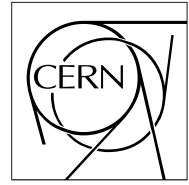


The Compact Muon Solenoid Experiment

CMS Note

Mailing address: CMS CERN, CH-1211 GENEVA 23, Switzerland



May 19, 1998

SPECIFICATIONS FOR LEAD TUNGSTATE CRYSTALS PREPRODUCTION

E. Auffray, M. Lebeau, P. Lecoq, *CERN, Geneva Switzerland*

M. Schneegans, *LAPP, Annecy, France*

Abstract

After 3 years of extensive R&D the Bogoroditsk Techno-Chemical Plant in Russia is ready to enter a preproduction phase where 6000 Lead Tungstate crystals will be produced for the CMS electromagnetic calorimeter during a period of about 18 months. Although this is a demonstration test for the mass production, these crystals will have to be installed in the calorimeter and have therefore to comply with the very severe constraints imposed by the ambitious performance goal of the calorimeter in the high radiation environment of LHC. This paper describes and justifies the specifications for these preproduction crystals.

1-Introduction

At the end of 1994, a vigorous Research and Development program was started in Russia and China to define the technology for the mass production of more than 80000 Lead Tungstate crystals, 23cm long, for the CMS electromagnetic calorimeter (ECAL). The ambitious performance goal of the ECAL and the high radiation levels at LHC impose strict requirements on several parameters of these crystals, such as a good light yield (in order to not be dominated by statistical fluctuations in the reconstructed energy resolution), a fast decay time without contamination by slow components (to avoid pile-up problems) and a very small light yield loss under irradiation (to allow precise corrections from the monitoring system). Moreover, precise dimensions will guarantee a good hermiticity of the detector. Last but not least, all these performances must be obtained using simple technologies which should guarantee an affordable price for CMS, imposing a target price of 1.5\$/cc.

During 3 years, the Bogoroditsk Techno-Chemical Plant in Russia has produced several hundreds of full size crystals with continuous improved performances, which were intensively tested in thousands of laboratory measurements of different nature, as well as in several test beam campaigns. It is now important to confirm the validity of the crystal optimisation on a scale in mass production conditions. For this purpose, a preproduction of 6000 crystals of the ECAL barrel will take place, 1000 crystals in 1998 and 5000 in 1999. Realistic specifications (defined for the barrel) will guarantee a good detector performance with the minimum implication on the crystal cost. This preproduction will help fine tune these specifications for the mass production and understand the production yields.

We consider 3 domains for the specifications, defining the acceptance tests and procedures which will be performed at the production centre before shipment, and in the regional centres at the reception of the crystals (all measurements are made at a temperature of 18°C):

- geometry
- optical properties
- radiation tolerance

In addition a visual inspection will be performed on each crystal in order to detect obvious defects before any measurement.

2- Visual properties

Some parameters of the crystal quality such as possible colouring, cracks or defects of any sort can easily be seen by eye and a check list will allow to immediately reject crystals presenting such obvious defects (see section 6).

After delivery in the regional centers, the crystals will be unpacked and first go to a visual inspection procedure. There, they will be checked for possible damage, for correct surface treatment and chamfers and for absence of obvious defects in their bulk or near to their surface. The presence of a label with an identifying barcode, stuck on the correct face, as shown in annex1, will also be checked.

This visual inspection is a short task which will allow immediate rejection of a crystal without spending more time on its characterisation.

3-Geometry

3.1. Introduction to the geometry

The dimensions and shapes specified in this document are related to the geometry version 5.2 of the calorimeter barrel part [1]. There are 17 different shapes, each one in right-hand and left-hand versions. A set of one right-hand and one left-hand crystals of the same type is called a flatpack [2] because they can be put together to form a single simple geometrical shape (Annex. 1). Contrary to appearance, the solids thus produced are not pyramidal frustums, as the side faces do not converge to a single apex. Each shape is entirely defined by a set of six lateral dimensions, the length -identical for all types- and the angular relationship between the indicated reference faces - identical for all types. Other angular values -e.g. between opposite side faces or adjacent non-reference faces- may be used for production purposes but have no contractual value.

3.2. Geometrical data

All dimensions and dimensional tolerances are given in millimeters. All surface finishes are given in micrometers.

The face numbering is given in table 1, the crystal being seen with its small end in front and its reference side face (N°6) down:

front	rear	left	right	up	down
1	2	3	4	5	6

Table 1: Definition of the face numbering

In the right-hand version, face N°3 is normal and face N°4 is slanted with respect to face N°6.

In the left-hand, version face N°3 is slanted and face N°4 is normal with respect to face N°6.

The reference faces are faces N°6, N°2 and N°3 (right-hand) or N°4 (left-hand). These three faces form a **rectangular reference trihedron**. Face N°1 is parallel to face N°2, but its position is defined as normal to face N°6 and N°3 (right-hand) or N°4 (left-hand).

3.3. Dimensional data

The six **lateral dimensions AF, BF, CF, AR, BR and CR** are given in annex 1 to a precision of 0.01 mm. Suffix F defines the dimensions of face N°1 (F for Front) and suffix R defines the dimensions of face N°2 (R for Rear). A is the dimension across face N°5, B across face N°3 (right-hand) or N°4 (left-hand) and C across face N°6. The crystal **length L** is the distance between faces N°1 and N°2.

Chamfers are made on all 12 edges. Chamfer should be cut to the following limits:

$$0.3 \leq \text{chamfer} \leq 0.7 \text{ mm.}$$

The surface finish of chamfers can be left at a roughness of 0.5 μm (lapping).

Surface finish of faces N°1, 2, 3 (right-hand) or 4 (left-hand), 5 and 6 is a **polished finish** (roughness $R_a < 0.02 \mu\text{m}$) produced on a polishing machine equipped with a special polishing cloth and using diamond abrasive of grain size 3 μm in emulsion. From the surface finish provided by the previous operation (lapping), about 10 minutes are necessary to reach the required surface finish.

Surface finish of faces N°3 (left-hand) or 4 (right-hand) is a **semi-polished finish** (roughness $R_a \approx 0.20 \mu\text{m}$) using the same equipment as for polishing, but for a shorter time. The detailed polishing method is defined in ref. [3].

3.4. Tolerances

The tolerance ranges specified below have been achieved on tests performed at Bogoroditsk and CERN when the processing method was validated [4,5,6,7]. This degree of precision is required to achieve the tight hermeticity of the detector, in particular the average crystal face-to-face distance of 0.5mm, in which the dimensional tolerance amounts to 0.1mm, the air gap necessary for elastic deformation to 0.1mm, the alveolar walls for 0.2mm inside an alveolar and 0.4mm between alveolar. Special care will be taken to apply the tolerance criteria to the inspection measurements: in particular the measuring accuracy of the 3-dimensional measuring machine will be added to the tolerance, and measurements repeated in case of marginal or infringing values.

Dimensional tolerances for L, AF, BF, CF, AR, BR and CR are all **+0 -0.100 mm** to nominal.

Angular tolerances for faces normal to references are specified in **perpendicularity**. The relevant tolerance is **0.050 mm** across a maximum length of 25 mm.

Planarity for all faces should be kept within **0.020 mm**.

Surface finish tolerances will be assessed by **visual inspection** and comparison to samples (polished and semi-polished finishes). In case of incorrect light uniformity, sampling **roughness measurements** will be performed on the semi-polished face N°3 (left-hand) or 4 (right-hand). The measurements will be made across and along the face in 8 points as illustrated on annex 2.a. The average measured value should abide the following conditions:

$$0.15 \mu\text{m} \leq \text{Ra average} \leq 0.25 \mu\text{m} \text{ with } \sigma \leq 0.10 \mu\text{m}$$

3.5. Dimensional inspection procedure and definition of the measurements

The mentioned **rectangular reference trihedron** will be used to position and define all measurements, using a three dimensional measuring machine to reconstruct the actual geometry from a pre-determined set of points and compare it to the nominal geometry.

On faces N°1 and N°2, nine points will be measured, the eight border points being not farther from the edge than 2 mm. On faces N°3 to 6, 15 points will be measured, the 12 border points being not farther from the edge than 2 mm. The layout of the measuring points is given in annex 2.b. The position of the points will be adapted to the supporting conditions of the measuring machine.

The set of points measured on any given face will define an **average plane**, using the mean square method. The plane parallel to the average plane and containing the outmost measured point is the **outmost plane**. The plane parallel to the average plane and containing the inmost measured point is the **inmost plane**. The position of this plane is of course known to the accuracy of the measuring machine.

The **maximum measure of length L** is computed as the distance of the outmost point in face N°1 to the outmost plane of face N°2. The **minimum measure of length L** is the distance of the inmost point in face N°1 to the inmost plane in face N°2. The minimum and maximum of the length should be measured within the tolerance margin +0 ; -0.100 mm, i.e.:

$$L - 0.100\text{mm} \leq L \text{ min (measured)} < L \text{ max (measured)} \leq L$$

The lateral dimensions AF, BF, CF, AR, BR and CR are computed by the intersection of the side faces (N°3 to 6) with the end faces (N°1 and 2). The intersection of the outmost side planes with the average end planes produces the **maximum measured lateral dimensions**. The intersection of the inmost side planes with the average end planes produces the **minimum measured lateral dimensions** (Annex 3). The minimum and maximum of lateral dimensions should be measured within the tolerance margin +0 ; -0.100 mm, e.g.:

$$AF - 0.100\text{mm} \leq AF \text{ min (measured)} < AF \text{ max (measured)} \leq AF$$

Flatness tolerance is measured as the distance between inmost and outmost planes for every of six faces. Because of the polishing method, no accidental point is expected, out of a regular surface, easy to represent by a reasonable number of points (15 in this case).

Perpendicularity of a measured face to a reference face is the angular error $\Delta\alpha$ between the average plane of the measured face and the average plane of the reference face, multiplied by the transversal rear dimension of the measured face (Annex 4), e.g. for face N°3 with respect to face N°6 of a right-hand crystal, α nominal = 0 and :

$$-0.050 \text{ mm} \leq \Delta\alpha \times BR \leq +0.050 \text{ mm}$$

3.6. Crystal identification

During the last processing operations crystals will receive an identification number. This number will be encoded in a bar code on a label, stuck on face N°1 as shown in annex 1, and be recorded in a database [8].

4- Optical properties

4.1-General considerations

A complete optical characterisation of all the crystals would require too much time and in some cases heavy equipment which is not compatible with a production facility which has to measure several tens of crystal per day. This is why only relatively simple and fast properties, such as optical transmission, decay time and light yield will be systematically measured. For parameters which are difficult to measure directly, studies were made to find correlations with other properties more easily accessible. This is particularly true for the radiation hardness, as will be explained in the next section. For such cases, the correlated property will be systematically measured, whereas the direct parameter will be studied on a sampling basis, allowing to confirm the validity of the correlation. Some other parameters which do not have a direct impact on the detector performance,

such as emission spectra, will also be measured on a sampling basis in order to check the stability of the production parameters.

4.2- Optical transmission

Transversal and longitudinal transmission spectra across PbWO_4 crystals are rather easy and fast measurements. They give useful indications on the crystal optical quality and have some correlation with radiation hardness (see section 5). In particular several years of intense R&D have shown that:

- in order not to affect the light collection uniformity in the crystal, a threshold on the longitudinal transmission at ~ 420 nm guarantees a minimum absorption length of ~ 80 cm,
- a low transmission at 600 nm indicates large core defects, which may affect the light collection and locally the radiation hardness,
- crystals showing an absorption band at 350 nm usually have a weak radiation hardness,
- crystals with the rise of their transversal transmission spectra variable with the position of the measurement along their length will be difficult to uniformize in light collection and also present non-uniform radiation hardness (see Fig. 1).

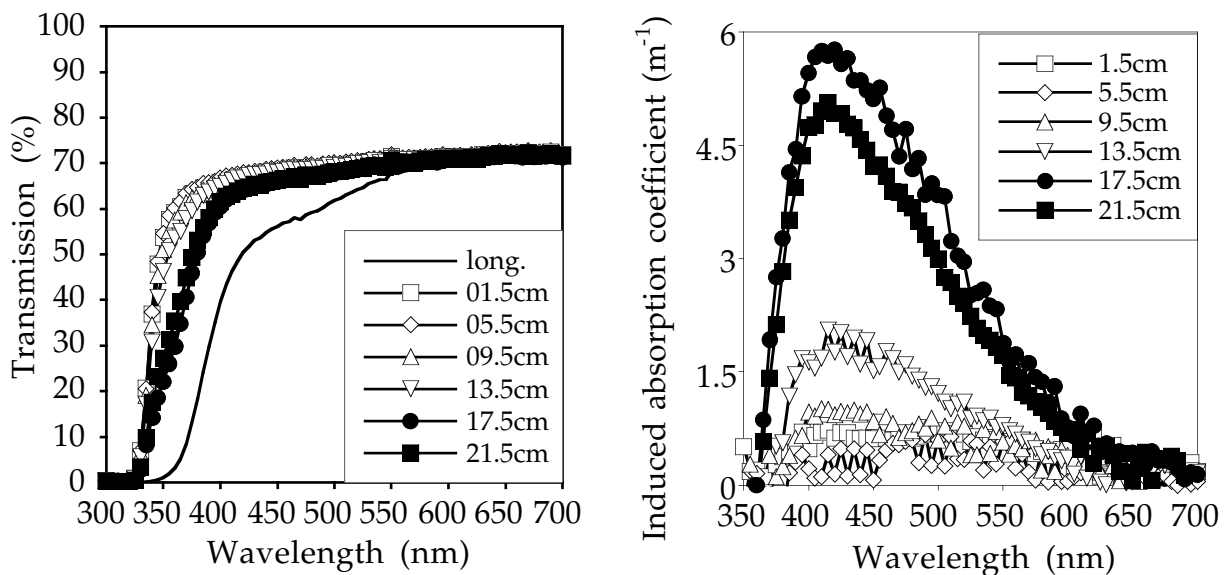


Fig. 1 : Longitudinal and transversal transmission and induced damage in a non uniform crystal

For these reasons, transmission thresholds are set on the longitudinal transmission at 3 wavelengths and a limit on the wavelength dispersion at 50% (absolute value) transversal transmission measured along the crystals is fixed. An additional condition will be discussed in section 5.

4.3- Light yield

The PWO crystal light yield is critical for the photostatistics contribution to the energy resolution (stochastic term) and for the signal over electronic noise ratio (noise term). The measured light yield of a crystal channel depends on the light emission spectrum and on the quantum efficiency and matching factor of the photodetector. The optimization of the crystals produced by the Bogoroditsk plant leads to an emission spectrum peaked at 420 nm. For such crystals, in several beam test campaigns, a value of ≈ 2 photoelectrons per MeV was measured at a temperature of 18°C, with one EG&G avalanche photodiode (APD) in a gate of 100ns.

In the final configuration of the calorimeter, the crystal section will be larger than the one referred above, and two APDs will be glued at the rear face of the crystals, resulting in an increased light signal of ≈ 3 photoelectrons per MeV, which will guarantee acceptable contributions to the energy resolution. This level corresponds to ≈ 8 photoelectrons per MeV measured with a Philips PM 2262B covering completely the back face with a 1.5 index of refraction silicon coupling grease (Rhodorsil), which is the level we request as minimum light yield for the preproduction.

The non-uniformity of light collection is not a specification as such. Only the roughness range of one side face will be indicated (see paragraph 3.3).

4.4- Decay time

Lead tungstate scintillation is intrinsically fast (15ns) but impurities or defects are often at the origin of slow components (in the 100ns to μ s range) or even afterglow (ms). The impact of such components on the pulse shape reconstruction and on pile-up is still under detailed studies, but it is generally agreed that if at least 90% of the light is emitted in less than 100ns the effect induced by slow components should be negligible. On the other hand, as afterglow is very often correlated with bad radiation hardness, we have therefore decided to limit the afterglow to less than 0.5% of the peak amplitude with a ^{60}Co counting rate of 1Mhz, as described in ref. [9].

5- Radiation hardness

5.1- How to predict radiation hardness from optical properties

Radiation hardness is a parameter for which systematic and direct measurements are not possible. It is therefore important to find correlated properties as already discussed in section 4.1.

In order to study the possibility of a reliable radiation hardness prediction of PbWO_4 crystals using the optical transmission characteristics, 63 crystals from 4 last R&D batches produced in the second half of 1997 have been investigated in terms of the transmission characteristics and of radiation hardness. This was done in order to evaluate new- or to verify

already existing-correlations between both measurements, and thus to define selection criteria for crystal acceptance.

In a first approach, a set of characteristic criteria for the longitudinal and lateral transmission has been defined:

Tlong (longitudinal transmission) at 350nm \geq 10%, Tlong at 420nm \geq 55%, Tlong at 600nm \geq 65%, and a wavelength dispersion for a transversal transmission at 50% below 6nm for 6 measurements, every 4 cm starting at 1.5cm from the front face.

This set of transmission criteria has been applied on the 63 crystals. These criteria allow to reject only bad quality crystals. But, some of the accepted crystals have an unacceptable light yield loss of more than 10% after low dose rate irradiation, thus requiring another rejection criterion.

Therefore, based on earlier experience with the slope characteristics of the transmission curves (classification on 3 types [10]), the slope of the band edge (defined on Fig. 2) has been plotted against the light yield loss after front irradiation with ^{60}Co source at a dose rate of 15rad/h (Fig.3).

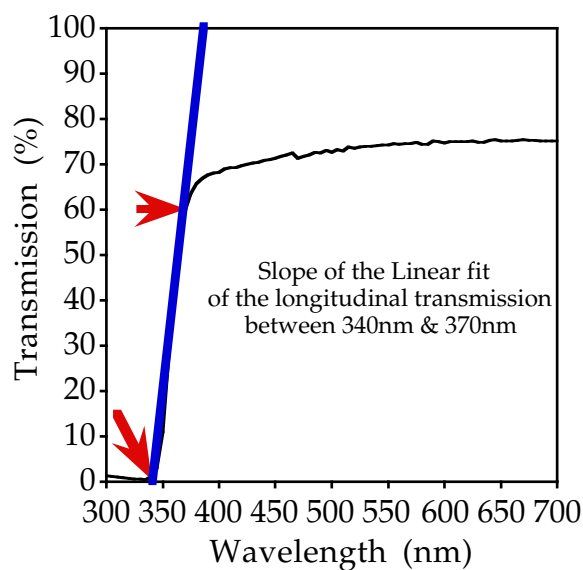


Fig. 2 : Definition of the slope of the band-edge

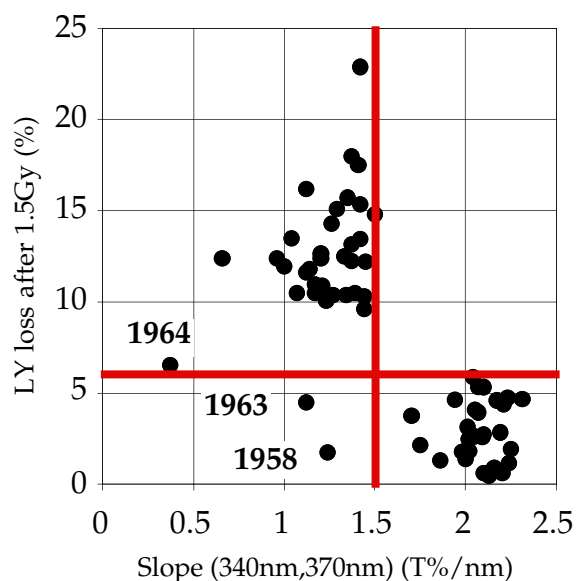


Fig. 3 : Correlation between Light yield loss and the slope

A good correlation between the two parameters is shown. Crystals with a slope of the band edge larger than 1.5%/nm have a light yield loss of less than 6% after low dose rate irradiation. Thus, the slope of the band edge can be considered as an additional rejection criterion and will be a specification .

5.2 - Radiation hardness specifications

It is first of all important to check that the total damage does not exceed a certain level when the crystal is fully saturated in all its volume. This is defined by a defect density above which more complex phenomena, like collective process and molecular defect stabilization can take place. This level corresponds to a defect density at the ppm level, which in the case of Lead Tungstate would represent $3 \cdot 10^{17}$ color centers per cm^3 . The corresponding induced absorption should therefore not exceed 1.5m^{-1} at the peak emission wavelength (420nm) when the crystal is irradiated from the side at a dose of at least 50krads and a dose rate of typically 10krad/h. These conditions are easily obtained at any ^{60}Co therapy unit like the one we are using at the Geneva Cantonal Hospital.

The second aspect is related to the light yield loss of the crystals when operating the calorimeter at LHC. Calculations about the LHC expected radiation levels show that the barrel will be exposed to a dose rate of typically 15 rad/h at high luminosity at the region of shower maximum in the crystal (at about 3 cm from the crystal entrance face) decreasing by a factor of 10 at the back of the crystal [11]. It is not easy to reproduce this dose profile for systematic measurements but it seems that a front ^{60}Co irradiation at the same dose rate gives a reasonable estimate of the damage. This point is important as it would allow us to test the crystals with an existing facility which can measure several crystals at the same time, as long as these crystals have been shown to have uniform properties. In order to confirm this point a set of 10 good quality crystals (very transparent, good light yield, fast decay, less than 10% light yield loss for front irradiation) have been irradiated with our TIS front irradiation set-up [12] with 15 rad/h of ^{60}Co . After annealing, the same crystals were irradiated from the side at the X5 setup using a ^{137}Cs source at the same dose rate of 15rad/h. In both cases the light yield loss was recorded when the crystal had reached its dynamical saturation level [13]. For the lateral irradiation case, a scan of longitudinal transmission measurements was made on the entrance face in order to control that the crystal was saturated through all its thickness. Results of the light yield losses in both cases, together with the band edge slope as defined in the previous section, are shown in Table 2.

The results show that there is a factor of at most 2 between the two conditions of irradiation. Considering that the lateral irradiation is a severe test as it produces a constant profile along the crystal which does not take into account the factor 10 attenuation predicted at LHC, it is safe to conclude that a limit of 6% light yield loss for the front irradiation setup would guarantee a loss at LHC of less than 10%, which is a value considered to be acceptable for the monitoring system. This limit of 6% for the front irradiation light yield loss has also the advantage of being easily predictable from the slope of the absorption band edge (see section 5.1).

It must be noticed in addition that the operating conditions of LHC with a sequence of fills and running periods will not induce continuous irradiation conditions to the crystals, allowing some

recovery during the fills. Under such conditions, it was shown that the residual fluctuations of the crystal light yield losses are a factor 5 to 10 smaller than the dynamical saturation level [14].

As several tests with neutron irradiations never showed any specific problems, it is not intended to specify radiation tolerance with neutrons.

Crystal #	Band edge Slope %/nm	% LY loss TIS (sat) front	% LY loss X5 (sat) lateral
2002	1.99	3.4	6
2004	1.3	9.5	18.6
2005	1.3	8.6	16.4
2036	1.4	7.2	13.8
2045	1.5	6.1	11.1
2020	2.17	2.7	4.6
2021	2.05	4.2	6.05
2022	2.02	1.6	2,4
1976	2.09	2.4	4.5
1894	1.39	8.2	14

Table 2: Light yield loss for front (TIS) and lateral (X5) irradiation at 15rad/h

6- Conclusions

The list of specifications for the barrel preproduction crystals in Bogoroditsk is the following:

Visual properties

- Presence of one label correctly positioned on face 1 (see 3.6)
- No visible cracks, chips or scratches, missing material, surface flaws
- No visible veil or core defect
- Transparent and colorless
- All faces polished but face 3 (left handed) or 4 (right handed) unpolished

Geometry (see definitions in section 3)

- All transversal and longitudinal dimensions within **+0mm, -0.1mm**
- Angular precision \leq **0.05mm**
- Planarity within **0.02mm** for all faces
- Chamfers between **0.3 and 0.7 mm**
- Polished faces with Roughness **Ra < 0.020 μ m**
- Face 3 or 4 unpolished with Roughness **Ra = 0.20 μ m**

Optical properties

- Longitudinal transmission (absolute values)
 - $\geq 10\%$ at 350nm
 - $\geq 55\%$ at 420nm
 - $\geq 65\%$ at 600nm
- Transversal transmission
 - For T=50% $\partial\lambda \leq 6$ nm, for 6 measurements every 4cm, the first one at 1.5cm from the front face
- Slope of the band edge S measured on longitudinal transmission between 340 and 370nm
 - S >1.5 %/nm
- Light yield ≥ 8 pe⁻ /MeV at 18°C
 - with a Phillips 2262B PM covering all the back face of the crystal
 - with a n=1.5 silicon coupling grease type Rhodorsil
 - in a gate of 100ns
 - measured at 8cm from front face
- Decay time
 - LY(100ns)/LY(1 μ s) > 90%
 - Afterglow $\leq 0.5\%$ of the peak amplitude with a ⁶⁰Co counting rate of 1Mhz

Radiation hardness

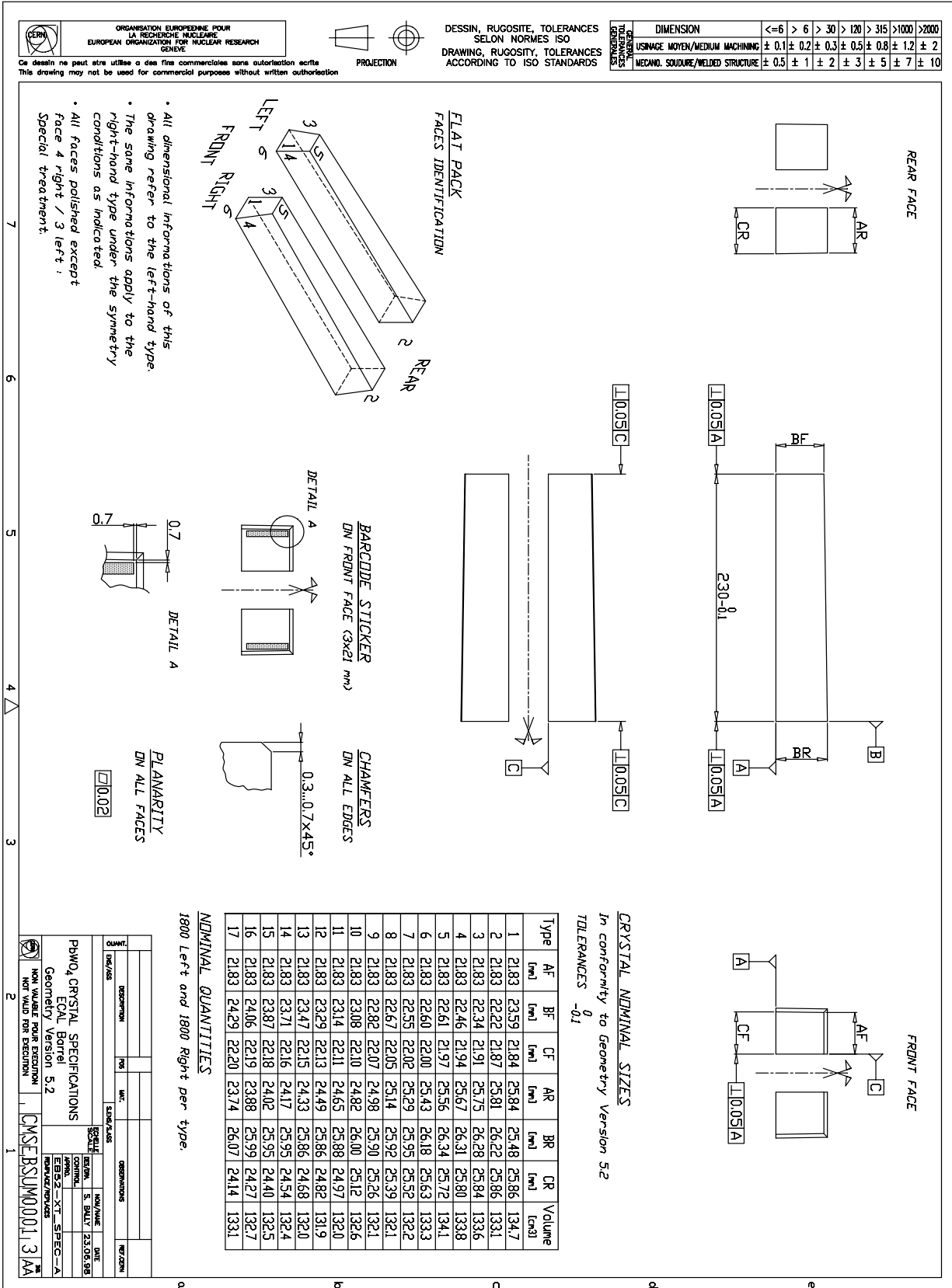
- Induced absorption for full saturation of the crystal
 - $\mu \leq 1.5$ m⁻¹ at 420nm
 - lateral ⁶⁰Co irradiation, > 50krad, > 10krad/h
- light yield loss < 6%
 - front ⁶⁰Co irradiation
 - 200rad, 15rad/h
- No recovery time constant shorter than 1 hour

Acknowledgments

The authors would like to thank their colleagues from the CMS-ECAL group for useful and constructive discussions. They are particularly grateful to J. Bourges and T. Otto from the group TIS/RP and J.P. Peigneux and A. Singovski from LAPP for invaluable help during irradiation studies.

References

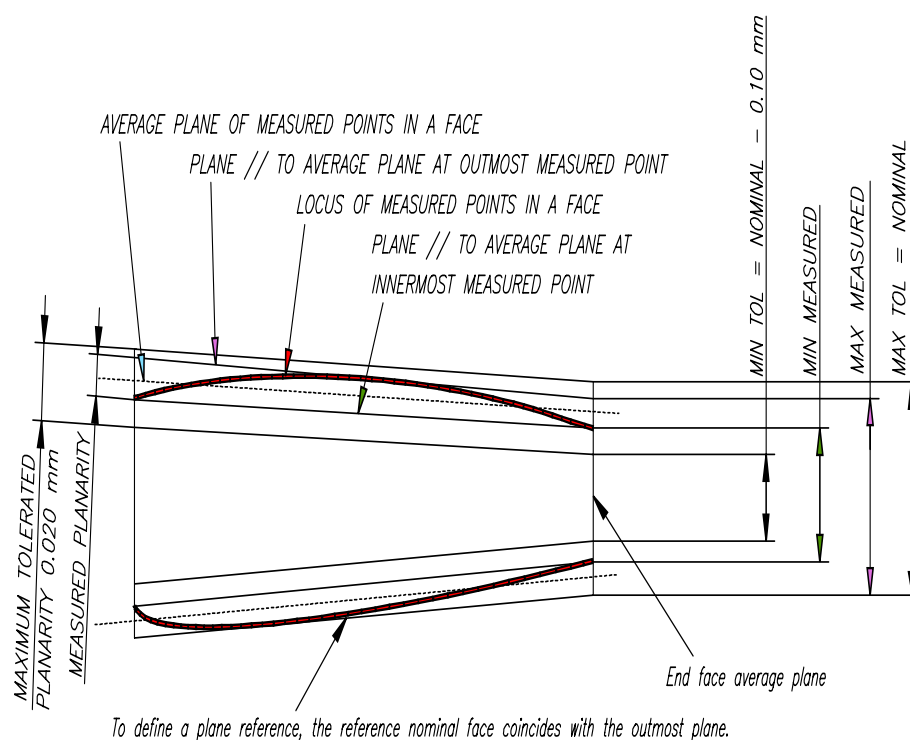
- [1] S. Bally et al. Geometry version 5.2 for the CMS ECAL Barrel. To be published as a CMS Note.
- [2] L.G.Denton. Flat-pack Crystal Geometry for use in the CMS Electromagnetic Barrel Section. CMS TN/95 - 208.
- [3] M. Lebeau. Protocol for the mechanical processing of the PbWO_4 crystals of the CMS ECAL Barrel. To be published as a CMS Note
- [4] . A. N. Annenkov et al. Cutting of five PbWO_4 crystals in industrial prototype conditions. CMS note 1997/29. March 1997.
- [5] A. De Forni et al. Cutting of five PbWO_4 crystals on the CERN prototype cutting machine. CMS note 1997/028. March 1997.
- [6] M. Lebeau. Principles of the cutting method proposed for the PbWO_4 crystals of the CMS Electromagnetic Calorimeter. CMS note 1997/024. March 1997.
- [7] A. N. Annenkov et al. Cutting of PbWO_4 crystals in industrial prototype conditions. Influence of the crystal lattice orientation on the cutting conditions and tooling. Scint'97 International Conference on Inorganic Scintillators, Shanghai, September 22-25, 1997.
- [8] J.-M. Le Goff et al. C. R. I. S. T. A. L./ Concurrent Repository & Information System for Tracking Assembly and production Lifecycles - A data capture and production management tool for the assembly and construction of the CMS ECAL detector. CMS NOTE/1996 - 003
- [9] M. Nikl et al, Slow components in the Photoluminescence and Scintillation Decays of PbWO_4 Single Crystals, Phys. stat. sol. (b) 195, 311 (1996)
- [10] E. Auffray et al, Scintillation characteristics and radiation hardness of PWO scintillators to be used at the CMS electromagnetic calorimeter at CERN, Proceedings of SCINT95, Delft, The Netherlands, August 95, p282
- [11] M. Huhtinen, ECAL Technical board minutes 09 March 98
See also ECAL TDR appendix A, CERN/LHCC 97-33, 15 Dec 97
- [12] C. D'Ambrosio et al, Low dose rate irradiation set-up for scintillating crystals, NIM A 388 (1997)
- [13] A.N. Annenkov et al, Systematic study of the short-term instability of PbWO_4 scintillator parameters under irradiation, Radiation Measurements Vol 29 N°1 p27-38, 1998, and CMS Note 1997-055
- [14] E. Auffray et al, Progress in the radiation hardness of PWO scintillators for the CMS calorimeter, Proceedings of the International conference on Inorganic Scintillators and their Applications, SCINT97, p 199, Shanghai, Sept. 97
See also ECAL TDR p 41



Annex 1

Annexe 3 – 19 June 1998

CRYSTAL SHAPE AND DIMENSION TOLERANCE CONVENTIONS



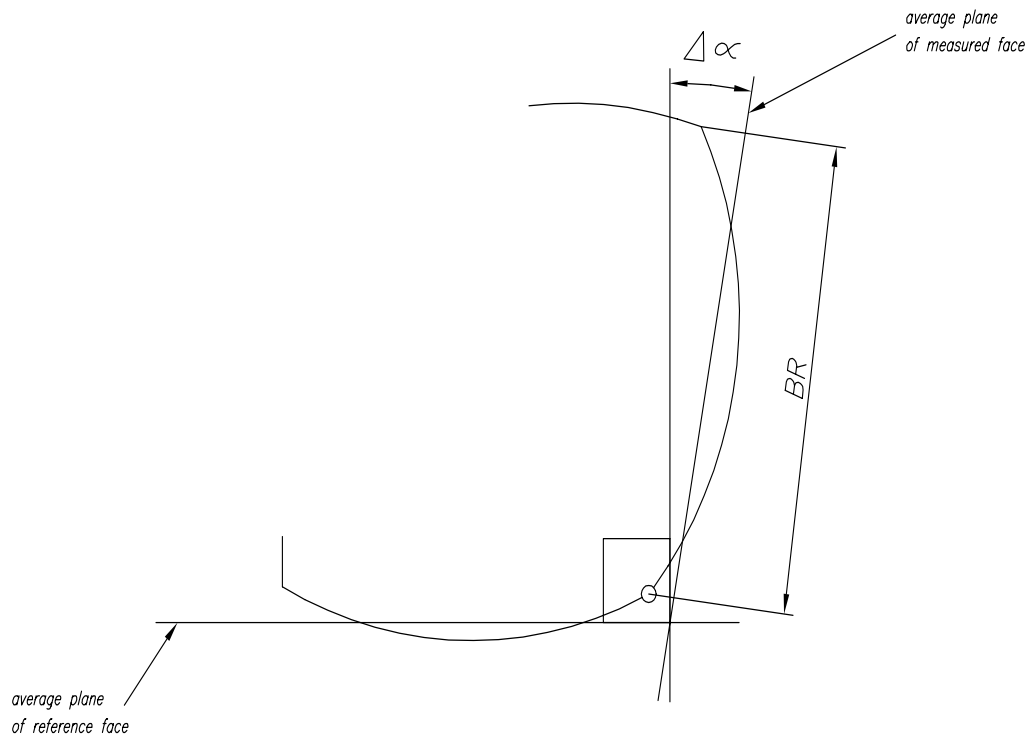
IN THE ILLUSTRATED CASE SHAPES AND DIMENSIONS ARE ACCEPTABLE:

- 1) THE PLANARITY IS WITH IN THE TOLERANCE GAP
 all measured points are contained between two planes parallel to the average plane of the points, (the inmost and outmost planes) the distance of which is smaller or equal to the planarity tolerance of 0.020 mm provided this condition is fulfilled, the planes are not necessarily parallel to the nominal plane of faces
- 2) THE DIMENSIONS ARE WITHIN THE TOLERANCE MARGINS
 the outmost planes intersect the end faces (average plane) to produce a dimension smaller than the maximal tolerance (nominal dimension)
 the inmost planes intersect the end faces (average plane) to produce a dimension larger than the minimal tolerance (nominal - 0.10 mm)

Annex 3

Annexe 4 - 19 june 1998

DEFINITION OF PERPENDICULARITY



Annex 4