Karl Fischer Moisture Titrator



Operation Manual

Please read this manual thoroughly before actual use for measurment.

Ver.09 A/N 98-595-0242

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1. General

1-1. Introduction

We appreciate your patronage by purchasing MKS-500 Karl Fischer Moisture Titrator, which is the volumetric KF titrator to determine water content in liquid or solid sample materials. The instrument is designed easy to use and produce measurement results accurately in a short span of time.

[Feature]

- Measurement operation is easy to perform.
 Routine measurement can be performed by [Pre-Titr.] and [Start] key only.
- Measurement results are digitally shown with high reproducibility. The reproducibility of standard 10mL burette unit is ±0.005mL.
- The instrument is GLP/GMP conformed. This unit performs functions required by GLP/GMP including printout and instrumental precision check.
- Various interfaces are standard The standard interface includes RS232C for PC, balance and printer.
- Charging and draining solvent is simple.
 The optional Manual absorb and drain unit (98-433-0134) makes the delivery and discharge of solvent easy while reducing the uncomfortable odor of KF reagent.

It is recommended to peruse and keep this manual near your system for the best performance and efficiency of the system.

Note!

If you should find any description different from the specification of your instrument, follow the instruction accompanied with the unit.

1-2. About the manual

Operate the apparatus with this manual by your side at all times.

You will find some descriptions marked as shown below. They are intended to call your careful attention.

1. Possible danger such as human body injury or death:

<u>Warning!</u> Accidents such as injury to human body or death are possible unless the warning is strictly observed.

2. Possible danger such as loss of property:



3. Failure of performance

Note!

Product may not perform in full performance to the specification unless the note is observed.

- It is prohibited to copy or make use of a part or all of this Manual.
- If you should find any questions or need more information other than in this manual, please contact your local dealer or sales representative.
- We will not be held responsible for any failure or damage or loss arising from measurement results by the instrument.

1-3. ASafety symbols

Always observe these signs and introductions.

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



You must ground earth wire of power cable.



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

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

Danger of electric shock if not grounded to earth.

\land WARNING!

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





Danger of fire if a wrong fuse is loaded.

WARNING!

Do not use volatile chemical or work in flammable gas.



Danger of explosion inside the instrument.

\Lambda WARNING!

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



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

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



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

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



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

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



Danger of fire, shock or malfunctioning of the unit.

Caution!

About the place for installation:

Avoid a place under any of the following conditions in order to maintain performance and reliability of the instrument.

- A place under vibration
- A place under direct sun light
- A place where corrosive gas exists.
- A place near strong electro-magnetic field or heavy power fluctuation
- The ambient temperature is 5 to 35°C. Do not use under excessive condition.
- The ambient humidity is less than 85%RH. Avoid higher humidity.

About power source:

- The power source is $AC100 \sim 120V$ or $200 \sim 240V$. Frequency is 50 or 60Hz.
- Supply power direct from power outlet.
- Do not supply power from power tap where other lines share the power.

About place for storage:

- If the instrument is not going to be used for an extended period of time, it is recommended to clean the titration flask and dry it, then keep them overhauled in a desiccator. Use the carton box in which the instrument was first delivered.
- Avoid a place dusty or highly humid, or too hot or too cold.

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.

2. Installation

2-1. Supplied parts

The delivered carton box contains MKS-500 Main unit, accessories and operation manual. Please check the delivered parts with the checklist below. If you should find any missing or broken part or parts, contact your local dealer or sales representative.

(1) Main unit (MKS-500)			1 unit
(2) Cylinder with piston (10mL)		12-006-5200-48	1 piece
(3) Burette cover	r	20-040-4300-48	1 piece
(4) Burette stopp	ber	20-040-4400-48	1 piece
(5) Switching va	lve	98-430-0050	1 piece
(6) Screw			4 pieces
(7) Power code	(for AC 100/110/120V)	98-320-3198	1 piece
	(for AC 220/230/240V)	98-320-3461	1 piece
	(for UK AC 230/240V)	98-320-4199	1 piece
	(for China)	64-000-1800-48	1 piece
(8) AC-3P adapt	er (for AC 100/110V)	98-320-3199	1 piece
(9) Reagent bott	le cap (for KF)	98-433-0066	1 piece
(10) Ground wir	e (for AC 100/110V)	98-433-3331	1 piece
(11) S-shape titration vessel		98-740-3001	1 set
(12) Titration vessel stopper		98-433-3435	1 piece
(13) Twin platinum electrode/KF		98-103-M714	1 piece
(14) Stirrer rotor (25mm)		98-500-3141	1 piece
(15) Joint KF		20-040-4700-48	1 piece
(16) Tip of diffu	sion proof nozzle	12-006-4900-48	1 piece
(17) KF grease (5g)	98-433-3138	1 piece
(18) Dessicant tu	ibe A	98-433-3116	2 peaces
(19) Piston extraction rod		98-551-5002	1 piece
(20) Wrench (8mm)		98-514-3339	1 piece
(21) + driver		98-514-3164	1 piece
(22) Drain bottle		98-500-3134	1 piece
(23) Operation n	nanual	98-595-0241	1 copy
(24) Warranty			1 copy

2-2. Installation of switching valve

Install the switching valve (with tube connection) on Main unit as shown below.

(1) Install the valve with its mark facing front, and fix with screws.



If you find it difficult to install the valve properly, the convex part of the valve and concave part of Main unit may be deviated. In this case, turn the knob manually so that both convex and concave parts are matched.



2-3. Connecting of power cable and Installation of burette

Install 10mL cylinder, burette cover and burette holder on Main unit. Before you install the cylinder, supply power according to the following steps.

<u>∕∕Warning!</u> The earth wire must be grounded. Danger of electric shock without grounding!

(1) Make sure the power switch is in OFF position.



(2) Plug in the power cord into the power inlet.

(3) Plug in the power cord to the power outlet.





<3P outlet> The 3P plug has ground terminal in it and ground wire is unnecessary.

<2P outlet> Connect AC-3P adapter to the 2P plug, and ground the green wire to the earth terminal.

(4) Turn on the power.

(5) When [Pre-Titr] appears on display, press [Δ] key.

(6) Insert the piston rod into the piston catch hole securely, and press $[\nabla]$ key to lower the piston while holding the burette upright.



(7) When the piston stops, tighten the burette holder, and put on the burette cover.



2-4. Installation of titration flask

<u>Warning!</u> Turn the titration flask top with its holding screw tightened. If it turns without screw fixed, it may break.

- (1) Undo the holding screw.
- (2) Set the titration flask top as shown below, and fix it by screw available from (1).
- (3) Put a stir bar in titration flask, and apply KF grease over the area indicated below (connect area between flask and the top).
- (4) Turn the flask top from titration position to 90 degree counterclockwise, and open the clip of flask holding lever on the top to set the titration flask.



(5) Move the titration flask over to titration position.

2-5. Installation of tube line

(1) Insert the tip of anti-diffusion nozzle into KF joint.



(2) Connect the tubes and titration nozzle as shown below.



Do not loose Teflon stopper at the tip of CRPT20 tube.



2-6. Installation of desiccant tube, port plug and electrode

(1) Lightly apply KF grease over sliding area of the electrode, desiccant tube and port plug, and insert each of them into the position.



(2) Insert the electrode plug into the connector on the rear of Main unit, and fix it by turning the outer screw of the plug.



The above procedure completes the assembly of the unit.

2-7. Parts name and functions

2-7-1. Front view



(1) Burette cylinder

Titration goes on in this burette.

(2) Switching valve

Switch the course of titration reagent.

(3) LCD

Liquid crystal display with back light.

(4) Keypad

The keys control titration and set up parameters.

(5) Reagent bottle

Use the bottle of commercial titration reagent.

(6) Titration nozzle

Reagent is discharged through this nozzle.

(7) Detection electrode (Twin Pt electrode)

It detects potential change.

(8) Port plug

This sample is injected by removing the plug.

(9) Power switch

The power of the unit is turned on or off by this switch.



(10) Electrode port

The cable from detection electrode is connected.

(11) Printer port

The cable from printer is connected here.

(12) Balance port

The cable from balance is connected here.

(13) RS232C port

Personal computer is connected here.

(14) Power inlet

The power cable is plugged in here.

2-7-3. Key layout and function



	Method key	Select titration type. (direct/indirect/factor/calib)
Method		
	Titration key	Kay to select titration parameter
	Thranon Key	Key to select thration parameter.
Titration		
	Result key	Key to set up calculation and print format.
Result	-	
mesun		
	Sample key	Key to set up parameters for sample including operator's name, sample
Sample		number, sample size, etc.
	Eupotion kov	Var to perform functions
	Function key	Eurotion 0: Reagant factor and confirmation
Function		Function 0. Reagent factor and commutation
		Function 2: Auto statistics
		Function 2: Deletion of data
		Function 4: Calibration (History)
		Function 5: Initialization of memory
	Satur kov	Variate select Seture function
	Setup key	Setur 0: Degister
Setup		Setup 0. Register
		Setup 1. Date and time
		Setup 2: Secure a secure
		Setup 5: Sample name
		Setup 4. Serial number and version number
		Setup 5: LCD originaless adjustment
		Setup 6: Calibration setup
		Setup 7. Lock operating keys

Print	Print key	Key to print out data
Stirrer	Stirrer key	Key to turn ON/OFF the stirrer and set stir speed
АРВ	APB key	Key to select a number of purge times
\bigtriangleup	Discharge key	Key to discharge reagent
\bigtriangledown	Suction key	Key to suck reagent into burette
$\Box \nabla$	Purge key	Key to fill reagent and purge
Pre-Titr	Pre-Titr. key	Key to start pre-titration
Start	Start key	Key to start titration
Reset	Reset key	Key to abort titration or dosing
Disp.	Disp key	Switch display and sample input (manual/balance)
	. key	Key to enter decimal point "."
STU 1 9	Alphanumeric key	Key to enter parameters
	– key	Key to enter parameters and switch the sample input.
<u> </u>	Up key	Key to move the cursor upward
↓	Down key	Key to move the cursor downward
<u></u>	Left/right key	Key to move the cursor to the left or right and to select parameter
<u></u>	Bs. Key	Key to erase the preceding character
Cir.	Clr. Key	Key to clear all entries
جا	Enter key	Key entry is confirmed by this key.

3. Before measurement

3-1. Initial display

When the power of MKS-500 is turned on, the initial display appears as follows:



After a while, the display changes as shown below.

D < < < I	01-01
[Pre-	Titr.]

<Main display>



3-2. Filling the reagent

Note!

Appropriate solvent and reagent have to be selected according to the sample.

For example, for sample which includes ketone or aldehyde, use reagent for ketone.

Use the bottle of commercially sold KF reagent as it is, however, if the bottle neck is different in size (e.g., Merck reagent bottle), transfer the solution into a bottle with correct neck size.

Sample	RdH's Reagent		Remark
General sample	For general use	Composite 5	
		Composite 2, etc	
Sample including	For ketone	Composite 5K, etc	Use solvent for ketone.
ketone or aldehyde			

(1) Attach the tube and desiccant tube to the bottle cap and fix the bottle by turning around as illustrated.



- (2) Press [APB] key and set up a number of purge.
- (3) Press [APB] key to show the display as follows:



Select purge times by numeric key and confirm by [] key.

- (4) Press $[\Delta \nabla]$ key to purge.
- (5) Press $[\Delta]$ key and fill the tube line with reagent.

Note!

By pressing $[\triangle \nabla]$ key, the reagent is purged for the selected number of times. When 0 is selected, it continues purging until [Reset] or [Start] key is pressed.

3-3. Delivery of solvent and pre-titration

(1) Take off the port plug and insert the funnel.



- (2) Add 50mL solvent. (just about the whole Pt element covered with the solvent when stirred)
- (3) Set the port plug and press [Pre-Titr.] key to dehydrate the cell. When the cell is dehydrated, the cell condition is ready to start measurement.

Note!

Appropriate solvent must be chosen according to the sample. For example, for a sample which contains ketone or carboxycyl base, use solvent for ketone, and for a sample which is strong alkali like amine, add pH buffer like salicylic acid to the solvent.

Sample	Commercially availa	ble solvent	Remarks
General sample other than below	For general use	Solvent ML Solvent MI, etc.	
With ketone, aldehyde	For ketone	Solvent CE, etc.	Use titration solution for ketone.
Petroleum products like lubricant, fats and oil	For fats and oil	Solvent CM, etc.	
Saccharides like millet jelly	For saccharides	Solvent FM	
With alkali like amine	Adjust pH by adding citric acid or salicylic acid to solvent	Solvent MS, etc.	Adjust additive depending on sample.
With hydrochloric acid	Adjust pH by adding buffer to solvent	Hydranal buffer, etc.	Adjust additive depending on sample.

3-4. Date and time setup

The MKS-500 is equipped with clock function, and can print out measurement date and time. The following description explains how to set up display as well as time adjustment.

3-4-1. Display format

- (1) Press [Setup] key.
- (2) Use [1] or $[\uparrow]$, $[\downarrow]$ key to select "Date&Time" and press $[\lrcorner]$ key.
- (3) Select "Display" by $[\leftarrow]$, $[\rightarrow]$ key and press $[\downarrow]$ key.
- (4) Select the balance by $[\leftarrow]$, $[\rightarrow]$ key and press $[\dashv]$ key.

- Display format that can be selected -

Format	Example of printout
DD/MM/YYYY	23/01/2000
YYYY/MM/DD	2000/01/23
MM/DD/YYYY	01/23/2000

3-4-2. Date and time setup

- (1) Press [Setup] key.
- (2) Use [1] or $[\uparrow]$, $[\downarrow]$ key to select "Date&Time" and press $[\downarrow]$ key.

(3) Select "Date&Time Set" by $[\leftarrow]$, $[\rightarrow]$ key and press $[\dashv]$ key.

(4) Present time is shown. Press $[\downarrow]$ key.

(5) Enter date and press $[\downarrow]$ key.

(6) Enter time and press $[] \downarrow$] key.

Setup(0-6)? 1 Date&Time

Date&Time? _____Display→

Display? YYYY/MM/DD \rightarrow

Setup(0-7)? 1 Date&Time

Date&Time? Date&Time Set→

Date&Time: 2000/02/23 15:42

DateYYYY/MM/DD? 2000/02/23

Time?

15:42

4. Basic operation

For routine measurement by MKS-500, just follow the guiding message on display.

4-1. Measuring steps



4-2. Method setup

The MKS-500 is equipped with 4 Methods, and a correct Method has to be chosen for the desired measurement.

(1) Press [Method] key.

(2) Select the desired Method by $[\leftarrow], [\rightarrow]$ key and press $[\dashv]$ key.

```
Method?
Direct→
```

Method	Discription	Remark
Factor	for factor measurement	See 4-2-1. Factor measurement of reagent
Direct	for routine measurement	See 4-2-2. Sample measurement
Calib	for calibration	See 4-2-3. Calibration
Indirect	for measurement with ADP-511S	See 4-2-4. Measurement with the evaporator
	Evaporator	

4-2-1. Factor measurement of reagent (Factor)

It is necessary to measure the factor of Karl Fischer reagent before sample measurement. Factor shoud be measured once a week to a month.

* Factor : moisture concentration reacted with 1mL of KF reagent

- (1) Press [Method] key.
- (2) Select "Factor" by $[\leftarrow], [\rightarrow]$ key and press $[\downarrow]$ key.
- (3) Deliver 50mL solvent in the cell (just to cover the Pt electrode).
- (4) Press [Pre-Titr] key to dehydrate the measuring cell.
- (5) When dehydrated, "Sample In" appears on display.When "Drift" appears, press [Start] key.
- (6) Press [Stirrer] key to stop stirring. Fill the syringe with standard substance (or pure water) and weigh Wt1 by the balance.
- (7) Take off the port plug and quickly inject standard substance, and close the inlet by the stopper.
- (8) Weigh Wt2 of syringe by the balance.

Method? Factor→

Operator? $0 \rightarrow K \in M 0 0$

<u>Note!</u> See "4-5. Sample parameter" for input sample parameters.

(10) Press [Start] key to start factor measurement of reagent.

- (11) When EP is detected, the results appear on display.To repeat measurement, repeat step (6) to (11).
- (12) Press [Function] key for auto statistics. The calculated results are automatically input for factor. The factor value can be confirmed on "Entry and confirmation of Reagent factor" on Function 0.

F I> 1.2345mg Measurement

R e s u l t 4 . 9 8 0 6 m g / m L

<u>Note!</u> Reagent for factor measurement Factor of Karl Fischer reagent has to be checked and maintained for volumetric KF moisture titration. For factor measurement, typical reagents are pure water of which concentration is known, sodium tartrate dihydrate or commercially available water methanol standard liquid.

 Standard substances
 Enter theoretical value in % unit. Typical standard substances are shown below: Water standard 10(mg/g) 1.00%
 Sodium tartrate dihydrate 15.66%
 Pure water 100.00%

Collect the standard substance in order to make the titration volume of Karl Fischer reagent 2 to 5mL.

4-2-2. Sample measurement (Direct)

- (1) Press [Method] key.
- (2) Select "Direct" by $[\leftarrow]$, $[\rightarrow]$ key and press $[\downarrow]$ key.
- (3) Deliver 50mL solvent in the cell (just to cover the Pt electrode).
- (4) Press [Pre-Titr] key to dehydrate the measuring cell.
- (5) When dehydrated, "Sample In" appears on display. When "Drift" appears, press [Start] key.
- (6) Press [Stirrer] key to stop stirring. Fill the syringe with sample and weigh Wt1 by the balance.
- (7) Take off the port plug and quickly inject standard (or pure water), and close the inlet by the stopper.
- (8) Weigh Wt2 of syringe by the balance.
- (9) Press [Sample] key and enter sample parameters.

D < < < I	01-01
Pre-	Titration

D	>	01-01
	Sample	ln

Operator? $0 \rightarrow$ KEM00

<u>Note!</u> See "4-5. Sample parameter" for input sample parameters.

- (10) Press [Start] key to start moisture measurement.
- (11) When EP is detected, the results will be shown. To repeat measurement, repeat step (6) to (11).

R e s u l t 2 . 5 0 1 %

< Handling after completing measurements >

Keep the titration nozzle - filled with KF reagent - in the solvent in the titration flask.

Store MKS-500 unit after completely draining out the reagent from the burette and rinsing the inside of burette with Methanol or the like when MKS-500 in not used for an extended time period. In addition, it is recommended that the titration flask be rinsed, dried out and stored in a desiccator with the flask disassembled.

Caution!

Karl Fischer reagent may precipitate crystalline formation if left unused for more than a week. Clean the system before it leaks by attempting discharge the precipitating liquid.

4-2-3. Calibration (Calib)

(1) Press [Method] key.

(2) Select "Calib" by $[\leftarrow], [\rightarrow]$ key and press $[\downarrow]$ key.

- (3) Deliver 50mL solvent in the cell (just to cover the Pt electrode).
- (4) Press [Pre-Titr] key to dehydrate the titration cell.
- (5) When dehydrated, "Sample In" appears on display. When "Drift" appears, press [Start] key.
- (6) Press [Stirrer] key to stop stirring. Fill the syringe with standard liquid and weigh Wt1 by the balance.
- (7) Take off the port plug and quickly inject standard (or pure water), and close the inlet by the stopper.
- (8) Weigh Wt2 of syringe by the balance.
- (9) Press [Sample] key and enter sample parameters.

Method? Calib→

C < < < I	01-01
Pre – Titr	ation

С	>	01-01
	Sample	ln

Operator? $0 \rightarrow K E M 0 0$

<u>Note!</u> See "4-5. Sample parameter" for input sample parameters.

(10) Press [Start] key to start measurement.

(11) When EP is detected, the results appear on display. To repeat measurement, repeat step (6) to (11).

С	l > 1.2345mg
	Measurement

Result 1.0045%

(12) Press [Function] key for auto statistics.

Note!

The results of calibration test are obtained and stored in the main unit when auto statistics is performed.

The results can be confirmed on the calibration (History) of "Function".

4-2-4. Measurement with the evaporator (Indirect)

When an evaporator is connected to the MKS-500, solid sample like iron ore or plastics can be measured. For details of operating the evaporator, refer to its manual. The below chart shows how to set up Method, Titration and Result (calculation, Blank) necessary for indirect method.

<Setup Method>

For measurement with Evaporator, set up method and measuremnt conditions as shown follows.

	Setup values	Remarks
Method	Indirect	Method for indirect titration (evaporation method)

<Setup Titration parameter>

	Setup values	Remarks
Titr. Speed (1-6)	3	
End Time (s)	0	
Final Volume: mL	0.01	
t(stir): s	0	
t(max): s	(1200)	Change t(max) for the sample.
Drift Titration	On	
Max Volume: mL	(20.0)	

<Setup Result parameter>

		Setup values	Remarks
Calculation	Calc Formula?	(g→%)	
Blank	Blank?	Yes→	Correct the moisture volume in carrier gas.
	Time: s?	600	ex) 10mg carieer gas moisture for 10 minutes
	Value: mg?	10.000	Set "Time:s?" to 600 (60s×10 minutes), and
			"Value: mg?" to 10.
Report	Report?	Short→	

4-3. Titration parameter

The MKS-500 has titration parameters for each Method and measurement conditions can be set up according to the sample and reagent. More precise and faster measurement can be performed under appropriate conditions.

(1) Press [Titration] key.

(2) Select "Titr.Speed" out of 1 to 6 and press [] key.

<Titr. Speed>

Titr. Speed depends on reaction of titration solution and sample liquid.

Titr. Speed

- 1: When Merck's reagent is used. (Titr.Speed: fast)
- 2: When Mitsubishi reagent is used.
- 3: When Riedel-de Haen's Composite is used.
- 4: When Riedel-de Haen's Titrant is used.
- 5: for over-titration
- 6: for over-titration (Titr.Speed: slow) for over-titration, increase Titr.Mode by 2 to 3 steps.
- (3) End Time: Enter EP wait time and press $[\dashv]$ key.

Endpoint is reached when excessive reagent continues for more than preset time. Typically 30 seconds. (according to JIS) For samples with ketone of which EP is unclear, set this setting shorter.

(4) Enter Final volume and press $[\downarrow]$ key.

Set up the minimum KF reagent to dose around the EP. Typically 0.01mL. For samples with ketone of which EP is unclear, set this setting larger.

(5) Select Detector Mode by $[\leftarrow], [\rightarrow]$ key and press $[\dashv]$ key.

Normal : EP detection for routine measurement

- Oil : Select this mode when titrating using oils or ketones de hydrated solvent or when titration does not end in excess of KF reagent.
- (6) Enter t(stir): stir time before titration and press [→] key.When it takes time to dissolve the sample or an evaporator is connected.
- (7) Enter t(max): limited titration time and press [↓] key.Normal: 0s

When Evaporator is connected, select sample evaporation time.

Titr. Speed(1-6)?

End Time:s? 30

```
Final Volume:mL?
30
```

Detector Mode? Normal→

t(stir):s? 0 t(max):s? 0

- (8) Select Drift Titration by $[\leftarrow], [\rightarrow]$ key and press $[\dashv]$ key.
 - On : the titration cell is being dehydrated.
 - Off : after dehydrated, quickly inject sample.
- (9) Set up Max volume and press $[\downarrow]$ key.

When it reaches the max volume, it terminates titration regardless of endpoint detection.

Drift	Titration?
	O f f \rightarrow

ivi a X	
	20.00

4-4. Result parameter

The MKS-500 has the result parameter for each Method, and desired calculation formula or print format can be selected. Also side reaction and moisture of carrier gas can be compensated.

4-4-1. Calculation formula

Calculation formula can be set up when Method is direct or Indirect. When it is Factor or Calibration, calculation formula is automatically selected according to the Method.

(1) Press [Result] key.

(2) Select "Calculation" by [0] or $[\uparrow]$, $[\downarrow]$ key and press $[\lrcorner]$ key.

Result(0-2)? 0 Calculation

(3) Select calculation formula by [←], [→] key and press [⊥] key.
 mgH2O : detected moisture content

mgH2O : detected moisture content
g→% : water content in weight is calculated in %
g→ppm : water content in weight is calculated in ppm
mL→% : water content in volume is calculated in %
mL→ppm : water content in volume is calculated in ppm

Calc. Formula?

m g H 2 O \rightarrow

Description of calculation formula

	Calculation formula	Description
Method: Direct or Indirect		
mL	Data	Titration volume (mL)
mgH ₂ O	$Data \times F - Blank$	Water content in mg
g→%	$\frac{(Data \times F - Blank)}{Wt1 - Wt2} \times 0.1$	Water content in weight to calculate in % unit
g→ppm	$\frac{(Data \times F - Blank)}{Wt1 - Wt2} \times 1000$	Water content in weight to calculate in ppm unit
mL→%	$\frac{(Data \times F - Blank)}{V \times Dens.} \times 0.1$	Water content in volume to calculate in % unit
mL→ppm	$\frac{(Data \times F - Blank)}{V \times Dens.} \times 1000$	Water content in volume to calculate in ppm unit
When Method: Factor		
g→mg/mL	$\frac{(Wt1-Wt2)\times Conc}{Data}$	To obtain factor of titration solution (mg/mL)
When Method: Calibration		
g→%	$\frac{(Data \times F - Blank)}{Wt1 - Wt2} \times 0.1$	Water content in weight to calculate in % unit

Data	Titration Volume of Karl Fischer reagent (mL)
F	Factor of Karl Fischer reagent (mg/mL)
Blank	Blank level (see next page for details)
Wt1	Sample weight (sample+sampler; g)
Wt2	Sample weight (sampler; g)
V	Sample size (mL)
Dens.	Density of the sample (g/mL)
Conc	Concentration of factor reagent (%)
4-4-2. Blank setup

Blank level can be set up when Method is Direct or Indirect. Water content can be compensated when the sample generates side reaction or when Evaporator is connected.

Blank is not used in normal titration.

- (1) Press [Result] key.
- (2) Select "Blank" by [1] or $[\uparrow]$, $[\downarrow]$ key and press $[\downarrow]$ key.
- (3) Select Blank (water compensation) by [←], [→] key and press [↓] key.
 Yes : compensate water content
 - No : no compensation
- (4) Enter unit time and press [↓] key.
 Enter 0s for compensating side reaction. Enter other than 0s for compensation of carrier gas.
- (5) Enter compensated water content and press $[\dashv]$ key.
 - For compensating water of carrier gas, enter the compensated water content of unit time preset at step (4).

Calculation of blank value

 \cdot When Time = 0s

Blank = Value

 \cdot When Time \neq 0s

Blank = (Value/Time) × M.Time (titration time: s)

R e s u l t (0 – 2) ?	1
В	lank

Blank? Yes→

Time:s? 0

Value:mg? 0

4-4-3. Print format

Desired print format can be selected according to the purpose of resulting data.

(1) Press [Result] key.

(2) Select "Report" by [2] or $[\uparrow]$, $[\downarrow]$ key and press $[\downarrow]$ key.

- (3) Select print format by $[\leftarrow], [\rightarrow]$ key and press $[\dashv]$ key.
 - Off : Printout of measurement results is disabled
 - Short : Only measurement results are printed.
 - Middle : Measurement results, sample and titration solution parameters are printed.
 - GLP : Measurement results and GLP items are printed.

For details of printout, see next page.

Note!

See "5-1-1. Printer" for setting printer.

Report

Blank?	
	Short→

	Short	Middle	GLP	Printout (GLP)
Header *1	0	0	0	Model :MKS-500
				S/N :******
				Ver :1.00
				*** R e s u l t ***
Sample No.	0	0	0	Sample No. 01-01
Date	0	0	0	Date 2000/01/08 13:45
Method			0	Method Direct
		_		Sample
Sample name	0	0	0	*************
Sample ID		0	0	
Reagent name		0	0	Reagent Name
				(Write reagent name after printing)
Factor		0	0	Reagent Factor
				5.0000 mg/mL
Calculation formula	0	0	0	Calc. No. g>%
Sample Size *2	0	0	0	Wt1 5.000 g
				(Vol 5.000 mL)
				Wt2 0.000 g
				(Dens 1.000 g/mL)
				Net 5.000 g
				(Conc 100.0 %)
Blank value			0	Blank 0.000 mg
Measurement results	0	0	0	Result 5.0000 mg
				1.0000 mL
				0.1000 %
Mesauring time			0	Titr.Time 00:02:15
Operator's name			0	Operator <u>***********</u>

*1: Printed out when power is turned on and Method is changed.

*2: Sample size changes by preset Method and calculation formula.

4-5. Sample parameter

The sample parameters of MKS-500 are operator's name, sample name, sample number, sample size, etc. Measurement results are calculated according to the Method and parameters for titration, results and sample. The parameter of step (6) and after changes according to the Method and calculation formula.

(1) Press [Sample] key.

(2) Select the operator's name by numeric key or by $[\leftarrow], [\rightarrow]$ key and	Operator?
press [↓] key.	$0 \rightarrow$
	K E M 0 1
(3) Enter sample No. and press []] key.	Sample No.?
	0 1 - 0 1
(4) Select sample name by numeric key or by $[\leftarrow], [\rightarrow]$ key and press $[\downarrow]$	S.Name?
key.	$0 \rightarrow$
	Sample01
(5) Enter sample ID and press [↓] key.	Sample ID
(6) Enter weight of Wt1 (sample+sampler) and press $[]$ key.	Weight1:g?
	5.000
(7) Enter Wt2 (sampler) and press [] key.	Weight2:g?
	0.000
(8) Enter sample volume: mL and press $[\downarrow]$ key.	Volume:mL?
	5,000
(9) Enter sample density (g/mL) and press [] key.	Density:g/mL?
	1 0 0 0
	1.000
(10) Enter water concentration (%) of standard reagent and press [, 1] key	Conc.:%?
	100 0
	100.0

Parameter	Range		Me	ethod	
		Direct	Indirect	Factor	Calib
Titration			·		
Titr. Speed	1~6	3	3	3	3
End Time	0~99s	30s	0s	30s	30s
Final Volume	0~9999mL	0.01mL	0.01mL	0.01mL	0.01mL
Detector Mode	Normal/Oil	Normal	Normal	Normal	Normal
t(stir)	0~9999s	0s	0s	0s	0s
t(max)	0~9999s	0s	1200s	0s	0s
Drift Titration	On/Off	Off	Off	Off	Off
Max.Volume	0~100mL	10mL	10mL	10mL	10mL
Result					
Calculation		g→%	g→%	g→mg/mL	g→%
Blank	Yes/No	No	Yes	No	No
Time	0~9999s		1200s		
Value	0~99.99mg		0mg		
Report	Off/Short/	Middle	Middle	Middle	Middle
	Middle/GLP				
Sample		1	T	1	ſ
Operator					
Sample No.	00-00~99-99	01-01	01-01	01-01	01-01
S.Name					
Sample ID					
Weight1	0~99999999g	5.000	5.000	5.000	5.000
Weight2	0~99999999g	0.000	0.000	0.000	0.000

4-6. Default value of prameters

4-7. Operating stirrer

4-7-1. On/Off stirrer

Note!

Do not turn on or off the stirrer during pre-titrating or drift titrating in order to avoid excessive titration.

When [Stirrer] key is pressed, the stirrer is turned $On \rightarrow Off$ or $Off \rightarrow On$.

4-7-2. Setting stir speed

(1) When [Stirrer] key is pressed for more than 2 seconds, the display changes as below and stir speed can be adjusted.

(2) Select stir speed and confirm by [,] key. Typically select 3 to 5. For viscous sample, select 5. 0 means the stirrer keeps stirring regardless of [Stirrer] key.

5. Description of Setup

The Setup key can set up GLP/GMP requirements including operator's name and sample name as well as output of interface including printer as follow:

Items that can be set up:

- 0. Interface setup
 - a) Printer
 - b) RS-232C
 - c) Balance
- 1. Date and time
- 2. Operator's name
- 3. Sample name
- 4. Serial number and version number
- 5. LCD brightness adjustment
- 6. Calibration setup
- 7. Lock operating keys

5-1. Interface setup

This instrument is equipped with interface for printer, balance and RS-232C. Printer prints out measurement results and conditions. Balance can output sample weight into the instrument. RS-232C can download data to PC by optional data acquisition software (SOFT-CAP).

5-1-1. Printer

Measurement results and conditions can be printed out. The below steps show how to set up a printer.

- (1) Press [Setup] key.
- (2) Use [0] or $[\uparrow]$, $[\downarrow]$ key to select "Interface" and press $[\lrcorner]$ key.
- Setup(0-7)? 0 Interface

- (3) Select "Printer" by $[\leftarrow], [\rightarrow]$ key and press $[\downarrow]$ key.
- (4) Select the printer by $[\leftarrow]$, $[\rightarrow]$ key and press $[\dashv]$ key.

Interface? Printer→

Printer?

 $I D P - \rightarrow$

-Printer that can be chosen-

Item	Printer
IDP-	IDP-100 impact dot matrix printer (made by Kyoto Electronics)
DP-	DP-500 thermal dot printer (made by Kyoto Electronics)
Other	Serial communication printer
	(SEIKO DPU-414, CITIZEN CBM-910, CITIZEN CBM-270, etc.)
Other2	Canon BJM-70 + 98-020-0017 Serial parallel converter (with a cable)

Note!

Other printer than IDP-100 or DP-500 may not print correctly. Select other printer of which digital configuration conforms to the specification of this instrument. -Digital configuration of printer for MKS-500-

- Pin configuration See 15. External I/O interface pin configuration
- 2. Protocol

Baud rate	4800 bps
Parity	none
Data length	8
Stop bit	1 bit

- 3. Data string ASCII
- 4. Printout characters

One line

more than 24 characters (Other printer) more than 48 characters (Other 2 printer)

5-1-2. RS-232C setup

This instrument can output data to a personal computer via RS-232C interface. With the data acquisition software (SOFT-CAP), the exported data can be edited as well as used in commercially available spreadsheet software.

Note!

If communication error occurs frequently, slower the baud rate. For details of RS-232C specification, refer to its manual.

a) SOFT-CAP

This optional software is Windows98/SE/ME/2000/XP/Vista compatible software by which the data can be downloaded to PC via RS232C into Microsoft Excel workbook or stored in CSV format. Also to start or reset Main unit can be controlled by this software once installed in PC.

<Receiving data>

The data acquisition software can download the measurement results as follows:

1) Directly export to Microsoft Excel workbook.

2) Stores data in CSV file so that Lotus 1-2-3 software can be used.

<Sending data>

Once this software is installed, the personal computer can control Main unit by sending start or reset signal.

Note!

For details, refer to the manual for data capture software (SOFT-CAP 98-595-0086).

b) External output via RS-232C

<Introduction>

This instrument is equipped with RS-232C which is standard bit serial transmittance of data communication. The RS-232C is standardized for data communication between modem and terminal.

This instrument is set to the terminal mode as a peripheral of personal computer. Most of the RS-232C interface equipped in commercially available personal computer are set to terminal mode, however, data communication is available even among peripherals. (Some computers can switch the mode between modem and terminal.)

<Signal level>

It conforms to JIS X5101.



The above bit pattern shows character "A"(41H of ASCII code).

<Connector>

The connector conforms to JIS X5101 male 9 pins and its pin assignment is as below:

Pin number	Signal name	Direction
2	RXD (Receive Data)	IN
3	TXD (Transmit Data)	OUT
4	DTR (Terminal Ready)	OUT
5	SG (Signal Ground)	
6	DSR (Data Set Ready)	IN
7	RTS (Request to Transmit)	OUT
8	CTS (Can Transmit)	IN

[Caution]

TXD and RXD data signals are negative logic while CTS, RTS, DSR and DTR are positive.

<Data I/O via RS-232C>

For details of RS-232C commands and data format, refer to the separately available manual for RS-232C.

b) Baud rate setup

- (1) Press [Setup] key.
- (2) Use [0] or $[\uparrow]$, $[\downarrow]$ key to select "Interface" and press $[\lrcorner]$ key.

(3) Select "RS232C" by $[\leftarrow], [\rightarrow]$ key and press $[\downarrow]$ key.

(4) Select data speed by $[\leftarrow], [\rightarrow]$ key and press $[\downarrow]$ key.

Setup(0-7)? 0
Interface
Interfece?
RS-232C→
Baud Rate?
$9~6~0~0 \rightarrow$

Other communication conditions are fixed as shown below.

· Parity no	ne
-------------	----

|--|

• Stop bit 1 bit

5-1-3. Balance setup

When the MKS-500 is connected to a balance, the sample weight can be input direct from the balance. The below steps show how to set up a balance.

(1) Press [Setup] key.

(2) Use [0] or $[\uparrow]$, $[\downarrow]$ key to select "Interface" and press $[\neg]$ key. S e

(3) Select "Balance" by $[\leftarrow]$, $[\rightarrow]$ key and press $[\downarrow]$ key.

(4) Select the balance by $[\leftarrow]$, $[\rightarrow]$ key and press $[\downarrow]$ key.

Setup(0-7)? 0 Interface

Interface? Balance→

Balance? $\mathsf{K} \to \mathsf{M} \to \mathsf{K}$

- Balance that can be connected to MKS-500 -

Balance maker or brand	Type of Balance
KEM	Kyoto Electronics
	AE-118DR, etc.
Mettler	Mettler Toledo balance
A&D	A&D balance
Shimadzu	Shimadzu Corp. balance
Sartorious	Sartorius Inc. balance

Note!

Some other balance when connected may not output sample weight correctly.

For details, contact your local dealer.

5-2. Date and time setup

The MKS-500 is equipped with clock function, and can print out measurement date and time. The following description explains how to set up display and time adjustment.

5-2-1. Display format

- (1) Press [Setup] key.
- (2) Use [1] or $[\uparrow]$, $[\downarrow]$ key to select "Date&Time" and press $[\neg]$ key.
- (3) Select "Display" by $[\leftarrow]$, $[\rightarrow]$ key and press $[\downarrow]$ key.
- (4) Select the balance by $[\leftarrow]$, $[\rightarrow]$ key and press $[\dashv]$ key.

- Display format that can be selected -

Format	Example of printout	
DD/MM/YYYY	23/01/2000	
YYYY/MM/DD	2000/01/23	
MM/DD/YYYY	01/23/2000	

5-2-2. Date and time setup

- (1) Press [Setup] key.
- (2) Use [1] or $[\uparrow]$, $[\downarrow]$ key to select "Date&Time" and press $[\lrcorner]$ key.

(3) Select "Date&Time Set" by $[\leftarrow]$, $[\rightarrow]$ key and press $[\downarrow]$ key.

(4) Present time is shown. Press $[\downarrow]$ key.

(5) Enter date and press $[\downarrow]$ key.

(6) Enter time and press $[] \downarrow$] key.

Setup(0-7)? 1 Date&Time

Date&Time? _____Display→

Display? YYYY/MM/DD \rightarrow

Setup(0-7)? 1 Date&Time

Date&Time? Date&Time Set→

Date&Time: 2000/02/23 15:42

DateYYYY/MM/DD? 2000/02/23

Time?

15:42

5-3. Registration of operator's name

Up to ten operator names can be registered in order to conform to GLP/GMP requirement. (Up to fifteen letters can be input for operator name.) The operator's name is automatically printed with resulting data.

(1) Press [Setup] key.	
(2) Use [2] or $[\uparrow]$, $[\downarrow]$ key to select "Regist. Operator" and press $[\downarrow]$ key.	Setup(0-7)? 2
	Regist. Operator
(3) Select "Regist.No." by $[\leftarrow], [\rightarrow]$ key and press $[\downarrow]$ key.	Regist. No.(0-9)?
	0
(4) Enter operator's name and press [→] key.	Operator? A
	K E M 0 1

On the display for operator's name entry, "A", "a" and "1" are shown at the upper right corner of display. Enter characters as follows:

- (1) Press [Disp.] key and select entry mode. Each time [Disp.] key is pressed, entry mode changes as "Capital letter (A)→Small letter (a)→Numeral (1)→Capital letter (A)".
- (2) Enter characters. After entries are finished, press [\downarrow] key.

Example: To enter [C], set the mode to capital letter and press [7^{ABC}] 3 times.

To enter	[2], set the mode to numeral and	press [2 ^{RST}]	once.
----------	----------------------------------	---------------------------	-------

Key	Numeric mode	Capital (A) mode	Small (a) mode
0 ^{Blk}	0	Space	Space
1^{STU}	1	S→T→U→S	s→t→u→s
2^{VWX}	2	$V \rightarrow W \rightarrow X \rightarrow V$	$V \rightarrow W \rightarrow X \rightarrow V$
3^{YZ}	3	Y→Z→Y	y→z→y
$4^{ m JKL}$	4	$J \rightarrow K \rightarrow L \rightarrow J$	j→k→l→j
5 ^{MNO}	5	$M \rightarrow N \rightarrow O \rightarrow M$	$m \rightarrow n \rightarrow o \rightarrow m$
6 ^{PQR}	6	$P \rightarrow Q \rightarrow R \rightarrow P$	$p \rightarrow q \rightarrow r \rightarrow p$
7^{ABC}	7	A→B→C→A	a→b→c→a
$8^{\rm DEF}$	8	$D \rightarrow E \rightarrow F \rightarrow D$	$d \rightarrow e \rightarrow f \rightarrow d$
9^{GHI}	9	G→H→I→G	g→h→i→g
.()		$(\rightarrow) \rightarrow ($	$(\rightarrow) \rightarrow ($
+/×	-	$+ \rightarrow / \rightarrow \times \rightarrow +$	$+ \rightarrow / \rightarrow \times \rightarrow +$

5-4. Sample name

Up to ten sample names can be registered in order to conform to GLP/GMP requirement. (Up to fifteen letters can be input for sample name.) The sample name is automatically printed with resulting data.

(1) Press [Setup] key.

(2) Use [3] or $[\uparrow]$, $[\downarrow]$ key to select "Regist. Samp. Name" and press $[\downarrow]$	Setup(0-7)? 3
key.	Regist.Samp.Nam
	е
(3) Select "Regist.No." by $[\leftarrow], [\rightarrow]$ key and press $[\downarrow]$ key.	Regist. No.(0-9)?
	0
(4) Enter sample name and press [] key. *1	Operator? A
	Sample001

*1 Regarding how to enter sample name, refer to registration of operator's name.

5-5. Serial number and version number

The MKS-500 stores serial number and version number. These numbers are necessary for repair or technical inquiry.

(1) Press [Setup] key.

(2) Use [4] or $[\uparrow]$, $[\downarrow]$ key to select "Serial/Version" and press $[\lrcorner]$ key.

Setup(0-7)? 4 Serial/Version

(3) The serial number and version number are shown.

Serial	: N D 0 1 2 3 4
Version	:1.00

5-6. LCD brightness adjustment

The brightness of LCD can be adjusted by changing the contrast as follows:

(1) Press	[Setup]	key.
-----------	---------	------

(3) Adjust contrast by numeric key and press $[\dashv]$ key.

Setu	p(0-7	7)?	5
	LCD	Contr	ast
LCD	Cont	rast	3

5-7. Setup conditions for calibration

The MKS-500 is equipped with self check function in order to conform to GLP/GMP. The below chart shows the steps to set up necessary conditions.

(1) Press [Setup] key.		
(2) Use [6] or $[\uparrow]$, $[\downarrow]$ key to select "Calibration" and press $[\neg]$ key.		Setup(0-7)? 6
		Calibration
(3) Input theoretical value (%) and press $[\downarrow]$ key.	*1	STD Value:%?
		1.00
(4) Input permissible error (%) and press [↓] key.	*2	Permis.Error:%?
		0.10

 *1 Enter theoretical value in % unit. Typical standard substances are shown below: Water standard 10(mg/g) 1.00% Sodium tartrate dihydrate 15.66%

Pure water 100.00%

*2 Enter permissible error in % unit. If calibrated results are within the permissible error, "OK" is judged, and if not, "NG" is judged.

5-8. Parameter lock function

The MKS-500 is provided with the parameter lock function to prevent erroneous change of active parameter.

- (1) Press [Setup] key.
- (2) Use [7] or [\uparrow], [\downarrow] key to select "Lock" and press [\downarrow] key.
- (3) Use $[\leftarrow]$, $[\rightarrow]$ key to select "Set \rightarrow " and press $[\downarrow]$ key.
- (4) Input password with numeric key and press $[\Box]$ key.
- (5) Input password with numeric key again and press [] key.

Setup(0-7)?	7	7
		‹

Lock? Set→

Set Password

Confirm password

6. Description of Function

By [Function] key, recalculation or data processing by auto statistics and instrumental check can be performed. The functions by this key are as follows:

- Setup items -
 - 0. Reagent factor and confirmation
 - 1. Recalculation
 - 2. Auto statistics
 - 3. Deletion of data
 - 4. Calibration (History)
 - 5. Initialization of memory

6-1. Reagent factor and confirmation

The MKS-500 stores KF reagent factor, and uses the value for calculation in measurement. The factor can be obtained by Method Factor.

(1) Press [Function] key.

(2) Select "Reagent Factor" by [0] key or $[\uparrow]$, $[\downarrow]$ key and press $[\lrcorner]$ key.

Function(0-5)? 0 Reagent Factor

(3) To change the factor, input the value and press [→] key.To confirm the factor, just press [→] key.

Factor:mg/mL? 5.000

6-2. Re-calculation

The MKS-500 can store up to 100 measurements per each Method. If a wrong sample size is input, the correct measurement results can be obtained by recalculation.

(1) Press [Function] key. (2) Select "Re-calculation" by [1] key or $[\uparrow], [\downarrow]$ key and press $[\downarrow]$ key. Function(0-5)?1 Re-culculation Re-calc No.? (3) Enter the lower sample number for re-calculation and press [] key. (0 - 29)?29(4) Titration volume is shown. Press $[\downarrow]$ key. Titr. Volume: 2.000mL Moisture: (5) Water content is shown. Press $[] \downarrow$ key. 10.000mg (6) Calculation formula is shown. Press $[\downarrow]$ key. Calc. Formula $g \rightarrow \%$ Time:s? (7) Enter unit time and press $[\downarrow]$ key. * This may not appear depending on setting. 0 (8) Enter water content and press $[\downarrow]$ key. Value:mg? * This may not appear depending on setting. 0 (9) Enter Wt1 and press $[\downarrow]$ key. Weight1:g? * This may not appear depending on setting. 5.000(10) Enter Wt2 and press $[\downarrow]$ key. Weight2:g? * This may not appear depending on setting. 0.000Volume:mL? (11) Enter sample size V and press $[\downarrow]$ key. * This may not appear depending on setting. 5.000 (12) Enter sample density D (g/mL) and press $[\downarrow]$ key. Density:g/mL? * This may not appear depending on setting. 5.000 Conc.:%? (13) Enter concentration of standard and press [] key. * This may not appear depending on setting. 100.0 (14) The results of recalculation will appear. Result: Press [Print] for printout of the results. 0.2000%

6-3. Statistics

The deviation or fluctuation of measurement results can be evaluated by auto statistics.

Number of data	:	The number of data used for statistics.
Mean value	:	The average value of the data
Standard deviation (SD)	:	This is obtained from the root of probability variance, and generally
		used for evaluation of the deviation of data.
Relative standard deviation (RSD)	:	This is the coefficient variance, and generally used for evaluation of
		the deviation of data.

(1) Press [Function] key.

(2) Use [2] or $[\uparrow]$, $[\downarrow]$ key to select "Auto Statistics" and press [,] key.	Function(0-5)? 2
	Auto Statistics
(3) Select data printout or not (On/Off) by $[\leftarrow], [\rightarrow]$ key and press $[\downarrow]$ key.	Data Print?
	$O f f \rightarrow$
(4) The results and mean value after statistics appear. Press $[-]$ key.	Result: 10
	Mean: 2.456
(5) The SD and RSD appear. Press [Print] key to print out the results of	SD : 0.0246
statistics.	RSD : 1.0000

6-4.Deletion of data

The MKS-500 can delete the results of wrong measurement by auto statistics.

- (1) Press [Function] key.
- (2) Use [3] or $[\uparrow]$, $[\downarrow]$ key to select "Data Deletion" and press $[\lrcorner]$ key.

(3)	Enter	the	data	number	to	be	deleted	and	press	[]	key.
-----	-------	-----	------	--------	----	----	---------	-----	-------	----	------

(4) The results and mean value after statistics appear. Press $[\dashv]$ key.

1.000

20*

6-5. History of calibration

The MKS-500 stores the history of calibration to conform to GLP/GMP. The history can be checked by this function.

(1) Press [Function] key.

(2) Use [4] or \uparrow , \downarrow key to select "Calib (History)" and press \downarrow key.

<Description> Data No .: date results Data (criterion)

6-6. Initialization of memory

The MKS-500 can initialize preset parameters and settings in order to reset the system to default value.

- (1) Press [Function] key.
- (2) Use [5] or $[\uparrow]$, $[\downarrow]$ key to select "Memory Clear" and press $[\downarrow]$ key.

Function(0-5)? 5 Memory Clear

(3) Use $[\leftarrow], [\rightarrow]$ key to select data to be initialized and press $[\downarrow]$ key. Measured Data : Deletion of measurement data Method Para : Initialization of measuring conditions

All Parameters : Deletion of data and initialization of conditions

(4) Use $[\leftarrow], [\rightarrow]$ key to select "Yes" and press $[\downarrow]$ key.

The data chosen at step 3 will be initialized.

Memory Clear?

All Parameter \rightarrow

Execute? ${\sf Y} \mathrel{e} {\sf s} \rightarrow$

(3) The check record appears. The data can be scrolled by $[\uparrow], [\downarrow]$ key.

Function(0-5)? 4 Calib (History)

0:2000/01/23 OK 0.9981 (1.0000)

7. Maintenance

7-1. Karl Fischer grease

Lightly apply KF grease around the sliding area between the electrode and its holder or sample inlet and its port plug.



Check once a week to see if the sliding area is locked. If it is locked, apply KF grease lightly. Do not put too much grease since the water in the grease affects drift level, making the cell difficult to be dehydrated.

Caution!

If checking the sliding area is neglected, the KF grease may be solidified. Once the area is solidified, use alcohol to dissolve the solid.

7-2. Replacement of silica gel

When the silica gel changes to reddish color, its hygroscopic capacity goes down.

Replace it with new gel.



Lightly apply KF grease around the sliding area between desiccant tube and its holder.

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.

7-3. Replacement of KF reagent

The Karl Fischer reagent can be used up once its factor is measured before measurement, however, if its factor is significantly reduced, it is recommended to replace it. After all the reagent in the burette is discharged, replace it with [APB] key.

7-4. Cleaning the electrode

When the electrode is deteriorated and the potential becomes unstable, causing fluctuating measurement results, rinse the electrode with concentrated nitric acid and clean with alcohol (methanol, etc.) and then, wipe off with clean gauze and dry.



7-5. Cleaning titration flask

- (1) Drain out the solvent in the flask.
- (2) Turn the flask lid 90 degrees counter-clockwise and open the holding lever to remove the flask.



- (3) Take off the port plug and clean the flask with neutral detergent.
- (4) After the flask is dried by a dryer, cool it in a desiccator.

7-6. Cleaning the burette unit

(1) Drain out the reagent in the burette and remove the reagent bottle.



(2) Fill the burette with methanol and discharge. Repeat a few times.



(3) Remove the burette cover and loosen the burette holder, and press $[\Delta]$ key to remove the burette.



(4) Pull out the piston head, and clean the burette and piston head with methanol.



- (5) Reassemble the burette and fill it with methanol, and discharge.
- (6) Discharge all of the methanol in the burette.

Caution!

The Karl Fischer reagent will be crystallized if left over for more than a week. It may cause clogging of the titration nozzle or the tube line, causing eventual leaking.

Discharge the reagent in the burette and clean it before it is stored.

7-7. Replacement of piston head

The piston head may be deteriorated by friction after excessive use for a long period of time. It is time to replace the head if reagent permeates through the lower part of the head after the reagent is delivered in the burette.



(1) Remove the piston head with a piston rod.



(2) Squeeze in the piston rod into the new head.



(3) Return the head into the burette cautiously not to scratch the head.



7-8. Replacement of switching valve

Replace the cock if reagent leaks from it due to crystallization of reagent or after use for an extended period of time.

- (1) Detach all the tube lines connected to the cock.
- (2) Undo 4 screws to remove the cock.
- (3) Install the new cock with the marking in front, and fasten by the screws.



(4) Connect all the tube lines.

7-9. Replacement of titration nozzle

- (1) Disconnect the tube from the joint.
- (2) Disconnect the joint from the nozzle.
- (3) Insert the new nozzle into the joint.
- (4) Connect the joint.



7-10. Replacement of tube

Replace the tube if leaking is observed at the joint. Use the supplied wrench for the switching valve. Use fingers to tighten other joints.

Caution!

When a tube is replaced, connect the tube with its contact part vertical. Also care should be taken not to attach any dirt or stains on the junction in order to avoid leaking.

8. Troubleshooting

8-1. Error messages and remedies

Error message	Trouble	Possible cause and remedies
Regist.Over!	The electrode detects very high	• Remove any foreign particle attached to the
Check Electrode	resistance.	electrode.
		• The electrode may be contaminated by extracting
		solvent.
		• Check electrode if any lead is broken.
		• Reduce sample amount if liquid resistance of
		sample is high.
Electrode Open!	No electric current flows into	• Check any loose connection of electrode.
Check Electrode	the electrode.	• Check any broken electrode lead.
		• Check electrode if contaminated by extracting
		solvent.
Meas. Over!	It reaches maximum present	• Reduce sample amount.
Check Samp. Size	volume.	• Increase reagent factor.
Over Titr.!	Iodine in extracting solvent is	• Raise potential by adding water.
Inject Water	excessive.	• Slow down titration speed to avoid excessive
		iodine.
Parameter NG!	Titration is started with wrong	• Check if both End time=0 and t(max)=0 are
Correct Setting!	parameters.	selected.
		• Check if t(stir)>t(max) is selected.
Burette Error!	The switching valve does not	• Check if the cock is locked.
Check burette	work.	• Check the cock sensor.
Preamp Error!	Preamplifier is not working.	• Contact your local dealer or sales rep.
Contact Dealer		
Pre-titr. Over	Pre-titration reached	• Use new extracting solvent.
Check solvent	40mL.	• Re-start pre-titration.
D. Mode Error!	Detection mode is not	• Change detection mode.
Check D. Mode	appropriate.	
V/F Error	Preamplifier is not working	• Contact your local dealer or sales rep.
Contact Dealer	properly.	
Data not exist	The relevant titration data is	• Press any key.
Press any key	not available.	

8-2. When Stirrer does not work



8-3. When the burette does not work properly



8-4. When air bubble exists in the burette



How to degas KF reagent

Air bubbles are generated when KF reagent is moved from a dark cool place to a warm room. Use an aspirator or leave it after well shuffled.

8-5. When titration ends with excessive KF reagent



Note!

Change the extracting solvent or add masking agent to the sample which contains substance for side reaction like ketone, aldehyde, amine, etc.

8-6. When titration does not reach an endpoint or takes too much time



Note!

Change the extracting solvent or add masking agent to the sample which contains substance for side reaction like ketone, aldehyde, amine, etc.

8-7. When reproducibility of measurement is poor



8-8. When crystallization or clogging due to KF reagent is observed

The Karl Fischer reagent crystallizes as the time goes by. When the titration nozzle or switching valve is clogged, follow below steps for cleaning:

8-8-1. Titration nozzle is clogged

- (1) Remove the titration nozzle.
- (2) Dip it in 60°C warm water or clean in an ultrasonic cleaner.
- (3) After clogging is removed, clean with alcohol.
- (4) After dried, connect the nozzle and tube.

8-8-2. Switching valve is clogged

- (1) Disconnect the connecting tubes to the cock. If KF reagent flows out, wipe it off with tissue paper.
- (2) Fill the cock with ethanol by a dripping pipette, and leave it for more than 30 minutes.
- (3) Remove the ethanol in the cock by the dripping pipette.
- (4) Repeat step (2) and (3).
- (5) Reassemble the connections.

<u> Warning!</u>

The Karl Fischer reagent is a toxic solution. A good care should be taken when handle the reagent. If splashed reagent touches your skin, wash it away with running water.

ACaution!

When KF reagent is filled and left over for more than a week, check any crystallization, and if any is found, clean it immediately. Do not attempt to discharge reagent without cleaning.
8-9. Glass sliding area is solidified

Follow below steps if glass sliding area is locked by solidified KF grease.

- (1) Drain out the liquid in titration flask.
- (2) Heat the locked area by a hair dryer to remove the grease.

Do not attempt to remove the solidified part by tapping or excessive force.

The titration flask and electrode may be broken and hurt yourself.

9. Display messages and meaning

Display message	Meaning
Auto Statis.	Automatic statistical calculation by the processor
Auto Statistics	
Balance	Select a balance to be connected. When [-] key is pressed, the value is stored.
Baud Rate	Communication speed via interface
Blank	Compensation of water content by side reaction or from carrier gas
Calculation	Calculation parameter
Conc	Abbreviation of concentration of water of standard substance
Data	Measurement results
Data Bits	Communication data bit number
Data Deletion	Erase data
Data List	Sampling list of data per unit time
Data Not Exist	Error message when data is not available.
Data Print	Select to print or not resulting data in auto statistics
Date&Time	Calendar date and hour
Density	Density of a sample
Detector Mode	Detecting mode: typically "normal" but for sample of high resistance like
	petroleum, use "Oil".
DP-	DP-500 thermal dot printer made by Kyoto Electronics
Drift	Condition in drift titration with reagent added to excessive water in titration vessel.
Drift Titration	Select drift titration to dehydrate the permeated water
End Time	Time to wait for endpoint, typically 30 second.
Factor	Factor of reagent or method for measuring Factor
Function	A software device to execute data processing or event
GLP	Abbreviation of requirements by Good Laboratory Practice
IDP-	IDP-100 impact dot printer made by Kyoto Electronics
Interface	Digital connecting device to RS232C, printer or balance
Low Sample No.	Individual sample number in low order (range: 00 to 99)
Max. Volume	Maximum titration volume to terminate the titration
Mean	Average amount of data
Meas. No.	Ordinal number of measurement
Measurment	Measurement in progress
Memory Clear	Initialize data in memory
Method	Measurement form
	MKS-500 has 4 Methods.

Display message	Meaning
Moisture	Water content
Operator	A name of operator for the instrument
Off	Something not turned on or set
On	Something turned on or set
Other	Printer not specified
Other 2	Printer for A4 size printout
Parity	A part of communication protocol
Permis. Error	Permissible error in check measurement
Potential	Electric potential in titration vessel
Press [Start] Key	Press [Start] key
[Pre-Titr.]	Initial display in reset condition before starting titration
Pre Titration	Display of pre-titration in progress
Printer	Select and set up a printer
Purge	Purge dry or setting a number of purge times
Reagent Factor	Setting the Factor of reagent
Re-calculation	Re-calculate data by changing formula, sample size or constant
Report	Print format to select
Result	Measurement results or setting data calculation, blank, printout
RSD	Abbreviation of relative standard deviation, same as CV%
RS-232C	Interface for external output or communication
S	Abbreviation of second
Sample	Sample parameter
Sample In	Press [Start] key after sampling
Press [Start] Key	
Sample No.	Ordinal number of a sample consists of high order (group number) and low order
	(individual) number, each range; 00-99
SD	Standard deviation
Serial No.	Individual number of production of the instrument
Setup	A software device for data processing or control
Short	Short fixed printing format
Size	Sample parameter in weight or constant, etc.
STD Value	Value of standard substance for precision check
Stir. Speed	The stirring speed by stir bar
Stir. Time	Time set on t(stir). Stirring time before titration starts

Display message	Meaning			
Titration	Titration parameter			
Titr. Speed	Titration speed			
Titr. Volume	Titration volume			
t(max)	Time to terminate titration			
t(stir)	Stirring time before titration starts			
Volume	Amount of sample or titration in mL			
Wt1	Weight of sample and sampler			
Wt2	Weight of sampler after sample is delivered			
0s	Zero second. T(max)=zero means no limit time.			
(Cursor brinks)) Enter characters by numeric key, dot or [Disp.] key			
(Cursor lit)	lit) Select by $[\uparrow], [\downarrow], [\leftarrow], [\rightarrow]$ key.			
_ (under-bar)	Parameter shown on display			

10. Others

10-1. Parts list





Optional parts and components

Part Code	Part name	Qty	Remark	Sketch
ADP-511S	Evaporator	1	Temp.: room ~ 300°C	
ADP-512	Evaporator for ore	1	Temp.: room ~ 130°C room ~ 1000°C	
ADP-512S	Evaporator for high temp.	1	Temp.: room ~ 1000°C	
ADP-513	Evaporator for oil	1	Temp.: room ~ 200°C	
ADP-344	Heat extractor for sugar samples	1	Temp.: room ~ 60°C	
98-350-0001	Conversion connector	1	9P-25P	and a second sec
IDP-100-10 IDP-100-11 IDP-100-12	IDP-100 series impact dot matrix printer	1 1 1	100 V 120 V 230 V with cable	
98-740-3002	N-shape flask	1	50~130mL	
98-740-3011	Bent type sampler	1	Bent type	

Part Code	Part name	Qty	Remark	Sketch
98-740-3020	C-bent type sampler	1	Sealed type	
98-740-3012	Spoon type sampler	1	For viscous sample	
98-740-3013	Oil sampler	1		
98-740-3014	Liquid gas sampler	1	100mL SUS pressure vessel	
98-740-3026	Straight light sampler	1		
98-740-3030	Sampler for viscous sample	1		
98-740-3024	C-eggplant sampler	1		
98-740-3025	Light sampler	1		
98-740-3031	Pressure sampler for viscous sample	1		
98-500-3166	Syringe (5mL)	1	For cock leaning	
98-500-3225	Syringe (20mL)	1		
98-433-3407	Syringe side stopper	1	With septum	

Part Code	Part name	Qty	Remark	Sketch
98-433-0134	Manual absorb and drain unit	1		Bette holder 1 Bette holder 1 Bette holder 2 Bette holder 2 Bette holder 3 Bette holder 3 Bette holder 3

Reagent and solvent

Part Code	Part name	Qty	Remark
98-812-4008	Composite 5	1	
98-812-4007	Composite 2	1	
98-812-4018	Composite 5K	1	For ketone
98-812-4035	Solvent ML	1	
98-812-4029	Solvent MI	1	
98-812-4039	Solvent MS	1	
98-812-4030	Solvent CM	1	For oils
98-812-4031	Solvent FM	1	For sugar
98-812-4032	Solvent CE	1	For ketone
98-812-4034	Solvent ME	1	For gas
98-812-4048	Water methanol 2	1	250mL
98-812-4044	Water methanol 5	1	500mL
98-812-0001	Water standard	1	Factor 10, 10 amples/set
98-820-3269	Silica gel	1	500g



10-3. External I/O interface pin configuration

The conector confirms to JIS X5101 with 9 pin male type.

(1) RS-232C



(2) Balance



(3) Printer



10-4. Technical data

Type and model	MKS-500 Karl Fischer Moisture Titrator			
Method	Karl Fischer volumetric titration			
Range	1) Water content: 0.1 to 500mg H_2O , 0.001 to 100%			
	2) Titration volume: 0.005 to 100.000mL			
Control method	By the built-in microcomputer			
Endpoint detection	Polar potential detected by twin Pt electrode with liquid resistance compensated			
Endpoint sense	 Setting range: 1 to 99 seconds Potential stat to maintain endpoint 			
Special function	 1) Titration speed control by 3 steps 2) Drift titration : maintain dehydration of titration flask 3) Start delay : range 0 to 9999 seconds 			
Display	 1) 16 digits by 2 lines LCD 2) Display contents: results in mL, mgH2O, concentration and guiding messages 			
Titration method	4 methods1) Direct: direct titration2) Indirect: indirect titration (by evaporator)3) Factor: factor measurement4) Calib: calibration			
Endpoint indication	Display and beep			
Computation	 Concentration calculation Statistics including mean value, SD and RSD Calculation of factor measurement 			
Alarm	Inhibited key entry with error message including excessive titration setup, abnormal polar potential, liquid resistance, etc.			
Power burette	 Cylinder: 10mL, Precision: ±0.015mL, Reproducibility: ±0.005mL Change-over delivery and discharge reagent by switching valve Backlash function 			
Required solvent	Min. 30mL, Max. 100mL (with S-type flask)			
External output	 RS-2332C for printer RS-232C for Balance RS-232C for Personal computer 			
Option	 Printer (IDP-100-10/11/12) Manual absorb and drain unit (98-433-0134) 			
Ambient condotion	Temperature: 5 to 35°CHumidity: less than 85%RH			
Power source	AC100 to 120V and AC200 to 240V, 50/60Hz			
Power consumption	35W			
Dimension	288mmW×468mmD×610mmH			
Weight	Approx 12.5 kg			

10-5. Principle of measurement

Water content in the presence of base and alcohol reacts with iodine and sulfur dioxide quantitatively as follows:

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

Karl Fischer moisture determination is based on this reaction. Add sample solution after the solvent in titration vessel is dehydrated by KF reagent. Quantitative titration of water is complete with KF reagent, of which factor is determined in advance (mgH_2O/mL) by standard water-methanol. Titration is progressed by detecting polar potential of the electrode while controlling titration speed.



10-6. Caution in transport

Caution: When transporting MKS-500 (e.g. send for repairs), be sure to detach the switching valve from base. Failure may result in the breakage of it.

(1) Unloosen the four fastening screws and detach the switching valve.



11. Warranty

- The product you have purchased passed standard test and inspection in plant before shipment. Quality of the product is covered by one-year warranty except consumable parts.
 Some conditions or performance in use may not be covered when the product is in the warranty period.
- 2. For repair or maintenance servicing, please contact your local dealer or distributor where you purchased the product.
- 3. Before you call the dealer, read the manual ("7. Maintenance" and "8. Troubleshooting"), and recheck the trouble.

When you contact your dealer, please notify:

Production No. of your instrument Details of the trouble Person to contact

- 4. Parts and consumables can be purchased from your dealer. Parts for discontinued model will be supplied for further seven years.
- 5. This warranty does not cover claims caused by the followings.
 - (1) Modification by unauthorized person
 - (2) Splashed by or dipped in water (the product is not waterproof)
 - (3) Use under conditions or range other than specified
 - (4) Operated in other method than specified in this manual
 - (5) Dropped or given physical shock
 - (6) Erroneous handling or misuse
 - (7) Use under extraordinary environmental condition
 - (8) Caused by fire, earthquake, lightning or Act of God
- 6. The manufacturer, dealer or distributor of the product will not be responsible for any loss or damage whether economical or physical related directly or indirectly in use of the product.

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