PHARMA DEVILS

QUALITY ASSURANCE DEPARTMENT

| STANDARD OPERATING PROCEDURE | |
|---|---|
| **Department:** Quality Assurance | **SOP No.:** |
| **Title:** Preparation of Validation/Qualification Protocol and Report | **Effective Date:** |
| **Supersedes:** Nil | **Review Date:** |
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## 1.0 OBJECTIVE:

To lay down a Procedure for Preparation of Validation/Qualification Protocols and Reports.

## 2.0 SCOPE:

This SOP is applicable for Preparation of Validation/Qualification Protocols and Reports at …….

## 3.0 RESPONSIBILITY:

**QA (Officer/Designee):** Preparation, Distribution (to Respective Department), Revision, Retrieval and Destruction of this SOP. Review, Approval, Training and effective implementation of this SOP. Review of Protocols and Reports.

**Respective Departments:** Preparation and Review of Protocols and Reports.

## 4.0 ACCOUNTABILITY:

**Head QA:** Ensure Training and Implementation of this SOP. Custodian of soft copy and hard copy of Master copy of this SOP. To ensure distribution to concerned Departments, Training and Effective Implementation of this SOP. Approved of all Qualification Protocol & Report.Custodian of soft copy and hard copy of Validation/Qualification Protocols and Reports.

**Respective Departments Heads:** Ensure the Review of contents of Protocols and Reports. Approval of all Qualification Protocol & Report.

## 5.0 DEFINITIONS

**5.1 Protocol:** A Protocol is a written set of instructions broader in scope than a Standard Operating Procedure (SOP).

**5.2 System:** Referred in this SOP means Facility/Equipment/Instrument or Utility.

**5.3 Activity:** Any monitoring or measuring or Qualification step.

**5.4 Validation/Qualification:** Documented act of proving that any Facility/Utility/Equipment/ Instrument/System actually lead to expected results.

**5.5 URS (User Requirement Specification):** Document indicating User Requirements and Specifications of any Equipment/Utility/System, submitted to Vendor(s) to ensure that the Equipment/Utility/System meet the intended purpose.

**5.6 Design Qualification (DQ):** Documented verification that the proposed design of the Facilities, Systems and Equipment is suitable for intended purpose.

**5.7 Installation Qualification (IQ):** Documented verification that the Facilities, Systems and Equipment as installed or modified, comply with the approved design and the Manufacturer's recommendation.

**5.8 Operational Qualification (OQ):** Documented verification that the Facilities, Systems and Equipment as installed or modified, perform as intended throughout the anticipated Operating Range and is operating consistently as per the established specification or design criteria.

**5.9 Performance Qualification (PQ):** Documented verification that the Facilities, Systems and Equipment as connected together, can perform effectively and reproducibly, to perform approved process and deliver product specification consistently.

**5.10 Re-Qualification:** Planned, periodic Qualification to prove that any Facility/Utility/Equipment/ Instrument/System actually are in validated state.

**5.11Modification:** Any significant change which may alter the validated state of Facility/Utility/ Equipment/Instrument/System.

**5.12Relocation:** Change in location of any Utility, Equipment or System.

## 6.0 PROCEDURE:

## 6.1 GENERAL GUIDELINES FOR PREPARATION, APPROVAL OF QUALIFICATION AND VALIDATION PROTOCOL AND REPORTS:

**6.1.1** Protocols and Reports shall be written in English Language by using Microsoft Word.

**6.1.2** The person performing the Process or activity of respective department/QA Department shall prepare the Protocols and Reports.

**6.1.3** Initiator shall have adequate knowledge, Training and experience in the related Areas of activity.

**6.1.4** All the points in the Protocol / Report shall be numbered sequentially and sub paragraph of the Protocol / Report be also numbered sequentially with an incremental number derived from the heading number. Bullets may be use for sub paragraph of Protocol / Report.

**6.1.5** Initiator shall check the completeness of draft Protocols and Reports and send to the Head of the Department for review.

**6.1.6** Protocols and Reports shall have reference of related document such as Pharmacopoeia and Guidelines published by various Regulatory Authorities. Wherever necessary illustrations and drawing shall be indicated to provide better clarity and understanding of the Process / System.

**6.1.7** The reviewer shall check the adequacy, accuracy, correctness and completeness of draft Protocols and Reports.

**6.1.8** Upon receipt of the comments (if any), same shall be reviewed and incorporated in the Protocols and Reports.

**6.1.9** Final draft soft copy of protocol & report shall be provided to QA and the soft copy in other Departments shall be deleted in presence of QA person.

**6.1.10** QA shall take the printout of protocol & report for signature.

**6.1.11** Print out of protocol & report shall be provided to Respective Departments for signature.

**6.1.12** All Protocols and Reports shall be prepared by Operating Officer / Executive of concerned Department, Checked by Operating Manager of concerned Department/ Head of related activity.

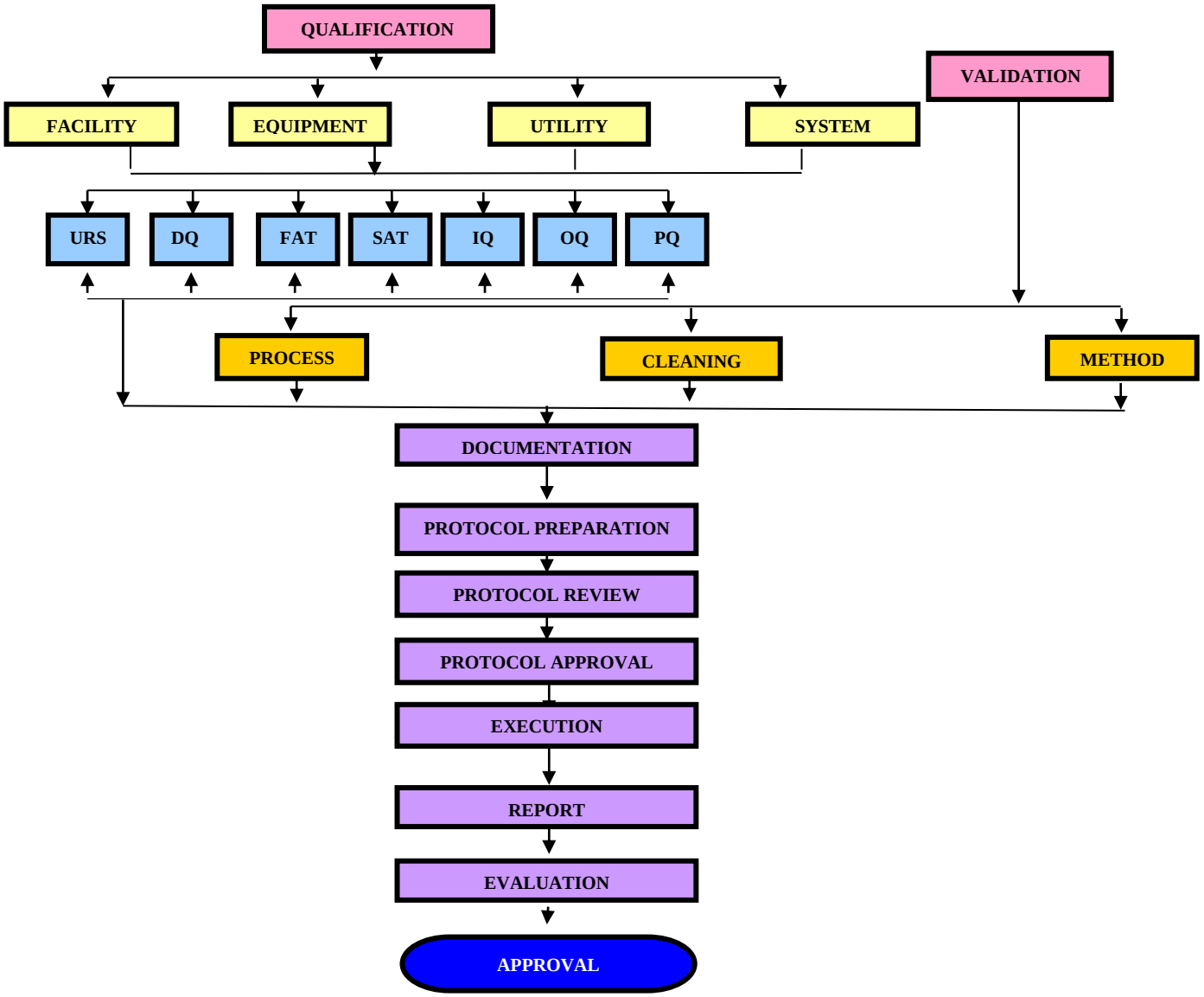
**6.1.13** Upon signature of Respective Department, signed off copy of Protocol & Report shall be sent back to Department Head for Approved by and Final approved by Head QA

**6.1.14** Soft Copy of protocol and report prepared by the User Department and transferred to Head QA and shall be deleted in presence of QA person.

## 6.2 PREPARATION OF QUALIFICATION PROTOCOL CUM REPORTS:

**6.2.1** Qualification Protocol shall be separate for DQ, IQ, OQ and PQ for each activity and report also separate.

**6.2.2** The activity flow for Qualification and Validation process is as:



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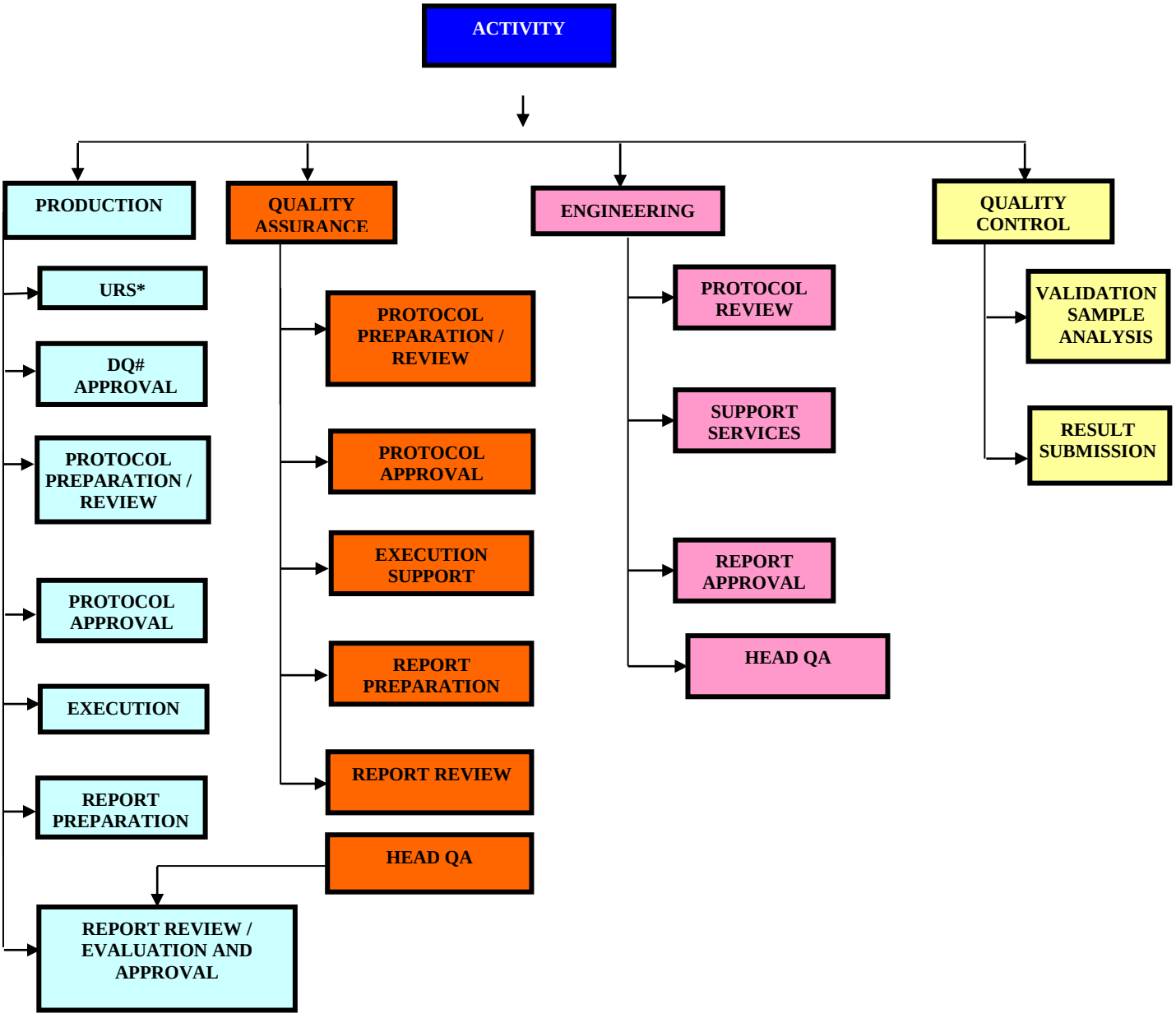
**6.2.3** The Responsibility and activity flow for Qualification and Validation process is as



**6.2.4** Preparation of Protocol shall be initiated after receipt of approved Specification and Design.

**6.2.5** **User Requirement Specification (URS)** (Where ever applicable):

**6.2.6** **Design Qualification:**

**6.2.6.1** Design Qualification shall be provided by the vendor.

**6.2.7** **Operational Qualification:**

**6.2.7.1** Operational Qualification shall be provided by the vendor

**NOTE:** If there is no Define of department on Approval page. Concerned and cross functional Department review the data and final approval of Head Quality assurance Department.

**6.2.8** **Performance Qualification (PQ)**:

**6.2.8.1** **Performance Qualification Protocol:**

**6.2.8.1.1** Performance Qualification Protocol shall be prepared as per format shown in **Annexure-I.**

**6.2.8.2** **Content of Header and Footer:**

**Header:**

**6.2.8.2.1.1** The Header of Performance Qualification Protocol shall have the Name of Organization (Including Name of Location). Header shall have the "Logo" of Organization in Left corner on Top and Title **"Performance Qualification Protocol for----------"** in center written in Bold and Capital letter of font size 12, format shown in **Annexure-I.**

**6.2.8.2.1.2** **Protocol No.:** Shall be written as per current version of respective SOP.

**6.2.8.2.1.3 Revision No.:** Shall be written in Times New Roman, Normal and Font size 12.

**6.2.8.2.1.4 Effective Date:** Effective Date shall be written in the form of **DD/MM/YY** in respective column **(Effective date :)**

**6.2.8.2.1.5 Page No.:** Shall be written in Bold and Normal of Font Size 12. The Page Number shall be mentioned in 'X of Y' format. **For Example:** If a Protocol contains 60 pages then the first page of the Protocol shall be 1 of 60 and the second page shall be 2 of 60 respectively. Shall be Pre-printed.

**Footer:**

**6.2.8.2.2.1 Format No.:** 09 Normal and Capital font size printed on the left corner of the page after

Footer, out of page border and shall be printed as **Annexure-I** with revision No. on all pages of the Protocol.

**6.2.8.3 Content of Body Part:**

The format of Performance Qualification Protocol shall contain the following information but not limited to:

**Equipment (for vessels) / Instrument / Utility Qualification:**

**1.0** Protocol Approval
**2.0** Objective
**3.0** Scope
**4.0** Responsibility
**5.0** Training
**6.0** Equipment / Instrument / Utility Details
**7.0** System Description
**8.0** Reason for Qualification
**9.0** Site of Study
**10.0** Frequency of Qualification
**11.0** Pre Qualification Requirements
**12.0** Procedure
**13.0** Annexure
**14.0** Reference
**15.0** Documents to be attached
**16.0** Deviation from Pre-defined Specification, if any
**17.0** Change Control, if any
**18.0** Review(Inclusive of follow u action, if any)
**19.0** Conclusion
**20.0** Recommendation
**21.0** Abbreviations
**22.0** Revision History

**For Equipment Qualification except Vessels:**

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**1.0** Protocol Approval

**2.0** Objective

**3.0** Scope

**4.0** Responsibility

**5.0** Equipment Details

**6.0** System Description

**7.0** Reason for Qualification

**8.0** Site of Study

**9.0** Frequency of Qualification

**10.0** Pre Qualification Requirements

**11.0** Tests & Checks

**12.0** Checklist of All Tests & Checks

**13.0** References

**14.0** Documents to be attached

**15.0** Non Compliance, if any

**16.0** Deviation from Pre–Defined Specification, If Any

**17.0** Change Control, If Any

**18.0** Abbreviations

**19.0** Revision History

**Water System Qualification:**

**1.0** Protocol Approval

**2.0** Objective

**3.0** Scope

**4.0** Responsibility

**5.0** Training record

**6.0** Pre Qualification Requirement

**7.0** System Description

**8.0** Reason for Qualification

**9.0** Site study

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**13.0** Acceptance Criteria

**14.0** Reference

**15.0** Documents to be attached

**16.0** Non Compliance

**17.0** Deviation from Pre-defined Specification, if any

**18.0** Change Control, if any

**19.0** Abbreviations

**20.0** Revision History

**6.2.9 Performance Qualification Report:**

**6.2.9.1** On the basis of Qualification Protocol, Qualification Report shall be prepared as per **Annexure-II**.

**6.2.9.2 Content of Header and Footer:**

**6.2.9.2.1 Header:**

**6.2.9.2.1.1** The Header of Performance Qualification Report shall have the Name of Organization (Including Name of Location). Header shall have the "Logo" of Organization in Left corner on Top and Title **"Performance Qualification Report for----------"** in center written in Bold and Capital letter of font size 12, format shown in **Annexure-II.**

**6.2.9.2.1.2** **Report No.:** Shall be written as per current version of respective SOP and shall be Pre-printed.

**6.2.9.2.1.3** **Protocol No.:** Respective Protocol No. shall be provided. Protocol no. shall be Pre-printed.

**6.2.9.2.1.4** **Revision No.:** Shall be written in Times New Roman, Normal and Font size 12.

**6.2.9.2.1.5** **Effective Date:** Effective Date shall be written in the form of **DD/MM/YY** in respective column **(Effective date :)**

**6.2.9.2.1.6** **Page No.:** Shall be written in Bold and Normal of Font Size 12. The Page Number shall be mentioned in 'X of Y' format. **For Example:** If a Protocol contains 60 pages then the first page of the Protocol shall be 1 of 60 and the second page shall be 2 of 60 respectively. Shall be Pre-printed.

**6.2.9.2.2 Footer:**

**6.2.9.2.2.1** **Format No.:** 09 Normal and Capital font size printed on the left corner of the page after

Footer, out of page border and shall be printed as per **Annexure-II** with revision no. on

All pages of the Report.

**6.2.9.3** **Content of Body Part:**

The format of Performance Qualification Report shall contain the following information but not limited to:

**Equipment (Vessels)/Instrument/Utility Qualification Report:**

**1.0** Report Pre-Approval

**2.0** Objective

**3.0** Scope

**4.0** Responsibility

**5.0** Equipment / Instruments / Utility Details

**6.0** Reason for Qualification

**7.0** Site of Study

**8.0** Frequency of Qualification

**9.0** Annexure

**10.0** Reference

**11.0** Documents to be attached

**12.0** Deviation from Pre-defined Specification, if any

**13.0** Change Control, if any

**14.0** Review (inclusive of follow up action, if any )

**15.0** Conclusion

**16.0** Recommendation

**17.0** Abbreviations

**18.0** Revision History

**19.0** Report Post Approval

**For Equipment Qualification except Vessels:**

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**Water System Qualification:**

**10.0** Training of Execution team

**11.0** Tests and checks

**12.0** Checklist of all tests and checks

**13.0** Documents to be Attached

**14.0** Non Compliance

**15.0** Deviation from Pre-defined Specification, if any

**16.0** Change Control, if any

**17.0** Review (inclusive of follow up action, if any)

**18.0** Conclusion

**19.0** Recommendation

**20.0** Revision History

**21.0** Abbreviations

**22.0** Report Post Approval

**Area Qualification:**

**1.0** Report Pre-Approval

**2.0** Objective

**3.0** Scope

**4.0** Responsibility

**5.0** Training Record

**6.0** Equipment Details

**7.0** Reason for Qualification

**8.0** Site of study

**9.0** Frequency of Qualification

**10.0** Pre-Qualification Requirements

**11.0** Procedure

**12.0** Checklist for tests and Checks

**13.0** References

**14.0** Documents to be attached

**15.0** Non Compliance

**16.0** Deviation from Pre-defined Specification, if any

**17.0** Change Control, if any

**18.0** Review (inclusive of follow up action, if any)

**19.0** Conclusion

**20.0** Recommendation

**21.0** Abbreviations

**22.0** Revision History

**23.0** Report Post Approval

**6.2.9.3.1** The Qualification Report shall be reviewed by Head of User Department, QC / Engineering / Production Head (as applicable) and approved by Head QA. The approval of Qualification Report is considered as handover of the system to user Department for its intended purpose.

**6.2.10 Preparation of Validation / Re-Validation Protocols:**

**6.2.10.1** All Validation / Re-Validation Protocols shall be prepared as per format **"Validation Protocol (Specimen)"** as shown in **Annexure-III.** The format of Validation / Re-validation

Protocol shall contain the following information.

**6.2.10.2 Process Validation Protocol:**

Process Validation Protocol shall contain the following Headings but not limited to:

**1.0** Protocol Approval

**2.0** Objective

**3.0** Scope

**4.0** Responsibility

**5.0** Training Record

**6.0** Reason for Validation

**7.0** Process Validation Methodology

**8.0** Master Document Verification

**9.0** Process Validation Pre requisite

**10.0** Product Information

**11.0** Manufacturing Formula

**12.0** Specifications and Standard Test Procedure

**13.0** Equipment Qualification Verification

**14.0** Process Flow Diagram

**15.0** Methodology and Sampling Procedure

**16.0** Process Steps and Critical Process Variables for Validation

**17.0** Sampling and Analysis Plan

**18.0** Sampling Locations

**19.0** Acceptance Criteria

**20.0** Revalidation

**21.0** Deviations

**22.0** Validation Stability Plan

**23.0** Validation Report

**24.0** Conclusion

**25.0** Reference Documents

**26.0** List of Attachments

**27.0** Abbreviations

**28.0** Revision History

**6.2.10.3 Analytical Method Validation Protocol:**

Analytical Method Validation Protocol shall contain the following Heads but not limited to:

1. Protocol Approval
2. Objective
3. Scope
4. Responsibility
5. Training Details
6. Instrument Calibration Verification
7. Procedure

8. Materials and Instruments Used
9. Study Design
10. Procedure for Method Validation
11. Validation Acceptance Criteria
12. References
13. Summary Report
14. Deviation (if any)
15. Abbreviations
16. Revision History

**6.2.10.4 Process Simulation Study (Media Fill) Protocol:**

Process Simulation Protocol shall contain the following Heads but not limited to:

1. Protocol pre Approval
2. Objective
3. Scope
4. Responsibility
5. Re-qualification Criteria
6. Frequency of Validation
7. Number of Runs
8. Duration of Runs
9. Size of Run
10. Selection of Media
11. Interventions (Worse Case Study)
12. Incubation and Examination of Media-Filled Units
13. Interpretation of Data and Acceptance Criteria
14. Analytical Support
15. Environmental Monitoring
16. Training Details
17. Equipment Qualification / Instrument Calibration Verification
18. Description of Process Simulation Study Methodology

19. Post Media Fill Cleaning
20. Media fill process for three piece filling machine (eye/ear drops)
21. Procedure
22. Monitoring of Process Parameters
23. Critical Parameters and Acceptance Criteria
24. Sampling Plan
25. Deviations
26. Definitions
27. References
28. Abbreviations
29. Revision History

**6.2.10.5 Stability Study Protocol:**

Stability Protocol shall contain the following Heads but not limited to:

1. Protocol Approval
2. Objective
3. Scope
4. Responsibility
5. Reason for Stability Studies
6. Training Details
7. Types of Studies
8. Specification and Test Methodology Information
9. Test results Acceptance Criteria
10. Reference to Analytical Validation [Stability Indicating]
11. Study Design and Condition
12. Stability Testing Matrix
13. Data Analysis Details
14. Stability Study Report
15. Out of Specifications
16. Deviations

17. Change Control
18. Equipment and Storage Malfunction
19. Commitment
20. Conclusion
21. Reference Documents
22. List of Attachments
23. Abbreviations

**6.2.10.6 Hold Time Study Protocol (For Product):**

Hold Time Study Protocol shall contain the following Heads but not limited to:

1. Protocol Approval
2. Objective
3. Scope
4. Responsibility
5. Reason for Validation
6. Site of Study
7. Training Details
8. Methodology
9. Master Document Verification
10. Product Details
11. Manufacturing Formula
12. Manufacturing Procedure
13. Hold Time Study Procedure
14. Sampling and Analysis Plan
15. Acceptance Criteria
16. Revalidation Criteria
17. Frequency of Validation
18. Change Control and Deviation
19. Conclusion
20. References

21. Documents to be Attached
22. Revision History
23. Abbreviations

**6.2.10.7 Hold Time Study Protocol (For Dirty/Cleaned Equipment):**

1. Protocol Pre-Approval
2. Objective
3. Scope
4. Responsibility
5. Accessories Details
6. Reason for Validation
7. Site of Study
8. Type of Validation
9. Acceptance Criteria
10. Methodology / Hold Time Study Procedure
11. Sampling Details
12. Photographs of Swab Sampling Location
13. Revalidation Criteria
14. Frequency of Validation
15. Change Control and Deviation
16. Conclusion
17. References
18. Documents to be Attached
19. Recommendation
20. Revision History
21. Abbreviations

**6.2.10.8 Cleaning Validation Protocol:**

Cleaning Validation Protocol shall contain the following Heads but not limited to:

1. Protocol Approval
2. Objective

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- ✓ Linearity of API Working Standard Solution
- ✓ Linearity and Recovery in Swabs Spiked with API
- ✓ Recovery of Drug from Swabs Sampled from Spiked SS Plates
- ✓ Lowest Detection Limit
- ✓ Record of Batch Manufacturing & Events
- ✓ Testing Record

**6.2.10.9 Product Development Protocol:**

Product Development Protocol shall contain the following heads but not limited to:

**1.** Approval:

Prepared By : F & D Officer / Executive

Reviewed By : Manager-F&D

Approved By : Head-F&D

**2.** Objective

**3.** Scope

**4.** Responsibility

**5.** Pre-formulation Study:

- ➢ Patent Information
- ➢ Reference Product Information
- ➢ Sourcing of Active Pharmaceutical Ingredients, Excipients & Packaging Material
- ➢ Analytical Details

**6.** Reference Product (Market Sample) Evaluation:

- ➢ Product Name :
- ➢ Generic Name :
- ➢ Strength :
- ➢ Dosage Form :
- ➢ Administration :
- ➢ Manufactured By :
- ➢ Marketed By :

- Composition :
- Storage Condition :
- Pack Profile :
- Primary Packing
- Secondary Packing
- Physico-Chemical Properties:

**7.** Literature Review:

- Physico-Chemical Properties of API:
  - Description
  - Solubility
  - Chemical Name
  - Chemical Structure
  - Molecular Weight
  - Molecular Formula
  - Pharmacopoeial Status of Drug Substance
  - Pharmacopoeial Status of Drug Product
  - Storage Condition
- Literature Review of Drug Product:
  - Introduction of Drug Product
  - Adult Dose
  - Pharmacokinetics:
  - Absorption
  - Distribution
  - Metabolism
  - Elimination
  - Pharmacokinetics Parameter Bioavailability (%)
  - Plasma Half Life (hrs)
  - Volume of Distribution (lit/hrs)
  - C max (µg / ml)

- T max (hrs)
- Bound in Plasma (%)
- Adverse Effects
- Administration
- Storage
- Design Input Specifications:
- Active Pharmaceutical Ingredient Specification
- Excipients Specification
- Product Specification
- Risk Assessment

- Development Pharmaceutics:
  - Development Flow Chart
  - Prototype Qualitative Formula
  - Brief Manufacturing Process
  - Equipment / Tooling
- Development Stages:
  - Lab Scale Development
  - Validation Batch
- Activity Schedule:

| | | |
|---|---|---|
| Literature / Patent Survey | : | F&D |
| API / Raw Materials / PM Sourcing | : | Purchase |
| Change Parts Procurement | : | Purchase |
| Reference Sample Procurement | : | Purchase |
| Reference Sample Characterization | : | F&D |
| Analytical Method Development (API) | : | F&D |
| Compatibility Study | : | F&D |
| Lab Scale Development | : | F&D |

- Analytical Method Development

(Finished Product) (Stability Indicating) : F&D

- Lab Scale Stability Batches / Stability : F&D
- Analytical Method Validation : F&D
- Generation of Tentative MFR / Specification : F&D
- Lab Scale up Batch / Stability : F&D
- Bio / Pivotal Batch / Stability : F&D
- Dossier Submission : F&D

8. References
9. Revision History
10. Abbreviations

**6.2.10.10 System / Utility / other Validation Protocol:**

System / Utility / Thermal Mapping / BET Validation / Sterility Test Validation/General Validation Protocols and any Others Validation Protocol shall contain the following Heads but not limited to:

1. Protocol Approval
2. Objective
3. Scope
4. Responsibility
5. Reason for Validation
6. Site of Study
7. Training Details
8. Methodology
9. Master Document Verification
10. Acceptance Criteria
11. Validation / Re-Validation Criteria
12. Frequency of Validation
13. Change Control and Deviation
14. Conclusion
15. Recommendation

16. References
17. Documents to be Attached
18. Deviation From Pre-Defined Specification, If any
19. Change Control, If any
20. Revision History
21. Abbreviations

**6.2.11 PREPARATION OF VALIDATION REPORT:**

**6.2.11.1** All Validation Reports shall be prepared as per format **"Validation Report (Specimen)"** as shown in **Annexure-IV.** The format of Validation / Re-Validation Report shall contain the following information.

**6.2.11.2 Process Validation Report:**

Process Validation Report shall contain the following Heads but not limited to:

1. Report Pre-Approval
2. Objective
3. Scope
4. Responsibility
5. Training Records
6. Master Document Verification
7. Equipment Details
8. Raw Materials and Primary Packing Materials
9. Dispensing Verification
10. Critical Process Variables
11. Environmental Monitoring of Manufacturing Area
12. Semi-Finished Product Analysis
13. Finished Product Analysis
14. Packing
15. Yield Statement
16. Attachments
17. Deviation (if any)

18. Conclusion
19. Recommendation
20. Abbreviations
21. Report Post Approval

**6.2.11.3 Analytical Method Validation Report:**

Analytical Method Validation Report shall contain the following Heads but not limited to:

1. Report Pre-Approval
2. Objective
3. Scope
4. Responsibility
5. Training Records
6. Instrument Calibration Verification
7. Materials & Instruments Details
8. Table for System Suitability Study
9. Table for Specificity
10. Table for Accuracy
11. Table for System Precision
12. Table for Method Precision
13. Table for Solution Stability Study
14. Table for Linearity and Range
15. Linearity Curve
16. Range Curve
17. Table for Method Intermediate Precision
18. Table for Robustness
19. Table for Filter Paper Interference
20. Table for Summarized Validation Report
21. Attachments
22. Deviation (if any)
23. Conclusion

24. Recommendation
25. Abbreviations
26. Report Post Approval

**6.2.11.4 Process Simulation Study (Media Fill) Report:**

Process Simulation Study (Media Fill) Report shall contain the following heads but not limited to:

1. Report Pre-Approval
2. Objective
3. Scope
4. Responsibility
5. Training Attendance Record
6. Master Document Verification
7. Detail of Media and Primary Packaging Materials used
8. Growth Promotion Test of Media (SCDM)
9. Equipments Details
10. Air Handling Unit (AHU) Qualification Verification
11. Utility Qualification Verification
12. Instrument Calibration Verification
13. Machine Parts and Accessories Sterilization
14. Three Piece Filling Machine
15. Environmental Monitoring of Three Piece Filling and Sealing
16. Container Closer Integrity (Leak Test & Clarity) Test
17. Microbiological Analysis Results
18. Interventions During Filling and Sealing (worst case condition)
19. Inspection of Filled and Sealed Containers
20. Environmental Monitoring Record of Incubation Room / Chamber
21. Observation of Media Filled Containers after Incubation
22. Post GPT of Deactivated Containers
23. Destruction of Incubated Containers after Inspection

24. Batch Yield
25. References
26. Documents to be Attached
27. Non compliance
28. Deviation from Pre-defined Parameters, if any
29. Change Control, if any
30. Review (inclusive of follow up action, if any)
31. Conclusion
32. Recommendation
33. Abbreviations
34. Revision History
35. Report Post Approval

**6.2.11.5 Stability Study Report:**

Stability Study Report shall contain the following heads but not limited to:

1. Report Pre-Approval
2. Objective
3. Scope
4. Responsibility
5. Training Records
6. Product Details
7. Active Ingredient Details
8. Excipients Details
9. Container Closure System Details
10. Stability Study test Conditions Results
11. Attachments
12. Deviation (if any)
13. Change Control

14. Out of Specification
15. Conclusion
16. Recommendation
17. Abbreviations
18. Report Post Approval

**6.2.11.6 Hold Time Study Report (For Product) :**

Hold Time Study Report shall contain the following heads but not limited to:

1. Report Pre-Approval
2. Objective
3. Scope
4. Responsibility
5. Training Record
6. Master Documents Verification
7. Environmental Monitoring Record
8. Hold Time Study Results
9. Change control and Deviation
10. References
11. Documents to be Attached
12. Review (Inclusive of Follow Up Action, If any)
13. Conclusion
14. Recommendation
15. Abbreviations
16. Report Post Approval

**6.2.11.7 Hold Time Study Report (For Dirty / Cleaned Equipments):**

Hold Time Study Report (For Dirty/Cleaned Equipments) shall contain the following heads

But not limited to:

1. Report Pre-Approval
2. Objective

3. Scope
4. Responsibility
5. Training Record
6. Master Documents Verification
7. Environmental Monitoring Record
8. Hold Time Study Results (Bioburden Report, Record of Environment Monitoring,
   Surface Monitoring & Personnel Monitoring)
9. Change control and Deviation
10. References
11. Documents to be Attached
12. Review (Inclusive of Follow Up Action, If any)
13. Conclusion
14. Recommendation
15. Abbreviations
16. Report Post Approval

**6.2.11.8 Cleaning Validation Report:**

Cleaning Validation Report shall contain the following heads but not limited to:

1. Report Pre-Approval
2. Objective
3. Scope
4. Responsibility
5. Determination of Acceptance Criteria
6. Training Records
7. Swab Limit Calculation
8. List of Instruments with Calibration Status
9. List of Equipments with Qualification and SOP Status
10. Equipment Train with Surface Area
11. List of Swab / Rinse Location

12. Number of Washing Cycles / Quantity of Water Used for Cleaning
13. Stability of API Working Standard Solution
14. Blank Swab Detector Response for Analysis of API
15. Linearity of API Working Standard Solution
16. Linearity and Recovery in Swabs Spiked with API
17. Recovery of Drug from Swabs sampled from Spiked SS Plates
18. Lowest Detection Limit
19. Lowest Quantifiable Limit
20. Record of Batch Manufacturing & Cleaning Events
21. Record of Sampling & Testing
22. Testing Record
23. Conclusion
24. References
25. Deviation
26. Recommendation
27. Abbreviations
28. Report Post Approval

**6.2.11.9 Product Development Report:**

Product Development Report shall contain the following heads but not limited to:

1. Objective
2. Scope
3. Responsibility
4. Reference Product Information
5. Literature Search (Review)
6. Pre-Formulation Studies
7. Reference Product Evaluation
8. Formulation Development
9. Three lab Scale Batches
10. Container Closure System

**11.** API & Excipient Specification

**12.** Analytical Testing Procedure for Raw Materials

**13.** Primary Packaging Material Specification

**14.** Primary Packaging Material Standard Test Procedure

**15.** Product Specifications

**16.** Finished Product Standard Test Procedure

**17.** Materials Safety Data Sheet of Raw Materials

**18.** Final Formula and Method of Preparation

**19.** Plan of Validation & Commercial Batches

**20.** Formulation Characterization Studies

**21.** Summary

**22.** Conclusion

**23.** Recommendation

**24.** References

**25.** Approval:

Prepared By : F & D Officer / Executive

Reviewed By : Manager-F&D

Approved By : Head-F&D

**26.** Abbreviations

**6.2.11.10 System / Utility / General Validation Reports / other Validation Report:**

System / Utility / Thermal Mapping / BET Validation / Sterility Validation and any Others Validation Report shall contain the following heads but not limited to:

**1.** Report Pre-Approval

**6.2.12.1** Objective

**2.** Scope

**3.** Responsibility

**4.** Training Records

**5.** Master Document Verification

6. Equipment / Utility Details
7. Critical Parameters / other Required Heads
8. References
9. Documents to be Attached
10. Deviation from Pre-defined Parameters, if any
11. Change Control, if any
12. Review (inclusive of follow up action, if any)
13. Conclusion
14. Recommendation
15. Abbreviations
16. Report Post Approval

**6.2.12 Preparation of Re-Qualification Report:**

**6.2.12.2** In case of major change in Equipment /Area / HVAC/ LAF / DPB / DGSC system the requalification protocol shall be prepared considering all the factors that affected due to the modification or change or based on the recommendation of change control document and accordingly the tests shall be included in the protocol.

**6.2.12.3** In case of periodic requalification as per the defined frequency, Individual master copy of Requalification Report shall be prepared for Equipment / Area / HVAC / LAF / DPB / DGSC as per Format **"Re-Qualification Report (Specimen)"** as shown in **Annexure-IV.**

**6.2.12.4** Qualification Protocol of respective Equipment / Area / AHU / LAF / DPB / DGSC shall be followed for procedure and Acceptance Criteria of respective tests and Check; Requalification Protocol shall not be prepared and the Number of respective Qualification Protocol shall be recorded as Reference Protocol No. in Header of Re-Qualification Report**.**

**6.2.12.5** Master Re-Qualification Report No. shall have Pre-printed on first Page of Report as per QA SOP, Titled **"Protocol and Report Numbering System"**. Provision shall be provided to fill the details of individual Equipment /Area / AHU / LAF / DPB / DGSC in the Report.

**6.2.12.6** After photocopying of Master Re-Qualification Report, all pages shall be stamped as **"CONTROLLED COPY"** with green colour ink and sign and date with Black Ink Ball Point Pen as per SOP, Titled **"Documentation and Data Control"**.

**6.2.12.7** QA Officer / Executive shall issue the Master Re-Qualification Report for each Equipment / Area / AHU / LAF / DPB /DGSC.

**6.2.12.8 Content of Header and Footer:**

**6.2.12.7.1 Header:**

**6.2.12.7.1.1** The Header of Performance Re-Qualification Report shall have the Name of Organization (Including Name of Location). Header shall have the "Logo" of Organization in Left corner on Top and Title **Performance Re-Qualification Report for**….. **ID No.…** in center written in Bold and Capital letter of font size 12, format shown in **Annexure-IV**.

**6.2.12.7.1.2 Report No.:** Shall be written in Times New Roman, Normal and Font size 12.

**6.2.12.7.1.3 Revision No.:** Shall be written in Times New Roman, Normal and Font size 12.

**6.2.12.7.1.4 Reference Protocol No.:** Reference Protocol No. shall be written in respective column with blue colour ball ink pen provided by QA Personnel at the time of Issuance of individual control copy.

**6.2.12.7.1.5 Page No.:** Shall be written in Bold and Capital of Font Size 12. The Page Number shall be mentioned in 'X of Y' format. **For Example:** If a Report contains 20 pages then the first page of the Report shall be 1 of 20 and the second page shall be 2 of 20 respectively. Shall be Pre-printed.

**6.2.12.7.2 Footer:**

**6.2.12.7.2.1 Format No.:** 09 Regular and Capital font size printed on the left corner of the page, out of page border and shall be printed as per **Annexure-IV** with revision no. on all pages of the Report.

**6.2.12.9 Content of Body Part:**

The Performance Re-Qualification Report shall contain the following information but not limited to:

**6.2.12.8.1** Re-Qualification Report shall contain the following information.

| STANDARD OPERATING PROCEDURE | |
|---|---|
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**1.0** Report Pre-Approval

**2.0** Objective

**3.0** Scope

**4.0** Responsibility

**5.0** Equipment Details

**6.0** System Description

**7.0** Reason for Re-Qualification

**8.0** Site of Study

**9.0** Frequency of Qualification

**10.0** Pre-Qualification Requirements

**11.0** Tests and Checks

**12.0** References

**13.0** Documents to be attached

**14.0** Deviation from Pre-defined Specification, if any

**15.0** Change Control, if any

**16.0** Review (Inclusive of follow up action, if any)

**17.0** Conclusion

**18.0** Recommendation Abbreviations

**19.0** Report Post Approval

**6.2.13 Layout of Protocol Cum Reports, Protocols and Reports:**

**6.2.13.1** All Protocols and Reports shall contain Header, Footer and Body. Formats for Protocols and Reports are shown in **Annexure-I, II, III, IV, V, VI, VII,**

**6.2.13.2** The Line Spacing between two points or title and subtitle shall be 1.0 / 1.5 and font style shall be Times New Roman 12 font.

**6.2.13.3** Paper Width 8.5”, Height 11.5” (Paper Size Custom) and Margin Top, Left, Right 24pt and Bottom Margin 31pt.

**6.2.13.4** Protocols and Reports (Master Copy) shall be printed on A4 size plain white colored Paper (75 GSM) using **“Times New Roman”** Font with Black Ink.

**6.2.13.5** Printing shall be done on one side of the paper only.

**6.2.13.6** All Protocols and Reports contents shall be covered by Single Borderline (Line width ½ pt).

**6.2.14 Numbering System of Protocols and Reports:**

**6.2.14.1** All Protocols and Reports should have a unique Reference Number, which shall identify that document. The document should have a cross-reference.

**6.2.14.2** Once a number is assigned to any Protocol and Report, the same number shall not be assigned to any other Protocols and Reports.

**6.2.14.3** Protocols and Reports Numbering shall be done as per current version of SOP**.**

**6.2.15 Font Size of Header, Footer and Body Contents:**

| NAME OF CONTENT | FONT SIZE |
|---|---|
| **HEADER:** | |
| Logo (On Left Hand Side Corner of the Page) | Height-0.75''and Width-0.63'' |
| Name of the Organization, Location,<br>Title of URS, Protocol and Report, URS / Protocol / Report No., Page No. (In the form of X of Y), | 12 Bold and Capital |
| **FOOTER:** | |
| Format No. | 09 Capital Normal |
| **BODY:** | |
| Main Heading and Table Heading | 12 Bold and Capital |
| Sub Heading | 12 Normal and Bold |
| Table Contents Except Headings | 12 Normal |
| **FIRST PAGE CONTENT:** | |
| Title of Protocol and Report | 28 Bold and Capital with Double Line Space |
| Table Content | 12 Bold and Capital |
| Space between lines | 1.15 |
| Margin of page | Width8.5,Height 11.5,Margin top |

| NAME OF CONTENT | FONT SIZE |
|---|---|
| | 2"Left 1"Right 0.4"and Bottom Margin 2" |

**6.2.16 Storage of Protocols and Reports:**

**6.2.16.1** All Master Copy / Soft Copy of Approved Protocols and Reports shall be stored in QA Department with Password Protected System and Data Backup shall be kept in Information Technology (IT) Department.

**6.2.16.2** If Protocol is revised, previous version of Protocol shall be made Obsolete and shall be archived for Historical purpose.

**6.2.17 Control, Issuance and Execution of Protocols and Reports:**

**6.2.17.1** After approval of Protocol & Reports, shall be stamped as '**MASTER COPY'** in Blue color ink on all the pages and shall be signed by **Blue Ink Ball Point Pen.**

**6.2.17.2** Effective date shall be entered in all pages of respective header column **(Effective Date :)** with blue colour ink ball pen.

**6.2.17.3** After photocopying of Master Protocol & Reports, shall be stamped as **"CONTROLLED COPY"** with green colour stamp (all pages) and sign and date with Black Ink Ball Point Pen and shall used for execution purpose.

**6.2.17.4** Master Index of Protocols and Reports shall be maintained by QA Department in format **"Master Validation / Qualification / Product Development Report Index"** as shown in **Annexure-VII**. In case of Requalification / Revalidation, Title of format shall be modified as **"Master Revalidation & Requalification Index".**

**6.2.17.5** Index of Protocols and Reports shall be updated once in a Year or whenever required,

**6.2.17.6** QA shall issues the protocol & report for Execution to respective Department by QA and details shall be recorded in format **"Protocol / Report Issuance & Retrieval Record"** as shown in **Annexure-VIII**.

**6.2.17.7** After completion of Qualification / Validation, a Qualification / Validation Report shall be compiled. The Protocol / Report shall be reviewed by User Department i.e. QC / Engineering / Production Head (as applicable), Approved by Head QA.

**6.2.17.8** If any discontinuation of Protocol and Report by using **"DISCONTINUED COPY"** stamp as per SOP.

**6.2.17.9** In case, if any Protocol and Report is submitted to the External Agencies (i.e. Regulatory, Customers / Partners etc.) by using **"UNCONTROLLED COPY"** stamp as per SOP.

**6.2.17.10** Copy of Protocol and Report that is meant only for Information / Reference purpose shall be made by Photostat of Master Copy / Executed Copy (in case of executed Protocol / Report) by using **"REFERENCE COPY"** stamp as per SOP.

**6.2.17.11** All Equipment, Area & Utility Qualification Documents Index shall be prepared as per Format **"Master Equipment / Area / Utility Qualification Documents Index"** as shown in **Annexure-V.**

**6.2.17.12** Master Validation/ Qualification / Product Development Protocol Index shall be prepared as per format **"Master Validation / Qualification / Product Development Protocol Index"** as shown in **Annexure-VI.**

Master Validation/ Qualification / Product Development Report Index shall be prepared as per format **"Master Validation / Qualification / Product Development Report Index"** as shown in **Annexure-VII.**

**6.2.17.13** Previous copy of protocol & report by using **"OBSOLETE COPY"** stamp and refer as per SOP.

**6.2.18 Revision of Protocol & Report:**

**6.2.18.1** Any change required in Protocol & Report shall be done only after Approval of **"Change Control"**.

**6.2.18.2** After Approval of Change Control QA Officer / Executive shall incorporate the changes in soft copy of Protocol & Report.

**6.2.18.3** Revision No., Change Control No. Details of Changes, Reason for Change shall be written under heading **Revision History** in **Qualification & Validation Protocols.**

**6.2.19 Archival of Protocols and Reports:**

**6.2.19.1** All Qualification and Validation Documents (i.e. Process Validation Protocols / Reports, Stability Data & Documents, Qualification & Validation Documents for Critical Utilities / **System / Equipments etc.) shall be retained till the life of the Organization.**

## 7.0 ABBREVIATIONS:

| | |
|---|---|
| AHU | Air Handling Unit |
| API | Active Pharmaceutical Ingredients |
| DQ | Design Qualification |
| DS | Design Specification |
| F&D | Formulation & Development |
| FAT | Factory Acceptance Test |
| FS | Functional Specification |
| GPT | Growth Promotion Test |
| HPLC | High Performance Liquid Chromatography |
| ID No. | Identification Number |
| IQ | Installation Qualification |
| IT | Information Technology |
| Ltd. | Limited |
| OQ | Operational Qualification |
| PDF | Portable Document Format |
| PQ | Performance Qualification |
| Pvt. | Private |
| QA | Quality Assurance |
| QC | Quality Control |
| SAT | Site Acceptance Test |
| SCDM | Soybean Casein Digest Agar Medium |
| SOP | Standard Operating Procedure |
| SS | Stainless Steel |
| URS | User Requirement Specification |
| VMP | Validation Master Plan |

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|---|---|
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## 8.0 ANNEXURES:

| ANNEXURE No. | TITLE OF ANNEXURE | FORMAT No. |
|---|---|---|
| Annexure-I | Performance Qualification Protocol (Specimen) | F01-00 |
| Annexure-II | Performance Qualification Report (Specimen) | F02-00 |
| Annexure-III | Validation/Revalidation Protocol (Specimen) | F03-00 |
| Annexure-IV | Validation/Re-Qualification Report (Specimen) | F04-00 |
| Annexure-V | Master Equipment/Area/Utility Qualification Documents Index | F05-00 |
| Annexure-VI | Master Validation/Qualification/Product Development Protocol Index | F06-00 |
| Annexure-VII | Master Validation/Qualification/Product Development Report Index | F07-00 |
| Annexure-VIII | Protocol/Report Issuance & Retrieval Record | F08-00 |

## 9.0 DISTRIBUTION:

Master Copy Quality Assurance Department

Controlled Copy No. 01 Quality Assurance Department.

Controlled Copy No. 02 Production Department

Controlled Copy No. 03 Quality Control Department

## 10.0 REFERENCES:

- A WHO guide to Good Manufacturing Practice (GMP) requirements (Part 2: Validation).
- Validation Master Plan.

## 11.0 REVISION HISTORY:

| Revision No. | Change Control No. | Details of Changes | Reason of changes | Effective Date | Done By |
|---|---|---|---|---|---|
| 00 | Not Applicable | Not Applicable | New SOP | | |

| STANDARD OPERATING PROCEDURE | |
|---|---|
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## ANNEXURE-I
## PERFORMANCE QUALIFICATION PROTOCOL (SPECIMEN)

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| | | **EFFECTIVE DATE:** | |
| | | **PAGE No.:** | |

**PERFORMANCE QUALIFICATION**

**PROTOCOL**

**FOR----------**

| | |
|---|---|
| **EQUIPMENT / INSTRUMENT ID No.** | |
| **LOCATION** | |
| **DATE OF QUALIFICATION** | |
| **SUPERSEDE PROTOCOL No.** | |

FORMAT NO.: …….

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| | | **EFFECTIVE DATE:** | |
| | | **PAGE No.:** | |

**1.0 PROTOCOL APPROVAL:**

**PREPARED BY:**

| DESIGNATION | NAME | SIGNATURE | DATE |
|---|---|---|---|
| OFFICER / EXECUTIVE (DEPARTMENT) | | | |

**REVIEWED BY:**

| DESIGNATION | NAME | SIGNATURE | DATE |
|---|---|---|---|
| EXECUTIVE/MANAGER (DEPARTMENT) | | | |
| HEAD (ENGINEERING) | | | |
| HEAD (PRODUCTION) | | | |
| HEAD (QUALITY CONTROL) | | | |

**APPROVED BY:**

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

# STANDARD OPERATING PROCEDURE

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| **Department:** Quality Assurance | **SOP No.:** |
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| **Supersedes:** Nil | **Review Date:** |
| **Issue Date:** | **Page No.:** |

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| | | **REVISION No.** |
| | | **EFFECTIVE DATE:** |
| | | **PAGE No.:** |

**2.0 OBJECTIVE:**

**3.0 SCOPE:**

**4.0 RESPONSIBILITY:**

| DEPARTMENTS | RESPONSIBILITIES |
|---|---|
| | |

**5.0 EQUIPMENT DETAILS:**

**6.0 SYSTEM DESCRIPTION:**

**7.0 REASON FOR QUALIFICATION:**

**8.0 SITE OF STUDY:**

**9.0 FREQUENCY OF QUALIFICATION:**

**10.0 PRE-QUALIFICATION REQUIREMENTS:**

**11.0 TESTS & CHECKS:**

**12.0 CHECKLIST OF ALL TESTS & CHECKS:**

**13.0 REFERENCES:**

**14.0 DOCUMENTS TO BE ATTACHED:**

**15.0 NON COMPLIANCE, IF ANY:**

**16.0 DEVIATION FROM PRE-DEFINED SPECIFICATION, IF ANY:**

**17.0 CHANGE CONTROL, IF ANY:**

**18.0 ABBREVIATIONS:**

**19.0 REVISION HISTORY**

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|---|---|
| **Department:** Quality Assurance | **SOP No.:** |
| **Title:** Preparation of Validation/Qualification Protocol and Report | **Effective Date:** |
| **Supersedes:** Nil | **Review Date:** |
| **Issue Date:** | **Page No.:** |

## ANNEXURE-II
## PERFORMANCE QUALIFICATION REPORT (SPECIMEN)

**Page 01**

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| | **PERFORMANCE QUALIFICATION REPORT FOR ----------** | **PROTOCOL No.:**<br>**REVISION No.**<br>**REPORT No.:**<br>**REVISION No.**<br>**PAGE No.:** |

**PERFORMANCE QUALIFICATION**

**REPORT**

**FOR**

**----------**

| | |
|---|---|
| **EQUIPMENT / INSTRUMENT ID No.** | |
| **LOCATION** | |
| **DATE OF QUALIFICATION** | |
| **SUPERSEDE REPORT No.** | |

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**1.0 REPORT PRE- APPROVAL:**

**PREPARED BY:**

| DESIGNATION | NAME | SIGNATURE | DATE |
|---|---|---|---|
| OFFICER / EXECUTIVE (DEPARTMENT) | | | |

**REVIEWED BY:**

| DESIGNATION | NAME | SIGNATURE | DATE |
|---|---|---|---|
| EXECUTIVE/MANAGER (DEPARTMENT) | | | |
| HEAD (PRODUCTION) | | | |
| HEAD (ENGINEERING) | | | |
| HEAD (QUALITY CONTROL) | | | |

**APPROVED BY:**

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

FORMAT NO.: …….

# STANDARD OPERATING PROCEDURE

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|---|---|
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**2.0 OBJECTIVE:**

**3.0 SCOPE:**

**4.0 RESPONSIBILITY:**

| DEPARTMENTS | RESPONSIBILITIES |
|---|---|
| | |

**5.0 EQUIPMENT DETAILS:**

**6.0 PRE-QUALIFICATION REQUIREMENTS:**

**7.0 TESTS & CHECKS:**

**8.0 CHECKLIST OF ALL TESTS & CHECKS:**

**9.0 DOCUMENTS ATTACHED:**

**10.0 NON COMPLIANCE:**

**11.0 DEVIATION FROM PRE-DEFINED SPECIFICATION, IF ANY:**

**12.0 CHANGE CONTROL, IF ANY:**

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

**14.0 CONCLUSION:**

**15.0 RECOMMENDATION:**

**16.0 ABBREVIATIONS:**

**17.0 REVISION HISTORY:**

**18.0 REPORT POST APPROVAL**

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**17.0 REPORT POST APPROVAL:**

**PREPARED BY:**

| DESIGNATION | NAME | SIGNATURE | DATE |
|---|---|---|---|
| OFFICER / EXECUTIVE (DEPARTMENT) | | | |

**REVIEWED BY:**

| DESIGNATION | NAME | SIGNATURE | DATE |
|---|---|---|---|
| EXECUTIVE/MANAGER (DEPARTMENT) | | | |
| HEAD (PRODUCTION) | | | |
| HEAD (ENGINEERING) | | | |
| HEAD (QUALITY CONTROL) | | | |

**APPROVED BY:**

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

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| STANDARD OPERATING PROCEDURE | |
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## ANNEXURE-III
## VALIDATION PROTOCOL (SPECIMEN)

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| | ** PROCESS VALIDATION / STABILITY STUDY / ANALYTICAL METHOD VALIDATION / MEDIA FILL / PRODUCT DEVELOPMENT PROTOCOL FOR ---------- | PROTOCOL No.: | |
| | | REVISION No. | |
| | | EFFECTIVE DATE: | |
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**PROTOCOL**

**FOR**

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| | | REVISION No. | |
| | | EFFECTIVE DATE: | |
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FORMAT NO.:……

* * May vary depending on validation type

| | | | |
|---|---|---|---|
| | **** PROCESS VALIDATION / STABILITY STUDY / ANALYTICAL METHOD VALIDATION / MEDIA FILL / PRODUCT DEVELOPMENT PROTOCOL FOR ----------** | **PROTOCOL No.:** | |
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1. **PROTOCOL APPROVAL:**

**PREPARED BY:**

| DESIGNATION | NAME | SIGNATURE | DATE |
|---|---|---|---|
| OFFICER / EXECUTIVE (DEPARTMENT) | | | |

**REVIEWED BY:**

| DESIGNATION | NAME | SIGNATURE | DATE |
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| EXECUTIVE/MANAGER (DEPARTMENT) | | | |
| HEAD (PRODUCTION) | | | |
| HEAD (ENGINEERING) | | | |
| HEAD (QUALITY CONTROL) | | | |

**APPROVED BY:**

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

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* * May vary depending on validation type

# STANDARD OPERATING PROCEDURE

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| **Department:** Quality Assurance | **SOP No.:** |
| **Title:** Preparation of Validation/Qualification Protocol and Report | **Effective Date:** |
| **Supersedes:** Nil | **Review Date:** |
| **Issue Date:** | **Page No.:** |

**Other Pages**

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| | ** PROCESS VALIDATION / STABILITY STUDY / ANALYTICAL METHOD VALIDATION / MEDIA FILL / PRODUCT DEVELOPMENT PROTOCOL FOR ---------- | PROTOCOL No.: | |
| | | REVISION No. | |
| | | EFFECTIVE DATE: | |
| | | PAGE No.: | |

2. **OBJECTIVE:**
3. **SCOPE:**
4. **RESPONSIBILITY:**

| DEPARTMENTS | RESPONSIBILITIES |
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| | |

5. **Re-qualification Criteria**
6. **Frequency of Validation**
7. **Number of Runs**
8. **Duration of Runs**
9. **Size of Run**
10. **Selection of Media**
11. **Interventions (Worse Case Study)**
12. **Incubation and Examination of Media-Filled Units**
13. **Interpretation of Data and Acceptance Criteria**
14. **Analytical Support**
15. **Environmental Monitoring**
16. **Training Details**
17. **Equipment Qualification / Instrument Calibration Verification**
18. **Description of Process Simulation Study Methodology**
19. **Post Media Fill Cleaning**
20. **Media fill process for three piece filling machine (eye/ear drops)**
21. **Procedure**
22. **Monitoring of Process Parameters**
23. **Critical Parameters and Acceptance Criteria**
24. **Sampling Plan**
25. **Deviations**
26. **Definitions**
27. **References**
28. **Abbreviations**
29. **Revision History**

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* * May vary depending on validation type

| STANDARD OPERATING PROCEDURE | |
|---|---|
| **Department:** Quality Assurance | **SOP No.:** |
| **Title:** Preparation of Validation/Qualification Protocol and Report | **Effective Date:** |
| **Supersedes:** Nil | **Review Date:** |
| **Issue Date:** | **Page No.:** |

## ANNEXURE-IV
## VALIDATION/REVALIDATION REPORT (SPECIMEN)

**Page 01**

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|---|---|---|---|
| | **** PROCESS VALIDATION / STABILITY STUDY / ANALYTICAL METHOD VALIDATION / MEDIA FILL / PRODUCT DEVELOPMENT REPORT FOR ----------** | **PROTOCOL No.:**<br>**REVISION No.**<br>**REPORT No.:**<br>**REVISION No.**<br>**PAGE No.:** | |

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**REPORT**

**FOR**

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| **SUPERSEDE REPORT No.** | | | |
| **VALIDATION BATCH NUMBERS** | | | |
| **VALIDATION BATCH SIZE** | | | |

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**Page 02**

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| | **** PROCESS VALIDATION / STABILITY STUDY / ANALYTICAL METHOD VALIDATION / MEDIA FILL / PRODUCT DEVELOPMENT REPORT FOR ----------** | **PROTOCOL No.:**<br>**REVISION No.**<br>**REPORT No.:**<br>**REVISION No.**<br>**PAGE No.:** | |

**REPORT CONTENTS**



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| | ** PROCESS VALIDATION / STABILITY STUDY / ANALYTICAL METHOD VALIDATION / MEDIA FILL / PRODUCT DEVELOPMENT REPORT FOR ---------- | PROTOCOL No.: | |
| | | REVISION No. | |
| | | REPORT No.: | |
| | | REVISION No. | |
| | | PAGE No.: | |

1. **REPORT PRE -APPROVAL:**

**PREPARED BY:**

| DESIGNATION | NAME | SIGNATURE | DATE |
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| OFFICER / EXECUTIVE (DEPARTMENT) | | | |

**REVIEWED BY:**

| DESIGNATION | NAME | SIGNATURE | DATE |
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| EXECUTIVE/MANAGER (DEPARTMENT) | | | |
| HEAD (PRODUCTION) | | | |
| HEAD (ENGINEERING) | | | |
| HEAD (QUALITY CONTROL) | | | |

**APPROVED BY:**

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

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** May vary depending on validation type

# STANDARD OPERATING PROCEDURE

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| **Department:** Quality Assurance | **SOP No.:** |
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| **Supersedes:** Nil | **Review Date:** |
| **Issue Date:** | **Page No.:** |

**Other Pages**

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| | **** PROCESS VALIDATION / STABILITY STUDY / ANALYTICAL METHOD VALIDATION / MEDIA FILL / PRODUCT DEVELOPMENT REPORT FOR ----------** | **PROTOCOL No.:** | |
| | | **REVISION No.** | |
| | | **REPORT No.:** | |
| | | **REVISION No.** | |
| | | **PAGE No.:** | |

**2. OBJECTIVE:**
**3. SCOPE:**
**4. RESPONSIBILITY:**

| DEPARTMENTS | RESPONSIBILITIES |
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**5. Objective**
**6. Scope**
**7. Responsibility**
**8. Training Attendance Record**
**9. Master Document Verification**
**10. Detail of Media and Primary Packaging Materials used**
**11. Growth Promotion Test of Media (SCDM)**
**12. Equipment's Details**
**13. Air Handling Unit (AHU) Qualification Verification**
**14. Utility Qualification Verification**
**15. Instrument Calibration Verification**
**16. Machine Parts and Accessories Sterilization**
**17. Three Piece Filling Machine**
**18. Environmental Monitoring of Three Piece Filling and Sealing**
**19. Container Closer Integrity (Leak Test & Clarity) Test**
**20. Microbiological Analysis Results**
**21. Interventions During Filling and Sealing (worst case condition)**
**22. Inspection of Filled and Sealed Containers**
**23. Environmental Monitoring Record of Incubation Room / Chamber**
**24. Observation of Media Filled Containers after Incubation**
**25. Post GPT of Deactivated Containers**
**26. Destruction of Incubated Containers after Inspection**
**27. Batch Yield**
**28. References**
**29. Documents to be Attached**
**30. Non compliance**
**31. Deviation from Pre-defined Parameters, if any**
**32. Change Control, if any**
**33. Review (inclusive of follow up action, if any)**
**34. Conclusion**
**35. Recommendation**
**36. Abbreviations**
**37. Revision History**

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| | ** PROCESS VALIDATION / STABILITY STUDY / ANALYTICAL METHOD VALIDATION / MEDIA FILL / PRODUCT DEVELOPMENT REPORT FOR ---------- | PROTOCOL No.: | |
| | | REVISION No. | |
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**1.0 REPORT POST APPROVAL:**

**PREPARED BY:**

| DESIGNATION | NAME | SIGNATURE | DATE |
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| OFFICER / EXECUTIVE (DEPARTMENT) | | | |

**REVIEWED BY:**

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| EXECUTIVE/MANAGER (DEPARTMENT) | | | |
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| HEAD (QUALITY ASSURANCE) | | | |

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| STANDARD OPERATING PROCEDURE | |
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| **Department:** Quality Assurance | **SOP No.:** |
| **Title:** Preparation of Validation/Qualification Protocol and Report | **Effective Date:** |
| **Supersedes:** Nil | **Review Date:** |
| **Issue Date:** | **Page No.:** |

## ANNEXURE-V
## MASTER EQUIPMENT/AREA/UTILITY QUALIFICATION DOCUMENTS INDEX

**As On Date:**
**Revision No.:**
**Page No.:**

| S. No. | Area / Equipment / Utility Name | ID No. | DQ Protocol No. | IQ Protocol No. | OQ Protocol No. | PQ Protocol No. | Remarks |
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| --- | Prepared By Officer/Executive | Checked By Department Head | Approved By Head Quality Assurance |
|---|---|---|---|
| **Name** | | | |
| **Signature** | | | |
| **Date** | | | |

| STANDARD OPERATING PROCEDURE | |
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| **Department:** Quality Assurance | **SOP No.:** |
| **Title:** Preparation of Validation/Qualification Protocol and Report | **Effective Date:** |
| **Supersedes:** Nil | **Review Date:** |
| **Issue Date:** | **Page No.:** |

## ANNEXURE-VI
## MASTER VALIDATION/QUALIFICATION/PRODUCT DEVELOPMENT PROTOCOL INDEX

**As On Date:**
**Revision No.:**
**Page No.:**

| S.No. | Protocol Title | Protocol No. | Effective Date | Remarks |
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| --- | Prepared By<br>Officer/Executive | Checked By<br>Department Head | Approved By<br>Head Quality Assurance |
|---|---|---|---|
| **Name** | | | |
| **Signature** | | | |
| **Date** | | | |

| STANDARD OPERATING PROCEDURE | |
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| **Department:** Quality Assurance | **SOP No.:** |
| **Title:** Preparation of Validation/Qualification Protocol and Report | **Effective Date:** |
| **Supersedes:** Nil | **Review Date:** |
| **Issue Date:** | **Page No.:** |

## ANNEXURE-VII
## MASTER VALIDATION/QUALIFICATION/PRODUCT DEVELOPMENT REPORT INDEX

**As On Date:**
**Revision No.:**
**Page No.:**

| S. No. | Report Title | Protocol No. | Report No. | Remarks |
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| --- | Prepared By<br>Officer/Executive | Checked By<br>Department Head | Approved By<br>Head Quality Assurance |
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| **Name** | | | |
| **Signature** | | | |
| **Date** | | | |

| STANDARD OPERATING PROCEDURE | |
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| **Department:** Quality Assurance | **SOP No.:** |
| **Title:** Preparation of Validation/Qualification Protocol and Report | **Effective Date:** |
| **Supersedes:** Nil | **Review Date:** |
| **Issue Date:** | **Page No.:** |

## ANNEXURE-VIII
## PROTOCOL/REPORT ISSUANCE & RETRIEVAL RECORD

**Protocol/Report Title:**

| S. No. | Date | Protocol /Report No. | Revision No. | Report No. | Revision No. | Location | Issued By Sign & Date | Received By Sign & Date | Retrieved On | Retrieved By Sign & Date | Storage Location/ File No. | Remarks |
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