



## K90365 COULOMETRIC KF TITRATOR

### *OPERATION AND INSTRUCTION MANUAL*

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Petroleum Testing & Analysis Instrumentation • Custom Design & Manufacturing



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# **CERTIFICATE OF CONFORMANCE**

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## **AKF5000 Compact Coulometric Karl Fischer Titrator K90365**

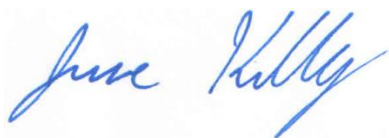
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This certificate verifies that part number K90365, AKF5000 Compact Coulometric Karl Fischer Titrator, was manufactured in conformance with the applicable standards set forth in this certification.

**Specifications:**

ASTM D1533  
ASTM D4928  
ASTM D6304  
IP 386  
IP 438  
API MPMS Chap. 10.9  
BS 60814  
ISO 10101-3  
ISO 10337  
ISO 12937

This unit is tested before it leaves the factory, to ensure total functionality and compliance to the above specifications and ASTM standards. Test and inspection records are on file for verification.



**Jesse Kelly**  
**Application Engineer**  
**Koehler Instrument Company**

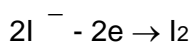
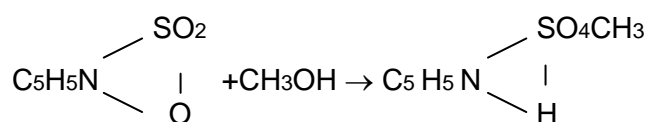
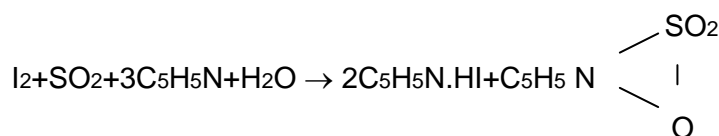


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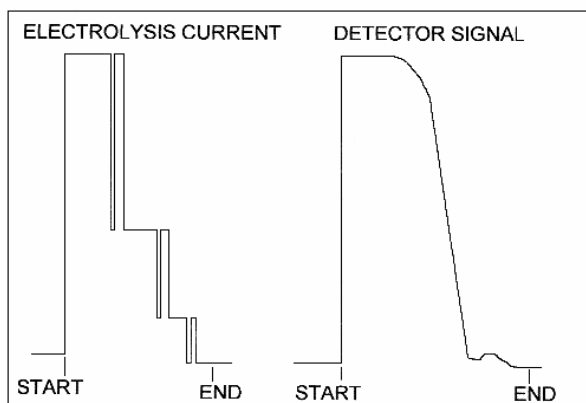
## PRINCIPLE OF MEASUREMENT

Karl Fischer titration is simply a means to measure water content of samples. Modern instruments, such as the AKF5000, use the coulometric principle, whereby the water present in the sample is coulometrically titrated to a predefined end point at which there is a minute excess of free iodine present. Stoichiometrically, 1 mole of water will react with 1 mole of iodine, so that 1 milligram of water is equivalent to 10.71 coulombs of electricity. Combining the coulometric technique with Karl Fischer titration, AKF5000 titrators determine the water content of the sample by measuring the amount of electrolysis current necessary to produce the required iodine. This is an absolute technique which does not require calibration of the reagents.



Using the latest pulse current technology and the patented “ACE” control system, (Patent No.GB2370641), the AKF5000 automatically selects the appropriate titration speed dependent upon the amount of water present in the sample. The titration speed is reduced as the end point is approached, and when the titration is completed the instrument prints out and displays the results.

## SCHEMATIC OF TYPICAL TITRATION



## SPECIFICATIONS &amp; FEATURES

	<b>Koehler AKF5000</b>
<b>Titration method</b>	<b>Coulometric Karl Fischer Titration</b>
<b>Electrolysis control</b>	<b>Patented “ACE” control system (GB2370641)</b>
<b>End point detection</b>	<b>AC polarisation</b>
<b>End point indication</b>	<b>Visual display / print out / acoustic beep</b>
<b>Display</b>	<b>40 character alphanumeric backlit LCD</b>
<b>Measuring range</b>	<b>1µg – 10 mg water</b>
<b>Moisture range</b>	<b>1 ppm – 100 % water</b>
<b>Max. sensitivity</b>	<b>0 • 1µg</b>
<b>Max. titration speed</b>	<b>2 mg per minute</b>
<b>Max. electrolysis current</b>	<b>400 ma</b>
<b>Drift compensation</b>	<b>Automatically controlled</b>
<b>Start delay time</b>	<b>0 – 30 minutes user programmable</b>
<b>Min. titration time</b>	<b>0 – 30 minutes user programmable</b>
<b>Power supply</b>	<b>90-264 VAC, 47-63 Hz, 12V DC car adapter/internal battery</b>
<b>Precision</b>	<b>10 - 100µg ± 3µg, 100µg – 1 mg ± 5 µg, above 1 mg ± 0.5%</b>
<b>Calculation modes</b>	<b>User programmable calculations:- Weight/weight, Volume/density, Volume/volume, Weight/Dilution ratio.</b>
<b>Display format</b>	<b>µg, mg/kg, ppm, %</b>
<b>Print format</b>	<b>µg count plus calculated mg/kg, ppm, % water</b>
<b>Statistics</b>	<b>Upto 99 runs, user programmable</b>
<b>Method storage</b>	<b>10 programmable methods</b>
<b>Sample ID number</b>	<b>User programmable</b>
<b>Printer</b>	<b>42 character high speed thermal printer</b>
<b>Stirrer speed</b>	<b>Microprocessor controlled</b>
<b>Battery life</b>	<b>8 hours running time from fully charged</b>
<b>Charging time</b>	<b>Fully charged 14 hours</b>
<b>Battery low indication</b>	<b>Display &amp; print out indication</b>
<b>Dimensions</b>	<b>10 x 9½ x 5 inches (250 x 245 x 120 mm)</b>
<b>Weight</b>	<b>6 pounds (3 kg)</b>
<b>Carry case</b>	<b>Optional</b>
<b>Calendar / clock</b>	<b>Analysis time &amp; date print out</b>

## UNPACKING & PARTS LIST

Inside the shipping carton you will find a yellow plastic case which contains the glassware and accessories pack and the user manual. The AKF5000 titrator is located underneath the glassware case (or inside the optional carry case if ordered). The mains power lead, power pack and in-car adapter are inside the carry case lid compartment.

Remove all of these items and check that they have arrived undamaged.



After unpacking your AKF5000 check that the glassware/accessories pack contains:-

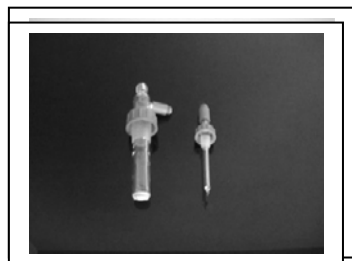
<u>Item</u>	<u>Description</u>	<u>Low Drift Cell</u>
1 pce	Titration Vessel	71- 0102
1 pce	Detector electrode	71- 0103
1 pce	Generator electrode	71- 0104
1 pce	Desiccant tube & cap	71- 0106
1 pack	Injection septa (10)	71- 9507
1 pce	Glass syringe	71- 9508
1 pce	Luer needle	71- 9509
1 bottle	Molecular sieve	71- 9512
1 pce	Stirrer bar	71- 9514
1 pce	Funnel	71- 9515
1 pce	Thermal paper roll	71- 0119

### ACCESSORIES

1 pce	Mains lead	71- 4014 20
1 pce	Mains power pack	71- 401607
1 pce	In Car Adapter	71- 401421
1 pce	User Manual	71- 9517
1 pce	Dust cover	71- 301301
1 pce	Carry case (optional)	71- 201109



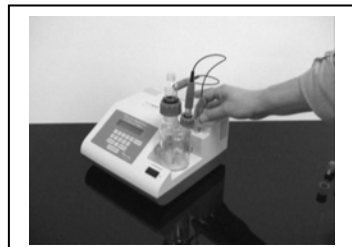
## AKF5000 TITRATION CELL STEP BY STEP ASSEMBLY INSTRUCTIONS



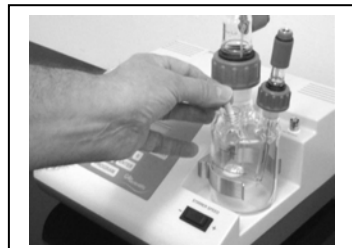
The glass joints do not require Teflon sleeves or grease. The special screw fittings seal the vessel from ingress of moisture from the atmosphere and are easily released, even after several weeks.



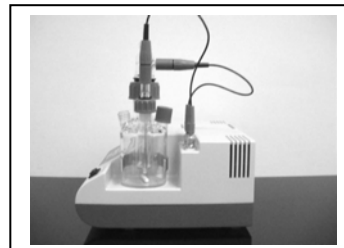
Position electrodes on titration vessel. It is not necessary to overtighten these screw joints.



Connect leads to generator and detector electrodes. Generator electrode lead connects to socket marked "GENERATOR" – detector electrode lead connects to socket marked "DETECTOR"

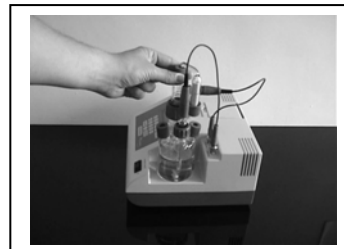


Place stirrer bar inside titration vessel



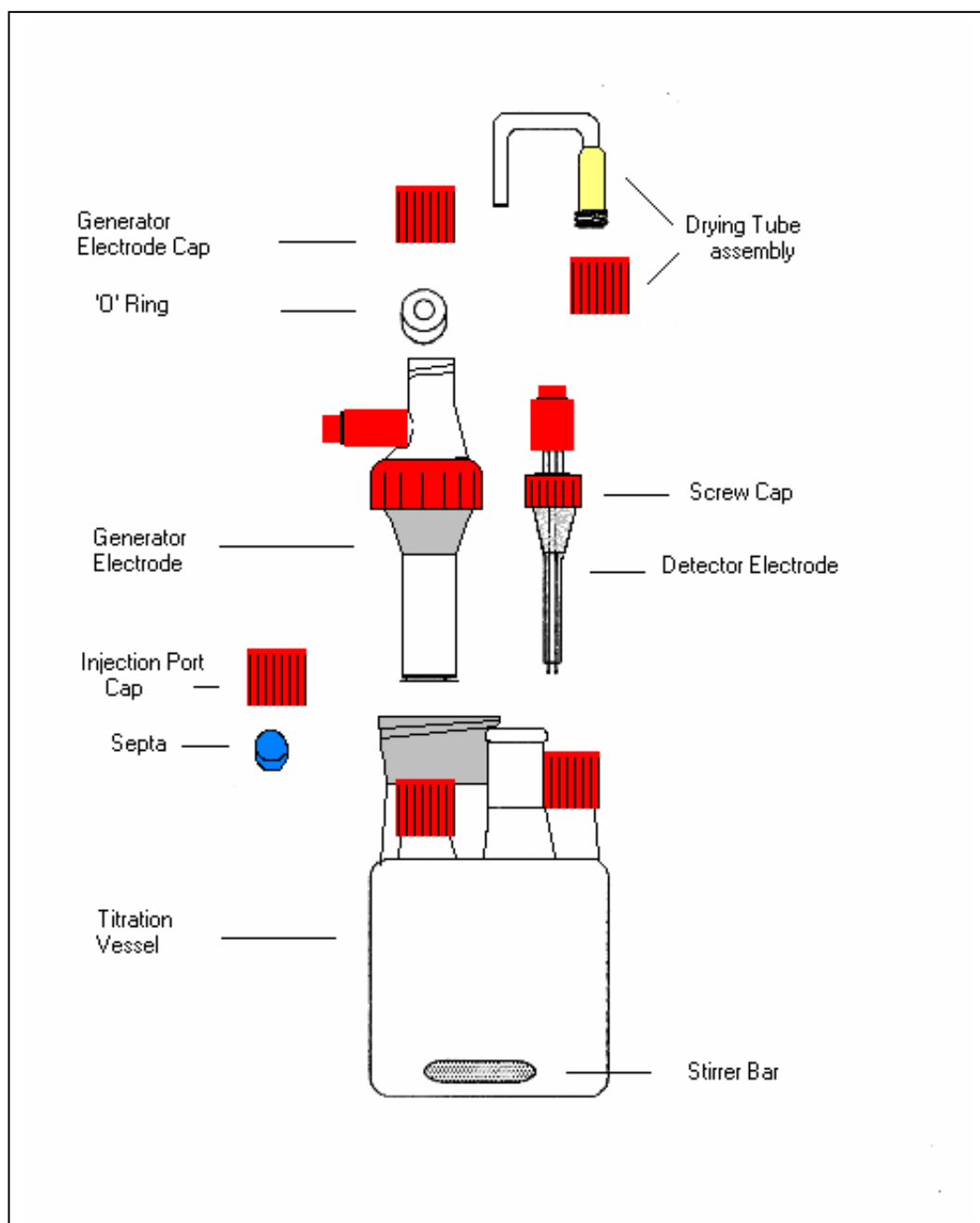
Fit injection septa into plastic screw cap connectors and locate onto injection ports of titration vessel.

Fill drying tube with molecular sieve and locate on top of generator electrode



The assembled titration cell is now ready to be charged with reagents prior to use. Please be sure to assemble correctly every time the cell is cleaned or re-filled. It is recommended that the titrator is **NOT** switched on until after the titration vessel has been charged with reagents. This will avoid damage being caused to the electrodes by the stirrer bar

## AKF5000 TITRATION CELL



## FILLING & CHANGING REAGENTS

The AKF5000 has been designed to operate with all major coulometric Karl Fischer reagents, however optimum performance can be obtained by using Cou-Lo Formula reagents which have been specifically formulated for use with these instruments. For most routine applications 100ml of Formula “A” (anode reagent) and 5ml of Formula “C” (cathode reagent) are used. When analysing Transformer oils, Crude oils and other petroleum products Formula “A” is especially suited as it contains other solvents to improve sample miscibility and solubility.

*(When analysing samples of Ketones, amines or others which may interfere with the reaction it is advisable to use specialised reagents which can be obtained from various suppliers).*



Although reagents can be poured into the titration vessel whilst it is located on the titrator, we recommend that the vessel and electrodes are removed from the instrument whilst this procedure is performed to avoid reagent spillage onto the instrument casing. *(Any spillage onto the instrument casing should be wiped off immediately to avoid damage or staining).*



Remove the drying tube and injection septa. Using the funnel supplied, charge the titration vessel up to the lower line with Formula “A” reagent. For your convenience these reagents are supplied in “single shot” bottles which contain 100ml so it is not necessary to measure any volumes – simply pour in the complete bottle

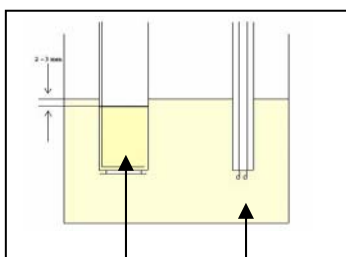


Also using the funnel, charge the inner chamber of the generator electrode with Formula “C” reagent which are supplied in “single shot” 5 ml vials which have “safety snappers” pre-fitted thereby reducing risk to the operator. *(It is not necessary to clean the funnel between reagents.)*



Reconnect the drying tube and injection septa so that the titration vessel is sealed from ingress of atmospheric moisture.

Locate the complete titration vessel onto the titrator and connect the electrode leads onto the appropriate sockets



Cathode Reagent    Anode Reagent

The colours of anode and cathode reagent are only an illustration to show that there are two different liquids. These are not necessarily the exact colours of the reagents being used.

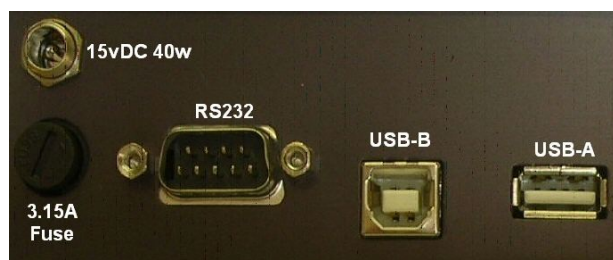
The AKF5000 titrator is now ready to be switched on.

## DESCRIPTION

### Front Panel



### Rear Panel



Power Jack:-	Nominal 15v DC 40w. Supply Input
Fuse:-	3,15amp T type Time delay
RS232:-	Standard 4 wire RS232 Input / Output Port.
USB-B:-	Direct cable to PC / Results Manager Port.
USB-A:-	Removable Flash Memory Store Port.

#### RS232 Port. –


For updating titrator software, connecting to other equipment for remote controlling (software specific protocols to be provided).


#### USB-B Port. –

For connecting directly to PC to export data to Results Manager and enable results to be saved on files for future reference. (See Results Manager User Manual section)

#### USB-A Port. –

For exporting results data to a Removable Flash Drive (memory stick), when running sample tests in the field or remote locations, and subsequent transfer to PC at a later date for loading onto Results Manager files.

To use the memory store, insert Flash Drive into USB port A, titrator will see it and a symbol  will appear on the display.

The symbol will appear blacked out while the drive is being written to and then as  when idle.

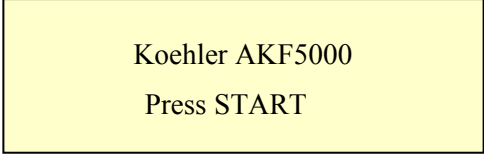
**The drive should only be removed when in the idle mode.**

To read data saved on drive insert it into PC USB port. Start Results Manager, load selected file and then save in the format you require, see Results Manager user manual for full instructions.

There is a separate power ON/OFF switch on the rear panel. Whilst connected to mains power supply, a trickle charge is continuously applied to the battery. It is therefore recommended that the instrument is used from mains power supply whenever possible so that the battery is kept fully charged. Maximum battery operating time is 8 hours continuous use. This may be reduced if repeated high water contents are being determined or if the printer is being continually used.

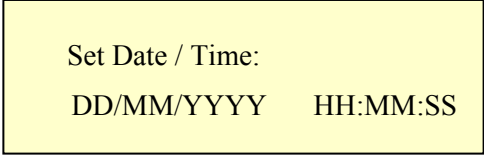
## SETTING THE DATE AND TIME

1. Switch on the AKF5000 and wait for it to display the following:



Koehler AKF5000  
Press START

2. Hold down the **START** key until the AKF5000 displays:-



Set Date / Time:  
DD/MM/YYYY HH:MM:SS

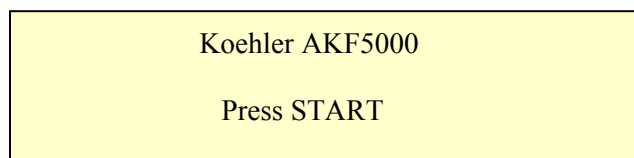
3. Use the  decimal point key to move the cursor across the screen.

4. Use the numerical keys to set / alter the required number

5. When completed press the **ENTER** key to save the changes and exit.

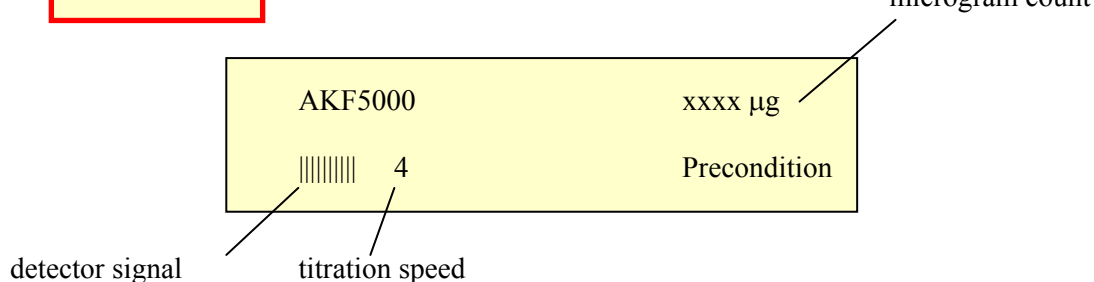
## CONNECTION & START UP PROCEDURE

1. Assemble glassware (see Assembly Instructions)
2. Charge vessel and generator electrode with reagents (see Filling & Changing Reagents)
3. Locate assembled titration vessel onto the AKF5000
4. Connect electrode leads
5. Connect mains lead / power cord
6. Switch ON (using switch on rear panel) and wait for display to show:-

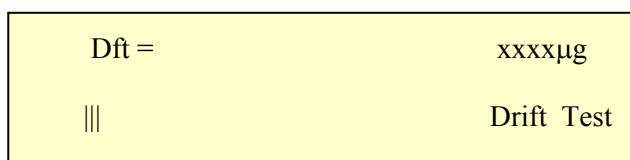


(If required adjust the stirrer speed using the black toggle switch on the front of the instrument.)

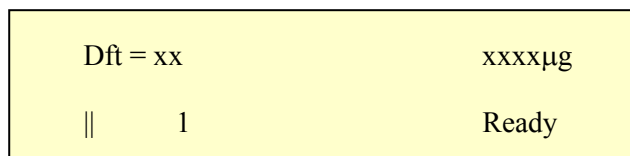
7. Press **START** and allow the instrument to precondition (equilibrate).



At the end of the precondition period the display will show:-



After the initial drift has been calculated, in micrograms of water per minute, the AKF5000 is ready for operation.



The background drift value will usually slowly decrease with time as the titration vessel becomes drier and more stable. Although it is possible for the titrator to be used at high drift values, it is advisable to wait until the drift value is below 20  $\mu\text{g}/\text{minute}$ , and stable, before commencing, especially for low water content samples in the ppm ranges. The lower, and more stable the drift – the more accurate the result.

## PROGRAM FUNCTIONS

When the AKF5000 displays Ready you can enter titration parameters, recall or edit existing methods, enter new method files, etc., by using the **PROGRAM**, **ENTER**, **CLEAR** and **•** keys.

Key Functions:-

**PROGRAM**

press PROGRAM key to scroll through menus listed.

**ENTER**

allows parameter values for the displayed menu to be entered into memory.

**CLEAR**

this key has two functions:

- (1) If you have finished entering data, press CLEAR and the AKF5000 will return to the Ready condition.
- (2) If you have started to enter an incorrect value, press CLEAR and the AKF5000 will display the previously stored value, you can now select the correct value and press ENTER.

**•**

decimal point key allows scroll through of individual parameters in each menu. When the required parameter is displayed, press ENTER to store it.

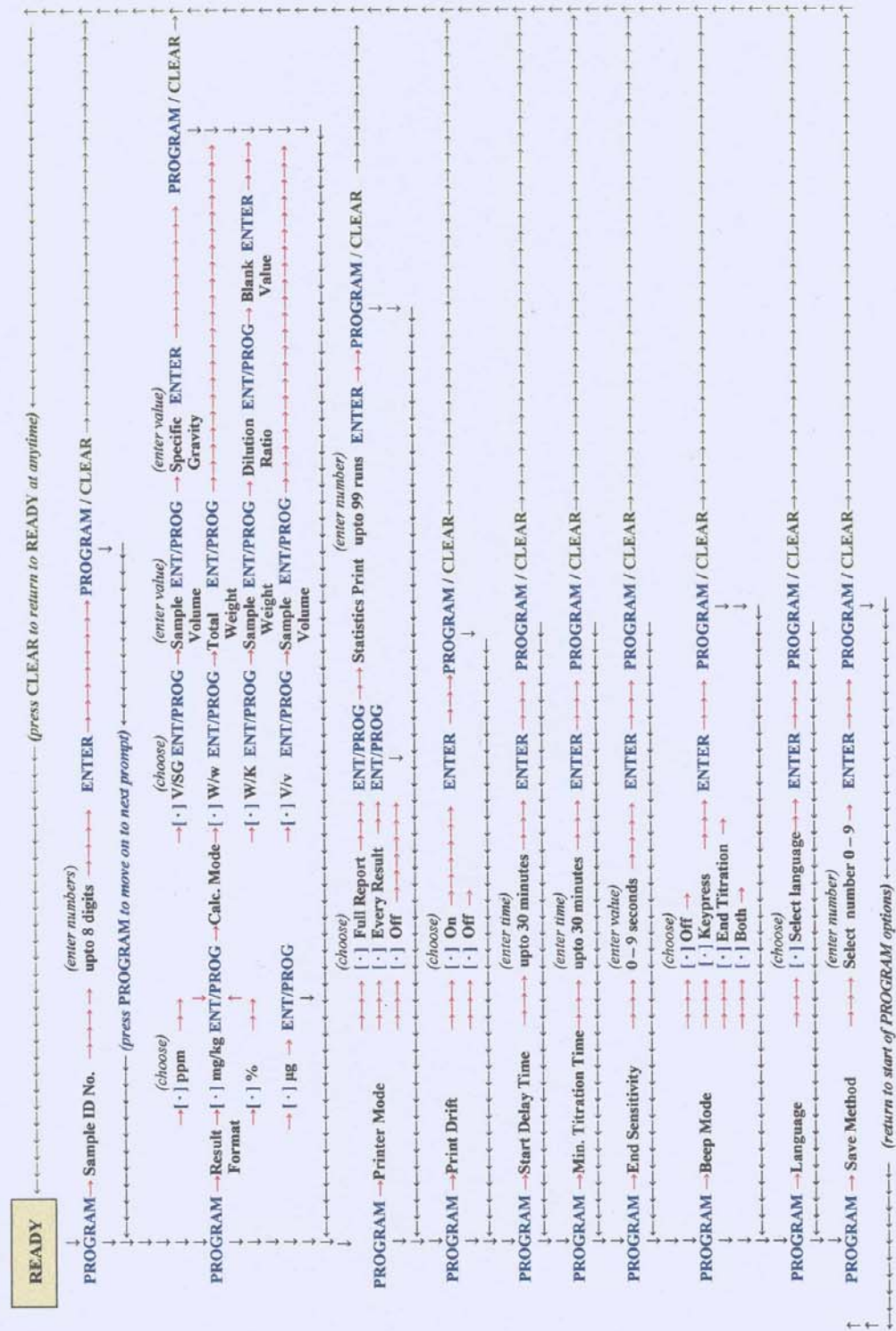
## MENU LIST

Sample ID Number	- upto 8 digits
Result Format	- µg, ppm, mg/kg or % water
Calculation Mode	- V/SG, W/W, W/K or V/v
Printer Mode	- off, every result or full report
Statistics Print	- upto 99 runs, will print min., max., mean, SD & CV
Print Drift	- on or off
Start Delay Time	- upto 30 minutes
Min. Titration Time	- upto 30 minutes
End Sensitivity	- 0 – 9 seconds
Beep Mode	- off, keypress, end titration or both
Language	- select language
Save Method	- store upto 10 methods

See PROGRAMMING A METHOD for an explanation of each Menu List parameter



## PROGRAMMING FLOW CHART





## PROGRAMMING A METHOD

After the initial drift has been calculated, in micrograms ( $\mu\text{g}$ ) of water per minute, the AKF5000 is ready for operation. It is now possible to step through the PROGRAM parameters and enter the required calculation data.

Dft = xx	xxxx $\mu\text{g}$
1	Ready

The Flow Chart on the previous page may prove helpful. Press the PROGRAM key until the required option is displayed then simply follow the Flow Chart to enter the specific data values. Pressing PROGRAM will move you on to the next prompt, pressing CLEAR will return to Ready condition. It is not necessary to step through all of the programme functions for every method, for example, if you only require to change the

Beep Mode from Off to Both – simple press PROGRAM until display shows Beep Mode, press the decimal point key  $\bullet$  until display shows Both, then press ENTER and CLEAR to return to Ready.

### SAMPLE ID NUMBER

Press PROGRAM → Display will show → Key upto 8 numbers → Press ENTER

<b>PROGRAM</b>	Sample ID No.	Sample ID No 12345678	<b>ENTER</b>
----------------	---------------	--------------------------	--------------

If you do not want to enter any further parameters simply press CLEAR to go back to the Ready condition. Press PROGRAM if you want to continue entering other parameters

### RESULT FORMAT

Press PROGRAM → Display will show

<b>PROGRAM</b>	Result Format $\mu\text{g}$
----------------	--------------------------------

Results can be selected in  $\mu\text{g}$ , ppm, mg/kg or % water.

If you want to select the format already stored in memory (and shown on display), press PROGRAM and the instrument will go to the next prompt. If you want to change format to give results in ppm, mg/kg or % you can scroll through these options by pressing decimal point key  $\bullet$  until the required format is displayed, then press ENTER to store this in memory and PROGRAM to move onto the next prompt. If you have selected a result format in ppm, mg/kg or % the instrument will prompt you for the necessary calculation mode and data values.

Result Format %	<b>ENTER</b>	<b>PROGRAM</b>	Calculation Mode V / SG
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The display shows the calculated water content according to the programmed Result Format and Calculation Mode program settings, ie. whatever has been chosen to be shown/printed at the end of the titration. The only exception to this is in the W/w mode when the Tare Weight has not yet been entered. In this case the display will show the  $\mu\text{g}$  count. The PROGRAM key can be used to enter the Tare Weight at any time during the titration, once this has been done the display will start to show the calculated result in ppm, mg/kg or % water.

## CALCULATION MODE

The AKF5000 has four standard calculation modes in memory. Each of these allow the instrument to divide the microgram count by the gram weight of sample introduced so that the calculated result can be printed out in either ppm or percentage of water.

Press the decimal point key  $\bullet$  to scroll through the options available.

Select the calculation mode required and press ENTER.

The display will prompt you with the appropriate parameters.

Enter the numerical value for each parameter and press ENTER.

The four standard calculations are:-

### 1. Weight difference W/w

*(This is the most accurate calculation and is suitable for almost all types of samples, powders, liquids, gases, etc.)*

$$\text{Result (ppm or \%)} = \frac{\text{microgram count}}{W - w}$$

Where W = total weight of sampler + sample (gm)  
w = tare weight of empty sampler (gm)

After entering the total weight (W) the instrument will return to the Ready condition. The Tare weight (w) can be entered at any time during the titration by pressing PROGRAM, or at the end of titration. For ease of operation it is possible to enter the Total weight (W) as 0.0000gm, then place the full sampler on the balance and tare to zero. Thereafter it is only necessary to enter the weight difference (tare weight w) for each titration. This procedure reduces potential data entry errors.

### 2. Weight/Dilution ratio W/K

*(Use this method if you need to dissolve a sample in another solvent. Suitable for samples which will not dissolve in the anode reagent or which might interfere with the KF reaction)*

$$\text{Result (ppm or \%)} = \frac{\text{microgram count} - bl}{W} \times K$$

where:

W = weight of sample dissolved (gm)

K =  $\frac{\text{total volume of diluent + sample}}{\text{volume injected into Cou-Lo Aquamax}}$

bl = micrograms of water for same injected volume of diluent (μg)

e.g.

Accurately weigh approximately 2 gm of sample into a 20ml stoppered graduated flask. Make up to the volume line with suitable solvent / diluent. Using appropriate syringe inject 100μl of diluted sample into the titration cell. Calculation parameters for this example would be:-

W = 2 gm  
K =  $\frac{20\text{ml}}{100\mu\text{l}} = 200$   
bl = microgram count of 100μl injected volume diluent

**3. Volume / Specific Gravity V/SG**

*(The most widely used technique for liquid samples. However, care must be taken to ensure removal of all air bubbles from the syringe)*

$$\text{Result (ppm or \% )} = \frac{\text{microgram count}}{V \times SG}$$

where V = volume of sample (ml)  
SG = specific gravity

**4. Volume V/v**

$$\text{Result \% volume} = \frac{\text{microgram count}}{V \times 1.0}$$

where V = volume of sample (ml)

This is essentially the same as V / SG mode, except that the sample S.G. is fixed at 1.0

**PRINTER MODES**

Selecting this program allows the printer to be turned ON or OFF. It also enables you to print out the titration parameters being used at that time. Simply scroll through the options using the decimal point key

- until the required mode is displayed, then press ENTER.

Options available:

- Off - no result print out
- Every result - will print result after every titration, first result also prints all calculation input data.
- Full Report - will print results after every titration and then print out statistical data after a selected number of titrations.

**STATISTICS PRINT**

The AKF5000 can be programmed to print out the statistical data from a series of results when using "Full Report" print mode. If no number has been selected, the AKF5000 will automatically clear after 99 runs. If 3 or more runs have been selected the Aquamax will print out the minimum, maximum, mean, SD & CV values.

**PRINT DRIFT**

The AKF5000 can be programmed to print out the drift value for each titration. This is a useful indicator of titration cell stability. If drift is above 25 µg / minute print will show \*\*XX\*\*. This is a warning that a high drift value was present at start of the titration.

## DELAY TIMES

The AKF5000 has been programmed to accept two different types of delay times, one at the start of titration and one at the end as a minimum titration time.

### START DELAY TIME

The AKF5000 has a preset delay of five seconds which allows samples to be dispersed into the reagents before the titration starts. Some samples, particularly powders and viscous samples, may need additional time to fully dissolve. By selecting the start delay program, you can increase this time by upto 30 minutes.

Display shows:

Start Delay Time 00 mins 00 secs
-------------------------------------

Select required time delay and press ENTER.

Whilst a delay time is in operation, the display will show “add sample” and the clock counter will show the time remaining before the start of titration. A start delay can be overridden by simply pressing START

### MINIMUM TITRATION TIME

For some applications, such as certain gas samples or when using the AKF5000 combined with a solid sample vaporiser unit it can prove useful to delay the end point of the titration. By selecting this parameter you can increase the minimum titration time by upto 30 minutes.

Display shows:

Min. Titration Time 00 mins 15 secs
--

Select required minimum time and press ENTER.

### END SENSITIVITY

The sensitivity of detector signal as the end point is approached can be set from 0 – 9 seconds. The default value is 3 seconds and there are very few occasions when this may need to be altered. Decreasing this value can prove useful if samples cause interference, e.g. some Lube Oil samples.

### BEEP MODE

Four options are available for the audible alarm (beeper) and they can be scrolled through using the decimal point key **.** until the required one is displayed. Then press ENTER.

The options are:

- |               |   |
|---------------|---|
| Off           | - beeper inactive   |
| Keypress      | - beep once every time a key is pressed                               |
| End titration | - beep three times at the end of a titration                          |
| Both          | - beep after every keypress and three times at the end of a titration |

## LANGUAGE

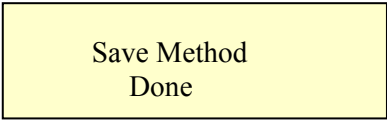
The AKF5000 can be programmed to show all display prompts and print outs in various languages. Use the decimal point key **.** to select language then press ENTER.

Choose from English, Francais, Espanol, Portugues, Deutsch & Magyar.

## SAVE METHOD

If you want to save a method for future use, this can be done by pressing a numerical key, 0 - 9, and ENTER. All titration parameters, printer mode, beeper mode, etc. are stored automatically.

Display then shows:



Save Method  
Done

Then press CLEAR and the instrument will revert back to the Ready condition.

## RECALL METHOD

If any methods have been saved to memory then the first prompt (before Sample ID number) will be "Recall Method"

When this prompt is displayed, simply press the number of the method file you wish to recall and press ENTER. There are 10 method files available numbered from 0 to 9. The titration parameters etc will all be automatically recalled. Press PROGRAM to view the recalled method parameters or press CLEAR and the instrument will go back to the Ready condition.

If you do not want to recall a method, press PROGRAM to view the next prompt.

**EXAMPLE COULOMETRIC METHODS**

ASTM D 1533	Water Determination of Electrical Insulating Oils by Coulometric Karl Fischer Titration
ASTM D 4928	Water Determination of Crude Oils by Coulometric Karl Fischer Titration
ASTM D 6304	Water Determination of Petroleum Products by Coulometric Karl Fischer Titration
IP 386	Water Determination of Crude Oils by Coulometric Karl Fischer Titration
IP 438	Water Determination of Petroleum Products by Coulometric Karl Fischer Titration
IEC 60814 BS EN 60814	Water Determination of Insulating Liquids, Oil Impregnated Pressboard by Coulometric Karl Fischer Titration
ISO 10101-3	Water Determination of Natural gas by Coulometric Karl Fischer Titration
ISO 10337	Water Determination of Crude Petroleum by Coulometric Karl Fischer Titration
ISO 12937	Water Determination of Petroleum Products by Coulometric Karl Fischer Titration

## EXAMPLE APPLICATIONS

### LIQUID SAMPLES

Liquid samples are usually introduced into the titration vessel by using a syringe. The greater the amount of water introduced into the titration cell will cause the titration to take longer and also consume the reagents more quickly. It is recommended that the smallest representative amount of sample be used to ensure fastest titration speed and maximum sample throughput before reagents have to be replenished. Supplied as standard with every AKF5000 titrator is a 1ml syringe and luer needle. These are suitable for injection of oil samples and water standards. Other volume syringes are commercially available from various suppliers. Please ensure that the needle is long enough to reach into the anode reagent.

This guide may be of assistance when selecting injection volume of samples:-

Expected water content.....			Suggested sample size
1	- 10	ppm.....	1 or 2 ml
10	- 100	ppm.....	1 or 2 ml
100	- 500	ppm.....	0.5 or 1 ml
500	- 1000	ppm.....	0.5 or 1 ml
0.001	- 0.01	% .....	1 or 2 ml
0.01	- 0.1	% .....	1 or 2 ml
0.1	- 0.5	% .....	0.5 or 1 ml
0.5	- 1.0	% .....	0.5 or 1 ml
1.0	- 5.0	% .....	0.2 ml
above	5.0	% .....	0.1 ml or less

Please note that these volumes are only a guide. You may wish to experiment with sample size to determine the optimum balance between repeatability, reagent depletion and speed of analysis.

### CRUDE OILS

(IP 386 & 438, ASTM D1533, D4928, D6304, API MPMS Chapter 10.9, ISO 10337 & 12937)

Most coulometric reagents contain Methanol as the main solvent and as such it is possible that waxy deposits from crude oils can “drop out” and contaminate the electrodes, requiring more frequent and thorough cleaning of the glassware. This problem can be reduced by using Cou-Lo “Formula A” anode reagent which has been specially formulated for this application. The most common interference in crude oil samples is usually caused by mercaptans or sulphides. Samples containing alkyl groups will react stoichiometrically so that:

100 ppm Mercaptans would show as approximately 30 ppm water

100 ppm Sulphides would show as approximately 50 ppm water

In normal practice oils containing less than 500 ppm mercaptans or sulphides are regarded as having no appreciable effect. Repeatability problems are usually caused by sampling inaccuracies or improper homogenisation of the sample.

## TRANSFORMER/INSULATING OILS

(ASTM D 1533, BS EN 60814, IEC 60814)

Due to the viscosity of these samples, and the fact that typical water content levels are in the range 5 – 40 ppm, it is usually necessary to inject sample volumes of 1 ml. This means that upto 50 samples would fill the titration cell. Provided that the other criteria governing reagent life, (total water titrated and reagent age), have not been reached, then it is possible to switch off the titrator and allow the oil to separate from the reagent. The oil can then be siphoned off and the instrument switched on again. After this operation it may take upto 30 minutes before the instrument settles down to a stable baseline. If it takes longer than 30 minutes then it is advisable to clean the cell and recharge with fresh reagents. For optimum performance use Cou-Lo “Formula A” reagent.

## TURBINE / LUBRICATING OILS

For most Lube Oil applications a sample size of 1ml is sufficient. However, if it is suspected that the water content is above 1 %, then a sample size of 0.2 to 0.5 ml is suggested.

Some lubricating oils contain additives which can coat the detector electrode or block the glass frit of the generator electrode. These problems can be reduced by modifying the Anode reagent with 20 – 40 % volume Chloroform.

## KETONES AND AMINES

These can react with the Methanol present in most coulometric reagents resulting in inaccurate results and, in extreme conditions, titration end point being unattainable. For samples containing ketones, amines or aldehydes the use of specially formulated coulometric reagents for ketone samples is recommended.

## POWDER SAMPLES

One of the main advantages of the coulometric technique is that reagents do not have to be replenished after every analysis, therefore the introduction into the titration cell of solid samples, which do not dissolve, is not recommended.

Three options available:-

- Powder samples which readily dissolve in the Anode reagent can be introduced using the optional powder sampler, part no. 2000-9511. Calculation mode W/w.
- Powder samples which cannot be dissolved in the Anode reagent should be extracted or dissolved in a suitable solvent and then an aliquot injected. Calculation mode W/K.



## GAS SAMPLES

Gas samples can be introduced by fitting a needle onto the gas sampling bomb and selecting Calculation mode W/w. Alternatively, gas samples can be bubbled through the titration vessel and the passed volume measured. For this method we suggest using the optional Low Drift Cell (LDC-0101) complete with gas analysis kit (LDC-0121).

1. Fit the gas inlet and outlet adapters in place of the injection septa.
2. Remove the drying tube and seal the top of electrode using the injection port septa and screw cap.
3. Connect outlet adapter to wet gas flow meter.
4. Flush the sample lines and allow instrument to stabilise.
5. Press START and allow gas sample to flow through the cell at an approximate flow rate of 0.5 litres/minute.
6. Turn off gas flow and allow titration to reach end point.

Calculation

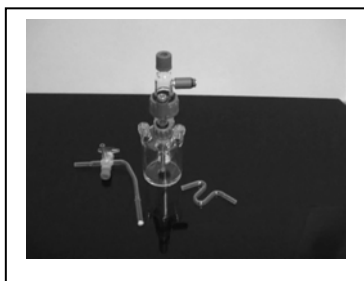
$$W = \frac{G \times (273 + t) \times 22.4}{V \times 273 \times 18}$$

W = moisture content

G = microgram count

V = gas volume, litres

t = water temperature of wet gas flow meter (°C)



Gas sample adapter



Fitted to vessel

## HOW TO RUN A TEST

Please read the section PROGRAMMING A METHOD then use the PROGRAMMING FLOW CHART before running any samples,

### Example: Transformer oil sample by V/SG

Program the AKF5000 with parameters for the analysis. For Transformer Oil samples, these parameters are usually:-

Result Format	= mg/kg or ppm
Calculation Mode	= V/SG
Sample Volume	= 1.0 ml
Sample SG	= 0.875

Other parameters such as Sample ID number, Printer Mode etc are optional.

### ANALYSIS METHOD

1. Confirm that AKF5000 is in “Ready” mode
2. Flush 1.0ml syringe several times (minimum 6 times) with sample
3. Fit luer needle and flush through with sample
4. Draw sample into syringe beyond the 1.0ml marking
5. Invert syringe so that any air bubbles can be ejected through the needle and adjust syringe plunger to the 1.0 ml mark
6. Wipe off excess sample from outside of needle using a clean, dry tissue or paper towel
7. Pierce needle through injection septa of titration vessel (1 – 2 cm)
8. Press START
9. Push needle into anode reagent and inject sample
10. Withdraw needle from titration vessel
11. Read result, in mg/kg (ppm) water, on display and printout
12. Repeat steps 2 – 11 if duplicate result required

**Example: Powder sample by W/w**

Program the AKF5000 with parameters for the analysis.

**ANALYSIS METHOD (1)**

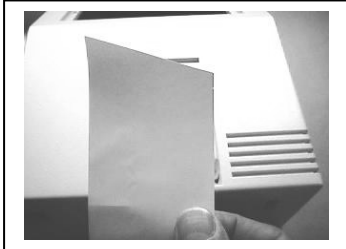
1. Confirm that AKF5000 is in “Ready” mode
2. Place a suitable amount of sample in the powder sampler (p.n. 2000-9511) and seal with stopper provided.
3. Weigh sampler
4. Press PROGRAM until Calculation Mode displayed then select W/w
5. Enter the sampler weight when prompted by display, then press ENTER / CLEAR
6. Remove the injection septa port, press START, and pour the sample inside the titration vessel ensuring that sample does not stick to the inside of the vessel wall.
7. Reweigh the sampler.
8. The net weight of sampler can be entered either during the titration by pressing PROGRAM or waiting until the end of titration when display will prompt TARE Weight. Enter the weight then press CLEAR.

**ANALYSIS METHOD (2)**

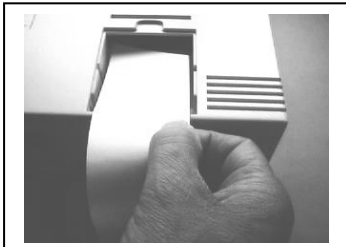
1. Confirm that AKF5000 is in “Ready” mode
2. Place a suitable amount of sample in the powder sampler (p.n. 2000-9511) and seal with stopper provided.
3. Place sampler on balance and tare to zero
4. Press PROGRAM until Calc. Mode displayed then select W/w
5. Enter the sample weight as 0.0 gram, then press ENTER / CLEAR
6. Remove the injection septa port, press START, and pour the sample inside the titration vessel ensuring that sample does not stick to the inside of the vessel wall.
7. Reweigh the sampler.
8. The weight difference of sampler can be entered either during the titration by pressing PROGRAM or waiting until the end of titration when display will prompt TARE Weight. Enter the weight then press CLEAR.

## LOADING THE PRINTER

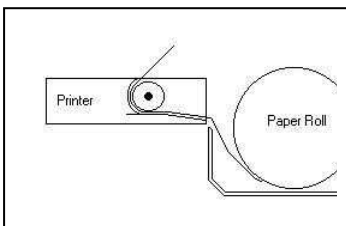
To load the paper into the printer, first open the paper roll holder lid above the print head and remove the old roll and rollers. Now fit rollers to new paper roll (LDC-0119), load as shown and insert the paper into the slot at the rear of the print head. As soon as the printer sees the paper it will automatically load it. To avoid damage to the printer mechanism it is advisable to only use the correct part number (LDC-0119) thermal paper rolls.



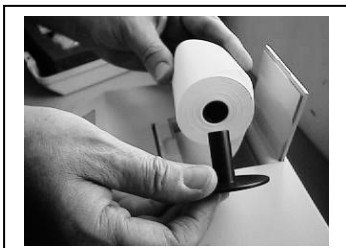
Cut the end of the paper roll at a slight angle as shown. This will make it easier to feed into the printer



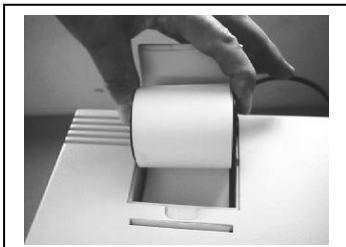
The mechanism has optical sensors which detect the paper and will auto feed.



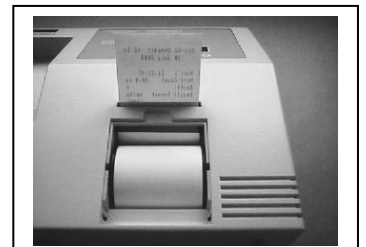
Schematic of paper feed



Fit rollers onto both sides of paper roll



Locate paper roll in to compartment



## PRINT-OUTS

The AKF5000 allows you to select different print modes. If you choose to have the printer on, you will receive a hard copy of the result obtained, together with the selected parameters for that particular titration every time a titration is performed. Options available;

- Off - no result print out
- Every result - will print result after every titration, first result also prints all calculation input data.
- Full Report - will print results after every titration and then print out statistical data after a selected number of titrations.

The AKF5000 automatically increments the next run number every time you press the start button. The run counter is reset to zero when power is switched off and back on again. A duplicate set of results can be printed out simply by holding down the start button for 3 seconds. Results memory is cleared after duplicate print out or when power is switched off.

### PRINT OUT EXPLANATION

AKF5000	
	V 2.3v
Serial No.	71000
Calibrated	DD/MM/YYYY
Run Date	
Run: 1	
Run Time	
Nett Count	0.0 µg
Drift	00
Result Format	%
Calculation Mode	V/SG
Volume	1.0000 ml
Density	1.0000
Res:	0.0000%
Run: 2	
Run Time	
Nett Count	0.0 µg
Drift	00
Res:	0.0000%

### EXAMPLE OF V/SG CALCULATION

Titrator model  
 Software version  
 Titrator serial number  
 Date titrator calibrated  
 Date sample run  
  
 Run number & time of analysis  
 Nett microgram count (after drift correction)  
 Drift value at start of titration  
 Result format selected  
 Calculation mode selected  
 Sample volume  
 Sample density

Calculated result water content

Subsequent runs of the same sample and calculation parameters show run number, run time, nett count, Drift value and calculated result.

AKF5000	
	V 2.3v
Serial No.	71000
Calibrated	DD/MM/YYYY
Run Date	
Run: 1	
Run Time	
Nett Count	0.0 µg
Drift	00
Result Format	%
Calculation Mode	W/w
Total Weight	0.0000 gm
Tare Weight	0.0000 gm
Sample Weight	0.0000 gm
Res:	0.0000%

### EXAMPLE OF W/w CALCULATION

Run number & time of analysis  
 Nett microgram count (after drift correction)  
 Drift value at start of titration  
 Result format selected  
 Calculation mode selected  
 Weight of syringe plus sample  
 Weight of syringe after sample injected  
 Weight of sample analysed

Calculated result water content

## DRIFT RATE COMPENSATION

At the start of any titration, the drift value in the top left corner of the screen (in micrograms per minute), is stored in memory. This ensures that the displayed result is corrected for any ingress of atmospheric moisture during the titration period.

Whilst in READY condition, the AKF5000 continually compensates for any drift caused by atmospheric moisture ingress or reagent decomposition and displays this information, which is updated every 10 seconds. If the drift value is above 25  $\mu\text{g}$  /minute the display will show High Drift **\*\*XX\*\***. This is a warning that the value is high, however the instrument can still be used by simply pressing START to continue. Provided the drift rate is stable then it is usually acceptable to continue operation. The AKF5000 titration cell design can provide drift values down to less than 5  $\mu\text{g}$  /minute but can also operate at much higher values. The maximum drift value is 60  $\mu\text{g}$  /minute but we recommend operating up to a maximum of 25 whenever possible.

If the drift value is above 60  $\mu\text{g}$  per minute the display will read  
“EXCESS DRIFT - SEE INSTRUCTION MANUAL”

Most common causes of excess drift are:

Titration vessel not properly sealed (check septa & fittings).

Reagent almost depleted (clean & recharge the cell).

Sample introduced before pressing START key.

Trace moisture on cell walls.

If excess drift occurs, switch the instrument off, remove titration cell from clamp, gently swirl the anode reagent around the cell walls, replace cell in clamp, switch instrument on and allow to precondition.

## OVERTITRATION

If the detector electrode senses too much Iodine in the titration cell, usually caused by instrument being left unoperated for a long period, the display will read :-  
“OVERTITRATION - SEE INSTRUCTION MANUAL”

Simply add 3 - 5 microlitres of water, or a small amount of a known wet sample, until the detector signal bars on bottom left of display are activated. Then instrument will automatically carry out its precondition.

## REAGENT LIFE

There are three main factors which govern the life of reagents, and therefore, the frequency of cleaning and recharging the titration cell:

1. The physical size of the titration cell allows for 50 – 60 mls of sample to be added. For the analysis of most liquid samples (e.g. solvents) the AKF5000 normally only requires a few microlitres to be injected, therefore the maximum volume of 50 – 60 mls should not become a limiting factor. (Oil samples are an exception and are dealt with elsewhere in this manual).
2. The total amount of water that can be analysed before saturation of the reagents varies slightly from one reagent manufacturer to another. Typically a 100 ml charge of Anode reagent (Formula “A”) will analyse up to 1 gm water, whilst a 5 ml charge of Cathode reagent (Formula “C”) will analyse up to 250 mg water. Considering that the injected sample volume is normally small, and also that the analysis is usually for the determination of low levels of water, these water capacity values are not normally a limiting factor.
3. Similar to all other Karl Fischer reagents, coulometric reagents will deteriorate when exposed to sunlight and with increases in temperature. Placing the instrument in direct sunlight or near to a heat duct can decrease the reagent life. As reagents age, the titration speed will reduce and the drift value will increase.

## CLEANING GLASSWARE

### IMPORTANT

The platinum mesh and wires of the electrodes are fragile and can be easily bent or broken. Extreme care **MUST** be taken when disassembling, cleaning and reassembling the titration cell. The generator electrode is the most expensive part of the AKF5000 glassware. Coulometric reagents are flammable and toxic, so care should always be taken when handling them and when cleaning the glassware. Whenever possible this should be done in a safe area, e.g. inside a fume cupboard. Reagents should not be recharged while the titration cell is still attached to the titrator. See page 8 on charging procedure.

Under normal circumstances the titration cell can be used for a large number of samples before having to replenish the reagents. Once the reagents have been exhausted, or when the titration cell maximum volume has been reached, it should simply be necessary to:-

1. DISASSEMBLE titration cell
2. EMPTY the titration cell and generator electrode
3. RINSE all parts with methanol
4. DRY all parts
5. REASSEMBLE glassware
6. RECHARGE with fresh reagents

If the cell is heavily contaminated then it may be necessary to clean it more thoroughly. For oil samples, cleaning with chloroform or xylene is suggested, whilst for salt deposits a water wash may be required. Use whichever solvent is most suited for the sample type. The titration vessel can even be cleaned with hot soapy water and a bottle-brush. However, after cleaning with suitable solvent, all glassware parts, **MUST** be rinsed inside and out with methanol. They can then be dried with a warm air blower, such as a domestic hair dryer, placed in a low temperature oven at 40 – 50 ° C, or left in a desiccator.

After being fully dried, reassemble the titration cell and charge with fresh reagents.

**THE MORE THOROUGHLY THAT THE TITRATION CELL IS CLEANED AND DRIED, THE FASTER THE INSTRUMENT WILL STABILISE READY FOR OPERATION AFTER RECHARGING REAGENTS.**

Under normal conditions, the AKF5000 should be ready for operation within 5 – 10 minutes after reassembly, however it could take up to 20 minutes before to completely stabilise if the drying procedure has not been properly carried out.

## **TROUBLESHOOTING**

### **OVERTITRATION**

Detector senses too much Iodine. Usually caused by old reagents or stirrer switched off. Check stirrer speed. Add 3-5 microlitres of water until detector bars activate, and allow to precondition.

### **EXCESS DRIFT**

Indicates excess moisture ingress or condensation on cell walls. Switch OFF. Remove titration cell from clamp and gently swirl anode reagent around the cell walls. Reconnect, switch ON, and allow to Precondition.

### **LONG PRECONDITIONING TIME**

Normally caused by glassware not being sufficiently dried before assembly. Re-clean glassware and electrodes, dry thoroughly and charge with fresh reagents.

### **PROGRESSIVELY LOWER RESULTS FOR SAME SAMPLE**

Moisture contamination of syringe. Flush syringe and needle with sample several times before beginning new test.

### **POOR REPEATABILITY FOR SAME SAMPLE**

Sample size probably too small or moisture contamination of syringe. Increase sample size and flush syringe and needle several times before beginning new test.

### **WARNING LETTERS ON PRINT OUT**

Warning letters will be printed after time of each analysis to signify the following:-

U – Uncalibrated, E – Calibration expired, B – Battery level low, A – ACE system active

### **INSTRUMENT CARE**

Although the instrument casing is highly resistive to most solvents care should be taken to ensure that any spillage is wiped off immediately to avoid staining or possible damage to the paint surface.

## **WARRANTY**

Under the conditions of our warranty, all faults which are proved to be due to material, construction or manufacturing defects, which occur within 24 months of the delivery date, will be repaired or replaced, (at our discretion), free of charge at our premises. Freight costs will be chargeable along with any inspection work which was not necessitated by manufacturing or material defects. Breakages of glassware or electrodes are not covered by this guarantee. In the event of instrument failure during the warranty period, written authorisation must be obtained from the manufacturer prior to shipping.