Glass
SERVICE

Technical Data and Technical Terms for Glass Tube for Pharmaceutical Use

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SOLUTIONS FOR THE GLASS INDUSTRY

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Technical Data and Technical Terms for Glass Tube for Pharmaceutical Use

Physical and chemical properties of glass

1 Expansion coefficient

2 Density

3 Strain point log η = 14.5 (°C)

4 Annealing Point   log η = 13.0 (°C)

5 Softening Point   log η = 7.6   (°C)

6 Working Point   log η = 4.0   (°C)

1 Coefficient of expansion

The coefficient of expansion "α, is the increasing of the length suffered by the unit-length of the sample coming from the increasing of the temperature of 1°C in the range between 0°C and 300°C. It is expressed in °C -1 and measured by the dilatometer.
The coefficient of expansion could change with the annealing and therefore the values are referred to the annealed glass.

2 Density

It is the weight per unit volume. Unit of measurement is gr/cm3.
The density could change with the annealing and therefore the values are referred to the annealed glass.

3 Strain Point

It is the temperature at which the internal stresses are reduced to low values in one hour
It is determined by the extrapolation of the annealing-data through the method of the lengthening of the fiber: method ASTM C336

4 Annealing Point

It is that temperature when the internal stresses, caused by a quick cooling because of working, can be removed in a few minutes.
It is measured according to the standard norm ASTM C336

5 Softening Point

It is that temperature when a calibrated glass-fiber lengths increase under its weight with a pre-established rate. It is measured according to the norm ASTM C338.
In this range of temperature glass deforms considerably under its own weight.

6 Working Point

It is that the temperature when the glass gets a viscosity of 104 poises. At this temperature viscosity glass is enough soft to be worked.

7 Light Transmittance

It is that the quantity of light radiation transmitted (I) from a body of glass when it is submitted to an incidence-light-radiation (I0)
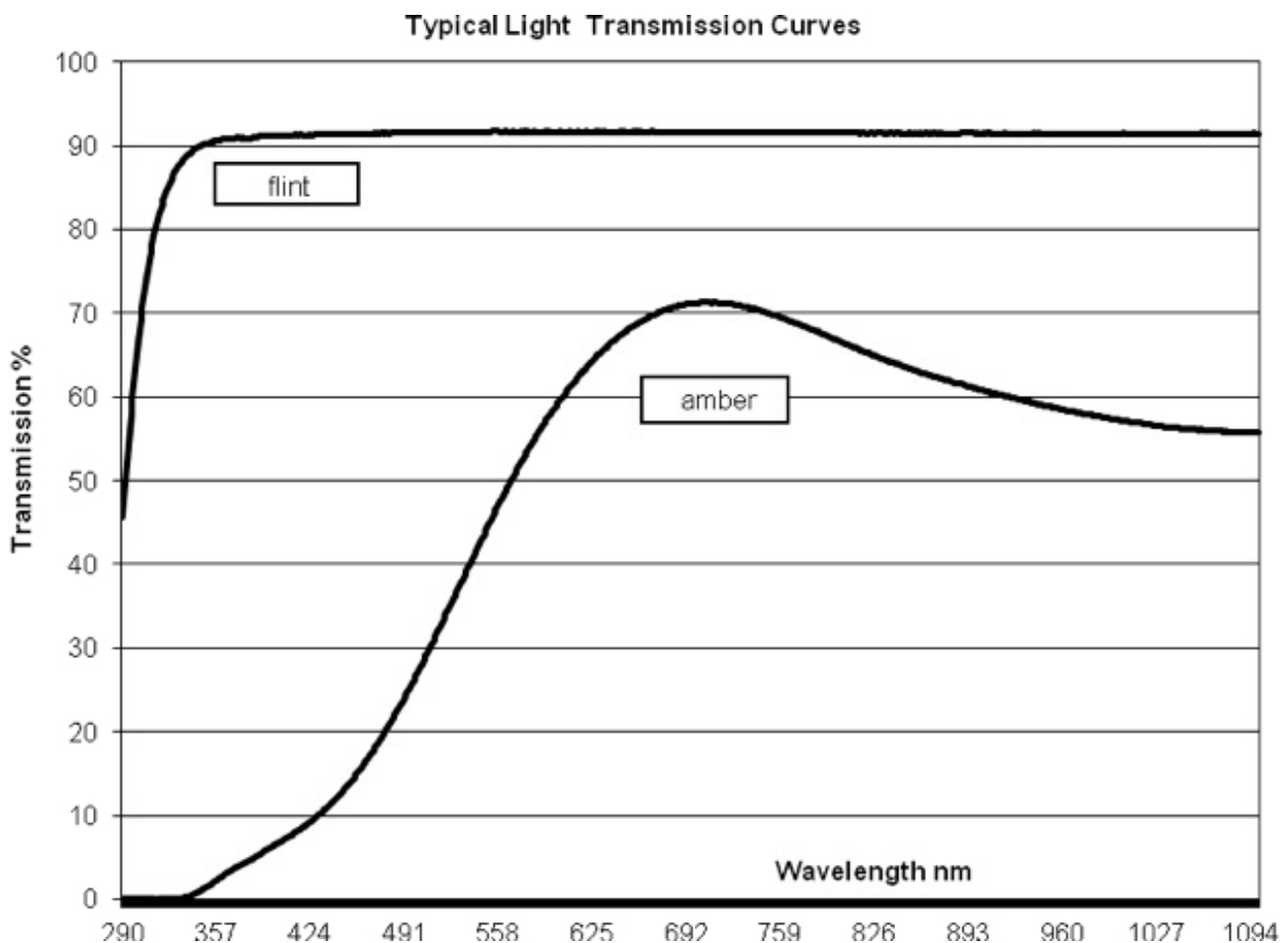
$$T(\%) = \frac{I}{I_0} \times 100\%$$

The transmission of light is highly conditioned by the annealing process.
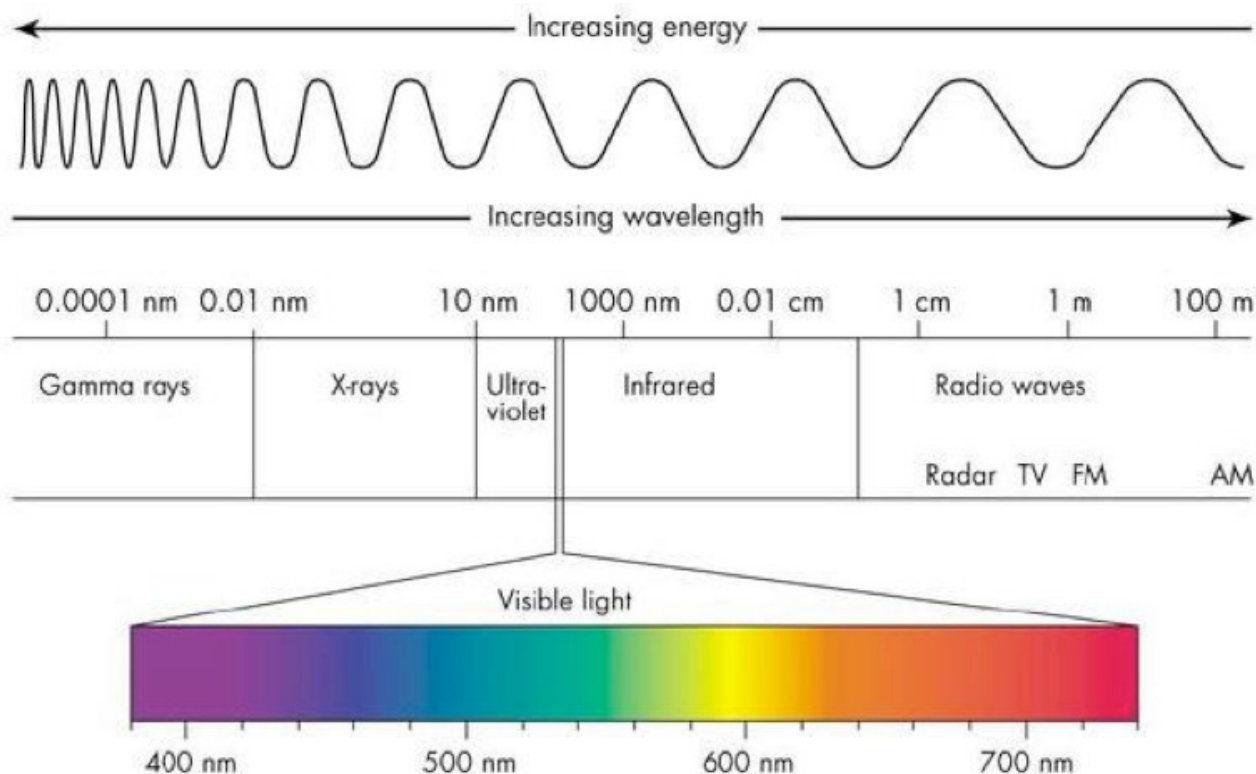
8 Chemical Durability

It is that the resistance against the chemical attack of the environment. It is expressed accordingly to the quantity of the released alkaline elements.

Light Transmission Curve

Typical Light Transmission Curves

Spectrum of light

Chemical Durability - test on powdered glass

The norms in force on the pharmacopeia are briefly listed below:

| Norm	USP 29	E.P. 5àed	JP XIV	Chinese	ISO 719	ISO 720	DIN 12116	ISO 695
Sample	10 g	10 g	5 g	GB 12416.2-90 10 g	2 g	10 g	Size	Size
Grain size	>300m<425 m	>300m<425 m	>300m<850 m	>300m<425 m	>300m<500 m	>300m<425 m	400 cm²	10-15 cm²
Conditions	121°C/30 min	121°C/30 min	100°/120 min	121°C/30 min	98°C/60 min	121°C/30 min	HCl boiling/6h	Alk solution 102,5°C/180 min
Attack	50 ml	50 ml	50 ml	50 ml	50 ml	50 ml	Test consists in defining the loss of weight in in mg/dm²	Test consists in defining the loss of weight in in mg/dm²
Indicator	Methyl red	Methyl red	Bromocresol methyl red	Methyl red	Methyl red	Methyl red		
Titatrion	HCl 0,02 M	HCl 0,02 M	H_2SO 0,01 M	HCl 0,02 M	HCl 0,01M	HCl 0,02 M		
Ranges	1 ml/10g	1 ml/10g	0,3 ml	class 1 0,1ml/1	HGB 1 0,1ml/1g	HGA 1 0,1ml/1g	S1 0 - 0,7	A2 75 - 175

Variations of alkali release after the transformation depending on the transforming temperature

During the conversion of glass tube to vials, performed by forming-machine and by the annealing of formed items, the neutral characteristics of the glass are modified as a result of outcropping of the alkaline ions.

The temperatures of the flames of the forming-machines and the further annealing influence the chemical neutrality of the shaped-vial.

The overheating of the tube during the forming let the alkaline ions come to the glass-surface deteriorating the neutrality of the glass. The annealing fixes the alkaline ions on the siliceous grating again. The equilibrium between T of transformation and the further annealing determines the final chemical neutrality of the tube.

In the following diagram you find an example of the loss of neutrality depending on the Temperature- transformation.
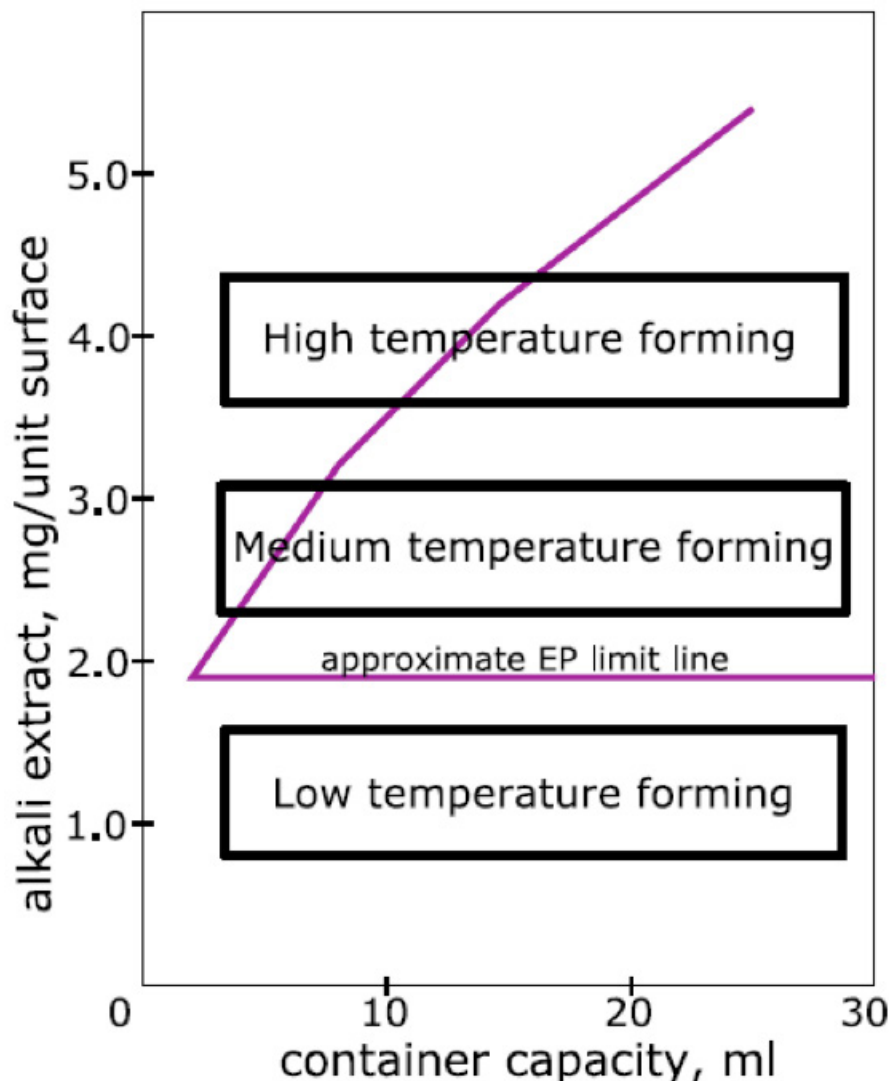
Annealing Cycle of the containers

Below is shown an example of a typical annealing cycle suitable for the production of articles produced by conversion of borosilicate glass tube.
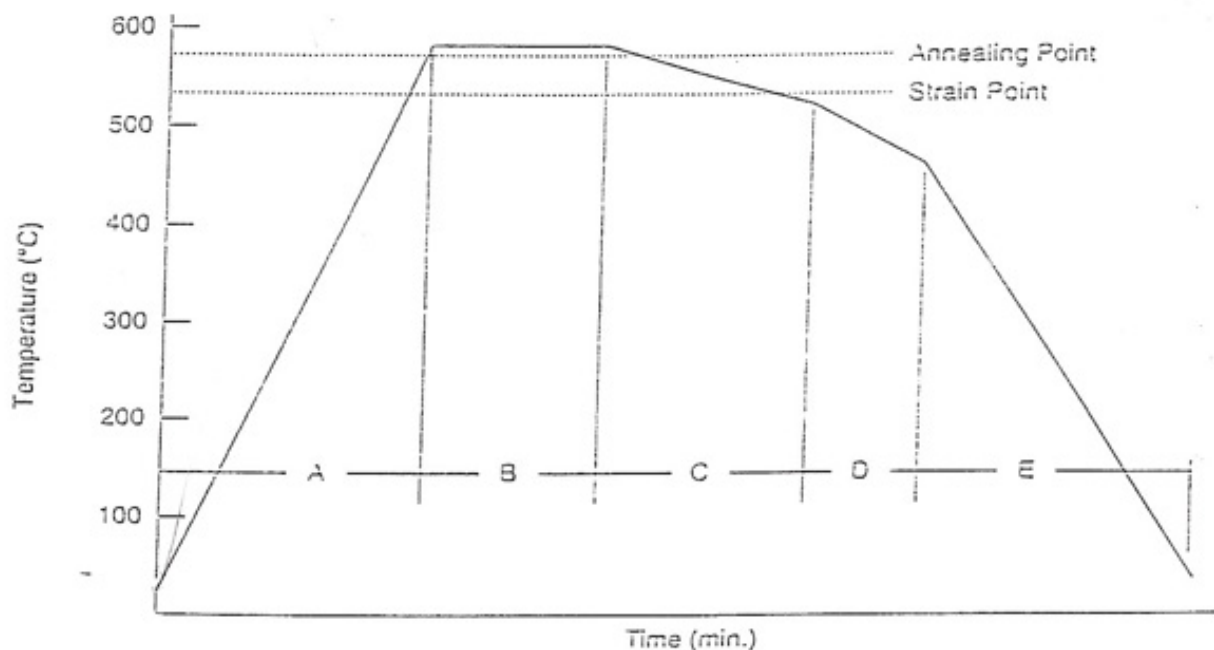
Example of an annealing cycle

phase	Description			
A)	Warm up to 5°C higher than the annealing point with an increase of temperature of 200 °C/min			
B)	Keep the temperature on accordingly to the indicative following schema (the below temperature are relative at one glass type and can be change with glass batch composition variation)			
	Amber (°C)	Flint (°C)	Thickness (mm)	Time (min)
	535	550	0,50	1,0
	535	550	0,75	1,5
	535	550	1,5	3,0
C)	Begin cooling after 5°C below the Strain Point with a rate of 20 °C/min			
D)	Cool up to 55°C below the Strain Point with a rate of 40 °C/min			
E)	Cool up to ambient temperature with a rate of 200 °C/min.			

Tab. 2

Standard Range of the sizes of the tubes and tolerance range

As an example here following we have listed some sizes of the diameters which can be manufactured by DANNER technology. For further info refer to the producers catalogues.

External Diameter (mm)	Compatible Thickness (mm)	Tolerance of diameter / thickness (+/- mm)	
4.0 – 5.9	0.30 – 0.55	0.13	0.02
	0.60 – 0.75	0.15	0.03
	0.8 – 1.00	0.15	0.04
6.0 – 8.9	0.35 – 0.55	0.13	0.02
	0.60 – 0.95	0.15	0.03
	1.00 – 1.15	0.15	0.04
	1.20 – 1.40	0.20	0.04
9.0 – 14.9	0.40 – 0.55	0.13	0.02
	0.60 – 0.95	0.15	0.03
	1.00 – 1.15	0.20	0.04
	1.20 – 1.35	0.20	0.04
	1.40 – 1.60	0.25	0.05
15.0 – 17.9	0.45 – 0.60	0.15	0.03
	0.65 – 1.05	0.20	0.04
	1.10 – 1.35	0.20	0.04
	1.40 – 1.60	0.30	0.05
18.0 – 19.9	0.55 – 1.05	0.20	0.04
	1.10 – 1.35	0.25	0.05
	1.40 – 1.60	0.30	0.05
20.0 – 24.9	0.65 – 1.05	0.20	0.04
	1.10 – 1.35	0.25	0.05
	1.40 – 1.60	0.30	0.06
25.0 – 29.9	0.75 – 1.05	0.25	0.04
	1.10 – 1.35	0.25	0.05
	1.40 – 1.60	0.30	0.06
30.0 – 34.9	1.00 – 1.35	0.30	0.05
	1.40 – 1.60	0.40	0.08
35.0 – 39.9	1.20 – 1.35	0.75	0.10
	1.40 – 1.60	0.75	0.10
40.0 – 50.0	1.40 – 1.60	1.00	0.10
	1.65 – 1.95	1.00	0.12

Tab. 3

General criteria for the quality and the dimensions of the glass

The quality of glass tubes is established for the use of containers in pharmaceutical field accordingly to the standard technologies. The tube will be considered defective if the criteria, defined by the technical specifications, will not be respected.

In case of particular needs, different from the ones established by the technical specifications, these should be subject for special agreements customer/producer.

Definitions

OD = Outer Diameter;

ID = Inner Diameter;

WT = wall thickness of the tube

- OD and ID have to be in the range of tolerances in each point of the tube
- Roundness and conical do not cause conditions of out-tolerance for OD and ID
- Defects of glass, as nodes and stones (in one acceptable range) can exceed from the tolerances OD without creating defects.

The comparison of the results of the test can be made **only if identical check-procedures are adopted.**

The visible characteristics must be inspected only by unaided eye without zooming optical device and under normal light-conditions.
An inspection area, with normal illumination, should be as follows:

- A desk with two 40W white-light fluorescent lamps, installed about 1 m above the inspection-table with a black non-reflecting background.

AQL. (Acceptable Quality Level). When we refer on dimensional defects the values are summarized to give just one accumulated level of defect. The resulting - AQL – aggregate must be specified for each defect.
The AQL is defined as the level of quality, which, over a continuous series of batch selected for the purpose of inspection by sampling, can be considered as the limit for a satisfactory process, on average.

Sampling and measurements of the quality parameters and defects of the tube

Methods of test

Visible defects are evaluated to the unaided eye therefore without any type of optical zooming device. Measurement and classification of the defects can be made subsequently with the help of optical zooming device and appropriate measure-systems listed below.

The visible characteristics will be inspected without the help of optical zooming device under a normal light source. For the characteristics not visible to the unaided eye, please refer to the instructions below.

An area with normal lighting can be described as below:

- A desk with two lamps of 40 Watt, (neon) which have to be installed at 1 meter over the inspection-desk with a contrast on an opaque-black bottom.

Do not consider defective those defects, which are not easy visible to the unaided eye and normal light conditions. For defects measure refer to the instructions below.

Plan for sampling for routine check

A simple check-plan for routine-checks of production follows timing and quantities with reduced samplings quantity, for example:

1) Every four hours take from each production line 5 (20<OD<50) to 10 tubes (4<OD<20) sample number variation on the size base.
2) The necessary parameters (diameter, thickness, etc. as listed below) are measured on each tube following the methodologies described further. Moreover, visible defects such as lines, stones, cords, knots are to be counted on each tube and the result shall be compared to a standard quantity of glass, for example 5 Kg. This is required to get an overview of the defects without the influence of the sizes of the tube and of the phenomena occurring therefore.
For the defects defined as "lines" it is useful to know their thickness, whether they are closed or open, and their subdivision based on their length according to the following schema:

<2 cm
>2 <5 cm
>5 <15 cm
>15 <50 cm
> 50 cm
continuous line

On the same Inspection form you will write also the data-collection concerning breakings, scratches, dirty, cracks and glazing.

Inspection form in enclosure no.1 (quality recording form)

Statistic-test method

The statistic test is used for the exact characterization of the production-quality or in case of production lot customer claim.

The test is affected on one significant sample quantity, which can differ accordingly to the AQL-requested-level and to lot size quantity to be analyzed. The value of AQL establishes the level of the requested quality. For each quality-level the table 4 shows the quantity of samples to analyze and the quantity of acceptable defects.

Tab.4

Lot size	AQL (n/c; quantity of samples/acceptance figure)									
	0,025	0,10	0,25	0,40	0,65	1,0	1,5	2,5	4,0	6,5
501-1200	500/0	125/0	50/0	125/1	80/1	80/2	80/3	80/5	80/7	80/10
1201-3200	500/0	125/0	200/1	125/1	125/2	125/3	125/5	125/7	125/10	125/14
3201-10000	500/0	125/0	200/1	200/2	200/2	200/5	200/7	200/10	200/14	200/21
10001-35000	500/0	500/1	315/2	315/3	315/5	315/7	315/10	315/14	315/21	200/21
35001-150000	500/0	500/1	500/3	500/5	500/7	500/10	500/14	500/21	315/21	200/21
150001-500000	500/0	800/2	800/5	800/7	800/10	800/21	800/21	500/21	315/21	200/21
> 500000	2000/1	1250/3	1250/7	1250/7	1250/14	800/21	800/21	500/21	315/21	200/21

Random Sample schedule for pharmaceutical tubing per ANSI/ASQC z1-4-1993
Simple random sample, general inspection level II, single sampling plans for normal inspection Lot = 1 pallet
n= Random sample size (number of testing unit evaluated)
c= acceptance figure (lot is accepted if number of defects in random samples is less than or equal to the acceptance figure)
BTU= Basic Testing Unit (BTU or audit sample size) is a 5 kg random sample (ex. 500 Tube of 10 gr each) from a minimum of 5 individual pack AQL= Acceptance Quality Level

Example

Tube OD=10,75 mm ; WT = 0,5 mm ; length 1500 mm ; tube weight 70 gr

Weight of 1 box is 20 kg; 1 pallet contains 44 boxes; number of tubes for 12540, if we take the lot size of 12540 tubes, the sample for AQL 0,25 becomes according to tab.4 - 315 tubes and among these 2 can be defective.

The errors of dimensions have to be cumulated.
Example: if a tube of 1500 mm has two points out of diameter these defects have to be considered as 2 even if they are on the same tube.

Dimensional defects

1) External diameter OD

It is that the maximum distance, in straight line, between two points of the external surface of the tube, measured on a plane perpendicular to the axis of the tube.

Each tube will be defined "defective" and therefore thrown way if on the whole tube length a continuous portion over 100 mm of it a discrepancy more than the diameter-tolerance has been detected.

AQL = 0.25 (cumulative)
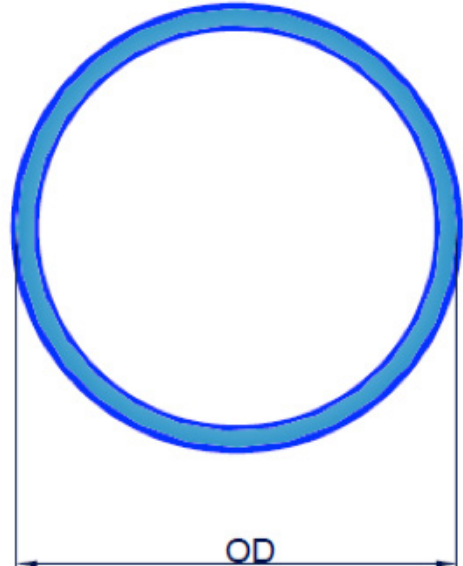
All the points, measured on the tube, have to be in the range of the foreseen tolerances. Roundness and Conical shape do not produce any effect of "out-of-diameter".
Superficial defects of the glass such as stones and nodules, within acceptable ranges, can exceed the tolerance of OD and do not produce the "out-of-diameter" defect.

Out of the production line, the OD measurement can be effected by a mechanical comparator having an accuracy of 0.01mm and installed on a mechanical support where it can slide perpendicularly to the axis of the tube and equipped with a "V"-shaped-base which will be sufficiently long to stabilize the tube to be measured.

2) Internal diameter ID

It is the maximum distance between two points on the internal surface of the tube on a plane perpendicular to the axis of the tube.
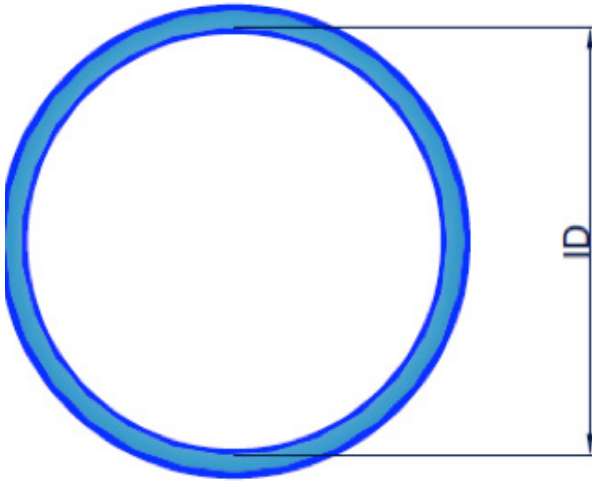
The measurement of the internal diameter can be effected out of the production line by a spring-caliper with double head; it has an accuracy of +/- 0.01mm.

Each tube will be defined "defective" and therefore thrown away if on the whole tube length has been detected a continuous portion over 100 mm of it a discrepancy more than the diameter-tolerance

(Other method to measure the ID is by calibrated stoppers "pass through/don't pass trough").

Surface defects of glass such as stones and nods within acceptable limits can exceed the tolerance of ID and do not produce "out-of-diameter" defect.

AQL = 0.25 (cumulative)

Measure desk

A measure desk as indicate in the following picture can use for several geometric dimensional control. Thickness, bow, out of the lineup.
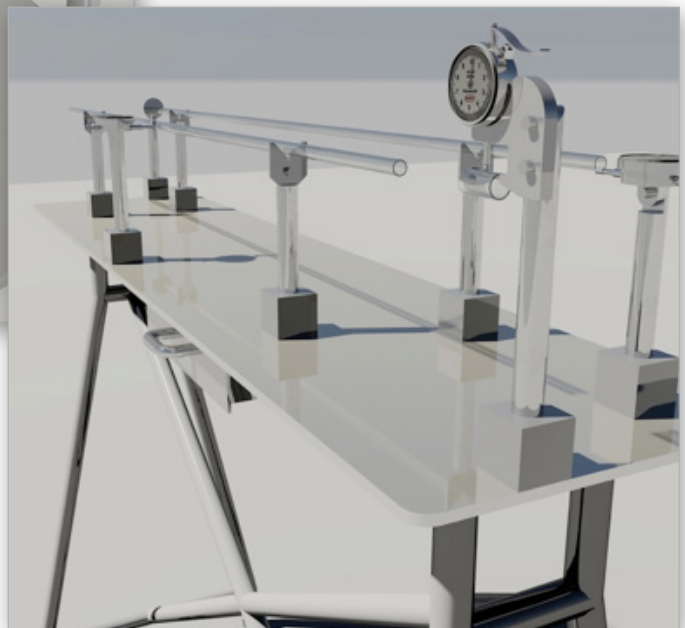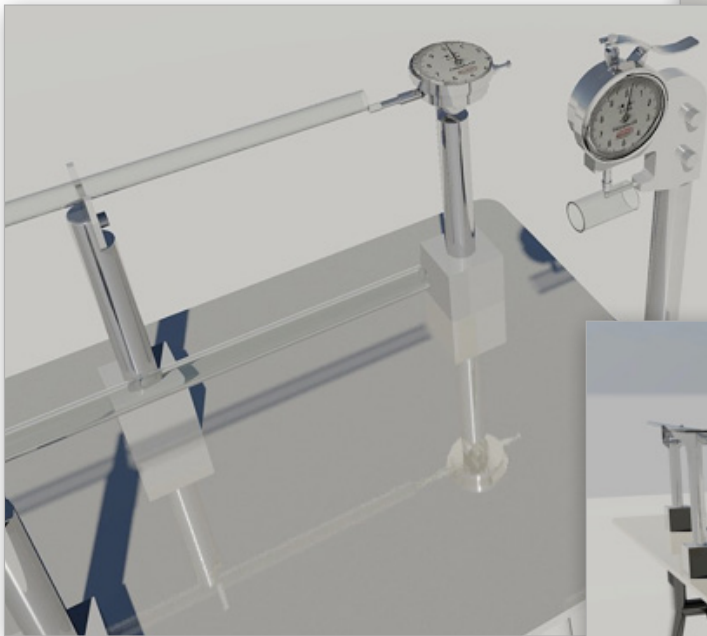
3) Thickness

It is the minimum distance measured between the internal and the external surface of the tube.

Each tube is considered defective and therefore rejected if the average of the measured thicknesses is out of tolerance.

AQL = 0.25 (cumulative)
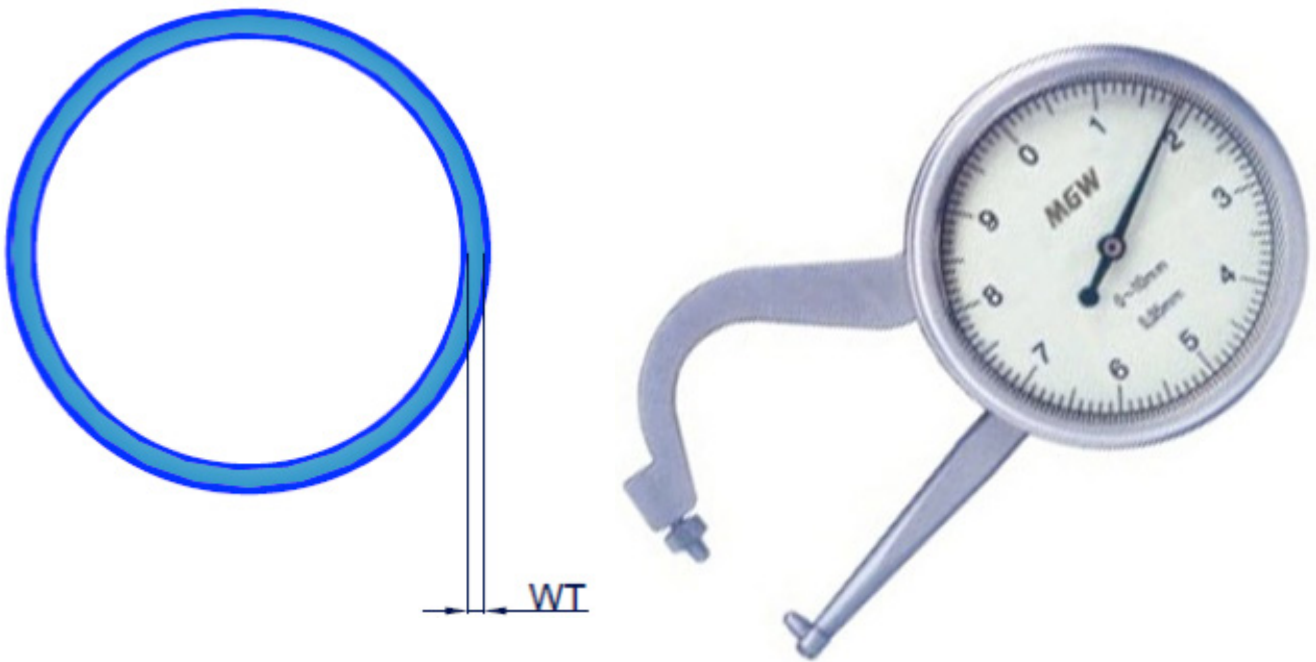
Measurement of the thickness will be done in different points of the tube between a point into the internal surface and one into the external one using a micrometer installed on a support which has an accuracy of 0.01mm. The thickness is defined by the average between the maximum and minimum measured-thickness.
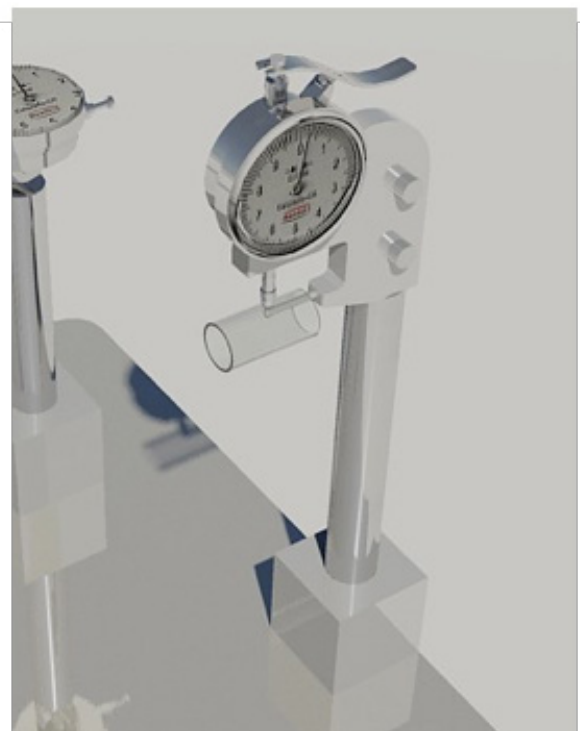
4) Oval-shaped (Out of Round)

It is the difference between the maximum and the minimum of the external diameter (OD) measured on the circumference of the tube, perpendicular to the axis of the tube itself.

$$Oval\ shaped = Max\ OD - Min\ OD$$

For measurement is used the same device as for measurement of external diameter.
Acceptable criteria for oval-shaped tubes depend on external diameter according to the following table:

| OD	Max value of ovalization
< 25.0 mm	0.4% of OD
25 – 35 mm	0.6 % of OD
> 35 mm	0.8 % of OD

AQL = 0.25 (cumulative)

5) Lopsided (siding) defect

It is the difference between the maximum and minimum thickness measured on the same section of the tube, perpendicular to the longitudinal axis.

For measuring can be adopted the same instrument used for measurement of thickness. Acceptable criteria = 6 % of the thickness of the tube. (This value can be diversified for the tubes which have a thinner wall – for ampoules = 6%, and for thicker tubes – vials = 8%)

AQL = 0.25 (cumulative)

6) Lenght
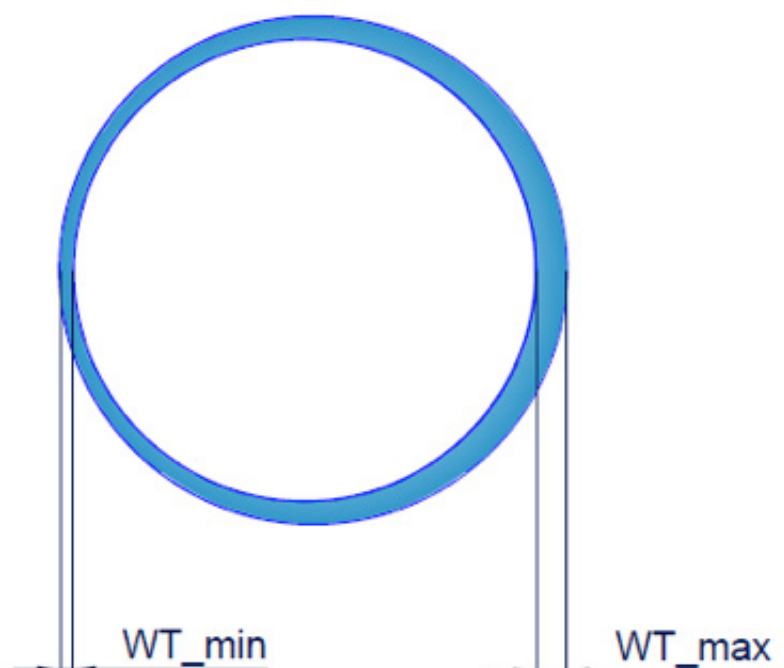
The standard lenght of the tube is 1500 +7/- 0 mm. Different sizes are possible on request. A mechanical meter measures it


L=1500 +7 / -0 mm

AQL = 0.25 (cumulative)

7) Out of the lineup (tube end not perpendicular to the tube axis)

It is the case, when the tip of the tube which is not orthogonal to the longitudinal axis of the tube itself.

It is measured after polishing and it is the distance T between the two planes, which are parallel each other and perpendicular to the axis of the tube.

The tube to be tested is placed on roller-supports fixed on a flat basement. The tube is positioned against a flat surface which is perpendicular to the axis of supports and which is of bigger dimensions of the tube to be measured; the contact point has to be clean and free from roughness.
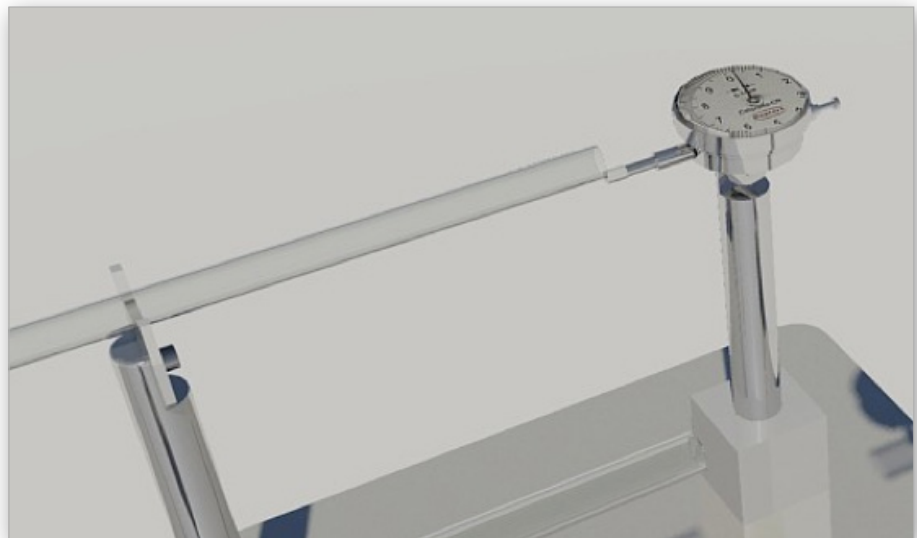
Make the tube rotate for 360°C supporting it on a flat surface and the difference of length is measured by placing on the free-extremity of the tube a comparator with an accuracy of +/-0.1mm. the measurement will be repeated on the other extremity.

The out of lineup error value is the biggest of length deviation established by the two measurements.

Tube is defective if the dimension T is bigger than 1.8 mm of the external diameter size.

$$T > OD + 1,8$$

AQL = 6.5

8) Bow

It is the definition of the maximum deviation of the center tube axis in comparison with a horizontal plane, when the tube has suffered a permanent deformation forming a bow.

Measurement is done positioning close to the ends of the tube on two horizontal supports of about 20 mm and making the tube rotate of 360° on its central axis. In the middle of the tube length a comparator is installed; the cursor of this comparator touches the tube. During the rotation of 360° the micrometer will measure the maximum deviation caused by the curvature. The value of the curvature is ½ of the deviation measured by the micrometer after 360° tube rotation.
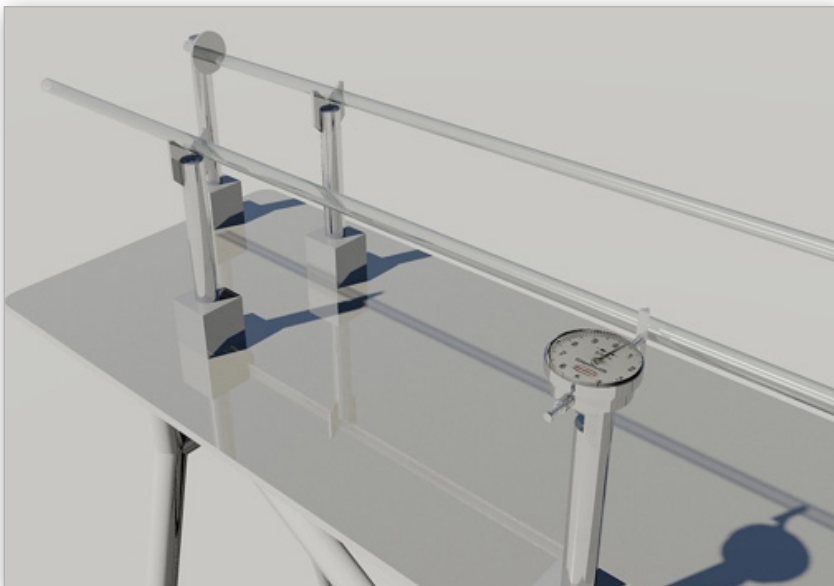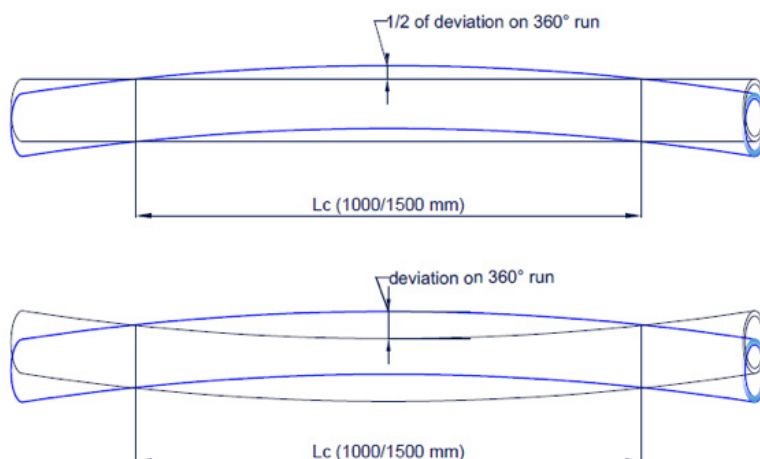

The maximum deviation can be referred to the length of 1500 mm or of 1000 mm.

The acceptability criteria of the bow are in function of the external diameter as shown in the following table:

	OD	Max deviation
Simple bow	4 – 6 mm	1.5 mm / 1500 mm
Simple bow	>6 mm	0.90 mm / 1000 mm
high bow	>/= 4 mm	0.20 mm / 200 mm

PS. High blow indicate a snake tube shape

AQL = 0.25 (cumulative)

Visual quality check

8) Closed lines

Lines are stretched-shaped-gaseous inclusions in the glass which form in the walls of the tube and which do not disturb neither internal nor external surface.

They form because of presence of gas bubbles in the glass melt. These bubbles are stretched during the forming process and because of this stretching form lines of different length, accordingly to the bubble dimension.
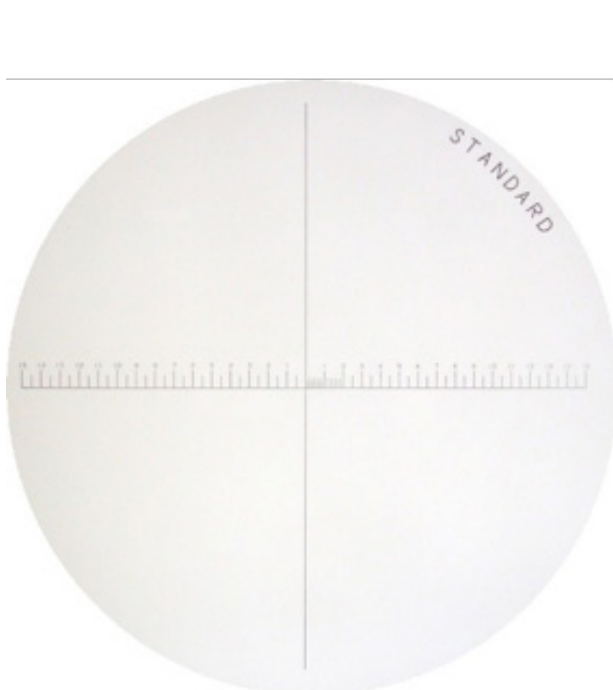
Length of aggregated lines is the sum of each line with longer than 15 mm.

About the method of check and measurement see chapter "Check Methods".

Lines are considered as defects if:
1) Width is above 0.1 mm
2) Length is above 500 mm
3) (according to Japanese specification) on a tube of 10 m are detect lines with length above 20 mm and the sum of these exceed 1500 mm (i.e. on a tube of 1500 mm cannot be present lines of >20 mm if their sum exceeds 225 mm.)

AQL = 0.25

Lines are detected and counted on a light desk. An optical micrometer is used to measure width. Lengths can be measured by a metal ruler.

9) Open lines

Important for the production of syringes.

The open lines come from stretched-shaped gaseous inclusions, which create a channel on one of the surfaces of the tube, either internal or external one.

The sum of all the lines, which have a length higher than 2 mm gives the aggregated length.

About the method of check and measurement see chapter "Check Methods".

These lines are considered as defects if:

lenght	Level AQL
< 2 mm	No defect
>2mm <100 mm	2.5
>100 mm	0.25

They are measured by same method used for closed lines.

Remark:
Some tube-suppliers (Schott for ex) classify the opened lines only for the production of syringes. In other case they are classified with the closed lines.

10) Stones and knots

Stones are opal inclusions typically coming from the wear-off of furnace refractory.

Knots are transparent glass-inclusions coming from non-homogeneity of the glass.

They are measured in the same way of the closed lines.

Attention: the measurement has to be effected only on the core of the defect and not on the glass-cavity, which surrounds the defect.

About the method of check and measurement see chapter "Check Methods".

Stones and knots constitute defectif:
1) Stones and knots with nucleus size > 0.5 < 0.8 mm ..AQL 2.5
2) Stones and knots with nucleus size > 0.8 mm ..AQL   0.25
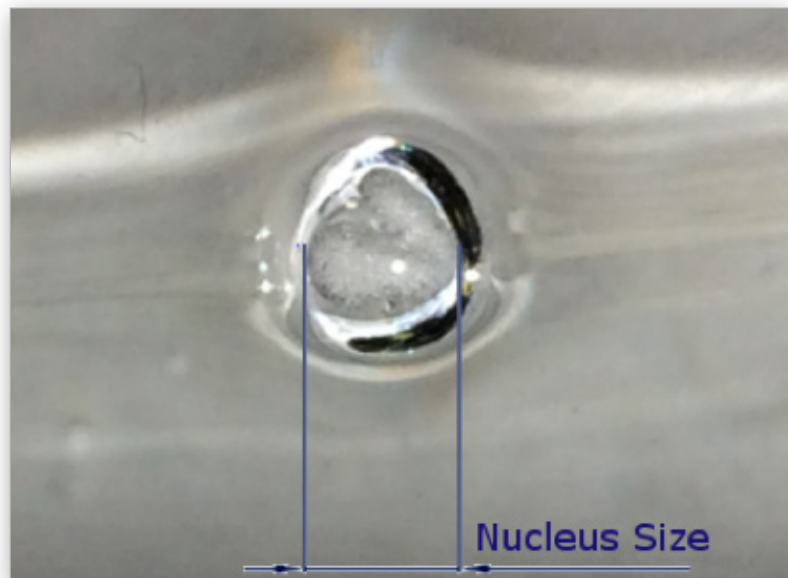
11) Cracks

They are fracture-lines without a complete separation of the surfaces involved.

A surface showing breakages on a tube or a container will reduce significantly the mechanical strength.

Surfaces with cracks and/or polished ends of the tube visible at unaided eye (not optical magnified device) with cracks longer than 2 mm are not accepted.

Acceptable criteria: Surface cracks: none
                            End cracks (first 3 cm from booth tube ends): AQL 0.65

About the method of check and measurement see chapter "Check Methods".

12) Scratches

They are defined as slight abrasion of the surface, which do not penetrate into the glass thickness significantly.

Normally scratches have insignificant effect on the mechanical properties of the glass.

About the method of check and measurement see chapter "Check Methods".

Definition of the defect:
1) Scratches of width >0.2mm and length > 100 mm
2) Two or more scratches with width > 0.2mm and an aggregate length > 150mm
3) Radial scratches width >0.2 mm which concerns half of the entire tube circumference

AQL = 1.5

13) Glass particles (fragments)

They are defined as little and thin glass fragments, which typically are found on the internal surface of the tube and originate from cutting and polishing operations.

An ultrasonic system and a further air-blow into the tube highly reduces the presence of fragments.

About the method of check and measurement see chapter "Check Methods".

The inspection of the tube is more effective if it is made using a fluorescent lamp and a desk covered by a black cloth as a background.

Definition of defects and acceptability; permissible particles for a tube of 1500 mm length.

The measurement of particles ca be effected using a magnifier up to a X8 or an optical micrometer.

OD	Number of measuring particles >0.2 fino a 0.5 mm	Number of measuring particles > 0.5 mm	AQL
<8.5 mm	11	1	0.25
>8.2 up to < 15 mm	15	2	0.65
>15 up to < 25 mm	20	3	1.5
>25 up to < 34.9 mm	30	4	2.5

14) Finishing of the tube-ends -reburning

The tube reburnig process melt by oxy-gas burner the tube ends to avoid the cutting risk and increase the tube mechanical stress resistance The tube end quality will be increase with reburning operation.

The reburning is:
1. Light: the cutting edge is reduced by a minimal glass edge melting
2. Medium: the tube ID is reduced by 20-30% by reburnig edge melting
3. Heavy: the tube ID is reduced by 70% by reburning edge melting. Usually the heavy reburning is use for bottle production on vertical forming machine

AQL = 0.65

About the method of check and measurement see chapter "Check Methods".

The defect is the difference between the internal diameter of the tube and the diameter of the polished reduction area.

For measuring use the mechanical caliper.

15) Dirt

It is given by any substance as grease, oil, powder or other organic and inorganic materials, which can visible at unaided eye (not optical device) under a normal light.

In particular:

for the internal surface are not allowed grease and oil. Impurities, which are >0.5mm, with AQL = 0.1 not attached, are considered as defects.

for the external surface impurities, which can be easily removed, are not considered as defects. Are counted only the impurities, which cannot be removed easily:
a) Grease and oil are defects as AQL = 0.1
b) Powder >2mm is defect with AQL = 0.65
c) Carbon (graphite) by contact with moving parts along the line AQL = 1.25

Each defect is counted as unique. Example: if one tube shows two oil-spots we consider two defects.

About the method of check and measurement see chapter "Check Methods".

APPENDIX 1. SAMPLING PLAN

It has to be effected according to the following norms:

simple sampling-plan for routine-tests of the production normally can follows a simplify test methods and quantities, for example:
- Take from each production line from 5 to 10 tubes each 4 hours.
- On each tube determine, according to the described methods, the requested parameters: (diameter, thickness, etc., as listed above). Moreover, it is necessary to count the visible parameters as lines, stones, knots and cords on each tube and resume the result in comparison to a standard quantity of glass, for example 5 kg. (this is necessary to have a picture of the defects which are not influenced by the dimension of the tube and therefore by the occurred phenomena) For lines is also useful to know if they are closed or opened, their thickness and their distribution accordingly to the length as per the following schema:

<2 cm
>2 <5 cm
>5 <15 cm
>15 <50 cm
> 50 cm

In the same form will be indicated information about cracks, scratches, dirt, glass fragments and polishing defects.
The test result can be record in the specific form.

Record module in enclosure 1 (quality recording form)

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