AquaLab

Water Activity Meter

Operator's Manual
For Series 4TE, 4TEV, DUO
Version 4

Decagon Devices, Inc.

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Contents

1. Introduction

Welcome to Decagon's AquaLab Series 4TE, 4TEV, and DUO, the industry standard for measuring water activity (a_w). AquaLab is the quickest, most accurate, and most reliable instrument available for measuring water activity. Whether you are researching or working on the production line, AquaLab will suit your needs. It is easy to use and provides accurate and timely results.

About this Manual

Included in this manual are instructions for setting up your AquaLab, verifying the calibration of the instrument, preparing samples, and maintaining and caring for your instrument. Please read these instructions before operating AquaLab to ensure that the instrument performs to its full potential.

Customer Support

If you ever need assistance with your AquaLab, or if you just have questions or feedback, there are several ways to contact us:
NOTE: If you purchased your AquaLab through a distributor, please contact them for assistance.

E-mail
support@decagon.com
Please include your name, contact information, instrument serial number(s), and a description of your problem or question.

sales@decagon.com
Please include your name, address, phone number, the items you wish to order and a purchase order number. Credit card numbers should always be called in.

Phone
1-800-755-2751 (USA and Canada Only)
1-509-332-2756 International

Our Customer Support and Sales Representatives are available Monday thru Friday.

Fax
1-509-332-5158

Warranty

AquaLab has a 30-day satisfaction guarantee and a three-year warranty on parts and labor. Your warranty is automatically validated upon receipt of the instrument. We will contact you within the first 90 days of your purchase to see how the AquaLab is working for you.

Seller's Liability

Seller warrants new equipment of its own manufacture against defective workmanship and materials for a period of three years from date of receipt of equipment (the results of ordinary wear and tear, neglect, misuse, accident and excessive deterioration due to corrosion from any cause are not to be considered a defect); but Seller's liability for defective parts shall in no event exceed the furnishing of replacement parts Freight On Board the factory where originally manufactured. Material and equipment covered hereby which is not manufactured by Seller shall be covered only by the warranty of its manufacturer. Seller shall not be liable to Buyer for loss, dam-

age or injuries to persons (including death), or to property or things of whatsoever kind (including, but not without limitation, loss of anticipated profits), occasioned by or arising out of the installation, operation, use, misuse, nonuse, repair, or replacement of said material and equipment, or out of the use of any method or process for which the same may be employed. The use of this equipment constitutes Buyer's acceptance of the terms set forth in this warranty. There are no understandings, representations, or warranties of any kind, express, implied, statutory or otherwise (including, but without limitation, the implied warranties of merchantability and fitness for a particular purpose), not expressly set forth herein.

2. About AquaLab

AquaLab is the fastest and most accurate instrument for measuring water activity, giving readings in five minutes or less. Its readings are reliable, providing ±0.003 a_w accuracy. The instrument is easy to clean and checking calibration is simple.

AquaLab Model and Options

Series 4TE: User-selectable internal temperature control model, uses thermoelectric (Peltier) components to maintain internal temperature.

Series 4TEV: Uses both a chilled-mirror dewpoint sensor and a capacitance sensor for measuring non-volatile and volatile substances, respectively. Either sensor is easily selected using the instrument's menu system.

Series 4TE DUO: Uses chilled-mirror dewpoint and programmed models obtained from isotherm data to give the user both water activity and moisture content simultaneously in five minutes or less.

AquaLab 4 Instrument Specifications

Water Activity Range: 0.050 to 1.000 a_w
Water Activity Accuracy: ±0.003 (4TE Dew Point Mode)
Water Activity Accuracy: ±0.015 (4TEV Capacitance Mode)
Water Activity Resolution: 0.0001
Read Time[1]**:** ≤5 min.
Sample Temperature Range: 15 to 50° C
Sample Temperature Accuracy: ±0.2° C

Sample Temperature Resolution: 0.01° C
Sample Dish Capacity: 15ml Full
Operating Environment: 5 to 50° C 20 to 80% Humidity
Case Dimensions: 26.7 x 17.8 x 12.7cm
Weight: 3.1 Kg
Case Material: Lustran 433 (ABS) with fire retardant
Display: 64 x 128 Graphical
Data Communications: RS232A Serial, 9600 to 115200 baud
Power: 110 to 220 VAC, 50/60Hz
Warranty: 3 year parts and labor

[1] On samples with no significant impedance to vapor loss

AquaLab 4 DUO Specifications

Moisture Content Repeatability: 0.02%
Accuracy to Moisture Content Ref.: 0.1% to 0.5%

AquaLab and Water Activity

Water activity (a_w) is a measurement of the energy status of the water in a system. It indicates how tightly water is "bound", structurally or chemically, within a substance. Water activity is the relative humidity of air in equilibrium with a sample in a sealed measurement chamber. The concept of water activity is of particular importance in determining product quality and safety. Water activity influences color, odor, flavor, texture and shelf-life of many products. It predicts safety and stability with respect to microbial growth, chemical and biochemical reaction rates, and physical properties. For a more detailed description of water activity as it pertains to products, please refer to Chapter 3 of this manual, titled " Water Activity Theory".

How AquaLab Works

AquaLab uses the chilled-mirror dewpoint technique to measure the water activity of a sample. In an instrument that uses the dewpoint technique, the sample is equilibrated with the head-space of a sealed chamber that contains a mirror and a means of detecting condensation on the mirror. At equilibrium, the relative humidity of the air in the chamber is the same as the water activity of the sample. In the AquaLab, the mirror temperature is precisely controlled by a thermoelectric (Peltier) cooler. Detection of the exact point at which condensation first appears on the mirror is observed with a photoelectric cell. A beam of light is directed onto the mirror and reflected into a photo detector cell. The photo detector senses the change in reflectance when condensation occurs on the mirror. A thermocouple attached to the mirror then records the temperature at which condensation occurs. AquaLab then signals you by beeping and displays the final water activity and temperature.

In addition to the technique described above, AquaLab uses an internal fan that circulates the air within the sample chamber to reduce equilibrium time. Since both dewpoint and sample surface temperatures are simultaneously measured, the need for complete thermal equilibrium is eliminated, which reduces measurement times to less than five minutes.

AquaLab and Temperature

Samples not read at room temperature during the read cycle will equilibrate with the AquaLab's temperature before the water activity is displayed. Large temperature differences will cause longer reading times, since a complete and accurate reading will not be made until the sample and the instrument are within 2°C of each other.

There are several advantages in having a temperature-controlled water activity meter. A few major reasons are:

1. **Research purposes**. Temperature control can be used to study the effects of temperature on the water activity of a sample, make a comparison of the water activity of different samples independent of temperature, and conduct accelerated shelf-life studies or other water activity studies where temperature control is critical. There are many shelf-life, packaging, and isotherm studies in which temperature control would be very beneficial.

2. **To comply with government or internal regulations** for specific products. Though the water activity of most products varies by less than ± 0.002 per °C, some regulations require measurement at a specific temperature. The most common specification is 25°C, though 20°C is sometimes indicated.

3. **To minimize extreme ambient temperature fluctuations**. If the environmental and AquaLab temperatures fluctuate by as much as ± 5°C daily, water activity readings will vary by ± 0.01 a_w. Temperature control eliminates variations due to changes in ambient conditions.

Series 4TE/4TEV/4TE-DUO

The AquaLab Series 4TE models have thermoelectric components installed to allow the instrument to maintain a set chamber temperature. The temperature is set using the configuration menu of any of the Series 4 models.

Chilled Mirror Dewpoint Limitations

AquaLab's limitation is its ability to accurately measure samples with high concentrations (typically >1%) of certain volatiles such as ethanol or propylene glycol, which can condense on the surface of the chilled mirror. The extent of the effect is determined by how readily the material volatilizes, which is both concentration- and matrix-dependent. Therefore, even if your sample contains materials that could volatilize, it may still be possible to make accurate readings using the chilled mirror dewpoint sensor.

AquaLab Series 4TEV which incorporates both a chilled mirror sensor and a capacitance sensor for measuring volatile substances is Decagon's solution for products containing volatile materials. If you are unsure if you need the TEV model, please call and discuss your product with a Decagon Representative. Refer to Chapter 8's section titled "Volatile Samples" or call Decagon for more details.

3. Water Activity Theory

Water is a major component of foods, pharmaceuticals, and cosmetics. Water influences the texture, appearance, taste and spoilage of these products. There are two basic types of water analysis: moisture content and water activity.

Moisture Content

The meaning of the term moisture content is familiar to most people. It implies a quantitative analysis to determine the total amount of water present in a sample. Primary methods for determining moisture content are loss on drying and Karlf Fisher titration, but secondary methods such as infrared and NMR are also used. Moisture content determination is essential in meeting product nutritional labeling regulations, specifying recipes and monitoring processes. However, moisture content alone is not a reliable indicator for predicting microbial responses and chemical reactions in materials. The limitations of moisture content measurement are attributed to differences in the intensity with which water associates with other components.

Water Activity

Water activity is a measure of the energy status of the water in a system, and thus is a far better indicator of perishability than water content. *Figure 1* shows how the relative activity of microorganisms, lipids and enzymes relate to water activity. While other factors, such as nutrient availability and temperature, can affect the relationships, water activity is the best single measure of how water affects these processes.

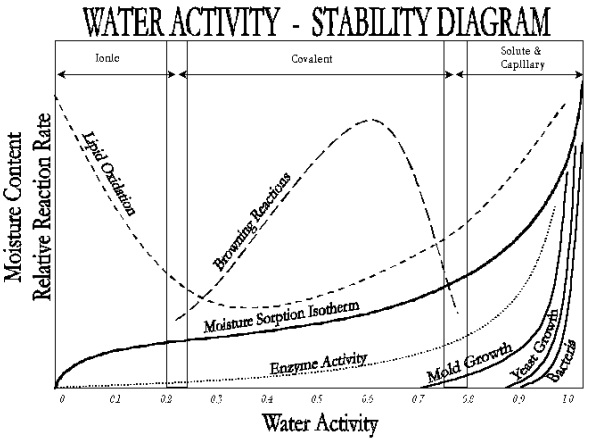


Fig. 1: Water Activity Diagram—adapted from Labuza

Water activity of a system is measured by equilibrating the liquid phase water in the sample with the vapor phase water in the head-space and measuring the relative humidity of the head-space. In the AquaLab, a sample is placed in a sample cup which is sealed inside a sample chamber. Inside the sample chamber is a fan, a dew point sensor, a temperature sensor, and an infrared thermometer. The dewpoint sensor measures the dewpoint temperature of the air in the chamber, and the infrared thermometer measures the sample temperature. From these measurements, the relative humidity of the head-space is computed as the ratio of dewpoint temperature saturation vapor pressure to saturation vapor pressure at the sample temperature. When the water activity of the sample and the relative humidity of the air are in equilibrium, the measurement of the head-space humidity gives the water activity of the sample. The purpose of the fan is to speed equilibrium and to control the boundary layer conductance of the dewpoint sensor.

In addition to equilibrium between the liquid phase water in the sample and the vapor phase, the internal equilibrium of the sample is important. If a system is not at internal equilibrium, one might measure a steady vapor pressure (over the period of measurement) which is not the true water activity of the system. An example of this might be a baked good or a multi-component food. Initially out of the oven, a baked good is not at internal equilibrium; the outer surface is at a lower water activity than the center of the baked good. One must wait a period of time in order for the water to migrate and the system to come to internal equilibrium. It is important to remember the restriction of the definition of water activity to equilibrium.

Temperature Effects

Temperature plays a critical role in water activity determination. Most critical is the measurement of the difference between sample and dewpoint temperature. If this temperature difference were in error by 1°C, an error of up to 0.06 a_w could result. In order for water activity measurements to be accurate to 0.001, temperature difference measurements need to be accurate to 0.017°C. AquaLab's infrared thermometer measures the difference in temperature between the sample and the block. It is carefully calibrated to minimize temperature errors, but achieving 0.017°C accuracy is difficult when temperature differences are large. Best accuracy is therefore obtained when the sample is near chamber temperature.

Another effect of temperature on water activity occurs when samples are near saturation. A sample that is close to 1.0 a_w and is only slightly warmer than the sensor block will condense water within the block. This will cause errors in the measurement, and in subsequent measurements until the condensation disappears. A sample at 0.75 a_w needs to be approximately 4°C above the chamber temperature to

cause condensation. The AquaLab warns the user if a sample is more than 4°C above the chamber temperature, but for high water activity samples the operator needs to be aware that condensation can occur if a sample that is warmer than the block is put in the AquaLab.

Water Potential

Some additional information may be useful for understanding what water activity is and why it is such a useful measure of moisture status in products. Water activity is closely related to a thermodynamic property called the water potential, or chemical potential (μ) of water, which is the change in Gibbs free energy (ΔG) when water concentration changes. Equilibrium occurs in a system when (μ) is the same everywhere in the system. Equilibrium between the liquid and the vapor phases implies that (μ) is the same in both phases. It is this fact that allows us to measure the water potential of the vapor phase and use that to determine the water potential of the liquid phase. Gradients in (μ) are driving forces for moisture movement. Thus, in an isothermal system, water tends to move from regions of high water potential (high a_w) to regions of low water potential (low a_w). Water content is not a driving force for water movement, and therefore can not be used to predict the direction of water movement, except in homogeneous materials.

Factors In Determining Water Potential

The water potential of the water in a system is influenced by factors that effect the binding of water. They include osmotic, matric, and pressure effects. Typically water activity is measured at atmospheric pressure, so only the osmotic and matric effects are important.

Osmotic Effects

Osmotic effects are well known from biology and physical chemistry. Water is diluted when a solute is added. If this diluted water is

separated from pure water by a semi-permeable membrane, water tends to move from the pure water side through the membrane to the side with the added solute. If sufficient pressure is applied to the solute-water mixture to just stop the flow, this pressure is a measure of the osmotic potential of the solution. Addition of one mole of an ideal solute to a kilogram of water produces an osmotic pressure of 22.4 atm. This lowers the water activity of the solution from 1.0 to 0.98 a_w. For a given amount of solute, increasing the water content of the systems dilutes the solute, decreasing the osmotic pressure, and increasing the water activity. Since microbial cells are high concentrations of solute surrounded by semi-permeable membranes, the osmotic effect on the free energy of the water is important for determining microbial water relations and therefore their activity.

Matric Effects

The sample matrix affects water activity by physically binding water within its structure through adhesive and cohesive forces that hold water in pores and capillaries, and to particle surfaces. If cellulose or protein were added to water, the energy status of the water would be reduced. Work would need to be done to extract the water from this matrix. This reduction in energy status of the water is not osmotic, because the cellulose or protein concentrations are far too low to produce any significant dilution of water. The reduction in energy is the result of direct physical binding of water to the cellulose or protein matrix by hydrogen bonding and van der Waal forces. At higher water activity levels, capillary forces and surface tension can also play a role.

Sorption Isotherms

Relating Water Activity to Water Content

Changes in water content affect both the osmotic and matric binding of water in a product. Thus a relationship exists between the water activity and water content of a product. This relationship is

called the sorption isotherm, and is unique for each product. Besides being unique to each product, the isotherm changes depending on whether it was obtained by drying or wetting the sample. These factors need to be kept in mind if one tries to use water content to infer the stability or safety of a product. Typically, large safety margins are built into water content specifications to allow for these uncertainties.

While the sorption isotherm is often used to infer water activity from water content, one could easily go the other direction and use the water activity to infer the water content. This is particularly attractive because water activity is much more quickly measured than water content. This method gives particularly good precision in the center of the isotherm. In order to infer water content from water activity, one needs an isotherm for the particular product. Decagon sells an Isotherm Generator called the AquaSorp IG or you can also have Decagon run the isotherm for a fee.

For example, if one were using the AquaLab to monitor the water content of dried potato flakes, one would measure the water activity and water content of potato flakes dried to varying degrees using the standard drying process for those flakes. An isotherm would be constructed using those data, and the water content would be inferred using the measured water activity of samples and that isotherm. We have an upgrade available to Series 4TE users that would allow you to determine moisture content and water activity simultaneously. This instrument is called the Series 4TE DUO.

The importance of the concept of water activity of foods, pharmaceuticals, and cosmetics cannot be over emphasized. Water activity is a measure of the energy status of the water in a system. More importantly, the usefulness of water activity in relation to microbial growth, chemical reactivity, and stability over water content has been shown.

4. Getting Started

Components of your AquaLab

Your AquaLab should have been shipped with the following items:

- **AquaLab water activity meter**
- **Calibration Certificate**
- **Power cord**
- **RS-232 interface cable**
- **100 disposable sample cups**
- **Operator's Manual**
- **Quick Start guide**
- **Cleaning Kit**
- **3 vials each of the following verification solutions:**

  1.000 a_w Distilled Water

  0.760 a_w 6.0 molal NaCl

  0.500 a_w 8.57 molal LiCl

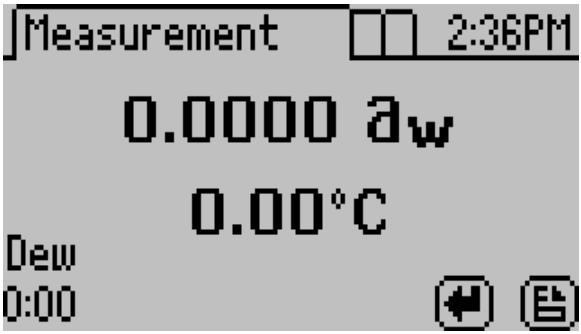
  0.250 a_w 13.41 molal LiCl

Choosing a Location

To ensure that your AquaLab operates correctly and consistently, place it on a level surface. This reduces the chance that sample material will spill and contaminate the sample chamber. Also select a location where the temperature remains fairly stable to avoid temperature changes that can affect accuracy. This location should be well away from air conditioner and heater vents, open windows, etc. Place the AquaLab in a location where cleanliness can be maintained to prevent contamination of the sample chamber.

Preparing AquaLab for Operation

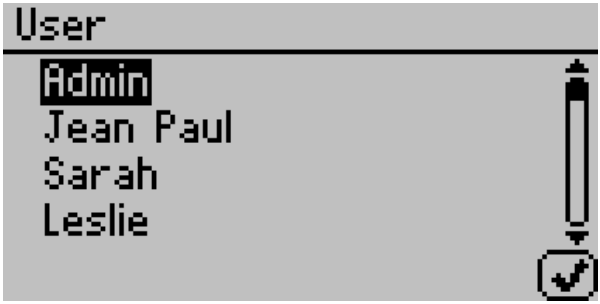
After finding a good location for your AquaLab, plug the power cord into the back of the unit. The ON/OFF switch is located on the lower left corner of the AquaLab's back panel. When the AquaLab is turned on, you should see a model name/number screen and then the main screen as shown below.



The main screen shows the water activity (a_w) in the middle of the screen and the sample temperature right below. On the Series 4TEV model you will also see either DEW or CAP indicating whether you are using the dewpoint or capacitance sensor respectively.

NOTE: In order to provide the most accurate readings, your AquaLab should be allowed a 15 minute warm-up period.

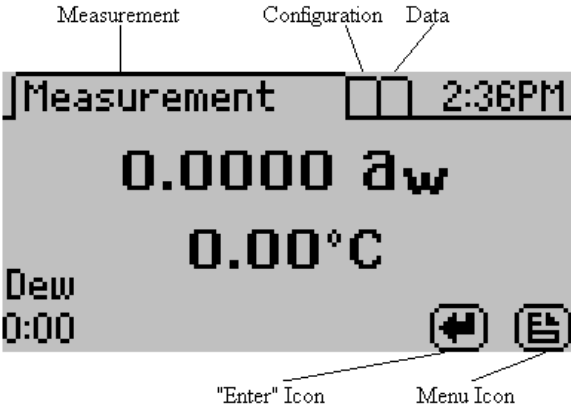
If users have been setup on the instrument, the following screen will appear instead of the main screen. (See Chapter 5 for more information on administrative settings and user setup).



Select the appropriate user and login to begin.

5. Menus

At the top of the display screen there are three tabs: Measurement, Configuration, and Data. These tabs indicate the three menus you can access. To change between the tabs press the right most button below the document icon.

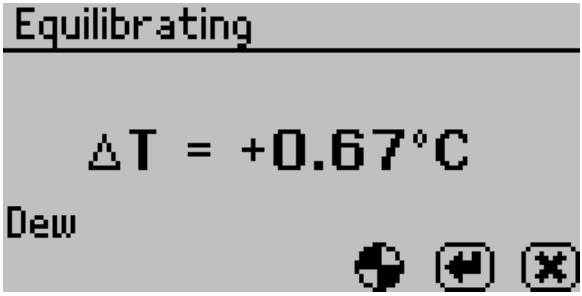


The enter icon is the read or enter button. Once the latch is set to the read position, the document icon will switch to an "X" icon, which allows the user to stop the current reading. During a reading, pressing enter again will restart the reading.

Measurement Tab

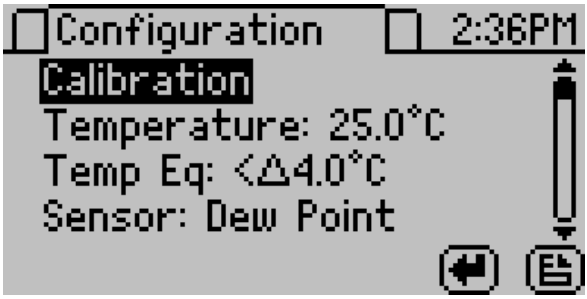
The measurement tab, as seen above, is the main screen which displays each time you turn on your AquaLab. If this screen doesn't appear, refer to Chapter 12 for troubleshooting instructions. As mentioned earlier, the water activity and sample temperature are displayed on the screen.

Pushing the right or left arrow keys will change the display to a temperature equilibration screen shown below. This screen shows the temperature difference between the sample temperature and the block temperature.

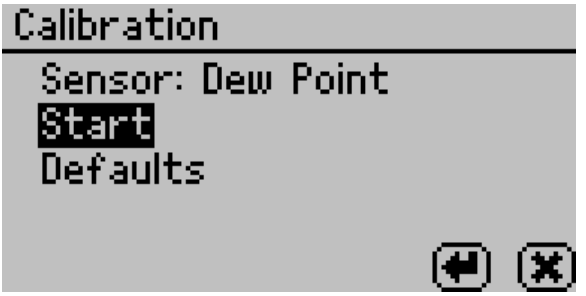


Configuration Tab

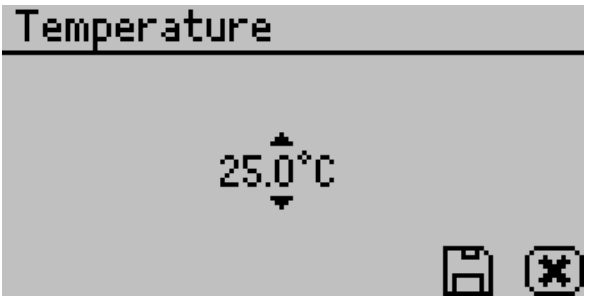
When at the configuration screen, pressing the up and down arrow keys moves the cursor through the various configuration options Press the left and right arrows to page through the options. The enter button will allow you to change the highlighted setting.



Calibration:
Pressing the Enter button when Calibration is highlighted starts the verification process. For more details on the verification procedure refer to Chapter 7. You may also reset the calibration to the factory defaults by highlighting the Defaults option and pressing Enter. This will reset all options to the way they were when the instrument arrived at your location.



Temperature:
The default temperature is 25°C. Press the enter button to change the temperature setting. The AquaLab Series 4TE models may be set between 15 and 50°C by 0.1°C intervals. Using the up and down arrows, set the AquaLab to your desired temperature and press the save button.



Temp Eq:
The Temperature Equilibration option allows you to set the level of temperature equilibration desired before the water activity measurement begins. The range is 0.5 to 4.0°C. A setting of 4.0°C begins the measurement immediately (assuming the sample is not >4.0°C above or below the block temperature). A setting of 0.5 °C will cause the instrument to wait until the sample temperature is within <0.5°C of the block temperature before starting the water activity measurement.

Sensor:
In the AquaLab Series 4TEV model only, this option indicates the selected sensor type, either dewpoint or capacitance (The Series 4TE models will always be Dewpoint). Pressing Enter when the Sensor option is highlighted allows you to change between a chilled mirror dewpoint or capacitance sensor for sampling with or without volatiles, respectively.

Mode:
Users may choose between single, continuous, or custom mode by pushing the save button.

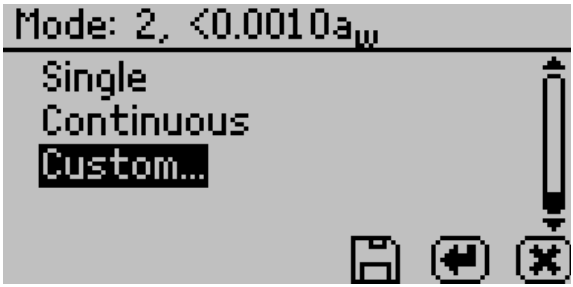
Single Mode
Single mode reads the sample once, after which the instrument notifies you that it is finished and the water activity and temperature are displayed on the screen.

Continuous Mode
Continuous mode reads your sample until you open the chamber lid or stop the test using the stop button. AquaLab reads the sample, displays the water activity and temperature, then begins another read cycle without further input from the user. Between samples, the machine will signal you with beeps. This mode eliminates the possibility of moisture exchange with the environment outside the chamber in between readings. A time on the bottom left of the screen tracks the cumulative read time. All readings taken during continuous mode are saved on the instrument's memory if the autosave feature is selected (see Auto Save below). If AquaLab is connected to a computer using AquaLink RG (See Chapter 11), all readings taken during continuous mode will be downloaded to the AquaLink RG software.

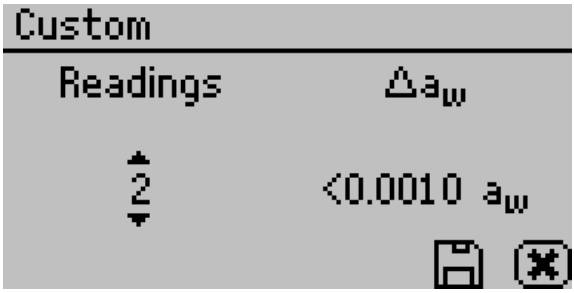
Custom Mode
Custom mode allows a sample to be read multiple times until a desired level of stability is achieved. The user determines how many consecutive tests they want to be within a given water activity stability setting. For instance, the customer can choose to have 4 consecutive tests be within +/- 0.001a_w. The instrument will continue to run tests until it records 4 tests that are within +/- 0.001a_w and then will stop and report the value of the final test. If autosave is turned on, all test readings will be saved to the instruments memory, but only the final reading will appear on the main

measurement screen. If AquaLab is connected to a computer using AquaLink RG (See Chapter 11), all readings taken during a custom mode test will be downloaded to the AquaLink RG software.

On the mode screen, at the top of the page, will appear the current mode settings with the number of tests appearing first, followed by the stability value (Δa_w). Pressing enter with the custom mode highlighted will allow the number of tests and stability settings to be changed.



To change the number of readings, use the right/left arrow buttons to highlight the number under Readings, and then use the up and down buttons to change to any value between 2 and 9.

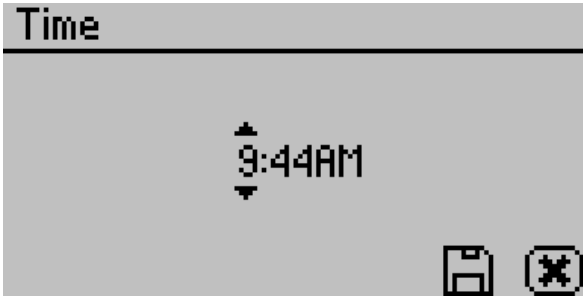


To change the stability setting, use the right/left arrow buttons to

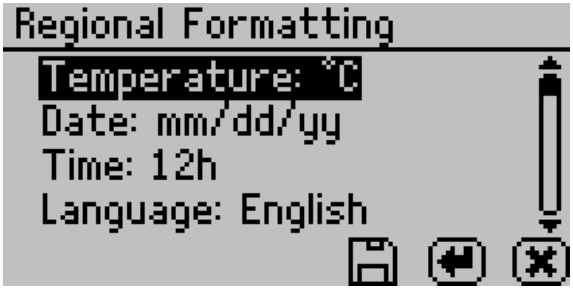
highlight the number under Δa_w, and then use the up and down buttons to change to any value between 0.0005 and 0.0030. To save the settings and finish, press the save button (to exit without updating, press the cancel button). The mode screen will now appear with the updated custom settings appearing at the top of the screen. Press the save button to return to the configuration screen and begin using the custom mode (To exit without updating, press the cancel button).

Date:
AquaLab Series 4 models now have an internal calendar and clock. The time and date are recorded with each water activity reading. Pressing Enter when the Date option is highlighted allows you to set the date in the instrument. Press the left and right arrows to change between the month, day and year. Press the up or down arrows to change any of the individual values.

Time:
Pressing Enter when the Time option is highlighted allows you to set the current local time. Press the up or down arrows to change any of the individual values. Press the left or right buttons to change between hour and minutes. The hour setting automatically changes between AM and PM.



Regional Formatting:
Allows you to configure how all Series 4 models will display information. You may choose the temperature scale (Celsius vs Fahrenheit), the date display (mm/dd/yy vs. dd/mm/yy), the hour format (12 vs 24 hour) and the language.



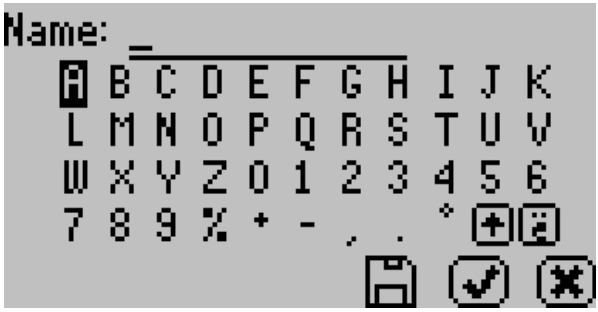
Admin Settings

Allows you to create an administrator password as well as create, edit and delete additional users.

The admin option allows the administrator to grant or block access to some or all of the configuration options in all Series 4 models. For example: If the administrator wanted to make sure that all samples were read at 25°C the administrator would set their temperature to 25°C and then would lock all other users out of that configuration screen. This is accomplished by entering the Access function and selecting the desired option to toggle it on and off.

Additionally you can lock and unlock all of them at once. For example, if you do not want John Doe changing the instruments measurement temperature, the administrator can lock that function for John. The areas that can be locked are calibration, temperature, temperature equilibration, sensor selection, mode, date/time, region, password, auto-save, number of beeps, contrast, and delete functions.

User Setup:
Users can be added, edited or deleted from this screen. An alphabet screen will appear where a name can be entered using lower case, upper case and accents.



NOTE: User setup is not required for instrument operation. It is in place for users wanting to be compliant with 21 CFR Part 11 or who want to maintain the settings they have selected.

Auto Save:
AquaLab Series 4 models have the ability to store water activity readings within the instrument. By selecting Auto Save "On" every water activity reading will be automatically stored in the instruments internal memory. AquaLab Series 4 can store up to 10,000 records before the memory is full. If you select Auto Save "off" then no data is automatically stored, although any individual reading may be manually stored at any time.

To manually store a water activity or append an annotation to the active reading that has been autosaved. Press the save icon button after the water activity measurement is completed. Pressing the icon opens a "name" screen. You may give this reading a name by pressing the arrow buttons to highlight the letter and then pressing the

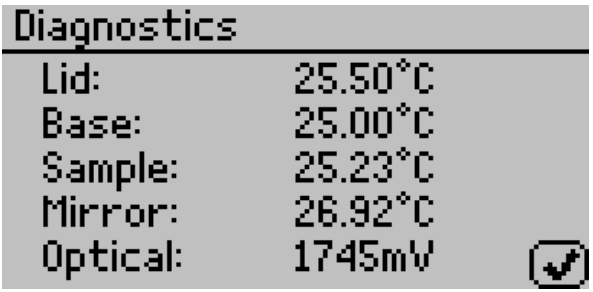
"Check" icon button. Press the save icon to save this data record with the name you have specified.

NOTE: Pressing the save icon button without giving it a name will save the reading without a name.

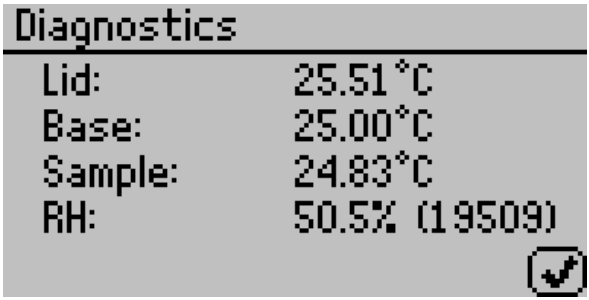
Beeps:
Allows you to set the reading finished notification from 4 beeps to continuous beeps. You may also turn the audible notification off.

Contrast:
Allows you to set the contrast of the screen to your liking. Viewing the screen from a sitting versus a standing position may require contrast adjustment for the best visibility in that position.

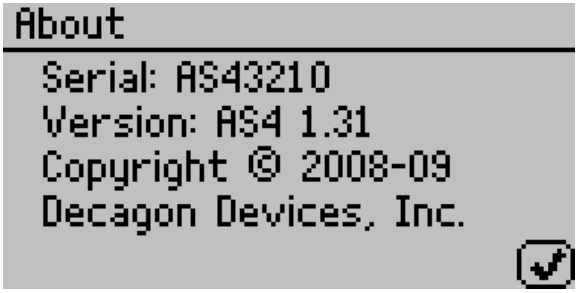
Diagnostics:
For the chilled-mirror dewpoint sensor it provides you with lid, base, sample and mirror temperature as well as an optical voltage.



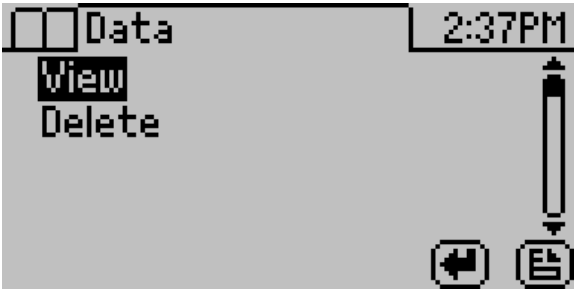
For the capacitance sensor (TEV Models only) it provides you lid, base, and sample temperatures as well as relative humidity.



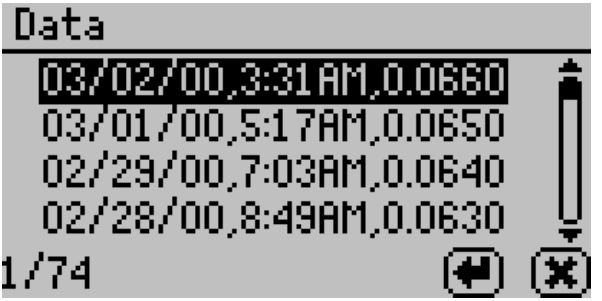
About:
This screen provides important information including the serial number and code version of your instrument.



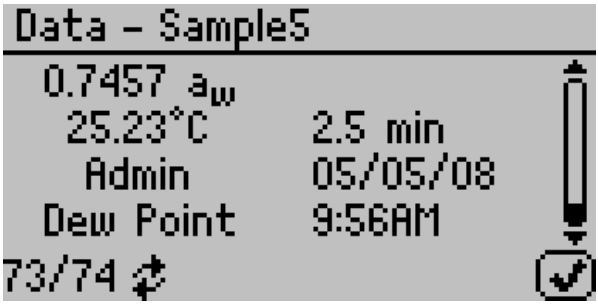
Data Tab



View:
This selection will allow you to view your stored measurements. The up/down arrows will move you through the stored data with the most recent measurements at the top of the table. You may also press the left and right arrows to page quickly through the data. See Chapter 11: Computer Interface for information about downloading these readings to a computer.

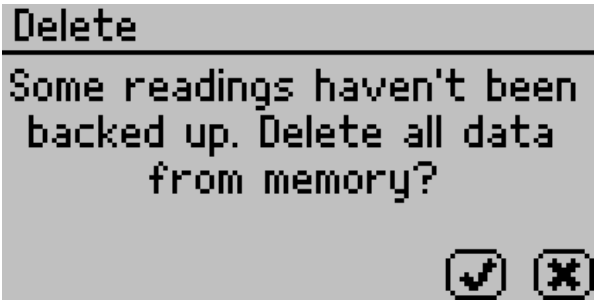


When you are viewing the summary screen, you may press the enter button on a highlighted reading to get detailed information on the reading as shown below.



The information shown is the water activity of the sample, the temperature, the time the reading took, the user who ran the test (if setup), the date of the reading, the sensor used (4TEV only), the time the reading was taken, and the sequence number of the stored reading.

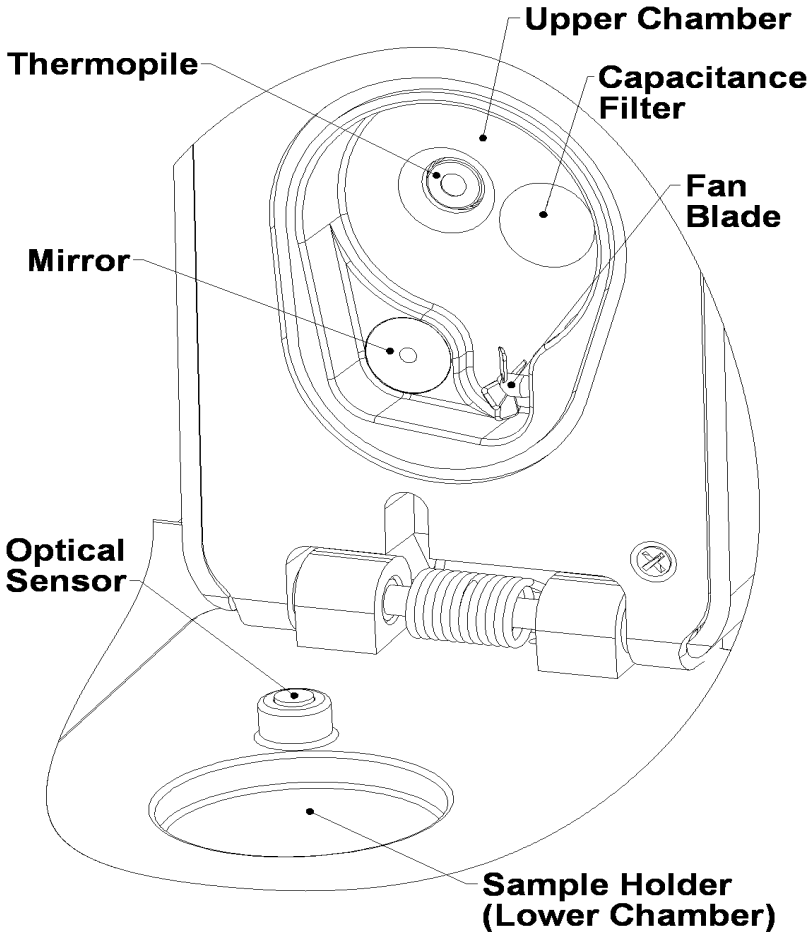
Delete:
Selecting this option will delete all of the information currently stored in the instrument. If you have not backed up this information you will be reminded of this by the following message:



*NOTE: You will **NOT** be able to recover deleted data.*

6. Cleaning and Maintenance

Keeping your AquaLab clean is vital to maintaining the accuracy of your instrument. Dust and sampling debris can contaminate the sampling chamber and must therefore be regularly cleaned out. To clean your instrument, carefully follow these instructions and refer to the labeled diagram below.



Purpose

The purpose for the cleaning procedure is to remove grease, dirt and other soluble substances which can absorb/release water during verification, calibration, and/or sample testing. For a smooth and even dew formation, it requires the mirror to be perfectly clean. If there are any contaminants (e.g. fingerprints) on the mirror, the dew will form unevenly and thus affect the accuracy of the reading.

Materials Needed

- A thin plastic rod or other non-metal implement
- Distilled Water
- Isopropyl Alcohol (IPA) or Decagon Cleaning Solution
- Kimwipes®

You may also purchase the AquaLab Cleaning Kit which comes with all the above materials except the Isopropyl Alcohol and Distilled Water.

NOTE: Wash your hands with soap and water and/or use clean lab gloves before starting the cleaning procedure. This will prevent oils from contaminating the cleaning materials, the sample chamber and/or the sensors.

Cleaning the Block and Sensors

Accessing the Sample Chamber

Turn the power off on your AquaLab. If latched, move the lever over to the open position. Lift the chamber cover to expose the sample chamber and sensors. The sample chamber consists of all surfaces inside the red o-ring when the lid is closed.

NOTE for Series 4TEV: If cleaning an AquaLab Series 4TEV, follow the cleaning procedures listed below being careful not to get cleaning solution or alcohol on the capacitance sensor filter (see illustration on

previous page) Repeated exposure of cleaning materials or contaminants to the filter may cause inaccurate readings. If the filter appears contaminated, replace it while being careful not to disturb the sensor behind the filter.

Cleaning Procedure:

Cleaning your AquaLab is a multi-step procedure which involves washing, rinsing, and drying for each specific area as outlined below:

1. <u>Cleaning the Sample Chamber</u>

Note: Be extremely careful not to damage the fan blades (see illustration) when cleaning the chamber.

a. Remove any debris that may have collected within or around the sample chanber.
b. Wrap a NEW Kimwipe around the end of the thin plastic rod (spatula) and moisten it with isopropyl alcohol or Decagon Cleaning Solution. *Note: Do NOT dip a <u>used</u> Kimwipe into your container of IPA or cleaning solution (the IPA or cleaning solution will become contaminated).*
c. WASH—Clean upper chamber, o-ring, and all surfaces of the block within the o-ring. You may need to replace the Kimwipe if it becomes too dirty during this process.
d. Clean lower block with a fresh Kimwipe. Be sure to clean the entire block surface.
e. RINSE—Repeat steps b-d using new Kimwipes with distilled water.
f. DRY—Repeat steps b-d using new, dry Kimwipes to help remove any moisture remaining from the cleaning.
g. Visually inspect the sample chamber for cleanliness. Re-clean if necessary. *Note: Do not reuse Kimwipes.*

2. Clean the Mirror

a. Wrap a new Kimwipe around the end of the thin plastic rod (spatula) and moisten it with isopropyl alcohol or Decagon Cleaning Solution.
b. WASH—Swipe the moistened Kimwipe across the mirror once. (A single swipe is usually sufficient to remove contaminants.)
c. RINSE—Repeat steps a-b using new Kimwipes moisted with distilled water instead of cleaning solution.
d. DRY—Repeat steps a-b using new, dry Kimwipes to help remove any moisture remaining from the cleaning.
e. Visually inspect the mirror for cleanliness. Re-clean if necessary.

3. Clean the Thermopile and Optical Sensor

a. Wrap a new Kimwipe around the end of the thin plastic rod (spatula) and moisten it with isopropyl alcohol or Decagon Cleaning Solution.
b. WASH—Swipe the moistened Kimwipe across thermopile and optical sensor. (A single swipe across the sensor is usually sufficient to remove contaminants.)
c. RINSE—Repeat steps a-b using new Kimwipes moistened with distilled water instead of cleaning solution.
d. DRY—Repeat steps a-b but use a new, dry Kimwipe to help remove any moisture remaining from the cleaning.
e. Visually inspect the thermopile and optical sensor for cleanliness. Re-clean if necessary.

4. Additional Drying Time

a. Visually inspect the sample chamber and sensors for contaminants, including moisture. If necessary, repeat the cleaning process using new Kimwipes.
b. Let stand for about 5 minutes to ensure the sample chamber is dry.

Verification of Calibration

After you have cleaned the chamber and other parts of your AquaLab, it is important to check the instrument's performance in order to correct for any linear offset that may have occurred during the cleaning process.

Before you check the instrument we recommend that you run a sample of the activated charcoal pellets provided in your AquaLab cleaning kit. This cleans the air inside the chamber, helping it come back to a stable sampling environment.

Verify the linear offset against known verification standards according to the procedure described in the next chapter. If a linear offset has occurred, refer to "adjust for linear offset" section in Chapter 7 for directions on how to correct for linear offset. If, after adjusting for linear offset, your instrument is still not reading samples correctly, please contact Decagon for support.

7. Verification and Calibration

It is important to verify AquaLab's water activity calibration against known standards to guarantee optimal performance and accuracy. Decagon recommends verification daily, once per shift or before each use.

Water Activity Verification

AquaLab uses the chilled-mirror dewpoint technique to determine water activity. Because this is a primary measurement of relative humidity, no calibration is necessary; however, it is important to verify for linear offset periodically. The components used by the instrument to measure water activity are subject to contamination which may affect the AquaLab's performance. When this occurs, it changes the accuracy of the instrument. This is what is called a "linear offset." Therefore, frequent verification assures you that your AquaLab is performing correctly. Linear offset is checked by using two different verification standards.

Verification Standards

Verification standards are specially prepared unsatuarated salt solutions having a specific molality and water activity constant which are accurately measurable. The verification standards that were sent with your initial shipment are very accurate and readily available from Decagon. Using verification standards to verify accuracy can greatly reduce preparation errors. For these reasons, we recommend using standards available through Decagon for the most accurate verification of your AquaLab's performance.

Performance Verification Standards come in five water activity levels: 1.000, 0.984, 0.760, 0.500, and 0.250 a_w. The standards are

produced under a strict quality assurance regime. Please contact Decagon Devices to order additional standards via sales@decagon.com or 1-800-755-2751.

Verification Standard @ 25°C	Water Activity
Distilled Water	1.000 ±0.003
0.5m KCl	0.984 ±0.003
6.0m NaCl	0.760 ±0.003
8.57m LiCl	0.500 ±0.003
13.41m LiCl	0.250 ±0.003

NOTE: If you need to obtain a Material Safety Data Sheet (MSDS) for any of these standards, a printable version is available on our website at www.decagon.com/msds.

To use a verification standard, remove the twist top and pour the contents into an AquaLab sample cup. If for some reason you cannot obtain Decagon's verification standards and need to make a saturated salt solution for verification, refer to Appendix A.

Verification of Calibration

When to Verify for Linear Offset

Linear offset should be checked against two known verification standards daily, either once per shift or before each use. Linear offset should never be verified solely against distilled water, since it does not give an accurate representation of the linear offset. For batch processing, the instrument should be checked regularly against a known standard of similar water activity. It is also a good idea to check the offset with a standard of similar water activity when the general

water activity range of your sample is changing. Checking the water activity of a standard solution will alert you to the possibility of unit contamination or shifts in the linear offset from other causes.

Note: The verification process is the same for both the dewpoint and capacitance sensors in TEV models except that the accuracy for the capacitance sensor is ± 0.015 a_w.

Verification

To verify for linear offset of your AquaLab, do the following:

1. Choose a verification standard that is close to the water activity of the sample you are measuring. Note: The AquaLab needs to warm up for approximately 15 minutes to make accurate readings.

2. Empty a vial of solution into a sample cup and place it in the AquaLab's testing chamber. Make sure that your standard is as close to the instrument temperature as possible.

Note: Make sure the rim and outside of the sample cup are clean.

3. Carefully close the lid and move the lever to the READ position.

4. Take two readings. The water activity readings should be within ± 0.003 a_w of the given value for the verification standard. See **Appendix B** for the correct water activity value of Decagon's standards at temperatures other than 25°C.

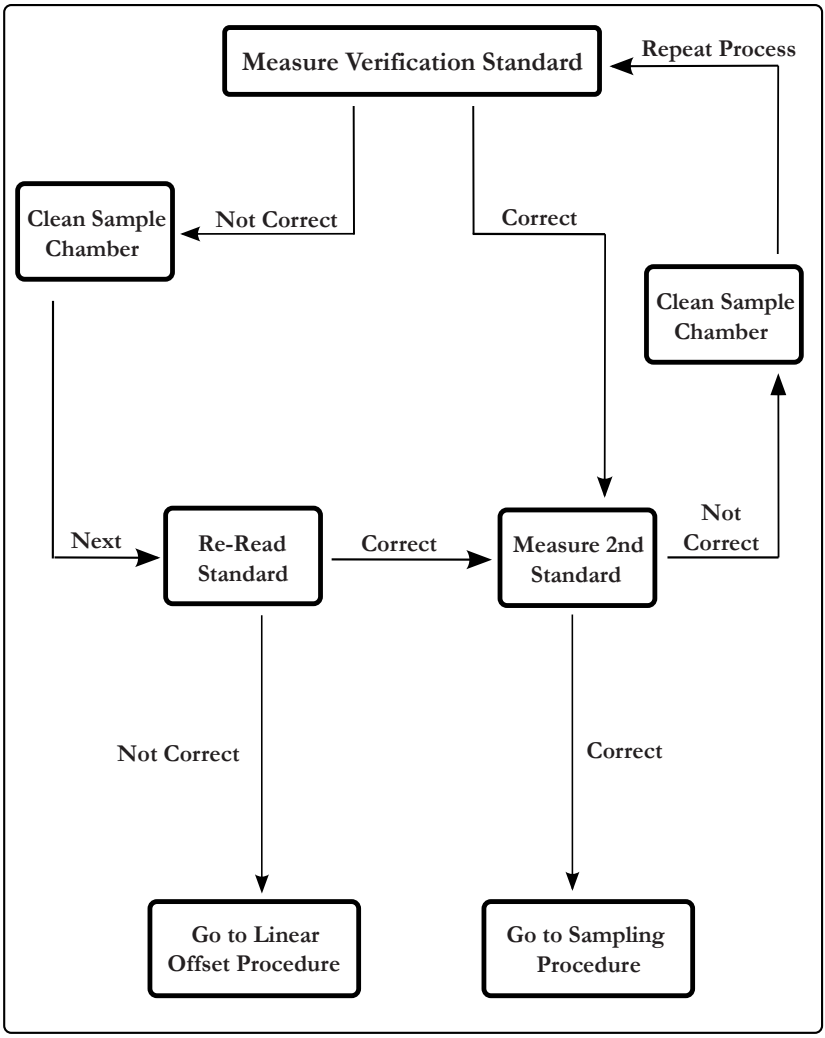
5. If your AquaLab is reading within ±0.003 a_w of the verification standard, chose a second verification standard that would border the range of water activity you plan to test. For example, if you plan

to test for water activity readings ranging between 0.713 and 0.621 you should use the 6.0M, NaCl ($0.76a_w$)standard for your first verification and the 8.57M LiCl ($0.50a_w$) for the second verification.

6. Prepare a sample cup of the second verification standard and make two readings. The second water activity reading for the second verification standard should be within ±0.003 a_w.

7. If either of the verification standards is not correct, it is probably due to contamination of the sensor chamber. For cleaning instructions, see Chapter 6. After cleaning, repeat verification from step two.

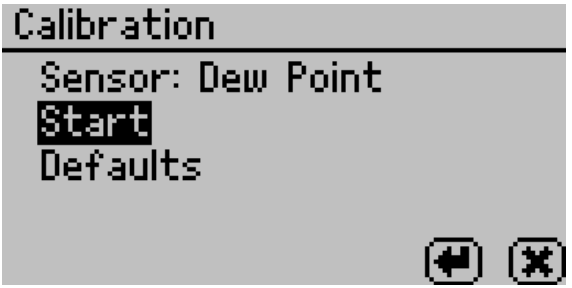
8. If you are consistently getting readings outside the water activity of your first verification standard by more than ±0.003 a_w, a linear offset has probably occurred. In this case, adjust the reading to match the verification standard's correct value as outlined in the next section.



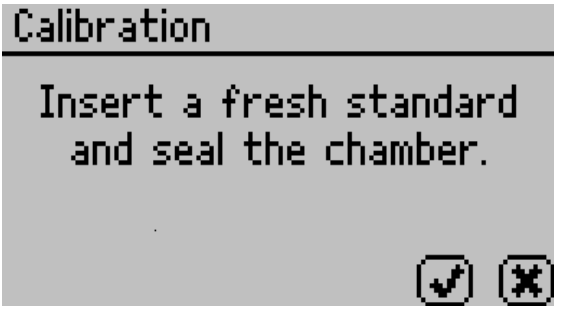
This flowchart is a graphical representation of the directions given above for checking for linear offset.

Adjust for Linear Offset

1. Once you are certain a linear offset has occurred, toggle to the Configuration tab by pressing the Menu icon button. Calibration is the first option highlighted in the configuration tab. Press the Enter icon button to begin the verification process. You will be guided through the linear offset routine through on screen commands. The following screen will appear:



2. Press the Enter button to start the linear offset process. To return to the main menu, press the cancel button. After pressing the enter button, the following screen will appear:



3. Empty the whole vial of solution into a sample cup. We recommend using the 6.0 NaCl (0.76a_w). Do not adjust for the offset us-

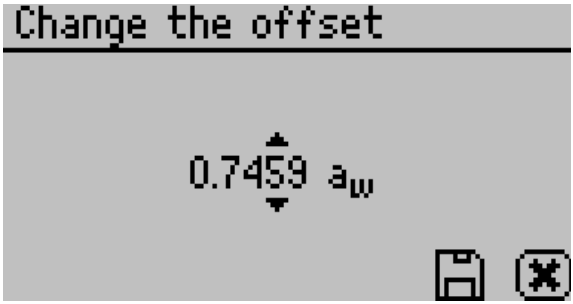
ing distilled water. Ensure the rim and outside of the cup are clean. Place the sample cup in the AquaLab's sample chamber.

NOTE: The same verification standard may be used to verify and adjust the linear offset.

4. Carefully close the lid and move the lever to the READ position. Press the Check icon button to begin testing.

NOTE: If you decide at this point not to continue with the linear offset program, just return the lever to the OPEN position or press the cancel button and you will be returned to the previous screen.

5. After your AquaLab has finished measuring the verification standard, it will display the following screen:



6. Press the up and down arrows to adjust the water activity reading to its proper value for the particular verification standard you are measuring. When the correct value is displayed, press the Save icon button to store this new value. To cancel and return to the main menu, press the cancel button and no changes will be made.

7. Re-measure the verification standard again in normal sampling mode. It should read the proper value (within ±0.003 a_w) at a given

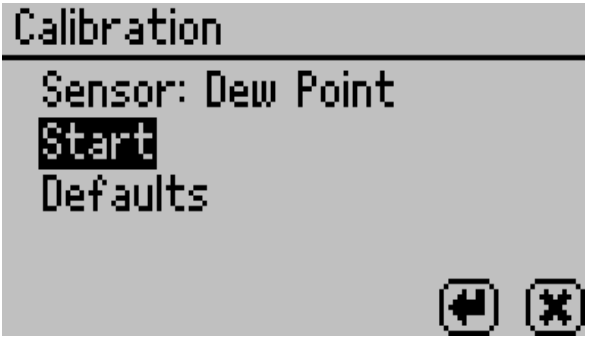
temperature for your particular standard (see Appendix B for temperatures other than 25°C).

Measure the water activity of a second verification standard according to the verification procedure described above. If both verification readings are within ±0.003 a_w then the instrument is ready to begin testing.

If you still have incorrect verification standard readings after cleaning the chamber and adjusting for linear offset, contact Decagon by email at support@decagon.com or by phone at 509-332-2756 (800-755-2751 in US and Canada) for further instructions. If you purchased your Decagon instrument from one of our international distributors, please contact them for local service and support.

How to Restore Factory Defaults
To restore original calibration settings, do the following:

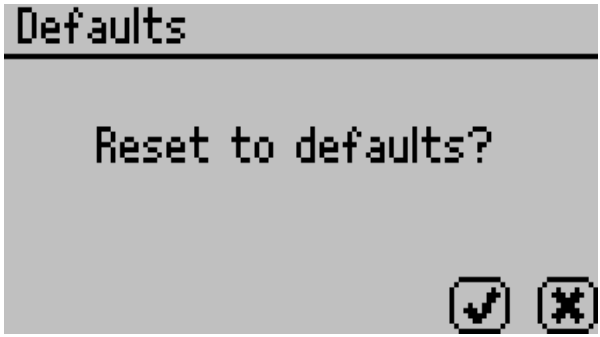
1. Toggle to the Configuration tab by pressing the Menu icon button. Select Calibration and press the Enter button.



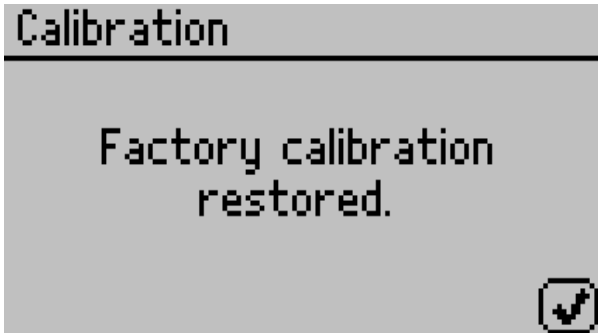
2. Scroll down to Defaults and press the Enter icon button to access

the Restore Factory Defaults routine. To cancel and return to the main menu, press the Cancel icon button. After pushing the Enter icon button, the following screen will appear:

NOTE: For TEV models make sure you have the correct sensor selected.



3. To restore the factory calibration values, press the Check icon button. To cancel and return to the main menu, press the cancel button. After pressing the Check icon button, the following screen will appear:



4. To return to the main menu screen, press the Check icon button.

8. Sample Preparation

Proper sample preparation is an important step in keeping your AquaLab clean and achieving repeatable results. Careful preparation and loading of samples will lengthen time between cleanings and help you avoid downtime.

Preparing the Sample

1. **Make sure the sample to be measured is homogeneous**. Multi-component samples (e.g., muffins with raisins) or samples that have outside coatings (like deep-fried, breaded foods) can be measured, but may take longer to equilibrate. For samples like these, AquaLab may take more than five minutes to give an accurate reading, or may require multiple readings of the same sample. Measuring the water activity of these types of products is discussed in detail later in this chapter (see Samples Needing Special Preparation).

2. **Place the sample in a disposable sample cup, completely covering the bottom of the cup, if possible.** AquaLab is able to accurately measure a sample that does not (or cannot) cover the bottom of the cup. For example, raisins only need to be placed in the cup and not flattened to cover the bottom. A larger sample surface area increases instrument efficiency by providing more stable infrared sample temperatures. It also speeds up the reading by shortening the time needed to reach vapor equilibrium.

3. **Do not fill the sample cup more than half full. Overfilled cups will contaminate the sensors in the sensor chamber.** Filling the sample cup will not make the readings faster or more accurate. There only needs to be enough sample in the cup to allow the water in the sample to equilibrate with the water in the vapor phase and not

change the moisture content of the sample. Covering the bottom of the sample cup provides enough sample to get an accurate reading.

4. **Make sure the rim and outside of the sample cup are clean.** Wipe any excess sample material from the rim of the cup with a clean Kimwipe. Material left on the rim or the outside of the cup can contaminate the sensor chamber and be transferred to subsequent samples.

5. **If a sample will be read at some other time, put the sample cup's disposable lid on the cup to restrict water transfer.** For long-term storage, seal the lid by placing tape or Parafilm® completely around the cup/lid junction. It is necessary to seal the cup if it will not be measured immediately.

6. **Be consistent in sample preparation practices.**
If you crush, grind, or slice your sample, be consistent in the method you use in order to obtain reproducible results.

Samples Needing Special Preparation

AquaLab reads most materials in five minutes or less. Some samples, however, may require longer reading times, due to their nature. These materials need additional preparation to ensure quick, accurate readings. To find out whether special sample preparation is necessary, take several readings to see if readings (a_w and time) stabilize. If continued readings take longer than six minutes, remove the sample and take a reading of a verification standard. This will ensure the sample itself is causing the long read time, and that there is not a problem with your instrument. If the verification standard also takes longer than six minutes to sample, the chamber may be dirty. Refer to Chapter 6 for cleaning procedures.

Coated and Dried Samples

Samples with coatings such as sugar or fat often require multiple readings, because it takes longer for them to equilibrate. If this is the case for your samples, it is not a problem with your instrument; it simply means that your particular sample takes longer than most to equilibrate.

To reduce the time needed to take an water activity reading for coated or dried samples, you can crush or slice the sample before sampling. This increases the surface area of the sample, thus decreasing reading times. Keep in mind, however, that modifying some samples may alter their water activity readings.

For example, a candy may have a soft chocolate center and a hard outer coating. The water activity reading for the center and the outer coating are different, so one would need to evaluate which part of the sample needed to be measured before crushing it. When the candy is crushed, the water activity will represent the average water activity of the entire sample; whereas leaving the candy whole will give a reading for the coating, which may act as a barrier to the center.

Slow Water-Emitting Samples

Some extremely dry, dehydrated, highly viscous water-in-oil (butter), high fat, or glassy compositions may require multiple tests due to their slow water-emitting properties. This is because the slow emission of water decreases the change in water activity sufficiently that the instrument determines the test to be complete, even though changes in water activity are still occuring. The most effective way to test these types of samples is to run them in the AquaLab using the continous or custom mode and wait for the water activity readings to stablize.

For faster reading, it is important to have the water activity of the chamber at or below the water activity of these type of samples. This causes the sample to release water to the vapor phase and equilibrate with the chamber. If the water activity of the head-space is greater than this type of sample, a long period of time will be required to reach equilibrium and the water activity of the sample may be affected.

Volatile Samples

AquaLab will give accurate readings on most samples. However, samples with certain volatiles in high enough concentrations may give inaccurate water activity values. This is because the volatiles condense on the mirror during the reading process, but do not evaporate from the mirror as water does. As a result, the reading on volatiles will not be accurate. The concentration of volatiles that will cause interference is variable and matrix dependent. The most effective method to determine if volatiles are a problem is to look for incorrect standard readings after reading the sample.

Decagon's Series 4TEV is designed for measuring volatiles such as propylene glycol and ethanol. The Series 4TEV contains both a chilled-mirror dewpoint and a capacitance sensor. Simply choose the sensor you want to use from the menu in the instrument. The only difference in operation is a lower accuracy of ±0.015 a_w for the capacitance sensor. All other operations and features will be the same, including measurement times and adjusting for linear offset. After measuring volatiles with the volatiles sensor it is a good idea to clean the chamber and run charcoal before switching to the dew point sensor.

Low Water Activity

When a sample's water activity value is below the cooling capacity of

the Series 4, your AquaLab will display an error message indicating the lowest reading it attained on that particular sample. See Chapter 12's troubleshooting problem #5 for possible solutions.

If your sample is not below 0.03 a_w but is still getting the error message, refer to **Chapter 12** for other possible explanations.

Samples not at Room Temperature

Samples that are 4°C colder or warmer than the instrument (chamber) temperature will need to equilibrate to instrument temperature before a fast, accurate reading can be made. Rapid changes in temperature over short periods of time will cause the water activity readings to rise or fall until the temperature stabilizes. When the temperature stabilizes within one or two degrees of the chamber temperature, you can proceed with normal measurements.

High-water activity samples that are warmer than the chamber temperature can cause condensation inside the measuring chamber, which will adversely affect subsequent readings. A warning message appears (Sample too hot) if the sample temperature is more than 4°C above chamber temperature. If this message appears, immediately remove the sample from the instrument, place a lid on the cup, and allow the sample to cool to within 4°C of the instrument before measuring.

Samples that are lower than 4°C of the instrument's temperature will cause long read times. The sample temperature must be within one or two degrees of the chamber temperature before fast, accurate readings can be made.

NOTE: Powdery substances can be blown by the fan so be sure not to overfill the sample cup and verify the cleanliness of the sample chamber before reading a new sample.

9. Taking a Reading

Measurement Steps

Once you have verified for cleanliness, calibration and prepared your sample, you are ready to take readings. The process is simple:

- Move the chamber lever to the Open position and lift the chamber lid.
- Check the top lip and outside of the sample cup to make sure they are free from sample residue and that sample cup isn't overfilled. (remember, an over-filled sample cup may contaminate the chamber's sensors).
- Place your prepared sample cup in the chamber.
- Close the chamber lid and move the lever to the Read position. This will seal the chamber and start the reading.

In 1 to 2 minutes, the first water activity measurement will be displayed on the LCD. Length of read times may vary depending on temperature differences between the chamber and your sample, and other properties of your sample.

How AquaLab Takes Readings

AquaLab's reading cycle continues until the rate of change of three consecutive readings are less than 0.0005 a_w of each other. The instrument crosses the dew threshold numerous times to ensure equilibrium and the accuracy of readings. When the instrument has finished its read cycle, the water activity is displayed, the read time is displayed, the spinning measurement icon is replaced by the Store

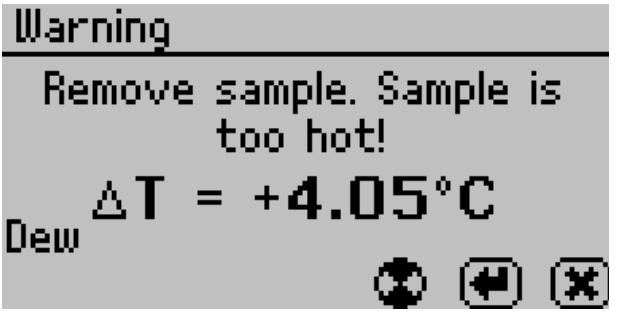
icon and, if enabled, you will hear a series of beeps.

Cautions!

- **Never leave a sample in your AquaLab after a reading has been taken. The sample may spill and contaminate the instrument's chamber if the instrument is accidentally moved or jolted.**

- **Never try to move your instrument after a sample has been loaded. Movement may cause the sample material to spill and contaminate the sample chamber.**

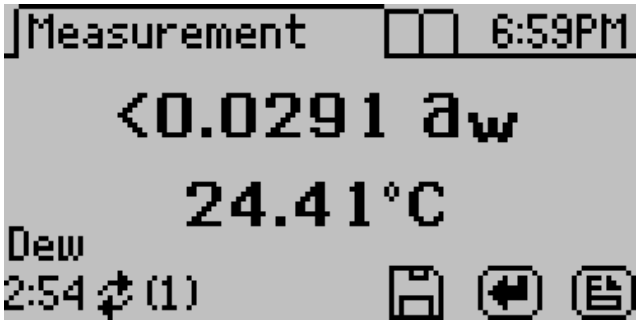
- **If a sample has a temperature that is 4°C higher (or more) than the AquaLab's chamber, the instrument will beep and display a warning as shown below. Remove the sample until it is at room temperature.**



Although the instrument will measure warmer samples, the readings may be inaccurate. Warm samples can cause condensation in the chamber if they have a high water activity. It is best to remove the sample from the instrument, place a lid on the cup and allow the sample to cool before reading.

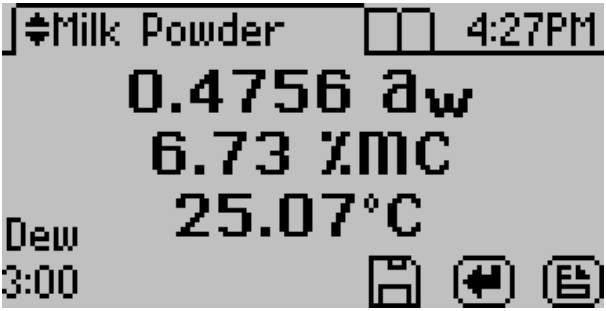
- The physical temperature of the instrument should be between 15 - 50°C. Between these ambient temperatures, AquaLab will measure samples of similar temperature quickly and accurately. The AquaLab Series 4TE has temperature control capabilities that enable it to read samples at temperatures different from ambient temperature, but no higher than 50°C.

- If a sample has a water activity lower than about 0.03, AquaLab will display the < symbol (see below) notifying you that your sample is too dry to be accurately measured by the AquaLab.



- If you know that your sample's water activity is above what the screen is telling you, your instrument's sensors may have been contaminated and will need to be cleaned (see **Chapter 6**) or serviced (see **Chapter 13**).

10. Duo Operation (Optional)



Previously, measuring moisture content and water activity required different instruments. Now it is possible to determine both moisture content and water activity with one machine. The Series 4TE can be upgraded to Series 4TE DUO which can display moisture content simultaneously with water activity. To calculate moisture content using water activity requires an understanding of the relationship between the two parameters. This relationship, referred to as the moisture sorption isotherm, is complex and unique to each product type. A product's isotherm can be used to calculate moisture content based on a water activity measurement. This is most easily accomplished using a model that characterizes the isotherm. For additional information about sorption isotherms and models, please refer to Chapter 3. The DUO generates water activity values just as a Series 4TE, but then it uses preloaded product specific isotherm models to calculate moisture content and present it on the screen with the water activity. For information about upgrading your Series 4TE to a Series 4TE DUO, please contact Decagon Devices.

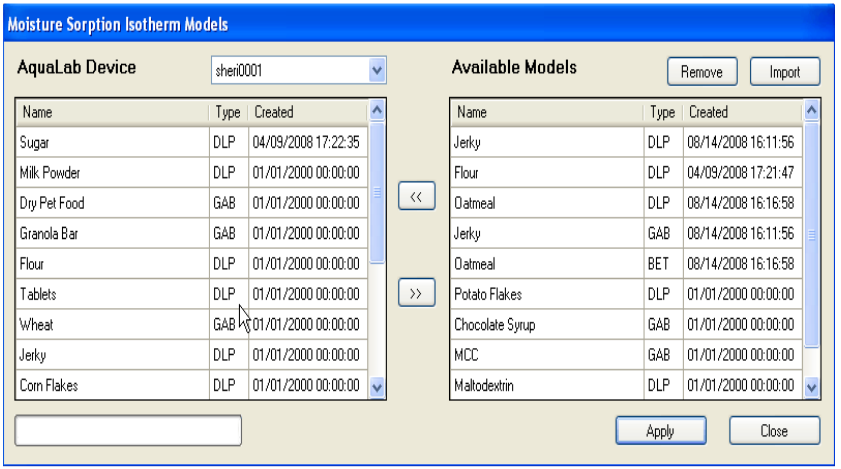
Obtaining Product Isotherm Models

Since the isotherm relationship for each product is unique, each product's isotherm model must be determined experimentally. This only needs to be done once, but must be done prior to testing moisture content with the DUO. To obtain an isotherm model for a product, samples of the product need to be sent to Decagon for isotherm analysis. Decagon will send instructions and a packing kit to assist in submitting samples for analysis. Upon completion of the isotherm, Decagon will generate a product model file to be loaded onto the instrument.

Loading and Organizing Product Models

A Product's model must be loaded into the Series 4TE DUO before it can calculate moisture content. Each product must have its own model and the model can either be loaded at the factory by Decagon or by using the AquaLink RG software program. This software is included with each DUO purchase or upgrade. Product model files generated by Decagon are sent to customers via email and can then be loaded into the instrument by connecting to the instrument using the AquaLink RG software. The software uses a model loading tool to add and remove product models from the Series 4TE DUO,

allowing the user to control and organize their product models. The AquaLink RG software can also download data (including moisture content) from the instrument, present the data in table form, filter the data, and print reports.

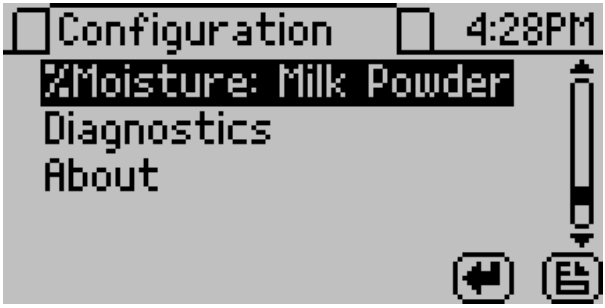


Measuring Moisture Content

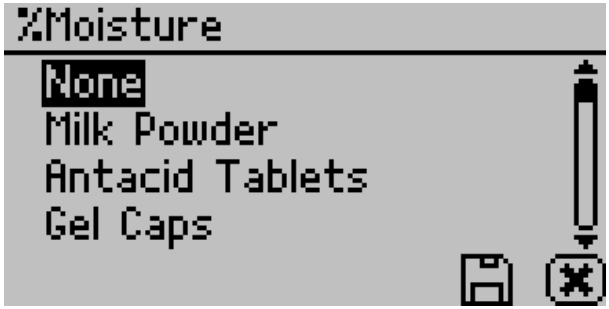
With the product models loaded into the instrument, the Series 4TE DUO can generate moisture content and water activity simultaneously.

Selecting a Product for Analysis

- With the Series 4TE DUO turned on, toggle to the configuration screen by pressing the menu button.
- At the configuration screen, scroll down and select moisture content.



- A list of available models will be listed by name.



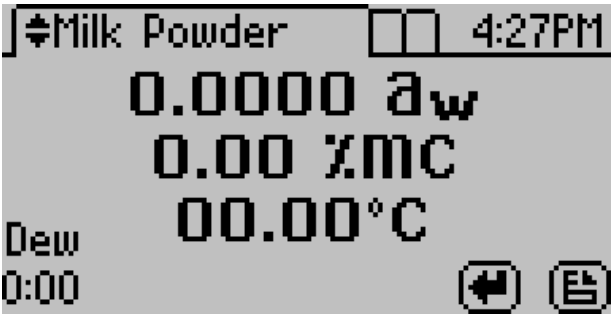
- Select the model for the product to be analyzed. Selecting "None" will not select any model.

Taking a Reading

- Readings are taken with the DUO the same as outlined in Chapter 9. First, return to the main screen.

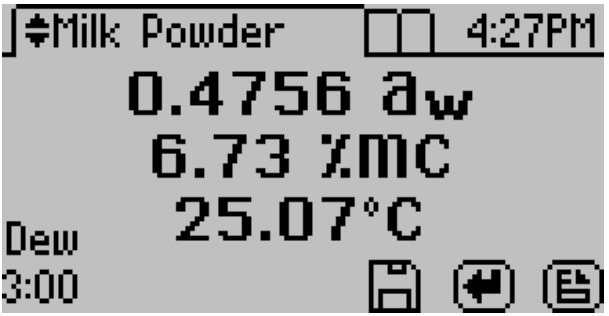
- The product chosen for analysis will be shown in the tab at the top of the screen. If a different product is desired to be analyzed, it is possible to scroll through all of the available product models on the screen by pressing the up and down buttons. This elimi-

nates the need to return to the configuration screen to change products. When the tab at the top shows "Measurement", no model is selected and only water activity will be displayed on the screen.

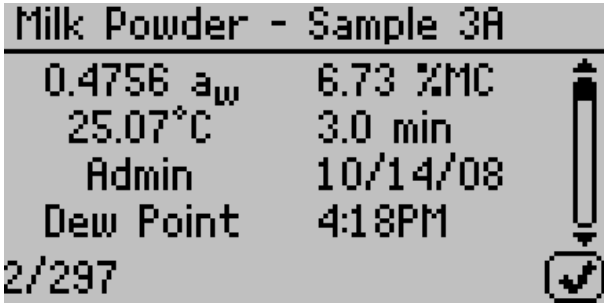


- Place a sample in the chamber and begin testing by sliding the lever left to the read position. For information about sample preparation, please see Chapter 8 and for additional information about running a test, please see Chapter 9.

- When the test is complete, the screen will display the water activity and moisture content for the product selected. If the wrong model was selected by accident, the up and down buttons can be used to toggle through all of the product models and the moisture content value will adjust based on the model selected.



- The test can be saved to the instrument's memory by pressing the button under the save icon. An annotation can be added if desired. If autosave has been selected, the data will already be saved but without any annotation.



The results can be viewed by moving to the data screen (press the right most button, which is below the document icon, to toggle between tabs) as shown in Chapter 5 under the Data Tab section. The only difference will be that moisture content data will now appear in the upper right column on the detailed information screen.

11. Computer Interface

Your AquaLab was shipped to you with a standard RS-232 interface cable. Using this, you can load data to a computer using Decagon's AquaLink RG program or your computer's terminal program for further analysis and storage.

AquaLink RG

An optional software program, AquaLink Report Generator (RG), is available for use with your AquaLab. AquaLink RG is a Windows based program designed for data collection and customized report generation for all Series 4 AquaLab models. AquaLink RG logs water activity, temperature, time of measurement, and date stamps along with other information. AquaLink RG also has sample identification and comment fields that you can use to help annotate the data your AquaLab is gathering.

A 30 day trial cd of this program is attached to the front cover of this manual. If you are interested in purchasing the full version of AquaLink RG, contact Decagon or your local distributor. If you have purchased the AquaLab 4TE DUO you will automatically receive the full version of AquaLink RG with your manual.

Using Windows Hyperterminal

To use Hyperterminal with your AquaLab, follow these steps:

- On your computer, press the Start button and select Programs > Accessories > Hyperterminal and click on the Hyperterminal icon.

- At the prompt, choose a name for this program (AquaLab is a good one) and choose an arbitrary icon above to represent it. In future downloads, you will be able to click on this icon in have it already set up for you to download. Click the OK button.

- A pop-up menu labeled "Connect To" will appear. Click on the scroll bar on the bottom of the screen labeled "Connect Using" and select the COM Port your RS-232 cable is connected to.

- A pop-up menu labeled "COM Properties" will appear, showing the port settings for the COM port you selected. Make sure the settings are the following: Bits per second, 9600; 8 databits, no parity, 1 stop bit, and flow control set to hardware. Click OK.

- Plug your RS-232 cable to the COM port you selected and connect it to your AquaLab. Begin sampling. AquaLab's data will be displayed on screen as it samples.

- When you are finished sampling, you can print the data in the terminal session, or cut and paste it to a spreadsheet or text editor. To save the data, go into the Transfers menu and select "Capture text," and designate where it should be saved.

12. Troubleshooting

AquaLab is a high performance, low maintenance instrument, designed to have few problems if used with care. Unfortunately, sometimes even the best operators using the best instruments encounter technical difficulties. Below is quick reference guide that will direct you to detailed solutions of some problems that may occur. If these remedies still don't resolve your problem, then please contact Decagon for help (see Customer Support in Chapter 1). Here is a list of some problems that may occur.

NOTE: If you purchased your Decagon instrument from one of our international distributors, please contact them for local service and support.

Troubleshooting Quick Guide

Troubleshooting Quick Guide (Continued)

1. PROBLEM:
AquaLab won't turn on.

SOLUTIONS:
1) Check to make sure your power cord is securely attached to the back of the instrument and it is plugged into the power outlet.

2) A power surge may have caused a fuse to blow. To change the fuses, follow these instructions:

   a. Unplug the power cord.

   b. Locate the panel where the power cord plugs in. The fuse box

is on the right side of that panel. Press in on the release tab and pull the fuse-holder out. Pull the broken fuse(s) out and replace with a 1.25 Amp 250V fuse.

Caution: **Do not use any other kind of fuse or you will risk damage to your instrument as well as void your warranty.**

c. Replace the fuse-holder and push it into the fuse-well until the release tab snaps in place.

d. Re-connect the power cord and turn your instrument on. If the fuse blows again, a failed component may be causing the problem. Contact Decagon to make arrangements for repairs.

2. PROBLEM:
Readings are slow or inconsistent.

SOLUTIONS:
1) The sample chamber may be dirty. Refer to Chapter 6 for directions on cleaning the sample chamber.

2) Some products absorb or desorb moisture very slowly, causing measurements to take longer than usual, and nothing can be done to speed up the process. Refer to Chapter 8 for further explanation.

3) Your sample may contain volatiles. Volatiles are known to cause unstable readings, because they condense on the surface of the chilled mirror and alter readings. Please refer to the volatiles section in Chapter 8 for hints on reducing difficulties with measuring samples with propylene glycol. If you have further questions regarding the measurement of volatiles contact Decagon.

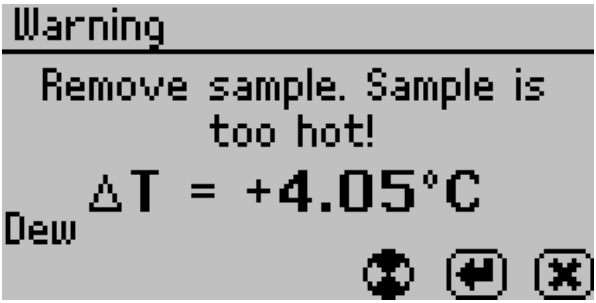
4) A fan blade in the block chamber may be broken or bent. If even salt standards take a long time to read, and the sample chamber is clean, you may have a broken chamber fan blade. This is especially likely if you have just cleaned the chamber. If you suspect this may have happened, contact Decagon for details on replacement.

3. PROBLEM:
Water activity readings on verification standards are too high/low and a linear offset adjustment cannot be made any higher/lower.

SOLUTIONS:
1) The thermopile in your chamber, which measures sample temperature, may have become contaminated. Refer to Chapter 6 for directions on cleaning.

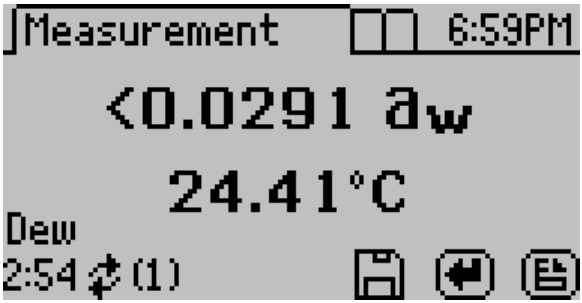
2) The chamber mirror may be dirty. Refer to Chapter 6 for directions on cleaning.

4. PROBLEM:
Message on screen displays the following:



SOLUTION:
Your sample's temperature is too high for the instrument to equilibrate with it in a reasonable amount of time. The instrument and sample need to be in temperature equilibrium before accurate measurements can be made. Therefore, very cold samples will take a very long time to measure for the same reason. To avoid this problem, make sure to only measure samples that are at the same temperature as the instrument.

5. PROBLEM:
Message on screen displays the following (example):



SOLUTIONS:
1) The sample is too dry for the instrument to read accurately. If your sample has a water activity that is less than below the detection limits of the instrument, this message will come up. Essentially, it means that there is not enough sample moisture to condense on the mirror and provide a reading.

2) The mirror may be dirty. Try cleaning the mirror and chamber and measuring the sample again.

6. PROBLEM:
Message on screen displaying dew point sensor failure.

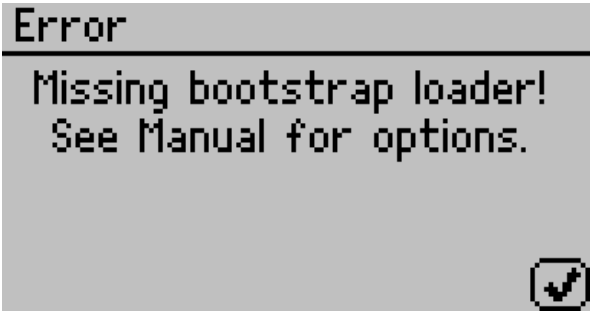
SOLUTION:
The Cooler is damaged and will need to be serviced by Decagon. See Chapter 12 for detailed instructions.

7. PROBLEM
Verification is not correct.

SOLUTIONS:
1) The sample chamber and mirror need to be cleaned. See Chapter 6 for detailed cleaning instructions. If verification is still not correct, then linear offset has occurred.

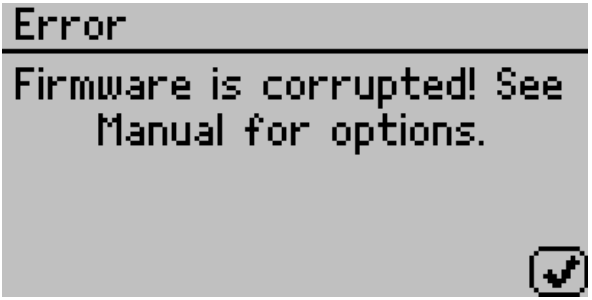
2) Verify and Adjust for Linear Offset. After you have cleaned the sample chamber and mirror (Chpt. 7) you will need to use a Verification Standard to verify and adjust for Linear Offset as described in Chapter 7.

8. PROBLEM:
Message on screen displays the following:



SOLUTION:
The instrument cannot download new firmware updates. To download new firmware to the Series 4 models, or to stop this message from displaying, the instrument must be serviced by Decagon.

9. PROBLEM:
Message on screen displays the following:



SOLUTION:
The firmware on the instrument is corrupted and needs to be reloaded. To download new firmware to the Series 4 models, the instrument must be serviced by Decagon.

10. DUO PROBLEM:
Test was run with wrong model.

SOLUTION:
1) On the measurement screen, toggle to the correct model using the up and down arrow keys. The moisture content value will be updated to correspond with the model selected.

2) If the correct model is not available, the model may not be loaded on the instrument.

   a. To determine which models are loaded on the instrument, cycle to the menu tab, select Moisture Content and then the loaded models will appear.

3) If the correct model is not available, load the appropriate model using Aqualink RG Software. The AquaLab DUO can only hold a total of 20 models at any one time. You may need to remove a model using the RG Software before you can add a new one. Any model that is removed from the instrument will be stored and may be reloaded again later.

11. DUO PROBLEM:
Moisture Content displayed is not correct.

SOLUTION:
1) Model selected may not be correct for the product being tested.

   a. Toggle through the available models to find a more appropriate model.

   b. If the model is correct but not giving correct moisture content values it may be necessary to generate a new model for

the product or update an existing model. For information about generating or updating a model, contact Decagon Devices.

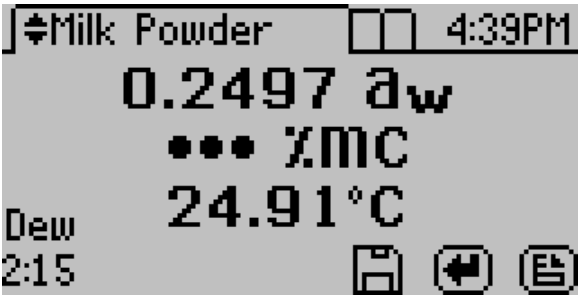
12. DUO PROBLEM:
Moisture content does not show up on the screen.

SOLUTION:
Moisture content has not been activated.
1) Toggle to menu tab, select moisture content, and select the appropriate model.

   a. If no models appear in moisture content screen, models will need to be reloaded using AquaLink RG software.

   b. If moisture content is not an active selection, the DUO feature may not be active. Content Decagon Devices to learn how to activate the DUO feature.

13. DUO PROBLEM:
Message on the screen displays the following:



SOLUTION:

1) When a moisture content reading is not shown, the water activity or temperature for that reading is beyond the scope of the moisture sorption isotherm. This can happen under the following conditions:

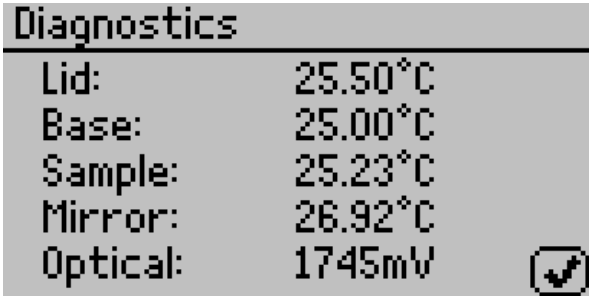
   a. The isotherm equation calculates a moisture content that is less than 0% or greater than 100% with the given water activity.

   b. The control temperature is significantly different than the isotherm temperature.

Make sure that the sample's water activity and the instrument's controlling temperature are within the scope of the selected moisture sorption isotherm model.

Diagnostic Screen

If, after cleaning your instrument and reading the other troubleshooting hints, you have reason to believe that one of the components of your AquaLab may be causing measurement error, you may access a screen that will display values for component performance. This is done by navigating to the Configuration tab and then by scrolling down to the diagnostics option. Press enter and you will be given a list of components and their values.



This screen shows typical values for the dew point method. Lid, base and sample temperatures may fluctuate but should not change more than 0.03 degrees. Typical ranges for the lid, base and sample temperatures is between 24.5 and 25.5 degrees.

If the mirror temperature is at lid temperature, the cooler has failed and must be replaced. If the mirror is below the lid temperature or appears to be random, the thermocouple wire is broken and must be repaired.

A typical optical range is between 500 mV and 2900 mV .

For capacitance mode, not shown here, the RH percentage should always be between 0 and 100%.

13. Support and Repair

NOTE: If you purchased your AquaLab from one of our international distributors, please contact them. They will be able to provide you with local support and service.

When encountering problems with your AquaLab (that can't be resolved with the help of this manual), please contact Decagon Customer Support at support@decagon.com, 800-755-2751 (US and Canada), 509-332-2756 (International) or fax us at (509) 332-5158. Please have the serial number and model of the instrument ready.

All AquaLabs returning to Decagon for servicing must be accompanied with a Return Material Authorization (RMA) form. Prior to shipping the instrument, please contact a Decagon customer support representative to obtain an RMA.

Shipping Directions:
The following steps will help to ensure the safe shipping and processing of your AquaLab.

1) Ship your AquaLab in its original cardboard box with suspension packaging. If this is not possible, use a box that has at least 4 inches of space between your instrument and each wall of the box.
2) Place the AquaLab in a plastic bag to avoid disfiguring marks from the packaging.
3) Don't ship the power cord or serial cable.
4) If the original packaging is not available, pack the box moderately tight with packing material (e.g. styrofoam peanuts or bubble wrap), ensuring the instrument is suspended in the packing material.

5) Include a copy of the RMA form in the shipment. Please verify the ship to and bill to information, contact name, and problem description. If anything is incorrect please contact a Decagon representative.
6) Tape the box in both directions for added support.
7) Include the RMA number in the attention line on the shipping label.

Ship to:
Decagon Devices Inc.
ATTN: RMA (insert your RMA #)
2365 NE Hopkins Court
Pullman, WA 99163

Repair Costs

Manufacturer's defects and instruments within the three-year warranty will be repaired at no charge. Non-warranty repair charges for parts, labor and shipping will be billed to you. An extra fee may be charged for rush work. Decagon will provide an estimated repair cost, if requested.

Loaner Service

Decagon has loaner instruments to keep you measuring water activity while your instrument is being serviced. If your AquaLab is still under calibration warranty or you have a service plan with your instrument, there is no charge for the loaner service.

14. Further Reading

Water Activity Theory & Measurement

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Appendix A

Preparing Salt Solution

If you choose to mix a saturated salt solution for use as a verification standard, we recommend that you use the approved AOAC method. This method is as follows:

1. Select a reagent-grade salt and place it in a test container to a depth of about 4cm for more soluble salts (lower a_w), to a depth of about 1.5 cm for less soluble salts (high a_w), and to an intermediate depth for intermediate salts.

2. Add distilled water in increments of about 2mL, stirring constantly.

3. Add water until the salt can absorb no more water, evidenced by the presence of free liquid. Keep the amount of free liquid to the minimum needed to keep the solution saturated with water. If you plan on using this solution over a long term period, seal the solution well to prevent losses from evaporation. Below is a table of saturated salt solutions and their respective water activities at various temperatures. Please note that these values are based on averaged published data, and the standard errors shown reflect Greenspan's standard error for each salt solution, not the AquaLab's accuracy in measuring the salt. AquaLab measures all samples with an accuracy of ±0.003 a_w.

Table 2: Water Activity of Selected Salt Solutions

Saturated Solution	a_w at 20° C	a_w at 25° C
Lithium Chloride	0.113 ± 0.003	0.113 ± 0.003
Magnesium Chloride	0.331 ± 0.002	0.328 ± 0.002
Potassium Chloride	0.432 ± 0.003	0.432 ± 0.004
Magnesium Nitrate	0.544 ± 0.002	0.529 ± 0.002
Sodium Chloride	0.755 ± 0.001	0.753 ± 0.001
Potassium Chloride	0.851 ± 0.003	0.843 ± 0.003
Potassium Sulfate	0.976 ± 0.005	0.973 ± 0.005

Adapted from Greenspan (1977). Rounded to nearest thousandth.

4. Saturated salt solutions are very temperature-sensitive and their values are not as accurate as the verification standards offered by Decagon.

Appendix B

Temperature Correction of Decagon's Verification standards

Temp. (°C)	H2O	0.5m KCL	6.0m NaCl	8.57m LiCl	13.4m LiCl
15.0	1.000	0.984	0.761	0.492	0.238
20.0	1.000	0.984	0.760	0.496	0.245
25.0	1.000	0.984	0.760	0.500	0.250
30.0	1.000	0.984	0.760	0.504	0.255
35.0	1.000	0.984	0.760	0.508	0.261
40.0	1.000	0.984	0.760	0.512	0.266

Aqualab will measure these standards to $\pm 0.003 a_w$

Appendix C

AquaLab Verification Standards Application Note

Using AquaLab is easier then ever. Pre-packaged standard salt solutions are immediately available for performance verification, saving you time and money. Validation and documentation for GMP and GLP has also become easier. Operate your instrument with certainty and insure the quality of your food product by using low cost precision salt solutions.

No need to purchase and store reagent grade salts.

No additional laboratory equipment necessary.

Avoid solution handling and mixing errors.

Save technician time.

The AquaLab should be verified against a known salt standard daily. For high use or batch processing, the instrument should be checked regularly against a known salt standard of similar water activity. Checking the water activity of a standard solution will alert the operator to the possibility of contamination of the unit or shifts in the linear offset from other causes.

Now, you can verify AquaLab performance with confidence. Performance Verification Standards come in four water activity levels; 0.984, 0.760, 0.500, and 0.250 a_w. The standards are produced under a strict quality assurance regime. The accuracy of the standards is

verified by an independent third party and are shelf stable for one year. Order your calibrtion salt standard of similar water activity today.

Uncertainties Using Saturated Salt Solutions

The water activity values listed in our operator's manual for saturated salts were reprinted from Greenspan (1977). His method for determining water activity was to combine all of the available data from tests by other researchers. He did not set up any experiments of his own. The uncertainty he published is due to variation among the results from the different methods. There are, therefore, limitations to the accuracy of these values. The instrumentation available for making water activity measurements is much better now than it was in 1977, so improved standards are needed.

Saturated salt solutions can be prepared by several methods. The AOAC method involves starting with salt and adding water in small increments, stirring well with a spatula after each addition, until salt can absorb no more water as evidenced by free liquid (where it will take on the shape of the container but will not easily pour). This method gives the most accurate readings, but only for a short time unless great care is taken to prevent water gain or loss. When a salt standard is prepared so that it consists mostly of liquid with a few crystals in the bottom, it can result in a layer of less than saturated solution at the surface which will produce a higher reading than anticipated. Conversely, solid crystals protruding above the surface of the liquid can lower the readings. To comply with Good Laboratory Practices (GLP), a saturated salt solution must read within reasonable analytical error of the accepted published value for a given temperature.

Why AquaLab Verification Standards are Superior

Our research indicates that unsaturated salt solutions make much

better standards than saturated salts. Robinson and Stokes (1965) give activity coefficient for various salt solutions. These can be used to compute the water potential, or partial specific Gibbs free energy, of the water in the solution using;

$$\Psi = -\phi\gamma cRT$$

where Ψ is the water potential, ϕ is the number of active particles per molecule of solute (i.e., 2 for NaCl), γ is the activity coefficient, c is the concentration of the solute (mol kg^{1}), R is the gas constant (8.314 J mol^{-1} K^{-1}), T is the Kelvin temperature. Water potential is related to water activity by the equation;

$$a_w = \exp(\frac{\Psi M_w}{RT})$$

where M_w is the molecular weight of water (0.018 kg mol^{-1}). When equations 1 and 2 are combined a simplified equation for water activity is obtained;

$$a_w = \exp(-\phi\gamma cM_w)$$

For example, equation 3 gives the a_w in a 6M NaCl solution, (M_w = 0.018 kg mol^{-1}, ϕ = 2, and = 1.271; from tables in Robinson and Stokes, 1965) as

$$a_w = \exp(-2 \times 1.271 \times 6 \times 0.018) = 0.760$$

It is important to note that equation 3 has no explicit temperature dependence. Available data on temperature dependence of γ indicates that its variation is less than ± 2% over the range 0 to 50°C for

NaCl (Lang, 1967) and KCl (Campbell and Gardner, 1971) and no other terms have any temperature dependence.

A further advantage of unsaturated salts is that there is no solid phase present to affect the water activity of the solution. Salt in saturated solutions can exist in different states and result in uncertainty in the water activity values.

Instructions for Using Decagon's Verification Standards
Simply empty one vial of standard solution into a sample dish and place the dish immediately into the AquaLab for measurement. Each vial will fill a sample dish to just less than half full. The following table shows the expected values.

Verification Standard	Water Activity
Distilled H2O	1.000 ±0.003
0.5m KCl	0.984 ±0.003
6M NaCl	0.760 ±0.003
8.5M LiCl	0.500 ±0.003
13.4M LiCl	0.250 ±0.003

NOTE: If you need to obtain a Material Safety Data Sheet (MSDS) for any of these standards, a printable version is available on our website at www.decagon.com/msds.

Verify the AquaLab is functioning properly with any two of these solutions. We recommended that you choose a standard from the range in which you are measuring and distilled water (or another solution from the table).

1. Place the verification standard (do not start with water) in AquaLab for measuring. When a final reading is reached, check it against

the value listed above. If it within ±0.003, place your second solution in the drawer for testing. It should read the value ±0.003 listed in the table above. If the readings are within the expected values your verification is complete.

2. If the first solution does not read within ±0.003 of the expected value, then you need to adjust the linear offset so that the solution reads correctly (see Chapter 7). When you are finished measuring both standards, the readings should be within ±0.003 of the predicted values.

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Declaration of Conformity

Application of Council Directive:	89/336/EEC
Standards to which conformity is declared:	EN81-1 EN500082-1
Manufacturer's Name:	Decagon Devices, Inc. 2365 NE Hopkins Ct. Pullman, WA 99163 USA
Type of Equipment:	AquaLab water activity meter.
Model Number:	Series 4TE, Series 4TEV Series 4DUO
Year of First Manufacture:	2008

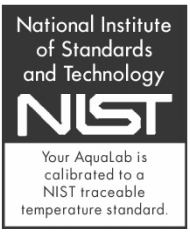
This is to certify that the AquaLab water activity meter, manufactured by Decagon Devices, Inc., a corporation based in Pullman, Washington, USA meets or exceeds the standards for CE compliance as per the Council Directives noted above. All instruments are built at the factory at Decagon and pertinent testing documentation is freely available for verification. This certification applies to all AquaLab Series 4 models, including, but not limited to, the 4TE, 4TEV and DUO.

Certificate of Traceability

Decagon Devices, Inc
2365 NE Hopkins Court
Pullman WA 99163

Tel: (509) 332-2756
Fax: (509) 332-5158
support@decagon.com

This is to certify that AquaLab water Activity Meters are manufactured utilizing temperature standards with calibration traceable to the National Institute of Standards and Technology (NIST).



Index

S

T

U

V

W