OPERATIONAL QUALIFICATION PROTOCOL FOR COLLOID MILL

PHARMA DEVILS	OPERATIONAL QUALIFICATION PROTOCOL CUM REPORT FOR COLLOIDAL MILL	PROTOCOL No.:

Signing of this Operational Qualification Protocol indicates agreement with the Validation Master Plan approach of the equipment. Further if any changes in this protocol are required, protocol will be revised and duly approved.

PREPARED BY:

Organization	Name	Signature	Date
Engineering			

CHECKED BY:

Organization	Name	Signature	Date
Engineering			

APPROVED BY:

Organization	Name	Signature	Date

PHARMA DEVILS	OPERATIONAL QUALIFICATION PROTOCOL CUM REPORT FOR COLLOIDAL MILL	PROTOCOL No.:

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1. OBJECTIVE

The objectives of this Operational Qualification (OQ) are as follows:

- 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 demonstrate that the system will operate reproducibly and consistently within its operating range.
- To confirm the suitability of the Standard Operating Procedures for all routine activities associated with the system.

Following execution of the protocol a summary report will be written and approved. All results, conclusions, exceptions and variances will be addressed and final disposition of the equipment will be stated. Successful completion of this protocol and approval of the summary report will verify that the Colloid Mill meets all the acceptance criteria and is ready for PQ.

2. SCOPE

- This protocol covers all aspects of Operational Qualification for the Colloid Mill serving Tablets, Capsules, Dry Syrup, Dry Injection and Oral Manufacturing Facility. Scope incorporates qualification of all Colloid Mill components such as Hopper, Three way cock system, Stator Rotor

This protocol will define the methods and documentation used to qualify the Colloid Mill. Successful completion of this protocol will verify that the Colloid Mill meets all acceptance criteria and is ready for Performance Qualification.

3. RESPONSIBILITIES

Engineering Validation Personnel

The following are the responsibilities of Jacobs Engineering Validation Personnel:

- Preparation, Review and submission of OQ Protocol.
- Ensures that the protocol is in compliance with current TPL policies and procedures.
- Ensures that the content is sufficient, clearly defined technically sound and accurate.
- Ensures compliance with design specifications.

The following are the primary responsibilities of the ALL Validation Personnel:

- Overall cGMP compliance for OQ
- Review and Pre-Approval of OQ Protocol
- Execution of this OQ protocol
- Document Control of OQ Protocol until such document is completed, approved and after.
- Regulatory Compliance Review of the completed OQ Protocol
- Review and Approval of the executed OQ Protocol.

4. SYSTEM DESCRIPTION

The Colloid Mill and its associated equipment are designed to process pharmaceutical products in accordance with cGMP principles. The Colloid Mill is used for Homogenizing, Emulsifying, Dispersing
& comminuting of liquids including highly viscous products

Qualification activities for the Colloid Mill incorporate the following system components:

- Hopper
- Three way cock system
- Stator Rotor

5. DOCUMENTATION REQUIREMENTS

The OQ File should include:

- This OQ Protocol.
- All printouts and handouts generated during the qualification procedure.
- Any laboratory test results or their referenced location.

- A Signature Sheet [*Appendix 1*], where all people, performing the qualification tests, are listed.
- Any change control actions that may have occurred during the qualification activities.
- Any variances, exceptions or investigation reports generated during the qualification activities.

6. DATA COLLECTION

All individuals executing this Protocol shall complete the *Signature Sheet [Appendix I]*. All personnel shall have suitable documented training or experience.

All approvals shall be made in *BLACK* ink.
All data entry shall be made in *BLACK* nk.

All corrections to this Protocol, which are not retyped, are to be made in *BLACK* ink. All written corrections to this Protocol or to data entered in this Protocol should be made by using a single line to delete the error. The person who makes the correction shall initial and date it and add comment to explain reason for correction.
After performing the qualification tests, collect all relevant printouts and certificates and retain for inclusion in the OQ File. If more Data Sheets or Variance Sheets are required, they are to be attached to this Protocol as *Appendices* and to be listed in *Section 13. List of Appendices*.

7. CHANGE CONTROL

Any changes or modifications to the system shall be performed in accordance with the TPL Project Change Control Procedure (SOP No: …….).

Change Control Forms raised during the execution of this OQ will be filed along with the protocol. An assessment will be made for each change to determine whether or not any re-validation is required.

8. PRE-QUALIFICATION REQUIREMENTS

The results of any tests should meet the limits and acceptance criteria specified in the test documents. Any deviations (as per BQA-017) or issues should be rectified and documented prior to OQ commencing. Open action items resulting from these tests shall be listed in the Comments section.

8.1 System Pre-requisites

S.No.	Description of Pre-requisite	Completed Yes or No	Verified By	Date
1	Verify that the IQ of the Colloid Mill has been executed and approved. IQ Protocol Document No: IQ /F/CLM-04/00	Yes/No*		
2	Verify that Site Acceptance Tests (SAT) of the Colloid Mill has been executed and approved.	Yes/No*		
3	Verify that the safety walk through has been completed and that the system is safe to use.	Yes/No*		
Verify that authorised drafts of the following procedures (SOP / PMI) relevant to operation of the Colloid Mill are available.				
4	SOP of Colloid Mill Operation	Yes/No*		
5	SOP of Colloid Mill Maintenance.	Yes/No*		
6	SOP of Colloid Mill Cleaning /Washing	Yes/No*		

7	Verify that all critical instruments associated with the system will be in a calibrated state during OQ execution.	Yes/No*		

Note:- * -Circle one, which is appropriate.

8.2 Test Equipment Calibration

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 Name	Equipment Owner	Equipment Number	Due Date	Signature	Date

Reviewed by		Date	

9. TESTS AND CHECKS

9.1 SOP Verification

9.1.1 Purpose

To verify the accuracy of Standard Operating Procedures applicable to the Colloid Mill.

9.1.2 Method

Obtain a controlled copy of each SOP referenced within section 9.1.4. During the course of OQ testing, perform each operation according to the instruction indicated within the appropriate SOP. Mark with a highlighter pen each instruction or statement within the SOP which is verified and in accordance with the actual practice. Write any differences from actual practice in red ink on the copy of the SOP. On completion, write "Operational Qualification - SOP Verification" on the marked-up copy of the SOP, sign & date it and attach as an appendix to the OQ protocol together with any other raw data such as printouts.
Ensure all SOP's identified in Section 9.1.4 are evaluated and checked.

9.1.3 Acceptance Criteria

At the completion of OQ testing, all standard operating procedures referenced within section 9.1.4 will be annotated to correctly reflect the applicable method instruction(s) required to obtain intended operation or function result.

9.1.4 Results

Enter the SOP's into the table below and verify that they have been evaluated and checked. Incorporate the marked up SOPs as an appendix to the OQ report together with any other raw data such as printouts

SOP Number	SOP Description	SOP accurate after check [Y/N]	Initial / Date
	SOP-Colloid Mill Operation & Cleaning		

Comments:

Reviewed by		Date	

9.2 System Start-Up and Shutdown Test

9.2.1 Objective

To verify that the system components will power-up and start as defined by the design documentation.

9.2.2 Method

Follow instructions in the Test Method column of section 9.2.4 to test the start-up and shutdown of each system component. Obtain approval from the Production, Electrical and Mechanical Departments (where applicable) prior to this test and attach the approval slip as an appendix to this protocol. Record all observations in section 9.2.4 and attach any raw data printouts as an appendix to this protocol.

9.2.3 Acceptance Criteria

All Start-up and Shutdown functions operate correctly as specified in the following document:

- *System Operating and Maintenance Manual* Colloid Mill

Specific acceptance criteria for each test are provided in the tables in section 9.2.4.

9.2.4 Results

Test Method	Expected Result	Actual Result	Acceptable [Y/N]	Initial / Date
Shutdown Procedure				
Switch "OFF" the mill by operating green push button situated at back side of mill.	Machine Stops and recurs to safe mode.			
Power-Up and Start Test				
Observe colloid Mill physically	Visual Inspection of colloid mill (discharge spout, three-way cock assembly, hopper, stator rotor)			
Operate cock handle	Cock close			
Switch "ON" the mill by operating green push button situated at back side of mill.	Anticlock wise rotation on top view.			
Adjust milling gap from maximum to minimum	Visual observation on scale "0-15" No.			

Equipment Operated by		Date	

Comments:

Reviewed by		Date	

9.3 System Functionality Tests

9.3.1 Objective

To verify Colloid Mill components functionality.

9.3.2 Method

Prior to this test, power up and start-up each component as described in Section 9.2.4: *Power Up and Start Test*. Operate each item as described in Section 9.3.4 to test the functionality of the system. Record all observations in the Actual Results column in Section 9.3.4.

9.3.3 Acceptance Criteria

All aspects of control for individual components integrated within the Colloid Mill shall function as specified in the expected results column in Section 9.3.4.

9.3.4 Results

Test Method	Expected Result	Actual Result	Acceptable [Y/N]	Initial / Date
Switching on the Power and Utilities to the System				
Switch on the power to Colloid mill	Machine is ready to start.			
Monitor and Log the readings.	Log the following readings: Voltage. 415 ± 10 %Volts			
Colloid Mill & its Components				
Observe colloid Mill physically	Visual Inspection of colloid mill (discharge spout, three-way cock assembly, hopper, stator rotor)			
Check hopper from inside	No foreign material or unwanted things lying there			
Operate cock handle	Cock close			
Switch “ON” the mill by operating green push button situated at back side of mill.	Check the direction of rotation by peeping in hopper in anticlock wise rotation on top view.			
Adjust milling gap from maximum to minimum	Visual observation on scale “0-16” No.			

Equipment Operated by		Date	

Comments:

Reviewed by		Date	

9.4 System Emergency Shutdown Stop

9.4.1 Objective

To verify that the emergency stop function activation shuts down the system in an appropriate manner.

9.4.2 Method

Ensure system is running under normal operating procedures. Press the emergency stop button and follow instructions in the Test Method column in section 9.4.4. Record all observations in the Actual Result column in section 9.4.4 and attach any raw data printouts as an appendix to this protocol.

9.4.3 Acceptance Criteria

Component comprising the system shut down in a safe and controlled manner when the emergency stop button is pressed. All pumps and motors will trip. An alarm condition is registered with audible alarm.

9.4.4 Results

Test Method	Expected Result	Actual Result	Acceptable [Y/N]	Initial /Date
Press Stop Button while the system is running in normal operating mode	The system shuts down in a safe and controlled manner.			

Equipment Operated by		Date	

Comments: **Stop Button acts as Emergency button.**

Reviewed by		Date	

9.5 Valve Operational Test

9.5.1 Objective

To ensure that valves located at throughout the Colloid Mill operates correctly and can be accessed safely.

9.5.2 Method

Locate each valve listed in Section 9.5.4. Perform the test by manually opening and closing the valve. Verify that all valves can be accessed safely and that each valve can be fully opened and closed. Record results following testing in section 9.5.4.

9.5.3 Acceptance Criteria

Each valve can be accessed safely.
Each valve can be operated at full open and full closed positions.

9.5.4 Results

Valve Check	Expected Result	Valve Tag No	Actual Result	Acceptable [Y/N]	Initial / Date
Verify that each valve can be assessed safely. Verify that each valve operates and seals correctly.	Valve can be accessed safely. Valves operate and seal correctly.	Three way Cock Valve			

Equipment Operated by		Date	

Comments:

Reviewed by		Date	

9.6 Confirmation of Critical Parameter and Full Function Testing

9.6.1 Objective

To confirm that the critical parameter and full function of the Colloid Mill are as defined below:-

- Non-metallic contact parts such as gaskets 'O' rings and other elastomers coming in contact with the product is of food grade quality.
- The lubricants used are of food grade and they do not come into contact with product or product contact parts.
- Facility is available for setting and varying gap between rotor & stator

9.6.2 Method

Follow the test methods described in section 9.6.4 for various parameters under test.
Record the observation in 9.6.4 actual results column.
Attach supporting documents, as applicable, in the appendix.

9.6.3 Acceptance Criteria

The critical operational parameters and full function testing on the Colloid Mill has been identified and completed satisfactorily.

9.6.4 Results

Test Method	Expected Result	Actual Result	Acceptable [Y/N]	Initial / Date
Product contact Parts				
O Rings & gaskets to be of Food Grade – Verification of Test Certificates	Material to Confirm Food Grade quality			
Lubricants are Food Grade & does not come in contact with the Product				
Visual Inspection & test certificates from Vendor	Lubricants are Food Grade & does not come in contact with the Product			
Setting and Varying gap between Rotor & Stator				
Adjust milling gap from maximum to minimum	Visual observation on scale "0-15" No.			

Equipment Operated by		Date	

Comments:

Reviewed by		Date	

9.7 Loss of Utilities

9.7.1 Objective

To verify the loss of utilities supplies will not affect or damage the Colloid Mill and that the subsequent return of any failed utility does not pose a threat to the system, the system's operator and the product quality.

9.7.2 Method

Check the electrical supply with voltmeter.

9.7.3 Acceptance Criteria

The Colloid Mill shall raise an alarm and revert to the scenario's listed in the results section below on the isolation of:

9.7.4 Results - Not Applicable

Test method	Expected Result	Actual Result	Acceptable [Y/N]	Initial/Date

Equipment Operated by		Date	

Comments:

Reviewed by		Date	

10. CHECKLIST OF ALL TESTS AND CHECKS

This checklist is provided to ensure that all tests or checks required for this protocol have been executed.

Reference No.	Tests or Checks	Executed [Y/N]	Comment
9.1	SOP Verification		
9.2	Colloid Mill System Start-Up and Shutdown Test		
9.3	Colloid Mill System Functionality Test		
9.4	Colloid Mill Emergency Shutdown Test		
9.5	Valve Operational Test		
9.6	Conformation of critical parameters		
9.7	Loss of Utilities		Not Applicable

Comments:

Reviewed by		Date	

11. VARIANCE SHEET

Report any deviations from the acceptance criteria or exceptions from protocol instructions in the Record Sheet as described in SOP:" Handling Of Deviations" Record the total number of exceptions / deviations reported during the qualification activities of this Protocol. Record the Deviation Number and Title in the Table below. Include all Deviation Record Sheets in the IQ File.

TOTAL NO. OF EXCEPTIONS / DEVIATIONS = _____

Exception / Deviation No.	Exception / Deviation Title	Status

Comments:

Reviewed by		Date	

12. REFERENCES

The Principle Reference is the following

- Master Validation 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.

The following references are used to give addition guidance

- FDA/ISPE Baseline Pharmaceutical Engineering Guide-Volume 5:- Commissioning and Qualification Guide, First Edition / March 2001.
- Code of Federal Regulations (CFR), Title 21, Part 210, *Current Good Manufacturing Practice (cGMP) in Manufacturing, Processing, Packing, or Holding of Drugs*, General. April 1, 1998.
- Code of Federal Regulations (CFR), Title 21, Part 211, *Current Good Manufacturing Practice (cGMP) for Finished Pharmaceuticals*, April 1, 1998.
- EU Guide to Good Manufacturing Practice, Part 4, 1997.
- European Commission's working party on control of medicines and inspections document, *Validation Master Plan, Design Qualification, Installation & Operational Qualification, Non Sterile Process Validation, Cleaning Validation*, October 1999.
- GAMP Guide, Validation of Automated Systems in Pharmaceutical Manufacture, Version 4.0, December 2001.
- SOP -"Handling of Deviations".
- SOP -"Change Control Procedure".

13. LIST OF APPENDICES

Appendix No.	Document Title

14. SUMMARY (TPL BADDI)

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15. APPROVALS

The following approvals signify that the OQ is complete and acceptable and that the system is ready for PQ Execution.

EXECUTED BY:

Organization	Name	Designation	Signature	Date

REVIEWED BY:

Organization	Name	Designation	Signature	Date

APPROVED BY:

Organization	Name	Designation	Signature	Date