Karl Fisher Moisture Titrator



# **Operation Manual**

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

Ver.02 A/N 69-001-0501-48

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## 1. Introduction

#### 1-1. Foreword

The MKC-520 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-520]

- <u>Easy to operate</u> 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 H<sub>2</sub>O water-methanol.
- <u>Variety of standard interface</u> Interface for Computer, Balance and Printer is standard
- <u>Bromine index · number can be measured.</u> Not only water content but bromine index · number of petroleum products can be measured.
- <u>One-component cell can be used. (Option)</u> One-component cell allows for easy maintenance in replacing reagents and lower running cost.
- Dispenser for Karl Fischer reagent is standard equipment.

The reagent dispenser as standard equipment eliminates troublesome replacements. The open air does not go into a titration cell at the time of reagent exchange. Therefore, the stability after reagent exchange is quick.

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.

# 🕂 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 of magnetic stirrer, 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.
- In draining waste liquid, dispose waste liquid before the drain pot is filled up with it; failure to follow this caution can be the cause of the gushing of waste liquid from the rubber globe.

#### 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. Unpacking

Below is the flow chart from unpacking the delivered instrument to starting actual measurement:

Unpack the carton box and check the supplied parts.	(see 1-5)
↓	
Ready for start-up.	(see 4-2)
↓	
Check the voltage of power source and voltage select	or, and identify
the loaded power fuse.	(see 4-1)
↓	
Connect the stirrer to Main unit, and place the titration	on cell in
position and connect the electrode.	(see 5-1)
$\downarrow$	
Set up the titration cell and supply anolyte and cathol	lyte.
	(see 5-2)
	(300 3 2)
↓	(300 3 2)
↓ Turn on the power and start pretitration.	(see 5-2)
$\downarrow$ Turn on the power and start pretitration. $\downarrow$	(see 5-2)
↓ Turn on the power and start pretitration. ↓ Select each parameter for the sample you are going to	(see 5-2) (see 5-2)
↓ Turn on the power and start pretitration. ↓ Select each parameter for the sample you are going to set up the Method.	(see 5-2) (see 5-2) o measure, and (see 8)
↓ Turn on the power and start pretitration. ↓ Select each parameter for the sample you are going to set up the Method. Setup Sample file (see 10-6) and Factor value (see10-	(see 5-2) (see 5-2) o measure, and (see 8) 8) if necessary.
↓ Turn on the power and start pretitration. ↓ Select each parameter for the sample you are going to set up the Method. Setup Sample file (see 10-6) and Factor value (see10- ↓	(see 5-2) (see 5-2) o measure, and (see 8) 8) if necessary.
↓ Turn on the power and start pretitration. ↓ Select each parameter for the sample you are going to set up the Method. Setup Sample file (see 10-6) and Factor value (see10- ↓ Make settings for Setup function.	(see 5-2) (see 5-2) o measure, and (see 8) 8) if necessary. (see 9)
↓ Turn on the power and start pretitration. ↓ Select each parameter for the sample you are going to set up the Method. Setup Sample file (see 10-6) and Factor value (see10- ↓ Make settings for Setup function. ↓	(see 5-2) (see 5-2) o measure, and (see 8) 8) if necessary. (see 9)

## 1-5. 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.

Jointa		ľ	мкс-520-D	MKC-520-N
(1)	Main unit	MKC-520	1 unit	1 unit
(2)	Power cord with earth (AC 100/120V area)	. 98-320-3198	1 pce	1 pce
	(AC 220/230/240V area)	. 98-320-3461		
	(for UK)	. 98-320-4199		
	(for China)	. 64-000-1800-4	8	
(3)	Adapter for power connector (AC 100V area only)	. 98-320-3199	1 pce	1 pce
(4)	Earth wire (AC 100V area only)	98-433-3331	1 pce	1 pce
(5)	Magnetic stirrer	. 12-001-8600-4	8 1 pce	1 pce
(6)	Titration cell holder	. 98-433-3496	1 pce	1 pce
(7)	Stirrer cable	. 98-428-0031	1 pce	1 pce
(8)	Titration cell (transparency)	. 20-040-4100-4	8 1 pce	1 pce
(9)	Stirrer rotor (35mm)	. (98-500-3362)	1 pce	1 pce
(10)	Inner burette (2-component burette)	. 98-433-0006	1 pce	_
	(1-component burette)	. 98-103-0002	_	1 pce
(11)	Twin platinum electrode / KF	98-103-M713	1 pce	1 pce
(12)	Desiccant tube A	. 98-433-3116	1 pcs	1 pce
(13)	Port plug (19/25)	98-550-0073	1 pce	1 pce
(14)	Syringe inlet (with septum)	. 98-433-3407	1 pce	1 pce
(15)	KF grease (5g)	. 98-433-3138	1 pce	1 pce
(16)	Septum (10pcs/set)	98-523-3161S	1 set	1 set
(17)	Bottle holder	. 98-560-0017	1 pce	1 pce
(18)	Polyethylene bottle (1L)	. 69-000-2800-4	8 1 pce	1 pce
(19)	Reagent bottle cap (with plug)	98-430-0052	1 pce	1 pce
(20)	Rubber globe for drain	98-433-0105	1 pce	1 pce
(21)	Reagent bottle cap	98-430-0058	1 pce	1 pce
(22)	Rubber globe	. 98-433-0069	1 pce	1 pce
(23)	Plug for titration flask	98-550-0076	1 pce	1 pce
(24)	Desiccant tube	. (98-444-6542)	1 pce	1 pce
(25)	Injection tube	. 98-433-0120	1 pce	1 pce
(26)	Drain tube	98-433-0121	1 pce	1 pce
(27)	Bottle holder 1	(98-569-0001)	1 pce	1 pce
(28)	Bottle holder 2	(98-569-0002)	1 pce	1 pce
(29)	Bottle holder 3	(98-569-0003)	1 pce	1 pce
(30)	Bottle stand	. (98-569-0004)	1 pce	1 pce
(31)	Washing bottle	. 98-500-3134	1 pce	1 pce*
(32)	Funnel	. 98-500-3159	1 pce	1 pce

(33) Anode adjuster	98-075-3411	1 pce	1 pce*
(34) Operation manual	69-001-0501-48	1 copy	1 copy
(35) Quick reference	69-001-0505-48	1 copy	1 copy

## Note:

Parts with parenthesized part code have unique order units or packing forms different from others. Therefore, please refer to the section "17. 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  $2 \times 96500$  coulombs, or 10.72 coulombs/ 1 mg H<sub>2</sub>O. The total amount of moisture can thus be determined by measuring the total consumption of electricity.



## 3. Parts name and function





#### Key configuration





## 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 13-2-6. 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.







< 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. Connect with Magnetic stirrer

Connect the magnetic stirrer to Main unit.



## Caution!

Turn OFF power of Main unit before plug in Stirrer cable in order to avoid electric trouble in the unit.

## 5-2. Titration cell setup

#### 5-2-1. Assembly of the titration cell

- 1) Fix the cell holder to the magnetic stirrer.
- 2) 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.



3) 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.



#### 5-2-2. Installation of KF reagent dispenser

- 1) Insert the rubber globe onto the reagent bottle cap.
- 2) Connect the drain tube to the reagent bottle cap (with rubber stopper), and the injection tube to the reagent bottle cap.
- 3) Fix the cap (with plug) to the polypropylene bottle.
- 4) Fix the desiccant tube to the rubber globe.
- 5) Connect the reagent bottle to a commercially sold KF reagent bottle filled with anolyte.



## Note:

If loosely squeezed it in, pressurized air may leak and it may cause malfunction of dispensing KF reagent. There are two kinds of rubber globes, one for drain and the other for discharge of reagent. Both of them are indicated by the joint on top of each.

- 6) Install the desiccant tube onto the reagent bottle holder.
- 7) Place the reagent bottle in the bottle holder. If the outside diameter of reagent bottle does not match the holder, use the bottle holder and bottle stand as shown below.
- 8) Connect the tube for drain and injection to the Plug for titration flask as shown below respectively.



9) Insert the Plug for titration flask carefully into the titration cell. At this point, apply a small amount of KF grease on slide contact area.



## 5-3. Connecting additional measuring unit

The MKC-520 can perform 2 ways of titration if the optional Stirrer unit E or EP and Titration cell unit is connected. For example, the first cell is used for regular titration and the second cell for evaporation or ketone measurement, or the first cell for the moisture and the second for bromine index/ number.

- 1) Connect the supplied stirrer cable between MKC-520 and the optional stirrer unit E/EP as shown below.
- 2) Connect the supplied stirrer cable (0.3m) between the two stirrers as shown.



- \* Standard stirrer is 1ch, and additional stirrer is 2ch.
- 3) Install the titration cell according to 5-2. Titration cell setup.

## 5-4. Initial display

When the power is turned on, the message (1) will appear on display for two seconds. Then, the message (2) will appear, which keeps on until pretitration starts.



## 6. Basic operating method

## 6-1. Selecting Cell No. and Channel

#### Note:

The MKC-520 needs to change titration control depending on titration cell type. Therefore, the cell type appropriate for titration has to be configured. If a wring cell is selected, it may cause erroneous measurement results.

Press [Method] key.

		shows cell channel
	< METHOD > Protect	C/e I./. No.
METHOD 1: METHOD 2:	Off On Off On	1 - 2 C o m p . 1 - 2 C o m p .
METHOD 3: METHOD 4:	<u>Off</u> On Off On	1 - 2 C o m p . 1 - 2 C o m p . 1 - 2 C o m p .
METHOD 5:	<u>Off</u> On	1 - 2 C o m p .

Select Cell Number with [-/Disp] key according to the cell and channel to be used.

			*
Cell No.	Cell ch.	Titr. cell	Option
1–2Comp.	1ch	2 components	
2–2Comp.	2ch	2 components	Stirrer unit E or EP and Titration cell unit
1–1Comp.	1ch	1 component	
2–1Comp.	2ch	1 component	Stirrer unit E or EP and Titration cell unit

Each time [-/Disp] key is pressed, display of Cell No. changes one after another from the top.

- 1 ch is the measuring unit, which is standard supplied.
- 2 ch is the additional measuring unit.
- Select 2 components for regular measurement using anode/cathode reagent with the electrolytic electrode with diaphragm.
- Single component is for measurement using anode reagent only and diaphragm-less electrode.

## 6-2. Filling reagent and draining

#### < Catholyte >

Inject the 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.



#### Note:

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

#### < Anolyte >

To fill the reagent, pump the rubber globe with fingers while holding the seal of reagent bottle cap. 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 stop filling, detach your finger which is holding the seal. To drain it out, pump the rubber globe of drain bottle a few times. The used reagent in the cell transfers to the waste bottle.



## 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.

## 6-3. Pretitration

The Karl Fisher reagent after a period of storage may have absorbed moisture, and correct measurement for water content cannot be expected if it is directly used.

Press [Pre-Titr.] key to start pretitration. The display will show the message of pretitration going on. After pretitration is finished, it will shift to Standby mode. (6-4)



## Note:

- When the indication of unstable drift is displayed, possible causes would be a leakage, the expiration of reagent life or the like. Then the countermeasures would be the application of the KF grease onto the ground portion of the titration cell, the replacement of reagents or the like.
- 2. Each parameter can be set up during pretitration.

## 6-4. Main display

The Main display message will appear after pretitration is finished.



Displays sample number waiting for measurement or being measured.

## Note:

When reliable measurement results are expected, operate moisture determination by "Stable" mode.

## 6-5. Routine measurement

1) Press [Pre-Titr.] key to dehydrate the measuring cell.

When the drift stabilizes after dehydration, the message "Stable" appears on display.

Cell-1 No. 01-01 Drift (µg/s)	[METHOD 1]
0.02	Stable
Press	(Start) key

2) Press [Start] key.

The message "Inject sample and press [Start] key" on display.

- 3) Inject sample liquid by a syringe into the measuring cell.
- 4) Press [Start] key.

Measurement is started and the display shows "Measurement" with reading of water content.

Cell-1 No. 01-01 Drift (µg/s)	[METHOD 1] Moisture (µg)
10.5	921.5
Measu	rement

5) When the measurement is finished, the display shows the results with beep.

Cell-1 No. 01-01 Drift (µg/s)	[METHOD 1] Moisture (µg)	
0.03	1001.3	
Result		

When a printer is connected, the results will be printed out at the same time.

## 6-6. Sequence of measurement

Routine measurement like direct titration of liquid sample by MKC-520 can be performed without difficult key operation.

Simply follow the dialog message on display.



#### 6-7. Example of measurement

The example shows a measurement of moisture of Dichloroethane with default parameters. Three measurements were performed resulting with as good 0.3% RSD.

Sample material	Dichloroethane
Catholyte	Coulomate CG
Anolyte	Coulomate AG
Detection electrode	Twin Pt electrode
	M-713
Electrolysis	Inner burette
	(2-component burette)
	(98-433-0006)

#### Measurement parameter

[Titration]				
Date 1999/0 Titr. Cell No Method t(stir) t(stir) Drift Stop : Cont. Gain Stanble Start Oven	)6/10 .1	13:00 1 0s 15s 0s Rel 0.1 ug/s 5.0 0.1 ug/min Manual off		
[Calculation]				
Date 1999/0 Titr. Cell No Calc. No. Unit Weight Drift Comp	)6/10 .1	13:00 1 ug Variable Auto		

#### Result

```
* * * R e s u l t * * *
Sample No.
                                        01-01
Date 1998/04/10 13:11
Titr. Cell No.1
Wt1 33.3160 g
Wt2 27.3584 g
Net 5.9576 g
Result 269.1 ug
45.169 ppm
    * * * R e s u l t * * *
Sample No.
                                        01-02
Date 1998/04/10 13:14
Titr. Cell No.1
Wt1 27.3585 g
Wt2 21.4480 g
Net 5.9105 g
Result 269.4 ug
45.580 ppm
    ***Result***
Sample No.
                                        01-03
Date 1998/04/10 13:18
Titr. Cell No.1
Wt1 21.4438 g
Wt2 15.3890 g
Net 6.0548 g
Result 269.9 ug
                            44.576 ppm
 <Auto Statistics>
Date 1998/04/10 13:23
Sample No.(High) 01
Method 1
Results
                                         3
                             45.108 ppm
0.4120 ug
0.9134 %
Mean
SD
RSD
```

## 7. Initialization and Default values

## 7-1. Default value of parameters

Param	leter	Default
Method		1
Protect		Off
Cell		1-2 Comp.
Titration	t(stir)	0s
	t(wait)	15s
	t(max)	0s
	Drift Stop	Rel
	Rel	0.1 μ g/s
	Abs	0.1 μ g/s
	Control Gain	5.0
	Stable	$0.1\Delta \mu$ g/min
	Start	Manual
	Oven	Off
	ADP-	
	Oven Temp.	150°C
	Pre Treat	2
	Back Purge	180s
	Cell Purge	120s
	Sampler Purge	180s
Result		Calculation
Calculation	Calc.No.	1
	Unit	$\mu$ g
	Weight	Variable
	Drift Comp.	Auto
	Manu.	0.0

Param	neter	Default
Report		Short
Variable	Sample No.	On
	Date&Time	On
	Lot No.	Off
	Method	Off
	Calc. No.	Off
	Size	On
	Result	On
	Drift	Off
	Blank	Off
	Reagent Capa	Off
	Titration Time	Off
	Factor	Off
	Operator	Off
Titr. Curve	Sampling Interval	30s
	Display	Off
	Print Meas. Data	Off
	Print Graph	Off
Sample	ID	0
	Operator	
	Sample No.	01-01
	Blank	0.0µg
	Lot No.	-
	Wt1	5.000g
	Wt2	0.0g
	Wt0	5.000g
	В	0.0g
	Α	0.0ppm
	V1	1.000mL
	Dens	1.000g/mL
	V2	1.000L
	Temp.	25.0°C
Param	leter	Default
------------------	-----------------	---------------
Function		Reagent Capa.
Reagent		
Reagent Capacity	Titr. Cell No.	1
	[Anode]	
	Capacity	0mg
	Alarm set	Off
	Alarm	99 day
	Reagent Life	1000mg
	Reagent Name	
	Lot No.	
	[Cathode]	
	Capacity	0mg
	Alarm set	Off
	Alarm	99 day
	Reagent Life	300mg
	Reagent Name	
	Lot No.	
Sample	Sample File	Off
Oven / Purge	Back Purge	1800s
	Cell Purge	600s
Factor	Cell-1	1.00
	Cell-2	1.00
Calibration	Titr. Call No.	1-2 Comp.
	Calibration	Off
	Standard Value	1000ppm
	Tolerance	$\pm$ 30ppm
Periodic Ck.	Next Check Date	/ /
	Alarm	Off
	Internal	1 month
	Display	List
	Memory Clear	No
Memory Clear		Off
Stirrer		4

# 7-2. Default of each Method

	Default of each Method					
Measurement	Direct measuren sample	nent of liquid	Measurement of powder sample b	Measurement of solid and powder sample by ADP-511		
			Evaporator		Measurement	
Parameter	1	2	3	4	5	
Titration						
t(stir)	0s	0s	0s	0s	0s	
t(wait)	15s	15s	15s	15s	15s	
t(max)	0s	0s	1200s	1200s	0s	
Drift Stop	Rel	Rel	Off	Off	Off	
Rel	0.1 μ g/s	0.1 μ g/s	0.1 μ g/s	0.1 μ g/s	1.0 μ g/s	
Control Gain	5.0	5.0	5.0	5.0	5.0	
Stable	$0.1 \ \mu \ \text{g/min}$	$0.1 \ \mu \ \text{g/min}$	$0.1 \ \mu \ \text{g/min}$	$0.1 \ \mu \ \text{g/min}$	0.5 μ g/min	
Start	Manual	Manual	Auto	Auto	Manual	
Oven	Off	Off	ADP-	ADP-	Off	
Oven Temp.			150°C	150°C		
Pre Treat			2	2		
Back Purge			180s	180s		
Cell Purge			120s	120s		
Sample Purge			180s	180s		
Result						
Calculation						
Calc. No.	1	2	2	2	7	
Unit	$\mu$ g	ppm	ppm	ppm	ms/100g	
Weight		Variable	Variable	Variable	Variable	
Drift Comp.	Auto	Auto	Auto	Auto	Auto	
Drift	0.0	0.0	0.0	0.0	0.0	
Report	Short	Short	Short	Short	Short	
Titr. Curve						
Sampling Interval	30s	30s	30s	30s	30s	
Display	Off	Off	Off	Off	Off	
Print Meas. Data	Off	Off	Off	Off	Off	
Print Graph.	Off	Off	Off	Off	Off	

	Default of each Method						
Measurement	Direct measuren sample	nent of liquid	Measurement of powder sample b Evaporator	Bromine Index and Value Measurement			
Parameter	1	2	3	4	5		
Sample							
Sample No.	01-01	01-01	01-01	01-01	01-01		
Lot No.	-	-	-	-	-		
Blank	0.0						
Wt1		5.000	5.000	5.000	5.000		
Wt2		0.000	0.000	0.000	0.000		
D					1.0		

# Note:

The above parameters can be changed, and when initialized, their values will return to the above parameters.

# 8. Extension of Measuring Operation

## 8-1. Entry of parameters

#### 8-1-1. General

Moisture measurement by MKC-520 can be made on default settings without changing initial parameters, however, depending on samples or how they are going to be measured, more precise and faster measurement can be performed by selecting appropriate parameter values.

Below is the list of parameters.

Parameter in [] can be entered by characters or numbers using numeric key, [·] key or [-/Disp.] key. Indicated cursor and underlined parameters can be selected by [ $\uparrow$ ], [ $\downarrow$ ], [ $\leftarrow$ ] or [ $\rightarrow$ ] key.

## 8-1-2. Method parameter

Method can be selected here with parameters including Titration, Calculation and Print parameter. Each parameter is saved independently from Method 1 to 5. Default values differ from Method 1 to 5, however, each of them can be modified freely. The parameters you have selected can be protected by Protect function.

Method 7 4 1 0

8 9

5 6

2 3

-/Disp.

Method	11	2	2	3	3	2	1	5	5
Titratio	n								
Resul	t								

Press [ Method ] key:

	< M E T H O D	>
	Protect	Cell No.
METHOD 1:	Off On	1-2 Comp.
METHOD 2:	<u>Off</u> On	1-2 Comp.
METHOD 3:	<u>Off</u> On	1-2 Comp.
METHOD 4:	<u>Off</u> On	1-2 Comp.
METHOD 5:	<u>Off</u> On	1-2 Comp.

The display changes as shown above by selecting Method number for desired titration or settings using [ $\uparrow$ ] or [ $\downarrow$ ] key. Protect function can be chosen by [ $\leftarrow$ ] or [ $\rightarrow$ ] key.

The channel of measuring cell and its kind can be selected by [-/Disp] key. (Typically use 1-2 Comp.) The display will return to Main display by [ $\downarrow$ ] key.

Cell No.	Cell ch.	Titr. cell	Option
<u>1–2Comp.</u>	<u>1ch</u>	2 components	
2–2Comp.	2ch	2 components	Stirrer unit E or EP and Titration
			cell unit
1–1Comp.	1ch	1 component	(Stirrer unit P)
2–1Comp	2ch	1 component	Stirrer unit E or EP and Titration
2 icomp.	2011		cell unit

## 8-1-3. Titration parameter

Parameters to control titration are selected here including how to detect endpoint or set up drift criteria.

Message display after [ Titration ] key is pressed:

< TITRATI	0N >	[METHOD 1]	▼
t(stir) t(walt) t(max) Drift Stop Rel	[ [ : Off [	0]s 15]s 0]s <u>Rel</u> Abs 0.1]μg/s	

Select item by  $[\uparrow], [\downarrow], [\leftarrow], [\rightarrow]$  key and enter desired numbers or conditions by  $[\downarrow]$  key.

t(stir)	: Where the sample you going to measure is a type of sample which takes time in dissolving in the anolyte and dehydrated, you can set up waiting time (=stir only) until titration (electrolysis) starts after the sample is injected.
t(wait)	: Where the sample has two inflection points and may reach endpoint by drift, the shortest possible time for titration termination is necessary to set up. Titration will not terminate during the wait time.
t(max)	<ul> <li>The longest time for titration is limited by this setup. Thus, total titration time is: t(stir)+t(wait)+t(max)</li> <li>Printed measurement time is t(wait)+t(max) and dose not include t(stir) t(max)=0 means titration will not terminate by time limit, for instance, where Evaporator is connected or minute moisture content continues to be extracted in the anolyte or titration does not end by drift.</li> </ul>
Drift Stop	: Off Rel Abs
	You can define a drift stop for reaching endpoint.

Off	:	Endpoint will not be detected by drift stop. It is the time total of
		t(stir)+t(wait)+t(max), for example, where endpoint has to be determined when most
		of the moisture content has been titrated while the sample releases small amount of
		moisture slowly in evaporation or when you want to know extracted moisture from a
		sample by unit time.
Rel	:	This is commonly used direct method of EP detection by drift stop, where endpoint is
		determined when the drift during titration reduces below [Drift at start]+[preset value].
Abs	:	This is another EP detection by drift stop, which determines endpoint when the drift
		during titration reduces below [ preset value] regardless of drift at start. Here, t(max)
		must be defined. Otherwise, it will not terminate titration if the drift goes up higher
		than the preset values.

Note : Criteria for drift stop must be defined when Rel or Abs is selected.

Message display when [Page down] key is pressed. ([ $\downarrow$ ] or [ $\downarrow$ ] key can be used if the cursor stays at the bottom line):

```
< TITRATION > [METHOD 1] ▲
Control Galn [ 5.0]
Stable [0.1]∆µg/min
Start :<u>Manual</u> Auto
Oven :<u>Off</u> ADP- CHG-
```

Here, you select each parameter on <TITRATION> second display.

Select item by cursor using  $[\uparrow], [\downarrow], [\leftarrow], [\rightarrow]$  key and enter desired amount or conditions and then, confirm by  $[ \downarrow ]$  key.

Control Gain : Select speed of electrolysis. (Normal 5.0) If reaction speed is too fast, reduce the speed to avoid over-titration. Select a larger amount for a sample which contains sample water and would take a long time to reach the endpoint.

Stable	:	This status appears when the change in drift $\Delta \mu$ g/min goes below preset amount that you entered. For example, if "Stable" does not appear for titration due to unstable moisture in the carrier gas for evaporation, select higher setting. But the higher setting will lead to deviation from true amount and to less precise results.
Start	:	When titration(electrolysis) start is set by sample injection(stir) after [Start] key is pressed, select Auto or Manual count-down to start. When Auto is selected, titration will start for testing titration without sample or for measurement of a sample of small amount of moisture.
Oven	:	Select to control ADP or CHG or not to control.
Off	:	Not to control ADP or CHG-
ADP-	:	Select ADP- when Evaporator is connected. ADP is selected, set up the following parameters by pointing ADP- by cursor to turn on " $\blacktriangle$ " on display.
CHG-	:	Select " CHG - " to control CHK-501.

Display after [Page down] is pressed:  $([\downarrow], [\downarrow]]$  key will work if the cursor stays at the bottom line)

< TITRATION >	[METHOD 1] ▲
Oven Temp. Pre-Treat(1~3) Back Purge Cell Purge SamplerPurge	[ 150]°C [1] [ 180]s [ 120]s [ 180]s

Here, you select each parameter on  $\langle \text{TITRATION} \rangle$  third display. Select item by cursor using  $[\uparrow], [\downarrow], [\leftarrow], [\rightarrow]$  key and enter desired amount or conditions and then, confirm by  $[\downarrow]$  key.

Oven Temp. : Select temperature for vaporizing by Evaporator, depending on, for instance, melting point of the sample or dissociation degree of crystallized water. (Max. 300°C)

Pre -Treat (1 to 3)	:	Select by method of sampling (Details follow):
		Pre-Treat 1 (using eggplant type sampler)
		Pre-Treat 2 (direct injection to the sampler)
		Pre-Treat 3 (using sample boat)
Back Purge	:	Set up time for carrier gas to purge between the sample inlet of Heater tube and the
		boat outlet. (normal 180s) See the figure 1.
Cell Purge	:	Set up time for carrier gas to purge between the sample inlet of Heater tube and
		titration cell. (normal 120s) See the figure 3.
Samplar Durga		Set up time for corrier and to purge inside the complex (normal 180s). Set up on
Sampler Furge	·	Set up time for carrier gas to purge inside the sampler. (normal 180s) Set up on
		Pre-treat 1 only. See the figure 2.

Press [titration] to exit from the setup.

## **Pre Treat 1** (using eggplant type sampler):

Prepare sample in the eggplant sampler and set it to the inlet. Purge through Back purge (Fig.1), Sampler purge (Fig.2) and Cell purge (Fig.3), and then, when drift reaches stable level, start measurement with carrier gas flowing through.

## Pre Treat 2 (direct injection to the inlet):

Purge through Back purge (Fig.1) and Cell purge (Fig.3), and when drift reaches stable level, start measurement with carrier gas flowing through.

## **Pre Treat 3** (using sample boat):

Dry the sample boat first, and then, transfer the weighed sample in boat into Heater tube. Purge through Back purge (Fig.1) and Cell purge (Fig.3), and then, when drift reaches stable level, start measurement with carrier gas flowing on.



Fig.1 (Back purge)

Fig.2 (Sampler purge)

Fig.3 (Cell purge)

Note : The eggplant type sampler is replaced with a ball plug for Pre-treat 2 or 3.

#### 8-1-4. Result parameter

Parameters on how to process measurement results are set up here including concentration calculation of data, formatting report on data and visualize data list in graphic chart.

Screen display after [Result] key is pressed:



Select item by cursor using  $[\uparrow]$ ,  $[\downarrow]$  key, and open each setup message display by  $[\downarrow]$  key.

#### 1. Calculation parameter

Set up parameters for concentration

< < Calculation >> [METHOD 1] ▼
Calc. No. (1 ~ 6) [2]
Unit :ppm %
Weight :Flxed <u>Variable</u>

Enter each parameter on **Calculation>** first display. Select item by cursor using  $[\uparrow], [\downarrow], [\leftarrow], [\rightarrow]$  key and enter desired amount and conditions, and then, confirm by  $[\downarrow]$ key.

Calc. No. (1 to 6) : Select equation for computation. Commonly used formula for moisture is Eq.1, and for concentration is Eq.2. For details, see "Calculation Formula".

Unit
 Select unit for calculation results. For Calc.No.2, 3, 4, 5, 6, select ppm or %, and for Calc.No.1, μ g or mg.

Weight : Select how to enter weight of sample. "Fixed" is concentration calculation by constant sample weight. "Variable" is calculation by individual sample weight at each time. This setting is for Calc.No.2, 3, 6 only, and not available for Calc.No.1, 4, 5. The weight is entered by [Sample] key.

Screen display after [ Page down] is pressed: ( $[\downarrow]$ , [ $\downarrow$ ] key works when the cursor stays at the bottom line)

Enter parameters on **<Calculation>** second display. Select item by cursor using  $[\uparrow], [\downarrow], [\leftarrow], [\rightarrow]$  key, and enter numerals and conditions, and confirm by  $[\downarrow]$  key.

Drift Comp.	: Off Manu. Auto
	Select how to correct drift level.
Off	: Drift is not corrected.
	This parameter is selected when total moisture amount including drift is the object.
Manu.	: Drift level is corrected manually; for Instance when mean value is going to be used to compensate a large difference of drift at start and at the end.
Auto	: Drift level is corrected automatically by the drift level at time when [Start] key is pressed. This is commonly practiced way of compensation.
Drift	: This is manually entered value for drift correction. Select Manu. on Drift Comp, and enter the value.

#### Calculation formulas

Calc. No.	Purpose	Equation	
1	for water content unit: µg, mg	$F \times Moisture (\mu g)$	
2	Concentration when liquid or solid sample is weighed unit: ppm, %	$F \times \frac{Moisture}{Wt1 - Wt2}$ (ppm)	
3	for concentration when liquid or solid sample is dehydrated in extracting solvent and out of which part of water is weighed unit: ppm, %	$F \times (\frac{\text{Moisture }}{\text{Wt1-Wt2}} \times \frac{\text{B+Wt0}}{\text{Wt0}} - \frac{\text{A} \times \text{B}}{\text{Wt0}}) \text{ (ppm)}$	
4	for concentration by measuring volume of liquid sample unit: ppm, %	$F \times \frac{\text{Moisture}}{V1 \times \text{Dens}}$ (ppm)	
5	for concentration by measuring cubic of gaseous sample unit: ppm, %	$F \times \frac{(\text{Moisture}) \times 22.4}{V2 \times 18} \times (1 + \frac{\text{Temp.}}{273}) \text{ (ppm)}$	
6	for concentration of water from solid sample dehydrated in extracting solvent out of which part of water is weighed and measured. (Sample is not soluble in solution) unit: ppm, %	$X = \frac{\text{Moisture}}{\text{Wt1-Wt2}} \times (\frac{\text{B}}{\text{Wt0}} + \frac{\text{X}}{10^6}) - \frac{\text{A} \times \text{B}}{\text{Wt0}}$ Water content is obtained from X multiplied by F×X	

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

Moisture (μg) : Net water amount 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 Function6)

Data (µg) : Total water content which is the amount of entire water that has been titrated (electrolysis) in the titration cell.

Drift ( $\mu$ g/s)	: Drift
	The moisture which enters into titration cell from ambient air or carrier gas.
t (s)	: Measuring time
	The time counted from start of titration with sample injected down to termination of titration.
	Define " $t(wait) + t(max)$ " to end titration within desired time.
Blank (µg)	: Blank value
	The amount of water that has entered into the cell from other source than the sample and
	has to be deducted from the titration.
Wtl (g)	: Total weight of a sample and the syringe before the sample is injected.
Wt2 (g)	: Total weight of a sample and the syringe after the sample is injected. Therefore, net
	weight of sample injected is Wt1-Wt2.
Wt0 (g)	: Sample weight into extracting solvent
	In the indirect method where sample is dipped in the extracting solvent and part of the
	sample is measured. This is the weight of the sample in the solvent.
B (g)	: Extracting solvent
	The amount of solvent that has been used in indirect method.
A (ppm)	: Water concentration of solvent
	Concentration of water in the extracting solution used for dehydrating the sample in indirect method.
VI (mL)	: Sample size
	The amount of sample measured in volume that has been injected.
Dens (g/mL)	: Density of sample
	The density of sample measured in volume that has been injected.
V2 (L)	: Gas sample
	Gas sample measured in volume.
Temp (°C)	: Gas temperature
	Temperature of sample gas at time of measurement.

#### 2. Report parameter

When optional printer is connected, parameters for printing the results of titration and calculation are selected here:



Enter each parameter on **<Report>** first display. Select desired print format by cursor using  $[\leftarrow]$ ,  $[\rightarrow]$  key, and confirm by  $[\downarrow]$  key. Below is the description of each print parameter:

1) Short (short form):

When short form is selected for printing, the following items will be printed out:

Sample No. Date & Time Titr. Cell No. Size Result



## Note:

- 1. The content of Size differs according to the selected equation number.
- 2. "-Stop by Reset-" will be printed out only when titration is aborted by [Reset] key while t(max) is defined on Titration parameter and Drift Stop is set to Off.

#### 2) GLP (GLP Form):

When GLP form is selected, the following items are printed to conform to GLP requirements:

Sample No. Date & Time Lot No. Titr. Cell No. Method Calc. No. Lot No. Size Result Drift Blank Reagent capa. Titration time Factor Operator Name

*** Result ***					
Sample No. 01-01		$\leftarrow$ Sample number			
Date 10/1	12/1998 14:52		$\leftarrow$ Date and time		
Method		1	$\leftarrow$ Method number		
Titr. Cell N	0.	1	← Cell channel		
Calc. No.2			$\leftarrow$ Calculation formula No.		
Lot No.	NBA968012		$\leftarrow$ Lot number		
Wt1	42.5384	g	$\leftarrow$ Syringe + sample weight	)	
Wt2	42.0213	g	$\leftarrow$ Weight of syringe	> Size	
Net	0.5171	g	$\leftarrow$ Sample weight	J	
Factor	1.00		← Factor		
Drift	0.01	ug/s	← Drift level		
Blank	0.0	ug	← Blank level		
Result	1734.8	μg	$\leftarrow$ Amount of water	٦	
	3354.9	ppm	< Amount of water	Calculation Result	
A. Capa.	30	mg	$\leftarrow$ Concentration of weight		
C. capa	30	mg	← Reagent life		
Titr.Time	00:01:48				
Stop by	Deet		$\leftarrow$ Time for titration		
- Stop by Rest -		( When tituation is about a d	[D a sat] [.a		
Name: KEMTARO		$\leftarrow$ when illration is aborted b	by [Reset] key		
		$\leftarrow$ Operator's name			
			· · · · · · · · · · · · · · · · · · ·		

## Note:

- 1. The content of Size differs according to the selected equation number.
- 2. "-Stop by Reset-" will be printed out only when titration is aborted by [Reset] key while t(max) is defined on Titration parameter and Drift Stop is set to Off.

#### 3) Variable:

Enter parameters after " $\mathbf{\nabla}$ " sign turns on display when the cursor points Variable.

When Variable is selected, reporting items can be changed and only selected items are printed out.

Point Variable by cursor and press [page down]. ( $[\downarrow]$  or [ $\downarrow$ ] key also can be used )

<< Report >>	[METHOD 1] ▲▼
Sample No. Date & Time Lot No. Method Calc. No.	: Off On : Off <u>On</u> : <u>Off</u> On : <u>Off</u> On : <u>Off</u> On

Enter parameters on <Report> second display.

Select item by cursor using  $[\leftarrow]$ ,  $[\rightarrow]$  key and confirm by  $[ \downarrow ]$  key. Press on to print out.

Press [Page Down]. ( $[\downarrow]$ ,  $[\downarrow]$  key will work when the cursor stays at the bottom line on display.)

<< Report >>	[METHOD 1] ▲▼
Size	: Off On
Result	: Off <u>On</u>
Drift	: Off <u>On</u>
Blank	: <u>Off</u> On
Reagent Capa.	: <u>Off</u> On

Enter parameters on <Report> third display.

Select item by cursor using  $[\leftarrow]$ ,  $[\rightarrow]$  key and confirm by  $[ \downarrow]$  key.

Press [Page Down]. ( $[\downarrow]$ ,  $[\downarrow]$  key will work when the cursor stays at the bottom line on display.)

<< Report >>	[METHOD 1] ▲
Titration Time	: Off On
Factor	: <u>Off</u> On
Operator Name	: <u>Off</u> On

To exit, press [Result] key.

#### 3. Titr. Curve parameter

The titration graph is made by water per unit time and accumulated titration volume, and it will be displayed and printed out. From this titration curve, the time when 100% water is titrated can be known. This is most used in distillation method.

```
<< Titr. Curve >> [METHOD 1]
Sampling Interval [ 30]s
Display : Off
Print Meas. Data : Off
Print Graph. : Off
Form 1 Form 2
```

Enter parameters on <Data List> display.

Select item by cursor using  $[\uparrow], [\downarrow], [\leftarrow], [\rightarrow]$  key and enter numerals conditions, and confirm by  $[\downarrow]$  key.

Sampling Interval	:	Select time interval for sampling data including titration volume or accumulated volume.
Display	:	Off Form1 Form2 Select display of titration(electrolysis) per set time and accumulated amount of titrated water after measurement is over.
Off	:	This means it will not display nor print the graph.
Form1	•	The accumulated titrated water is printed in graphic form. The axis of accumulated amount is shown in percent, and the evaporation rate against 100% water at EP is shown on display or printed out.
Form2	:	Titration volume (electrolysis) per set time and accumulated amount are shown in graph on display or printed out.
Print Meas. Data	:	Select to print or not the sample data at time interval. The sampled data is printed out at real time.
Print Graph.	:	Off, Form1, Form2 Select to print or not graphic chart or the titrated volume (electrolysis) and accumulated water of titration.

## Note:

Graphic chart can be printed out when IDP- or DP- Printer is connected. (IDP- or DP- must be selected on Setup parameter for Print)



## 8-1-5. Sample parameter

Enter parameters for the sample including Sample No., Sample weight and Sample file (see Function 4). Settings on Sample parameter depend on Result parameter (0) and calculation parameter (Calc. No.).

Press [Sample] key.



- When Size Only of Function 4 is "On", it is not displayed.

Select ID and operator's name by  $[\leftarrow], [\rightarrow]$  key and confirm by  $[\downarrow]$  key. Operator's name can be registered on "2.Regist Operator".

When Calculation is Calc. No.1:

	< SAMPLE >	
Sample Lot No. Blank	[01] - [01] [ ] [ 0.0] μg	

Enter each parameter on **<Sample>** display.

Select item by cursor using  $[\uparrow], [\downarrow], [\leftarrow], [\rightarrow]$  key and enter numerals and conditions, and confirm by  $[ \downarrow]$  key.

Sample No.	:	The sample is numbered here.
		The numbers are divided by higher order number for grouping (up to 100[00]) and
		low order number for individual numbering (up to 100[00]). Low order number is
		incremented, and High order number, if changed, will not be included in statistics.
Lot No.	:	Enter Lot number by numerals and characters using numeric key, [ $\cdot$ ] and [ -/Disp ]
		key.

Blank : This is the amount of water to be subtracted from measurement results. For example, when a sample is injected with inlet port opened, certain amount of Blank which is determined in advance has to be deducted from the measurement results.

The above entry will finish the setup for Sample on Calc.No.1.

For Calc.No.2 to 6, enter Wt1, Wt2, Wt0, B, A, V1, Dens, V2, Temp. parameter that appear under the lot number on display.

To exit from setup, press [Sample] key.

# 8-2. Special keys

Other than keys for parameter settings, titration control, numerals and the cursor, etc., MKC-520 has the following three special keys:

[ Stirrer ] key	:	Controls stirring speed
[ Print ] key	:	Prints out parameters and data
[ -/Disp. ] key	:	Switches display of titration volume to titration potential
		Reads weight when Balance is connected.
		Displays alphabets on Lot No. entry.

## 8-2-1. [Stirrer] key

Screen display when [Stirrer] key is pressed on Main:

Cell-1 No. 01-01 Drift (µg/s)	[METHOD 1]		
Speed (0~9)	[4]		
Press	Start	key	

Enter desired number of speed by numeric key on the second line of the above display. (normal 4 to 6, 0:stop) To exit from setup, press [Stirrer] key again.

## 8-2-2. [Print] key

Each parameter can be printed out by the following key entry:

Key entry	Printout
[ Method ],[ Print ]	All parameters on preset Method
[ Titration ],[ Print ]	Titration parameters on preset Method
[ Result ],[ Print ]	Parameters of Calculation, Report and Data List on preset Method
[ Result ],[ 0 ],[ ↓ ],[ Print ]	Calculation parameters on preset Method
[ Result ],[ 1 ],[ ↓ ],[ Print ]	Report parameters on preset Method
[ Result ],[ 2 ],[ ⊣ ],[ Print ]	Data List parameters on preset Method
[ Sample ],[ Print ]	Sample parameters on preset Method
[ Setup ],[ Print ]	Setup parameter
[ Function ],[ Print ]	Function parameter

# Note:

When Sample File is "On", all parameters set in Sample file. (prints for the number of samples on first display, and for the individual sample on second display)

It will not print out Sample parameter only.

## 8-2-3. [ -/Disp. ] key

Display of titration volume or potential in mV can be switched by this key during titration. The weight of sample from Balance can be read by this key when Balance is connected.

# Note: Do not press [-/Disp.] key when Balance is not connected.

## 8-3. Inhibited key entry

Following key entries are inhibited during titration:

Alarm will be activated by beep if any of the following keys is pressed to attempt parameter change:

[ Titration ] [ Function ] [ Setup ] [ Print ] (except [ Result ] and [ Sample ])

[Result] key will work but cannot change 0.Calculation.

Therefore, only [ Reset ], [ Sample ], [ -/Disp. ], or [ Stirrer ] key works during measurement.

## 8-4. Initialize to default values of Method and parameter

All the stored parameters can be cancelled and reversed to default value. (See 10-11.Memory Clear)

# 9. Setup function

## 9-1. Introduction

The MKC-520 has the following six Setup functions to control data and user interface.

Setup 0	:	Interface setup
Setup 1	:	Calendar setup
Setup 2	:	Register Operator
Setup 3	:	Check Serial number and Ver. number
Setup 4	:	Adjust LCD brightness
Setup 5	:	Display setup

## 9-2. Setup 0 - Interface setup

The MKC-520 has three user interface including RS-232C, Printer and Balance. Protocol to match such peripherals electronically can be set up as follows:

## 9-2-1. RS-232C

a) About Data Capture Software (SOFT-CAP)

The SOFT-CAP (Option) is a Windows® 95-compatible software which allows MKC-520 to be linked with a personal computer through RS232C port. This enables you to download the measurement results on MKC-520 to the Microsoft® Excel workbooks or store them in CSV format. In addition, this software allows you to control the operations on MKC-520 (start-up of measurement, resetting and the others) from a PC.

#### 1. Receiving Features

"Data Capture Software" allows arbitrarily-selected measured results to :

- 1) be transmitted directly to the Microsoft® Excel workbooks.
- 2) be read on lotus 1-2-3® spread sheets.

#### 2. Sending Features

- 1) Start of titration.
- 2) Start of pre-titration
- 3) Reset of Measurement
- 4) Reset batch calculation.

#### Note:

# For more details, see the instruction manual for "Data Capture Software" (98-595-0086).

Transmission through RS-232C Interface

1) Introduction

MKC-520 uses RS-232C, one of approved bit serial transmission standard specifications, to exchange data with the computer.

RS-232C interface is so specified that data may be transmitted correctly between modem (modulator and demodulator) and terminal (peripheral equipment).

MKC-520 is set on the terminal mode as one such peripheral equipment. Many current personal computers today are equipped with RS-232C specification interface and set on the terminal mode. Data can also be exchanged simply between peripheral equipment. (Depending on computer, modem and terminal modes can be switched).

2) Signal level

Conforming to JIS C 6361.

Logic	Level
1	-3 ~ -15 [V]
0	+3 ~+15 [V]



This shows the bit pattern of transmission of character "A" (ASCII code 41H).

#### 3) Connector

The connector conforms to JIS C 6361 with 9 pins of male shape used. Pin configuration is shown below.

Pin No.	Signal name	Direction
2	RXD (Receive data)	IN
3	TXD (Send data)	OUT
4	DTR (Terminal ready)	OUT
5	SG (Signal ground)	
6	DSR (Data set ready)	IN
7	RTS (Request to send)	OUT

# Note: The digital configuration via RS-232C is described in MKC-520 Manual.

b) Setup Parameters

The MKC-520 can be controlled or its measurement results can be read by external device through RS-232C.

Press [Setup] key:



The display will change as above.

Point "0.Interface" by cursor using  $[\uparrow]$ ,  $[\downarrow]$  key and confirm by  $[\downarrow]$  key.



The display changes as above, and point RS-232C by cursor using  $[\leftarrow], [\rightarrow]$  key and confirm by  $[ \downarrow ]$  key .

	<< R	S-232	C > >	
Baud Rate:	300	600	1200	
	2400	4800	9600	
Parity :	Even	Odd	<u>None</u>	
Stop Bits :	<u>1</u> 2			
Data Bits :	7 <u>8</u>			
Soft H.S. :	<u>Off</u> O	n		

Then, the display will change as above, and point the required digital setup by cursor using  $[\uparrow], [\downarrow], [\leftarrow], [\rightarrow]$  key and confirm by  $[ \downarrow]$  key.

To close the setup, press [Setup] key.

# <u>Note:</u> The digital configuration via RS-232C is described in MKC-520 Manual.

#### 9-2-2. Printer

The MKC-520 offers optional printer to print out measurement results and parameter settings.

Press [Setup] key:



The display changes as above, and point 0. Interface by cursor using  $[\uparrow]$ ,  $[\downarrow]$  key and confirm by  $[\downarrow]$  key

<< Interface >> ▼ RS-232C Printer Balance			
RS-232C Printer Balance	<< Interface >>	▼	
	RS-232C Printer Balance		

The display changes as above, and point Printer by cursor using  $[\leftarrow]$ ,  $[\rightarrow]$  key and confirm by  $[\downarrow]$  key.

	<< Prim	nter >>		
IDP-	DP-	GA-	Other	
101	DI	Сл	01101	

The display changes as above, and point the desired printer by cursor using  $[\leftarrow], [\rightarrow]$  key and confirm by  $[\downarrow]$  key.

Each parameter for printer stands for the following makes:

When Other printer is selected, the display will turn on "▲" sign, and proceed to the following instruction. To close setup after IDP-, DP- or GA- is selected, press [Setup] key.

- IDP- : Impact dot matrix printer IDP-100 by Kyoto Electronics
- DP- : Thermal printer DP-500 by Kyoto Electronics
- GA- : Dot printer GA-42 by Mettler-Toledo
- Other : Printer other than the above

When Other is selected, communication format becomes Variable. Point "Other" by cursor and confirm by [هـ] key.

Select item by cursor using  $[\uparrow], [\downarrow], [\leftarrow], [\rightarrow]$  key and confirm by  $[\downarrow]$  key. To exit, press [Setup] key.

<u>Note:</u> For digital configuration, refer to the users manual of your printer.

## 9-2-3. Balance

The MKC-520 can read sample weight from Balance by just pressing [-/Disp.] key. The communication protocol is already set up for leading electronic balances, and all you have to do is to select the name of your balance.

Press [Setup] key:

	< SETUP >
0.	Interface
1.	Date & Time
2.	Register Operator
3.	Serial No./Version No.
4.	LCD Contrast
5.	Display & Beep
	• •

Select "0.Interface" by cursor using  $[\uparrow]$ ,  $[\downarrow]$  key and confirm by  $[\lrcorner]$  key.

<<	nterface >	>>	▼
R S - 2 3 2 C	Printer	Balance	

Select "Balance" by cursor using  $[\leftarrow]$ ,  $[\rightarrow]$  key and confirm by  $[\downarrow]$  key.

	<< Balance >>		
KEM	Shimadzu	Sartorius	
Mettler	A & D		

Select the name of your balance by  $[\leftarrow]$ ,  $[\rightarrow]$  key and confirm by  $[\downarrow]$  key. To exit, press [Setup] key.

# 9-3. Date & Time (Setup1)

The MKC-520 has built-in clock, and date and time of measurement can be printed out. Once correct date and time is input, there will be no need to correct them unless backup battery runs down.

Press [Setup] key:

<< SETUP >>	
0. Interface	
1. Date & Time	
<ol><li>Register Operator</li></ol>	
3. Serial No./Version	Νo.
4. LCD Contrast	
5. Display & Beep	

Select "Data & Time" by cursor using  $[\uparrow]$ ,  $[\downarrow]$  key and confirm by  $[\downarrow]$  key.

<< Date & 1	∏ime >>	
11/24/1998	17:01	
Month: [ <b>1</b> 1] Day : [24] Year : [1998]	Hour Minute	: [17] : [01]

Select the item by cursor using  $[\uparrow]$ ,  $[\downarrow]$  key and confirm by  $[\downarrow]$  key. To exit, press [Setup] key.

## 9-4. Register Operator (Setup 2)

The operator's name can be registered on MKC-520. It can also be printed out together with measurement results.

Press [Setup] key:

```
< SETUP >
0. Interface
1. Date & Time
2. Regist Operator
3. Serial No./Version No.
4. LCD Contrast
5. Display & Beep
```

Select "2. Regist Operator" by cursor using  $[\uparrow]$ ,  $[\downarrow]$  key and confirm by  $[\downarrow]$  key.

```
<< Regist Operator >>
ID (0 ~ 9) [*]
Operator [ ]
```

Enter ID of the operator. 10 operators are numbered by 0 to 9 ID, and the sample operation is controlled by ID. When the cursor comes to the top of Operator, press [-/Disp] key to show the next display, where operator's name can be registered.

<< N	ame >>		
t I		]	
ABCDEFGHI JKLMNOPQR STUVWXYZ	abcdefghl jklmnopqr stuvwxyz	12345 67890 End	

Select desired character by cursor using  $[\uparrow], [\downarrow], [\leftarrow], [\rightarrow]$  key and confirm by  $[\downarrow]$  key. Point a character one after another by blinking cursor and input as above. One character already input can be erased by [Bs] key. All characters can be erased by [Clr.] key. When all the desired characters are input, point "End" by cursor, and confirm by  $[\downarrow]$  key. The display will return to <Setup>. When [Setup] key is pressed, the display will return to Main display.

## 9-5. Checking Serial number and Version number (Setup 3)

Your MKC-520 has individual number and version number registered in planet before shipment to identify the product by its options and specification. If you want to know the information, follow the below steps:

Press [Setup] key:



Select "3. Serial No." by cursor using  $[\uparrow]$ ,  $[\downarrow]$  key and confirm by  $[\downarrow]$  key.

<< Serial No. / Version No. >> Serial No. : NLAxxxxx Version No. : x. xx

The serial number and version number appear on display. To exit, press [Setup] key.

## Note:

Should a product need repairing due to a failure, please make sure to let us know the Serial number and the Software version number for the product.

# 9-6. Adjusting brightness of LCD (Setup 4)

The LC display can be adjusted to the desired brightness.

Press [Setup] key:



Select "4. LCD Contrast" by cursor using  $[\uparrow]$ ,  $[\downarrow]$  key and confirm by  $[\lrcorner]$  key.



Adjust the brightness by moving the cursor  $[\leftarrow], [\rightarrow]$  key. To exit, press [Setup] key.

# 9-7. Display setup (setup 5)

Result time and language used can be changed.

Press [Setup], [5], []] key one after another. The display will change to the below.

[First display] (\*This display shows languages, English, Japanese and Mandarin.)

< <	Display & Beep >>	•
Result Time	[10]s	
Sample No.	:Off <u>On</u>	
Language	:English <u>Japanese</u>	
	Mandarin	

Select the item by  $[\uparrow], [\downarrow], [\leftarrow], [\rightarrow]$  key and enter desired setting and confirm by  $[\downarrow]$  key. Press either [Page down] or [Page up] to change displays below.

[Second display]

	<< Display & Beep >>	
Веер	:Off <u>On</u> (1 ~ 4) : [1]	

Result Tim	:	How long the measurement results stay on display can be adjusted to your need.
		Select desired time length in second. After display of the results is selected, the
		results will return to Main display. If "0"s is selected, the results will stay on display
		until [↓] key is pressed.

Sample No. : Select whether you like Sample No. displayed at the upper left corner of the screen.

Language	:	Language used for message on display.
		A language selection is subject to a version of softwares JC and K.
		JC version [English/Japanese/Mandarin]
		K version[English/Korean]

Beep	:	Select electronic beep or not.
(1-4)		Choose rhythm of beep sound.
		Effective only when "Beep:On" is selected

To close the setup, press [Setup] key.

# 10. Special function

## 10-1. Introduction

The MKC-520 has the following ten special functions:

Function 0	:	Reagent
Function 1	:	Recalculation
Function 2	:	Auto statistics
Function 3	:	Data deletion
Function 4	:	Sample file
Function 5	:	Oven/Purge
Function 6	:	Factor setup
Function 7	:	Calibration
Function 8	:	Periodic check
Function 9	:	Memory clear

## 10-2. Reagent (Function 0)

The Karl Fischer reagent for coulometric titration has reagent life for measurement of water content. Generally, 1000mg  $H_2O$  for 100mL analyte and 300mg $H_2O$  for 5mL catholyte are the approximate limit of capacity. (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-520 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 reading to zero when reagents are replaced.

Alarms when it reaches the limit. (Beep alarm can be switched by on/off)

Further, when the optional unit such as Stirrer unit E or Stirrer unit EP is used, the recordings of the reagent capacity, alarm sound and the history of reagent replacement will be possible for both Channel 1 and Channel 2.
Press [Function] key:

<	FUNCTION >
<ol> <li>Reagent</li> <li>Recalculation</li> <li>Auto Statis.</li> <li>Data Deletion</li> <li>Sample</li> </ol>	5. Oven/Purge 6. Factor 7. Calibration 8. Periodic Ck. 9. Memory Clear

Point "0. Reagent Capa." by cursor using  $[\uparrow], [\downarrow], [\leftarrow], [\rightarrow]$  key and enter numerals and conditions, and confirm by  $[ \downarrow ]$  key.

<< Reage	ent >>	
Titr. Cell No.	:12	
0. Reagent Capacity 1. History (List) 2. History (Detail)		

Select titration cell channel by  $[\leftarrow]$ ,  $[\rightarrow]$  key. Always select 1 if optional stirrer unit E/EP is not connected. Select the item by  $[\uparrow]$ ,  $[\downarrow]$  key and confirm by  $[\downarrow]$  key.

- Reagent Capacity : Set up the amount for alarm and reset electrolytic current accumulation by zero entry when the liquid is exchanged.
- History (List) : Shows a list of record of reagent exchange.
- History (Detail) : Shows a list of detailed record of reagent exchange.

Select "0. Reagent Capacity" and confirm by [↓] key.

<< Reagent	Capacity (Cell-1)>>	7
[Anode]		
Replaced	: 09/11/1998	
Capacity	[ 10] mg	
Alarm Set	: Off <u>On</u>	
Alarm	[ 1000] mg	
Reagent Life	[ 99] day	

Select item on [Anode] by cursor using  $[\uparrow], [\downarrow], [\leftarrow], [\rightarrow]$  key and enter numerals and conditions, and confirm by  $[\downarrow]$  key.

Replaced	: The date when anolyte was replaced. The date when "0" is input on "Capacity" appears here.
Capacity	: Accumulated electrolytic current. Enter "0" when anolyte is replaced.
Alarm Set	: Select on/off beep alarm when Capacity reaches the limit.
Alarm	: Enter anolyte capacity limit (mgH2O). It turns on only when Alarm Set is On.
Reagent Life	: Set up a number of days for replacement. It turns on only when Alarm Set is On. Alarm activates when the number of days elapses since "0" is input on "Capacity".



Reagent Name : Point the cursor and press [-/Disp] key to change the display for entry of reagent name.

<< Reagent Name >>
[ ]
ABCDEFGHI abcdefghI 12345
JKLMNOPQR jkImnopqr 67890
STUVWXYZ stuvwxyz .- End

Enter anolyte name just like "Setup 2. Register Operator".

Lot No. : Point the cursor and press [-/Disp] key to change the display for entry of Lot number.

# Note:

The alarm activates by message on display and by beep after the titration is over. Press [] key to stop alarm and return to the active display.

Press [Page down] to page for [Cathode] display. ( $[\downarrow]$ ,  $[\downarrow]$  key works when the cursor stays at the bottom line on display)

<< Reagent	Capacity (Cell-1)>>	▲▼
[Cathode]		
Replaced	:_09/11/1998	
Capacity	[ 10] mg	
Alarm Set	: Off <u>On</u>	
Alarm	[ 1000] mg	
Reagent Life	[ 99] day	

Select item on [Cathode] by cursor using  $[\uparrow]$ ,  $[\downarrow]$ ,  $[\leftarrow]$ ,  $[\rightarrow]$  key and enter numerals and conditions, and confirm by  $[\neg]$  key.

Replaced	: The date when catholyte was replaced.
	The date when "0" is input on "Capacity" appears here.
Capacity	: Accumulated elecrolytic current,
	Enter "0" when catholyte is replaced.
Alarm Set	: Select on/off beep alarm when Capacity reaches the limited.
Alarm	: Enter catholyte capacity limit (mgH $_2$ O). It turns on only when Alarm Set is On.
Reagent Life	: Set up a number of days for replacement.
	It turns on only when Alarm Set is On. Alarm activates when the number of days elapses since "0" is input on "Capacity".

```
<< Reagent Capacity (Cell-1)>>
[Cathode]
Reagent Name [ ]
Lot No. [ ]
```

Reagent Name : Enter the name of reagent for electrolysis.

Lot No. : Enter the lot number of reagent.

Enter the name of reagent for electrolysis and the lot number just like anolyte.

### 10-3. Recalculation (Function 1)

You can recalculate the data when wrong Lot number or sample weight is input. Recalculated results will be displayed on the screen and can be printed by pressing [Print] key, with the header "[Recalculation]" instead of \*\*\* Result \*\*\* and (#) marking to Sample number for identification.

Press [Function] key.

<	FUNCTION >
<ol> <li>Reagent</li> <li>Recalculation</li> <li>Auto Statis.</li> <li>Data Deletion</li> <li>Sample</li> </ol>	5. Oven / Purge 6. Factor 7. Calibration 8. Periodic Ck. 9. Memory Clear

Point "1. Recalculation" by cursor using  $[\uparrow], [\downarrow], [\leftarrow], [\rightarrow]$  key and confirm by  $[\downarrow]$  key.

<< Recalcula	tion >>	▼
Sample No. Lot No. Moisture Calc. No. Unit	: 01 - [ <b>0</b> 1] [ LBN62351] : 1002.1 μg : 1 : μg	

Sample No.	: Sample number for recalculation
Lot No.	: Lot number of the sample for recalculation
Moisture	: The amount of water of measurement results for recalculation (This parameter cannot be changed)
Calc. No.	: Equation for recalculating the measurement results (This parameter cannot be changed)
Unit	: Unit for the results for recalculation (This parameter cannot be changed)

# Note: Only parameters in [ ] can be changed.

Select Calc.No.2 and press [Page down]. ([ $\downarrow$ ], [ $\downarrow$ ] key works when the cursor stays at the bottom line on display)

<< Recalculation >> ▼ Drift : 0.03 µg/s Blank [ 0.0] µg

Select the item for correction by cursor using  $[\uparrow]$ ,  $[\downarrow]$  key.

Drift : The corrected value of drift level for recalculating the results. (This parameter cannot be changed)

Blank : Blank value for the results for recalculation

### Note:

- 1. The parameters that can be changed for recalculation are only parameters in [ ].
- 2. The contents of display differ according to calculation formula.

Press [page down] key. ([,],  $[\downarrow]$  key works when the cursor stays at the bottom line on display)



The recalculated results will be shown on display. Press [Print] key to print out the results. To exit, press [Function] key.

### 10-4. Auto statistics (Function 2)

The MKC-520 can batch calculate the measurement results of samples under the same group number (High order sample No.). (It will process statistics by pressing [,...] key without changing high order number of the sample thereafter.)

Press [Function] key:

<	FUNCTION >
<ol> <li>Reagent</li> <li>Recalculation</li> <li>Auto Statis.</li> <li>Data Deletion</li> <li>Sample</li> </ol>	5. Oven / Purge 6. Factor 7. Calibration 8. Periodic Ck. 9. Memory Clear

Point "2. Auto Statis." by cursor using  $[\uparrow], [\downarrow], [\leftarrow], [\rightarrow]$  key and confirm  $[\downarrow]$  key.

< <	Auto Statistics >>	
Data Print Result Mean SD RSD	: <u>Off</u> On : 5 : 1001.2 μg : 1.2013 μg : 0.1200 %	

The results of statistical calculation are shown on display. Press [Print] to print it out. To exit, press [Function] key.

Data Print	:	Select whether print out the statistics or not.
Result	:	The number of data that have been batch calculated.
Mean	:	The average amount of the data.
SD	:	Standard deviation of the data.
RSD	:	Relative standard deviation of the data.

#### Note:

If statistics is tried without data, there goes beep alarm with message "No Data!". The display returns to FUNCTION after the error message with alarm.

# 10-5. Data deletion (Function 3)

Unnecessary data for statistics can be deleted. It will be printed out with "(\*)" mark where deletion is made.

Press [Function] key:

< F	UNCTION >
0. Reagent	5. Oven / Purge
1. Recalculation	6. Factor
2. Auto Statis.	7. Calibration
3. Data Deletion	8. Periodic Ck.
4. Sample	9. Memory clear

Select "3. Data Deletion" by cursor using  $[\uparrow], [\downarrow], [\leftarrow], [\rightarrow]$  key and confirm by  $[\downarrow]$  key.

< <	Data Deletion >>	<b>▲</b> ▼
Low Sa	mple No. [01]	
No.	Data [µg]	
* 0 0	1002.1	
0 1	1000.9	
02	1001.5	

Enter the data No. or point the data to be deleted by cursor using  $[\uparrow]$ ,  $[\downarrow]$  key and delete/recall data by [-/Disp] key.

After the data is deleted, the display will return to <FUNCTION>.

Press [Function] key to return to Main display.

# 10-6. Sample File (Function 4)

You can specify sample conditions and measuring method for a number of measurements in advance by Sample File. This function is effective especially useful when Multiple sample Changer is connected.

Press [Function] key:

< FU	NCTION >
0. Reagent	5. Oven / Purge
1. Recalculation	6. Factor
2. Auto Statis.	7. Calibration
3. Data Deletion	8. Periodic Ck.
4. Sample	9. Memory Clear

Select "4. Sample File" by cursor using  $[\uparrow], [\downarrow], [\leftarrow], [\rightarrow]$  key and confirm by  $[\lrcorner ]$  key.

< <	Sample >>
Sample File	: Off On
Size Only	: Off On
Weight ← Conc.	[ 1000]ppm
Weight	: 0.50g

Sample File

On	:	Set sample conditions and measuring method for a number of measurements
Off	:	Set sample for one of measurement
Size Only		
On	:	move cursor to wt1, set only weight (Wt1, wt2)
Off	:	set all of sample parameters
Weight $\leftarrow$ Co	nc. :	Enter estimated moisture content of a sample. Concentration unit used for current
		calculation parameters is adopted.
		Sample size obtained by calculation

Select On or Off by cursor using  $[\leftarrow]$ ,  $[\rightarrow]$  key and confirm by  $[\dashv]$  key.

### Note:

When Sample File On is selected, [METHOD 1] will be displayed in reverse.

Press [Sample] key:



Select ID and operator's name by  $[\leftarrow], [\rightarrow]$  key and confirm by  $[\downarrow]$  key. Operator's name can be registered on "2.Regist Operator".

	< SAMPLE >	▼
Meas. No. Next Meas. Method Statistics Blank	[100] [ 1] : Fixed <u>Variable</u> : Off On [ 0.0] μg	

Enter conditions for Sample File. Move cursor using  $[\uparrow], [\downarrow], [\leftarrow], [\rightarrow]$  key.

Meas. No.	:	The number of measurement to preset
Next Meas.	:	The next number after preset measurements
Method	:	Select Fixed or Variable
Statistics	:	Select ON/OFF statistics of the group of sample when high order number has been
		changed in Sample File.
Blank	:	Enter Blank value of sample.

Select Calc. No. 2 and press [Page down] ( $[\downarrow]$ ,  $[\downarrow]$  key works when the cursor stays at the bottom line on display)

	< SAMPLE >	▼
No. [1]	Method [1]	
Sample No.	[01] - [01]	
Lot No.	[ LBN62351]	
Form	: <u>Sample</u> Blank	
Wt1	[ 5.000]g	
Wt2	[ 0.0]g	

Select the item by cursor using  $[\uparrow], [\downarrow], [\leftarrow], [\rightarrow]$  key. To close the setup, press [Function] key.

No.	:	The number of preset measurement data.
Form	:	Select measurement of Sample or Blank for the sample. When Blank is selected, the
		value in previous display will be input. If the Blank is plural, the average amount will
		be taken.
Method	:	Select Method, which appears when "Variable" was selected on previous display.
Sample No.	:	The sample is numbered here.

#### Note:

- 1. Settings on sample parameter depend on calculation parameter.
- 2. While Sample File is On, [Sample] key works for Sample File.
- 3. Sample File is not to be "On" when Method No.5 is selected.
- 4. Error massage appears at titration when Cell No. of Methods is not the same.

# 10-7. Oven/Purge - Aging Evaporator (Function 5)

When Evaporator is connected, aging by purging the instrument is necessary before it is used for measurement. (see the manual of Evaporator for its details)

Press [Function] key:



Select "5. Oven/Purge" by cursor using  $[\uparrow], [\downarrow], [\leftarrow], [\rightarrow]$  key and confirm by  $[\downarrow]$  key.



Here select the time length for purging. Normal 1800s for back purge and 600s for cell purge. Aging will start when [Start] key is pressed.

Press [Function] key to return to Main display.

## 10-8. Factor setup (Function 6)

Coulometric titration by MKC-520 does not require Factor, however, measurement results can be adjusted using this function.

Press [Function] key:

< FUNCT	TION >
0. Reagent	5. Oven / Purge
1. Recalculation	6. Factor
2. Auto Statis.	7. Calibration
3. Data Deletion	8. Periodic Ck.
4. Sample	9. Memory Clear

Select "6. Factor" by cursor using  $[\uparrow], [\downarrow], [\leftarrow], [\rightarrow]$  key and confirm by  $[\lrcorner]$  key.

```
<< Factor >>
Cell-1 [ 1.00]
Cell-2 [ 1.00]
```

Enter desired numeral for correction and press  $[\Box]$  key.

Typically select 1 and enter the factor of Cell-1. When optional Stirrer unit E or Stirrer unit EP is used, enter the factor according to the cell channel. To finish setup, press [Function].

### 10-9. Calibration (Function 7)

High performance of MKC-520 can be maintained by checking the precision and accuracy of measurement using water standard.

Press [Function] key:

< FUNC	TION >
0. Reagent	5. Oven / Purge
1. Recalculation	6. Factor
2. Auto Statis.	7. Calibration
3. Data Deletion	8. Periodic Ck.
4. Sample	9. Memory Clear

Select "7. Calibration" by cursor using  $[\uparrow], [\downarrow], [\leftarrow], [\rightarrow]$  key and confirm by  $[\downarrow]$  key.

```
<< Calibration >>
Titr. Cell : 1-2Comp.
Calibration : Off On
Standard Value [ 1000]ppm
Tolerance ±[ 30]ppm
```

Point the desired item by cursor using  $[\uparrow], [\downarrow], [\leftarrow], [\rightarrow]$  key and enter numerals and conditions, and confirm by  $[\dashv]$  key.

Titr. Cell	:	Select the channel and sort of the titration cell the records of which are to be checked. Usually, select "1-2. Comp.". When One-component cell or the optional Stirrer unit E/EP is used, make the setting according to the channel of the titration cell to be checked.
Calibration	:	Select on/off check mode.
Standard Value	:	Enter the value of the water standard that you have prepared.
Tolerance	:	Comparison criterion with water of the standard.

To exit, press [Function] key.

When Check Mode On is selected, the upper right corner of display changes from [Method] to [Calib.], and changing each parameter will be restricted.

Cell-1 No. 01-0 Drift (µg/s)	1	[Calib.]	
0.02		Stable	
Press	Start	key	

After the standard is measured, the water content of the standard solution and criterion will be displayed together with statistical results.

The measurement results will be stored together with the last ten (10) measurements, which can be checked and confirmed on Function 8. The date when precision is checked will be automatically stored on Function 8, and the next check date will be shown on display.

To return to regular Method, select Check Mode: Off on Function 7.

# 10-10. Periodic check (Function 8)

The MKC-520 is designed to maintain high precision and accuracy in measurement, however, in order to confirm its performance, the instrument can be checked periodically and its history of maintenance can be reviewed by this Function.

Press [Function] key:

< FUNC	TION >
<ol> <li>Reagent</li> <li>Recalculation</li> <li>Auto Statis.</li> <li>Data Deletion</li> <li>Sample</li> </ol>	5. Oven / Purge 6. Factor 7. Calibration 8. Periodic Ck. 9. Memory Clear

Select "8. Periodic ck." by cursor using  $[\uparrow], [\downarrow], [\leftarrow], [\rightarrow]$  key and confirm by  $[\downarrow]$  key.

<< Periodic	Check >>	▼
Next Check Date	: 09/12/199	8
Titr. Cell No.	: 1	2
Alarm	: Off	On
Interval (1 ~ 12)	[1]month	
Display	: <u>List</u>	Detail
Memory Clear	: <u>No</u>	Yes

Point the desired item by cursor using  $[\uparrow], [\downarrow], [\leftarrow], [\rightarrow]$  key and enter numerals, and confirm by  $[\lrcorner]$  key.

Next Check Date	:	Shows the next check date.
Titr. Cell No.	:	Usually, select "1". When the optional unit such as Stirrer unit E or Stirrer unit EP is used, select "1" or "2" according to the titration cell to be checked.
Alarm	:	Select on/off message alarm with beep. If "On" is selected, a message appears at start-up and the end of titration.
Interval (1 to 12)	:	Enter a number of month to check.
Display	:	Select the list of history of last ten checks or the detail of individual record.

List	: Show the list of last 30 checks by date and criterion.
Detail	: Shows the detail of each check record of measurement.
Memory Clear	: Select Yes or No to erase the check. If "Yes" is selected, all records and parameters on Function 8 will be initialized.

Other page of List and Detail can be opened by [Page down] key.

Example of List display:

<< Periodic	Check	(Cell-1) >	·> ▼▲
No. Date	Std.	Mean	OK/NG
1.09/10/1998	1000	1001.3	ΟK
2. 08/10/1998	1000	1005.5	NG
3.07/10/1998	1000	1001.6	ΟK
4.06/10/1998	1000	994.5	NG
5. 05/10/1998	1000	999.9	ΟK

Example of Detail display:

<< Periodic	Check (Cell-1) >>	$\mathbf{A}$
Νο.	: 1	
Date	: 09/10/1998	
Standard Value	: 1000 ppm	
Tolerance	:± 5 ppm	
Mean (n= 5)	: 1001.3 ppm	
Result	: OK	

To exit, press [Function] key.

### 10-11. Memory clear (Function 9)

Initialize the memory space to the default when you wish to start over again or when you find instrumental error due to the stored memory. Setup function and Function 8 will not be initialized due to the nature of parameter.

Press [Function] key:

```
< FUNCTION >
0. Reagent
1. Recalculation
2. Auto Statis.
3. Data Deletion
4. Sample
FUNCTION >

5. Oven / Purge
6. Factor
7. Calibration
8. Periodic Ck.
9. Memory Clear
```

Select "9. Memory Clear" by cursor using  $[\uparrow], [\downarrow], [\leftarrow], [\rightarrow]$  key and confirm by  $[\downarrow]$  key.

```
< Memory clear >
Press return, turn OFF Power
And turn ON again.
All parameters can be
Initialized by those steps.
```

To clear the memory, press [] key, and turn OFF power at beep, and then, in about 5 seconds, turn it ON. The stored data will be cleared by this procedure.

Memory Initialized

Hit any Key

The display message confirms the memory has been cleared. Press  $[\downarrow]$  key. Electronic beep will sound and the display returns to the initial display.

# 11. Measurement by additional functions

### 11-1. Routine measurement

#### 11-1-1. Manual start by Calc. No.2

Titration starts from standby mode after pretitration is finished.

Press [Start] key

Cell-1 No. 01-01 Drift (µg/s)	[METHOD 1]
0.02	Stable
→ Sample	In
"Press	<sub>Start</sub> key"

The display shows the message "Sample In" and "Press Start key" alternately. Measure weight of the sample and inject it into the titration cell by syringe.

Press [Start] key:

Cell-1 No. 01-01 Drift (μg/s)	[METHOD 1] Moisture (µg)	
10.5	921.5	
Measurement		

Titration starts with "Measurement" message, showing drift level and titrating moisture at real time. Weigh the empty syringe after the sample is released.

Cell-1 No. 01-01	[METHOD 1]
	Wt1 [ 40.4965]g Wt2 [ 39.6232]g
Input	Weight

When the titration is finished, the display changes as above with beep. Then, input the weight before the sample was injected for Wt1, and weight after the sample has been released for Wt2.

Cell-1 No. 01-01 Drift (µg/s)	[METHOD 1] Concentration (ppm)
0.03	1001.3
Result	

The display message changes as above after the weights are input, and if Printer is connected, it will print out the measurement results.

The display will return to Main by [  $\dashv$  ] key.

# Note:

- 1. Sample weight can be input by Sample key during or before titration starts.
- 2. It should be noted when Calc. No. 3 to 6 is selected, no further message of requesting weight input other than for Wt1 and Wt2 will appear on display.

### 11-1-2. t(stir) setup

When t(stir) is selected and [Start] key is pressed, the display will show the stirring time being counted before it goes to titrating mode.

Cell-1 No. 01-01	[METHOD 1]
	(s)
Stir. Time	12
Please	wait

#### 11-2. When Balance is connected

The MKC-520 can read information on sample weight from Balance by [-/Disp.] key and calculate concentration.

Connect the cable from Balance to the port for balance on rear panel of MKC-520. (see 9-2-3.) Set up protocol to Manufacturer's instruction on digital configuration of the balance.

Press [Sample] key:

< SAMPLE >		
Sample No.	[ <b>0</b> 1]-[01]	
Lot No.	[ ]	
Blank	[ 0.0]µg	
Wt 1	[ 5.000]g	
Wt 2	[ 0.0]g	

Enter Sample No. , Lot No. and Blank, and point Wt1 by cursor.

Weigh the syringe with sample in it.

After the reading of weight becomes stable, press [-/Disp.] key to read the weight from balance, and confirm by  $[\Box]$  key. Return the display to Main by [Sample] key. (or you can leave it)

Press [Start] key:



The display will show the message "Sample In" and "Press Start key" alternately. After the sample is injected, weigh the syringe.

Press [Start] key.

Cell-1 No. 01-01 Drift [µg/s]	[METHOD 1] Moisture (µg)
10.5	921.5
Measurement	

Titration will start and the display shows the drift and moisture content during titration.

Cell-1 No. 01-01	[METHOD 1]
	Wt 1 [ 40.4965] Wt 2 [ 39.6232]
Input	Weight

When the titration is finished, the display will change with beep. Point Wt2 with cursor and press [-/Disp.] key to read the weight of syringe after sampled, and confirm by [-] key.

Cell-1 No. 01-01 Drift (µg/s)	[METHOD 1] Concentration (ppm)
0.03	1001.3
Result	

The display will change showing the measurement results. It will print out the results if Printer is connected. The display will return to Main when  $[\downarrow]$  key is pressed.

#### Note:

- 1. Wt2 can be input by pointing by cursor even while titrating.
- 2. It should be noted when Calc. No. 3 to 6 is selected, other items than Wt2 can only be input by [Sample] key.
- 3. It reads absolute weight even if Wt1-Wt2<0.

### 11-3. When Evaporator is connected

The MKC-520 can measure moisture which exists in solid sample or a sample hard to dissolve in anolyte by vaporizing moisture of the sample.

Explanation on how to start Pretreat 1 manually:

Prepare sample in the eggplant type sampler and after its weight is measured, attach it to the heater tube, and press [Start] key.



The display changes as above with message "Oven Start", and then, quickly will turn to the below message "Back Purge 180", "Oven Purge" to start back-purge.



It starts counting down the time when back-purge starts.



When back-purge is finished, the message will ask you to set sampler in position and "Press Start" alternately, and press stopper hole.

Cell-1 No. 01-01	[METHOD 1]
	(S)
Samp. Purg	e 178
Please V	Vait

The display changes with message "Sampler Purge Time 180", "Oven Purge".

Cell- Drift	1 Νο. (μg/s	0 1 - 0 1 )	[METHOD 1]	
0.0	3	Oven	Start	
	<u> </u>	Sampler	Open	
		"Press	key"	

After sampler is purged, the display changes as above with message "Sampler Open", "Press Start" alternately, and press [Start] key after sliding sampler position off the ball stopper hole.



The display changes as above with message "Cell Purge 180", "Oven Purge", and cell purge time is counted down.



After the cell is purged, the display will turn to standby mode "Ready" as the above, and wait until it sets in "Stable" status of drift.

Cell-1 Drift (,	No. 01-01 μg/s)	[METHOD 1]
0.03		Stable
Γ	→Drift	OK
	"Press	s <sub>Start</sub> key"

When the drift sets in stable status, the display changes as above with alternating message "Drift OK" and "Press Start", and then, press [Start] key.



When the drift becomes stable with message from "Ready" to "Stable", the display changes as above. Turn the sampler and place the sample on the boat, and press [Start] key.

Cell-1 N Drift [µ	o. 01-01 g/s]	[METHOD 1]
0.03	Boat	t Mov'n
	Please	Wait

The display changes as above with message "Boat Moving".

The sample boat moves to the heating unit.

Cell-1 No. 01-01 Drift (μg/s)	[METHOD 1]
0.03	Stable
Press	Start key

After the boat has moved in, the display will change as above with message "Press Start". Then, press [Start] key.

Cell-1 No. 01-01 Drift [µg/s]	[METHOD 1] Moisture (µg)
10.5	921.5
Measurement	

When [Start] key is pressed, the display will change to the above with message "Measurement".

Cell-1 No. 01-01	[METHOD 1]
	Wt 1 [ 40.4965]g Wt 2 [ 39.6232]g
Input	Weight

When titration is finished, the display changes to the above with message "Input Weight". When sample boat returns to the inlet, remove the sampler and measure its weight, and then, enter Wt1 and Wt2.

Cell-1 Νο. 01-01 Drift (μg/s)	[METHOD 1] Concentration (ppm)
0.03	1001.3
Result	

The display changes as above with message "Results" showing the results.

(It will print out the results if Printer is connected)

Press [  $\downarrow$  ] key to return the display to standby mode.

### 11-4. Bromine number and index

The degree of unsaturated hydrogen carbide in oil and petroleum products is indicated by bromine number or index. The volumetric test method is specified in ASTM D2710, JIS K2605 and K2435. The coulometric method with this unit is easier than volumetric, however, it is recommended to refer to the volumetric testing procedure and specifications.

#### < Bromine number and index >

Bromine number	:	The amount of bromine (unit: g/100) consumed in 100g sample
Bromine index	:	The amount of bromine (unit: mg/100) consumed in 100g sample

#### < Principle of measurement >

The unsaturated hydrogen carbon reacts with bromine as follows:	
$R-CH = CH-R^{-} + Br_2 \rightarrow R-CHBr-CHBr-R^{-} $	1)

In coulometric titration, bromine is generated by electrolysis of anolye containing bromine ion:	
$2Br^{-} \rightarrow Br_2 + 2e^{-}$	2)

When generated bromine is consumed according to Eq. (1), the electrode detects bromine consumption, and continues generating bromine according to Eq. (2).

Such bromine is generated in proportion to the electricity determined by Faraday's Law. From Eq.(1), Bromine reacts with coupled C=C evenly (1:1). Thus, one mol of bromine (159.8g) is equivalent to  $2 \times$  96500 coulomb, which means 1.2 coulomb/1mgBr<sub>2</sub>.

Based on the above principle, the electricity consumed in electrolysis is converted to the exiting bromine.

#### Note:

Follow the below instructions in order to obtain correct measurement results:

- 1. Replace anode and cathode reagent with new one respectively each day.
- 2. When the anolyte turns to white turbidity, change it with new one.
- 3. When the same anolyte and catholyte are continuously used, the measurement results may produce larger amount in value than expected. Change the anolyte in this case.
- 4. Do not share the titration cell with moisture titration.

#### 11-4-1. Preparation of reagent

Use the following reagent for measurement of bromine number or index:

Anode reagent	:	Mixture of Acetic acid (high grade) 600mL; Methanol (high grade) 260mL;	
		1M-Potassium bromide solution 140mL. Blend it well and use 100mL each time.	
Cathode reagent	:	0.2M-Potassium chloride solution. Use 5mL each time.	
Check solution	:	0.05Wt-Cyclohexene toluene mixture (Aprox. 93 ~ $102$ mgBr <sub>2</sub> /100g). Theoretical	
		value is calculated by the below formula:	

Theoretical value (mg Br<sub>2</sub>/100g) =  $\frac{159.83(\text{bromine molecular mass}) \times \text{cyclohexene}(g)}{82.15(\text{cyclohexene molecular mass})} \times \frac{100}{\text{toluene}(g) + \text{cyclohexene}(g)} \times 1000$ 

#### 11-4-2. Setting Cell Number

Press [ Method ] key:

	< METHOD >	
	Protect	Cell No.
METHOD 1:	<u>Off</u> On	1 - 1 C o m p .
METHOD 2:	<u>Off</u> On	1 - 2 C o m p .
METHOD 3:	<u>Off</u> On	1 - 2 C o m p .
METHOD 4:	<u>Off</u> On	1 - 2 C o m p .
METHOD 5:	Off On	1 - 2 C o m p .

The display changes as above and select Method 5 by  $[\uparrow]$ ,  $[\downarrow]$  key.

When additional stirrer unit is used, select Cell number according to the channel of measuring cell to be used.

Cell No.	Cell ch.	Titr. cell	Option
1–2Comp.	1ch	2 components	
2–2Comp.	2ch	2 components	Stirrer unit E or EP and Titration
			cell unit

# 11-4-3. Titration parameter

Parameters to control titration are selected here including how to detect endpoint or set up drift criteria. Message display after [ Titration ] key is pressed:

< TITRATION	> [METHOD	5]	▼
t (stir) t (wait) t (max)	0]s 15]s 0]s		
Drift Stop	Off <u>Rel</u> Abs		
Rel	0.1]µg/s		

Select item by  $[\uparrow], [\downarrow], [\leftarrow], [\rightarrow]$  key and enter desired numbers or conditions by  $[ \downarrow]$  key.

t(stir)	: Where the sample you going to measure is a type of sample which takes time in dissolving in the anolyte and dehydrated, you can set up waiting time (=stir only) until titration (electrolysis) starts after the sample is injected. For example, this is useful for a sample which is hard to dissolve in anolyte and takes time to react with bromine.
t(wait)	: Where the sample has two inflection points and may reach endpoint by drift, the shortest possible time for titration termination is necessary to set up. Titration will not terminate during the wait time.
t(max)	<ul> <li>The longest time for titration is limited by this setup. Thus, total titration time is: t(stir)+t(wait)+t(max)</li> <li>Printed measurement time is t(wait)+t(max) and dose not include t(stir) t(max)=0 means titration will not terminate by time limit, for instance, where Evaporator is connected or minute moisture content continues to be extracted in the anolyte or titration does not end by drift.</li> </ul>
Drift Stop	: Off Rel Abs You can define a drift stop for reaching endpoint.
Off	: Endpoint will not be detected by drift stop. It is the time total of t(stir)+t(wait)+t(max), for example, where endpoint has to be determined when most of the moisture content has been titrated while the sample releases small amount of moisture slowly in evaporation or when you want to know extracted moisture from a sample by unit time.

- Rel : This is commonly used direct method of EP detection by drift stop, where endpoint is determined when the drift during titration reduces below [Drift at start]+[preset value].
- Abs : This is another EP detection by drift stop, which determines endpoint when the drift during titration reduces below [preset value] regardless of drift at start. Here, t(max) must be defined. Otherwise, it will not terminate titration if the drift goes up higher than the preset values.

Notes : Criteria for drift stop must be defined when Rel or Abs is selected.

Message display when [ Page down] key is pressed. ([ $\downarrow$ ] or [ $\dashv$ ] key can be used if the cursor stays at the bottom line):

< TITRATION >	[METHOD 5] ▲
Control gain	[■ 5.0]
Stable	[ 0.5]∆µg/min
Start	: <u>Manual</u> Auto
Oven	: <u>Off</u> ADP- CHG-

Here, you select each parameter on <TITRATION> second display.

Select item by cursor using  $[\uparrow], [\downarrow], [\leftarrow], [\rightarrow]$  key and enter desired amount or conditions and then, confirm by  $[ \downarrow ]$  key.

Control Gain	: Select speed of electrolysis. (Normal 5.0)			
	If reaction speed is too fast, reduce the speed to avoid over-titration.			
	Select a larger amount for a sample which contains sample water and would take a long			
	time to reach the endpoint.			
	Use a larger number for a sample of high bromine index number which takes a long			
	time to reach the endpoint.			
Stable	: This status appears when the change in drift $\Delta \mu g/min$ goes below preset amount that you entered. For example, if "Stable" does not appear for titration due to unstable moisture in the carrier gas for evaporation, select higher setting. But the higher setting will lead to deviation from true amount and to less precise results.			
Start	: When titration (electrolysis) start is set by sample injection(stir) after [Start] key is			
Stuit	pressed select Auto or Manual count-down to start. When Auto is selected titration			
	will start for testing titration without sample or for measurement of a sample of small			
	bromine index number.			

#### 11-4-4. Result parameter

Parameters on how to process measurement results are set up here including concentration calculation of data, formatting report on data and visualize data list in graphic chart.

Screen display after [Result] key is pressed:



Select item by cursor using  $[\uparrow]$ ,  $[\downarrow]$  key, and open each setup message display by  $[\downarrow]$  key.

#### 1. Calculation parameter

Set up parameters for concentration

<< Calculation >> [METHOD 5] Calc. No. (7 ~ 8) [7] Unit : mg/100g g/100g Weight : Fixed <u>Variable</u>

Enter each parameter on **<Calculation>** first display. Select item by cursor using  $[\uparrow], [\downarrow], [\leftarrow], [\rightarrow]$  key and enter desired amount and conditions, and then, confirm by  $[\downarrow]$  key.

- Calc. No. (7 & 8) : Select equation for computation. For details, see "Calculation Formula".
- Unit : Select a unit for calculation result. Select g/100g for bromine number and mg/100g for bromine index.

Weight : Select how to enter weight of sample. "Fixed" is concentration calculation by constant sample weight. "Variable" is calculation by individual sample weight at each time. This setting is for Calc.No.2, 3, 6 only, and not available for Calc.No.1, 4. The weight is entered by [Sample] key.

Screen display after [ Page down] is pressed: ( $[\downarrow], [ \downarrow ]$  ] key works when the cursor stays at the bottom line)

Enter parameters on **<Calculation>** second display. Select item by cursor using  $[\uparrow], [\downarrow], [\leftarrow], [\rightarrow]$  key, and enter numerals and conditions, and confirm by  $[\dashv]$  key.

Drift Comp.	Off Manu. Auto
	Select how to correct drift level.
Off	Drift is not corrected. This parameter is selected when total bromine amount including drift is the object.
Manu.	Drift level is corrected manually; for Instance when mean value is going to be used to compensate a large difference of drift at start and at the end.
Auto	Drift level is corrected automatically by the drift level at time when [Start] key is pressed. This is commonly practiced way of compensation.
Drift	This is manually entered value for drift correction. Select Manu. on Drift Comp, and enter the value.

#### Calculation formulas

Eq. No.	Purpose	Equation
7	Concentration when liquid sample is weight unit: mg/100g, g/100g	$F \times \frac{Moisture}{Wt1 - Wt2} \times D \times 0.1(mg/100g)$
8	For Concentration by measuring volume of liquid sample unit: mg/100g, g/100g	$F \times \frac{\text{Moisture}}{V1 \times \text{Dens}} \times D \times 0.1 (\text{mg}/100\text{g})$
	* "Moisture" in the equation is iden	ntical to "Data – Drift x t – Blank".
Moisture ( $\mu$ ;	g) : Net Bromine in a sample Bromine obtained by sub total Bromine titrated (el	e otracting "Drift value x titrating time and Blank value" from lectrolyzed) in the cell.
F	: Factor value that is coeff (see Function6)	ficient related to the results.
Data ( µ g)	: Total bromine content w (electrolysis) in the titrat	hich is the amount of entire bromine that has been titrated ion cell.
Drift ( $\mu$ g/s)	: Drift The moisture which ente	rs into titration cell from ambient air or carrier gas.
t (s)	: Measuring time The time counted from st titration. Define "t(wait) + t(max)	tart of titration with sample injected down to termination of " to end titration within desired time.
Blank ( µ g)	: Blank value The amount of bromine t and has to be deducted fr	hat has entered into the cell from other source than the sample rom the titration.
Wt1 (g)	: Total weight of a sample	e and the syringe before the sample is injected.
Wt2 (g)	: Total weight of a sample weight of sample injecte	e and the syringe after the sample is injected. Therefore, net d is Wt1-Wt2.
V1 (mL)	: Sample size The amount of sample m	easured in volume that has been injected.
D	: Dilution rate of sample	

#### Note:

It is necessary for measurement of bromine number to dilute the sample with toluene. The amount of toluene for dilution is calculated as follows:

Toluene (g) = sample (g) × expected bromine number (g/100g) / 0.2

- 1) Turn on the power of MKC-520.
- 2) Press [Pre-Titr.] key to dehydrate the measuring cell.

When the drift stabilizes after dehydration, the message "Drift Stable" appears on display.



3) Press [Start] key.

The message "Inject sample and press [Start] key" on display.

4) Inject sample liquid by a syringe into the measuring cell.

Bromine index (mg/100g)	Sampled amount (g)
below 10	10~15
10~50	5~10
50~100	3~5
100~200	1~3
over 200	~1

- For bromine number, take approximately 1g sample diluted with toluene.
- Prepare sample according to the above chart.

#### 5) Press [Start] key.

Measurement is started and the display shows "Now Titrating" with reading of bromine content.



6) When the measurement is finished, the display shows the results with beep.

Cell-1 No. 01-01 Drift (µg/s)	[ΜΕΤΗΟΟ 5] Br2 (μg)	
0.1	1001.3	
Result		

When a printer is connected, the results will be printed out at the same time.

# 11-4-6. Example of usual measurement

The example shows a measurement of bromine			
index of cyclohexene with default parameters.			
Three measurements were Performed resulting with			
as good 0.3% RSD.			
Sa	ample meterial	Cyclonexene	
С	atholyte	Potassium Chlomide	
А	nolyte	mixed Solvent	
		(СНЗСООН, МеОН,	
		KBr)	
D	etection electrode	Twin Pt electrode	
		M-713	
E	lectrolysis	Inner burette	
		(2-component burette)	
		(98-433-0006)	
Measurement parameter			
[Titration]			
	Date 1999/06/10 Titr. Cell No. Method t(stir) t(stir) t(stir) Drift Stop : Cont. Gain Stanble Start : Oven :	0 13:00 1 0s 15s 0s Rel 1.0 ug/min 5.0 1.0 ug/min Manual off	
[Calculation]			
	Date 1999/06/10 Titr. Cell No. Calc. No. Unit : Weight : Drift Comp :	) 13:00 1 1 ug Variable Auto	

Result		
* * * R e s u l t * * *		
Sample No.	01-01	
Date 1999/0 Titr. Cell No. Wt1 Wt2 Net Result	6/10 13:11 . 1 33.3160 g 32.3584 g 0.9576 g 944.2 ug 98.6 mg/100g	
***R	esult***	
Sample No.	01-02	
Date 1999/0 Titr. Cell No. Wt1 Wt2 Net Result	6/10 13:14 . 1 32.3585 g 31.4480 g 0.9105 g 894.1 ug 98.2 mg/100g e s u l t * * *	
Sample No.	01-03	
Date 1999/0 Titr. Cell No. Wt1 Wt2 Net Result	6/10 13:18 . 1 31.4438 g 30.3890 g 1.0548 g 1041.1 ug 98.7 mg/100g	
<auto statistics=""></auto>		
Date 1999/06/10 13:23 Sample No.(High) 01 Method 1		
Results	3	
Mean SD RSD	98.500 mg/100g 0.2646 ug 0.2640 %	

# 12. Printer

The MKC-520 when connected to optional Printer can print out measurement results, date of measurements, Operator's name, etc.

### 12-1. Printers connected to MKC-520

The built-in interface for printer installed in MKC-520 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-520. 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-520.


### 13. Maintenance

### 13-1. Daily checkup

### 13-1-1. Karl Fischer grease

Twin platinum electrode, inner burette, syringe inlet, desiccant tube, port plug, and plug for titration flask 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) Heat the glass contact areas with a hair dryer to melt the grease.

# <u> Warning!</u>

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.

### 13-1-2. 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.



#### 13-1-3. Changing the desiccant

The desiccant tube is removed from a titration cell like the clause of an application of KF grease. And the desiccant tube is removed from a bottle holder.

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 >

< Desiccant tube >

#### 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.

#### 13-1-4. Replacement of Karl Fischer reagent

< Replacement of the anolyte (anode reagent) >

Change the anolyte when the accumulated moisture measurement (=reagent life) has reached 1000mgH<sub>2</sub>O 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) Drain out the old anolyte using the dispenser. Press and pump the rubber globe on top of the waste bottle a few times to transfer the liquid into the waste bottle.



2) Fill the titration cell with new reagent. Hold the seal attached to the top of regent bottle with your finger, and pump the rubber globe. The reagent will flow into the cell. 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 stop filling, detach your finger which is holding the seal.



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.

### 13-2. Other Maintenance

### 13-2-1. 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.

### 13-2-2. 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.

### 13-2-3. 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.

### 13-2-4. 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.



### 13-2-5. 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.

### 13-2-6. 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>/!\</u> Warning!

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

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

### 13-2-7. 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.

# 14. 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-component cell or 1-component cell

# 15. Troubleshooting

# 15-1. Error messages and remedies.

Error message	Trouble	Remedies
"ADP Error!"	Tried to work ADP-511S	Check the connection with EXT LED.
↓ ↓	without MKC-520 connected.	If not lit, check Titration paramete
"Check ADP"		("Oven" must be "On")
"Electrode Open!"	No current from electrode	Check broken lead from electrode,
\$		loose connection or unplugged
"Check Electrode"		terminal.
"Electrode short!"	Potential is too low.	*Check contact of platinum electrode.
\$		*Raise potential by injecting water.
"Check Electrode"		
"Meas. Over!"	Exceeds 100mgH <sub>2</sub> O by one	*Replace catholyte(anolyte too if
\$	measurement	necessary) and pretitrate until drift
"Reduce Sample"		stabilizes, then try with less sample.
"C.Capa. Over!"	Catholyte capacity exceeds preset	*Replace catholyte and reset capacity.
\$	life.	
"New Catholyte!"		
"Catholyte Old!"	Alarm date has passed	*Replace catholyte and reset alarm.
\$	catholyte.	
"New Catholyte!"	-	
"Check Day!"	It is precision Check day.	*Check precision.
"XX Days to Check!"	It is warning for periodic check.	*Check when it comes due.
"Check Day Past!"	It has passed due date for	*Perform maintenance check
	checking.	immediately.
"Current Error!"	No electrolysis is generating.	*Sample resistance is too high.
\$		*Broken lead from terminals or loose
"Check Buret"		contact
		*Unplugged connector

Error message	Trouble	Remedies
"No Data!"	Tried statistics without data.	*Perform measurements and collect
		data.
"Over Titr!"	It is over-titrating.	*Clean inner buret if stained.
\$		*Inject a little water in the cell if it
"Inject Water"		occurs during pretitration.
"A.Capa. Over!"	Electrolysis has exceeded preset	*Replace anolyte and reset capacity.
\$	alarm.	
"New Anolyte!"		
"Anolyte Old!"	Anolyte has passed due date.	*Replace anolyte and reset alarm
\$		
"New Anolyte!"		
"Parameter NG!"	t(max):0s, Drift stop off are preset.	*Enter t(max) or select Drift stop :
\$		rel↑/abs↑.
"Correct setting"		
"Preamp Error!"	Preamplifier is malfunctioning.	*Contact your local dealer or sales
\$		rep.
"Contact Dealer"		

### Note:

To exit from Error message, press [Reset] key.

### Note:

If the error cannot be remedied by yourself, please contact your local dealer or sales representative.

### 15-2. 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.

### 15-3. Stirrer does not work properly



### 15-4. Drift level is too high



### 15-5. It runs into over-titration



### 15-6. Poor repeatability or no EP found



Mo	isture	Content		2	Sample	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	%	10	~	20	g

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

### 15-7. 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.

# Marning!

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

# 16. Display message and glossary

# 16-1. Display message and meaning

Display	Meaning
А	Concentration(ppm) of extracting solvent in indirect method, necessary for Eq. 3 and 6.
Abs	Titration terminates when titrated water goes below preset level.
ADP-	Parameter on Oven at time of connection.
Ageing	Purge out moisture inside Heater tube of Oven before measurement.
Alarm Set	Select alarm when the set level is reached.
Alarm	Set up level for alarm.
Anode	Setting on anolyte solution.
Auto	Automatic
Auto Statis.	Batch calculation of sample data by group.
Auto Statistics	
В	Amount of extracting solvent(g) for indirect method, necessary for Eq. 3 and 6
Back Purge	Moisture inside Evaporator(from sample inlet port to boat exit) has to purged out by carrier gas.
Balance	Electronic balance to weigh sample.
Baud Rate	an item of electronic configuration for interface protocol.
Blank	Water content intentionally added to the titration cell. (Constant water content like moisture in carrier gas for evaporation is called Drift)
Br2	Amount of detected bromine.

Display	Meaning
Br2 Index	Bromine index of measurement results
Br2 Number	Bromine number of measurement results
Calculation	Parameter for computation.
Calc.No.	The numbering of equation formula to be selected.
Capacity	Accumulated electrolytic current(water-converted).
Calibration	Select calibration of measurement.
	Turns on when Method is in Calibration.
Cathode	Setting on catholyte solution.
Cell	Select a kind of channel of titration cell
Cell-1	Factor value of cell ch-1
Cell-2	Factor value of cell ch-2
Cell No.	Select cell ch
Cell Purge	Purge moisture existing from sample inlet port of Heater tube to the titration cell.
CHG-	Parameter of Oven when connecting to Multiple sample Changer.
Concentration	The amount of water in percentage after measurement.
Control Gain	Coefficient of electrolytic progression.
D	Dilution rate of sample
Data	Date of measurement results.
Data Bits	The number of bit in communication interface.
Data Deletion	Erasing entered or saved data.
Data List	Sampling list of data per unit time.
Data Print	Select to print out each measurement results in batch calculation.
Date&Time	Setting date and time
Day	Set up a number of days for alarm.
Dens	Density of sample, necessary for Eq.4.
Detail	Information detailing periodic checks in the past.

Display	Meaning
Display	Setting on display message.
DP-	Thermal dot printer DP-500 by Kyoto Electronics.
Drift	Moisture existing in ambient air which enters the titration cell and cannot be avoided.
Drift Comp.	Correction of drift level during titration.
Drift Stop	Detect endpoint by drift level when it goes below preset level.
English	Message expressed in English language.
Even	Even parity of related to calculation results.
Factor	Coefficient related to calculation results.
Fixed	A setting that cannot be changed.
Form1	Titration graphic chart showing accumulated water with 100% at endpoint.
Form2	Titration graphic chart showing accumulated water with titrated moisture per unit time.
Function	Program to execute data processing or activation.
GLP	Print format conforming to Good Laboratory Practice.
Graphic Form	Selection of graphic style of titration curve.
Hour	Setting of the hour.
IDP-	Impact dot matrix printer IDP-100 by Kyoto Electronics.
Interface	Parameter on interface for RS232C, Balance or Printer.
Interval(1-12)	Enter a number of months for periodic check.
Japanese	Message on display expressed in Japanese language.
Language	Select language in English or Japanese.
LCD Contrast	Adjust brightness of LC display screen.

Display	Meaning
List	A list of periodic checks in the past.
Lot No.	Lot number of measured sample.
Low Sample No.	Individual number for sample (00 to 99).
Manu.	Input by key entry.
Manual	
Mean	Average amount of calculation results.
Meas No.	Numbering of measured sample.
Measure No.	
Memory Clear	Erase saved memory and initialize to default.
Method	Titration form. MKC-520 has 5 Methods individually stored with parameters
	for titration, results, sample, etc.
Minutes	Setting of time by minute.
Moisture	Display of water content.
Month	Setting of time by month.
Name	Operator's name.
Next Check Date	The number of days to the next check day after precision check has been done.
Next Meas.	Sample number in Sample file for next measurement.
None	No parity for parity in communication interface.
Odd	Odd parity in communication interface.
OK/NG	Criterion of precision check.
On	A mode or program to be activated or activating.
Other	Printers other than those printers which interface for printing is installed.

Display	Meaning
Oven	Selection to control Evaporator.
Oven/Purge	Setup of ageing and its start of evaporation.
Oven Temp.	Heating temperature of Evaporator.
Parity	A digital configuration for communication interface.
Periodic Ck	Precision check due periodically.
Periodic Check	
Potential	Voltage of anode current.
Precision Ck	Select Precision Check mode or not.
Precision Check	
Press Pre-Titr key	Message on Initial display asking Operator to press
	[ Pre-Titr. ] key.
Press Start key	Press [ Start ] key.
Pre Treat	Selection of how to feed sample into Evaporator.
Printer	Setup and selection of Printer to be connected.
Print Time	Unit time to sample data for printing out titration graphics.
Protect	Select or not to inhibit key entry to protect preset parameters (Titration, measurement result, sample) for each Method.
Reagent Capa.	The electric current of accumulated electrolysis (converted to H <sub>2</sub> O) of anolyte
Reagent Capacity	and catholyte.
Reagent Life	Enter the number of reagent effective days. If it elapses, the alarm activates.
Reagent name	Enter the name of reagent for anolyte and catholyte
Recalculation	Setting of recalculation parameters.
Rel	Abbreviation of "Relative". Titration ends when detected moisture during
Replaced	Replace the reagent. The date when "0" is input on Capacity.

Display	Meaning
Report	Setting of result parameters.
Result	Select parameters on calculation of titration data, display of the results, titration graphic curve, etc. Also, it measurement results.
Result Time	Select the length of time to show measurement results on display. If 0s is selected, the display continues until [ ] ] key is pressed.
RSD	Abbreviation of Relative Standard Deviation, which shows percentage of standard deviation divided by average value. RSD means the same as Coefficient Variance CV (%).
RS-232C	Parameter to communicate with external computer.
S	Abbreviation of time unit of second.
Sample	Parameter for sample.
Sample File	Data records filed by sample.
	Enter by [ Sample ] key the order of filing and measuring conditions, sample conditions, etc.
Sample No.	It means sample number . The number is divided into two numbers (each 00 to 99); high order number for grouping and low order for individual number. If a high order number is changed, the data of the group is regarded as a different group and the relating data will be erased.
Sampler Purge	Purge moisture in Sampler by carrier gas.
Sampling Interval	Time interval of sampling measurement data.
SD	Standard deviation.
Serial No.	Instrument production number registered in plant before shipment, also displayed on rear panel.
Setup	Setup function to execute settings on measurement results processing and data management.
Short	Short form of printed record.
Size	Prints sample weight and constants.
Soft H.S.	Soft handshake is a communication parameter via RS232C.
Stable	Enter changing ratio of drift to show stability.
Standard Value	Property of standard substance.

Display	Meaning
Start	Setting on how to start measurement.
Std.	Water content of the standard used for periodic check.
Stir.Time	Time selected for t(stir).
	Stirring continues for this time without titrating while time is counted down.
Stop Bits	The number of stop bit for interface communication.
Temp.	The value required when Eq.5 is selected.
	It is the temperature of gas for measurement of gaseous sample of which volume is calculated.
Titration	Titration parameter
Titration Time	Select or not to print titration time.
Tolerance	Input range of criterion for the results of precision check.
t(max)	Input maximum length of time for measurement.
	Measurement ends in t(stir)+t(wait)+t(max).
t(stir)	Time setting during which stirring sample continues until titration starts.
t(wait)	Time setting for titration from start to the end during which titration does not stop.
Unit	Select unit for calculation results.
Variable	Variable setting, not fixed.
Version No.	Version number of the instrument
V1	Sample volume (mL) necessary when Eq.4 is selected.
V2	Volume of gas(L) necessary when Eq.5 is selected.
Weight	Set up weight input for sample.
Wt1	Weight of sampler with sample in it. (g)
Wt2	Weight of sampler after sample is released. (g)
Wt0	Parameter necessary when Eq.3 or 6 is selected for indirect titration method, that is, sample size(g) injected into solvent for the sample to be dissolved.

Display Meaning			
Year	Calendar year		
Os	Zero second. t=0(max) means no limit of time.		
ADP Error!	Error message when [Start] is pressed while "Oven" is set to "ADP-" without Evaporator is connected.		
Oven Start	It is preparing Evaporator for measurement		
Oven Burge	It is purging Evaporator inside system		
Electrode Open!  Check Electrode	Error message when no current is picked up by Electrode.		
Electrode Short!  Check Electrode	Error message Electrode is electrically shorted.		
Sampler Open    Press Start key	Slide the ball stopper of Eggplant-type sampler to open the vent hole, and press [Start].		
Sampler Close	Slide the ball stopper of Eggplant -type sampler to close the vent hole, and press [Start].		
Samp. Purge	Sampler purging time during which time is counted down.		
Input Weight	Input weight of the sample.		
Boat Mov'n	Sample boat is moving from sample inlet to Oven.		
Sample In    Press Start key	Introduce sample and press [Start] key.		
Speed	Select stir speed.		
Cell Purge	Cell purging time during which time is counted down.		
Ready	Message of Ready mode of titration cell on Main display.This message appears when drift goes down below $10.0 \mu$ g/s.		

Display	Meaning
Result	Measurement results after compensated and calculated.
Measurement	It is titrating the sample.
Meas. Over!	Error message when more than 100mg water is detected during a
\$	measurement.
Reduce Sample	
Capa. Over!	Error message when electrolysis of cathlolyte reaches alarming limit.
\$	
New Catholyte!	
Catholyte Old!	Error message when use of catholyte elapses preset limit date.
\$	
New Catholyte!	
Check Day!	Message when it is the day for check date after passing preset period.
X Days to Check	Message of number of days due for periodic check.
Check Day Past!	Message when it has passed the date for periodic check.
Current Error!	Error message when electrode resistance of inner burette is too high.
\$	
Check Buret	
Over Titr.!	Error message when anolyte potential goes down too low causing overtitration.
\$	
Inject Water	
Data not exist!	Error message when data does not exist in memory space.
Stable	Message of Stable mode of titration cell on Main display, changing from
	Ready mode.
	This mode is for precise measurement of sample when rate of drift change goes
	down below preset rate, usually 0.1µg/min.
Back Purge	Backpurging time during which time is counted down.
Capa. Over!	Error message when electrolysis of anolyte reaches preset alarming limit.
\$	
New Anolyte!	

Display	Meaning
Anolyte Old!	Error message when use of anolyte reaches preset limit.
\$	
New Anolyte!	
Parameter NG!	Error message when [Start] is pressed while t(max):0s and
\$	Drift Stop: Off are selected.
Correct Setting	
Preamp Error!	Error message when preamplifier malfunctions.
\$	
Contact Dealer	
Pre-Titr.	It is pretitrating.
<b>▲</b>	The display can return to previous page by [Page up] or $[\uparrow]$ key.
	(The cursor also works when it stays on the upper line)
▼	The display can go ahead to the next page by [page down], $[\downarrow]$ or Return key.
	(the cursor also works when it stays on the bottom line)
(Cursor blinking)	Enter letters or numbers using numeric key, [ · ] or [-/Disp.] key.

### 16-2. Glossary

Word	Meaning		
Ageing	See "Ageing" on display.		
Sampler purge	See "Sampler Purge" on display.		
Evaporator	An oven which vaporizes moisture in solid sample and send it by carrier gas to titration.		
Cell purge	See "Cell Purge" on display.		
Multiple sample changer	An automatic sampler with revolving carrousel to turn a number of sample vials on it for continuous measurement.		
Drift	See "Drift" on display.		
Purge	To push out moisture by carrier gas.		
Back purge	See "Back Purge" on display.		
Parameter	Individual condition or constant for titration or calculation.		
Factor	See "Factor" on display.		
Blank	See "Blank" on display.		
Method	A form of measurement composed of parameters.		

### 17. Others

### 17-1. Part list







### 17-1-1. 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	Vesting tools Seguring (10)postal Proved Proved Acode adjuster
12-006-7400-48	Titration cell unit (two solution) set	1	Transparent cell Two-component cell Twin platinum electrode / KF and other attachment	Vienhing bottle Septem (10poniae) KF grease Funnel Anode adjuster
12-006-6400-48	Titration cell unit (single solution) set (with a cock)	1	Transparent cell 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	
98-560-0011	Vat	1		
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	

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	
98-500-3225	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	
CHK-501	Multiple sample changer	1	40 to 300°C	
KF-Win/ER	Moisture titrator software compliant with electronic records	1		
IDP-100-10 IDP-100-11 IDP-100-12	Impact dot printer	1 1 1	100V 120V 230V 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	Ĭ
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	

# 18. System chart


## 19. Pin assignment of I/O interface

1) RS232C



2) Balance



3) Printer



## 20. Specification

Specification	Contents
Type and Model	MKC-520 Karl Fischer Moisture Titrator
Measuring method	Karl Fischer coulometic Titration method
Control method	Constant current pulse time control
Endpoint detection	AC polarization
Display of titration	Pre-titration (status of excess water)
	Ready (status of rough measurement)
	Stable (status of precise measurement)
Indication of EP	Electronic beep
Stirring method	Magnetic stir motor with spinner in cell
	Stirring speed adjustable by 10 steps
Titration cell	100mL (max. 150mL) 2 channel (option)
Measuring range	10µg to 100mg H <sub>2</sub> O
Display resolution	0.1µg
Reproducibility	within 0.3%CV (n=10)/water-methanol 1mgH <sub>2</sub> 0
Drift compensation	Automatic (cancelable by key entry)
Content	$H_2O 0.1 \mu g$ to 999999 $\mu g$ (display at end of titration)
	Br <sub>2</sub> 0.9µg to 9999999µg
Diagnostic function	Electrolytic current, measured reading, over-titration, anolyte life, catholyte life,
	electrode short or open, parameter entry, preamplifier malfunctioning, inhibited
	key entry
External control	Balance; automatic weight transfer
	Computer; RS232C interface
	Printer; date, sample size, water content, measured time, etc.
	(printer is external, IDP-100 recommended)
Ambient condition	5 to 35°C below 85%RH
Power source:	AC 100 / 120 / 220 / 230 / 240 V
	50 / 60 Hz
Power consumption	Approx. 50 W
Dimension	1) Main unit : $288 (W) \times 468 (D) \times 215 (H) mm$
	2) Stirrer : 118 (W) × 225 (D) × 330 (H) mm
	3) Solvent change unit : $240(W) \times 170(D) \times 405(H) \text{ mm}$
Weight	Approx.10kg
CE marking	EMC : EN61326
	LVD : EN61010-1 conformed

## 21. Warranty and After-Sale Service

- 1. The product you have purchased passed factory inspection and testing prior to shipment, and its quality is guaranteed by free of charge replacement during warranty period except consumable parts provided the instrument has been under normal use and operation, however, depending on operational and environmental condition under which the instrument has been in use may require chargeable service work.
- 2. For service during and after warranty period, please contact your local dealer or distributor.
- Read the manual thoroughly before you decide to call for service.
  When you should need servicing, please provide with the following information:

Production number of unit Description of the trouble Person to contact

- 4. Parts and spares can be purchased separately and will be available for seven (7) years after termination of production of the model.
- 5. This warranty does not cover claims due to any of the following conditions:
  - 1) Any modification or specification change by an unauthorized person
  - 2) Damage by splashed water (the instrument is not water-proof)
  - 3) Use in range or condition other than specified
  - 4) Operated in other way than specified in the manual or negligence of maintenance
  - 5) Physical force given to the instrument during transportation or move
  - 6) Use of parts or reagent other than specified
  - 7) Caused by use under extreme ambient or environmental condition
  - 8) By fire, riots, earthquake, lightning, or Act of God in any form or manner
- 6. Escape clause

Under no circumstances will Manufacturer be liable for any damage, whether incidental, consequential or other, or for any other remedy arising from any loss, damage, expenses or inquiry in connection with use of the article.



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