

AQUAPAL III
COULOMETRIC KARL FISHER

CSC SCIENTIFIC COMPANY, INC.

MANUFACTURERS AND DISTRIBUTORS OF LABORATORY EQUIPMENT
2810 Old Lee Highway, Fairfax VA 22031
1-703-876-4030 1-800-458-2558 FAX:703-280-5142

SCIENTIFICGEAR LLC

MANUFACTURERS AND DISTRIBUTORS OF LABORATORY EQUIPMENT
1984 Isaac Newton Square, Reston, VA 20190
1-703-608-1349 1-800-998-6429 FAX:800-998-6429

Table of Contents

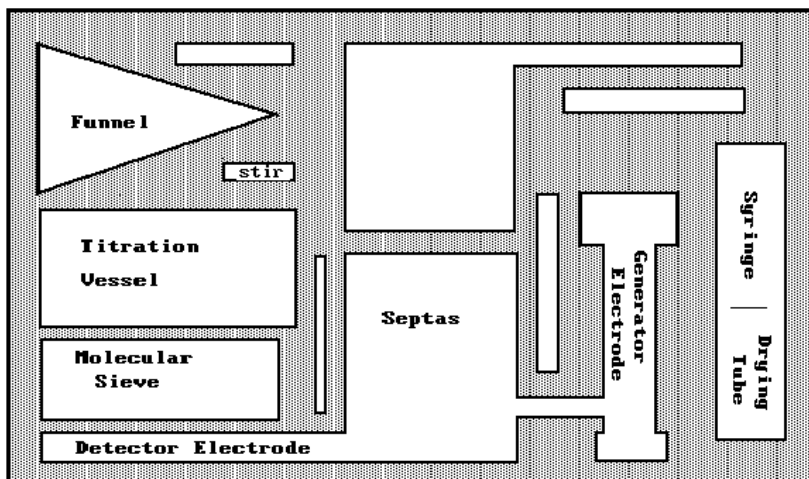
Introduction	1
Getting Started	3
Display	8
Running a Test	9
Keypad	13
Applications	17
Syringe Contamination	21
Cleaning the Glassware	25
Shipping the Aquapal	29
Troubleshooting	Appendix

Upon arrival, your CSC Aquapal III will be packaged in 3 different containers:

- A. The Aquapal III Titrator
- B. Glassware and accessories kit
- C. Karl Fischer reagents (supplied by Crescent Chemical) If needed

Unpack the Aquapal, examine the glassware and be familiar with:

- A. titration vessel
- B. generator electrode, large cap and O-ring
- C. detector electrode, small cap and O-ring
- D. stirrer bar
- E. plastic funnel
- F. septas (2 from bag of 50)
- G. Small caps (2) and O-rings for sealing
- H. Drying tube, medium cap and O-ring
- I. Molecular sieve

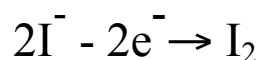
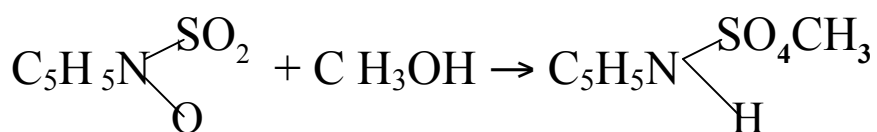
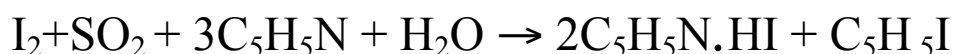


*Your Karl Fischer reagents will include:

- A. Anode solution (Hydranal A, AG, AG-H or AK)
 - B. Cathode solution (Hydranal C, CG or CK)
- OR
- C. Anode Solution for one reagent units (Hydranal AGH)

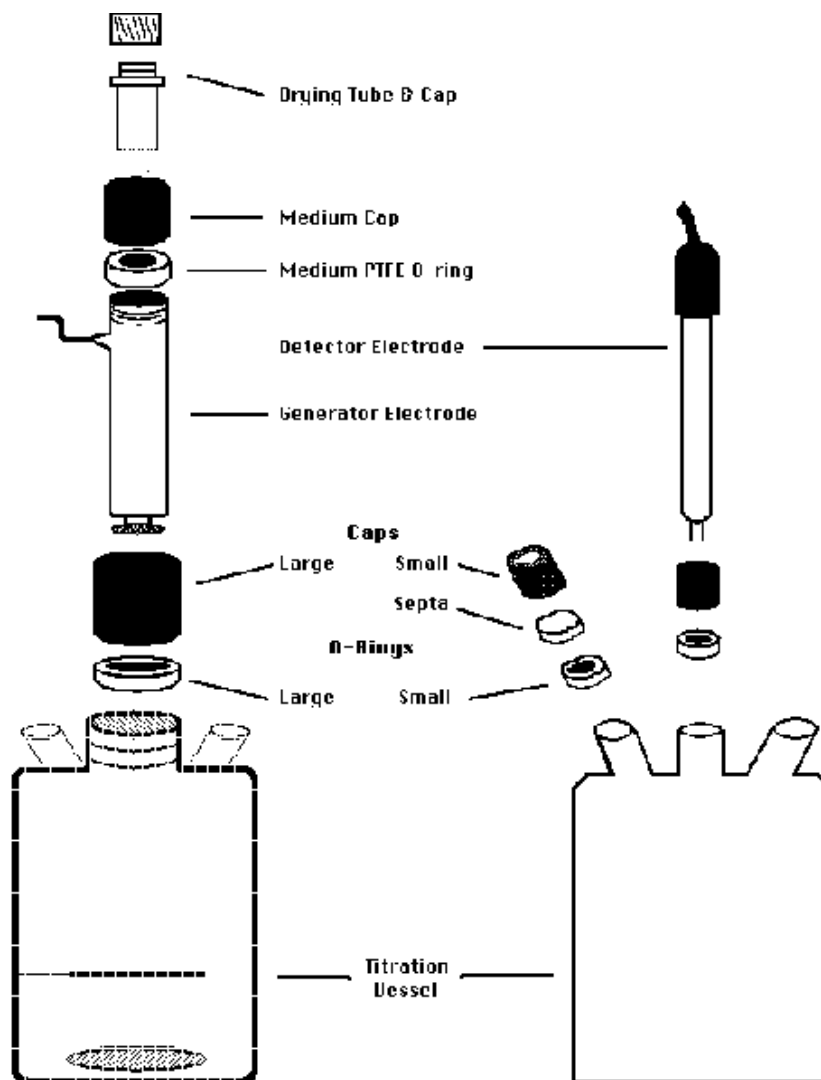
Principle of Measurement

The CSC Aquapal III produces iodine at the wire mesh of the generator electrode. When electricity is conducted across the mesh, one mole of the created iodine consumes one mole of water. One milligram of water is equivalent to 10.71 coulombs of electricity. The Aquapal measures the electrical current needed to create iodine and remove the existing water.



Employing a stepped pulse current, the CSC Aquapal III automatically selects titration speed depending on the amount of water present. As the end point is approached, the titration rate slows until the detector signal reaches and maintains the original baseline value. This end point is a minute excess of free iodine. At that time the CSC Aquapal subtracts any background drift, then calculates and prints the water content.

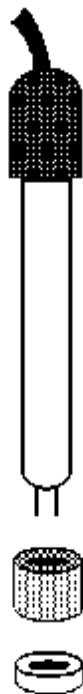
Getting Started Glassware



Getting Started

Assembling the Glassware

1. Detector Electrode: Slide the small cap onto the detector electrode with threads towards the two-pronged end. Position a small O-ring onto the detector electrode. Slide the O-ring so it fits snug in the cap.

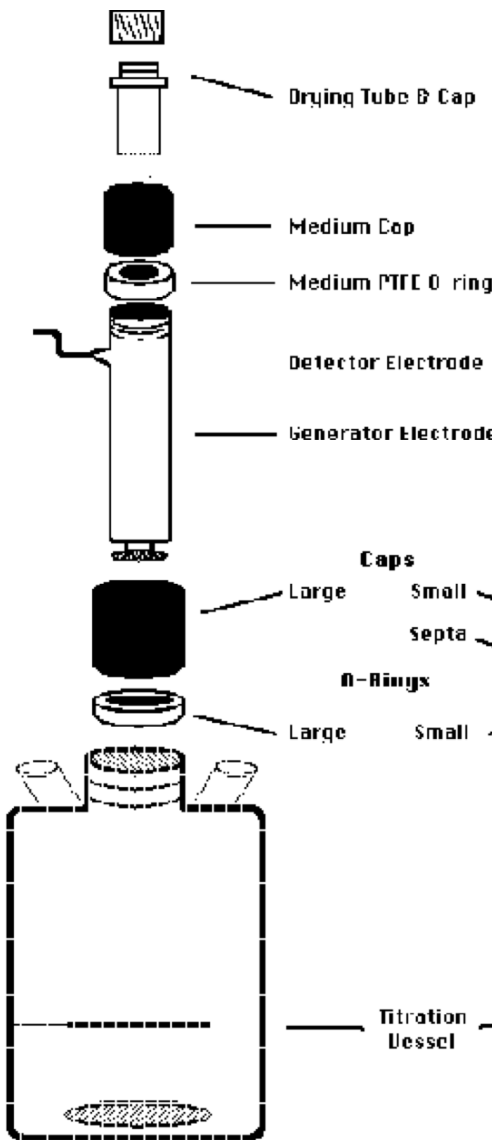


2. Vessel: The titration vessel has three smaller threaded holes. Working with the center thread, insert the detector electrode into the titration vessel. Tighten cap to seal.

NOTE: The detector electrode should slide into the vessel as far as it will go. There may be more than 1/2" between the detector probes and the vessel bottom

Getting Started

Assembling the Glassware (continued)



3. Generator Electrode: Position the large cap onto the generator electrode with threads towards the platinum mesh. Place the large O-ring onto the generator electrode. Slide the O-ring so it fits snug in the cap. Insert the generator electrode into titration vessel **leaving a 1/2" (15 mm) clearance**. Tighten cap to seal.

NOTE: May already be assembled.

4. Stirrer Bar: Place a stirrer bar in the bottom of the titration vessel. Check clearance so bar will not damage the platinum mesh of the generator or the probes of the detector.

5. Drying Tube: Completely fill drying tube with molecular sieve. Set aside.

6. Caps/Septas/O-rings: Insert one septa into each of the smaller two caps with PTFE-treated side down (towards threads) followed by an O-ring for sealing. You will see the yellow side of the septa through the top of the cap. Set aside.

NOTE: The O-rings for sealing have PTFE lining (white) for protection against chemical reagents. These are the same O-rings used with the detector electrode.

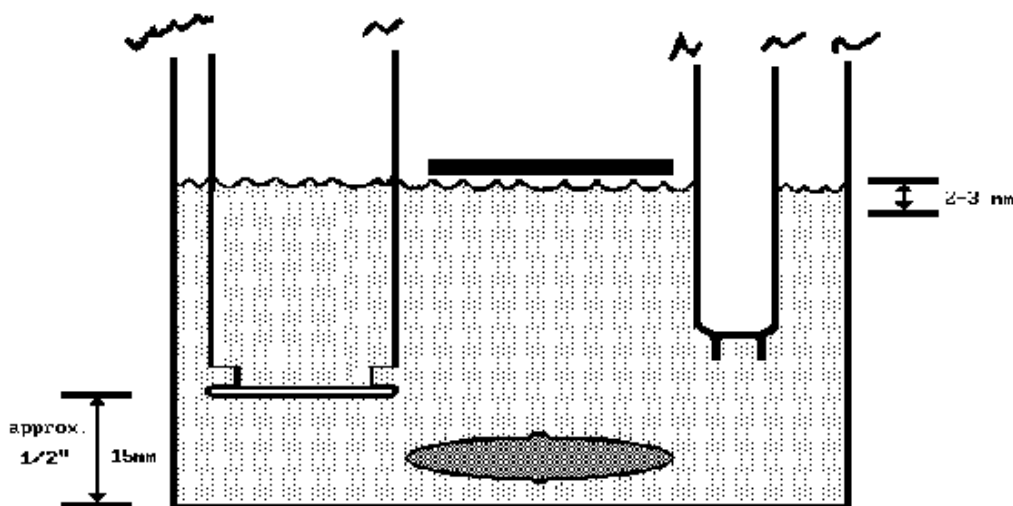
Getting Started Adding Reagents

1. **If you are using a 1 reagent system (fritless).** Using the funnel, fill titration cell **up to the TOP line** with anode "AG-H" reagent (100ml). This is the only reagent needed when using the single reagent system.
2. **If you are using a 2 reagent system.** Using the funnel, fill titration cell **up to the TOP line** with anode "A" reagent. Then take the cathode solution "C" and add 5ml to the generator electrode. *Note this is only necessary if you have a 2 reagent system*
3. **IF YOU ARE UNSURE AS TO WHICH SYSTEM YOU HAVE PLEASE CONTACT CSC SCIENTIFIC AT: 703-876-4030, 800-458-2558 or hlevi@cscscientific.com**

NOTE: If you are using a single reagent system, you only need to use the one reagent, the generator electrode is configured slightly different and does not have a frit at the base.

Hydranal-brand are specially formulated so no monitoring of A/C levels is needed. Stability is guaranteed regardless of cathode level.

2. **Caps & Septas:** Attach and seal the two small caps/septas and O-rings onto the slanted threads.
4. **Drying tube:** If not already attached, screw medium cap and O-ring onto top of generator electrode. Slide drying tube with filled molecular sieve into the top of the medium cap.



Getting Started

Connection and Start-up

1. Place the assembled titration vessel in the clamp and secure.
Looking at the front of the instrument, position the vessel with the generator electrode on the left and the detector electrode on the right.
2. Connect electrode cables to sockets. The detector electrode connects to the upper socket.
The generator electrode with the red-tagged cable connects to the lower socket with the red dot.
3. Attach power cable to the back of the instrument.
4. Press **POWER** switch.
5. Adjust stirrer motor, balancing the speed somewhere between a fast and slow swirl. Excess turbulence may create a higher drift rate. Please note that the stirrer bar can damage the platinum mesh on the generator electrode.
6. Press **CELL** button.
7. Allow the Aquapal to 'Precondition'.

Precondition:

Preconditioning happens when the instrument is turned on with the cell switch on or if the cell switch is turned off then on again. In preconditioning the instrument is removing any excess water from the cell, when complete the display will show a drift value and Ready (see next page).

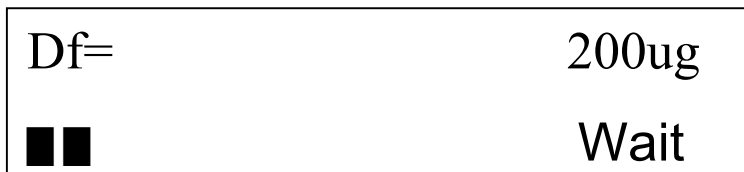
If the drift value is greater than 70 the instrument will automatically go to preconditioning, this is all so indicated on the print out each time it happens.

Its normal for the instrument to go through several preconditioning cycles particularly after the reagent has been changed, this is due to moisture getting into the cell after it has been opened.

The Aquapal Display



This screen indicates the instrument is preconditioning, (removing excess moisture from the cell). The Aquapal will go into preconditioning when power is applied with the cell switch on or when the cell switch is turned off then on.



After preconditioning the Aquapal starts to calculate drift in ug per minute.



The Aquapal is now ready to run a test.



This display shows the Aquapal running a test the ug count will increment and the detector indicator bars will reduce as the test Progresses to the end point.

Instrument set up.

Before you start running tests you will need to set how the results are printed or displayed.

There are 3 print and one display option for printing or displaying the results after a test.

To access these options press the **F5** Key then press 1,2,3 or 4 then **ENTER** to select the following.

1 = No print out after the completion of a test.

2 = Only the ug count is printed the completion of a test.

3 = This is option will print the results after the completion of a test.
The printout will include Run Number, Drift value, ug count and result in PPM or %
After you press **F5, 3, ENTER** the display will ask you to enter the following.

Sample ID number
Result format PPM or %
Calculator Function W/w , V/SG or W/K (see page 15 for definitions)

4 = This option will display the result in PPM or % (no print out) after the completion of a test.
After you press **F5, 4, ENTER** the display will ask you to enter the following.

Sample ID number
Result format PPM or %
Calculator Function W/w , V/SG or W/K (see page 15 for definitions)

This option is useful if you run out of paper or you just don't want to print the results.

Note: If you select V/SG you will also be given the option to have the display to count in PPM or % rather than the normal ug count.

Changing Calculator Function

When the Aquapal cell switch is off or the display shows

Df=	200ug
■■	Wait

or

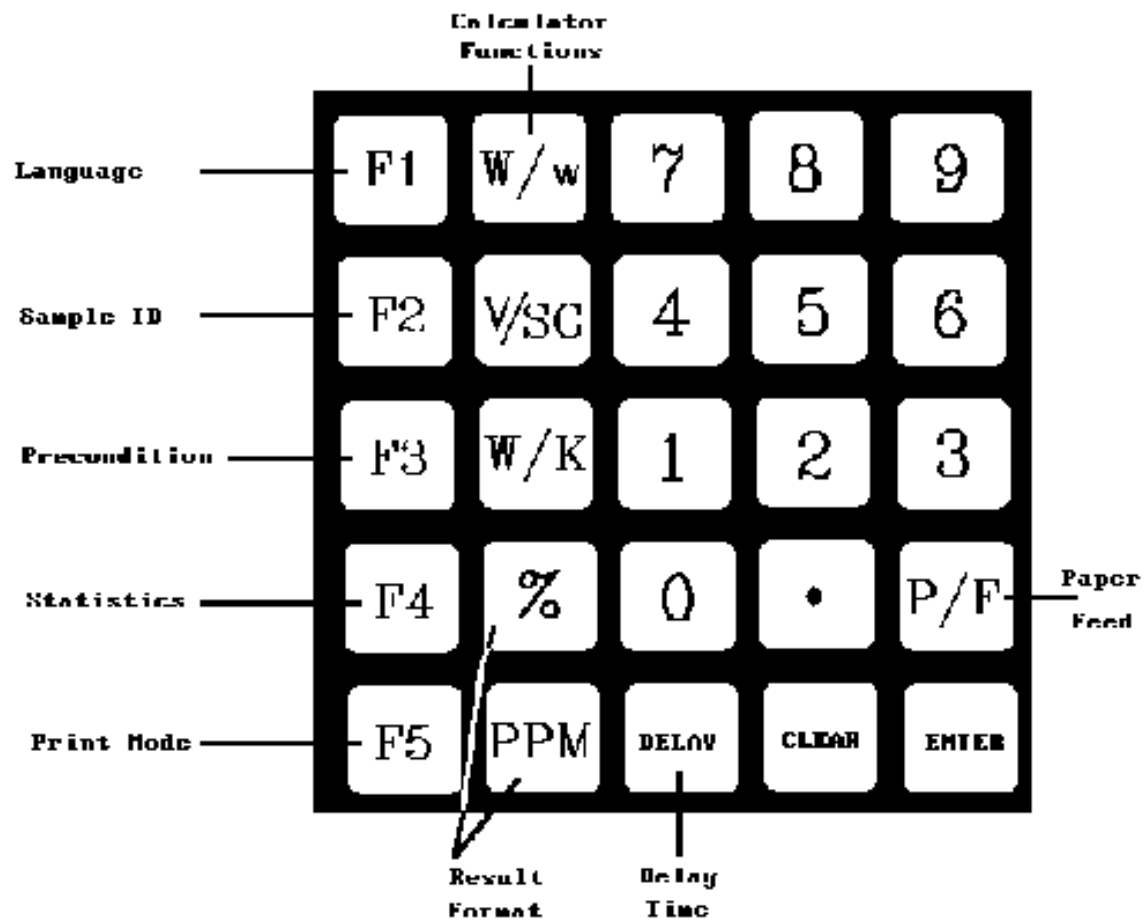
Df= 6	200ug
■■	Ready

You can press any of the Calculator Function keys W/w , V/SG or W/K to enter new values.

Note: To change the result format PPM or %you must press F5 and go through the setup menu.

The Keypad

The Keypad



Keypad Description

Function Keys

F1 - Function Menu

Not available in this version.

F2 - Sample I.D. number

Press **F2**, then **1** through **999999**, then **ENTER**.

For sample analysis I.D. Entering Sample I.D. will not affect data already in memory (i.e. V/SG - volume and specific gravity do not have to be reentered).

Decimal is allowed to further distinguish Sample I.D.
(i.e. 12.456)

F3 - Optional memory file

F4 - Statistics

Press **F4** and display shows:

PRINT STATS = 1 DELETE RESULT = 2

Press **1** and all statistics will print out.

Press **2** and display shows:

DELETE RESULT?

Select run number to delete (i.e. **2** or **15**) then press **ENTER**.

Continue selecting run numbers to delete.

Press **ENTER** again. Choose **F4** then **1**

The Aquapal will print statistics on all remaining results.

The Aquapal automatically prints statistics after analyzing 50 samples. Run number is then reset to zero. Statistics are calculated based on your results in PPM or percent, not ug count.

F5 - Print mode

No print out:	press F5 , then 1 , then ENTER .
Print ug count only:	press F5 , then 2 , then ENTER .
Full printout:	press F5 , then 3 , then ENTER .
Results displayed and no printout:	press F5 , then 4 , then ENTER .

Keypad Description

Calculator Function Keys

W/w - Weight over weight

Calculates water content by weight of your sample. The Aquapal divides the ug count by sample weight and prints in selected result format. Your sample weight is determined by the Aquapal when you enter total and tare weights.

The Aquapal also calculates based on sample weight only. At full syringe prompt, enter "0". After test at empty syringe prompt, enter sample weight. Results print.

V/SG - Volume over specific gravity

Calculates water content by your sample size and specific gravity.

W/K - Weight over dilution

Calculates water content of a diluted or dissolved sample. Used with powder samples or when any sample is mixed with a solvent before addition. A blank value is run on the carrier solvent. Weigh and enter the sample. The Aquapal subtracts the blank value from the ug count and prints in selected result format.

Result format

% - The Aquapal calculates and prints your results in percent.

PPM - The Aquapal calculates and prints your results in parts per million.

Both result format and calculator function remain stored in memory even when power is *off*.

Note: To change the result format you must press F5 and go through the setup menu.

Keypad Description

Delay time

The Aquapal has a standard delay time of six seconds. This allows time for the sample to disburse into the reagents after pressing the **START** button.

The default delay time of six seconds cannot be removed, but can be extended.

In some instances, six seconds may not be long enough for the detector signal to recognize water content. This delay time can be changed to allow for sample viscosity, powder samples or other special applications.

To set delay time:

Press **DELAY** key. Display will read:

Start Delay Min.Sec _____

Type in the extra time required in minutes and seconds, separated by a period. Then press **ENTER**.

Example: For 10 seconds type **00.10** then **ENTER**.

For 7 minutes, 27 seconds type **07.27** then **ENTER**.

After pressing the **START** button "Delay" appears at the lower right side of the display. This indicates the delay time is in operation. Cancel the delay during a particular test by pressing the **START** button again.

The delay time remains in memory for all future tests. Cancel by entering delay time of 00.00 (zero) seconds.

Applications

Suggested Sample Size

1 - 15 PPM	2 ml
15 - 100 PPM	1 or 2 ml
100 - 500 PPM	1 ml
500 - 1000 PPM	0.5 or 1 ml
0.001 - 0.01 %	1 or 2 ml
0.01 - 0.1 %	1 ml
0.1 - 0.5 %	0.5 or 1 ml
0.5 - 1.0 %	0.5 ml
1.0 - 5.0 %	0.2 ml
above 5.0 %	100 ul

ASTM standards may recommend a 10 to 20 ml sample for lower moisture ranges (10 PPM). This is due to imprecise instruments available when investigations were made. Because of the increased accuracy of the Aquapal III, you'll achieve reliable results with a small sample.

You may wish to experiment with sample size to achieve the proper balance between repeatability, reagent depletion and analysis speed.

Oil Samples

Transformer/Insulating Oils

Typical water content of transformer oils is 10 to 20 PPM. It is usually necessary to inject sample volumes of 1 to 2 ml. If the Aquapal is calculating by volume (V/SG) you may need to use a 2 ml sample to achieve repeatability. If you're using the weight (W/w) function and a four-place balance, a 1 ml sample should be adequate.

25 to 50 samples can fill the titration vessel. It is possible to turn off the titrator and allow the oil to separate from the reagent. The oil can then be siphoned off and the Aquapal turned back on. It may take 30 minutes for the Aquapal to 'Precondition' to the proper baseline. If it takes longer than 30 minutes it is advisable to clean and recharge the cell with fresh reagents.

Transformer oil specifications vary on specific gravity. In addition, oil density can change with age or addition of contaminants. Many transformer experts use 0.88 or 0.867 for a specific gravity value. You may wish to consult your oil supplier.

Applications

Turbine/Lubricating Oils

A 1 ml sample is sufficient for most lube oil applications. If a seal has broken, or you believe water content is above 1%, use a 0.2 to 0.5 ml sample.

A few lubricating oils have additives which coat the detector electrode or clog the frit in the generator. If this occurs, avoid future difficulties by mixing 25 to 40% Chloroform with your anode "A" reagent. Consult your reagent supplier or call CSC for further information.

EHC fluids have an approximate specific gravity of 1.16.

Turbine oil has an approximate specific gravity of 0.86.

Crude Oils

Occasionally, crude oils won't mix with your methanol-based reagents. These crude oil deposits "drop out" and contaminate the electrodes, requiring more frequent and thorough cleaning of the glassware. Avoid this situation by mixing 30% Xylene with your anode "A" reagent. Xylene or chloroform will dissolve your sample and help you achieve reliable results.

The most common interference with crude oil is caused by mercaptans and sulfides. Samples containing alkyl groups will react stoichiometrically so that:

100 PPM mercaptans show as approximately 30 PPM water
100 PPM sulfides " " 50 PPM water

Repeatability problems are usually caused by sampling inaccuracies and improper homogenization, not sulfides. Oils containing less than 500 PPM mercaptans or sulfides should not affect your results.

Fuels/Fuel Oil/Gasoline/Diesel

Fuels typically in the 200 to 300 PPM range will need a 1 ml sample. Calculate specific gravity by using the API number as follows:

$$\frac{141.5}{\text{API\#} + 131} = \text{Specific Gravity}$$

Approximate specific gravity values:

- Gasoline 0.736 to 0.75
- Kerosene 0.79
- Diesel 0.88

Applications

Ketones and Amines

Ketones and amines react negatively with the methanol in standard Karl Fischer reagents. In addition to giving inaccurate results, they can coat the detector electrode or clog the frit (glass diaphragm) on the generator.

If your samples contain amines, Ketones or Aldehydes use the reagents specially formulated for ketone solutions. Hydranal AK and CK are recommended.

When cleaning the glassware, use a methanol-free solvent such as acetone. You also may wish to "bake" the cleaned titration vessel in an oven at 50 degrees Centigrade. Methanol residue escalates the formation of ketals which clog the frit.

Reagent life shortens when working with ketones. Ketone applications consume the reagent and the cell will need to be recharged more often.

Applications

Gas Samples

1. Remove the two caps/O-ring/septas from the angled threads on the titration vessel.
2. Replace the PTFE-coated (white) O-rings with gas sealing O-rings provided with the Gas Accessories kit.
3. Thread gas inlet and outlet tubes through gas sealing O-rings. 4. Tighten caps to seal.
5. Remove the drying tube and seal the top of the generator electrode with the blanking disk.
6. Connect the outlet tube with a wet gas meter (flow meter).
7. Flush the lines and allow the Aquapal to re-equilibrate.
8. Making sure gas is bypassing the titration vessel with the stopcock valve, prepare gas to flow at 0.5 litre/minute.
9. Press **START** and introduce a sufficient (and reproducible) sample into the titration vessel.
10. Turn off the gas flow.
11. Allow the instrument to reach an end point.

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

W = moisture content (ug per liter)
 G = ug count
 V = gas volume (liters)
 t = water temp (C) in wet gas meter

12. For applications involving a drying oven and nitrogen gas, use the **W/w** function. Input weight of sample. Tare weight will be zero. Since the Aquapal is analyzing a known sample there is no need for a gas flow meter.

NOTE: The **DELAY** key is useful in gas applications. This allows you sufficient time to introduce a sample and then run your test. See further explanation under Keypad - Delay Mode.

An optional gas sampling kit containing tubes, seals and samplers can be purchased through CSC for use with all drying ovens.

A note about the new CSC Vaporizer:

The CSC Vaporizer uses a special gas sampling tube not addressed here. A unique internal rotameter controls nitrogen delivery - gas flow meter is unnecessary. Refer to CSC Vaporizer manual for easier testing method.

Syringe Sampling Technique

Proper syringe sampling

Air bubbles add moisture to your sample. If bubbles are seen when filling the syringe, tighten needle with a twisting motion.

1. Fill the syringe **past** your target volume.
2. Turn syringe upside down.
3. Tap side of syringe so air bubbles to rise to the top.
4. Hold **clean** paper towel over tip of needle.
5. Depress plunger slightly, forcing air bubble out, so plunger tip rests on target volume.
6. Wipe needle with another **clean** section of the paper towel.
7. Check septa for oil spills that attract moisture.

Syringe Contamination

Moisture contamination in your syringe causes improper results. The symptoms of contamination are:

- * **High results which drop to lower results.**
(i.e. 400 PPM, then 200, 80, 20, 20 PPM on same sample)
- * **Mixed results. High and low results.**

Oil build up forms in the syringe and needle through improper cleaning. Repetitive samples flush out the moisture attracted by the oil. This is why your results start high, then go down.

It's best to clean your syringe and needle when daily tests are completed. Then separate needle, plunger and glass and allow to dry. The next day your syringe will be clean and dry.

Clean the syringe

Flush out the syringe and needle with methanol or isopropyl alcohol **5 to 10 times**.

Remove the needle and flush out the syringe with the same alcohol.

Separate the plunger from the syringe. Lay needle, syringe and plunger on a paper towel and allow to dry for a few minutes.

After reassembling syringe and needle, fill syringe to capacity with first sample. Empty into waste container.

Fill and empty syringe **5 to 10** times with sample to remove any moisture left by the alcohol.

Drift Rate Compensation

The Aquapal automatically compensates for drift after a test. It stores the displayed drift value in memory when you press the **START** button. At the end of a titration, the display flashes while the Aquapal calculates drift and test time. It then subtracts the calculated drift before printing test results.

After running a test and after 'Preconditioning', the Aquapal calculates drift for 30 seconds. It then updates this value every 15 seconds. The **START** button remains inactive until the Aquapal calculates initial drift.

"**EXCESS DRIFT >70ug/min**" displays if drift exceeds 70. This indicates one of three problems:

- 1) Titration vessel not sealed properly (check septas/caps).
- 2) Moisture in space above reagent (see below).
- 2) Reagent life almost depleted (change reagents).
- 3) Sample added before pressing **START**.

If excess drift occurs, turn the **CELL** switch off and then on again.

If moisture condenses inside the vessel:

- 1) Turn **Cell** off, then **Power** off.
- 2) Unclamp the vessel.
- 3) **SWIRL** the anode solution along the wall of the vessel.
- 4) Turn **Power** ON, then **Cell** ON.

Instrument will "Precondition", removing excess moisture.

Reagent Life

The size of the titration vessel allows for sample addition of 50 to 60 ml. Maximum volume, in most instances, should not be a limiting factor. Refer to the Applications section of this manual for further information on sample size.

Reagent life is the total amount of water analyzed before saturation. Depending upon reagent brand, 100 ml of anode "A" reagent or Single reagent "AGH" analyze up to 1 gram (1 million ug) water. 5 ml of cathode "C" reagent analyze up to 250 mg (250,000 ug) water.

Sunlight and temperature increases will deteriorate Karl Fischer reagents. Placing the Aquapal in direct sunlight or near a heat duct can decrease the life of your reagents. One charge of reagents typically lasts three to four weeks if you're not running a test.

The Aquapal's optional carrying case is an ideal protection from sunlight. Covering the Aquapal with a dust cover is also effective.

Cleaning the Glassware

The most important thing to remember:

The Generator electrode is extremely fragile. The platinum mesh and wires can be easily bent or broken. The Generator electrode is the single most expensive piece in the Aquapal.

Normal Cleaning Procedures

- A. Empty the titration vessel and generator electrode.
- B. Rinse vessel and electrodes with methanol.
- C. Dry all glassware.
- D. Reassemble and recharge with fresh reagents.

Special Cleaning Procedure

Thorough cleaning is sometimes needed when residue begins to collect on vessel or electrodes. These instructions will detail DISASSEMBLY, CLEANING and REASSEMBLY of the titration vessel. Ketone applications should use acetone as a cleaner. Also refer to ASTM standards for your particular application.

A. Preparing for cleaning

1. For best cleaning results you'll need:
 - a. sealable glass container for reagent waste
 - b. Methanol (preferable - Isopropyl Alcohol is adequate)
 - c. plastic funnel
 - d. soap and water
 - e. paper towel or napkin
 - f. read all chemical precautions associated with your reagent
2. Prepare a sealable glass container for reagent waste. If opening is small, use the plastic funnel to empty waste into the jar.
3. Unlatch the vessel clamp. Disconnect cables for generator & detector electrodes.
4. Ketone applications with special reagents should use acetone instead of methanol.

Cleaning the Glassware

B. Disassembly

1. Unscrew the cap holding detector electrode, leaving cap and O-ring attached to electrode. Rotating the entire detector electrode (which will also turn the cap) may be easier. Remove electrode/cap/O-ring from titration vessel. Set aside.
2. Unscrew cap holding generator electrode, leaving cap and O-ring attached. Remove from titration vessel. Remove Drying Tube from top of generator electrode and set aside. Unscrew cap and O-ring for drying tube from top of generator electrode.
3. Carefully pour reagent waste into your glass container. Use funnel if necessary. Set generator electrode gently aside.
5. Unscrew the caps, O-rings and septas from the titration vessel.
5. Being careful not to drop the stirrer bar into your glass container, empty waste reagent from titration vessel.
6. Remove magnetized stirrer bar with something metallic or turn the vessel upside down on a paper towel.

Be sure the glassware is dry before adding fresh reagents.

Cleaning the Glassware

C. Cleaning

1. Rinse vessel inside and out with a splash of methanol. Because methanol absorbs moisture and evaporates quickly, any liquid left in the vessel is usually water.
2. In extreme conditions (if glassware is still dirty), clean titration vessel with soap and water, then flush with hot water. Rinse with methanol.

OR

Reassemble all glassware, fill with methanol and soak overnight.

NOTE: If residue still exists you may eventually need a bottle brush from a local grocery or drug store. Removing crude oil deposits may require a solvent such as chloroform, Xylene or hexane.

3. Drying time can be accelerated by using a blow dryer, desiccator or low temperature oven at 40-50 degrees Celsius.

NOTE: If moisture is present in vessel or generator electrode your start-up 'Precondition' time may be one hour instead of ten minutes.

4. Using methanol, rinse generator electrode inside and out. (Set aside gently).
5. If generator is still dirty, or if salt deposits have formed inside the electrode, fill the generator electrode (up to the hole) with methanol. Stand electrode in an empty titration vessel overnight. This will wash out any deposits in the generator. A long cotton swab is also effective on salt deposits.
6. Rinse detector electrode and stirrer bar with methanol. Set aside to dry.

Cleaning the Glassware

D. Reassembly

1. Place stirrer bar in clean, DRY vessel.
2. Reattach detector electrode and generator electrode.
3. Check clearance of generator electrode and stirrer bar.
4. Screw one cap/O-ring/septa to angled thread. Replace septas.
5. Using clean, dry funnel, charge (fill) titration cell to the line with anode "A" reagent.
6. Charge generator electrode with cathode "C" reagent so the level is 1/8" (2-3 mm) BELOW the vessel reagent.
7. Attach and seal other cap/O-ring, septa.
8. Screw medium cap to top of generator electrode. Change material in drying tube. Slide in drying tube.
9. Place assembled titration vessel in vessel clamp and secure.
10. Connect electrode cables. Red-tagged generator cable connects to lower socket.
11. Press power switch, adjust stirrer, then press Cell button. Allow the Aquapal to 'Precondition'.

Shipping the Aquapal

Care must be taken with the glassware when shipping the Aquapal via UPS.

DO NOT disassemble caps from Generator Electrode. Slide the large cap down to cover and protect the wire mesh from accidental damage during shipment. Even the foam packing in the accessories box can damage the wire mesh.

DO NOT pack reagents in same box as instrument or glassware.

Provide at least 1-2 inches of protective foam packing around instrument and glassware box.

See page 2 for location of glassware in the accessories box.

Appendix

Calculator Functions

$$W/w = \frac{\mu g}{W - w}$$

μg = microgram count

W = total weight of syringe + sample (gm)

w = tare (empty) weight of syringe (gm)

$$V/SG = \frac{\mu g}{V \times SG}$$

μg = microgram count

V = volume of injected sample (ml)

SG = specific gravity of sample

$$W/K = \frac{(\mu g - bl)}{W} \times K$$

μg = microgram count

W = sample weight (gm)

K = dilution ratio

bl = blank / diluent (μg)

W/K Example: 1/3 sample + 2/3 solvent
 (20 grams) + (40 grams)

μg = microgram count

bl = solvent blank * 2/3

W = weight of solvent + sample

$K = \frac{\text{weight of solvent + sample}}{\text{weight of sample ONLY}}$

Statistical Calculations

**Mean
(MN)** $\bar{X} = \frac{X_1 + X_2 + X_3 + \dots + X_n}{n}$

**Standard
Deviation
(SD)** $SD = \sqrt{\frac{(X - X_1)^2 + \dots + (X - X_n)^2}{n - 1}}$

**Coefficient
of variation
(CV)** $CV = \frac{SD \times 100}{\bar{X}}$

Troubleshooting

Problem	Cause	Remedy
Over Titration	Detector electrode senses too much iodine. Usually caused when stirrer is not active <u>and</u> CELL is on.	Make certain stirrer is spinning. Add 3-5 µg of water (less than a drop) to anode solution.
	CELL on when starting Aquapal.	CELL off, then POWER on. Adjust stirrer. Then CELL on.
	*Cable connection or <u>bent</u> detector electrode probes.	Remove detector from vessel and bend probes to parallel.
Excess Drift >34µg/min	Indicates excess moisture. Usually caused by adding sample before pressing START .	Turn CELL off and on. Check Septas for air tight seal.
	Excess moisture in headspace or on walls of vessel.	Remove vessel from clamp and SWIRL , so anode solution rises along wall to absorb moisture.
0 PPM / 0.0% or 0 µg result	Sample injected after six second delay. May also result in 'Excess Drift'.	Turn CELL and POWER off and on. Allow to 'Precondition'.
Df=25 or more	Indicates excess moisture. Reagents may be depleted.	Wait one minute before running your next test. If high drift continues, change reagents.
Power on but won't Run.	Cell is off or electrode attached To the wrong connectors	Turn CELL on. Check electrode connector

Troubleshooting

Problem	Cause	Remedy
Added new reagents and 'Precondition' takes more than one hour.	Titration vessel and/or electrodes not properly cleaned and DRIED. Glass diaphragm (frit) in generator may be clogged.	Reclean vessel and electrodes using methanol. DRY THOROUGHLY WITH HEAT GUN OR OVEN. See Cleaning the Glassware.
Bars on Aquapal display are half height.	Generator electrode not plugged in.	Check electrode leads. Generator (red) lead plugs in to bottom connector.
	Reagents are depleted.	Change reagents.
	Glass diaphragm (frit) in generator may be clogged.	Change reagents
No bubbles inside Generator Electrode with power ON, cell ON & 5 bars shown on display	Possible Bad wire on Generator	Remove vessel from clamp. Stir off, power & cell off. Move vessel far to right of Aquapal, stretching wire. Turn ON power and cell & Move wire looking for bad Connection
Changer Reagents and Precondition runs continuously going from 2 to 3 bars	Moisture in head space	Power & cell OFF. Swirl vessel. Power & cell ON. Increase stir speed to pull moisture from head space into reagent
	Possible bad connection on Detector Electrode.	Call CSC at 703-876-4030

Troubleshooting Improper Results

If Your Results Are:

It might be:

Try:

Repeatable but values keep getting lower. i.e. 17,16,17 PPM then 5,7,6 PPM for the same sample.

Moisture contamination of your syringe. Typically happens when moisture condenses in a syringe overnight or a few hours

Cleaning the syringe and needle by flushing with methanol 5-10 times to get the moisture out. Then flush a 2 -3 times with your sample before beginning a new test.

Repeatable then one high reading on the same sample. i.e. 10, 10,11, then 26.

Moisture contamination of the needle or septa.

Wiping down the outside of needle with clean towel. Even a fingerprint or moisture from a dirty rag can cause a high result.

OR

Change septa, clean and dry caps.

Non repeatable moistures for the same sample.

Sample size too small, moisture contamination of syringe or needle or sampling technique.

Increase sample and/or syringe size, see above for moisture contamination.

Non uniform distribution of water in your sample.

Mixing the sample thoroughly to ensure the water is distributed evenly. Water in some samples will sink to the bottom of the container.

Call CSC for suggestions on sampling technique for your application or consult ASTM guidelines.

CSC Scientific
1-800-458-2558

Do's and Don'ts
For First Time Users

DO

Make sure the stir motor knob is turned counter-clockwise (OFF) before turning on the power. This way you don't damage the electrodes by having the stir speed too fast.

POWER ON, then adjust stirrer, then press CELL button.

Flush your syringe and needle with alcohol at end of daily testing. Then separate plunger, glass and needle and allow to air dry. They'll be dry and ready to run the next day.

If you're using plastic disposable syringes, flush syringe and needle. SAVE your needle and throw out the syringe.

If you haven't run the Aquapal in a few days, try "swirling" the vessel to get moisture from the walls of your vessel into the "A" solution. Then turn on to Precondition.

When changing reagents, try drying your glassware with a heat gun or in an oven at 50 degrees C. Or let dry over-night. The more care you take in COMPLETELY drying your glassware, the shorter your precondition time

DON'T

Do not rinse your syringe with alcohol before running a test. Most alcohols have 10-30% WATER (10% = 100,000 PPM) and will contaminate the next sample you run.

Don't inject samples with free water into the Aquapal. If you're testing for water content, free water usually means you have a problem. By injecting a sample with free water, you'll probably deplete your reagents and have to change them.

