



HOLD TIME STUDY PROTOCOL CUM REPORT FOR ORAL LIQUID

**HOLD TIME STUDY PROTOCOL
FOR
ORAL LIQUID**

PROTOCOL NO.	
SUPERCEDES	Nil
PROTOCOL EFFECTIVE DATE	
REPORT EFFECTIVE DATE	



HOLD TIME STUDY PROTOCOL CUM REPORT FOR ORAL LIQUID

CONTENTS

0.0 PROTOCOL PREAPPROVAL.....3

1.0 OBJECTIVE.....4

2.0 INTRODUCTION.....4

3.0 RESPONSIBILITIES.....4

4.0 PROCEDURE.....5

5.0 MATRIX & SPECIFICATION FOR HOLD TIME STUDY.....6-9

6.0 DEVIATION AND CHANGE CONTROL.....10

7.0 SUMMARY AND CONCLUSION.....10

8.0 REFERENCE.....10

9.0 ABBREVIATION.....10

10.0 POST APPROVAL.....11



PHARMADEVILS

QUALITY ASSURANCE DEPARTMENT

HOLD TIME STUDY PROTOCOL CUM REPORT FOR ORAL LIQUID

1.0 PROTOCOL PREAPPROVAL

PROTOCOL PREPARED BY:

DEPARTMENT	NAME	DESIGNATION	SIGNATURE	DATE
QUALITY ASSURANCE				

PROTOCOL REVIEWED BY:

DEPARTMENT	NAME	DESIGNATION	SIGNATURE	DATE
PRODUCTION				
QUALITY CONTROL				

PROTOCOL APPROVED BY:

DEPARTMENT	NAME	DESIGNATION	SIGNATURE	DATE
QUALITY ASSURANCE				



HOLD TIME STUDY PROTOCOL CUM REPORT FOR ORAL LIQUID

1.0 Objective:

The objective of this protocol is to study the effect of “Hold Time” on the bulk of to verify that during the hold period, the Product shall meet predetermined quality specifications. The protocol also details the responsibilities and activities associated with this Hold Time Study.

2.0 Introduction:

The Samples of subjected to Hold Time Study shall be stored under simulated conditions and shall be analyzed as per the established specifications at different time points of study as specified.

Analytical methods for testing of bulk of have been satisfactorily adopted into the QC laboratory.

On completion of the study, a report shall be prepared and final conclusion shall be drawn in the same. It shall be implemented in routine manufacturing. This document shall form the basis of the production scale batches. Any variation to the established Hold Time shall be explained and justified, in accordance with the change control procedure.

3.0 RESPONSIBILITIES:

S.No	Department	Responsibilities
01	Production	Review of Hold time study protocol and Report
02	Quality Control	Review of Hold time study protocol & report Analysis of samples and reporting the results
03	Quality Assurance	Preparation, Review & Approval of Hold time study protocol & report, Sampling and compilation of results

4.0 PROCEDURE:

4.1 Bulk hold time study:

Test the sample from the bulk from the three batches at Zero (0) time point. Store sample from three batches for a period of specific time period given different process stage under simulated conditions at Temp. NMT 27°C and Relative humidity NMT 50 %.

S.No	Name of stage	Sample Qty. Required for Hold time study
01	Un-Filtered Solution	2.00 ltr
02	Filtered Solution	2.00 ltr

Section 5.0 (Matrix for Hold Time Study).

Acceptance criteria:

Bulk sampled at different time points shall meet the acceptance criteria as mentioned in Table No. 1.



HOLD TIME STUDY PROTOCOL CUM REPORT FOR ORAL LIQUID

5.0 Matrix for hold time study

5.1.1 Matrix for hold time study of Un-Filtered solution

Storage Time	Sample Quantity for testing	Tests to be performed
Type of Sample: Un-Filtered solution		
Initial	300 ml (Chemical + microbiological sample)	Description, pH Value, wt. per ml & Assay, Micro
01 Day	300 ml (Chemical + microbiological sample)	Description, pH Value, wt. per ml & Assay, Micro
02 Days	300 ml (Chemical + microbiological sample)	Description, pH Value, wt. per ml & Assay, Micro
05 Days	300 ml (Chemical + microbiological sample)	Description, pH Value, wt. per ml & Assay, Micro
07 Days	300 ml (Chemical + microbiological sample)	Description, pH Value, wt. per ml & Assay, Micro

5.1.2 : specifications for Un-Filtered solution

S.No.	Tests	Limits
01	Description	
02	pH value	
03	Wt. per ml	
04	Assay	
Additional Test		
05	Microbial limit test	
	A. Total viable aerobic count	
	Aerobic bacteria	NMT 10 ² cfu/gm
	Fungi	NMT 10 ¹ cfu/gm
	B. Pathogens	
	1. <i>E. coli</i>	Absent



HOLD TIME STUDY PROTOCOL CUM REPORT FOR ORAL LIQUID

5.2.1 Matrix for hold time study of Filtered solution

Storage Time	Sample Quantity for testing	Tests to be performed
Type of Sample: Filtered solution		
Initial	300 ml (Chemical + microbiological sample)	Description, pH Value, wt. per ml & Assay, Micro
01 Day	300 ml (Chemical + microbiological sample)	Description, pH Value, wt. per ml & Assay, Micro
02 Days	300 ml (Chemical + microbiological sample)	Description, pH Value, wt. per ml & Assay, Micro
05 Days	300 ml (Chemical + microbiological sample)	Description, pH Value, wt. per ml & Assay, Micro
07 Days	300 ml (Chemical + microbiological sample)	Description, pH Value, wt. per ml & Assay, Micro

5.2.2 : specifications for Filtered Solution

S.No.	Tests	Limits
01	Description	
02	pH value	
03	Wt. per ml	
04	Assay	
Additional Test		
05	Microbial limit test	
	A. Total viable aerobic count	
	Aerobic bacteria	NMT 10 ² cfu/gm
	Fungi	NMT 10 ¹ cfu/gm
	B. Pathogens	
	1. <i>E. coli</i>	Absent



HOLD TIME STUDY PROTOCOL CUM REPORT FOR ORAL LIQUID

6.0 DEVIATION AND CHANGE CONTROL:

.....
.....
.....
.....
.....
.....
.....
.....

7.0 SUMMARY AND CONCLUSION:

.....
.....
.....
.....
.....
.....
.....

8.0 REFERENCE:.

ICH, FDA, EMEA and Annexure IV WHO TRS 992

9.0 ABBREVIATION:.

S.No	Abbreviation	Extended Form
01	SOP	Standard Operating Procedure
02	HTSP	HOLD Time Study Protocol
03	LOD	Loss on Drying



PHARMADEVILS
QUALITY ASSURANCE DEPARTMENT

HOLD TIME STUDY PROTOCOL CUM REPORT FOR ORAL LIQUID

10.0 REPORT POSTAPPROVAL:

COMPILED BY:

DEPARTMENT	NAME	DESIGNATION	SIGNATURE	DATE
QUALITY ASSURANCE				

REVIEWED BY:

DEPARTMENT	NAME	DESIGNATION	SIGNATURE	DATE
PRODUCTION				
QUALITY CONTROL				

APPROVED BY:

DEPARTMENT	NAME	DESIGNATION	SIGNATURE	DATE
QUALITY ASSURANCE				



PHARMADEVILS
QUALITY ASSURANCE DEPARTMENT

HOLD TIME STUDY PROTOCOL CUM REPORT FOR ORAL LIQUID

Annexure I

RESULTS OF UN-FILTERED SOLUTION

BATCH NO.		MFG. DATE	
BATCH SIZE		EXP. DATE	

Sr. No.	Tests	Limits	0 hours (Initial)	01 Days	02 Days	05 Days	07 Days
01	Description						
02	pH Value						
03	Wt.per ml						
04	Assay						

Additional Test

05	Microbial limit test						
	A. Total viable aerobic count						
	Aerobic bacteria	NMT 10 ² cfu/g					
	Fungi	NMT 10 ¹ cfu/g					
	B. Pathogens						
	1. E. coli	Absent					

Compiled By _____

Reviewed By _____



HOLD TIME STUDY PROTOCOL CUM REPORT FOR ORAL LIQUID

Annexure II

RESULTS OF FILTERED SOLUTION

BATCH NO.		MFG. DATE	
BATCH SIZE		EXP. DATE	

S.No.	Tests	Limits	0 hours (Initial)	01 Days	02 Days	05 Days	07 Days
01	Description						
02	pH Value						
03	Wt.per ml						
04	Assay						

Additional Test

05	Microbial limit test						
	A. Total viable aerobic count						
	Aerobic bacteria	NMT 10 ² cfu/g					
	Fungi	NMT 10 ¹ cfu/g					
	B. Pathogens						
	1. E. coli	Absent					

Compiled By _____

Reviewed By _____