# Specificities for DQW Crab Cavity Surface Treatment

| | | | |
|---|---|---|---|
| EDMS No. | | | |
| AUTHORS | | | DATE |
| Silvia Verdú-Andrés (BNL) | | | Dec 17, 2015 |
| REVISIONS | | | |
| *Rev. No.* | *Description of changes* | *Reviewer* | |
| 1.0 | First version | | |
| | | | |

### *Description*

This document provides the specifics for the Buffer Chemical Polishing (BCP) and the High Pressure Rinsing (HPR) of the SPS Double-Quarter Wave (DQW) crab cavity. It provides the guidelines to follow in the surface treatment of the cavity parts prior to Electron Beam Welding (EBW) and of a fully assembled cavity.[1]

### *Contents*


1. Cavity description
2. Processing and assembly plan
3. Chemical etching of parts prior to EBW
    3.1. Inspection of functional surfaces
    3.2. Surface machining to correct imperfections
    3.3. Protection of acid-sensitive regions
    3.4. Degreasing, rinsing and drying
    3.5. Etching, rinsing and drying
4. Surface treatment of fully assembled cavity
    4.1. BCP
    4.2. HPR
    4.3. Draining and drying
5. Safety
6. References




[1] The procedure is based on the BNL procedure "Specification for Superconducting RF Components", CAD 1244, on the CERN procedure "Superconducting RF cavities. Chemical polishing of niobium RRR> 300", EDMS 1388794 and on the BCP procedure of JLAB.

## 1. Cavity description

The SPS DQW crab cavity has two large ports terminated with DN100CF flanges (beam ports), four medium ports terminated with DN63CF flanges (three ports for the extraction of HOMs and one port for the FPC) and one small port terminated with a DN35CF flange (pickup port). Fig. 1 shows the port distribution for the SPS DQW crab cavity. Note that the port distribution is not symmetric.

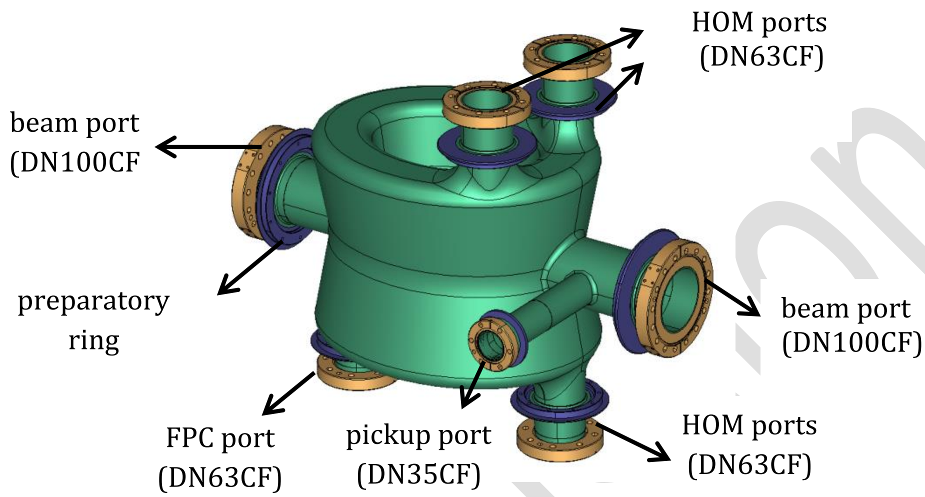


**Figure 1. Port distribution in SPS DQW crab cavity.**

The total cavity weight (including flanges and preparatory rings) is 55 kg. The cavity has an inner surface of approximately 0.737 $m^2$ for a total volume of about 0.022 $m^3$. From beam port to beam port, the cavity length is 659.6 mm (including flanges). The maximum cavity width is 410.5 mm (including pickup port) and the maximum cavity height (from FPC port to HOM port) is 495.71 mm, as shown in Fig. 2. All these dimensions correspond to the cavity at room temperature. Table 1 summarizes these numbers. For further information, refer to CERN technical drawing "LHCACFCA0001-vAB" in EDMS [1]. The step format file with the manufacturing cavity model is also in EDMS [2].

**Table 1. Main dimensions of cavity and weight.**

| CAVITY PARAMETER | | UNIT |
|---|---|---|
| Maximum length | 659.6 | mm |
| Maximum height | 495.71 | mm |
| Maximum width | 410.5 | mm |
| Inner surface | 0.737 | $m^2$ |
| Inner volume | 0.022 | $m^3$ |
| Weight (incl. flanges and prep rings) | 55 | kg |

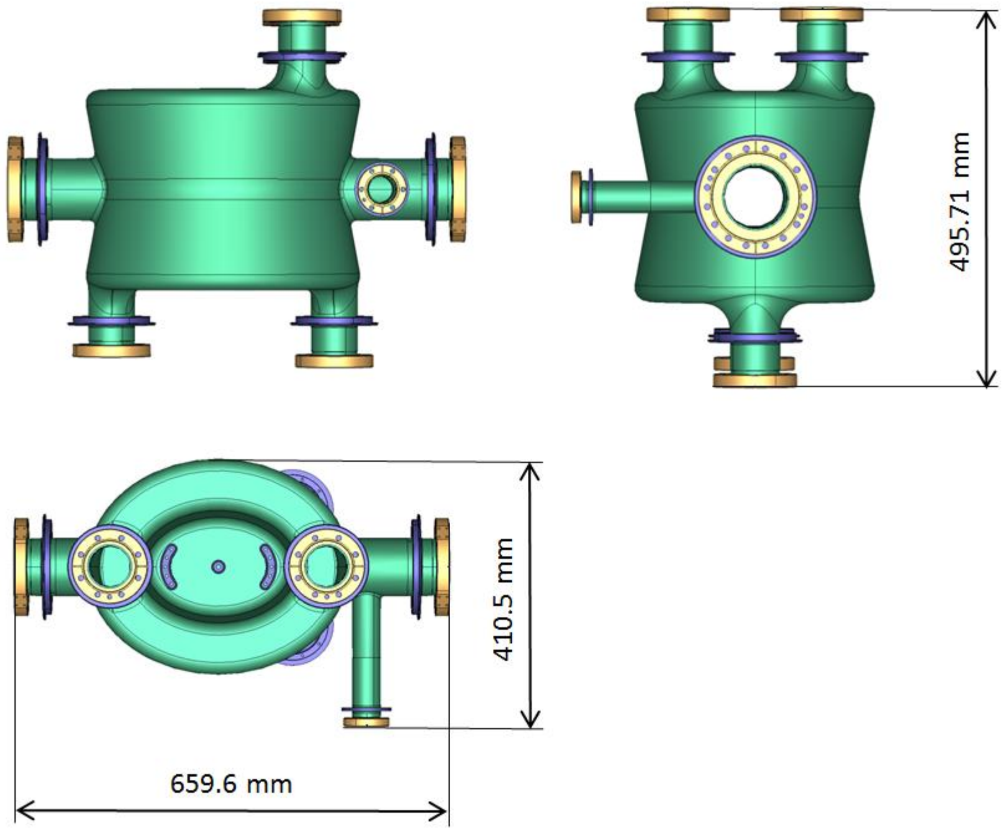


**Figure 2. Main dimensions of SPS DQW crab cavity.**

## 2. Processing and assembly plan

The main cavity preparation steps after bare cavity fabrication are listed in Table 2 [3]. Note that some steps (step #30-31, #41-43) listed in Ref [3] have been omitted in Table 2.

**Table 2. Main cavity preparation steps after bare cavity fabrication.**

| STEP # | STEP NAME |
|---|---|
| 10 | Incoming inspection |
| 11 | Chemical polishing |
| 12 | Tuner tab and stiffener welding (including warm tuning tabs) |
| 13 | EBW of subassemblies |
| 19 | Final trimming of subassemblies |
| 20 | Chemical polishing |
| 21 | Final EBW of the cavity |
| 27 | Tuning with frequency check |
| 28 | Dimensional control and pre BCP thickness measurement |
| 29 | Bead pull measurement |
| 30 | Optical inspection |
| 33 | Thickness measurement |
| 34 | ***Bulk chemical polishing*** |
| 35 | Visual examination |
| 36 | Thickness measurement |
| 37 | Resonator frequency check |
| 38 | Bead pull measurement with FPC (dummy) and/or HOM couplers |
| 39 | Heat treatment |
| 40 | ***Light chemical polishing*** |
| 41 | Thickness measurement |
| 45 | Visual examination |
| 46 | Manual HPR |
| 47 | High pressure water rinse |
| 48 | Cleanroom assembly |
| 49 | Evacuation and helium leak test |
| 50 | 120 C low temperature bake |
| 51 | RF acceptance tests at cold temperature (without and with HOM couplers) |

## 3. Chemical etching of parts prior to EBW (ID: #11 & #20)

All surfaces exposed later to ultra-high vacuum are functional surfaces that can critically influence RF operation.

Fig. 3 contains the process flowchart for the chemical etching of parts. The main steps are summarized below:

- All functional surfaces must be inspected for imperfections.
- The surfaces must receive, after shaping or after reworking (if any) and before welding (within 8 hours), a chemical polishing treatment by etching at least 20 μm on each side.
- The etching can be performed only on the welding surfaces or on the whole part, depending on the shape and size of the part.
- Acid-sensitive regions must be protected as described below.
- Before etching is performed, the surfaces to be etched must be degreased, rinsed and dried.
- After etching the surfaces must be rinsed and dried.
- If the time from cleaning to welding is more than 8 h an additional chemical polishing of 3 μm of the welding area shall be performed (only locally) immediately before welding.

Special guidelines for each step are provided in the following paragraphs:

**1. Inspection of functional surfaces**

- All functional surfaces must be inspected for imperfections before welding.
- All functional surfaces must be free of scratches and mechanical damage with a size larger than 15 μm, free of inclusions of foreign material, free of visible surface oxides, free of fingerprints, and free of silicone.
- If this requirement is not fulfilled, reworking can be carried out (see Section 3.2.), being restricted to areas of defects.
- Note that weld joint areas are particularly sensitive to inclusions.

**2. Surface machining to correct imperfections**

- Imperfections shall be removed by reworking and subsequent BCP treatments.
- Reworking must be avoided as much as possible and it must be restricted to areas of defects to produce a smooth surface.
- Imperfections shall be removed with a metallic grinding tool or an aluminum oxide wheel. Recommended grinding wheel material is an aluminum oxide abrasive embedded in fibrous material or equivalent and shall be approved by CERN&BNL representative.
- Cutting, grinding and abrasive tools shall be niobium dedicated and shall not be used on other materials.
- Grinding of defects at the “inner” surface shall produce a smooth surface and a smooth transition to the untreated surrounding area.
- During rework operations surface temperatures on niobium surfaces shall not exceed 150°C.
- No deep scratches shall be produced at the ground area.

## 2.1. Protection

- Whenever applicable, surface/regions that must not be exposed to etching shall be protected. For instance, brazed joints of flanges and cavity tubes shall be protected during chemical polishing of the cavity. Different methods can be applied for this purpose. The CERN&BNL representative should approve the method used.

## 2.2. Degreasing, rinsing and drying

- In preparation for welding, the parts shall be degreased in a detergent under ultrasonic agitation for a minimum of 30 minutes. (Time shall be taken as reference value as it is function of the geometry, type and amount of contamination.) The use of other product shall be subjected to CERN&BNL representative for approval.
- Ultrasonic agitation shall be done at least with 5 W/l at 40 to 80 kHz.
- Subsequently, rinsing with de-ionized (DI), filtered (0.2 micron particle size) water jet precedes all etching operations.
- Drying of components shall be carried out in laminar airflow in a clean room of class 100 or better.
- The parts shall not be touched or supported in places close to an electron beam weld joint or an RF surface to avoid contamination by drying stains.
- Appropriate clean gloves shall be worn when handling the parts.

## 2.3. Etching, rinsing and drying

- Acid mixture: HF (49% conc.), $HNO_3$ (69.5% conc.) and $H_3PO_4$ (85% conc.) in a volume ratio of 1:1:1.
- [For etching of the whole part] The parts must be completely immersed in the acid solution and slightly shaken inside the bath to avoid the formation of bubbles.
- The solution bath shall not exceed a temperature of 15°C. At this temperature the removal rate in fresh acid is approximately 1μm/minute for niobium and ~ 50% higher for Nb55Ti, but removal rate also depends on agitation of the parts.
- Acid baths for Nb parts and Nb55Ti parts shall be separated.
- The acid mixtures shall be replaced when the niobium content reaches 15 g/l and the removal rate is roughly reduced to half of its value for fresh acids.
- Before EB welding all component surfaces shall have a layer of 20 μm removed (removal rate and thickness refers to a single surface only).
- The parts shall then be immersed in de-ionized (DI), filtered (0.2 micron particle size) water within 10 seconds to avoid overheating of the surfaces during reaction with the acid films on the surfaces. High Pressure Rinsing (HPR) shall be performed with de-ionized (DI), filtered (0.2 micron particle size) water multiple times until the effluent exiting the component has a resistance > 17.5 MΩ. Inlet pressure of the rinsing system shall be between 1000 psi and 1200 psi.
- DI water specifications: The demineralized water shall be according to type II of ASTM D1193-99e1 "Standard Specification for Reagent Water".
- The drying operation shall be performed in laminar flow of a clean room class 1000 (ISO 6 according to ISO 14644-1).

- If the etching does not occur in a clean room, the pieces must be transported in a demineralized water bath to the clean room and then dried.
- EBW shall be executed within 8 hours after BCP treatment in order to prevent contamination. If this is not possible, an additional “refreshing” of the surface by the removal of 3 μm shall be done prior to welding.

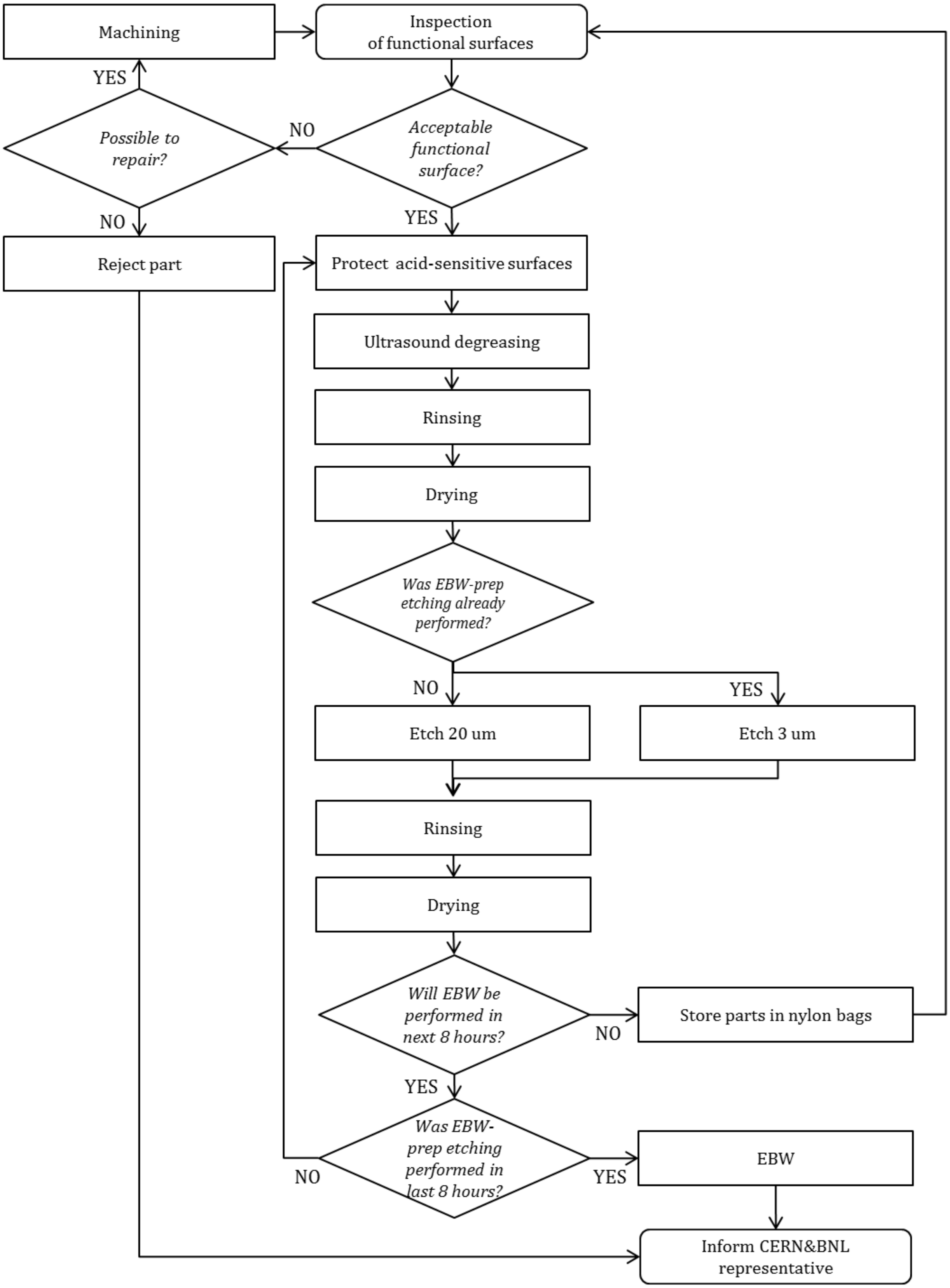


**Figure 3. Diagram flow for chemical etching of parts prior to EBW.**

# 4. Surface treatment of fully assembled cavity

The DQW crab cavity surface will be polished using the Buffer Chemical Polishing (BCP) method. The surface treatment is completed with an ultrasonic bath, a High Pressure Rinsing (HPR) and drying in a clean environment.

## 4.1. Bulk and light BCP

The guidelines for both bulk and light BCP of the DQW crab cavity are:

- Description of BCP device:
  - The BCP device is located at ISO Class 4 clean room. Any personnel performing the BCP procedure should follow the gowning protocol of clean room.
  - The BCP device can take two configurations (see Fig. 4):
    a) Horizontal configuration which allows continuous cavity rotation during BPC treatment.
    b) Vertical configuration for enhanced draining of acid after BCP treatment.
  - The BCP device allows continuous acid flow for cavity etching and continuous $N_2$ flow for evacuation of fumes resulting from BCP treatment.

- Acid specifications:
  - Acid mixture: HF (49% conc.), $HNO_3$ (69.5% conc.) and $H_3PO_4$ (85% conc.) in a volume ratio of 1:1:1.
  - Acid temperature: below 15$^{o}$C. Higher temperatures increase hydrogen absorption and also lead to a rougher surface.

- Acid circuit (see Fig. 5):
  - Continuous acid flow (about 1-2 GPM) while the cavity is constantly rotating.
  - Cavity rotation speed is such that the sheer speed is about ~1 cm/s at the cavity surface.
  - Cavity rotation direction does not change during one BCP iteration.
  - Acid must cover at least half of the cavity volume.
  - Beam ports will be used as inlet and outlet ports for the acid circuit.
  - As soon as the acid temperature stabilizes below 15°C after filling, the flow speed of acid should be reduced to minimum values for stable operation and homogeneous material removal.

- Acid draining: to be performed following steps described in Section 5 in order to avoid acid stagnation.

- DI water specifications: The demineralized water shall be according to type II of ASTM D1193-99e1 “Standard Specification for Reagent Water”.

- Hardware assembly:
  - Location of O-rings (or gaskets) should be designed to prevent the acid contact with braze joints and flange surfaces. For example, leaving a lip on all niobium ports that sticks out 0.5 mm inches and provides a good positive seal against the Viton. If that lip is not there it can also be machined into the mating plastic piece.

    - Beam tube flanges should be parallel to the floor during hardware installation to ensure proper O-ring centering.
    - Use a crisscrossing pattern when tightening the bolts/nuts with socket wrench/stationary wrench combo.
    - Tighten a little each time to avoid an uneven clamping effect on the flange surface. Do NOT over tighten, as this can lead to drastic bending in the flange itself OR stripping of the bolt head.

- BCP for the DQW crab cavity:
    - Cavity setup is the same for bulk and light BCP.
    - The frequency sensitivity due to BCP is -170 kHz for 210 mm thickness removal according to CST simulations.
    - ANSYS simulations… (Thomas Jones.)
        - Most-etched region expected in …
        - Least-etched region expected in …
    - <u>Bulk BCP</u> (ID: #34): The total thickness removal during bulk BCP treatment for the DQW crab cavity should be around 150 µm average. The maximum and minimum thickness removal values for the bulk BCP treatment are given in Table 3. The bulk BCP treatment will be performed in 4 iterations. Inlet and outlet ports will be interchanged after every iteration. It is recommended to remove about 50 µm in the first 2 iterations (25 mm in each iteration) and the remaining 100 µm in the later 2 iterations. The first 2 iterations should serve to understand the etch rate according to thickness gage measurements and expected etch rate.
    - <u>Light BCP</u> (ID: #40) The total thickness removal for each light BCP treatment for the DQW crab cavity should be around 30 µm average. The maximum and minimum thickness removal values for the light BCP treatment are given in Table 4. The light BCP treatment will be performed in 2 iterations. Inlet and outlet ports will be interchanged after every iteration. About 15 µm should be removed in each iteration.

- Instrumentation:
    - About 4 cavity regions are found as critical during thickness removal. These regions are the two capacitive plates (so-called cavity domes) and the two inductive plates (so-called cavity rings). Ideally a minimum of one thickness gauge should be placed on the outside of the surface to actively monitor the thickness removal during BCP. If only one thickness gauge is available, it should be placed on one of the cavity central plates to monitor the thickness removal during the buffer chemical polishing procedure. The gauge position is chosen based on the fact that the cavity frequency is especially sensitive to the distance between the two cavity central plates. The preferred location of the thickness gauge is shown in Fig. 6.
    - The temperature of the cavity surface can increase significantly due to the action of the acids used in BCP. Install thermocouples at different positions in the cavity to monitor the temperature during the BCP treatment.
    - Use witness sample???????

- Monitor resistivity of water during cavity rinsing performed after draining acid???? It was included in general specification document created for both RFD and DQW.

Steps for one BCP treatment:

- Degrease cavity in ultrasonic bath prior to assembly.
- Install High-Density Polyethylene (HDPE) cap joint with Teflon Encapsulated Viton O-ring to both DN100 port flanges. Install HDPE blind flanges to DN63 and DN35 port flanges. Use stainless steel screws, washers, and silicon bronze nuts.
- Install thickness gages on cavity surface. See Fig. 6 for recommended gage location.
- Mount cavity in BCP device.
- Leak test with DI water. If leaks are present, tighten nuts and/or fixtures as needed.
- Place BCP device in vertical configuration for water draining.
- Dry cavity overnight.
- Set BCP device in horizontal configuration.
- Record amount of material to be removed in $\mu$m.
- Record thickness gages reading prior to BCP treatment.
- Turn on water curtain for temperature control of cavity during BCP.
- Start the BCP treatment and record start time. See Table 5 for other quantities that should be recorded during BCP treatment.
- Monitor etch rate during the etching process and thickness gage controller while BCP is performed. The operator must stop the process when predetermined amount of material has been removed.
- Stop the BCP treatment and record end time. Calculate actual BCP treatment time in minutes.
- Turn off water curtain.
- Record ultrasonic thickness gage reading after BCP treatment.
- Place BCP device in vertical configuration for acid draining.
- Rinse cavity with DI water repeatedly (about 4 iterations) immediately right after acid is drained from the cavity.
- Drain DI water from cavity. If possible, spin the cavity with some tilting for better evacuation of liquid.
- Dismount cavity from BCP device. Disconnect hoses and chemistry hardware from cavity.
- Submerge the cavity in an ultrasonic degreasing bath right after it has been rinsed (procedure described in following Section "Degreasing and HPR"). Salts formed by the action of the acid on niobium can solidify on the cavity interior surface and make a crust, which can be difficult to remove with HPR. Therefore, the cavity must go to the ultrasonic bath with detergent immediately after being drained and rinsed.

Fig. 7 summarizes the steps for BCP in a flow diagram.

Equipment and ancillary:

- Ultrasonic bath cleaner.
- BCP device with water curtain, allowing vertical and horizontal configurations.
- Cavity frame.
- Adaptors to attach cavity frame to the BCP device.
- Acid hoses, $N_2$ hoses.
- 2 Viton O-rings for DN100 ports, 4 Viton disks for DN63 ports and 1 Viton disk for DN35 port.

- 4 HDPE blind flanges for DN63CF flange ports and 1 for DN35CF flange port.
- 2 HDPE cap joints for DN100CF flange ports.
- 4 ultrasound thickness gages.
- Several thermocouples.
- SS bolts, SiBr nuts, washers.

Note: All equipment and ancillary must be approved for use in clean room class 100 environment.

**Table 3. Specification for bulk BCP: thickness removal in different cavity regions.**

| | |
|---|---|
| *Thickness removal limits for bulk BPC* – 150 µm (average) | |
| Most etched region | Max. 180 µm |
| Least etched region | Min. 120 µm |
| No. of iterations to complete a 150 µm average thickness removal | 4 |

**Table 5. Specification for bulk BCP: thickness removal in different cavity regions.**

| | |
|---|---|
| *Thickness removal limits for light BPC* – 30 µm (average) | |
| Most etched region | Max. 50 µm |
| Least etched region | Min. 15 µm |
| No. of iterations to complete 30 µm average thickness removal | 2 |

**Table 4. Data record for BCP treatment.**

| | |
|---|---|
| Cavity ID | |
| Cavity surface area (m$^2$) | |
| Cavity volume (L) | |
| Niobium density (g/L) | |

| BCP iteration ID | | #1 | #2 | #3 | #4 | #5 | #6 |
|---|---|---|---|---|---|---|---|
| Date | | | | | | | |
| Inlet port ID | | | | | | | |
| Outlet port ID | | | | | | | |
| Acid volume (L) | | | | | | | |
| Niobium volume in acid (%) | | | | | | | |
| Temperature ($^{o}$C) | | | | | | | |
| Acid etch rate (µm/min) | | | | | | | |
| Acid fill duration (min) | | | | | | | |
| Acid process duration (min) | | | | | | | |
| Acid dump duration (min) | | | | | | | |
| Drain duration (min) | | | | | | | |
| Total etch duration (min) | | | | | | | |
| Rinse duration (min) | | | | | | | |
| Thickness removed (µm) | Expected | | | | | | |
| | Gage #1 | | | | | | |
| | Gage #2 | | | | | | |
| | Gage #3 | | | | | | |
| | Gage #4 | | | | | | |
| Cumulative thickness removal (µm) | | | | | | | |

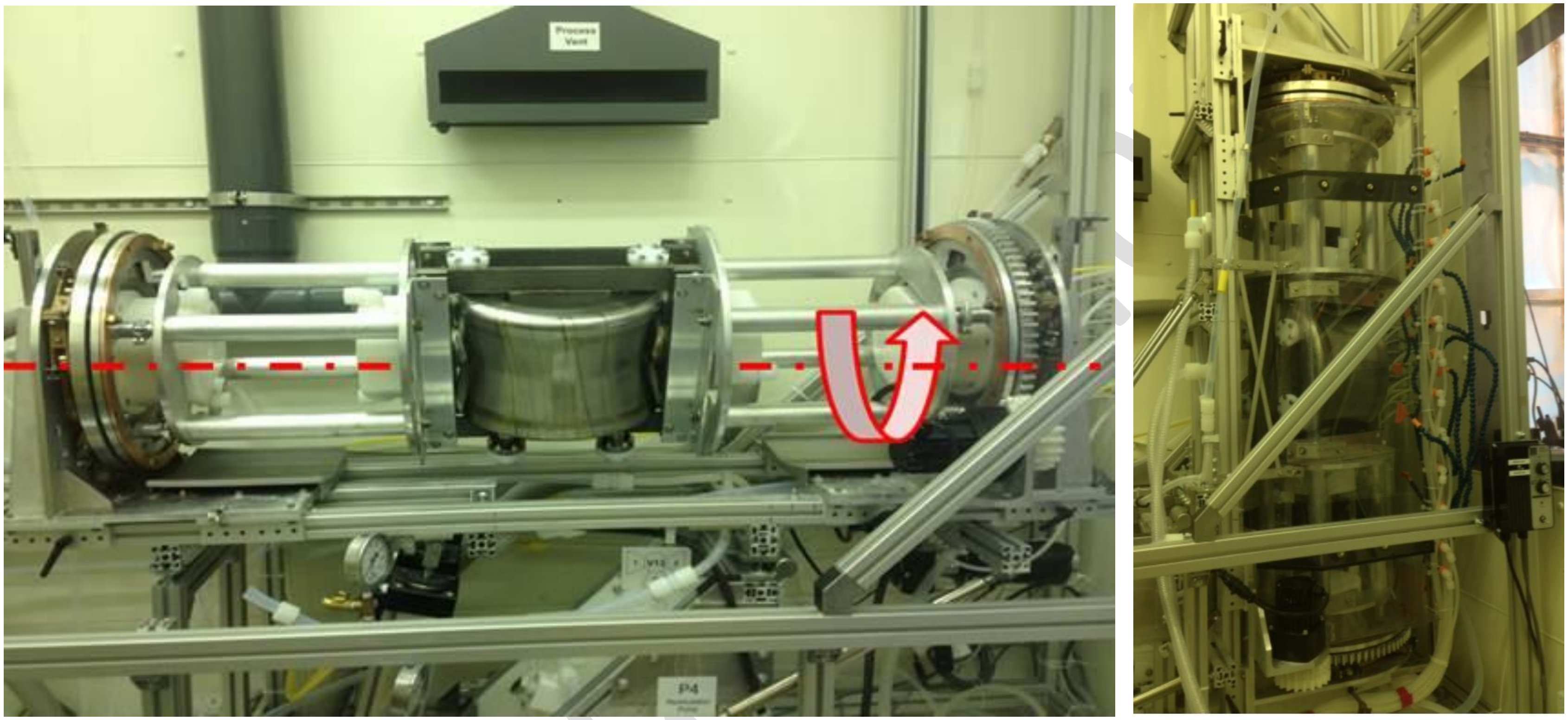

**Figure 4. [Left] Horizontal configuration for BCP device. [Right] Vertical configuration for BCP device.**

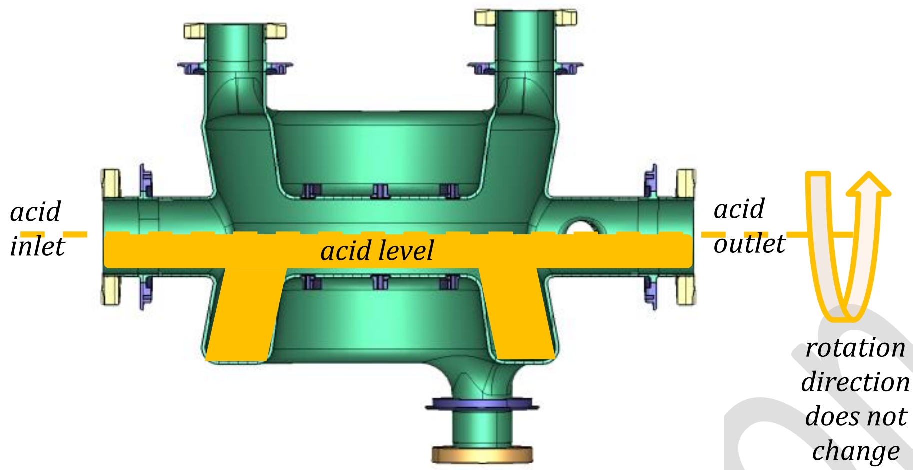


**Figure 5. Sketch of the acid circuit desired for a DQW crab cavity. One of the beam ports is used as the inlet port and the other beam port is used as the outlet port. The acid volume is such that the acid covers at least half of the cavity volume. The acid flow is continuous and the cavity is constantly rotating during the BCP treatment.**

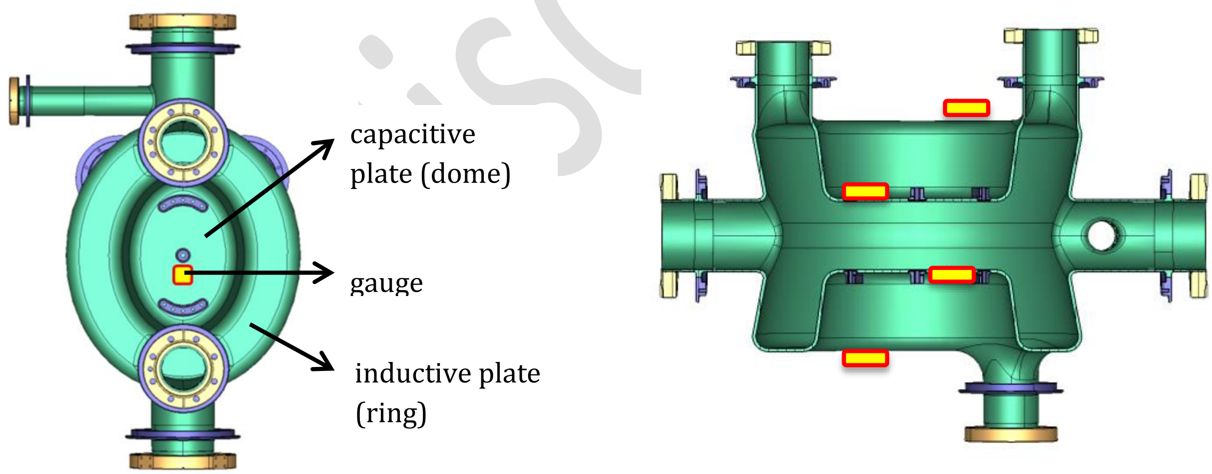


**Figure 6. [Left]If only one thickness gauge is available, the preferred location for the gauge is on one of the cavity central plates (position marked by yellow square). [Right] Preferred location for the thickness gauges when at least four gauges are available: one gauge should be placed on each of the inductive plates and on each of the capacitive plates.**

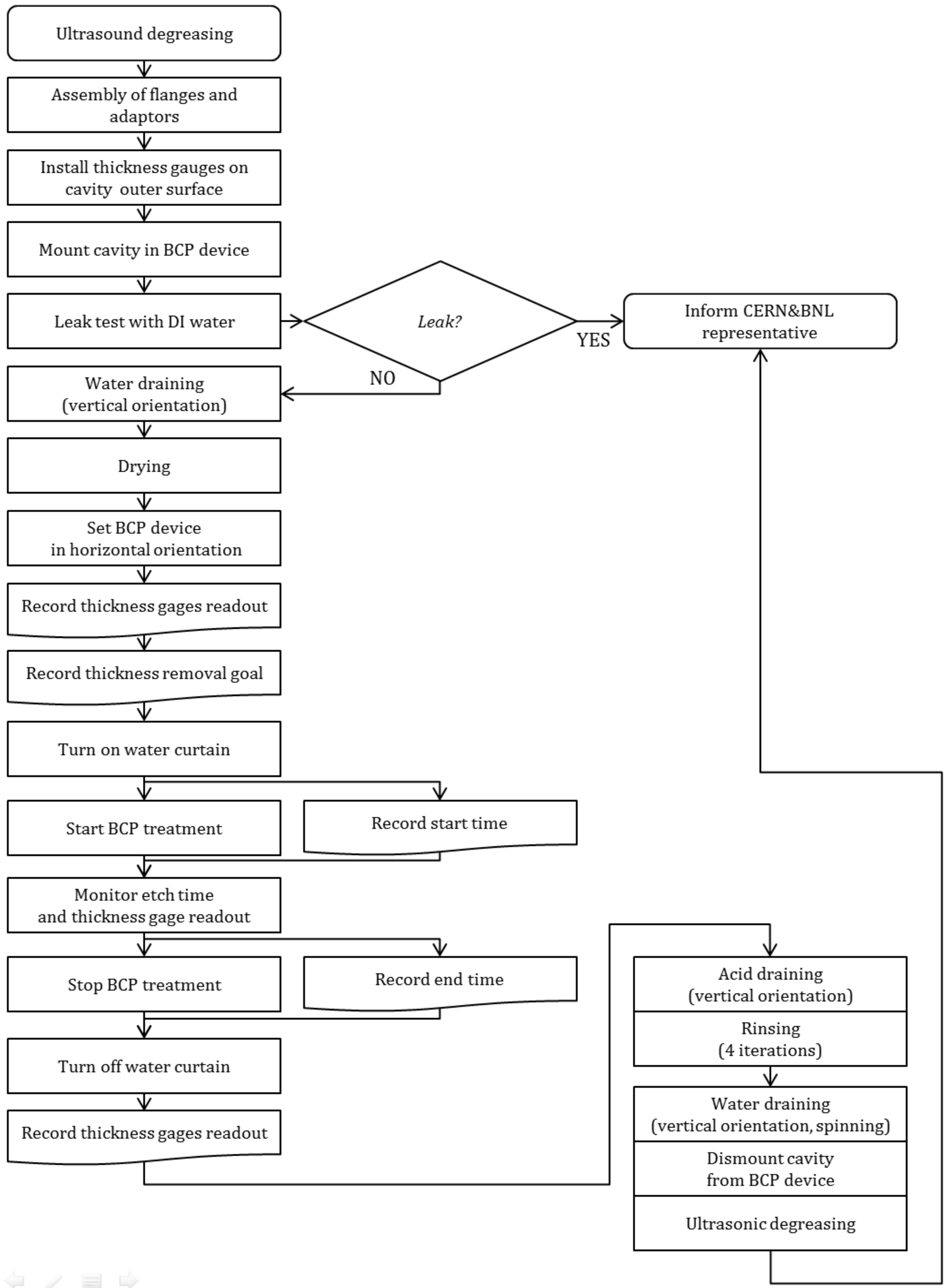


**Figure 7. Flow chart for bulk and light BCP treatment.**

## 4.2. Degreasing and HPR (ID: #47)

The HPR device is located at ISO Class 4 clean room. Any personnel performing the BCP procedure should follow the gowning protocol of clean room.

The main steps for HPR on the DQW crab cavity are:

- Immerse the cavity in detergent solution at about 50°C for 1 hour.
- Rinse the cavity in a bath of water. Let the cavity stay in the bath until the HPR system is ready.
- Install the cavity in the HPR device, being the cavity in vertical configuration as shown in Fig. 8.
- Perform fast HPR (2 inches per minute advance with 1200 psi deionized water at ambient temperature) accessing from one of the beam ports and going all the way through to the top of the cavity.
- Flip cavity and perform fast HPR from the other beam port.
- Turn cavity and perform fast HPR from small ports.
- Flip cavity and perform fast HPR from the other small ports.
- Turn cavity and perform slow HPR (1 inch per minute) through the beam port with the pick-up tube, going up and down for at least three times.
- Cover the opening of beam and pickup ports to avoid dust going into clean cavity. The cover should not block the exit of water from the ports. It should just act as an “umbrella”.
- Flip the cavity over to get the cavity rinsed through the other beam port.
- Perform slow HPR through the other beam port, going up and down for at least three times.
- Cover the opening of the beam port. The cover should not block the exit of water from the port.
- Turn the cavity so that it stays in horizontal position.
- Perform slow HPR through both HOM ports.
- Cover the opening of the ports. The cover should not block the exit of water from the port.
- Flip the cavity over to get the cavity rinsed through the FPC and HOM ports.
- Cover the openings of the ports. The cover should not block the exit of water from the ports.
- Perform slow HPR through FPC and HOM ports.
- Drain cavity as indicated in the following Section “Draining and drying”.

Equipment and ancillary:

- Ultrasonic bath cleaner.
- HPR device.
- Cleanroom lift with rotating arm.
- Cavity frame.
- Adaptors to attach cavity frame to the lift.
- 2 blind DN100CF flanges, 4 blind DN63CF flanges and 1 blind DN35CF flange.
- OFE copper gaskets for DN100, DN63 and DN35 ports.
- SS bolts, SiBr nuts, washers.

Note: All equipment and ancillary must be approved for use in clean room class 100 environment.

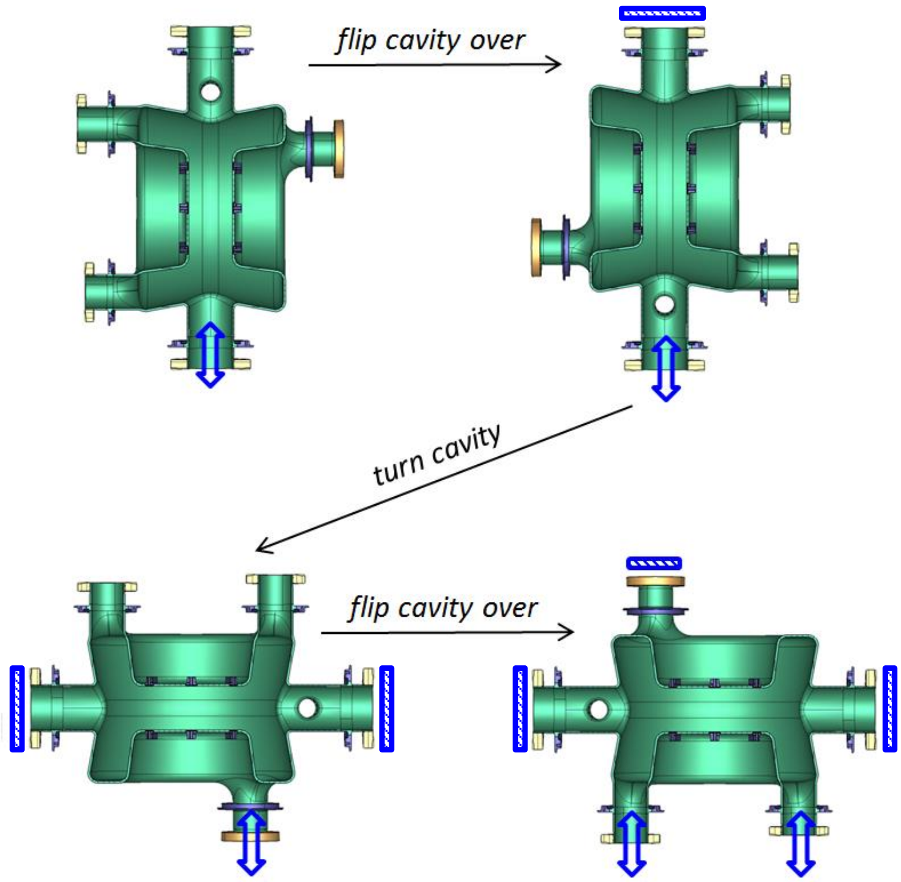


**Figure 8. Steps for SPS DQW crab cavity HPR. Note that cover flanges are not preventing water from coming out of the ports.**

## 4.3. Draining and drying

The draining and drying procedures should occur in an ISO Class 4 clean room. Any personnel performing the BCP procedure should follow the gowning protocol of clean room.

- The angle α is not zero (α =10.6 degrees); therefore, the cavity should be rotated and drained through beam pipe and FPC ports with an angle larger than θ to avoid stagnation in the cavity dome.
- Once the main cavity body is dry, the cavity should be turned over the pickup tube to let the pickup tube dry up. Fig. 8 shows both configurations.
- The cavity ports should then immediately sealed with blanked flanges and their corresponding copper gaskets.

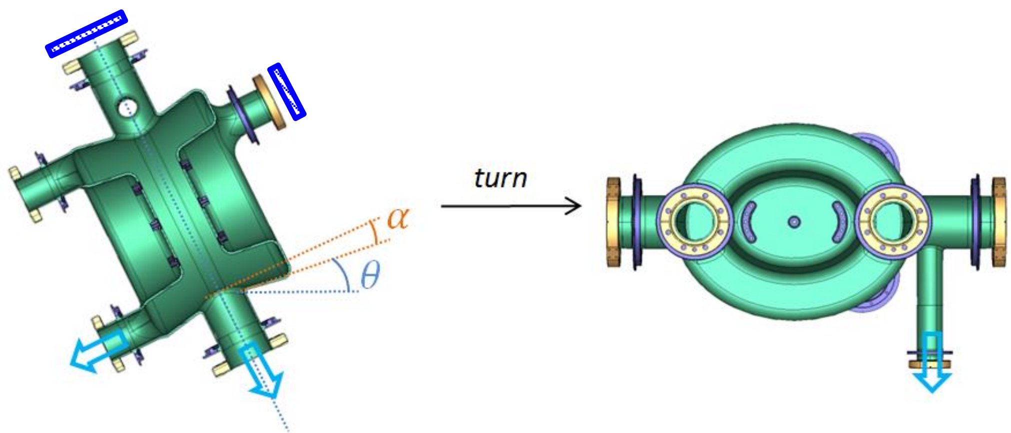


**Figure 8. Draining and drying configurations.**

# 4. Safety

- OSHA Applicable Regulations.

# 5. References

[1] DQW Crab Cavity CERN technical drawing "LHCACFCA0001-vAB" in EDMS: https://edms.cern.ch/document/1400295/AB.

[2] Step format file with the manufacturing cavity model in EDMS: https://edms.cern.ch/document/1396447/2.

[3] Annex 6.3 manufacturing process (version November 2015).