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# OPERATIONAL QUALIFICATION

# PROTOCOL CUM REPORT

# FOR

# NITROGEN GAS GENERATION AND DISTRIBUTION SYSTEM

# CAPACITY: 10 $Nm^3$/HR

| | |
|---|---|
| **EQUIPMENT ID. No.** | |
| **LOCATION** | **UTILITY BLOCK** |
| **DATE OF QUALIFICATION** | |
| **SUPERSEDES PROTOCOL No.** | **NIL** |

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## 1.0 PROTOCOL PRE – APPROVAL:

**INITIATED BY:**

| DESIGNATION | NAME | SIGNATURE | DATE |
|---|---|---|---|
| OFFICER/EXECUTIVE (QUALITY ASSURANCE) | | | |

**REVIEWED BY:**

| DESIGNATION | NAME | SIGNATURE | DATE |
|---|---|---|---|
| HEAD (PRODUCTION) | | | |
| HEAD (ENGINEERING) | | | |

**APPROVED BY:**

| DESIGNATION | NAME | SIGNATURE | DATE |
|---|---|---|---|
| HEAD (QUALITY ASSURANCE) | | | |

## 2.0 OBJECTIVE:

- To verify that the equipment operates in accordance with the design and user requirements as defined by set Acceptance Criteria and complies with relevant cGMP Requirements.
- To verify the Operational features of Nitrogen gas generation & distribution system and to ensure that it produces desired Quality & rated output according to manufactures specifications.
- To verify all the Operational features from user point of view of the Equipment, Cleaning Procedure, Start up & Shut down Procedure and Safety Features.

## 3.0 SCOPE:

- The scope of this operational qualification protocol cum report is limited to qualification of **Nitrogen gas system (Make – Mass Gas air Systems Pvt. Ltd.)** to be installed in utility block at …….
- This Protocol will define the methods and documentation used to perform OQ activity the Nitrogen gas system for OQ. Successful completion of this Protocol will verify that Nitrogen gas generation & distribution system meets all acceptance criteria and ready for Performance Qualification.

## 4.0 RESPONSIBILITY:

The Validation Group, comprising of a representative from each of the following departments, shall be responsible for the overall compliance of this Protocol cum Report:

| DEPARTMENTS | RESPONSIBILITIES |
|---|---|
| **Quality Assurance** | • Initiation, Authorization, Approval and Compilation of the Operational Qualification Protocol cum Report.<br>• Co-ordination with Production and Engineering to carryout Operational Qualification.<br>• Monitoring of Operational Qualification Activity. |
| **Production** | • Review & Pre Approval of Protocol cum Report.<br>• To Co-ordinate and support for Execution of Qualification study as per Protocol.<br>• Post Approval of Operational Qualification Protocol after Execution. |
| **Engineering** | • Review & Pre Approval of Protocol cum Report.<br>• Co-ordination, Execution and technical support in Nitrogen gas system Operational Qualification Activity.<br>• Calibration of Process Instruments.<br>• Responsible for Trouble Shooting (if occurs during execution).<br>• Post Approval of Operational Qualification Protocol after Execution |

## 5.0 EQUIPMENT DETAILS:

| | |
|---|---|
| **Equipment Name** | **NITROGEN GAS GENERATION & DISTRIBUTION SYSTEM** |
| **Equipment ID No.** | |
| **Model** | **PSA Based model** |
| **Manufacturer's Name** | **MASS GASAIR SYSTEMS PVT. LTD.** |
| **Supplier's Name** | **MASS GASAIR SYSTEMS PVT. LTD.** |
| **Capacity** | **10$Nm^3$/hr.** |
| **Outlet Pressure** | **5.5 $Kg/cm^2$** |
| **Place of Installation** | **UTILITY BLOCK** |

**6.0 SYSTEM DESCRIPTION:**

| | | |
|---|---|---|
| **Type** | **:** | **PSA BASED NITROGEN PLANT** |
| **Capacity** | **:** | **10 $Nm^3$/hr.** |
| **Purity** | **:** | **99.5%** |
| **Outlet Pressure** | **:** | **5.5 $Kg/cm^2$** |
| **Dew Point** | **:** | **(-) 40 °C** |

- PSA (Pressure Swing Adsorption) Based Nitrogen Plant is to produce Nitrogen gas form Atmospheric compressed air. Air passes through Carbon Molecular Sieves (CMS) at a certain pressure, the moisture, Oxygen and $CO_2$ are selectively adsorbed, and balance nitrogen comes out and collects in the receiver.
- Compressed air first collects in air receiver at 7.0 $kg/cm^2$ pressure and then goes to PSA module through air filter module. The air receiver has been providing to avoid air pressure fluctuation so that a constant flow & pressure will available during plant operation. One high efficient air filter has been provided at the outlet of air receiver to arrest dust particles from nitrogen gas before enter in PSA module.
- This is a specially designed composite bed type PSA module having two towers filled with special grade of Activated Alumina and second generation of high efficient Carbon Molecular Sieves (CMS) to produce 99.5% pure Nitrogen. As compressed air passed through PSA module, moisture from compressed air is adsorbed in Alumina Bed and oxygen & carbon dioxide are selectively adsorbed in CMS bed, balance Nitrogen collected in surge vessel at the outlet of PSA Module.
- Surge Vessel is a vertical, cylindrical type vertical pressure vessel. Surge vessel has been provided to collect outlet nitrogen before send to storage tank / user point.
- One Oxygen analyzer connected with this vessel to measure oxygen impurity in the product nitrogen. Nitrogen from surge vessel now goes to user point through flow meter and backpressure control valve at required flow and pressure. To avoid impure high oxygen content in nitrogen on 3-way vent valve has been provided with an interlock of oxygen analyzer. In case oxygen content high as purity limit nitrogen will vent out in the atmosphere till purity comes with in desired limit.

## 7.0 PRE – QUALIFICATION REQUIREMENTS:

### 7.1 Verification of Documents:

- DQ Protocol Cum Report
- IQ Protocol cum Report
- Draft SOP for Operation & Cleaning of Nitrogen gas system
- Draft SOP for Preventive Maintenance of Nitrogen gas system
- Technical specification of equipment

### 7.1.1 Procedure:

- Verify the above mentioned documents for availability, completeness and approval status
- If any deviation is observed the same has to be recorded giving reasons for deviation and approved. Deviation should be approved by Authorized person.
- Approved Drawings and supporting documents would form a part of the OQ Protocol cum report.

### 7.1.2 Acceptance Criteria:

All the documents should be available, complete and approved by respective authorities.

## 8.0 CRITICAL VARIABLES TO BE MET:

### 8.1 Verification of documents:

The results of any tests should meet the limits and acceptance criteria specified in the test documents.

Any deviations or issues should be rectified and documented prior to OQ commencing.

| S. No. | Document Name | Document/SOP No. | Completed (Yes/No) | Checked By (Engineering) Sign/Date | Verified By QA Officer/Exe. Sign/Date |
|---|---|---|---|---|---|
| 1. | Executed and approved Design Qualification document | | | | |
| 2. | Executed and approved Installation Qualification document | | | | |
| 3. | SOP for operation & Cleaning of Nitrogen gas distribution & generation system | | | | |
| 4. | Draft SOP for Preventive Maintenance of Nitrogen gas distribution & generation system | | | | |

**Checked By**
**(Production)**
**Sign/Date:** ……

**Verified By**
**(Quality Assurance)**
**Sign/Date:** ……

**Inference:**

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**Reviewed By**
**(Manager QA)**
**Sign/Date:** ……

**8.2 Test Equipment Calibration:**

Verify that all critical parts associated with the equipment will be in a calibrated state. Review the calibration status for the test equipment to be utilised and record the calibration due dates in the table below. All Equipment/Instrumentation must remain within the calibration due date for the duration of OQ test for which the item is used. If a due date potentially occurs during the testing period then the instrument must be recalibrated before it can be utilised.

| Equipment/ Instruments Name | Equipment/ Instrument ID | Calibration On | Due On | Observed By Sign / Date |
|---|---|---|---|---|
| | | | | |
| | | | | |
| | | | | |
| | | | | |
| | | | | |
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**Checked By**
**(Production)**
**Sign/Date:** ……

**Verified By**
**(Quality Assurance)**
**Sign/Date:** ……

**Inference:**

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**Reviewed By**
**(Manager QA)**
**Sign/Date:** ……

**8.3 Operational and Functional Checks:**

Operate the Nitrogen gas generation & distribution system as per Manufacturer's Manual/SOP and Check for the following functions of the Equipment. The Equipment should function as desired.

| Parameter | Acceptance Criteria | Observation | Observed By (Engineering) (Sign/Date) |
|---|---|---|---|
| Check that all electrical are ok | Control panel is getting power supply. | | |
| Ensure that there is no leakage in Cylinder valve | No leakage should observed | | |
| Check the zeroing all the pressure gauge | all the pressure gauge should be at 0 | | |
| Check all the drain valves of equipments are closed. | Drain valve should be closed | | |
| Ensure the pressure achieve up to min. 7.0 Kg/cm²g in air receiver. | Pressure should be up to min. 7.0 Kg/cm²g in air receiver. | | |
| Minimum pressure in the absorbing towers goes to 6.8Kg/cm²g. | Minimum pressure in the absorbing towers should be 6.8Kg/cm²g. | | |

**Checked By**
**(Production)**
**Sign/Date:** ……

**Verified By**
**(Quality Assurance)**
**Sign/Date:** ……

**Inference:**

……

……

……

**Reviewed By**
**(Manager QA)**
**Sign/Date:** ……

## 8.4 START-UP & SHUT DOWN PROCEDURE

- The area near the unit should be kept clean.
- Check that all electrical are ok. Control panel is getting power supply.
- Check the zeroing all instruments.
- Ensure that drain values of all equipments are closed.
- Open the inlet air valve of air receiver.
- Ensure the pressure achieve upto min. 7.0 Kg/cm²g in air receiver.
- Switch 'ON' the main switch & control supply switch.
- Observe the operation of the PSA Module for some time and see that all
- The valves are working properly. Check the minimum pressure in the absorbing towers goes to 6.8 Kg/cm²g. Make sure that air pressure inside the towers should be never less than 6.8 Kg/cm²g.. Check the operation of solenoid and change over valves.
- When 5.0 Kg/cm²g pressure archive in the surge vessel the open the out let valve of nitrogen plant.
- Make sure that no any flange joint or socket is leaking.

## 8.5 NORMAL SHUT DOWN PROCEDURE: -

- Close outlet valve of nitrogen plant.
- Close inlet valve of air receiver.
- Switched off the PSA system.. Switched off the control supply.
- Don't disturb any other valve and oxygen analyzer setting. Close

**8.6 Power Failure Verification:**

| Item | Acceptance Criteria | Observation | Observed By (Engineering) Sign/Date |
|---|---|---|---|
| Main Power shut down | Equipment stops in safe and secure condition | | |
| Main Power Restored | Equipment can be restarted with no problems or adverse conditions. | | |

**Checked By**
**(Production)**
**Sign/Date:** ……

**Verified By**
**(Quality Assurance)**
**Sign/Date:** ……

**Inference:**

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**Reviewed By**
**(Manager QA)**
**Sign/Date:** ……..…

## 9.0 REFERENCES:

**The Principle Reference is the following:**

- Validation Master Plan.
- Schedule-M – "Good Manufacturing Practices and Requirements of Premises, Plant and Equipment for Pharmaceutical Products."
- WHO Essential Drugs and Medicines Policy, QA of Pharmaceuticals, Vol-2 – Good Manufacturing Practices and Inspection.

## 10.0 DOCUMENTS TO BE ATTACHED:

- Operation And Maintenance Manual
- Copy of Draft SOPs
- Any Other Relevant Documents

## 11.0 DEVIATION FROM PREDEFINED SPECIFICATION IF, ANY:

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## 12.0 CHANGE CONTROL, IF ANY:

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## 13.0 REVIEW (INCLUSIVE OF FOLLOW UP ACTION, IF ANY ):

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## 14.0 CONCLUSION:

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## 15.0 RECOMMENDATION:

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## 16.0 ABBREVIATIONS:

| | | |
|---|---|---|
| No. | : | Number |
| WHO | : | World Health Organization |
| QA | : | Quality Assurance |
| PVT. | : | Private |
| Ltd. | : | Limited |
| ID | : | Identification |
| No. | : | Number |
| UB | : | Utility Block |
| PSA | : | Pressure Swing Adsorption |
| CMS | : | Carbon molecular sieve |
| Kg | : | Kilo gram |
| °C | : | Degree centigrade |
| Mg | : | milligram |
| $m^3$ | : | meter cube |
| OQ | : | Operational qualification |

## 17.0 POST APPROVAL:

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| OFFICER/EXECUTIVE (QUALITY ASSURANCE) | | | |

**REVIEWED BY:**

| DESIGNATION | NAME | SIGNATURE | DATE |
|---|---|---|---|
| HEAD (PRODUCTION) | | | |
| HEAD (ENGINEERING) | | | |

**APPROVED BY:**

| DESIGNATION | NAME | SIGNATURE | DATE |
|---|---|---|---|
| HEAD (QUALITY ASSURANCE) | | | |