
International Standard



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.

International Standard ISO 78/3 was developed by Technical Committee ISO/TC 47, *Chemistry*, and was circulated to the member bodies in March 1982.

It has been approved by the member bodies of the following countries:

Australia	Germany, F.R.	Portugal
Austria	Hungary	Romania
Belgium	India	South Africa, Rep. of
Brazil	Italy	Sri Lanka
China	Korea, Rep. of	Switzerland
Czechoslovakia	Mexico	Thailand
Egypt, Arab Rep. of	New Zealand	USSR
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The member body of the following country expressed disapproval of the document on technical grounds:

Netherlands

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.

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Preferred order for the layout

For comments,
see clause
(or sub-clause)

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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 rearrange 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.