



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.:

1.0 OBJECTIVE:

To lay down a procedure for Operation, cleaning and calibration of Karl –Fischer Apparatus.

2.0 SCOPE:

This Procedure is applicable for Karl Fischer Apparatus, (MAKE : Metrohm 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 **Titrimo**
- 5.1.9 Select the required method using **<recall meth>**, which can be accessed by help of **<user meth>** key which is on keyboard. Use arrows key to select your method and then pressing **<enter>** key loads the reqd method in RAM. The method name comes on first line extreme right side of your screen.
- 5.1.10 Press **<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 neutralize the access moisture present in it. This conditioning is referred as **DRIFT**
- 5.1.12 After conditioning **<DRIFT OK>** is displayed.
- 5.1.13 To start your analysis, again press **<start>** key either on front panel of instrument or on keyboard.



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.:

5.1.14 Feed sample in titration vessel and <enter> sample weight and wait for 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 Grade)

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 Correlation coefficient are within the given limit.

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 during 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 dispensed.

5.3.6 Press <FILL> Key.

5.3.7 Weigh the dispensed volume.

5.3.8 Repeat 9 discharges of different volume.

5.3.9 Calculate the actual volume.



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.:

--	--	--	--	--	--	--	--	--	--

9.0 REFERENCES:

Not Applicable

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



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)	



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.:

BURETTE CALIBRATION

(Frequency : 3Months)

Actual temp of water : _____

Factor : _____

Wt. 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 ± 0.5 % of displayed vol.
1						
2						
3						
4						
5						
6						
7						
8						
9						
10						



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.:

CONCLUSION

INSTRUMENT WORKING SATISFACTORY

INSTRUMENT NOT WORKING SATISFACTORY

PERFORMED BY			CHECKED BY		
NAME	SIGN.	DATE	NAME	SIGN.	DATE

