

Methods for

Analysis of glass —

**Part 3: Glasses of the potassium
oxide-lead oxide-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 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 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 1958 and was technically equivalent to the procedure for the analysis of lead crystal glass published by the Society in 1955. The Society revised the procedure in 1972 and this standard is technically equivalent to the revision; due acknowledgement is made to the Society of Glass Technology. This revision of BS 2649-3 supersedes the 1958 edition, which is withdrawn.

Standard glass no. 8 was used in the development of the test procedures described in this standard. In order to enable analysts to compare the methods described in this standard with others in use, samples of standard glass no. 8 may be purchased from the Society (see the note). The analyses of the standard glass were examined statistically and the mean composition and limits of 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 2649-3:1958 and this edition are as follows.

- 1) Potassium is determined as potassium tetraphenylborate instead of as potassium chlorate.
- 2) Iron is determined colorimetrically using 1,10-phenanthroline hydrate instead of the mercaptoacetic acid (previously known as thioglycollic 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 16, 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 full lead crystal glass and lead crystal glass as specified in section 2 of BS 3828:1973 having as the main constituents silica (SiO_2), lead (II) oxide (PbO) and potassium oxide (K_2O).

This standard describes methods for determining silica (SiO_2), lead (II) oxide (PbO), aluminium oxide (Al_2O_3), calcium oxide (CaO), magnesium oxide (MgO), potassium oxide (K_2O), loss on heating at 550 °C, iron (as Fe_2O_3), arsenic (as As_2O_3), titanium (IV) oxide (TiO_2) and *diboron trioxide* (B_2O_3).

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. 8, 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 lead content is determined gravimetrically as lead (II) sulphate on the glass and reported as lead (II) oxide.

The aluminium content is determined gravimetrically as aluminium oxide on the filtrate from the lead determination after correction for 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 potassium content is determined gravimetrically as potassium tetraphenylborate on the glass, and is reported as potassium oxide.

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 arsenic content is determined on the glass by titration in carbon tetrachloride against potassium iodate and reported as *diarsenic trioxide*.

The titanium content is measured absorptiometrically at 410 nm, and is reported as titanium (IV) oxide.

The boron content is determined by titration against sodium hydroxide using a pH meter, and is reported as *diboron trioxide*.

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 lead and aluminium contents of the glass are determined by titration of the EDTA complex against the standard zinc solution before and after the addition of ammonium fluoride.

Sodium, potassium, calcium, magnesium and iron may be determined spectrometrically from a prepared solution of the glass.

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 Ammonium fluoride (NH_4F)

3.2.2 Hydrazinium (1 +) chloride ($\text{NH}_2\text{NH}_2 \cdot 2\text{HCl}$)

3.2.3 Hydrogen sulphide (H_2S)

3.2.4 Mannitol [$\text{CH}_2(\text{OH}) \cdot (\text{CHOH})_4\text{CH}_2\text{OH}$]

3.2.5 Oxalic acid [$(\text{COOH})_2 \cdot 2\text{H}_2\text{O}$]

3.2.6 Potassium bromide (KBr)

3.2.7 Sodium carbonate, anhydrous (Na_2CO_3)

3.2.8 Sodium hydroxide, pellets (NaOH).

3.3 Reagent solutions

3.3.1 General. The following designations are used for the solutions of acids and ammonia: the concentrated reagents are specified, where necessary, in grams per litre; the dilute reagents are specified by giving the proportions by volume in which the appropriate concentrated reagent is to be diluted with water. Thus, concentrated hydrochloric acid solution is described as hydrochloric acid solution, 425 g/L, and hydrochloric acid solution (1 + 4) refers to the solution obtained by diluting one volume of concentrated hydrochloric acid solution with four volumes of water. Water shall be the solvent or diluent unless otherwise specified.

3.3.2 Acetate buffer solution, pH = 5.3.

Dissolve 27.2 g of sodium acetate trihydrate ($\text{CH}_3\text{COONa} \cdot 3\text{H}_2\text{O}$) in water and dilute to 1 L. Dilute 2.9 mL of glacial acetic acid [CH_3COOH] to 250 mL and mix the two solutions.

3.3.3 Ammonia solution, dilute ammonia solution [NH_4OH] 634 g/L (1 + 1).

3.3.4 Ammonia solution, diluted (1 + 19).

3.3.5 Ammonium molybdate solution, 80 g/L.

Dissolve 80 g of ammonium molybdate tetrahydrate ($(\text{NH}_4)_6\text{Mo}_7\text{O}_{24} \cdot 4\text{H}_2\text{O}$) in water, filter if necessary, and dilute to 1 L. Store in a polyethylene bottle. Discard after 4 weeks, or earlier if any appreciable deposit is observed.

3.3.6 Ammonium nitrate solution, 20 g/L.

Dissolve 20 g of ammonium nitrate (NH_4NO_3) in 1 L of water, add a few drops of the methyl red indicator solution (3.4.1) and then add the ammonia solution (3.3.3) until the indicator just turns yellow.

3.3.7 Ammonium oxalate solution ($(\text{COONH}_4)_2 \cdot \text{H}_2\text{O}$), 1 g/L.

3.3.8 Diammonium hydrogen orthophosphate solution, 100 g/L. Dissolve 100 g of diammonium hydrogen orthophosphate [$(\text{NH}_4)_2\text{HPO}_4$] in 1 L of water and filter through a close filter paper (4.3) if any precipitate is present.

3.3.9 L-ascorbic acid solution, 0.1 mol/L.

Dissolve 0.890 g of L-ascorbic acid [$\text{Q.CO.C(OH):C(OH).CH.CH(OH).CH}_2\text{OH}$] in 100 mL of water. Discard after 1 month.

3.3.10 Carbon tetrachloride (CCl_4)

3.3.11 Citric acid solution

[$\text{C(OH)(COOH)(CH}_2\text{COOH)}_2 \cdot \text{H}_2\text{O}$], 100 g/L.

3.3.12 Hydrochloric acid solution (HCl), concentrated, 425 g/L.

3.3.13 Hydrochloric acid solution, diluted (1 + 1).

3.3.14 Hydrochloric acid solution, diluted (1 + 4).

3.3.15 Hydrochloric acid solution, diluted (1 + 9).

3.3.16 Hydrochloric acid solution, diluted (1 + 19).

3.3.17 Hydrochloric acid solution, diluted (1 + 99).

3.3.18 Hydrochloric acid solution, 0.1 mol/L.

3.3.19 Hydrochloric acid solution, (1 + 99) saturated with hydrogen sulphide gas (3.2.3).

3.3.20 Hydrofluoric acid solution, (HF), 452 g/L.

3.3.21 Hydrogen peroxide solution, (H_2O_2), 60 g/L.

3.3.22 Nitric acid solution (HNO_3), concentrated, 994 g/L.

3.3.23 Perchloric acid solution, (HClO_4), 924 g/L.

3.3.24 Perchloric acid solution, dilute (1 + 4).

3.3.25 Potassium tetraphenylborate saturated wash solution. Prepare immediately before use.

Add 35 mg of the potassium tetraphenylborate to 300 mL of water in a glass stoppered bottle. Shake the mixture at 5 min or 10 min intervals over a period of 60 min to 90 min then filter through a medium filter paper (4.3).

NOTE Potassium tetraphenylborate [$\text{KB}(\text{C}_6\text{H}_5)_4$] may be prepared by adding 10 mL of the sodium tetraphenylborate reagent solution (3.3.28) drop by drop to a continuously stirred solution containing 90 mL of the standard potassium chloride solution (3.5.6) and three drops of 0.1 mol/L acetic acid solution [CH_3COOH]. After 30 min filter off the precipitate on a sintered glass filter-crucible of porosity grade designation P16 complying with BS 1752, and wash 10 times with 2 mL to 3 mL portions of cold water. Dry the precipitate for 1 h at $110 \pm 10^\circ\text{C}$, and store in a stoppered bottle.

3.3.26 Sodium hydroxide solution [NaOH], carbonate-free, 0.5 mol/L.

3.3.27 Sodium hydroxide solution, carbonate-free, 0.05 mol/L.

3.3.28 Sodium tetraphenylborate reagent solution. Prepare immediately before use. Dissolve 1.7 g of sodium tetraphenylborate [$\text{NaB}(\text{C}_6\text{H}_5)_4$] in 50 mL of water in a glass stoppered bottle. Add about 0.5 g of moist aluminium hydroxide gel ($\text{Al}(\text{OH})_3 + \text{H}_2\text{O}$), and break up any solid lumps before shaking to disperse the gel throughout the solution. Shake the mixture at 5 min intervals over a period of 20 min and then filter the solution using a “medium” filter paper (4.3). If the first portion of the filtrate is cloudy, return it to the filter paper and collect the clear filtrate in a clean vessel.

3.3.29 Sulphuric acid solution, diluted (1 + 1).

Add 500 mL of sulphuric acid solution (H_2SO_4), 1 800 g/L, to 500 mL of water slowly, carefully and with stirring; much heat is evolved.

3.3.30 Sulphuric acid solution, diluted (2 + 98).

3.3.31 Sulphuric acid solution, diluted (1 + 9).

3.3.32 Silver nitrate solution, (AgNO_3), 25 g/L. Prepare immediately before use.