

Recommendations for

A standard layout for methods of chemical analysis by gas chromatography

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- British Gas Corporation
- British Pharmacopoeia Commission
- Chemical Industries Association Ltd
- Department of Energy
- Department of Industry — Laboratory of the Government Chemist
- Electricity Supply Industry in England and Wales
- Institute of Fuel
- Institute of Petroleum
- Institution of Gas Engineers
- Ministry of Defence
- National Coal Board
- Scientific Instruments Manufacturers' Association
- SIRA Institute
- Standardization of Tar Products Tests Committee

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Foreword

This British Standard, prepared under the direction of the Chemical Divisional Council, gives recommendations for a standard layout for methods of chemical analysis by gas chromatography. These recommendations have been compiled, in view of the diversity of apparatus and working methods available, to enable methods to be drafted in such a way as to ensure a stated reproducibility in results.

It is recognized that such a standard cannot be regarded as exhaustive in its treatment. Users should therefore include any parameter not specifically mentioned therein, but known to affect the precision of the method under consideration, in the most appropriate part of the recommended layout. On the other hand, items in the recommendations, if not applicable, should be omitted.

The clauses and subclauses in such a document should be numbered consecutively in accordance with a point system.

Although based on recommendations given in ISO 2718, the content of this standard is different in that

- a) it has been essentially aligned, for uniformity, with the more general recommendations given in the more recently published ISO 78-2 and in BS 0-3;
- b) it gives more detailed guidance on the implementation of the recommendations;
- c) it contains an example of a gas chromatographic method of analysis drafted in accordance with the recommendations. (It is emphasized, however, that this example serves only to illustrate the use of the recommendations and does *not* constitute a British Standard method for the determination of styrene in polystyrene by gas chromatography.)

This standard should be used in conjunction with BS 3282, BS 0-3.

NOTE The clause numbering of section 2 of this standard is to some extent duplicated in section 4. Care should therefore be taken, when making reference to this standard, to give both the section and clause numbering.

A British Standard does not purport to include all the necessary provisions of a contract. Users of British Standards are responsible for their correct application.

Compliance with a British Standard does not of itself confer immunity from legal obligations.

Summary of pages

This document comprises a front cover, an inside front cover, pages i and ii, pages 1 to 20, an inside back cover and a back cover.

This standard has been updated (see copyright date) and may have had amendments incorporated. This will be indicated in the amendment table on the inside front cover.

Section 0. General

0.1 Scope

This British Standard gives recommendations for the layout for methods of chemical analysis by gas chromatography.

0.2 References

The titles of the publications referred to in this standard are listed on the inside back cover.

Section 1. Recommended standard layout

The following standard layout is recommended for methods of chemical analysis by gas chromatography.

Title

- 1 Scope and field of application
- 2 References
- 3 Definitions
- 4 Principle
- 5 Materials required
- 6 Apparatus
- 7 Sample
- 8 Procedure
- 9 Expression of results
- 10 Notes on procedure, particularly with reference to hazards
- 11 Test report
- 12 Bibliographical references
- 13 Appendices

Guidance on the use of this layout is given in section 2 of this standard.

Section 2. Notes on the application of the recommended standard layout

NOTE The numbering of the clauses and subclauses in this section refers to those used in the recommended standard layout (see section 1).

Title

The title of the publication or standard should express its contents concisely and unambiguously, mentioning briefly the products concerned and the nature of the analysis.

The title should always contain the words “by gas chromatography” and should be phrased either “Determination of in by gas chromatography”, or “The analysis of by gas chromatography”. The phrase “the purity of” should not be used.

1 Scope and field of application

This clause should contain precise details of the subject of the standard. The field of application, particularly, should be fully described and should contain the following information:

- a) the determination to be made;
- b) the products for which the method is suitable, in sufficient detail to prevent ambiguity;
- c) the physical state of the sample [unless clearly defined by b)];
- d) the range(s) of concentration covered by the method;
- e) known limitations or interference in the method, arising from the state of the sample;
- f) any other special features of the method.

To avoid repetition, where many substances are to be determined, a) and d) may be more conveniently tabulated with retention times or other practical data elsewhere in the standard. Clause 1 then need only include a reference to the clause containing the table.

2 References

This clause should list, or make reference to, all other standards that are indispensable for the application of the method.

3 Definitions

If it is necessary, for clarity, to give definitions of certain terms used in the standard, these definitions should be given in this clause.

4 Principle

This clause should give a concise summary of the method used, from receipt of the original sample to the calculation and reporting of results, and the statement should be suitable for use as an abstract in other documents.

It should make particular reference to the following:

- a) the method of obtaining a suitable chromatogram (including preparation of sample);
- b) features of the apparatus or technique;
- c) the method by which results are calculated.

5 Materials required

The purpose of this clause is to ensure that all materials used are sufficiently defined for selection. Hazards should be indicated where they exist and, in these circumstances, reference should be made to clause **10** of the standard layout. The materials used should be listed as given in **5.1** to **5.3**.

5.1 Carrier gas and auxiliary gases. The carrier gas and auxiliary gases should be named, together with the supply pressure requirements. The purity of the gases should be specified if necessary to the proper functioning of the method. Reference should be made, where appropriate, to established methods for determining the purity of the gases and for purifying them.

5.2 Materials for use in calibration and sample treatment. The materials to be used for calibration purposes or as sample diluents, sample solvents or internal standards should be named and limits for the impurities given; suitable criteria should be given for the specific limits of peaks that interfere, by reference to **8.2**.

5.3 Materials for the preparation of columns. The materials used to prepare the column, including, if relevant, any solvent used to disperse the stationary phase, should be specified. Details of how to pack the column should, however, be specified in **6.3**.

6 Apparatus

This clause should specify the apparatus and facilities necessary to perform within the stated reproducibility of the method. The details given in **6.1** to **6.7** are intended for guidance. The efficiency of the column and of the recording apparatus should always be stated, preferably by reference to **6.3.4** and **8.3.3** respectively.

NOTE A British Standard specification for gas chromatographic equipment is in course of preparation. When available its use is recommended, where appropriate, in specifying the components of the apparatus.

6.1 General description

6.1.1 Gas chromatographic system. The chromatographic system required should be briefly described, e.g. 'Dual flame ionization detector, temperature-programmed chromatograph with heated injection block and column back-flush facilities'.

6.1.2 Flow diagram. For clarity, it is recommended that a flow diagram be incorporated in all but the simplest cases.

6.1.3 Characteristics of the assembly (type of oven, temperature regulation, etc.) , special devices

6.1.3.1 Column temperature

- a) *Isothermal temperature control range.* The upper and lower temperature limits of the method should be specified. Nominal precision of temperature control may be given if required.
- b) *Temperature programming.* The programme required for the method should be stated, together with nominal precision limits for the temperature/time relationship.
- c) *Temperature read-out.* Minimum facilities for checking the temperature should be specified.

6.1.3.2 Detector temperature. The upper and lower range of temperature limits should, and the nominal precision of temperature control may, be specified and should be compatible with the required test performance.

6.1.3.3 Special devices. Specialized equipment such as precolumn reaction units and back-flush systems should be adequately described. Where appropriate, suitable diagrams should be included.

6.1.4 Devices for the control of pressure or of the flow of carrier gases and auxiliary gases. The methods used for controlling the flow of carrier gases or other gases (such as those used for flame ionization detection or for back-flushing) should be specified, e.g. control of flow rate or pressure.

Where gas flow is programmed, the minimum and maximum flow rates and/or pressures should be specified, together with the precision limits for the programme profile. Suitable equipment for checking flow rates and/or pressure should normally be specified, together with the required discrimination. The units should comply with the appropriate requirements of 8.1.

6.2 Injection equipment

6.2.1 Injection device. The injection device, which is not normally permanently attached to the apparatus, should be specified in respect of type, capacity and, if necessary, the manufacturer's reference, e.g. "10 µl glass injection syringe" or "10 ml gas-tight syringe".

6.2.2 Injection system. The injection system is normally an integral part of the chromatographic apparatus and should be specified by type, e.g. "direct on-column", "heated injection block", "gas sampling valve with a sample volume of 20 ml".

Where appropriate, the material of construction (including that used for the injection septa), special design features, manufacturer's reference and lubricants (where used) should be stated.

Where the injection system is required to be heated, the appropriate nominal range of operating temperatures should be specified. Details should preferably be given of suitable temperature monitoring facilities.

6.2.3 Flow splitter. If inlet splitters are required, nominal split ratios should be stated. If it is important, materials of construction and layout should be described; if necessary, the manufacturer's reference should be given.

6.3 Columns (and precolumn if required)

6.3.1 Number. The number of columns should be stated.

6.3.2 Column details. This subclause should specify the column system required. The following information should always be given.

- a) The wall material, including any special pretreatment required.
- b) The packed length, in metres, and nominal internal diameter, in millimetres.
- c) Additional relevant information such as wall thickness, the composition of any end plugs used, details of connections and requirements for sealing compounds.

The form of column should be specified only when necessary. The expected life of the column should be stated, either in hours at operating conditions or as a number of injections.

This subclause should also indicate such requirements for the preparation of the column as are necessary to comply with the requirements of clause 8.

Column packing should be specified in 6.3.3.

6.3.3 Packing

6.3.3.1 General. This subclause should specify such requirements for the preparation of the column packing as are necessary to comply with the requirements of clause 8.

The column packing should be clearly identified and should be characterized by the retention parameters of selected calibration compounds. If a physical or chemical property of a packing component can be correlated with the retention behaviour, this property should be included in the description.

It is preferable to use commercially available materials. However, if the liquid phase or active solid is not commercially available, adequate details of preparation should be given.

The method of packing, if critical to the method, should be adequately described.

These details should be suitably incorporated in 6.3.3.2 to 6.3.3.6.

6.3.3.2 Support. Special treatment of the solid support, if required by the method, should be described.

6.3.3.3 Stationary phase. The mass of liquid phase and of the solid support, and the method of incorporating a liquid phase on a solid support, or the method of coating open tubular columns, should be given, or reference made to published literature. Detailed description should be given of any column not commercially available.

6.3.3.4 Active adsorbent. When an active solid is used, either alone or modified with an additive, procedures should be given for its preparation and/or treatment, or reference made to published literature. Detailed descriptions should be given of any materials not commercially available.

6.3.3.5 Other packing. Packing materials not already included in 6.3.3.2 to 6.3.3.4 should be specified in sufficient detail to facilitate their preparation and/or treatment.

6.3.3.6 Conditioning. The method of conditioning, if critical, should be described. It is recommended that the temperature(s) and time(s) of conditioning should be stated, together with the carrier gas purity and flow rate.

6.3.4 Efficiency and resolution

6.3.4.1 Efficiency under given conditions. It is desirable to quote the minimum theoretical plate number for a given amount of a specified compound.

6.3.4.2 Resolution under given conditions. The allowable peak resolution on the critical separations should be specified in terms such as the minimum value of the resolution R or the maximum value of $\frac{\text{valley height}}{\text{smaller peak height}}$.

Valley height and peak height should be measured from the base line and should be exemplified on the typical chromatogram (see 8.4.2).

6.4 Detector. The detector type should be specified, together with evidence of its suitability for use in the method under consideration. Such information should include details of the essential assembly and materials of construction.

NOTE If radioactive sources are used, a warning should be inserted at this point and reference made to clause 10.

The performance characteristics of the detector or detector/amplifier system should be specified, together with the attenuation and the time constant.

Additional information may be required in particular cases.

The following are examples.

- a) *Electron capture detector voltage supply.* The nature of the supply should be stated, i.e. direct current or pulsed. In the latter case, the pulse repetition rate, pulse width and pulse amplitude should be specified.
- b) *Katharometer supply.* The value of the bridge current (or voltage, element temperature or resistance), together with its stability requirements, should be specified.