



PHARMA DEVILS
QUALITY ASSURANCE DEPARTMENT

PROCESS VALIDATION REPORT FOR SUGAR SPHERE

**PROCESS VALIDATION REPORT
FOR
SUGAR SPHERE [FILM COATED (#18#24)]**



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1.0 SUMMARY PLAN OF STUDY:

1.1 PRODUCT AND BATCH DETAILS:

Process Validation of **Sugar Sphere [Film Coated (#18#24)]** was carried out on three consecutive batches. The details are as under.

BATCH DETAILS:

S. No.	BATCH NUMBER	BATCH SIZE	MFG. DATE	EXP. DATE	COMMENCEMENT DATE	COMPLETION DATE
1		150.0 KG				
2		150.0 KG				
3		150.0 KG				

1.2 PRODUCT DETAILS:

Generic Name	Sugar Sphere [Film Coated (#18#24)]
License No.
Product Code
Description	White to off white round spherical film coated pellets
Shelf Life	48 Months
Batch Size	150.0 Kg
Storage Condition	Store in dry place, below 30 ⁰ C



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2.0 REFERENCE DOCUMENTS

S.No.	DOCUMENTS	SPECIFICATION No.
1.	Master Formula Record	
2.	Batch Manufacturing Record	
3.	Finished Product Specification	

3.0 MANUFACTURING EQUIPMENT AND ACCESSORIES USED IN THE PROCESS:

The various equipment's used for the Process Validation of **Sugar Sphere [Film Coated (#18#24)]** are as follows:

S.No.	EQUIPMENT	EQUIPMENT NUMBER
1.	Sifter	
2.	Fluid Bed Coater	
3.	Multi MILL	
4.	Semi Auto Coater 48"	
5.	Solution Preparation vessel	
6.	Solution Storage Tank	
7.	Tray Drier	
8.	Tray Drier	

All critical equipment's were verified for their Installation, Operational and Performance Qualification. Further all the equipment was cleaned and operated as per relevant SOP's as indicated in the Process Validation Protocol.



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4.0 ANALYTICAL REPORT NUMBERS AND QUANTITY OF ALL RAW MATERIALS:

RAW MATERIAL	UOM	STD. QTY./ 150.0 KG	BATCH NO.					
			QTY USED	A.R NUMBER	QTY USED	A.R NUMBER	QTY USED	A.R NUMBER
Sugar (Powder) #300	kg	97.845	97.845		97.845		97.845	
Maize Starch	kg	21.000	21.000		21.000		21.000	
PG Sugar #40#50	kg	30.780	30.780		30.780		30.780	
Povidone (K-30)	kg	0.375	0.375		0.375		0.375	
Purified Water	kg	15.000	15.000		15.000		15.000	
Coating Materials								
HPMC E-5	kg	4.500	4.350		4.380		4.365	
\$ Purified Water	kg	72.000	69.600		70.080		69.840	



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5.0 STAGE WISE ENVIRONMENTAL CONDITIONS:

S.No.	STAGE	BATCH NUMBER					
		Temp. (°C)	RH (%)	Temp. (°C)	RH (%)	Temp. (°C)	RH (%)
1.	Dispensing	23.0°C	48.0%	21.0°C	49.0%	21.0°C	45.0%
2.	Sugar sphere manufacturing stage	24.0°C	46.0%	23.0°C	48.0%	24.0°C	46.0%
3.	Coating stage	23.0°C	48.0%	24.0°C	49.0%	24.0°C	49.0%

6.0 EXPERIMENTAL PLAN, RESULTS AND DISCUSSION:

The following critical parameters were monitored in this study:

PROCESS PARAMETERS:

- In Process check parameters like Appearance and particle size after sugar sphere formation.
- Analysis after coating stage of sugar sphere as per specification.

ENVIRONMENTAL CONDITIONS:

- HVAC system as well as LAF units was conforming for Installation, Operational and Performance Qualification. Differential Pressure as well as Viable counts in HVAC system and LAF units were monitored on regular basis as per relevant protocols and the same were found to be conforming to specifications.
- Purified water plant, holding tanks and distribution loops were qualified as per the protocol and the same were found to be conforming to specifications.



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6.1 PROCESS PARAMETERS RESULTS:

6.1.1 MANUFACTURING OF SUGAR SPHERE:

6.1.1.1 Milling of sugar (Powder) observations:

Specified	Standard	Observation
Screen size / integrity	0.5 mm	Complies

6.1.1.2 In process checks during sugar sphere manufacturing:

Parameters	Limits	Observations		

In process checks during sugar sphere formation stage

Pan RPM	1-15 RPM	09-12 RPM	09-12 RPM	09-12 RPM
Peristaltic pump RPM	5-15 RPM	06-11 RPM	06-11 RPM	06-11 RPM
Atomizing air pressure	0.5-1.0 kg/cm ²	0.5 kg/cm ²	0.5 kg/cm ²	0.5 kg/cm ²

Observations of drying (In tray drier)

Inlet temperature	40 °C -50 °C	40 °C -45 °C	40 °C -45 °C	40 °C -45 °C
LOD (after final drying)	1.0%-1.5%	1.2%	1.2%	1.2%



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6.1.1.3 Sizing/sifting of uncoated Sugar Sphere

Sifting of sugar sphere through #18#24

Specified	Standard	Observation		
Sieve size	#18	Complies	Complies	Complies
Sieve size	#24	Complies	Complies	Complies
Sieve integrity	Should be ok	Complies	Complies	Complies

6.1.2 Coating Stage/ Process (In FBC)

6.1.2.1 In process checks of Coating Stage

Parameters	Limits	Observations		
In process checks during sugar sphere formation stage				
Inlet temperature	40 °C -50 °C	42 °C -46.2 °C	41.8 °C -46.0 °C	42 °C -45 °C
Bed temperature	35 °C -40 °C	35 °C -39.2 °C	35 °C -38 °C	35 °C -40 °C
Peristaltic pump RPM	15-20 RPM	18-20 RPM	18-20 RPM	19-20 RPM
Atomizing air pressure	2.0-2.5kg/cm ²	2.0 kg/cm ²	2.0 kg/cm ²	2.0 kg/cm ²
Blower RPM	2000-2500 RPM	2050 RPM	2050 RPM	2050 RPM



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6.1.2.2 Sizing/sifting of coated Sugar Sphere:

Sifting of sugar sphere through #18#24

Specified	Standard	Observation		
Sieve size	#18	Complies	Complies	Complies
Sieve size	#24	Complies	Complies	Complies
Sieve integrity	Should be ok	Complies	Complies	Complies

6.1.2.3 Results of different locations from FBC:

6.1.2.3.1 Results of 1st batch

Batch no.			
Sampling locations	Description (White to off white round spherical pellets)	LOD (1.0%-1.5%)	Assay (NMT 92% of sucrose)
1	Complies	1.42%	89.38%
2	Complies	1.38%	89.61%
3	Complies	1.40%	89.56%
4	Complies	1.37%	89.58%
5	Complies	1.42%	89.58%



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6.1.2.3.2 Results of 2nd Batch

Batch no			
Sampling locations	Description (White to off white round spherical pellets)	LOD (1.0%-1.5%)	Assay (NMT 92% of sucrose)
1	Complies	1.38%	91.07%
2	Complies	1.46%	90.79%
3	Complies	1.27%	90.42%
4	Complies	1.25%	91.06%
5	Complies	1.34%	90.56%

6.1.2.3.3 Results of 3rd batch:

Batch no			
Sampling locations	Description (White to off white round spherical pellets)	LOD (1.0%-1.5%)	Assay (NMT 92% of sucrose)
1	Complies	1.29%	90.65%
2	Complies	1.18%	90.89%
3	Complies	1.21%	90.76%
4	Complies	1.32%	90.35%
5	Complies	1.27%	90.54%



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6.1.2.4 Results of final Sugar Sphere:

PARAMETER	ACCEPTANCE CRITERIA			
Description	White to off White pellets	Complies	Complies	Complies
Identification				
By TLC	The principle spot in the chromatogram obtained with the test solution is similar in the position, color and size to the principle spot in the chromatogram obtained with reference solution	Complies	Complies	Complies
By Chemically	A dark-blue color is produced, which disappears on heating	Complies	Complies	Complies
By Chemically	An orange precipitate is formed immediately.	Complies	Complies	Complies
Solution S	Filter under vacuum to obtain a clear solution	Complies	Complies	Complies
Pellet size	NLT 90% is passed through 18# NLT 90% is retained on 24#	98.18% 96.86%	98.14% 97.43%	99.91% 92.64%
LOD	NMT 5.0%	1.26%	1.25%	1.28%
Sulphated Ash	NMT 0.2%	0.05%	0.03%	0.03%
Bulk density	0.55 g/ml-0.80 g/ml	0.774 g/ml	0.775 g/ml	0.775 g/ml
Heavy metals	NMT 5 ppm	Less than 5 ppm	Less than 5 ppm	Less than 5 ppm
Assay	NMT 92% of sucrose, calculated on the dried basis.	90.69%	90.87%	90.32%
MICROBIAL LIMITS:				
1. TAMC	NMT 500 cfu/gm	30 cfu/gm	20 cfu/gm	30 cfu/gm
2. TYMC	NMT 50 cfu/gm	Less than 10 cfu/gm	Less than 10 cfu/gm	Less than 10 cfu/gm
Pathogens 1.E. Coli 2.Salmonellae	Absent/gm Absent/10 gm	Absent/gm Absent/10 gm	Absent/gm Absent/10 gm	Absent/gm Absent/10 gm



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The formalized, final 3-batch validation sequence provides the necessary process validation document required to show product reproducibility and a manufacturing process in a state of control.

The test data and results show process reproducibility and consistency among validated batches of **Sugar Sphere [Film Coated (#18#24)]**. Description, LOD, assay, Particle size, have been addressed in in process or testing. All the parameters fall well within in-house acceptance criteria for **Sugar Sphere [Film Coated (#18#24)]**.

Testing has been sufficient to establish process reproducibility and demonstrate, with a high degree of certainty that the product, **Sugar Sphere [Film Coated (#18#24)]** And process are under control.

7.0 STAGE WISE YIELD VARIFICATION:

S.No.	STAGE	ACCEPTANCE CRITERIA OF YIELD (IN %)	OBSERVED YIELD (IN %)		
			BATCH NO.		
1	Coating Stage	NLT 95.00 %	95.23%	96.100%	96.81%

8.0 REASON OF VALIDATION:

To establishing documented evidence which provide a high degree of assurance that the process will consistently produce a product meeting its predetermined specifications and quality attributes.

9.0 CONCLUSION:

Based on the analytical data and comparative results of the process step of three batches, All the critical quality attributes involved in the manufacturing are acceptance criteria, ensure the desired product quality. It's concluded that process validation of **Sugar Sphere [Film Coated (#18#24)]** is validated and process is under control.

10.0 RECOMMENDATIONS :

All the process parameters and chemical results found satisfactory hence no additional recommendation required.



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11.0 REPORT APPROVAL:

PREPARED BY

S.No.	NAME	DESIGNATION	SIGNATURE	DATE

REVIEWED BY

S.No.	NAME	DESIGNATION	SIGNATURE	DATE

APPROVED BY

S.No.	NAME	DESIGNATION	SIGNATURE	DATE

AUTHORISED BY

S.No.	NAME	DESIGNATION	SIGNATURE	DATE