PHARMA DEVILS

QUALITY ASSURANCE DEPARTMENT

VALIDATION PROTOCOL FOR LUBRICATION OF INGREDIENTS IN PLANETARY MIXER

1. Objective:

To validate the process of Lubrication of Ingredients, so as to establish that the blend uniformity of active ingredient achieved at standard Lubrication time (as given in BMR) and the physical parameters like Pour Bulk Density, Tapped Density, Loss on drying/Water content and particle size distribution of the final Lubricated Granules are within the specified ranges as per BMR.

2. Scope:

Applicable to all lubrication operation in PLM.

3. Principle:

Lubrication of raw materials due to rotation of Stirrer in Planetary Mixer.

4. Site of the Study:

Hormone Department.

5. Responsibility:

Production :

Quality Control :

Engineering :

Quality Assurance:

(Individuals to be named in the report)

6. Description of the Equipment to be used:

PLANETARY MIXER: λ

CODE No. : Details to be recorded in the report.

CAPACITY : Details to be recorded in the report.

RPM (STIRRER) : Details to be recorded in the report.

λ *Date of Equipment Qualification to be recorded in the Report.*

Sampling thief and Bulk sampler Code No. to be recorded in the validation report.

7. BMR and Standard Operating Procedure (SOP) to be followed:

i) SOP for operating Octagonal Blender : To be recorded in Validation Report
ii) SOP for sampling with Sampling thief : To be recorded in Validation Report
iii) SOP for checking Loss On Drying (LOD) : To be recorded in Validation Report
iv) SOP for Pour Bulk and Tapped density : To be recorded in Validation Report
v) SOP for water content (if applicable) : To be recorded in Validation Report
vi) SOP for Particle Size Distribution : To be recorded in Validation Report
vii) Batch Manufacturing Record : Formulation Code No. and Manufacturing Code No. To be recorded in Validation Report.

8. Control:

8.1 Requirement:

i. Active Pharmaceutical Ingredient to be used should meet the requirement of Specification (Name of the active ingredient, Quantity required, Pharmacopoeia grade and the test involved for raw material testing with result and Analytical Reference number should be recorded in Validation report).

ii. Validated Analytical Methods for estimation of active ingredient (Reference Analytical Validation protocol number / Reference Specification No. to be recorded in the report).

iii. Analytical Reference number for validation Technical Information Sheet (T. I. Sheet).

8.2 Calibration:

i) Calibration of RPM & Timer of Planetary Mixer. Details to be recorded in the report.

ii)Calibration of test apparatus. Details to be recorded in the report.

8.3 Training:

Availability of Training Record of Personnel involved in the Validation exercise should be recorded in the Validation Report

8.4 Precaution:

Safety aspects while operation of equipment and process must be ensured.

9. Validation Procedure:

9.1 Load the sifted ingredients in the Planetary Mixer as per the sequence given in the Batch Manufacturing Record (BMR).

9.2 Operate the Planetary Mixer as per SOP.

9.3 Sample one to three unit doses in triplicate from 10 positions at various depths as shown in sampling plan on Page 6, with the help of a sampling thief, at standard lubrication time specified in the BMR. Send these samples to Quality Control for Content Analysis.

9.4 Sample approximately 40 gm from top, middle, bottom position of the Planetary Mixer at standard lubrication time. Make a composite sample of 120 gm.

100gm for Particle size distribution
10 gm for pour bulk density and Tapped density.
2 to 3 gm for LOD
5 g for water content (Q.C. analysis)(If applicable)

9.5 The results should be reported as Individual test results and calculate % RSD.

9.6 Check the appearance of lubricated granules after completion of lubrication operation and record the same in report.

10. Acceptance criteria:

The Optimal time decided should conform to the following:

10.1 Uniform distribution of the lubricated granules, when checked visually.

10.2 Content of Active Ingredient upon testing as per Quality Control Specification should be within the specified limit.

10.3 The Relative Standard Deviation (RSD) of content of Active Ingredient sampled at the optimal time should be less than 5%.

10.4 Result of physical parameters of the lubricated granules sampled at the standard lubricated time (as given in BMR)

11. Non Compliances:

Details of deviations (including justification of acceptance) done to successfully carry out the validation exercise and any OOS results obtained should be checked. (Attach the details in the Validation report).

12. Type of Validation:

Concurrent Validation/Re-validation

13. Frequency:

1) Concurrent validation : Three consecutive successful validation exercises.
2) Re-validation (Periodic) : One validation exercise within five years.
3) Revalidation (after major change): Three consecutive successful validation exercises.

14. Results/Observations:

Record the observations during the study and results obtained from Quality Control Department in the Validation Report.

15. Summary of findings of experiment (inference):

Summarize the findings of the Validation Study to draw an inference.

16. Recommendation (Including requirements of any additional Documentation):

Record the recommendations based on the interpretation of the results of the Validation Report.

17. Team approval:

The individuals who have performed the Validation Study, supervised the validation, completed the records, performed the testing of the product should approve the validation report.

18. Review (inclusive of follow up action, if any):

The Validation Report should be reviewed by Unit Quality Assurance and Unit Head. The report should include any follow up action if required.

19. Approved by:

Unit Quality Assurance and Unit Head should finally approve the validation Report.

20. Attachments:

Annexure (if any) attached to the Validation Report should be recorded.

21. Abbreviations:

VP	: Validation Protocol
Lt.	: Litres
No.	: Number
A.R.No.	: Analytical Reference Number
RSD	: Relative Standard Deviation
mg	: Milligram
OOS	: Out of Specification
%	: Percent
SOP	: Standard Operating Procedure
BMR	: Batch Manufacturing Record
RPM	: Revolution Per Minutes

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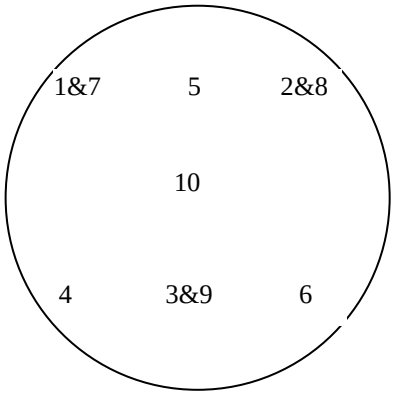
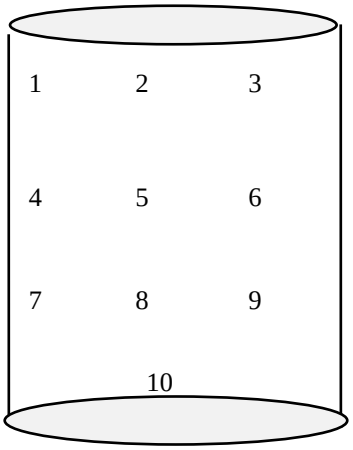
SAMPLING PLAN FOR BLENDING/LUBRICATION

TOP VIEW OF PLANETARY MIXER (PLM)



SIDE VIEW OF PLANETARY MIXER



SAMPLING POSITIONS

POSITION 1,2 & 3 : Top Layer of the bed.

POSITION 4,5 & 6 : 0.5 feet from top layer of the bed

POSITION 7,8 & 9 : 1 feet from top layer of the bed

POSITION 10 : Bottom centre where the tip touches the PLM.