Inspection QA Plan for PbWO₄ Crystal Tests in the EEEMCal

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Abstract

This ITP document will provide an overview on the PbWO4 (PWO) crystal properties that need to be verified as part of the overall EEEMCal QA plan. The procedures and hardware used to determine these properties will be outlined as well as the recording and reporting methods used.

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1. PWO CRYSTAL PROPERTIES

Lead Tungstate (PWO) crystals have been one of the default choices for homogenous electromagnetic calorimetry. The crystal's optical properties are susceptible to issues in the mechanical features. These include physical defects like scratches, chips, and others. Depending on their locations these can have a large impact on properties like transmission and light yield. In order to ensure that the crystals have good quality and uniformity we monitor several properties.

- Visual Quality: This is an indicator of the overall quality of the crystal. We have a rigorous visual
 inspection regiment that records and quantifies the number and severity of external and internal physical
 defects.
- Mechanical Dimensions: The crystals are cut and polished to a precise specification listed in the
 procurement documents. These dimensions have an impact on the stacking tolerances in the full detector
 assembly.
- Optical Transmission: Optical transmission is an essential quality of these crystals, both in terms of
 the ability of the crystal to produce scintillation light but also its ability to transport that light to our
 optical sensors.
- **Light Yield**: This is a physical property of the scintillator that is a measure of the number of photoelectrons that are produced by scintillation as a function of the energy of the incident particle that was the cause, usually reported in Pe/MeV for our applications. This value and the uniformity of it throughout the delivery of the articles is crucial for the overall uniformity of the detector.

To summarize, these lead tungstate crystals scintillators have several properties that are significant factors which have a direct impact on the overall quality and suitability of the material for use in a particle detector. These properties are what we will be measuring and tracking to ensure that the individual articles match with our required specifications. With these properties in agreement with our specifications, the crystals will provide accurate and consistent particle data.

2. PROCESSES AND PROCEDURES

2.1. Visual Quality

Upon initial receipt of the lot we conduct the individual inspection of each crystal with careful consideration given to the kinds of flaws we have experience in identifying as well as any new issues that may arise [3]. The original, individual manufacturer packaging is retained so as to allow for re-wrapping after the inspection is complete. This inspection is done prior to any other work being done on the crystals in order to establish a baseline for the individual crystal's features. The visual quality of the crystals is categorized by a very detailed physical inspection of their outer surface and mechanical features of the individual blocks as well as the internal features. This inspection is focused on looking for several kinds of defects that we have experienced while conducting business with a previous vendor which we no longer purchase from due to these issues. The list of physical defects we are experienced with include deep surface scratches, or large regions of high scratch counts, surface pitting, chips, and internal bubbles. All of these defects can have a negative effect on the optical properties on the crystals, both by directly decreasing the transmission and as an extension the light yield which is the case of the bubbles or by impacting the uniformity of the internal reflection [1]. In our experience this inspection process is effective at early identification of the kinds of defects that will have major impacts on the physics performance of the crystals thereby certifying if a crystal is suitable for use in the detector [1]; it also is essential for our record keeping process as it serves as our

initial ingestion step for the crystals at JLab. Details of the visual inspection procedure will be provided in section three.

2.1.1. In-Process Testing

Prior to shipment and throughout the manufacturing CRYTUR does a visual inspection of the crystals to ensure no aberrant articles are sent.

2.1.2. Incoming Inspections or Acceptance Testing

Upon completion of the visual inspection process of the entire lot (~110 crystals per) the lot will be initially accepted by the consortium designated person responsible for inspection at JLab, and further tests will be conducted on a subset (20 crystals per lot) to examine the other parameters mentioned in this document.

2.1.3. Verification Testing

During the wrapping stage in the detector assembly process, prior to the final assembly, the crystals will be visually inspected again to ensure no damaged articles are installed.

2.1.4. Failures and Non-Conformances

In the case that any of the tested PbWO4 crystals are outside of the range defined in the specification, the crystals will be sent back to the manufacturer and replaced. For nonconformances identified by JLab, BNL's concurrence on disposition shall be required.

2.2. Mechanical Dimensions

The mechanical dimensions are recorded for the subset of 20 crystals per lot after the visual inspection process has concluded, and the measurements are made in the same space as the visual inspection. The crystals are square prisms, with dimensions on the order of 2.05x2.05x20.00cm (sometimes these sizes will be reported in mm and will be noted as such when this occurs. This QA process of recording the crystals has two main impacts, one is to maintain a rigorous QC check to ensure that the articles delivered are within spec, while the second is to provide the engineers designing the EEEMCal with the necessary information as to the mechanical dimensions of the components. The measurements are made with digital height gauge against a granite slab table. The measurement is treated in this way in order to determine the height of the crystals over flat. This is the dimension of the crystal that is of interest as it is how they are stacked when in the detector. The total height stack is what is nominally the limiting factor, or at least the most likely issue that could arise in large matrix stacking. Both being undersized or oversized can be detrimental [3]. Oversizing can lead to misalignment or other physical hardware defects, while under sizing can lead to both mechanical issues but also problems in detector resolution as well. The details of the mechanical dimension measurement will be provided in section three.

2.2.1. In-Process Testing

The mechanical dimensions of the individual crystals are recorded by CRTUR during their manufacturing process after the final stage of polishing is concluded and prior to shipment. These numbers are provided to us via a digital spreadsheet, becoming part of our recording documentation, and are used as a crosscheck comparison to our own measurements [2].

2.2.2. Incoming Inspections or Acceptance Testing

These mechanical dimension measurements will be conducted after the primary visual inspections and initial acceptances have been completed for the respective articles.

2.2.3. Failures and Non-Conformances

In the case that any of the tested PbWO4 crystals are outside of the range defined in the specification, the crystals will be sent back to the manufacturer and replaced. For nonconformances identified by JLab, BNL's concurrence on disposition shall be required.

2.3. Optical Transmission

The optical transmission of the crystal material is a crucial component of the detector's overall performance, and it is directly tied to the light yield of the crystal. It has several influencing factors which can include surface quality as well as the chemical composition. 20 crystals from each lot will be selected to be subjected to the wavelength measurement procedure. We measure the optical transmission of light by direct injection of monochromatic light produced by a monochromator setup located in the ARC building at JLab in partnership with Carl Zorn and the Detector Support Group (DSG). The optical transmission is calculated via a simple formula.

$$\%T = \frac{Sample\ Current\ (nA)}{Reference\ Current\ (nA)}\ x\ 100$$

The sample and reference current are measured by a photomultiplier coupled to an integrating sphere which collects the light injected through the sample. Wavelengths between 300nm and 795nm are injected resulting in a transmission curve for the material. We then compare this to a typical transmission curve which has been established throughout our previous work on these crystals. The details of the transmission measurement will be provided in section three.

2.3.1. In-Process Testing

CRYTUR measures the transmission of the individual crystals prior to shipment and provides us with a spreadsheet containing the transmission each at three critical wavelengths, these being 360nm, 420nm, and 620nm. We use these measurements to compare to our own for cross calibration.

2.3.2. Incoming Inspections or Testing

After the visual inspection and dimensions measurements are taken 20 crystals will be transported in batches of ten to the ARC building where their transmission measurements will be measured and recorded. Following this they will be returned to their lot.

2.3.3. Failures and Non-Conformances

In the case that any of the tested PbWO4 crystals are outside of the range defined in the specification, the crystals will be sent back to the manufacturer and replaced. For nonconformances identified by JLab, BNL's concurrence on disposition shall be required.

2.4. Light Yield

The light yield of the crystals and the uniformity of this property throughout the delivery process is a key parameter we will monitor. This will be achieved by setting up multiple test stands at individual institutions to supplement the setup at JLab which has been utilized for the previous shipments delivered for other detectors. There will be a testing campaign initiated by sending a rotation of different test articles to each institution with which to calibrate their test stands, these test articles being rotated around each institution until the setup dependent discrepancies are understood. Once these differences are known then subsets of test articles will be distributed for testing.

2.4.1. In-Process Testing

CRYTUR measures the light yield of each crystal prior to shipment and they measure several different properties of it, this information is provided in the same spreadsheet that contains the mechanical dimensions they measure.

2.4.2. Transportation and Handling

Prior to final detector assembly, the crystals will always be transported within their manufacturer provided boxes for movement to and from the various test stands and the individual test setups at JLab. This will minimize the amount of time that any crystal is unprotected as we have found that this is the most significant control we have in the overall long-term safety of the crystals. After the detector is assembled sufficient safety measures will be put in place to ensure that the overall detector stack is safe for transportation.

3. EXPERIMENTAL/TEST SETUPS

3.1. Visual Inspection Method

The first component of the visual inspection process consists of a detailed examination of the individual crystals directly following their arrival from the shipping and receiving bay into the clean room. We perform the inspection looking for physical damage to the crystals or other internal issues that would impair their performance in the detector so as to remove any aberrant crystals before they get put into inventory. This inspection is performed with indirect light so as to catch the surface features in the reflection of the light sources, this makes them significantly more visible than other methods we have tried. It is typical to find a few small scratches which are not significant enough to have any impact on the physics properties, occasionally we encounter a crystal that is completely free of any external items. These results are recorded in a digital spreadsheet and include categories for chips and scratches as well as the quality of the chamfers on the corners of the crystals[1].





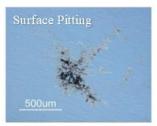




Figure 1. Examples of physical issues or damage that were encountered on crystals from a vendor we worked with previously.

The second part of the inspection is conducted with the aid of a green laser pointer (eye safe) which is used to shine light through the long axis of the crystal so as to reveal any internal bubbles, large bubbles are visible to the eye but the smaller distributions of bubbles we have found to be most easily identifiable with the laser.



Figure 2. A typical crystal image taken with the laser being shined along the long axis, not the small region of bubbles to the left of the image.

It is customary to find a small number of microscopic bubbles in each crystal but what we are looking for are large regions of high-density bubbles that would have an impact on the light transmission of the crystal. Previously other vendors we have purchased from were prone to having distinct layers of bubbles that occupied the entire volume of the crystal for sections. Each crystal is inspected with this laser and the resulting description of the bubbles are noted in the spreadsheet as well. There is also a picture taken of each crystal with the laser pointer illuminating the bubbles internally which is saved into the same online file system as the spreadsheet.

3.1.1. Resource Requirements

Our required resources include a green laser pointer and a magnifying desk lamp. Also, various cleaning supplies are used to remove any dust or other material from the crystal prior to inspection, these include isopropanol, chem wipes, and powder free nitrile gloves for handling.

3.1.2. Test Conditions

The testing conditions are clean space with good lighting for the initial inspection preferably with a movable light source. The second part requires very good low light conditions (pitch black) ideally so as to see the bubbles.

3.2. Mechanical Dimension Measurement Method

The measurements are made with a Mitutoyo QMH-14" B digital height gauge on a precision granite surface plate, the height gauge has a resolution of 0.0005mm. There is a 35µm layer of Tedlar® that we put down under the crystal to prevent scratches to the surface of the crystal during the process and we have zeroed the height gauge before each use with this material in place so that it doesn't impact the measurements, it is not compressible at these pressures and is replaced periodically as necessary due to wear. This allows us to accurately and quickly measure the dimensions of the crystals which we record in a google sheets spreadsheet which is backed up periodically.



Figure 3. The Mitutoyo QMH-14" B digital height gauge in the NPS Clean Room on the granite slab table.

We measure the crystal at 4 points sequentially along each side of the surface of the crystal to determine its dimensions. This starts at the end of the crystal that has the serial number on it and proceeds to the other end. We then rotate it 90° around the long axis and repeat the same measurements in the same pattern as the first four. Taking them in this way allows us to establish the height of the crystal along the entire surface relative to the flat surface we're zeroed to. These measurements are then put into a spreadsheet in the order 1-8 with 1-4 corresponding to the first side, side A in the figure, and 5-8 to side B.

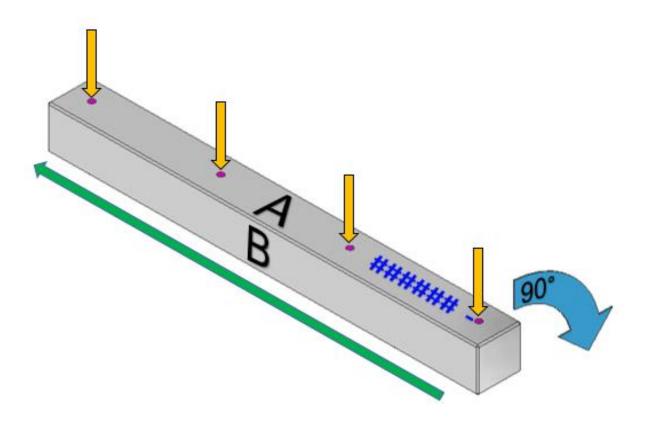


Figure 4. A diagram showing the measurement methods of the crystal to determine the mechanical dimensions. The magenta dots and the yellow arrows denote the individual measurement points where and in what direction the measurement occurred. The green arrow shows the direction of measurement progression and the blue arrow denotes the rotation. Blue #'s denotes the serial number location.

3.2.1. Test Conditions

Clear space around the measurement platform clear of obstructions and good lighting

3.2.2. Equipment

A Mitutoyo QMH-14" B digital height gauge and a stable granite slab table (quote surface finish quality), and gloves for handling of the crystals.

3.3. Optical Transmission Measurement Method

The optical transmission measurement is conducted in the ARC building in the DSG lab space on the second floor in room L125. In this lab space they maintain a custom experimental setup which is built around a monochromator coupled to a xenon arc lamp installed inside a dark box. The resulting light is then collimated and sent across a sample table which is mounted to a horizontal translation stage and into an integrating sphere coupled to the readout photomultiplier. There is also a mechanical filter wheel which blocks the aperture to the integrating sphere when the dark box is open. The monochromator and the translation stage are controlled via a computer using a custom LabView software program.



Figure 5. The setup in the DSG lab space in the ARC building showing the monochromator on the right with collimating lenses mounted to it, the light source is located behind the cloth baffle wall behind it. The translation stage in the middle of the frame supports the sample platform, and the integrating sphere with its filter wheel and photomultiplier are located on the left of the frame.

This sample platform allows us to measure up to 5 crystals in rapid sequence with the resulting data being saved into text files for later analysis. Fast analysis is available directly on the same computer that controls the setup as well and we typically use that for producing the transmission curves for the crystals during the measurement process.

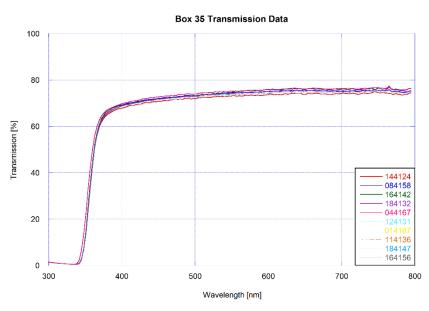


Figure 6. Typical set transmission curve Box 35 used as example all values passing.

The different curves are then evaluated and compared to the numbers that CRYTUR provides at the three wavelengths of interest and the results are input into the digital spreadsheet.

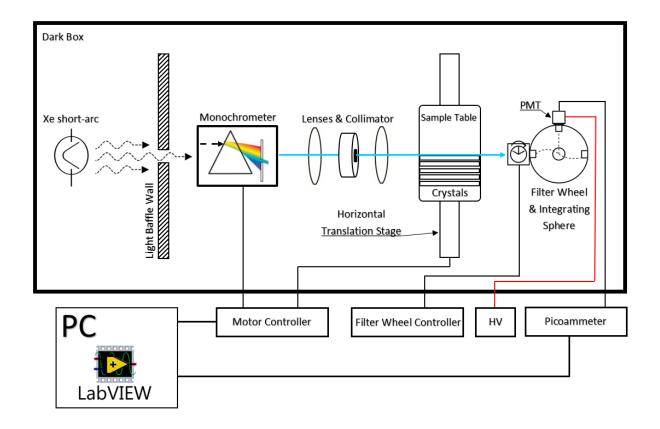


Figure 7. Schematic diagram of the transmission setup in ARC L125.

3.3.1. Test Conditions

This measurement is made in the ARC building in a custom dark box built and maintained by the DSG group and Carl Zorn which provides a dark, low noise environment.

3.3.2. Equipment

The hardware required for this measurement is a monochromator coupled to a Xenon lamp. The sample platform is mounted to a linear stage controlled by a stepper motor. There is also an integrating sphere coupled to a photomultiplier for light collection along with a filter wheel functioning as a shutter. All of this hardware is housed inside a large dark box which is pneumatically actuated. Gloves for handling of the crystals.

4. ENVIRONMENT, SAFETY & HEALTH CONSIDERATIONS

The procedures will be implemented in a way consistent with the environment, safety, and health policies of the relevant work areas. Within Jefferson Lab the process is described in the ES&H manual Chapter 3200, Work Planning and Control Program and at BNL within the SBMS: "Work Planning & Control for Experiments and Operations". For this work following best work practices, the following personal protection equipment and materials are used:

Powder Free Nitrile Gloves.

Also, for viewing of the bubbles within the crystals we use of a Class 2 laser of a green color so as to clearly see the bubbles while mitigating the risk involved.

5. RECORDS AND DOCUMENTATION

5.1. Manufacturer/Producer Records

For every item that is manufactured, the manufacturer will be responsible for maintaining records (travelers) of all raw materials that are used in the fabrication process, and document the processes and procedures that were used for production. The resultant documentation will be compiled into a report and will be provided to the project as part of the deliverable, which will be reviewed, validated, and then placed in the central data repository:

(https://docs.google.com/spreadsheets/d/1Z4A3S21vsW8xjCyKdAkVi-PlkrPyTHU7CfPtDaW4W74/edit?usp=sharing).

CRYTUR provides us with a digital copy of their own inspection and measurement records for each lot delivered upon notification of shipment. These measurements are compiled into the online spreadsheet to consolidate all the information into a central location. Each crystal has a unique serial; the project Google Sheet is the functional traveler-of-record. All entries (measurements, scans) are keyed to the serial numbers of the individual crystals.

Each box shipped also includes a physical inventory sheet that is scanned and uploaded to the same file location as the digital spreadsheet.

5.2. Deliverable Documentation and Records

We will provide the final copy of the spreadsheets for final documentation of the received shipments from CRYTUR, this serves as our traveler during the inspection process. It will include all the resulting measurements of crystal parameters that are measured.

We will also deliver a RIR (Receiving Inspection Report form) for each large delivery (~1000) PO from BNL of crystals received for record keeping purposes. This document will include the list of the articles received as well as statistical evaluation of the variables recorded. Controlled records including vendor COA, inventory scans, inspection data, and any NCRs will reside in the project's central Google Drive repository version history enabled.

6. REFERENCES

- Horn, T., Berdnikov, V. V., Ali, S., Asaturyan, A., Carmignotto, M., Crafts, J., Demarque, A., Ent, R., Hull, G., Ko, H.-S., Mostafavi, M., Muñoz-Camacho, C., Mkrtchyan, A., Mkrtchyan, H., Nguyen Trung, T., Pegg, I. L., Rindel, E., Somov, A., Tadevosyan, V., Trotta, R., Zhamkochyan, S., Wang, R., & Wood, S. A. (2020). Scintillating crystals for the Neutral Particle Spectrometer in Hall C at JLab. Amsterdam, Netherlands: Nuclear Instruments and Methods in Physics Research Section A: Accelerators, Spectrometers, Detectors and Associated Equipment. https://doi.org/10.1016/j.nima.2019.163375
- 2. CRYTUR (2024). PWO_DimensionProtocol_1072025 Turnov, Czech Republic CRYTUR, spol. s r.o. https://wiki.jlab.org/cuawiki/images/7/73/PWO_DimensionsProtocol_1072025.pdf
- 3. Crafts J., Horn T., (2025) *QA Testing of PbWO4 Crystals for EEEMCal* Newport News, VA, Thomas Jefferson National Accelerator Facility. https://indico.bnl.gov/event/29015/contributions/110514/attachments/63711/109353/TIC%20Meeting %20QA%20Testing%20of%20PbWO4%20Crystals%20for%20EEEMCal.pdf