

CSC SCIENTIFIC COMPANY, INC.

Manufacturer and Distributor of Laboratory Equipment
2799C Merrilee Drive, Fairfax, VA 22031
703 876 4030, 800 458 2558, FAX 703 280 5142
info@cscscientific.com www.cscscientific.com

Version
v5.9

Table of Contents

Introduction	1
Getting Started	3
Training Checklist	8
Display	9
Running a Test	11
Keypad	15
Applications	20
Proper Syringe Sampling	24
Cleaning the Glassware	29
Shipping the Aquapal	34
Troubleshooting	Appendix

Introduction

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

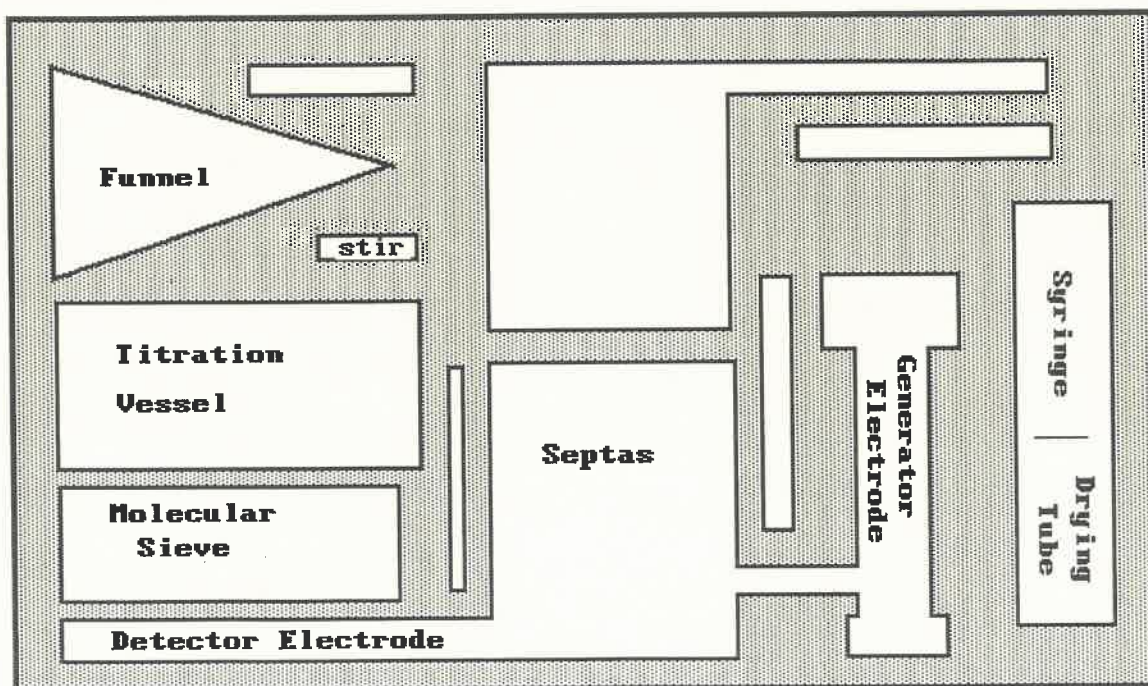
- A. The Aquapal III Titrator
- B. Glassware and accessories kit
- C. Karl Fischer reagents (supplied by Crescent Chemical)

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:

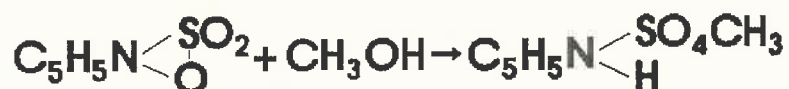
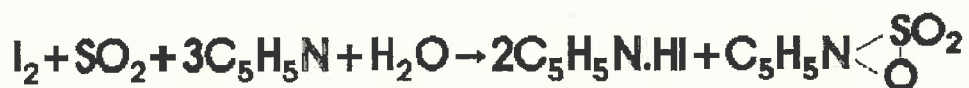
- A. Anode solution (Hydranal A, AG, AG-H or AK)
- B. Cathode solution (Hydranal CG or CK)



Principle of Measurement

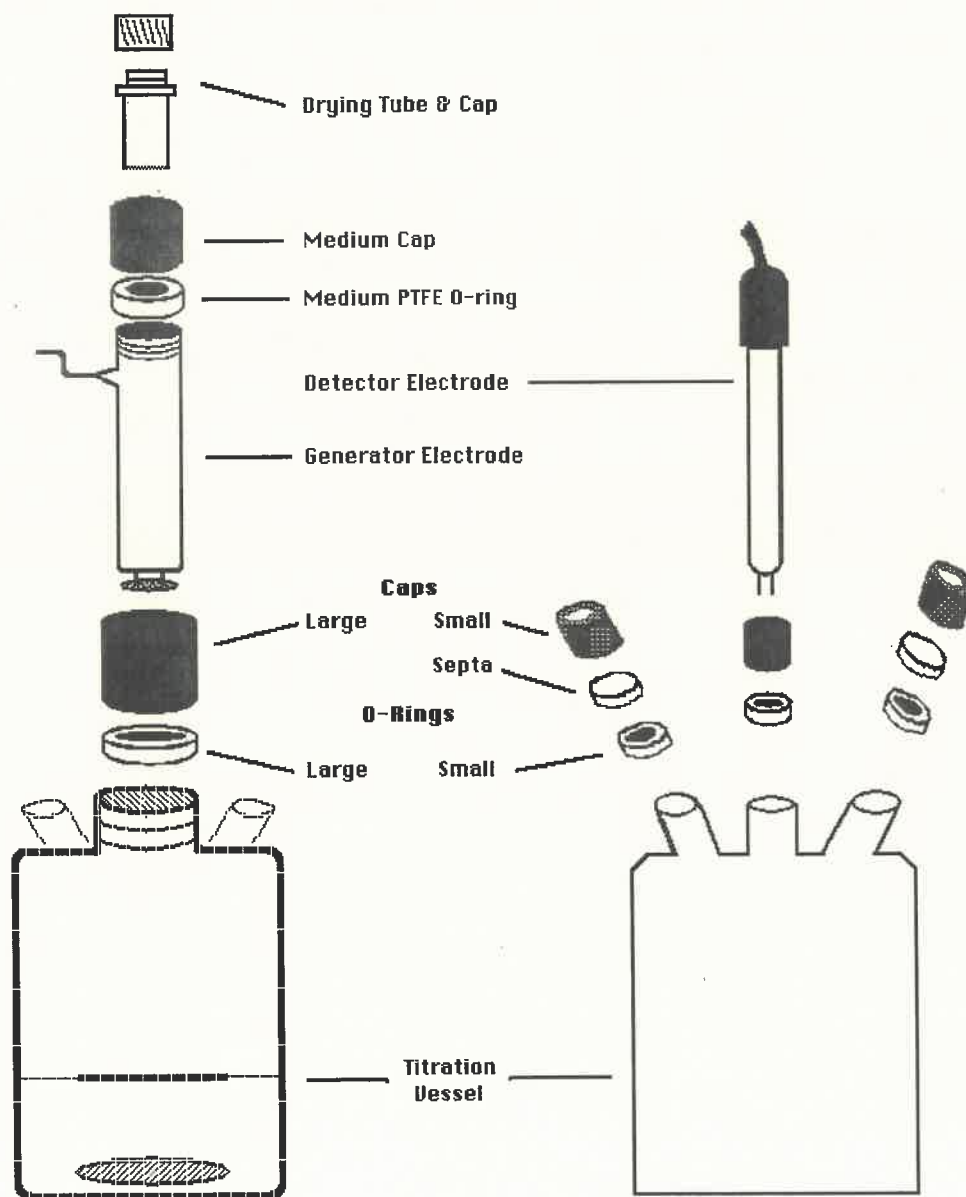
The CSC Aquapal III produces iodine at the wire mesh of the generator electrode. When electricity is conducted across the mesh, one mole of the created iodine consumes one mole of water. One milligram of water is equivalent to 10.71 coulombs of electricity. The Aquapal measures the electrical current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



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: Slide the generator electrode down into vessel as far as it will go. Then check to make sure stir bar won't hit the wire mesh.



Getting Started

Assembling the Glassware (continued)

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

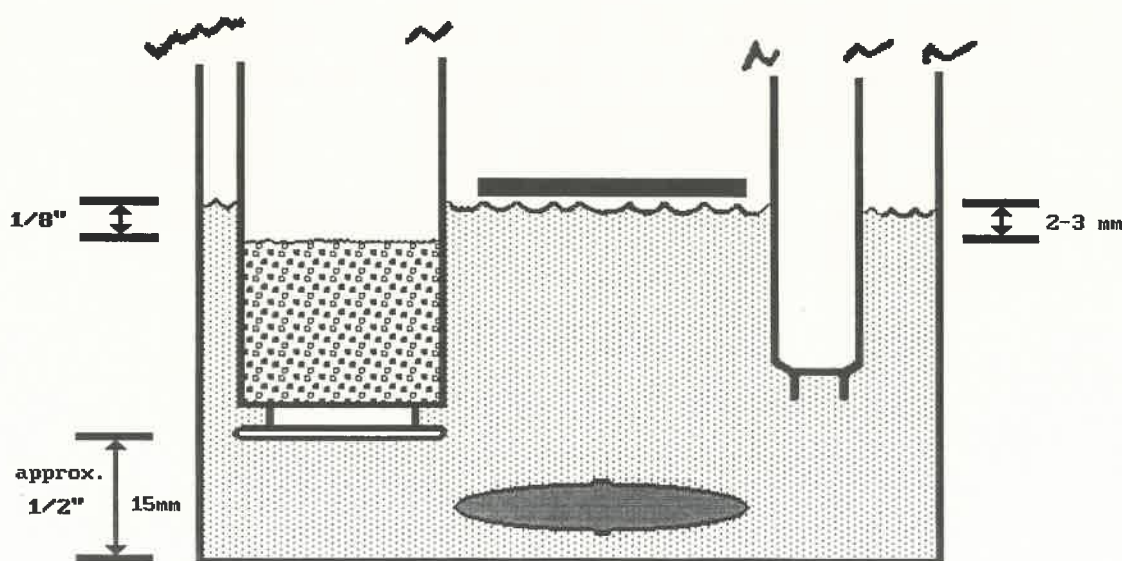
Adding Reagents

1. Reagents, Vessel and Generator: Using the funnel, fill titration cell up to the line with anode "A" reagent. Break open ampoule containing cathode "C" reagent and empty entire ampoule into the generator electrode. If cathode is in a bottle rather than an ampoule, use a syringe to charge generator with cathode "C".

NOTE: If using a reagent other than Hydranal-brand, you must keep cathode "C" at least 1/8" below anode "A" level. Check daily, either reducing "C" or adding "A".

Hydranal-brand reagents have a specially formulated cathode (CG) solution - 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. Do not over-tighten caps.
3. 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.



Start-up

Glassware

1. Before placing vessel in clamp, SWIRL the *anode* "A" solution along the inside walls of titration vessel. The swirling action picks up any moisture condensed on the inside wall of the vessel.
2. Place the assembled titration vessel in the clamp and secure. Position the vessel with the generator electrode on the left and the detector electrode on the right.
3. Check that black caps on vessel are snug enough to prevent moisture from entering the vessel but not overtightened. The generator electrode should NOT move freely inside the large cap.

Connections

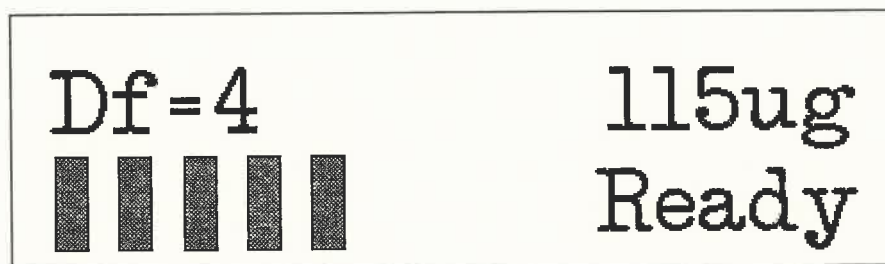
1. Connect electrode cables to sockets. The detector electrode connects to the upper socket. The generator electrode with the red-tagged cable connects to the lower socket with the red dot.
2. Attach power cable to the back of the instrument.
3. Press **POWER** switch.
4. Adjust stirrer motor, balancing 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.
5. Press **CELL** button.
6. Allow the Aquapal to 'Precondition'.

Training Checklist

After initial set-up, the first time user should know:

- ☐ 1. Assembly of glassware (page 3)
- ☐ 2. Aquapal display (page 9)
- ☐ 3. Specifics for testing a particular application
 - A. Running a test (usually V/SG - by volume - page 12)
 - B. Press **START** then inject sample
 - C. Proper syringe sampling (page 24)
 - D. If disposable syringes, use one each day
- ☐ 4. Error Messages (appendix)
 - A. Excess Drift - create by injecting sample w/o pressing START
 - B. Excess Drift - understanding and solving error message (page 27)
 - C. 0 PPM - press START w/o injecting sample
- ☐ 5. Swirling the vessel before turning on will speed Precondition.
- ☐ 6. Oil can be removed when Aquapal is turned off without changing reagents (page 20).
- ☐ 7. When to change reagents (page 28)

The Aquapal Display

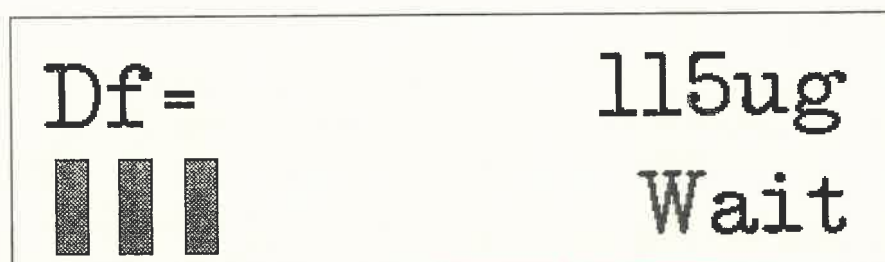


Df = Drift tracking

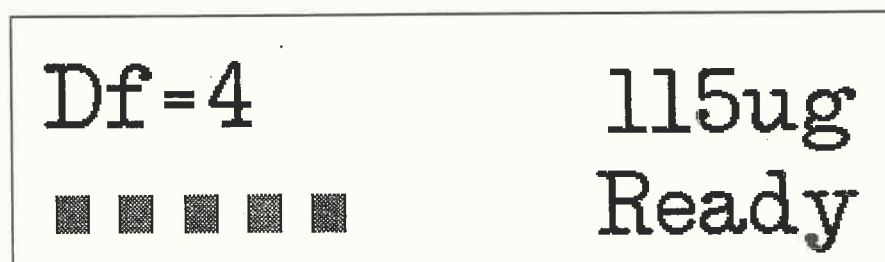
115ug = Microgram count

 = Detector signal

 = Current monitor



After test or Precondition - calculating Drift




Current Monitoring in effect

The Aquapal Display

Precondition: Initial drying mode when turned on. Water is titrated (removed) from the vessel during this mode.

ug: Microgram count. The Aquapal counts the number of micrograms water titrated during precondition and testing. 100,000 ug equals about one drop of water.

Detector Signal:

 Shows moisture level (1 to 5) in vessel. As water is removed, detector signal lowers (number of bars decreases) and titration speed slows down.

Wait: In "Wait" Mode, calculating Drift (Df). No tests can be run when in this mode.

Ready: Aquapal is "Ready" to run a test.

Running a Test

The Aquapal can step you through each point in the testing process.

Press **F5** then **3** then **ENTER** to start full printout mode.

The Aquapal will prompt you for each step needed to run a test.

Many CSC Aquapal users prefer a simpler operation: Pressing start, injecting their sample and getting their results.

Follow the suggestions on the next three pages for easier operation.

Running a test

Results by Volume

START @ CELL on. Stirrer should be rotating.

1. Press **PPM** or **%**
2. Press **U/SG**
3. Display will prompt you to enter volume in ml.

Example: press **1** then **ENTER**

OR **0** **.** **5** then **ENTER**

4. Display prompts you to enter the Specific Gravity of your sample.

Example: press **.** **8** **8** **0** then **ENTER**

(Transformer Oils are .86 - .88)

5. Wait for display to read *"Ready"*.
6. Press **Start**.
7. Inject sample -- needle goes through septa and into reagent.
8. The Aquapal calculates and prints your result.
9. To continue testing with same volume and specific gravity repeat steps 6 and 7. Volume and specific gravity entered earlier are stored in memory.
10. Statistics for a group of samples can be printed by pressing **F4**

Running a test

Results by Weight - Full and Empty Syringe

START & CELL on. Stirrer should be rotating.

1. Press **PPM** or **%**

2. Press **W/w**

3. Enter full syringe weight in grams (max. 6 digits)

Example: press **1** **5** **.** **2** **2** then **ENTER**

4. Wait for display to read "Ready".

5. Press **Start** button.

6. Inject sample -- needle goes through septa and into reagent.

7. Display will prompt you for tare weight (weight of empty syringe).

Example: press **1** **4** **.** **3** **2** then **ENTER**

8. The Aquapal calculates and prints your results.

9. For further testing repeat steps 2-7.

10. After samples have been analyzed, statistics (mean, standard deviations and coefficient of variance) can be printed by pressing **F4**

Running a test

Results by Weight - Sample Weight Only

START & CELL on. Stirrer should be rotating.

1. Press **PPM** or **%**

2. Press **W/w**

3. When display asks for full syringe weight, enter "0".

Example: press **0** **.** **0** then **ENTER**

4. Wait for display to read "Ready".

5. Press **Start** button.

6. Inject sample -- needle goes through septa and into reagent.

7. Display prompts for tare weight - ENTER SAMPLE WEIGHT ONLY.

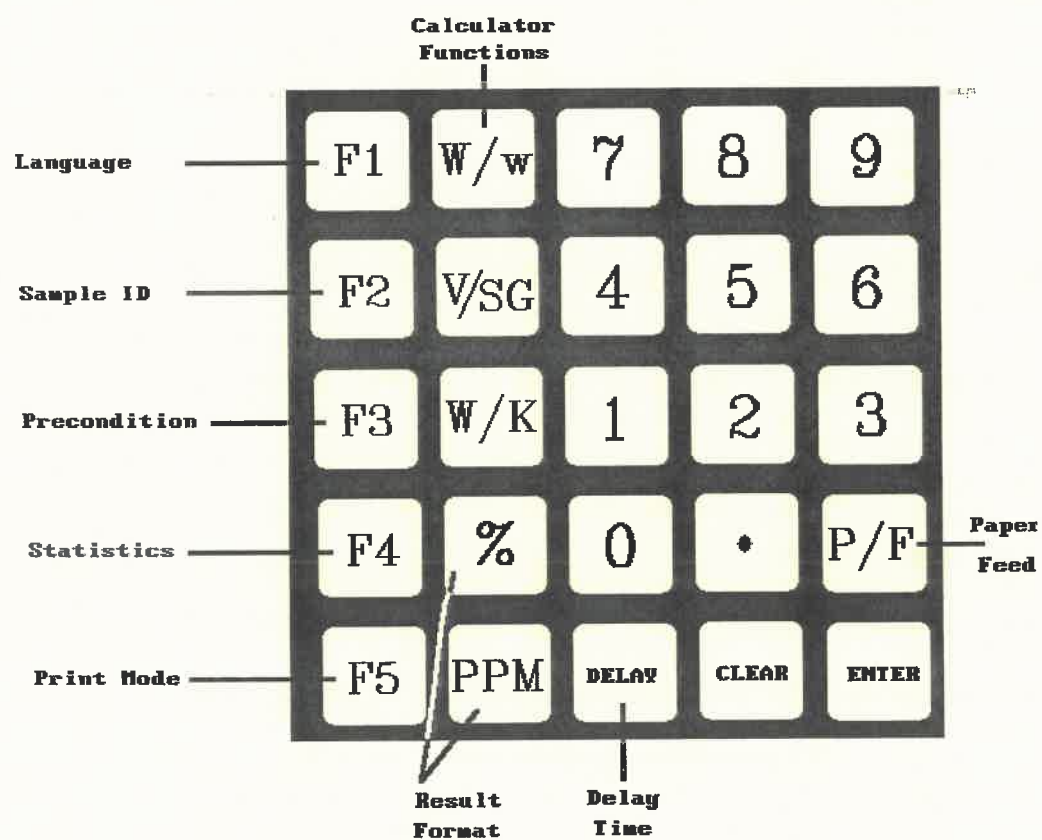
Example: press **1** **.** **3** **2** **3** then **ENTER**

8. The Aquapal calculates and prints your results.

9. For further testing repeat steps 5, 6 and 7.

10. After samples have been analyzed, statistics (mean, standard deviations and coefficient of variance) can be printed by pressing **F4**

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**. (Decimal allowed).

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

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** and then **1**.
The Aquapal will print statistics on all remaining results.

The Aquapal automatically prints statistics after analyzing 99 samples. Run number is then reset to zero. Statistics are calculated by 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 mixing any sample 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.

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. Increase the delay time to allow for sample viscosity, powder samples or other special applications.

To set delay time:

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



2. Type in the extra time required in minutes and seconds, separated by a period.

Then press **ENTER**.

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

7 min., 27 seconds type **07.27** then **ENTER**.

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

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

*Cancel DELAY during a test by pressing
the START button a second time.*

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 less precise 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. 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 three- or four-place balance, a 1 mL sample is 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. Syphon off the oil and turn ON the Aquapal. If "Precondition" time takes longer than 30 minutes, it is best to clean and recharge the cell with fresh reagents.

Transformer oil specifications vary on specific gravity. In addition, an oil's density can change from 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 enough 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 that 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.

Mercaptans and sulphides cause the most common interferences in crude oil applications. Samples containing alkyl groups will react stoichiometrically so that:

100 ppm mercaptans show as approximately 30 ppm water
100 ppm sulphides " " 50 ppm water

Improper sampling, not side reactions, cause most repeatability problems. Oils containing less than 500 ppm mercaptans or sulphides 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.

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 that clog the frit.

Reagent life shortens when working with ketones. Ketones consume reagents faster than most other applications.

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
 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 testing 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.

*Oil from fingertips, if on the needle,
can cause unrepeatable results.*

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 after completing daily tests. Then separate the needle, plunger and glass and allow to dry. The next day your syringe will be clean and dry.

If using plastic disposable syringes, use a new disposable syringe each day. If high or mixed results are seen, try a new syringe.

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.

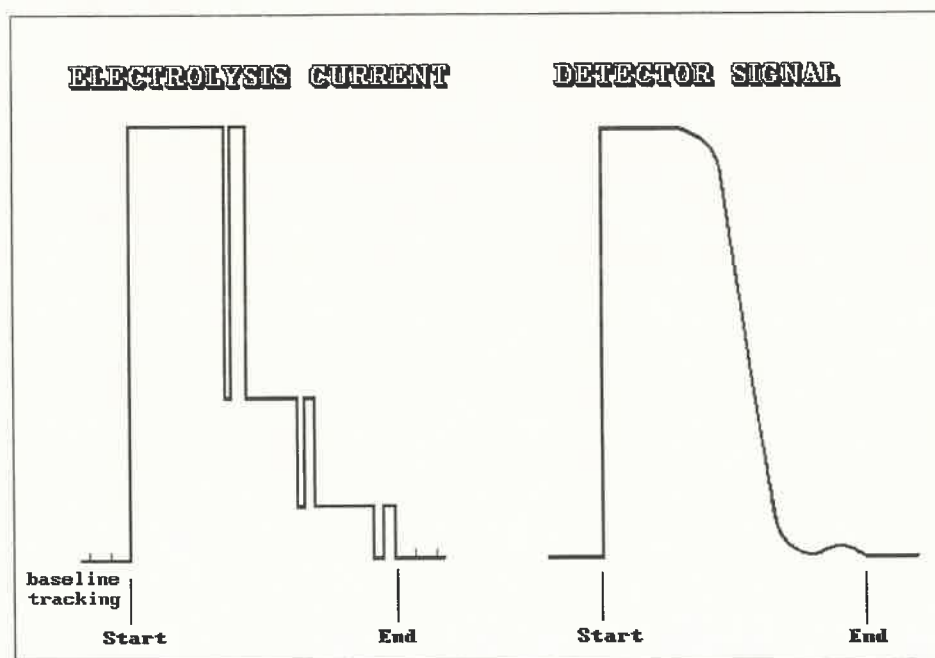
After reassembling syringe and needle, fill syringe to capacity with first sample. Empty into waste container.

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

Current Monitoring Technique

Some applications require mixing a breakdown solvent (i.e. xylene, chloroform) with the anode reagent to prevent deposits on the electrode. Solvents (and samples such as crude oil) introduced to the anode solution increase the electrical resistance in the titration vessel. At higher resistance levels the generator electrode would normally be unable to operate at 100% efficiency, leading to falsely high results.

Using a patented technique, the CSC Aquapal III monitors the efficiency of its electrolysis current. When the Aquapal finds an unstable current it automatically searches for a steady level. This automatic step-down assures accurate results through proper operation of the generator electrode.



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 >34ug/min**" displays if drift exceeds 34. This indicates one of four problems:

- 1) Titration vessel not sealed properly (check septas/caps).
- 2) Moisture in space above reagent (see below).
- 3) Reagent life almost depleted (change reagents).
- 4) 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

Reagents added to the vessel

Once reagent is added to the vessel, reagent life depends on two factors:

- 1) water introduced through samples
- 2) water introduced by background drift

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. Typical reagent life is three to four weeks.

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

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

Reagents in the bottle

Hydranal reagents in unopened bottles have no shelf life according to the manufacturer. Experience has shown that once opened, reagents last six to twelve months.

Knowing When to Change Reagents

Drift runs consistently high ($Df > 25$) before reagents need changing. High drift will not affect results.

When test time noticeably increases, change reagents. This means if your test time is typically one minute, you should change reagents when test time takes two to four minutes.

If "Precondition" time is longer than 40 minutes, and reagents have not been changed recently (within the past two weeks), reagents may be depleted and should be changed.

It's best to change molecular sieve and the pored septa when changing reagents.

v1.9 12/15/94

Cleaning the Glassware

Normal Cleaning Procedures

- A. Empty the titration vessel and generator electrode.
- B. Rinse vessel and electrodes with methanol or alcohol.
- C. DRY all glassware.
- D. Change new septa and molecular sieve.
- E. Reassemble and add new reagents

Special Cleaning Procedure

Thorough cleaning is sometimes needed when residue begins to collect on vessel or electrodes. These instructions detail DISASSEMBLY, CLEANING and REASSEMBLY of the titration vessel. 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. Use acetone only for ketone applications (AK/CK reagents).

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 also turns 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.
4. 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.

READ THIS!

1. Glassware is usually only broken during cleaning.
2. The generator electrode is especially fragile (and expensive) due to platinum mesh and wires.
3. Make sure glass is **DRY** before adding 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 try 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. Accelerated drying time by using a blow dryer, desiccator or low temperature oven at 40-50 degrees Celsius.

NOTE: It's very important to completely **DRY** all glassware before adding reagents. If moisture is present in vessel or generator electrode your start-up '*Precondition*' time may be one hour instead of ten minutes.

4. Rinse generator electrode with methanol. (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, and NEW septa.
8. Change out molecular sieve in drying tube.
9. Screw medium cap to top of generator electrode. Slide in drying tube.
10. Check caps and seals for snugness. Swirl vessel.

Shipping the Aquapal

Be careful with 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 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 (ug)

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

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

Statistical Calculations**Mean
(MN)**

$$\bar{X} = \frac{X_1 + X_2 + X_3 + \dots + X_n}{n}$$

**Standard
Deviation
(SD)**

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

**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. Inject 1-3 samples into vessel (introducing a small amount of water).
	CELL on when starting Aquapal.	CELL off, then POWER on. Adjust stirrer. Then CELL on.
	*Cable connection or <u>bent detector</u> electrode probes.	Remove detector from vessel and bend probes to parallel.
Excess Drift > 34 ug/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.
	Reagents depleted if more than three (3) weeks old.	Change reagents.
0 ppm / 0.0% or 0 ug result	Sample injected after six second delay. May cause '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 tests won't run.	CELL is off or electrodes 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. Reagents are depleted. Glass diaphragm (frit) in generator may be clogged.	Check electrode leads. Generator (red) lead plugs in to bottom connector. Change reagents.
No bubbles inside Generator Electrode with Power ON and 5 Bars show 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 Power and Cell ON, move wire looking for bad connection.
Changed reagents. Precondition runs continuously going from two to three bars.	Moisture in Headspace area of vessel.	Power and Cell OFF. Swirl vessel. Power and Cell ON. Increase stir speed to pull moisture from headspace into anode solution.

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.

Cleaning the syringe and needle by flushing with methanol 5-10 times to get the moisture out. Then flush a few 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.

Not repeatable 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.

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.

Notes

CSC Scientific: **1-800-458-2558**
1-703-876-4030

