
International Standard



78/2

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Layouts for standards — Part 2 : Standard for chemical analysis

Plans de normes — Partie 2 : Norme d'analyse chimique

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (ISO member bodies). The work of developing International Standards is carried out through ISO technical committees. Every member body interested in a subject for which a technical committee has been set up 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.

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.

International Standard ISO 78/2 was developed by Technical Committee ISO/TC 47, *Chemistry*.

This first edition was submitted directly to the ISO Council, in accordance with clause 5.10.1 of part 1 of the Directives for the technical work of ISO. It cancels and replaces pages 11 to 23 of ISO Recommendation R 78-1969, which had been approved by the member bodies of the following countries :

Australia	Hungary	South Africa, Rep. of
Austria	Iran	Spain
Belgium	Israel	Switzerland
Brazil	Italy	Thailand
Cuba	Netherlands	Turkey
Czechoslovakia	New Zealand	United Kingdom
Egypt, Arab Rep. of	Poland	USA
France	Portugal	USSR
Germany, F. R.	Romania	

The member body of the following country had expressed disapproval of the document on technical grounds :

India

It also cancels and replaces ISO Guide 18-1978.

Layouts for standards — Part 2 : Standard for chemical analysis

0.1 Introduction — Drawing up standards according to standard layouts

It is recommended that standards for methods of chemical analysis should be drawn up in accordance with part 3 of the Directives for the technical work of ISO, which gives general guidance, and using the layout of which details are given on the following pages.

It should always be remembered, in making use of this layout, that it is for guidance only. It should be adapted to suit any special requirements. There may be no need for all the subdivisions provided : no account should be taken of those not required.

With the object of helping to solve problems of drafting and layout which arise in drawing up standards for chemical products and for methods of chemical analysis, ISO/TC 47 has established a series of standard layouts :

- layout for a standard for a chemical product, with notes on its application¹⁾;
- layout for a standard method of chemical analysis, with notes on its application²⁾;
- standard layouts for instrumental analyses, with notes on their application³⁾.

A standard for a chemical product should form a consistent whole. In addition to specifying the characteristics required of the product, it should state how to determine these characteristics. A standard method of chemical analysis has a rightful place in the text of a standard for a chemical product. However, to shorten the text, the method of chemical analysis may be issued as a separate standard; and if a general method of chemical analysis applicable to the product exists, the standard for the product may merely refer to it.

The adoption of a standard form of layout and drafting ensures

- that no important point is overlooked in the preparation of the standard;

- that the various items of information to be included in the standard are always given in the same order;
- that any desired clause may be found rapidly, whatever the origin or scope of the standard.

(This is important particularly if a partial translation of a text is being studied or two texts are being compared.)

0.2 Scope and field of application

With a view to the simplification, rationalization and standardization of methods, reagents and equipment used in test laboratories, and so that each International Standard or other international document prepared in this field should be drafted in terms as comprehensible as possible, particularly to readers whose mother tongue is not the language in which the document is written, this part of ISO 78 sets out a number of principles which should be applied for the layout and wording of methods of chemical analysis.

0.3 General

0.3.1 Plan of document

In drafting the methods of analysis, the subjects should be dealt with in the order shown in the layout on page 3 but any clause or sub-clause which may be unnecessary in a particular case should be omitted and others, if required, should be added in the most appropriate places.

0.3.2 Numbering of clauses and sub-clauses

The clauses and sub-clauses from beginning to end of each document, including documents containing several sections each dealing with a different method of test, should be numbered consecutively, using arabic numbers, in accordance with the point system described in ISO 2145, *Documentation — Numbering of divisions and subdivisions in written documents*.

1) The layout for a standard for a chemical product is given in ISO 78/1, *Layouts for standards — Part 1 : Standard for a chemical product* (at present at the stage of draft).

2) i.e. this part of ISO 78 : this cancels and replaces the layout for a standard method of chemical analysis given in ISO/R 78-1969, pages 11 to 23.

3) Namely, ISO 78/3, *Layouts for standards — Part 3 : Standard for molecular absorption spectrometric analysis* (at present at the stage of draft), ISO 78/4, *Layouts for standards — Part 4 : Standard for atomic absorption spectrometric analysis* (at present at the stage of draft) and ISO 2718 (which will become ISO 78/5, *Layouts for standards — Part 5 : Standard for gas chromatographic analysis*).

0.3.3 Units and symbols

0.3.3.1 General

The units, signs and symbols laid down in International Standards¹⁾ or, failing these, those already well known internationally, should be used if possible.

The symbols for units of measurement should be used if they are preceded by a number expressed in figures. In other cases, these units should preferably be written out in full (except where used as column headings in tables of numerical values).

0.3.3.2 Millilitre

The millilitre (ml) may be used as the special name for the cubic centimetre (cm³), in accordance with the decision of the Twelfth General Conference of Weights and Measures (Paris, October 1964).

10.3.3.3 % Sign ("percentage" in English and "pourcentage" in French)

The % sign should be used in all cases where this sign is preceded by figures, unless otherwise indicated (for example, for typographical reasons). It should be followed by the indication (*m/m*) or (*V/V*) depending on whether the percentage is by mass or by volume.

0.3.4 Choice of methods of test

As far as possible and except in certain industries, if this rule would be contrary to justified and well-established practice

which it is desirable to retain, the same test methods for a given characteristic in related chemical products should be adopted in all ISO documents and their wording should be as similar as possible.

0.3.5 Chemical nomenclature

The directives prepared by the International Union of Pure and Applied Chemistry (IUPAC) on the nomenclature of chemicals of high purity and the way of spelling and printing their names should be applied. It is advisable to put the trivial name of the reagent in parentheses after the IUPAC name when it first occurs but, thereafter, only the IUPAC name should be used.²⁾

It is recommended that the use of trade names and registered trade names be avoided, if possible.

For commercial chemicals (basic chemicals for industrial use), the trivial name should be given in the title and in the "Scope and field of application" clause(s); the corresponding IUPAC name for the pure product should be given in parentheses after the trivial name but, thereafter, only the trivial name need be used.

0.3.6 Numerical values and tolerances

Whenever a parameter, for example a temperature or a time period, is quantified, it is important to consider the need to specify a tolerance on its value.

1) See in particular ISO 31, *Quantities, units and symbols*, and ISO 1000, *SI units and recommendations for the use of their multiples and of certain other units*.

2) However, the trivial name of the reagent may be used in the remainder of the text if by so doing the presentation is made more readable.

Preferred order for the layout

(see 0.3.1)

	For comments see clause
Title	1
Warning and safety precautions	2
Introduction	3
Scope	4
Field of application	5
References	6
Definitions	7
Principle	8
Reactions	9
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Numbered
clauses

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Notes on the application of the layout for a standard method of chemical analysis

1 Title

The title of the document should express concisely and without ambiguity the products to which the method of test applies, the constituent or the characteristic to be determined and the nature of the determination.

Example :

Sodium hexafluorosilicate for industrial use — Determination of iron content — 1,10-Phenanthroline spectrometric method.

2 Warning and safety precautions

If the product being analysed or the reagents or the procedure are dangerous, it is essential to draw attention to the hazards and to describe the precautions necessary to avoid them. This information should be printed in capital letters or in bold type

- immediately after the title of the standard if the danger to be encountered is due to the product being analysed;
- after the name of the reagent or material if the danger to be encountered is due to a particular reagent or material;
- as stated in 13.1 if the danger to be encountered is due to the procedure.

This part of the text should not be numbered.

3 Introduction

If special information on the method is required, it should be included in this clause, which is given the number zero (0).

4 Scope

This clause of the document should state succinctly the method of analysis of test and specifically the products to which it applies.

It should contain any additional, useful, information which could not be included in the title.

5 Field of application

This clause should contain sufficient information to enable the user to judge quickly whether the standard is applicable to the products being considered, or whether limitations exist.

It should contain an indication of the products to which the method of analysis applies and the limits of contents between which the method can be used without alteration.

These limits should take into account the presence of the other components of the product or products in question and of their own limiting contents.

If interferences arise in a method, they should be mentioned in this sub-clause. If it is necessary to provide for modifications to the basic method, for instance to ensure the elimination of certain interfering factors, these modifications should be treated as special cases. These special cases should be indicated in the "Field of application" clause and the corresponding modifications should be described in the "Special cases" clause.

NOTES

1 It is sometimes necessary to provide several methods for the determination of a given component, depending, for example, on the composition of the product or on its differing contents of that component, or the accuracy required, each method having its own field of application. A clear distinction should be drawn between the individual fields of application of the methods laid down if the document includes several methods.

2 In simple cases, it may be preferable to amalgamate the clauses "Scope" and "Field of application" into a single clause entitled "Scope and field of application".

6 References

This clause should give a list of ISO documents which are necessary for the application of the International Standard.

NOTE — Documents which have merely served as references in the preparation of the standard should be indicated in the "Bibliography" clause or mentioned in the "Introduction" clause.

7 Definitions

This clause should give any definitions of terms used in the text that may be necessary for its complete understanding.

8 Principle

This clause should indicate the essential steps of the method used (preferably using substantive phrases), the basic principles and the properties of which use is made and, if appropriate, the reasons justifying the choice of certain procedures.

9 Reactions

This clause should indicate the essential reactions, if they are considered necessary for the comprehension of the text or the calculations. These reactions should, if appropriate, be expressed in ionic form.

The reactions are given only for guidance and do not claim to settle any controversial questions. They justify the calculations made from the data obtained in the determinations and may lead to a better understanding of the method, especially if

several successive changes occur in the state of oxidation of the element being determined.

Any additional information required should be given either in a note to this clause or in the clause "Notes on procedure".

10 Reagents and materials

10.1 General

The title of this clause should be "Reagents" or "Materials" or "Reagents and materials", as appropriate. This clause should begin, if applicable with the following sentence :

"During the analysis, unless otherwise stated, use only reagents of recognized analytical grade and only distilled water or water of equivalent purity."

It should give the list of all the reagents and materials used during the test, together with their essential characteristics (concentration, density¹⁾, etc.) and should specify, if necessary, their degree of purity in relation to their specific use. The list should not include products used only in the preparation of these reagents.

If necessary, the precautions to be taken for storing the reagents, and the time for which they may be stored, should be specified.

If a standard volumetric solution or other standard solution is called for, its preparation and, if necessary, its standardization should be described in this clause. In other cases, these particulars are not necessary, but if general methods for the preparation and checking of certain reagents used are the subject of an International Standard, a reference to the latter should be made.

If the absence of an interfering component in the reagents has to be verified, the details of the tests to be used for this purpose should be given.

It is desirable to draw up this clause in the following systematic order :

- products (excluding solutions) used in their commercially available form;
- solutions or suspensions (excluding standard volumetric solutions and standard solutions), with their approximate concentrations stated;
- standard volumetric solutions and standard solutions;
- indicators;
- auxiliary materials (drying agents, etc.).

The reagents and materials should be identified by a serial number. By showing this number, in parentheses, in the "Pro-

cedure" clause after the name of the reagent or material, it is possible, by referring back to the "Reagents and materials" clause, to avoid repeating all the characteristics of the reagent or material in question and thus to keep the text short. It is not, however, necessary to repeat this reference every time if no ambiguity is introduced.

10.2 Products used in their commercially available form

In the list of reagents and materials, products used in their commercially available form should be described unambiguously, giving the particulars necessary for their identification (for example, the chemical formula, concentration, etc.) and, for solid products in particular, the presence of any water or crystallization.

10.3 Aqueous solutions

In the list of reagents and materials, solutions for which the solvent is not specified are aqueous by convention. The use of the term "aqueous" in the list is superfluous.

10.4 Solutions with defined concentration

If a solution has to be prepared to a concentration specified on a volume by volume or mass by volume basis, the temperature at which the volume or volumes are measured should be given wherever measurement at ambient temperature would have a significant effect on the accuracy of the determination.

The following equivalent terms in the official ISO languages [English, French (F) and Russian (R)] should be used.

10.4.1 Standard volumetric solution

F : Solution titrée

R : Титрованный раствор

10.4.1.1 Definition

Solution for titrimetric analysis, the concentration of which is defined accurately.

10.4.1.2 Expression of concentration

The concentrations of these solutions should be expressed as amount-of-substance (molar) concentrations with the unit mole per cubic metre (mol/m^3) or mole per litre (mol/l)²⁾. The concentration shall be represented either by a whole number (for example, 2 mol/m^3 , 1 mol/l) or by a decimal number (for example, 0,06 mol/m^3 , 0,1 mol/l). In certain cases, the concentration of the solutions may be expressed as mass concentrations, in which case the unit may be, for example, grams per millilitre (g/ml).

1) **density** (symbol ρ) : Quotient of the mass by the volume. (See ISO 31/3, *Quantities and units of mechanics*.)

2) Throughout the rest of this document, only the unit mole per litre (mol/l) is used.

The amount-of-substance concentration of component B (symbol c_B) is defined as the amount of substance of component B divided by the volume of the mixture. (See ISO 31/8, *Quantities and units of physical chemistry and molecular physics*.)

The base SI unit of amount of substance is the mole, defined as follows :

The mole is the amount of substances of a system which contains as many elementary entities as there are atoms in 0,012 kg of carbon 12. When the mole is used, the elementary entities must be specified and may be atoms, molecules, ions, electrons, other particles, or specified groups of such particles.

Thus, when using the molar concentration (amount-of-substance concentration) the elementary entity must be specified.

Examples :

$c(\text{NaOH}) = 1 \text{ mol/l}$ is the molar concentration corresponding to that hitherto described as 1 N, that is containing 40 g of sodium hydroxide per litre. The elementary entity is the molecule of sodium hydroxide.

$c(1/2 \text{ H}_2\text{SO}_4) = 3 \text{ mol/l}$ is the molar concentration corresponding to that hitherto described as 3 N, that is containing $3 \times 49 \text{ g}$ of sulphuric acid per litre. The elementary entity is the half-molecule of sulphuric acid.

$c(\text{H}_2\text{SO}_4) = 1,5 \text{ mol/l}$ is the molar concentration corresponding to that hitherto described as 3 N, that is containing $1,5 \times 98 \text{ g}$ of sulphuric acid per litre. The elementary entity is the molecule of sulphuric acid.

$c(\text{H}_2\text{SO}_4) = 1 \text{ mol/l}$ is the molar concentration corresponding to that hitherto described as 1 M or 2 N, that is containing 98 g of sulphuric acid per litre. The elementary entity is the molecule of sulphuric acid.

$c(1/5 \text{ KMnO}_4) = 0,1 \text{ mol/l}$ is the molar concentration corresponding to that hitherto described as 0,1 N, in the case of reaction in acid medium, that is containing 3,16 g of potassium permanganate per litre. The elementary entity is the one-fifth-molecule of potassium permanganate.

$c(\text{HgCl}) = 1 \text{ mol/l}$ is the molar concentration corresponding to that hitherto described as 1 N, that is containing 236,04 g of mercury(I) chloride per litre. The elementary entity is the half-molecule of mercury(I) chloride.

$c(\text{Hg}_2\text{Cl}_2) = 1 \text{ mol/l}$ is the molar concentration corresponding to that hitherto described as 1 M

or 2 N, that is containing 472,08 g of mercury(I) chloride per litre. The elementary entity is the molecule of mercury(I) chloride.

$c(\text{Hg}_2^{2+}) = 1 \text{ mol/l}$ is the molar concentration corresponding to that hitherto described as 1 M or 2 N, that is containing 401,18 g of the mercury(I) cation per litre. The elementary entity is the mercury(I) cation.

$c(1/2 \text{ Ca}^{2+}) = 1 \text{ mol/l}$ is the molar concentration corresponding to that hitherto described as 1 N, that is containing 20,04 g of the calcium cation per litre. The elementary entity is the half-cation of calcium.

Hitherto, the terms normality and molarity (symbols N and M respectively) were used in this context, and the corresponding expressions in these terms are shown above.

10.4.2 Standard reference solution

F : Solution étalon de référence

R : Образцовый раствор

10.4.2.1 Definition

Solution used as a reference solution for standardizing other solutions. It is either prepared from a primary standard or standardized by some other means.

10.4.2.2 Expression of concentration

The concentrations of these solutions should be expressed in the same way as those of standard volumetric solutions (10.4.1).

10.4.3 Standard solution

F : Solution étalon

R : Эталонный раствор

10.4.3.1 Definition

Solution of accurately known concentration of an element, an ion, a compound or a group derived from the product used for its preparation.

10.4.3.2 Expression of concentration

The concentrations of these solutions should be expressed in grams per litre or in sub-multiples of this unit.

10.4.4 Standard matching solution¹⁾

F : Solution témoin

R : Контрольный раствор

1) This English term is used solely as a generic term for these solutions and each solution should be defined more precisely by the appropriate adjective (for example, "standard colorimetric solution", "standard turbidimetric solution").

10.4.4.1 Definition

Solution of which the relevant characteristic is exactly known or defined (for example colour, turbidity) and is used to assess the test solution in relation to that characteristic. It may be prepared from solutions 10.4.1, 10.4.2, 10.4.3 or other solutions with the required characteristic.

NOTE — The method of preparation of the standard matching solutions should normally be given in the sub-clause "calibration" (see 13.7).

10.4.4.2 Expression of concentration

The concentrations of these solutions should be expressed in accordance with the indications of 10.4.1.2, 10.4.2.2 or 10.4.3.2.

10.4.5 Other solutions

10.4.5.1 If the concentration of a solution is to be given on a mass by mass or volume by volume basis, it should be expressed as a percentage in the following way :

... % (m/m) or ... % (V/V)

10.4.5.2 If the concentration is expressed in terms of the dimensionally heterogeneous units of mass and volume, the concentration should be expressed in grams per litre or, if appropriate, in sub-multiples thereof.

10.4.5.3 If a solution is prepared by dilution of another specified solution, the following conventions should be observed :

- the expression "diluted $V_1 \rightarrow V_2$ " means that the volume V_1 of the specified solution is diluted in such a way as to give a total volume V_2 of final mixture;
- the expression "diluted $V_1 + V_2$ " means that the volume V_1 of the specified solution is added to the volume V_2 of the solvent.

NOTE — Expressions such as " $V_1 : V_2$ " or " V_1/V_2 " which have different meanings in different countries should not be used.

In the same way, traditional or conventional denominations of solutions other than those given above (for example "hydrogen peroxide, 12 volumes") should not be used.

10.5 Indicators

Indicators shall be designated by their systematic chemical names and, if possible, by their Colour Index numbers, and never by any trade name. If diluted indicators are specified, their concentrations shall be given either as percentages by volume in the case of indicators that are liquids or as grams per litre in the case of solutions of solids.

11 Apparatus

This clause should indicate the significant characteristics of the apparatus used during the analysis or the test, other than of usual laboratory apparatus.

The items of apparatus should be identified by a serial number. By showing this number, in parentheses, in the "Procedure" clause after the name of the item, it is possible, by referring back to the "Apparatus" clause, to avoid repeating all the characteristics of the apparatus in question and thus to keep the text short. It is not, however, necessary to repeat this reference every time the item is mentioned if no ambiguity is introduced.

If appropriate, reference should be made to International Standards concerning laboratory glassware and related apparatus.

It is advisable to illustrate special types of apparatus and their assembly by means of a diagram drawn in accordance with the International Standards concerning technical drawings, if these are applicable.

Any checking of the functioning of the assembled apparatus should be described in the "Procedure" clause in a sub-clause headed "Check test" (see 13.4).

The special requirements of any apparatus that is not in common use should be given in this clause, especially if they play a significant part in the procedure or if they constitute a factor in the safety, reproducibility and accuracy of the method.

12 Sampling and samples

12.1 Definitions

The following equivalent terms in the official ISO languages [English, French (F) and Russian (R)] should be used. The following definitions are taken from ISO 6206, *Chemical products for industrial use — Sampling — Vocabulary*.

12.1.1 Laboratory sample

F : Échantillon pour laboratoire

R : Лабораторный образец

A sample as prepared for sending to the laboratory and intended for inspection or testing.

12.1.2 Test sample

F : Échantillon pour essai

R : Образец для испытаний

A sample prepared from the laboratory sample and from which test portions will be taken.

12.1.3 Test portion

F : Prise d'essai

R : Проба для анализа

The quantity of material drawn from the test sample (or, if both are the same, from the laboratory sample) and on which the test or observation is actually carried out.¹⁾

12.2 Sampling

In principle, sampling for the preparation of the laboratory sample is independent of the chemical analysis as such, and it is generally sufficient to refer to the relevant standard dealing specifically with this question, or to the corresponding clause of the product standard. If no texts exist, this sub-clause may include a sampling plan and sampling procedure including guidance on the means of avoiding alteration of the product and taking into account the International Standards concerning the applications of statistical methods.

If necessary, this sub-clause should also give any useful information on the mass or volume of the laboratory sample, the characteristics of the containers for its storage (for example type, capacity, air-tightness) and the storage conditions to be used.

12.3 Preparation of the test sample

This sub-clause should give all the necessary information for the preparation of the sample from which the test portions will be drawn. This sample is prepared from the "laboratory sample" specified in 12.2.

In each case, all the steps in the preparation should be stated (for example, grinding, drying) together with appropriate information on the characteristics of the sample thus prepared (for example particle size distribution, approximate mass or volume) and, if necessary, those of any containers to be used for storage (for example type, capacity, air-tightness) and the storage conditions themselves.

13 Procedure

This clause may be divided into as many sub-clauses as there are operations or sequences of operations to be carried out.

Each sequence of operations should be described accurately, using the imperative mood.

If the number of steps in the procedure is large, it is recommended that the sub-clause covering each step should be further subdivided, using the point numbering system, with each element corresponding to a given analytical operation and including all indispensable preliminary operations.

If the method to be described is already given in another document, the phrase "use the method specified in . . ." or "use

one of the methods specified in . . ." should be used, with an indication of any modification, if necessary.

The "Procedure" clause should normally include the following sub-clauses²⁾.

13.1 Safety precautions

If there are risks during the procedure (for example explosion, fire or toxicity) for which special precautions are necessary, a cautionary statement should be included in this sub-clause in capital letters or in bold type.

If necessary, more detailed advice on safety procedures and first-aid measures can be given in an annex.

13.2 Test portion

This sub-clause should give all the information necessary for the drawing of the test portion from the test sample (or from the laboratory sample, if both are the same) prepared as indicated in 12.3. If appropriate, it should state the method of weighing or measuring this test portion (for example, using a weighing pipette). It should state the mass or volume of the test portion, the precision with which this has to be measured and, if necessary, any other relevant characteristics.

This sub-clause should also indicate if several test portions are required in order to take account of the manner in which the result of the determination is derived.

13.2.1 Test portion defined by mass

If the test portion is defined by mass, the measurement generally falls into one of the following classes :

13.2.1.1 First case

A definite quantity of the test sample is weighed with the accuracy specified.

Two recommended methods of expression are illustrated by the following example :

Example :

$$"m = 5,000 \pm 0,001 \text{ g}"$$

or

"Weigh, to the nearest 0,001 g, 5,000 g of the test sample."

1) In certain cases, the term "test portion" can be used to describe a product of previous steps in the procedure applied to the original test portion (see 13.2.3).

2) In certain cases, other sub-clauses should be included specifying, for example, the number of replicate determinations, additional tests such as determination of moisture, use of standard sample, etc.

13.2.1.2 Second case

An approximate quantity of the test sample is taken and weighed with the accuracy specified. The recommended method of expression is illustrated by the following example :

Example :

"Weigh, to the nearest 0,001 g, approximately 2 g of the test sample."

In general, the expression "weigh approximately . . . g of the test sample" should be interpreted as signifying that the mass to be taken is within $\pm 10\%$ of the specified mass of the test portion. If a smaller tolerance is required, this should be shown as illustrated by the following example :

Example :

"Weigh, to the nearest 0,001 g, 1,9 to 2,1 g of the test sample."

13.2.2 Test portion defined by volume

If the test portion is defined by a given volume, there are two recommended methods of expression (13.2.2.1 and 13.2.2.2) for indicating the accuracy to which test portions are to be measured.

13.2.2.1 If an apparatus having a known precision is required, its accuracy should be stated in the "Apparatus" clause.

Example :

"4.1 **Pipette.** 50 ml, complying with ISO 648, class A."

The identification number of this apparatus should then be shown in the text of the procedure.

Example :

"Measure 10 ml of the test solution with the pipette (4.1)."

13.2.2.2 If no apparatus is prescribed, the accuracy of the measurement should be indicated in the text of the procedure.

Example :

"Take 10,0 ml of the test solution"

or

"Take $10 \pm 0,05$ ml of the test solution."

13.2.3 Test portion resulting from previous steps in the procedure

Finally, if the test portion is the product of previous steps in the procedure (for example, filtrate, precipitate, residue), its origin should be clearly identified by means of a letter.

Example :

"Solution 'A' — Filtrate 'C' from the determination of . . ."

13.3 Blank test

This sub-clause should indicate all the conditions for carrying out the blank test, if this is necessary to verify the purity of the reagents.

The blank test should be carried out in parallel with the determination, by the same procedure, using the same quantities of all the reagents as in the determination (except in the case of titrimetric methods, when the volumes of the standard solutions will obviously be different), but omitting the test portion.

In certain cases, this absence of the test portion may cause the conditions of the blank solution to differ from those of the test solution and interfere with the application of the method. In such cases, the modifications in the procedure that have to be used in the blank test to allow for these differences, while retaining, if necessary, the same quantities of reagents as in the determination itself, should be clearly laid down in this sub-clause.

13.4 Check test

If it is necessary to make a preliminary check of the apparatus (for example, to check the regularity of combustion in a furnace) or to verify the validity of the application of the method by using a certified reference material, synthetic samples or natural products of known purity, this sub-clause should give all the details necessary to carry out the check.

13.5 Matching test

If it is necessary to allow for, or to eliminate, the interference of certain phenomena (for example, "background" colour), an appropriate test should be provided in this sub-clause, with all the necessary details of the procedure.

13.6 Determination(s) or test(s)

Each sequence of operations should be described accurately, using the imperative mood, and the test should be set out in an easily readable form in suitable sub-clauses and paragraphs in order to facilitate the description, the understanding and the application of the procedure.

Should it be necessary, in the course of these operations, to retain the product resulting from one of these steps in the procedure (for example, filtrate or precipitate) for use as a "test portion" in a later determination, this should be clearly stated, and provision made for it to be given a reference symbol or letter by which it can be identified again later when it is required in the course of the analysis.

Example :

"Keep filtrate 'D' for the determination of . . ."

and, when it is used :

"Use filtrate 'D' from the determination of . . ."

13.7 Calibration

If the method requires calibration, this operation should be the subject of a separate sub-clause located at the most appropriate point in the "Procedure" clause. This sub-clause should give all necessary details of the operations to be carried out. If the operations relating to the calibration are identical to those relating to the determination itself, or to a part of it, then one of the two sub-clauses should make reference to the other.

Example :

". . . Proceed as described in the sub-clause 'Determination', from the . . . th paragraph to the . . . th paragraph inclusive."

14 Expression of results

14.1 Method of calculation

This sub-clause should indicate the method for calculating the results. The following should be stated :

- the manner in which the results are to be expressed;
- the complete expression used for the calculation, including letter symbols;
- the simplified expression, if appropriate;
- the meaning of the letter symbols in the expression;
- the meaning of any numerical factors used in the expression;
- the units in which these symbols and factors are expressed;
- the number of decimal places to be shown in the results.

The following letter symbols are recommended :

- m* (lower case italic) for mass;
- V* (upper case italic) for volume;
- c* (lower case italic) for concentration.

NOTES

1 If a given letter symbol has to be applied for different values of the same quantity, numerical subscripts (0, 1, 2, . . .) should be added to the symbol (for example : $m_0 - m_1 - m_2$).

2 For titrimetric methods, it is recommended that one of the following alternatives be employed for the method of calculation :

- a) give in the formula used for the calculation a symbol (*c*) representing the actual concentration, expressed in moles per litre, of the standard volumetric solution used (in this case, the numerical factor, if there is one, will be related to 1,00 ml of 1,000 mol/l solution);
- b) give in the formula used for the calculation the numerical factor corresponding to 1,00 ml of standard volumetric solution having exactly the concentration specified in the "Reagents and materials" clause. In this case the following note is to be added :

"NOTE — If the concentration(s) of the standard volumetric solution(s) used is (are) not exactly as specified in the list of reagents and materials, an appropriate correction should be made."

Example :

Consider a titrimetric method for the determination of the alkalinity of a substance, carried out using a standard volumetric hydrochloric acid solution as titrant. The specified concentration of the hydrochloric acid is 0,2 mol/l. The result is to be expressed as a percentage by mass of potassium hydroxide (KOH).

The method of calculation could then be :

- a) "The alkalinity, expressed as a percentage by mass of potassium hydroxide (KOH), is given by the formula

$$\frac{V \times c \times 0,056 \times 100}{m}$$

$$= \frac{V \times c \times 5,6}{m}$$

V is the volume, in millilitres, of the hydrochloric acid solution [give the reference number from the list of reagents and materials] used;

c is the actual concentration, expressed in moles of HCl per litre, of this solution;

m is the mass, in grams, of the test portion;

0,056 is the mass, in grams, of potassium hydroxide corresponding to 1,00 ml of hydrochloric acid solution, $c(\text{HCl}) = 1,000 \text{ mol/l}$.

The result should be expressed to two decimal places."

- b) "The alkalinity, expressed as a percentage by mass of potassium hydroxide (KOH), is given by the formula

$$\frac{V \times 0,0112 \times 100}{m}$$

$$= \frac{1,12 V}{m}$$

where

V is the volume, in millilitres, of the hydrochloric acid solution [give the reference number from the list of reagents and materials] used;

m is the mass, in grams, of the test portion;

0,0112 is the mass, in grams, of potassium hydroxide corresponding to 1,00 ml of hydrochloric acid solution, $c(\text{HCl}) = 0,200 \text{ mol/l}$.

The result should be expressed to two decimal places.

NOTE — If the concentration of the standard volumetric solution used is not exactly as specified in the list of reagents and materials, an appropriate correction should be made."