VALIDATION PROTOCOL FOR LUBRICATION OF INGREDIENTS IN OCTAGONAL BLENDER

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 Lubrication of ingredients in Octagonal Blender.

3. Principle:

Lubrication of ingredients due to tumbling action in the Octagonal Blender.

4. Site of the Study:

Hormone Department.

5. Responsibility:

Production :
Quality Assurance :
Quality Control :
Engineering :
(Individuals to be named in the report)

6. Description of the Equipment to be used:

OCTAGONAL BLENDER λ
CODE No. : *To be recorded in Validation Report*
CAPACITY : *To be recorded in Validation Report*
RPM : *To be recorded in Validation Report*
λDate of Equipment Qualification to be recorded in the Report.

Sampling thief, Sampling bullet and Bulk sampler Code No. to be recorded in the Validation report.

7. Standard Operating Procedure (SOP) & Batch Manufacturing Record (BMR) 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. Controls:

8.1 Requirements:

i. Active Pharmaceutical Ingredient to be used should meet the requirement of Specification (Name of the active ingredient, Quantity required, Pharmacopoeial

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 Octagonal Blender. 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 Report.

8.4 Precautions:

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

9. Validation Procedure:

9.1 Load the Ingredients in the Octagonal Blender as per the sequence given in the Batch Manufacturing Record (BMR).

9.2 Operate the Octagonal blender as per SOP.

9.3 Sample one to three unit doses in triplicate from 10 positions at various depths as shown in the sampling plan on Page No. 6 with the help of a sampling thief at standard lubrication time as specified in the BMR. Send these samples to Quality Control for analysis of blend uniformity.

9.4 Sample approximately 40 gm from top, middle, bottom position of the octagonal blender 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 of content should be reported as Individual test results.

9.6 % RSD should be calculated for the results of content. (To be done by QC)

9.7 Check the appearance of lubricated granules visually after lubrication operation and record the observations in the report.

10. Acceptance criteria:

The Optimal time decided should confirm to the following:

10.1 Uniform distribution of the blend (Visual Inspection).

10.2 Content of Active Ingredient upon testing at intervals of standard lubrication time as per Quality Control Specification should be within limits.

10.3 Relative Standard Deviation (RSD) of blend uniformity at standard lubrication time (as given in BMR) should be less than 5%.

10.4 Result of physical parameters of the lubricated granules sampled at the standard 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:

1) Concurrent validation
2) Re-validation

13. Frequency:

1) Concurrent validation : Three consecutive validation exercises.
2) Re-validation (Periodic) : One validation exercise – should not exceed five years
3) Revalidation (after major change): Three consecutive validation exercises.

14. Results/Observation:

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 follow up action if required.

19. Approved by:

Validation Report should be finally approved by Unit Quality Assurance and Unit Head.

20. Attachment:

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

21. Abbreviations:

VALIDATION PROTOCOL FOR LUBRICATION OF INGREDIENTS IN OCTAGONAL BLENDER

Lt.	: Litres
No.	: Number
A.R.No.	: Analytical Reference Number
RSD	: Relative Standard Deviation
Gms.	: Grams
OOS	: Out of Specification
SOP	: Standard Operating Procedure
BMR	: Batch Manufacturing Record
T. I. Sheet	: Technical Information Sheet
API	: Active Pharmaceutical Ingredient
RPM	: Revolution Per Minutes

PHARMA DEVILS

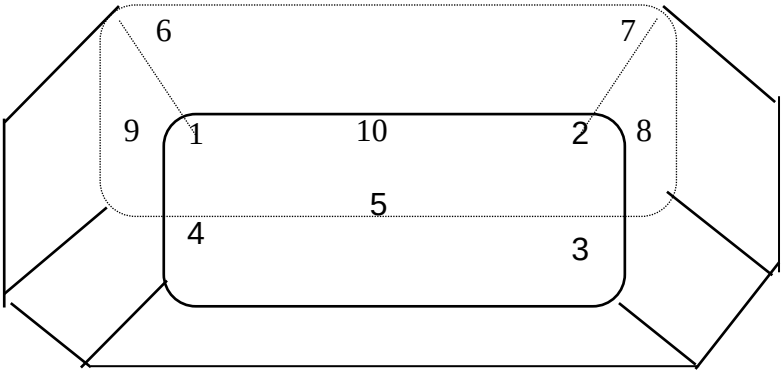
QUALITY ASSURANCE DEPARTMENT

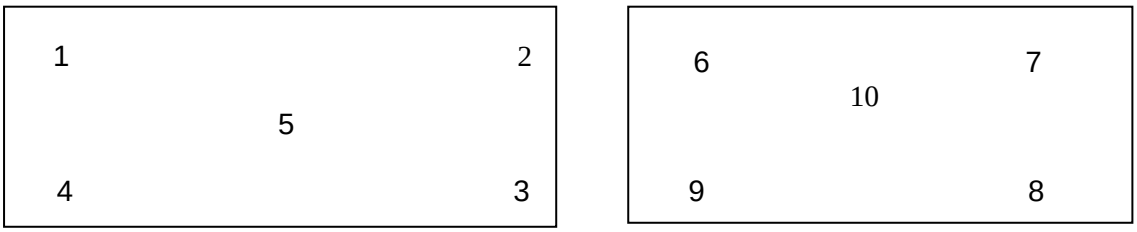
VALIDATION PROTOCOL FOR LUBRICATION OF INGREDIENTS IN OCTAGONAL BLENDER

SAMPLING POSITIONS FOR LUBRICATION VALIDATION

Vertical cross-section of the Octagonal Blender:

Sample the lubricated granule at various depths and positions (Positions as shown below) Using a sampling thief. Follow SOP No. CQA-57 for usage of the sampling thief.





TOP LAYER BOTTOM LAYER

Position 1,2, 3 & 4 : Top layer of the bed towards the periphery.

Position 5 : Middle layer of the bed at centre.

Position 6,7,8 & 9 : Bottom layer of the bed towards the periphery.

Position 10 : Bottom layer of the bed at centre.

NOTE:

1. **Depth for sampling should be decided depending on the respective blender capacity.**
2. **Total height of material bed should be established and based on actual height of bed; sampling layer should be decided as Top (25%), Middle (50%) and Bottom (75%)**