
International Standard



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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (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 authorized 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.

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

Australia	France	Netherlands
Austria	Germany, F.R.	Poland
Belgium	Hungary	South Africa, Rep. of
China	India	Switzerland
Czechoslovakia	Italy	Thailand
Egypt, Arab Rep of	Korea, Rep. of	USSR

No member body expressed disapproval of the document.

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Layouts for standards — Part 4: Standard for atomic absorption spectrometric analysis

0.1 Introduction

This part of ISO 78 supplements ISO 6956, which summarizes the principle of atomic absorption, lists the components of a spectrometer and fixes the required conditions during its use. The basic principles and the essential recommendations in common with all procedures and the general conditions of reproducibility and accuracy of the mean of the determinations are also given in ISO 6956.

This layout involves the use of appropriate terminology, indicated in ISO 6955. In other respects, the presentation and the wording of clauses given in this layout should comply with the principles stated in ISO 78/2.

The layout itself is followed by a section "Notes on the application of the layout for a standard method of analysis by atomic absorption spectrometry", which deals with the wording of clauses and sub-clauses used in the layout.

0.2 Scope and field of application

This part of ISO 78 establishes a layout for standard methods of analysis using atomic absorption spectrometry and gives some recommendations for their presentation and wording.

0.3 References

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

ISO 690, *Documentation — Bibliographical references — Essential and supplementary elements.*

ISO 6955, *Analytical spectroscopic methods — Flame emission, atomic absorption and atomic fluorescence — Vocabulary.*

ISO 6956, *Atomic absorption spectrometry — Introduction to use.*¹⁾

1) At present at the stage of draft.

Preferred order for the layout

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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 atomic 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 atomic absorption spectrometry; 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 ISO 6955;
- 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 Introduction

This clause can usefully make reference to ISO 6956 and specially to its annex about safety, and to ISO 6955 for the correct nomenclature.

3 Field of application

This clause shall contain exact indications on the field of application of the method (definition of products to which the method is applicable, limit of content for which the method is valid). In addition to these indications it is necessary to report the interfering constituents as well as their contents which give interferences (see sub-clause 5.2 of ISO 6955) or the interferences of other origin which render inapplicable the specified procedure.

This clause should also indicate any special cases and the clauses in which they will be dealt with.

4 References

This clause must contain a complete list of all documents or International Standards which are indispensable for the application of the specified method of analysis.

NOTE — This list shall not include technical documents which are merely used as references in the preparation of the method; such documents can be mentioned in the clause « Bibliography ».

5 Principle

This clause should 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, addition of complexing agents and spectrochemical buffers) preceding the stage corresponding to the atomic absorption spectrometry and, on the other, the characteristics of this stage.

With regard to this last statement, it shall mention:

- a) the type of atomizers (see clause 2 of ISO 6955);
- b) the wavelength(s) of the line(s) used;
- c) the type of detection and correction of non-specific attenuation;
- d) the type of method (see sub-clause 6.1 of ISO 6955), namely:
 - 1) direct method of determination,
 - 2) determination by the bracketing technique,
 - 3) (analyte) addition technique,
 - 4) reference-element technique,
 - 5) indirect method.

6 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 the set of calibration solutions shall be prepared.

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

7 Apparatus

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

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On the other hand, it should define the particular equipment and for the spectrometer it should indicate

- a) the source(s) emitting the characteristic lines;
- b) the type and the characteristics of the atomizer (type of burner, type of gases, etc.);
- c) the maximum permissible spectral bandwidth for the wavelengths of the spectral line(s) and the necessity or not of a device for correcting random fluctuations;
- d) other special devices.

Where necessary, include a reference to ISO 6956 with regard to the performances which may be required and for checking the condition of the equipment.

8 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. In some circumstances, preliminary tests should be foreseen [designed, for example, to detect or correct non-specific attenuation (see ISO 6956)]. 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.

8.1 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 given in sub-clause 13.2 of ISO 78/2.

8.2 Treatment of test portion (or Preparation of test sample for presentation to instrument)

The mass or the volume of the test portion for analysis 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 test sample for presentation to instrument.

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

8.3 Preparation of the blank test

This sub-clause should indicate how to prepare the blank test solution (see definition 5.4.4 of ISO 6955) before introducing it into the atomizer.

In certain cases, the indications will conveniently be made by reference to the sub-clause dealing with the preparation of the test sample for presentation to instrument (case of a sample not containing the analyte).

8.4 Preparation of the set of calibrating solutions

This sub-clause should indicate the concentration of standard solutions which shall be used to form the different members of the set of calibrating solutions, from the zero member to the member containing the maximum permissible quantity of the element to be determined.

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 concentration of analyte in the test sample for presentation to instrument in order to obtain optimum conditions, after checking that the possible interferences remain corrected and that others do not appear.

8.5 Calibration and determination

8.5.1 Spectrometric measurements

This sub-clause should indicate

- a) the wavelength(s) at which measurements should be made;
- b) if necessary, the observation height (see ISO 6955 clauses 2.3.13);
- c) in the case of flames, the characteristics of the flame (oxidizing or reducing);
- d) in the case of a flameless atomizer, the exact indications of the permissible amount of analyte for each measurement, the time and the temperature of different stages of the cycle leading to atomization, as well as the quality of materials constituting the atomization enclosure and its geometry;
- e) the solvent blank for zero absorbance adjustment of the instrument (see definitions 5.4.2 of ISO 6955 and 4.2.4 of ISO 6956).

8.5.2 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.

8.5.3 Blank test

This sub-clause should be included if the blank test solution differs from the zero number of the set of calibrating solutions and is then used to correct the measurements carried out on the samples.

9 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 8.5.2;
- b) the result of the measurement obtained according to 8.5.1;
- c) if necessary, the result of the measurement obtained according to 8.5.3;
- d) the quantity of test portion (8.1) used.

In addition it should show clearly the units employed.

10 Precision

The clause should bring together, in addition to precision (repeatability and reproducibility), all the factors capable of defining the suitability of the method i.e. the sensitivity, the accuracy and the reliability (see definitions 6.2.1, 6.2.5 and 6.2.9 in ISO 6955).

11 Special cases

This clause should bring together all the alterations in the procedure necessitated by the presence or the absence of specific elements in the product to be analysed.

It is preferable for the sake of greater clarity, even if the alteration is very slight, to draft a clause entitled "Special case" rather than describe the alteration in a note.

However, if the alterations involve very important modifications, it is preferable to draft another document.

12 Bibliography

If it is necessary to mention bibliographical references, follow the form provided in ISO 690.

