Design Qualification

Microprocessor Based
Karl Fischer Titrator
Model: µAquaCal$_{10+}$

DESIGN QUALIFICATION

Protocol Number : ASIPL/DQ/

Company Name :

Location :

Instrument : Microcontroller based Karl Fischer Titrator

Model : µAquaCal$_{10+}$

Instrument Sr. No. :

Protocol Approved By : Analab Scientific Instruments Pvt. Ltd.

Date :

Signature :

Name :

Analab Representative	Checked by End User	Approved by Head of Department

DESIGN QUALIFICATION

"Design qualification (DQ) defines the functional and operational specifications of the instrument ". DQ should ensure that instruments have all the necessary functions and performance criteria that will enable user to be successfully implemented for the intended application and to meet business requirements.

Technical Specification :

- Method of Detection : Conductometric / Volumetric
- Moisture Detection Range : Better than 100 ppm to 100% (depends upon type of sample)
- Dispensing Resolution : 0.01ml
- Magnetic Stirrer : Built-in with soft touch Key speed controller
- Auto Neutralization : On / Off - users selectable - reduces the time between analysis & gives accurate results
- Stirring Time : 00:00 to 99:00 (mm:ss) – users adjustable
- End Point Sensitivity : 80-100 - users adjustable
- End Point Time : 1-20 seconds - users adjustable
- End Point mV : 1000-3000 mV - users adjustable
- End Point Detection : By Audio & Visual Alarm
- Over titration Alarm : Audio / Visual Alarm at 15 ml to save KF Reagent
- Display of Reading : Moisture in ppm & percentage (%)
- Auto Drain : For cleaning reaction vessel - useful for large & multiple volume sample
- Flush : For cleaning dispensing path
- Average KF Factor : Manual Calculation
- Dispenser Calibration : Onsite by users
- Dispenser MOC : SS-316L & Teflon
- Dispenser Type : Motorized
- Teflon Tube : To transfer KF reagent from reagent bottle to reaction vessel
- Material of Construction : Epoxy Based Powder Coated Metallic Body / GMP Enclosure made up of Stainless Steel
- Type of Electrode : Dual Platinum - Combined Type
- Input : Electrode with Banana Connector
- Error Messages : “Over KF”, “KF Factor Error”
- Short Link : To diagnose problem either with Instrument OR Electrode
- Display : 16 x 2 Line Alphanumeric Backlit LCD
- Keypad : Membrane waterproof polycarbonate soft touch keypad compliance to UL/ROHS
- Power Supply : 230VAC ±10%, 50 Hz

The supplied Instrument is according to the Purchase Order and as per specification.

Date :

Signature :

Name :

Analab Representative	Checked by End User	Approved by Head of Department

DESIGN QUALIFICATION

The Design Qualification (DQ) for the Microprocessor based Karl Fischer apparatus has been reviewed and was found acceptable for use.

INSTRUMENT : Microcontroller based Karl Fischer Titrator
MODEL : µAquaCal$_{10+}$
SR.NO. :
FINAL REPORT :
REVIEWED AND
ACCEPTED BY :
(Name & Designation)
DATE :

Customer	
Department	
Head of Dept.	
End User	
Date	
Signature	
Seal of the Company	

Analab Representative	
Date	
Signature	

Date :

Signature :

Name :

Analab Representative	Checked by End User	Approved by Head of Department

Installation Qualification
&
Operational Qualification

Microcontroller Based
Karl Fischer Titrator
Model: µAquaCal$_{10+}$

Installation & Operational Qualification

Protocol Number	**:**	ASIPL/IQ-OQ/
Company Name	**:**	
Location	**:**	
Instrument	**:**	Microcontroller Based Karl Fischer Titrator
Model	**:**	µAquaCal$_{10+}$
Instrument Sr. No.	**:**	
Protocol Approved By	**:**	Analab Scientific Instruments Pvt. Ltd.

Date :

Signature :

Analab Representative	Checked by End User	Approved by Head of Department

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Date :

Signature :

Analab Representative	Checked by End User	Approved by Head of Department

1 INSTALLATION QUALIFICATION:-

Installation qualification establishes that the ordered instrument is received as designed and specified, that it is properly installed in the specified environment, and that this environment is suitable for the operation and use of the instrument.

1.1. Instrument Identification & Verification

Customer Purchase Order No. _____ Date_____

Main System Contents.

Sr. No.	Description	PO Qty	Received Qty.	Remark	
				Ok	Not ok
01	Micro Controller based Karl Fischer Titrator Model : µAquaCal$_{10+}$	01			
02	250 reaction Vessel	01			
03	Dual Platinum combined Electrode	01			
04	Teflon Paddle	01			
05	Teflon assembly	01			
06	Moisture Trap	02			
07	250 ml Amber Reservoir Bottle	01			
08	Dispensing Tube	02			
09	Micro Tips	02			
10	Teflon tube for dispensing path	01			
11	Power cord	01			
12	Operational & Instruction manual with SOP	01			
13	Calibration test certificate (Without Traceability Documents)	01			
14	Warranty Card	01			
15	DQ/IQ-OQ/PQ Documents				

The supplied Instrument is according to the Purchase Order and as per specification.

Date :

Signature :

Analab Representative	Checked by End User	Approved by Head of Department

1.2. Pre-Installation Requisites & Verification

Check the installation place for fulfillment of manufacturer's recommendation. (Utilities such as Power requirement, necessary chemicals and environmental conditions such as humidity, temperature, light and dust etc.(Please refer Pre-Installation requisites attached with this document.)

Item	Condition	Observation	
		Ok	Not Ok
Instrument & Accessories appearance check	No Damage and Breakage to instrument & Accessories.		
Installation Table space Leveling & Vibration.	As per requirements.		
Power Requirements • Connections sockets • Supply Voltage • Earthing (Earth to Neutral Potential	 • 5 Amps. 02 no. • 230 V AC +/- 10% • Not more than 3 (Three) Volts.		
Environment conditions. 1) Sunlight 2) Air Draft 3) Room Temp. 4) Humidity 5) Dust 6) Flammable, Toxic & Corrosive Vapors	 Away from sunlight No direct fan or AC 15 ° ~ 50° C 20 ~ 80 % Free from dust Free from vapor.		

Date :

Signature :

Analab Representative	Checked by End User	Approved by Head of Department

1.3. Installation Qualification Verifications

1.3.1. Instrument Identification & Verification

SR. NO.	DESCRIPTIONS	RESULT	
		Yes	No
A	Items received as per Purchase Order		

1.3.2. Pre-Requisites Inspection

SR. No.	DESCRIPTIONS	RESULT	
		Yes	No
A	Instruments & Accessories Checked for Damages		
B	Table Space & Leveling		
C	Power Supply Availability		
D	Environmental Requirements		

1.3.3. Standard & Optional Accessories (as ordered)

SR. NO.	DESCRIPTIONS	RESULT	
		Yes	No
A	250 reaction Vessel		
B	Dual Platinum combined Electrode		
C	Teflon Paddle		
D	Teflon assembly		
E	Moisture Trap for reaction vessel & for reservoir		
F	250 ml Amber Reservoir Bottle		
G	Dispensing Tube		
H	Micro Tips		
I	Teflon tube for dispensing path		
J	Power Cord		
K	Operational & Instruction manual with SOP		
L	Calibration test certificate (Without Traceability Documents)		
M	Warranty Card		
N	DQ/IQ-OQ/PQ Documentation (Optional)		
P			

Date :

Signature :

Analab Representative	Checked by End User	Approved by Head of Department

2 Operational Qualification

Operational Qualification (OQ) is the process of demonstration that an instrument will function according to its operational specifications in the selected environment.

2.1. Customer Orientation & Training

Orientation for the end user/s will be performed as per the Instruction Manual.

Sr. No.	Orientation Topic	Status	Verified	
			Yes	No
1	Instrument Setup/Connectivity with Accessories, Peripherals and Mains etc.	Explained / Shown		
2	Instrument Front Panel – Key Function	Explained / Shown		
3	Instrument Back Panel – Connection	Explained / Shown		
4	Instrument Mode & its function • Neutralization • Calibration • Analysis • Utilities	Explained / Shown		
5	Instrument & Accessories Maintenance	Explained / Shown		

The Training on the above topics is given to following personnel

Personnel Trained	Designation	Location	Verified

The Instrument orientation and training were carried out in accordance with the Instruction Manual and found satisfactory.

Date :

Signature :

Analab Representative	Checked by End User	Approved by Head of Department

2.2. Operational Checking & Verification

Ensure all the connection of glassware, electrode, and dispensing unit with the instrument. (Pl. refer instruction manual)

2.2.1. Power on Check

Requirement Condition & Observation	Remark	
	Pass	Fail
➢ Instrument will display Manufacturer's company name and then holds at Customer Name.		
➢ Stirrer status shows		

2.2.2. Electrode check

- Short both electrode input from back panel and observe audio signal after 20 Sec in Neutralization mode □**Yes** □ **No**
- Open both electrode input from back panel and observe reagent mL increasing. □**Yes** □ **No**

2.2.3. Dispenser Validation check

(Use fresh reagent to calibrate Dispenser)

Trial	Actual Volume in Measuring Cylinder (mL)	Volume on Instrument Display (mL)	Average Volume (mL)
1	10.000		
2	10.000		
3	10.000		

2.2.4. Dispense Flow Linearity Test

Trials	Actual Volume in Measuring Cylinder (mL)	Volume on Instrument Display (mL)	Tolerance (ml)
1	2.00		1.95 – 2.05
2	5.00		4.95 - 5.05
3	10.00		9.95 -10.05

Date :

Signature :

Analab Representative	Checked by End User	Approved by Head of Department

2.3. Summary of Operational Checking & Verification

SR NO.	DESCRIPTION	RESULT	
		Yes	No
1	Power ON Check		
2	Electrode Input Check		
3	Dispensing validation check		
4	Dispense flow linearity check		

All system components and functions are operating satisfactorily.

The Operational check and the verification were carried out in accordance with the operating Manual and found satisfactory.

Date :

Signature :

Analab Representative	Checked by End User	Approved by Head of Department

Installation & Operational Qualification

The Installation Qualification (IQ) & Operational Qualification (OQ) final reports for the Microcontroller based Karl Fischer Titrator has been reviewed and was found acceptable for use.

INSTRUMENT : Micro controller based Karl Fischer Titrator
MODEL : µAquaCal10+
SR.NO. :
FINAL REPORT :
REVIEWED AND
ACCEPTED BY :
(Name & Designation)
DATE :

Customer	
Department	
Head of Dept.	
End User	
Date	
Signature	
Seal of the Company	

Analab Representative	
Date	
Signature	

Date :

Signature :

Analab Representative | Checked by End User | Approved by Head of Department

Performance Qualification

Microcontroller Based
Karl Fischer Titrator
Model: µAquaCal$_{10+}$

PERFORMANCE QUALIFICATION DOCUMENT

Protocol Number	**:**	ASIPL/PQ/
Company Name	**:**	
Location	**:**	
Instrument	**:**	Microcontroller based Karl Fischer Titrator
Model Number	**:**	µAquaCal$_{10+}$
Instrument Serial Number	**:**	
Protocol Prepared By	**:**	Analab Scientific Inst. Pvt. Ltd. Vadodara

Date :

Signature :

Name :

Analab Representative	Checked by End User	Approved by Head of Department

PERFORMANCE QUALIFICATION

Performance Qualification is the process of demonstrating that an instrument consistently performs according to a specification appropriate for its use.

Note: PQ should always be performed under conditions that are like routine sample analysis.

1. Aim

To ensure that the Karl Fischer Titrator is properly calibrated with traceable instruments/System & with injection of fix water quantity. The test will check and confirm that the correct moisture values of different solutions are achieved.

2. Scope

Performance Qualification must be performed in the case of new installation of an instrument OR change in the location and / or as per FDA requirements.

3. Responsibility

The responsibility for the Performance Qualification lies with the system user.

4. Preparation for the Test

4.1. Apparatus Required:

A. Karl Fischer Reagent - 250 ml
B. Dried Methanol bottle (KF Grade)-250ml
C. Distilled Water - 50 ml
D. Silicon Grease - 50 gm.
E. Dropper Bottle OR 10 µL syringe for calibration
F. Dry Di sodium Titrate (DST) - 250 Gms.
G. Analytical Balance (with 0.1 mg accuracy)
H. Tissue Paper Roll
I. Butter paper for solid samples
J. Wash Bottle – 1 No.
K. Calculator

Date :

Signature :

Name :

Analab Representative	Checked by End User	Approved by Head of Department

Note :

1. Ensure that the electrode is connected to the instrument.
2. Dry Reaction Vessel properly and fix all joints with silicon grease.

5. Standardization of Instruments Using Water :

5.1. Fill reagent bottle fully 250 mL and add KF grade Methanol in Reaction vessel up to tip of Electrodes.

5.2. Start stirrer by pressing stirrer on / off key.

5.3. Press Mode key and select Neutralization mode to neutralize methanol.

5.4. Repeat above step 5.3 two to three times to confirm neutralization of methanol

5.5. Once Neutralization over, Enter in Calibration mode and select calibration type "Calibration with water by volume

5.6. Add 10 µl water with syringe, after adding water, press Enter key. KF reagent starts entering in vessel and ml display will start increasing.

5.7. Once End point achieved, buzzer starts.

5.8. Note down ml reading of KF dispensed.

5.9. Repeat above steps i.e. 5.5 to 5.7 three to five times. Take average of display reading.

5.10. Calculate "WATER EQUVIVALANT FACTOR or KF strength with given formula.

Result: (Water Equivalent Factor)

Water Equivalent factor = mg. of water added (10 µltr.= 10 mg of Water)

Burette Reading in ml.

Injected volume with Syringe	ml Reading on Display	Water Equivalent Factor
	Average Factor →	

Date :

Signature :

Name :

Analab Representative	Checked by End User	Approved by Head of Department

6. Determination of Moisture in unknown Sample:

Di sodium Tartarated is used as an unknown sample as it has fixed percentage of water content (15.67% ± 5% or as per mention by manufacturer). It does not decompose and is not hygroscopic.

6.1. Weigh 0.150 Gram (fine powdered) DST by accurate balance and insert in vessel.

6.2. Dilute it properly in methanol with stirring for approx. 1 to 2 minute

6.3. Select analysis with Solid sample by weight from "Utility01>Analysis Parameter>sample type.

6.4. Press start key, reagent will start adding in vessel.

6.5. After getting END point, buzzer starts, and display will show ml. reading of KF reagent use for getting neutralize the sample. Note down ml reading of reagent consume.

6.6. Feed Gross weight and tare weight when instrument ask, instrument will calculate % and PPM of moisture of sample.

6.7. Repeat above for two to three times to confirm.

Result: (Using 150 mg. Di-Sodium Tartrate)

$$\% \text{ Moisture in Sample} = \frac{\text{Reagent consumed in ml} \times \text{AV. Factor}}{\text{Wt. of Sample in gram} \times 1000} \times 100$$

No of Times Sample added	ml. Reading on Display	% Moisture found in Sample
1		
2		
3		
	Average Result…… →	

NOTE: Expected result is 15.66% +/- 5.0 %

Date :

Signature :

Name :

Analab Representative	Checked by End User	Approved by Head of Department

7. Record of Chemical used:

K/F Reagent	
Manufacturer	
Batch No / Quality	
Date of Opening	
Di sodium Tartarated	
Manufacturer	
Batch No / Quality	
Date of Opening	

7.1. List of Attachment (if any) :

Date :

Signature :

Name :

Analab Representative	Checked by End User	Approved by Head of Department

PERFORMANCE QUALIFICATION

The instrument has been validated using traceable glassware and Chemicals. The test protocols have been filled in correctly and test results are documented. The system can be released for appropriate use.

INSTRUMENT NAME : Microcontroller Based Karl Fischer Titrator

MODEL : µAquaCal$_{10+}$

SR.NO. :

FINAL REPORT REVIEWED AND ACCEPTED BY (Name & Designation) :

DATE :

Customer	
Department	
Head of Dept.	
End User	
Date	
Signature	
Seal of the Company	

Analab Representative	
Date	
Signature	

Date :

Signature :

Name :

Analab Representative	Checked by End User	Approved by Head of Department