Karl Fisher Moisture Titrator



Operation Manual

Please read this manual thoroughly in advance for the best performance of the equipment.

Ver.05 A/N 69-001-0601-48

Table of Contents

Page

1. Introduction	1
1-1. Foreword	1
1-2. About the manual	2
1-3 Assafety symbols	3
1-4. Supplied parts	
2. Principle of measurement	7
3. Parts name and function	
4. Before power is turned on	
4-1. Line voltage and power fuse	
4-2. Power cable	
5. Preparation before measurement	14
5-1. Titration cell setup	14
5-2. Initial screen display after turning on power	
5-3. Display of main screen	
5-4. Prohibited key operations	
6. Basic operating method	
6-1. Filling reagent and draining	
6-2. Pre-titration	
7. Measuring operations	
7-1. General description	
7-2. Titration parameter	
7-3. Calculation parameters	
7-4. Parameters for Report	
7-5. Parameters for Sample	
8. Example of actual operation	
8-1. Status diagram of titration	
8-2. Titration	
8-3. Titration with Balance	
9. Setup	
9-1. General	
9-2. Interface (Setup 0)	
9-2-1. RS-232C	
9-2-2. Balance	
9-2-3. Printer	
9-3. Date & Time (Setup 1)	
9-4. User Name (Setup 2)	
9-5. Serial No. (Setup 3)	
9-6. Cell Type (Setup 4)	
10. Function	
10-1. General	

10-2. For Reagent life control and Alarm setting (Function 0)	
10-3. Recalculation (Function 1)	
10-4. Factor value setup (Function 2)	
10-5. Initialize to default (Function 3)	
11. Initializing parameters and default values	40
12. Printer	42
12-1. Printers connected to MKC-501	
12-2. Connection to Printer	
13. How to confirm product version number	
14. Maintenance	44
14-1. Daily checkup	44
14-1-1. Karl Fischer grease	44
14-1-2. Replacement of septum	45
14-1-3. Changing the desiccant	45
14-1-4. Replacement of Karl Fischer reagent	46
14-2. Other Maintenance	
14-2-1. Storage of the instrument	
14-2-2. Cleaning the electrode	
14-2-3. Cleaning the titration cell	
14-2-4. Cleaning the inner burette and how to dry it	
14-2-5. Cleaning the drain tube	51
14-2-6. Replacement of power fuse	
14-2-7. Replacing the clock battery	
15. Karl Fischer reagent	
16. Troubleshooting	
16-1. Error messages and remedies	
16-2. Troubles in power source	54
16-3. Stirrer does not work properly	55
16-4. Drift level is too high	
16-5. It runs into over-titration	
16-6. Poor repeatability or no EP found	
16-7. Glass contact area jammed	
17. Display message and glossary	60
18. Others	
18-1. Part list	
18-2. Option	65
19. System chart	68
20. Pin assignment of I/O interface	69
21. Specification	
22. Warranty and After-Sale Service	71

1. Introduction

1-1. Foreword

The MKC-501 you have purchased is Karl Fischer Coulometric Moisture Titrator, by which you can measure micro amount of water content which exists in liquid or in solid sample material. The measurement is easy to perform, fast in operation with its results of high precision and accuracy.

[Feature of MKC-501]

Easy to operate Very simple operation just pressing [start] key.

<u>Displays measurement results with high reproducibility</u> Guarantees below 0.3% Refractive Standard Deviation (RSD) at measurement of 1mg H₂O water-methanol.

<u>Variety of standard interface</u> Interface for Computer, Balance and Printer is standard

In order to gain the maximum performance and satisfactory results by the instrument, it is recommended to peruse this manual, and keep it near your system so that you are in quick access to the necessary information you are looking for while at work.

Note:

If you should find some specification in this manual which may differ from the system you have purchased, please always refer to the specification that accompanies your instrument when first delivered.

1-2. About the manual

This manual must be always in ready access from your instrument in order to not only assist you operate the whole process of titration but also to secure safety of yourself and others during the measurement and data processing.

You will find from place to place the following symbol marks for safety:

1. Where danger exists of physical injury or even possible death

<u>Warning!</u> This warning means danger of physical injury or severe damage leading to possible death.

2. Where danger exists of property damage



3. Where precaution must be noted for best performance of the instrument

Note:

If this note is ignored, you may not obtain best performance of the instrument or even warranty may not be covered.

- * It is prohibited to copy a part of or all of this manual without Manufacturer's consent.
- * If you should find any questions or need more information other than in this manual, contact your local dealer or sales representative.
- * Manufacturer will not be liable for any loss or damage due to use of measurement results by the instrument.

1-3. ASafety symbols

Always observe these signs and instructions.

You must observe cautionary messages and warnings in order to protect yourself as well as prevent others from physical injury or property damages.





Ground the green wire of adapter if power tap is 2-pin outlet.

3-pin plug has earth line to ground by itself when plugged in.

Danger of electric shock if not grounded to earth.

\land WARNING!

Use the same type and rating of fuse. Be sure to plug out power cord before replace the fuse.





Danger of fire if a wrong fuse is loaded.

WARNING!

Do not use volatile chemical or work in flammable gas.



Danger of explosion inside the instrument.

\Lambda WARNING!

Wear safety glasses, gloves or protective mask if necessary, and well ventilate the room.



Danger of injury on your skin or in the eyes by splashing chemical. Also your windpipe may get hurt if toxic gas is breathed in.

Unplug the power cord when the unit can be troubled or exposed to a lightning.



Failure to observe this caution may result in a damage to the instrument.

Do not operate in a way other than specified in the manual.



Danger of fire, electric shock or damage to the instrument.

Do not open housing case or overhaul the unit for repair except by an authorized service person.



Danger of fire, shock or malfunctioning of the unit.

About place for installation

Avoid the use of this instrument under the environment described below. (Failure can lead to the degradation of performance and reliability of the system.)

Operation of devices with strong electric motors using common power source Near strong magnetic/electric field Use of power source with too variable load Location of strong vibration Exposure to direct sunlight Location with large temperature difference Exposure to corrosive gas Exposure to extreme heat (Operation temperature: 5 to 35 C (41 to 95 F)) Exposure to high humidity (Over 85%RH)

About power source

Power for this instrument is $AC100/120/220/230/240V \pm 10\%$ and 50/60Hz. Supply power direct from the outlet, and do not share power from a tap. Do not put any obstacle around power outlet just case of need for plugging out power cord to avoid the possible danger of the whole system in trouble.

About place for storage

Store in a desiccant container the disassembled titration cell as they are after cleansed and dried, if it is not going to be operated for a long period of time. It is recommended to pack the main unit in the carton box in which the instrument was first delivered.

Avoid the places for storage under inadequate ambient conditions such as extremely high/low temperature, high humidity or heavily dusty atmosphere.

About use

Karl Fischer reagents are toxic chemicals. Therefore, please handle in a well ventilated room and be aware of its danger.

When a reagent etc. is spilt to Main unit or the connectors, there is a possibility of malfunction.

Other caution

Do not use such a solvent as alcohol, acetone, thinner or the like for cleaning this instrument. Doing so may adversely affect the instrument, e.g. deformation, discoloration or cracks. When cleaning this instrument, wipe it with a soft cloth or tissue paper, after applying detergent diluted with water to the soft cloth or tissue paper and adequately wringing out excess water in order not to allow water drops to fall.

Environmental condition

This instrument is designed for the indoor use under the environmental conditions specified in the Section 1.4 of CE marking (LVD, 73/23/EEC, EN61010-1) and the use of the Category II of Overvoltage and the Pollution Level 2.

1-4. Supplied parts

Check the supplied parts with the standard parts list in below. If you should find any parts missing or broken, contact your local dealer or sales representative.

		М	KC-501-D	MKC-501-N
(1)	Main unit 1	MKC-501	1 unit	1 unit
(2)	Power cord with earth (AC 100/110/120V area)	98-320-3198	1 pc	1 pc
	(AC 220/230/240V area) 9	98-320-3461		
	(for UK)	98-320-4199		
	(for China)	64-000-180-048		
(3)	Adapter for power connector (AC 100/110V area) 9	98-320-3199	1 pc	1 pc
(4)	Earth wire (AC 100/110V area)	98-433-3331	1 pc	1 pc
(5)	Titration cell (transparency)	20-040-4100-48	1 pc	1 pc
(6)	Stirrer rotor (35mm) ((98-500-3362)	1 pc	1 pc
(7)	Inner burette (2-component burette)	98-433-0006	1 pc	-
	(1-component burette)	98-103-0002	-	1 pc
(8)	Twin platinum electrode / KF 9	98-103-M713	1 pc	1 pc
(9)	Desiccant tube A	98-433-3116	1 pc	1 pc
(10)	Port plug (19/25)	98-550-0073	1 pc	1 pc
(11)	Syringe inlet (with septum)	98-433-3407	1 pc	1 pc
(12)	KF grease (5g)	98-433-3138	1 pc	1 pc
(13)	Septum (10pcs/set)	98-523-3161S	1 pc	1 pc
(14)	Washing bottle	98-500-3134	1 pc	1 pc*
(15)	Funnel	98-500-3159	1 pc	1 pc
(16)	Anode adjuster	98-075-3411	1 pc	1 pc*
(17)	Operation manual (CD-ROM)	12-02906	1 pc	1 pc

Note:

Parts with parenthesized part code have unique order units or packing forms different from others. Therefore, please refer to the section "18. Part list" when ordering these parts.

The accessories marked with "*" are not used for this instrument. These accessories will be needed when the titration cell unit with a diaphragm is purchased separately.

2. Principle of measurement

In the Karl Fisher moisture content measurement, water reacts with iodine and sulfur dioxide in the presence of base and alcohol.

 $H_2O + I_2 + SO_2 + CH_3OH + 3RN \rightarrow [RNH]SO_4CH_3 + 2[RNH]I....(1)$

In the volumetric titration, iodine is added as a titrant. In the coulometric technique, iodine is electrolytically generated in the anolyte, which contains iodide.

 $2I \rightarrow I_2 + 2e^{-}$ (2)

As long as water is present in the titration cell the generated iodine reacts according to (1).

As soon as all the water reacts, excess of iodine appears in the anolyte. This iodine is detected by the platinum electrode and the iodine production is stopped. According to Faraday's law, the quantity of iodine produced is proportional to the current generated. In equation (1), I_2 and H_2O react with each other in proportion 1:1.

Therefore a mole of water (18g) is equivalent to 2×96500 coulombs, or 10.72 coulombs/ 1 mg H₂O. The total amount of moisture can thus be determined by measuring the total consumption of electricity.



3. Parts name and function







0g	Key to select numerals.
$\left(\begin{array}{c} \leftarrow \end{array} \right) \left[\begin{array}{c} \rightarrow \end{array} \right]$	Key to move the cursor.
۲	Key to confirm the key entry.
Esc.	Key to return the parameters on display to the previous screen.
Bs.	Key to reverse a character or number by one space.
Cir.	Key to clear the key entry that has been made.

4. Before power is turned on

4-1. Line voltage and power fuse

Before power is turned on, make sure the voltage selector and line voltage of power source. If the voltage selector shows a different voltage, correct it by the following steps:

- 1) Open the cover above power plug-in receptacle by a flat head screwdriver.
- 2) Pull out the selector wheel and put it back to the box so that the selected voltage can be seen when the cover is closed.
- Make sure the loaded fuse conforms to the below rating chart: For replacing the fuse, see 14-2-5. Replacement of Power Fuse.

AC	Line voltage	Fuse	Hz
100 V	90 ~ 121 V	T3.15 A/250V	50/60
120 V	103 ~ 132 V	T3.15 A/250V	50/60
220 V	198 ~ 242 V	T1.6A/250V	50/60
230 V	207 ~ 253 V	T1.6A/250V	50/60
240 V	216 ~ 264 V	T1.6A/250V	50/60

4) Close the cover and confirm correct voltage can be seen on voltage selector.



Warning!

For continued protection against risk of fire, replace only with same type and rating of fuse.

4-2. Power cable

1) Make sure the power switch is in Off position.



2) Plug in the supplied power cable on the back of unit.



3) Connect the power cable to the power outlet.



< 3-pin plug > The 3 pins plug has an earth terminal and grounds to the earth by itself.



< 2-pin plug > Attach an adapter for power connector to the plug and ground the green wire to the earth terminal.

Warning!

The earth wire must be grounded. If not, there exists a danger of electric shock.

5. Preparation before measurement

5-1. Titration cell setup

1) Put a stirrer rotor into the cell, and install the inner burette, the electrode, desiccant tube A, the port plug and syringe inlet.



Note:

Make sure to apply KF grease around glass sliding area.

Warning!

When handling the inner burette, do not hold the housing (black resin area) and sliding area of desiccant tube in order to avoid breakage.



2) Place the titration cell onto the titration holder, and plug in the cable from the inner burette and the electrode. Tighten the plug screws firmly.



5-2. Initial screen display after turning on power

C. Moisture Titr.	When MKC-501 is switched on, the message on the left will be displayed for about two seconds.
	_
Pre - Titr	In about two seconds, the display will turn to the screen on the left and begin to blink. Blinking status continues till pre-titration starts.

Note:

When another pre-titration becomes necessary for some reasons, the above "Pre-Titr" message will appear again even after a pre-titration has been completed.

5-3. Display of main screen

$03 \rightarrow \text{Stable}$	Completi
	following

Completion of pre-titration after turning on power will lead to the following main screen display.

Note:

"Ready" or "Stable" indication will be variably displayed depending on the cell's inside conditions.

5-4. Prohibited key operations

The following key operations cannot be accepted while carrying out pre-titration.

Pressing the keys to allow for changing parameter settings such as [Titration], [Result], [Function] or [Setup] key or the [Print] key will result in making a warning sound.

Only [Reset], [Sample], [-/Disp.] or [Stirrer] key will work during pre-titration or measurement—other keys will not work singly.

6. Basic operating method

6-1. Filling reagent and draining

< Catholyte >

Inject 5mL catholyte into the inner burette using a syringe. (The lower line outside the cell shows approx.5mL.)

To drain out the liquid, use the supplied washing bottle for draining.



< To fill the reagent >

< Drain out >

Note:

When one component cell is used for inner burette, catholyte is not needed.

< Anolyte >

Fill the titration cell with 100mL reagent for two component cell, and fill the titration cell with 150mL reagent for one component cell. (The lower line outside the titration cell indicates approx.100mL line for anolyte, and the middle point between upper and lower line marked outside the titration cell indicates approx.150mL line for anolyte.)

To drain out the liquid, use the supplied washing bottle for draining.



< To fill the reagent >

< Drain out >

Note:

When reagents for Ketones are used, residue of alcohols in the titration cell may lead to the failure of successful measurements. In this event, rinse the inside of the titration cell and replace the Anolyte.

6-2. Pre-titration

When the Karl Fischer reagents are stored under inadequate conditions, the higher level of the residual water in them will give an adverse effect on water determination. Therefore, it is quite important to remove the residual water in the titrant as much as possible.

Pre - Titr	After the power is turned on, the initial screen will appear, followed by displaying the blink message on the left of the screen.
↓	_
.00 * Pre - Titr	Pressing [Pre-Titr] key will turn the screen display to the left one and start pre-titration. And when a pre-titration is completed, the
	titrator will be in the stand-by mode.
↓	
$.03 \Rightarrow \text{Stable}$	After the power is turned on and a pre-titration is completed, the following main screen will be displayed.

7. Measuring operations

7-1. General description

In ordinary measurements on MKC-501, difficult key operations are not required. Routine measurement by MKC-501 is simply started by [Start] key, and can be aborted by [Reset] key.

Display messages and key entry are related like this:

Parameters of message ending with " \uparrow " is selected on display by [-/Disp.] key, and confirmed by [\downarrow] key. For parameters ending with "?", use numeric key and confirm by [\downarrow] key.

The contents of each parameter are shown below:

[Titration]

Para	ameter	Range	Remarks
t (stir) [s]	?	0 – 999 s	
t (wait) [s]	?	0 – 999 s	
t (max) [s]	?	0 – 9999 s	
Drift Stop :	rel ↑	rel ↑ / abs ↑ / off ↑	
rel [µg/s]	?	0.00 - 9.99	This will be displayed when "rel" is
			selected in "Drift stop" setting.
abs [µg/s]	?	0.00 – 9.99	This will be displayed when "abs" is
			selected in "Drift stop" setting.

[Result] – Calculation ↑

Parameter	Range	Remarks
Unit : μg ↑	$_{ m \mu g}$ \uparrow / mg \uparrow / ppm \uparrow / % \uparrow	These parameters will be displayed
Weight : var ↑	var ↑ / fix ↑	when "Calculation" is selected.
Drift comp : manu ↑	manu \uparrow / auto \uparrow / off \uparrow	
Drift?	0.0000 – 999.9	This will be displayed when "manu" is
Blank?	0.00 - 999.9	selected in "Drift comp" setting.

Short ↑	GLP ↑	
\downarrow	\downarrow	The parameters on the left will be
Date & time	Date & time	printed according to the format selected.
Sample No.	Serial No.	
Wt1	Sample No.	
Wt2	Lot No.	
Net	Wt1	
Result	Wt2	
	Net	
	Factor	
	Result	
	Titr. Time	
	Drift	
	Blank	
	Name	

$[{\sf Result}] - {\sf Report} \uparrow$

[Sample]

Parameter	Range	Remarks
Sample No. ? XX–XX	00–00 - 99–99	
Lot ?	10	
Wt1 ?	0.000000 - 999.9999	
Wt2 ?	0.000000 - 999.9999	

7-2. Titration parameter

Here you can set the control parameters for the titration. Press [Titration] key.

Entry Key	Display	7	Description
	t(stir) [s]?	0	Set 'Waiting time' before starting titration
[L_]	t(wait) [s]?	15	Set the time during which titration is not stopped. Titration won't stop unless this time period elapses. (Minimum 15 s)
[+-]	t(max) [s]?	0	Set the maximum titration time. Titration will stop even while carrying out titration when this time period elapses. (When '0' is set, there is no time limit for titration)
[+]			there is no time mint for titration.)
	Drift stop : off \uparrow		Titration will not end by drift level; it lasts till t(max).
[– /Disp.]			
	Drift stop : rel ↑		Titration will end when the drift level becomes lower than either 'Input value + the reading at start – Drift comp is set to off \uparrow ' or 'Input value + set value – Drift comp is set to manu \uparrow '.
[– /Disp.]			
	Drift stop : abs	\uparrow	Titration will end when the drift value
			becomes lower than the input value.

Note:

Do not select Drift Stop:off and t(max)=0 at the same time. If selected, the alarm will beep when titration starts.

7-3. Calculation parameters

The four calculation parameters are part of the result parameters you can select in a method. Press [Result] key.

Entry Key	Display	Description		
	Calculation ↑			
[-]				
	Unit∶g ↑	Set the calculation result unit		
[– /Disp.]	:			
	:			
	Unit∶g ↑			
[-]				
	Weight : fix \uparrow	Select fixed or variable		

Entry Key	Display	Description
[– /Disp.]		
	Weight : var ↑	Select when sample weight is input for each
[↓]		measurement.
	Drift comp : off \uparrow	No compensation for drift value is made.
[– /Disp.]		
	Drift comp : auto ↑	Auto compensation for drift value is made. The
:		object of this compensation is the drift level
		when [Start] key is first pressed.
[– /Disp.]	:	
	Drift comp : manu ↑	Manual compensation for drift value is made.
[↓]		
	Drift? 0.0	Drift is entered here [g/s]
[₊]		
	Blank? 0	Blank is entered here [g]
[₊]		

Formula No.	Description	Formula	
1	Moisture calculation	$F \times (Moisture) (g)$	
	Result in g or mg.		
2	Concentration calculation for weighed		
	liquid or solid samples.	$F \times \frac{(Moisture)}{W(1-W/2)}$ (ppm)	
	Result in ppm or %(w/w)	wti – wt2	

* "Moisture" in the equation is identical to "Data – Drift x t – Blank".

Moisture	:	Net water amount (g)
		Water obtained by subtracting "Drift value x titrating time and Blank value" from total water
		titrated (electrolyzed).
F	:	Factor value that is coefficient related to the results. (see 10-4)
Data	:	Result of the titration (g)
Drift	:	Drift Level (g/s)
		When Drift comp:auto is selected, this value represents the drift at time [Start] key is pressed.
		When Drift comp:manu is selected, this is manually input value.
t	:	Measurement time (s)
		When Drift stop:off \uparrow is selected,t=(t(stir)+t(wait)+t(max)) (s)
		When Drift stop:rel \uparrow /abs \uparrow is selected, t= <t(stir)+t(wait)+t(max) (s)<="" td=""></t(stir)+t(wait)+t(max)>
Blank	:	The value for subtraction of result (g)
Wt1	:	Weight of syringe with sample in it
Wt2	:	Weight of empty syringe
		Therefore, the sample weight = $Wt1-Wt2$

Note:

Even when ppm or % is selected for calculation results, if Wt1-Wt2=0, the display will show the results in mg and printout in g and mg.

7-4. Parameters for Report

The report parameters allow you to define the format of your report for a particular method. Press [Result] key.

Entry Key	Display	Description
	Calculation ↑	
[– /Disp.]		
	Report ↑	Set the report parameters
[↓]		
	Short ↑	With Short format, Date & Time, Sample
[– /Disp.]		No., Wt1, Wt2, Net and Result are
		automatically printed out.
	GLP ↑	With GLP format, Date & Time, Serial No.,
		Sample No., Wt1, Wt2, Net, Factor, Result,
		Titration time, Drift, Blank and Name are
		automatically printed out.
[-]		

		Г			
Below shows comparison of print format between Short form and GLP:					
<short form<="" td=""><td>></td><td></td></short>	>				
Date	:	date and time of measurement			
Sample No.	:	sample number			
Wt1	:	weight of syringe and sample weight			
Wt2	:	weight of syringe			

weight of sample

amount of water (concentration or weight)

* * * R e s u l t * * *

Date	04/12/09 11:01
Sample No.	00-34
Wt1	42.5384g
Wt2	42.0213g
Net	0.5171g
Result	1734.8 g
	3354.9ppm

<glp form=""></glp>	>	
Date	:	date and time of measurement
Serial No.	:	production number of the instrument
Sample No.	:	sample number
Lot No.	:	lot number
Wt1	:	weight of syringe and sample
Wt2	:	weight of syringe
Net	:	sample weight
Factor	:	factor value
Result	:	amount of water(concentration or weight)
Titr. Time	:	measurement time
Drift	:	drift value
Blank	:	blank value
Name	:	name of operator

* * * R e s u l t * * *					
Date 04	/12/09 10:57				
Serial No.	NCAxxxx				
Sample No.	00-33				
Lot No.	A960123				
Wt1	42.5384 g				
Wt2	42.0213 g				
Net	0.5171g				
Factor	1.00				
Result	1728.7 g				
	3343.0 ppm				
Titr.Time	00:01:48				
Drift	0.04 g/s				
Blank	0 g				
Name	KEMTARO				

Net

Result

:

:

7-5. Parameters for Sample

Desired parameters on the sample can be selected including sample number, lot number and sample size for concentration calculation.

Press [Sample] key.

Entry Key		Displa	a y	Description
	Sample	No. ?	00 – 00	Enter sample number by numeric key.
[0], [1]				
	Sample	No. ?	01 – 00	Enter sample number of high order.
[+-]	Comple	No. 0	01 00	
[0] [1]	Sample	NO. ?	01 – 00	
[0], [1]	Sample	No. ?	01 – 01	Enter sample number of low order.
[-]		-		1
	Lot	?		Enter lot number by [-/Disp.], [•] and
$[3], [\rightarrow], [0], [\rightarrow], [5], [\rightarrow],$				numeric keys.(up to ten characters)
[•], [•], [→], [5], [→], [2]				
r 11				
[+-]	Lot	?	305-52	
		-		
	Wt1	?	5.0000	
[4], [•], [1]				
[2], [3] , [5]	\\/+1	2	4 1005	Enter weight(g) of (guringe genule)
[.]]	VVLI	ſ	4.1235	Enter weight(g) of (syringe+sample).
r-1	Wt2	?	0.0000	Enter weight(g) of syringe after sample is
[4], [•], [0]				injected.
[1], [7] , [3]				
	Wt2	?	4.0173	
[4]				

Sample parameter can be input during titration.

If Weight of calculation parameter is var \uparrow and sample size is not entered, the display message will ask Operator for Wt1 and Wt2 after titration is finished.

Sample number of low order will be added up after each measurement.

Note:

If Wt1-Wt2 is set to zero(Wt1-Wt2=0) and ppm or % is selected for calculation results, it will be shown in mg and printed in g or mg.

8. Example of actual operation

8-1. Status diagram of titration



8-2. Titration

Entry Key	Display			Description
	.01	\Rightarrow	Ready /Stable	This shows a stand-by state waiting for the
[Start]				start of titration.
	.01	\Rightarrow	Samp.In.	Inject a sample into the titration cell here,
				followed by pressing [Start] key.
			\$	
	.01	\Rightarrow	Start	Then electrolysis begins and titration is
[Start]				carried out.
	43	*	45.8 g	Also, both detected moisture amount and
				titrated water amount are displayed on the
				LCD.
	.03		50.3 g	When titration is completed, the buzzer
				sounds to notify the end of titration, followed
				by displaying moisture amount or
				concentration.
[]				
	.02	\Rightarrow I	Ready/Stable	Return to the stand-by state waiting for the
				start of titration.

Ordinary measurement can be made in the stand-by state for titration following the pre-titration described in the section 6-4.

Note:

The MKC-501 will shift to Reset condition after titration is finished, however, it is recommended to press [,] key after measurement results are recorded so that the condition of the measuring cell can be checked to see if it is in Ready or Stable.

Reset condition can be reached by other key than [\downarrow], and other display screen can be reached by pressing such keys as Parameter Setup or key for separate display message screen.

Note:

If Drift Stop:off \uparrow is selected and [Reset] key is pressed during titration, the measurement results until [Reset] key is pressed will be printed out.

8-3. Titration with Balance

With the MKC-501 you can enter the sample weight manually using the keypad or automatically when a balance is connected. You can activate the weight transfer by pressing the [-/Disp.] key Connect MKC-501 and Balance.

Input sample number and lot number by Sample key, and leave it in waiting condition for Wt1. Measure the weight of syringe filled with sample for measurement. Place the syringe on Balance and wait until it becomes stable. Then, press [-/Disp.] key. The Wt1 will be read by MKC-501.

Press Start key and inject the sample, and then press Start to titrate. Place the syringe after the sample is injected. When the display message asks for Wt2 after titration is over, press [-/Disp.] key to read Wt2. Calculation results will be displayed at the end.

Entry Key	Disp	lay	Description
	.01⇒Ready/S	table	Waiting for titration start.
[Sample]			
	Sample No.?	00 - 00	Enter sample number.
[0], [1]			
	Sample No. ?	01 – 00	Enter sample number of high order.
[⊷]		04 00	
[0] [4]	Sample No. ?	01 – 00	
[0], [1]	Sample No. 2	01 01	Enter sample number of low order
]	Sample No. :	01 - 01	Ener sample number of low order.
	Lot ?		Enter lot number by [-/Disp.]. [•] or numeric
			keys.
[3], [→], [0], [→], [5], [→],			
[•], [•], [→], [5], [→], [2]			
	Lot ?	305-52	
[↓]			
	Wt1 ?	5.0000	
[– /Disp.]			
	VVt1 ?	4.3259	Read weight from Balance (syringe+sample)
[Start]			Start utration.
	01⊸Samn In		Now inject sample into the cell
	101>0amp. m. ↓		row, inject sumple into the cen.
	01⇒Start		
[Start]			

Entry Key	-	Display	Description
	33 * 1 ⁻	124.7 g	Electrolysis begins and titration starts, and the display will show the amount of water per unit time and then titrated amount of water.
	Wt2	? 0.0000	Beep sounds to tell the end of titration with message asking for Wt2.
[-/////	Wt2	? 4.0146	Weight of syringe has been read from Balance.
	.03	4176.2ppm	Measurement results are calculated and displayed.
[-]			
	.02 ⇒	Ready/Stable	Returns to Reset condition.

Note:

Even if the amount of Wt1-Wt2 becomes in minus, its absolute value will be read.

9. Setup

9-1. General

The MKC-501 has 5 functions for additional possibilities, such as Interface, data & time, Name, Serial No. Setup No.

0.Interface 1.Date & Time 2.2.Name 3.Serial No. 4.Cell Type

9-2. Interface (Setup 0)

The MKC-501 has three interface for the connection, RS-232C, Balance and Printer. With this function you can set the configuration of the interface.

9-2-1. RS-232C

MKC-501 can communicate with external device via RS-232C cable. Here you will set up necessary protocol to match digital configuration.

Press [Setup] key.

Entry Key	Display	Description
	Setup [0-4] ?	
[0]		
	Setup [0-4] ? 0 ‡	
	Interface	
[↓]		
	RS232C↑	Select the RS-232C.
[⊷]		
	Baud rate : 4800	Select the Baud rate.
[/Dien]		
[-/Disp.]	Baud rate [·] 9600↑	Choose from 300, 600, 1200, 2400, 4800 and
		9600 Baud.
[+]		
	Parity : even↑	Select the Parity. Choose from even, odd and
		none.
[– /Disp.]		
	Parity : odd ↑	
[+]		
	Data bits : 7	Select the Data bits. Choose from 7 and 8.

Entry Key	Display	Description
[– /Disp.]		
	Data bits : 8↑	
[₊]		
	Stop bits : 1↑	Select the Stop bits. Choose from 1 and 2
[– /Disp.]		
	Stop bits : 2↑	
[⊷]		
	Soft HS : off↑	Select the Soft Hand Shake. Choose from off
[– /Disp.]		and on.
	Soft HS : on↑	
[⊷]		
	Setup [0-4] ?	
[Esc.]		

<u>Note:</u>

For details of data communication via RS-232C, refer to MKC-501 Serial Communication Manual.

9-2-2. Balance

When an electronic balance is connected to MKC-501, sample weight from Balance can be read automatically to the MKC-501. Here is how to enter keys to match your Balance. Press [Setup] key.

Entry Key	Display	Description
	Setup [0-4] ?	
[0]		
	Setup [0-4] ? 0 ‡	
	Interface	
[⊣]		
	RS232C↑	Select the RS-232C.
[– /Disp.]		
	Balance↑	Select the Balance.
[⊣]		
	Bal : KEM ↑	Select the type of Balance.
	:	
[– /Disp.]	:	
:	Bal : A & D↑	Choose from KEM, Mettler, A & D,
[↓]		Shimadzu and Sartorius
	Setup [0-4] ?	
[Esc.]		

9-2-3. Printer

Optional printer can be connected as follows. Press [Setup] key.

Entry Key	Display	Description
	Setup [0-4] ?	
[0]		
	Setup [0-4] ? 0	
	↓ Interface	
[[]	Interface	
	RS232C1	Select the RS-232C.
[- /Disp.]		
	Balance↑	Select the Balance.
[– /Disp.]		
r. 11	Printer	Select the Printer.
[+]	Printer : IDP-↑	Select the type of printer.
[– /Disp.]		
	Printer : DP-↑	Choose from IDP-,DP-, and Other
[– /Disp.]		The parameters of IDP- and DP- are fixed.
r n	Printer : Other	
[+-]	Baud rate : 4800↑	Select the Baud rate
[– /Disp.]	:	
:	Baud rate : 9600↑	Choose from 300, 600, 1200, 2400, 4800 and
[⊷]		9600 Baud.
	Parity : even↑	Select the Parity. Choose from even, odd and
[_ /Disp]		none.
	Parity : odd↑	
[+]	,	
	Data bits : 7↑	Select the Data bits. Choose from 7 and 8
[– /Disp.]		
r n	Data bits : 8	
[+]	Stop bits : 1↑	Select the Stop bits Choose from 1 and 2
[– /Disp.]		
	Stop bits : 2↑	
[⊷]		
	Setup [0-4] ?	
[⊑SC.]		

9-3. Date & Time (Setup 1)

With this function you can set Date and Time.

Once date and time is input on MKC-501, the measurement date and time will be automatically printed out on Printer. The stored date and time will be kept in good time by clock function of MKC-501 unless backup battery runs down or the date and time is changed otherwise. Press [Setup] key.

Entry Key	Display	Description
	Setup [0-4] ?	
[1]		
	Setup [0-4] ? 1 ‡	
	Date & Time	
[]		
	YY MM DD? 950601	Enter year, month and day
[0], [4], [0], [1]		
[3], [0]		
	YY MM DD? 040130	
[⊷]		
	HH MM? 1000	Enter hour and minute
[1], [3], [2], [5]		
	HH MM? 1325	
[↓]		
	Setup [0-4] ?	
[Esc.]		

9-4. User Name (Setup 2)

With this function you can set User name. Press [Setup] key.

Entry Key	Display	Description
	Setup [0-4] ?	
[2]		
	Setup [0-4] ? 2	
	¢	
	Name	
[↓]		
	_	Enter User name
[– /Disp.]		
	A	Use the [-/Disp.], [•] and numeric keys.
$[\rightarrow]$		
	A_	
[– /Disp.]		
	A <u>A</u>	
[↓]		
	Setup [0-4] ?	
[Esc.]		

<u>Note:</u> Characters can be shifted automatically by keeping [-/Disp] and [•] keys pressed.
9-5. Serial No. (Setup 3)

With this function you can check the serial No. Press [Setup] key.

Entry Key	Display	Description
	Setup [0-4] ?	
[3]		
	Setup [0-4] ? 3	
	1	
	Serial No.	
[+]		
	NCXxxxx	The serial number is displayed
[⊷]		
	Setup [0-4] ?	
[Esc.]		

Individual serial number of the unit is input in plant before shipment.

Note:

Serial number can only be checked and confirmed but not for changing the number itself.

9-6. Cell Type (Setup 4)

Set the cell type for measurement (2-component or 1-component). Press [Setup] key.

Entry Key	Display	Description
	Setup [0-4] ?	
[4]		
	Setup [0-4] ? 4	Select "Setup 4".
	1	
	Cell Type	
[↓]		
	with Diaphragm	
[– /Disp.]		
	Diaphragmless	Select the cell type with $[\leftarrow] [\rightarrow]$ key.
[– /Disp.]		* with Diaphragm: 2-component cell (with
		diaphragm)
		Selected when two reagents, Anolyte and
		Catholyte, are used.
	with Diaphragm	* Diaphragmless: 1-component cell (without
[-]		diaphragm)
	Setup [0-4] ?	Selected when only Anolyte is used.
[Esc.]		

10. Function

10-1. General

The MKC-501 has 4 functions for additional possibilities, such as reagent capacity, recalculation, factor, memory clear.

Function No.

- 0. Reagent capacity
- 1. Recalculation
- 2. Factor
- 3. Memory clear

10-2. For Reagent life control and Alarm setting (Function 0)

The Karl Fischer reagent for coulometric titration has reagent capacity for measurement of water content. Generally, 1000mg H₂O for 100mL anolyte and 300mg H₂O for 5mL catholyte are the approximate limit of capacity for moisture measurement.(For details, see Instruction of the reagent you have purchased.) Exhausted overdue reagent will lead to longer titration time, too low in the results or too high in the drift.

The MKC-501 can show you update reagent capacity that has been calculated from coulomb used in electrolysis during moisture titration, and will let you know by alarm when the reagent has reached its preset limit of capacity.

Function 0 provides the following functions:

Displays reagent capacity both anolyte and catholyte. Clear incremented capacity to zero when reagents are replaced. Enter limit value for capacity. (Beep alarm can be switched by off/on.).

Press [Function] key.

Entry Key	Display	Description
	Function [0-3]?	
[0]	Function [0-3] ? 0	
[₊]		
	A. Capa. [mg]? xxxx	Accumulated electrolytic current (moisture-converted) of anolyte is displayed (mg H ₂ O)
[0]		Enter 0 on anolyte replacement to delete
	A. Capa. [mg]: 0	accumulated value (reagentcapacity)
[+]	C. Capa. [mg]? xxxx	Accumulated electrolytic current (moisture-converted) of catholyte is
		displayed (mg H ₂ O)
[0]	C. Capa [mg]? 0	Enter 0 on catholyte replacement to delete accumulated value (reagent capacity).
[-]	Alarm set : off↑	Select alarm/no-alarm status for reagent capacity.
[– /Disp.]		
[Enter]	Alarm set : on↑	
	A.Alarm [mg]? 1000	Enter alarm limit (mg H ₂ O) of anolyte
[5], [0], [0]	A.Alarm [mo]? 500	capacity.
[]		
[2] [0] [0]	C.Alarm [mg]? 300	Enter alarm limit (mg H ₂ O) of catholyte canacity
[-], [], []	C.Alarm [mg]? 200	-up unity.
[⊷] [Esc.]	Function [0-3] ?	

Note:

How to cancel alarm and display when Alarm set: on \uparrow is selected: when preset moisture-converted reagent life is reached, alarm by beep and blinking message will be activated. To cancel the alarm, press any key (eg; []] key, [Esc.] key, Numeric key, etc.) just like for ordinary operation after measurement is finished. It is recommended to replace the reagents immediately after the alarm has been turned off.

10-3. Recalculation (Function 1)

If expected measurement results have not been obtained due to wrong parameter settings or erroneously input sample weight, the measurement can be recalculated with correct parameters.

The recalculated results are printed out with (#) mark.

Press [Function] key.

Entry Key		Displ	ay	Description
	Funct	tion [0-3]	?	
[1]				
	Funct ‡	tion [0-3]	?1	Select Function 1 for recalculation
	Reca	lculation		
[-]				
	Unit :	g↑		Select result unit. (g, mg, ppm, %)
[– /Disp.]		:		
:	Unit :	ppm↑		
[-]				
	Wt1	?	5.0000	To correct sample weight, use [•] or numeric
[4], [•], [3], [7]	\\/+1	2	1 27	keys for Wt1, Wt2.
с II	ννιι	£	4.37	
[-]	Wt2	?	0.000	
[3], [•], [8], [6]		-		
	Wt2	?	3.86	
[⊣]				
	Drift	?	0.03	The first display is for drift value.
[↓]				
	Blank	(?	0	Then, enter Blank value.
[-]				
c 13	1325.	.4ppm		Recalculated results are displayed and printed.
[]	Funct	tion [0 2]	2	
[Esc.]	FUNC	uon [0-3]	ſ	

Note:

Even when ppm or % is selected, if Wt1-Wt2 is zero(Wt1-Wt2=0), the recalculated results will be shown in mg, and printed in g or mg.

Note:

For recalculation with new factor value, enter the corrected factor before recalculation.

Shown below is an example of recalculated results in ordinary print format.

Printed in short form			
[Recalculati	[Recalculation]		
Date 04/12/0	09 11:01		
Sample No.	00-34	(#)	
Wt1	42.5384	g	
Wt2	42.0213	g	
Net	0.5171	g	
Result 1734.8 g			
3354.9 ppm			

[Recalculation]		
Date 04/12/0	09 10:57	
Serial No.	NCA	xxxxx
Sample No.	00-33	(#)
Lot No.	A960123	
Wt1	42.5384	g
Wt2	42.0213	g
Net	0.5171	g
Factor	1.00	
Result	1728.7	g
3343.0ppm		
Titr. Time	00:01:48	
D	rift 0.04	g/s
Blank	0	g
Name K	EMTARO	

Printed in GLP form

10-4. Factor value setup (Function 2)

The MKC-501 is Karl Fischer Moisture Titrator and does not require Factor value unlike volumetric titration, however, the factor can be set up in order to adjust measurement according to the effect of sample or the syringe that is used for sampling.

This factor value will be added in the equation for calculation. Press [Function] key.

Entry Key	Display	Description	
101	Function [0-3] ?		
[2]			
	Function [0-3]?2	Select Function 2 for factor entry.	
	ţ		
	Factor		
[⊷]			
	Factor ? 1.00	Enter factor value by [•] and numeric keys.	
[0], [•], [9], [9], [8]			
	Factor ? 0.998	Confirm the factor value.	
[+]			
	Function [0-3]?		
[Esc.]			

10-5. Initialize to default (Function 3)

All the stored parameters can be cancelled and reversed to default values initially input in plant before shipment by Function 3. Press [Function] key.

Entry Key	Display	Description
[3]	Function [0-3] ?	
	Function [0-3] ? 3 ‡	Select Function 3 to clear saved data.
	Memory clear	
[↓]		
	Memory clr. : off↑	
[– /Disp.]		
	Memory clr. : on↑	Select to clear memory.
[]	Function [0-3]	When beep sounds, turn off the switch, and then, turn it on after five (5) seconds.
[_]]	SRAM Init.	Press any key (eg; [هـ] key). It beeps.
[⊷]	C. Moisture Titr.	This message turns on for two (2) seconds.
	Pre-Titr	All the data has been cleared, and it returns to initial display.

Note:

Setup function will not be affected by initializing the parameter.

11. Initializing parameters and default values

Para	meter	Default value
Titration	t(stir) [s] ?	Os
	t(wait) [s] ?	15s
	t(max) [s] ?	0s
	Drift Stop	rel 0.1 g/s
Result		Calculation↑
Calculation	Unit	g↑
	Weight	fix↑
	Drift comp	auto↑
	Drift comp : manu↑	
	Drift?	0.0 g/s
	Blank ?	0 g
Report		Short↑
Sample	Sample No. ?	00-00
	Lot ?	
	Wt1 ?	5.000g
	Wt2 ?	0.0000g
Set up		
Interface		RS232C ↑
RS232C	Baud rate	4800 ↑
	Parity	None 1
	Data bits	8 1
	Stop bits	1 1
	Soft HS	Off ↑
Balance		КЕМ↑
Printer		IDP- ↑
Other	Baud rate	4800 ↑
	Parity	None ↑
	Data bits	8 1
	Stop bits	1 ↑

The procedure of initialization of parameters to default is described in 10.5. All the data of parameters are reversed to default values, which are preset in plant before shipment.

Entry	Display	Description
Set up		
Date & Time	YY MM DD	950601
	HH MM	0000
Name		
Serial No.		NCXxxxxx
Function		
Reagent capacity	A.Capa. [mg] ?	Omg
	C.Capa. [mg] ?	0mg
	Alarm set	off ↑
	Alarm set : on↑	
	A.Alarm [mg] ?	1000 mg
	C.Alarm [mg] ?	300 mg
Recalculation	Unit	g↑
	Wt1 ?	5.0000 g
	Wt2 ?	0.0000 g
	Drift ?	0.0 g/s
	Blank?	0 g
Factor		1.00
Memory clr.		off ↑
-		
Stirrer	Speed [0-9]	4

Note: The Setup function is not affected by the initialization.

12. Printer

The MKC-501 when connected to optional Printer can print out measurement results, date of measurements, Operator's name, etc.

12-1. Printers connected to MKC-501

The built-in interface for printer installed in MKC-501 is for Kyoto Electronics IDP-100 Dot printer, and Mettler-Toledo GA-42 printer. Other printers can also be connected when digital configuration is set up to MKC-501. The recommended printer is IDP-100.



12-2. Connection to Printer

All you have to do is to connect the printer cable to the port for printer on rear panel of MKC-501.



13. How to confirm product version number

The product ver. number varies depending on what options and specifications you have selected for your MKC-501.

Follow the below procedure to check its number:

With power turned on, make sure it shows Initial message or Main display. Then, press [0], [0], [0] and press $[\downarrow]$ key. The product number will appear for two seconds. (5-2)

Ver X.XX

Note:

This procedure must be worked on Initial display or main display.

14. Maintenance

14-1. Daily checkup

14-1-1. Karl Fischer grease

Twin platinum electrode, inner burette, syringe inlet, desiccant tube, and port plug are removed from a titration cell. Apply KF grease around glass contact areas. Check those parts once a week to ensure they rotate smoothly. If not, apply thin coating of grease. Do not apply too much grease as it may penetrate the titration cell and increase the background owing to the water content of the grease.



<u>Caution!</u> Check the glass joints from time to time so that applied grease will not solidifies.

If grease on the glass contact areas becomes hard and the respective parts are difficult to separate, take the following steps;

- 1) Discharge anolyte and catholyte.
- 2) When using a glass port plug, warm it up with a hair dryer or something similar to soften KF grease before removing. When using a PTFE port plug, remove it after cooling the titration flask in a freezer for about five (5) minutes.

🗥 Warning!

Care should be taken not to get burned when heating the unit. Do not try and open solidified jointed parts by force. Glassware may break into piercing pieces for injury.

Do not warm up a PTFE port plug when removing it as doing so may inflate the material and the titration flask may be broken.

14-1-2. Replacement of septum

The syringe inlet is removed from a titration cell like the clause of an application of KF grease. Change the syringe inlet port septum occasionally. An old septum is easily broken and allows air into titration cell to increase the background.



14-1-3. Changing the desiccant

The desiccant tube is removed from a titration cell like the clause of an application of KF grease. Replace the desiccant with new one when its moisture absorption turns down to reddish color. Be sure to apply KF grease around sliding area between the titration cell and desiccant tube A.



< Desiccant tube A >

Note:

If the nozzle chip slips from the desiccant tube when replacing the desiccant, ensure that you return the nozzle chip in the tube with its trenches downward as shown above.

14-1-4. Replacement of Karl Fischer reagent

< Replacement of the anolyte (anode reagent) >

Change the anolyte when the accumulated moisture measurement (=reagent life) has reached 1000mgH₂O after the last replacement with new one, when the surface of anolyte exceeds the upper line on cell wall when sample liquid is discharged into the cell or when drift level goes up.

1) Suck the old anolyte out of the supplied washing bottle.



2) Fill the titration cell with new reagent. Fill the titration cell with 100mL reagent for two component cell, and fill the titration cell with 150mL reagent for one component cell. (The lower line outside the titration cell indicates approx.100mL line for anolyte, and the middle point between upper and lower line marked outside the titration cell indicates approx.150mL line for anolyte.)



3) After the anode is filled with new anolyte, press [Clear] button and set the Now life to zero "0".

< Replacement of the catholyte (cathode reagent) >

Change the catholyte when the accumulated moisture measurement (=reagent life) has reached 300mgH₂O after the last replacement with new one or when drift level goes up.

1) Suck the old catholyte out to the supplied washing bottle.



2) Fill the cathode with 5mL of new catholyte.



 After the cathode is filled with new catholyte, press [Clear] button and set the Now life indicator to zero "0".

Note:

Negligence of replacing cathode reagent will cause higher drift level, foreign objects generated around the diaphragm and may lead to measurement errors. The optional single component titration cell does not need the above process. The lower line on cell wall indicates approximately 5mL filling.

14-2. Other Maintenance

14-2-1. Storage of the instrument

Store the instrument, if it is not going to be operated for a long period of time in a place where there is no direct sunlight or under no vibration, and the place is dry, not humid. It is recommended to pack it in the carton box in which the instrument was first delivered.

Store in a desiccant container the disassembled titration cell, inner burette and electrode as they are after cleansed and dried.

14-2-2. Cleaning the electrode

If the electrode is heavily stained and the potential is unstable and measurement reading fluctuates, cleanse it with nitric acid, and after cleaning by methanol, wipe off with clean gauze.

14-2-3. Cleaning the titration cell

- 1) Remove Detection electrode, Inner burette and Sampling port stopper, and then drain out the reagent.
- 2) Wipe off the grease around the sliding area with methanol.
- 3) Rinse by neutral detergent under running water.
- 4) After drying the glassware in a heater, either cool them in desiccator or dry them.

Note:

Insufficiently dried glassware may cause higher drift level.

14-2-4. Cleaning the inner burette and how to dry it

Periodical cleaning of inner burette is recommended since if the inner burette is stained, the electrolysis reaction will not run smoothly, and may cause a longer time length in measuring process with measurement results higher than theoretical value.

< Cleaning with alcohol: general method >

- 1) Turn off all the powers.
- 2) Disconnect the electrodes from their ports.
- 3) Take out both anolyte and catholyte.
- 4) Wipe off grease around sliding area with methanol.
- 5) Rinse the inner burette with methanol, and fill it with approximately 10mL of methanol, and then, put it in a beaker. Fill the beaker with methanol up to the level of methanol inside the inner burette, and leave it for about 30 minutes.
- 6) After the above 5), dry the inner burette.

< Cleaning with chromic acid mixture >

If foreign objects are observed on diaphragm and platinum surface, use chromic acid mixture instead of methanol for cleaning.

Chromic acid mixture : 1.5g approx. potassium dichromate dissolved in 100mL of concentrated sulfuric acid

Caution!

Chromic acid mixture is a very strong oxidizing reagent. When handling this chemical, protect yourself with gloves and glasses. If it touches your skin, immediately rinse it with running water.

- 1) Follow the same steps as above for methanol.
- 2) Drain out the chromate inside the cell, and rinse it with pure water for 5 to 6 times until yellowish color disappears.
- 3) Clean the inner burette with methanol or with alcohol.
- 4) After cleaning, dry the inner burette.

A Caution!

Chrome is a heavy metal. Do not discard the used mixture or rinsing solvent as wastewater. First, dilute the collected chromic acid mixture down to 1% concentration, and then, reduce it. After confirming no Cr^{6+} is contained in it, adjust its pH to 7.5 ~ 8.5. Filter the liquid, and store the precipitation.

For more details, refer to the corresponding documents regarding how to dispose of heavy metals.

< How to dry the inner burette >

Dry it in a decompression dryer for more than 2 hours.

Below sketch shows an example of commercially sold drying under reduced pressure.





Diagram of Decompression dryer

Commercially available vacuum dryer

Caution!

Dry the inner burette itself only after removed from the titration cell in order to avoid possible breakage of inside ceramic diaphragm.

Note:

Use a hair dryer if a compression dryer is not available. With a hair dryer, dry the inner burette well enough as long as for more than 10 minutes, especially dry the diaphragm until it is really dried. Any residue of moisture will cause high drift level.

< Distance adjustment between anode electrode and diaphragm >

If the anode electrode in the inner burette and the diaphragm are too close together, electrolysis reaction will not run in normal condition. Use the supplied anode adjuster to adjust the distance in between.



14-2-5. Cleaning the drain tube

- 1) Remove the tube line, and clean it with ethanol.
- 2) Connect the tube, and inject solvent, and see if it is drained out smoothly.

Note:

KF reagent contains substantial amount of salt. If it is not used for an extended period of time, the salt may precipitate out of solution and clog the pipe. Rinse and clean all tube lines if not to be used for an extended period of time.

14-2-6. Replacement of power fuse

- 1) Turn off the power and plug out the cord.
- 2) Open the box cover above power inlet by a flat screwdriver.
- 3) Pull out the two-fuse holder using the screw driver.
- 4) Replace the broken fuse by inserting new fuse to the same arrow direction of cover and holder, and close the cover.



<u> Warning!</u>

Danger of electric shock. Turn off power and plug out the cord before work on fuse.

<u>Caution!</u> There are two fuses. It is recommended to change them all at a time.

14-2-7. Replacing the clock battery

If the clock does not function correctly, the inside battery needs to be replaced with new one. Ask your local dealer for its replacement.

15. Karl Fischer reagent

For Karl Fischer titration, appropriate reagent must be selected to the sample that you are going to analyze. Below chart shows the type of sample and its corresponding reagents available on the market.

Karl Fischer reagent Corresponding sample	Coulomat (Riedel-de Haën)	Aquamicron (MITSUBISHI)	Remarks
General titration	Coulomat AG*	Aquamicron AX	Coulomat AG/CG,
Alcohols	Coulomat CG	Aquamicron CXU	Aquamicron AX/CXU
Hydrocarbons			are non-organic
Ethers		Aquamicron AS	chlorines.
Esters	Coulomat AD**	Aquamicron CXU	
Ketones	Coulomat AK*		Formaldehyde can only
	Coulomat CG-K		be titrated among other
			aldehydes.
	Coulomat AK*	Aquamicron AKX	Coulomat CG-K
	Coulomat CK	Aquamicron CXU	Aquamicron CXU are
			non-organic chlorines.
Gases	Coulomat AG Oven*	Aquamicron AX	
	Coulomat CG	Aquamicron CXU	
		Aquamicron AX	
	Coulomat AG*, Blended	Aquamicron CX	
	solution with Ethylene		
	glycol (Blend ratio/ AG:		
	Ethylene glycol=3:1)		
	Coulomat CG		
	Coulomat AD**		
Fats and Oils	Coulomat AG-H*	Aquamicron AX	Coulomat AG-H/CG,
	Coulomat CG	Aquamicron CXU	Aquamicron AX/CXU
		Aquamicron AS	are non-organic
		Aquamicron CXU	chlorines.
Amines	Coulomat AG	Aquamicron AX	To add, neutralize a basic
	Coulomat CG	Aquamicron CXU	amine with an acid.
	Coulomat AD**		
	To use Coulomat AG,	To use Aquamicron	
	Add acetic acid,	AX, add 10g	
	Salicylate or benzoic	salicylate acid to	
	Acid to 20% of 100mL	100mL Aquamicron	
	of Coulomat AG	AX	

Note) ****** possible to use for only single component cell

* possible to use for 2-compoonent cell or 1-component cell

16. Troubleshooting

16-1. Error messages and remedies.

Error message	Cause	Rectification
A.Capacity Over!	Accumulated electrolytic current	Replace anolyte.
	(moisture-converted)of anolyte has	Reset A.capacity value.
	exceeded anolyte capacity limit.	
C.Capacity Over!	Accumulated electrolytic current	Replace catholyte.
	(moisture-converted)of catholyte has	Reset C.capacity value.
	exceeded catholyte capacity limit.	
Current Error!	No electrolytic current flows.	Sample liquid resistance is too great.
		Check anode and cathode for breakage
		or poor contact.
		Make sure connectors are plugged in.
Elect Open!	No current to Detection electrode.	Check electrode for breakage or poor
		contact.
		Make sure connectors are plugged in.
Elect Short!	Detection electrode is shorted or cell	Check electrode connector for short-
	potential is very low.	circuit.
		Check cell potential and add water to
		raise above 200mV.
Meas. Over!	Moisture content measured has exceeded	Replace catholyte (anolyte if necessary),
	100 mg H ₂ O.	make background level stable and
		remeasure a smaller quantity sample.
Over Titr.!	Over-titration.	Clean anode if stained with foreign
	Excessive iodine is generated by anolyte	matter.
	with time.	Move unit to another place if exposed
		to direct sunlight.
		In case of pre-titration, add a little water
		to cell.
Para set miss!	time(max):0s and Drift stop off ↑ have	Set Drift stop rel \uparrow or abs \uparrow .
	both been set.	Set t(max).
Pre Amp Err-XX	Inside Pre Amp of instrument is not	Please contact your local dealer or
	working correctly.	distributor.

16-2. Troubles in power source



Line voltage and fuse ratings

AC	Line	Voltage	Fuse	Hz
100 V	90 -	121 V	T3.15 A/250V	50/60
120 V	103	- 132 V	T3.15 A/250V	50/60
220 V	198	- 242 V	T1.6 A/250V	50/60
230 V	207	- 253 V	T1.6 A/250V	50/60
240 V	216	- 264 V	T1.6 A/250V	50/60

Caution!

We accept no responsibility whatsoever for any fault in or damage to the electrical circuit caused by wrong setting of the voltage selector.

Caution!

For continued protection against risk of fire: Replace only with same type and rating of fuse.

16-3. Stirrer does not work properly



16-4. Drift level is too high



16-5. It runs into over-titration



16-6. Poor repeatability or no EP found



Moisture Content				Sampl	e Size		
50	~	100	%	10			mg
10	~	50	%	10	~	20	mg
1	~	10	%	10	~	50	mg
0.1	~	1	%	10	~	100	mg
0.01	~	0.1	%	100mg	~	1.0	g
0.001	~	0.01	%	1	~	10	g
0.0001	~	0.001	l %	10	~	20	g

For reproducibility of measurement results, moisture content and sample size relations in below chart is important.

16-7. Glass contact area jammed

If grease on the glass contact areas becomes hard and the respective parts are difficult to separate, take the following steps;

- 1) Discharge anolyte and catholyte.
- 2) Heat the glass contact areas with a hair dryer to melt the grease.



Do not try and open solidified jointed parts by force. Glassware may break into piercing pieces for injury.

17. Display message and glossary

Display	Printout	Meaning	
A.Alarm[mg]?	Reagent capacity A.Alarm 1000mg	Enter anolyte capacity alarm limit(mg H ₂ O).	
abs[g/s]?	Drift stop:abs 0.1 g/s	Enter drift stop value.	
A.Capa.[mg]?	Reagent capacity A.Capa. 10mg	Accumulated electrolytic current (moisture-converted) of anolyte is displayed (mg H ₂ O). Enter 0 when replacing anolyte.	
A.Capacity Over!		Error message when accumulated electrolytic current (moisture-converted) of anolyte has exceeded capacity alarm limit.	
Alarm Set:on↑	Alarm set:on	Select reagent capacity alarm.	
Bal:KEM↑	Balance :KEM	Select balance.	
		(KEM, Mettler, A&D, Shimadzu, Sartorius)	
Balance↑	<balance></balance>	Setting of balance parameters.	
Baud Rate:1200↑	Baud rate:1200	Set baud rate (300,600,1200,2400,4800,9600).	
Blank?		Enter blank. (subtracting from the result)	
Calculation↑		Setting of calculation parameters.	
C.Alarm[mg]?	Reagent capacity C.Alarm 300mg	Enter catholyte capacity alarm limit (mg H ₂ O).	
C.Capa.[mg]?	Reagent capacity	Accumulated electrolytic current of catholyte is	
	C.Capa. 10mg	displayed (mg H ₂ O). Enter 0 when replacing catholyte.	
C.Capacity Over!		Error message when accumulated electrolytic current of catholyte	
		has exceeded the preset catholyte capacity alarm limit.	
Current Error!		Error message when there is no electrolytic current.	
^		Check Inner Burette.	
Data bits:7	Data bits:7	Select Data bits. (7,8) when using RS-232C	
Date & Time	Date 96/02/13 16:34	Set Date & Time	
Drift?	Drift 0.00 g/s	Set recalculation parameter.	
Drift comp: auto↑	Drift comp: auto	Select drift compensation.	
		Auto : drift before titration is used	
		off : no drift compensation	
		manu : entry of drift value	
Drift Stop: rel ^T	Drift stop: rel 0.1 g/s	Select drift stop.	
		rel : stops when drift is below	
		(entered value + drift before determination).	
		abs : stops when drift is below entered value	
		off : no drift stop, termination after t(max).	
Elect Open!		Detection electrode is defective (open).	

Display	Printout	Meaning	
Elect Short!		Detection electrode is defective (short-circuit).	
Factor?	Factor 1.00	Enter factor. (multiplication of result)	
Function [0-3]?		Enter Function number.	
GLP↑	GLP Format	Measurement results are printed out in the GLP format.	
HH MM?		Enter hour and minute.	
Interface	Interface	Set up the parameter for Interface.	
Lot ?	Lot No.	Enter lot number.	
Meas. Over!		Error message when the measured moisture content has exceeded 100 mg H_2O .	
Memory clr.:off↑		Message to reinitialize all parameters.	
Name	Name	Enter user's name.	
Over Titr.!		Over-titration.	
		Excessive iodine has been generated by the anolyte.	
Para set miss!		Error message when time limit parameter is at 0s and Drift	
		stop is off.	
Parity:even↑	Parity:even	Set parity when using RS-232C.	
		Select odd parity, even parity or no parity.	
Pot.		Shows voltage inside of titration vessel.	
Pre Amp Err-XX		Pre Amp Error.	
Pre-Titr		Pretitration in progress.	
		Waits for pre-titration by blinking.	
Printer:IDP [↑]	Printer:IDP-	Select printer.	
Printer↑	<printer></printer>	Setting of printer parameters.	
Ready		Water content inside the cell changes within 10.0 g/s, and	
		is "Ready" for measurement but before the drift is stable.	
		For accurate measurement wait until "Stable" sign	
		appears.	
Reagent capacity	Reagent capacity	Select reagent capacity display and alarm limit.	
Recalculation↑	[Recalculation]	Setting of recalculation parameters.	
Report↑	[Report]	Setting of result parameters.	
rel[g/s]?	Drift stop:rel	Enter drift stop value.	
	0.1 g/s	(entered value+drift before determination)	
RS232C↑	<rs232c></rs232c>	Setting of RS-232C parameters.	
Samp.In.		After sample injection, press [Start] key again	
Start			
Sample No. ?	Sample No.	Enter the sample number.	
Serial No.	Serial No.	Check the serial number	
Setup[0-3]?		Enter setup No.	

Display		Printou	t	Meaning
Soft HS:off↑		Soft HS	:off	Select off/on of software handshake when using RS-232C.
Short↑		Short For	mat	Measurement results are printed out in the Short format.
Speed[0-9]?				Enter stirrer speed (0:stop)
SRAM Init.				This message appears when memory is initialized to default.
Stable				Means precise moisture can be measured since water content fluctuates within 0.1Δ g/min.
Stir.Time:5				Time until electrolysis starts is counted down.
Stop bits:2↑		Stop bits	:2	Set stop bit (1or2) when using RS-232C.
t(max)[s]?:0		t(max)	0s	Enter t(max) limit.
t(stir)[s]?:0		t(stir)	0s	Enter t(stir) time.
t(wait)[s]?:15		t(wait)	15s	Enter t(wait) time. (minimum 15s)
Unit: g↑		Unit	: g	Select results calculation unit. (g, mg, ppm, %)
Weight:fix↑		Weight	: fix	Select sample weight fixed or variable.
			5.0000g	
Wt1.Wt2		Wt1	2.3456g	Manual input of sample weight
		Wt2	2.1234g	Transfer sample weight from balance.
YY MM DD?		Date 95/0	6/01 00:00	Enter year, month and day.
*	(blink)	Pre-Titr		Pretitration in progress.
⇒	(blink)	Ready / S	table	Measurement ready.
*		112.3 g		Sample measurement in progress.

18. Others

18-1. Part list





18-2. Option

Part code	Part name	Qty	Remarks	Sketch
20-040-4200-48	Titration cell (with a cock)	1	Transparent cell with a drain cock	
12-006-7300-48	Titration cell unit (single solution) set	1	Transparent cell One-component cell Twin platinum electrode / KF and other attachment	Visiching bottle Septum (Spossier) WF grease Funnel Arocka adjuste
12-006-7400-48	Titration cell unit (two solution) set	1	Transparent cell Two-component cell Twin platinum electrode / KF and other attachment	Washing bottle Septurn (10ccriset) Funnel Anode osjuter
12-006-6200-48	Titration cell unit (single solution)	1	Transparent cell with a drain cock One-component cell Twin platinum electrode / KF and other attachment	
12-006-6100-48	Titration cell unit (two solution)	1	Transparent cell with a drain cock Two-component cell Twin platinum electrode / KF and other attachment	
12-006-6300-48	Titration cell unit (two solution) set (with a cock)	1	Transparent cell with a drain cock Two-component cell Twin platinum electrode / KF and other attachment	
12-006-6400-48	Titration cell unit (single solution) set (with a cock)	1	Transparent cell with a drain cock One-component cell Twin platinum electrode / KF and other attachment	

Part code	Part name	Qty	Remarks	Sketch
98-433-0117	Sampling set	1	For powders	
98-433-3389	Liquefied gas sampler	1		
98-433-3393	Adapter for gas	1	Bubbler for integrating flow meter	
98-433-0131	Manual absorb and drain unit	1		Bittle bilder 1 Bittle bilder 2 Bittle bilder 3 Bittle bilder 3 Bittle bilder 3 Bittle bilder 3
98-500-3225	Syringe 20mL	1		
98-030-0002	Connecting cable	1	9pin-9pin	
SOFT-CAPE	Data capture software	1		
ADP-511S	Evaporator	1	Room to 300 C	
ADP-512	Evaporator for ores	1	Room to 200 C Room to 1000 C	
ADP-512S	Evaporator for high temperature	1	Room to 1000 C	

Part code	Part name	Qty	Remarks	Sketch
ADP-513	Evaporator for oil sample	1	Room to 200 C	
IDP-100-10	Impact dot printer	1	100V	
IDP-100-11		1	120V	
IDP-100-12		1	230V	100 / 100 /
			With cable	
98-350-0001	Conversion connector	1	9P-25P	

Regent and standard solution

Part code	Part name	Qty	Remarks	Sketch
98-812-4055	Coulomat AG	1	Anolyte for general purpose 500mL	
98-812-4056	Coulomat CG	1	Catholyte for general purpose 5mL 10pcs/set	
98-812-4057	Coulomat AG-H	1	Anolyte for oil 500mL	Ĭ
98-812-4017	Coulomat AK	1	Anolyte for ketone 500mL	Ĭ
98-812-4033	Coulomat CG-K	1	Catholyte for ketone 5mL 10pcs/set	
98-812-0002 98-812-0003	Water standard	1	Factor 1.0 Factor 0.1 10pcs/set	

19. System chart


20. Pin assignment of I/O interface

(1) RS232C



(2) Balance



(3) Printer



21. Specification

Name and model	MKC-501 Karl Fisher Moisture Titrator
Measurement method	Karl Fisher coulometric titration method
Control method	Constant current pulse time control
Endpoint detection	AC polarization
Titration status display	Pre-titr (Excessive moisture state)
	Ready (Measurable state)
	Stable (Stable drift state)
Endpoint indication	Electronic: Audible
Stirring method	Magnetic stirrer
	Stirring speed is set by key entry
Titration cell volume	100 mL (150 mL max)
Measurable range	10 g to 100 mg H_2O content in test sample
Display resolution	0.1 g H ₂ O
Reproducibility	Coefficient of variance from measurement of 1mg H ₂ O in methanol is below 0.3%
	(n=10)
Drift	Automatic compensation function is selectable.
	Drift is shown on the display at all times.
Moisture quantity display	0.1~9999999 g
Alarms	Electrolysis current, Measurement value, Overtitration, Reagent life (anolyte/
	catholyte), Electrode contact(short/open), Parameter error, Preamplifier error,
	Inhibited key entry
External I/O	Printer : IDP-100 prints Date and time, Sample ID, Sample weight, H ₂ O
	content, etc.
	Balance : Automatic weight reading
	Computer : RS-232C Interface
Ambient condition	Temperature : 5 to 35°C
	Humidity : below 85%RH
Interfaces (standard)	Electronic balance is connectable and weighing automatically inputtable.
	Connectable through RS-232C interface to an external computer.
	Printer is connectable.
Power supply	AC 100/120/220/230/240 V
	50/60Hz
Power consumption	Aprox.50W
Outer dimensions	Main unit: $288(W) \times 468(D) \times 475(H)$ mm
Weight	Aprox.7.5 kg
CE marking	EMC : EN61326
	LVD . ENOIDID-I conformed

22. Warranty and After-Sale Service

1. Warranty Period

Three (3) years from the date of receipt of this product or the date of installation by KEM service personnel or by authorized personnel.

2. Warranty Details, After-sales Service

This product passed the strict inspections of KEM and, except for consumables, KEM warrants this product, under normal use, for three (3) years from the date of receipt of this product or the date of installation by KEM service personnel or by authorized personnel. (Parts and consumables will be supplied for at least seven (7) years after discontinuation of this product.)

Should an initial failure occur during the warranty period, KEM will decide whether to replace the product or to correct defects.

This product can be repaired at user's site by KEM service personnel or by authorized personnel. Note that secondhand or pre-owned products are not covered by warranty.

"KEM Warranty Guidelines" is available on our website (www.kyoto-kem.com).

3. Exclusion

Warranty shall be void where:

- any part is replaced or any repair or remodeling is performed by unauthorized personnel;
- unauthorized service parts, spare parts and/or consumables are used;
- the user does not follow the instructions for installation, correct use, maintenance and/or storage, resulting in malfunction;
- the user does not follow the ranges and/or conditions stated in the product brochure, flyer or specifications;
- periodic checks and/or maintenance is not performed;
- breakage and/or malfunction is caused by careless handling such as, but not limited to, exposing to or submerging in water, or dropping down;
- breakage and/or malfunction is caused by excessive force applied to glassware or plastics;
- malfunction or leakage is caused by sample properties (corrosivity, solid materials, etc.);
- malfunction is caused by any device, part and/or chemical other than those supplied by KEM;
- overuse has led to fatigue or wear of parts;
- items are consumables or wearing parts;
- this product has been moved or transported to another place once accepted and installed;
- breakage and/or malfunction is caused by conditions beyond control of KEM including, but not limited to Acts of God such as fire, earthquake, lightning strike, flood, etc.;
- failure or malfunction of the diaphragm pump is caused by samples;
- parts including, but not limited to the touchscreen, keypad or LCD, are broken due to improper or inadequate handling such as spilling chemicals;
- items are consumables, accessories or wearing parts such as, but not limited to, electrodes, glassware, tubing and pump seals, or parts which are in direct contact with samples and/or reagents and are considered consumables due to normal wear.

KEM is also unable to offer warranty and related services of repairs and maintenance checks of any kind once specifications, capability, features and/or functions of this product as well as its parts are changed, altered or remodelled by unauthorized personnel.

4. Disclaimer

KEM is not held liable, during or after the warranty period, regardless of whether loss or damage is caused by any event beyond control of KEM, or it is the user's opportunity loss and/or lost earnings caused by failure or malfunction of KEM products, or with or without predictability of KEM, for loss or damage resulting from a particular reason, secondary loss or damage, accident compensation, damage to products other than those supplied by KEM, and any other incidental compensation.

KEM is also not held liable for physical and/or economic loss or damage resulting from the use of KEM products, or loss of stored data during repair or servicing of such product.

KEM KYOTO ELECTRONICS MANUFACTURING CO., LTD.

Overseas Division: Yamawaki Bldg 9F 4-8-21 Kudan Minami, Chiyoda-ku, Tokyo 102-0074 JAPAN FAX: +81-3-3237-0537, Phone: +81-3-3239-7333 E-mail: kem.overseas-tokyo@kyoto-kem.com <u>URL:http://www.kyoto-kem.com</u> Factory: 56-2 Ninodan-cho,Shinden Kisshoin,Minami-ku,KYOTO 601-8317,JAPAN

FAX: +81-75-691-9961, Phone: +81-75-691-4122