

Methods for

Analysis of glass —

Part 1: Glasses of the
soda-lime-magnesia-silica type

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Society of Glass Technology

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Foreword

This Part of BS 2649 has been prepared under the direction of the Laboratory Apparatus Standards Committee in close cooperation with the Society of Glass Technology, which has been concerned for many years with standardization of the methods of chemical analysis of glass and of the raw materials and refractories used in its manufacture. The Society published a monograph on the subject in 1929 and further investigations, started in 1944, resulted in the publication of recommended procedures for the chemical analysis of various types of glass. Procedures for soda-lime and borosilicate glasses were published in 1950, and for lead crystal glass in 1954.

The investigations carried out under the auspices of the Society included cooperative analyses in a number of laboratories and discussion at intervals of the results obtained. The following general conclusions were reached:

- a) preliminary analyses of standard samples of glass by the methods previously in use by the collaborating laboratories usually served to emphasize that parts of the procedures required closer specification;
- b) in order to obtain good agreement among all analysts taking part in the analysis of any single glass, it was necessary to agree on procedures specified in great detail;
- c) the variations in procedures necessary for the analysis of a wide variety of types of glass were so extensive that no general overall scheme of analysis was possible, and the procedures should therefore be provided separately for the analysis of each of the important main types of glass.

The Society subsequently agreed to a suggestion that the recommended procedures should, after revision in the light of further work, be published as a British Standard. As a result the following Parts of BS 2649 "*Methods for the analysis of glass*" were published.

- *Part 1:1955: Recommended procedure for the analysis of glasses of the soda-lime-magnesia-silica type;*
- *Part 2:1957: Recommended procedure for the analysis of soda-boric oxide-alumina-silica glasses of high silica and boric oxide content;*
- *Part 3:1958: Recommended procedure for the analysis of potassium-oxide-lead oxide-silica glasses;*
- *Part 4:1963: Recommended procedure for the analysis of fluoride-opal glasses.*

Parts 2 and 4 have since been withdrawn because they were obsolete.

This Part of BS 2649 was first published in 1955 and was technically equivalent to the procedure for the analysis of soda-lime-magnesia-silica glass published by the Society in 1954. The Society revised the procedure in 1971 and this standard is technically equivalent to the revision; due acknowledgement is made to the Society of Glass Technology. This revision of BS 2649-1 supersedes the 1955 edition, which is withdrawn.

Standard glass no. 1 was used in the development of the test procedures, but because of the diminution of the stock of this glass, another glass of similar composition (standard glass no. 5) was used to test the more rapid techniques described in this standard. In order to enable analysts to compare the methods described in this standard with others in use, samples of the standard glass no. 5 may be purchased from the Society (see the note). The analyses of the standard glass were examined statistically and the mean composition and limits for error are given for information in Appendix A.

NOTE Society of Glass Technology, "Thornton", 20 Hallam Gate Road, Sheffield S10 5BT.

The principal differences between BS 2649-1:1955 and this edition are as follows.

- 1) Potassium is determined as potassium tetrphenylborate instead of as potassium hexanitritocobaltate (III) (previously known as potassium cobaltinitrite).

2) Iron is determined colorimetrically using 1,10-phenanthroline hydrate instead of by the mercaptoacetic acid (previously known as thioglycolic acid) method.

3) Rapid methods suitable for control analysis are included.

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Summary of pages

This document comprises a front cover, an inside front cover, pages i to iv, pages 1 to 14, 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.

1 Scope

This Part of BS 2649 describes procedures for the chemical analysis of glass of the soda-lime-magnesia-silica type, which, for the purposes of this standard, is defined as glass containing silica (SiO_2) as its main constituent, with sodium oxide (Na_2O), calcium oxide (CaO) and/or magnesium oxide (MgO) as its other major constituents, and considerably lower proportions of aluminium oxide (Al_2O_3), iron (III) oxide (Fe_2O_3) and potassium oxide (K_2O). These procedures are recommended for any glass of this type in which fluorine (F), diboron trioxide (B_2O_3) and diarsenic trioxide (As_2O_3) are either absent or present to the extent of not more than 0.5 % (*m/m*) each.

This standard describes methods for determining silica (SiO_2), aluminium oxide (Al_2O_3), calcium oxide (CaO), magnesium oxide (MgO), sodium oxide (Na_2O), potassium oxide (K_2O), loss on heating at 550 °C, sulphur [as sulphur trioxide (SO_3)], iron (as Fe_2O_3) and titanium (IV) oxide (TiO_2).

This standard includes both “referee” methods of analysis, for which the results are required to have a high degree of accuracy and the time factor is a secondary consideration, and “rapid” methods of analysis for the determination of the major constituents, which are entirely suitable for routine purposes. The determination of the major constituents is common to both referee and rapid methods.

NOTE 1 The mean composition and limits of error of standard glass no. 5, for use in comparing the methods contained herein with other methods, are given in Appendix A.

NOTE 2 The titles of the publications referred to in this standard are listed on the inside back cover.

2 Principle

2.1 Referee methods

The silica content is determined gravimetrically by evaporation as the fluoride from the glass.

The aluminium content is determined gravimetrically as aluminium oxide on the residue of the silica determination after correction for residual silica, titanium (IV) oxide and iron (III) oxide.

The calcium content is determined gravimetrically as calcium oxide on the filtrate from the aluminium oxide determination.

The magnesium content is determined gravimetrically as magnesium pyrophosphate on the filtrate from the calcium determination, and is reported as magnesium oxide.

The sodium content is determined gravimetrically as sodium zinc uranyl acetate on the glass, and is reported as sodium oxide.

The potassium content is determined gravimetrically as potassium tetraphenylborate on the glass, and is reported as potassium oxide.

The sulphur content is determined gravimetrically as barium sulphate on the glass, and is reported as sulphur trioxide.

The iron content is measured colorimetrically using 1,10-phenanthroline hydrate solution at 510 nm on the glass, and is reported as iron (III) oxide.

The titanium content is measured absorptometrically at 410 nm on the glass, and is reported as titanium (IV) oxide.

2.2 Rapid methods

The major portion of the silica is determined on the glass gravimetrically by the evaporation of the fluoride, and the minor portion, i.e. that which remains in the solution, colorimetrically as the yellow molybdosilicate at 420 nm.

The aluminium content is determined on the glass by titration of the EDTA complex against standard zinc solution.

The calcium and magnesium contents are determined on the glass by titrating buffered solutions against EDTA solution.

The sodium and potassium contents are measured using a flame photometer.

3 Reagents

3.1 General

All the reagents shall be free from such amounts of impurities as might affect the accuracy of the determination in question. Analytical reagent grade will normally be suitable but provision is made for blank determinations if these are considered to be essential.

Water produced by distillation, ion exchange, or other methods and complying with grade 3 of BS 3978 shall be used throughout.

CAUTION. There is an inherent danger in the handling, even by trained personnel, of all chemicals. Some chemicals have to be handled with greater care than others. It is important that the laboratory safety precautions be observed at all times.

3.2 Solid and gaseous reagents

3.2.1 Hydrogen sulphide (H_2S).

3.2.2 Magnesium nitrate [$\text{Mg}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$].