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Foreword

This translation has been made based on the original Japanese Industrial Standard revised by the Minister of International Trade and Industry through deliberations at Japanese Industrial Standards Committee in accordance with the Industrial Standardization Law. Consequently **JIS C 6704 : 1992** is replaced with **JIS C 6704 : 1997**.

Attention is drawn to the possibility that some parts of this Standard may conflict with a patent right, application for a patent after opening to the public, utility model right or application for registration of utility model after opening to the public which have technical properties. The relevant Minister and the Japanese Industrial Standards Committee are not responsible for identifying the patent right, application for a patent after opening to the public, utility model right or application for registration of utility model after opening to the public which have the said technical properties.

In this revision, the structure of Standard, standard values, requirement, test methods, etc. are revised for conformance with International Standard **IEC 758 : 1993-04**, *Synthetic quartz crystal—Specifications and guide to the use*.

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Synthetic quartz crystal

Introduction Though quartz crystal being raw materials of quartz crystal units for oscillators had depended upon natural quartz crystal imported from Brazil, production of quartz crystal units for oscillators increased by mass production of synthetic quartz crystal by a hydrothermal synthetic method, and Japanese Industrial Standard of synthetic quartz crystal was established in March, 1975. Thereafter, standards for Z-bar synthetic quartz crystal and lumbered synthetic quartz crystal were added in 1981, and the revision related to standardization with International System of Units was executed in 1992. Besides, IEC 758 : 1993-04, *Synthetic quartz crystal—Specifications and guide to the use* being the corresponding International Standard was revised largely conforming to diversification of uses of synthetic quartz crystal, and issued as the second edition in April, 1993.

This Standard is a JIS prepared based on IEC 758 without changing technical contents for the corresponding parts, but the items and contents which are not specified in the corresponding International Standard are added as Japanese Industrial Standard.

Parts to which the dotted underline is applied in this Standard are items which do not exist in the original International Standard.

Section 1 Specification for as-grown synthetic quartz crystal

1.1 Scope This Japanese Industrial Standard applies to synthetic quartz single crystals intended for manufacturing piezoelectric elements for frequency control and selection.

Remarks : The corresponding International Standard to this Standard is given below.

IEC 758 : 1993-04 *Synthetic quartz crystal—Specifications and guide to the use*

1.2 Normative reference The normative reference to this Standard is given below.

JIS C 6701 *Quartz crystal units for oscillators*

1.3 Definitions For the main terms used in this Standard, the following definitions apply.

1.3.1 hydrothermal crystal growth Literally this means crystal growth in the presence of water, elevated temperatures and pressures by a crystal growth process. The industrial synthetic quartz growth processes utilize alkaline water solutions confined within autoclaves at supercritical temperatures and pressures. The autoclave is divided into two chambers: the dissolving chamber, containing raw quartz chips at the higher temperature; the growing chamber, containing cut seeds at the lower temperature.

1.3.2 synthetic quartz crystal A single crystal of a quartz grown by the hydrothermal method. The crystal is of either handedness and in the “as grown” condition or in lumbered processed condition.

1.3.2A as-grown synthetic quartz crystal Synthetic quartz crystal grown but not yet processed.

1.3.2B as-grown Y-bar As-grown synthetic crystal grown by use of long bar seed in Y-direction.

1.3.2C as-grown Z-bar As-grown synthetic crystal grown by use of plate seed having Z-face which is long in Y-direction as main face.

1.3.3 synthetic quartz crystal batch Synthetic quartz crystals grown at the same time in one autoclave.

1.3.4 seed The rectangular cross-section quartz plate or bar used as a nucleus for crystal growth.

1.3.5 growth zones The regions of a synthetic quartz crystal resulting from growth along different crystallographic directions (see Fig. 1a).

1.3.6 orientation of a synthetic quartz crystal Orientation of its seed with respect to the orthogonal axes specified in 1.3.7.

1.3.7 definition of orthogonal axial system of a quartz crystal X-axis, Y-axis, and Z-axis intersecting each other at right angles as given in Fig. 2. As given in Fig. 5b, Y-axis and Z-axis which are slanted by θ° around X-axis as the centre are called Y'-axis and Z'-axis respectively. When pressure is applied along the X-axis, a direction which causes positive charge is called positive. A right-handed rectangular coordinate is used for right-handed quartz, and left-handed rectangular coordinate is used for left-handed quartz.

1.3.7A AT-cut plate A quartz crystal piece in which the main surface is parallel to X-axis, and slanted by about 35° from Z-axis and by about -3° from r-face (see Fig. 3a).

1.3.7B r-cut plate A quartz crystal piece in which the main surface is parallel to r-face (see Fig. 3a).

1.3.7C X-cut plate A quartz crystal in which the main surface is parallel to X-face (see Fig. 3b).

1.3.7D Y-cut plate A quartz crystal in which the main surface is parallel to Y-face (see Fig. 3b).

1.3.7E Z-cut plate A quartz crystal in which the main surface is parallel to Z-face (see Fig. 3b).

1.3.8 dimensions Dimensions pertaining to growth on a Z-cut seed rotated less than 20° from the Y-axis.

1.3.8.1 gross dimensions The maximum dimensions along the X-, Y'-, and Z- or Z'-axes measured along the X-, Y'- and Z'-axes.

1.3.8.1A effective Z dimension The minimum dimensions in Z ($\theta = 0^\circ$) or Z'-axis direction as given in Fig. 1c, which is represented by Z_{eff} .

1.3.8.1B minimum Z dimension The minimum distance to Z-surface from seed surface as given in Fig. 1d, which is represented by Z_{min} .

1.3.9 inclusions Any foreign material within a synthetic quartz crystal, visible by examination of scattered light from a bright source with the crystal immersed in a refractive index-matching liquid.

1.3.9.1 seed veil The array of inclusions or voids at the surface of the seed upon which a crystal has been grown.

1.3.10 dopant Any additive used in the growth process which may change the crystal habit, chemical composition, physical or electrical properties of the synthetic quartz batch.

1.3.11 pre-dimensioned bar Any bar whose "as grown" dimensions have been altered by sawing, grinding, lapping, etc., to meet a particular dimensional requirement.

1.3.12 impurity concentration The concentration of impurities is given in 10^{-6} units relative to silicon atoms.

1.3.13 dislocations A kind of lattice defect. Linear defects in the crystal due to misplaced planes of atoms.

1.3.13A etch channels Tubular void which appears along dislocations when quartz crystal is etched.

1.3.13B autoclave A high pressure container for growing synthetic quartz crystal.

1.3.13C right-handed quartz or left-handed quartz Right-handed quartz is quartz crystal of right-handed optical rotary power, and left-handed quartz is quartz crystal of left-handed optical rotary power, according to classification by the optical rotary power of quartz crystal.

1.3.13D twin Twin means an aggregate according to crystallographic law in which two or more crystals are joined each other in a symmetrical relation to a specific surface or axis, and the following classification exists.

- a) **electrical twins** A crystal in which two pieces of either right-handed quartz or left-handed quartz having a common Z-axis, and a mutual discrepancy of 180° on X-axes, are joined each other.
- b) **optical twins** A crystal in which right-handed quartz and left-handed quartz are joined having a common Z-axis.

1.3.13E infrared absorption coefficient α -value Though OH-joint lies as a defect in a crystal lattice, a specific wavelength in the infrared region is absorbed thereby. This term means the absorption coefficient calculated from the absorption rate. Since existence of the OH-joint gives an influence on mechanical inner loss of a vibrator, infrared absorption coefficient α -value is used as an index expressing the mechanical inner loss of the vibrator. Hereafter, the infrared absorption coefficient α -value is called the α -value.

1.4 Standard values

1.4.1 Orientation of the seed Standard values for orientation of the seed are: Z-cuts, rotated X-cuts, minor rhombohedral (z-minor) cut, 1.5° rotated Z-cut, 2° rotated Z-cut, 5° rotated Z-cut and 8°30' rotated Z-cut, the Z'-axis of the last four seeds being rotated as shown in Fig. 2.

1.4.2 Inclusion density The grade by inclusion density in Z-region (pieces per 1 cm³) shall be as given in Table 1.

Table 1 Grade by inclusion density

Grade \ Range of size μm	Maximum value of inclusion density for each grade			
	10-30	30-70	70-100	>100
Ia	2	1	0	0
Ib	3	2	1	1
I	6	4	2	2
II	9	5	4	3
III	12	8	6	4

Users requiring a grade in only one or more of the size ranges may designate their requirement as the grade followed by the appropriate size range.

1.4.3 Infra-red quality indication, α_{3500} , α_{3585} , α_{3410} An infra-red extinction coefficient value (α -value) of synthetic quartz shall be as listed under the appropriate heading for α_{3500} , α_{3585} , or α_{3410} in the following Table 2 for the various grades:

Further, α_{3500} , α_{3585} , α_{3410} are absorption coefficients of respective number of waves 3 500 cm⁻¹, 3 585 cm⁻¹, 3 410 cm⁻¹.

Table 2 Infra-red extinction coefficient value (α -value)

Grades	Maximum value of α -value of each grade		
	α_{3500}	α_{3585}	α_{3410}
Aa	0.026	0.015	0.075
A	0.033	0.024	0.082
B	0.045	0.050	0.100
C	0.060	0.069	0.114
D	0.080	0.100	0.145
E	0.120	0.160	0.190



1.4.4 The frequency versus temperature characteristics When the test is carried out according to 1.5.7, the frequency versus temperature characteristics of synthetic quartz crystal shall satisfy the following with respect to the series resonance frequency at 25 °C.

at 15 °C 0.5 to 1.5×10^{-6}

at 35 °C -0.5 to -1.5×10^{-6}

1.4.5 Etch channel density, ρ The etch channel density, ρ , per cm², for each grade shall comply with the listings in the following Table 3:

Table 3 Grade of etch channel density

Grades	Maximum number ρ per cm ²
1	10
2	30
3	100
4	300
5	600

1.5 Requirements and measuring methods

1.5.1 Orientation of seed The orientation of the seed shall be along specified directions, with a deviation of $\pm 30'$ from nominal.

1.5.2 Handedness of seed The handedness of the seed shall be specified, either right-hand or left-hand (see Fig. 2).

1.5.3 Synthetic quartz crystal dimensions Dimensions of as-grown synthetic quartz crystal are specified in the detail specification.

1.5.3.1 Dimension along Y or Y'-axis The dimension shall be the dimension of the maximum part as specified in Fig. 1d.

1.5.3.2 Dimension along Z or Z'-axis The dimension along the Z or Z'-axis shall be the dimensions of the maximum part to +X side as given in Fig. 1c.

1.5.3.2A Z_{eff} or Z'_{eff} dimension Z_{eff} or Z'_{eff} , as given in Fig. 1c, shall be dimension of the minimum part in Z or Z' axis direction.

1.5.3.2B Z_{min} , Z'_{min} dimension The dimension shall be as given in Fig. 1d.

1.5.3.3 Dimension along X-axis The gross dimension along the X-axis shall be as specified in Fig. 1c.

1.5.4 Seed dimensions

1.5.4.1 Z or Z'-dimension The Z or Z'-dimension of the seed shall be less than 3 mm.

1.5.4.2 X-dimension The dimension X of the seed shall be specified in the detail specification.

1.5.5 Imperfections

1.5.5.1 Twinning There shall be no electrical or optical twinning in the usable region.

1.5.5.2 Cracks and fractures There shall be no cracks or fractures in the usable region.

1.5.5.3 Inclusion density There are two methods as the test method. Either of them shall be selected as required.

Method 1 Inclusions, within size ranges required by the user, are counted visually per cm^3 in sample volumes within a crystal, using a stereo binocular microscope, operating at 30 to 40 magnification, equipped for counting within a circular field, and with a calibrated reticle scale for determining particle sizes, intense side illumination (such as halogen lamps) over a black matt background, an index matching fluid ($n = 1.55$, approximately) when needed for transparency, and appropriate means to measure the dimensions of the sample volumes counted.

Method 2 When application of method 1 is difficult, the judgement is carried out by comparing the specimen with the limit sample of the same tolerable number of inclusions given in the standard value. When transparency is required, the test is carried out in the liquid with the refractive index close to that of quartz, or with such liquid is coated thereon. In that case, the tolerable limit sample to be used is determined between the manufacturer and the users. A preparing example of the tolerable limit sample is stated in Annex C (informative).

1.5.5.3.1 Sampling In order to save time, labor, and cost, sampling is generally carried out by the method independently simplified as agreed upon between the supplier and the user. For the more precise inspection method, it is determined as agreed upon between the manufacturer and the user, the sampling is necessarily carried out by an effective statistical method so that the measured crystal satisfies the agreed quality assurance level test, and shall sufficiently represent the lot.

1.5.5.3.2 Batch sampling In most batch sampling, a suitable sample bar or group of bars is chosen to represent the batch population. The number of bars shall depend on the number in the batch, the type of crystal, the intended application, the separation between the mean and the target inclusion densities and the AQL confidence level requirement needed to provide sufficient assurance that the batch inclusion density in each size range shall be below their applicable grade test limits. The sample bar group shall reasonably represent the batch with respect to inclusion densities. Deviations, if any, are allowed and shall be towards higher, not lower, inclusion densities for safe assurance.

1.5.5.3.3 Volumes within a bar A group of volumes within each sample bar is next chosen for inclusion counting. The boundaries of the volumes are defined by the area of the microscope's focal field (or of a square reticle's outline) and either the bar's height or the length range of the microscope's depth adjustment chosen for use. It is necessary

to determine and total the volumes throughout which counts are accumulated. The volumes selected for counting should include mainly regions (usually Z-growth zones as in Fig. 3) whose material will be present and active in the finished devices, and should not avoid dense inclusion volumes within these regions. The number of volumes per bar shall be at least six or more for reasonable statistical confidence.

The sample volume locations within a bar shall be appropriately distributed in its X-, Y-, Z-axes to include the variations of the inclusion density with these independent variables. Typical synthetic quartz bars (Fig. 3a) are long in the Y- and small in the X- and Z-axes dimensions. Normally, the greatest variation of inclusion density appears over a zone's grown direction, e.g. the Z-dimension in the Z-zone. Thus for large Z-crystals, the sampled regions shall be located at varied Z-distances from the seed to ensure that the bar's range of Z is well represented by the group of sample volumes. Similarly, any noted variations over Y or X shall be sampled, if such variations are present.

To aid in distributing sample volumes within a typical bar, its lesser X-surface is marked with transverse Z'-lines, perpendicular to the seed at regular 10 mm intervals over the Y-length of the surface. Sample volumes for inclusion counting are chosen as needed from within each rectangle formed by the marks and the crystal surfaces. To locate the sample volumes at varied distances from the seed, in small crystals (where a Z-zone measures less than double the microscope's field diameter), they should be alternated near and far from the seed. For larger crystals, the volumes should be sequenced in Z over its range to ensure that each inclusion band is represented in the sample volumes. Several frequently used sampling plans are illustrated in Annex A (informative).

1.5.5.3.4 Inclusion counting The Inclusion counting shall be as follows.

The circular or square field of view chosen for counting within its marked rectangle is scanned vertically over its chosen X-height within the microscope's range of depth adjustment, as follows.

If the sample is a Y-bar with a relatively small X-height, the scans will be at a series of sites varied in Z along its length. Beginning in a rectangle positioned at one end of the usable zone, an inclusion count is taken in an X-cylinder (or parallelepiped) volume. Starting slightly below the lesser X-surface (and not counting surface material), all visible inclusions in focus are categorized and counted in each of the size categories required by the customer's order: 10 μm to 30 μm , 30 μm to 70 μm , 70 μm to 100 μm , and greater than 100 μm . The microscope is then lowered, and the newly focused inclusions counted and added into their size categories. This process is continued through the chosen X-height; the procedure is repeated at the next sample position, and so on.

The counts from the bar's sample sites in each of the four size categories are summed by category and divided by the calculated total of the sampled volumes, to obtain an average count per cm^3 for each category in one bar. The count averages in each category from all the sample bars from a run are averaged and recorded as required to represent the size distribution for the run. Maximum and minimum bar averages may also be recorded, if desired or required. A numerical example is offered in Annex B (informative).

1.5.6 Evaluation of infra-red quality by α -measurement For the α -value, transmittance is measured by using a Y-cut sample at a wave number of $3\,500\text{ cm}^{-1}$, $3\,585\text{ cm}^{-1}$ or $3\,410\text{ cm}^{-1}$, $3\,800\text{ cm}^{-1}$ or $3\,979\text{ cm}^{-1}$ being as reference, with an infra-red spectrophotometer. There are the following two methods as the test method, and either of them is selected as required.

Method 1 The wave number is fixed, and variation of the transmittance due to position is measured by moving the test sample.

Method 2 A test sample is fixed, and the transmittance at a specific wave number is measured at plural positions of the sample.

1.5.6.1 Preparation of the Y-cut sample A test sample of the following specification is prepared from the central part of Y-axis dimension of the test crystal. The α -value reflects the fluctuation in Z dimension. Therefore, the sample of average Z dimension in the lot, or the sample of the maximum Z dimension in which the α -value is considered the largest is used for measurement.

- a) Cut direction: Y-cut
- b) Thickness: 5.0 mm to 10.0 mm
- c) Surface polishing: mirror finish or polishing finish with homogenized mixture of $3\text{ }\mu\text{m}$ mean particle size

1.5.6.2 Calibration of a standard Y-cut slice in an infra-red spectrophotometer The infra-red spectrophotometer is turned on, allowed to warm and fully stabilize, then calibrated. The normal daily calibration includes its transmission (0 % – 100 %) or absorbance ($\log T = 1.0 - 0$) limit settings, chart speed and synchronized sample scanning arrangement. For evaluation and normal use, a 1.5 mm width aperture is located in the sample beam. For the lowest α -value measurements a 5 mm width aperture may be required. The aperture's height shall not exceed the X-dimension of the seed. Transmittance is adjusted at 100 %, while the sample is not attached and it is confirmed that the 100 % line being kept in a specific state by moving the wave number from $4\,000\text{ cm}^{-1}$ to $2\,000\text{ cm}^{-1}$. When the 100 % line is not kept constant, balance of the infra-red spectrometer is adjusted.

Then a polished Y-cut standard reference slice is placed first in the sample holder, which is then mounted in the scanning device. The wave number control is set at a background setting (outside, but near the OH absorption band), usually $(3\,800 \pm 3)\text{ cm}^{-1}$, and the sample is translated through the beam with synchronized chart advance at the fixed wave number. Such scanning is done only in the Z-growth zones of the Y-cut slice (illustrated in Figs. 1 and 3). In certain cases where background noise may be a problem, such as single beam operation, a higher background wave number $(3\,979 \pm 3)\text{ cm}^{-1}$, may be used for lowered background noise. If the background scan trace is not reasonably flat outside the original seed's boundaries, a thin film of fluorolube grease shall be applied to both major surfaces of the semi-polished Y-cut slice. Baseline changes shall not exceed 0.02 absorption units during a background scan. After the sample has completed a successful scan at background, the wave number is adjusted to the chosen $(3\,410, 3\,500\text{ or }3\,585 \pm 3)\text{ cm}^{-1}$. The sample is returned to its original position and the chart paper rerolled to the position where its wave number scan began. The sample is then scanned to plot its infra-red absorption at this wave number in

the absorption band. The calibration α -values (maximum and minimum) are calculated from this reference scan, using the equation:

$$\alpha = (\log_{10} T_1 - \log_{10} T_2) / t$$

where, T_1 : transmittance at reference wave number
(3 800 cm^{-1} or 3 979 cm^{-1})

T_2 : transmittance at absorption wave number
(3 500 cm^{-1} , 3 585 cm^{-1} or 3 410 cm^{-1})

t : thickness of test sample expressed in cm

The spectrophotometer is considered in proper calibration if its $\alpha_{\max}$ and $\alpha_{\min}$ readings are repeatable within ± 0.004 units of the standard's values for them. A standardization correction may be calculated as needed to bring the instrument's reading on a standard slice to an accepted value, and used while current.

1.5.6.3 Test measurement of a Y-cut slice An α -value is measured under the following conditions.

a) **Measuring wave number:**

- 1) Reference wave number zone 3 800 cm^{-1} or 3 979 cm^{-1}
- 2) Absorption wave number 3 500 cm^{-1} , 3 585 cm^{-1} or 3 410 cm^{-1}

b) **Slit:** The slit of the following dimensions is placed to a sample beam side.

- 1) Width 1.5 mm to 5.0 mm
- 2) Height X dimension of seed or not exceeding 5 mm

c) **Test region:** The infrared radiation is transmitted through the Z region part of test sample. However, a region ± 2.0 mm from the centre of seed and a region grown exceeding the aimed lumbered dimension are rejected from the evaluation.

d) **Direction of test sample:** A test sample is placed on a test stand with its +X direction faced upwards.

After calibration, each test sample (mirror finished sample is preferable) is scanned in the reference wave number region and at wave numbers of 3 500 cm^{-1} , 3 585 cm^{-1} or 3 410 cm^{-1} . In the case of polishing finish, an oil film is necessarily applied onto a sample surface. The α -value is calculated according to the formula stated above.

1.5.7 Frequency versus temperature characteristics The specifications for the quartz crystal unit for evaluating the frequency versus temperature characteristics shall be as follows and measurements shall be as follows:

- frequency 10 MHz $\pm$ 10 kHz (fundamental);
- location of specimen Z-zone;
- orientation of plate AT-cut; 35°13' $\pm$ 30";
- shape of plate square flat and parallel plate with one edge along the X-axis;
- lateral dimensions of plate 8 mm $\times$ 8 mm;