



STANDARD OPERATING PROCEDURE

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| Department: | SOP No.: |
| Title: Operation & Calibration of Karl Fisher | Effective Date: |
| Supersedes: Nil | Review Date: |
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1.0 OBJECTIVE:

To lay down a procedure for operation and calibration of Karl Fisher.

2.0 SCOPE:

This SOP is applicable for operation and calibration of Karl Fisher in the QC department.

3.0 RESPONSIBILITY:

Officer, Executive, Sr.Executive - Quality Control Department.
Head - Quality Control.

4.0 PROCEDURE :

4.1 Operation:

4.1.1 KF Titration:

4.1.1.1 Ensure that instrument is cleaned and free from dust.

4.1.1.2 Connect the instrument to power supply. Switch on the instrument on the front panel.

4.1.1.3 A Stirrer speed controller is provided to adjust the desired stirrer speed.

4.1.1.4 A keyboard having 16keys is provided on the front panel. The numeric keypad consists of 0 to 9 keys along with a decimal key. Theses keys are used to enter values of sample ,density etc.

4.1.1.5 Press START key.

4.1.1.6 The message will appear as-

Add Sample
Ex. Time =10 secs.

4.1.1.7 The 4th line shows the stirring time (the set stirrer time is 10 secs, time can vary from 1 sec to 240 sec). When the set stirring time decrements to “00”

The display will indicate

Add Sample
Ex. Time “00”



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4.1.1.8 Then dispensing until will start adding K/F Reagent into the beaker through delivery tube.
The display will indicate as-

TITRATION STARTED
00 : 01 ml
EXCESS WATER
Conf. Time: 20 Secs

4.1.1.9 Second line of the display indicate the consumption of K/F Reagent in ml.

4.1.1.10 Third line indicates the presence of moisture. i.e. Excess Water

4.1.1.11 Forth line of the display indicates the end point duration. The end point should last for 20 secs.
When the time counts down to "00", It indicates the end point is reached.

4.1.1.12 This display will change to

K/F REAGENT READING
06:52 ml

4.1.1.13 To make titration vessel moisture free, follow the above procedure (i.e. para6.1) 2 to 3 times. This indicates that the methanol is neutralized (or titration vessel is moisture free) and 1 stage of titration is over.

4.1.2 KF Factor:

4.1.2.1 Verify the KF factor on daily basis or as and when required.

4.1.2.2 Press CAL key and add 10 µl of distilled water in the titration vessel.

4.1.2.3 The display will indicate-

Add 10 MICRO LIT
Distilled water
And
Press Enter.

Press Enter Key

4.1.2.4 The display will indicate-

CAL IN PROGRESS
00: 00 ml.
EXCESS WATER
Conf.Time: 20 sec.

4.1.2.5 The message appearing on the will be



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CAL. IN PROGRESS
02:25 ml.
CONF. TIME: 00 Secs.

4.1.2.6 After the audio signal, the message will appear as-

K/F Reagent Reading=02:25

4.1.2.7 The above message will remain for few seconds and message will change over to-

Factor = 04:4444
K/ F Reagent Reading = r 02:25 ml.
Repeat New factor ??
CONT. / END / CLEAR

4.1.2.8 If one wants to verify factor (i.e. second time and third time.)

4.1.2.9 Repeated the procedure for 4.1.2.3 to 4.1.2.7

4.1.2.10 The calculate only average of three titer factors.

4.1.2.11 Press SELECT key END message will start flashing. Then Press ENTER key.

4.1.2.12 The message will appear as

WAIT
CALCULATING AVERAGE
FACTOR

4.1.2.13 The above message will remain for few seconds, and will change over to-

Av. Factor =
Press ENTER to count

4.1.2.14 % RSD of three KF value should not be more than 1.0 % record the factor in Annexure-I

The KF factor should be between “4.5 – 6.5”.

4.1.3 Sample Analysis :

4.1.3.1 Before starting the analysis ensuring followings:

4.1.3.2 The instrument is clean, free from dust and calibrated and Check KF Factor.



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- 4.1.3.3 All tubing is connected properly.
- 4.1.3.4 Required sensor/electrode is attached in proper condition.
- 4.1.3.5 Press STARTS key. Operate the procedure 4.1.1.6 to 4.1.1.13
- 4.1.3.6 Sample should be added after pressing the start key only.
- 4.1.3.7 The display should appear as-

| |
|---------------------------------------|
| KF.R READING 02.88ml |
|---------------------------------------|

- 4.1.3.8 To find out % Enter the sample quantity. Then enter and press %/ppm key the display will appear as % of water. Same procedure follows in liquid sample.

4.2 Monthly Calibration:

- 4.2.1 Operating Procedure for Calibration as follow 4.1.1.6 to 4.1.1.13
- 4.2.2 Take weigh disodium tartrate 100.0 mg in KF Titrate. determine the water Disodium tartrate.
- 4.2.3 Take at least 3 times determine the water disodium tartrate for the same weight and record the percentage of Water in Annexure -II
- 4.2.4 Limit of water in Disodium Tartrate: $15.66 \pm 2\%$.
- 4.2.5 Frequency (KF Factor): Daily
- 4.2.6 Calibration :Monthly
- 4.2.7 Record the use of instrument in instrument logbook.

5.0 ANNEXURE (S):

- Annexure – I: Daily Factor verification of Karl Fischer.
- Annexure – II: Monthly Calibration Record for Karl Fisher.

6.0 REFERENCE (S):

7.0 ABBREVIATION (S)/ DEFINITION (S):

- KF : Karl Fischer
- RSD: Relative Standard Deviation



PHARMA DEVILS
QUALITY CONTROL DEPARTMENT

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REVISION CARD

| S.No. | REVISION No. | REVISION DATE | DETAILS OF REVISION | REASON (S) FOR REVISION | REFERENCE CHANGE CONTROL No. |
|-------|--------------|---------------|---------------------|-------------------------|------------------------------|
| 01 | 00 | --- | --- | New SOP | --- |



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DAILY FACTOR VERIFICATION OF KARL FISCHER

Reference SOP No.

| | | | |
|---------------------------|--|--------------------------|--------|
| Location | | Page No. | 1 of 1 |
| Manufactured By | | Model No. | |
| Identification No. | | Date of performed | |

| S.No. | Distilled water | Karl Fischer use (in ml) | KF Factor | Mean value of KF Titer | % RSD (NMT 1.0%) | KF Factor (Between 4.5 to 6.5) |
|-------|-----------------|--------------------------|-----------|------------------------|------------------|--------------------------------|
| 1. | 10 μ l | | | | | |
| 2. | 10 μ l | | | | | |
| 3. | 10 μ l | | | | | |

Opinion: The Karl Fischer Apparatus is Ok / Not Ok for analysis.

Performed By:

Checked By:

Date

Date:

ANNEXURE II

MONTHLY CALIBRATION RECORD FOR KARL FISHER

Reference SOP No.:

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|----------------------------|--|-----------------------------|--|
| Manufactured By | | Model No. | |
| Identification No. | | Frequency | |
| Date of Calibration | | Calibration Due Date | |

| S.No. | Wgt. of Di-Sodium Tartrate (in mg) | Karl Fischer use (in ml) | Water % | Limit 15.66 ± 2 % |
|--------------|---|---------------------------------|----------------|--------------------------|
| 1. | | | | |
| 2. | | | | |
| 3. | | | | |

Opinion: The Karl Fischer Apparatus is Ok / Not Ok for analysis.

Calibrated By:

Checked By:

Date

Date: