this part of ISO 9555, the definitions given in ISO 772 and the following definitions apply.

**3.1 tracer:** Ion, compound or radionuclide introduced into a flow system to follow the behaviour of some component of that system. It is necessary that the tracer, which can be observed, behave in exactly the same fashion as the component to be followed, whose behaviour cannot easily be observed.

**3.2 dilution method:** Method in which the discharge is deduced from the determination of the ratio of the concentration of the tracer injected to that of the tracer at the sampling cross-section (see also 3.9).

**3.3 constant rate injection method:** Method of measuring the discharge in which a tracer is injected at a constant and known rate at one cross-section and its dilution is measured at another section downstream where complete mixing has taken place and the concentration plateau has been reached.

**3.4 sudden injection method; integration**

**method:** Method of measuring the discharge in which a known volume of tracer is injected over a short time at one cross-section and its dilution is measured at another cross-section downstream where complete mixing has taken place, over a period sufficient to allow all the tracer to pass that cross-section, so that the mean dilution of tracer during this period can be determined.

**3.5 multiple injection:** Simultaneous injection of tracer at several points in the injection cross-section, with the aim of improving transverse mixing in a given measuring reach.

**3.6 constant level tank:** Equipment for the injection of the concentrated solution at constant rate, in which, using an overflow weir, a constant head is maintained above a selected nozzle or orifice.

**3.7 Mariotte vessel:** Equipment for injection of the concentrated solution at constant rate. The constant rate injection is achieved by means of an airtight vessel that is provided with an orifice plate or nozzle at its bottom portion. The liquid flows through the restriction and air enters the vessel through a tube, maintaining atmospheric pressure at the lower end of the tube, set at a determined height above the restriction. The head on the restriction and consequently the discharge from the vessel remain constant, independently of the level of the liquid in the vessel.

**3.8 floating siphon:** Equipment for injection of the concentrated solution at constant rate. The solution is taken from a vessel by means of a siphon that is fixed on a float. The lower end of the siphon is fitted with an orifice plate or nozzle. The head on the restriction and consequently the discharge from the vessel remain constant, independently of the level of the liquid in the vessel.

**3.9 dilution ratio; dilution factor**

(1) For the constant rate injection method, the ratio between the concentration of the injected tracer solution and the concentration of added tracer detected at the sampling cross-section when steady conditions have been reached.

(2) For the sudden injection method, the ratio between the concentration of the injected tracer solution and the mean, over the sampling period, of the concentration of added tracer detected at the sampling cross-section.

**3.10 sampling cross-section; sampling station:**

Cross-section of an open channel downstream of the injection cross-section, at which samples are taken or in which the concentration is directly measured.

**3.11 reach:** Length of open channel between two defined cross-sections.

**3.12 degree of mixing:** Measure of the extent to which mixing has been achieved in a cross-section downstream of the injection of tracer, varying from near zero in a cross-section immediately downstream of the injection to 100 % at a cross-section in which the tracer has been completely mixed across the whole cross-section.

**3.13 mixing length:** Distance, measured along the general path of flow, between the injection cross-section and the downstream cross-section at which the specified degree of mixing is obtained. For given conditions, this mixing length is not a fixed value: it varies according to the admissible values for the degree of mixing. The higher the specified degree of mixing, the longer the mixing length.

**4 Symbols and units****4.1 Symbols**

The symbols used in this part of ISO 9555 are given below. Other symbols not listed here are used only in passing and are defined where used.

Symbol	Explanation
$\bar{b}$	Average stream surface width between the injection and sampling cross-sections
$c_0$	Background concentration of tracer
$c_1$	Concentration of tracer in injection solution
$c_2$	Concentration of added tracer
$c'_1$	Concentration of diluted injection solution ( $c'_1 = c_1/D'$ )
$c'_{ii}$	Relative concentration corresponding to standard dilution $D'$
$D$	Dilution factor
$D'$	Estimated dilution factor used in dilution of injection solution
$D'_i$	Standard dilution
$\bar{d}$	Average depth of flow between injection and sampling cross-sections
$E$	Transverse mixing coefficient
$G$	Instrument response
$g$	Acceleration due to gravity
$L$	Mixing length



Symbol	Explanation
$M, m$	Mass
$m$	Number of sampling points
$n$	Number of samples or measurements
$Q$	Stream discharge
$Q'$	Estimated stream discharge
$q$	Injection rate
$r_h$	Mean hydraulic radius
$S$	Energy slope
$T_c$	Duration of steady state concentration
$T_i$	Duration of injection
$T_p$	Time of passage of tracer through the sampling cross-section
$t$	Elapsed time, usually taking as the origin the start of injection
$t_a$	Time of arrival of first molecule of tracer at a given sampling cross-section
$t^*$	Mean time of travel
$V$	Volume of tracer solution injected
$\bar{v}$	Mean velocity
$v^*$	Shear velocity, equal to $(gr_h S)^{1/2}$
$x$	Degree of mixing
$\epsilon$	Level of accuracy of analysis
$\theta$	Lowest temperature likely to be met during gauging

## 5 Principles of the different dilution methods

The basic principle of dilution methods is the addition of a suitably selected tracer to the flow. Downstream of the injection point, when vertical and lateral dispersion throughout the flow is complete, the discharge may be calculated from the measurement of the dilution of the tracer.

If this tracer, or a substance with analytical characteristics similar to the injected tracer, was present in the flow before the injection, the increase in concentration of tracer due to the injection is known as the "concentration of added tracer". The methods set out in this part of ISO 9555 permit the calculation of the discharge regardless of the prior concentration of tracer, analogous with background noise, as long as this can be assumed constant during the measurement.

### 5.1 Constant rate injection method

A solution of concentration  $c_1$  of a suitably chosen tracer is injected at a constant rate  $q$  into a cross-section located at the beginning of the measuring reach of the channel, in which the discharge  $Q$  remains constant for the duration of the gauging.

At a second cross-section downstream of this reach, at a sufficient distance for the injected solution to be uniformly diluted, the concentration is measured for a sufficient period of time and at a sufficient number of points to ensure that good mixing has been obtained and that the concentration of added tracer  $c_2$  has attained a constant value. Under these conditions, if all of the tracer injected passes through the sampling cross-section, the discharge rate of tracer at the injection points is equal to that passing through the sampling cross-section:

$$qc_1 = (Q + q)c_2$$

Hence:

$$Q = q \frac{c_1 - c_2}{c_2} \quad \dots (1)$$

In general  $c_1$  is much greater than  $c_2$  and this leads to a simplification of equation (1) as follows:

$$Q = q \frac{c_1}{c_2} \quad \dots (2)$$

The discharge  $Q$  can therefore be determined by comparing the concentration of the injection solution with that measured at the sampling cross-section of the channel.

## 4.2 Subscripts

c	Concentration
z	Exponent

### NOTES

1 A bar (–) is used above the symbol to indicate an averaging process; further averaging is indicated by a double bar (≡).

2 For convenience in the use of this part of ISO 9555, some of the symbols and their definitions are not consistent with those defined in ISO 772.

## 4.3 Units of measurement

The units of measurement used in this part of ISO 9555 are those of the International System of Units (SI).

## 5.2 Sudden injection method

A volume  $V$  of a solution of concentration  $c_1$  of a suitably chosen tracer is injected over a short period into a cross-section located at the beginning of the measuring reach of the channel, in which the discharge  $Q$  remains constant for the duration of the gauging.

At a second cross-section downstream of this reach, at a distance sufficient for the injected solution to be uniformly diluted, the concentration of tracer  $c_2$  is determined over a period of time sufficiently long to ensure that all the tracer has passed through the second cross-section.

If all the tracer injected passes through the sampling cross-section, the following equation is true:

$$M = Vc_1 = Q \int_{t_a}^{\infty} c_2(t) dt \quad \dots (3)$$

where

- $M$  is the mass of tracer injected;
- $V$  is the volume of injected solution;
- $c_1$  is the concentration of tracer in the injected solution;
- $Q$  is the discharge in the channel;
- $c_2(t)$  is the concentration of added tracer at the fixed sampling point over the time interval  $dt$ ;
- $t$  is the elapsed time, taking as origin the instant at which the injection started;
- $t_a$  is the time of arrival of the first molecule of tracer at the sampling cross-section.

This equation requires that the value of the integral

$$\int_{t_a}^{\infty} c_2(t) dt$$

be the same at every point of the sampling cross-section. This condition is satisfied if and only if the injected solution is well mixed with the water in the channel.

In practice, the presence of the tracer is no longer detectable at any point in the sampling cross-section after a certain time ( $t_a + T_p$ ). The value  $T_p$  is known as the "time of passage of the tracer cloud through the sampling cross-section".

Let

$$\bar{c}_2 = \frac{1}{T_p} \int_{t_a}^{t_a + T_p} c_2(t) dt$$

The practical condition of good mixing is that  $\bar{c}_2$  is identical at all points of the section, hence

$$Q = \frac{Vc_1}{T_p \bar{c}_2} \quad \dots (4)$$

$V/T_p$  is the mean discharge of added tracer in the sampling cross-section, and is analogous with the injection rate  $q$  defined in 5.1.

## 6 Characteristics common to both methods

### 6.1 General formula for the calculation of discharge

Relations (2) and (4) can be written in the general form:

$$Q = kD \quad \dots (5)$$

For measurement by constant rate injection,  $D$  is the dilution factor ( $= c_1/c_2$ ) and  $k = q$ .

For measurement by sudden injection,  $D = c_1/\bar{c}_2$  and  $k = V/T_p$ .

The dilution factor,  $D$ , which is the fundamental concept of the dilution method, is determined by comparative analysis of samples of the injection solution and samples taken from the sampling cross-section.

This determination, although certain aspects are peculiar to the tracer and to the associated method of analysis, rests on several general principles which are presented below. The details of the specific methods of determination for the commonly-used tracers are given in ISO 9555-2, ISO 9555-3 and ISO 9555-4.

### 6.2 Separate determination of tracer concentrations in the channel and in the injection solution

The practice of dilution gauging shows that in most cases the pursuit of maximum effectiveness and convenience leads to a reduction in the total quantities injected, and to a dilution factor,  $D$ , which attains high values, of the order of  $10^3$  to  $10^8$ .

All analytical procedures, whatever the tracer, the apparatus or the method of analysis, are based on the existence of a physical relation between the concentration of tracer and a measurable quantity  $G = G(c)$ , for which the analytical instrument provides a value

$G_i$  corresponding to  $c_i$ . Experience shows that it is difficult, if not impossible, directly to obtain comparable accuracy for independent measurements of concentrations varying in so high a ratio as  $D$  with the same instrument or method of analysis.

### 6.2.1 Direct determination of $c_1$ and $\bar{c}_2$

Procedures for the direct determination of  $c_1$  and  $\bar{c}_2$  are not recommended and thus are not discussed in this part of ISO 9555.

### 6.2.2 Indirect determination of $c_1$ and direct determination of $\bar{c}_2$

Where the analytical method leads to an instrument response which is a linear function of concentration in the range from zero to the usual value of  $c_2$ , the following procedure of indirect determination of  $c_1$  can be used. The solution of concentration  $c_1$  is diluted by a factor  $D'$ , of the same order as  $D$ , to obtain concentrations  $c'_1 = c_1/D'$  and  $c_2$  of the same order of magnitude. This permits use of the same method for the determination of  $c'_1$  and  $c_2$  with comparable accuracy. For this purpose, a sample of injection solution is diluted by the gravimetric method using a precision balance, or by the volumetric method using flasks, calibrated pipettes or microburettes, preferably with water taken from the flow upstream of the injection cross-section or before the test.

This method of determination is a simplified variant of the procedure described in 6.3.

## 6.3 Direct determination of the dilution factor

The method of comparative or standard dilutions is particularly recommended because it minimizes the effects of systematic errors in the determination.

### 6.3.1 Principle

After estimating  $D'$  from the dilution factor  $D$  [using formula (5)] from the test conditions and from the estimate  $Q'$ , however rough, of the discharge  $Q$  to be measured, the method consists of preparing a series of standard dilutions of a sample of the injected solution, having known dilution factors  $D'_1, D'_2, \dots, D'_n$  surrounding the estimated value  $D'$ .

The analytical equipment is then used as a comparator to determine the value of the dilution factor  $D$ . The basic requirements of the equipment are thus sensitivity and stability. The latter is necessary only during

the analysis of samples corresponding to a single flow measurement.

Experience shows that the ratio  $D'/D$  rarely falls outside the range 0,5 to 2. It is therefore possible to use the analytical equipment in the vicinity of its optimum range of sensitivity and accuracy.

Let  $G'_1, G'_2, \dots, G'_n$  be the responses of the analytical equipment for standard dilution factors  $D'_1, D'_2, \dots, D'_n$  and let  $G_1, G_2, \dots, G_n$  be the responses of the analytical equipment for given river samples corresponding to unknown dilutions  $D_1, D_2, \dots, D_n$ .

It is then necessary to obtain the dilutions  $D_i$  corresponding to the responses  $G_i$  by using the relation between  $D'$  and  $G'$  established for the analytical equipment.

This relationship is established either graphically or by using a mathematical function.

As the response of analytical instruments is generally a linear function of concentration, it is logical to use the concept of relative concentration  $c'_{ri} = 1/D'_i$ .

The graphical representation of the relation  $G = G(c_i)$  is a simple means of checking its linearity and the accuracy of preparation of the standard dilutions.

### 6.3.2 Application to measurement by the constant rate injection method

When the gauging is performed in good conditions, the scatter of the  $G_i$  about their mean  $G$  is small, and a small number of standard dilutions suffices to define the response function over the range used ( $D'/2$  to  $2D'$ ).

### 6.3.3 Application to measurement by the sudden injection method

The necessary range of  $G'_i$  varies according to the method of sampling selected (see 10.4.2). In the case of a mean sample (see 10.4.2.3) or several mean samples (see 10.4.2.4), conditions differ little from those of the constant rate injection method.

In the case of discrete samples (see 10.4.2.2) or continuous recording, the concentration  $c_{2i}$  of the samples may vary over a very large range, and it is necessary to determine the response of the analytical equipment by a larger number of standard dilutions distributed over an interval which could extend for example from  $D'/10$  to  $10D'$ .

### 6.3.4 Preparation of standard dilutions

#### 6.3.4.1 Definition

The set of standard dilutions required is defined by the number of standard dilutions, their range and their distribution around the central value.

The range is a function of the accuracy with which field conditions permit the estimation of the dilution factor  $D'$ , which corresponds to the central value<sup>1)</sup>. The set of dilutions will comprise five to seven values, in a sequence approximating a geometric sequence.

For example, using either dilution  $D_i$  ( $= 1/c_{ri}$ ) or relative concentration ( $c_{ri}$ ), most purposes can be served by selecting seven consecutive values from the following reference series, multiplied by a factor of  $10^2$ . The range can be extended by choosing alternate values.

For relative concentrations:

0,8; 1; 1,25; 1,5; 2; 2,5; 3; 4; 5; 6; 8; 10; 12,5

For dilutions:

12,5; 10; 8; 6,4; 5; 4; 3,2; 2,5; 2; 1,6; 1,25; 1; 0,8

The establishment of standard ranges of this type, associated with a detailed laboratory procedure, allows significant savings of time in preparation and analysis in the laboratory or in the field, gives measurements which are more comparable, and aids in the choice of the most appropriate equipment for the smallest suitable number of dilutions.

Two methods can be used to obtain the sets of standard dilutions: the volumetric method and the gravimetric method.

#### 6.3.4.2 Volumetric method

This method uses calibrated glassware (i.e. pipettes, flasks and microburettes) to prepare the required series of standards by successive dilution operations. Essential precautions for obtaining suitable accuracy by this method are as follows:

- select, from the apparatus conforming to the relevant standards, that which is most convenient to use and provides an accuracy appropriate to the problem;
- avoid in particular pipettes of volume less than 10 ml and flasks of volume less than 250 ml.

One-mark bulb-type pipettes, which are most easily handled, are preferred;

- follow scrupulously the recommendations for use laid down in the relevant standards;
- maintain all glassware in a perfect state of cleanliness and discard any equipment which is chipped or stained.

#### 6.3.4.2.1 Use of pipettes and flasks only

This is the least sophisticated and the least expensive procedure, but it is not always possible to obtain conveniently the values proposed for the reference series. A guide to the procedure and a list of recommended equipment is presented in annex A.

To obtain the best results, the following additional recommendations are useful:

- one series of pipettes and flasks should be clearly marked and reserved for the initial dilution operations which involve the highest concentrations and the greatest risks of persistent contamination, despite washing;
- each pipette should be rinsed several times with the solution which it is to deliver;
- it is recommended that tests of the effectiveness of cleaning procedures be performed regularly, for example by rinsing supposedly clean equipment with distilled water and examining for tracer in the rinse water;
- the work plan of the laboratory should be rigorously organized to separate the zones of preparation of samples, for first dilutions of intermediate concentration and for standard dilutions, to avoid accidental contamination.

Disadvantages of this method are

- the inconvenience of its use in the field;
- in the case of large dilution factors, the number of successive dilutions necessary, which prolongs the operation and leads to a possible loss of accuracy.

The advantage of this method is that the equipment can be easily obtained at moderate cost, even for equipment of the highest accuracy.

1) This range is also dependent on the method used (see 6.3.2 and 6.3.3)



Although the methods set out in 6.3.4.2.2 and 6.3.4.3 are preferred, it is desirable to have the necessary equipment for the application of this method in emergencies.

### 6.3.4.2.2 Microburettes

Microburettes (also known as microsyringes) consist of a cylinder containing a piston which is driven by a manually controlled micrometer device (hand-operated type) or by an electromechanical device; some types permit the delivery of volumes adjustable continuously from 0,01 ml to 0,5 ml with an accuracy better than 0,5 %. Thereby it is possible to obtain very conveniently suitable ranges of standards distributed around the reference dilution  $D'$ , by injecting directly into a series of beakers containing for example 50 ml of river water. Under these conditions and depending on the final accuracy required, it may be necessary to carry out a strength correction of the dilution due to the addition of the volume  $V$  delivered by the microburette into the reference volume,  $V_{\text{ref}}$ , of the standard dilution,  $[V/(V_{\text{ref}} + V)]$  instead of  $V/V_{\text{ref}}$ .

It is possible, using for the first dilution either a pipette and a flask or another microburette, to obtain dilution factors of  $10^6$  or  $10^7$  with only two successive dilution operations.

The advantages of microburettes are:

- a reduction in the amount of glassware necessary, in the length of the dilution operations and in the quantities of background water necessary for the standard dilutions;
- a consistent accuracy, practically independent of the dilution factor. Moreover, their compact size permits their use in the field.

As in the use of the pipette-flask procedure, it is possible to define predetermined ranges of standards, in order to distribute the dilutions evenly around the estimated value, avoiding the loss of time caused by calculation for each analysis and limiting the necessary glassware (see annex B).

### 6.3.4.3 Gravimetric method

This method uses similar glassware, but does not rely on its precise calibration, the dilution factor of each successive operation being determined by weighing on a precision balance. The sequence of operations is described below:

- a) Determine the volume  $V$  of the required diluted solution and the volume  $V_1$  of the solution to be

diluted considering the desired dilution factor  $D = V_1/V$ .

- b) Fill a small vessel, e.g. an ampoule or syringe, of suitable capacity with a volume approximately equal to  $V_1$  of the solution to be diluted, seal the ampoule and weigh it: its mass is  $m_2$ .
- c) Weigh an empty flask of volume  $V_0$ ; its mass is  $M_1$ . Fill it partly with the diluent water, then add to it the contents of the ampoule.
- d) Top up to volume  $V_2$  with the diluent water and weigh: its mass is  $M_2$ .
- e) Weigh the ampoule with its residue: its mass is  $m_1$ .
- f) Calculate the exact value of the dilution factor by the relation:

$$D'_i = \frac{M_2 - M_1}{m_2 - m_1}$$

This process should be repeated for each dilution operation.

It is necessary to use a highly accurate balance for the weighing operations. Such equipment is costly, but the gravimetric method can give very accurate results for large dilution factors. This method is particularly recommended for radioactive tracers, because it permits remote handling and with these tracers the number of standard dilutions is much reduced.

## 6.4 Influence of background — Clarification

### 6.4.1 Background noise

If the conditions of preparation of the standard dilutions  $D'_i$  are such that they are strictly comparable with the samples taken from the sampling cross-section, there is no need to take account, in calculation of the discharge, of the initial concentration  $c_0$  of tracer, or of a substance having similar analytical characteristics, existing in the flow before the addition of  $c_2$ , as long as it can be considered constant over the duration of the measurement. It is sufficient if the standard dilutions are prepared with water taken from the flow to be measured, either upstream of the point of injection during the measurement, or in the sampling cross-section before the measurement. The procedure recommended in 6.3 permits the calibration of the measuring instrument, taking into account implicitly the effect of  $c_0$ .

A sample of background water with no tracer added may be used as a blank, which should be treated in

exactly the same way as the other samples to be analysed; it is then possible to estimate the overall "background noise" of the analysis, which may partially be caused in particular by the presence of tracer in the flow.

This operation is essential where standard dilutions have not been made up with water taken from the flow. However, as this blank is usually equivalent to a very high value of the dilution factor, the reading corresponding to it is far outside the working range adopted for the determination of  $D$ . A significant risk of inaccuracy can thus result and this reading cannot be used in the determination of the response function  $G' = G'(c')$ . This result is nevertheless a useful element in the interpretation of the analysis.

#### 6.4.2 Sample pretreatment (clarification of samples and standard dilutions)

Where samples are brought from the field to the laboratory for analysis, certain tracers may be subject to losses caused by adsorption onto organic or inorganic sediments. Adsorption problems encountered in the use of specific tracers are detailed in ISO 9555-2, ISO 9555-3 and ISO 9555-4. However, the practice of decantation and filtration recommended applies to any tracer, and is outlined in this subclause.

Adsorption onto sediment is most likely to be a problem with finely-divided suspended material, which has a high surface area. If the river water contains suspended solid matter, several remedies may be considered.

- a) A simple decantation may be sufficient to clear the samples. In this case the standard dilutions shall be made up with background water carefully agitated beforehand. The flasks containing samples and standard dilutions shall be of identical shape, and the decantation times shall also be identical.
- b) If decantation does not suffice to clear the water, samples and standard dilutions prepared with turbid background water may be filtered, as soon as possible after collection, using a portable vacuum filtration unit or syringe-mounted filters. Glass-fibre filters capable of removing all material larger than  $2\ \mu\text{m}$  are suitable for general use; smaller particles, down to  $0,45\ \mu\text{m}$ , can be removed by membrane filters. It is necessary to change the filter if it becomes clogged, or if samples of different concentrations are to be filtered, and to saturate the filter with the solution to be filtered before each operation. For this purpose a certain quantity of the solution to be analysed is

filtered and discarded, only the liquid subsequently filtered being retained for measurement.

- c) If the water is laden with colloidal clay material and clogs the filters rapidly, it is possible to accelerate the preliminary decantation by adding to the samples a known volume of a solution of aluminium sulfate for simple cases, or of sodium silicate followed by aluminium sulfate for the more difficult cases.

Preliminary tests will be necessary with all sample treatments to establish that no apparent or real changes in tracer concentration result, for example from uptake or release of tracer by the filter media. It is recommended that samples and standard solutions be treated in a rigorously identical fashion.

#### 6.5 Accuracy and limitations of the dilution method

The general formula for the calculation of discharge

$$Q = kD$$

can be written

$$Q = kD'r_c$$

where

$D'$  is the standard dilution closest to the experimental value  $D$ ;

$r_c$  is a correction coefficient (often close to unity) determined by the measuring instrument.

It is clear that the uncertainty in  $Q$  is the combination of the uncertainties in  $k$ ,  $D'$  and  $r_c$ .

The uncertainty in  $k$  ( $q$  or  $V/T_p$ ) is generally easily reduced by using classical methods of repeated sampling and measurement (see 9.3 and 10.2).

The uncertainty in  $D'$  is one of the factors limiting the field of application of the method. In fact, for a given tracer, injection device and measuring instrument the maximum measurable discharge is proportional to the ratio.

$$D' = \frac{c_{1\ \text{max}}}{c_{2\ \text{min}}}$$

where

$c_{1\ \text{max}}$  is the maximum injectable concentration;

$c_{2\ \text{min}}$  is the minimum measurable concentration.

This ratio can reach values in excess of  $10^7$  with certain tracers. It is necessary therefore to prepare standard dilutions of this order with an accuracy approaching 1 % (gravimetric method or volumetric method followed meticulously, see 6.3.4).

The uncertainty in  $r_c$  depends on the method of analysis, on the instrument and on the concentration  $c_2$ , and it increases as  $c_2$  approaches  $c_{2\text{ min}}$ . It also involves the sensitivity of the analysis, which can be defined as the ratio,  $\Delta G/\Delta c_2$ , of the change in response of the instrument to the change in concentration around the usual values of  $G$  and  $c_2$  analysed.

## 7 Choice of tracer

The tracers to be used in practice shall conform as closely as possible to the ideal tracer, whose characteristics are described in 7.1.

### 7.1 Characteristics required of a measuring tracer

#### 7.1.1 Hydraulic and physicochemical characteristics

A good flow tracer shall have a hydrodynamic behaviour identical with that of molecules of water. This property shall not be altered by a change of chemical form or possibly of phase due to an interaction with the medium, nor by variations of temperature which can occur in the course of the gauging.

When the application of a tracer requires the use of a solvent, the solution obtained shall have a density close to that of the flow, to avoid segregation in the zone around the injection point.

The tracer shall not be retained by parts of the channel (material or vegetation) nor by material in suspension in the flow, in such proportions as to affect the desired accuracy of the measurement.

The tracer shall be stable in very dilute solution in natural waters under the influence of light and temperature, so as to permit a delay in analysis which may arise from the remoteness of certain gauging sites.

The tracer shall have the greatest possible solubility in water, to ensure convenience of use in gauging.

The tracer shall be analysable at the lowest possible concentration, to extend its use to the measurement of large flows.

### 7.1.2 Metrological characteristics

There shall be a well-defined physical or chemical relationship  $G = G(c)$  between a measurable quantity  $G$  and the concentration  $c$  of the tracer in aqueous solution.

This relation shall be stable and precise over a large range of concentration around the usual value  $c_2$  for samples taken from the sampling cross-section.

Examples are: the disintegration rate of a radioactive tracer, the Beer-Lambert Law for tracers analysed by colorimetry, the relation between concentration and conductivity for certain chemical tracers, the intensity of fluorescence for certain dye tracers, etc.

It shall be possible to establish this relation with an instrument which shall combine the following essential qualities:

- high sensitivity to low values of  $c_2$ ,
- high stability over the period of analysis.

It is also desirable that the apparatus be sturdy, proven, easily available and of reasonable cost, and that it can be operated by a technician who is trained but not specialized, and maintained easily and inexpensively.

## 7.2 Criteria for comparison

### 7.2.1 Preliminary note

The choice of tracer depends on whether the aim is to find a tracer and an associated analytical procedure which covers a vast field of application and can be used for a large number of measurements, or on the contrary to find the optimal solution for a particular isolated application.

In these two cases the tracer shall satisfy the criteria enumerated in 7.1, but it is necessary, in making a choice, always to associate three elements: tracer, method of analysis and analytical instrument.

### 7.2.2 Definition of criteria

#### 7.2.2.1 Minimum measurable concentration, $c_{2\text{ min}}$ , at a level of accuracy $\varepsilon$ fixed at a given probability $P$ (for example 95 %)

For each combination of tracer, method of analysis and analytical instrument, the relation  $\varepsilon = f(c_2)$  is es-