ANNEX II

METHODS FOR DETERMINING THE CHEMICAL AND PHYSICAL PROPERTIES OF CATEGORIES OF CRYSTAL GLASS

1. CHEMICAL ANALYSES

1.1. BaO and PbO

1.1.1. Determination of the combination BaO + PbO

Weigh, to within 0·0001 grammes, approximately 0·5 grammes of powdered glass and place in a platinum dish. Moisten with water and add 10 millilitres of a 15% solution of sulphuric acid and 10 millilitres hydrofluoric acid. Heat in sand bath until white fumes are given off. Allow to cool and treat again with 10 millilitres hydrofluoric acid. Heat until reappearance of white fumes. Allow to cool and rinse the sides of the dish with water. Heat until reappearance of white fumes. Allow to cool, carefully add 10 millilitres of water, then transfer to a 400 millilitres beaker. Rinse the dish several times with a 10% sulphuric acid solution and dilute to 100 millilitres with same solution. Boil for 2-3 minutes. Leave to stand overnight.

Pass through a filtering crucible of 4 porosity, wash first of all with a 10% solution of sulphuric acid, then two or three times with ethyl alcohol. Dry for one hour in an oven at 150 °C. Weigh BaSO4 + PbSO4.

1.1.2. Determination of BaO

Weigh, to within 0·0001 grammes, about 0·5 grammes of powdered glass and place in a platinum dish. Moisten with water and add 10 millilitres of hydrofluoric acid and 5 millilitres perchloric acid. Heat in sand bath until white fumes are given off.

Allow to cool and add a further 10 millilitres hydrofluoric acid. Heat until reappearance of white fumes. Allow to cool and rinse the sides of the dish with distilled water. Heat again and evaporate until almost dry. Start again with 50 millilitres of a 10% solution of hydrochloric acid and heat gently to aid dissolution. Transfer to a 400 millilitres beaker and dilute to 200 millilitres with water. Bring to boil and pass a current of hydrogen sulphide through the hot solution. When the precipitate of lead sulphide drops to the bottom of the beaker, turn off the hydrogen sulphide. Pass through a fine filter paper and wash with cold water saturated with hydrogen sulphide.

Boil the filtrates and then, if necessary, reduce them by evaporation to 300 millilitres. Add to boiling mixture 10 millilitres of a 10% solution of sulphuric acid. Remove from heat and leave to stand for at least four hours.

Pass through a fine filter paper, wash with cold water. Calcine the precipitate to 1050 °C, and weigh the BaSO4.

1.2. Determination of ZnO

Evaporate the filtrates from the separation of BaSO4 so as to reduce their volume to 200 millilitres. Neutralise with ammonia in the presence of methyl red and add 20 millilitres of N/10 sulphuric acid. Adjust the pH to 2 (pH meter) by adding N/10 sulphuric acid or N/10 caustic soda whichever the case, and precipitate the zinc sulphide in the cold by passing a current of hydrogen sulphide. Let the precipitate settle for four hours, then collect on a fine filter paper. Wash with cold water saturated with hydrogen sulphide. Dissolve the precipitate on the filter by pouring through it 25 millilitres of a hot 10% solution of hydrochloric acid. Wash the filter with boiling water until a volume of about 150 millilitres is obtained. Neutralise with ammonia in the presence of litmus paper, then add 1-2 grammes solid urotropine to buffer the solution

to about pH 5. Add a few drops of a 0·5% freshly prepared aqueous solution of xylenol orange and titrate with an N/10 solution of Complexon III until the pink changes to citron yellow.

1.3. Determination of K2O

by precipitation and weighing of potassium tetraphenylborate.

Procedure:	2 grammes of glass are attacked, after crushing and sieving, by 2 millilitres concentrated HNO3 **[**[X1]15 millilitres NClO4**]** 25 millilitres HF

Editorial Information

X1 Substituted by Council Directive No 69/493/EEC of 15 December 1969 on the approximation of the laws of the Member States relating to crystal glass (Official Journal of the European Communities, No L 326, p. 36),.

in a platinum dish on a water-bath then in a sand bath. After dense fumes of perchloric acid have been given off (continue until dry), dissolve with 20 millilitres of hot water and 2-3 millilitres concentrated HCl.

Transfer to a 200 millilitres graduated flask and adjust to volume with distilled water.

Reagents: 6% solution of sodium tetraphenylborate: dissolve 1·5 grammes of the reagent in 250 millilitres distilled water. Remove the light cloudiness which remains by adding 1 gramme of hydrated aluminia. Shake for five minutes and filter, taking care to re-filter the first 20 millilitres obtained.

Washing solution for the precipitate: prepare a little of the potassium salt by precipitation in a solution of about 0·1 grammes KCl to 50 millilitres N/10 HCl into which the solution of tetraphenylborate is poured while stirring, until precipitation ceases. Filter through a sinter. Wash with distilled water. Dry in a desiccator at room temperature. Then pour 20-30 milligrammes of that salt into 250 millilitres of distilled water. Stir from time to time. After thirty minutes, add 0·5-1 gramme of hydrated alumina. Stir for a few minutes. Filter.

Method of operation: Take an aliquot of the acid digest corresponding to about 10 milligrammes of K2O. Dilute to about 100 millilitres. Slowly add the reagent solution, about 10 millilitres per assumed 5 milligrammes of K2O, while gently stirring. Allow to stand for a maximum of fifteen minutes then filter through a tared sintered crucible of porosity 3 or 4. Wash with washing solution. Dry for thirty minutes at 120 °C. Conversion factor 0·13143 for K2O.

1.4. Tolerances

± 0·1 in absolute value for each determination. If the analysis gives a lower value, within the tolerances, than the limits fixed (30, 24 or 10%), the average of at least three analyses must be taken. If that average is greater than or equal to 29·95, 23·95 or 9·95 respectively, the glass must be accepted in the category corresponding to 30, 24 and 10% respectively.