INTERNATIONAL STANDARD 2718

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Standard layout for a method of chemical analysis by gas chromatography

Plan normalisé de méthode d'analyse chimique par chromatographie en phase gazeuse

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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 2718 was drawn up by Technical Committee ISO/TC 47, *Chemistry*, and circulated to the Member Bodies in April 1972.

It has been approved by the Member Bodies of the following countries :

Austria	India	South Africa, Rep. of
Belgium	Ireland	Spain
Chile	Israel	Sweden
Czechoslovakia	Italy	Switzerland
Denmark	Netherlands	Thailand
Egypt, Arab Rep. of	New Zealand	Turkey
France	Portugal	United Kingdom
Germany	Romania	U.S.S.R.

This International Standard has also been approved by the International Union of Pure and Applied Chemistry (IUPAC).

No Member Body expressed disapproval of the document.

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Standard layout for a method of chemical analysis by gas chromatography

0 INTRODUCTION

Gas chromatography is a separatory and analytical technique which is being utilized increasingly, particularly for check analyses within the same laboratory, for check analyses for acceptance between laboratories, for analyses of unknown samples, and for analytical research.

Because of the diversity of apparatus and working methods, it seems desirable to establish a standard layout for the basic characteristics and instructions of a technical nature which should be specified for either of the following purposes :

– to present a report of an experimental investigation or of a check analysis in a scientific publication or in a document of a commercial nature. The authors of such publications should give all the necessary instructions to enable the document to be understood and the procedure to be followed with the aid of identical or closely related equipment.

– to draw up procedures capable of application in laboratories equipped with different apparatus. Only those details that are sufficient to allow the method to be put into practice and reproducible results to be obtained, within determined limits of precision, should be given.

The list of characteristics enumerated in this standard layout should not be regarded as exhaustive, nor is there any intention to imply that all these characteristics should be given and explained in publications and procedures. The authors or editors of such documents should select from this list the characteristics necessary to achieve one or other of the two objectives mentioned above.

In particular, from the point of view of standardization, the details given in a particular standard should be decided at the conclusion of an experimental investigation carried out in several laboratories and after a comparative analysis of the results.

STANDARD LAYOUT

NOTES ON THE APPLICATION OF THE STANDARD LAYOUT

1 TITLE

The title of the publication or standard shall express its contents without any ambiguity, mentioning briefly the products concerned and the nature of the analysis.

2 SCOPE AND FIELD OF APPLICATION

This clause shall contain precise details of the subject of the investigation or standard, and particularly of the field of application of the method described. It shall include a brief summary of the investigation or standard which shall be suitable for use as an abstract.

3 MATERIALS

Mention any hazard present and cross-refer to clause 9, "Hazards".

3.1 Carrier gas and auxiliary gases

- Nature and commercial description.
- Content of impurities and method of purification.

3.2 Materials for the preparation of calibration samples.

3.3 Cross-refer to 4.3 for column details, including packing and preparation.

4 APPARATUS

4.1 Type of apparatus

4.1.1 Characteristics of the assembly (oven, temperature regulation . . .), special devices.

4.1.2 Devices for the control of pressure, or of the flow of carrier gas and auxiliary gases.

4.2 Injection device

- Type.
- Material.
- Characteristics.
- Miscellaneous accessories.

4.3 Columns (and pre-column)

4.3.1 *Number*

- Grouping and switching of the columns.

4.3.2 *Column*

- Nature of the column (material and treatment).
- Length and packed length.
- Internal and external diameters.
- Form (U, W or helical . . .).
- Radius of any curve.

4.3.3 *Packing*

4.3.3.1 Support

- Nature (description, batch . . .).
- Particle size.
- Treatment, if any.

4.3.3.2 Stationary phase

- Chemical nature.
- Degree of impregnation : indicate, without ambiguity, the proportions of stationary phase and support (for example, in grams of stationary phase per 100 g of support, or as the percentage by mass of stationary phase in relation to the total of support + phase).

4.3.3.3 Active adsorbent

- Nature.
- Particle size and/or other physical characteristics.
- Activation.

4.3.3.4 Other packing

- Modified adsorbents.
- Porous polymers.

4.3.3.5 Method of packing and mass introduced.

4.3.4 *Efficiency and resolution*

4.3.4.1 Efficiency under given conditions.

4.3.4.2 Resolution under given conditions.

4.4 Flow splitter

4.5 Detector

- Type.
- Characteristics.
- Associated electronic device (characteristics of the electrometer).

4.6 Recorder and/or integrator

- Type.
- Sensitivity.
- Impedance.
- Response time.

4.7 Electronic data processing system

4.8 Accessories

5 SAMPLE

- Nature :

  name;

  description;

  supposed composition.

- Method of sampling and storage.
- Special considerations that may be involved during the analysis (volatility, stability, explosivity, toxicity, etc. with reference to clause 9 "Hazards", as appropriate).
- Preliminary treatment (distillation, dissolution, methylation or any other treatment).

6 PROCEDURE

6.1 Setting up the apparatus

6.1.1 *Injector*

- Temperature and permitted limits.

6.1.2 *Oven and column*

6.1.2.1 Temperatures

- Isothermal conditions :

  temperature and permitted limits.

- Programmed conditions :

  initial period and temperature;

  programme of rise in temperature;

  temperature and final period.

6.1.2.2 Flow of carrier gas

- Isobaric conditions :

  rate of flow;

  pressures at inlet and outlet of the column.

- Programmed conditions :

  initial period and initial flow rate;

  programme of flow variation;

  back flushing.

6.1.3 *Flow splitter*

- Split ratio.

6.1.4 *Detector*

- Temperature and permitted limits.
- Electrical controls.
- Regulation of auxiliary gases.

6.1.5 *Recorder*

- Chart speed.
- Initial attenuation.

6.1.6 *Integrator*

6.2 Calibration

6.2.1 *Methods of calibration*

The following are the most generally used methods of calibration :

6.2.1.1 Internal normalization method

All the constituents are eluted and all the peaks measured. The measurements are normalized to 100, with the calibration factor determined either by calculation (see 6.2.3) or by measurement at each analysis (see 6.2.2.1).

6.2.1.2 Internal standard method

A known quantity of a known substance, whose peak does not interfere with any other peaks, is added to the sample. The measurement of the peaks of the different constituents, corrected by the respective calibration factors, is compared to that of the peak of the added known substance.

6.2.1.3 External standard method

A certain quantity of the sample and an equal amount of a known synthetic mixture are subjected in turn to chromatography. The measurements of the resultant peaks are compared.

6.2.1.4 Absolute method

A known quantity of sample is injected. By measurement of the peaks, and depending on the adjustment of the apparatus, the quantity of constituent corresponding to each peak is determined on the basis of a general relationship of peak area to mass. This relation is derived from, for example, gravimetric, volumetric or coulometric analysis and can be presented in the form of tables, curves, charts, etc.

6.2.1.5 Addition method

The following are subjected in turn to chromatography :

– the original sample. The peaks corresponding to the constituent to be determined and to a constituent giving a neighbouring peak are measured.

– a reference mixture of a quantity m of the sample and a known quantity m_1 of the constituent to be determined. The peaks corresponding to the constituent to be determined and to the constituent with the neighbouring peak are again measured.

The conditions of application of the five methods of calibration mentioned in 6.2.1.1 to 6.2.1.5 are summarized in the table below.

6.2.2 *Standard mixtures*

6.2.2.1 Operating frequency :

– calibration sample used for the determination of factors, whether once only or for periodical recalibration (the period to be indicated);

– calibration samples used at each analysis.

6.2.2.2 Method of preparation of the standard mixture or of the series of standard mixtures.

6.2.2.3 Conditions specific to the use of standard mixtures in chromatography :

– injection of known or identical quantities;

– conditions for reproducibility in the regulation of the apparatus;

– possible subsequent or simultaneous analysis of the standard mixture.

6.2.3 *Presentation of the calibration data*

6.2.3.1 In the form of curves :

– selection of scales;

– method for drawing up charts;

– particular forms of presentation.

6.2.3.2 In the form of factors :

– method of calculation from the results of chromatography of standard mixtures; indicate the units;

– method of calculation from theoretical data (gas density balance, Sternberg Gallaway and Jones' method, etc.);

– approximate estimations; the case where the factors are considered to be equal to 1; state and explain approximations when appropriate; rules to be followed in the case of unidentified peaks.

6.3 Test

6.3.1 *Preparation of the test portion* (in the case of the internal standard method) or of the reference mixture (in the case of the addition method).

– Volume or mass of the sample.

– Volume or mass of the standard mixture or addition.

– Method of mixing the standard mixture with the sample.

Method	Eluted constituents	Detector	Volume injected	Operating conditions of the chromatograph
Internal normalization method	All constituents	Linear	Optional	Stable during the analysis
Internal standard method	Constituents to be determined	Linear	Optional	Stable during the analysis
External standard method	Constituents to be determined	Linear	Reproducible	Stable during at least two consecutive analyses
Absolute method	Constituents to be determined	Optional	Known reproducible	Stable during a series of analyses reproducible from one day to another
Addition method	Constituents to be determined	Linear	Optional	Stable during at least two consecutive analyses

6.3.2 *Introduction of the test portion* (and of the reference mixture in the case of the addition method)

- Quantity injected.
- Procedure.

6.3.3 *Recording*

- Attenuation and changes in the attenuation and chart speed.
- Period.

6.4 Examination of the chromatograms

6.4.1 *Standard chromatograms*

6.4.2 *Qualitative analysis*

- Order of elution of the constituents (peaks).
- Retention characteristics.
- Specification of possible interferences.
- Complementary methods of identification.

6.4.3 *Quantitative analysis*

- Measurements effected (height of peaks, product height by retention distance, triangle constructed on the peaks, triangulation, planimetry, cutting and weighing, integration, etc.).
- Calculations :

  transfer to calibration curves or the use of charts;

  formulae to be applied.
- Special methods.

7 EXPRESSION OF RESULTS

7.1 Qualitative analysis

- Number of constituents found.
- Names of the constituents identified.

7.2 Quantitative analysis

- Results and method of expression of the results.
- Limit of accuracy of the results.
- Limit of detection.

8 TEST REPORT

This clause is intended to remind the operator that the following particulars are to be included in the test report :

a) authority for the method used, for example, a citation of an official reference or of a reference from the scientific literature;

b) the results, the method of expression used and, possibly, the formula for the calculation;

c) any special details, as well as any unusual features noted during the analysis;

d) any operation not included in the method, or regarded as optional.

9 HAZARDS

Materials known to present a hazard are to be noted as such in the appropriate clause, normally under clause 3. In addition, all hazards are to be grouped together and detailed in clause 9 under an appropriate heading, together with information about the precautions to be taken or reference to a suitable source of such information. Examples of headings which may be required are :

- Toxic hazards
- Explosive hazards
- Radioactive hazards.

ANNEX

CALCULATION FORMULAE DEPENDING ON THE METHOD OF CALIBRATION USED

A.1 INTERNAL NORMALIZATION METHOD

$$x_i = \frac{100 \times K_i \times A_i}{\Sigma K_i \times A_i}$$

where

x_i is the content, expressed as a percentage, of the substance i in the sample;

K_i is the coefficient of proportionality for the substance i;

A_i is the measurement of the peak, determined in accordance with 6.4.3.

A.2 INTERNAL STANDARD METHOD

$$x_i = \frac{100 \times m_E \times A_i \times K_{E,i}}{m \times A_E}$$

where

x_i is the content, expressed as a percentage, of the substance i in the sample;

m is the mass, in grams, of the sample;

m_E is the mass, in grams, of the known substance added;

A_i is the measurement of the peak, determined in accordance with 6.4.3;

A_E is the measurement of the peak of the known substance;

$K_{E,i}$ is the coefficient of proportionality relating to the compound i in comparison to the known substance; $K_{E,i} = A'_E/m'_E$ is determined separately with a known mass (m'_E) of known substance to which a peak area A'_E corresponds.

A.3 EXTERNAL STANDARD METHOD

$$x_i = E_i \frac{A_i}{A_E}$$

where

x_i is the content of the substance i in the sample;

E_i is the content of the compound i in the synthetic mixture;

A_i is the measurement of the peak i on the chromatogram of the sample;

A_E is the measurement of the peak i on the chromatogram of the synthetic mixture.

A.4 ABSOLUTE METHOD

Reference should be made to the tables, curves and charts, as described in 6.2.1.4.

A.5 ADDITION METHOD

$$x_i = \frac{100 \times A_{i,1} \times m_i}{m \times [A_{i,2} - A_{i,1}\ (A_{j,2}/A_{j,1})]}$$

where

x_i is the content, expressed as a percentage, of the substance i in the sample;

$A_{i,1}$ is the measurement of the peak i on the chromatogram of the sample;

$A_{i,2}$ is the measurement of the peak i on the chromatogram of m g of sample with the addition of m_i g of the constituent to be determined (reference mixture);

$A_{j,1}$ is the measurement of the peak j in the vicinity of the peak i on the chromatogram of the sample;

$A_{j,2}$ is the measurement of the peak j on the chromatogram of the sample with addition (reference mixture).