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International Standard ISO 78/3

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Chemistry — Layouts for standards — Part 3: Standard for molecular absorption spectrometry

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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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Brazil	Italy	Sri Lanka
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INTERNATIONAL STANDARD ISO 78/3-1983 (E)

Chemistry — Layouts for standards — Part 3: Standard for molecular absorption spectrometry

0.1 Introduction

The products dissolved in a solution analysed by methods using molecular absorption spectrometry, the principles of the methods adopted and the operating conditions employed are extremely varied. Thus, the solutions on which the spectrometric measurements are made vary greatly in composition. The absorbance of any one of these solutions is generally equal to the sum of the characteristic absorbances of the various constituents.

The principle of each method of analysis using molecular absorption spectrometry consists of judiciously selecting the composition of solutions which will be measured so that the value of the characteristic absorbance due to each constituent to be determined can be obtained by difference.

0.2 Scope and field of application

This part of ISO 78 establishes a layout for standard methods of analysis using molecular absorption spectrometry for examining solutions in the visible and near ultra-violet regions, and gives some recommendations for their presentation and wording.

The layout itself is preceded by a clause "Terminology and definitions", which supplements the information given in ISO 6286. It is followed by a section "Notes on the application of the layout for a standard method of analysis by molecular absorption spectrometry", which deals with the wording of clauses and sub-clauses used in the layout.

0.3 References

ISO 78/2, *Layouts for standards — Part 2: Standard for chemical analysis.*

ISO 6286, *Molecular absorption spectrometry — Vocabulary — General — Apparatus.*

0.4 Terminology and definitions

0.4.1 set of calibration solutions: A set of reference solutions having different concentrations of the constituent to be determined.

Each member of the set:

— is prepared without addition of the sample submitted for analysis;[1)]

— contains a known quantity of the constituent to be determined;

— is prepared in such a way that the presence of the constituent to be determined is shown by a detectable absorption and an absorption of the same magnitude as the sample solution.

NOTE — The member of the set in which the quantity of the constituent to be determined intentionally added is zero, is called the zero member.

0.4.2 test solution: A solution made up from a test portion of the sample submitted for analysis and prepared in such a way that the presence of the constituent to be determined is shown by an absorption.

0.4.3 blank test solution: A solution prepared in the same way as the test solution, but so that it does not contain the constituent to be determined. With this intention, the test portion used for making up the test solution is

— replaced by an equal quantity of sample which has been treated in such a way as to ensure that the constituent to be determined is totally absent from it, without its other characteristics being changed, or

— replaced by an equal quantity of a substance, whose the composition is apart from the absence of the constituent to be determined, identical or very similar to that of the sample, or,

— replaced by a substance which is inert under the conditions of the determination (water for example) and which does not contain the constituent to be determined, or

— simply omitted.

NOTE — The particular procedures must be based on a selection from these four possibilities.

1) In methods based on the addition technique however, each member of the set contains the sample submitted for analysis, and in this case the zero member corresponds to the sample solution.

0.4.4 compensation solution: A solution designed to take into account the particular absorption of the reagents and, possibly, the absorption of the constituent to be determined in the sample. For this purpose, it is prepared in such a way that, if the constituent to be determined is present, it is not shown by an absorption.

To do this, the procedures usually adopted consist either of seeing that the initial reaction producing the absorbing compound does not take place (omission of one of the reactants if it does not itself absorb at the wavelength used for measurement, modification of the order of reagent addition, etc.) or in destroying the absorbing compound formed (oxidation, reduction, formation of a complex, etc.).

Depending on circumstances, the procedure may include the following compensation solutions:

a) calibration compensation solution;

b) zero member compensation solution;

c) sample compensation solution;

d) blank test compensation solution.

All the reagents which are used to make these solutions are found in those at the same concentration as in the corresponding solutions (members of the set of calibration solutions, zero member, sample solution, blank test solution).

0.4.5 Sample solution

A solution made up from the test solution and prepared by dilution in function of the sensitivity of the apparatus and of the calibration range selected, on which the measurement of absorbance is carried out.

Preferred order for the layout

	For comments, see clause (or sub-clause)
Title	
Warning and safety precautions	
Introduction	
Scope	
Field of application	
References	
Definitions	
Principle	2
Reactions	
Reagents and materials	3
Apparatus	4
Sampling and samples	5
Procedure	6
Test portion	6.1
Preparation of test solution	6.2
Preparation of the blank test (or **Blank test**)	6.3
Calibration	6.4
Preparation of reference solutions and of the set of calibration solutions	6.4.1
Formation of the absorbing compound	6.4.2
Calibration compensation solution	6.4.3
Spectrometric measurements	6.4.4
Analytical function (or **Plotting calibration graph**)	6.4.5
Determination	6.5
Formation of the absorbing compound	6.5.1
Sample compensation solution	6.5.2
Spectrometric measurements	6.5.3
Blank test	6.5.4
Expression of results	7
Special cases	
Remarks	
Test report	
Schematic representation of procedure	
Bibliography	
Annexes	

NOTE — Clauses and sub-clauses should be numbered consecutively using arabic numbers and the point numbering system.

Notes on the application of the layout for a standard method of analysis by molecular absorption spectrometry

1 General

The clauses included in the layout scheme (headings in capital letters) are strictly those given in ISO 78/2.

The notes below concern only the clauses and sub-clauses whose content is directly related to analysis by molecular absorption spectrometry carried out in the visible range or the near ultra-violet; for the other clauses, see ISO 78/2.

The aim in elaborating this part of ISO 78 has been to provide editors with a guide which can be used wherever possible.

Where this standard layout cannot be followed exactly, it will be for editors to modify the recommended layout, adapting it to analytical demands.

In particular, the description of certain determinations will not require the inclusion of all the clauses or sub-clauses provided in the layout, while in other cases extra clauses or sub-clauses may occasionally be needed.

Consequently, the only inflexible rules are the following:

a) use the terminology given in this part of ISO 78 and in ISO 6286;

b) where possible and justifiable, include the clauses and sub-clauses indicated in the layout, and arrange them in the sequence indicated (it is possible and even advisable to re-arrange them where strictly following the plan would lead to sub-clauses having little meaning);

c) only ignore instructions given in this part of ISO 78 if they are not applicable.

2 Principle

This clause shall indicate concisely the essential steps of the method (preferably using substantive phrases), excluding details relating to the procedure.

It shall thus, on the one hand contain the physical and chemical principles of preparations (e.g. ashing, solution preparation, extraction, separation, complexing) preceding the stage corresponding to the molecular absorption spectrometry and, on the other, the characteristics of this step.

With regard to this last statement, it shall

— mention the nature and the details of formation of the absorbing compound;

— draw attention, where necessary, to the kinetics of the reaction in formation of this compound;

— indicate the wavelength at which the measurement should be made, stating whether it corresponds to the absorption spectrum maximum and, if not, stating the reasons.

3 Reagents

This clause, set out in accordance with the details given in ISO 78/2, shall indicate in particular how the standard solution for the members of set of the calibration solutions shall be prepared.

The quality of reagents should be precisely indicated and also, where necessary, the processes of checking and purification.

4 Apparatus

It is not necessary to include in this list ordinary laboratory apparatus required to carry out the method.

On the other hand, it may be useful to indicate the minimum requirements with which the spectrometric equipment shall comply and, in particular, the maximum permissible spectral bandwidth for the wavelengths used and the permissible variation with regard to the nominal wavelength.

If use of optical cells of particular thickness is required, it is necessary to specify it.

Where necessary, include a reference to ISO 6286 with regard to the performances which may be required and for checking the condition of the equipment.

5 Sampling and samples

It is recalled that, according to ISO 78/2, a sub-clause headed "Preparation of the test sample" shall be included if necessary in this clause, giving all the details regarding the preparation (from the laboratory sample) of the test sample from which the test portion shall be taken.

6 Procedure

It should be remembered that the layout that is the subject of this part of ISO 78 should not be regarded as universally applicable. This is why, for example, certain procedures should be regarded as preliminary tests (designed, for example, to determine how pH corrections should be made) or control tests (e.g. to check whether the quantities or nature of the reagents used are well adapted to the particular conditions of the medium), to be carried out on one or more solutions the preparation of which is the subject of this clause. It is then for the editor to decide how the corresponding sub-clauses should be inserted between the sub-clauses provided for in the layout, how to make the text understood and how best to carry out the method.

The following is the only inflexible rule: as far as possible, include each time all the sub-clauses indicated in this clause and arrange them in the sequence given.

6.1 Test portion

The recommendations to follow for editing the sub-clause concerning the amount of sample for analysis by mass or by volume are those which are given in the sub-clause 13.2 of ISO 78/2.

6.2 Preparation of test solution

The mass or the volume of test portion is given as a guide; as it is a function of the sensitivity of the apparatus and of the calibration range selected, it may be reduced or increased. According to this hypothesis, the editor should indicate the maximum and minimum permissible concentrations of the sample in the sample solution and/or in the test solution.

This sub-clause should indicate precisely all physical and chemical treatments which the test portion for analysis has to undergo prior to the development of the absorbing compound. The editor should not hesitate to describe the minor details capable of influencing the quality of the results.

6.3 Preparation of the blank test (or Blank test)

The heading of this sub-clause depends on its contents: it will be "Preparation of the blank test" if the step corresponding to the formation of the absorbing compound is not included in it. Otherwise it will be "Blank test".

The contents of this sub-clause are also essentially variable in the two cases above:

a) sometimes it will be very detailed. For example, the absence in the blank test solution of certain constituents of the product submitted for analysis (or from the test solution) may lead to conditions of the medium (e.g. pH) which would differ from those of the sample solution (and which could therefore possibly affect the determination) if the methods of preparation were not mentioned in detail in this sub-clause;

b) sometimes it might simply refer to the sub-clause on "Preparation of test solution". This is particularly the case where the solution which replaces the test solution in carrying out the blank test is prepared in the same manner as this solution;

c) sometimes, even, the zero member of the set of calibrations solutions may, in certain cases, replace the blank test solution.

6.4 Calibration

6.4.1 Preparation of reference solutions and of the set of calibration solutions

This sub-clause should indicate how the reference solutions should be prepared by giving the volumes of standard solution which shall be used to form the different members of the set of calibration solutions, from the zero member to the member containing the maximum admissible quantity of the element to be determined and also giving the thickness of the optical cells to which this range is adapted.

The range of the set of calibration solutions is given as a guide, being a function of the instrumental sensitivity, it can be varied slightly. In the latter case, it is for the operator to adjust the final dilution of the test solution in order to obtain optimum conditions.

6.4.2 Formation of the absorbing compound

This sub-clause should describe in detail all the operations that needed to be carried out on each member of the set in order to obtain the solutions for filling the optical cells.

In particular, if necessary

— attention should be drawn to the kinetics of the reaction of formation of the absorbing compound and the waiting time to be observed between the various additions of reagents and also the time required for the purpose of obtaining or not obtaining complete formation of the absorbing compound: the absence of any indication, particularly in the latter case, implies that the absorbing compound is formed completely and instantaneously;

— the temperature at which it is necessary to carry out the determination should be indicated;

— the duration of stability of the absorbing compound should be indicated.

Absence of these indications will be interpreted as follows:

a) no waiting time is necessary at all;

b) the operations take place at ambient temperature;

c) the absorbance of the absorbing compound has not been checked over a long period.

6.4.3 Calibration compensation solution

If a compensation solution over the set of calibration solutions is necessary, this sub-clause (either in full, or by indicating the modifications in the treatment of the members of the set) will give all the instructions necessary to obtain the solution to be used in the optical cell.

6.4.4 Spectrometric measurements

This sub-clause shall indicate the following details.

6.4.4.1 The wavelength at which (or the minimum and maximum wavelength between which) the measurements should be made, also indicating whether or not this corresponds to the maximum of the absorption spectrum.

Nevertheless, in most cases the user should plot the absorbance curve as a function of the wavelength, for indicating — with regard to the equipment at his disposal — the particular wavelength to be used.

6.4.4.2 The reference for zero absorbance adjustment of the instrument.

This reference may be

a) preferably, water or the solvent used in the method;

b) the calibration compensation solution;

c) the blank test solution;

d) the zero member of the set.

The nature of the determination (characteristics of the product analysed, properties of the absorbing compound used, peculiarities of the procedure, etc.) may make it a matter of indifference which of the above-mentioned possibilities is adopted. Thus, when possible, some operators prefer to use the zero member of the set [which normally allows calibration graphs (without translation) passing through the origin], whereas others prefer to take measurements relative to water (which allows any unusual variation to be detected).

In such a case, it is better to draft the method prescribing that the zero member is used as reference (which results in slightly shorter wording) and to indicate in a note that "water may also be used as reference".

On the other hand, when it is essential to use a specific reference, it is as well to state this fact, for example by introducing, in editing, the adverb "necessarily".

6.4.4.3 The temperature at which the measurements should be taken.

The absence of instructions to this effect will be interpreted in the following manner: between 10 and 40 °C, no special precautions are necessary, provided that the temperatures of the solutions on which measurements are being made, including those of the reference solutions, are sufficiently close for variations due to dilatation to be negligible.

NOTE — For single-beam instruments it was necessary to check their performance when using at different temperature between 10 and 40 °C.

6.4.5 Analytical function (or Plotting calibration graph)

This sub-clause may sometimes be replaced by a phrase incorporated in the preceding sub-clause, for example "Plot the analytical curve from the values obtained".

In other cases, however, it may be appropriate to give detailed instructions. It is useful to indicate for at least one of the members of the range an approximate value for the absorbance which should be obtained.

6.5 Determination

6.5.1 Formation of the absorbing compound

This sub-clause, analogously with sub-clause 6.4.2, to which it may, moreover, merely refer, should describe the operations necessary to obtain the solution (sample solution) for introduction into the optical cell.

6.5.2 Sample compensation solution

This sub-clause, if required, is analogous to sub-clause 6.4.3, relating to the calibration compensation solution.

6.5.3 Spectrometric measurements

This sub-clause should include the same requirements as those set out in the corresponding sub-clause (6.4.4) relating to spectrometric measurements carried out on the members of the set of calibration solutions.

The reference against which the instrument is set on zero absorbance may be

a) water or another solvent;

b) the sample compensation solution;

c) the blank test solution;

d) the zero member of the set of calibration solution.

When there are several equally valid possibilities, it is best to draft the procedure in those terms which allow the shortest wording and indicate in a note that "another such solution may equally well be used as reference, provided that the use made of the value obtained is modified in consequence".

On the other hand, when it is essential to use a specific reference, it is necessary to well indicate.

6.5.4 Blank test

It is only worthwhile including this sub-clause if sub-clause 6.3 is limited to the preparation of the blank test.

7 Expression of results

This clause should indicate how the value of the parameter sought should be calculated starting from

a) the analytical function obtained according to 6.4.5;

b) the result of the measurement obtained according to 6.5.3;

c) if necessary, the result of the measurement obtained according to 6.5.4;

d) the quantity of test portion (6.1) used for the preparation of the test solution;

e) the dilution factor between the test solution and the sample solution.

In addition, it should show clearly the units employed.

NOTE — It will also be recalled that, according to ISO 78/2, it is in this clause that a sub-clause headed "Precision" should be included, if necessary.