

# model *CX-2*

Water Activity Meter

Operator's Manual Version 3.0

Decagon Devices, Inc.

AquaLab Table of Contents

# **Table of Contents**

## **1. Introduction 1**

About this Manual 1 Customer Service 1 Phone: 1 Fax: 2 E-mail: 2 Warranty 3 Note to our AquaLab Users 3 Seller's Liability 3

## 2. About AquaLab 5

How it works 5 Features: 8

## **3. Getting Started 9**

Components of your AquaLab 9 Choosing a Location 9 Checking and Changing Voltage Settings 10 Preparing AquaLab for Operation 12

## 4.Linear Offset and Verification Standards 14

What is Linear Offset? 14 Verification Standards 14 Saturated Salt Solutions 15 AquaLab's Verification Standards 16 When to Verify for Linear Offset 18 How to Verify and Adjust for Linear Offset 18

## **5. Sample Preparation 21**

Table of Contents

Preparing the Sample 21 Materials Needing Special Preparation 22 Coated and Dried Samples 23 Speeding up Read time 23 Dehydrated Samples 24 Propylene Glycol 24 Low Water Activity 25 Samples not at Room Temperature 25

## 6. Taking a Reading 26

How AquaLab takes Readings 27 Cautions 27

## 7. Temperature Control 29

Important Hints Concerning the CX-2T 31

## **8.** Computer Interface 33

Output Format 33 Using Hyperterminal in Windows 95 and Windows NT 34 Using Terminal for Windows 3.0 and 3.11 35 Using AquaLink 35

## 9. Theory: Water Activity in Foods 38

## **10. Cleaning and Maintenance 40**

Tools Needed 40 Cleaning the Sensor Chamber Block 41 Cleaning the Fan Filter 45 Checking Calibration 46

## **11. Repair Instructions 48**

Shipping Directions 48 Repair Costs 49

AquaLab Table of Contents

**Loaner Service 50** 

## 12. Troubleshooting 51

**Problems and Solutions 51** 

## 13.Further Reading 56

Water Activity Theory and Measurement 56 Food Quality and Safety 59 Water Activity and Microbiology 60 Water Activity in Foods 63 Meat and Seafood 63 Dairy Products 65 Fruits and Vegetables 66 Baked Goods and Cereals 67 Beverages/Soups/Sauces/Preserves 68 Pharmaceuticals 69 Miscellaneous 70

## **Appendix A: Salt Solutions 72**

**Preparing Salt Solution 72** 

# 1. Introduction

Welcome to Decagon's **AquaLab model CX-2**, 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. We hope you find this manual informative and helpful in understanding how to maximize the capabilities of your AquaLab.

# **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 the AquaLab to ensure that the instrument performs to its full potential.

## **Customer Service**

If you ever need assistance with your AquaLab, or if you just have questions, there are several ways to contact us:

## Phone:

Our **toll-free customer service number** is available to our customers in the US and Canada, Monday through Friday, between 8 a.m. and 5 p.m. PST at **1-800-755-2751**.

## Introduction

For our customers outside of the US and Canada, our regular telephone number is **(509) 332-2756**.

## Fax:

Our fax number is **(509) 332-5158.** When you fax us, please include your AquaLab's serial number, your name, address, phone and fax number along with a description of your problem so we can help you with your problem and then get back to you as soon as possible.

#### E-mail:

If you need technical support, you can send us messages via email at **support@decagon.com**. Again, please include as part of your message your AquaLab's serial number, your name, address, phone, fax number, and return e-mail address.

If you have a question about your application with AquaLab, please send your message with the above information to **aqualab@decagon.com**.

AquaLab Introduction

## Warranty

AquaLab has a 30-day satisfaction guarantee and a one year warranty on parts and labor. To validate your warranty, please complete and return the warranty card included with this manual. You can return your warranty information by fax, email, phone or simply by mailing in the postage-paid card. Please include all of the information requested on the warranty card. It is necessary for Decagon to have your current mailing address and telephone number in case we need to send updated product information to you.

# Note to our AquaLab Users

We understand that some of our references to scientific terminology in this manual may not meet the criteria of some members of the scientific community. We apologize for this. However, this manual is written to aid the end user in understanding the basic concepts and theories of water activity, enabling them to use our instrument with confidence. Every effort has been made to ensure that the content of this manual is correct and scientifically sound.

# Seller's Liability

Seller warrants new equipment of its own manufacture against defective workmanship and materials for a period of one year 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 F.O.B. 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

## Introduction

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 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

Water activity  $(a_w)$  is the measurement used to determine how tightly water is structurally or chemically bound within a substance. Not only is it a vapor pressure ratio, but it can also be considered equal to the relative humidity of air in a sealed chamber that is equilibrated with a substance. One of the most common uses for the AquaLab is to measure the  $a_w$  of food samples.  $a_w$  influences color, odor, flavor, texture and shelf-life of a food product. If  $a_w$  is not controlled, the quality and safety of a product may suffer as a result. For a more detailed description of water activity in food samples, please refer to Chapter 9, titled "Theory: Water Activity in Foods" of this manual.

# How it works

AquaLab uses the chilled-mirror dew point technique to measure the  $a_w$  of a sample. This technique is a primary measurement method of relative humidity. When a sample is measured with AquaLab, a stainless steel mirror within the chamber is repeatedly cooled and heated. As it does so, dew is continually forming and evaporating. The instrument's fan circulates air in the sensing chamber, which speeds up the equilibration process. Each time dew forms on the mirror, AquaLab measures the temperature and calculates  $a_w$  of the sample. Each  $a_w$  value is saved and compared to previous values as it makes new

#### AquaLab About AquaLab

readings. When the  $a_w$  values of any two readings are less than .001 apart, the instrument has reached equilibrium and the measurement process stops. AquaLab then signals you by beeping. The final water activity and temperature of the sample is then displayed.

With AquaLab, temperature control is unnecessary for most applications. The change in  $a_w$  due to temperature change for most materials is less than .002 per degree Celsius. Therefore, model CX-2 is ideal for the measurement of samples at room temperature. However, samples that are not at room temperature during the read cycle will equilibrate to the temperature of AquaLab. This variable temperature will cause longer reading times, since a complete and accurate reading will not be made until the sample and the instrument are within 2 degrees of each other. For applications where temperature control is critical, AquaLab can be operated in a controlled temperature chamber or room, or you may purchase an AquaLab model CX-2T.

AquaLab is the fastest instrument for measuring water activity, giving readings in five minutes or less. Its readings are precise, providing  $\pm 0.003$  accuracy. The instrument is easy to clean and checking calibration is simple.

AquaLab has a few limitations, however. One of these is its ability to measure certain samples with high concentrations of propylene glycol, which can condense on the surface of the chilled mirror. Not all volatiles react this way; glycerol, for example, has posed no problems, and most other alcohols used to flavor foods are also measurable. If your sample contains propylene glycol, it is still possible to make accurate readings. Call Decagon for more details. AquaLab may also have trouble measuring extremely dry substances (with an  $a_w$  of less than 0.1). Some very dry or dehydrated foods absorb or desorb moisture in such a way that their readings may take longer than five minutes. Black pepper, for example, causes faulty readings at first, but after the initial gas has dissipated, the sample can be read accurately.

About AquaLab

# **Features:**



# 3. Getting Started

# **Components of your AquaLab**

Your AquaLab should have been shipped with the following items:

- AquaLab main unit
- Power cord
- 100 disposable sample cups
- Operator's Manual
- 3 vials each of the following verification solutions: .760 a<sub>w</sub> NaCl
  .500 a<sub>w</sub> LiCl
  .250 a<sub>w</sub> 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 inside of the instrument. To protect the internal electrical components, and to avoid inaccurate readings, place your AquaLab in a location where the temperature remains fairly stable. This location should be well

Linear Offset and Verification Standards

away from air conditioners, heaters, open windows, outside doors, refrigerator exhausts, or other items that may cause rapid temperature fluctuation.

# **Checking and Changing Voltage Settings**

Users in the United States and Canada will not have to change AquaLab's voltage setting. Users in other countries, particularly those which supply power at 220V or 240V, should check the voltage setting before using the instrument. Your AquaLab may sustain damage if the voltage is incorrectly set.

AquaLab will operate at both 50 and 60Hz and at any voltage near 110, 120, 220, or 240 volts. The voltage setting can easily be changed by rotating a tab inside the power module. The power module is located in the lower right corner of the instrument's back panel.



• Unplug AquaLab's power cord before checking or changing the voltage.

- Slide the plastic cover to the left to expose the fuse chamber.
- Remove the fuse by pulling out the fuse pull lever (located under the fuse to the right).



When the fuse has been removed, the voltage board, a small light-colored board located in the bottom of the fuse chamber, will be visible. The current voltage setting is displayed in the back left hand corner. If this voltage matches the voltage of the power source to be used, replace the fuse and continue setting up AquaLab. If the two do not match, the voltage tab needs to be changed to match the correct line voltage.

To change the voltage:

- Remove the tab by inserting a small screwdriver or awl into the hole at the center bottom of the power module chamber.
- Slide the tool under the tab and hook it into the hole in the center front of the tab. You may need to hold the fuse pull

Getting Started

lever as you slide the tab gently out. The tab allows you to select between four voltage settings: 100, 120, 220, and 240. If your power source does not exactly match one of these settings, choose the closest setting.

- Align the tab by rotating it until the number corresponding to the voltage you want is right side up and facing you. Slide the tab back into position. The correct voltage should now be displayed in the back left hand corner.
- Carefully replace the fuse and slide the plastic cover over the fuse chamber.

# **Preparing AquaLab for Operation**

After finding a good location to do your sampling and checking the voltage, plug the power cord to the back of the unit. Before turning it on, pull open the sample drawer (turn the knob to the "OPEN/LOAD" position). An empty disposable sample cup is usually placed upside-down in the drawer to protect it during shipment. Remove this sample cup and turn the instrument on. The ON/OFF switch is located on the lower left corner of the AquaLab's front panel. When you turn it on, all the segments on the LCD will appear (like "8's" across the screen). Within 30 seconds, the display should show all zeroes.



In order to provide the most accurate readings, AquaLab should

AquaLab Getting Started

ideally be allowed a warm-up period of 15 minutes to an hour after turning it on. This allows the air inside the AquaLab to equilibrate to the temperature of its surroundings. It is also helpful to prepare a sample and take some readings during this equilibration period in order to warm up the sample chamber. For instructions on how to prepare samples and take readings, refer to Chapters 5 and 6 of this manual.

# 4.Linear Offset and Verification Standards

# What is Linear Offset?

AquaLab uses the chilled mirror dewpoint technique for measuring water activity. Because this is a primary measurement method of relative humidity, no calibration is necessary; however, it is important to check for linear offset periodically. The components that the instrument uses to measure  $a_w$  are subject to changes that may affect AquaLab's performance. These changes are usually the result of chamber contamination. When this occurs, it changes the accuracy of your readings. This is what is called a "linear offset." Therefore, frequent linear offset verification can assure you that your AquaLab is performing correctly. Linear offset can be checked by using a salt solution and distilled water.

AquaLab should be checked for linear offset daily. For high use or batch processing, the instrument should be checked more often against a known standard of similar water activity. Checking the water activity of a standard solution and distilled water will alert you to the possibility of contamination of the unit or shifts in the linear offset from other causes.

# Verification Standards: Two choices

Verification standards are specially prepared salt solutions that have a specific molality and water activity that is constant and accurately measurable. There are two types of verification solutions: saturated and non-saturated. The verification standards that were sent with your initial shipment are nonsaturated solutions, and they are readily available from Decagon Devices. These particular standards are accurate, easy to use, and are not temperature-sensitive, like saturated solutions can be. Most importantly, they greatly reduce preparation errors. Because of these reasons, we recommend using the standards provided by Decagon for the most accurate verification.

Decagon's Performance Verification Standards come in three water activity levels; 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.

## **Saturated Salt Solutions**

The water activity values listed in Appendix A 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. 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

Linear Offset and Verification Standards

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.

#### AquaLab's Verification Standards

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;

$$\Psi = \phi \gamma c R T \tag{1}$$

where  $\Psi$  is the water potential,  $\phi$  is the number of active particles per molecule of solute (i.e., 2 for NaCl),  $\gamma$  is the activity coefficient, c is the concentration of the solute (mol kg<sup>-1</sup>), R is the gas constant (8.314 J mol<sup>-1</sup> K<sup>-1</sup>), and 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 kg mol<sup>-1</sup>). When equations 1 and 2 are combined, a simplified equation for water activity is obtained;

$$a_w = \exp(-\phi \gamma c M_w)$$

(3)

For example, equation 3 gives the  $a_w$  in a 6M NaCl solution, ( $M_w = 0.018$  kg mol<sup>-1</sup>,  $\phi = 2$ , and  $\gamma = 1.271$ ; 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  $\gamma$  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.

Verification Standard	Water Activity
.5M KCl	0.983 ±0.003
6M NaCl	$0.760 \pm 0.003$
8.5M LiCl	$0.500 \pm 0.003$
13.3M LiCl	0.250 ±0.003

Linear Offset and Verification Standards

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.

# When to Verify for Linear Offset

Linear offset should be checked against a known verification standard on a frequent (if not daily) basis. Linear offset should never be adjusted when measuring 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  $a_w$  It is also a good idea to check the offset with a standard of similar  $a_w$  when the general water activity range of your sample is changing. Checking the  $a_w$  of a standard solution will alert you to the possibility of contamination of the unit or shifts in the linear offset from other causes.

# How to Verify and Adjust for Linear Offset

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

1. Choose a verification standard that is close to the  $a_w$  of the sample you are measuring. Each of the verification standards supplied by Decagon has its  $a_w$  labeled. Before you begin sampling, make sure that your standard is at ambient temperature before you load it into the sample drawer, and that your AquaLab has warmed up long enough to make accurate readings.

- 2. Empty the whole vial of solution into a sample cup and place it in the AquaLab's sample drawer.
- 3. Carefully slide the drawer closed, being especially careful so the solution won't splash or spill and contaminate the chamber.
- 4. Turn the drawer knob to the READ position to make an  $a_w$  reading. Make two readings. The  $a_w$  readings should be within  $\pm$  .003 of the given value for your salt solution.
- 5. If your AquaLab is reading within .003 of the salt solution, prepare a sample cup half full of distilled water and make two readings—the first reading may be low. The  $a_w$  reading should be 1.000 ± .003. If your salt reading is correct and your distilled water reading is not, it is probably due to contamination of the sensor chamber. For cleaning instructions, see Chapter 10. After cleaning, repeat these instructions.
- 6. If you consistently get readings that are outside of the  $a_w$  of your salt solution by more than ±.003, a linear offset has probably occurred. In this case, adjust the reading on the AquaLab to the correct value. This is done by twisting the potentiometer located on the left side of the screen with a small flat-head screwdriver while the AquaLab is still beeping.
- 7. After adjusting for linear offset, prepare a sample cup half full of distilled water and make two readings. The  $a_w$  reading for the distilled water should be 1.000 ±.003. If it is not within this range, clean the sensor chamber (see Chapter 10) and repeat these instructions. If it is reading within this

Linear Offset and Verification Standards

range, you can now measure samples accurately.

8. If, after adjusting for linear offset and cleaning the chamber, you still are getting incorrect readings when reading verification standards, contact Decagon at 509 332-2756 (1-800-755-2751 in US and Canada) for further instructions.



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

# **5. Sample Preparation**

Your AquaLab will continually provide accurate water activity measurements as long as its internal sensors are not contaminated by improperly-prepared samples. Careful preparation and loading of samples will lengthen time between necessary cleanings and will help you avoid costly repairs and down time.

# **Preparing the Sample**

To prepare a sample, follow these steps:

- 1. Make sure the material to be sampled is homogeneous. Samples that combine wet and dry ingredients (i.e., muffins with raisins) or samples that have outside coatings (like deep-fried, breaded foods) can be measured, but will take longer to equilibrate. For samples like these, AquaLab may take more than five minutes to give an accurate reading, or it may require multiple readings of the same sample. Measuring the  $a_w$  of these types of foods is discussed more in-depth later in this chapter (see Materials 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, but a larger sample surface

Sample Preparation

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!
- 4. Make sure that the rim and outside of the sample cup are clean. Wipe any excess sample material from the rim of the cup with a clean tissue. Material left on the rim or the outside of the cup will contaminate the sensor chamber and will be transferred to subsequent samples. The rim of the cup is pushed up to form a vapor seal with the sensor block when the drawer knob is turned to the READ position. Therefore, any sample material left on the cup rim will be transferred to the block, preventing this seal and contaminating future samples.
- 5. If the same sample will be read at some other time, put the sample cup's disposable lid on the cup to restrict water transfer. To seal the cap, place tape completely around the cup/lid junction. It is necessary to seal the cup if it will be a long time before the measurement is made.

# **Materials Needing Special Preparation**

AquaLab reads most materials in less than five minutes, depending on which mode you are operating in. Some samples, however, may require longer reading times, due to the nature of the material you are sampling. These materials need additional preparation to ensure quick, accurate readings. To find out whether special sample preparation is necessary, take a reading and see how long it takes to find the water activity. If it takes longer than six minutes, remove the sample and take a reading of a verification standard. This will ensure that 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, refer to Chapter 12 of this manual for more information.

# **Coated and Dried Samples**

Samples that have coatings such as sugar or fat often require longer reading times. This is because it takes longer for the samples to equilibrate. If this is the case for your samples, don't worry that something is wrong with your instrument; it simply means that your particular sample takes longer than most to exchange water with its outside environment.

## Speeding up Read time

To reduce the time needed to take an  $a_w$  reading for coated or dried samples, one thing you can do is crush the sample before putting it in the sample cup. Crushing increases the surface area of the sample, thus decreasing reading times. Keep in mind, however, that crushing some samples may alter their  $a_w$ readings. For example, a candy may have a soft chocolate center and a hard outer coating. The  $a_w$  reading for the center and the outer coating are different, so you need to evaluate which part of the sample you need to measure before crushing it. When the candy is crushed, the  $a_w$  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.

Another way to speed up readings for coated and dried samples

#### AquaLab Sample Preparation

is to restart your AquaLab during its reading cycle. To do this, start the read cycle (turn the sample drawer knob to the READ position) and then wait 60 seconds. Then turn the sample drawer knob from READ to the OPEN/LOAD position, then back to READ. The instrument should beep again and the display should show zero for the  $a_w$  reading. This action essentially speeds up sample equilibration by restarting the read cycle while keeping the chamber sealed.

# **Dehydrated Samples**

Some extremely dry samples, such as dehydrated foods, because of their moisture sorption characteristics, also have increased reading times. AquaLab may require up to ten minutes to reach an accurate measurement of  $a_w$ . Nothing can be done to decrease the reading times of some dehydrated samples.

# **Propylene Glycol**

AquaLab will give accurate readings on most alcohols. However, samples with high levels of propylene glycol require special sampling procedures to get accurate readings. If your sample contains propylene glycol in levels of less than 10%, there will probably be no effect on the accuracy of consecutive  $a_w$  readings. Samples that contain propylene glycol in concentrations higher than 10% will not damage the instrument, but  $a_w$  values for consecutive samples will not be accurate unless certain precautions are taken. Propylene glycol condenses on the mirror during the reading process, but it never evaporates from the mirror, as water does. As a result, the very first reading will be somewhat accurate, but subsequent readings will not be accurate unless you clear the condensed propylene glycol out of the chamber after each reading. This is

done by running a sample consisting of activated charcoal after each propylene glycol-bearing sample. Another option is to clean the chamber, as described in Chapter 10.

# Low Water Activity

Samples that have an  $a_{\rm w}$  of less than .03 cannot be accurately

measured. However, samples with such low  $a_w$  values are rare.

When a sample's  $a_w$  value is less than .03, AquaLab will display an error message informing you that your sample is too dry to be read. If your sample is not extremely dry but is still getting the error message, refer to the troubleshooting section of this manual for other possible explanations.

# Samples not at Room Temperature

Samples that are 4 degrees colder or warmer than the instrument (chamber) temperature will need to equilibrate to ambient temperature before a fast, accurate reading can be made. Rapid changes in temperature over short periods of time will cause the  $a_w$  readings to rise or fall until the temperature stabilizes. Because the AquaLab is always comparing its previous and current results, it will not terminate the read cycle until its compared readings are less than .001 apart.

Initial readings on colder or warmer samples should not be averaged. Such measurements will not be accurate. When the temperature stabilizes within one or two degrees of the chamber temperature, you can proceed with normal measurements.

# 6. Taking a Reading

Once you have prepared your sample, you are ready to take readings. The process is simple:

- 1. Turn the sample drawer knob to the OPEN/LOAD position and pull the drawer open.
- 2. Place your prepared sample in the drawer. Check the top lip of the cup to make sure it is free from sample residue (remember, an over-filled sample cup may contaminate the chamber's sensors).
- 3. Carefully slide the drawer closed, being especially careful if you have a liquid sample that may splash or spill and contaminate the chamber.
- 4. Turn the sample drawer knob to the READ position to seal the sample cup with the chamber. This will start the read cycle. In about 3 minutes, the first  $a_w$  measurement will be displayed on the LCD. Length of read times may vary depending on how dry your sample is.

*Note:* Samples that have a large difference in  $a_w$  from previous samples may need extra time to reach equilibrium, since some of the previous sample's atmosphere stays in the chamber after measurement.

AquaLab Taking a Reading

# How AquaLab takes Readings

AquaLab's reading cycle continues until two consecutive readings are within 0.001 of each other. The instrument crosses the dew threshold numerous times to ensure the accuracy of readings. When the instrument has finished its read cycle, the  $a_w$  is displayed, accompanied by the beeper.

## 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.
- Do not overfill the sample dish.
- 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.
- Take special care not to move the sample drawer too quickly when loading liquid samples, in order to avoid spilling.
- If a sample has a temperature that is four degrees higher (or more) than the AquaLab's chamber, the instrument will alert you to cool the sample before reading. Although the instrument will measure warmer samples, the readings may be inaccurate.
- The physical temperature of the instrument should be between 5°-43°C. Between these ambient temperatures, AquaLab will measure samples of similar temperature

Taking a Reading

quickly and accurately. AquaLab model CX-2T has temperature control capabilities that enable it to read samples at temperatures different from ambient temperature, but no higher than 43°C.

- If a sample has an  $a_w$  of lower than .03, AquaLab's display will read "LO" to notify you that your reading is too low. If you know that your sample's water activity is above .03, and you still get the "LO" message, your instrument's sensors have probably been contaminated and will need to be cleaned or serviced. Please refer to Chapter 11 for more information.
- A reading of "L0" is also displayed if the instrument has not detected the formation of dew on the mirror during one complete read cycle. This is an indication of contamination and the instrument should be cleaned.
- To measure powdery samples, take the following precautions to prevent contamination of the chamber. When first loading the sample, cover the case fan in the rear of the instrument with a piece of cardboard until the drawer is closed and locked. this will prevent the fan, which is always on, from blowing your sample around inside of the instrument and chamber. Once the sample drawer is pushed all the way in, take the card away from the fan.

# 7. Temperature Control

In the past, water activity instruments have needed temperature control to make accurate sample readings. AquaLab takes precision measurements of the air dew point temperature and the sample temperature, then uses a microprocessor-controlled algorithm to convert these temperatures and other critical information into a water activity reading. AquaLab's water activity readings are therefore accurate without temperature control.

Users constructing and studying moisture sorption isotherms, referencing a particular temperature, or testing samples more than four degrees Celsius higher than the ambient temperature may want to have temperature control. There are several options for controlling AquaLab's temperature. One is to use an incubator or hot/cold room set at the desired temperature. This method is usually satisfactory for users with access to those facilities. AquaLab is also available with a temperature control feature which allows it to be connected to an external water bath.

Temperature control can be purchased as a feature on the AquaLab model CX-2T instrument or retrofit in an existing AquaLab model CX-2. Water is circulated from a user-provided water bath through the sample drawer base and a temperature tray. The temperature tray holds two samples and equilibrates them to the temperature of the drawer. This speeds the reading

## Temperature Control

process. However, the water bath should only be used for higher than ambient samples. The temperature of the whole instrument needs to be altered when reading samples colder than AquaLab. The coldest ambient temperature of AquaLab and the cold samples should not be below 5°C.

If you have purchased AquaLab with the temperature control feature, you should have received:

- AquaLab fit with water ports and internal circulating base
- Temperature Tray
- Tygon tubing for connections
- 4 Elbows, 2 with valves (for the rear of AquaLab)
- 2 without valves (for the sample tray)

The drawer should be placed in the feed loop from the water bath and the temperature tray should be placed in the return path. This keeps sample temperatures below the block temperature to ensure that unwanted condensation does not occur.

#### AquaLab Temperature Control



Diagram of CX2T setup

*Note: Water temperature settings and water circulation are controlled by the user-provided water bath.* 

# **Important Hints Concerning the CX-2T**

- The CX-2T should be hooked up to a running water bath for at least one-half hour before the unit is turned on. This allows the water bath to reach a desired and accurate temperature.
- When using your CX-2T, it is important that the temperature of the drawer and the temperature of the sample are the same. The sample to be measured should sit in the temperature tray for at least 3 minutes before any measurements are taken.
- When running the CX-2T with a water bath, the instru-

Temperature Control

ment's case fan must always be turned off. When the water bath is not employed, the fan must remain on. The switch controlling the case fan is located on the instrument's back panel above the fan filter.

- The case fan keeps the block temperature constant when the water bath is not in use. This helps to ensure accurate readings. When a water bath is employed, the water maintains the block temperature. If the case fan is on at this time it will cool the block to a temperature lower than the sample causing condensation within the block and faulty readings.
- As the temperature of the CX-2T increases, water activity readings will decrease. It is necessary to adjust for an offset when the instrument is run at different temperatures. Instructions for this process are located in Chapter 7 of this manual.

*Note: Do not try to chill the CX-2T using a water bath. Dew will form throughout the sample chamber causing accuracy errors. To read chilled products, the entire instrument must be chilled.* 

For more detailed information regarding the operation of the CX-2T, please refer to Appendix A.
# 8. Computer Interface

Your AquaLab has the capability to send water activity data to your computer for further analysis and storage. To do this, you can purchase an AquaLab RS-232 interface cable and a terminal program called AquaLink or you can use the RS-232 cable with your computer's own terminal program. The RS-232 cable is specially designed for use with the CX-2, so other types will not work.

## **Output Format**

AquaLab data is output in comma delimited ASCII format. Each time the AquaLab makes a measurement, one line of information will be sent to the computer. Here is an example illustrating the format in which it appears on screen:

187,.405,20.77,6

The first number that appears is from the datalogger and is not relevant to your measurments. The second is the water activity value. The third is the temperature in degrees Celsius at the timeof measurement, and the fourth is the time in hours and minutes since AquaLab was turned on. If AquaLab is left on for more than 24 hours, the clock will reset to zero.

Computer Interface

# Using Hyperterminal in Windows 95 and Windows NT

To use Hyperterminal with your AquaLab, follow these steps:

- 1. Press the Start button and select Programs > Accessories > Hyperterminal and click the Hyperterminal icon.
- 2. 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.
- 3. 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.
- 4. 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: Baud rate, 300; 8 databits, no parity, 1 stop bit, and flow control set to hardware. Click OK.
- 5. 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.
- 6. When you are finished sampling, you can print the data in the terminal session, or save it.

## **Using Terminal for Windows 3.0 and 3.11**

If you are operating under Windows 3.0 or 3.11, you can use the Terminal program to log your AquaLab's data as it reads. Follow these instructions:

- 1. Connect your RS-232 cable between the AquaLab and your computer. Remember which COM port you are using.
- 2. Double-click on the Accessories icon in Program Manager. Open the icon labeled "Terminal."
- 3. Select "Communications" from the Settings menu. Set the baud rate to 300, and make sure the other settings are as follows: 8 data bits, 1 stop bit, no parity. Select the COM port that you will be using. Click OK.
- 4. Select "Receive text file" from the Transfers menu. A pop-up window will appear. Type in a name for this terminal session and click OK.
- 5. Begin sampling with your AquaLab. The data will be displayed on screen as you sample.
- 6. When you are finished, click on the "Stop" button on the bottom left corner of the screen. Terminal will save your data to the filename you indicated earlier.
- 7. If you wish, you can open this file in a spreadsheet program and analyze the data further.

## **Using AquaLink**

AquaLink is a program that can only be used in DOS. Therefore,

Computer Interface

if your computer uses Windows 95 or higher, you may not be able to use AquaLink. If this is the case, refer to the directions above for using HyperTerminal.

- 1. Install AquaLink to your hard drive or run it from the floppy disk.
- 2. Type "AquaLink" in the AquaLink directory to execute the program.
- 3. Press return and begin taking readings with AquaLab. The readings will be displayed on screen under three headings: time, a<sub>w</sub>, and temperature. The time readings are real clock time according to your computer. If your computer's clock is set to the correct time, the time readings will be accurate.
- 4. Save listed readings to disk at any time by pressing the F1 key.
- 5. When you select F1, the program will prompt you to enter a file name. If you wish to save data to a different drive or directory than the one where AquaLink is stored, specify that drive and directory with the file name. All data will be saved to this file.
- Note: If you wish to use a spreadsheet to analyze your data, you may want to use a particular extension. For example, comma delimited files must have the extension ".xls" to be read by Microsoft Excel. Check your spreadsheet user's manual for more information.

AquaLink can display up to 2000 readings before saving. However, we recommend saving more frequently to guard against data loss. If you enter the same file name at the file name prompt, AquaLink will append data to the existing file. No data will be lost.

6. To exit the program, press F2. If some of the data has not been saved, AquaLink will prompt "Data not saved. Are you sure you want to quit? (y,n)." Make sure your data has been saved and then exit.

## 9. Theory: Water Activity in Foods

Water Activity  $(a_w)$  is a measurement of water energy. It indicates the amount of "free water" in a sample. "Free water" refers to the water molecules in a sample that are not chemically or physically bound.

 $a_w$  is equivalent to Equilibrium Relative Humidity (ERH), which is the ratio of water vapor pressure above any sample to the water vapor pressure of pure water at the same temperature. Samples with no "free" water will have an  $a_w$  of 0.000, while a sample such as pure water will have an  $a_w$  of 1.000.

There are two basic types of water analysis. The first is a quantitative or volumetric analysis to determine the water content of a sample. Typically the sample is first weighed, then oven dried and reweighed to measure its total water content. But merely knowing the water content of a sample does not give an indication of the physical properties of that water. The question is not how much water is in a sample, but how much of that water is available to be used by microorganisms or other destructive agents.

The second type of water analysis, the measurement of water activity, answers this question. Water activity is an energy measurement which defines the amount of unbound water in a sample. Because microorganisms require water for survival, too much "free water" in a sample can serve as a medium for microbial reproduction, travel, and contamination. If water within a sample is held with enough force, microorganisms will not be able to exert the energy required to obtain water necessary for their subsistence. Because of this,  $a_w$  is commonly used in the evaluation of quality and safety of foods, drugs, and cosmetics.

Energy measurements are influenced by potentials that affect the binding of water. These include matric potentials, chemical bonding energies, osmotic potentials, gravitational potentials, and pressure potentials. Matric, chemical bonding, and osmotic potentials are very important in their relation to water activity in foods. By altering any of these potentials, the  $a_w$  of a sample may also be altered.

Matric potential refers to the sample's structure and its ability to hold water within that structure through capillary and surface tensions. Chemical bonding energy refers to the energy of chemical/water bonds within a sample. Osmotic potential refers to not only the potential energies exerted on water across semipermeable membranes, but also to the energy a microorganism might exert on a sample in order to take on water. If the water within a sample is tightly bound, a microorganism will find it hard to survive.

If you would like more information about water activity in foods, please contact Decagon. We have a collection of papers written about water activity in a variety of applications. We can send you a copy of the articles, or you may find what you need in the reference section of this manual.

# 10. Cleaning and Maintenance

The accuracy of your AquaLab is vitally dependent on keeping your instrument clean. Because of this, we include a prepared cleaning kit with each AquaLab to ensure that you have the right tools for effective cleaning. Dust and sampling debris can contaminate the sampling chamber and must therefore be regularly cleaned out. To clean your instrument, carefully follow these instructions.

## **Tools Needed**

- Philips screwdriver
- AquaLab Cleaning Kit

If you have used up the components of your cleaning kit, you may order another from Decagon, or you can gather the following materials:

• <u>lint-free</u> tissues (like Kimwipe<sup>®</sup>) Note: Do not use cotton swabs, since they can leave an adhesive residue that can contaminate the mirror and block surfaces.

- Tweezers
- Distilled Water

Isopropyl Alcohol

## **Cleaning the Sensor Chamber Block**

- 1. Unplug your AquaLab.
- 2. Remove the eight screws that secure the cover, located on each side of the AquaLab case. Remove the cover.



Location of the case screws

3. Using a phillips screwdriver, remove the four screws that secure the fan assembly to the block.



Location of the block screws

4. Carefully remove the fan assembly by lifting it straight up from the block. There is a small fan on the under side of this lid. The little blades break easily and AquaLab will not work properly when one breaks. Treat this fan with care.

Cleaning and Maintenance

5. AquaLab's sensor mirror is located on top of the block between two round ports. Take a blunt-headed swab from your cleaning kit or a small piece of lint-free tissue wrapped around the end of the tweezers, and dip it into the distilled water so that it is wet but not soaked. Gently wipe the mirror.



Location of the sensor mirror

Note: If the mirror will not come clean using distilled water, a small amount of 99% isopropyl or ethyl alcohol may be used followed by distilled water rinses. Be careful, if the swab is too wet with alcohol, the seal around the mirror will be broken and require repair at Decagon.

Getting the mirror clean could be likened to getting laboratory glassware clean. First, use the necessary cleaners and then follow with several rinses.

After a thorough cleaning, it is possible to check the effectiveness of the cleaning by running the instrument with the top off. It is important when you do this that you unplug the fan motor. Follow the wire from the motor on top of the block lid to the small circuit board. Disconnect this plug. Turn the knob to read. After about one minute the mirror will begin to fog. Even fog formation over the entire mirror is an indication of a good cleaning. Further cleaning of the mirror surface can be achieved by wiping the fog from the mirror as it forms with tissue as previously described. Fogging will continue until the display reads "LO" or the knob is turned to open. This procedure can be repeated as often as desired.

Taking care not to damage the fan blade by dropping it or running the fan while it is out of the block is of utmost importance. Any broken fan blade must be replaced before it will be possible to get reliable  $a_w$  readings.

- 6. Clean all surfaces of the block with the other swabs (dampened with water) from the cleaning kit, or a damp lint-free tissue, including the port holes on top.
- 7. Lift up the sensor block and gently clean the thermopile (it looks like an eye) located on the bottom of the block.



Location of AquaLab's Thermopile

Cleaning and Maintenance

- 8. Clean the lid of the chamber, especially the small optical sensor found near the fan.
- 9. Clean the sample drawer and its base. Remove any debris from the inside of AquaLab.
- Note: When replacing the sample drawer, be sure it is fit correctly into the base so that it will engage the switch on the bottom of the drawer. Caution-- if the switch lever on the drawer gets behind the switch lever on the base, it will break off the lever causing numerous problems.
- 10. Recheck for debris, then carefully insert the fan assembly into the block. Align it so that the four screw holes match.
- 11. When replacing the sensor block, make sure that the two alignment pins located on the lower section of the block are lined up with the holes on the drawer base. You may have to gently force the pins into the holes in the base.
- 12. Insert the four screws with the metal washers and O-rings into the holes. Turn them counter-clockwise until a slight drop in the screw height is felt or seen. Turn the screws clockwise until they are secure. All four screws should be tightened equally to form a seal between the fan and the block.
- Note: These screws may resist tightening. If there is any resistance <u>do not force them</u>! Turn the screw counterclockwise until a little drop is felt and then turn it clockwise to tighten. If these screws are forced it will cause stripping and they will not fully tighten. This in turn will prevent the chamber from sealing as it should

and any following readings will be inaccurate.

- 13. Adjust the screws so that there is pressure on the sample cup when the knob is turned. This ensures that the chamber is sealed. Test the tightness of the screws by placing an empty sample cup in the drawer and turning the knob to read. If the knob is too easy to turn, the screws need to be tightened.
- 14. Your instrument is equipped with O-rings under the screws, don't compress them. The O-rings help ensure proper sealing of the chamber during the read cycle without over-tightening of the screws.
- 15. Replace AquaLab's outer case. Make sure the holes in the case align with the holes in the main unit before inserting and tightening the screws.
- 16. Note: The outer case should always be in place when measuring  $a_w$ . For ease of cleaning, the cover screws could be left out or reduced to one or two.

## **Cleaning the Fan Filter**

Your AquaLab's fan filter is designed to limit the intake of debris from the environment. The accuracy of the instrument is dependent on a clean chamber. Therefore, the fan filter should be routinely checked and cleaned. You may observe that your sample temperatures rise faster than usual when your fan filter becomes increasingly clogged with dust. A clogged filter will reduce airflow inside your instrument, making it more difficult to maintain the correct temperature balance between the block and the sample. To clean the fan filter, do the following:

Cleaning and Maintenance

- 1. Unplug your instrument.
- 2. Locate the fan guard / filter at the back of the case. Pry off the top plate of the fan guard to expose the foam rubber filter.



location of the fan filter

- 3. Remove the filter and rinse it under running water, with the dusty side down to avoid forcing dust back into the filter.
- 4. Pat the filter dry with paper towels.
- 5. Replace the filter and snap the filter plate back in place.

## **Verifying Operation**

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 cleaning procedures. Check the response of your instrument by measuring the water activity of both a verification standard and a distilled water sample.

If a linear offset has occurred, refer to Chapter 4 for directions on how to correct for linear offset. If, after adjusting for linear offset your instrument is still not reading samples correctly, contact Decagon for technical support.

# **11. Repair Instructions**

If your AquaLab ever needs to be sent in for service or repair<sup>\*</sup>, call Decagon at **(509) 332-2756** or **1-800-755-2751** (US and Canada), or fax us at **(509) 332-5158**. We will ask you for your address, phone number, and serial number. For non-warranty repairs, we will also ask for a purchase order number, a repair budget, and billing address.

\*Note: If you purchased your AquaLab from one of our international distributors, please contact them before contacting Decagon. They may be able to provide you with local service and help you remedy the problem.

## **Shipping Directions**

When you ship your instrument back to us, please include with it a document with the complete shipping address, name and department of the person responsible for the instrument, and (most importantly) a description of the problem. This information will better help our technicians and our shipping department to expedite repair on your instrument and ship it back to you in good time.

Following are steps that will help in safely shipping your instrument back to us:

1. If possible, ship your AquaLab back to us in its original cardboard box with foam inserts. 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. Put your instrument in a plastic bag to avoid disfiguring marks from the packaging.
- 3. Don't ship your AquaLab to us with the power cord; we have plenty here to use with your instrument.
- 4. If you aren't using the foam inserts, pack the box moderately tight with packing material, like styrofoam peanuts.
- 5. Tape the box in both directions so it cannot be broken open in shipment.
- 6. Include necessary paperwork so your repair can be processed quickly. This should include your name, address, serial number, phone and fax numbers, purchase order, and a description of the problem.

#### Ship to:

Decagon Devices Inc. ATTN: Repair Department 950 NE Nelson Court Pullman, WA 99163

## **Repair Costs**

Manufacturer's defects and instruments within the one-year warranty will be repaired at no cost. For non-warranty repairs, costs for parts, labor, and shipping will be billed to you. We have a \$50 minimum repair charge. An extra fee will be charged for rush work. Decagon will provide an estimated repair cost, if

Repair Instructions

requested.

## **Loaner Service**

We have loaner instruments that can be provided while your instrument is being serviced. There is, however, a limited number of loaner instruments. They are granted on a "firstcome-first-served" basis. This service is in place to help you if your AquaLab needs service during critical operations.

## 12. Troubleshooting

AquaLab is a high performance instrument, designed to have low maintenance and few problems if used with care. Unfortunately, sometimes even the best operators using the best instruments encounter technical difficulties. Here is a list of some problems you may occur, as well as an explanation of the error messages your instrument may give you. If you have encountered a problem that isn't addressed here, or if these remedies still don't resolve your problem, contact Decagon at 1-800-755-2751 or (509) 332-2756 (for those not in the US or Canada).

## **Problems and Solutions**

<u>PROBLEM</u>: AquaLab won't turn on.

SOLUTION:

- Check to make sure your power cord is securely attached the back of the instrument, and into the power outlet.
- A power surge may have caused a fuse to blow. To change the fuses, follow these instructions:
  - 1. Unplug the power cord from the wall and the instrument.

- 2. 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.
- 3. Pull the broken fuse(s) out and replace with a 1 Amp 250V fast blow fuse.

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

- 4. Replace the fuse-holder and push it into the fuse-well until the release tab snaps in place.
- 5. 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.

#### **PROBLEM:**

# The instrument's display doesn't go to "zeroes" a short while after turning it on.

#### SOLUTION:

• Some of the AquaLab's components may be damaged. The "garbage" that shows on the display when AquaLab is first turned on should disappear after about thirty seconds. If it doesn't, some of the internal components may be damaged, and you may have to return your AquaLab for repair. See Chapter 11 for repair instructions.

#### <u>PROBLEM</u>: **Readings are slow or inconsistent.**

#### SOLUTION:

- **The sample chamber may be dirty.** Refer to Chapter 10 of the manual for directions on cleaning the sample chamber.
- Some foods absorb or desorb moisture so slowly that measurements take longer than usual, and nothing can be done to speed up the process. Refer to Chapter 5 for further explanation.
- Your sample may contain propylene glycol. This compound is known to cause unstable readings, because it condenses on the surface of the chilled mirror and alters readings. Please refer to the propylene glycol section in Chapter 5 for hints on reducing difficulties with measuring samples with propylene glycol. If you have further questions regarding the measurement of propylene glycol, contact Decagon.

*Note:* As yet, propylene glycol is the only volatile know to act unpredictably. Glycerol and other alcohols used to flavor foods can usually be measured without problems; however, some aromatics may also caused unstable readings. If this seems to be a problem for you, contact Decagon.

• A fan blade may be broken inside the block. If even salt standards take a long time to read, and the sample chamber is clean, you may have a broken fan blade. This is especially

Troubleshooting

likely if you have just cleaned the chamber. If you suspect this may have happened, contact Decagon for details on replacement.

• The mirror or cooler may have become detached from the sample block. Contact Decagon before sending it in for repair.

#### **PROBLEM:**

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

#### SOLUTION:

- The thermopile in your chamber, which measures sample temperature, may have become contaminated. Refer to Chapter 10 for directions on cleaning.
- If you weren't using Decagon's performance verification standards, high readings may indicate that the salt solution you are using is not in equilibrium.

#### **PROBLEM:**

When measuring samples, AquaLab displays a "LO" message.

#### SOLUTION:

- **The sample you are trying to read may be too dry** for the AquaLab to make an accurate measurement.
- The mirror may be dirty. Refer to Chapter 10 for instruc-

tions on cleaning.

- Your AquaLab's optical sensor may not be detecting the formation of of dew on the mirror. Refer to Chapter 10 for instructions on cleaning.
- One of the chamber components may be damaged. Contact Decagon for details.

#### **PROBLEM:**

# The potentiometer twists all the way around, making it difficult or impossible to adjust for a linear offset.

#### **SOLUTION:**

• **Do not force the potentiometer.** It was designed to only rotate one full turn. If you can twist it all the way around, it is broken and your AquaLab must be returned for repair. See Chapter 11 for repair instructions.

#### **PROBLEM:**

# While using AquaLab at elevated temperatures the instrument displays "zeroes" or "garbage" on the screen.

#### **SOLUTION:**

The water bath or environmental temperature is above 40° C. Cool the water bath and lower the environmental temperature. AquaLab will not be damaged by these higher temperatures. If the problem persists after cooling, carefully clean the block. If this doesn't solve the problem, please contact Decagon for technical assistance at 1-800-755-2751 or aqualab@decagon.com.

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## **AquaLab** *Further Reading*

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# **Appendix A: Salt Solutions**

## **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, as 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 intend on using this solution over a long term period, make sure to seal the solution well to prevent losses from evaporation. Below is a table of saturated salt solutions and their respective water activities at differ-

#### AquaLab Appendix A

ÿ		
Saturated Salt Solution	$\mathrm{a_w}$ at 20° C	$\mathrm{A_w}$ at 25° C
Lithium Chloride	.113 ± .003	.113 ± .003
Magnesium Chloride	.331 ± .002	.328 ± .002
Potassium Carbonate	.432 ± .003	.432 ± .004
Magnesium Nitrate	.544 ± .002	.529 ± .002
Sodium Chloride	.755 ± .001	.753 ± .001
Potassium Chloride	.851 ± .003	.843 ± .003
Potassium Sulfate	.976 ± .005	.973 ± .005

## ent temperatures. Please note that these values are

**Table 1: Water Activity Table for Selected Salt Solutions** 

Adapted from Greenspan (1977). Numbers rounded to nearest thousandth.

based on averaged published data. Saturated salt solutions are temperature-sensitive and their values are not as accurate as the verification standards offered by Decagon.

# Index

## **Symbols**

"**LO**" 43

### Α

appendix A 72

## С

case fan 32 cautions 27 checking calibration 46 chilled products 32 chilled-mirror technique 5 cleaning 40, 42, 45 fan filter 45 sensor block 41 coated samples speeding read time for 23 components 9 cotton swabs 40 customer service 1 CX-2T 29

## D

dehydrated samples 24 dew point 29 downloading using Terminal for Windows 35 dried samples speeding read time for 23 dry samples 7

### E

e-mail address 2 error messages 51

## F

fan blade 41, 43 fan filter 32 fax number 2 features 8

## L

linear offset 55 causes for 14 definition 14 how to adjust for 18, 18–20 when to verify for 18 loaners 50 location 9 low water activity 25, 28

## Μ

maintenance 40
manual 1
microbial growth
related to water activity 38

## 0

**offset** 32 **O-rings** 44, 45

### P

pharmaceuticals 69 powdery samples 28 preparing salt solutions 72 propylene glycol 6, 24, 53

#### R

read time 6 long read time 6, 22, 53 readings cautions 27 how Aqualab takes 26, 27 taking readings 26 references 56 baked goods and cereals 67 beverages, soups, sauces, preserves 68 dairy products 65 food quality and safety 59 meat and seafood 63 microbiology 60 pharmaceuticals 69 water activity theory 56 relative humidity 38 repair costs 49 repair instructions 48

## S

salt solutions uncertainty using saturated solutions 15 salt standards. See verification standards sample cup 45 sample cups filling level 22 sealing 22 sample drawer 44 sample preparation 21 sample temperature and CX2-T 31 saturated salts 72 seller's liability 3 shipping 48

## Т

technical assistance 55 technical support 1 telephone number 1 temperature hot samples 27 of instrument 27 samples not at room temp. 25 temperature control 6, 29 theory water activity 56 troubleshooting 51

## V

verification standards 14 equations 16 table of values 17 voltage settings 12

#### W

warm-up 13 warranty 3 water activity Aqualab and 5 definition 5 low 28 water bath 29