

QUALITY ASSURANCE

# PROCESS VALIDATION REPORT OF CALCITRIOL & MULTIVITAMINS SOFTGEL CAPSULES

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#### 1.0 REPORT APPROVAL:

Function	Department	Name	Signature	Date
Prepared By	Quality Assurance			
	Production			
Reviewed By	Quality Control			
	Engineering			
Approved Pro	Head - Production			
Approved By	Head - QA			



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#### 2.0 VALIDATION TEAM\*:

Department	Name	Designation	Signature	Date

#### **BATCHES UNDER VALIDATION**

Sr. No	Batch No.	Manufacturing Date	Expiry Date
1			
2			
3			

<sup>\*</sup> The validation team includes production. QA, QC, and engineering as applicable

**Reference Validation Protocol No.: ......** 



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3.0 INTRODUCTION:	
Calcitriol & Multivitamins Softgelatin Capsules has been developed using	the Soft Gel Encapsulation
Technology. The batch conducted during the Validation exercise shall be setup	for stability study, and other
parameters monitored periodically and shall reviewed by the validation task force	ee.
OBJECTIVE:	
The objective of this exercise is to develop a <b>PROCESS VALIDATION REP</b>	ORT to demonstrate that the
critical operation involved in the manufacturing process of product are capab	
batches which meet the limits of acceptance criteriaa.	ne of consistently producing
batches which meet the limits of acceptance criteriaa.	
JUSTIFICATION FOR VALIDATION:	
MONITORING:	
All the critical unit operation, process parameter specified in the Batch Manufac	eturing Record monitored and
results are recorded in the validation.	
TYPE OF VALIDATION:	



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#### 4.0 EQUIPMENT CALIBRATION / QUALIFICATION RECORD

#### **BATCH NO.:**

Equipment	Location	Equipment No.	In Calibration / Qualification	Calibration / Qualification Due	Recorded By/Date	Checked By/Date
Reverse Laminar Air Flow	Dispensing Area					
Balance	pensii					
Balance	Dis					
Balance						
Balance						
Balance	Area					
Balance	Production Area					
Balance	Produ					
Balance						
Gelatin Cooking Vessel	Gelatin mass manufacturing area					
Gelatin Feeding Tank	Gelati					



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Batch Size: BMR No.:							
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Equipment	Location	Equipment No.	In Calibration / Qualification	Calibrati Qualificati Due		Recorded By/Date	Checked By/Date
Gelatin Feeding Tank							
Hotplate							
Colloidal Mill							
Sieves 60#, 100#,80#	ë						
Medicament mixing unit.	Medicament preparation area						
Medicament Holding Vessel	para						
Hot Plate	t pre						
Colloidal Mill	men						
Vibro Sifter	edica						
Ultrasonic Sonicator	$\Sigma$						
Encapsulation M/C	ion						
Tumbler Dryer	Encapsulation area						
Degreasing Pan	Enca						
Capsule Inspection Table	ecti						
Capsule shorting Machine	Inspecti on area						
Reviewed by : _				Date:			
	Va	llidation					



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#### **BATCH NO.:**

Equipment	Location	Equipment No.	In Calibration / Qualification	Calibration / Qualification Due	Recorded By/Date	Checked By/Date
Reverse Laminar Air Flow	Dispensing Area					
Balance	pensii					
Balance	Dis					
Balance						
Balance						
Balance	Area					
Balance	ction					
Balance	Production Area					
Balance	[					
Gelatin Cooking Vessel	Gelatin mass manufacturing area					
Gelatin Feeding Tank	Gelatii manufactı					



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CAPSULES			
Batch Size:	BMR No.:		
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Equipment	Location	Equipment No.	In Calibration / Qualification	Calibration / Qualification Due	Recorded By/Date	Checked By/Date
Gelatin Feeding Tank						
Hotplate						
Colloidal Mill						
Sieves 60#, 100#,80#	ea					
Medicament mixing unit.	on ar					
Medicament Holding Vessel	Medicament preparation area					
Hot Plate	t pre					
Colloidal Mill	men					
Vibro Sifter	edica					
Ultrasonic Sonicator	M					
Encapsulation M/C	tion					
Tumbler Dryer	Encapsulation area					
Degreasing Pan	Enca					
Capsule Inspection Table	Inspection area					
Capsule shorting Machine	Inspe					

Reviewed by :		Date:
	Validation	



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CAISULES			
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#### **BATCH NO.:**

BATCH NO			In	Calibration /		
Equipment	Location	Equipment No.	Calibration / Qualification	Qualification Due	Recorded By/Date	Checked By/Date
Reverse						
Laminar Air Flow						
	Dispensing Area					
Balance	pensii					
Balance	Dis					
Balance						
Balance						
Balance	Area					
Balance	ction					
Balance	Production Area					
Balance	, ,					
Gelatin						
Cooking Vessel	ss ; area					
. 65561	in ma turing					
Gelatin Feeding Tank	Gelatin mass manufacturing area					



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CAI	SULES
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Equipment	Location	Equipment No.	In Calibration / Qualification	Calibration / Qualification Due	Recorded By/Date	Checked By/Date
Gelatin Feeding Tank						
Hotplate						
Colloidal Mill						
Sieves 100#,80#	a					
Medicament mixing unit.	Medicament preparation area					
Medicament Holding Vessel	parat					
Hot Plate	t pre					
Colloidal Mill	men					
Vibro Sifter	edica					
Ultrasonic Sonicator	M					
Encapsulation M/C	ion					
Tumbler Dryer	Encapsulation area					
Degreasing Pan	Enca					
Capsule Inspection Table	Inspection area					
Capsule shorting Machine	Inspe					

Reviewed by:	Date:
Valida	tion



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#### **5.0 RAW MATERIAL RATIONALE:**

The raw materials shall be tested to ensure that the materials are of the acceptable quality prior to their use in the manufacturing. All the excipients shall be tested as per the Pharmacopoeial monograph of respective excipients. The details of reference monographs, vendor and analytical reference number are given below:

	Reference			A. R. No.			
Raw Material	Monograph	Name of vendor	B. No.:	B. No.:	B. No.:		
Gelatin IP (200 Bloom)							
Glycerin IP							
Sorbitol Solution (70 %) (Non-Crystallising) IP							
Methyl Paraben IP							
Propyl Paraben IP							
Fumaric Acid IP							
Colour Ponceau 4R Supra IHS							
Titanium Dioxide IP							
Purified Water IHS*							
Calcitriol IP @							
Calcium Carbonate IP (BD-NLT 1g/ml)							
Vitamin K2-7 IHS #							
Mecobalamin JP £							
Magnesium oxide heavy IP							
Zinc Sulphate Monohydrate BP							
L-Methyl Folate Calcium IHS \$							



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	Reference			A. R. N	0.
Raw Material	Monograph	Name of vendor	B. No.:	B. No.:	B. No.:
Natural Vitamin E IHS					
Methyl Paraben IP					
Propyl Paraben IP					
White Bees Wax IP					
Hydrogenated Vegetable Oil BP (Lubritab)					
Butylated Hydroxytoluene IP					
Butylated Hydroxyanisole BP					
Lecithin USP-NF					
Disodium Edetate IP					
Citric Acid IP (Anhydrous)					
Refined Soya Oil BP					
Medium Chain Triglycerides BP (Hariol-538)					
Refined Soya Oil BP (For Fill Weight Setting & Machine Flush)					
Light Liquid Paraffin IP (For Machine Lubrication & Ribbon Lubrication)					



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ch Size:					BMR No	).:
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6.0 USAGE OF RA	W MATERI	AL (ACTIVE)				
Active Material	B.N	lo. A.	R. No.	API	B.No.	Assay (%)
Calcitriol						
						Limit: NLT 97.0 & NMT 103.0
Active Material	B.No.	A. R. No.	API B.N	o. A	ssay (%)	LOD (%,w/w
Calcium Carbonate						
				I	Limit: Between 0 to 100.5	Limit: NMT 2.0 % (w/w)
Active Material	B.No.	A. R. No.	API B.N	0.	Assay	LOD (%,w/w
Vitamin K2-7						
					Limit: LT 2500	Limit: NMT 8.0 % (w/w)



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		CAPSUL	E S		
Batch Size:				BMR No.	
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Active Material	B.No.	A. R. No.	API B.No.	Assay (%)	Water
Mecobalamin					(%,w/w)
				Limit: NLT 98.0	Limit: NMT 12.0 % (w/w)
Active Material	B.No.	A. I	R. No.	API B.No.	Assay (%)
Zinc Sulphate Monohydrate					
					Limit: NLT 99.0 to NMT 101.0
Active Material	B.No.	. A. I	R. No.	API B.No.	Assay (%)
Heavy Magnesium oxide					

Limit: NLT 98.0 to NMT 100.5



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			1		T
Active Material	B.No.	A. R. No.	API B.No.	Assay (%)	Water (%,w/w)
L-Methyl folate					
L-Wethyl Iolate					
				Limit: NLT 95.0	Limit: NMT 25.0 % (w/w)
Reviewed by :	Validatio		Date	e:	



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CAPSILES	

CAISULES				
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#### 7.0 GELATIN MANUFACTURING PROCESS PARAMETERS & RESULTS:

#### 7.1 Gelatin mass manufacturing parameter and results

Hot water circulation Temp.							
Batch No.	D (	Gelatin	<b>Process Time</b>		Observed	Recorded By	
Datch No.	Date	Preparation	Start	End	Temp.	(Sign & Date)	

Reviewed by:_		Date:
-	Validation	



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CAPSIII ES	

CALSULES				
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#### FREQUENCY FOR EVERY 60 MIN

				Observation			Recorded	
Sr. No.	Date	Time	Vacuum Applied in mm Hg	Gelatin Cooking Vessel Temp.	Gelatin Mass Temp.	Stirrer Speed	Appearance of gelatin	By (Sign & Date)
Batch No	o.:							
Gelatin I	Mass mai	nufacturir	ıg:					
1								
2								
3								
4								
5								
6								
7								
8								
9								
10								

Reviewed by:		Date:
•	Validation	



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CITI SCEED				
Batch Size:	BMR No.:			
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#### FREQUENCY FOR EVERY 60 MIN

				Ol	oservatio	n		Recorded
S.No.	Date		Vacuum Applied in mm Hg	Gelatin Cooking Vessel Temp	Gelatin Mass Temp.	Stirrer Speed	Appearance of gelatin	By (Sign & Date)
Batch No	o.:							
Gelatin I	Mass man	ufacturin	ıg:					
1								
2								
3								
4								
5								
6								
7								
8								
9								
10								



**Batch Size:** 

### PHARMA DEVILS

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# PROCESS VALIDATION REPORT OF CALCITRIOL & MULTIVITAMINS SOFTGEL CAPSULES

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ol No.:						<u>  ]</u>	<b>Page No.:</b> 19	of 53
Reviewed	l by :		alidation		]	Date:		
		V	alidation					
FREQUE	ENCY FO	OR EVEI	RY 60 MIN					
					bservatio			Recorded
Sr. No.	Date	Time	Vacuum Applied in	Gelatin Cooking	Gelatin Mass	Stirrer Speed	Appearance of gelatin	(Sign &
			mm Hg	Vessel Temp	Temp.			Date)
Batch No	<b>.:</b>							
Gelatin M	Iass mai	nufacturi	ng:		1	T	T	T
1							_	
2								
3								
4								
5								
6							_	
7							=	
8							-	
9							-	
10							-	
					<u> </u>	l		<u> </u>
Reviewed	lbv:					Date:		



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#### 8.0 MATURATION OF GELATIN MASS IN GELATIN HOLDING TANK (FREQUENCY AT 1 HR)

Sr. No.	Date	Time	Set Temp.	Observed Temp.	Air Bubble	Uniformity of mass	Recorded By (Sign & Date)
Batch N	No.:						
Gelatin	Mass hold	ing tank	ID:				
1							
2							
3							
4							
5							
6							
7							
Gelatin	Mass hold	ing tank	ID:				
1							
2							



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		CAPS	SULES		
Batch Size:		BMR No.:			
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				1	
3					
4					
5					
6					
7					
Reviewed by :			Date:		

teviewed by:	Date:	
•	-	

Validation

S.No.	Date	Time	Set Temp.	Observed Temp.	Air Bubble	Uniformity of mass	Recorded By (Sign & Date)
Batch 1	No.:			•		•	,
Gelatir	Mass hold	ing tank	ID:			<b>.</b>	
1							
2							
3							
4							
5							
6							
7							
Gelatir	Mass hold	ing tank	k ID:				
1							
2							



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Dotah Cizat			CAPSUL		DMD No		
Protocol No.:	Satch Size: Protocol No.:				<b>BMR No.: Page No.:</b> 22 of 53		
3							
4							
5							
6							
7							
Reviewed by	y:			Date: _			
		Validation					

Sr. No.	Date	Time	Set Temp.	Observed Temp.	Air Bubble	Uniformity of mass	Recorded By (Sign & Date)
Batch N	No.:						
Gelatin	Mass hold	ling tank	ID:				
1							
2							
3							
4							
5							
6							
7							
Gelatin	Mass hold	ing tank	ID:				
1							



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				BMR N Page N				

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<b>,</b>		

Validation

Sr. No.	Date	Time	Set Temp.	Observed Temp.	Air Bubble	Uniformity of mass	Recorded By (Sign & Date)
Batch 1	No.:						
Gelatin	Mass hold	ing tank	k ID:				
1							
2							
3							
4							
5							
6							
7							



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Gelatir	n Mass holdi	ng tank	iD:		
1					
2					
3					
4					
5					
6					
7					

Reviewed by:		Date:
•	Validation	

Sr. No.	Date	Time	Set Temp.	Observed Temp.	Air Bubble	Uniformity of mass	Recorded By (Sign & Date)
Batch N	No.:						
Gelatin	Mass hold	ding tank	iD:				
1							
2							
3							
4							
5							
6							



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Batch Size: B					
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7					
Gelatin Mass ho	olding tank ID :				
1					
2					
3					
4					
5					
6					
7					

Reviewed by:		Date:
	Validation	

Sr. No.	Date	Time	Set Temp.	Observed Temp.	Air Bubble	Uniformity of mass	Recorded By (Sign & Date)
Batch I	No.:						
Gelatin	Mass hold	ing tank	iD:				
1							
2							
3							
4							
5							



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Batch	Size:						BMR No.:	
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	6							
	6							
	7							
	Gelatii	n Mass holdi	ing tanl	k ID:				
	1							
	2							
	3							
	4							
	5							
	6							
	7							
,	Review	ved by :		1		Date: _		
			V	alidation				

#### 9.0 MEDICAMENT MANUFACTURING PROCESS PARAMETER

#### 9.1 MEDICAMENT PREPARATION

Sr. No.		Parameter				
	Batch No.	Stirring Start (Time/Date)	Stirring Stop (Time/Date)	Mesh No. Used	Vacuum Applied in mm Hg	Recorded By (Sign & Date)



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•	Date
Validation	

#### 10.0 SAMPLE COLLECTION SUMMARY

Product	Calcitriol & Multivitamins					
Sample		Sample quantity	(in gm)			
Location↓	Batch No.	Batch No.	Batch No.			
T1						
T2						
Т2						
T3						



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	_ Date:		
alidation			
Calcitriol & Mult	ivitamins		
Batch No.	Batch No.	Batch No.	
	_ Date:		
	Calcitriol & Mult Sample quantity i Batch No.	Calcitriol & Multivitamins Sample quantity in gm Batch No. Batch No.	Calcitriol & Multivitamins Sample quantity in gm Batch No. Batch No. Batch No.

# 11.0 RESULTS OF ASSAY FOR MEDICAMENT Batch No.

Sampling	% Assay				
location	Calcitriol	Calcium carbonate	Mecobalamin	Vitamin K2-7	L-Methyl Folate
T1					



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n Size:				BMR N	0.:
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T2					
T3					
M1					
M2					
M3					
B1					
B2					
В3					
B4					
Mean					
Minimum					
Maximum					
Checked By/ Date					
		·	·		
Reviewed by:	Validation		Date:		_
	vandation				
Result for comp	osite sample				
Batch No.					



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		CAPSULES		
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% of Assay & Des	cription			
Calcitriol	Calcium carbonate	Mecobalamin	Vitamin K2-7	L-Methyl Folate
Description				
Checked By/ Date				
Reviewed by:	Validation	Г	Pate:	_

Batch No.



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col No.:				Page No.:	31 01 33
Sampling	% Assay				
Sampling location	Calcitriol	Calcium carbonate	Mecobalamin	Vitamin K2-7	L-Methy Folate
T1					
T2					
Т3					
M1					
M2					
M3					
B1					
B2					
В3					
B4					
Mean					
Minimum					
Maximum					
Checked By/ Date					

Validation



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		CAPSULES		
Size:			BMR	No.:
col No.:			Page N	<b>No.:</b> 32 of 53
Result for compos	site sample			
Batch No.	•			
% of Assay & Des	 scription			
Calcitriol	Calcium carbonate	Mecobalamin	Vitamin K2-7	L-Methyl Folate
Description				
Checked By/				



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#### Batch No.

Sampling	% Assay				
location	Calcitriol	Calcium carbonate	Mecobalamin	Vitamin K2-7	L-Methyl Folate
T1					
T2					
Т3					
M1					
M2					
M3					
B1					
B2					
В3					
B4					
Mean					
Minimum					
Maximum					
Checked By/ Date					

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ch Size:		BMR No.:			
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Result for compos	ite sample				
_	<b>-</b>				
Batch No.					
% of Assay & Des	cription				
Calcitriol	Calcium carbonate	Mecobalamin	Vitamin K2-7	L-Methyl Folate	
Description					
Checked By/					
Date					
Reviewed by:		Ι	Date:		
	Validation			- <del></del> -	



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# 12.0 SAMPLES COLLECTED DURING ENCAPSULATION FOR IN-PROCESS PARAMETERS

Batch No.	Sample Stage	Sample Quantity	Sampled By/Date
	Minimum Speed		
	Maximum Speed		
	Optimum Speed		
	Full Hopper		
	Half Hopper		
	End Hopper		
	Initial stage		
	Middle Stage		
	End stage		
	Minimum Speed		
	Maximum Speed		
	Optimum Speed		
	Full Hopper		
	Half Hopper		
	End Hopper		
	Initial stage		
	Middle Stage		
	End stage		
	Minimum Speed		
	Maximum Speed		
	Optimum Speed		
	Full Hopper		
	Half Hopper		
	End Hopper		
	Initial stage		
	Middle Stage		
	End stage		



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CAPSULES		
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**NOTE:** In-process testing shall be carried out as per frequency in BMR and the recordings shall be done in the BMR and the results shall be evaluated.

# 13.0 INDIVIDUAL IN-PROCESS TEST DATA AND EVALUATION OF IN PROCESS DATA:

DATA.									
INDIVIDUAL IN-PROCESS TEST DATA (SPEED)									
Batch Number:		Da	Date:						
TE OTO			RESULTS						
TESTS			Minimum Speed ( )		Maximum Speed ( )		Optimum speed( )		
Description									
Gelatin Feed Tank temperature (°C)		е							
Duct temperature (°C)									
Segment temperature (°C)									
Spreader Box temperature (°C)		,	Left	Right	Left	Right	Left	Right	
		,							
Ribbon Thickness (From ribbon continuously formed) (mm)		on							
Cavity No	1	2		3	4	5	6	Avg. Net Wt.	
Minimum Speed ( )									
Gross Wt.									
Shell Wt.									
Net Wt.									
Maximum Speed ( )									
Gross Wt.									
Shell Wt.									
Net Wt.									
Optimum Speed ( )									



QUALITY ASSURANCE

		CAPSUL	ES			
<b>Batch Size:</b>				BMR I	No.:	
<b>Protocol No.:</b>				Page N	<b>Vo.:</b> 37 of 53	}
	1	T				
Gross Wt.						
Shell Wt.						
Net Wt.						
Reviewed by:	Validation		Date:		_	

	INI	DIVID	U <b>AL IN</b>	-PRO	CESS T	EST	DATA (F	HOPPER )			
Batch Number	:		Date:								
7	PEGTG						RES	ULTS			
1	TESTS		I	Full H	lopper		Half	f Hopper		End H	opper
Description											
Gelatin Feed T (°C)	ank temperatur	re									
Duct temperatu	ıre (°C)										
Segment temper	erature (°C)				ı						
Spreader Box t	emperature (°C	C)	Let	ft	Righ	t	Left	Righ	nt	Left	Right
Ribbon Thickn continuously fo	,	on									
Cavity No	1		2		3		4	5		6	Avg. Net Wt.
Full Hopper											
Gross Wt.											
Shell Wt.											
Net Wt.											
Half Hopper											
Gross Wt.											
Shell Wt.											
Net Wt.											
End Hopper											



QUALITY ASSURANCE

ch Size:		BMR No.:	}
tocol No.:		Page No.:	38 of 53
Gross Wt.			
Shell Wt.			
Net Wt.			

	IN	DIVID	UAL II	N-PR	OCESS	TES	ST DATA (S	TAGE)		
Batch Number:			Date:							
TI	ESTS						RESU	LTS		
11	2313		Initial Stage			Midd	le Stage	End S	Stage	
Description										
Gelatin Feed Ta (°C)	nk temperatur	re								
Duct temperatur	re (°C)									
Segment temper	rature (°C)				T					
Spreader Box te	ader Box temperature (°C)		Left Righ		t	Left	Right	Left	Right	
Ribbon Thickne continuously for		on								
Cavity No	1		2		3		4	5	6	Avg. Net Wt
Initial Stage										
Gross Wt.										
Shell Wt.										
Net Wt.										
Middle Stage										
Gross Wt.										
Shell Wt.										
Net Wt.										



QUALITY ASSURANCE

Size:				MR No.:	
ol No.:			P	age No.: 39	9 of 53
End Stage					
Gross Wt.					
Shell Wt.					
Net Wt.					
Reviewed by:	Validation	Di	ate:		·
EVALUATION:		 			



QUALITY ASSURANCE

h Size:	BMR No.:
ocol No.:	<b>Page No.:</b> 40 of 53
5	<b>D</b>
Reviewed by:	Date:
Validation	

	IN	DIV	IDUAL I	N-PR	OCESS 7	TEST DATA	(SPEED)					
Batch Number:	:		Date:									
т	ESTS		RESULTS									
1.	ESIS		Minimum Speed ( )			Maximum S	peed ( )	Optimum speed( )				
Description												
Gelatin Feed T (°C)	ank temperatur	re										
Duct temperatu	ıre (°C)											
Segment tempe	erature (°C)											
Spreader Box t	Spreader Box temperature (°C)		Lef	t	Right	Left	Right	Left	Right			
Ribbon Thickn continuously fo	·	on										
Cavity No	1		2		3	4	5	6	Avg. Net Wt.			
Minimum Spe	eed ( )											
Gross Wt.												
Shell Wt.												
Net Wt.												
Maximum Sp	eed ( )											



QUALITY ASSURANCE

		CAPS	SULES			
Batch Size:				BMR	No.:	
Protocol No.:				Page 1	<b>No.:</b> 41 of 53	
Gross Wt.						
Shell Wt.						
Net Wt.						
<b>Optimum Speed</b>	( )					
Gross Wt.						
Shell Wt.						
Net Wt.						
Reviewed by:	Validation		Date:			

	IND	IVID	UAL IN	-PR	OCESS T	EST	DATA (F	HOPPER)		
Batch Number:			Date:							
T	Бажа						RES	ULTS		
1.	ESTS		Full Hopper				Half	f Hopper	End I	Hopper
Description										
Gelatin Feed Ta (°C)	ank temperatur	·e								
Duct temperatur	re (°C)									
Segment temper	rature (°C)									
Spreader Box te	Spreader Box temperature (°C)		Le	ft	Righ	t	Left	Right	Left	Right
Ribbon Thickne continuously fo	·	on								
Cavity No	1		2		3		4	5	6	Avg. Net Wt.
Full Hopper										
Gross Wt.										
Shell Wt.										
Net Wt.										



QUALITY ASSURANCE

Size:			MR No.:	2 652
col No.:		P	age No.: 42	2 of 53
Half Hopper				
Gross Wt.				
Shell Wt.				
Net Wt.				
End Hopper				
Gross Wt.				
Shell Wt.				
Net Wt.				

	IN	DIVII	OUAL I	N-PR	COCESS	TES	T DATA (	STAGE)		
Batch Number:			Date:							
Ti	ESTS						RES	ULTS		
11	Loto		Initial Stage			Mid	dle Stage	End S	Stage	
Description										
Gelatin Feed Ta	nk temperatur	e								
Duct temperatur	re (°C)									
Segment temper	rature (°C)									
Spreader Box te	emperature (°C	)	Let	ft	Righ	t	Left	Right	Left	Right
Ribbon Thicknet continuously for		on								
Cavity No	1		2		3		4	5	6	Avg. Net Wt.
Initial Stage										
Gross Wt.										
Shell Wt.	·							·		



QUALITY ASSURANCE

CAPSUI	LES			
		Page	e <b>No.:</b> 43 of :	53
<u> </u>				
 				<b>_</b>
 Validation	Validation	Validation	Page    Date:	BMR No.:   Page No.: 43 of :



QUALITY ASSURANCE

tch Size:	BMR No.:
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Reviewed by:	Date:
Validation	

	IN	DIV	IDUAL I	N-PR	OCESS '	TES	ST DATA	(SPEED)			
Batch Number:			Date:								
T	ESTS						RESU	JLTS			
1	ESIS		Minimu	m Spe	eed ( )	M	Iaximum S	peed ( )	Optimum spe	Optimum speed( )	
Description											
Gelatin Feed T	ank temperatui	re									
Duct temperatu	re (°C)										
Segment tempe	erature (°C)										
Spreader Box t	emperature (°C	<b>'</b> )	Lef	ť	Right		Left	Right	Left	Right	
•	• ` `										
Ribbon Thickn continuously for	•	on									
Cavity No	1		2		3		4	5	6	Avg.	



QUALITY ASSURANCE

Size:				BMI	R No.:	
col No.:		Page	<b>No.:</b> 45 o	f 53		
						Net W
Minimum Speed	( )	I	I			
Gross Wt.						
Shell Wt.						
Net Wt.						
Maximum Speed	( )		·			•
Gross Wt.						
Shell Wt.						
Net Wt.						
Optimum Speed	( )					
Gross Wt.						
Shell Wt.						
Net Wt.						

INDIVIDU	JAL IN-PRO	CESS TEST	DATA (HO	PPER)						
Batch Number:	Date:									
TECTC		RESULTS								
TESTS	Full H	lopper	Half H	Iopper	End Hopper					
Description										
Gelatin Feed Tank temperature (°C)										
Duct temperature (°C)										
Segment temperature (°C)										
Spreader Box temperature (°C)	Left	Right	Left	Right	Left	Right				



QUALITY ASSURANCE

Batc	h Size:					BMR	No.:	
Prote	ocol No.:					Page	<b>No.:</b> 46 of	53
	Ribbon Thickner continuously for		1					
	Cavity No	1	2	3	4	5	6	Avg. Net Wt.
	Full Hopper			•				
	Gross Wt.							
	Shell Wt.							
	Net Wt.							
	Half Hopper							
	Gross Wt.							
	Shell Wt.							
	Net Wt.							
	End Hopper							
	Gross Wt.							
	Shell Wt.							
	Net Wt.							
	Reviewed by:	Valida	ntion		Date:_			•

INDIV	IDUAL IN-PR	OCESS TES	ST DATA (ST	TAGE)						
Batch Number:	Date:									
TITL CATHO		RESULTS								
TESTS	Initia	l Stage	Middle	e Stage	End St	tage				
Description										
Gelatin Feed Tank temperature (°C)										
Duct temperature (°C)										
Segment temperature (°C)										
Spreader Box temperature (°C)	Left	Right	Left	Right	Left	Right				



QUALITY ASSURANCE

Size:					BMR	No.:	
col No.:					Page I	<b>No.:</b> 47 o	f 53
							<u> </u>
Ribbon Thicknes continuously form		bon					
Cavity No	1	2	3	4	5	6	Avg Net W
Initial Stage							12.22
Gross Wt.							
Shell Wt.							
Net Wt.							
Middle Stage							
Gross Wt.							
Shell Wt.							
Net Wt.							
End Stage		<b>-</b>	<u> </u>	<b>-</b>		<u> </u>	l l
Gross Wt.							
Shell Wt.							
Net Wt.							
Reviewed by: _		idation		Date:_			
EVALUATIO	N:						
	<b></b>						<b>-</b>
	·						
	<b></b>						



QUALITY ASSURANCE

# PROCESS VALIDATION REPORT OF CALCITRIOL & MULTIVITAMINS SOFTGEL CAPSULES

atch Size:	BMR No.:
otocol No.:	<b>Page No.:</b> 48 of 53
Reviewed by:	Date:
Validation	<u> </u>
, andanon	

#### 14.0 DRYING DETAILS OF CAPSULE:

Batch No.	Drying	Started At	Drying	End At	Total Daving Time
Daten No.	Date	Time	Date	Time	Total Drying Time



QUALITY ASSURANCE

15.0 SA	AMPLE (	COLLEC	CTION AND L	OD RESU	JLTS:					
S.N.	Batch No.	Dry	ring Time	Sampl Quanti		Desci	ription	Sam	npled By	LOD Resu Limit 6.0- 10.0%
1		After 3	2 hr of drying							
2		After 3	7 hr of drying							
3		After 4	2 hr of drying							
1		After 3	2 hr of drying							
2		After 3	7 hr of drying							
3		After 4	2 hr of drying							
1		After 32	2 hr of drying							
2		After 3	7 hr of drying							
3		After 4	2 hr of drying							
								•		
Revie	wed by : _		Validation			Ι	Date:			



QUALITY ASSURANCE

Batch Size:	BMR No.:
Protocol No.:	<b>Page No.:</b> 50 of 53

Test  Description  Identification  For Calcitriol (By HPLC)	Red coloured, oval shaped, softgelatin capsules containing pale brown to pink coloured viscous oily paste.  The retention time of the principal peak in the chromatogram of sample preparation should correspond to that of the principal peak in the chromatogram of standard preparation as obtained in the	
Identification  For Calcitriol	softgelatin capsules containing pale brown to pink coloured viscous oily paste.  The retention time of the principal peak in the chromatogram of sample preparation should correspond to that of the principal peak in the chromatogram of standard preparation as obtained in the	
For Calcitriol	The retention time of the principal peak in the chromatogram of sample preparation should correspond to that of the principal peak in the chromatogram of standard preparation as obtained in the	
	chromatogram of sample preparation should correspond to that of the principal peak in the chromatogram of standard preparation as obtained in the	
	"Assay" for Calcitriol.	
For Vitamin K2-7 (By HPLC)	The retention time of the principal peak in the chromatogram of sample preparation should correspond to that of the principal peak in the chromatogram of standard preparation as obtained in the "Assay" for Vitamin K2-7.	
For Mecobalamine (By HPLC)	The retention time of the principal peak in the chromatogram of sample preparation should correspond to that of the principal peak in the chromatogram of standard preparation as obtained in the "Assay" for mecobalamine.	
( I	By HPLC) For Mecobalamine	principal peak in the chromatogram of sample preparation should correspond to that of the principal peak in the chromatogram of standard preparation as obtained in the "Assay" for Vitamin K2-7.  The retention time of the principal peak in the chromatogram of sample preparation should correspond to that of the principal peak in the chromatogram of sample preparation should correspond to that of the principal peak in the chromatogram of standard preparation as obtained in the



QUALITY ASSURANCE

Batch Size:	BMR No.:
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S.	Specification		BATCH NO:		
No	Test				
	For L-Methyl folate	The retention time of the principal peak in the chromatogram of sample preparation should correspond to that of the principal peak in the chromatogram of standard preparation as obtained in the "Assay" for L-Methyl Folate.			
3	Average net content (mg)	1875 + 5.0% (1781.3 - 1968.8)			
4	Average Weight (mg)	2250.0 + 5.0 % (2137.5 - 2362.5)			
5	Uniformity of weight (mg)	Not more than two of individual weights deviate from the average weight by more than 7.5 % and none deviates by more than 15 %.			
6	<b>Disintegration Time</b> (min)	Not more than 60			
7	Assay  Calcitriol  (By HPLC, as [C <sub>27</sub> H <sub>44</sub> O <sub>3</sub> ]) -mcg per capsule -% label claim	Not less than 0.225 Not less than 90.0			



QUALITY ASSURANCE

Batch Size:	BMR No.:
Protocol No.:	<b>Page No.:</b> 52 of 53

S.	Test	Specification	BATCH NO:	
No				
	Calcium Carbonate			
	(By Titrimetry, as CACO <sub>3</sub> )			
	-mg per capsule	Not less than 1125.0		
	-% label claim	Not less than 90.0		
	Mecobalamin			
	(By HPLC)			
	-mcg per capsule			
	-% label claim	Not less than 1350.0 Not less than 90.0		
	Vitamin K2-7	Not less than 90.0		
	(By HPLC)			
	-mcg per capsule			
	-% label claim	Not less than 40.5		
	L-Methyl Folate	Not less than 90.0		
	(By HPLC)			
	-mcg per capsule			
	-% label claim	Not less than 720.0		
	Zinc (By AAS)	Not less than 90.0		
	-mg per capsule	Not less than 6.75		
	-% label claim	Not less than 90.0		
	Magnesium			
	(By AAS)			
	-mg per capsule	Not less than 45.0		
	-% label claim	Not less than 90.0		
		· ·		

Reviewed by:	Date:	
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QUALITY ASSURANCE

Size:		BMR No.:	
ocol No.:		<b>Page No.:</b> 53 of 53	
Valid	ation		
17.0 PERCENTAGE YIE	LD AT THE END OF INSPECTION:		
Batch No.	% yield	Limit*	
* Yield Limit is tentative an	d will be finalised after 10 or more prod	uction batches.	
	_		
D 11	D-4		
Reviewed by:Valid			
	Date: ation  REMAINING SAMPLES:  Destruction done by / Date	Checked by / Date	
18.0 DESTRUCTION OF	REMAINING SAMPLES:		
18.0 DESTRUCTION OF	REMAINING SAMPLES:		
18.0 DESTRUCTION OF	REMAINING SAMPLES:		
18.0 DESTRUCTION OF  Batch No.:	REMAINING SAMPLES:	Checked by / Date	
18.0 DESTRUCTION OF  Batch No.:	REMAINING SAMPLES:  Destruction done by / Date  e batch are destroyed as per SOP No  Date:	Checked by / Date	



QUALITY ASSURANCE

19.0 SUMMARY, CONCLUSION AND A	APPROVAL REPORT:



QUALITY ASSURANCE

### PROCESS VALIDATION REPORT OF CALCITRIOL & MULTIVITAMINS SOFTGEL CAPSILES

ch Size:		capsules ze: BMR No.:	
tocol No.:			<b>Page No.:</b> 55 of 53
	Validation		
CONCLU	SION:		
Calcitriol 6	& Multivitamins Batch Nos.:	,and	manufactured in th
facility as	the validation batch meet the sp	ecification of Softgel. The	process of manufacturing wa
carried as 1	per the approved Batch Manufact	uring records and process of	of Manufacturing is validated.
_	ocess Validation batches has be		_
_			•
cGMP requ	uirement.		
	Batch No.	A.R.No. of	Finished Product
APPROV	AL		
	HEAD – PRODUCTION	HEAD – QC	HEAD – QA
SIGN &			

#### **REVISION CARD**

S. No.	RPV No.	Reason for Revision	Change Control No.
1		New	



QUALITY ASSURANCE

CHIBCEES	
Batch Size:	BMR No.:
Protocol No.:	<b>Page No.:</b> 56 of 53