AquaSorp

Isotherm Generator

Moisture Sorption Isotherm Generator

Operator's Manual

Version 3.0

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1. Introduction

Welcome to Decagon's AquaSorp Isotherm Generator, an automatic isotherm generator from the world leaders in water activity measurement. The AquaSorp is the only automatic isotherm generator that utilizes the Dynamic Dewpoint Isotherm (DDI) method. This revolutionary method makes it possible to generate complete isotherms with hundreds of data points quickly and accurately. We hope you find this manual informative and helpful in understanding how to maximize the capabilities of your AquaSorp.

About this Manual

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

Customer Service

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

Phone/Fax
Toll-Free: (US, Canada Only) 1-800-755-2751
Tel: (509) 332-2756
Fax: (509) 332-5158
E-mail: support@decagon.com. Please include your serial number, a contact name, phone number and address with a description of your problem.

Warranty

The AquaSorp has a 30-day satisfaction guarantee and a one year warranty on parts and labor. To validate your warranty, please complete and return your warranty card included with this manual, or register online at http://www. decagon.com/aw/aquasorp_registration. You can return your warranty information by fax, e-mail, or phone. Please include all of the requested information so we may better assist you with future needs. It is important for Decagon to have your current mailing address and telephone number in case we need to send updated product information to you.

Note to Our AquaSorp 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); but Seller's liability for defective parts shall in no event exceed the furnishing of replacement parts F.O.B. the factory where originally manufactured. Material and equipment covered hereby which is not manufactured by Seller shall be covered only by the warranty of its manufacturer.

Seller shall not be 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 AquaSorp

The AquaSorp Isotherm Generator is an automatic moisture sorption isotherm generator which rapidly creates detailed adsorption and desorption isotherm curves.

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:

- Monolayer moisture content determination

- Determine critical water activity or moisture content limits for crispness, hardness, and flow properties.

- Optimize moisture contents at a safe water activity that maximizes moisture and avoids over drying.

- Determine shelf-life and storage stability of a product.

- Predict packaging requirements based on sorption properties of a product.

- Determine the equilibrium water activity of a mixture of dry ingredients.

- Determine the degree of crystallinity of powders.

- Determine the level of amorphous material in a product.

- Determine critical water activities for phase transitions.

- Determine the relationship between water activity and glass transition temperature.

- Determine the relationship between water activity and crystallization.

- Determine hysteresis levels for a product.

- Determine the moisture sensitivity of a product.

- Determine the equilibrium moisture content at a given water activity.

- Allow rapid moisture content determination from water activity analysis through isotherm curve.

Measurement Method

The AquaSorp creates isotherms using a water activity and gravimetric analysis method called Dynamic Dewpoint Isotherm (DDI). The AquaSorp 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 an integral part of the measurement chamber to ensure humidity saturation and minimize temperature fluctuation. The AquaSorp 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. The integrated air pump eliminates the need for gas cylinders. This allows the Aqua-Sorp to generate robust isotherms with hundreds of data points much faster than other isotherm methods because the sample does not have to equilibrate to a known humidity level.

Limitations

The AquaSorp may not be able to analyze samples with high concentrations of certain volatiles like propylene glycol or ethanol. Specific volatile materials can interfere with dewpoint measurements by chilled-mirror sensors. The AquaSorp uses a chilled mirror sensor to measure water

activity. Not all volatiles or concentrations will be a problem, but it is important to note that the presence of some volatiles could influence the accuracy of the isotherm.

Finally, the AquaSorp sample chamber can have some humidity memory and it may be a good idea to precondition the chamber, especially when running low moisture capacity samples (such as crystalline or glassy samples).

Specifications
Water Activity Range: 0.03 to 0.95 a_w
Water Activity Accuracy: ±0.005 a_w
Water Activity Repeatability: ±0.003 a_w
Temperature Control Range: 15° to 40° C
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): 42.5 x 36.2 x 25.4cm
                16.75 x 14.25 x 10in. (L x W x H)
Weight: 42 pounds
Weight Accuracy: ± 0.1mg

3. Theory

Moisture sorption isotherms describe the relationship be-
tween water activity and moisture content at a specified
temperature. The nature of this relationship depends on the
interaction between water and other ingredients. Conse-
quently, the isotherm shape is unique to each product and
products with the same water activities may have different
moisture contents depending on capillary, surface, and col-
ligative effects. Products that lie in the low water activity,
<0.60 a_w, 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 a_w are high moisture products.

For ease of interpretation, isotherms are often classified as
one of three types (Figure 1).

• Type I isotherms are typical of anti-caking agents.
These types of ingredients absorb water onto polar sites
and into non-swelling capillaries, which results in high
amounts of moisture being held at low water activities.
When all these sites are filled up, further increases in
moisture content results in large changes in water activ-
ity.

• Type II isotherms describes most types of products.
The isotherms shape for these types of products is sig-
moidal, characterized by sharp changes in moisture con-
tent at low and high water activities, but small changes in
moisture content over the intermediate moisture range.

•   Type III isotherms are typical of crystalline sub-stances. For this type of isotherm, there is very little moisture gain initially because water is only interacting with the surface of the crystal through hydrogen bonds. Increasing the surface area of the crystal will increase the moisture content at low water activities. Eventually, as water activity increases, the water will dissolve the crystal (often called deliquescence). At this point, the moisture content starts to increase dramatically as the material goes into solution.

Moisture Sorption Isotherm Types
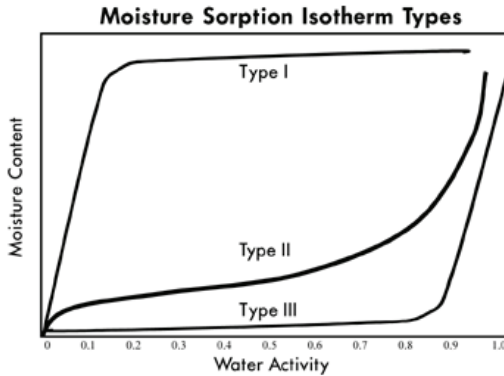
Figure 1. Brunauer classification of moisture sorption iso-therms: Type I = anti-caking agents, Type II = most foods, Type III = crystalline substances.

Constructing an isotherm consists of collecting water ac-tivity 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. Most isotherm methods consist of con-trolling water activity levels using saturated salt slurries,

acid solutions, glycerol solutions, or mechanical humidifiers. Equilibrium moisture contents are then determined at each water activity level. Equilibrium is determined based on 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. Automatic isotherm generators use the same principle, but track weight electronically and dynamically change the water activity levels once equilibrium is achieved. Decagon's AquaSorp Isotherm Generator uses the DDI method, which is discussed in detail below.

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 phenomena 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.
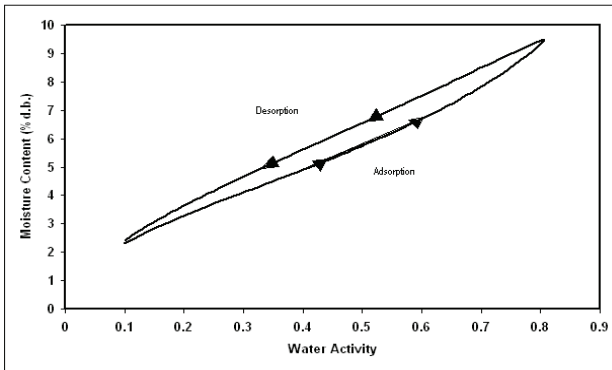
Figure 2. Full isotherm showing hysteresis.
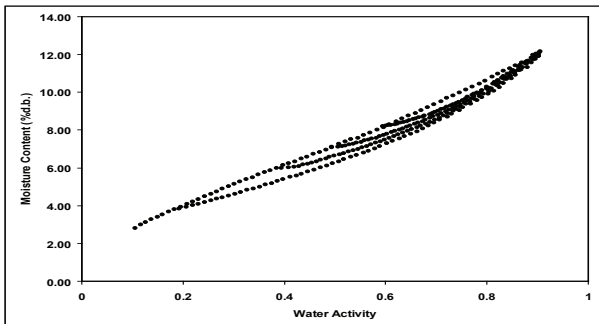
Figure 3. Scanning adsorption curves resulting from drying to different water activities.

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 proper-

ties 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 sample, these theories explain why the water content of a desorption process is greater than that for a wetting process.

• 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.

• 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.

• Structural changes within a solid matrix in which 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 some polar sites unavailable for water 'binding' prior to adsorption.

• Supersaturation. 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

It is possible to produce adsorption-desorption curves that appear to show hysteresis, but are just the result of not waiting long enough for equilibrium. Figure 4 shows two AquaSorp runs, one with a high flow rate and one with a low flow rate. Note that the apparent hysteresis is much worse at the high flow rate. A flow rate needs to be chosen such that further reductions in flow rate do not reduce the size of the hysteresis loop.
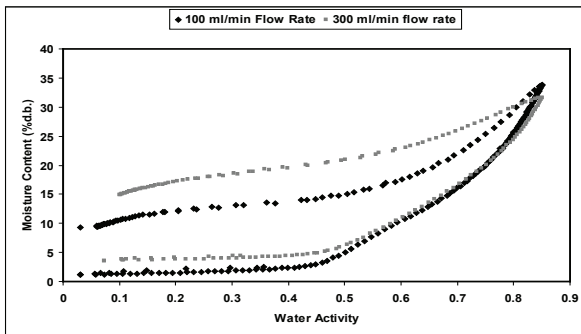

Figure 4. Changes in hysteresis levels when flow rate of wet and dry air is reduced from 300 ml/min to 100 ml/min.

Matrix changes

Figure 5 shows several cycles of an isotherm obtained on a sample of rice cereal. Note that the first wetting branch is substantially different from all subsequent branches. This could be termed hysteresis, since the path depends on the

wetting history of the sample, but the path can't be repeated. Something about the sample changed during the first wetting cycle, and is not changed back by drying, no matter how many drying cycles occur.

The water in the sample is "bound" to particle surfaces by various bonding mechanisms. When the configuration of the surface changes, possibly by conversion from glassy to crystalline form, or rearrangement of molecular structures, the binding sites change and the amount of water which can be "bound" at a given energy changes. Once these changes occur the wetting and drying paths coincide for as many cycles as one wants to make. These curves show what should be more properly called hysteresis.
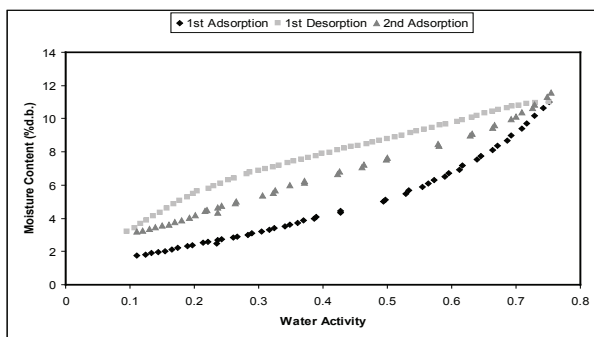

Figure 5. A moisture sorption isotherm curve showing a change in hysteresis due to a phase change during the 1st adsorption causing the 2nd adsorption curve to not match the 1st adsorption curve.

Working Isotherms

In some cases, it is desirable to determine how a product will adsorb or desorb water from its current condition. This is referred to as the working isotherm and is determined by wetting and drying the product from its current state. The isotherm 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 period can be observed at any point on the isotherm if the direction of the sorption is changed and the product exhibits hysteresis.

NOTE: Please refer to Chapter 7 for information about running a working Isotherm using the AquaSorp.

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. It will also show if a product exhibits hysteresis what impact that will have on the moisture content after drying to a given water activity.

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.

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. 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. A new model called the Double Log Polynomial (DLP) has proven to be even better than the GAB at characterizing complex isotherms. SorpTrac data analysis provides the coefficients for the BET, GAB, and DLP. The model equations are shown below.

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

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 same units. The constant (c) is calculated by: $c = e^{\frac{\Delta H}{R\,T}}$ Where (ΔH) is the surface interaction energy cal/mole, R is the gas constant and T is the Kelvin temperature .

$$\text{GAB} \quad m = \frac{m_o k_b C_1 a_w}{[(1 - k_b a_w)(1 - k_b a_w + C_1 k_b a_w)]}$$

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_1) is a constant in the range of 1 to 2000. In addition, (m_o) is the mono-layer water content in the same units as (m) and (a_w) is the water activity at moisture (m).

$$\text{DLP} \quad m = b_3 x3 + b_2 x2 + b_1 x + b_0$$

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

The DDI Isotherm Method Compared to Other Methods

The Dynamic Dewpoint Method(DDI) used by the Aqua-Sorp is a unique way of obtaining isotherms. Tradition-al isotherm methods depend on the equilibration of the sample to known water activities and then measuring the

equilibrium moisture content of the sample. This is most easily done by placing the sample in a sealed chamber over a saturated salt slurry in excess. Different water activity levels are achieved by using different salts. Adjusting a mixture of wet and dry air while monitoring the water activity with a sensor can also be used to control water activity. Different water activity levels are achieved by changing the levels of dry or wet air. Some instruments are programmed to automatically change the water activity in a dynamic stepwise progression. The sample is held at each water activity level until weight stops changing before moving to the next water activity. Common to all these isotherm methods is the dependence on equilibration to a known water activity level to determine each data point's water activity. Since true equilibration between the sample and the vapor source requires an infinitely long period of time, an apparent equilibrium when weight stops changing by a tolerable level is used. Increasing the tolerable weight change will speed up the isotherm process but calls into question the validity of the water activity values.

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. In addition, only water and desiccant are needed to run the isotherm.

The dynamic nature of the DDI method can present problems when trying to compare isotherms by DDI to isotherms created using other isotherm methods, especially isopiestic methods where equilibration times can be weeks. For most sample types, especially samples with fast vapor diffusion, penetration by water vapor into the whole sample is rapid and isotherms from DDI for these types of products will be comparable to other 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 adsorbed 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 isotherms constructed using other methods, resulting in higher levels of apparent hysteresis. Better agreement to other isotherms may be achieved, when using the DDI method, by reducing the sample size and lowering the wet or dry air flow rate to allow more moisture penetration into slow diffusing samples.

Since the DDI method, other automated isotherm generators, and traditional isopiestic methods can achieve different matrix states, none of which may be true equilibrium states. It is impossible to say that one method is better than another. The dynamic nature of the DDI method

may actually give a more correct picture of the sorption characteristics of a product in real conditions since samples are rarely exposed to changes in moisture in stepwise progression but instead in a dynamic progression. In addition, time dependent physical changes that can occur as a product equilibrates at different water activity levels over weeks may not occur in production situations where exposure to different moisture levels is dynamic. These physical changes may cause isotherms determined using traditional methods to be much different than those made using the DDI method.

The DDI method is the best method for tracking the sorption characteristics of a sample through the full isotherm since the same sample can be subjected to multiple adsorption and desorption events in succession across any water activity range. The resolution of the method eliminates the need for extrapolation and gives a detailed view of sorption events. A limitation of the DDI method is that it can not be used to determine kinetics of sorption at different water activity levels.

4. Getting Started

Components of your AquaSorp

The AquaSorp 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 AquaSorp is shipped with the following items:

- The AquaSorp Isotherm Generator Main Unit
- PC Computer with Monitor
- The SorpTrac Software Installation Disc
- Operators Manual
- Calibration Certificate / MSDS Certificates
- Power Cord
- RS-232 Interface Cable
- 5 Stainless Steel Sample Cups
- 2 Refillable Desiccant Tubes
- 1 Decagon Cleaning Kit
- 1 Water Bottle
- 3 Vials each of the following verification solution
    - 1.000 a_w steam distilled water
    - 0.760 a_w 6.0 molal NaCl
    - 0.500 a_w 8.57 molal LiCl
    - 0.250 a_w 13.41 molal LiCl
- 1 Phillips Screwdriver.
- 1 Flathead Screwdriver.
- 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.

The AquaSorp Isotherm Generator Essentials

Desiccant Tube

The desiccant tube provides dry air to the sample chamber and is critical to correct operation of the instrument. It needs to be maintained with blue desiccant to provide dry air to the instrument. For instructions on changing the desiccant or desiccant tube, see Step 2 of "Starting a new Test" in Chapter 5.

Message Center

The message center on the front of the instrument consists of three LED lights. Each light provides information about the AquaSorp depending on which one is lit and if it is solid or blinking.

| COLOR	STATE	MESSAGE
White	Solid	Power is on but the test is not running
White	Blinking	The Instrument and balance are thermally equilibrating.
Blue	Solid	A Test is running

4. Getting Started

Blue	Blinking	The Test is in pause mode and if not resumed in 3 minutes the Test will stop.
Red	Solid	A fatal error has occurred and the test was stopped.
Red	Blinking	An unusual condition has occurred and may need attention.
White/Blue/Red	Blinking in succession	Water Activity verification in process.

Sample Chamber

The sample chamber is located on top of the instrument under the lid, which is opened and closed using two thumbscrews. Turn both thumbscrews to the right to open and to the left to tighten. When closing the lid, it is important to tighten the screws down completely to ensure a good seal. Make sure you tighten the screws evenly or the lid could seat unevenly.

With the lid open and looking down at the instrument, the sample chamber can be seen on the left and the water chamber on the right. The water chamber provides saturated air to the sample chamber and needs to be kept filled between the two lines. Inside the sample chamber is the weighing platform, which is connected to the precision balance. The chilled-mirror dew-point sensor, which

is used to measure sample water activity, is in the lid and can be viewed by looking under the lid when in the open position.
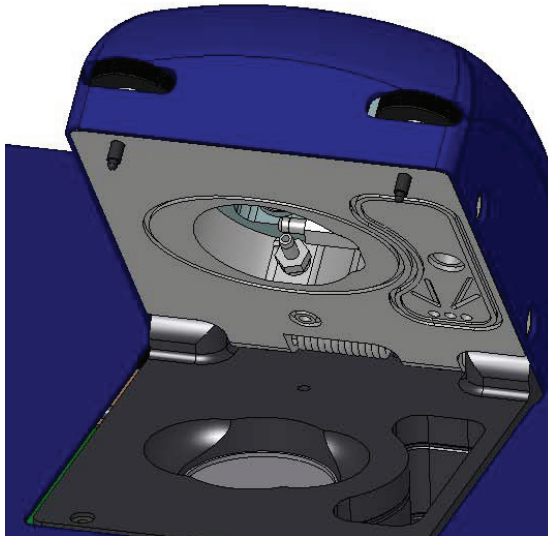
Preparing for Operation

Choosing a Location

To ensure that your AquaSorp operates correctly and consistently, place it on a level surface. There must be adequate room to house the AquaSorp and computer used to operate the AquaSorp. The instrument must be completely level to ensure that the precision balance works properly. To level the instrument, adjust the three feet until the bubble on the front of the instrument is centered. To protect the internal electrical components, and to avoid inaccurate read-

ings, place your AquaSorp in a location where the temperature remains stable. This location should be well away from air conditioner and heater vents, open windows, outside doors, refrigerator exhausts, or other items that may cause rapid temperature fluctuation. After finding a good location for the AquaSorp, plug the power cord into the back of the unit.

## Setting up and Connecting to the Computer

Set up the computer next to the AquaSorp according to the Dell instructions. Connect the computer to the Aqua-Sorp through the serial port on the back of the instrument with the supplied RS232 serial cable. The use of an uninterrupted power supply, or UPS, is recommended to avoid data loss.

The computer is pre-loaded with the SorpTrac software used to operate the AquaSorp. Turn on both the computer and the AquaSorp by pressing the power switches. The power switch on the AquaSorp is located next to the power cord on the back of the instrument. The AquaSorp requires a 60 minute temperature equilibration period after turning it on or following a power outage. Although you can still do data analysis during this equilibration, no test can be started until after it has completed temperature equilibration. The AquaSorp can be left on continually without harmful effects.

Setting up the Desiccant Tube

The desiccant tube must be loaded in the recessed area on the front of the machine before testing can begin. To load the tube, insert one black cone-shaped end into the left

inlet point on the front of the machine. This inlet is spring loaded and pressing the tube to the left will allow room to insert the right side of the tube into the right inlet. Make sure that both ends are seated in the inlets snugly.
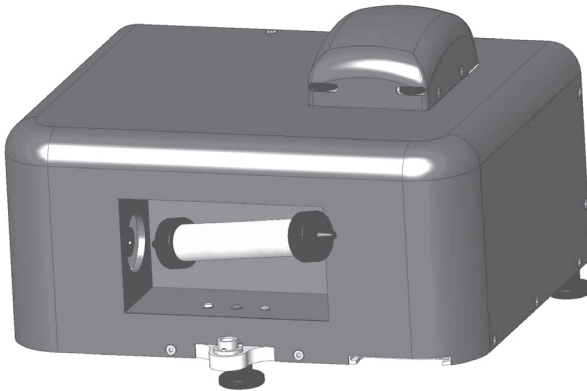
Preparing the Sample Chamber

The AquaSorp is shipped with a foam insert in the sample chamber to protect the chamber and the balance. This foam insert must be removed before a test can be performed. In addition, the weighing pan must be inserted into the chamber. To open the chamber and remove the insert, loosen the thumbscrews to the right and open the lid. Remove the foam insert and discard. Find the small round weighing pan and insert it into the bottom of the sample chamber. It should drop easily into the hole in the bottom of the chamber. The weighing pan should be located with the other components in the original packaging.

Setting up the Water Chamber

Water must be added to the water chamber before testing can begin and the chamber should be refilled when the water level falls below the bottom fill line. To fill the chamber, find the water bottle that was shipped with the AquaSorp. Fill the bottle with distilled or deionized water. Use the bottle to fill the water chamber to the top fill line.

5. SorpTrac Software

Overview

The AquaSorp uses the SorpTrac software to setup, collect and analyze test data. To run the software, go to Start > Programs > Decagon, and click on SorpTrac or double click the SorpTrac icon on the desktop. At the top of the screen are tools for connecting to the AquaSorp and for setting SorpTrac preferences. The "Connect Via" drop down box allows you to choose which communication port you will use to connect to the AquaSorp and the "Visible Data" drop down box allows you to change the current view from chart alone, chart and table, or table alone. At the top right of the screen is a current test status bar. In the center of the screen you will see a blank chart on the left and a blank table on the right.

As data is downloaded from the AquaSorp, the data points will be loaded into the table and displayed in the chart. At the bottom of the screen is a status bar that shows the isotherm settings for the current test.

A Closer Look at SorpTrac

The File Menu
The File menu consists of Export, Save Experiment, and Exit.

Export
The Export menu consists of two options for getting data out of SorpTrac.

Chart to Graphic
SorpTrac allows you to export your chart as a graphic bitmap (BMP) file. This can then be placed in other programs that allow the use of image files.

Table

Exports the current table in the Excel format (CSV). This file can be directly opened in Excel for further analysis or additional graphing.

Save Experiment

Saves the current experiment. This file can be opened in the Data Analysis Tool.

Exit

Closes the SorpTrac software.

The Actions Menu

The Actions menu consists of Connect/Disconnect (this menu item/button will change depending on the status of the instrument), New Test, Stop Test, Modify Test, Set Temp., Equilibration Clock, Download, and AquaSorp Information.

Connect/Disconnect

Selecting this will disconnect the AquaSorp from the SorpTrac software. This can also be done using the Connect/Disconnect button beneath the menus.

New Test

Starts a new test and opens up the test setup wizard.

Stop Test

Stops the current test.

Modify Test

This allows you to make changes to an experiment

that is running. Further information on what can be modified can be found in this chapter under the Modify Test section.

Set Temp.
Sets the desired test temperature of the AquaSorp.

Equilibration Clock
Displays the amount of time until the temperature equilibration is complete.

Download
Downloads the data points from the previous test.

NOTE: This option is only available before a new test is started. Starting a new test will erase previously acquired data points from the AquaSorp.

AquaSorp Information
Displays the Serial number, firmware version, etc. of the AquaSorp.
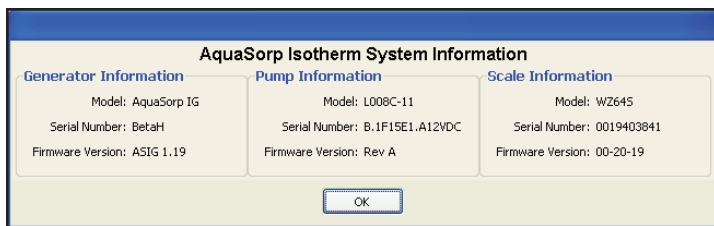
The Device Tools Menu
The Device Tools menu consists of Sync Time, Verification and Restore, and Erase Test.

Sync Time
This option will synchronize the time between the AquaSorp and your computer clock.

Verification and Restore
This menu option allows you to select the following calibration options: Water Activity Verification, Restore Factory Water Activity Calibration, Balance Verification, and Restore Factory Balance Calibration.

Water Activity Verification
This is used to verify the calibration of the chilled-mirror dewpoint sensor. Selecting this option will start a verification wizard that will guide you through the steps necessary to verify the calibration of the sensor. See Chapter 8 for more information about verifying the water activity.

Restore Factory Water Activity Calibration
This option will set the aater activity calibration back to the original factory settings.

NOTE: Clicking on "Yes" will restore the water activity calibration values to factory settings and cannot be undone.

Balance Verification
This is used to verify the calibration of the magnetic force balance. You will need the supplied 2g NIST traceable weight. SorpTrac will check the balance and ask if you want to offset the

measured weight to the actual weight See Chapter 8 for more information about verifying balance performance.

Restore Factory Balance Calibration
This resets the balance to the factory settings. A window will open asking if you want to make the change.

NOTE: Clicking on "Yes" will reset the balance to factory settings and cannot be undone.

Erase Test
This option erases the current test and all associated data. Make sure you save the data before selecting this option or all data will be lost.

The SorpTrac Tools Menu

The SorpTrac Tools Menu provides three drop-down menus: Data Analysis, Multiple Isotherm Analysis and Preferences.

Data Analysis
This menu option opens a new window for analyzing your data. A further description of Data Analysis is found in Chapter 7.

Multiple Isotherm Analysis
This menu option opens a window for analyzing multiple isotherms. This function is used to combine isotherm curves for comparison or to analyze work-

ing isotherms. More information about Multiple Isotherm Analysis is found in Chapter 7.

Preferences
The Preferences Menu opens a window with three tabs to customize SorpTrac. The tabs are General Program Options, Chart View Options, and Communications Options.

General Program Options
This menu option gives you general choices for customizing SorpTrac such as update options, clock options, data save locations, date/time format, etc.

Automatic Internet Version Check
This option allows SorpTrac to check Decagon's web site for updates to the SorpTrac program. You must have an internet connection and be connected to the web to use the automatic version checker.

Automatic Clock Synchronized to PC clock
This option synchronizes the AquaSorp's clock to the clock on your computer.

Date and Time Display in SorpTrac
Provides options for displaying the date and time.

Power Outage Restart
By default this box will not be checked and an isotherm test will stop if a power outage occurs. To make an isotherm test automatically resume, check the box.

NOTE: It is recommended that restart delay be left unchecked because power outages result in temperature fluctuation and this invalidates the isotherm experiment.

Location to Auto-Save Data

This option tells the SorpTrac where you want the data to be placed during an Auto-Save.

Chart View Options

This tab includes options for setting up the chart view in SorpTrac. Click on the "Chart View Options" tab to open this window.

Moisture Content Display: Wet or Dry Basis

This options allows you to set the method of display for the moisture content of the sample. Dry basis (d.b.) is the amount of water in the sample divided by the amount of dry material. Wet basis (w.b.) is the amount of water divided by the amount of wet material.

Clipboard Format

This option sets the format for pasting data into the Windows clipboard. You can choose between the Enhanced Meta (emf) format or a Bitmap (bmp) image format.

Moisture Content Displayed During Test Monitoring

This option will be selected by default if a known moisture content is entered during test setup. It will enable moisture content values to be shown during data collection instead of the weight values. If a known moisture content is not entered at setup, this option will not be selected and weights will be shown on the data collection screen.
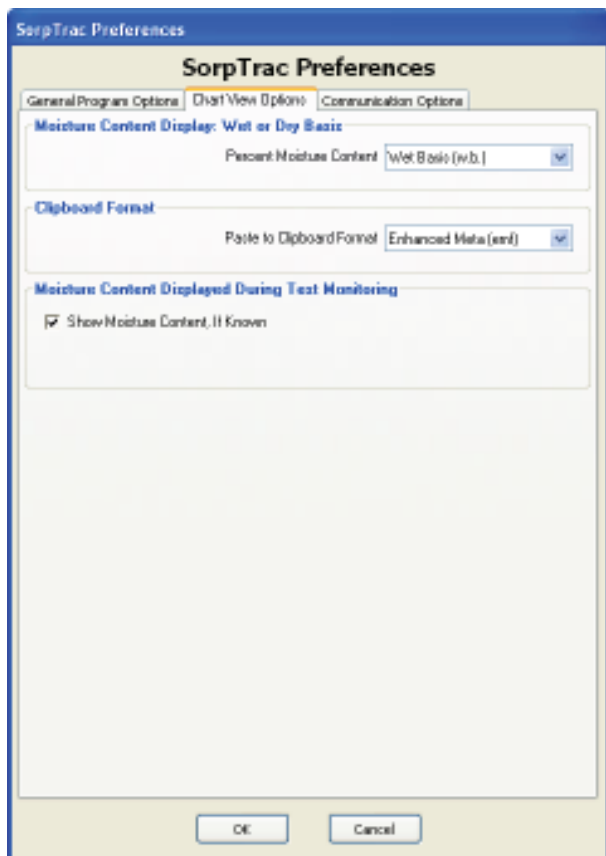
Communications Options

The communications tab gives you options for changing the way SorpTrac communicates with the AquaSorp. Click on the "Communication Option" tab to open this window.

NOTE: Only change these settings if you are having trouble communicating with the AquaSorp.

Communication Ports List

This option allows SorpTrac to force find all comm ports. Activating this option may give you ports that are unavailable to SorpTrac. This is a useful option if your USB to serial adapter doesn't appear in the "Connect Via" communication port list.

Device Commands Retries

Sets the number of times SorpTrac will try to connect to the AquaSorp. The default is 3 times before a communication error dialog box will appear.
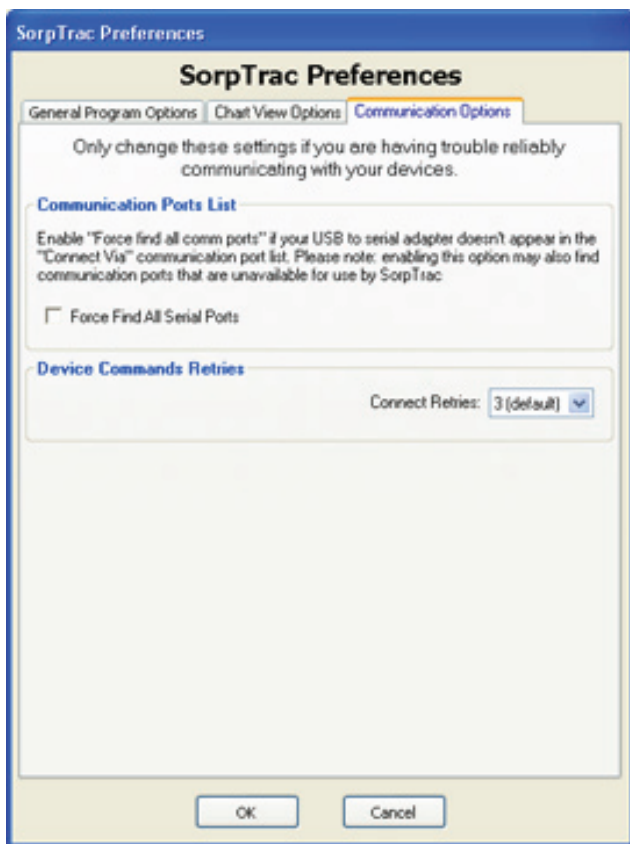
The Window Menu Option

This menu option is available after you have opened at least one Data Analysis Window via the SorpTrac Tools menu. You can open up to three Data Analysis Windows and this allows you to quickly switch between them.

The Help Menu Option

The Help menu option has one menu called "About Sorp-Trac" that will give you information about the SorpTrac software. Click anywhere within the window to close.

6. Running a Test

It is important to verify that the AquaSorp is performing correctly before starting a test using Decagon's Verification Standards. Please refer to Chapter 8 for instructions on how to verify your AquaSorp and adjust the calibrations if necessary.

Connect to the AquaSorp

SorpTrac software must be connected to the Aqua-Sorp to start a test. To connect, press the 'Connect' button at the top of the screen or go to Actions > Connect. If a previous isotherm has been run on the Aqua-Sorp and the data is still stored in the AquaSorp, a dialogue box will ask if the user wants to download the data. If this data has not been previously saved, select "yes" and save the data before proceeding with a new test.

NOTE: Make sure this data is saved before pressing the 'New Test' button, as this data will be erased when the new test button is pressed.

Setting the AquaSorp Temperature

The AquaSorp must be thermally equilibrated to a desired test temperature before a new test can be initiated. The desired isotherm temperature and the current instrument temperature can be found at the bottom of the data collection screen in the status bar. These temperatures are updated real time. Anytime the current temperature is more than ±1°C different from the desired temperature, an equilibration time is needed. The length of this equilibration time

will vary depending how far away the current temperature is from the desired temperature.

When the AquaSorp is turned on, an initial 60 minute warmup time for the balance is needed. The AquaSorp temperature is set to 25°C by default.

Equilibration Time Remaining to Start Test

⏱ 00:51:44 [Hide]
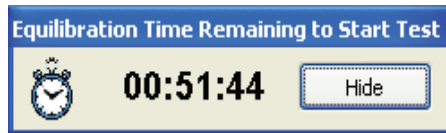
After the initial warmup, additional equilibration time should only be needed if the isotherm temperature is changed or the instrument is shutdown.

If an isotherm test temperature other than 25°C is desired, the temperature should be set prior to starting a new test. To change the desired isotherm temperature, press the "Set Temp" icon at the top of the screen to open the "Set Isotherm Temperature" page.
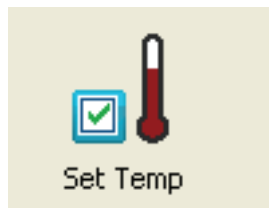
Set Temp

Any change to the target temperature will require an equilibration time, even if the instrument had previously equilibrated to a different temperature. The equilibration time

needed will be indicated in the text of the "Set Isotherm Temperature Page." Temperatures between 15°C and 40°C may be chosen. Enter the new isotherm test temperature using the up and down arrow buttons or by typing in the desired temperature and press "OK". The "Set Temp" icon at the top of the SorpTrac data collection page will then blink red, the white light on the front of the AquaSorp will blink, and the "New Test" icon will not be active until the temperature has equilibrated to the desired isothermal temperature.

Starting a new Test

To begin a test, press the 'New Test' button or go to Actions > New Test to open the test setup wizard.

A warning box will tell you that starting a new test will erase data on the instrument and ask if you want to proceed.
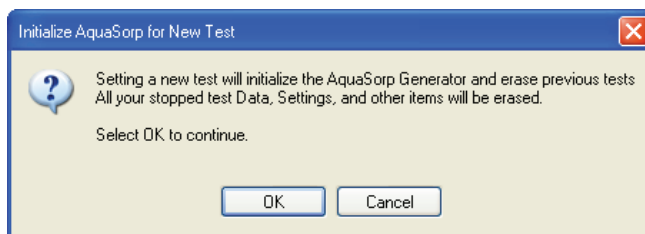
Make sure this data has been saved before pressing 'OK' as this data will be erased when the new test wizard begins. Pressing "cancel" stops the new test and does not erase the data. The wizard will guide you through the steps necessary to start an isotherm test. The steps of the wizard are described below:

Step1. Isotherm Temperature

This page shows the currently set isotherm temperature to allow the user to confirm it is correct. If a different temperature is desired, the user must cancel out of the test startup wizard and set the temperature using the "Set Temp" icon. Additional equilibration time will be needed if a change is made.

After a test is complete, the temperature of the AquaSorp can be maintained at the current isotherm test temperature or be allowed to return to the 25°C default temperature. The default setting is to maintain the temperature of the AquaSorp at the current test temperature. To change the setting back to 25°C when the test is over, just un-check the dialogue box at the bottom of the 'Isotherm Temperature' screen in Step 1 of the Test Setup Wizard called "Maintain AquaSorp Temperature Setting after test is completed. If subsequent isotherm tests are going to be run at the same temperature, it is advantageous to maintain the AquaSorp at this temperature to avoid equilibration time.

After verifying the isotherm temperature is correct and choosing whether to maintain temperature after the isotherm test is complete, press "Next" to advance to Step 2 of the wizard.
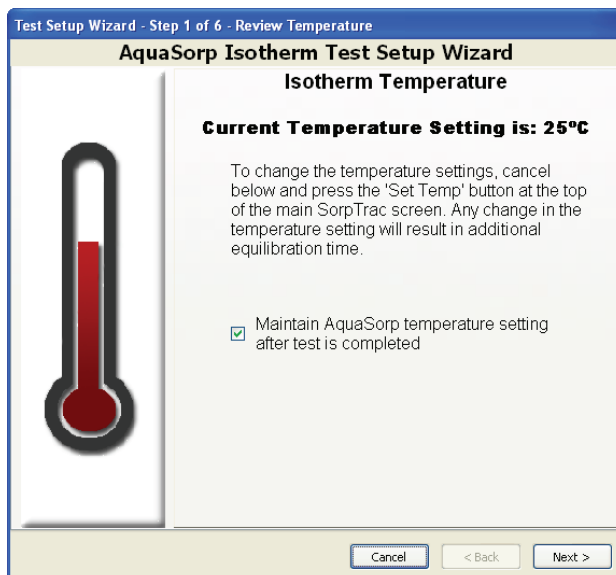
Step 2. Check Water and Desiccant Levels

Ensure the water level in the water chamber is between the two fill lines. The water chamber is located to the right of the sample chamber and should be filled with distilled water using the supplied water dispenser. Care should be taken to prevent spilling water into the sample chamber or down the side of the lid.

Verify there is enough blue desiccant in the desiccant tube to complete the experiment. If more than three quarters of the tube is pink, the desiccant tube should be changed. New desiccant tubes are available from Decagon or used desiccant tubes can be recharged by returning the tube to Decagon. Alternatively, the desiccant tube can be refilled

by removing one of the end caps, dumping out the pink desiccant (any remaining blue desiccant in the tube can still be used) and refilling with blue desiccant (additional desiccant can be purchased from Decagon). Fill the tube with desiccant and then lightly tap the tube on a hard surface while holding the open end up to pack the desiccant. If the tube is not full after tapping, add additional desiccant until full. When the water chamber and desiccant tube are ready, press "Next".

NOTE: Opening the chamber during a test will invalidate and stop the test due to exposure of the sample to the room environment. Make sure both the desiccant and water level are adequate before starting a test.

Step3. Zero the Balance

If there is a sample cup in the chamber, remove it. Close the lid and seal the chamber. The lid does not need to be sealed tight during this step, only closed. Press "Next." The balance will zero itself and the next step of the wizard will appear.
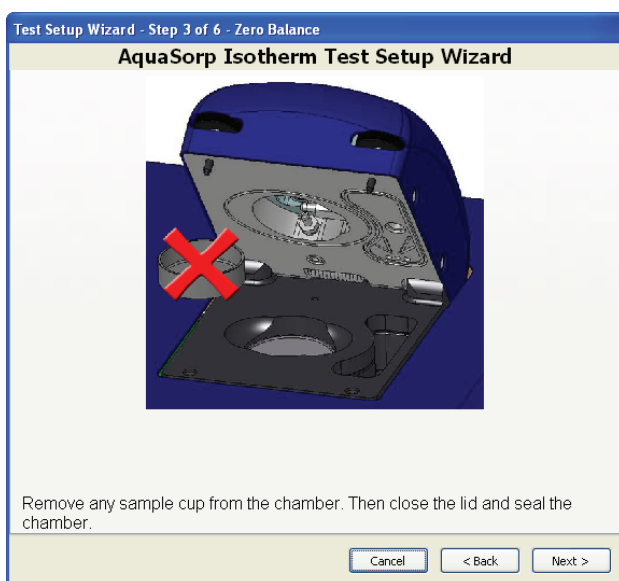
Step 4. Tare Cup

Open the lid and place an empty stainless steel cup into the sample chamber on the weighing platform. The platform

has a beveled rim and the cup should fit inside of the rim and lie flat on top of the platform. When the cup is properly inserted, close the lid and seal the chamber as described above. Then press "Next." The empty sample cup will be tared and the wizard will advance to the next step.

NOTE: This cup should be close to the isotherm test temperature. If ambient humidity is high (above 40% RH) and there is a large temperature divergence between the AquaSorp and the sample cup (more than 15°C), condensation could occur.
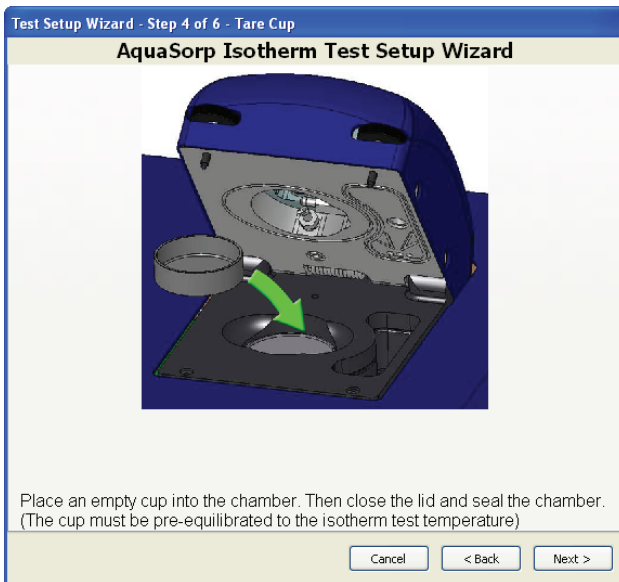
Step 5. Sample Insertion

Open the lid and remove the cup that was just tared. Place the sample to be tested into this tared cup. The sample

should be uniform and representative of the material to be tested. Avoid filling the sample cup more than half full and make sure the outside of the sample cup is clean. For most samples, adding just enough sample to cover the bottom of the cup should be sufficient.

For samples with slow water diffusion rates, increasing the surface area through grinding and keeping sample sizes small may improve sorption testing. However, if surface adsorption characteristics are desired, the sample should be kept in native state. Tablets and other coated samples should be crushed for analysis unless surface sorption characteristics are of interest. Samples with high viscosity and slow diffusion can potentially crust over so sample size should be kept small. For slow diffusing samples and powders, the optimal sample size is between 500 and 800 mg.

Use caution when placing the cup in the sample chamber to avoid spilling the sample. Insert the cup with the sample into the chamber, making sure the sample cup is seated properly on the weighing platform. Close the lid and seal the chamber by screwing down the thumbscrews until tight. Make sure you tighten the screws evenly or the lid could seat unevenly and cause a vapor leak. Then press "Next." The wizard will advance to the test setup screen.

NOTE: Like the sample cup above, the sample temperature should not be drastically different from the isotherm test to avoid condensation problems.

Test Setup Wizard - Step 5 of 6 - Weight Sample

AquaSorp Isotherm Test Setup Wizard

Place the sample into the tared cup. Insert the cup with the sample into the chamber. Then close the lid and seal the chamber. (The sample must be pre-equilibrated to the isotherm test temperature)

Cancel   < Back   Next >
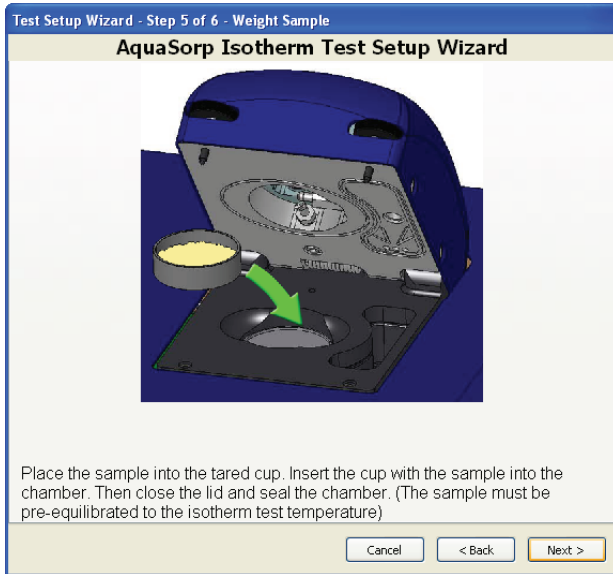
Step 6. The AquaSorp Isotherm Test Setup Screen

This screen sets the parameters for the isotherm analysis. It has several important elements including:

Test or File Name: Each experiment requires a unique name. This name will be used to identify the test as it is being run and to identify the data set once testing is complete. The name can be any combination of letters and symbols including spaces and should provide identifying information for the sample. A data file will automatically be saved with the test or file name in My Documents/Decagon/SorpTrac. If another default save location is desired, it can be changed by going to SorpTrac Tools > Preferences. This data file

will be updated with new data as it becomes available whenever SorpTrac is connected to the AquaSorp or if not connected, the data file will be updated with new data upon re-connecting to the AquaSorp. Upon completion of the isotherm test, this data file will contain all the isotherm data and can be used for data analysis (see Chapter 7 for instructions on using Data Analysis).

Test Description: This space can be used to re-cord any additional information about the ex-periment that might be helpful to the user.

There are two tabs on the lower portion of the Setup window. One is labeled "Isotherm Setup" and the other is labeled "Moisture Content Calculation".

Isotherm Setup: The Isotherm Setup Tab is used to set the parameters for the test.

Water Activity Limits: Use this section to set the upper and lower water activity limits for the isotherm. The minimum setting can be any value above 0.03 a_w, but it must always be lower than the maximum setting. The upper limit is 0.95 a_w. Default values are 0.10 a_w minimum and 0.85 a_w maximum. The limits used will depend on the water activity range the user wants to study. Some sample types can undergo phase transitions and possibly go into solution during adsorption. If these changes are not desired, the water activity limit should be set at a value that is lower than the critical water activity for the change. Keep

in mind that the critical water activity may not be known until after an initial adsorption test has been conducted.

Pump Flow Rate (ml/min): Use this section to set the rate air will be pumped into the isotherm chamber. Separate flow rates can be set for each direction. The minimum flow rate is 10 ml/min and the maximum flow rate is 1000 ml/min. Default setting is 300 ml/min. Faster flow rates may make the isotherm test faster, but may also result in fewer and less evenly spaced data points. For more information about choosing the correct flow rate for your sample, please contact Decagon Devices.

Sorption: Use this section to set the initial direction of the isotherm, the starting water activity, and the number of sorptions.

> **Starting Direction**: In this box, choose if the isotherm is to be started as desorption or adsorption.

> **Starting Water Activity**: Use the next box to determine if the isotherm will be started at the sample's current water activity level or if the sample will be saturated to the maximum water activity (desorption) or dried to minimum water activity (adsorption) before beginning the isotherm. If just full isotherms are desired, the min and max starting water activity are the most desirable. If the working adsorption or desorp-

tion isotherm data is desired from the current state of the sample, current starting water activity should be chosen.

Number of Sorptions: The final box is used to determine the number of sorption curves. Choosing "1" for the number of sorptions means one desorption or adsorption curve depending on the starting sorption direction. Choosing "2" for the number of sorptions will result in one desorption and one adsorption curve. Any number of curves may be chosen to a maximum of 20. Default setting is "2" sorptions. If current water activity was chosen as the starting water activity, the initial curve from the starting point to the minimum or maximum water activity (depending on the chosen initial isotherm direction) will count as one sorption. If maximum or minimum is chosen as the starting water activity, the first adsorption or desorption curve (depending on the chosen initial isotherm direction) will be identified as a quick adsorb or a quick desorb and will not count as one of the sorption curves.

Moisture Content Calculation: This tab is used to determine how moisture content will be calculated. The AquaSorp's balance tracks weight gravimetrically as the isotherm is running. To have meaning, these weights must be converted into moisture contents. The method used to determine moisture content when using the AquaSorp will depend on the users preferred moisture content method. If moisture content is available prior to the start of the test, it can be

entered at setup and data collected while the test is running will be displayed as moisture content on the data collection screen. If the moisture content information is not available at startup and is entered after completion of the test, the data collection screen will show the weight of the sample and not the moisture content.

Dry Weight Prediction: This method has been developed by Decagon to eliminate the need to perform a moisture analysis. It utilizes the desorption isotherm data below 0.40 a_w and oven dry water activity to predict the oven dry weight. This method cannot be used if a desorption curve is not going to be generated. The method works for most types of isotherms, but cannot be used for materials with desorption curves that flatten at low water activities and then rapidly lose water at water activities below 0.1 a_w.

Moisture Content Before Test: If the user knows the moisture content of the sample prior to beginning the isotherm test, select this button and enter the percent moisture content. To be accurate, this must be the moisture content of the sample at the time it is placed in the AquaSorp.

Set This Item at a Later Time: If the dry weight or moisture content of the sample is going to be determined after the analysis, select this button. This is the default selection.

Moisture Content Reference
This function allows the user to select whether mois-

ture content should be reported on a wet or dry basis. Alternating between the reporting methods will result in changes to the data since they are calculated differently. For wet basis, the amount of water is divided by the total weight of the sample (solids plus moisture) while for dry basis, the amount of water is divided by the dry weight (solids only). When using a known moisture content, it is important to know its basis and then select the right method on the setup screen as this will impact the way all other moisture contents are determined.

Finish: Pressing finish at the bottom of the page will start the isotherm experiment you have just set up.

Data Collection

Once the test is started, data points will be loaded into the chart and table as they are generated as long as the Sorp-Trac software is connected to the AquaSorp. In addition, the status bar at the top of the screen will show the current status of the test and the status bar at the bottom of the screen will show the current test settings.

Pressing "Disconnect" at the top of the screen stops real time updating, but data will still be generated and saved in the AquaSorp's internal memory. This means that once a test has been started, the SorpTrac software can be disconnected and the computer can be shutdown without affecting the test.

If the AquaSorp is turned off and automatic restart is not selected or if the stop button is pressed during the isotherm test, the data will not be lost and can be saved; however the isotherm test cannot be continued. If additional data points are needed, a new isotherm test must be started.

Tools Menu   Connect Via   Satus Menu

Chart View   Current Settings   Table View
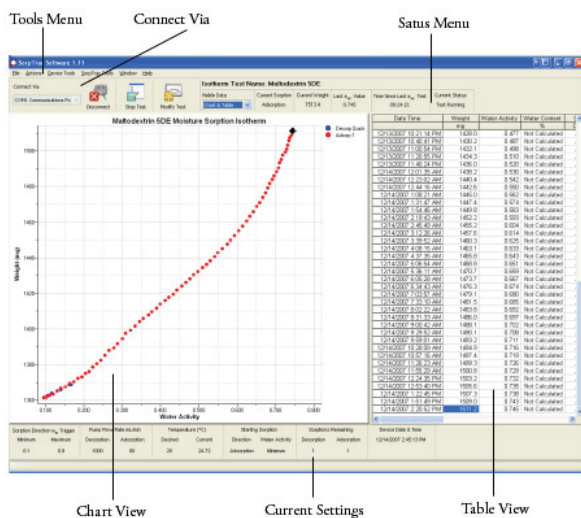
Saving Data

Upon completion of a test, a data file is automatically saved in the location described in preferences. If a different file name and/or location are desired, the test can be saved by going to File > Save Experiment and then entering a file name and location for the test and pressing "Save." The file name can be the same or different from the test name selected during test setup. In addition, both the chart and the table can be exported from the data collection screen for use in other programs. The graph is exported as an image file and the table is exported as a .csv data file. This is done by selecting File > Export and then selecting either "chart to graphic" or "table."

Modify Test

While the test is running, the a$_w$ limits flow rate for both adsorption and desorption, and the number of sorptions can be modified. To change these values, press the "Modify Test" button at the top of the screen or select Actions> Modify Test from the menu options at the top of the screen. (The modify test button will only be visible below the menus after a test has started. When idle, the button will be "Set. Temp").

The isotherm setup screen will appear. The a$_w$ minimum & maximum values, the flow rates and the number of sorptions can be updated on this screen. The starting direction and starting a$_w$ value settings cannot be changed and the number of sorption curves cannot be reduced to a number less than the number of curves already generated.

7. Analysis Tools

Data Analysis

SorpTrac software analyzes the completed test data using the GAB, BET, and Decagon's own polynomial equation called the Double Log Polynomial. Three different file types are used for data analysis in SorpTrac.

> • **Sorption Experiment Data file (.sxd)**. This file contains the data collected for each isotherm test. It is created automatically when an isotherm test is started or can be saved manually be selecting File > Save Experiment at the data collection screen. This file is then opened in the Data Analysis Tool to be analyzed.

> • **Analyzed Experiment Data file (.axd)**. This file is created by saving the results from the Data Analysis Tool. It contains the experimental data included in the analysis plus the isotherm model information.

> • **Multiple Experiment Data file (.mxd)**. This file is created by saving the results of the Multiple Isotherm Analysis Tool. It contains the data for all isotherms included in the analysis as well as the isotherm model information if an analysis was done.

To begin Data Analysis, select Tools > Data Analysis, to open the Data Analysis Window. The Data Analysis window consists of a chart area, a data table area, and an analysis results area as seen below.
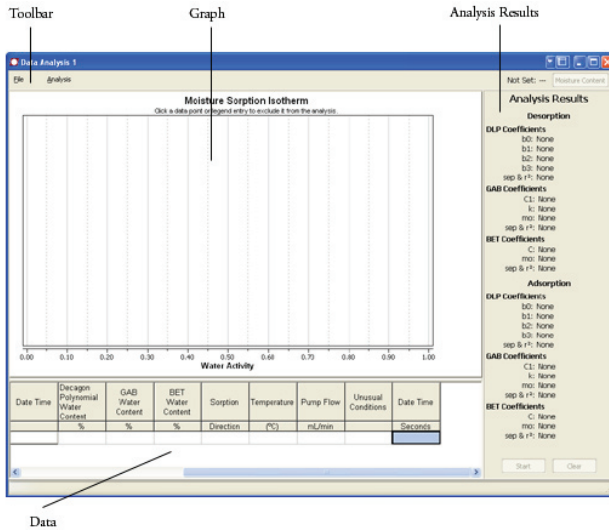
To open a test for analysis, select File > Open Test, select a previously saved test data file, and press "OK". The file extension for the test data file will be .sxd. The test data will be displayed in the chart and table. The chart shows the test name for the isotherm being analyzed.

Moisture Content Icon
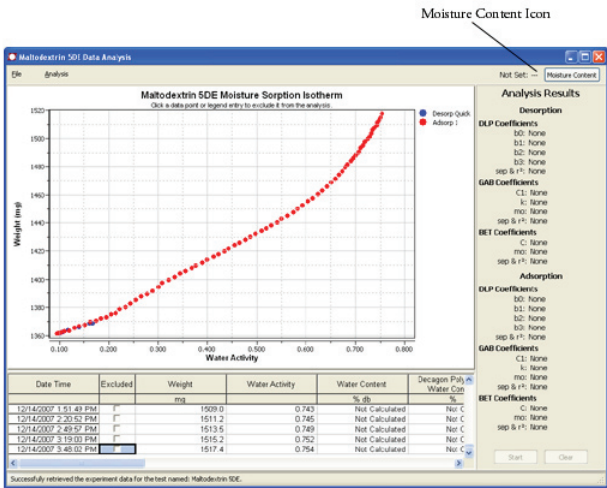
Multiple data analysis pages can be open simultaneously and more than one open data analysis page can use the same data set. Additional data analysis pages are opened by navigating to the data collection screen, selecting Tools > Data Analysis, and then opening an experiment as outlined earlier. You can quickly switch between Data Analysis Windows by selecting the Window menu item from the data collection screen.

Moisture Content

Before the isotherm curves can be analyzed, the sample weight data collected by the AquaSorp must be converted to a moisture content. If moisture content information was entered earlier during data collection, the moisture contents should already be displayed in the data table. To enter moisture content information, click the moisture content icon in the top right corner of the data analysis page. This brings up a moisture content window. This window con-

sists of several different sections including:

>**Test Name**:   The name of the test provided during data collection will appear here by default.

>**Test Description**:   If a test description was provided during data collection, it will appear here by default. A test description can be added or edited anytime using this screen.

Moisture Content Calculation: This section is used to choose the method to determine the moisture content of the isotherm data points. The possible methods include:

>**Dry Weight Prediction.** This method has been developed by Decagon to eliminate the need to perform a moisture analysis. It utilizes the desorption isotherm data below 0.40 a_w and oven dry water activity to predict the oven dry weight. This method cannot be used if a desorption curve has not generated. The method works for most types of isotherms, but cannot be used for materials with desorption curves that flatten at low water activities and then rapidly lose water at water activities below 0.1 a_w.

>**Oven dry weight**. This method is used if loss-on-drying is used for moisture content analysis. It is important that the dry weight be the weight of the actual isotherm sample after drying in a convection or vacuum oven. This is done by taking the isotherm sample from the instrument when the isotherm analysis is complete, and putting it directly into the oven. The
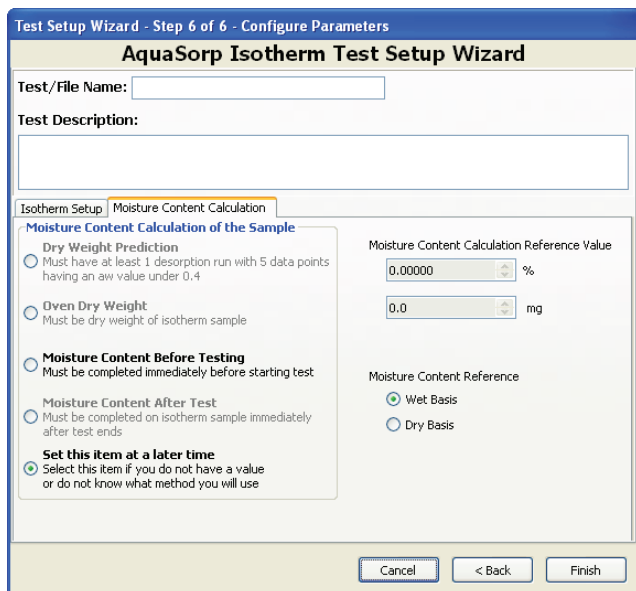
stainless steel cup with the sample used for the iso-
therm analysis can be placed directly in an oven The
oven should be the oven routinely used for moisture
content analysis. Keep in mind that any loss in sample
or errors in weighing will result in errors in moisture
content values for all isotherm data points.

To obtain the oven dry weight of the sample, weigh
the cup and sample after removing from the oven and
cooling in a desiccator. Next remove the sample from
the stainless cup and clean the cup. Weigh the clean,
dry and empty stainless steel cup. The oven dry weight
of the sample is the weight of the cup and sample mi-
nus the weight of the empty cup. Enter this value in
milligrams in the dialog box to the right of the oven
dry weight button.

Moisture content before testing. Use this method
if a moisture content analysis was performed on a
sub-sample either before or during the isotherm test.
If this method is chosen, it is vital that the moisture
content be performed on a sub-sample that is in the
same moisture condition as the isotherm sample at
the time the test was started. This method will be the
most common choice for those who use moisture
analysis methods other than loss-on-drying i.e. Karl
Fischer titrations. Enter the % moisture content value
into the top box to the right of the buttons. Sorp-
Trac will assign this moisture content value to the first
data point collected and calculate all other data points
based on this value.

Moisture content After Test: Use this section to enter a moisture content value from the sample determined after completing the isotherm experiment. This moisture content value can be determined by any appropriate method such as loss-on-drying or Karl Fisher. Enter the percent moisture content value determined on the sample at the end of the isotherm experiment into the top right of the buttons. SorpTrac will assign this moisture content value to the last data point collected and calculate all other data points based on this value.

Moisture Content Reference: This function allows the user to select whether moisture content should be reported on a wet or dry basis. Alternating between the reporting methods will result in changes to the data since they are calculated differently. For wet basis, the amount of water is divided by the total weight of the sample (solids plus moisture) while for dry basis, the amount of water is divided by the dry weight (solids only). When using a known moisture content, it is important to know its reporting basis and then select the right method on the setup screen as this will impact the way all other moisture contents are determined. Dry weight prediction is the default selection.

After selecting the appropriate Moisture Content determi-
nation, press "OK" at the bottom of the screen. The mois-
ture content window will close and return to the chart and
data table screen.

Running Data Analysis

With the moisture content data available, the isotherm
graphs can be analyzed to determine the isotherm equation
coefficients. If more than one adsorption or desorption
curves were generated, they will each be displayed on the
chart. Any of the curves can be excluded from the analysis
by clicking once on the curve name in the legend to the
right of the chart. The curve will then be removed from the
chart view. The word 'excluded' will now appear in paren-
theses next to the curve name in the legend at the right of

the chart indicating that the data for this curve will not be included in the data analysis. The data for excluded curves is not deleted and the curve can be re-included in the analysis by clicking the name in the legend again. If more than one curve is included in the analysis, all adsorption points from all included adsorption curves will be combined for the adsorption analysis and all desorption data points from all included desorption curves will be combined for desorption analysis. A single data point can also be eliminated from the analysis by double clicking on the data point in the chart view or by clicking on the excluded box in the data table next to the data point. When the data point is excluded, it will be replaced by an X in the chart view as seen in the following picture.
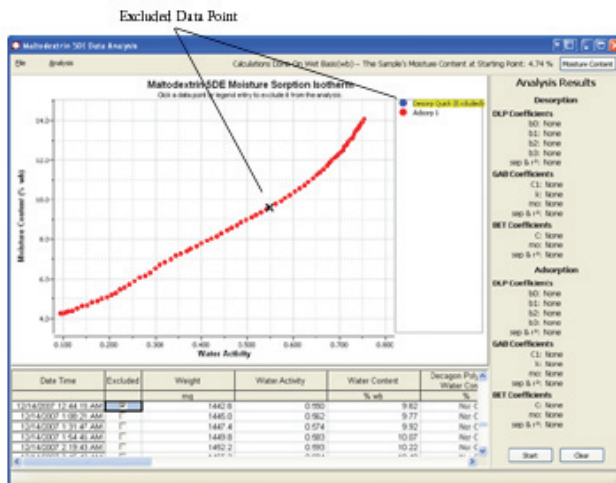
When the data points and curves excluded from the analysis have been selected, the analysis process is started by pressing the start button at the bottom right side of the data

analysis screen. When analysis is complete, the coefficient results will be displayed on the right side of the screen. The standard error of prediction (S.E.P.) and the R^2 are also displayed. The S.E.P. provides information about how well the models predict the actual data. For example, an S.E.P. of 0.05 means that the moisture content predicted by the isotherm model at any water activity is ±0.05 of the actual moisture content. The R^2 is an indication of the goodness of fit of the moisture content data generated by the models to the actual moisture content data. An R^2 value of 1.00 means the model perfectly matches the actual data.

The SorpTrac software provides separate analysis results for adsorption and desorption curves using the Double Log Polynomial (DLP equation), GAB, and BET isotherm models (for the BET model, only data points between 0 and 0.50 a_w are used for the analysis). The data points predicted by each equation are now displayed in the data table and the predicted curve is displayed in the chart view. Any of these curves can be hidden from view by clicking once on their name in the legend to the right of the graph. The word 'hidden' will appear after the model name.

The data table can be exported as a .csv file and the chart can be exported as an image file before or after pressing the start analysis button. However, if the table or chart is exported before pressing start, the information generated during analysis will not appear in the graphic or table. To export the table select File> Export> Table. To export the graph select File> Export> Chart to Graphic.

Once start has been pressed and analysis is complete, the

results can be saved. To save the results, select File> Save Analysis and choose the name and location to save the file. This file will be saved with an .axd file extension and will save the moisture content information and GAB, BET, and DLP information. To open a previously saved analysis file, select File> Open Analysis File and choose any .axd file to open. Once opened, new moisture content data can be entered by pressing the moisture content icon or the data set can be re-analyzed by pressing the clear button and the start button again. Any graphs excluded from the analysis originally will return when the clear button is pressed.

An isotherm report showing the isotherm graph and the table of coefficients can also be saved as a pdf file. To generate an isotherm report, go to File> Generate Report and choose a name and location for the isotherm report file.

Multiple Isotherm Analysis

The SorpTrac software includes a tool to view and analyze multiple isotherm tests together in the same chart. This function is used to compare isotherms as well as create and analyze working isotherms. To begin Multiple Isotherm Analysis, select SorpTrac Tools > Multiple Isotherm Analysis. This will open a new Multiple Isotherm Data Analysis page, which looks like the Data Analysis page, but with only one set of coefficients under Analysis Results.
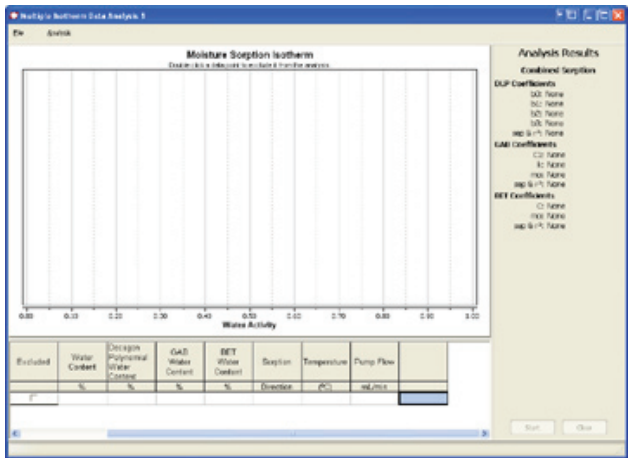
Overview of Multiple Isotherm Analysis

Multiple Isotherm Analysis utilizes axd files created using the Data Analysis Tool. It creates mxd files, which can be re-opened for further analysis or opened with other mxd or axd files for further comparisons and analysis. Since axd and mxd files already contain moisture content information, there is no need to enter moisture content information for Multiple Isotherm Analysis. When axd files are created, there is only one adsorption and one desorption curve, even if the original sxd file had multiple adsorption and desorption curves (Note: the adsorption and desorption curves in the axd files will include a combination of all of the data from all of the curves included in the original data analysis). The axd file can also have only an adsorption or desorption curve depending on what was included when data analysis was conducted. When an axd file is opened in Multiple Isotherm Analysis, all isotherm data

and curves in the axd file will be imported, (the isotherm model information will not be imported). Therefore, axd files that are created to be merged with other axd files using the Multiple Isotherm Analysis tool should only include the data or curves that are to be merged. Any unwanted data or curves should be excluded from the analysis before the axd file is created.

Each curve from each axd file to be combined will be identified in the Multiple Isotherm Chart by its test name and sorption direction allowing each curve to retain its original identity. However, if the combined data is analyzed using the start button at the bottom right corner of the Multiple Isotherm Analysis page, all of the data will be treated as one data set and one sorption curve. This means that for analysis, the data will not be distinguished by sorption direction or original axd file name. The analysis results will be one sorption curve with one set of coefficients.

For example, consider there are two axd files whose adsorption curves are to be compared. The original sxd files contain both an adsorption and desorption curve for both isotherm tests. To compare just the adsorption curves of the two tests, the desorption curves should be excluded from data analysis using the Data Analysis Tool. The results of the data analysis of just the adsorption curves for both tests should then be saved as axd files. These axd files can then be opened using the Multiple Isotherm Tool and compared on the same chart without the desorption curves. If the desorption curves were not excluded using the Data Analysis Tool, they too would be imported and would appear in the chart with the adsorption curves.

Running a Multiple Isotherm Analysis

To begin a new multiple isotherm project, select File > New. This will bring up the Multiple Isotherm Analysis Setup page. This page consists of two parts, the Files to Merge section and the Legend Name section.

Files to Merge

The Files to Merge section is used to select the isotherm files to be combined. To select a file, click on the open folder icon on the right side of the first blank space in the Files to Merge section. This will bring up a browsing window to find the first file to merge. This must be either an .axd file created during data analysis or an .mxd file created previously using Multiple Isotherm Analysis. At least two different files must be selected for merging and up to 10

files can be selected. To open additional files, repeat the process using the other blank lines.

Legend Name

By default, each curve from each file will be identified in the chart by the test name and the sorption direction. Test names that are too many characters will be shortened and if more than one curve has the same test name, they will be followed by ascending numbers (i.e. 1 then 2 and so forth). If a legend name other than the test name is desired, it can be entered in the corresponding blank line under Legend Name. The curves imported for that file will now be identified by the new legend name and the sorption direction.

Pressing 'OK' after selecting the files to merge will import the information and the combined isotherms will now appear together in the Multiple Isotherm Analysis page.

Pressing "cancel" will return to the blank Multiple Isotherm Analysis screen. If the files included in the Multiple Isotherm Analysis need to be changed, select File > Modify. This will bring up the Multiple Isotherm Analysis Setup Screen with the files and legend names (if any) chosen previously. The files can then be cleared, changed, additional files can be included, or the legend names can be changed. If a new Multiple Isotherm Analysis project is desired, selecting File > New will bring up a blank Multiple Isotherm Analysis Setup page.

Once the multiple isotherm files have been combined using the setup screen, the combined data will appear in the chart and table on the Multiple Isotherm Analysis screen. The legend names will be listed to the right of the chart. Clicking on a legend name in the chart will hide that curve from view and the word 'Hidden" will appear next to the legend name in parentheses. The chart and table of data can be exported at any time by selecting File > Export > Table or File > Export > Chart to Graphic. When a chart is exported, it will appear as it does on the screen and any curves that are hidden will be hidden in the image as well. The Multiple Isotherm Analysis information can be saved at any time as well (before or after clicking on the start button) by selecting File > Save. This will create an .mxd file. This mxd file can then be re-opened anytime by selecting File > Open. Only mxd files can be opened in this manner. The mxd file can also be combined with other axd and mxd files by selecting File > New and selecting the newly created mxd file along with the other files to be combined.

In many cases, all that will be desired with the Multiple

Isotherm Analysis tool is to compare multiple curves but not analyze them together. In these situations, it is not necessary to click the start analysis tool at the bottom right corner of the screen. However, if a combined analysis of the curves is desired, pressing the start button will generate the isotherm model values in the table and the coefficient values under Analysis Results. Unlike the Data Analysis Tool, clicking on a legend name in Multiple Isotherm Analysis hides the curve from view, but does not exclude it from the analysis. Individual data points can be eliminated by double clicking on them or clicking on the excluded box next to the data point in the data table.

The most obvious situation where analysis of the combined isotherms will be of value is for working isotherms. More information about working isotherms can be found below. Since all of the data imported into the Multiple Isotherm Analysis tool is considered one data set for analysis, only one set of coefficients will appear under Analysis Results. The results of the analysis can be cleared anytime by pressing the clear button. The results can also be saved by selecting File > Save. This will create an .mxd file as above but will now contain the isotherm model information. An analysis report showing the graph and coefficients can also be saved as a pdf file by selecting File > PDF Report.

Creating a Working Isotherm Using the AquaSorp

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

<u>Step 1.</u>

Generate two isotherm tests using the AquaSorp, 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.

> **Adsorption Curve:** One sub-sample is analyzed for adsorption from its current state. When setting up the test, any settings can be used for temperature, water activity limits, and flow rate, but at the Isotherm Test Setup screen of the Test Setup Wizard, Starting Sorption Direction must be **Adsorption**, Starting Water Activity must be **Current**, and Number of Sorptions must be **1**. The Test Name should identify the sample name and the sorption direction. Upon completion of the isotherm, the sxd file should be saved with a file name that identifies this isotherm as the adsorption curve of the working isotherm.

Desorption Curve: One sub-sample is analyzed for desorption from its current state. When setting up the test, any settings can be used for temperature, water activity limits, and flow rate, but at the Isotherm Test Setup screen of the Test Setup Wizard, Starting Sorption Direction must be **Desorption**, Starting Water Activity must be **Current**, and Number of Sorptions must be **1**. The Test Name should identify the sample name and the sorption direction. Upon completion of the isotherm, the sxd file should be saved with a file name that identifies this isotherm as the desorption curve of the working isotherm.

Step 2.

Analyze the adsorption and desorption curves separately using the Data Analysis Tool. Open the adsorption and desorption sxd files of the working isotherm in separate Data Analysis Tool windows. Enter the moisture content information for each curve as detailed in the Data Analysis section. Ideally, the same initial moisture content will be used for both isotherm curves since the two sub-samples used for isotherm analysis should have had the same initial moisture content. This also facilitates combining the curves in Multiple Isotherm Analysis. Data analysis should be then be conducted for each individual curve and the analyses saved as axd files as outlined above.

Step 3.
Combine the adsorption and desorption curves using the Multiple Isotherm Analysis Tool. In the Multiple Isotherm Analysis Tool, select to merge the axd files for adsorption and desorption as instructed above. Both curves should now appear in the same chart, each identified by their test name and sorption direction.

Step 4.
Analyze the combined curves as one working isotherm. Press the Start button at the bottom right corner of the page to analyze the two curves as a combined working isotherm. The results will show one working isotherm with the isotherm model information for the working isotherm. This working isotherm can now be saved as an .mxd file, exported as a graphic, exported as a data table, or generated as a pdf report as outlined above. In addition, multiple working isotherms can be compared using the Multiple Isotherm Tool by merging the .mxd files from several working isotherms.

8. Instrument Verification

To generate accurate isotherms, the AquaSorp must measure water activity and sample weight accurately. It is therefore important to verify the AquaSorp's water activity and weight measurements against known standards.

Water Activity Verification

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

Verification Standards

Verification standards are specially prepared salt solutions that have a specific molality and water activity that is constant and accurately measurable. The verification standards that were sent with your initial shipment are very accurate and readily available from Decagon Devices. These particular standards are accurate and easy to use. Most importantly, they greatly reduce preparation errors. Because of these reasons, only standards provided by Decagon can be used for verification of your AquaSorp's performance.

8. Instrument Verification

Performance Verification Standards come in four water activity levels: 1.000, 0.760, 0.500, and 0.250 a_w. The standards are produced under a strict quality assurance regime. The accuracy of the standards is verified and are shelf stable for one year. Several verification vials are included with the AquaSorp. Please contact Decagon Devices to order additional standards.

Verification Standards @25°C	Water Activity
Distilled Water	1.000 ±0.005
0.5m KCl	0.984 ±0.005
6.0m NaCl	0.760 ±0.005
8.57m LiCl	0.500 ±0.005
13.41m LiCl	0.250 ±0.005

To use a salt standard, remove the twist top and pour the contents into an AquaSorp sample cup.

When to Verify for Linear Offset

Linear offset should be checked against a known verification standard before beginning a new isotherm test. Linear offset should never be verified solely against distilled water, since it does not give an accurate representation of the linear offset. Decagon recommends that the AquaSorp be checked with a high and low salt standard, preferably the $0.760a_w$ and $0.250a_w$ verification solutions. Checking the a_w of a standard solution will alert you to the possibility of contamination of the unit or shifts in the linear offset from other causes.

How to Verify and Adjust for Linear Offset

To verify for linear offset of your AquaLab, select Device Tools > Verification and Restore > Water Activity Verification. The water activity verification wizard will then appear. The steps of the wizard include:

Step 1: Choose Water Activity Standard

Use this step to choose which water activity standard you will use to verify your AquaSorp. The most common choices will be 0.760a_w and 0.250a_w. The choices include:

0.250 a_w (13.41 molal LiCl)
0.500 a_w (8.57 molal LiCl)
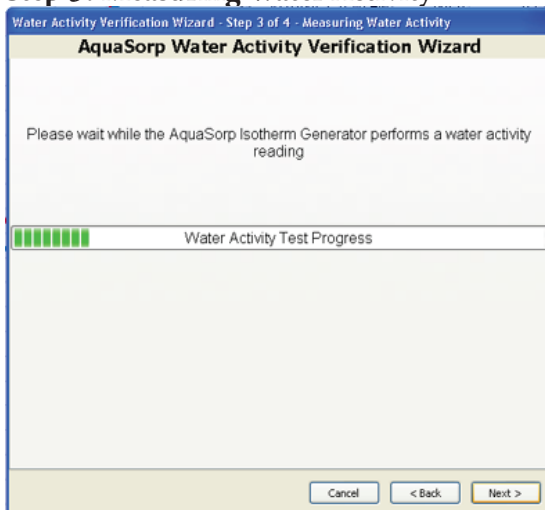0.760 a_w (6.0 molal NaCl)
1.000 a_w (Steam Distilled Water)

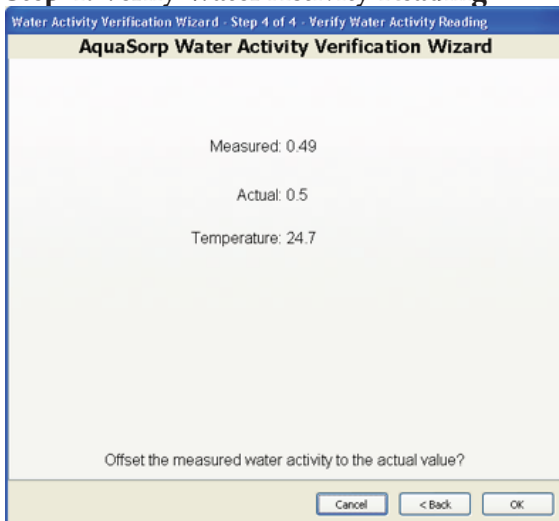**Step 2: Insert Water Activity Standard**

For this step, insert a cup with a fresh water activity standard into the sample chamber and seal the chamber.

Step 3: Measuring Water Activity

During this step, the AquaSorp will perform a water activity analysis on the water activity standard.

Step 4: Verify Water Activity Reading

When the water activity analysis in Step 3 is complete, the analysis results will be displayed as the temperature and measured a_w. The actual aw will also be displayed. To adjust the offset so the measured a_w will match the actual a_w, press "OK" at the bottom of the screen. The offset adjustment is done automatically and the verification wizard closes. Pressing "Cancel" on any screen of the wizard closes the verification wizard without saving the offset adjustment.

The verification process should be repeated for a second water activity standard. If the second standard reads correctly, the AquaSorp is ready to begin a test. If the second water activity standard requires a further offset, or if the offset on the first standard was larger than ±0.05a_w, the AquaSorp's testing chamber requires cleaning. For instructions on cleaning the instrument, please refer to Chapter 9. The calibration settings can be restored to the factory settings anytime by selecting Device Tools > Verification and Restore > Restore Factory Aw Calibration.

Balance Verification

The AquaSorp 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 AquaSorp and should be handled with tweezers and not bare skin. Balance performance should be verified before starting a new isotherm test

Note: The oils from your skin will affect the accuracy of the NIST traceable weight.

How to Verify and Adjust the Balance

To verify balance performance select Device Tools > Verification and Restore > Balance Verification. The balance verification wizard will then appear.

Step 1: Zero the Balance

During this step, nothing should be on the balance. Close the lid and press next to zero the balance.

Step 2: Insert Weight Standard

The next screen will ask you to insert the 2 gram NIST traceable weight standard included with the AquaSorp. Remember not to handle the weight with bare skin.

Step 3: Verify Weight Reading

The next screen will display the actual reading and the measured reading. If the measured value is different from the actual reading, press 'OK' to offset the weight to the correct value. If no offset is needed, press "Cancel". The balance settings can be restored back to the factory settings anytime by selecting Device Tools > Verification and Restore > Restore Factory a_w Calibration.

9. Maintenance and Cleaning.

The accuracy of the AquaSorp is vitally dependent on keeping your instrument clean. Dust and sampling debris can contaminate the sample chamber and must therefore be regularly cleaned. To clean the AquaSorp, carefully follow these instructions.

Tools Needed
- Decagon Cleaning Kit which includes:
    A plastic rod.
    Cleaning Solution
    Lint-free or sizing-free tissues (Kimwipes®)
    Isopropyl Alcohol (Not Included).

NOTE: Kimwipes® are ideal because they don't leave a lint residue, like most tissues. They also don't have any other compounds in the tissue that may contaminate the sensors in the AquaSorp's block. Also, never use cotton swabs to clean the block sensors. Most cotton swabs contain adhesives and other compounds that are released and transferred to the mirror and other surfaces, contaminating them.

Cleaning the Sample Chamber

If the sample chamber becomes dirty, wipe down the inside of the chamber with a wet Kimwipe followed by a dry Kimwipe. The lid containing the dewpoint block should be cleaned in a separate step as outlined below.

Dust and debris can fall below the balance plate and should be cleaned regularly. To remove the balance plate, gently lift up simultaneously on both sides of the platform. It may

be necessary to use some tweezers to assist with the removal of the plate. Avoid exerting any downward vertical force on the plate or brass shaft that sits below it as this can damage the precision balance. After wiping down the area under the plate, gently replace it by inserting it into the top of the brass shaft.
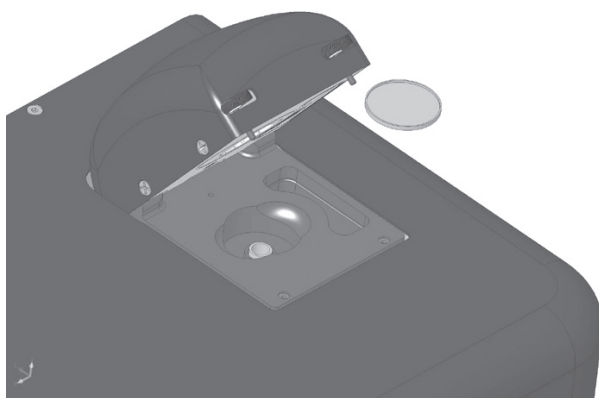
Cleaning the Dewpoint Sensor Block

Accessing the Block

1. Turn off the power on your AquaSorp (switch on back).

2. To remove the sample chamber lid, loosen and remove the 4 screws located on the left and right side of the sample chamber lid using the Phillips head screwdriver. Carefully lift the back of the lid and slide the lid forward past the thumbscrews on the front and lift to remove.

9. Maintenance and Cleaning
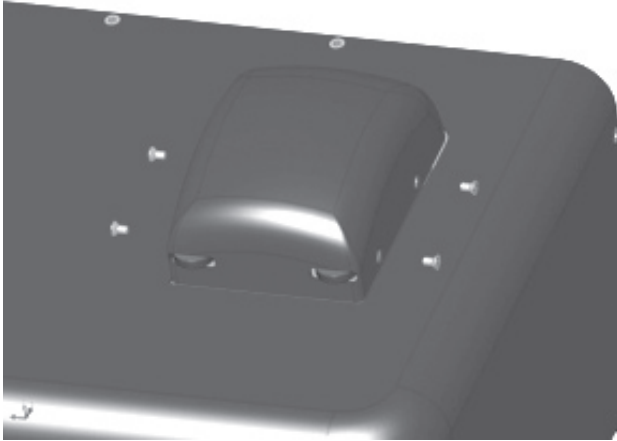
3. Removing the lid will expose the dew point sensor block. Remove the foam insulation square and unscrew the two thumbscrews that secure the sensor block. You need to use the flat head screwdriver to loosen the thumb screws.

4. Unplug the cable with the 20-pin socket that attaches the block to the main circuit board by releasing the two locking levers that are on either side of the socket. Also, unplug the lid sensor by pulling up on the small white plug at the front of the block.

5. Carefully lift the block straight up from its mount. Turn the block over to expose the chamber cavity.

Cleaning the Block

1. Wash your hands with soap and water to prevent oils from contaminating the Kimwipe tissue and being transferred to the mirror.

2. Wrap a Kimwipe strip around the plastic rod from the Decagon Cleaning Kit.

3. Cleaning your AquaSorp sensor block is a three-step procedure. First clean the sensors and block using an iso-propyl alcohol-moistened Kimwipe tissue. Clean the mirror, optical sensor, infrared temperature sensor and chamber surfaces.

4. Follow this with a Kimwipe tissue moistened with either Decagon's cleaning solution or distilled water.

5. Finally, wrap a new, dry Kimwipe around the plastic rod, and use it to thoroughly wipe the water or cleaning solution from the mirror, infra-red temperature sensors and chamber surfaces.

Reassemble the Block and Lid

After cleaning, return the block to its position on top of the lid and screw down the 2 thumbscrews. To ensure a good seal, tighten them with the flat-head screwdriver. Plug the 20 pin socket cable back into the block making sure the two locking levers lock back in place. Also, plug the small white lid sensor plug back in at the front of the block. Replace the insulation on top of the block and place the sample chamber lid back on. For proper fitting, make sure the 2 thumbscrews that are used to seal the sample chamber fit through the slots in the sample chamber lid. Secure with the 4 screws using the Phillips screwdriver.

Verify the water activity calibration as described in Chapter 8 after cleaning.

10. Troubleshooting

Problem	Possible Solution
Wont Turn on	• Power cord disconnected • Blown Fuse
Can't communicate with the AquaSorp	• RS232 cable not plugged in • Wrong Com port chosen
Weight is decreasing during adsorption	• Water tank has run dry • Pump is not working • Material is undergoing a phase change
Weight is increasing during desorption	• Desiccant tube has been used up • Pump is not working
Water Activity is decreasing during adsorption	• Water tank has run dry • Pump is not working • Material is undergoing a phase change
Water activity is increasing during desorption	• Desiccant tube has been used up • Pump is not working • Material is undergoing a phase change

Isotherm test has stopped premature	• The lid has been opened • The AquaSorp experienced a power failure
Isotherm test is taking unusually long time to complete, even at high flow rates	• Sample has very slow sorption properties • Lid is not closed tightly even if thumb screws seem to be tight. You may need to remove sample chamber lid (as when cleaning chamber) to further tighten tighten thumb screws

11. Further Reading

Applications for Moisture Sorption Isotherms

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Declaration of Conformity

Application of
Council Directive: 89/336/EEC

Standards to which   EN500082-1
conformity is declared: EN81-1

Manufacturer's Name: Decagon Devices,Inc.
                               2365 NE Hopkins Courtt
                               Pullman WA, 99163
                               USA

Type of equipment: AquaSorp Isotherm
                               Generator

Model Number: AquaSorp

Year of First Manufacture: 2007

This is to certify that the AquaSorp Isotherm Generator, manufactured by Decagon Devices, Inc., a corporation based in Pullman, Washington, USA meets or exceeds the standards for CE compliance as per the Council Directives noted above. All instruments are built at the factory at Decagon and pertinent testing documentation is freely available for verification. This certification applies to all AquaSorp Isotherm Generator models.

Certificate of Traceability

Decagon Devices, Inc.
2365 NE Hopkins Court
Pullman WA 99163
tel: (509) 332-2756
fax: (509) 332-5158
support@decagon.com

This is to certify that AquaSorp Isotherm Generators are manufactured utilizing weight and temperature standards with calibration traceable to the National Institute of Standards and Technology (NIST).

**National Institute
of Standards
and Technology**

NIST

Your AquaSorp is calibrated
to a NIST-traceable
weight and temperature
standard.

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