

	STANDARD OPERATING PROC	EDURE		
Depa	artment: Quality Control	SOP No.:		
Title	: Operation, Cleaning and Calibration of Karl –Fischer Apparatus	Effective Date:		
Supe	Supersedes: Nil Review Date:			
Issue Date: Page No.:				
1.0	OBJECTIVE: To lay down a procedure for Operation, cleaning and calibration of Kar	rl –Fischer Apparatus.		
2.0	SCOPE:			
	This Procedure is applicable for Karl Fischer Apparatus, (MAKE : Me	trohm Model: 787		
	Titrino) installed in Quality Control Department Laboratory.			
3.0	RESPONSIBILITY – Execution – Executive QC. Checking - Assistant Manager QC.			
4.0	ACCOUNTABILITY - Manager Quality Control			
5.0	PROCEDURE:			
5.1	OPERATING PROCEDURE:			
5.1.1	Check calibration status of instrument.			
5.1.2	If calibration is due, then, inform QC In-charge.			
5.1.3	Switch 'ON' the mains.			
5.1.4	Fill the reagent bottle with Karl Fischer reagent.			
5.1.5	Check the molecular sieves. They shall be light brown in colour. If dark	brown, replace them with fresh molecular		
	sieves. The molecular sieves can be regenerated.			
5.1.6	Switch ON the Power.			
5.1.7	Rinse the titration vessel with methanol.			
5.1.8	Switch on the Titrino using On/Off switch present at front panel of T	itrino		
5.1.9	Select the required method using <recall meth=""></recall> , which can be access on keyboard. Use arrows key to select your method and then pressing RAM.The method name comes on first line extreme right side of your	<enter> key loads the reqd method in</enter>		
5.1.10	Press <start></start> key either on front panel of instrument or on keyboard			
5.1.11	After pressing start the instrument conditions the methanol so as to n it. This conditioning is referred as DRIFT	eutralize the access moisture present in		
5.1.12	After conditioning <drift ok=""></drift> is displayed.			
5.1.13	To start your analysis, again press <start></start> key either on front panel of	f instrument or on keyboard.		



		EDURE		
Department: Quality Control SOP No.:				
Title: Operation, Cleaning and Calibration of Karl –Fischer Apparatus Effective Date:				
-	rsedes: Nil Date:	Review Date: Page No.:		
15500	Date.	1 age 110		
5.1.14	Feed sample in titration vessel and <enter></enter> sample weight and wait for	or the result.		
5.1.15	After completion of the analysis, result is displayed on the screen.			
5.2	CALIBRATION PROCEDURE:			
5.2.1	For Linearity:			
	Frequency: Once in a Month.			
5.2.1.1	Weigh about 50, 100, 150, 200, 250 mg of Disodium Tartarate (KF Grad	le)		
5.2.1.2	Operate the instrument as per the procedure given above.			
5.2.1.3	Note down the burette reading.			
5.2.1.4	Report the results in the given format as per Annexure-I.			
5.2.1.5	Check the correlation coefficient.			
5.2.1.6	Correlation coefficient should not be less than 0.99.			
5.2.1.7	The performance of the instrument is satisfactory if the obtained Correla	tion coefficient are within the give		
imit.				
5.3	Burette calibration:			
	Frequency: Once in 3 Months			
5.3.1	Use water for calibration.			
5.3.2	Mount the burette tip firmly on the stand rod, it must not be moved durin	g the test.		
5.3.3	Set dispensing and filling rate of the burette to 'max'.			
5.3.4	Take a dried beaker and tare the beaker.			
5.3.5	Discharge the volume into the beaker and note down the volume dispense	d.		
5.3.6	Press <fill> Key.</fill>			
5.3.7	Weigh the dispensed volume.			
5.3.8	Repeat 9 discharges of different volume.			



		STANDARD OPERATING P				
-	rtment: Quality Co		SOP No.:			
	1	g and Calibration of Karl – Fischer Appara				
-	rsedes: Nil		Review Date:			
Issue	Date:		Page No.:			
5.3.10	Note down the reading in the provided format Annexure – I.					
5.3.11	Maximum relative err	or of the 10ml exchange unit should NMT 0.5	5%.			
5.3.12	The performance of the	he burette is satisfactory if the obtained relativ	e error is within limit.			
5.3.13	If the calibration is no	t satisfactory then follow				
5.4	Cleaning Procedure	For Karl Fischer Apparatus				
	FREQUENCY: DAI	FREQUENCY: DAILY				
5.4.1	After completion of the testing, press "Arrow Key " pointing towards upward direction on front					
	panel of Ti Stand for	flushing of the Test solution into a storage bo	ttle.			
5.4.2	Remove the solution	from the Titration vessel and wash with meth	anol by pressing " Arrow			
	key" pointing towar	ds the downward direction on front panel of T	'i Stand.			
5.4.3	Open the Titration V	essel and clean the vessel and the electrode w	ith the help of the tissue paper.			
5.4.4	Switch 'OFF' the ins	strument and switch 'OFF' the mains.				
5.4.5	Collect the waste sol	ution in the waste beaker to be discarded to a	void corrosion.			
5.4.6	Clean the outer surfa	ce of the instrument with Isopropyl alcohol and	nd dry it with tissue paper.			
5.4.7	Record details of cle	aning in the log card of instrument				
6.0	SAFETY & PREC A Not Applicable.	AUTIONS:				
7.0	REVISION HISTO	RY:				
	Revision No.	Reason for Revision	Superseded from & Date			

Comu	Issuance Record			Withdrawal Record		Destruction Record		
Copy No.	Date	Dept. issued	Name / Signature of receiver	Issued By Name / Signature	Ву	Sign/ Date	Ву	Sign/ Date



STANDARD OPERATING PROCEDURE					
Department: Quality Control	SOP No.:				
Title: Operation, Cleaning and Calibration of Karl –Fischer Apparatus	Effective Date:				
Supersedes: Nil	Review Date:				
Issue Date:	Page No.:				

9.0 **REFERENCES:** Not Applicable

II ·····

10.0 ABBREVIATIONS & ANNEXURES

- SOP : Standard Operating Procedure
- QA : Quality Assurance
- No. : Number
- QC : Quality Control
- Dept. : Department
- % : Percentage
- ml : Millilitre
- mg : Milligram
- KF : Karl Fischer
- NMT : Not More Than

Annexure-I : Calibration Report of Karl Fischer Apparatus

Annexure-II: Cleaning Record for KF Apparatus



Г

PHARMA DEVILS QUALITY CONTROL DEPARTMENT

STANDARD OPERATING PROCEDURE					
Department: Quality Control	SOP No.:				
Title: Operation, Cleaning and Calibration of Karl –Fischer Apparatus	Effective Date:				
Supersedes: Nil	Review Date:				
Issue Date:	Page No.:				

ANNEXURE-I

CALIBRATION REPORT OF KARL FISCHER APPARATUS

CALIBRATION DATE	DATE OF LAST CALIBRATION	NEXT DUE FOR CALIBRATION

INSTRUMENT DETAILS							
INSTRUMENT NAME	INSTRUMENT MAKE	INSTRUMENT ID No.					

OBSERVATION

S.No.	Wt. taken of di-Sodium tartrate (mg)	Volume of KFR required (ml)
1		
2		
3		
4		
5		
Limit→	Correlation coefficient 'r': (NLT 0.99)	



STANDARD OPERATING PROCEDURE					
Department: Quality Control	SOP No.:				
Title: Operation, Cleaning and Calibration of Karl –Fischer Apparatus	Effective Date:				
Supersedes: Nil	Review Date:				
Issue Date:	Page No.:				

BURETTE CALIBRATION

(Frequency : 3Months)

Actual temp of water :FactorWt. of beaker (A)

S.no.	Displayed volume(ml) B	Wt. of beaker + water (g) C	Wt. of water(gm) D=(C-A) g	Actual Volume (ml) E =(FactorX D)	difference F=(E-B)	Tolerance <u>+</u> 0.5 % of displayed vol.
1						
2						
3						
4						
5						
6						
7						
8						
9						
10						



STANDARD OPERATING PROCEDURE				
Department: Quality Control	SOP No.:			
Title: Operation, Cleaning and Calibration of Karl –Fischer Apparatus	Effective Date:			
Supersedes: Nil	Review Date:			
Issue Date:	Page No.:			

CONCLUSION			
INSTRUMENT WORKING SATISFACTORY	INSTRUMENT NOT WORKING SATISFACTORY		

	PERFOMED BY			CHECKED BY	
NAME	SIGN.	DATE	NAME	SIGN.	DATE



STANDARD OPERATING PROCEDURE			
Department: Quality Control	SOP No.:		
Title: Operation, Cleaning and Calibration of Karl –Fischer Apparatus	Effective Date:		
Supersedes: Nil	Review Date:		
Issue Date:	Page No.:		

ANNEXURE-II

CLEANING RECORD FOR KF APPARATUS

NAME OF THE INSTRUMENT : MAKE : MODEL NO : IDENTIFICATION NO. :

Date of Cleaning	Cleaned By	Checked By	Remarks