29.12.69

#### COUNCIL DIRECTIVE

#### of 15 December 1969

on the approximation of the laws of the Member States relating to crystal glass

(69/493/EEC)

# THE COUNCIL OF THE EUROPEAN COMMUNITIES,

Having regard to the Treaty establishing the European Economic Community, and in particular Article 100 thereof;

Having regard to the proposal from the Commission; Having regard to the Opinion of the European Parliament<sup>1</sup>;

Having regard to the Opinion of the Economic and Social Committee;

Whereas, with regard to the use of a special description for crystal glass products and the consequent obligation concerning the composition of such products, there are differences between the rules of certain Member States; whereas those differences hinder trade in such products and can lead to distortions in competition within the Community;

Whereas those obstacles to the establishment and proper functioning of the common market can be eliminated by adoption of the same requirements by all the Member States;

Whereas, with regard to the descriptions laid down for the various categories of crystal glass and to the characteristics of those categories, the purpose of the Community provisions to be adopted is to protect both the buyer against fraud and the manufacturer who complies with those provisions;

Whereas implementation of a system of Community rules requires the establishment of standard methods for determining the chemical and physical properties of crystal glass products bearing descriptions laid down in this Directive;

# HAS ADOPTED THIS DIRECTIVE:

# Article 1

This Directive shall apply to the products falling within heading No 70·13 of the Common Customs Tariff.

#### Article 2

Member States shall take all necessary steps to ensure that the composition, characteristics of manufacture and labelling of the products referred to in Article 1, and all forms of publicity for such products, conform to the definitions and rules laid down in this Directive and in the Annexes thereto.

#### Article 3

Member States shall take all necessary steps to prevent the descriptions in column (b) of Annex I from being used commercially for products which do not have the corresponding characteristics specified in columns (d) to (g) of Annex I.

# Article 4

- 1. If a product covered by this Directive bears one of the descriptions listed in column (b) of Annex I, it may also bear the corresponding identifying symbol shown and described in columns (h) and (i) of that Annex.
- 2. Where a trade mark, the name of an undertaking or any other inscription contains, as a main part, as an adjective or as a root, a description appearing in columns (b) and (c) of Annex I or a description liable to be confused therewith, Member States shall take all necessary steps to ensure that that trade mark, name or inscription is immediately preceded by the following, in very prominent lettering:
- (a) the description of the product, where that product has characteristics specified in columns (d) to (g) of Annex I;
- (b) a statement of the exact nature of the product, where that product does not have characteristics specified in columns (d) to (g) of annex I.

<sup>&</sup>lt;sup>1</sup> OJ No C 108, 19.10.1968, p. 35.

# Article 5

The description and identifying symbols given in Annex I may appear on one and the same label.

# Article 6

The methods laid down in Annex II, and only those methods, shall be used to verify that products bearing descriptions and identifying symbols have the characteristics corresponding thereto as specified in columns (d) to (g) of Annex I.

# Article 7

Products intended for export from the Community shall not be subject to the provisions of this Directive.

# Article 8

Member States shall put into force the measures needed in order to comply with this Directive within

eighteen months of its notification and shall forthwith inform the Commission thereof. As soon as this Directive has been notified, Member States shall also ensure that they inform the Commission in time for it to submit its observations, of any subsequent drafts of main laws, regulations or administrative provisions which they propose to adopt in the field covered by this Directive.

#### Article 9

This Directive is addressed to the Member States.

Done at Brussels, 15 December 1969.

For the Council
The President
H. J. DE KOSTER

# ANNEX I List of crystal glass categories

No	Description of category		Characteristics				Labelling	
		Explanatory notes	Metal oxides (%)	Density	Refractive index	Surface hardness	Shape of symbol	Remarks
′—a—	<u></u> b	-c-	—d—	-c-	f	g	—h—	—i—
1	CRISTAL SUPERIEUR 30% CRISTALLO SUPERIORE 30% HOCHBLEIKRISTALL 30% VOLLOODKRISTAL 30%	Description may be freely used, whatever the country of origin or the country of destination	PbO ≥ 30%	≥ 3.00	×			Round label. Colour: gold $\phi \geq 1$ cm
2	CRISTAL AU PLOMP 24% CRISTALLO AL PIOMBO 24% BLEIKRISTALL 24% LOODKRISTAL 24%	The percentage figure re- fers to the lead oxide con- tent	PbO ≥ 24%	≥ 2.90	x			
3.	CRISTALLIN VETRO SONORO SUPERIORE KRISTALLGLAS KRISTALLIJNGLAS¹ SONOORGLAS²	Only the description in the language or languages of the country in which the goods are marketed may be used  Exception: On the German market pressed glass containing 18% PbO and having a density of at least 2.70 may be sold under the description 'PRESSBLEI-KRISTALL' or 'BLEI-KRISTALL GEPRESST' (in capital letters)	ZnO BaO PbO K₂O singly or together ≥ 10%	≥ 2.45	nD . ≥ 1·520			Square label. Colour: silver Side: ≥ 1 cm
4	VERRE SONORE VETRO SONORO KRISTALLGLAS SONOORGLAS		BaO PbO K₂O single or together ≥ 10%	≥ 2.40		Vickers 550 ± 20		Label in the shape of an equilateral triangle. Colour: silver Side: ≥ 1 cm

x nD  $\geq$  1.545 as criterion for an additional non-destructive determination of the products (at the time of import).

<sup>1</sup> In Belgium.

<sup>&</sup>lt;sup>2</sup> In the Netherlands.

#### 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 BaSO<sub>4</sub> + PbSO<sub>4</sub>.

#### 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 BaSO<sub>4</sub> 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 K<sub>2</sub>O

by precipitation and weighing of potassium tetraphenylborate.

Procedure: 2 grammes of glass are attacked, after crushing and sieving, by

- 2 millilitres concentrated HNO<sub>3</sub>
- 15 millilitres HCO<sub>4</sub>
- 25 millilitres HF

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

 $\pm$  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.

#### 2. PHYSICAL DETERMINATIONS

#### 2.1. Density

Method by hydrostatic balance to within  $\pm$  0.01. A sample of at least 20 grammes is weighed in air and weighed immersed in distilled water at 20 °C.

# 2.2. Refractive index

The index is measured on the refractometer to within  $\pm 0.001$ .

# 2.3. Microhardness

Vickers hardness is to be measured according to the standard ASTM E 92-65 (Revision 1965) but using a load of 50 grammes and taking the average of 15 determinations.