

Synthetic quartz crystal — Specifications and guide to the use

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ICS 31.140

National foreword

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**Synthetic quartz crystal –
Specifications and guide to the use
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Spécifications et guide d'utilisation
(CEI 60758:2004)

Synthetischer Quarzkristall -
Festlegungen und Leitfaden
für die Anwendung
(IEC 60758:2004)

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Foreword

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Endorsement notice

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SYNTHETIC QUARTZ CRYSTAL – SPECIFICATIONS AND GUIDE TO THE USE

1 Scope

This International Standard applies to synthetic quartz single crystals intended for manufacturing piezoelectric elements for frequency control and selection.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

IEC 60068-1:1988, *Environmental testing – Part 1: General and guidance*

IEC 60122-1:2002, *Quartz crystal units of assessed quality – Part 1: Generic specification.*

IEC 60410:1973, *Sampling plans and procedures for inspection by attributes*

IEC 61994 (all parts), *Piezoelectric and dielectric devices for frequency control and selection – Glossary*

3 Terms and definitions

For the purposes of this document, the following terms and definitions, as well as those given in IEC 61994, apply.

3.1

hydrothermal crystal growth

literally, crystal growth in the presence of water, elevated temperatures and pressures by a crystal growth process believed to proceed geologically within the earth's crust. The industrial synthetic quartz growth processes utilize alkaline water solutions confined within autoclaves at supercritical temperatures (330 °C to 400 °C) and pressures (700 to 2 000 atmospheres). 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 (see 7.1.2)

3.2

synthetic quartz crystal (also known as cultured quartz crystal)

single crystal of α quartz grown by the hydrothermal method. The crystal is of either handedness and in the as-grown condition

3.2.1

as-grown synthetic quartz crystal

single crystal quartz grown hydrothermally. As-grown refers to the state of processing and indicates a state prior to whatever treatment might occur after growth, excluding quality control operations

3.2.2

as-grown Y-bar

crystals which are produced using seed with the largest dimension in the Y-direction

3.2.3**as-grown Z-bar**

crystals in which the Z-grown sector is much larger than the X-grown sector. The relative size of the growth sector is controlled by the X-dimension of the seed

3.3**synthetic quartz crystal batch**

synthetic quartz crystals grown at the same time in one autoclave

3.4**seed**

rectangular parallelepiped quartz plate or bar to be used as a nucleus for crystal growth

3.5**growth zones**

regions of a synthetic quartz crystal resulting from growth along different crystallographic directions (see Figure 1)

3.6**orientation of a synthetic quartz crystal**

orientation of its seed with respect to the orthogonal axes specified in 3.7

3.7**orthogonal axial system of a quartz crystal****3.7.1**

axial system for quartz illustrated in Figure 2

NOTE The Z-cut seed may be oriented at an angle of less than 20° to the Y-axis, in this case the axial system becomes X, Y', Z'.

3.7.2**AT-cut plate**

rotated Y-cut crystal plate oriented at an angle of about $+35^\circ$ around the X-axis or about -3° from the z (minor rhombohedral)-face as shown in Figure 3

3.7.3**z (minor rhombohedral)-cut plate**

crystal plate parallel to the z (minor rhombohedral)-face as shown in Figure 3a

3.7.4**X-cut plate**

crystal plate perpendicular to the X-axis as shown in Figure 3b

3.7.5**Y-cut plate**

crystal plate perpendicular to the Y-axis as shown in Figure 3b

3.7.6**Z-cut plate**

crystal plate perpendicular to the Z-axis as shown in Figure 3b

3.8**dimensions**

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

3.8.1

gross dimensions

maximum dimensions along the X-, Y-, or Y'-, and Z- or Z'-axes measured along the X-, Y'- and Z'-axes

3.8.1.1

effective Z-dimension

as-grown effective Z dimension defined as the minimum measure in the Z ($\theta = 0^\circ$) or Z' direction in usable Y or Y' area of an as-grown crystal and described by Z_{eff} , as shown in Figure 1

3.8.1.2

minimum Z-dimension

minimum distance from seed surface to Z-surface described by Z_{min} as shown in Figure 1d

3.8.2

dimensions pertaining to growth on a Z-cut seed rotated more than 20° from the X-axis
(under consideration)

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. A particularly common inclusion is the mineral acmite (sodium iron silicate)

3.9.1

seed veil

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

3.9.2

etch channel

roughly cylindrical void that is present along dislocation line after etching a quartz crystal

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

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

3.12

impurity concentration

concentration of impurities relative to silicon atoms

3.13

dislocations

linear defects in the crystal due to misplaced planes of atoms

3.14

etch channel

roughly cylindrical void present along a dislocation line after etching a test wafer prepared from a quartz crystal

3.15**autoclave**

vessel for the high-pressure high-temperature condition required for growth of synthetic quartz crystal

3.16**right-handed quartz or left-handed quartz**

handedness of quartz crystal as determined by observing the sense of handedness of the optical rotation in the polarized light. Right-handed quartz is the crystal of dextrorotatory and left-handed quartz is the crystal of levorotary

3.17**twins**

twins follow laws of crystallography relating symmetrically to specific faces or axes.

The following types have been identified in synthetic quartz crystals:

a) **Electrical twins**

Quartz crystal in which regions with the common Z-axis exist showing a polarity reversal of the electrical X-axis.

b) **Optical twins**

Quartz crystal in which regions with the common Z-axis exhibit handedness reversal of the optical Z-axis

3.18**infrared absorption coefficient α -value**

coefficient (referred to as the α -value) established by determining the relationship between absorption of two wavelengths: one with minimal absorption due to OH impurity, the other with high absorption due to presence of OH impurities in the crystal lattice. The OH impurity creates mechanical loss in resonators and its presence is correlated to the presence of other loss-inducing impurities. The α -value is a measure of OH concentration and is correlated with expected mechanical losses due to material impurities. The infrared absorption coefficient α -value is determined using the following equation:

$$\alpha = \frac{1}{t} \log \frac{T_1}{T_2}$$

where

α is the infrared absorption coefficient;

t is the thickness of Y-cut sample, in centimetres;

T_1 is the per cent transmission at a wave number of 3 800 cm⁻¹ or 3 979 cm⁻¹;

T_2 is the per cent transmission at a wave number of 3 410 cm⁻¹, 3 500 cm⁻¹ or 3 585 cm⁻¹.

3.19**lumbered synthetic quartz crystal**

synthetic quartz crystal whose X- and Z- or Z'- surfaces in the as-grown condition have been processed flat and parallel by sawing, grinding, lapping, etc., to meet specified dimensions and orientation

3.19.1**lumbered Y-bar**

quartz bars which are lumbered from an as-grown Y-bar

3.19.2**lumbered Z-bar**

quartz bars which are lumbered from an as-grown Z-bar

3.20

reference surface

surface of the lumbered bar prepared to specific flatness and orientation with respect to a crystallographic direction (typically the X-direction)

4 Specification for as-grown synthetic quartz crystal

4.1 Standard values

4.1.1 Orientation of the seed

Standard orientation for the seeds are Z-cuts and rotated X-cuts, minor rhombohedral (z-minor) cut, 1°30' rotated Z-cut, 2° rotated Z-cut, 5° rotated Z-cut, and 8°30' rotated Z-cut, the Z'-axis of the latter three seeds being rotated as shown in Figure 2.

4.1.2 Inclusion density

The inclusion density (measured as in 4.2.5.3) for each grade shall not exceed the figures in any required size range for that grade listed in Table 1.

Table 1 – Inclusion densities for the grades

Grade/size range μm	Densities per cm^3			
	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.

4.1.3 Infra-red quality indications, α_{3500} , α_{3585} , α_{3410}

An infra-red extinction coefficient value (α -value) of synthetic quartz (measured as in 4.2.6) shall be as listed under the appropriate heading for α_{3500} , α_{3585} , or α_{3410} in Table 2 for the various grades:

Table 2 – Infra-red quality indications for the grades

Grades	Maxima			Pre-1987 ^a Q · 10 ⁶ units
	α_{3500}	α_{3585}	α_{3410}	
Aa	0,026	0,015	0,075	3,8
A	0,033	0,024	0,082	3,0
B	0,045	0,050	0,100	2,4
C	0,060	0,069	0,114	1,8
D	0,080	0,100	0,145	1,4
E	0,120	0,160	0,190	1,0

^a These Q-values were obtained from α -measurements and empirical correlation, and were in common usage prior to 1987. These are included here as the previous labels to maintain continuity through the change in emphasizing α -labels. α is the physical measurement now used to control and specify quality in synthetic quartz.



The test limits above either correspond to or are unchanged (except in the cases of grades B and D) from the α_{3500} limits that correspond to the Q -value grades listed in the first edition of IEC 60758. This earlier publication designated some of the same grades in terms of minimum indicated Q 's in 10^6 units, as follows:

- A = 3,0;
- B = 2,2 (basis used herein), changed from 2,4 in the earlier edition;
- C = 1,8;
- D = 1,4 (revised);
- E = 1,0 (the same as the earlier D-grade).

4.1.4 Frequency-versus-temperature characteristics (Figure 4 and 4.2.7)

The frequency-versus-temperature characteristics of synthetic quartz crystal units shall be assessed by determination of the fractional frequency deviation measured at 15 °C and 35 °C with respect to the series resonance frequency at 25 °C. The fractional deviation shall satisfy the following:

- fractional frequency deviation at 15 °C: +0,5 to $+1,5 \times 10^{-6}$;
- fractional frequency deviation at 35 °C: $-0,5$ to $-1,5 \times 10^{-6}$.

Measurement shall be made in accordance with 4.7.3 of IEC 60122-1.

4.1.5 Etch channel density ρ

When required, the etch channel density, ρ , per cm^2 (measured as in 4.2.8) for each grade, shall comply with the listings in Table 3.

Table 3 – Etch channel densities for the grades

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

4.2 Requirements and measuring methods

4.2.1 Orientation

The orientation of the seed shall be along specified directions, with a deviation of less than 30 min from nominal.

4.2.2 Handedness

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

4.2.3 Synthetic quartz crystal dimensions

The dimension shall be measured by calipers or point calipers which enable the hollow point of a synthetic quartz crystal to be measured (see Annex D).

4.2.3.1 Dimension along Y or Y'-axis

The dimension shall be as specified (see Figure 1d).

4.2.3.2 Dimension along Z or Z'-axis dimension shall be measured by a neck ipers

The dimension along the Z or Z'-axis shall be specified as the maximum dimension along the Z or Z'-axis in the greater X zone (see Figure 1c).

4.2.3.3 Dimension Z_{eff} or Z'_{eff}

The Z_{eff} or Z'_{eff} dimension shall be specified as the minimum dimension along the Z or Z'-axis (see Figure 1c).

4.2.3.4 Dimension Z_{min} or Z'_{min}

The dimension shall be as specified (see Figures 1c and 1d).

4.2.3.5 Dimension along X-axis

The gross dimension along the X-axis shall be as specified (see Figure 1c).

4.2.4 Seed dimensions

4.2.4.1 Z or Z'-dimension

The Z or Z'-dimension (i.e. thickness) of the Z-cut or rotated Z-cut seed shall be less than 3 mm, unless otherwise specified.

4.2.4.2 X-dimension

The dimension X of the seed shall be as specified.

4.2.5 Imperfections

4.2.5.1 Twinning

There shall be no electrical or optical twinning in the usable region. The existence of twinning shall be checked by visual inspection.

4.2.5.2 Cracks and fractures

There shall be no cracks or fractures in the usable region. The existence of cracks and fractures shall be checked by visual inspection.

4.2.5.3 Inclusion density

The following two measuring methods are used and either one may be chosen.

Method 1

Inclusions within stated ranges 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 either a circular or a square field and with a calibrated reticule scale for determining particle sizes, intense side illumination (such as halogen lamps) over a recessed black matt background, an index matching liquid ($n = 1,55$, approximately) for transparency, and means of measuring the dimensions of the sample volumes counted.

Method 2

In case it is difficult to apply method 1, crystals are compared with reference samples appropriately representing each grade range, immersing within an index matching liquid ($n = 1,55$ approximately) for transparency, or applying such liquid to the surface. The reference samples shall be agreed upon between the supplier and the user. An example for the reference sample selection procedure is given in Annex C.

4.2.5.3.1 Sampling

Because of the considerable costs in time, labour and money, some plan for sampling both bars and regions within the bars is normally used by agreement between the supplier and the buyer when quality control of either inclusion density or etch channel density is required.

Clearly, the preferable low-cost inspection situation is the one in which the densities of inclusions or etch channels are well below the test limits, and infrequent samples can be justified. Since such situations are not always attainable, more rigorous inspection strategies will sometimes be required for appropriate density control, and shall be found, worked out, and agreed upon between the supplier and the user.

Sound statistical methods are required in order to meet appropriate agreed-upon assured quality level tests and ensure that the crystals and the volumes counted within them are sufficiently representative. Since sampling procedures and statistical confidence tests are described in the literature, their principles will not be repeated here.

4.2.5.3.2 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.

4.2.5.3.3 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 focal field of the microscope (or the outline of a square reticule) and either the height of the bar or the length range of the depth adjustment of the microscope 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 Figure 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 (Figure 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, for example, the Z-dimension in the Z-zone (Figures 1a and 3). 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 field diameter of the microscope), 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.

4.2.5.3.4 Inclusion counting

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 (under side illumination, with its lesser X-surface up). 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 given in Annex B.

4.2.6 Evaluation of infra-red quality by alpha-measurement

The infra-red absorption per centimetre at one or more of 3 410, 3 500 or 3 585 wave numbers is measured in a Y-cut slice scan as the difference between the absorption at the chosen wave number and absorption in the background outside the band, at 3 800 cm^{-1} or at 3 979 cm^{-1} when using a single beam instrument. Since alpha are known to vary directly with the total Z-growth size distribution of the crystals in one batch, a user may designate his preference in the batch distribution for sample testing. Such choices are often either an average Z-crystal or a maximum Z-crystal (for worst-case maximum alpha-measurement).

The following two measuring methods are used and either one shall be chosen.

Method 1

The wave number is fixed and the positional transmission data are measured by scanning the sample.

Method 2

The sample is fixed and the transmission data are measured at chosen wave numbers on several points in the sample.

4.2.6.1 Preparation of the Y-cut slice

The synthetic quartz crystal to be sampled is mounted on a substrate then sliced with a quartz saw to yield at least one Y-cut slice whose thickness after lapping and polishing will fall in the range of 5 mm to 10 mm. The 5 mm thickness is appropriate for high α -material, to resolve its α -variations; the mid-range for medium α ; and the 10 mm thickness is appropriate for the lowest α -material to measure its small absorption.