

Vapor Sorption Analyzer



User's Manual

Version 1

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1. Introduction

The AquaLab Vapor Sorption Analyzer (VSA) is an automatic isotherm generator from the world leaders in water activity measurement. The VSA is the only automatic isotherm generator that can generate isotherms using both the Dynamic Vapor Sorption (DVS) method, which generates equilibrium isotherms, and the Dynamic Dewpoint Isotherm (DDI) method, which generates dynamic isotherms. By combining both methods in one instrument, the VSA makes it possible to investigate both dynamic matrix changes due to water sorption and the kinetics of those changes. This manual is for VSA operation and to be informative regarding the capabilities of your VSA.

About this Manual

Included in this manual are instructions for setting up your VSA which includes setting up an isotherm test, running a test, collecting data, and analyzing data. Please read the manual before operating the VSA to ensure your instrument performs to its full potential.

Customer Support

For service or assistance, there are several ways to contact us:

- **E-mail:** support@aqualab.com. Please include your serial number, a contact name, phone number and address with a description of your problem.
- **Phone/Fax:**
 - **Telephone:** (509) 332-5601
 - **Fax:** (509) 332-5158

Warranty

The VSA has a one year warranty on parts and labor. Your warranty is automatically validated upon receipt of the instrument. Decagon will contact you within the first 90 days of your purchase to see how the VSA is working for you.

Note to Our VSA Users

This manual is written to aid the end user in understanding the basic concepts of moisture sorption isotherms, enabling them to use our instruments with confidence. Every effort has been made to ensure 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. The seller's liability for defective parts shall in no event exceed the furnishing of replacement parts F.O.B. from 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 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 the VSA

The VSA Isotherm Generator is an automatic moisture sorption isotherm generator that can generate both dynamic and equilibrium moisture sorption isotherms.

Moisture Sorption Isotherms

The relationship between water activity (a_w) and moisture content at a given temperature is called the moisture sorption isotherm. This relationship is complex and unique for each product due to different interactions (colligative, capillary, and surface effects) between the water and the solid components at different moisture contents. An increase in a_w is almost always accompanied by an increase in water content, but in a non-linear fashion. Moisture sorption isotherms are sigmoidal in shape for most foods, although foods that contain large amounts of sugar or small soluble molecules have a J-type isotherm curve shape.

Isotherms provide information about product quality and safety. A few uses for isotherms include:

1. Monolayer moisture content determination.
2. Determine critical water activity or moisture content.
3. Limits for crispness, hardness, and flow properties.
4. Optimize moisture contents at a safe water activity that maximizes moisture and avoids over drying.
5. Determine shelf-life and storage stability of a product.
6. Predict packaging requirements based on sorption properties of a product.
7. Determine kinetics of sorption and water vapor diffusion coefficients

8. Determine the equilibrium water activity of a mixture of dry ingredients.
9. Determine the degree of crystallinity of powders.
10. Determine the level of amorphous material in a product.
11. Determine critical water activities for phase transitions.
12. Determine the relationship between water activity and glass transition temperature.
13. Determine the relationship between water activity and crystallization.
14. Determine hysteresis levels for a product.
15. Determine the moisture sensitivity of a product.
16. Determine the equilibrium moisture content at a given water activity.
17. Allow rapid moisture content determination from water activity analysis through an isotherm curve.

Measurement Method

The VSA is the only automatic isotherm generator that can create isotherms using both dynamic and static methods. To produce dynamic isotherms, the VSA uses the Dynamic Dewpoint Isotherm (DDI) method. To produce static or equilibrium isotherms, the VSA uses a controlled humidity and balance system commonly referred to as Dynamic Vapor Sorption (DVS).

The DDI method for dynamic isotherms is a water activity and gravimetric analysis method that controls neither water content nor water activity, but dries or wets the sample and measures water activity and water content during the wetting or drying process. Water content is determined by weighing the sample using a high precision magnetic force balance. Water activity is

determined using Decagon's patented chilled-mirror dewpoint sensor. Drying of the sample is imposed by flowing dry air from a desiccant tube across the sample. Wetting of the sample is imposed by saturating the air with water before it enters the chamber and flows across the sample. The water reservoir is mounted to the temperature controlled lower block to ensure humidity saturation and minimize temperature fluctuation.

The DVS method for static or equilibrium isotherms consists of tracking sample weight change as the sample is exposed to different controlled humidities. The sample is held at each humidity for a preset time interval or until a steady state weight change is achieved, the goal being to achieve equilibrium between the sample water activity and the controlled humidity. Typically, several humidity levels are chosen and preset during the setup process. The instrument then tracks equilibrium progress at each humidity level and automatically steps to the next humidity when equilibrium requirements are achieved. Weight change vs. time data is recorded allowing for determining kinetics of sorption for each humidity level. Using less stringent equilibrium settings for weight change or setting a reduced time interval at each step will speed up the isotherm test, but may give non-equilibrium results. In addition, setting smaller steps in humidity will increase the data resolution of the isotherm, but at the expense of much longer test times.

The VSA is a stand-alone instrument with an integrated pump system that eliminates the need for an external gas cylinder, however an external gas source can be used if desired. The instrument consists of a case which houses the power supply, air pump, balance, temperature controlled sample chamber, sensor block, sensor and temperature control electronics, water

reservoir, and desiccant supply. Setup is as simple as plugging the instrument in, installing the desiccant tubes, and filling the water reservoir. Test parameters can be set using an onboard interface or using a connected computer and VSA software package.

Specifications

Water Activity (Relative Humidity) Range: 0.03 to 0.95 a_w (3% to 95% RH) for 15 to 50°C , 0.03 to 0.90 a_w for 50 to 60°C

Water Activity (Relative Humidity) Accuracy: $\pm 0.005 a_w$ (0.5 %RH) (for volatiles setting, $\pm 0.02 a_w$)

Water Activity (Relative Humidity) Repeatability: $\pm 0.003 a_w$ (0.3% RH) (for volatiles setting, $\pm 0.02 a_w$)

Sample Weight Range: 500-5000 mg

Temperature Control Range: 15 to 60°C at STP. Instrument Control (sample temperature may vary)

Temperature Operating Range: 0 to 60°C

Humidity Operating Range: 10-90% non-condensing

Universal Power: 110 V to 220 V AC. 50/60Hz

Size (Footprint): 38.1 x 26.7 x 30.5cm (15 x 10.5 x 12in. (L x W x H))

Weight: 33 pounds

Weight Accuracy: $\pm 0.1\text{mg}$

3. Theory

Water profoundly influences product attributes such as quality and safety. To completely understand water relations in a product requires an understanding of the amount of water (moisture content) that can be held at a given energy state (water activity). Moisture sorption isotherms describe the relationship between water activity and moisture content at a constant temperature. The nature of this relationship depends on the interaction between water and other ingredients. The amount of water vapor that can be absorbed by a product depends on its chemical composition, physical-chemical state, and physical structure. Consequently, the isotherm shape is unique to each product type due to differences in capillary, surface, and colligative effects (Figure 1). Products that lie in the low water activity portion of the isotherm are often referred to as dry, those in the range of $0.60 a_w$ to $0.90 a_w$ are intermediate moisture products, and those having water activities higher than 0.90 are high water activity products.

For ease of interpretation, isotherms are often identified by Brunauer classifications (Brunauer, 1945). Most food and pharmaceutical products fall under type I, II, or III. Type I isotherms are typical of very hygroscopic materials. Type II (sigmoidal) isotherms are typical for intermediate moisture products. Type III (J-shaped) isotherms are typical for crystalline and coated materials. These general classifications proved useful when conducting isotherms on every product was not feasible due to time and labor constraints. However, with automation and improved speed, isotherms can easily be conducted on any product and the uniqueness of each isotherm

often proves more valuable than placing them in a common classification.

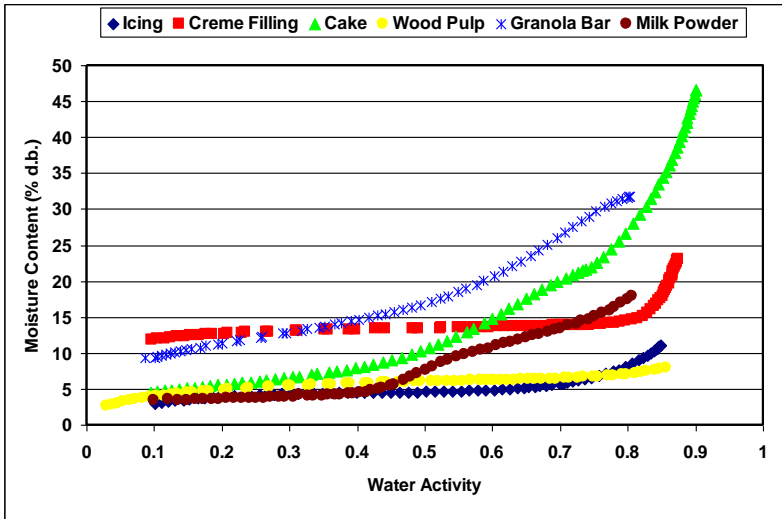


Figure 1. Moisture adsorption isotherms for icing (♦), crème filling (■), cake (▲), wood pulp (◆), granola bar (*), and milk powder (●) showing that the isotherm shape is unique to the product.

Moisture Sorption Isotherm Methods

Constructing an isotherm consists of collecting water activity and moisture content data at various points along the water activity range. The range of water activities used will depend on the situation, but normally run from 0.10 a_w up to 0.90 a_w . This can be accomplished by controlling water activity levels using saturated salt slurries or mechanical humidifiers and determining equilibrium moisture content at each water activity level. Equilibrium is assumed when the weight of the sample stops changing. This process is often accomplished using sealed chambers such as desiccators and the equilibration process can

take weeks. Additional challenges with this manual method include difficulty tracking the weight of the samples, removing the samples for weighing exposes them to ambient humidity, and at high humidities, there is the possibility of mold growth. Decagon's VSA Isotherm Generator uses the DVS method described above to speed up and automate the construction of equilibrium isotherms, eliminating the challenges of manual determination. In addition to equilibrium moisture contents at a given water activity, equilibrium isotherms provide information about kinetics of sorption and water vapor diffusion properties.

Moisture sorption isotherms can also be determined using dynamic methods such as the DDI method used by the VSA. The DDI method directly measures water activity while gravimetrically tracking weight, so there is no dependence on equilibration to known water activity levels to determine water activity. Adsorption occurs as saturated wet air is passed over the sample. Desorption is accomplished as desiccated air is passed over the sample. After a short period of time, airflow is stopped and a snapshot of the sorption process is taken by directly measuring the water activity and weight. The advantages of this method are increased analysis speed since the sample does not have to wait for equilibration to a known water activity and an unmatched level of resolution. This makes it possible for dynamic isotherms to produce high resolution isotherms in a matter of days instead of the weeks it would take to make a comparable isotherm using equilibrium techniques. The high resolution of dynamic isotherms makes them valuable for observing sudden changes in sorption properties associated with matrix changes such as glass transition.

The dynamic nature of the DDI method means that moisture contents may or may not be at equilibrium and it is possible for there to be differences between dynamic and equilibrium isotherms. For samples with fast vapor diffusion, penetration by water vapor into the whole sample is rapid and isotherms using the DDI method for these types of products will be comparable equilibrium methods. However, for samples with slow diffusion rates, moisture movement through the sample is slow and complete diffusion of moisture into and out of the sample may be slow enough to give the appearance of vapor equilibrium in the headspace during water activity analysis. In reality, the moisture has not had time to be completely absorbed by the sample. Isotherms for these types of samples developed using the DDI method may have lower moisture contents during adsorption and higher moisture contents during desorption than equilibrium isotherms, resulting in higher levels of apparent hysteresis.

Combining DVS and DDI in one instrument

A unique and important feature of the AquaLab VSA is that it can generate both dynamic and equilibrium isotherms. As explained above, both DVS and DDI methods have advantages and disadvantages. While the data they generate will agree in some cases, it is the uniqueness of the results from each method that gives them value. There is information that can only be obtained from dynamic isotherms such as critical water activities for glass transition. Similarly, there is information that can only be obtained by equilibrium isotherms such as kinetics of sorption. Therefore, to provide the most information about the moisture characteristics of a material, both types of isotherms are needed. The VSA can provide both DVS and DDI isotherms and even run them both on one sample.

Hysteresis

Figure 2 shows two isotherms, one obtained by wetting a sample from complete dryness and the other obtained by drying a sample from saturation. The arrows show the direction of the process. The water content at each water activity is higher during desorption (drying from high water content) than adsorption (wetting from low water content). This phenomenon is called hysteresis. The curves in Fig. 2 represent limits or bounding isotherms since they begin at water activities near zero and one. If a drying process reduces the water activity of a sample only part way to dryness, and the sample is then re-wet, it follows a path between the wetting and drying boundary curves, as shown in Fig. 3. These curves are called scanning curves, and there can be an infinite number of them depending on where drying stops and starts.

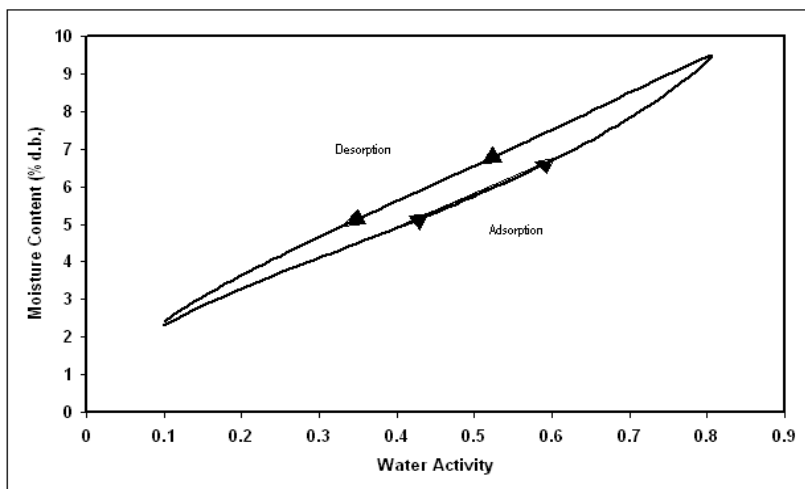


Figure 2. Full isotherm showing hysteresis.

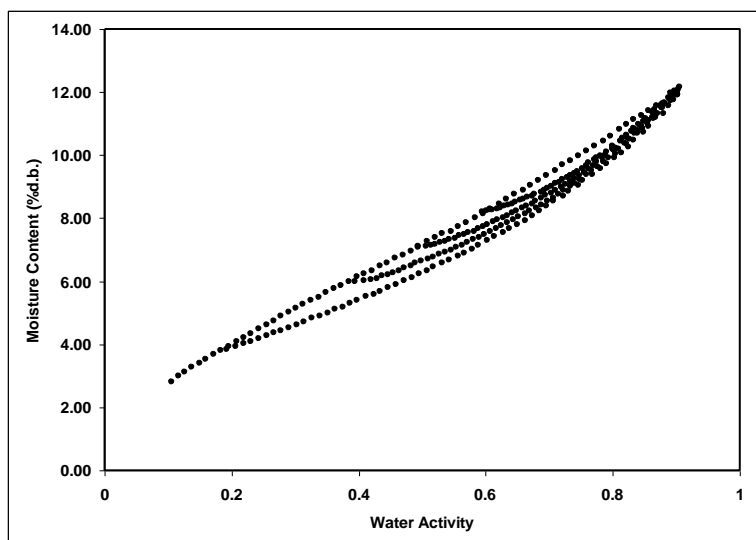


Figure 3. Scanning adsorption curves resulting from drying to different water activities.

These observations help clarify the point that an isotherm is not a single valued function. The water content for any given water activity value depends on the wetting and drying history of the sample.

It is possible to obtain isotherm data which appear to show hysteresis by failing to allow a sample to equilibrate at each step, or by inducing changes in the water binding properties of the matrix by wetting or drying. We prefer to treat these cases separately, and reserve the term hysteresis for situations where equilibrium is reached, but water contents of wetted and dried samples still differ because of their history.

Several plausible models exist for hysteresis. Theories are based on; capillary condensation of porous solids, phase

changes of non-porous solids, structural changes within a solid matrix, and supersaturation of some solutes during desorption. Depending on the composition of the sample, these theories explain why the water content of a desorption process is greater than that for a wetting process.

1. Capillary condensation of porous solids theory is illustrated by the 'ink bottle' model, in which pores and capillaries fill and empty differently. Such a pore fills when the water activity corresponding to the energy state of the larger radius is exceeded, but will empty only when the water activity drops below the energy state of the narrow neck radius.
2. A phase change of non-porous solids is illustrated by the fact that desorption from rubbery state can reach equilibrium faster due to increased molecular mobility, while adsorption into a glassy material can be slow due to restrictions in molecular mobility.
3. Structural changes within a solid matrix occur when the material swells and polar sites once obscured are now exposed to 'bind' with water. For example, hydrated protein contains many sites for water 'binding' before desorption while dehydrated protein have limited polar sites for water 'binding' prior to adsorption.
4. Some solutes may supersaturate below their crystallization water activity (non-equilibrium condition) and thus, hold more water as a_w is lowered. Foods with high sugar content frequently exhibit this phenomenon.

Non-Equilibrium

If diffusion of water into (adsorption) or out of (desorption) a material is slow and sufficient time is not allowed for complete diffusion, there will be a large amount of apparent hysteresis that could be reduced by allowing sample equilibration.

Matrix Changes

Figure 4 shows three different isotherm curves of spray-dried milk powder with different maximum water activities and different sorption histories. The boundary isotherm with a 0.80 a_w maximum experienced a phase change at 0.43 a_w , indicated by a sharp inflection point in the curve. The desorption curve for this isotherm appears to show hysteresis, especially below a water activity of 0.60. However, an isotherm, run on a sample wetted to a maximum water activity below the phase change, exhibits very little hysteresis. The lack of hysteresis in this isotherm indicates that the matrix changes that occur at 0.43 a_w are completely responsible for the apparent hysteresis.

Water in a sample is bound to particle surfaces by various bonding mechanisms. When the configuration of particle surfaces changes due to a phase change, binding sites change and the amount of water which can be bound at a given energy of water also changes. An isotherm curve of the phase changed sample does not show further phase transitions since simply drying the sample does not return it to an amorphous state.

These matrix changes represent a true physical change in the material. They are not reversible by drying, no matter how many drying cycles occur. Differences between the adsorption and

desorption curves in the initial isotherm is not true hysteresis since the sample matrix has experienced a physical change. Differences between sorption curves in the subsequent isotherms represent true hysteresis.

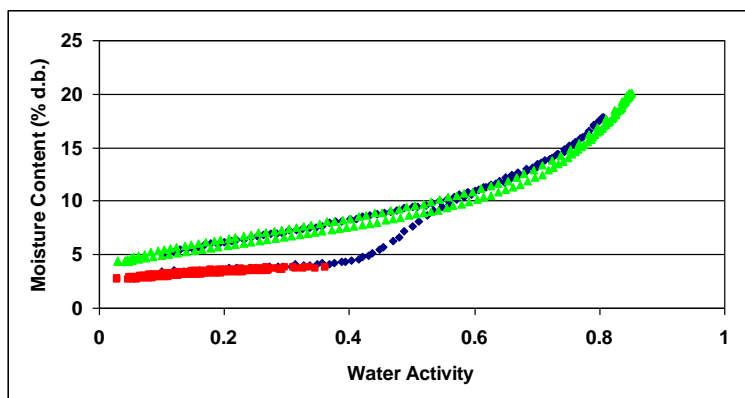


Figure 4. Moisture sorption isotherms for spray-dried milk powder showing a change in hysteresis due to a phase change at 0.43 a_w . When the isotherm is ended prior to the phase change at 0.43 a_w (■), there is little hysteresis. When the isotherm is ended above the phase change (◆), there is apparent, but not actual hysteresis due to the phase change. Subsequent isotherms run on the sample after experiencing a phase transition (▲) do not show an inflection point, are repeatable, and exhibit only small levels of hysteresis.

Working Isotherms

Full boundary isotherms give information about the physical characteristics of a product, show hysteresis, and are important in establishing critical control points, but in many cases a working isotherm will prove very useful. A working isotherm

shows how a product adsorbs and desorbs water from its current or typical condition. To create a working isotherm, the product is processed as usual, and then a scanning curve is created by wetting one sample from that point and drying a different sample from that same point. Figure 5 shows a working isotherm for wood pulp superimposed over its full boundary isotherm.

The scanning curve the product will initially follow depends on whether the product was previously wetted or dried to its current state. If a product was wetted to a certain water activity and then is dried back down, there will be an initial transition period as the product moves from the adsorption curve to the desorption curve. The same is true for a product that was previously dried and then wetted up. There will be an initial transition period as the product moves from the desorption curve to the adsorption curve. This transition region can be observed at any point on the isotherm if the direction of the sorption is changed and the product exhibits hysteresis as can be observed in Figure 5.

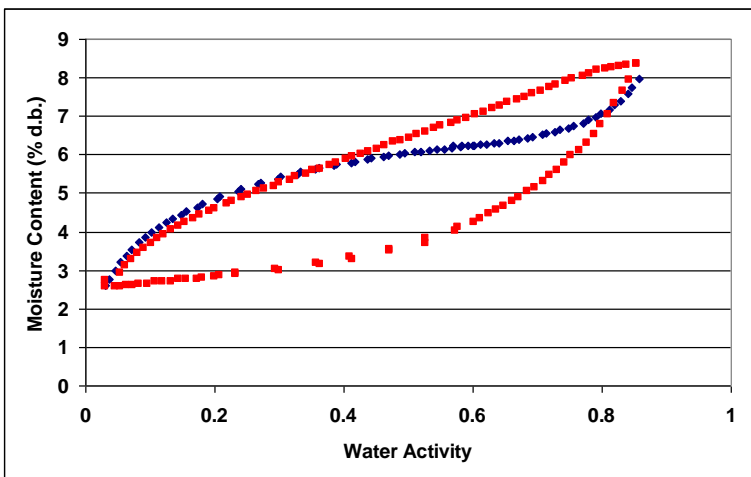


Figure 5. A working isotherm (♦) superimposed over a full isotherm (■) for wood pulp. There is a transition from the native starting point (0.60 a_w) on the working isotherm in both adsorption and desorption until the curves meet the bounding adsorption and desorption curves of the full isotherm at which point the working isotherm follows the full isotherm.

NOTE: Please refer to Chapter 8 for information about running a working Isotherm using the VSA.

Isotherm Models

Several different isotherm models have been proposed and compared in the literature. These models are necessary to predict the moisture content at a given water activity and are used to evaluate thermodynamic functions of water in foods. They are also necessary when using isotherm methods with low data resolution to interpolate between isotherm data points. While there are 270 proposed isotherm models, the most commonly used models are the GAB and BET. Since the BET model is only applicable up to 0.50 a_w , the GAB model is widely accepted as the most useful for characterizing isotherms across the entire water activity range. Its coefficients also have theoretical physical meaning such as providing monolayer moisture content. A new empirical model called the Double Log Polynomial (DLP) or Chi plot (Condon, 2006) has proven to be even better than the GAB at characterizing complex isotherms. The model equations are shown below.

$$\text{BET} \quad m = \frac{a_w m_o c}{(1 - a_w)[1 + a_w(c - 1)]} \quad (2)$$

Where m is the moisture in g/100 solids or g/g solids at water activity a_w and m_o is the monolayer value in the same units. The constant c is calculated by: $c = \exp\left(\frac{Q_s}{RT}\right)$ Where Q_s is the surface interaction energy in J/mole, R is the gas constant (8.314 J/mol K) and T (K) is the temperature.

$$\text{GAB} \quad m = \frac{m_o k_b c a_w}{(1 - k_b c)(1 - k_b a_w + k_b c a_w)} \quad (3)$$

Where m is the moisture in g/100 solids or g/g solids, k_b is a constant in the range of 0.70 to 1 and c is a constant in the range of 1 to 2000. In addition, m_o is the monolayer moisture content in the same units as m and a_w is the water activity at moisture m .

$$\text{DLP} \quad m = b_3 \chi^3 + b_2 \chi^2 + b_1 \chi + b_0 \quad (4)$$

Where m is the moisture in g/100 solids or g/g solids, $\chi = \ln[-\ln(a_w)]$ and $b_0 - b_3$ are empirical constants.

Uses for Moisture Sorption Isotherms

Moisture sorption isotherms provide valuable information. For anyone who dries or wets their product, the sorption isotherm serves as a drying and wetting curve and provides information about the moisture content of a product when dried or wetted to a specific water activity. It can be used to assist in process control by determining drying rates and optimal endpoints. If a product exhibits hysteresis, the isotherm will show what impact that will have on the moisture content after drying to a given water activity.

3. Theory

An additional function of the isotherm is moisture content prediction. Although water activity is a much better predictor of safety and quality than moisture content, there are times when it is necessary to know both water activity and moisture content as well as the relationship between the two parameters for a given product. Water content measurements can be inaccurate, time-consuming and require a precision balance. As an alternative to moisture content measurement methods, the sorption isotherm can be used to determine moisture content based on water activity, usually with better accuracy than actually running a moisture content analysis and in much less time.

Isotherms can be used to determine the effect of temperature on a product's water activity and moisture content. Isotherms conducted at several different temperatures will show the temperature at which a product, in a sealed package (at constant moisture content), will be at unstable water activity levels.

Isotherms can be very valuable for formulation and product development. By comparing the isotherms of different formulations, it is possible to determine if a product can be adjusted to allow higher moisture content at a given water activity or a lower water activity at a given moisture content. The result can be a moister product that is still shelf stable. For those producing multi-component products, it is possible using the isotherms of the two components to determine what the final water activity will be of the mixture without actually making the product. For dried products, the isotherm will predict the moisture content of the product when it is dried to a shelf stable water activity level.

Finally, sorption isotherms are valuable for shelf life prediction. A product's isotherm can be used to determine package requirements depending on the products sensitivity to moisture. It can also be used to determine the monolayer moisture content, which represents a products most stable state. The shape of the isotherm can provide information about the level of amorphous to crystalline material in a product. Changes in the slope of the isotherm indicate phase transitions and can provide information about critical water activities for maintaining texture properties and preventing caking and clumping. The water activity value where the glass transition temperature equals storage temperature or the crystallization temperature equals storage temperature can also be determined.

4. Getting Started

Components of VSA

The VSA requires a computer and software to generate and analyze isotherm data. Once a test has begun, the computer can be disconnected and re-connected without losing any data. Your VSA is shipped with the following items:

- The VSA Isotherm Generator Main Unit
- The VSA Software Installation Disc
- Operators Manual
- Calibration Certificate / MSDS Certificates
- Power Cord
- USB Interface Cable
- 2 Stainless Steel Sample Cups
- 2 Refillable Desiccant Tubes
- 1 Decagon Cleaning Kit
- 1 Water Bottle
- 3 Vials each of the following verification solution:

0.920 a_w 2.33 molal NaCl

0.760 a_w 6.0 molal NaCl

0.500 a_w 8.57 molal LiCl

0.250 a_w 13.41 molal LiCl

- 1 2g NIST traceable weight.

NOTE: Please keep the box your instrument arrives in. If it ever needs to be returned, it must be shipped in the original packaging.

Instrument Setup

1. Place AquaLab VSA on a flat, stable surface. Use the bubble level (Figure 1) and the adjustable feet to level the instrument. The small rubber feet on the back of the lower plate are to prevent tip-over and do not need to be touching the table.
2. Use AquaLab VSA where environmental temperature and humidity remain stable.
3. Plug in AquaLab VSA and turn on the power (switch in back). For best results, let the instrument warm-up for 2 hours before use.

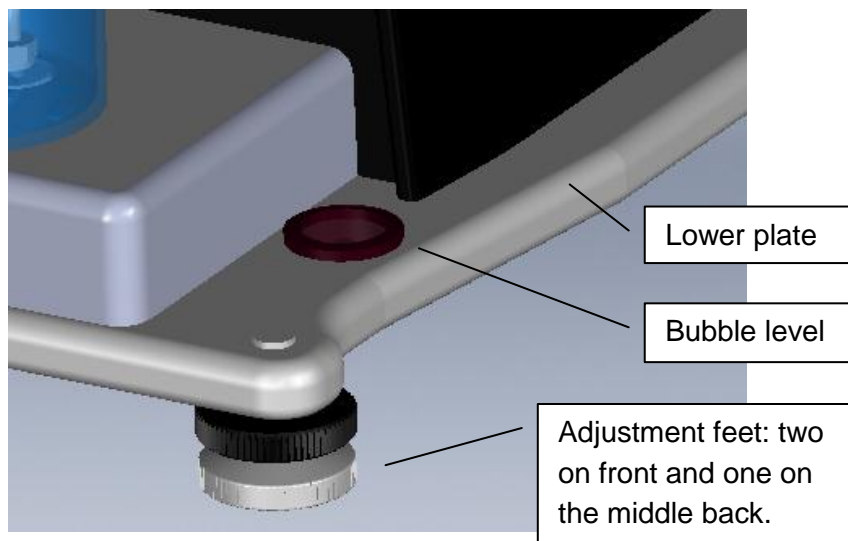


Figure 1: Adjustment and bubble level.

4. Install the 2 desiccant tubes in the front of the instrument.
 - Desiccant tubes are installed as shown in *Figure 2: Desiccant installation*. Before insertion make sure that the desiccant tube lid is firmly sealed onto the desiccant tube, remove black vinyl covers from air fittings. Lift the desiccant tube into the area above the desiccant receiver and then insert the fittings into the mating holes while rocking the tube slightly. Be sure to place the black fitting into the corresponding black hole. Once in place, the desiccant tube is sealed for flow purposes even if it may feel loose.

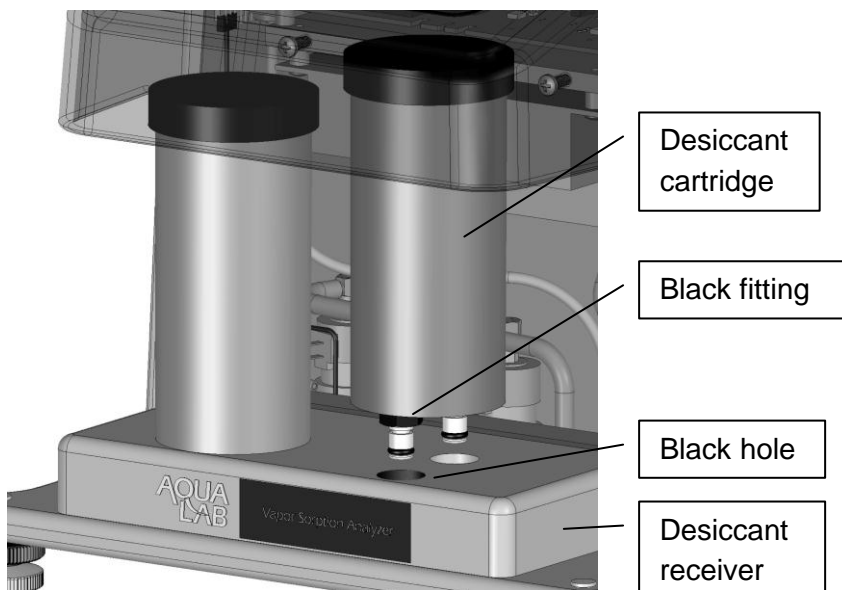


Figure 2: Desiccant installation

5. Install the air cylinder and weighing pan. With the lid open, inset the air cylinder and weighing pan into the testing block as shown in Figure 3. These parts remain in the instrument continually and are only removed for maintenance. A sample cup is not inserted unless a test is going to be run. The air cylinder is placed into the bore of the base block. It should slide into the block easily and settle at the bottom of the bore. The weight pan connects to the scale and holds the sample cup. For best results, verify cleanliness of all parts and avoid dropping the parts as damaged parts may not slide properly and affect performance.

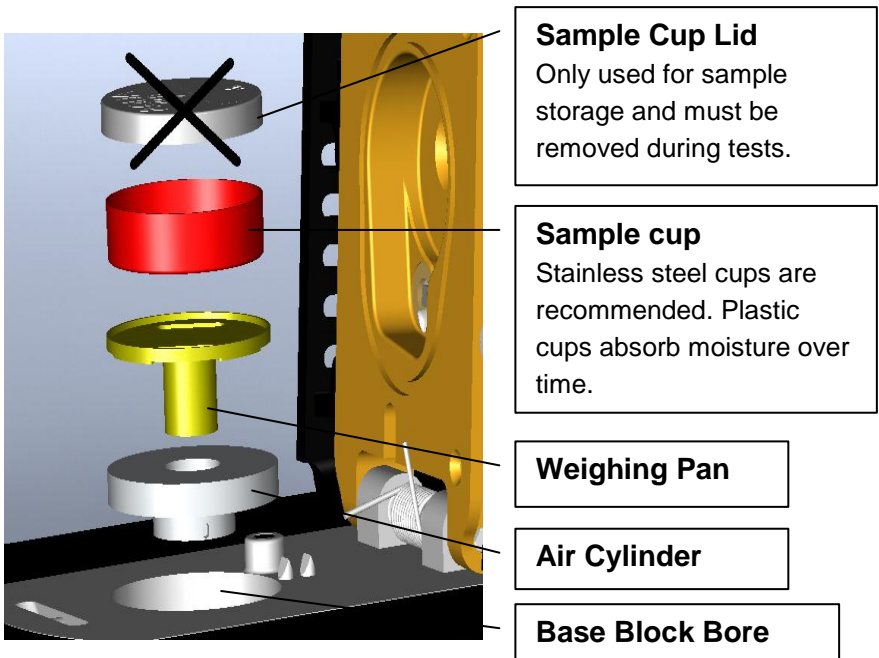




Figure 3: Sample cup and lower block assembly

6. Verify shipping did not damage the scale performance and test scale stability:
 1. Turn on the VSA and wait for it to warm up.
Note: The scale takes about 2 hours after being turned on to come to full equilibrium.
 2. Go to the Configuration menu and navigate to the diagnostics screen and press .
 3. Scroll down and choose diagnostics by pressing .
 4. Scroll down to the screen that shows the weight. Readings should vary no more than 0.1 mg.
 5. Lightly tap the table with one finger. If the readings vary more than 0.2 mg after tapping, the table holding the VSA is probably too unstable for accurate weight readings during a test and will need to be made more rigid or another location found for this equipment. Contact your AquaLab distributor if the scale readings are unstable after placing the unit on a stable structure.
7. Fill the water tank. Remove the black plug on the clear plastic tank fill extension located on the back right of the VSA. (see *Figure 4: Tank fill extension*). Fill the tank with steam distilled water until full to top. Reinstall the black plug once finished.

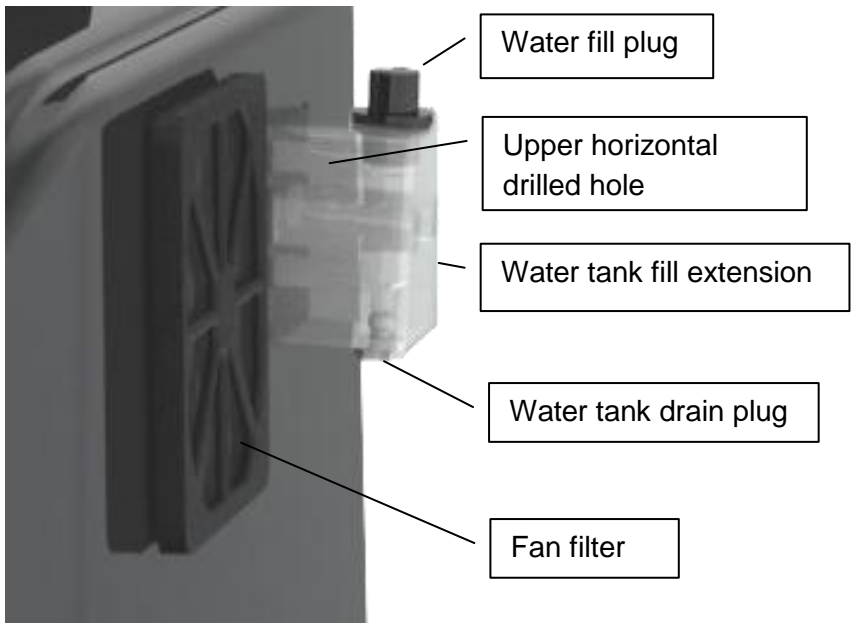


Figure 4: Tank fill extension

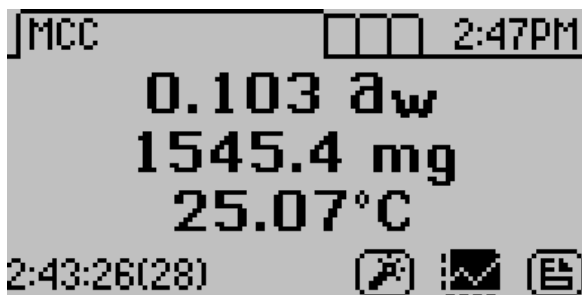
8. Figure 4 shows the location of a filter that will need to be removed from time to time when dust builds up on it. The housing around the filter is a snap on housing that is easily removed.
9. To remove the water from the water tank, the water tank drain plug is used.

5. User Interface and Menus

The AquaLab VSA is a stand-alone instrument that uses arrows and 3 buttons to navigate menus and begin tests. At the top of the display screen there are 4 tabs: Isotherm, Measurement, Configuration, and Data. These tabs indicate the 4 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.

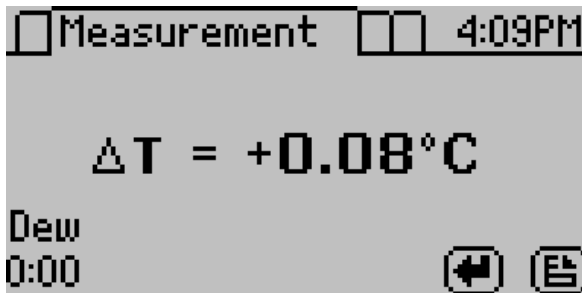
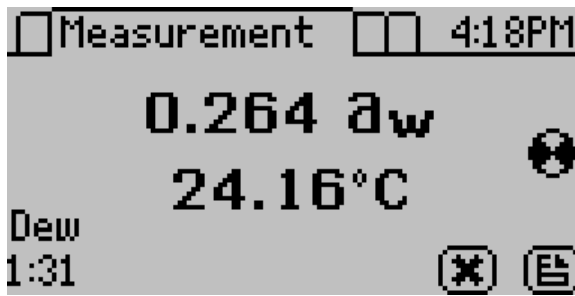
Isotherm Tab

The Isotherm tab, as seen below, is the main screen which displays each time you turn on your VSA. If this screen doesn't appear, refer to the chapter on troubleshooting. From this screen, a new test can be initiated by pressing the wizard icon (third button from right). If a test is already running, this screen will display the latest test values including water activity, weight (% weight change or % moisture content) and temperature. Pressing the 2nd button activates the chart view.



Measurement Tab

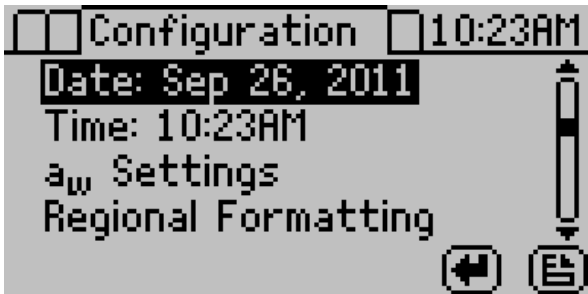
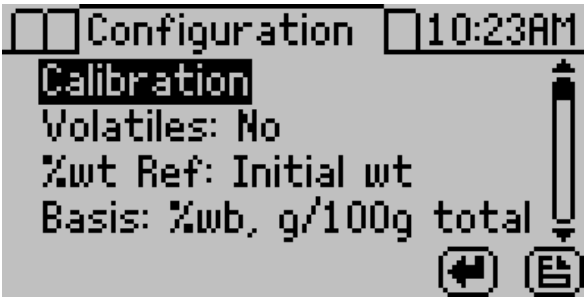
The Measurement tab allows you to take water activity readings of the sample in the sample chamber when not running a test. Pushing the right or left arrow keys will change the display to a temperature equilibration screen shown below. This screen shows the temperature difference between the sample temperature and the lid temperature.

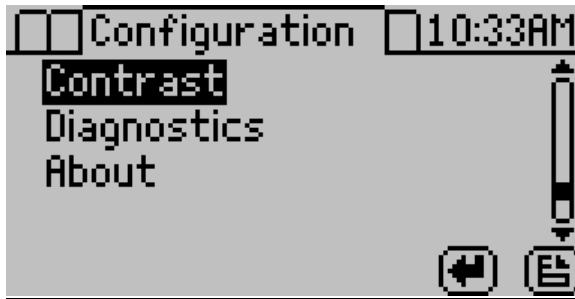


Configuration Tab

When at the configuration screen, pressing the up and down arrow keys moves the cursor through the various configuration options. Press the left and right arrows to page through the

options. The enter button will allow you to change the highlighted setting.

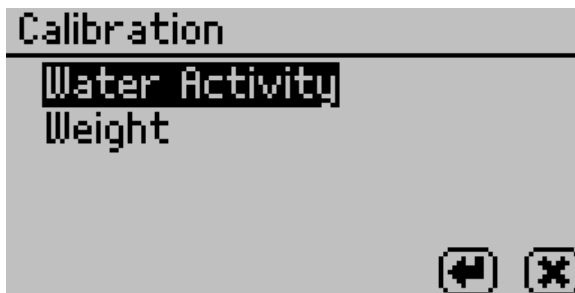




Calibration:

Pressing the Enter button when Calibration is highlighted opens the calibration menu where either a water activity or weight adjustment can be made.

For more details on weight and water activity calibration procedures refer to the chapter on Verification of Calibration. You may also reset the calibration to the factory defaults by highlighting the Defaults option in the calibration menu and pressing Enter. This will reset all options to the way they were when the instrument arrived at your location.



Volatiles:

The instrument will give accurate readings on most samples when the default volatiles setting is "Off". However, samples

with certain volatiles in high enough concentrations may give inaccurate water activity values. This is because the volatiles condense on the mirror during the reading process, but do not evaporate from the mirror as water does. As a result, the reading on samples with volatiles will not be accurate. The concentration of volatiles that will cause interference is variable and matrix dependent. The most effective method to determine if volatiles are a problem is to compare dew point readings to capacitance readings. If the dew point readings are more than 0.02 higher than the capacitance readings, volatiles are likely a problem and it is recommended that the volatiles setting be switched to "On". When the volatiles setting is turned on, the instrument only uses the capacitive relative humidity sensor for water activities measurements and isotherm tests. All other operations and features will be the same, including measurement times and adjusting for linear offset. Press enter to toggle the volatiles setting on and off.

Weight Reference:

Weight reference is the reference used when calculating the percent change in weight of the sample. This can be based on the initial weight of the test, the starting weight of the stage or the minimum weight during the test. This reference is used anytime a percent weight is calculated.

Basis:

The VSA generates water activity values and corresponding sample weights during moisture desorption and resorption. To complete the isotherm, the sample weights must be translated into moisture contents. Moisture content can be calculated in different ways and reported with a variety of different units. The

basis parameter allows the reporting of moisture content in two different ways:

Percent moisture on a wet basis (%wb, g/100g total)

$$\% \text{Moisture} = \frac{\text{initial weight} - \text{dry weight}}{\text{initial weight}} \times 100 = \frac{\text{grams of water}}{100 \text{ grams total}}$$

Percent Moisture on a dry basis (%db, g/100g solids)

$$\% \text{Moisture} = \frac{\text{initial weight} - \text{dry weight}}{\text{dry weight}} \times 100 = \frac{\text{grams of water}}{100 \text{ grams solid}}$$

For food applications, moisture contents are reported on a wet basis. A simple equation will convert between the wet and dry basis.

$$\% \text{ moisture dry} = \frac{\% \text{ moisture wet}}{100 - \% \text{ moisture wet}} \times 100$$

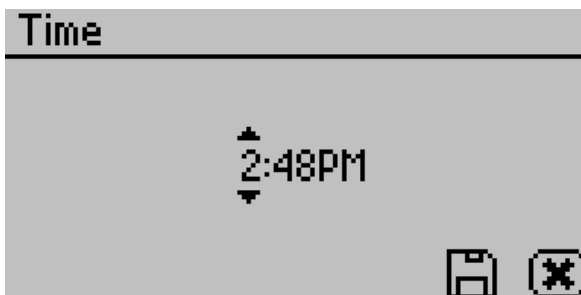
$$\% \text{ moisture wet} = \frac{\% \text{ moisture dry}}{100 + \% \text{ moisture dry}} \times 100$$

Date:

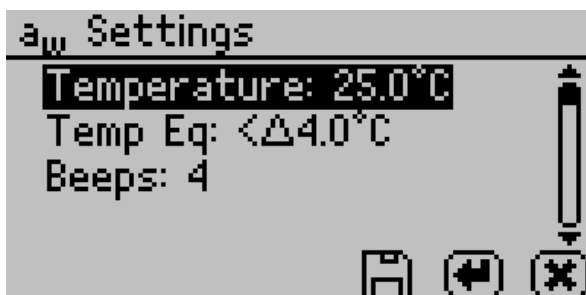
The VSA has an internal calendar and clock. The time and date are recorded with each water activity reading. Pressing Enter when the Date option is highlighted allows you to set the date in the instrument. Press the left and right arrows to change between the month, day and year. Press the up or down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

**Time:**

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

**a_w Settings:**

Settings related to water activity measurements can be updated here. These include default system temperature, temperature equilibration tolerance and beeps.

**Temperature:**

This setting is the temperature that the instrument maintains during water activity measurements. It also is the temperature that the instrument will return to after a test is complete. The default temperature is 25°C. Press the enter button to change the temperature setting. The VSA may be set between 15 and 50°C by 0.1°C intervals. Using the up and down arrows, set the VSA to your desired temperature and press the save button.

Temp Eq:

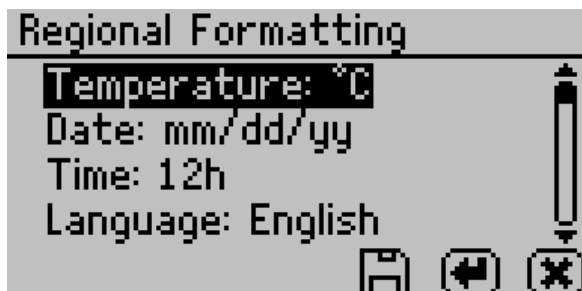
Temperature Equilibration Tolerance or “Temp Eq” sets the maximum difference allowed between the lid and the sample. This lid to sample temperature difference must be less than the Temp Eq in order to start a water activity measurement. If a sample is out of this range, the VSA will wait for the sample to equilibrate prior to beginning the water activity measurement.

Beeps:

Indicates the number of audible beeps after a water activity measurement is completed. This value can be set to off (0), 4, or infinity (∞).

Regional Formatting:

Allows you to configure how the VSA will display information. You may choose the temperature scale (Celsius vs Fahrenheit), the date display (mm/dd/yy vs. dd/mm/yy), the hour format (12 vs 24 hour) and the language. Press enter to switch the setting.

**Admin Settings:**

Allows you to create an administrator password as well as create, edit and delete additional users. The admin option allows the administrator to grant or block access to some or all of the configuration options in all Series 4 models. For example: If the administrator wanted to make sure that all samples were read at 25°C the administrator would set their temperature to 25°C and then would lock all other users out of that configuration screen. This is accomplished by entering the Access function and selecting the desired option to toggle it on and off. Additionally you can lock and unlock all of them at once. (For example, if you do not want John Doe changing the instrument's measurement temperature, the administrator can lock that function for John.) The areas that can be locked are calibration, temperature, temperature equilibration, sensor selection, mode, date/time, region, password, auto-save, number of beeps, contrast, and delete functions.

User Setup:

Users can be added, edited or deleted from this screen. To add a user, press the Enter button when the “New” is highlighted. To edit or delete a user, choose the target user and press enter. When creating a new user, an alphabet screen will appear where a name can be entered using lower case, upper case and accents.



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

Auto Restart:

Auto restart enabled the instrument to automatically resume the currently running test if there is a power failure of any kind. Press enter to toggle auto restart between on and off.

Lighting:

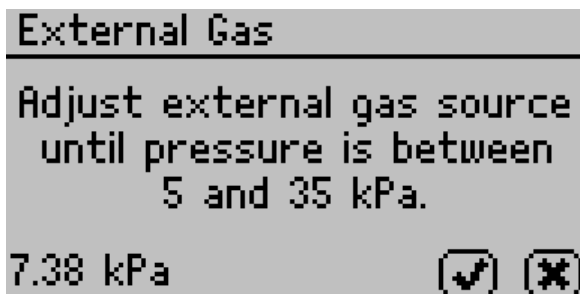
Turns the lighting in the desiccant tube area on or off.

External Gas:

Enables or disables the use of external gas. When External Gas is “On”, the external gas is used as the air source for desiccation and wetting of the sample. When External Gas is “Off” the internal pump uses ambient air as the air source.

NOTE: WHEN THE EXTERNAL GAS IS USED IT IS EXHAUSTED INTO THE ATMOSPHERE. BE SURE TO PROVIDE ADEQUATE VENTILATION AND SAFETY MEASURES WHILE USING AN EXTERNAL GAS SUPPLY WITH THE INSTRUMENT. COMBUSTABLE OR HAZARDOUS GASES ARE NOT RECOMMENDED.

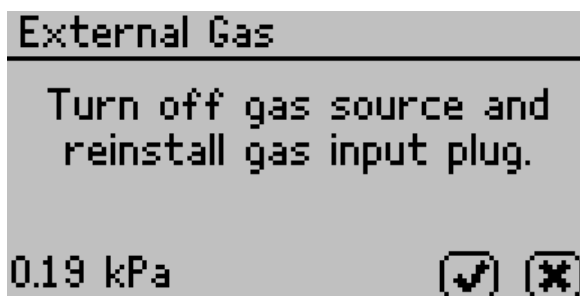
If the external gas is off and you want to turn it on, pressing enter causes the following screen to appear:



To start using external gas, remove the external gas plug from the back of the VSA and insert the desired gas source. Adjust the external gas pressure until it is between 5 and 35 kPa (0.7 to 5 psi). The check mark will be hidden until the gas is within the acceptable pressure range. Press Check to enable external gas or escape to cancel.

Note: When selecting a gas source, choose a gas supply with a filter system that eliminates oil and other particulates to help prolong the life of your VSA.

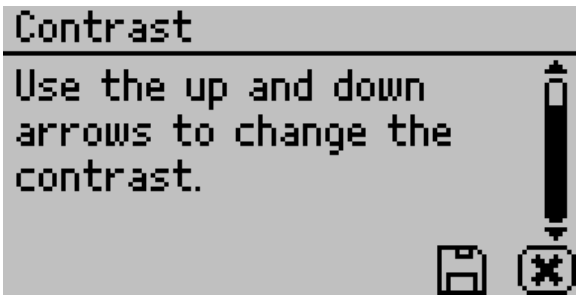
If the external gas is on and you want to turn it off, pressing enter causes the following screen to appear:



To stop using external gas, turn off the external gas source and remove the external gas tubing and adapters from the back of the instrument. Reinstall the external gas plug and then press the check to disable external gas or escape to cancel.

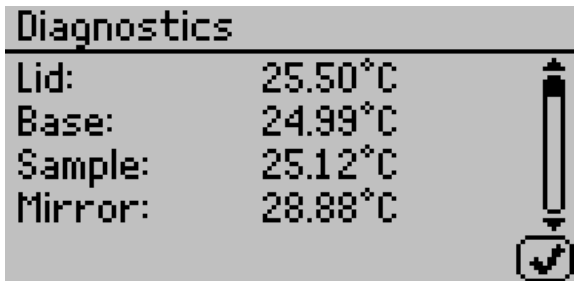
Contrast:

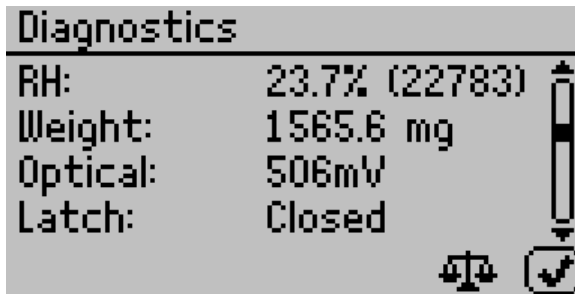
Allows you to set the contrast of the screen to your liking. Viewing the screen from a sitting versus a standing position may require contrast adjustment for the best visibility in that position.



Diagnostics:

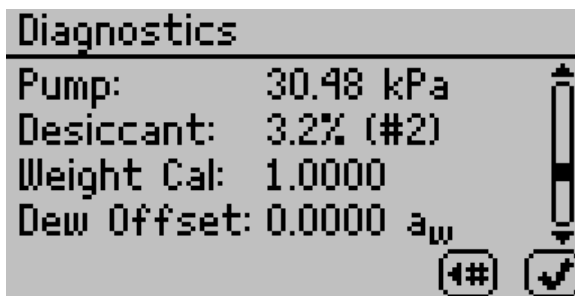
The diagnostics screen provides you with detailed information about all the sensors in the instrument. Refer to the Troubleshooting section for details on what kind of values you should expect for each sensor. Press up or down to page between the different diagnostics screens. On startup, the pump is turned on and flows dry air into the chamber so that the desiccating capacity of the tubes can be monitored.





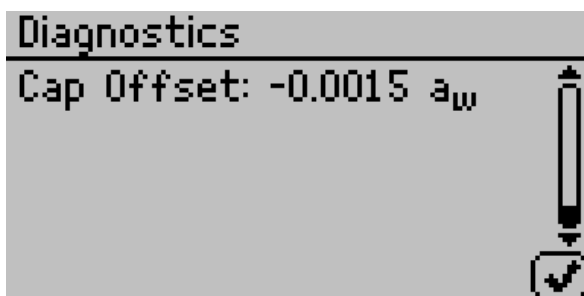
The weight icon on the bottom allows you to zero the balance so that you can take weight readings via the diagnostic screen.

The weight calibration value is the current calibration applied to balance readings. A value of one means no weight calibration is applied. The dew point and capacitance RH sensor offset values are the current linear offsets applied to water activity readings for the respective sensor type. A value of zero means that no water activity calibration is applied.



The number button on the bottom screen allows you to switch between the two desiccant tubes. If you press the number button with the arrow to the left, it switches to the left desiccant tube (#1). If you press the number button with the arrow to the

right, it switched to the right desiccant tube (#2). Any time the desiccant tube button is pressed the pump is turned on and dry air is blown into the chamber so that desiccating capacity of the tubes can be monitored.



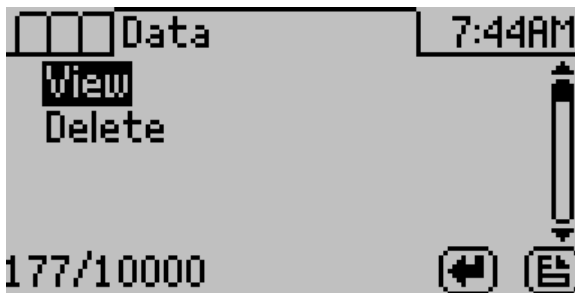
About:

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

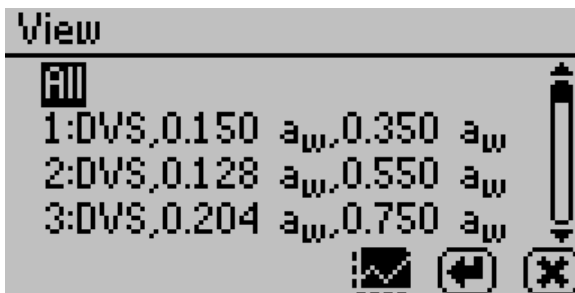




Data Tab

This tab allows you to view detailed information about the test stored in memory. You can view data as a list, view detailed data point information, and graph all or individual stages. You can also delete the data in memory.



View:

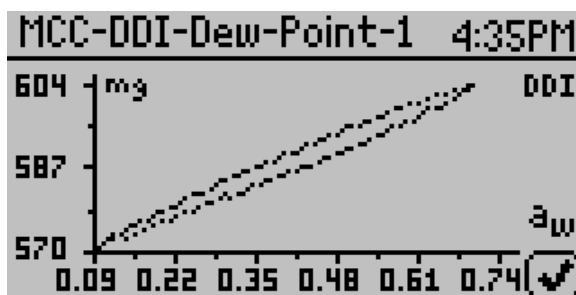


This selection brings you to a screen where you can decide how you want to view your data. You can choose to view your whole test at once by selecting "All" or you can view an individual stage. Once the desired portion of the test is selected, you can then choose to graph the data by pressing the graph icon  or by pressing the enter icon  to view summary information of

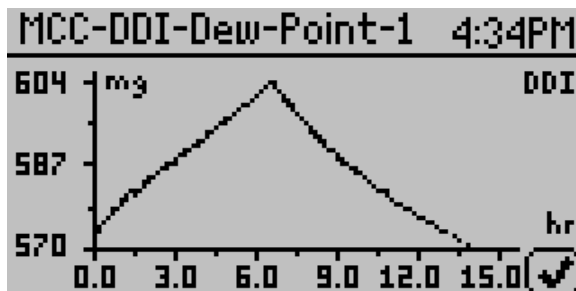
each data record. See Chapter 11: Computer Interface for information about downloading test data to a computer.

Graph Screen:

This selection shows the graph of the selected data from the previous screen. You can change the y-axis units by pressing the left or right arrows or change the x-axis units by pressing the up or down arrows. The title of the graph indicates the test name and the stage number (if applicable). The isotherm type is shown on the right hand side.



Example of a graph with water activity as the x-axis.



Example of a graph with elapsed time as the x-axis.

NOTE: For DVS isotherms, if water activity is the x-axis, only equilibrated data points appear on the chart. To view all the data points, the x-axis must be in terms of the time elapsed.

Summary Screen:

This selection will allow you to view your stored measurements. The up/down arrows will move you through the stored data with the most recent measurements at the top of the table. You may also press the left and right arrows to page quickly through the data. When you are viewing the summary screen, you may press the enter button on a highlighted reading to get detailed information on the reading as shown below.

Data	
0.129 a_w	
24.93°C	4.7 min
1322.7 mg	09/01/11
Dew Point	4:04PM
1/177	

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 sensor used, the time the reading was taken, and the sequence number of the stored reading.

Delete:

Selecting this option will delete all of the information currently stored in the instrument.

NOTE: You will NOT be able to recover deleted data so make sure you've backed up your data to the computer using the VSA downloader software package prior to deleting the data from the instrument.

6. Cleaning and Maintenance

Desiccant cartridge desiccant replacement.

Removal of the desiccant material

1. Orient the desiccant cartridge or tube (Figure 5: Desiccant Cartridge) so the lid or cap is up.
2. Remove the main lid.
3. Remove the foam located below the lid as shown in Figure 6: Desiccant Cartridge Foam.
4. Hold the small tubing at the top by curling the tubing over the top edge of the cartridge. This will prevent desiccant from getting into it when emptying the cartridge.
5. Pour out the desiccant material.

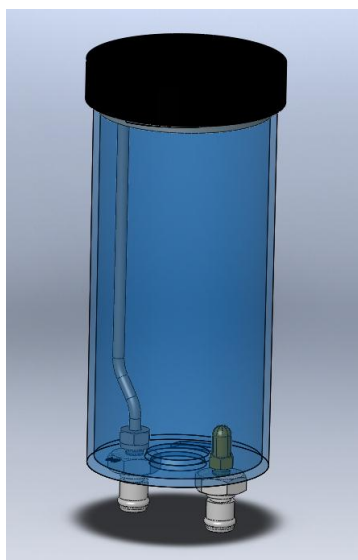


Figure 5: Desiccant Cartridge



Figure 6: Desiccant Cartridge Foam

Loading the desiccant into the desiccant cartridge.

1. Orient the desiccant tube so the opening is upward.
2. Hold the tubing to the side of the cartridge and looped over the edge of the cartridge.
3. Fill the cartridge to the top of the cartridge.
4. Shake or vibrate the desiccant cartridge to settle the material.
5. The top of the desiccant should be about 0.1 inches from the top.
6. Place in the foam at the top.
7. Place the small tubing across the filter and through the slit in the foam.
8. Wet with water the cartridge (bottle) lip or the edge of the washer adjacent to the bottle lip located in the lid as shown in Figure 7:Desiccant Lid.
9. Secure the lid to the desiccant cartridge
10. Verify the lid seal is correct by using the squeeze bottle (included with your VSA). Refer to Figure 8: Checking Desiccant Cartridge for leaks.
 - a. Have a pan or cup with about 1 inch deep water in the bottom



Figure 7: Desiccant Lid

- b. Hold one finger over the output fitting
- c. Pressurize the desiccant tube using the pressure bottle as shown in the figure. (Note: the bottle just needs to be squeezed with air in it, do not put water into the squeeze bottle!)
- d. Place the desiccant tube with the lid into the water. If the lid is not sealed, the water could flow into the desiccant cartridge. Hold the tube in the water only as long as needed to verify the test.
- e. Look for bubbles escaping from the lid. If there are many bubbles, the lid is not sealed. The washer will need to be reset. This requires that the lid be removed and the washer reset. The washer will need to be lubricated with water and lid re-installed.



Figure 8: Checking Desiccant Cartridge for leaks.

- 11. When the desiccant cartridge is placed into the desiccant receiver, the o-rings on the fittings will need to be wetted for ease of installation and to reduce wear.

Cleaning the Testing Chamber

Keeping your VSA clean is vital to maintaining the accuracy of your instrument. Dust and sampling debris can contaminate the sampling chamber and must therefore be regularly cleaned out.

Purpose

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

When to Clean:

The instrument should be cleaned if visual inspection indicates the chamber is dirty or as instructed in the verification flowchart.

Materials Needed

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

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

Before you begin:

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

Follow the cleaning procedures being careful not to get cleaning solution or alcohol on the capacitance sensor filter. Repeated exposure of cleaning materials or contaminants to the filter may cause inaccurate readings. If the filter appears to be

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

Turn the power off on your AquaLab VSA. If latched, move the lever over to the open position. Lift the chamber cover to expose the VSA sample chamber and sensors. The sample chamber



consists of all surfaces within the red o-ring when the lid is closed.

Cleaning Procedure:

1. Cleaning the Sample Chamber

Note: Be extremely careful not to damage the fan blades when cleaning the 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 thin plastic rod (spatula) and moisten it with isopropyl alcohol or Decagon Cleaning Solution. Note: Do NOT dip a used Kimwipe into your container of IPA or cleaning solution (the IPA or cleaning solution will become contaminated).

c. WASH—Clean upper chamber, o-ring, and all surfaces of the block within the o-ring. You may need to replace the Kimwipe if it becomes too dirty during this process.

d. Clean lower block with a fresh Kimwipe. Be sure to clean the entire block surface.

e. RINSE—Repeat steps b-d using new Kimwipes with distilled water.

f. DRY—Repeat steps b-d using new, dry Kimwipes to help remove any moisture remaining from the cleaning.

g. Visually inspect the sample chamber for cleanliness. Re-clean if necessary. Note: Do not reuse Kimwipes.

2. Clean the Mirror

a. Wrap a new Kimwipe around the end of the thin plastic rod (spatula) and moisten it with isopropyl alcohol or Decagon Cleaning Solution.

- b. WASH—Swipe the moistened Kimwipe across the mirror once. (A single swipe is usually sufficient to remove contaminants.)
- c. RINSE—Repeat steps a-b using new Kimwipes moistened with distilled water instead of cleaning solution.
- d. DRY—Repeat steps a-b using new, dry Kimwipes to help remove any moisture remaining from the cleaning.
- e. Visually inspect the mirror for cleanliness. Re-clean if necessary.

3. Clean the Thermopile and Optical Sensor

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

- e. Visually inspect the thermopile and optical sensor for cleanliness. Re-clean if necessary.

4. Additional Drying Time

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

7. Verification of Calibration

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

Before you check the instrument we recommend that you run a sample of the activated charcoal pellets provided in your VSA cleaning kit. This cleans the air inside the chamber, helping it come back to a stable sampling environment. It is important to verify VSA's water activity calibration against known standards to guarantee optimal performance and accuracy. AquaLab recommends verification before running a new isotherm test.

Water Activity Verification

AquaLab VSA uses both a capacitance relative humidity sensor and a chilled-mirror dew point technique to determine water activity. If volatiles is set to "yes" as described in Chapter 5, water activity verification will be performed on the capacitance sensor only, but if volatiles is set to "no", the dewpoint sensor will be verified. While the instrument does not require a routine full calibration, it is important to verify for linear offset periodically. The components used by the instrument to measure water activity are subject to contamination which may affect the VSA's performance. When this occurs, it changes the accuracy of the instrument. This is what is called a "linear offset."

Therefore, frequent verification assures the VSA 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 Decagon. Using verification standards to verify accuracy can greatly reduce preparation errors. For these reasons, we recommend using standards available through Decagon for the most accurate verification of your VSA's performance.

Performance Verification Standards come in six water activity levels: 1.000, 0.984, 0.920, 0.760, 0.500, and 0.250 a_w . The standards are produced under a strict quality assurance regime. Please contact Decagon Devices to order additional standards or order via the web at www.aqualab.com/reorder.

Verification Standard at 25°C	Water Activity
13.41m LiCl	0.250 \pm 0.003
8.57m LiCl	0.500 \pm 0.003
6.00m NaCl	0.760 \pm 0.003
2.33m NaCl	0.920 \pm 0.003
0.50m KCl	0.984 \pm 0.003
Distilled Water	1.000 \pm 0.003

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

Although Distilled Water is available as a verification standard. Decagon does not recommend its use with the AquaLab VSA.

To use a verification standard, remove the twist top and pour the contents into one of the stainless steel sample cups. Information about the standard's value and molality can be found printed on the outside of the plastic vial.

When to Verify for Linear Offset


Linear offset should be checked against two known verification standards before running a new isotherm test. Linear offset should never be verified solely against a single standard, since it does not give an accurate representation of the linear offset. For best results, the water activity verification for the VSA should be conducted using the 0.76 a_w and 0.25 a_w standards. Checking the water activity of a standard solution will alert you to the possibility of unit contamination or shifts in the linear offset from other causes.

NOTE: The verification process is the same whether the volatiles is set to yes or no, except that the accuracy for the capacitance sensor is $\pm 0.020 a_w$.

Verification

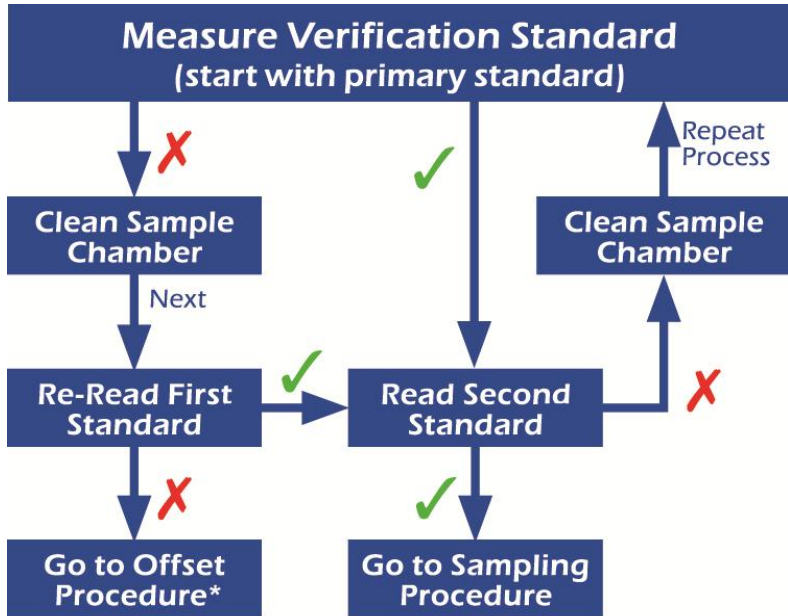
To verify for linear offset of your VSA do the following:

(Refer to the flowchart at the end of this section)

1. From the Isotherm Tab, navigate to the Measurement Tab by pressing .
2. Choose a 0.25 a_w standard.
3. Remove weighing pan.
4. Empty a vial of solution into a stainless steel sample cup and place it in the VSA's testing chamber. Make sure that

your standard is as close to the instrument temperature as possible. Note: Make sure the rim and outside of the sample cup are clean.


5. Carefully close the lid and move the lever to the READ position.
6. 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 A for the correct water activity value of Decagon's standards at temperatures other than 25°C.
7. If your VSA is reading within $\pm 0.005 a_w$ of the verification standard, choose a $0.76 a_w$ standard.
8. Prepare a sample cup of the second verification standard and make two readings. The second water activity reading for the second verification standard should be within $\pm 0.005 a_w$.
9. If either of the verification standards is not correct, it is probably due to contamination of the sensor chamber. For cleaning instructions, see Chapter 6. After cleaning, repeat verification from step two.
10. If you are consistently getting readings outside the water activity of your first verification standard by more than $\pm 0.005 a_w$, a linear offset has probably occurred. In this case, adjust the reading to match the verification standard's correct value as outlined in the next section.

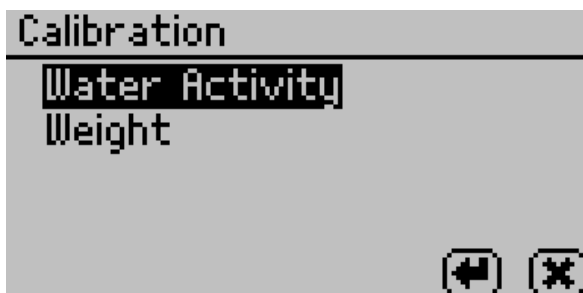


*See the verification section in the operator's manual

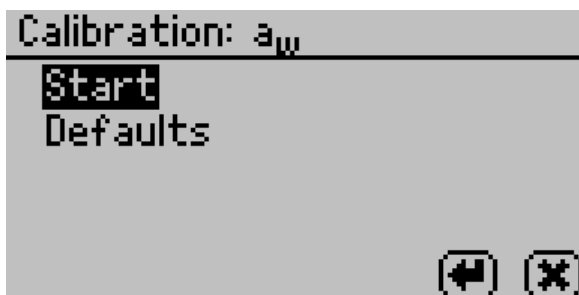
This flowchart is a graphical representation of the directions given above for checking for linear offset.


Adjust for Linear Offset for Water Activity


1. Once you are certain a linear offset has occurred, toggle to the Configuration tab by pressing the Menu icon button. Calibration is the first option highlighted in the configuration tab. Press the  icon button to bring up the following screen.






2. To offset for water activity, highlight water activity and press



 and the following screen will appear.


3. Highlight start and press . Follow the wizard on screen commands.
4. Empty the whole vial of solution into a sample cup. We recommend using the 6.0 NaCl (0.76 a_w). Do not adjust for the offset using distilled water. Ensure the rim and outside of the cup are clean. Place the sample cup in the VSA's sample chamber.
10. NOTE: The same verification standard may be used to verify and adjust the linear offset.

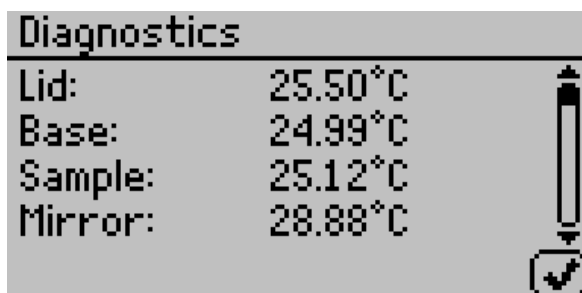
5. Carefully close the lid and move the lever to the READ position. Press the  icon button to begin testing.
11. NOTE: If you decide at this point not to continue with the linear off set program, just return the lever to the OPEN position or press the cancel button and you will be returned to the previous screen.
6. After your VSA has finished measuring the verification standard, it will display the test value.
7. 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  to store this new value. To cancel and return to the main menu, press  and no changes will be made.
8. Re-measure the verification standard again on the Measurement Tab. It should read the proper value (within $\pm 0.005 a_w$) at a given temperature for your particular standard (see Appendix A for temperatures other than 25°C).
9. 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 off set, contact AquaLab by email at support@aqualab.com or by phone at 509-332-5601 for further instructions. If you purchased your AquaLab instrument from one of our international distributors, please contact them for local service and support.

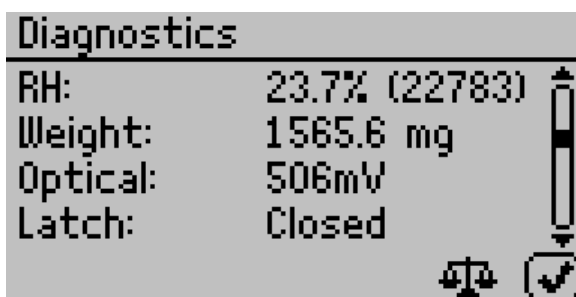
Verifying Weight Measurement

The VSA uses a magnetic force balance to gravimetrically track the weight of the sample during the isotherm test. The performance of this balance is verified against a NIST traceable 2 gram standard weight. This 2 gram weight is included with the VSA and should be handled with tweezers and not bare skin.

1. To verify the balance, toggle to the Configuration tab by pressing the Menu icon button. Scroll down to Diagnostics and press the  icon button to bring up the following screen.




2. Scroll down until you see weight on the screen.

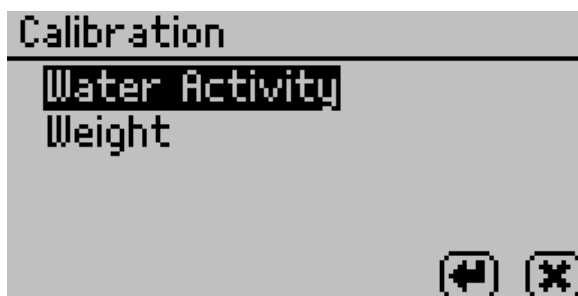



3. Close the lid and seal by pressing the lever to read.
4. Press the balance icon at the bottom to zero the balance.

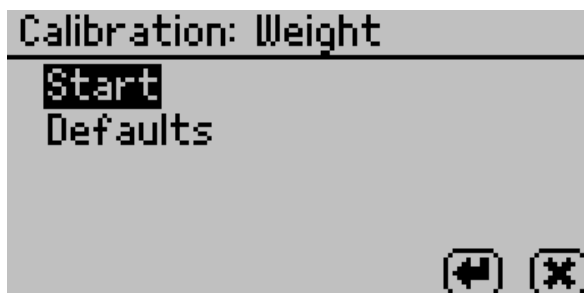
5. Open the lid and carefully place the 2 gram standard on the balance using glove and tweezers.
6. Close the lid and verify that the weight readings are within +/- 0.1 mg of 2000mg. If the weight reading is not correct, a balance adjustment may be needed.





Adjusting for Weight Measurement Offset

1. To adjust the balance, toggle to the Configuration tab by pressing the Menu icon button. Calibration is the first option highlighted in the configuration tab. Press the  icon button to bring up the following screen.



2. To adjust the balance, highlight weight and press  and the following screen will appear.






3. Highlight start and press . Follow the wizard on screen commands.
4. When asked to place the 2 gram weight on the balance, handle carefully with tweezers and gloves to prevent changing the weight of the standard.
5. Carefully close the lid and move the lever to the READ position. Press the  icon button to begin testing.
12. NOTE: If you decide at this point not to continue with the linear off set program, just return the lever to the OPEN position or press the cancel button and you will be returned to the previous screen.
6. After your VSA has finished measuring the weight verification, it will display the test value.
7. Press the up and down arrows to adjust the weight reading to its proper value for the 2 gram (2000 mg) standard. When the correct value is displayed, press  to store this new value. To cancel and return to the main menu, press  and no changes will be made.

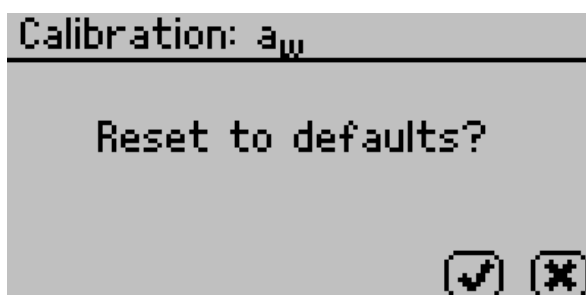
If you continue to have problems with weight readings, contact AquaLab by email at support@aqualab.com or by phone at 509-332-5601 for further instructions. If you purchased your AquaLab instrument from one of our international distributors, please contact them for local service and support.



How to Restore Factory Defaults

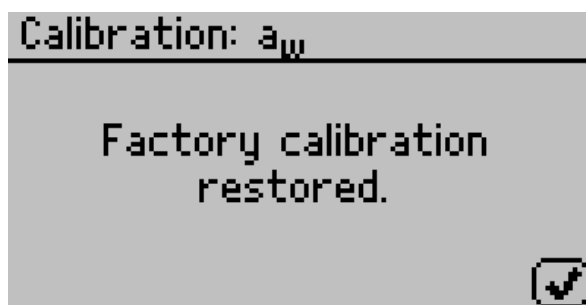
To restore original calibration settings, do the following:


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

2. Select water activity or weight, depending on which defaults you want to restore and press .
3. Scroll down to Defaults and press the  icon button to access the Restore Factory Defaults routine. To cancel and return to the main menu, press the Cancel icon button. After pushing the Enter icon button, the following screen will appear:



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



Similar screens will appear if the weight factory defaults are being restored, but the screen will say weight instead of water activity. To return to the main menu screen, press .

8. Running a Test



Becoming Familiar with Stages

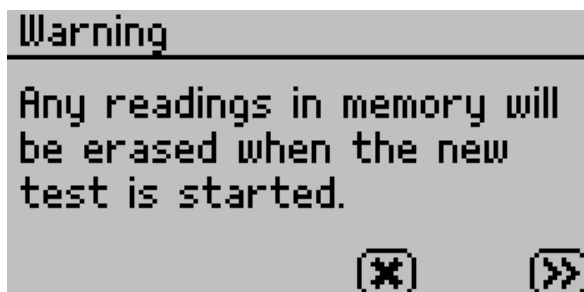
The AquaLab VSA uses test stages to maximize the utility of running an isotherm test. In the vocabulary of setting up and running moisture sorption isotherms with the VSA, an isotherm test is considered a collection of stages all performed on 1 sample. Each stage should be considered a unique portion of a test with its own settings while keeping in mind that all stages will be conducted on 1 sample and will be combined into 1 data set at the end of the isotherm test. This means that an isotherm test could consist of 1 stage or could consist of up to 20 stages. An example of an isotherm test with multiple stages might be running a sample through full adsorption and desorption using the static DVS method for Stage 1, then setting up Stage 2 to run the same test, but using the DDI method on the same sample. Another example would be to run a sample through full adsorption and desorption at 15°C for Stage 1, then, in Stage 2, switch the temperature to 25°C and run the same test on the same sample. This stage interface makes it easy to compare dynamic and static isotherms and utilize the advantages offered by both methods all on a single sample. It also makes it easy to edit currently running tests as seen below.



Running a Moisture Sorption Isotherm Test

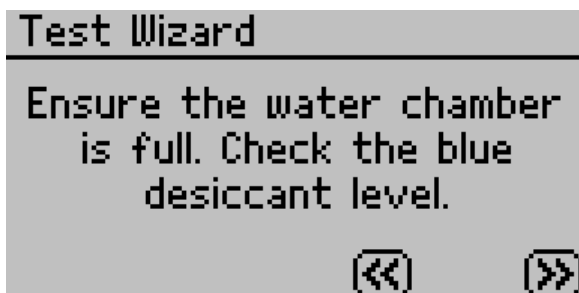
Before starting a test it is important to verify that the VSA is performing correctly before starting a test using Decagon's Verification Standards. Please refer to Chapter 7 for instructions on how to verify your VSA and adjust the calibrations if



necessary. Tests can also be setup using a wizard interface in the VSA Downloader software (see Chapter 9 for more information).

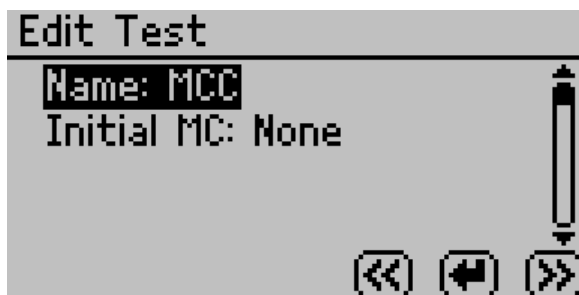
1. Before starting a test, set the Volatiles: Yes/No option in the configuration menu. If your sample has volatiles, select “Yes” otherwise select “No”.
2. On the main screen, select the test wizard 
3. A warning screen will appear letting the user know that any data points currently on the instrument from previous tests will be deleted when a new test is started. Press  to continue.






4. A new message reminding the user to make sure the water chamber is full and there is still active (blue) desiccant in the desiccant tubes. For instructions on filling the water chamber and replacing the desiccant, refer to Chapter 4 instrument setup. Press  to continue or  to go back.



5. The Edit Test screen will now appear. To provide a name for the test, press  with Name highlighted and then use the arrow keys to create the name. When finished, press . (The name of a previous test will appear by default.)





6. Enter initial moisture content if known (optional) by pressing  with Initial MC highlighted and use the up and down arrow to enter the correct moisture. The %wb refers to the moisture reporting basis, which can be either wet basis (%wb) or dry basis (%db). The moisture content basis is set as a global preference in the configuration menu (see section 5) . Press  to continue or  to go back.



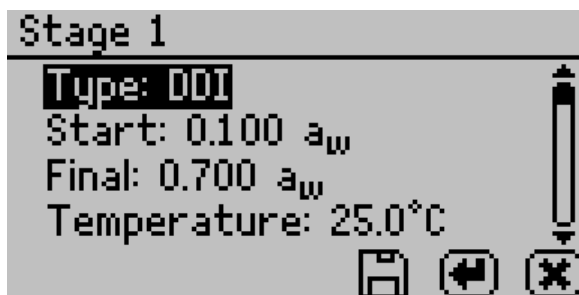
7. Setup each stage of the isotherm test. A stage is used to adjust the isotherm settings for an individual sample (up to 20 stages). Each stage is setup with the isotherm method (DDI or DVS) and the settings for that portion of the isotherm. Once a stage is created, it can be modified or deleted anytime before the test is started.
8. i.e. For milk powder, Stage 1 could use the DDI method to ramp quickly from 0.1 to 0.4 a_w and Stage 2 could use the DVS method to step from 0.4 to 0.8 a_w slowly in 0.05 a_w increments.






9. To setup a new stage, select "new" and press . If more than one stage is in memory, you will have the option of inserting the stage after any existing stage in the test. A screen with several parameters will appear. If a previous


test has been run, the stages for that test will be listed by default. These stages can be used as is or updated with new settings. *If you need to delete or edit a stage, go to the specific stage and press  and then select Edit or Delete.*



10. Select the isotherm method as either Dynamic Dewpoint Isotherm (DDI) or Dynamic Vapor Sorption (DVS). DDI will create a dynamic isotherm and DVS will create a static or equilibrium isotherm. The other choices will adjust based on which method, was selected.

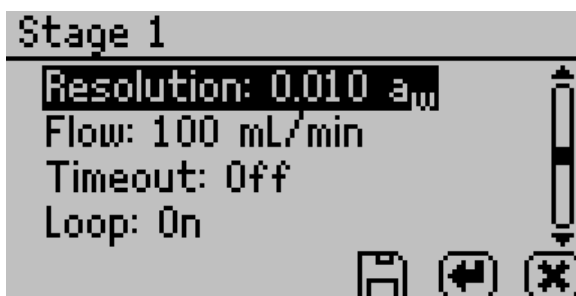



For DDI method





1. To choose the starting a_w , press  with "Start" highlighted and then cycle to the desired value using the up and down arrows. Any value between 0.03 and 0.95 a_w can be chosen including "current". Choosing "current" means that the stage will start at the initial a_w of the sample. Press  when finished.
2. To choose the final a_w , press  with "Final" highlighted and then cycle to the desired value using the up and down arrows. Any value between 0.03 and 0.95 a_w can be chosen including current (see "current" definition above).

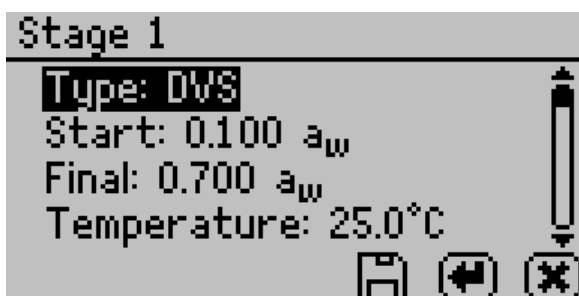
Selecting a final a_w that is lower than the starting a_w will result in desorption, while selecting a final a_w higher than the starting a_w will result in adsorption. Press  when finished.

3. To choose the temperature of the stage, press  with “Temperature” highlighted and then cycle to the desired temperature using the up and down arrow keys (hold down arrows for accelerated scrolling). Any temperature between 15-60°C can be chosen. Each stage can run at a unique temperature.
4. To choose the desired a_w resolution, press  with “Resolution” highlighted and then cycle to the desired value using the up and down arrows. Any value between 0.003 and 1.0 a_w can be chosen. The resolution for DDI determines the target a_w resolution. Setting a higher step value will make the test faster, but will decrease the a_w resolution. A DDI step of 0.01 a_w is recommended.









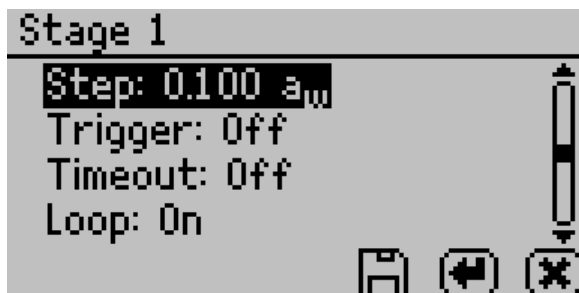
5. To choose the air flow rate, press  with “Flow” highlighted, and then cycle to the desired value using the up and down arrows. Any value between 10-600 ml/min can be selected. Higher flow rates will speed up the test, but may result in lower data resolution and non-equilibrium conditions. The default value of 100 ml/min is suitable for most products.


6. To choose to include a timeout setting, press  with "Timeout" highlighted, and then cycle to the desired value using the up and down arrows. Any value between 5.0 min to 30 days can be selected. The timeout determines the maximum time allotted to move from the starting a_w to the final a_w (DDI only) and is optional. The default value is off, which means there is no timeout value.
7. To choose to have the isotherm loop, press  with "Loop" highlighted to toggle between on and off. Turning loop on causes the isotherm to automatically return to the start a_w (with the same settings) once the final a_w has been achieved. This would create both an adsorption and desorption curve (or vice versa depending on the starting direction).
8. When finished adjusting the settings for the stage, press  to save and return to the edit stage screen. Press  to return to edit stage screen without saving changes.

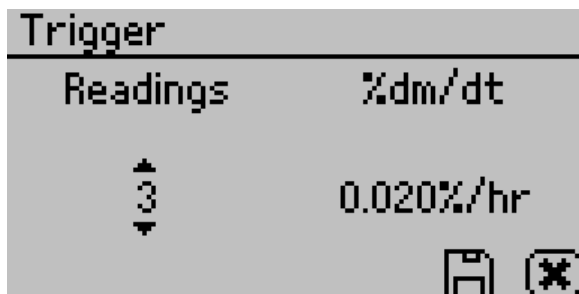


For DVS method




1. To choose the starting a_w , press  with "Start" highlighted, and then cycle to the desired value using the up and down arrows. Any value between 0.03 and 0.95 a_w can be chosen including "current". Choosing "current" means that the stage will start at the initial a_w of the sample. Press  when finished.
2. To choose the final a_w , press  with "Final" highlighted, and then cycle to the desired value using the up and down arrows. Any value between 0.03 and 0.95 a_w can be chosen including current (see "current" definition above). Selecting a final a_w that is lower than the starting a_w will result in desorption, while selecting a final a_w higher than the starting a_w will result in adsorption. Press  when finished.
3. To choose the temperature of the stage, press  with "Temperature" highlighted, and then cycle to the desired temperature using the up and down arrow keys. Any temperature between 15-60°C can be chosen. Each stage can run at a unique temperature.
4. To choose the desired a_w step, press  with "Step" highlighted and then cycle to the desired value using the up and down arrows. Any value between 0.003 and 1.0 a_w can be chosen. The step setting for DVS determines the a_w values for equilibration and consequently the resolution. A step setting of 0.10 a_w will result in humidity being controlled to 0.1, 0.2, 0.3 ... up to the final a_w , remaining at each level until either equilibrium is achieved based on the trigger value or the timeout value is reached. Setting a higher value will make the test faster, but will decrease the a_w resolution.



To choose the trigger %dm/dt value that will indicate equilibration, press enter with "Trigger" highlighted and a new window will open. Cycle to the desired trigger value using the up and down arrows. The trigger can be shut off or be any value between 0.001-1%/hr. The trigger value represents an acceptable change in mass per change in time to indicate equilibrium for a given step. Next, choose the number of readings or events meeting the trigger value that are required to achieve equilibration. For example, choosing 3 means that in order for a step to have reached equilibrium, 3 %dm/dt readings in row must be less than the set trigger value. Any value between 1 to 10 can be chosen. Setting a higher trigger value and a lower number of events will make the test faster, but may not result in complete equilibrium. A recommended starting point would be 2 events at 0.01%/hr. If the trigger is turned off, the sample will be held at each step for the time indicated by the timeout setting. After adjusting all settings, press  to save the settings and return to the stage setup screen.



5. To choose to include a timeout setting, press with “Timeout” highlighted and then cycle to the desired value using the up and down arrows. Any value between 5.0 min to 30 days can be selected. The timeout determines the maximum time allotted for each a_w step, NOT the time from starting a_w to final a_w . The default value is off, which means there is no timeout value and only the trigger value will determine the end of step. If both a trigger value and timeout value are set, whichever is reached first will determine the end of the step.
6. To choose to have the isotherm loop, press with “Loop” highlighted to toggle between on and off. Turning loop on causes the isotherm to automatically return to the start a_w (with the same settings) once the final a_w has been achieved. This would create both an adsorption and desorption curve (or vice versa depending on the starting direction).
7. When finished adjusting the settings for the stage, press to save and return to the edit stage screen. Press to return to edit stage screen without saving changes.
8. After setting up all stages on the edit stage screen, press the button.

9. Follow the message on the screen to place an empty stainless steel cup in the chamber and seal it. Press  to tare the cup.
10. Follow the message on the screen to place the sample in the cup and press  to weight the sample.
11. Press  to begin the test.

Using DDI to investigate Glass Transition

To investigate glass transition events using dynamic isotherms, a DDI test with the settings of initial water activity of 0.03-0.1 a_w , final water activity $>0.80 a_w$, flow rate of 30-40 ml/min, resolution of 0.01 a_w and no timeout should be used.

Creating a Working Isotherm Using the VSA

Working isotherms, as described in the Theory section in Chapter 3, can be generated using the VSA, but it requires several steps.


1. Generate two isotherm tests using the VSA, one for adsorption and one for desorption and both starting at the “current” water activity value. A working isotherm is an analysis of the sorption characteristics of a sample starting from its native state. To generate this data requires the analysis of two sub-samples in the same condition as the original sample.
2. **Adsorption Curve:** One sub-sample is analyzed for adsorption from its current state. The test is setup with one stage with desired settings for method (DDI or DVS), temperature, step value, flow rate etc., but the starting water activity must be **Current**, and the final water activity must be a value higher than the current water

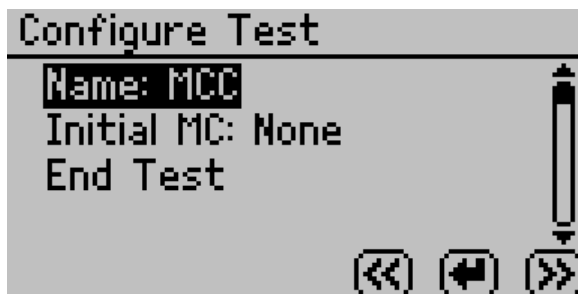
activity of the sample. Upon completion of the isotherm, the data is downloaded and the test saved using a name that will identify the sample name and the sorption direction.


3. **Desorption Curve:** A second sub-sample is analyzed for desorption from its current state. The test is again setup with one stage with desired settings for method (DDI or DVS), temperature, step value, flow rate etc., but the starting water activity must be **Current**, and the final water activity must be a value lower than the current water activity of the sample. Upon completion of the isotherm, the data is downloaded and the test saved using a name that will identify the sample name and the sorption direction.

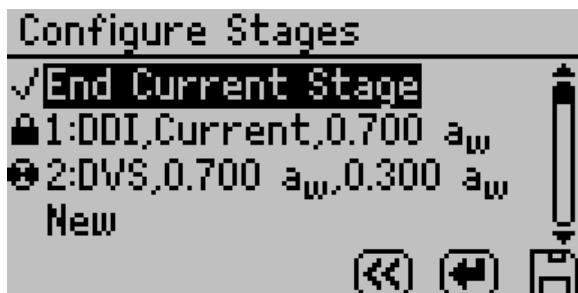
Editing a Running Isotherm Test




A test that is already running can be edited and additional stages can be added to a running test (inserted after the current running stage) at any time using the edit test function. This can be done using either the instrument interface or the software. To edit the test using the device interface:

1. Press the  icon as you did when starting a test. The following screen will appear.



2. Press the  icon to edit the selected test. The following screen will appear





3. The current running stage (spinner icon) and completed stages (lock icons) cannot be edited. To add additional stages, select new and press . Then setup each stage as outlined above. To edit an existing stage that is not currently running and is not complete, select it and press . Adjust the settings as instructed above.
4. To alter a currently running stage, insert a new stage after the currently running stage. Make sure to set the starting a_w to “Current” if you want the new stage to resume where the previous stage left off. Then select “End current stage” and click . The current stage will end and the newly created stage will start.

To edit a test using the software, see the VSA Downloader Software in chapter 9.

Running a Water Activity Test

(Can't be done when isotherm test is running)

The AquaLab VSA can also be used as a water activity meter. For best results and temperature control, we recommend you use a clean stainless steel cup for water activity testing and that you remove the weighing pan and place the cup directly on air cylinder when testing for water activity. If volatiles is set to yes as described in Chapter 5, water activity readings will be determined using the capacitance sensor, otherwise, the dewpoint sensor is used. To measure water activity

1. Press  until you are at the Measurement screen (2nd tab). Move latch left and/or press  to start a reading. The pinwheel will spin indicating a measurement is running.
2. The a_w reading for the sample and the temperature will display when finished.
3. To change between dew point and capacitive readings, you will need to toggle the Volatiles Yes/No option in the Configuration menu (Volatiles = Yes → Capacitive Sensor, Volatiles = No → Dew Point Sensor).

9. VSA Downloader Software

While the AquaLab VSA can operate as a stand-alone instrument, a computer must be attached to download test results. The VSA Downloader software makes it easy to download moisture sorption isotherm measurements from your VSA, view the data in table or chart form, export for use in other programs, and easily setup a new test with multiple stages. After installing the VSA Downloader on a computer, follow these steps to utilize software.

Connect

1. Connect your USB cable between the COM port on the Vapor Sorption Analyzer (VSA) and the USB port on your computer
2. Open the VSA Downloader software
3. Select the proper COM port in the toolbar
4. Click the Connect button in the toolbar

Configure and Start a New Test

1. Once your VSA is connected, click the Wizard button in the toolbar
2. Follow the Wizard's instructions and fill in the parameters of your test and each stage desired
3. After completing the Wizard, follow the instructions displayed on the VSA to start your test

Modify a Currently Running Test

Note: You can only modify stages that have not yet begun. If you wish to modify the current running stage, simply create a new

stage and adjust your stage behavior from the current reading of the VSA.

1. Connect to VSA running test
2. Click the Wizard button in the toolbar
3. Modify stages in the currently running test
4. Click Next
5. Click Finish and Allow the Wizard to finish communicating with the device

Download and Export

1. Once your VSA is connected and a test has been run, select Download in the toolbar
2. From the File menu or using the icon in the toolbar, select "Export..." to create an Excel compatible table
3. To view your data in table format, click the Table button in the toolbar
4. To view your data in chart format, click the Chart button in the toolbar

While the VSA Downloader does not have a hardcopy manual, extensive help files exist to guide you through the various features and processes of VSA Downloader. To find these help files in the VSA Downloader, click on the Help menu item and then select the VSA Downloader Help submenu item to display the help files.

10. Troubleshooting

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

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

Troubleshooting Quick Guide	
<u>If this problem occurs:</u>	
VSA won't turn on.....	Problem #1
Readings are slow or inconsistent	Problem #2
a _w reading on solution too high/low to adjust.....	Problem #3
Screen displays "Sample too hot"	Problem #4
Screen displays "a _w < 0.0"	Problem #5
Dew point sensor failure	Problem #6
The verification is not correct.....	Problem #7
Screen displays "Crystal failure"	Problem #8

Troubleshooting Quick Guide Continued

Screen displays "Contaminated Mirror"	Problem #9
Screen displays "Firmware is corrupted"	Problem #10
Screen displays "Missing bootstrap loader"	Problem #11
Screen displays "Annual calibration due"	Problem #12
Screen displays "No equilibration points available!" ..	Problem #13
Screen displays "Desiccant tank is low."	Problem #14
Displays Low or High System Pressure Warning.....	Problem #15
Power was reset.....	Problem #16
Screen indicates a Scale failure	Problem #17 (overload or underload)
Screen displays "Actuator failure."	Problem #18
Critical error starting a test	Problem #19
Can't communicate with the VSA	Problem #20
Weight is decreasing during adsorption.....	Problem #21
Weight is increasing during desorption	Problem #22
Isotherm test has stopped prematurely.....	Problem #23
Isotherm test taking unusually long time to complete	Problem #24

Troubleshooting Quick Guide Continued

Scale Readings are not stable or balance cannot be started.....	Problem #25
Scale calibration is failing	Problem #26
Desiccant cartridges are hard to install	Problem #27
Poor temperature control on the system	Problem #28
Diagnostic Screen	Problem #29

1.PROBLEM:

VSA won't turn on.

SOLUTIONS:

- 1) Check to make sure your power cord is securely attached to the back of the instrument and it is plugged into the power outlet.
- 2) A power surge may have caused a fuse to blow. To change the fuses, follow these instructions:
 - a. Unplug the power cord
 - b. Locate the panel where the power cord plugs in. The fuse box is on the right side of that panel. Press in on the release tab and pull the fuse-holder out. Pull the broken fuse(s) out and replace with a 1.25 Amp 250V fuse.

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

- c. Replace the fuse-holder and push it into the fuse-well until the release tab snaps in place.
- d. Re-connect the power cord and turn your instrument on. If the fuse blows again, a failed component may be causing the problem. Contact Decagon to make arrangements for repairs.

2.PROBLEM:

Reading are slow or inconsistent.

SOLUTIONS:

- 1) The sample chamber may be dirty. Refer to Chapter 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 will need to equilibrate to instrument temperature before an accurate reading can be made. (Refer to Chapter 8, Samples Not at Room Temperature.)
- 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 Chapter 8 for further explanation.
- 4) Your sample may contain volatiles. Volatiles are known to cause unstable readings, because they condense on the surface of the chilled mirror and alter the readings. Please refer to the volatiles section in Chapter 8 for hints on reducing difficulties with measuring samples with propylene glycol or ethanol. If you have further questions regarding the measurement of volatiles contact Decagon.
- 5) A fan blade in the block chamber may be broken or bent. If even salt standards take a long time to read, and the sample chamber is clean, you may have a broken

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

3.PROBLEM:

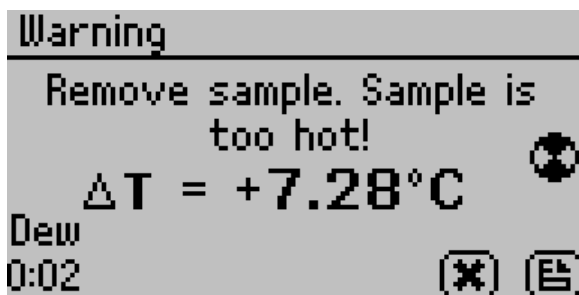
Water activity readings on verification standards are too high/low and a linear offset adjustment cannot be made any higher/lower.

SOLUTIONS:

- 1) The thermopile in your chamber, which measures sample temperature, may have become contaminated. Refer to Chapter 6 for directions on cleaning.
- 2) The chamber mirror may be dirty. Refer to Chapter 6 for directions on cleaning.

4.PROBLEM:

Message on screen displays the following:

**SOLUTION:**

Your sample's temperature is too high for the instrument to equilibrate with it in a reasonable amount of time. The instrument and sample need to be in temperature equilibrium before

accurate measurements can be made. Therefore, very cold samples will take a very long time to measure for the same reason. To avoid this problem, make sure to only measure samples that are at the same temperature as the instrument.

5.PROBLEM:

Message on screen displays the following:

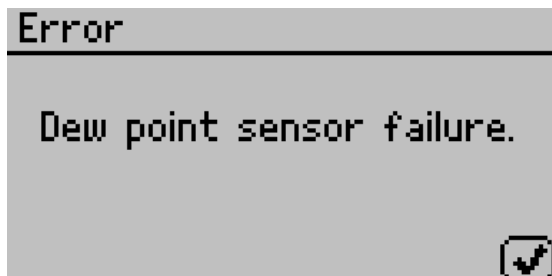


SOLUTIONS:

- 1) The sample is too dry for the instrument to read accurately. If your sample has a water activity that is less than below the detection limits of the instrument, this message will come up. Essentially, it means that there is not enough sample moisture to condense on the mirror and provide a reading.
- 2) The mirror may be dirty. Try cleaning the mirror and chamber and measuring the sample again.

6.PROBLEM:

Message on screen displaying dew point sensor failure.



SOLUTION:

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

7.PROBLEM

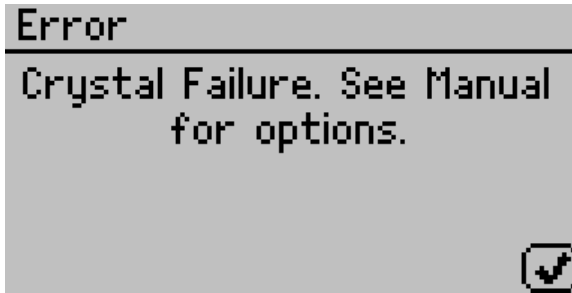
Verification is not correct.

SOLUTIONS:

- 1) The sample chamber and mirror need to be cleaned. See Chapter 6 for detailed cleaning instructions. If verification is still not correct, then a linear offset has occurred.
- 2) Verify and Adjust for Linear Offset. After you have cleaned the sample chamber and mirror (Chapter 7) you will need to use a Verification Standard to verify and adjust for Linear Offset as described in Chapter 7.

8.PROBLEM:

Message on screen displays the following:

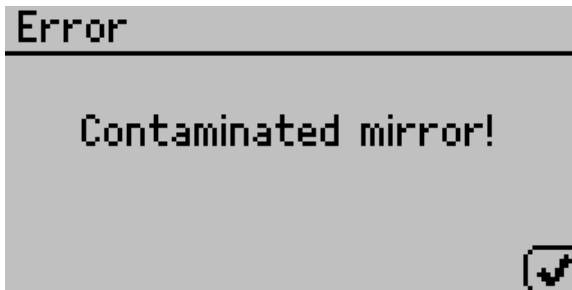


SOLUTION:

The crystal that runs the firmware is having trouble starting. Occasionally, cycling the power will solve the problem. If this message continues to appear, the instrument will need to be serviced by Decagon. See Chapter 13 for detailed instructions.

9.PROBLEM:

Message on the screen displays the following:

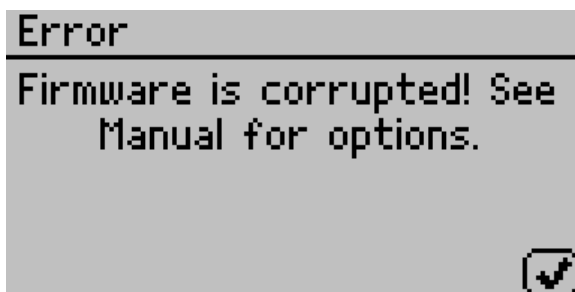


SOLUTION:

The mirror used for dew point measurements requires cleaning. Follow the instructions outlined in Chapter 6: Cleaning and Maintenance before trying to run your sample again. If this message continues to appear, contact your Decagon for further options.

10.PROBLEM:

Message on the screen displays the following:

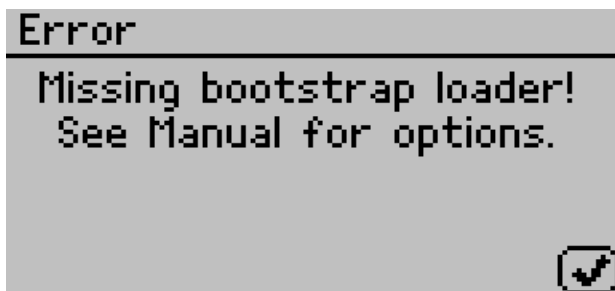


SOLUTION:

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

11.PROBLEM:

Message on the screen displays the following:



SOLUTION:

The instrument cannot download new firmware updates. To download new firmware to the VSA, or to stop this message from displaying, the instrument must be serviced by Decagon.

12. PROBLEM:

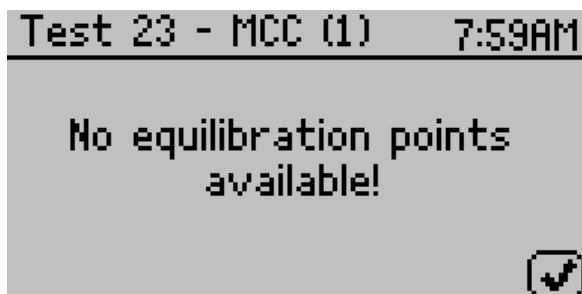
Message on the screen displays the following:

**SOLUTION:**

This message indicates that it's been at least a year since the instrument has been calibrated and serviced by Decagon or your local distributor. It is recommended that you return your instrument so that we can help you ensure you are getting the most accurate readings possible from your VSA. Click remind me later to be reminded in a couple of weeks, click Don't display again if you don't want to be reminded in the near future.

13.PROBLEM:

Message on the screen displays the following:



SOLUTION:

When graphing a test, this message indicates that there are no data points in which the weight has stabilized. The time it takes for an equilibration point to appear depends on the sample phase changes and stage trigger/ timeout specifications. To view detailed data other than equilibration points, press the up or down arrow.

14.PROBLEM:

Message on the screen indicates that a desiccant tube is low.

SOLUTION:

During a test, if the VSA determines that a desiccant tube is exhausted its drying capacity, the VSA will automatically switch to the other desiccant tube. When this event occurs, a message appears indicating that the exhausted desiccant tube needs to be replaced. See Chapter 4 for information on replacing the desiccant tube.

15.PROBLEM:

Screen displays a Low or High System Pressure Warning

SOLUTION:

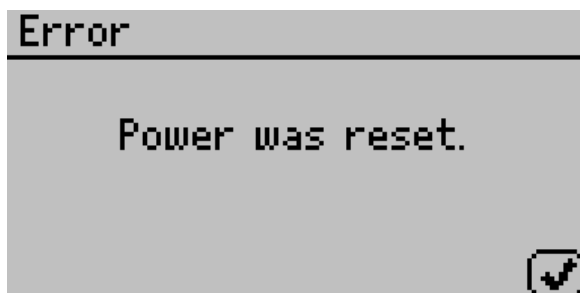
Anytime the VSA instrument requires air flow, it monitors the system pressure and sends a warning if the system pressure is outside the expected range for the current scenario. This may be caused by one of the following issues:

- 1) External gas is flowing when the external gas setting is off.
- 2) External gas is not flowing when external gas setting is on.
- 3) External gas pressure is outside the recommended range for running a test.
- 4) External gas input plug is not properly installed or missing when external gas is off.
- 5) Both Desiccant tubes are not properly installed or missing.
- 6) Water chamber plug is not properly installed or missing.
- 7) Pump system is failing

If you continue to receive a message after the above items have been checked, contact Decagon for further options.

16.PROBLEM:

Message on the screen displays the following:



SOLUTION:

Anytime during a test if the VSA instrument is reset due to a power outage or due to unforeseen circumstances, this message will appear on the screen. If Auto Restart is turned on, the test will automatically resume after a device reset. If Auto Restart is turned off, then the test will stop running when a device reset occurs. To prevent device resets, it's strongly recommended that you connect your VSA to a UPS.

17.PROBLEM:

Screen indicates a Scale failure, overload or underload

SOLUTION:

If the VSA is unable to communicate with the scale, the scale failure message will appear. If the scale is reading overload or underload, the balance is either misaligned or broken. If any of these instances occur the VSA will need to be serviced by Decagon.

18.PROBLEM:

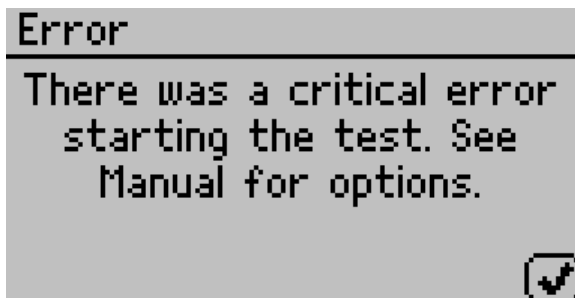
Screen displays "Actuator failure."

SOLUTION:

An actuator failure is caused when the air cylinder is unable to actuate. This may be caused by a dirty chamber, a faulty or damaged air cylinder, a misaligned scale shaft or a pump assembly problem. Clean the chamber including the air cylinder, the weighing pan, and the space beneath the air cylinder. If you continue to have problems, contact Decagon.

19.PROBLEM:

Message on the screen displays the following:



SOLUTION:

Verify that the following has been done before starting a test:

- The sample weight is at least 50 milligrams.
- The test must have a name.
- The test must contain at least one stage.

If you continue to receive this error, contact Decagon.

20.PROBLEM:

Can't communicate with the VSA

SOLUTIONS:

- 1) Usb cable is not plugged in.
- 2) Wrong com port chosen.
- 3) Restart VSA.
- 4) Restart communication software.

21. PROBLEM:

Weight is decreasing during absorption.

SOLUTIONS:

- 1) Water tank has run dry.
- 2) Plug on fill tank is loose.
- 3) Pump is not working.
- 4) Material is undergoing a phase change.
- 5) Scale needs to be checked for calibration.

22. PROBLEM:

Weight is increasing during desorption

SOLUTIONS:

- 1) Desiccant cartridge has been used up.
- 2) Pump is not working.
- 3) Desiccant tube/tubes leaking.
- 4) Scale needs to be checked for calibration.

23. PROBLEM:

Isotherm test has stopped prematurely.

SOLUTIONS:

- 1) Download the test with the VSA downloader.
- 2) Navigate to the Table view.
- 3) Scroll down to the last data record in the list.
- 4) Look in the special condition column to see what caused the test to stop prematurely.
- 5) Refer to the troubleshooting section that references the special condition indicated.

24. PROBLEM:

Isotherm test is taking an unusually long time to complete, even at high flow rates.

SOLUTIONS:

- 1) Sample has very slow sorption properties.
- 2) Water has run out – fill.
- 3) Desiccant is used up – replace.
- 4) Increase the flow rate.

25. PROBLEM:

Scale readings are not stable or the sample cups cannot be tared.

SOLUTIONS:

- 1) Unstable bench or support for the VSA.
- 2) Internal parts need to be cleaned. Lift out air cylinder and clean.
- 3) Interior parts have shifted and are contacting so weight cylinder will not slide.

26. PROBLEM:

Scale calibration is failing.

SOLUTIONS:

- 1) Air cylinder and internal parts need to be cleaned.
- 2) Air cylinder is not actuating. May be sticking due to contamination in chamber.

27. PROBLEM:

Desiccant cartridges hard to install.

SOLUTIONS:

Dampen the o-rings on the desiccant cartridge using water before installing

28. PROBLEM:

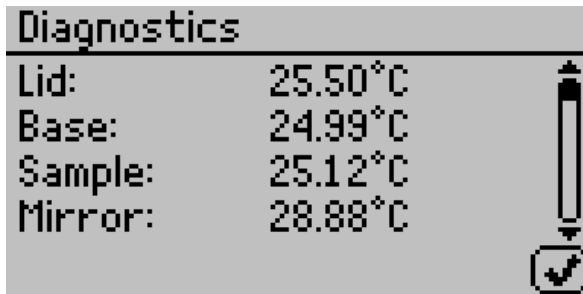
Poor temperature control on the system.

SOLUTIONS:

- 1) Clean the filters on the enclosure fans.
- 2) Check that external fan on the back of the unit is operational.

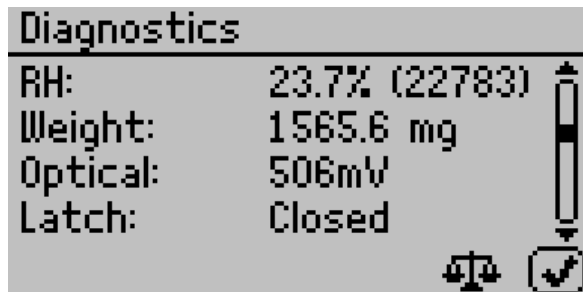
Diagnostic Screen

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



This screen shows typical values for the temperature sensors on the instrument. Lid, base and sample temperatures may fluctuate but should not change more than 0.03 degrees. Typical ranges for the lid, base and sample temperatures are between 24.5 and 25.5 degrees.

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

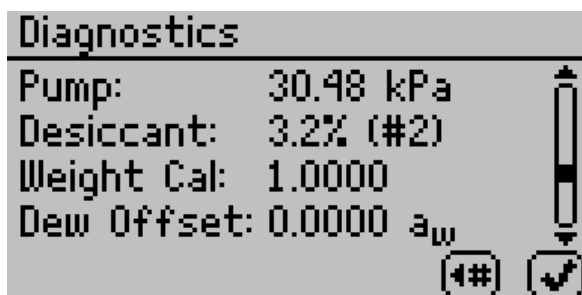


RH: This screen shows the capacitive sensor's RH percentage which should always be between 0 and 100%.

Weight: The weight measurement is updated in real-time and can be zeroed for measurements by pressing the weight icon (2nd button from the left). If the weight is reading overload or underload, the balance is broken and needs to be replaced. A negative value indicates that the balance needs to be re-zeroed if you want to take weight measurements.

Optical: A typical optical range is between 500 mV -- 2900 mV.

Latch: To determine if the latch is functioning properly, open and close the latch while monitoring the latch status on the screen. If the screen doesn't update the latch status when you open and close the latch, the latch magnet may need to be re-seated (with the correct polarity) or the Hall Effect sensor needs to be realigned with the magnet.

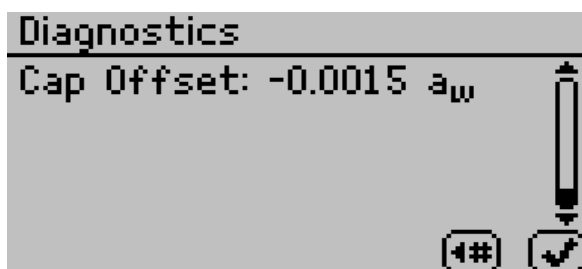


Pump: The pump pressure is only valid when the pump is active. When the pump is off, the pressure should be 0 kPa. This pressure reading also applies to an auxiliary air supply. When the air supply is active the pressure in the system shouldn't exceed 45 kPa. If the pump pressure continually reads zero (even when you press the switch desiccant button) then the pump system may need to be serviced.

Desiccant: The Desiccant humidity value indicates the effectiveness of the desiccant tubes. Initially when the diagnostic screen is turned on, the pump supplies flow through the active desiccant tube to help verify that it's able to desiccate properly. After 30 seconds of pumping, the desiccant humidity reading should be 5% or less. If the desiccant humidity is unable to reach 5% humidity, refer to Chapter 7 for instructions on replacing the desiccant tubes.

Weight Cal: The weight calibration saved a scalar into memory that's applied to all subsequent weight readings. A scalar of 1 means that the weight hasn't been changed from it's default factory calibration. If the scalar strays too far from 1, you may need to reverify the balance and also recertify the weight standard.

Dew Offset: The chilled mirror dew point calibration offset is zero if it's running off of the default factory calibration. This value indicates how large an offset was needed to be applied to the water activity readings inorder to read correctly.



Cap Offset: The capacitive RH calibration offset is zero if it's running off of the default factory calibration. This value indicates how large an offset was needed to be applied to the water activity readings inorder to read correctly.

Appendix A

Temperature Correction of Decagon's Verification Standards

Temp. (°C)	H ₂ O	0.5m KCL	2.33 m NaCl	6.0m NaCl	8.57m LiCl	13.4m LiCl
15.0	1.000	0.984	0.923	0.761	0.492	0.238
20.0	1.000	0.984	0.922	0.760	0.496	0.245
25.0	1.000	0.984	0.920	0.760	0.500	0.250
30.0	1.000	0.984	0.920	0.760	0.504	0.255
35.0	1.000	0.984	0.920	0.760	0.508	0.261
40.0	1.000	0.984	0.921	0.760	0.512	0.266

The Aqualab Vapor Sorption Analyzer will measure these standards to $\pm 0.005 a_w$.

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