TITLE: OPERATION AND STANDARDISATION OF KARL FISHER TITRATOR

1.0 PURPOSE:

To describe the operation and Standardization procedure for the KF Titrator.

2.0 SCOPE:

This procedure is applicable to the digital KF Titrator of Laboratory.

Make : VEEGO

Model : VEEGO/MATIC-MD+PC

Instrument No. : DIPL/QC/INS/KF/002

3.0 RESPONSIBILITY:

3.1 Analyst-QC is responsible to follow this SOP.

3.2 Head-QC/Designee is responsible for ensuring implementation of this SOP.

3.3 Head-QA/Designee is responsible for monitoring overall compliance of this SOP.

4.0 DEFINITIONS:

Nil

5.0 PROCEDURE:

5.1 OPERATION:

5.1.1 Clean and dry the reaction Vessel

5.1.2 Assemble the apparatus

5.1.3 Fill the reservoir with Karl Fisher reagent, ensure that delivery is free from air Bubbles

5.1.4 Switch on the apparatus and adjust the speed of the magnetic stirrer.

5.1.5 The Display will indicate.

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5.1.6 The message will remain "NO" for few Seconds and will change to

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5.1.7 Press START Key, the massage will appear as

Add Sample Conf. Time =10 secs

5.1.8 The 4th line shows the stirring time (the set stirring time is 10 secs, this is 10 secs, This time can vary from 1 sec to 20 sec). When the time decrements to "0". The Display will indicate as

Add Sample Conf. Time "10" secs

5.1.9 Then dispensing unit will start adding K/F reagent into the beaker through delivery Tube the display will indicate as -

TITRATION STARTED 00:01 ml Conf. Time: 20 secs

5.1.10 2nd Line of the display indicates the consumption of KF reagent in ml. 4th 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. The display will Indicates as -

TITRATION STARTED 06:52 ml Conf. Time: 00 secs

Note: when the addition of KF reagent is progress, the counts will increase, elapsed Time will indicate 20secs. When the addition of KF reagent stops, the time will start Decrementing. If the moisture still exists in the titration vessel, the addition of KF reagent will continue the elapsed time will indicate 20sec, again. When the total moisture is neutralized, the addition of KF reagent and the counting will stop, the (elapsed) time will Count down to"00" and audio signal will indicate titration is over.

5.1.11 The intermittent addition of reagent will not stop until the elapsed time of 20 sec a down Every time does not count down to '00' between two additions) the display

will indicate as -

K/F Reagent Reading 06:52 ml

5.1.12 To make titration vessel moisture free, follow the above procedure (i.e.: 1.7 to 1.10) 2 to 3 Times. This indicates that the methanol is neutralized (or titration vessel is moisture free)

5.2 **KF FACTOR:**

The titer factor can be determined in two ways.

5.2.1 **1st Procedure**

5.2.1.1 Press CAL-1 Key and add 10µl of distilled water in the titration vessel.

The display will Indicate

Add 10 µl distilled water and press Enter to Cont

5.2.1.2 Press ENTER Key The display will indicate

Add sample Conf. Time : 05 Secs

5.2.1.3 The 4th line indicate the external stirring time, when the time counts down to: 00". The Dispensing unit will start adding K/F Reagent in the titration vessel. The display will indicate -

TITARTIN STARTED 00:00 Conf. Time : 20 Sec

5.2.1.4 2nd line of the display indicates the consumption of KF reagent to neutralize the 10 mg of moisture added into titration vessel. Every time the addition takes place, the counts Increase accordingly.

5.2.1.5 4th line of the display indicates the time duration of end point. (i.e. 20secs) After the Completion of titration if the end point lasts for 20 secs, indicates the titration is over by an audio signal. For example if the display indicate

TITARTIN STARTED 02:00 Conf. Time: 00 Sec

and titration over is indicated by audio signal and the display indicates as

K/F reagent reading : 2.00

5.2.1.6 The above message will remain 'ON' for few seconds on the display and change over to

Wait calculating Factor

$$Formula: Titer\ factor = \frac{Wt \in mg's\ of\ water}{Reading \in ml\ of\ reagent}$$

As wt of 10µl of water is 10 mgs

It is $= \frac{10\,mgs}{2ml}$ = 5 mgs of H_2O per cc of reagent

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

Factor = 05.000
Repeat test for New factor
New factor
YES / NO

5.2.1.7 The printer port is provided at the rear of the instrument. If the 80 column dot matrix Printer is attached to the instrument with paper loaded and kept "ON" the heading and 1st Reading of the titer factor will be printed.

5.2.1.8 'YES' massage is flashing. If one wants to repeat the test to find out new factor, then press ENTER key the display will indicate as

Add 10µl distill water
And Press
Enter to cont.

Follow the procedure as per para 4.2.1 to 4.2.5

5.2.1.9 In this way, 6 difference readings to calculate the KF factor can be taken. After the 6th Factor, the display for example indicates as-

Factor = 05.0120
Repeat test for
New factor
YES/NO

5.2.1.10 This message will appear every time till it is decided that no new factor is necessary. (For Example we will take values of the first three factors) Press ENTER Key.

The message will appear as -

Factor 1 = 05.0000
Factor 2 = 05.1500
Factor 3 = 05.2530

ENTER TO CONT.

5.2.1.11 The factor 1 massage is flashing. On pressing 'ENTER' key again, 'factor 2' will be flashing. On Pressing ENTER key again, 'factor 3; will be flashing. Then pressing ENTER key again, the massage will change over to

Factor 4 = 05.0000
Factor 5 = 05.0350
Factor 6 = 05.0120

ENTER TO CONT.

'Factor 4' massage is flashing.

5.2.1.12 If operator wants to see the factor -1, 2, or 3 he has to press key

UP PG ---------- D

5.2.1.13 The massage will appear as -

Factor 1 = 05.0000 Factor 2 = 05.1500 Factor 3 = 05.2530 ENTER TO CONT.

Factor 1 massage is flashing.

If user wants to delete any factor (for example- factor 2 and Factor 3)

Press ENTER key The “Factor 2’ message will start flashing.

Press CE/CLEAN key, the factor 2 reading will be deleted and the row will become Blank.

Press ENTER key the “Factor 3’ message will start flashing .Press CE/CLEAN key, the factor 3 reading will be deleted and the row will become blank. The massage will appear as -

Factor 1 = 05.0000 Factor 2 = 05.1500 Factor 3 = 05.2530 ENTER TO CONT.

5.2.1.14 Instead of pressing three times ENTER key, one can press

UP PG ---------- D

key to change the massage

Factor 4 = 05.0000 Factor 5 = 05.0350 Factor 6 = 05.0120 ENTER TO CONT.

5.2.1.15 'Factor 4' message flashing. If user wants to delete the Factor 5, press ENTER key. 'Factor 5' massage will start flashing.

Press | CE / CLEAN | key

5.2.1.16 Then "Factor 5'reading will get deleted and that row will become blank.

The massage will appears as

Factor 4 = 05.0000 Factor 5 = 05.0350 Factor 6 = 05.0120 ENTER TO CONT.

Press ENTER Key two times.

5.2.1.17 The massage will appear as

WAIT Calculating Average factor

5.2.1.18 The above massage will remain 'ON' for few seconds.

5.2.1.19 The massage will appear as

Avg. Factor = 05.0040 PRESS ENTER TO CONT.

5.2.1.20 It will take the average of factor 1, 4&6. Deleting Factor Nos: 2, 3&5 as required. If the Factor reading are not to be deleted, it will take all the 6 readings of the titre factor and Calculate the average factor.

5.2.1.21 If user does not want to take all the 6 readings, if he wants to take only three readings, say After 3rd reading, i.e. the message appearing on the display for example is

Factor = 05.0000 Repeat test for New factor YES/NO

5.2.1.22 'YES' message is flashing..

5.2.1.23 Press Key

0 ------- UNIT SET

5.2.1.24 'NO' message will start flashing. Press ENTER Key.

Factor 1 = 05.0000 Factor 2 = 05.1500 Factor 3 = 05.2530 ENTER TO CONT.

5.2.1.25 Then the message will appear as -

5.2.1.26 For example we will use these above factors.

5.2.1.27 To go to next message, either press ENTER key three times or

Press

* PG	UP ----- DN

Key

Factor 4 = 05.0000 Factor 5 = 05.0350 Factor 6 = 05.0120 ENTER TO CONT.

5.2.1.28 The message will appear as

5.2.1.29 Press ENTER Key tree times, the message will appear as

WAIT Calculating Average Factor

5.2.1.30 The message will remain for few seconds and change over to, the message will appear as

AVG. FACTOR : 05.0040 PRESS ENTER TO CONT.

Note: If Printer is 'ON', it will print all the six titre factors and also the average value. It will also print the factor numbers which are deleted

5.2.2 **CAL II Key:**

On pressing this key, user can find out the titer factor by using Sodium –tart rate Dehydrate which is a standard powder known as DST. On pressing this key, the massage will appear as

Massage will appear as

ADD DST PRESS ENTER TO CONT.

For example: Add DST fine powder wt =115 mgs, press ENTER key.

5.2.2.1 Message will appear as

WAIT EXTRACTING MOISTURE EX TIME = 20 sec

When the time decrements to 00 secs. The dispensing unit will start adding the KF Reagent.

5.2.2.2 The display will appear as

Karl Fisher Reagent 00.10 ml Conf. time : 20 Sec

When the total moisture is neutralized, the conf. time will decrement to '00' secs.

5.2.2.3 The display will then appear as

Karl Fisher Reagent 03.05 ml Conf. time : 20 Sec

If for example 03.05ml of K/F reagent is consumed.

5.2.2.4 Then display will change to

KF Reading = 3.05 ml

5.2.2.5 The above message will remain 'ON' for few seconds and then it will change over to

ENTER DST WEIGHT 0.02355 gm Press ENTER to cont.

5.2.2.6 2nd line os indicating the weight of DST of last test.

5.2.2.7 To enter the new weight =115 mgs, press [0][.][1][1][5]

5.2.2.8 The display will appear as

Calibration Weight 000.115 mgs Any key to change Press Enter to cont.

5.2.2.9 If weight is not entered properly, proceed as under: Press any key the display will appear as

ENTER DST WEIGHT 000.115 mgs Any key to change Press Enter to cont.

5.2.2.10 Now enter the correct weight. Press ENTER Key

5.2.2.11 The display will appear as

Wait Calculating Factor

5.2.2.12 This message will remain for few seconds and will change over to

Factor = 05.9045 Repeat test for New factor YES / NO

5.2.2.13 Yes message is flashing. If one wants to continue, press ENTER Key.

5.2.2.14 If one wants to find out all six factor, follow the procedure as from para 4.2.7

5.3 FACTOR KEY

5.3.1 When the reagent is calibrated (i.e. titre Factor) by using CAL-1 and CAL-II key, the Reading of titer factor goes in memory. To see the reading if, necessary, press FACTOR Key.

5.3.2 The same key i. e factor key can be used to enter the titer factor manually if desired.

5.3.3 FACTOR key, the display will appear as - As an example, press

K.F REAGENT FACTOR
05.0000 mgs/ml
PRESS ENTER TO CONT.

(The figure displaying on the screen may be of last checking)

5.3.4 Suppose if one wants to enter the new titer factor which is manually calculated by way of pressing 'START' key and adding known quantity of moisture).
Such as titer factor = 6.25
Press [6][*][2][5] keys

5.3.5 The display will appear as

K.F REAGENT FACTOR
0006.25 mgs/ml
PRESS ENTER TO CONT.

Press ENTER Key.

5.3.6 The display will appear as

K.F REAGENT READING
01.40 ml

(The second line may be last test)

5.4 **TO TEST THE SAMPLES**

5.4.1 Press the START Key the display will appear as

ADD SAMPLE
CONF. TIME: 10 Secs

Note: Sample should be added after pressing the "START' key only

5.4.2 When the CONF.TIME decrements to 00 seconds. The dispensing unit will start adding the K.F Reagent and on the display the counting will start.

5.4.3 The display will appear as

TITRATION STARTED
00.10 ml
CONF. TIME: 10 Secs

Second line of the display shows the consumption of K/F reagent in ml. when the moisture is neutralized , the counting will stop , conf. time will start decrementing , If on Decrementing the time, the moisture still remains before the time reaches'00' the dispensing Unit will start again and will add the K/F Reagent. The counts will increase according. When the complete moisture is neutralized, the conf. time decrement to 00 seconds and the Audio signal will indicate the end point is reached.

5.4.4 The display will appear as

KFR READING
02.88 ml

Taking for granted, the titre factor of reagent =6.25

18 mgs =6.25x2.88

5.4.5 To find out % and PPM.

5.4.6 Enter the sample quantity.

5.4.7 Press

9
Sample Quantity

Key.

5.4.8 The display will appear as –

Sample quantity
00000003 gm/ml
Press Enter to Continue.

(The quantity which is showing on display may be of last test). To enter the weigh = 150 mgs, user has to enter the weight in Gms i.e.0.150 Gms.

5.4.9 Press [0] [.0] [1] [5] [0] the display will indicate as

Sample quantity 00000003 gm/ml Press Enter to Continue.

Press Enter Key

K.F.R. Reading 02.88 ml.

5.4.10 The Display will appear as -

7 %   PPM

5.4.11 Press Key.

WAIT Calculating % and PPM

5.4.12 The display will appear as -

% = 12.000 PPM = 120000.00 Mg of H_2O =18.0000

And the display will change over to - Press ENTER Key.

	Prepared by	Reviewed by	Checked by
Sign & Date			
Name	A.Navya	S.Prasad	Ch.Mahendar Reddy
Department	Quality Control	Quality Control	Quality Assurance

5.4.13 The display will appear as-

KFR Reading
02.88 ml

5.5 **TO TEST THE SAMPLES**

5.5.1 **To test the liquid Sample:**

Example- Methanol: Add 10ml, as Sample.

Press START Key. The display will appear as-

ADD SAMPLE
CONF. TIME=10 Secs.

5.5.2 **Note: sample should be added after pressing 'start' key.**

5.5.3 When the total moisture is neutralized, CONF. Time decrements to '00' Seconds

The Display will appear as-

TITRATION STARTED
00.10 ml.
CONF. TIME=20 Secs.

5.5.4 When the total moisture is neutralized, CONF.TIME decrements to '00' seconds

The display will appear as-

TITRATION STARTED
00.56 ml.
CONF. TIME=00 Secs.

(Quantity of reagent 00.56 is taken as example).

5.5.5 A audio signal will indicate the end point is established.

5.5.6 The display will indicate as-

KFR Reading
00.56 ml.

5.5.7 To find out %and PPM to enter the sample qty =10 ml.

5.5.8 Since the Sample qty added is ml, is necessary to enter the density of solvent, Methanol density such as 0.793

5.5.9 Press

9
SMPL QTY

Key.

5.5.10 The display will appear as-

SAMPLE QUANTITY
000003gm/ml
PRESS ENTER TO CONT

5.5.11 The third line indicating the gm/ml of sample quantity may be of last check. To enter the new value .Press [1] [0].The display will indicate as-

SAMPLE QUANTITY
0000010gm/ml
PRESS ENTER TO CONT

Press ENTER Key.

5.5.12 The display will appear as-

KFR Reading
00.56 ml

5.5.13 To enter the density, i.e. = 0.793, Press

8
DENSITY

Key.

The display will appear as-

DENSITY
00000010 kg/L.
PRESS ENTER TO CONT

Second line of the display is indicating the density as 1(which is the set value).

5.5.14 To enter the new density i.e.0.793, Press [0] [.] [7] [9] [3].

5.5.15 The display will appear as-

DENSITY
0000.793 mg/L.
PRESS ENTER TO CONT

Press ENTER Key

5.5.16 The display will appear as-

KFR Reading
00.56 ml

5.5.17 Press

7 %
PPM

Key.

5.5.18 The display will appear as -

WAIT
CALCULATING % and PPM

5.5.19 The above massage will appear for few seconds and will change to-

% = 0.044
PPM = 440.000
Mg of H_2O = 3.5000
PRESS ENTER TO EXIT

Press ENTER Key.

5.5.20 The display will appear as-

KFR Reading
00.56 ml

5.5.21 To test the next sample, press START key and follow the procedure as per Paragraph 4.4.2

**If the time is lost, then the fresh end point should be obtained and then press The START key.

5.6 **SOLVENT BLANK**: (i.e. Dilution of sample in a solvent)

5.6.1 Example: If the sample is having low moisture, it is not possible to add large quantity of sample or if the sample contains high % of moisture like 30% or more It requires to add very small quantity of sample. In this case, the dilution technique is suitable.

5.6.2 Example: For moisture of order of 0.1%, the sample quantity required is at least 10gms So that the mgs of H_2O available is 10 mgs

5.6.3 For such samples, the dilution technique is suitable.

5.6.4 Dissolve the sample quantity 10 gms in 20ml of methanol

5.6.5 Determine the moisture in the methanol also. I.e Blank Reading of methanol only Known as 'Solvent Blank'

5.6.6 Add 1 ml of methanol in the titration vessel.
(If time is lost, obtain the fresh end point by pressing the START key and wait till the Audio signal indicating fresh end point)

5.6.7 Press START key and follow the procedure as paragraph 4.4

5.6.8 Suppose the reading of K/F reagent to neutralize 1 ml of methanol =0.05ml

5.6.9 The display will appear as -

KFR Reading
00.56 ml

5.6.10 To enter the reading into solvent blank mode,

Press

5
SOLVENT BLANK

Key

5.6.11 The display will appear as -

SOLVENT BLANK
0000000 ml
PRESS ENTER TO CONT

5.6.12 To enter the volume, press [0],[.][0][5]

The display will appear as -

SOLVENT BLANK
00000.50 ml
PRESS ENTER TO CONT

Press ENTER Key

5.6.13 The display will appear as -

KFR Reading
00.05 ml

5.6.14 Now add 1 ml of diluted sample (without losing any time gap). (Remember sample is diluted by adding 10gms of sample in 20 ml of methanol)

5.6.15 Press START Key and follow the procedure as per paragraph 4.4.15 to 4.4.18

5.6.16 The display will appear as -

KFR Reading
01.15 ml

Taking for granted, the consumption of K/F reagent required for 1 ml of diluted sample.

5.6.17 To enter the sample quantity, i.e .0.500 Gms

(Since 20 ml of methanol =10 gms of sample, 1 ml of methanol =0.500 gms of sample)

5.6.18 Press

9
SMPL QTY

key

5.6.19 The display will appear as

SAMPLE QUANTITY
0000.500gm/ml
PRESS ENTER TO CONT

5.6.20 Press ENTER Key the display will appear as -

KFR Reading
01.15 ml

5.6.21 To find out % and PPM, Press

7	%
	PPM

Key

$$Formula = T.F \times \frac{(Reading - solvent\ blank\ reading)}{weight\ of\ the\ sample \in gms} \times 100$$

$$\% = 6.25 \times \frac{(1.55 - 1.05)}{0.500} \times 100$$

5.6.22 The display will appear as -

WAIT
CALCULATING % and PPM

5.6.23 This message will remain for few seconds and change to

% = 0.625
PPM = 6250.000
Mg of H_2O = 3.1250
PRESS ENTER TO EXIT

5.6.24 ENTER Key The display will appear as

KFR Reading
01.15 ml

5.7 **EXTRACTION TIME:**

5.7.1 In most of the liquid samples, the moisture is extracted by the methanol in Titration vessel, as soon as it is added. In the case of high viscous liquid and from some of the solid sample, moisture may not be extracted immediately. In such samples, Extraction time is essential to extract the moisture.

5.7.2 During this extraction time all functions are stopped. Only stirring is kept 'ON' to extract the moisture. When 'EXTRACTION TIME' is over, the dispensing unit will start adding K/F Reagent

5.7.3 The 'EXTRACTION TIME' is set to '10' seconds, but this can be varied from 1seconds to 240 seconds as per requirement.

5.7.4 The display is indicating as

KFR Reading 00.56 ml

Second line is showing the reading of K/F reagent may be of last test.

5.7.5 Press key

4 EX TIME

5.7.6 The display will appear as

EXTRACTION TIME 0010 Secs PRESS ENTER TO EXIT

Second line if display is showing set value of 10 seconds

5.7.7 To Change the time, say 20 seconds, press [2][0]. The display will indicate as

EXTRACTION TIME 0020 Secs PRESS ENTER TO EXIT

5.7.8 Press ENTER key. The display will indicate as –

KFR Reading 00.56 ml

5.7.9 If user wants to cancel the Ex. Time of 20 seconds and go to 'SET VALUE', it can be Done by pressing the CE key or, can over write.

Example:

EXTRACTION TIME
0020 Secs
PRESS ENTER TO EXIT

5.7.10 Press CE CLEAN key or Press [1][0].

5.7.11 The massage will appears as

EXTRACTION TIME
00.10 Secs
PRESS ENTER TO EXIT

5.7.12 Press ENTER Key. The display will indicate as

KFR Reading
00.56 ml

5.8 **CLEANING:**

5.8.1 Press CLEAN Key. The display will appear as

Are you ready for cleaning any key to EXIT Enter to continue

5.8.2 Press ENTER Key. The massage will appear as

Remove Glass adapter from K/F Reagent bottle and connect to methanol bottle

5.8.3 Press ENTER Key

Connect Glass STOPPER TO K/F Reagent Bottle

5.8.4 Press ENTER Key

WAIT CLEANING STARTED PRESS CLEAN TO STOP

5.8.5 The dispensing unit will start sucking the methanol. To ‘Stop’ in between, press ‘STOP’ key again. OR The system will ‘STOP’ automatically after few minutes.

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5.9 **BATCH NO:**

5.9.1 To enter batch number as 1234, press BATCH NO key. The display will appear as

BATCH NUMBER 0000 Press ENTER to cont.

5.9.2 Press [1][2][3][4] the massage will appear as

BATCH NUMBER 124 Press ENTER to cont.

5.9.3 Press ENTER key. The massage will appear as

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5.10 **DISPENSER CALIBRATION:**

5.10.1 **Calibration Frequency:** Every 2Months (±3 days)

5.10.2 Fill the sufficient amount of Methanol into reservoir and connect it to the "INLET' of the dispenser. Connect the 'OUT LET' of the dispenser to a small beaker.

5.10.3 Allow the dispenser to run for sometime so that the inlet and outlet tubes are filled with water. This can be done by running the clean cycle of the dispenser.

5.10.4 Thus the dispenser is now ready for calibration. After air bubble is removed.

5.10.5 On pressing the 'CLEAN' key the display will show the massage.

DO YOU WANT TO VALIDATE DISPENSER ANY KEY TO EXIT ENTER TO CONTINUE

5.10.6 If you want to continue with Validation or Calibration of the dispenser, Press ENTER Otherwise press any key to exit. On Pressing then ENTER key the display will show the Message

DISPENSER READY
ANY KEY TO EXIT
ENTER TO CONTINUE

5.10.7 If the dispenser is ready after removing air bubbles etc., then to continue with Calibration of the dispenser, press ENTER otherwise press any key to exit. On pressing the ENTER key the display will show the message.

SELECT MODE
VALIDATION/CALIBRATION
SLECT TO CHANGE
ENTER TO CONTINUE

5.10.8 Observe that the message ‘VALIDATE’ is bilking, on pressing the SELECT key the Message “CALIBATE’ will blink. Press SELECT to choose the either of the modes.

5.10.9 Press SELECTS to choose ‘CALIBRATION’ mode. Observe that on pressing the SELECT key the message ‘CALIBRATE’ is blinking. Now press ENTER to continue. On pressing the ENTER to continue. On pressing the ENTER key the display will Show the message.

DISPENSER READY

ANY KEY TO EXIT
ENTER TO CONTINUE

5.10.10 If the dispenser is ready after removing air bubbles etc., then to continue with calibration of the dispenser, Press ENTER otherwise press any key to exit. On pressing the ENTER key the display will show the message

ENTER REQUIRED VALUME
00.00ML

PRESS ENTER TO CONTINUE

5.10.11 Now enter the required volume to be dispensed for calibrating the dispenser, say 10.00ML, after entering the required volume the display will show.

ENTER REQUIRED VALUME
10.00ML

PRESS ENTER TO CONTINUE

5.10.12 Now press ENTER to continue the calibration. On pressing the ENTER the display Will Show

PLACE THE CYLINDER
AND
ENTER TO CONTINUE

5.10.13 Now place the measuring cylinder just below the delivery tip and press ENTER. The Display will show

WAIT
ADDING LIQUID
00.00ML

5.10.14 Observe that the dispenser is adding the Methanol into the beaker and display indicates Amount of Methanol being dispensed, after 10.00 ml water is dispensed, the dispenser Stops and the display shows the message.

ENTER DISPENSED VOLUME VALUME
00.00ML
PRESS ENTER TO CONTINUE

5.10.15 Now take the dispensed i.e. Volume of methanol. if the volume dispensed is 9.70 ml. Now on entering the volume dispensed as 9.70 ml the will show.

ENTER DISPENSED VOLUME VALUME
09.70 ML
PRESS ENTER TO CONTINUE

5.10.16 After entering the volume press ENTER. The display will show.

ENTER DISPENSED VOLUME VALUME
09.70 ML
ANY KEY TO CHANGE
PRESS ENTER TO CONTINUE

5.10.17 To edit the dispensed volume, press any key so that the volume can be edited. Otherwise Press ENTER Key to confirm. On pressing the ENTER Key the display will show

ARE YOU READY FOR
NEXT RUN
ANY KEY TO ABORT
ENTER TO CONTINUE

5.10.18 The same experiment is repeated thrice and the average volume is calculated. To continue with the calibration press ENTER Key and repeat the experiment again. Each time calculate the dispensed volume and enter it properly. If you do not want to Continue with calibration further, press any key to abort. In that case the average of whatever. After the calibration is over the display will show the Message with an. Audio signal for few seconds

CALIBRATION OVER

The message will be cleared automatically after few seconds.

5.11 **DISPENSER VALIDATION:**

5.11.1 **Validation Frequency:** Every 2 Months (±3days) Acceptance Criteria: ±0.20ml

5.11.2 Fill the sufficient amount of methanol into reservoir and connect it to the "INLET' of the dispenser. Connect the 'OUT LET' of the dispenser to a small beaker.

5.11.3 Allow the dispenser to run for sometime so that the inlet and outlet tubes are filled with water. This can be done by running the clean cycle of the dispenser.

5.11.4 Thus the dispenser is now ready for calibration. After air bubble is removed.

5.11.5 On pressing the 'CLEAN' key the display will show the massage.

DO YOU WANT TO
VALIDATE DISPENSER
ANY KEY TO EXIT
ENTER TO CONTINUE

5.11.6 If you want to continue with Validation of the dispenser, Press ENTER otherwise Press any key to exit. On Pressing then ENTER key the display will show the message.

DISPENSER READY
ANY KEY TO EXIT
ENTER TO CONTINUE

5.11.7 If the dispenser is ready after removing air bubbles etc., then to continue with validation of the dispenser, press ENTER otherwise press any key to exit. On pressing the ENTER Key the display will show the message.

SELECT MODE
VALIDATION/CALIBRATION
SLECT TO CHANGE
ENTER TO CONTINUE

5.11.8 Observe that the message “VALIDATE” is blinking, now press ENTER to continue for the validation of the dispenser. On pressing the ENTER key the display will show the massage.

DISENSER READY
ANY KEY TO EXIT
ENTER TO CONTINUE

5.11.9 If the dispenser is ready after removing air bubbles etc. then to continue with validation of the dispenser, press ENTER otherwise press any key to exit. On pressing the ENTER Key the display will show the message.

ENTER REQUIRED VOLUME 00.00ML PRESS ENTER TO CONTINUE

5.11.10 Now enter the required volume to be dispensed for validating the dispenser, Say 10.00ml. after entering the required volume the display will show.

ENTER REQUIRED VOLUME 10.00ML PRESS ENTER TO CONTINUE

5.11.11 Now press ENTER to continue the. On pressing the enter the display will show

PLACE THE CYLINDER AND ENTER TO CONTINUE

5.11.12 Now place the measuring cylinder just below the delivery tip and press ENTER. The Display will Show

WAIT ADDING LIQUID

5.11.13 Observe that the dispenser is adding the methanol into the beaker and the display Continues to show above message After 10.00 ml methanol is dispensed, the dispenser Stops and the display show the message

CHECK THE DISPENSER VALUME
PRESS ENTER TO CONTINUE

5.11.14 Measuring the methanol Volume. Repeat the validation three times

6.0 FORMATS / ANNEXURE(S):

6.1 KF Standardization Record : QC032-FM066

6.2 Calibration of KF Titrator : QC032-FM067

6.3 Validation of KF Titrator : QC032-FM068

6.4 Usage Log Book : QC048-FM086

7.0 CHANGE HISTORY:

Revision No.	Effective Date	Details of Revision	Ref CCF No.
00	07.06.2013	New Sop Introduced for KF Titrator	--
01	01.06.2014	Formats are the part of SOP. So prepared Separately	--
02	01.01.2017	SOP format make to in line with SOP-QA-001-04.	QC-CRF-025/16