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Recommendations for

A standard layout for methods of chemical analysis by gas chromatography

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Cooperating organizations

The panel responsible for the preparation of this British Standard, which has been working under the direction of the Chemical Divisional Council, consists of representatives from the following Government departments and scientific and industrial organizations:

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.

6.5 *Potentiometric recorders.* If measurements are to be made from potentiometric recorder charts, the following characteristics should be specified:

minimum chart width

span

total response time

chart speed

dead band

noise

The resolution and accuracy of all measuring equipment used, including rulers and chart divisions, should be stated.

6.6 *Integrators.* If an integrator is used, the minimum number of counts (or the equivalent analogue measurement) and the minimum linearity compatible with the requirements of **6.3.4** and **8.3.3** should be specified. Other factors that may require specification are the response time, dead band, drift and the setting of peak sensing controls.

In the case of integrators such as the ball-and-disc integrator, which are used in conjunction with a potentiometric recorder, the performance requirements necessarily apply to the overall combination.

6.7 *Accessories.* Specialized equipment should be adequately described. Where possible, suitable diagrams should be included and, where appropriate, manufacturers' references should be given.

7 Sample

7.1 General. Detailed sampling procedures are not included in these recommendations. Procedures for sampling specific products may be obtained from relevant standards, and general information on sampling is given in BS 5309-1 to BS 5309-4.

The conditions under which the laboratory sample is stored prior to analysis should be stated. Factors known to affect the stability of the sample should be indicated and suitable recommendations made to avoid deterioration.

7.2 Preparation of test portions. Sufficient detail should be given to ensure that a homogeneous representative test portion is obtained from the laboratory sample for analysis.

The following points should be considered:

a) technique(s) for changing the phase of the sample;

b) dilution of the sample;

c) chemical treatment of the sample;

d) extraction of the sample.

8 Procedure

This clause should specify, with appropriate details of measurement techniques, the operating temperatures, gas flow rates, duration of analysis, splitters, pressures and detectors necessary to comply with the requirements of **6.3.4** and **8.3.3**.

The sequence and timing of operations appropriate to the apparatus specified in clause **6**, together with any other relevant information of this kind, should be given.

8.1 Setting up the apparatus. Temperature should be expressed as nominal in degrees Celsius, in multiples of 5.

8.1.1 *Injector.* If the injection system is fitted in a separately controlled enclosure, the nominal temperature should be quoted.

8.1.2 ***Oven and column***

8.1.2.1 *Temperature.* Ideally, column temperatures should always be specified but it is usual, in practice, to specify the temperature of the column environment; when temperature programming is required, the programme(s) should be described in detail, using appropriate terms from the following list.

a) Suitable criteria for cooling and stabilizing before injection.

b) Nominal initial temperature and the time for which this is held after injection.

c) Linear programme rate (in nominal degrees Celsius per minute).

d) Nominal final temperature and the time for which this is held before cooling.

8.1.2.2 *Rate of flow of gases.* Gas flow rates are conveniently indicated in terms of retention time, in seconds, although volumetric units at 20 °C and 101.3 kPa pressure should be used where the retention time is small. The corresponding inlet pressures should be indicated where appropriate.

a) *Column.* The absolute retention time of the "air" peak should be specified in seconds; if less than 10 s, the column flow rate at a temperature of 20 °C and 101.3 kPa pressure should be specified in millilitres per minute.

b) *Auxiliary gas flow rates.* Auxiliary gas flow rates should be specified only when such information is necessary to obtain the performance defined in **6.3.4** and **8.3.3**, or when they deviate from the manufacturer's instructions.

c) Where temperature programmed operation is used, the gas chromatography equipment manufacturer's recommendations for balancing the columns should be followed.

8.1.3 *Flow splitter.* If an inlet or effluent gas flow splitter is used, the approximate ratio of the split should be specified, and a method included for its measurement under the conditions of use, as the ratio of the exit streams under the same temperature and pressure.

8.1.4 *Detector.* If the detector is fitted in a separately controlled enclosure, the nominal temperature should be specified.

Any other operating parameters specific to the detector that are necessary to meet the requirements of **6.3.5** and **8.3.3** should be specified at this point.

8.1.5 *Recorder.* A recorder, if used, should be specified so as to be set up in accordance with **6.5**.

8.1.6 *Integrator.* An integrator, if used, should be specified so as to be set up in accordance with **6.6**.

8.2 Calibration. This subclause describes the procedure used to calibrate the system. Various calibration procedures may be used but in most cases these involve the use of a series of mixtures of known concentrations covering the expected range for each of the compounds to be determined in the analysis. The method of calibration should be described in detail.

The method should specify that the retention data obtained from the calibration mixtures are used for designating peaks in the chromatogram of the sample.

8.2.1 *Methods of calibration.* Many methods of calibration exist; the most generally used are those given in **8.2.1.1** to **8.2.1.5**.

8.2.1.1 *Internal normalization.* All the components are eluted and all the peaks are measured. The measurements are normalized to 100 with the calibration factor determined either by calculation or by measurement at each analysis (see **8.2.3**).

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

The procedure used for the addition of these materials should be stated. The size and materials of construction of vessels to be used, and the method of stoppering, should be given. The quantities may be specified in units of volume or mass but the order of addition should be given.

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

8.2.1.4 *Absolute method.* A known quantity of sample is injected and peak measurements are carried out. Knowing the settings of the apparatus, the quantity of the component corresponding to each peak is then calculated or determined from tables, graphs, charts, etc.

8.2.1.5 *Calibration by addition.* The following are subjected in turn to chromatography.

a) The sample. The component to be determined corresponding to a peak measurement, $A_{i,1}$, and a component giving a neighbouring peak corresponding to a peak measurement $A_{j,1}$ (see section 3, clause **5**).

b) A reference mixture of a quantity, m, of the sample and a known quantity, m_i, of the component to be determined. A peak measurement, $A_{i,2}$, corresponding to the total component to be measured in the mixture, and a peak measurement, $A_{j,2}$, corresponding to the neighbouring peak (see section 3, clause **5**).

The conditions of application of the five methods of calibration mentioned above are summarized in Table 1.

8.2.2 ***Standard mixtures***

8.2.2.1 *Purity of components.* The purity and/or composition of the components used in making the calibration mixtures should be specified in accordance with **8.2.2.1** a) and **8.2.2.1** b). **8.2.2.1** c) gives recommendations for components containing impurities that cause interference for which corrections have to be made.

a) The composition of the components should be specified by reference to an analytical method that is different from the one being drafted, preferably not gas chromatographic.

b) The individual impurities in the components should be specified in terms of the maximum peak height or area permissible when the component is chromatographed under the standard conditions of the method being drafted. More severe limits should normally be specified separately on impurities with retention times likely to interfere with peaks to be measured in the mixture, if corrections for these interferences are not to be made.

c) If it is necessary to specify the use of components that contain impurities causing interferences for which corrections have to be made, the permissible levels of these impurities should be stated and a suitable means of correcting for the associated interferences should be given.

Table 1 — Conditions of application of methods of calibration

Method	Eluted components	Detector linearity	Volume injected	Operating conditions of the chromatograph
Internal normalization	All components	Necessary	Nominal	Stable during the analysis
Internal calibration	Components to be determined	Necessary	Nominal	Stable during the analysis
External calibration	Components to be determined	Necessary	Reproducible	Stable during at least two consecutive analyses
Absolute calibration	Components to be determined	Optional	Known and reproducible	Stable during a series of analyses reproducible from one day to another
Calibration by addition	Components to be determined	Necessary	Nominal	Stable during at least two consecutive analyses

8.2.2.2 *Operating frequency.* The minimum number of different calibration mixtures to be used should be specified. A minimum number of repeat injections for each mixture should also be specified.

8.2.2.3 *Method of preparation of the standard mixture or of the series of standard mixtures*

a) For calibration mixtures containing liquids and/or solids, precise details of the method of weighing should be given, including the size and type of container, and the order of addition of components.

b) For calibration mixtures containing gases, precise details of the method of mixing should be given, including the size of the container, the material of manufacture of the container, the method for obtaining adequate mixing and any pressure limitations.

The composition of the calibration mixtures should be specified.

8.2.2.4 *Conditions specific to the use of standard mixtures in chromatography.* The procedures for chromatographing the calibration mixture and for measuring and calculating the results are normally identical with those used for the sample. In these circumstances, it is sufficient only to make reference to the appropriate clauses associated with the sample.

8.2.3 *Presentation of the calibration data.* This may be in the form of graphs or factors.

8.2.3.1 *Calibration graphs*

a) The calibration results should be presented in the form of a graph for each component to be measured. Limits should be specified for the scale of a graph to ensure sufficient discrimination in its reading. Reference should also be made to **8.4**.

b) The calibration graph should take the form of a smooth curve and it should be specified that none of the associated calibration points lie more than a given percentage of their value from the curve. This limit should be in accordance with **9.2**.

8.2.3.2 *Factors.* The following information, as appropriate, should be given:

a) method of calculation from the results of chromatography of standard mixtures (the unit should be indicated);

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

c) approximate estimations, e.g. the case where the factors are considered to be equal to 1 (it should then be indicated that it is an approximation); rules to be followed in the case of unidentified peaks.

8.3 Test

8.3.1 *Preparation of the test portion or the standard mixture.* The following information, as appropriate, should be given in respect of the preparation of the test portion [in the case of internal calibration (see **8.2.1.2**)] or of the standard mixture [in the case of calibration by addition (see **8.2.1.5**)]:

a) the volume or mass of the sample;

b) the volume or mass of the standard solution (or addition);

c) the method of mixing the standard solution (or addition) with the sample.

8.3.2 *Introduction of the test portion.* Details of techniques should be given, and these should include at least the sample size and the duration of injection. With sampling valves, details of the procedure for filling the sampling valve should be given.

If special precautions are necessary to avoid contamination, these should be specified, together with the procedure for cleaning out the sampling device.

The minimum time between injections, to ensure that the various components are fully eluted, should be specified. Any time required for cooling and stabilization and/or back-flushing should also be given.

8.3.3 *Recording.* Details should be given of the following aspects of recording.

a) *Base line drift.* The maximum allowable drift in base line from the start to the end of the analysis should be specified in terms of a percentage full scale deflection at the lowest attenuation to be used during the analysis.

b) *Base line noise and wander.* The maximum allowable base line noise and wander, at all sensitivities used for the analysis, should be specified.

c) *Retention data.* Maximum and minimum limits of the retention volume or time of at least two compounds having peaks widely spaced on the chromatogram should be specified wherever possible.

d) *Peak height of sample components.* To ensure that an adequate signal to dead band ratio is available, the minimum peak height, or equivalent, for each component (whether measured by height or by area) should be specified.

e) *Peak height of internal standard.* If an internal standard is used in the method, the minimum peak height should be specified. The maximum difference between the internal standard peak height when a sample is analysed and the average internal standard peak height obtained during the calibration should be specified.

f) *Integrator counts.* The minimum number of counts for the smallest peak area to be measured by an integrator, and used in the calculation of results, should be specified.

g) *Peak width.* If peak width is to be measured, the minimum width of the narrowest peak to be used in the calculation of results should be specified.

8.4 Examination of the chromatograms

8.4.1 *Information required.* The following information should be made available for examination of the chromatograms.

a) *Identification.* A list should be given of the components known to exist in the test sample, including impurities that may occasionally be present.

The typical retention data of these components should be given for the method, with reference to the typical chromatogram. The method of measurement of the retention data (retention volume, retention distance, etc.) should always be stated, together with details of interpretation, e.g. relative retention time, retention index.

b) *Measurement of peak size.* The method of measurement of the peaks (peak height, integrator counts, etc.) should always be specified. The procedure for establishing the peak base should be given and, if necessary, exemplified by reference to the typical chromatogram. This is particularly important with partially resolved peaks.

8.4.2 *Typical chromatograms.* Each method should include at least one typical chromatogram of adequate size, on which the peak base should be drawn under each peak to be measured. The chromatogram should be clearly labelled with the following information:

a) a brief title of the analysis;

b) the injection point and approximate position of "air" peak (where appropriate);

c) the identity and approximate component concentration for each peak (the positions of any other possible components may also be indicated);

d) the internal standard peak, where appropriate;

e) the points at which the base line position should be determined for drift;

f) the periods during which base line noise and wander should be assessed;

g) the attenuation for each portion of the chromatogram, relative to the most sensitive setting equalling 1, e.g. a peak recorded at one quarter of this sensitivity should be marked "attenuation × 4";

h) the points at which flow switching or any other operation takes place;

i) the time scale;

j) the locations at which the component resolution should be determined.

The chromatogram should also indicate any necessary methods for allocation of areas of partially resolved peaks.

8.4.3 *Qualitative analysis.* Where applicable, the following should be specified:

a) order of elution of components;

b) retention characteristics;

c) possible interferences;

d) complementary methods of identification.

8.4.4 *Quantitative analysis.* Where applicable, the following should be specified:

a) methods of measurement, e.g. height of peaks, peak height multiplied by width, triangle constructed on the peaks, planimetry, cutting and weighing, integration, etc.;

b) calculations (transfer to calibration graphs or the use of charts; formulae to be applied);

c) special methods.

9 Expression of results

9.1 Qualitative analysis. As applicable, the way in which the following information is to be reported should be specified:

a) number of components found;

b) names of the components identified.

9.2 Quantitative analysis. This subclause should specify the way in which the test result is to be reported. In addition, it should give a quantitative statement of the agreement to be expected between test results for the analyses of the sample under the conditions laid down in the standard method. The two main parameters, repeatability and reproducibility, should be employed as indicated in **9.2** a) and b), so that the significance of results can be assessed. It may also be necessary to give a table or graph of precision data.

a) Repeatability. This should be in accordance with the recommendations given in BS 5497-1. Suggested wording is as follows:

> The difference between two single results found on identical test material by one operator using the same apparatus within the shortest feasible time interval will not exceed the repeatability value *r* on average more than once in 20 cases in the normal and correct operation of the method.

b) *Reproducibility*. This should be in accordance with the recommendations given in BS 5497-1. Suggested wording is:

> Single results on identical test material reported by two laboratories will differ by more than the reproducibility value *R* on average not more than one in 20 cases in the normal and correct operation of the method.

9.3 Unassigned peaks. Any peak to be reported, but not assigned a chemical identity by the method, should be designated in terms of retention value and, if applicable, the quantitative basis for its calculation stated.

10 Notes on procedure, particularly with reference to hazards

Warning should be given in clause **5** of hazards arising from the use of materials required for the analysis.

Where appropriate, the hazards should be grouped and detailed in clause **10** under an appropriate heading, together with information about the precautions to be taken (alternatively, reference may be made to a suitable source of such information listed in clause **12**). Examples of headings that may be required are:

a) toxic hazards;

b) explosive hazards;

c) radioactive hazards.

If no special hazards are known, this should be mentioned in clause **10**. However, a statement such as ”no hazards’ should never be used.

11 Test report

The test report should include the following particulars:

a) the reference of the method used;

b) the results and the method of expression used;

c) any unusual features noted during the determination;

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

12 Bibliographical references

If bibliographical references (i.e. those which do not refer to other standards) are considered necessary, they should be given.

NOTE Attention is drawn to BS 1629 and BS 4148.

13 Appendices

Appendices should be used when it is desirable to relieve the body of the document of detailed information that can be more conveniently presented in that form. They should bear titles and should be identified by a letter, starting at A and omitting I, O and X.

Section 3. Calculation formulae depending on the method of calibration used

1 Internal normalization method

The content of component i in the test portion expressed as a percentage is given by the formula

$$\frac{100 \times K_i \times A_i}{\Sigma KA}$$

where

K_i is the coefficient of proportionality for the component i

A_i is the measurement of the peak of component i, determined in accordance with the requirements of **8.4.4**

2 Internal standard method

The content of component i in the test portion, expressed as a percentage, by mass, is given by the formula

$$\frac{100 \times m_{\mathrm{E}} \times A_i \times K_{\mathrm{E},i}}{m \times A_E}$$

where

m is the mass of the test portion (in g)

m_{E} is the mass of the standard sample added (in g)

A_i is the measurement of the peak of component i, determined in accordance with the requirements of **8.4.4**

A_{E} is the measurement of the peak of the standard sample

$K_{\mathrm{E},i}$ is the coefficient of proportionality relating to the component i in comparison with the standard sample

3 External standard method

The content of component i in the test portion is given by the formula

$$E_i \frac{A_i}{A_{\mathrm{E}}}$$

where

E_i is the content of the compound i in the standard mixture

A_i is the measurement of the peak of component i on the chromatogram of the test portion

A_{E} is the measurement of the peak of component i on the chromatogram of the standard sample

4 Absolute method

Reference should be made to the tables, graphs and charts, as described in **8.2.1.4**.

5 Addition method

The content of component *i* in the test portion, expressed as a percentage, by mass, is given by the formula

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

where

$A_{i,1}$ is the measurement of the peak of component *i* on the chromatogram of the test portion

$A_{i,2}$ is the measurement of the peak of component *i* on the chromatogram of *m* grams of sample with the addition of m_i grams of the component *i* to be determined (matching mixture)

$A_{j,1}$ is the measurement of the peak *j* in the vicinity of the peak for component *i* on the chromatogram of the test portion

$A_{j,2}$ is the measurement of the same peak on the chromatogram of the test portion with addition (matching mixture)

m is the mass of the test portion (in g)

m_i is the mass added of the substance *i* to be determined (in g)

Section 4. Typical example of use of recommended standard layout

Introduction

This section sets out a gas chromatographic method drafted in accordance with the recommendations of this standard.

In drafting this example, clauses contained in the recommendations that are irrelevant to the method under consideration have been omitted. It should therefore be noted that, as the remaining clauses are numbered sequentially, the clause numbering of the example does not completely correspond to that of the recommendations.

NOTE The method given in this example does not constitute a British Standard (see the foreword).

Determination of styrene in polystyrene by gas chromatography

1 Scope and field of application

This document specifies a gas chromatographic method for the determination of styrene in all grades of styrene homopolymer. It is applicable to products having styrene contents, expressed as a percentage (*m*/*m*), in the range 0.02 to 5.00.

2 Principle

A known mass (approximately 2 g) of the sample is dissolved in dimethylformamide containing a known amount of 1,2,4-trimethylbenzene as internal standard. A portion of this solution is analysed by gas chromatography, the concentration of styrene being determined from the ratio of its peak height to that of the 1,2,4-trimethylbenzene.

The gas chromatograph is fitted with a column maintained at a temperature of 100 °C, packed with diatomaceous earth coated with polyethylene glycol (average relative molar mass 6 000), and a flame ionization detector.

3 Materials required

3.1 Carrier gas and auxiliary gases

3.1.1 *Nitrogen* at not less than 250 kPa pressure.

3.1.2 *Hydrogen*, at not less than 150 kPa pressure.

WARNING. This material is potentially explosive (see clause **8**).

3.1.3 *Air*, at not less than 250 kPa pressure.

3.1.4 *Methane.* Commercial grades, in cylinders, are normally suitable.

WARNING. This material is potentially explosive (see clause **8**).

3.2 Materials for the preparation of calibration samples

3.2.1 *Styrene,* containing not less than 99% styrene (see **6.2.2.1**).

Distil not more than 4 h prior to use and store at a temperature of 0 °C.

WARNING. Styrene monomer vapour is toxic (see clause **8**).

3.2.2 *1,2,4-trimethylbenzene,* not less than 90 % pure (see **6.2.2.1**).

WARNING. This material is toxic (see clause **8**).

3.2.3 *Dimethylformamide,* complying with the requirements specified in **6.2.2**.

Distil prior to use, rejecting the first 15 % of the distillate. This separates the small amount of "light ends" that could interfere with the measurement of very volatile components in the polymer sample.

WARNING. This material is toxic (see clause **8**).

3.2.4 *Dichloromethane (methylene chloride),* laboratory grade.

WARNING. This material is toxic (see clause **8**).

3.3 Materials for the preparation of columns

3.3.1 *Polyethylene glycol,* average relative molar mass 6 000.

3.3.2 *Diatomaceous earth,* particle size approximately 190 μm to 250 μm. The materials shall be such that the packed column complies with the requirements specified in **4.3.3** and **6.3.3**.

4 Apparatus

4.1 *General description*

4.1.1 *Gas chromatographic system.* The system comprises a gas chromatograph with a flame ionization detector and a heated injection block with disposable glass liner[1)].

4.1.2 *Characteristics of the assembly*

4.1.2.1 *Column temperature*

a) *Isothermal temperature control range.* Between 95 °C and 105 °C, controlled to within ± 0.5 °C.

b) *Temperature read-out,* capable of checking that the temperature is within the range 95 °C to 105 °C with a discrimination of at least 2 °C.

4.1.2.2 *Detector temperature.* Chromatographs without separate detector heating are normally suitable. If separate detector heating is used, the detector environment temperature should be within the range 95 °C to 200 °C.

4.1.3 *Gas controls and flow measurement*

Pressure regulators capable of controlling gas flows as follows.

a) *Nitrogen.* Flow rate of approximately 50 ml/min at a pressure of 200 kPa.

b) *Hydrogen.* Flow rate of 25 ml/min at a pressure of 100 kPa.

c) *Air.* Flow rates of 100 ml/min to 600 ml/min at a pressure of 200 kPa.

Pressure regulators supplied by manufacturers of gas chromatographic equipment are usually adequate. Normal cylinder head regulators should not be used to control the supply of hydrogen and nitrogen and should be replaced by pressure regulating devices with discrimination better than 10 kPa.

4.2 *Injection equipment*

4.2.1 *Injection device.* Syringe, 10 μl capacity, fitted with a needle of the length recommended by the gas chromatograph supplier.

4.2.2 *Injection system,* comprising a separately heated injection block, fitted with a disposable glass liner, capable of being controlled within the nominal temperature range 200 °C to 250 °C. Temperature monitoring facilities are recommended.

[1)] Satisfactory results have been obtained using a Parkin Elmer Model F11 or a Varian Aerograph Model 1200. Other gas chromatographs may be used if known to be suitable.

4.3 *Columns*

4.3.1 *Construction.* The column shall be constructed of copper tubing (cleaned by washing with acetone prior to packing), internal diameter 2 mm (nominal), packed length 3 m. When packed with the stationary phase (see **4.3.2.1**) it should have a lifetime of at least 1 year at operating conditions.

4.3.2 *Packing*

4.3.2.1 *Stationary phase.* Dissolve 15 g of the polyethylene glycol (see **3.3.1**) in approximately 50 ml of the dichloromethane (see **3.2.4**). Add 85 g of the diatomaceous earth (see **3.3.2**) to form a slurry. Heat on a water bath with occasional stirring until the solvent is removed. Dry in an oven controlled at a temperature of about 50 °C for 1 h. This will provide sufficient for three columns.

4.3.2.2 *Conditioning.* Purge for 16 h with the nitrogen (**3.1.1**) at a temperature of 100 °C using a flow rate of between 40 ml/min and 50 ml/min and with the column outlet disconnected from the detector.

4.3.3 *Efficiency and resolution.* The performance criteria specified in **4.3.3.1**, **4.3.3.2** and **6.3.3** shall apply to both the calibration mixtures and the samples.

4.3.3.1 *Efficiency.* The theoretical plate number, as measured on the 1,2,4-trimethylbenzene peak, shall be not less than 3 000.

4.3.3.2 *Resolution.* The value of the ratio

$$\frac{\text{valley height between 1,2,4-trimethylbenzene and styrene}}{\text{peak height of 1,2,4-trimethylbenzene}}$$

shall not exceed 0.02.

The valley is measured from the base line as shown on the typical chromatogram (see Figure 1).

4.4 *Flame ionization detector,* capable of satisfactory performance (see **4.3.3** and **6.3.3**) with a carrier gas flow rate of approximately 50 ml/min.

The detector/amplifier measuring system shall be capable of detecting 0.02 % of styrene in 0.5 µl of the sample under the conditions of the method. A matched attenuator is required. Attenuation in factors of 2 is recommended.

The amplifier time constant shall be less than 0.3 s.

4.5 *Potentiometric recorder,* having the following characteristics:

a) 1 s (nominal) maximum time for 98 % full scale response;

b) minimum chart width 200 mm;

c) suitable for a nominal chart speed of 10 mm/min;

d) dead band not greater than 0.3 % of full scale;

e) noise less than 0.4 % of full scale;

f) linearity better than 0.4 % of full scale.

The recommendations of the detector/amplifier manufacturer should be followed in selecting a suitable recorder.

The measurement of peak heights requires a scale with a discrimination of 1 mm. If digital integration is used, not less than 100 counts should be obtained for the smallest peak to be measured. The linearity should be better than 1 % over the range used. Most integrators sold for gas chromatographic use are suitable. Special facilities such as base line drift correction are not essential.

5 Sample

Store the sample in a suitably sealed container, preferably in cool conditions, to minimize loss of styrene by volatilization.

6 Procedure

6.1 Setting up the apparatus

6.1.1 *Injection system.* Maintain the temperature between 200 °C and 250 °C. Change the glass liner (see **4.1.1**) after 10 injections.

6.1.2 ***Oven and column***

6.1.2.1 *Column temperature.* Maintain the temperature at 100 °C (nominal).

6.1.2.2 *Rate of flow of carrier gas*

a) *Column.* Adjust to obtain a time in the range 27 s to 33 s for the "air" peak, calculated from the elution time of methane. This corresponds to an inlet pressure of approximately 200 kPa.

b) *Auxiliary gas flow rates.* Follow the apparatus manufacturer's recommendations.

6.1.3 *Detector temperature.* If separately heated, maintain the temperature of the detector enclosure between 95 °C and 200 °C.

6.2 Calibration. Calibrate by internal calibration.

6.2.1 *Preparation of internal standard solution.* Add 500 µl of the 1,2,4-trimethylbenzene (see **3.2.2**) to approximately 1 000 ml of the dimethylformamide (see **3.2.3**) contained in a suitable flask. Stopper the flask and mix the contents thoroughly.

6.2.2 ***Standard mixtures***

6.2.2.1 *Purity of components*

a) The purity of the styrene (see **3.2.1**) shall be not less than 99 % *m*/*m*.

b) The peak heights for impurities in the styrene (see **3.2.1**) shall be not greater than 2 % of the styrene peak height when 1 µl of a mixture of 1.5 g of the mixture containing the highest concentration of the styrene (see **3.2.1**) and 20 ml of the dimethylformamide (see **3.2.3**) is chromatographed under the conditions of this method. For any impurity eluted at a retention time that would interfere with the 1,2,4-trimethylbenzene (see **3.2.2**), the peak height shall be not greater than 1 % of the styrene peak height.

c) The purity requirement for the 1,2,4-trimethylbenzene is such that, when 1 µl of the internal standard solution (see **6.2.1**) is chromatographed under the conditions of this method, the peak heights of impurities shall be not greater than 2 % of the styrene peak height obtained when the mixture containing the highest concentration of styrene is chromatographed [see **6.2.2.1** b)].

6.2.2.2 *Operating frequency.* Carry out the calibration at least daily for routine operation and recalibrate whenever the apparatus has been shut down and restarted. The apparatus is satisfactory if the calibration has not changed by more than half the value of the repeatability.

6.2.2.3 *Preparation of standard mixtures.* Prepare a series of five standard mixtures of styrene in dimethylformamide having the following nominal concentrations of styrene:

Mixture	% **styrene** (*m*/*m*)
A	0.02
B	0.10
C	0.50
D	2.50
E	5.00

Prepare each mixture as follows.

Weigh accurately into a 100 ml one-mark volumetric flask approximately 50 ml of the dimethylformamide (see **3.2.3**). Add the appropriate volume of the styrene (see **3.2.1**) (between 20 µl and 5 000 µl is required) and reweigh. Dilute to the mark with more of the dimethylformamide and reweigh. Stopper the flask and mix well.

Calculate the exact concentration of styrene in the solution.

6.2.2.4 *Conditions specific to the use of the standard mixtures.* Use the procedure described in **6.3** for chromatographing the standard mixtures (see **6.2.2.3**) and measuring the results, except that 1.5 g of the appropriate standard mixture shall be used in place of the test portion.

6.2.3 ***Presentation of the calibration data***

6.2.3.1 *Calibration graph*

a) Prepare a graph in which the ratio of peak height of styrene to that of 1,2,4-trimethylbenzene is plotted against the mass, in grams, of styrene in the standard mixture. The scale of the graph shall be 0.01 per millimetre for the peak height ratio and 0.0005 g per millimetre for the mass of styrene.

b) The calibration graph should take the form of a smooth line such that no point lies more than half the value for repeatability (see Figure 2 and **7.1**) from the mean line. If such a point is found, check the whole apparatus for faults and repeat the entire calibration procedure.

Extrapolation of the calibration line to cover samples lying outside the calibrated range is not permitted.

6.3 Test

6.3.1 *Addition of internal standard to the test portion.* Weigh accurately approximately 1.5 g of the test sample (see clause **5**) into a 50 ml conical flask fitted with a ground glass stopper. Pipette 20 ml of the internal standard solution (see **6.2.1**) into the flask. Stopper the flask and shake it, using a mechanical shaker, until all the polymer has dissolved. This is generally complete within 1 h.

NOTE If the polymer contains fillers, cross-linking agents or some other materials, a cloudy or opaque solution will be obtained. This does not generally affect the analysis but care should be taken to ensure that any solid matter present does not block the syringe.

6.3.2 *Introduction of the test portion, operation of equipment and time between injections.* Inject a nominal fixed volume of between 0.5 µl and 1.0 µl of the prepared solution (see **6.3.1**) into the chromatograph. Leave the syringe needle in the injection block for 5 s.

In operating the equipment, make attenuation changes in accordance with the values obtained in **4.3.3** and **6.3.3** and as indicated on the typical chromatogram (see Figure 1).

Allow a minimum time of 25 min between injections.

6.3.3 *Recording.* During the test, ensure that recording complies with the following requirements.

a) *Base line drift.* The base line drift throughout the entire analysis, as shown on the typical chromatogram, shall be not greater than 1 % of full scale deflection at the lowest attenuation used during the analysis.

b) *Base line noise and wander.* The base line noise and wander shall be not greater than 1 % of full scale deflection throughout the duration of the chromatogram.

c) *Retention time.* The retention times shall fall between the following limits.

Compound	Retention time	
	minimum	maximum
	min	min
Styrene	6.5	7.5
1,2,4-trimethylbenzene	7.5	9.5

d) *Peak height of sample components.* The peak height to be measured for styrene shall be not less than 10 % of full scale deflection.

e) *Peak height of internal standard.* The peak height to be measured for 1,2,4-trimethylbenzene shall be not less than 40 % of full scale deflection.

The difference between the peak height of 1,2,4-trimethylbenzene measured for a sample and the average peak height of 1,2,4-trimethylbenzene obtained on calibration samples shall be not more than 10 %.

f) *Integrator counts.* If digital integration is used, not less than 100 counts should be obtained for the smallest peak to be measured.

6.4 Examination of the chromatograms

6.4.1 *Typical chromatogram.* A typical chromatogram for the analysis is shown in Figure 1.

6.4.2 *Identification.* A list of the compounds likely to be present, together with their typical relative retentions, is given below (see also Figure 1). The retention times measured on the calibration mixtures are used for identifying the peaks in the samples.

Compound	Typical relative retention time
Injection point	– 0.11
"Air" peak (methane)	0.00
Ethylbenzene, *p*-xylene	0.40
m-xylene	0.43
*iso*propylbenzene	0.52
o-xylene	0.57
n-propylbenzene	0.65
Ethylmethylbenzenes	0.72
Styrene	0.88
1,2,4-trimethylbenzene (internal standard)	1.00

Check for the absence of components eluting in the same position as 1,2,4-trimethylbenzene by chromatographing the same concentration of the sample dissolved in dimethylformamide but without addition of 1,2,4-trimethylbenzene.

6.4.3 *Determination of styrene monomer content.* Draw in the peak base and measure the peak heights, in millimetres, to the nearest 1 mm, of 1,2,4-trimethylbenzene and styrene as shown on the typical chromatogram (Figure 1).

Calculate the ratio of the peak height of styrene to the peak height of the internal standard solution (see **6.2.1**). From the calibration graph (see **6.2.3.1**) read off the mass, in grams, of styrene corresponding to this value.

The styrene monomer content, expressed as a percentage by mass, is given by the formula

$$\frac{m_1}{m_o} \times 100$$

where

m_1 is the mass of styrene found (in g)

m_o is the mass of the test portion (in g)

7 Expression of results

The considerations given in **7.1** and **7.2** shall apply in the interpretation of results obtained for the determination of the styrene content.

7.1 Repeatability. The difference in two test results obtained by the same operator with the same apparatus in a given laboratory under constant operating conditions on identical test material should, in the long run, in the normal and correct operation of the test method, not exceed the amount shown in Figure 2.

7.2 Reproducibility. The difference between two single and independent results obtained by different operators working in different laboratories on identical test material should, in the long run, in the normal and correct operation of the test method, not exceed the amount shown in Figure 2.

8 Hazards

8.1 Toxicity

a) Styrene monomer vapour is a toxic irritant; threshold limit value (TLV) 100 p.p.m. (see **10.1**).

b) Dimethylformamide is toxic by contact with the skin; TLV 20 p.p.m. (see **10.2**).

c) 1,2,4-trimethylbenzene is a depressant and a respiratory irritant (see **10.3**).

d) Dichloromethane, particularly its vapour, is toxic; TLV 500 p.p.m. (see **10.4**).

8.2 Explosivity. Hydrogen is explosive when mixed with air at concentrations ranging approximately from 4 % to 75 % (*v*/*v*). All joints and lines carrying hydrogen shall be regularly checked for leakage. Particular attention shall be paid to prevent leakage of hydrogen into a confined space.

Similar precautions should also be observed with methane which is explosive when mixed with air at concentrations ranging approximately from 5 % to 15 % (*v*/*v*).

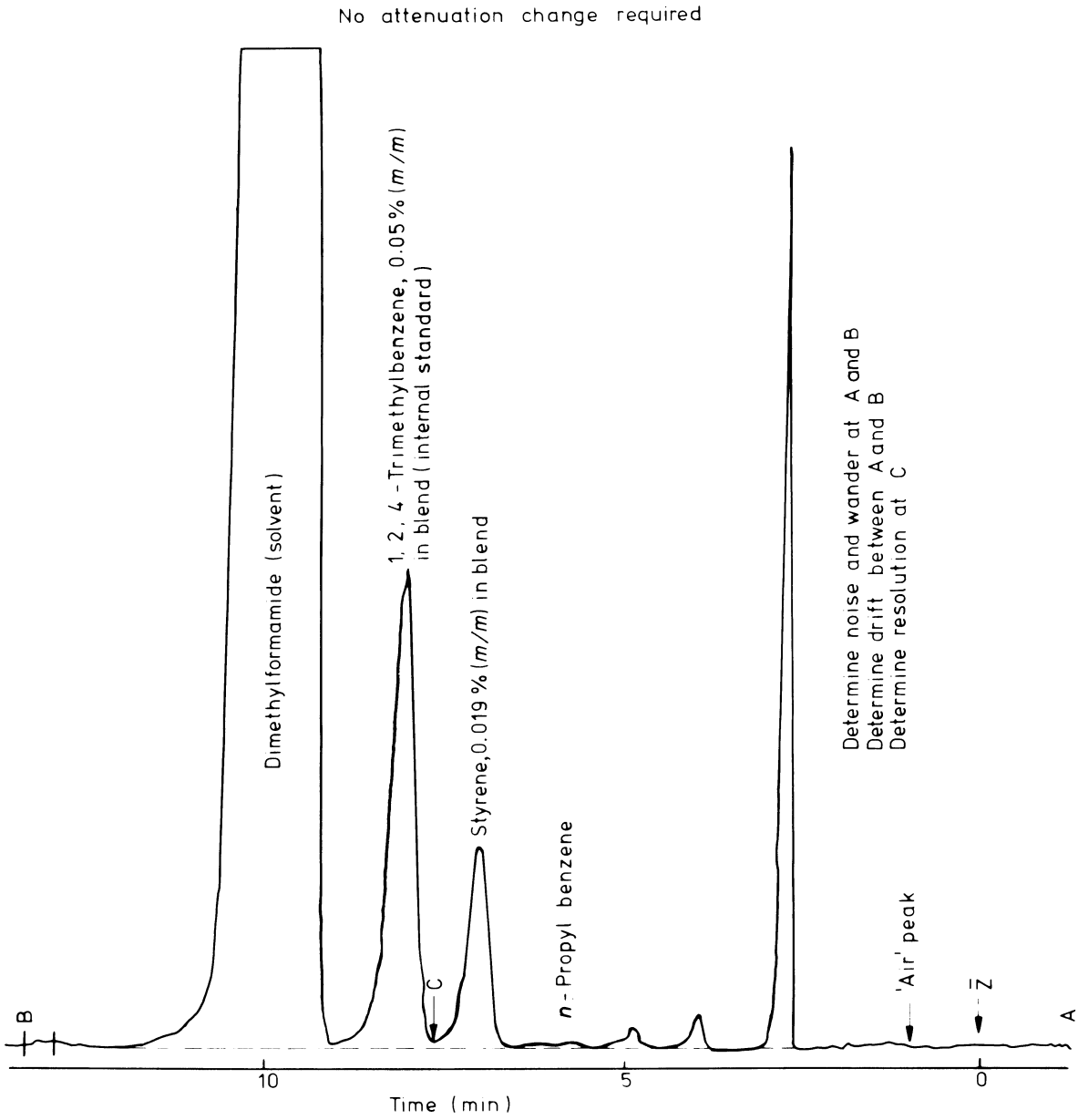

Figure 1 — Determination of styrene in polystyrene; typical chromatogram (not to scale)

9 Test report

The test report shall include the following particulars:

a) the reference of the method used;

b) the concentration of styrene monomer, as a percentage by mass, to the nearest 0.01 % for contents up to 1 % and to the nearest 0.1 % for contents greater than 1 %;

c) any unusual features noted during the determination;

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

10 Bibliographical references

10.1 Browning, E. Toxicity and metabolism of industrial solvents. Elsevier Publishing Co., 1965. p. 98.

10.2 Gray, C. H., ed. Laboratory handbook of toxic agents (second edition). The Royal Institute of Chemistry, 1966. p. 104.

10.3 Browning, E. Toxicity and metabolism of industrial solvents. Elsevier Publishing Co., 1965. p. 116.

10.4 Gray, C. H., ed. Laboratory handbook of toxic agents (second edition). The Royal Institute of Chemistry, 1966. p. 88.

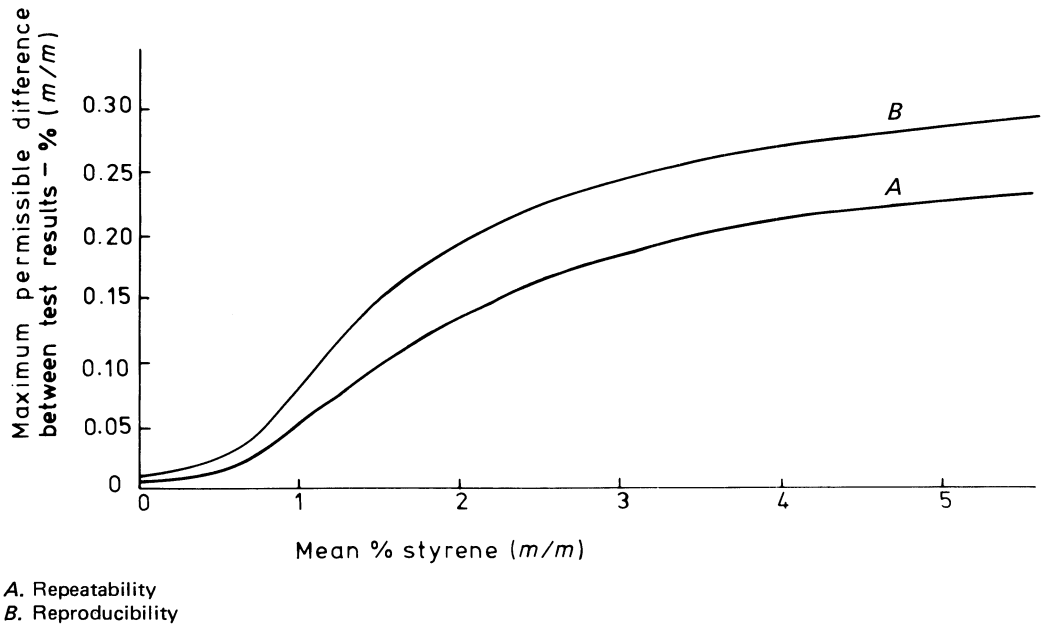

Figure 2 — Precision of test results

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Publications referred to

BS 0, *A standard for standards.*

BS 0-3, *Guide to drafting and presentation of British Standards.*

BS 1629, *Recommendation for references to published materials.*

BS 3282, *Glossary of terms relating to gas chromatography.*

BS 4148, *Specification for abbreviation of title words and titles of publications.*

BS 5309, *Methods for sampling chemical products.*

BS 5309-1, *Introduction and general principles.*

BS 5309-2, *Sampling of gases.*

BS 5309-3, *Sampling of liquids.*

BS 5309-4, *Sampling of solids.*

BS 5497, *Precision of test methods.*

BS 5497-1, *Guide for the determination of repeatability and reproducibility for a standard test method by inter-laboratory tests.*

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

ISO 2718, *Standard layout for a method of chemical analysis by gas chromatography.*

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