TDL Tunable Diode Laser Water Activity Meter

Operator's Manual



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1 Introduction

Welcome to your AquaLab Tunable Diode Laser (TDL). 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, the TDL suits your needs. It is easy to use and provides accurate and timely results.

1.1 Customer Support

If you ever need assistance with your AquaLab, have any questions or feedback, there are several ways to contact us. METER has Customer Service Representatives available to speak with you Monday through Friday, between 7 am and 5 pm Pacific time.

Note: If you purchased your AquaLab through a distributor, please contact them for assistance.

$\underline{\mathrm{Email:}}\\ \mathbf{support.food@metergroup.com} \ \mathrm{or} \ \mathbf{sales.food@metergroup.com} \\$

<u>Phone:</u> 1-509-332-5601

<u>Fax:</u> 1-509-332-5158

If contacting us by email or fax, please include as part of your message your instrument serial number, your name, address, phone, fax number, and a description of your problem or question.

1.2 About This Manual

This manual includes 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.

1.3 Warranty

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

1.4 Seller's Liability

Seller warrants new equipment of its own manufacture against defective workmanship and materials for a period of one year from the date of receipt of equipment.

Note: We do not consider the results of ordinary wear and tear, neglect, misuse, accident and excessive deterioration due to corrosion from any cause as defects.

The 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, damage 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 the 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.

1.5 General Safety Information

Please read through this documentation carefully before putting the instrument into operation. The documentation contains information and warnings which the user must follow in order to ensure safe operation. This instrument may only be operated in accordance with the specifications in this documentation.

This instrument has left the factory in a flawless state in terms of technical and electrical safety. To maintain this state and ensure non-hazardous operation of the instrument, the following instructions must be observed carefully.

- 1. Only personnel qualified by METER are authorized to carry out service work on the electrical components. When work is required a Certificate of Calibration will be issued upon completion of the work.
- 2. Never remove the housing of the instrument. The instrument could be damaged by this. There is also a risk of serious injury if the live components are touched. There are no parts inside the housing which can be serviced or replaced by the user.
- 3. An incorrect main power voltage can damage the instrument. Only operate this instrument with a main power voltage specified for it (see rear label).
- 4. This product is grounded through the grounding conductor of the power cord. To avoid electric shock, the grounding conductor must be connected to earth ground.
- 5. Should a fuse need to be replaced. Use only the fuse type and rating specified for this instrument.
- 6. If the instrument is used in a manner not specified by the manufacturer, the protection provided by the equipment may be impaired.

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 $\pm 0.005 \ a_w$ accuracy. The instrument is easy to clean and checking calibration is simple.

Note: UL has listed this product to applicable UL Standards and Requirements.

2.1 AquaLab TDL Instrument Specifications

Sensor Type: Tunable Diode Laser and Infrared Temperature

Water Activity Accuracy: ± 0.005 @ 25 °C

Water Activity Resolution: 0.0001 a_w

Water Activity Range: 0.000 to 1.000 a_w

Water Activity Repeatability: $\pm 0.001 \ a_w$

<u>Read Time¹</u>: ≤ 5 min.

Sample Temperature Control: 25 °C 15 to 50 °C (with calibration)

Sample Temperature Adjustment Increment: 1 °C

Sample Temperature Accuracy: ± 0.2 °C

Sample Temperature Resolution: 0.01 $^{\circ}\mathrm{C}$

Sample Dish Capacity: 15 mL full

Operating Environment: 4 to 50 °C; 0 to 90% Humidity non-condensing

<u>Case Dimensions</u>: $26.7 \ge 17.8 \ge 12.7 \text{ cm}$

Weight: 3.1 kg

Case Material: POLYLAC PA-765 (ABS) with fire retardant

Display: 64 x 128 Graphical

¹On samples with no significant impedance to vapor loss.

Data Communications: USB

<u>Power</u>: 110 to 220 VAC, 50/60 Hz

Warranty: One year parts and labor

2.2 AquaLab and Water Activity

Water activity (a_w) is a measurement of the energy status of the water in a system. The value 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 Section 3 of this manual, titled "Water Activity Theory."

2.3 How TDL Works

The TDL uses a tunable diode laser to measure the water activity of a sample. The sample is equilibrated with the head-space of a sealed chamber containing a tunable laser that shines light of a controlled wavelength at a detector that receives the light from the laser. At equilibrium, the relative humidity of the air in the chamber is the same as the water activity of the sample. In the TDL, the vapor pressure of the headspace in equilibrium is determined by the loss of signal strength from the laser caused by the presence of water vapor in the headspace. This vapor pressure is divided by the saturated vapor pressure at the sample temperature, which is measured using an IR sensor, to give water activity. AquaLab then signals you by beeping and displays the final water activity. Since the sample temperature is directly measured and no sensor is being used to detect humidity, all that is needed to make a measurement is vapor equilibrium, which can happen in as little as two to three minutes.

2.4 AquaLab and Temperature

Samples not read at room temperature during the read cycle equilibrate with the TDL temperature before the water activity is displayed. Large temperature differences cause longer reading times, since TDL cannot make a complete and accurate reading until the sample and the instrument equilibrate to within ± 4 °C. There are several advantages in having a temperature-controlled water activity meter. A few major reasons are:

- 1. **Research purposes.** Researchers can use temperature control 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. (See Section 14. Further Reading for more information)
- 2. Compliance 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. Minimization of extreme ambient temperature fluctuations. If the environmental and AquaLab temperatures fluctuate by as much as ± 5 °C daily, water activity readings vary by $\pm 0.01 a_w$. Temperature control eliminates variations due to changes in ambient conditions.

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.

3.1 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. There are two primary methods for determining moisture content: loss on drying and Karl Fisher titration, but you can also use secondary methods such as infrared and NMR. 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.

3.2 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. Researchers measure the water activity of a system by equilibrating the liquid phase water in the sample with the vapor phase water in the headspace and measuring the relative humidity of the head-space. First place a sample in a sample cup that seals inside the TDL sample chamber. Inside the sample chamber is a tunable diode laser and an infrared thermometer. The TDL determines the vapor pressure in the headspace and the infrared thermometer



Figure 1: Water Activity Diagram adapted from Labuza

measures the sample temperature. From these measurements, the relative humidity of the head-space is computed as the ratio of the partial pressure measured by TDL 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.

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. The AquaLab infrared thermometer measures the difference in temperature between the sample and the block. It is carefully calibrated to minimize temperature errors, but when temperature differences are large water activity can change during testing. 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 condenses water within the block. Condensation causes errors in the measurement, and in subsequent measurements until it evaporates. 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 TDL.

3.3 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 Activity

The water activity 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 Waals forces. At higher water activity levels, capillary forces and surface tension can also play a role.

3.4 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. METER sells an Isotherm Generator called the AquaLab Vapor Sorption Analyzer (VSA) or you can also have METER run the isotherm for a fee.

For example, if you were using the AquaLab to monitor the water content of dried potato flakes, you would measure the water activity and water content of potato flakes dried to varying degrees using the standard drying process for those flakes. You could then use that data to construct an isotherm and infer the water content using the measured water activity of samples and that isotherm.

We cannot overemphasize the importance of the concept of water activity for foods, pharmaceuticals, and cosmetics. 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

4.1 Components of your AquaLab

Your AquaLab should have been shipped with the following items:

- AquaLab water activity meter
- <u>Calibration certificate</u>
- <u>Power cord</u>
- <u>USB interface cable</u>
- 50 disposable sample cups
- Operator's Manual
- Quick Start Guide
- Cleaning kit
- Two vials each of the following verification solutions:

1.00 a_w USP Purified Water 0.984 a_w 0.50 mol/kg KCL 0.920 a_w 2.33 mol/kg NaCl 0.760 a_w 6.00 mol/kg NaCl 0.500 a_w 8.57 mol/kg LiCl 0.250 a_w 13.41 mol/kg LiCl

• AquaLink 4 Software Package

4.2 Choosing a Location

To ensure that your AquaLab operates correctly and consistently, place it on a level surface. This reduces the chance of sample material spillage or sample chamber contaminate. 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.

4.3 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 back panel. When the AquaLab is turned on, you should see a model name/number screen and then the main Measurement screen.

Warning: Only use the supplied power cord or one rated for your AquaLab 4 and certified for the country of use. The cord must be minimum of 18 AWG and have a rating for 10 Amps or greater.



The Measurement screen shows the water activity (a_w) in the middle of the screen, directly above the sample temperature.

Note: Allow your AquaLab a 15 minute warm-up period to ensure the most accurate readings.

If you have users set up, the User screen appears instead of the Measurement screen. (See Section 5 for more information on administrative settings and user setup.) Select the appropriate user to begin.

User	
Admin	Â
Jean Paul	
Sarah	
Leslie	Ų

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 switches to an "X" icon, which allows the user to stop the current reading. During a reading, pressing Enter again restarts the reading.

5.1 Measurement Tab

The Measurement tab, as seen above, is the main screen which displays each time you turn on your AquaLab. If this screen does not appear, refer to Section 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 changes the display to a temperature Equilibrating screen. This screen shows the temperature difference between the sample temperature and the lid temperature.



5.2 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 allows you to change the highlighted setting.



Calibration

Pressing the Enter button with Calibration highlighted starts the calibration process. For more details on the water activity verification and calibration procedures refer to Section 7. You may also reset the calibration to the factory defaults by highlighting the Defaults option and pressing Enter. This resets 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 may be set between 15 and 50 °C by 1.0 °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 causes the instrument to wait until the sample temperature is within < 0.5 °C of the block temperature before starting the water activity measurement.



Mode

Users may choose between single, continuous, custom, or low emitting 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 display 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 signals 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 memory if the auto save feature is selected (see Auto Save below). If AquaLab is connected to a computer using AquaLink 4 (See Section 11), all readings taken during continuous mode download to the AquaLink 4 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 four consecutive tests be within $\pm 0.001 a_w$. The instrument continues to run tests until it records four consecutive tests within $\pm 0.001 a_w$, then it stops and reports the value of the final test. If you turn auto save on, all test readings save to the instruments memory, but only the final reading appears on the main Measurement screen. If you keep the TDL connected to a computer using AquaLink 4 (See Section 11: Computer Interface), all readings taken during a custom mode test download to the AquaLink 4 software.

On the Mode screen at the top of the page is 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 allows you to change the number of tests and stability settings.



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 0.0200. To save the settings and finish, press the save button (to exit without updating, press the cancel button). The Mode screen now has the updated custom settings 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).

Low Emitting Mode: This mode is for samples that are slow to equilibrate such as vegetable oils, high fat samples, and high viscosity samples. AquaLab determines vapor equilibrium at the end of a test by comparing sequential water activity values, looking for a trigger value difference less than 0.0005 a_w . This very strict requirement is needed to ensure that the instrument provides the necessary accuracy. The low emitting mode gives the option of speeding up test time by adjusting the equilibrium trigger value. The typical setting is two water activity values that are within $\pm 0.0005 a_w$ of each other. This value can be adjusted to any value between 0.0003 and 0.0030 a_w .



Increasing the trigger value causes a subsequent reduction in instrument accuracy and precision, but results in a shorter test time. In certain cases, this loss in performance may be acceptable in order to speed up the analysis time. We recommend using using high trigger only after careful consideration of the impact on test results.

Date

AquaLab TDL models 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 with the Time option 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 AquaLab TDL displays 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.



5.3 Admin Settings

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



The Admin settings allow the administrator to grant or block access to some or all of the configuration options for all AquaLab TDL 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 lock all other users out of that configuration screen. Administrators may lock out users by entering the Access function and selecting the desired option to toggle it on and off. You can also lock and unlock all of them at once. (For example, if you do not want an individual changing the instruments measurement temperature, the administrator can lock that function for only certain individuals.) The areas that you can lock are calibration, temperature, temperature equilibration, sensor selection, mode, date/time, region, password, auto-save, number of beeps, contrast,

and delete functions.



User Setup

Administrators can add, edit, or delete users from this screen. An alphabet screen appears where you can enter a name 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 TDL models have the ability to store water activity readings within the instrument. By selecting Auto Save "On," the instrument automatically stores every water activity reading in the internal memory. AquaLab TDL can store up to 8,000 records before the memory is full. If you turn Auto Save "off" then the instrument does not store data automatically, although you may store any individual reading manually right after completing the test and before starting the next test.

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 saves the reading without a name. If you do not press the save icon after a reading, and the reading is autosaved, you cannot give it an annotation later.

Beeps

Allows you to set the reading finished notification from four 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 TDL water activity meter, the diagnostics screens provide you with an updated lid, base, sample, and laser temperature, as well as laser intensity, atmospheric pressure and water activity offset.



About

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



5.4 Data Tab



View

This selection allows you to view your stored measurements. The up/down arrows 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 Section 11 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 the Data - Sample screen shows.



The information shown is the water activity of the sample, the temperature, the test time, the user who ran the test (if setup), the date of the reading, the time the reading was taken, and the sequence number of the stored reading. The up and down arrows scroll through readings.

Delete

Selecting this option deletes all of the information currently stored in the instrument. If you have not backed up this information with AquaLink 4, TDL reminds you in the delete screen.



Note: It is impossible 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, so you must regularly clean your instrument. To clean your instrument, carefully follow these instructions and refer to the labeled diagram in Figure 2. METER also recommends you send your TDL in for annual factory calibration.



Figure 2: AquaLab Instrument Diagram

Purpose

The purpose for the cleaning procedure is to remove grease, dirt and other soluble substances which can absorb or release water during verification, calibration, and/or sample testing. For the TDL and the IR sensor to perform well, they must be clean and free from contaminants (e.g. fingerprints).

When to Clean

The instrument should be cleaned if visual inspection indicates the chamber is dirty or as instructed in Table 1 on page 35.

Cleaning Supplies

Your new instrument comes with the AquaLab Cleaning Kit. The AquaLab Cleaning Kit comes with all the materials needed to clean the instrument for about a year. Every time you send in your instrument for the annual calibration service, you receive a new cleaning kit. The AquaLab Cleaning Kit contains the following supplies.

- Spatula (a thin plastic rod)
- Deionized Water
- Cleaning Solution
- $Kimwipes^{\mathbb{R}}$

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

Note: You can substitute isopropyl alcohol for cleaning solution.

6.1 Cleaning the Block 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 orange o-ring when the lid is closed.

6.2 Cleaning an AquaLab TDL

Follow the cleaning procedures listed below. If you run out of solution, you can use isopropyl alcohol (IPA) instead.

6.3 Cleaning Procedure:

Cleaning your AquaLab is a multi-step procedure which involves washing, rinsing, and drying for each specific area as outlined below. (Refer to Figure 2 at the beginning of this section to identify component locations for cleaning.)

1. Cleaning the Sample Chamber

- (a) Remove any debris that may have collected within or around the sample chamber.
- (b) Wrap a NEW Kimwipe around the end of the spatula (thin plastic rod) and moisten it with cleaning solution.

Note: Do NOT dip a used Kimwipe into your cleaning solution (the cleaning solution becomes 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 through d using new Kimwipes with deionized water.
- (f) DRY Repeat steps b through d using new, dry Kimwipes to help remove any moisture remaining from the cleaning.

Note: Do not reuse Kimwipes.

2. Clean the TDL

- (a) Wrap a NEW Kimwipe around the end of the spatula and moisten it with cleaning solution.
- (b) WASH Swipe the moistened Kimwipe across the TDL emitter and detector lense once. (A single swipe is usually sufficient to remove contaminants.)
- (c) RINSE Repeat steps a through b using new Kimwipes moistened with deionized water instead of cleaning solution.
- (d) DRY Repeat steps a through b using a new, dry Kimwipes to help remove any moisture remaining from the cleaning.
- (e) Visually inspect the components for cleanliness. Clean again if necessary.

3. Clean the IR Sensor

- (a) Wrap a new Kimwipe around the end of the spatula and moisten it with cleaning solution.
- (b) WASH Swipe the moistened Kimwipe across IR Sensor. (A single swipe across the sensor is usually sufficient to remove contaminants.)
- (c) RINSE Repeat steps a through b using new Kimwipes moistened with deionized water instead of cleaning solution.
- (d) DRY Repeat steps a through b but use a new, dry Kimwipe to help remove any moisture remaining from the cleaning.
- (e) Visually inspect the IR Sensor for cleanliness. Clean again 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 at least five minutes to ensure the sample chamber is dry.
6.4 Verification of Calibration

After you have cleaned the chamber and other parts of your AquaLab, it is important to check the instrument 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 section. If a linear offset has occurred, refer to "adjust for linear offset" in Section 7 for directions on how to correct for linear offset. If, after adjusting for linear offset, your instrument is still not reading samples correctly, it may be time for an annual factory calibration. Contact Aqualab at support.foods@metergroup.com or 509-332-5601 for annual calibration.

7 Verification and Calibration

It is important to verify AquaLab water activity calibration against known standards to guarantee optimal performance and accuracy. METER recommends verification daily, once per shift or before each use. METER also recommends annual factory calibration to maintain optimal performance.

Note: To avoid inaccurate water activity readings, verification standards should be used once immediately after opening and not stored in sample cups for repeated use.

7.1 Water Activity Verification

AquaLab uses the TDL technique to determine water activity. Because this is a primary measurement of relative humidity, no calibration is necessary; but we recommend periodic verification for linear offset. The components used by the instrument to measure water activity are subject to contamination which may affect the AquaLab 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 unsaturated salt solutions having a specific molality and water activity value which are accurately measurable. The verification standards that were sent with your initial shipment are very accurate and readily available from METER. Using verification standards to verify accuracy can greatly reduce preparation errors. For these reasons, we recommend using standards available through METER for the most accurate verification of your AquaLab performance. Performance Verification Standards come in six water activity levels: 1.000, 0.984, 0.920, 0.760, 0.500, 0.250, and 0.150 a_w . The standards are produced under a strict quality assurance regime. Please contact METER to

order additional standards via sales.food@metergroup.com or 509-332-5601.

Verification Standard @ 25 $^{\circ}C$	Water Activity
17.18 mol/kg LiCl	0.150 ± 0.005
13.41 mol/kg LiCl	0.250 ± 0.005
8.57 mol/kg LiCl	0.500 ± 0.005
6.00 mol/kg NaCl	0.760 ± 0.005
2.33 mol/kg NaCl	0.920 ± 0.005
0.50 mol/kg KCl	0.984 ± 0.005
USP Purified Water	1.000 ± 0.005

Table 1: Verification Flowchart

Note: If you need to obtain a Safety Data Sheet (SDS) for any of these standards, a printable version is available on our website at http://sds.metergroup.com/.

To use a verification standard, remove the twist top and pour the contents into an AquaLab sample cup. Information about the standard value and molality can be found printed on the outside of the plastic vial. If for some reason you cannot obtain METER's verification standards and need to make a saturated salt solution for verification, refer to Appendix A.

7.2 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 steam 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 alerts you to the possibility of unit contamination or shifts in the linear offset from other causes.

Follow steps 1 through 8 to verify for linear offset of your AquaLab. (Refer to Figure 3: the Verification Standard Flowchart for a quick overview.)

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 TDL testing chamber. Make sure that your standard is as close to the instrument temperature as possible. See Section 8 for detailed instructions.

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

- 3. Carefully close the lid and move the lever to the Read position.
- 4. Take two readings. The water activity readings should be within $\pm 0.005 \ a_w$ of the given value for the verification standard. See Appendix B for the correct water activity value of METER's standards at temperatures other than 25 °C.
- 5. If your AquaLab is reading within $\pm 0.005 a_w$ of the verification standard, choose 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 8.57 mol/kg LiCl (0.50 a_w) standard for your first verification and the 6.00 mol/kg, NaCl (0.76 a_w) for the second verification.
- 6. Prepare a sample cup of the second verification standard and take two readings. The second water activity reading for the second verification standard should be within $\pm 0.005 a_w$.
- 7. If either of the verification standards is not correct, it is probably due to contamination of the sensor chamber. For cleaning

instructions, see Section 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 ± 0.005 a_w , a linear offset has probably occurred. In this case, adjust the reading to match the correct verification standard value as outlined in the next section.



Figure 3: Verification Standard Flowchart

Note: The Measure Verification Standard flowchart is a graphical representation of the Verification of Calibration directions.

Adjust for Linear Offset

1. Once you are certain a linear offset has occurred, toggle to the Configuration tab by pressing the Document icon button. Calibration is the first option highlighted in the Configuration tab. Press the Enter icon button to begin the verification process. The on screen commands guide you through the linear offset routine. The Calibration screen prompts you to start.



2. Press the Enter button to start the linear offset process. To return to the Configuration Screen, press the Cancel button. After pressing the Enter button, the Calibration screen prompts you to insert a fresh standard and seal the chamber.

Calibration
Insert a fresh standard and seal the chamber.

3. Empty the whole vial of solution into a sample cup. We recommend using the 6.00 NaCl (0.76 a_w). Do not adjust for the offset using steam distilled water. Ensure the rim and outside of the cup are clean. Place the sample cup in the TDL sample chamber.

Note: You may use the same verification standard 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 to return to the previous screen.

5. After your AquaLab has finished measuring the verification standard, it displays a Change the Offset 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 without saving changes and return to the main menu, press the Cancel button.
- 7. Re-measure the verification standard again in normal sampling mode. It should read the proper value (within $\pm 0.005 a_w$) 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 $\pm 0.005 \ a_w$ 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 METER by email at support.food@metergroup.com or by phone at 509-332-5601 for further instructions. If you purchased your METER instrument from one of our international distributors, please contact them for local service and support.

Note: A linear offset does not adjust the calibration for all water activity levels and should only be used if the user intends to measure water activity in a very small range.

Multi-Point Calibration for New Temperatures

1. The AquaLab TDL is optimized for performance at specific temperatures. If testing is to be conducted at new temperatures, an updated calibration will be needed. If a temperature setting that has not been used previously is chosen, an error message will appear instructing the user that the calibration needs to be updated.

Note: Factory calibrations performed by METER are locked and cannot be updated with a multipoint calibration, only with a linear offset as outlined above.

This update can be either a 1 point linear offset (see instruction for adjusting for linear offset) or a multi-point update, which requires reading multiple water activity standards.



- 2. Change instrument temperature setting to desired testing temperature.
- 3. You will need 0.25 a_w , 0.50 a_w , 0.76 a_w and 1.00 a_w unsaturated salt standards from METER to proceed with multi-point

calibration.

4. To perform a multi-point calibration, select Calibration from the Configuration tab. The Calibration screen will prompt you with options.



- 5. Highlight Type and select Enter to toggle to multi-point. You will be guided through the multi-point calibration routine through on screen commands.
- 6. Toggle to the Start button and press Enter to begin the multipoint calibration. Once you press Enter, the Calibration screen will prompt you to insert a fresh standard and seal the chamber.



- 7. Empty the whole vial of $0.25 a_w$ standard solution into a sample cup. Ensure the rim and outside of the cup are clean. Place the sample cup in the AquaLab sample chamber.
- 8. Carefully close the lid and move the lever to the Read position. Press the Check icon button to begin testing.

9. After your AquaLab has finished measuring the verification standard, a new screen will appear requesting that a 0.50 a_w standard be placed in the chamber. Repeat steps 6 through 7 for 0.50, 0.76, and 1.00 a_w standards.

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.

10. When measurements are complete on all four standards, a verification complete screen appears showing the testing results for each standard.



11. Pressing the X button will cancel the calibration process while selecting the Check Mark will bring up the optimize prompt. You can toggle the audio icon to turn beeping on and off.



- 12. To make adjustments to the calibration, select the check mark or select the X button to cancel without adjusting the calibration.
- 13. After optimizing the new calibration, the Calibration Complete screen will appear.



14. To save the new calibration changes, select the Save icon and the calibration screen will verify that the AquaLab saved your new multi-point calibration.



15. To discard the calibration changes and exit without saving, press the X button and the system will return a calibration canceled message.



- 16. Measure a 0.76 a_w verification standard again in normal sampling mode. It should read the proper value (within $\pm 0.005 a_w$) at a given temperature (see Appendix B of a second verification standard (0.25 or 0.50 a_w) according to the verification procedure described above. If both verification readings are within $\pm 0.005 a_w$ then the instrument is ready to begin testing.
- 17. If you have trouble reading the water activity of standards at temperatures other than 25 °C after performing a multi-point calibration, contact METER by email at support.food@metergroup.com or by phone at 509-332-5601 for further instructions. If you purchased your METER 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 Document 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 Default screen prompts you to reset defaults.



3. To restore the factory calibration values, select the Check icon. To cancel and return to the main menu, choose the Cancel button. After pressing the Check icon, the Calibration screen verifies restoration of factory calibration.



4. To return to the Main menu screen, select the Check icon.

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 lengthens time between cleanings and helps you avoid downtime.

8.1 Preparing the Sample

- 1. Make sure the sample to be measured is homogeneous. Multicomponent 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 section (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 contaminate the sensors in the sensor chamber. Filling the sample cup does 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 you want to save a sample for later, put the disposable sample cup lid on the cup to restrict water transfer. For longterm storage, seal the lid by placing tape or Parafilm[®] completely around the cup/lid junction.
- 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.

8.2 Samples Needing Special Preparation

AquaLab reads most materials in five minutes or less. However, the nature of some samples necessitates longer reading times. 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 ensures 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 test, the chamber may be dirty. Refer to Section 6 for cleaning procedures.

Coated and Dried Samples

Samples with high sugar or fat coatings 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 a 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. However, keep in mind 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 represents the average water activity of the entire sample; whereas leaving the candy whole gives a reading for the coating, which may act as a barrier to the center.

8.3 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 occurring. The most effective way to test these types of samples is to run them in the TDL using the continuous or custom mode and wait for the water activity readings to stabilize.

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, reaching equilibrium takes a longer period of time and it may affect the water activity of your sample.

8.4 Samples Not at Room Temperature

Samples that are 4 °C colder or warmer than the instrument (chamber) temperature need to equilibrate to instrument temperature before you can make a fast and accurate reading. Rapid changes in temperature over short periods of time cause the water activity readings to rise or fall until the temperature stabilizes. When the temperature stabilizes within an optimal 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 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 $^{\circ}$ C of the instrument temperature cause longer read times. The sample temperature must be within one or two degrees of the chamber temperature before you can take fast and accurate readings.

9 Taking a Reading

9.1 Measurement Steps

Once you have verified for cleanliness, calibration, and prepared your sample, you are ready to take readings. Follow steps 1 through 4.

- 1. Move the chamber lever to the Open position and lift the chamber lid.
- 2. Check the top lip and outside of the sample cup to make sure they are free from sample residue and that the sample cup is not overfilled.

Note: Over-filling the sample cup may contaminate the chamber sensors.

- 3. Place your prepared sample cup in the chamber. The sample cup lid must be removed while in the testing chamber for correct functionality.
- 4. Close the chamber lid and move the lever to the Read position. This seals the chamber and starts the reading.

In one to two minutes, the first water activity measurement displays on the LCD (this is an intermediate reading and not the final water activity). Length of read times may vary depending on temperature differences between the chamber and your sample, and other properties of your sample.

9.2 How AquaLab Takes Readings

The AquaLab reading cycle continues until the rate of change of three consecutive readings are less than 0.0005 a_w of each other. The instrument continually tracks the strength of the TDL signal 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 Save icon replaces the spinning measurement icon,

and, if enabled, you 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 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 chamber, the instrument beeps and displays a warning that the sample is too hot. Remove the sample until it is at room temperature.

Note: To check the differences in temperature between the sample and the chamber prior to beginning a read, set the sample in the chamber, close the lid without latching it, and press the right arrow button.



Although the instrument can 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

and 50 °C. The TDL can measure samples between these ambient temperatures quickly and accurately. The AquaLab TDL has temperature control capabilities that enable it to read samples at temperatures different from ambient temperature, but no higher than 50 °C.

10 Moisture Content Measurement



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.

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. Customers can use the product isotherm 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 Section 3.

The TDL generates water activity values and then uses preloaded product specific isotherm models to calculate moisture content and present it on the screen with the water activity.

Obtaining Product Isotherm Models

Since the isotherm relationship for each product is unique, each product isotherm model must be determined experimentally. This only needs to be done once, but must be done prior to testing moisture content with the TDL.

11 Computer Interface

Your AquaLab may connect to your computer using USB or RS232. Your AquaLab was shipped to you with a standard USB cable. Using this cable, you can send water activity data to a computer for further analysis and storage. The interface is run through the AquaLink 4 Software or a terminal communication program.

Note: You must install the USB driver before connecting the USB Cable to your computer. You can install from the USB included with your AquaLab or download here: www.aqualab.com/aqualink.

11.1 AquaLink 4 Software

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

A USB of this program was included with your instrument. Figure 4 shows a screen shot of the AquaLink 4 program.

	Connect Via			1 1	6	9/6	1
	COM4 Decagon	JCA	•	Connect Down	load Export	Models (Chart
 Date Time	Device	Water Activity	°C	Test Time	User	Туре	
2000-Jan-01 00:00:00	\$40001234	0.0000	0.00	0.0	Admin	Normal	De
2000-Jan-01 22:14:07	\$40001234	0.0010	1.43	3.0	Admin	Normal	De
2000-Jan-02 20:28:14	\$40001234	0.0020	2.86	2.9	Admin	Normal	De
2000-Jan-03 18:42:21	\$40001234	0.0030	4.29	5.9	Admin	Normal	De
2000-Jan-04 16:56:28	\$40001234	0.0040	5.72	5.8	Admin	Normal	De
2000-Jan-05 15:10:35	\$40001234	0.0050	7.15	5.7	Admin	Normal	De
2000-Jan-06 13:24:42	\$40001234	0.0060	8.58	5.5	Admin	Normal	De
2000-Jan-07 11:38:49	\$40001234	0.0070	10.01	5.4	Admin	Normal	De
2000-Jan-08 09:52:56	\$40001234	0.0080	11.44	5.3	Admin	Normal	De
2000-Jan-09 08:07:03	S40001234	0.0090	12.87	5.2	Admin	Normal	De
2000-Jan-10 06:21:10	S40001234	0.0100	14.30	5.1	Admin	Normal	De
2000-Jan-11 04:35:17	S40001234	0.0110	15.73	5.0	Admin	Normal	De
2000-Jan-12 02:49:24	S40001234	0.0120	17.16	4.8	Admin	Normal	De
2000-Jan-13 01:03:31	S40001234	0.0130	18.59	4.7	Admin	Normal	De
2000-Jan-13 23:17:38	S40001234	0.0140	20.02	4.6	Admin	Normal	De
2000-Jan-14 21:31:45	S40001234	0.0150	21.45	4.5	Admin	Normal	De
2000-Jan-15 19:45:52	S40001234	0.0160	22.88	4.4	Admin	Normal	De
2000-Jan-16 17:59:59	\$40001234	0.0170	24.31	4.3	Admin	Normal	De
2000-Jan-17 16:14:06	S40001234	0.0180	25.74	4.1	Admin	Normal	De
2000-Jan-18 14:28:13	\$40001234	0.0190	27.17	4.0	Admin	Normal	De
2000-Jan-19 12:42:20	\$40001234	0.0200	28.60	3.9	Admin	Normal	De
2000-Jan-20 10:56:27	\$40001234	0.0210	30.03	3.8	Admin	Normal	De

Figure 4: AquaLink 4 Screen Shot

11.2 AquaLink 4 Part 11 Compatible Software

This version of AquaLink 4 is available for customers needing to be CFR Part 11 compliant. The software contains the required elements to be used in a Part 11 compliance system.

11.3 Using a Communication Program

There are several terminal program options. METER has its own terminal program (DecaTerm) which can be downloaded from http://software.metergroup.com/DecaTerm.zip. Two other options are TeraTerm, which is a free program that can be found on the Internet and Hyperterminal which came standard with Microsoft Windows prior to Windows 7.

To use any of these terminal programs with your AquaLab, follow the instructions for the program with the following settings. Be sure to power on the AquaLab prior to connecting the USB interface cable to your computer.

- Choose correct Com port
- Set/Verify Com Properties

✓ Bits per second 9600
✓ 8 Databits

8 Databits

✓ No parity

- \checkmark 1 stop bit
- \checkmark Flow control set to none

After successfully connecting the AquaLab to your computer and upon completion of a water activity reading, the data displays in the terminal program in the format as follows: measurement time (minutes), sample temperature, and water activity. Table 2 shows an example data return.

Table 2: Terminal Data

Time since chamber was closed	Temperature (°C)	a_w
3.1,	24.3,	0.862

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 directs you to detailed solutions of some problems that may occur. If these remedies still do not resolve your problem, then please contact METER for help (see Customer Support in Section 1). Here is a list of some problems that may occur.

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

If this problem occurs:	Refer to:	
AquaLab does not turn on	Problem #1	
Readings are slow or inconsistent	Problem $#2$	
A_w readings on solutions are too high/low to ad-	Problem $#3$	
just		
Screen displays "Sample too hot"	Problem #4	
Verification is not correct	Problem $\#5$	
Screen displays "Crystal failure"	Problem $\#6$	
Screen displays "Pressure Sensor failure"	Problem $\#7$	
Screen displays "Firmware is corrupted"	Problem #8	
Screen displays "Readings are disabled"	Problem #9	
Test was run with wrong model	Problem #10	
%Moisture Content displayed is not correct	Problem #11	
%Moisture Content is not shown on screen	Problem $\#12$	
Returns no moisture content reading	Problem $\#13$	

Table 3: Troubleshooting Quick Guide

1. PROBLEM:

AquaLab does not 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 instructions a through d.
 - (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-A 250-V fuse.

Caution: Do not use any other kind of fuse or you 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) Connect the power cord and turn your instrument on. If the fuse blows again, a failed component may be causing the problem. Contact METER to make arrangements for repairs. (See Section 13)

2. PROBLEM:

Readings are slow or inconsistent.

SOLUTIONS:

- 1. The sample chamber may be dirty. Refer to Section 6 for directions on cleaning the sample chamber.
- 2. The temperature difference between the sample and the block chamber may be too great. The sample must equilibrate to instrument temperature before a making a fast and accurate reading. (Refer to Section 8)
- 3. 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 Section 8 for further

explanation.

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 IR Sensor in your chamber, which measures sample temperature, may have become contaminated. Refer to Section 6 for directions on cleaning.
- 2. The TDL lenses may be dirty. Refer to Section 6 for directions on cleaning.

4. PROBLEM:

Message on screen displays a warning that the sample is too hot.



SOLUTION:

Your sample 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 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:

Verification is not correct.

SOLUTION:

- 1. The sample chamber and components need to be cleaned. See Section 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 components you need to use a Verification Standard to verify and adjust for Linear offset as Section 7 describes.

6. PROBLEM:

Message on screen displays the following:



SOLUTION:

The crystal that runs the firmware is having trouble starting. Occasionally, cycling the power solves the problem. If this message continues to appear, METER needs to service the instrument. See Section 13 for detailed instructions.

7. PROBLEM:

Message on screen displays the following:



Pressure sensor failure!

SOLUTION:

The pressure sensor used during water activity measurements is not functioning correctly and needs to be replaced. METER needs to service the instrument. See Section 13 for detailed instructions.

8. PROBLEM:

Message on screen displays the error "Firmware is corrupted."



SOLUTION:

The firmware on the instrument is corrupted and needs to be reloaded. To download new firmware to the AquaLab TDL, the instrument must be serviced by METER.

11. PROBLEM:

Ran test 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 updates to correspond with the model you select.
- 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 Configuration tab, select %Moisture and then the loaded models appear.
- 3. If the correct model is not available, load the appropriate model using AquaLink 4 Software. The AquaLab TDL can hold a total of 100 models at any one time. You may need to remove a model using the Software or use the delete option in the %Moisture Calibration menu before you can add a new one. Any model that you remove from the instrument with AquaLink 4 stores in the software to use later.

12. 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 a model, contact METER for updating a model.

13. PROBLEM:

Moisture content does not show up on the screen.

SOLUTION:

Moisture content has not been activated.

- 1. Toggle to Configuration tab, select % Moisture, and select the appropriate model.
 - (a) If no models appear in moisture content screen, reload them using AquaLink 4 software.
 - (b) If moisture content is not an active selection, the model feature may not be active. Content METER to learn how to activate the model feature.

14. PROBLEM:

Message on the screen displays no moisture content reading.



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 conditions a or b.
 - (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 water activity and the instrument 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 displays values for component performance. Customers can access this Diagnostics screen by navigating to the Configuration tab and then by scrolling down to the diagnostics option. Press Enter and the TDL gives you a list of components and their values.

Diagnostics		
Lid:	25.20°C	Â
Base:	25.24°C	
Sample:	25.12°C	
LaserT:	26.68°C	<u> </u>
LaserI:	3893mV	_ 🕑
Diagnostics	∆=0.4	10°C
0ffset:25°C	:+0.0000 a _w	n
Pressure:	92.83 kPa	ري ال

13 Support and Repair

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

When encountering problems with your AquaLab (that you unable to resolve with the help of this manual), please contact METER Customer Support at support.food@metergroup.com, 509-332-5601 or fax us at 509-332-5158. Please have the serial number and model of the instrument ready.

AquaLab annual calibration is available. For details on sending your AquaLab to METER or your distributor in for calibration, contact us by phone or email at support.food@metergroup.com.

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

Shipping Directions

The following steps 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 four 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. Do not 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. On the RMA form, please verify the ship to and bill to information, contact name, and problem description. If anything is incorrect please contact a METER 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: METER Group, Inc. ATTN: RMA (insert your RMA #) 2365 NE Hopkins Court Pullman, WA 99163

13.1 Repair Costs

METER repairs manufacturer defects and instruments within the one year warranty at no charge. We bill non-warranty repair charges for parts, labor and shipping to you and may charge an extra fee for rush work. METER can provide an estimated repair cost, if requested.

13.2 Loaner Service

METER has loaner instruments available to keep you measuring water activity while your instrument is being serviced. Please contact customer support for pricing and availability of loaners. If your AquaLab is being serviced under warranty, you qualify for a free loaner.

14 Further Reading

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15 Appendix A

15.1 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.

Steps 1 through 4 detail the AOAC method.

- 1. Select a reagent-grade salt and place it in a test container to a depth of about 4 cm 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 2 mL, 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. Table 4 shows 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 accuracy in measuring the salt. AquaLab TDL measures all samples with an accuracy of $\pm 0.005 a_w$.

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 Carbonate	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

Table 4: Water Activity of Selected Salt Solutions

Note: Table 4 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 METER.

16 Appendix B

Temperature Correction of METER's Verification Standards

		0.50	2.33	6.00	8.57	13.41	17.18
Temp.	H_2O	mol/kg	mol/kg	mol/kg	mol/kg	mol/kg	mol/kg
(°C)		KCL	NaCL	NaCL	LiCl	LiCl	LiCl
15.0	1.000	0.984	0.923	0.761	0.492	0.238	0.140
20.0	1.000	0.984	0.922	0.760	0.496	0.245	0.145
25.0	1.000	0.984	0.920	0.760	0.500	0.250	0.150
30.0	1.000	0.984	0.920	0.760	0.504	0.255	0.155
35.0	1.000	0.984	0.920	0.760	0.508	0.261	0.160
40.0	1.000	0.984	0.921	0.760	0.512	0.266	0.165
50.0	1.000	0.984	0.894	0.740	0.517	0.275	0.172

Table 5: Water Activity of Selected Salt Solutions

Note: AquaLab TDL measures these verification standards to $\pm 0.005 a_w$.

17 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 alerts 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 seven water activity levels: 1.000, 0.984, 0.920, 0.760, 0.500, 0.250, and 0.150 a_w . The standards are produced under a strict quality assurance regime by an independent third party that verifies the standards and they are shelf stable for one year. Order your calibration 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 takes on the shape of the container but does 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 produces 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 coefficients for various salt solutions. Customers can use these activity coefficients to the water potential, or partial specific Gibbs free energy, of the water in the solution using;

$$\Psi = -\phi\gamma cRT\tag{1}$$

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/kg¹), R is the gas constant (8.314 J mol/kg⁻¹ K⁻¹), T is the Kelvin temperature. Water potential is related to water activity by the equation;

$$a_w = \exp\left(\frac{\Psi M_w}{RT}\right) \tag{2}$$

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

$$a_w = \exp(-\phi\gamma cM_w) \tag{3}$$

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

$$a_w = \exp(-2 \times 1.271 \times 6 \times 0.018) = 0.760 \tag{4}$$

It is important to note that equation 3 has no explicit temperature dependence. Available data on temperature dependence of γ indicates variation is less than $\pm 2\%$ 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 METER'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 fills a sample dish to just less than half full. Table 6 shows the expected values.

Note: If you need to obtain a Safety Data Sheet (SDS) for any of these standards, a printable version is available on our website at http://sds.metergroup.com/.

Verification Standard	Distilled H ₂ O
Water Activity	1.000 ± 0.005
0.50 mol/kg KCl	0.984 ± 0.005
2.33 mol/kg NaCl	0.920 ± 0.005
6 mol/kg NaCl	0.760 ± 0.005
8.5 mol/kg LiCl	0.500 ± 0.005
13.4 mol/kg LiCl	0.250 ± 0.005
17.18 mol/kg LiCl	0.150 ± 0.005

Table 6:	Verification	Standard	Expected	Values
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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 you reach a final reading, check it against the values in Table 6. If it is within ± 0.005 , place your second solution in the drawer for testing. It should read the value ± 0.005 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 ± 0.005 of the expected value, then you need to adjust the linear offset so that the solution reads correctly (see Section 7). When you are finished measuring both standards, the readings should be within ± 0.005 of the predicted values.

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18 Declaration of Conformity

Application of Council Directive:	$2004/108/\mathrm{EC}$ and $2011/65/\mathrm{EU}$
Standards to which conformity is declared:	EN 61326-1:2013 and EN 50581:2012
Manufacturer's Name:	METER Group, Inc 2365 NE Hopkins Ct. Pullman, WA 99163 USA
Type of Equipment:	AquaLab water activity meter.
Model Number:	AquaLab Tunable Diode Laser (TDL)
Year of First Manufacture:	2015
Laser Type:	Class 1

(Class 1 Lasers are safe under all conditions of normal use. Do not exceed maximum permissible exposure (MPE) when viewing a laser with the naked eye or with the aid of typical magnifying optics (e.g. telescope or microscope).)

The undersigned hereby declares on behalf of METER Group, Inc. that the above referenced products, to which this declaration relates, fully conform to the provisions of the Council Directives and standards referenced above.

Michel B Wadness

Michael Wadsworth Engineering Director 7-9-2015

19 Certificate of Traceability

METER Group, Inc. 2365 NE Hopkins Court Pullman WA 99163 USA

Tel: 509-332-5601 Fax: 509-332-5158 support.food@metergroup.com

METER Group, Inc. manufactures all AquaLab water activity meters according to accepted international temperature standards with traceable calibration.



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