DVS Advantage

Operation Manual

Revision: 1.6

1 Contents

2 Introduction

This Operation Manual is designed to help the user understand the basic operational principles of the Dynamic Vapour Sorption (DVS) Advantage instrument, and how to set up and operate the system to gain efficient use of both the instrument and the user's time.

If any questions remain unanswered upon reading this manual, please do not hesitate to contact either your SMS representative or SMS directly. Also available for on-line customer support are our application notes which can be found on our website at: http://www.smsuk.co.uk .

Figure 2.1: The Dynamic Vapour Sorption (DVS) Advantage system

3 Principles of Operation

The DVS Advantage is designed to accurately measure a sample's change in mass as it sorbs precisely controlled concentrations of water or organic vapours in an air carrier gas.

The sample is hung from a microbalance in a sample pan (an empty pan is usually hung on the other side of the balance as a 'reference'). Air carrying the test vapours is then passed over the sample at a well-defined flowrate and temperature. The sample mass readings from the microbalance then reveal the vapour adsorption/desorption behaviour of the sample.

A schematic diagram of a DVS Advantage system is shown below:

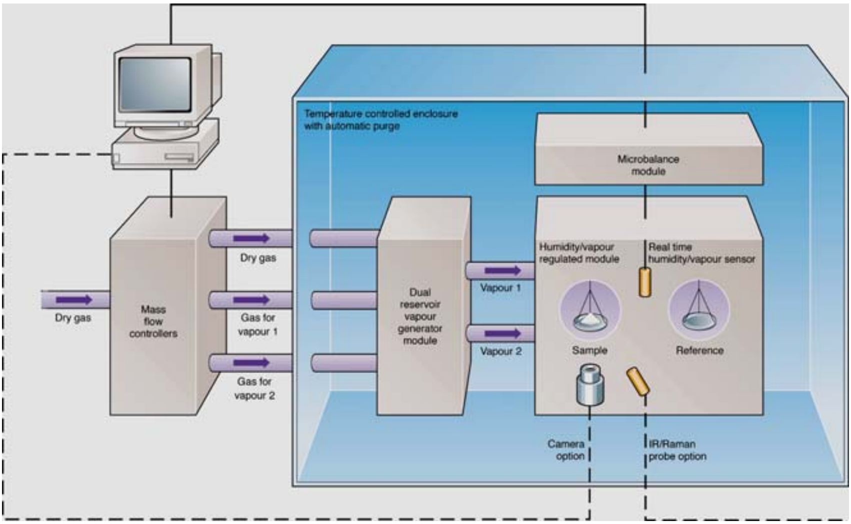


Figure 3.1: Schematic of a **DVS Advantage**.

At the heart of the DVS system is an ultra-sensitive recording microbalance capable of measuring changes in sample mass lower than 1 part in 10 million. This type of microbalance has very good long-term stability and is therefore ideally suited to the measurement of vapour sorption phenomena, which may take from minutes to days to achieve equilibrium.

The main instrument microbalance and stand is housed in a precisely controlled constant-temperature oven. This ensures a highly stable instrument baseline as well as accurate vapour generation control. In addition, the oven's temperature range (and so the instrument's experimental temperature range) is broad, spanning from 5-60^{o}C.

The required vapour pressures are generated by mixing dry and saturated vapour gas flows in the correct proportions, using mass flow controllers. A propriety optical vapour sensor as well as temperature probes are situated near the sample to verify and control system performance.

The instrument's microbalance mechanism is very sensitive to the sorption and desorption of moisture or organic vapours. Therefore a constant flow rate of a dry gas is used to purge the balance head. This facilitates the best performance in terms of baseline stability. The purge flow is automatically controlled so that the condensation of vapour in the balance head cannot occur.

The DVS Advantage instrument is fully automated via control from a dedicated PC microcomputer. A schematic of the instrument's sub-systems is shown below:

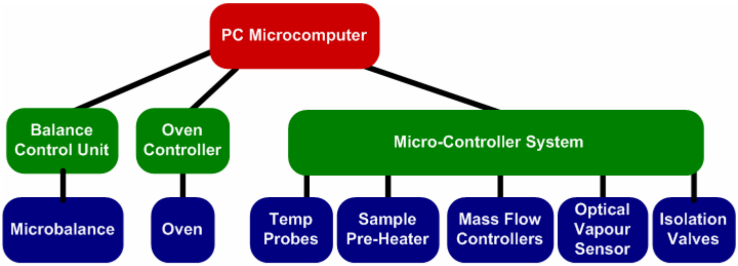


Figure 3.2: Schematic Diagram of **DVS instrument's sub-systems**.

The PC microcomputer is interfaced to the balance control unit via an RS-232 serial link. The oven is also controlled using an RS-232 serial link via a proprietary controller. An advanced microprocessor system is used to drive a range of important sub-component systems including:

- Temperature RTD probes
- Sample Pre-heater
- Mass Flow Controllers
- Optical Vapour Concentration Sensor
- Isolation Valves

This controller is linked to the PC microcomputer via a USB link.

The DVS Advantage software package supplied with the instrument provides a flexible and easy-to-use interface for setting up and running vapour sorption/de-sorption experiments.

In addition, the DVS Analysis Suite, which runs from within Microsoft Excel, provides a powerful environment for rapid plotting and data analysis (please refer to the DVS Analysis Suite User Manuals).

4 Instrument Installation

The DVS Advantage must be installed correctly by a qualified service engineer and it is strongly advised that your local SMS representative is contacted before decommissioning or moving an instrument from its installation site.

5 Minimum Computer Requirements

5.1 Windows 2000/XP Professional

The minimum computer requirements for Windows 2000/XP Professional (English Language version) are:

1. Pentium III processor (500 MHz)
2. 256 MB RAM.
3. Microsoft Internet Explorer 5.01 or later
4. 1 x available USB 2.0 port (if Dino-Lite USB video camera is to be used)

5.2 Windows Vista

The minimum computer requirements for Windows Vista (English Language version) are:

1. Dual Core 1.8 GHz (32 bit) processor.
2. 2 GB RAM.
3. Microsoft Internet Explorer 5.01 or later
4. 1 x available USB 2.0 port (if Dino-Lite USB video camera is to be used)

Note: To check what version of Internet Explorer is installed, view the **Help→About Internet Explorer** menu item when Internet Explorer is running.

6 DVS Operation

6.1 Hardware

The following points are designed to aid in developing a 'standard operating procedure' to ensure that the DVS is given the opportunity to deliver optimum results.

6.1.1 Starting up the DVS instrument:

The instrument will need restarting if there is a local power failure or a laboratory shutdown, in which case everything must be switched off at the power supply and all power switches put in the **OFF** position before commencing start-up.

Turn on the mains power to the computer, DVS Advantage Control Unit, oven and the Cahn microbalance Control Unit.

Then turn on:

1. The Cahn microbalance control unit via the switch at the rear. A red LED on the front panel will indicate that it is on.

2. The DVS Control Unit via a switch at rear. The lower green LED on the front of the unit will signify that the unit is powered up.

3. The computer provided with the instrument.

4. Turn on the Oven system via its front panel power switch.
5. Now turn on the carrier gas supply, which should be dry nitrogen regulated at a constant pressure of between 1 and 2 bar.
6. The DVS Advantage software supplied with the instrument may now be started up by double clicking

on the DVS Advantage icon on the computer main screen or menu. Following the software initialisation routine the DVS Advantage software main panel will be displayed.

The DVS instrument is now ready to be used.

6.1.2 Closing Down the DVS instrument

DVS instruments are designed for continuous laboratory usage. Therefore it is not necessary for the instrument to be shut down at the end of the working day - indeed it is intended that the DVS system will be working overnight!

The DVS Advantage Control Unit and the Cahn microbalance Controller are best left powered up at all times. The only need for powering down these units may arise during extended laboratory closures where local laboratory safety policy requires all instruments to be turned off. In these cases please follow the procedure below:

1. Exit the DVS Advantage software.
2. Turn off the carrier gas supply.
3. Turn off in the following order:
    a. The PC compatible computer.
    b. The DVS Control Unit main power via the switch on the back of the unit.
    c. The Cahn microbalance controller via its rear switch.
    d. The Oven via the front panel switch

4. Finally turn off the mains power to the computer, DVS Control Unit and the Cahn microbalance Controller.

WARNING!

If the power to the Cahn microbalance is turned off the Cahn microbalance unit will need to be re-calibrated after re-establishing RS232 communications between the balance and the computer (See Section 7.2).

6.1.3 DVS Sample Pans

The DVS Advantage instrument is supplied with a pair of precision quartz pans which are provided as a matched weight pair (European part No.C-WM-003A; North America No. QPNH13).

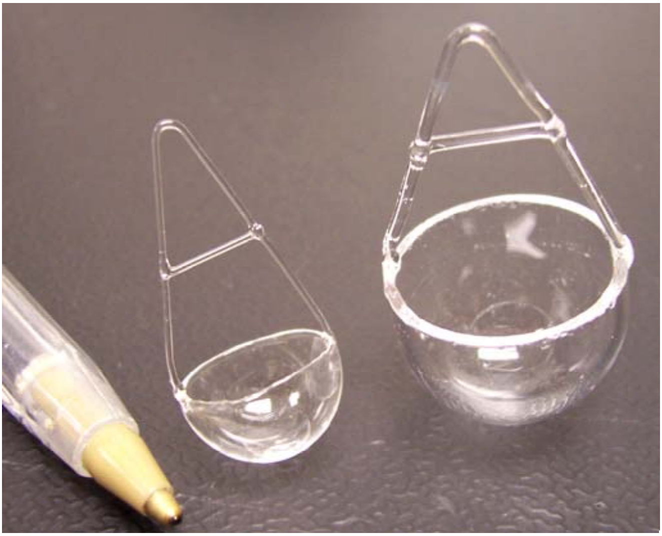


Figure 6.1: Quartz sample pans for a DVS-1 (left) and DVS-2 (right)

Larger glass pans are normally provided with the DVS Advantage 2 (see Figure 6.1 above).

The quartz pans will be suitable for general materials characterisation. They should be used as a pair (one for the reference position and one for the sample position) as their matched weights will minimise buoyancy drift. Please note, however, they are very fragile and will shatter if dropped.

Stainless steel mesh pans are also available for use with the DVS Advantage system for applications in which a solid sample pan is not necessary. Please contact Surface Measurement Systems for more information.

6.1.3.1 Preparation of the Sample Pans

An important issue for the accurate and reliable use of any gravimetric equipment is the cleanliness of the sample pan, and the elimination of static from the sample. Cleaning the cups is as much a question of remembering what not to do, as well as what to do.

Do not flame clean the pans, unless when using the platinum type. This results in micro-cracks forming on the surface of the glass and will result in a very high baseline shift between 0 and 98% p/p_0.

Do not use detergents to clean the pans - they are very difficult to remove and once again will result in a very high baseline shift.

Do not, having cleaned the pans, rub them with a cloth or tissue - this can create static build-up on the pan which may affect the DVS results or result in cup breakage.

Clean the pans using pure water from a wash bottle (or equivalent) (either HPLC grade or distilled and deionised water). Having cleaned the pans in water, rinse in a clean pure solvent such as ethanol (ethyl alcohol) or isopropyl alcohol (IPA), and then allow to air dry naturally.

If the sample pans have contamination which is difficult to remove there are two acceptable cleaning strategies available. One is to try a different solvent to help remove the contamination. This can also be combined with mild short-term ultrasonic cleaning. However, ultrasonic cleaning can occasionally result in pan breakage and thus should only be used as a last resort.

It is always good practice to have a least one spare pair of sample pans available.

If working under strict time constraints then it is a good idea to utilise a spare matched pair of pans and rotate them in sequence.

In order to achieve a satisfactory baseline in the DVS, it is important to establish two very important parameters:

1. That the pan is dry, and therefore the baseline at 0% p/p_o is stable.

2. That there is no static on the sample pans which could cause a baseline shift as the DVS moves to high vapour partial pressures.

The preferred method of establishing that both of these conditions have been met once the pan is hanging from the balance is as follows:

- Set the target moisture partial pressure to 0% manually, and allow 5-10 minutes for the pan to fully dry. Then check the baseline for stability - a criterion of less than 3 times the balance resolution (0.1 μg for DVS Advantage 1, 1μg or 10μg for DVS Advantage 2) per 20 seconds is the target. If the drift is always negative, then the pan may not yet be dry. If the drift is positive or erratic, however, then it is possible that there is static on the pans.

- Elimination of the static is easily accomplished by setting the target moisture p/p_o to 95% for a period of 5 minutes – this provides a conductive path to earth for the static. There may be a rapid change in mass as the vapour partial pressure passes through 60-80% as the static discharges. Once a stable baseline has been established at the high water vapour partial pressure, the target p/p_0 may be reset to the starting p/p_0 to be used for the next experiment.

- Once this humidity has been reached, again check for balance stability over a period of a few minutes. During this time prepare the method to be utilised, the sample etc.

- Finally, the DVS is ready to be ‘tared’ once balance stability has been established.

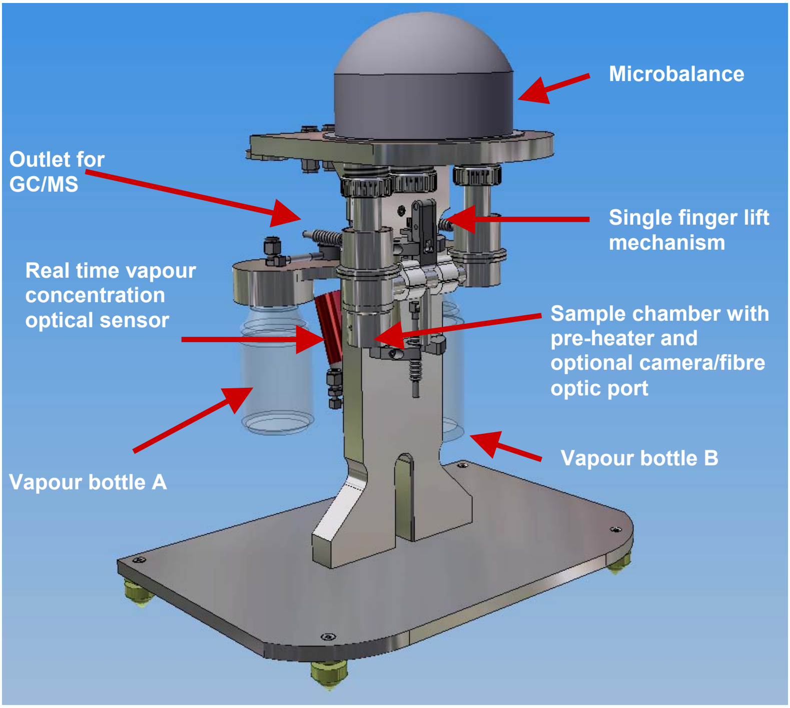


Figure 6.2: Layout of the **DVS Advantage stand assembly**

6.1.4 Sample Mass Considerations

Before loading the sample, it is wise to consider the likely change in mass (ie; vapour uptake) that may occur during the experiment. If the change in mass is likely to be less than 0.5%, then a sample mass of 50 mg or even 100 mg (for DVS Advantage 1) will produce a more acceptable signal-to-noise ratio. However, for changes greater than this 10-20 mg is normally more than adequate, and for very hydrophilic materials, less than 10 mg may in fact be more suitable (more vapour means more experimental time!).

If using the DMDT mode for the experiment (Sections 6.2.2, 9.5.2 and 9.6.1.1) the fact that ‘dm/dt’ is expressed as a percentage must also be considered. Therefore an equilibrium criterion of dm/dt = 0.002, which is suitable for a 10-20 mg sample, will be too high for a 100 mg sample (a value of 0.0005 would be more appropriate in the latter case).

It should also be remembered that samples such as compressed tablets will respond very slowly to partial pressure changes. This will necessitate the use of low dm/dt settings in experiments involving samples such as these.

6.1.4.1 Using Larger Sample Masses

The various DVS systems will handle a wide range of sample masses, though to use the maximum possible sample mass, counterweights or tare weights will need to be used:

DVS Advantage 1

This DVS system will measure a change in mass of up to 150mg in the sample mass without any need of counterweights. For example, for a 50mg sample a 100mg increase in mass could be measured. Using a counter or tare weight, samples of up to 1.5g in mass may be studied, though the maximum measurable change in sample mass remains 150mg.

DVS Advantage 2 - LOW MODE

A system in LOW MODE (default) will measure a change in mass of up to 1.0g in the sample mass without any need of counterweights. Using a counterweight, samples of up to 10g in mass may be studied, though the maximum measurable change in sample mass remains 1g.

DVS Advantage 2- HIGH MODE

This system will measure a change in mass of up to 10.0g in the sample mass without any need of counterweights. Using a counterweight, samples of up to 100g in mass may be studied, though the maximum measurable change in sample mass measured remains 10g.

The methodology for use of counterweights is as follows:

1. Estimate the weight of the sample – for this example, imagine that the sample mass is approximately 500mg and it is to be used in a DVS 1 system which can measure a maximum weight change of 150mg.

2. Place a 500mg calibration weight in the sample pan, and place 500mg of tare weights on the reference side of the balance system.

3. Tare the balance (Section 10.2.2).

4. Remove the calibration weight and substitute the sample into the sample pan (assuming the mass reported by the balance at this stage is -5.3678mg).

5. Select the **Configuration → Set Counter Weight Value** menu item in the control software (Section 9.3.3) and enter a counter weight value of 500mg.

6. The mass reading from the balance will now be 494.6322mg ie: 500mg - 5.3678mg.

6.1.5 Loading the sample

The procedure for loading a sample into the system is as follows:

- Firstly, open the DVS Advantage manifold so that the sample pan can be removed or replaced.

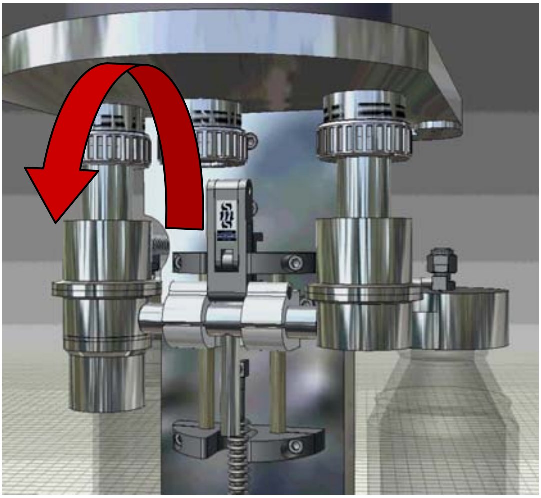


Figure 6.3: Release arm for sample access

Pulling forward the cam as indicated in Figure 6.3 will release the manifold and permit access to the sample pan area.

- The pan should be carefully removed using tweezers; it should not be removed by hand.

- Remember, the sample pan is contained in the LEFT-hand side of the manifold, and the reference pan is contained in the right-hand side. Note that the sample pan is placed within the pre-heater zone in the manifold.

Note: The sample must not be loaded into the DVS pan while it is hanging in the manifold! It must be loaded remotely on an analytical balance or on the bench, where a spillage can be easily cleaned up. The cup should only be placed back into the DVS once it has been sufficiently loaded with sample.

- Once loaded on the hang down wire, the sample should be allowed to settle for a short time before starting the experiment. This ensures that the first few data points are not affected by the swinging of the sample cup, and also allows the sample to equilibrate to the temperature and humidity of its new environment.

6.1.6 Controlling the Oven and Preheat Temperature

The DVS instrument is designed to perform measurements isothermally, over the temperature range specified for the system. The working temperature may be set using the DVS Advantage Software for both the DVS Advantage 1 and 2. Simply set the Target Sample Temperature to the desired value.

In addition, the DVS Advantage has a high-temperature local sample pre-heater (Section 6.1.7 below, and Section 9.5.1). This allows for rapid heating of the sample prior to the vapour sorption experiment and is useful for drying porous materials or for removing water from chemical hydrates.

Note: The microbalance MUST be recalibrated whenever the operational temperature of a DVS Advantage 1 and 2 is changed.

After making a small change in the system's operating temperature (<10°C), the user should wait a minimum of **one hour** to ensure the instrument has reached thermal equilibrium before commencing experiments at the new temperature.

This waiting period will increase with larger temperature changes (i.e. >10°C).

Monitoring the sample chamber temperature will give a good indication of the re-establishment of thermal equilibrium - the major rate-limiting factor in this process is the establishment of a new thermal equilibrium in the humidity generation bottles.

It is also important to remember that the partial pressure **MUST be set to 0% p/p$_o$** whilst changing the temperature of the instrument to avoid the possibility of condensation**.**

6.1.7 DVS Advantage 1 and 2 Preheat function

The DVS Advantage sample chamber is equipped with a small heating device which can heat the sample locally as the first stage of an experiment (Figure 6.2).

To use the preheat facility, the user must set up a **Preheat Method** by using the Method Menu of the DVS Advantage software (Section 9.5.1) to create a new Preheat Method.

This new Preheat Method should be saved and then incorporated into a **Sequence** (Section 9.6). Running the **Sequence** will allow the preheater to dry the sample in the manner requested.

6.2 Software – Quick Start to running an experiment

The following section is designed to get the user started with running experiments on the DVS instrument. Sections 9 and 10 give a more detailed description of the software functionality.

6.2.1 Initial Considerations

There are many factors to be considered prior to running an experiment on the DVS. For instance, the approximate uptake characteristics of the sample material, the amount of sample material available, and whether this experiment is intended to be a quick sample survey, or a final run to provide high quality publication data should all be taken into account.

Once the sample has been loaded into the instrument there are many different ways of setting up and running experiments. The following points may be used as a basic guide for setting up and running Methods on the DVS:

6.2.2 Method Stage Type – Stage-End Determinant

A DVS experimental Method is made up of a series of Preheating or Partial Pressure steps. Each of these steps is termed a Stage (Section 9.5).

In the case of a Partial Pressure Method, a key consideration is the amount of time each Stage will last. Usually the objective at the end of each Stage is for the sample to have reached equilibrium at the given Partial Pressure.

There are two Methods by which the end of a Stage can be determined; Time and dm/dt.

6.2.2.1 Time

(Refer also to Section 9.5.2.1)

This regimen is the simplest – a Stage will last for a specified period of time regardless of the state of equilibration of the sample i.e. if the sample has not yet reached equilibrium at the end of the allotted stage time, the instrument will move to the next stage regardless.

When to use Time-based experiments

Time-based experiments are useful in their simplicity.

Often – especially when testing samples whose sorption properties are unknown - it is desirable at first to use Time-based experiments to gain a rough idea of the time required for the sample to equilibrate at each Stage and the dm/dt reached at equilibrium. A very rough guide to running these initial exploratory experiments is to try a Stage time of 2 hours or more.

Thereafter dm/dt-based experiments may then offer the most convenient experimental configuration (see below).

6.2.2.2 DMDT

(Refer also to Section 9.5.2)
This uses the percentage rate of change of mass with time (dm/dt) to determine whether a sample has come to equilibrium. Using this as the Stage-End Determinant ensures that the sample reaches the required degree of equilibration before the Stage is deemed complete. If for some reason the sample does not reach equilibrium then the software will move on to the next stage after a specified maximum time has elapsed.

Suitable DMDT criteria

As outlined in Section 6.1.4, if using the DMDT mode the fact that 'dm/dt' is expressed as a percentage must be considered when determining the dm/dt criteria to set for sample equilibrium.

An equilibrium criterion of dm/dt = 0.002 is suitable for a 10-20 mg sample, but will be too high for a 100 mg sample (a value of 0.0005 would be more appropriate in the latter case).

When to use DMDT

It is recommended that, when using samples of unknown sorption behaviour, Time-based criteria be used in initial experiments. This will allow a rough determination of the time and dm/dt required for equilibration. DMDT methods may then be used if desired once an idea of the required Stage dm/dt is known.

6.2.3 Preliminary Settings

If drying the sample and/or the moisture behaviour of the sample is unknown, it is recommended to have the **Update Sample Weight an completion of first method stage** option activated (Section 9.5.2.1)

6.2.4 Initial Sample Weight

As soon as the sample has been loaded in the DVS and has stopped swinging, press the **M(0)** button to store the initial weight (Section 10.2.3).

6.2.5 Running an Automated Experiment

To run an automated experiment, follow the 5 easy steps outlined below:

1. First input the experiment details onto the **Run Experiment** panel (Section 0). These include Sample Name, Sample Description, the Output directory, etc. This provides useful information for later analysis of the DVS data.

2. Create any DVS Methods to be used via the **New Method** menu item (Section 9.5) that is located on the **Method** menu.

Note: An existing DVS method may be edited by selecting the **Edit Method** menu item (Section 9.5)

3. Once all DVS methods to be used in the experiment have been created, add them to a new DVS Sequence by using the **New Sequence** menu item located on the **Sequence** menu (Section 9.6.1.1).

Note: An existing sequence may also be used by selecting the **Load Sequence** menu item (Section 9.6.1.2).

4. Once a DVS sequence is loaded the experiment may be commenced. Select the **Run Experiment** panel (Section 0) and then click the **Run Sequence and Save Data** button. The DVS Advantage will instantly start sequentially running the Methods contained in the active DVS Sequence and saving the DVS data to the output directory specified.

   Once the experiment is in progress, the change in mass, humidity, Stage time, etc. can be monitored as the experiment progresses using the **Mass Graph** and **Instrument Data** panels (Sections 10.3 and 10.4).

   At any time during an experiment it is possible to gain an overall view of the data. Use the **Import DVS Data** function provided in the DVS Analysis Suite to analyse the data in Microsoft Excel® (please refer to the DVS Analysis Suite User Manual).

   Once the **Run Sequence and Save Data** switch of the Run Experiment panel has returned to the **OFF** state, the experiment has finished. However, after removing the sample pan for cleaning, the sample should be inspected visually. Has the sample changed colour, or turned into a solid or liquid? These observations constitute important and valuable information which can enhance the gravimetric data gained from the DVS.

5. Once the experiment has completed the results may be analysed using the DVS Analysis Suite software.

6.3 Organic Vapour Operation

The DVS Advantage is designed for use with water as well as a range or organic solvents. Please also refer to Section 9.4 for information on using the software with respect to solvent information.

6.3.1 Organic Vapour Leak Sensor

The DVS Advantage incorporates highly solvent-resistant 'O' ring seals, as well as the vapour detector safety interlock and an external gas exit for safe ventilation of the exhaust gases.

The exit vapour gas stream from the DVS Advantage should be vented to an appropriate location such as a fume cupboard.

The DVS Advantage's integrated organic leak detector will normally shut down all the power to the instrument in the event of a vapour leak inside the instrument incubator.

The sensitivity of the detector is factory-set for the most volatile vapours to be used and may only be adjusted by a qualified service engineer. In the event of a system shutdown the top LED on the DVS Advantage Control unit turns red.

The system will only reset when both of the following conditions have been met: the DVS Advantage software is re-started **AND** all traces of organic vapour have been removed from the incubator (green LED illuminated). This usually requires the incubator door to be left open for several minutes in order to dissipate the vapours. It should be noted that accidental tripping of the detector might occur during the filling/refilling of solvent vapours.

The system should not be restarted until the source of the vapour leak has been established. In the case where the sensor does not reset to a vapour-clear status, please contact Surface Measurement Systems.

6.3.2 Using Organic Vapours

SMS recommends only the organic solvents listed below which have been tested for use in the DVS. Only solvents of high purity should be used and HPLC-grade is recommended.

- Heptane
- Octane
- Nonane
- Decane
- Undecane
- Dodecane
- Cyclohexane
- Methanol
- Ethanol
- Acetone
- 2-Butanone
- Acetonitrile
- 1-Butanol
- Isopropanol
- Toluene
- Ethyl Acetate
- Butyl Acetate
- 1,4-Dioxane
- Dichloro-methane
- 1-Propanol

WARNING!

Solvents with very high flammability such as diethyl ether and tetrahydrofuran (THF) must **not** be used in the DVS Advantage.

Note: The use of any solvents not on this list should be discussed with SMS as they could constitute a safety hazard.

SMS will not be held responsible for the consequences of using non-recommended solvents.

6.3.2.1 Installing an organic solvent into the DVS Advantage system

Upon choosing a solvent, decant it into one of the smaller (250ml) pyrex bottles until it is approximately half-full. It is presumed that the bottle is already clean and free of any solvents from previous experiments. It is a wise idea to label the bottle with the name of the solvent contained within.

The bottle may be placed in the Bottle A or Bottle B position in the Advantage manifold - it should be screwed into the chosen position until hand-tight.

The system is also provided with a larger 500ml bottle and it is recommended that this be used for water. Again this bottle should be half-filled with either HPLC-grade or distilled and deionised water - the bottle should be labelled to indicate its contents.

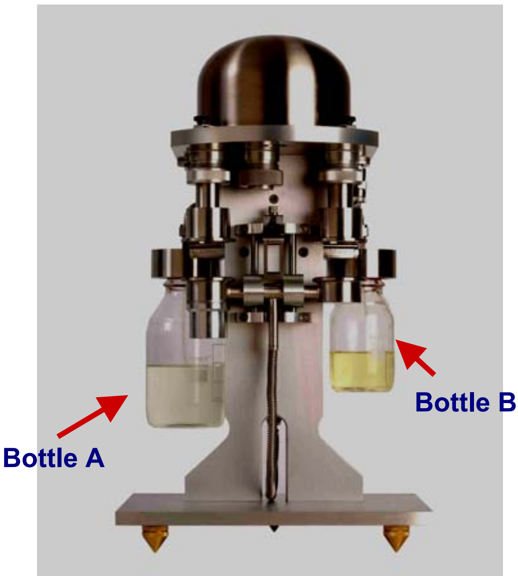


Figure 6.4: Solvent reservoirs on the DVS Advantage

6.3.2.2 Changing solvent reservoirs.

The DVS Advantage is designed to allow rapid changing of solvents. This is also facilitated by the availability of two solvent reservoir bottles designated Bottle A and Bottle B.

Once an experiment has finished, any bottle containing an organic solvent which is not required should be unscrewed from the Advantage manifold and sealed using the blue caps supplied. They may then be stored until required again.

It is then advised that an empty bottle be fitted to the manifold and 95% partial pressure be requested. This will allow dry nitrogen to be flushed through the system which will remove any vapour left from the previous experiments. Thirty minutes should be sufficient time for a complete system flush. This will need to be repeated for the other bottle if both solvents have been changed.

If a different solvent is then required in the system, the new solvent bottles may be mounted onto the manifold (again hand-tight only).

Note: It is important to remember to identify the new solvents by finding them on the list provided under **Solvents → Select Solvents** (Section 9.4.1) on the DVS software main panel.

This is a very important step as the instrument has no way of identifying the solvents that have been placed in the solvent bottles!

6.4 Optical Vapour Concentration Detector

A key feature of the DVS Advantage is the incorporation of a real time, in-line optical detector for measuring the vapour pressure of organic and inorganic solvents.

This optical sensor determines the temperature at which a liquid droplet film forms on a special optical mirror. This dew point temperature is then converted into the solvent partial pressure using the sample temperature and standard vapour pressure tables for the solvent in question.

The optical sensor is mounted at the rear of the DVS stand and in-line with the vapour flow stream prior to it entering the sample chamber (see Figure 6.2).

The DVS control software incorporates a number of special control features to maintain the cleanliness and performance of the optical mirror.

This optical sensor has proven to be a very effective tool for determining real organic vapour concentrations for a wide range of organic solvents. However, SMS cannot guarantee the sensor will operate for every organic solvent. Should there arise any problems or questions pertaining to the optical sensor please contact SMS.

7 Maintenance and Service

7.1 Refrigeration Maintenance

The Advantage incubator's refrigeration runs continuously when the instrument is in use. This being the case, it is possible for a build-up of ice on the incubator's refrigeration panel to form. This will result in a slow decline in temperature control over a period of months and may result in permanent damage to the cooling system if not rectified.

Note: It is strongly recommended that the incubator be regularly defrosted by turning it off for an hour or so <u>at least once every three months, but ideally once a month</u>, in order to avoid the build up of ice.

Note: If a large build-up of ice has formed, please be aware that a large amount of water will drain away via the drainage tube connected to the rear of the incubator.

This drainage tube is usually directed into a container or bottle – care must be taken that this container or bottle does not overflow.

As the incubator thaws, it is common for ice to drop down in the back of the incubator. This ice should be physically removed and not be left to sit inside the unit.

7.2 Calibrating the Microbalance

The microbalance needs to be calibrated against a certified calibration weight for optimum system performance. It is recommended that the microbalance be calibrated whenever:

- The microbalance is turned off;
- The DVS system is moved to a new location or repositioned in its current site;
- The experimental temperature of the incubator is changed, and;
- At least every month during routine usage.

Please refer to Section 9.7.1.2 for details of the microbalance calibration procedure.

8 Additional Hardware Issues

8.1 Time taken for the sample partial pressure to change after the target %p/p_o is changed

Around 90% of the requested change in sample p/p_o should be achieved within 3 to 4 minutes of altering the p/p_o set point.

For example, upon changing the set point from 20% p/p_o to 60% p/p_o, the sample partial pressure should reach 56% p/p_o within 4 minutes.

8.2 Proximity between the actual partial pressure and the requested value

In Open Loop control mode the DVS programme uses a look-up table in conjunction with an interpolation algorithm which takes into account temperature, gas flow rate and desired partial pressure in computing the ratio of dry to wet gas flow rates. The generated partial pressure should be within +/-1.5% p/p_o of the set point specified for commonly used flows and temperatures (e.g. 25°C and 200 sccm).

In the case of Closed Loop control, in which the vapour concentration from the optical vapour sensor is used to actively change the gas mix ratio, the vapour pressure generated should be within +/-1% p/p_o of the set point specified.

8.3 Vapour Pressure stability

Generally, the stability of the vapour pressure once equilibrium has been established will be typically +/-0.5%p/p_0.

Any problems with thermal stability might lead to slightly higher levels of drift.

8.4 Statically charged samples

Occasionally DVS users may come across samples that exhibit significant levels of static charge - especially those studying pharmaceutical materials. Static electricity has long been an important issue for microbalance users and must be treated with respect in those uncommon circumstances when it does occur. Statically charged powders will often exhibit unusual properties during sample preparation, such as strongly adhering to mortar/pestles, or to container walls. In the worst cases, transfer of static electricity to the sample hang-down wire will cause it to adopt a non-vertical alignment. Weight measurements associated with statically charged samples and sample cups will exhibit slow but significant drift. The build up of static may be prevented by using either an ionising antistatic gun, or a source of ionising radiation such as a polonium strip.

Care in cleaning the glass/quartz pans can minimise their becoming statically charged. Pans that have been cleaned with a solvent or water should be allowed to dry statically - the use of a hot air gun or dry air line to rapidly dry pans often leads to them becoming statically charged.

8.5 Hang-down wire falling off the system balance

If a hang-down wire falls off the system balance, follow the procedure outlined below to re-hang the wire:

1. The helmet which covers the microbalance must first be removed; loosen the four thumb screws securing the helmet, rotate the four holding flanges, and carefully lift the helmet vertically until it is clear of the pillars that guard the microbalance mechanism.

2. The hang-down wire must then be recovered. If it has dropped down into the DVS manifold, then a wire hook made from a paper clip may be used to carefully lift the hang-down wire out (care must be taken to avoid bending the hang-down wire while doing this).

3. Then using tweezers, drop the hang down wire through the holes that form the centre part of the flow chokes. It may take a few attempts to get the wire through both lower and upper flow chokes. Then, making sure that no excessive force is applied to the very sensitive balance arm, lift the wire on to the balance arm. Check that the hook for holding the sample pan is orientated in the correct direction prior to carefully replacing the helmet.

4. Finally, carefully finger-tighten all four securing flange screws to replace the balance helmet.

8.6 Purpose of the Purge Gas

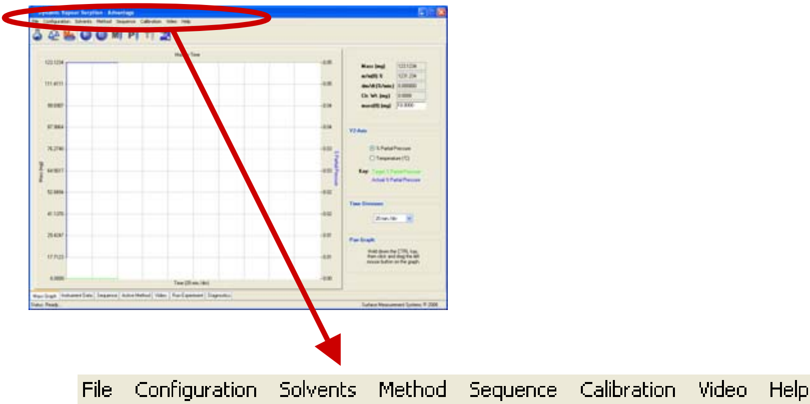
Experience has shown that microbalances may exhibit slight drift over long time periods if exposed to high partial pressures. The magnitude of this problem varies from balance to balance, but may typically be 10ug over a 24-hour period. By flowing a purge gas through the balance head at all times, this drift can be largely eliminated.

9 Software Toolbar

9.1 Introduction

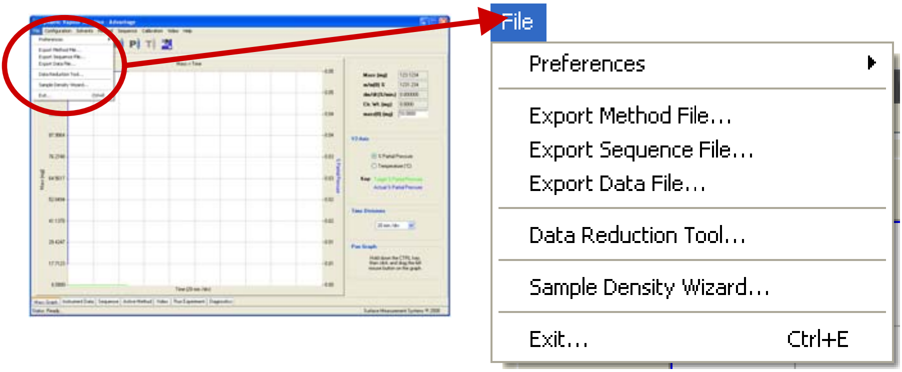
The Control software Toolbar allows the user to create the building blocks of an experiment –Method and Sequence files.

The toolbar options also allow the user to manage these files and to configure the instrument for optimum performance.

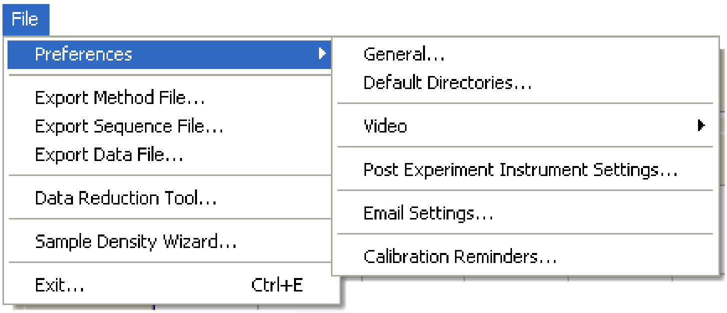


9.2 File

The **File** menu allows instrument preferences to be set, and for various DVS files to be exported into a .DAT format. The control software may also be exited from the File menu.



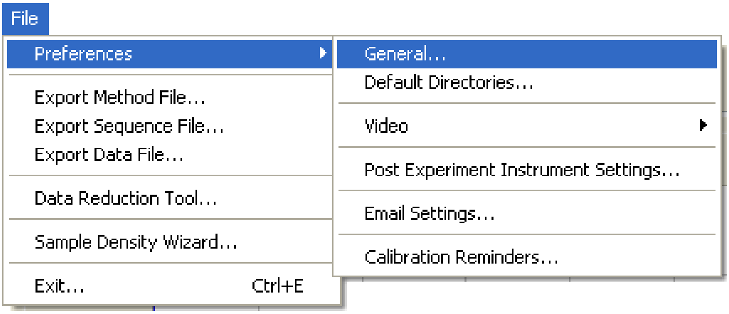
9.2.1 Preferences



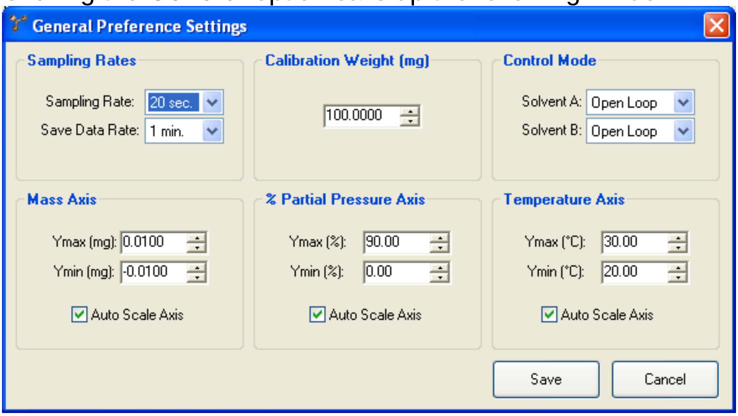
The **Preferences** option allows commonly used settings to be stored as preferences.

Note: The **General Preferences** menu item is **disabled** whenever a Sequence is loaded and/or data is being saved. The Sequence must be cleared and data saving stopped to enable this option for use – see Section 9.6 on Sequences.

9.2.1.1 General



Clicking the **General** option calls up the following window:



General Preference Settings may be specified for the following functions:

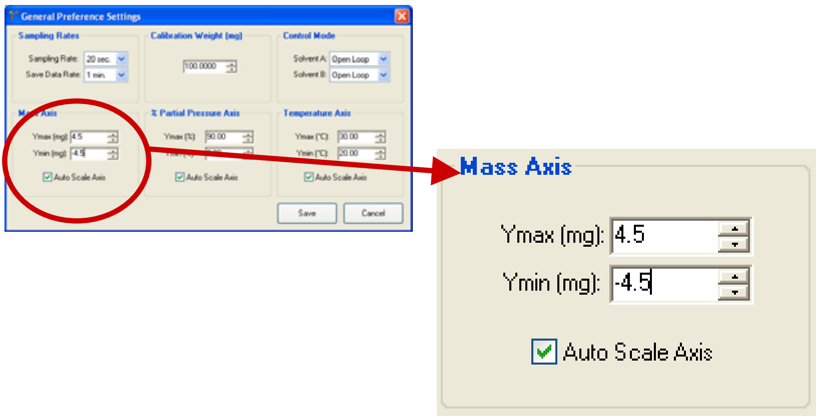
- Sampling Rates
- Calibration Weight
- Control Mode
- Mass/Partial Pressure/Temperature Axis

The **General Preference** settings are automatically loaded each time the DVS Advantage software is started. Each of these functions may be edited in other parts of the Control software – if they are, however, upon restarting the software they will return to the settings specified in the Preferences window.

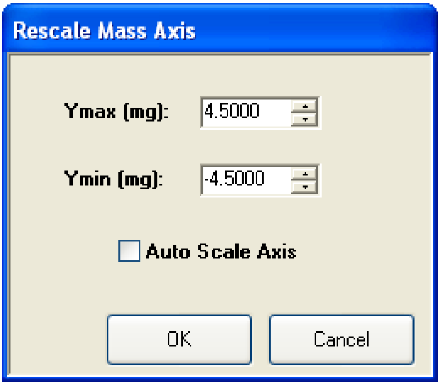
Details of how to edit the settings for these functions are contained in later sections of this manual.

In addition – any changes made in the General Preferences settings will not come into effect until either the Control software is re-started or the new settings are confirmed in the editing windows for each function.

For example, if the General Preferences window were used to edit the scaling on the mass axis:



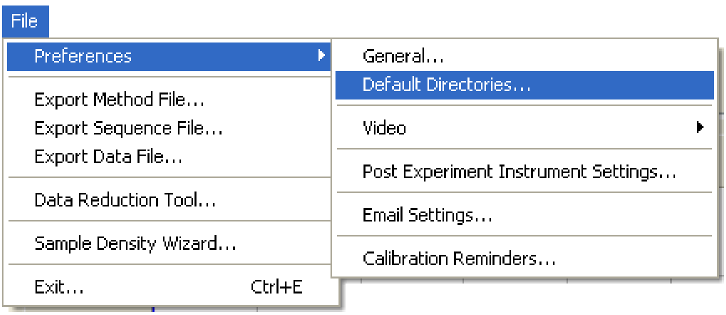
…then going to **Configuration → Rescale Mass Axis** (Section 9.3.2) will call up the editing window which will display the newly defined preferences:



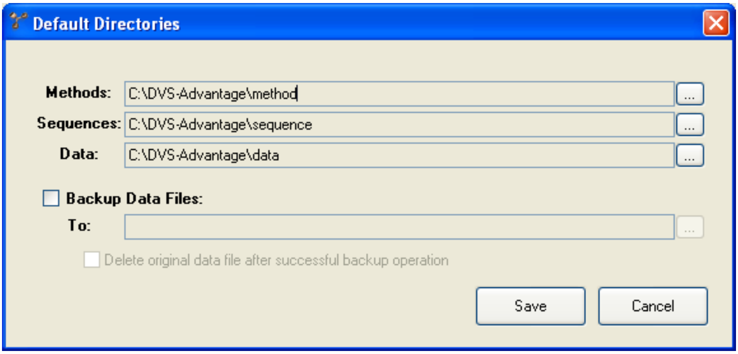
These new settings will not be implemented, however, until they are confirmed by clicking **OK** (or by restarting the Control software). The mass axis will then be rescaled according to the new settings.

Upon altering the settings in the **General Preference Settings** window, clicking **Save** will save the new settings. Clicking **Cancel** will retain the previous settings.

9.2.1.2 Default Directories

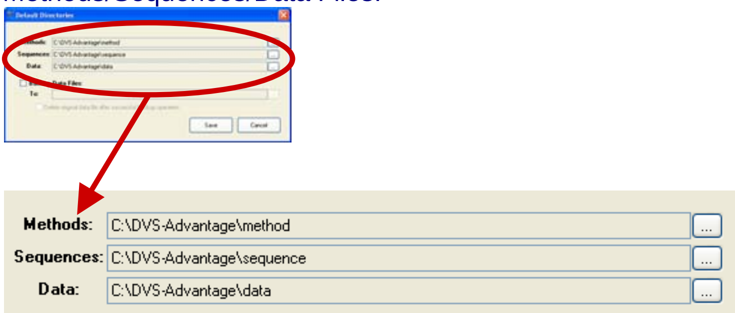


Clicking the **Default Directories** option calls up the following window:



These preferences allow the user to define where various DVS files will be saved by default.

Methods/Sequences/Data Files:



The default locations for Method, Sequence, and DVS Data files can be specified here.

The following default directories are in place upon software startup:

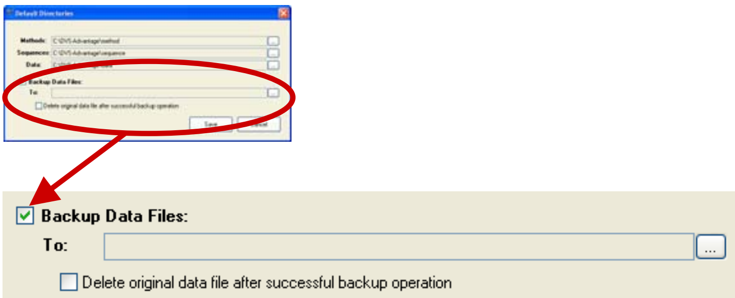
Methods: **C:\DVS-Advantage\method**

Sequences:	**C:\DVS-Advantage\sequence**
Data:	**C:\DVS-Advantage\data**

To choose a different directory, click on the  button at the end of the relevant Output Directory box – the following window will appear:

Upon selecting the desired folder and clicking **OK**, the newly specified directory appears in the Output Directory box.

Backup Data Files



This option specifies the backup directory to which a copy of the raw Data files will be stored once each DVS Method has completed. This option should be set to a directory location on the network.

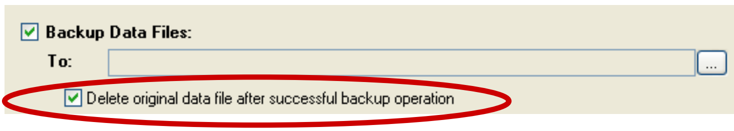
Using this option ensures that the DVS experiment is not interrupted by a network connection failure. As the Data files are saved to the network at the end of each Method, they can then be more easily incorporated into the user's normal IT back up procedures.

In order to activate the **Select Backup Directory** option, the tick-box next to the section header must be clicked:

Selection of the Backup Directory works in the same way as for the Method/Sequence/Data Directory, as shown above.

Delete original data file after successful backup operation:
If the raw data file backup operation is successful and this option is selected, all raw data files produced by the current DVS experiment that were saved to the Data Output directory (as specified in Step 1) are deleted.

This option is also activated using a tick-box:



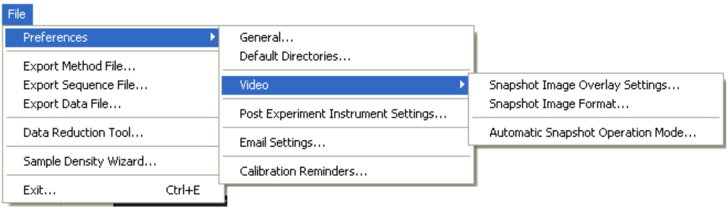
Note: If the backup operation is unsuccessful, the raw data files will not be deleted from the Data Output Directory. This ensures that the data will never be lost.

Save/Cancel

Once the Default Directories have been specified, the settings can be saved by clicking the **Save** button. Clicking **Cancel** will retain the original settings.

Note: In the case of the Default Directories Preferences described above, the settings become active as soon as they have been saved – the Control software does not need to be restarted to activate the newly specified preferences.

9.2.1.3 Video

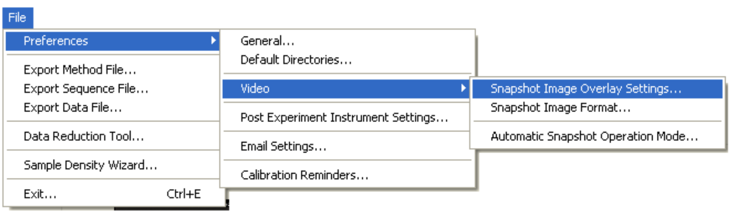


If the system has been fitted with the digital microscope option, then it has the capability to take snapshots of the sample. This can be done either manually or automatically.

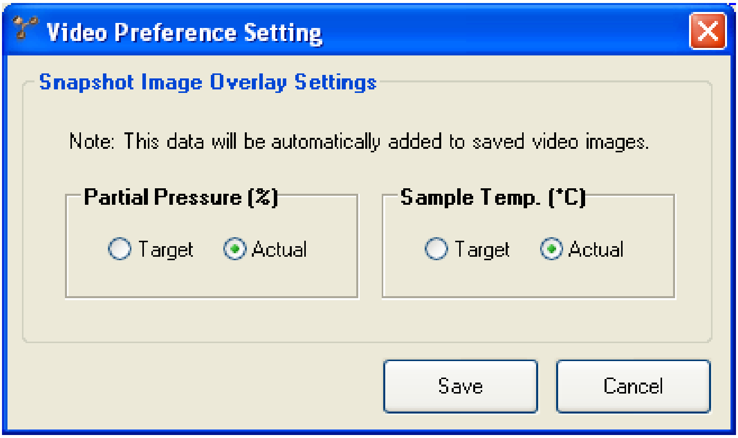
The **Video** menu item determines the kind of information that is overlaid on the sample pictures, the file format of these pictures and whether they are taken periodically or at the end of experimental Method Stages (see Section 9.5 for more information on Methods).

For more information on taking sample snapshots and formatting the snapshots themselves, please refer to Sections 9.8 and 10.7).

Snapshot Image Overlay Settings



Clicking this option calls up the following window:

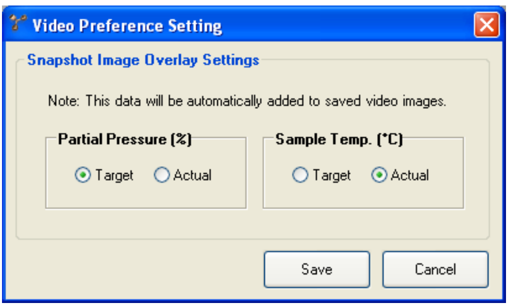


The **Video Preference Setting** window determines the type of system information that is overlaid on the sample snapshots.

The window above allows the user to select either the Target or Actual **Partial Pressure** and **Sample Temperature** to be displayed on the sample pictures (see also Section 10.4 on the Instrument Data panel).

Once the preferred data format is selected, clicking **Save** recalls the control software main interface. These preferences are retained through software re-starts.

Any snapshot of the sample then displays the selected data - along with the date. For instance, the following settings…..

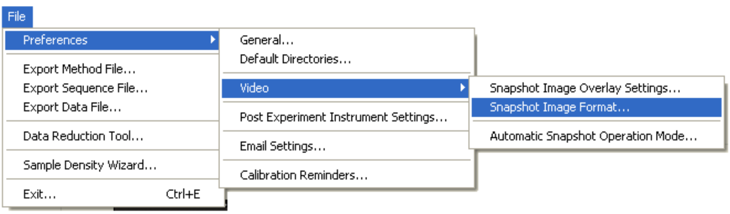


……would be displayed in the top, left-hand corner of the sample picture as shown below:

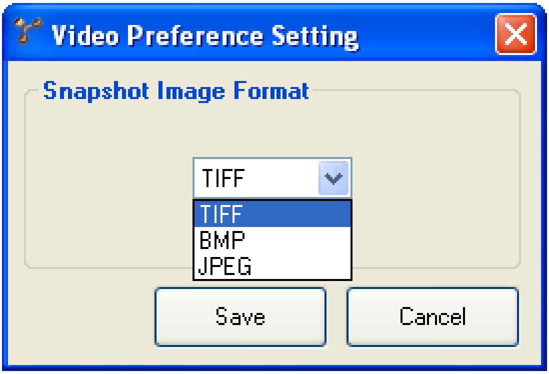
In addition, directory location of the picture file is recorded in the bottom left-hand corner of the frame.

For more information on taking sample snapshots, please refer to Section 10.7.

Snapshot Image Format



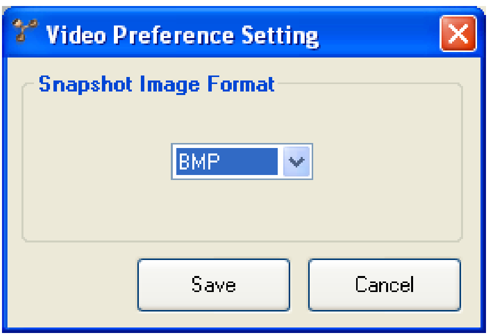
This option determines the file format of the sample pictures. Clicking **Snapshot Image Format** calls up the **Video Preference Setting** window, which allows the user to choose the file format of TIFF, BMP or JPEG:



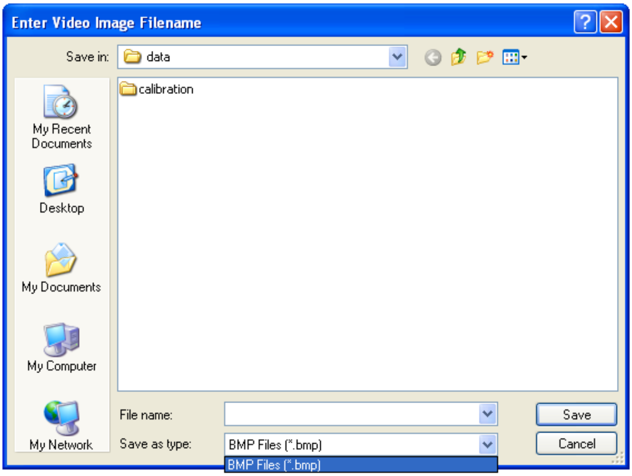
Clicking **Save** returns the control software main dialogue screen. These preferences are retained through software re-starts.

With these preferences stored, the next time a sample snapshot is taken, the user will be prompted to save the file in the selected format – no other format will be available unless the preference settings are changed.

For example, if the following preference were stored…..

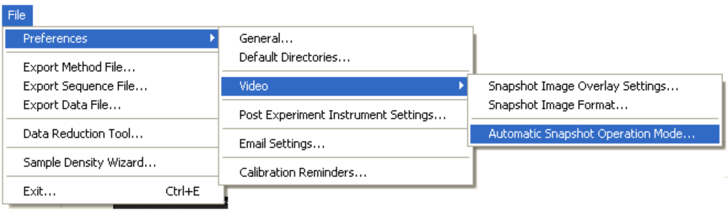


…..then, upon taking a sample snapshot, the user would be prompted to save the resulting picture file in BMP format only:



For more information on taking sample snapshots and saving the resulting files, please refer to Section 10.7.

Automatic Snapshot Operation Mode

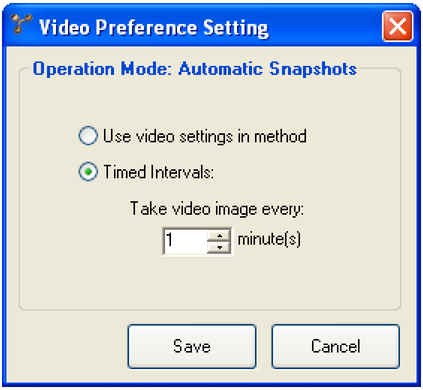


When data is not being saved, sample snapshots may be taken manually by clicking the **Take Snapshot** button on the **Video** tab (refer Section 10.7).

However, if data is being saved, then snapshots can only be taken according to the settings of a running Method (refer Section 9.5) or at pre-determined time intervals.

The **Automatic Snapshot Operation Mode** allows the user to choose between the options stated above.

Clicking the **Automatic Snapshot Operation Mode** option calls up the **Video Preference Setting** window:



The options available are:

Use video settings in method:
This option ensures that sample snapshots will be taken according to the settings in a running Method.

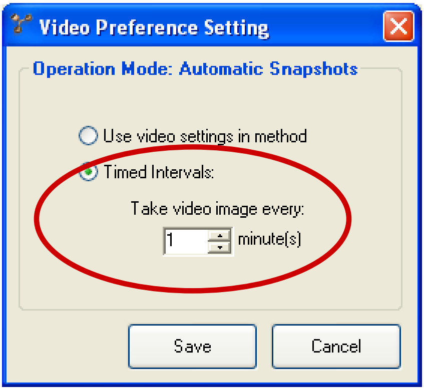
In order for any pictures to be taken in this case, a Method must be running, which has been set to activate the camera at the end of each Method Stage.

For more details on Method settings, please refer to Section 9.5).

Timed Intervals:

This option ensures that sample pictures will be recorded at the interval set (in minutes) whenever data is being saved – with or without running a Method.

Clicking the **Timed Intervals** option activates the data entry box entitled **Take video image every:** as shown below:



The number of minutes that may be entered ranges from 1 to 9999. The figure may be entered directly or by using the arrows at the side of the data entry box.

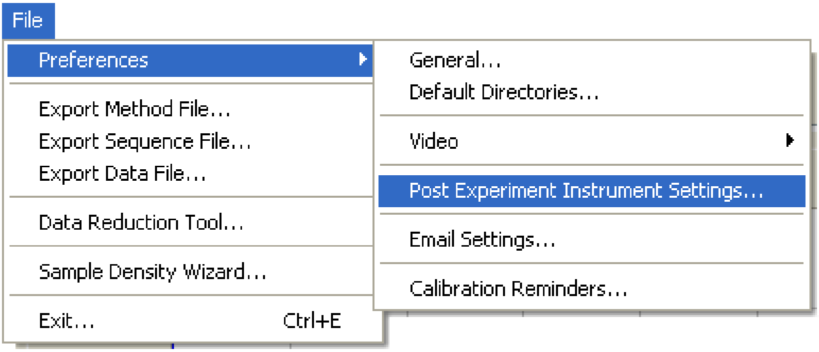
In the window shown above, clicking **Save** would ensure that a sample picture would be taken every one minute whenever data was being saved – either by running a Method or by saving data only.

The **Video Preference Settings**, once saved, are retained through software re-starts.

Whether saving pictures though Method settings or at Timed Intervals, the pictures taken are saved in the same directory as the system data files (refer Section 9.2.1.2).

Note: When data is being saved, the video preference option for **Automatic Snapshot Operation Mode** becomes inactive. However, the other video preference settings – **Snapshot Image Overlay Settings** and **Snapshot Image Format** may be altered during data saving.

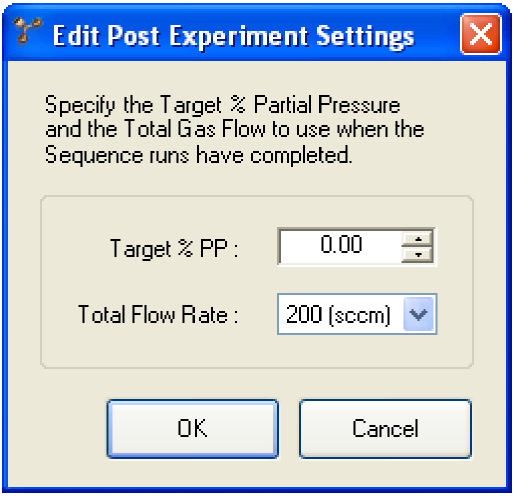
9.2.1.4 Post Experiment Instrument Settings



The **Post Experiment Instrument Settings** option allows the user to specify the post-experiment flowrate and partial pressure of the system.

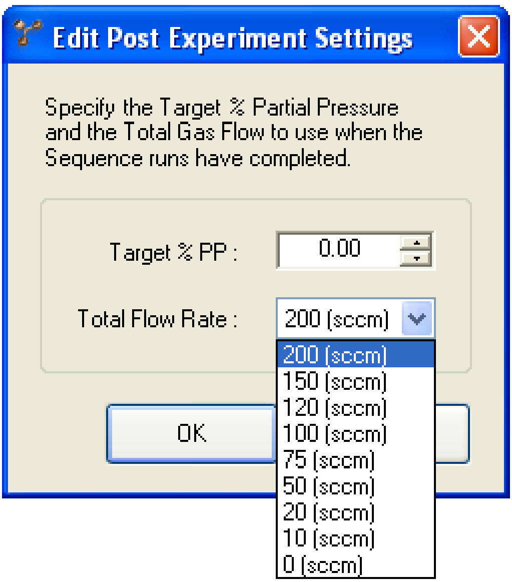
This is particularly useful if, for instance, the sample needs to be preserved under specific conditions before finally being removed from the DVS Advantage system.

Clicking the **Post Experiment Instrument Settings** calls up the **Edit Post Experiment Settings** window:



When first opened, the window will appear with default settings shown above.

The post-experiment **Target %PP** may be directly entered into the data-entry box or by using the up and down arrows to the box's right. The permissible target partial pressure values range from 0 to 100%.



The **Total Flow Rate** is set by using the drop-down list as shown below:

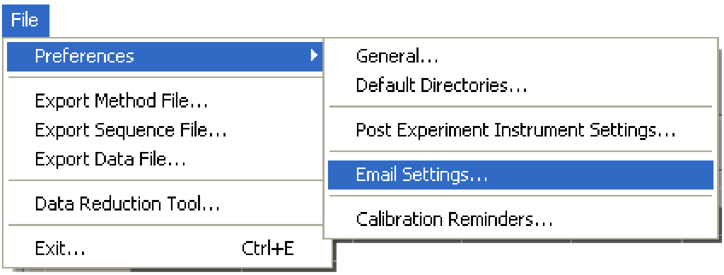
Only the Total Flow Rate values shown on the list above may be used. They can be set by either selecting them on the list, or by typing digits into the display box, which will call up the closest flowrate figure from the list.

Clicking **OK** will set the entered values, and return the DVS Advantage Control software main interface.

Clicking **Cancel** will also return the Control software main interface, but without setting the newly entered values.

Note: The Post Experiment Settings are retained through a software re-start – they do not revert to default values when the Control software is shut down.

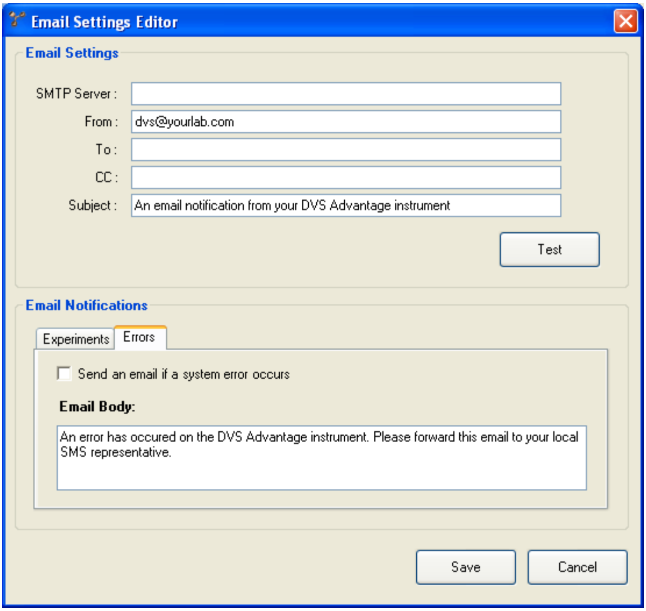
9.2.1.5 Email Settings



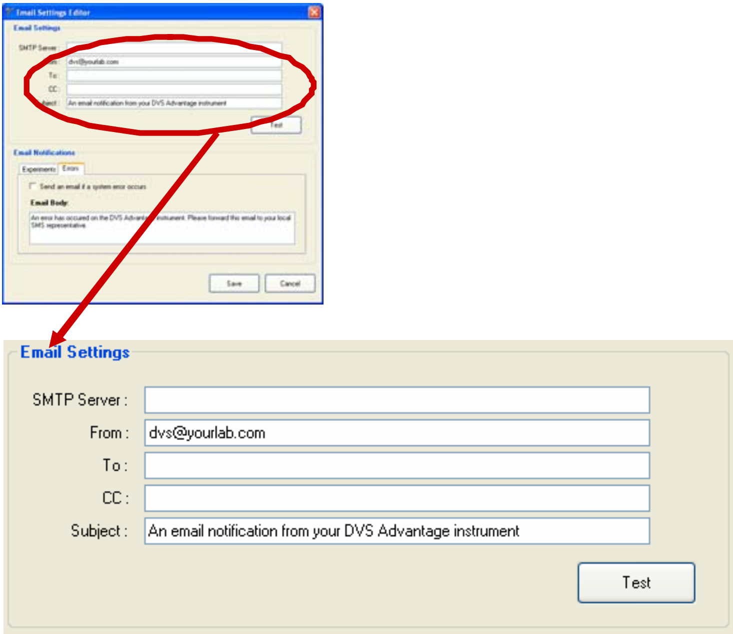
The control software can be set to send emails notifying the user of when an experiment has completed, and also when an error has occurred.

Clicking **File→Preferences→Email Settings…** calls up the

Email Settings Editor window:



Email Settings



This box allows the user to define the SMTP Server, the recipient addresses for the emails, and the email Subject header (the default message shown below may be edited).

It may be necessary to enlist the help of the local IT Administrator to help configure these settings.

SMTP Server

This needs to be set to the name of the e-mail relay server to be used. If the **SMTP Server** field is left blank, the name of the local SMTP server is used.

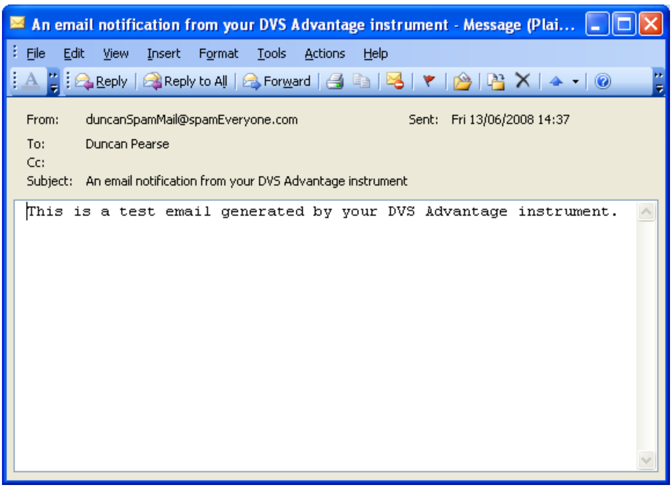
> **Note:** Windows XP computer administrators, please refer to Section 17.1.5.8 for more information on configuring SMTP for the email function to work.

Once the necessary details have been filled in, clicking the **Test** button will call up the following window:

Clicking **OK** returns the **Email Settings Editor** window.

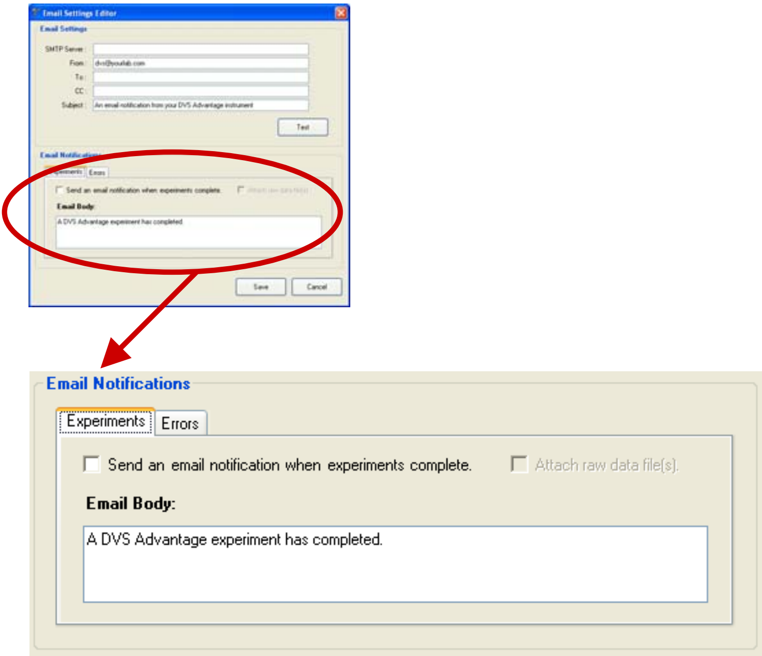
Shortly after the **Test** button is clicked, a test email of the form shown below is sent to the recipients:

Email Notifications



This box allows the user to specify the information that will be contained in, and attached to the emails sent from the DVS Advantage system.

Experiments tab

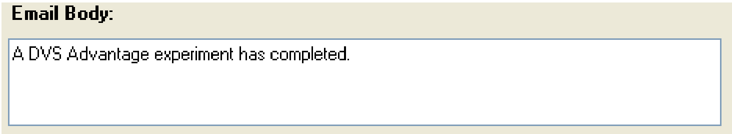


This tab specifies the content of emails sent regarding experiments.

The emails are activated by checking the box entitled **Send an email notification when experiments complete**. Checking this box also activates another check box entitled **Attach raw data file(s)**:

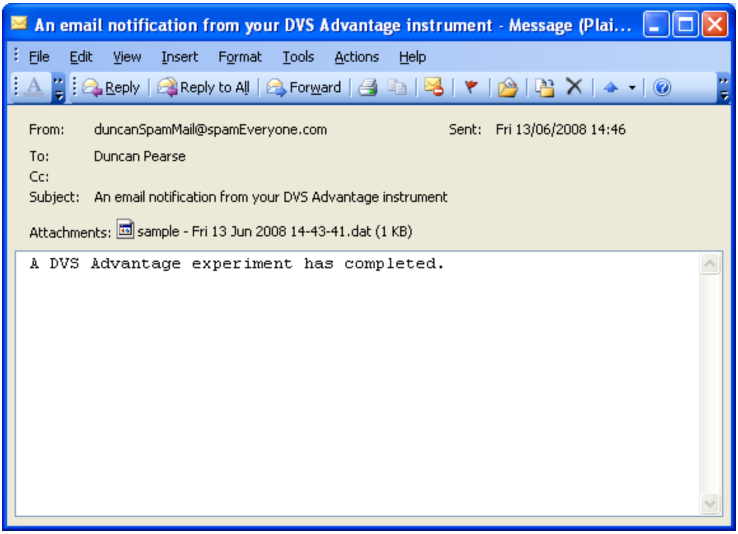
Checking this second box ensures that the DVS raw data file (.dat) is attached to the email when the experiment completes.

The email body box allows the user to specify any text which is to be included in the email notification:

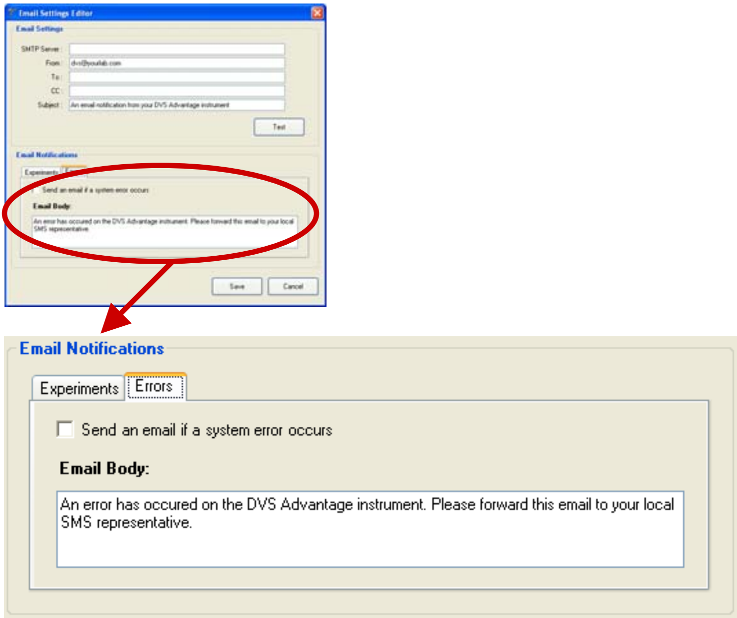


The default text shown above may obviously be edited as desired.

Once all the necessary options are specified, and a DVS experiment has completed, then an email of the form shown below is sent:


An email notification from your DVS Advantage instrument - Message (Plai...
File Edit View Insert Format Tools Actions Help
Reply Reply to All Forward
From: duncanSpamMail@spamEveryone.com
Sent: Fri 13/06/2008 14:46
To: Duncan Pearse
Cc:
Subject: An email notification from your DVS Advantage instrument
Attachments: sample - Fri 13 Jun 2008 14-43-41.dat (1 KB)
A DVS Advantage experiment has completed.

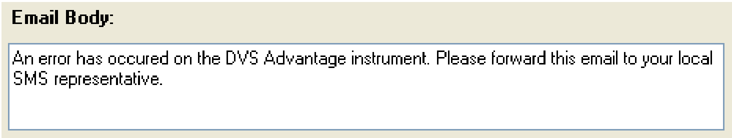
Errors tab


Email Notifications
Experiments
Errors
Send an email if a system error occurs
Email Body:
An error has occured on the DVS Advantage instrument. Please forward this email to your local SMS representative.

This tab specifies the content of emails sent regarding errors.

The emails are activated by checking the box entitled **Send an email if a system error occurs**.

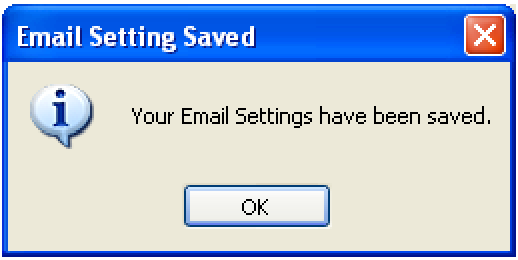
The email body box allows the user to specify any text which is to be included in the email error notification:



The default text shown above may obviously be edited as desired.

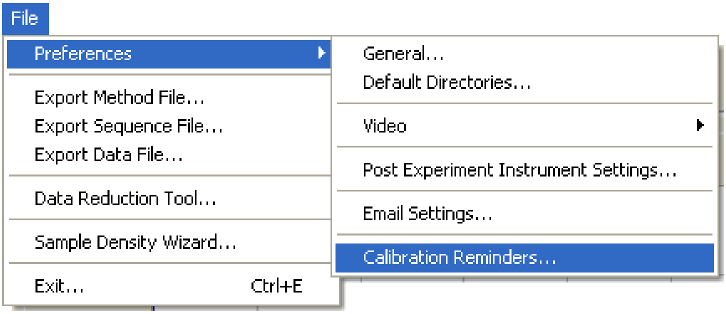
Once all the necessary options are specified, an email of similar form to those shown above is sent in the event of an error occurring.

After the desired email settings have been entered, clicking **Save** calls up the following confirmation message:



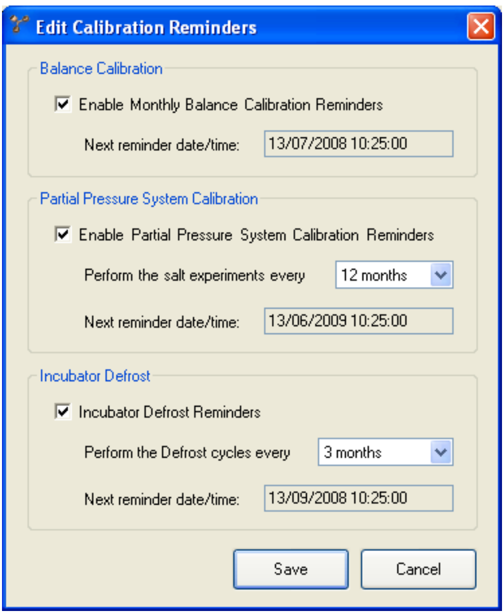
Clicking **OK** returns the DVS Advantage Control software main interface.

9.2.1.6 Calibration Reminders



The DVS Advantage Control Software can be set to provide the user with regular reminders to calibrate the system.

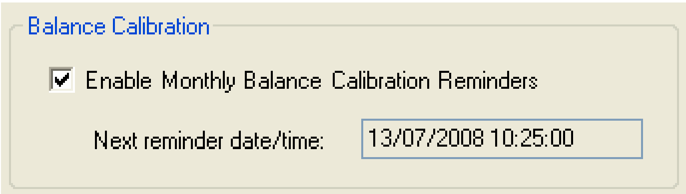
Clicking **File→Preferences→Calibration Reminders….** calls up the **Edit Calibration Reminders** window:



Note: The reminders are triggered by the completion of an experiment. If the system is sitting idle the reminders will not spontaneously activate – the system needs to be in use for the reminders to function.

The chosen reminder settings are retained through control software re-starts.

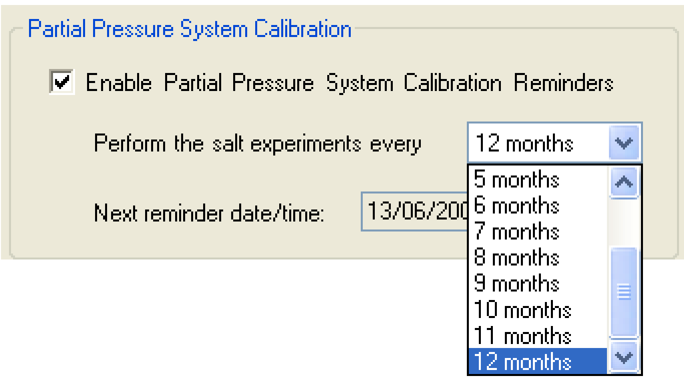
Balance Calibration



This reminder is set to monthly only, and can be activated by ticking the check-box. As shown above, the date on which the next reminder is due appears in the display box – it will be set to one month from when the check-box is ticked.

For more information on balance calibrations, please refer to Sections 7.2 and 9.7.1.2.

Partial Pressure System Calibration

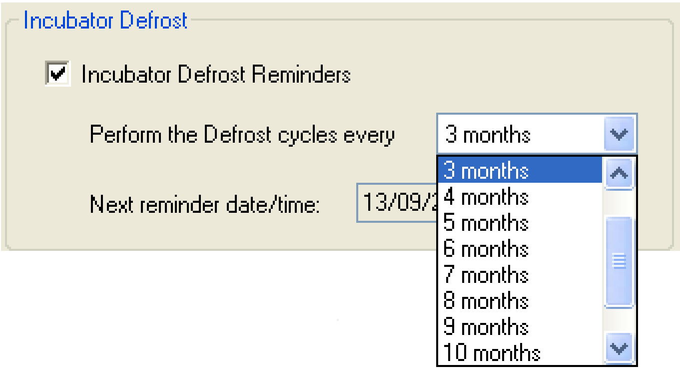


Partial pressure calibrations involve performing salt tests on the system (refer Appendix B, Section 12).

The reminders for these experiments are activated using the check-box indicated, and their frequency can be set using the drop-down menu shown above. Again, the due date for the next reminder is displayed in the window at the bottom of the panel.

It is recommended that the salt tests are performed at least every six months.

Incubator Defrost

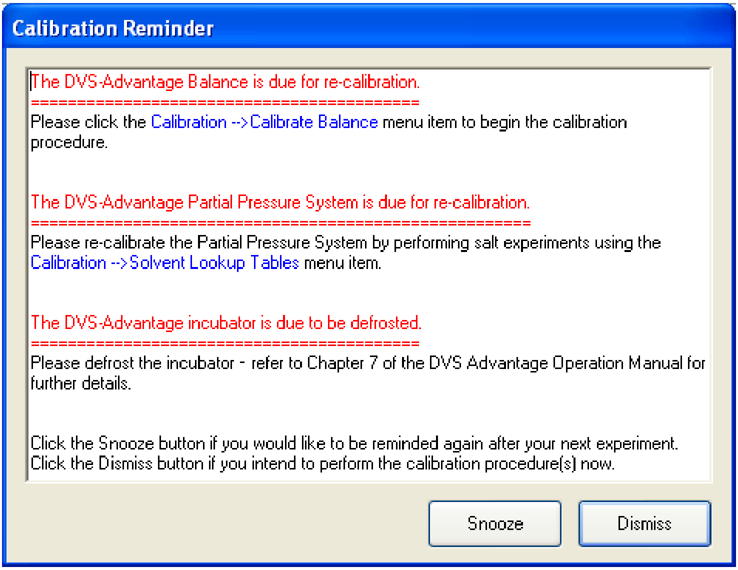


For more information on refrigeration maintenance please refer to Section 7.1.

The reminders for the defrosts are activated using the check-box indicated, and their frequency can be set using the drop-down menu shown above. The due date for the next reminder is displayed in the window at the bottom of the panel.

The window first appears with a defrost frequency of 3 months, which is the recommended minimum – however, the ideal frequency is monthly.

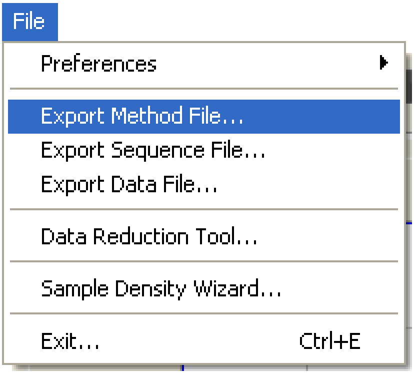
When an experiment has completed and a reminder is due, a Calibration Reminder window appears as shown below.



In the example shown above, all three reminders are activated. The **Calibration Reminder** will display all of the due reminders in one window. Each reminder provides instructions on where to find information on how to perform each calibration.

As indicated, the reminder also informs the user that clicking **Snooze** will ensure that the reminders re-appear after the next DVS experiment has run its course, and that clicking **Dismiss** will automatically update the reminders to appear at their next due date.

9.2.2 Export Method/Sequence/Data File



These functions export the details of a Method, Sequence or Data file to a Text format (.txt) file.

This is useful in cases where, for instance, the user may wish to analyse a Data file in a format other than Excel, or if the user wishes to view the details of a Method or Sequence file without using the Control software.

Clicking any of the **Export Method**, **Sequence** or **Data File** options calls up the following window (in this example, a **Method File** is being exported):

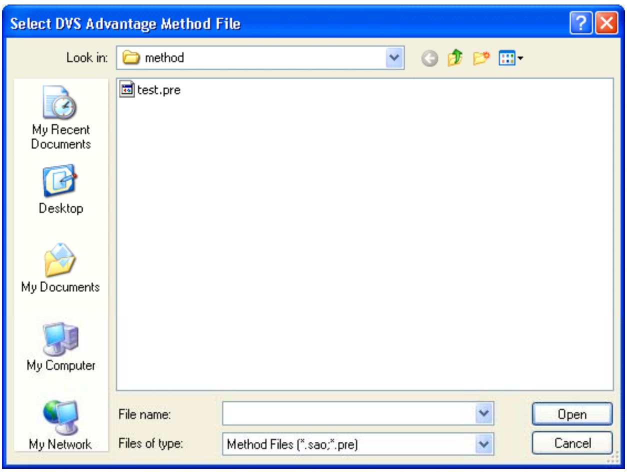


Figure 9.1: The **Select DVS Advantage Method File** window

The **Select DVS Advantage Method File** window opens the directory specified in the **Preferences** toolbar option (Section 9.2.1.2). If desired, the user may navigate to a different directory using the **Look in:** drop-down menu.

Upon selecting the file to be exported and clicking **Open**, the **Enter Target Export Filename** window appears:

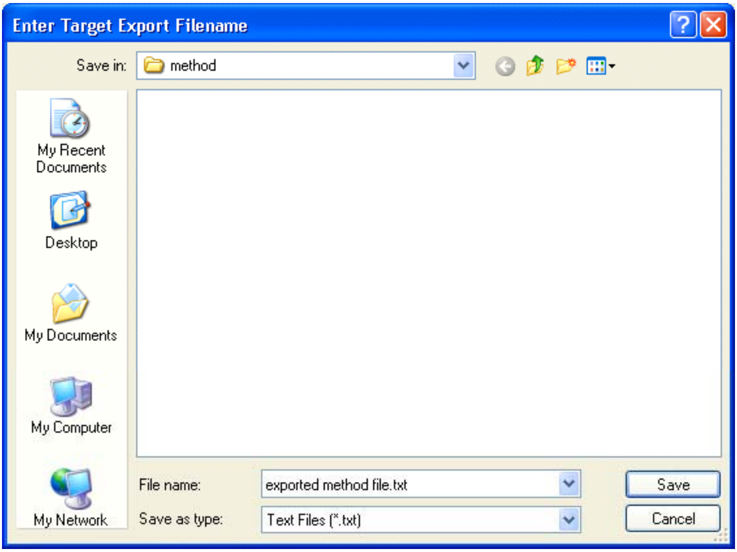
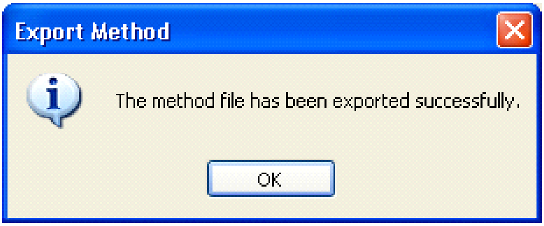


Figure 9.2: The **Enter Target Export Filename** window

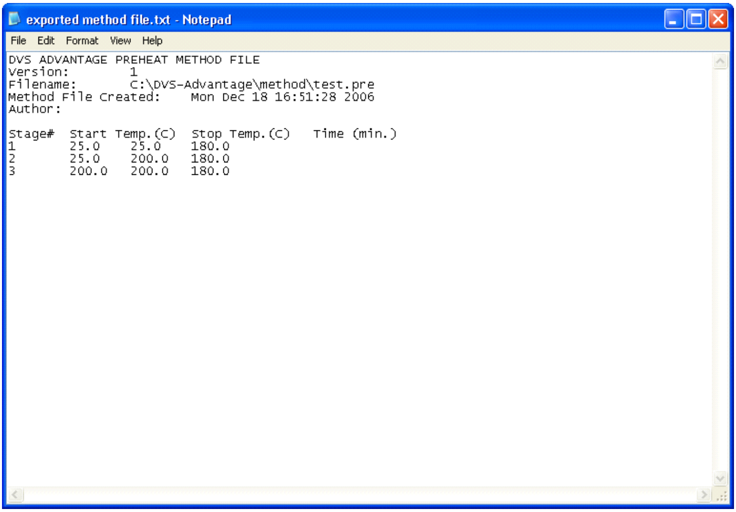
This window allows the user to specify the name of the exported .txt file and to save it in the desired directory. By default, the window navigates to the same directory as that specified in Preferences (as per above). The **Save in:** drop-down menu may be used to navigate to an alternative directory.

Upon clicking **Save**, the following dialog box appears:



Upon clicking **OK**, the Control Software interface is returned.

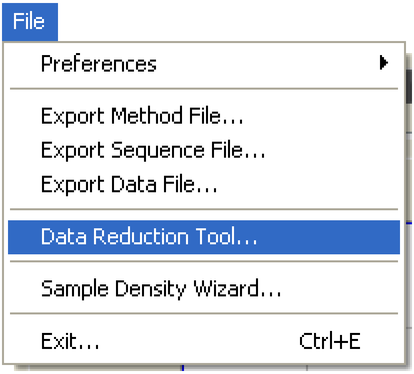
As an example, the .txt format of the exported Method file is shown below:




```
DVS ADVANTAGE PREHEAT METHOD FILE
Version:        1
Filename:       C:\DVS-Advantage\method\test.pre
Method File Created:    Mon Dec 18 16:51:28 2006
Author:

Stage#  Start Temp.(C)  Stop Temp.(C)   Time (min.)
1       25.0    25.0    180.0
2       25.0    200.0   180.0
3       200.0   200.0   180.0
```

9.2.3 Data Reduction Tool



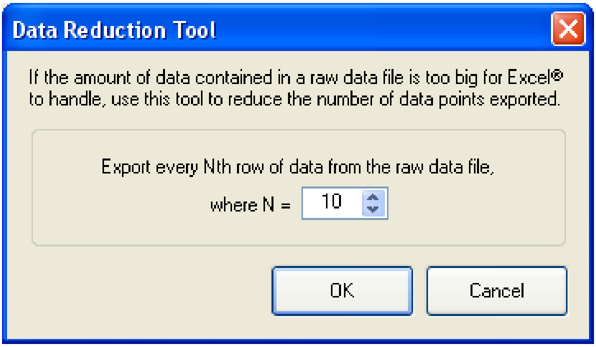
The **Data Reduction Tool** reduces the number of data points exported from a .dat file to Excel. This is especially useful in cases where the .dat file contains too many data points to fit into an Excel file.

Note: Although later versions of Excel can accommodate around one million data points, the current maximum of data points that the DVS Analysis Suite can handle is around sixty thousand, which is the same capacity as older versions of Excel (2003 and earlier).

Large .dat files will therefore need to be reduced to around sixty thousand data points at most if they are to be used with the Analysis Suite software.

Also Note: Using the Data Reduction Tool does not affect the contents of the .dat file – the whole file can be exported to Excel in full at a later stage if desired.

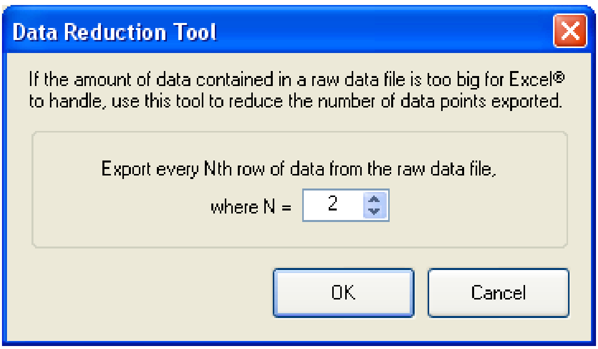
Clicking the **Data Reduction Tool** option calls up the following window:



The window features a data entry box which allows the user to define the level of data reduction required. The window will always open with the default setting of exporting every **10th** row of data to excel. This can be altered to anywhere between a minimum of **1** and a maximum of **600**.

If a figure below 1 is entered, then the software will revert to a default of 1, if a figure above 600 is entered, then likewise a default of 600 will be used.

In the example shown below, every 2nd data point is to be exported:



Once the desired level of data reduction has been specified, clicking **OK** initiates the Export Data File process explained above in Section 9.2.2.

The exported data file, once opened in Excel, will display a reduced number of rows according to that specified in the process above.

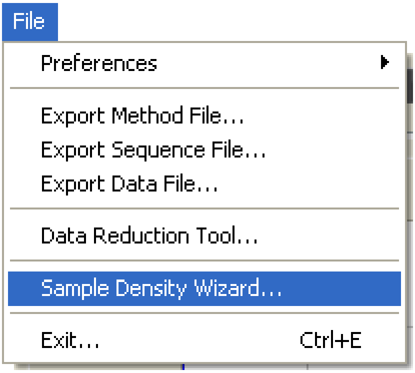
Figure 9.3 below compares the 'reduced data' file (with every 2nd data point exported to Excel) with the same file if it had been exported to Excel with no data reduction:

Time (min)	Mass
0.01	0
1.01	0
2.02	0
3.03	0
4.03	0
5.03	0
6.04	0
7.04	0
8.04	0
9.05	0

Time (min)	Mass
1.01	0
3.03	0
5.03	0
7.04	0
9.05	0
11.06	0
13.06	0
15.07	0
17.07	0
19.08	0

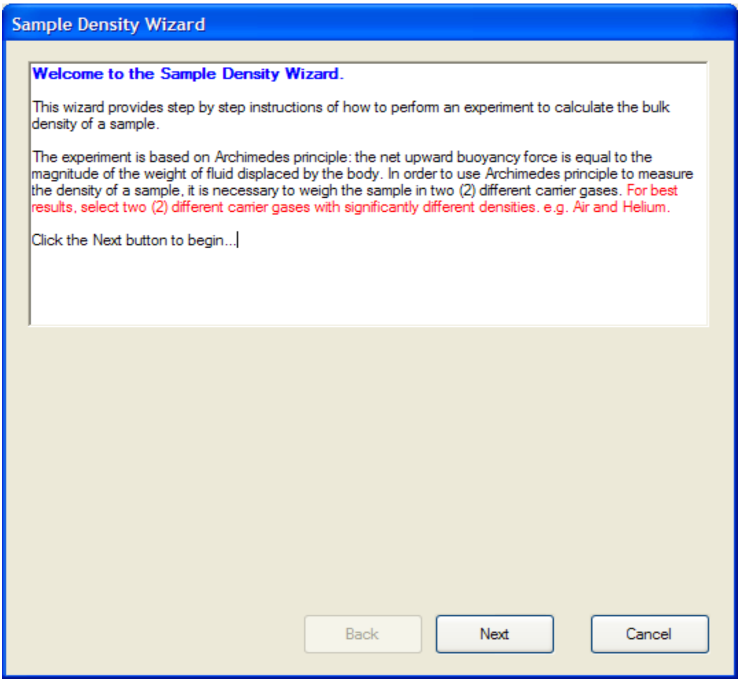
Figure 9.3: The same data file exported to Excel with no data reduction (left) and with every second data point exported (right) – the 'reduced data' will, naturally, occupy half the number of rows of the original.

9.2.4 Sample Density Wizard

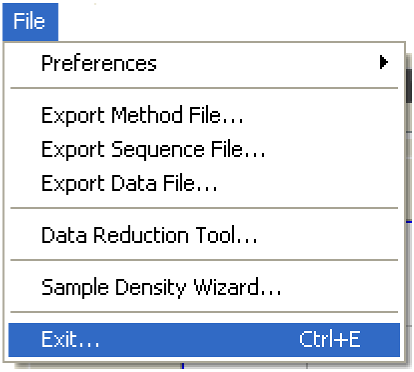


This Wizard provides clear, step-by-step instructions on how to use the DVS system to perform an experiment which allows the calculation of the bulk density of a sample:

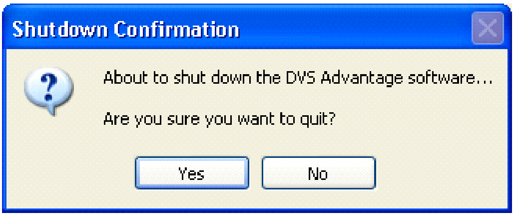
The experiment is based on Archimedes principle, and involves weighing the sample in two different carrier gases. The rest of the Wizard windows are not shown here, they are self-explanatory.



9.2.5 Exit

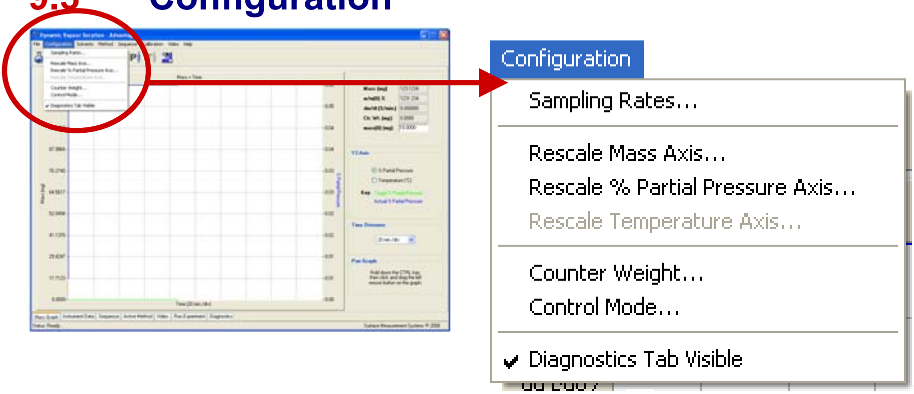


Clicking the **Exit** option calls up the following dialog box:



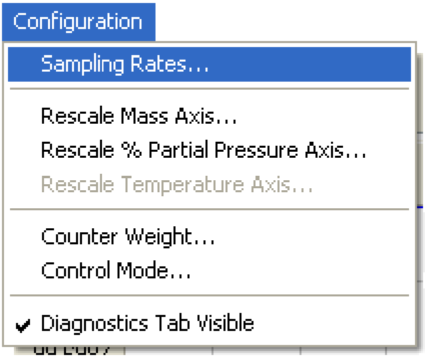
Clicking **Yes** shuts down the Control software. Clicking **No** returns the Control Software interface.

9.3 Configuration

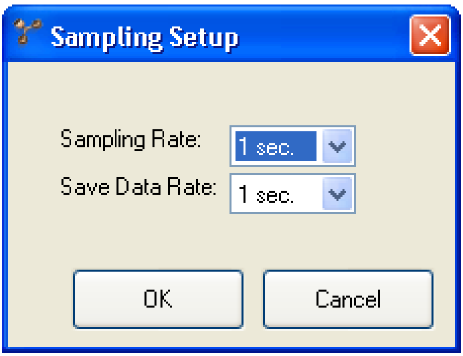


The **Configuration** option provides access to various panels and functions for changing Configuration settings which control the operation of the DVS instrument.

9.3.1 Sampling Rates



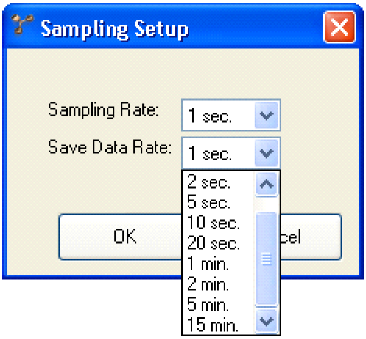
Clicking the **Sampling Rates** option calls up the following dialog box:



This window may be used to set both the interval at which the data is updated on screen (**Sampling Rate**) and the interval at which data is saved to the DVS raw data file on the hard disk (**Save Data Rate**).

The **Sampling Setup** window will open with the values set in the **General Preferences** Settings window (see Section 9.2.1.1).

To alter either value, simply click on the relevant edit box or the arrow next to it to reveal the drop-down menu which shows all the available sampling rates:

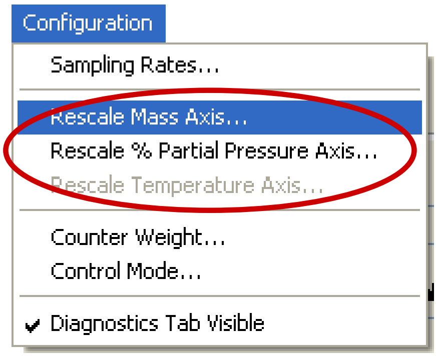


Clicking **OK** will set the new values, while clicking **Cancel** will reinstate the previous rates.

Note: The Sampling Rates menu item is **disabled** whenever data is being saved.

Data saving must be stopped to enable this option for use.

9.3.2 Rescale Mass/Partial Pressure/Temperature Axis

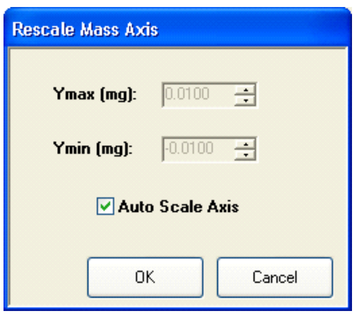


These options allow the vertical axes on the Mass Graph panel (Section 10.3) to be rescaled at any time before, during, or after an experiment.

The **Rescale Mass Axis** option will always be active, whereas either the **Rescale Partial Pressure Axis** OR **Rescale Temperature Axis** option will be active depending upon which is displayed on the graph (Section 10.3.2).

Note: The Rescale Axes options described above may also be accessed by right-clicking on the Mass Graph panel (Section 10.3.6) or by clicking the buttons which appear on the top of the Mass Graph panel (Section 10.3.1.2).

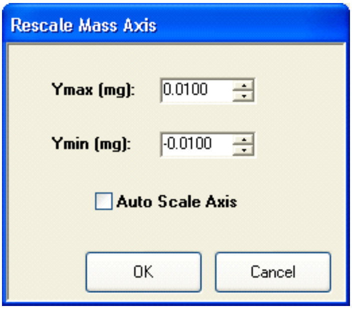
Clicking any of these options calls up the following window:



The example shown above is the **Rescale Mass Axis** window. The window will first appear with the settings defined in the Preferences settings (Section 9.2.1.1).

With the **Auto Scale Axis** option ticked, the axis will automatically scale to fit the entire range of values covered by the plotted parameter.

Un-checking the **Auto Scale Axis** box activates the **Ymax** and **Ymin** windows:

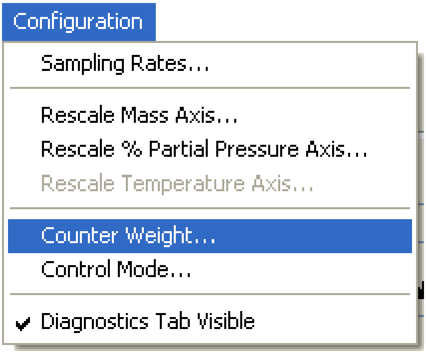


The units displayed for Ymax and Ymin will correspond with the parameter on the axis being rescaled – Mass (mg), Partial Pressure (%) and Temperature (°C).

The desired Ymax and Ymin values can either be entered directly, or the up and down arrows can be used to increase or decrease the limits.

Clicking **OK** then returns the Mass Graph panel showing the newly rescaled graph.

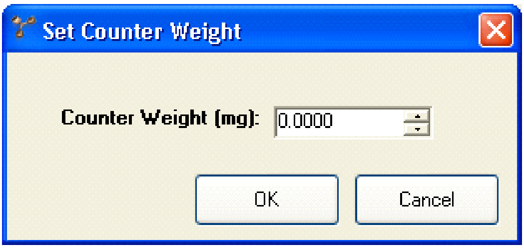
Note: The parameters entered when rescaling the graph will not be retained upon re-starting the Control software. To retain the new parameters, they must be entered in the **File →Preferences** option on the toolbar (see Section 9.2.1).



9.3.3 Counter Weight

This option changes the counter weight value setting in the software for those occasions when a counter weight is being used to analyse larger samples.

Clicking the **Counter Weight** option calls up the following window:

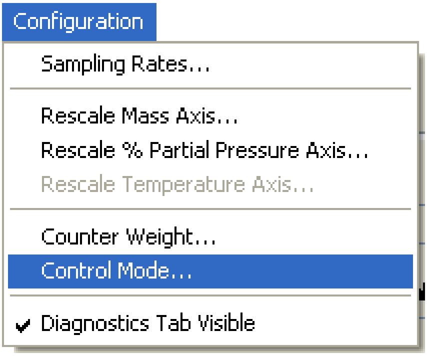


The counter weight value may be entered directly or by using the up and down arrows on the right hand side of the edit box. The value specified will be added to all mass readings from the balance.

Clicking **OK** will save the new data, while clicking **Cancel** will reinstate the previous setting.

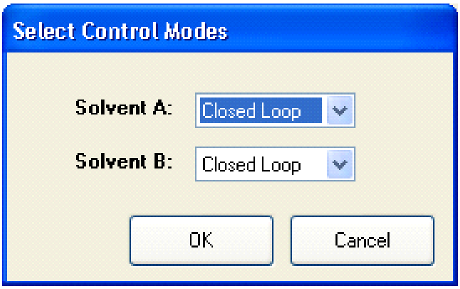
Note: The Counter Weight menu item is **disabled** whenever data is being saved. Data saving must be stopped to enable this option for use.

9.3.4 Control Mode

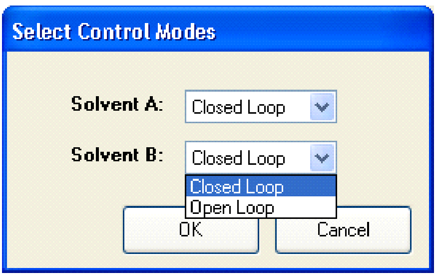


This function determines how the partial pressure generated from Solvent A (left-hand bottle) and Solvent B (right-hand bottle) is regulated.

Clicking the **Control Mode** option calls up the following window:



The control mode for each solvent may be set to **Open Loop** or **Closed Loop**:



Closed Loop: This involves using feedback control to regulate the amount of vapour that is generated in the DVS system.

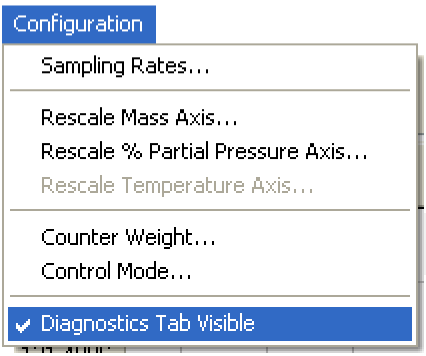
Closed loop control recognises if the actual amount of vapour in the system is incorrect and adjusts the system's vapour generation in order to reach the desired target partial pressure.

Open Loop: This involves using a Solvent Lookup Table (see Appendix D in Section 14) to determine what vapour generation is required for a given solvent at a given temperature to achieve a particular partial pressure. No feedback is used.

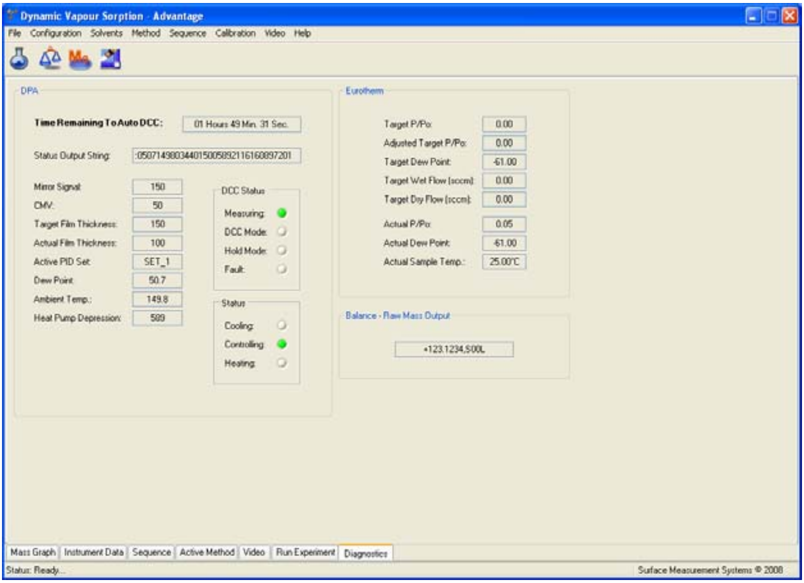
When the **Select Control Modes** window is first opened it displays the options selected in the Preference Settings (Section 9.2.1).

Note: The Control Mode menu item is **disabled** whenever a Sequence is loaded or data is being saved. The Sequence must be cleared or data saving stopped to enable this option for use.

9.3.5 Diagnostics Tab Visible

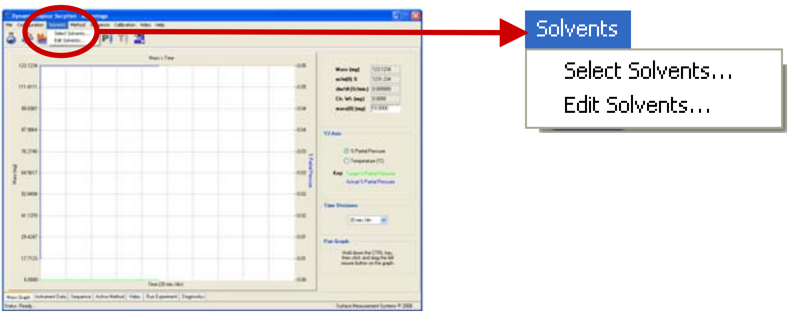


When this option is checked, the Diagnostics panel (shown below) is visible on the DVS Advantage Control Software main interface. When unchecked, the panel is hidden.



See Section 10.10 for more information on the Diagnostics panel.

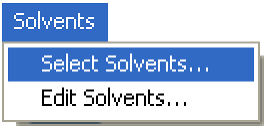
9.4 Solvents



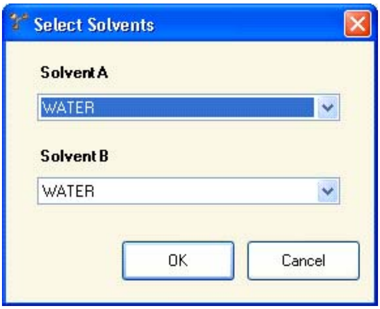
This option allows the user to specify which solvents are installed in the DVS. It also allows the user to update a database of solvents which may be used in the system.

Note: The Solvents menu is disabled whenever data is being saved. Data saving must be stopped before the menu may be used.

9.4.1 Select Solvents

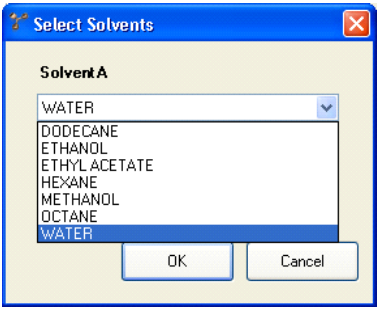


(See also Section 10.2.1).
Selecting this option calls up the following window:



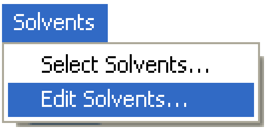
The **Select Solvents** window allows the user to specify which solvents are present in the system – Solvent A (left bottle) and Solvent B (right bottle). Refer also to Section 6.3.2.1 which details the changing of solvents in the DVS.

Clicking anywhere in the edit boxes or on the down arrows to their right will reveal a drop-down list of all the available solvents which are stored in the Control Software database:



Note: It is very important that the software correctly 'knows' which solvents are present in the system – otherwise it will cause the DVS system to generate incorrect partial pressures.

9.4.2 Edit Solvents



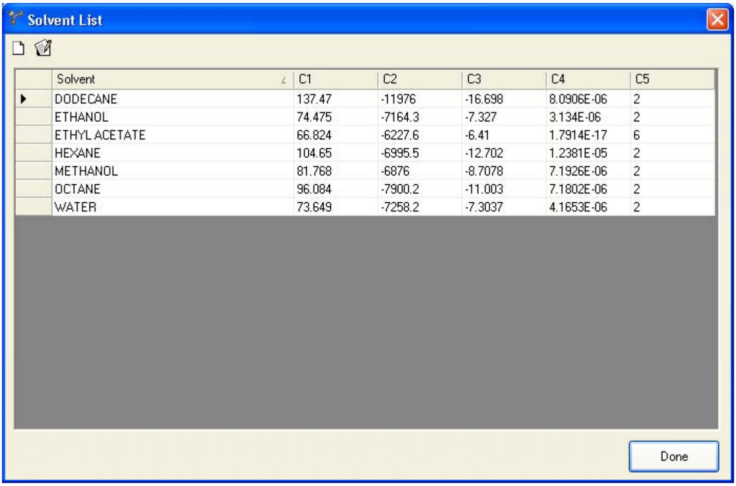
Note: The DVS Advantage uses an equation from Perry's Chemical Engineers Handbook (7th Edition) Table 2.6 to convert from Dew Point to Partial Pressure and vice versa.

This conversion equation requires five constants for each solvent involved in the calculation.

The constant values for a particular solvent can be found in **Perry's Chemical Engineers Handbook (7th Edition) Table 2.6**

When adding a solvent to the solvent database (Section 9.4.2) which is not contained in the Perry's table mentioned above, please refer to Appendix F (Section 16).

Clicking this option calls up the **Solvent List** window:



Solvent List

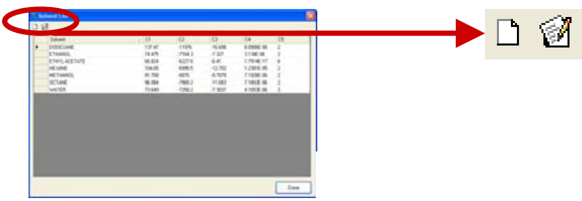
	Solvent	C1	C2	C3	C4	C5
▸	DODECANE	137.47	-11976	-16.698	8.0906E-06	2
	ETHANOL	74.475	-7164.3	-7.327	3.134E-06	2
	ETHYL ACETATE	66.824	-6227.6	-6.41	1.7914E-17	6
	HEXANE	104.65	-6995.5	-12.702	1.2381E-05	2
	METHANOL	81.768	-6876	-8.7078	7.1926E-06	2
	OCTANE	96.084	-7900.2	-11.003	7.1802E-06	2
	WATER	73.649	-7258.2	-7.3037	4.1653E-06	2

Done

The **Solvent List** shown above displays the constants used in the Perry's Handbook equation to calculate their partial pressures. This table allows the user to configure the solvents that may be used in the DVS system.

9.4.2.1 Creating and Editing Solvent entries

There are two buttons on the Solvent List toolbar: **Create a New Solvent Entry** and **Edit the Selected Solvent**, as shown below:



Create a new solvent entry

Clicking this button calls up the **Enter New Solvent** window:

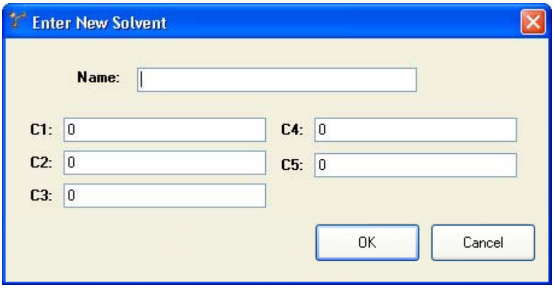


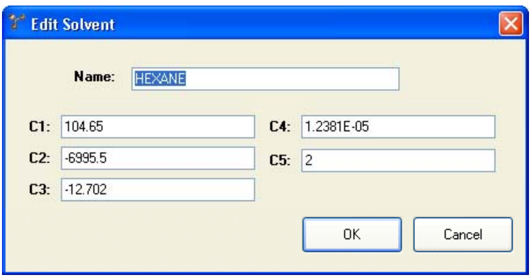
Figure 9.4: The **Enter New Solvent** window

This button may be used to create a New Solvent entry in the Solvent list. The solvent name and its corresponding constant values from **Perry's Chemical Engineers Handbook (7th Edition) Table 2.6** must be entered in the relevant edit boxes.

Clicking **OK** will then add the new entry to the list of available solvents.

Edit the selected Solvent 

Clicking this button calls up the **Edit Solvent** window:



The window will display whichever solvent was highlighted in the Solvent List.

This button may be used to edit the settings for an existing solvent entry in the Solvent list.

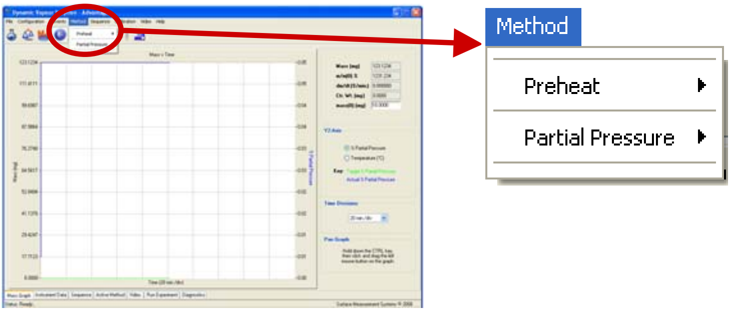
Note: Using this option should only be necessary if a mistake was made when previously entering the settings for a solvent.

The solvent name may be altered (except in the case of WATER) and also the corresponding constant values that come from **Perry's Chemical Engineers Handbook (7th Edition) Table 2.6.**

After the desired changes have been made, clicking **OK** will update the solvent details in the Solvent List.

9.5 Method

The **Method** menu can be used to create new DVS Methods OR to edit existing DVS Methods.



A Method file contains a series of pre-defined Preheating or Partial Pressure Stages. Methods are loaded in series to form a Sequence (Sections 9.6 and 10.5), which allows a

DVS experiment to run automatically without the need for any user involvement.

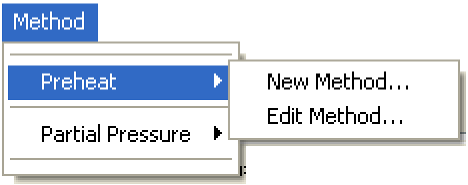
Note: The Method menu may also be called up by selecting the **Active Method** tab and right-clicking the mouse anywhere on the panel (see Section 10.6.4).

There exist three different types of DVS Advantage Methods:

Preheat Method:	Used to dry out the sample by controlling the preheat temperature over user-defined time periods. *The maximum preheat temperature setpoint is 200°C*.
Partial Pressure Method:	Used to control the amount of vapour that is sent into the sample area.
DCC method:	In order to maintain the accuracy of the Optical Sensor reading it is recommended that the user perform a Dynamic Contamination Correction (DCC) cycle prior to running a partial pressure method. To do this, simply load the "Clean.dcc" Method that can be found in the DVS Advantage Method directory into the Sequence immediately before the Method that is to be run. See Appendix E (Section 15) for more information regarding the Optical Sensor Dynamic Contamination Correction procedure.

Note: Running a Clean.dcc Method before each new Partial Pressure method in a Sequence is STRONGLY recommended.

9.5.1 Preheat



This option allows the user to create and edit Preheat Methods for DVS experiments.

Note: The maximum preheat temperature setpoint is 200°C.

9.5.1.1 New Method

Clicking **Method→Preheat→New Method…** calls up the **Preheat Method Editor** window:

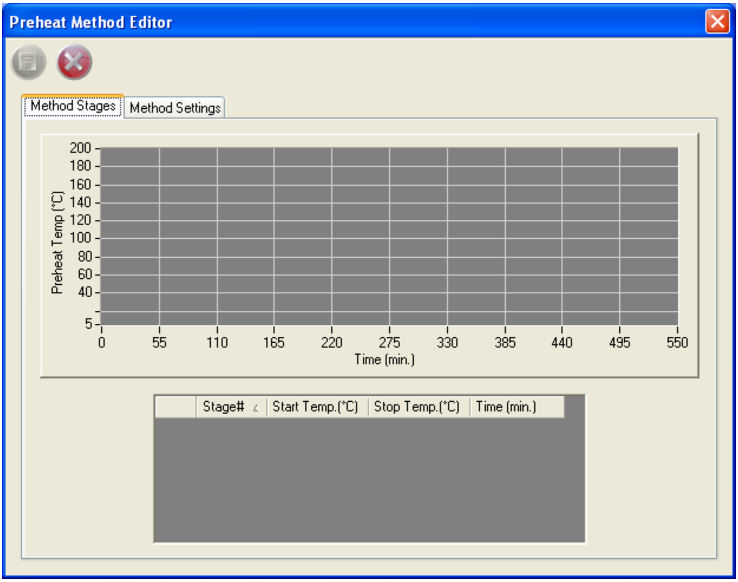
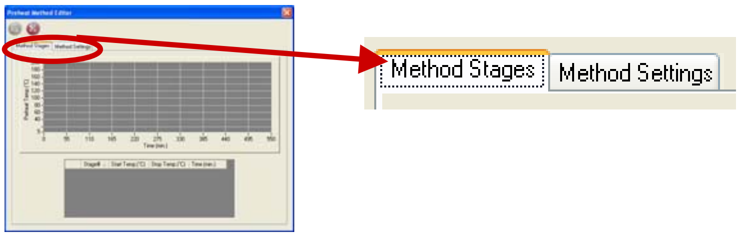
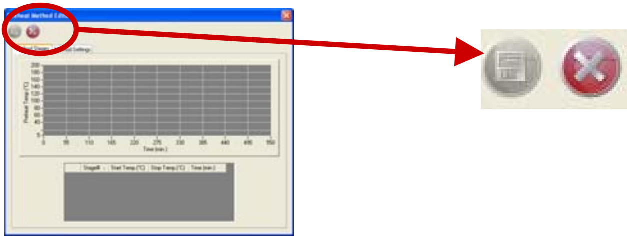


Figure 9.5: The **Preheat Method Editor** window

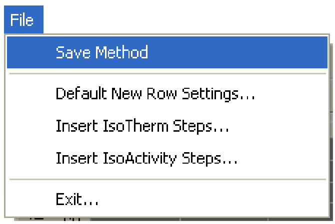
The **Preheat Method Editor** window looks similar to the **Active Method** panel on the main DVS Advantage dialog screen (Section 10.6) – it similarly features a **Method Stages** tab and a **Method Settings** tab.



At the top of the window are two buttons which allow the user to save Preheat Methods and to close the editor:



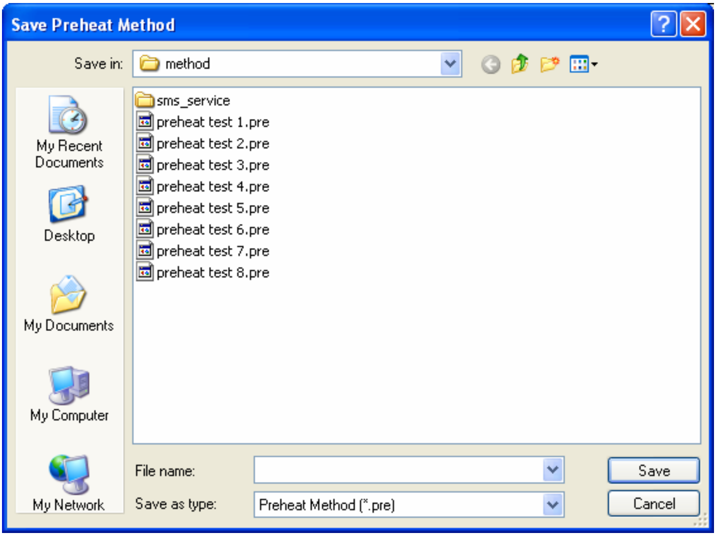
Save Method 



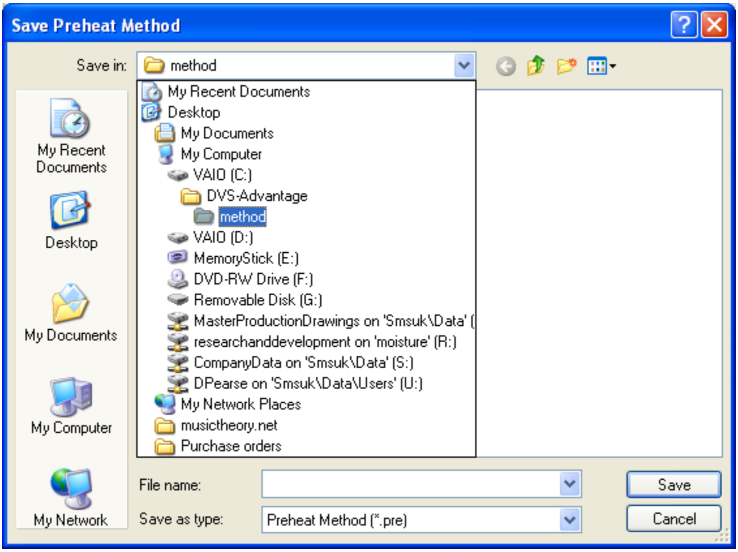
Clicking the **Save Method** button  saves a Preheat Method to a file after it has been formulated in the **Method Editor**.

Note: The **Save Method** option only becomes active after a Method has been created (see sections below).

Once the Save Method button has been pressed, a **Save Preheat Method** window is opened, requiring the user to name the new Preheat Method file:

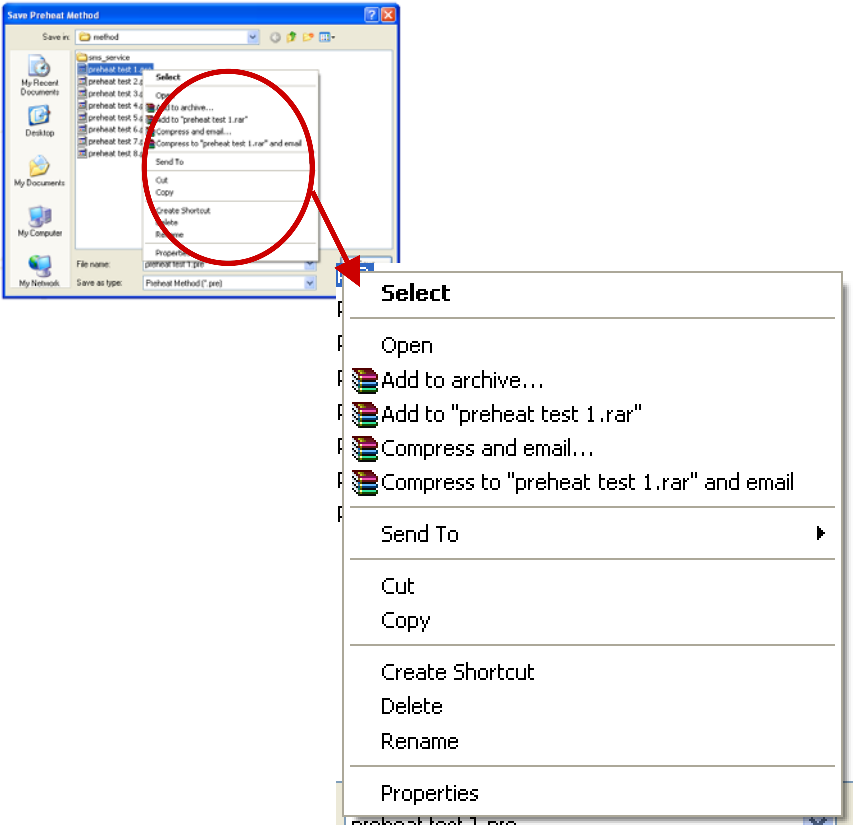


The **Save Preheat Method** window automatically navigates to the directory specified in **File → Preferences → Default Directories** (Section 9.2.1.2), but a different location may be specified using the **Save in** drop-down menu:



As the pictures above show, **Preheat Method** files are saved with the extension **.pre** (ie; **pre**heat).

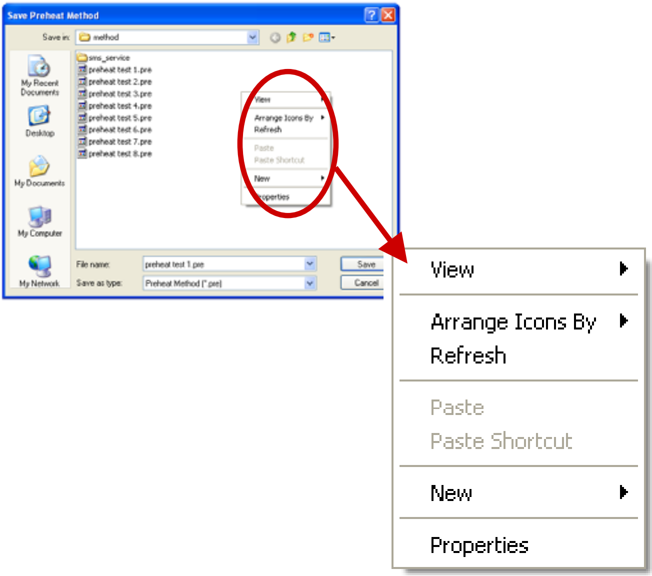
Normal Windows Explorer functions are available in the **Save Preheat Method File** window. Right-clicking on a pre-existing file in the window calls up the following menu:



Files can be deleted, renamed, etc, before the new Preheat Method file is saved.

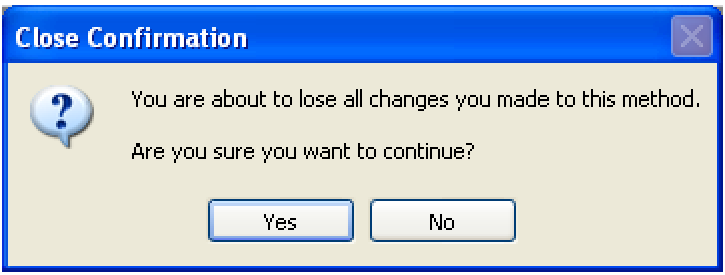
Right-clicking in a blank space in the window calls up the following menu:

After the new Preheat Method file has been named in the **Filename** box, clicking **Save** then saves the new Preheat Method and returns to the main DVS Advantage dialog screen.



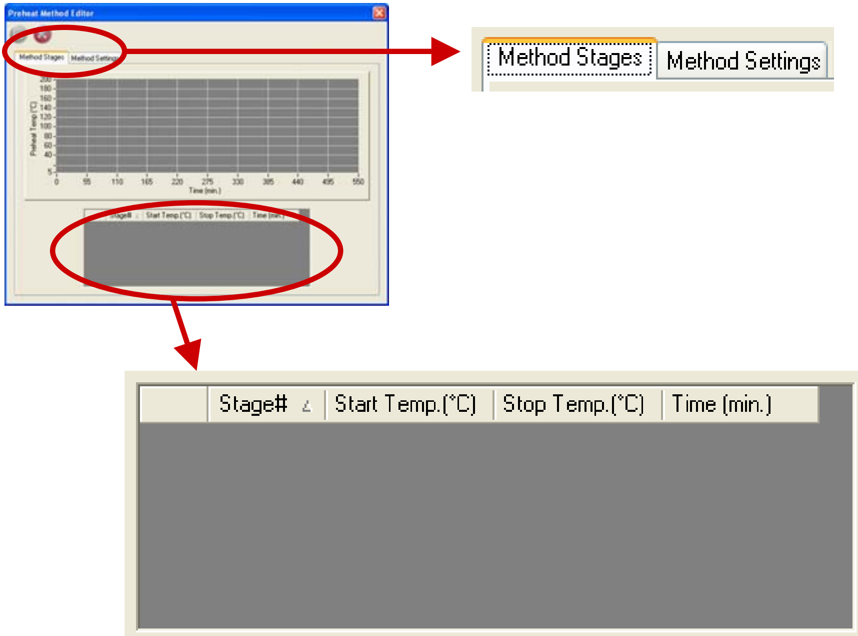
Close Method Editor 

Clicking the **Close Method Editor** button  before the Method has been saved will call up the following message:



Clicking **Yes** returns the DVS Advantage main interface, while clicking **No** returns **the Preheat Method Editor** window.

Method Stages Tab: Creating a Preheat Method



Rows Data

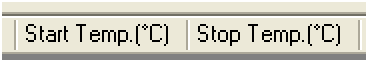
A Preheat Method is created by defining Rows (Stages) in the **Temperature Table** of the **Method Stages** tab. Each row (Stage) defines the following parameters:

Stage.#

This denotes the number of the Stage and cannot be edited - new Stages are added or appended, the numbers will change automatically.

Start and Stop Temp (°C)

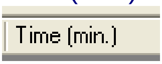
These values define the Temperatures at which each Preheat stage begins and ends, respectively.



If the Start and Stop Temp values are the same, then the Stage will be set at one constant temperature. If the values are different, then the Stage will ramp between the Start and Stop temperatures.

The minimum allowable Temp setpoint is 5°C
The maximum allowable Temp setpoint is 200°C

Time (min.)



This defines the amount of time taken (in minutes) for the Stage to go from the Start Temp to the stop Temp.

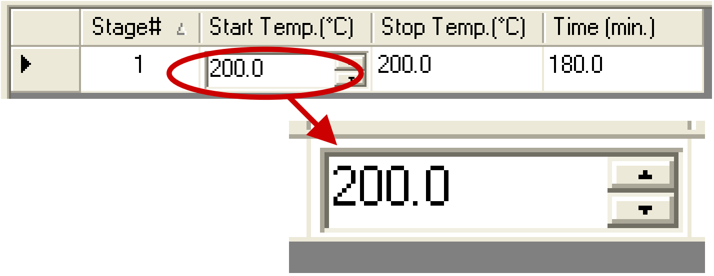
If the Start and Stop temperatures are the same, then the Time will define how long the Stage lasts on this constant temperature.

The minimum allowable Time setpoint is 2 min.
The maximum allowable Time setpoint is 9999 min.

If the Start and Stop temp are different, then the Time will define the period over which the temperature ramp occurs.

Performing operations upon Temperature Table data

Once Row(s) of data have been inserted into the Temperature Table (see below), the **Start** and **Stop** Temp, and the **Time** values may be edited by clicking on them – this activates a data entry box:



The desired values may be entered either directly or by using the toggle arrows – the arrows change the values by 1.0 °C or minute on each click. The newly entered value is confirmed by either clicking **Return** or selecting another cell in the table.

The values entered do not need to be whole numbers – decimal values are also accepted:

	Stage#	Start Temp.(°C)	Stop Temp.(°C)	Time (min.)
▶	1	180.7	132.5	169.8

In addition, it is possible to move from one cell to another in the **Temperature Table** by using the arrow keys on the control PC's keyboard (as happens in Microsoft Excel®).

Each cell is activated for editing when selected using the arrow key:

Stage#	Start Temp.(°C)	Stop Temp.(°C)
1	20.0	20.0
2	30.0	30.0
3	40.0	40.0

Stage#	Start Temp.(°C)	Stop Temp.(°C)
1	20.0	20.0
2	30.0	30.0
3	40.0	40.0

Stage#	Start Temp.(°C)	Stop Temp.(°C)
1	20.0	20.0
2	30.0	30.0
3	40.0	40.0

Selecting multiple Rows

Multiple rows may be highlighted (ie; *selected*) one at a time by holding down the **Ctrl** key while clicking the panels to their left, as shown below:

Stage#	Start Temp.(°C)	Stop Temp.(°C)	Time (min.)
1	20.0	20.0	180.0
2	30.0	30.0	180.0
3	40.0	40.0	180.0
4	50.0	50.0	180.0
5	60.0	60.0	180.0

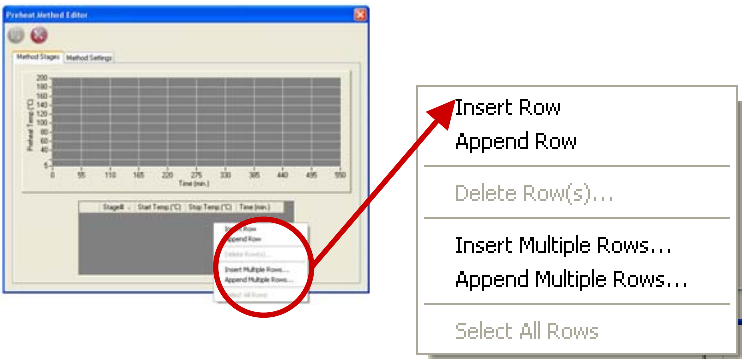
Alternatively, a group of rows may be selected at once by holding down the **Shift** key while clicking the first and last row in the group – all rows in between them will also be selected:

Stage#	Start Temp.(°C)	Stop Temp.(°C)	Time (min.)
1	20.0	20.0	180.0
2	30.0	30.0	180.0
3	40.0	40.0	180.0
4	50.0	50.0	180.0
5	60.0	60.0	180.0

Finally, a group of Rows can also be selected by highlighting one Row and dragging the cursor up or down along the Rows to be selected.

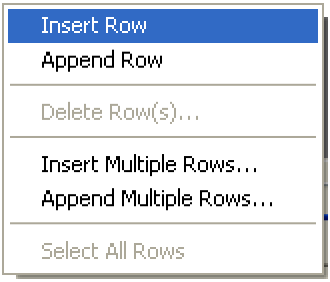
Inserting, Appending and Deleting Rows

Rows are added to, and deleted from the **Temperature Table** by right-clicking on the body of the table, which calls up the following menu of options:

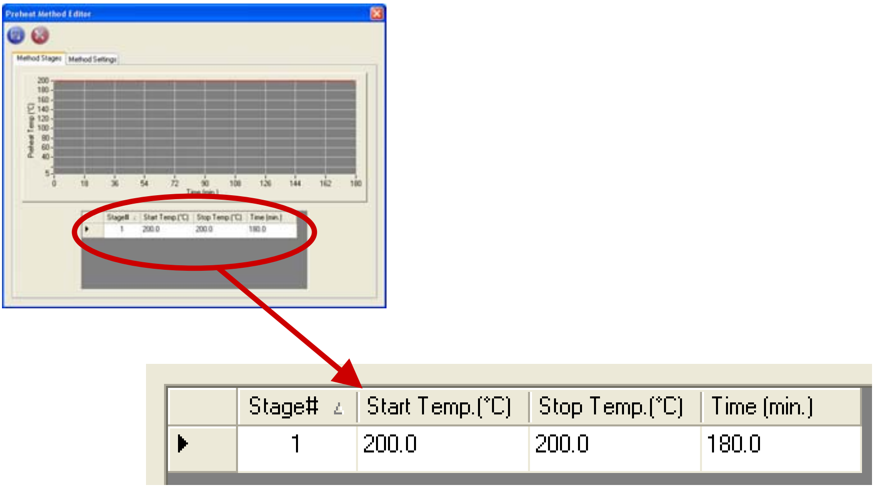


Note: The Delete Row(s) and Select All Rows options are inactive when the Temperature Table is empty.

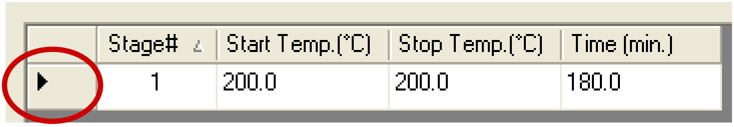
Insert Row



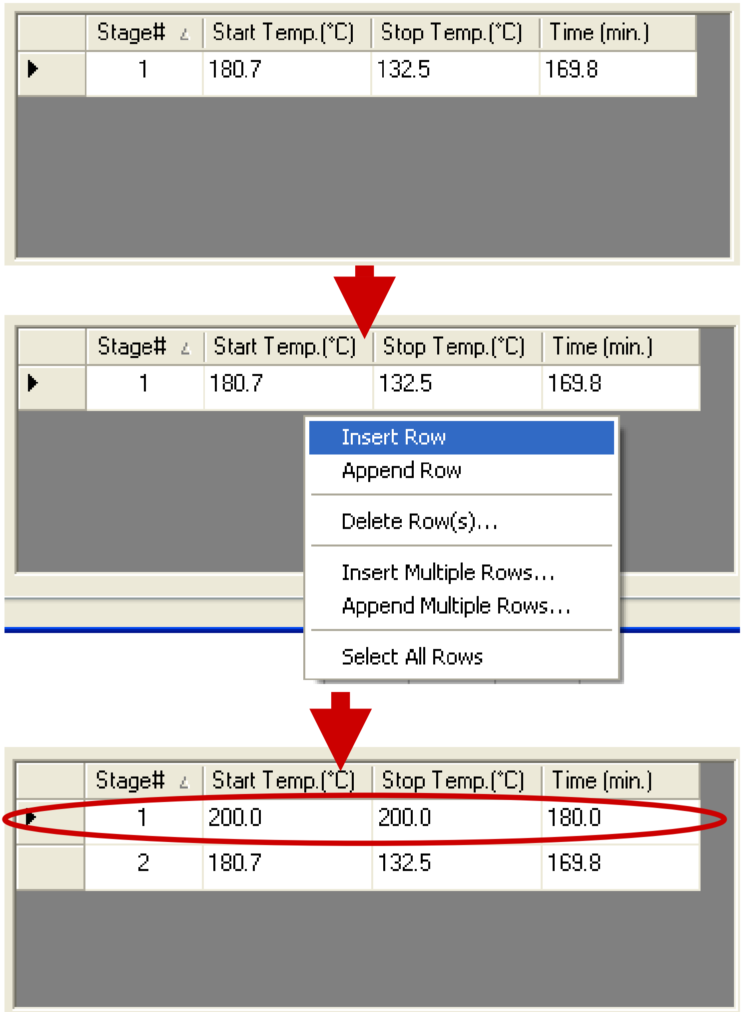
Clicking this option inserts a new row in the **Temperature Table** with default values (200°C for the **Start** and **Stop RH** values, **Time** equal to 180minutes):



The newly inserted row is denoted the *selected* row by means of an arrow appearing in its left-most panel:

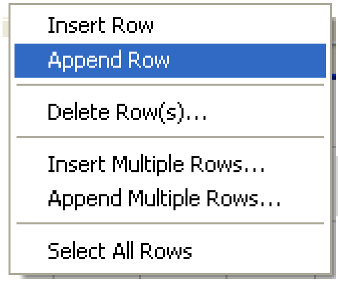


If the **Insert Row** option is used subsequently, the new row will be inserted *above* the *selected* row:

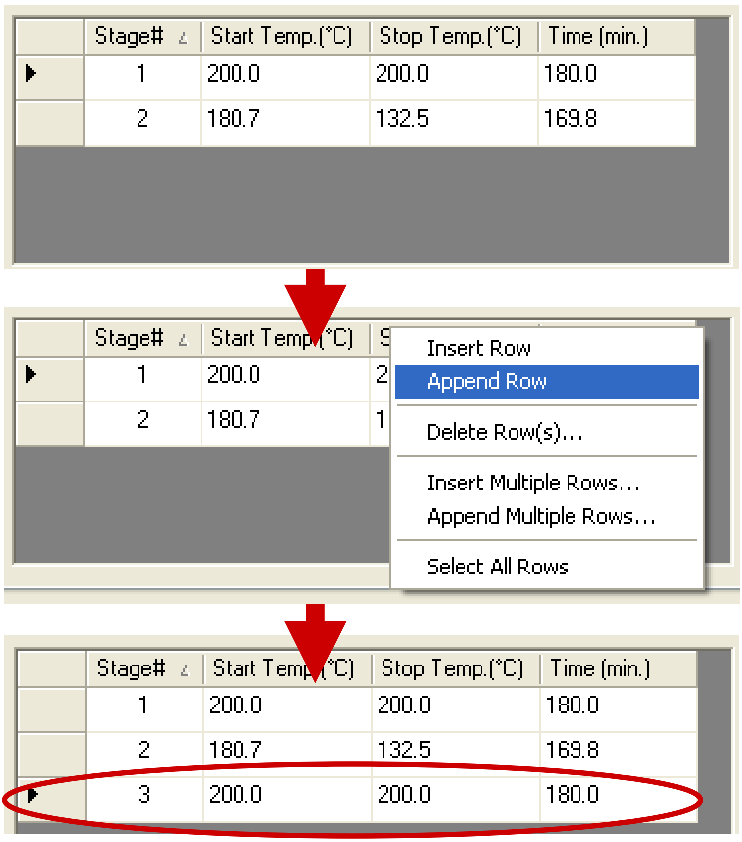


As shown above, the newly inserted Row appears with default values, and is now the selected row, as indicated by the arrow icon .

Append Row

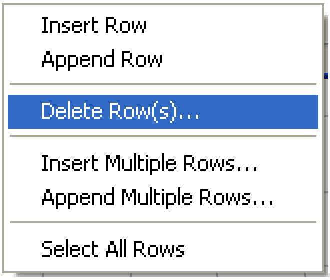


This function adds a new Row with default values to the end of the current list, regardless of which Row is currently selected:



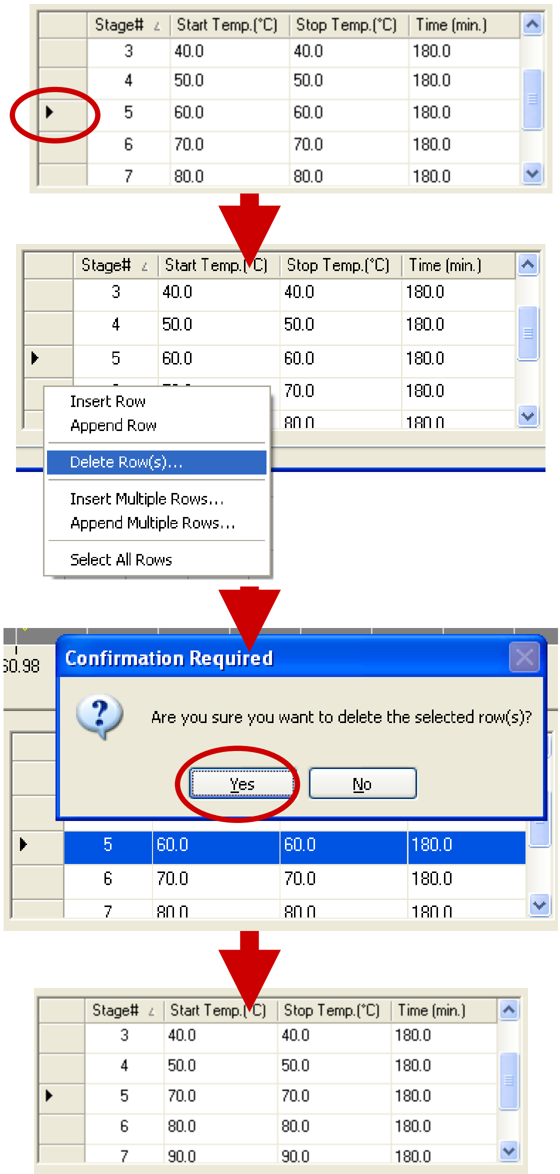
As shown above, the newly appended Row appears with default values, and is now the selected row, as indicated by the arrow icon .

Delete Row(s)…

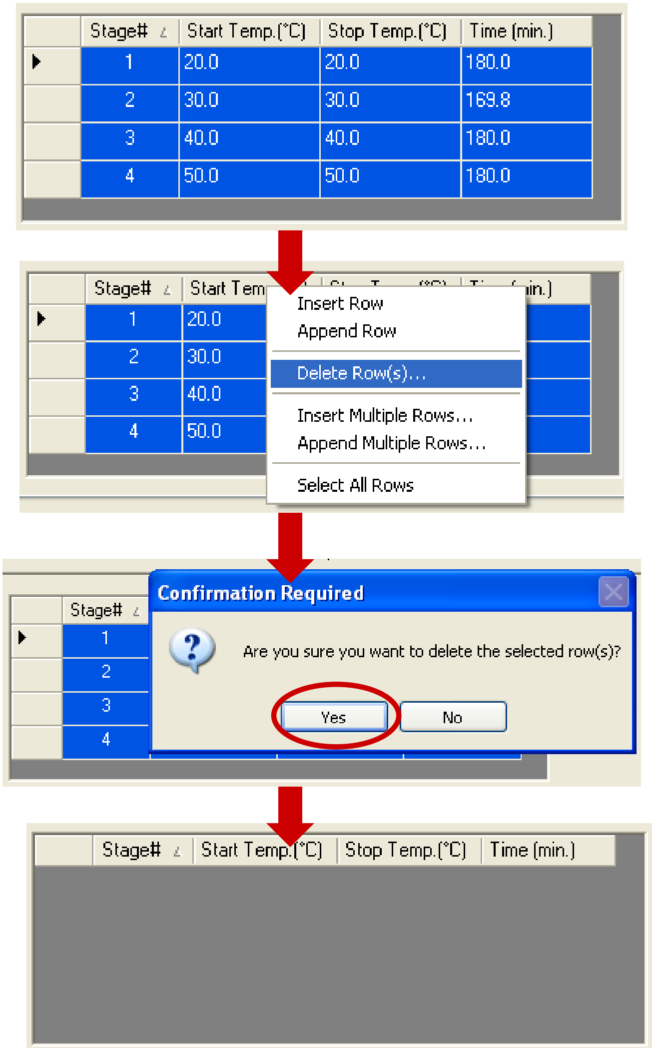


This function will delete the Row which is selected with the arrow , or multiple Rows if they are highlighted (in blue, as discussed above).

Deleting a Row selected with the arrow  takes place as follows:


Stage#
Start Temp.(°C)
Stop Temp.(°C)
Time (min.)
Insert Row
Append Row
Delete Row(s)...
Insert Multiple Rows...
Append Multiple Rows...
Select All Rows
Confirmation Required
Are you sure you want to delete the selected row(s)?
Yes
No

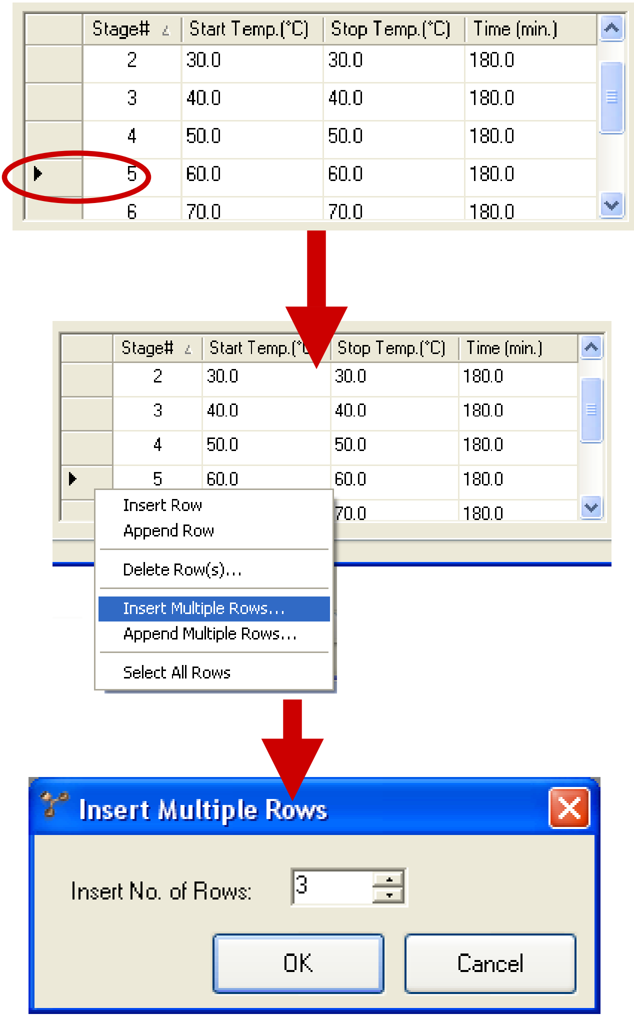
Once multiple Rows have been selected the deletion process may take place as follows:



Insert Multiple Rows

This function inserts one or more rows containing default values above the selected row.

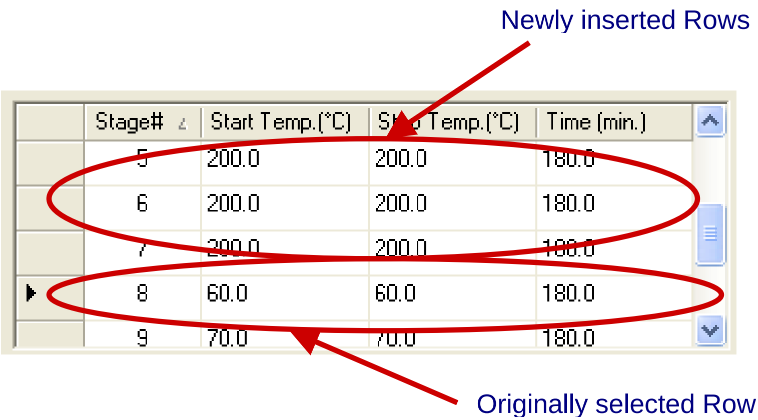
The process is as follows:



The Insert Multiple Rows window appears with a default value of 1 Row. Up to 50 Rows may be entered into the data entry box.

Upon entering the desired number of rows (in this case, 3) and clicking **OK**, the Preheat Method Editor is returned, with the new Rows inserted above the selected Row.

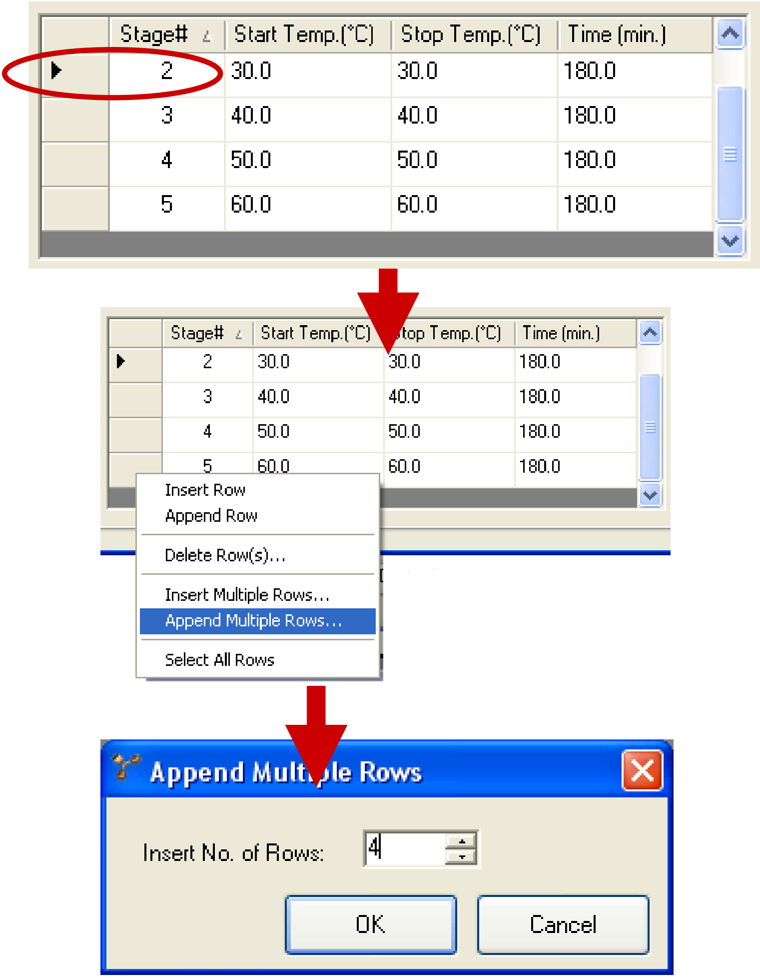
Note that the Row which was originally selected (in this case, the Row with 60°C Temp) remains selected after the multiple row insertion:



Append Multiple Rows

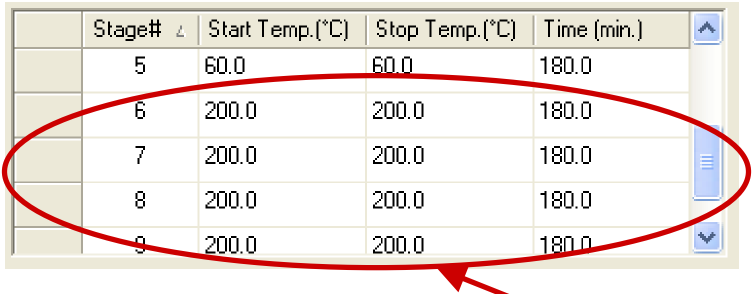
This function appends one or more rows containing default values to the end of the **Temperature Table**, regardless of which Row is selected.

The process is as follows:



The Append Multiple Rows window appears with a default value of 1 Row. Up to 50 Rows may be entered into the data entry box.

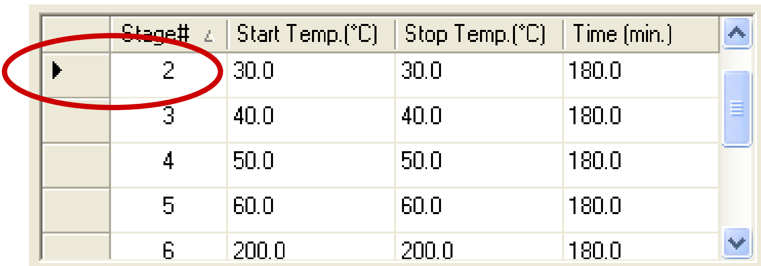
Upon entering the desired number of rows (in this case, 4) and clicking **OK**, the Preheat Method Editor is returned, with the new Rows appended to end of the Temperature Table.



	Stage#	Start Temp.(°C)	Stop Temp.(°C)	Time (min.)
	5	60.0	60.0	180.0
	6	200.0	200.0	180.0
	7	200.0	200.0	180.0
	8	200.0	200.0	180.0
	9	200.0	200.0	180.0

Appended Rows

The Row which was originally selected (in this case, Row 2, or Stage#2) remains selected after appending the multiple Rows:

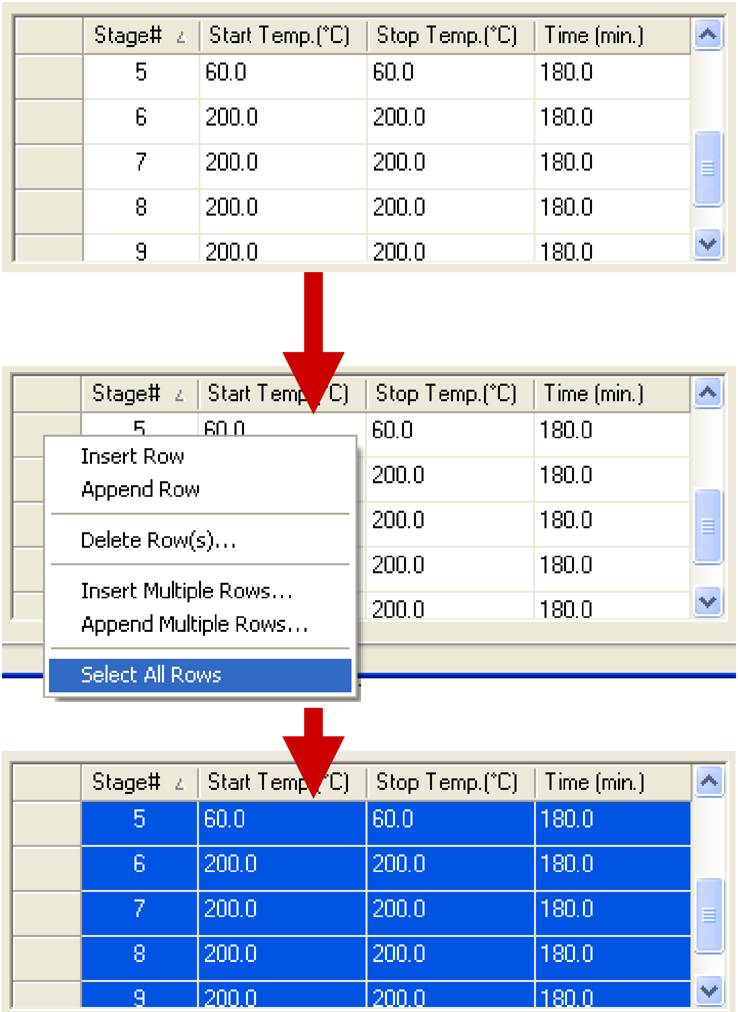


	Stage#	Start Temp.(°C)	Stop Temp.(°C)	Time (min.)
▶	2	30.0	30.0	180.0
	3	40.0	40.0	180.0
	4	50.0	50.0	180.0
	5	60.0	60.0	180.0
	6	200.0	200.0	180.0

Select All Rows

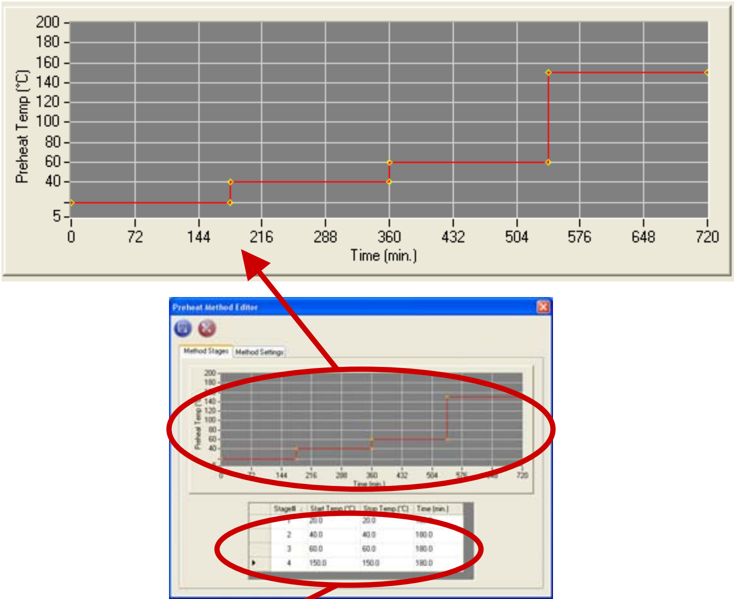
This option allows automatically selects all Rows that are in the Temperature Table, which is useful for carrying out subsequent operations, such as deletion

The process is as follows:



Preheater Graph

The **Preheater Graph** plots temperature vs time, and reflects the data entered into the **Temperature Table**:



	Stage#	Start Temp.(°C)	Stop Temp.(°C)	Time (min.)
	1	20.0	20.0	180.0
	2	40.0	40.0	180.0
	3	60.0	60.0	180.0
▶	4	150.0	150.0	180.0

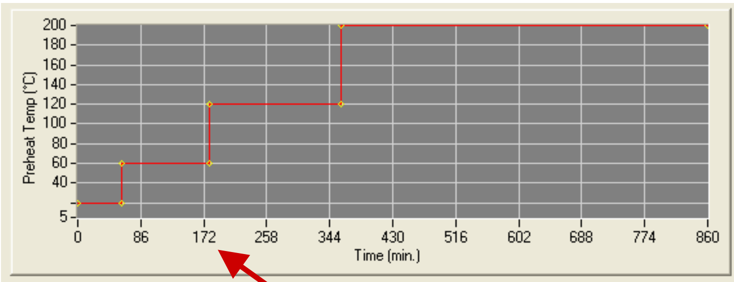
Both the Preheater Graph and Temperature Table also appear in the **Active Method Panel**, which displays running Methods. More information can be found in Section 10.6.

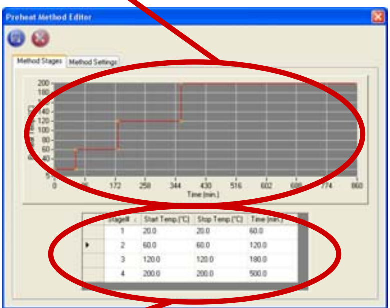
Creating temperature Steps and Ramps

Using a Preheat Method, the temperature can be made to change in discreet steps or to ramp gradually from a Start Temp to a Stop Temp over a specified period of time.

Temperature steps

Temperature steps are created simply by making the **Start** and **Stop Temps** of each Stage equal. The **Time** setting of each Stage then defines how long each temperature step will last:

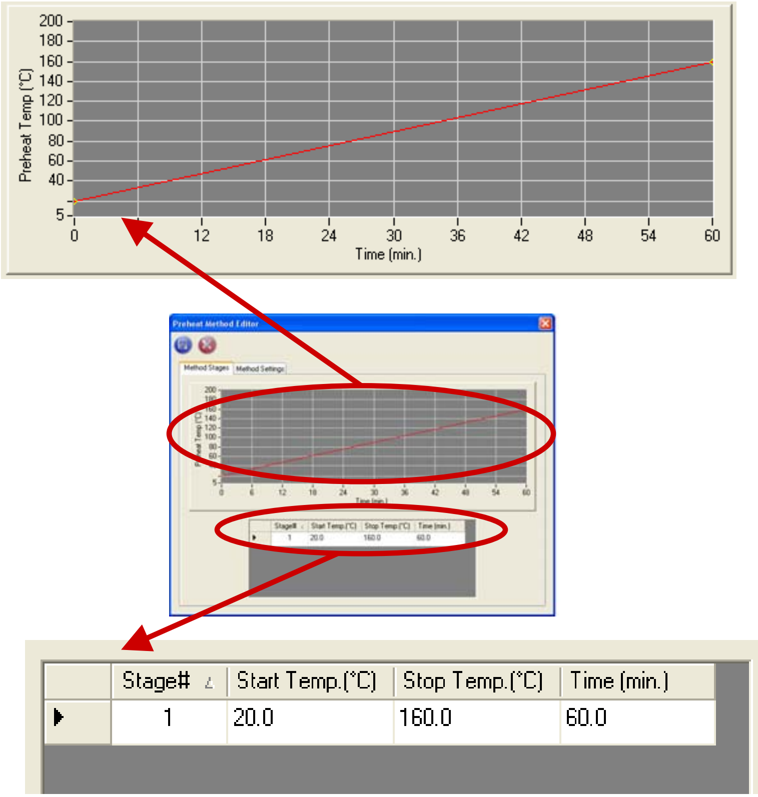




	Stage#	Start Temp.(°C)	Stop Temp.(°C)	Time (min.)
	1	20.0	20.0	60.0
▶	2	60.0	60.0	120.0
	3	120.0	120.0	180.0
	4	200.0	200.0	500.0

Temperature Ramps

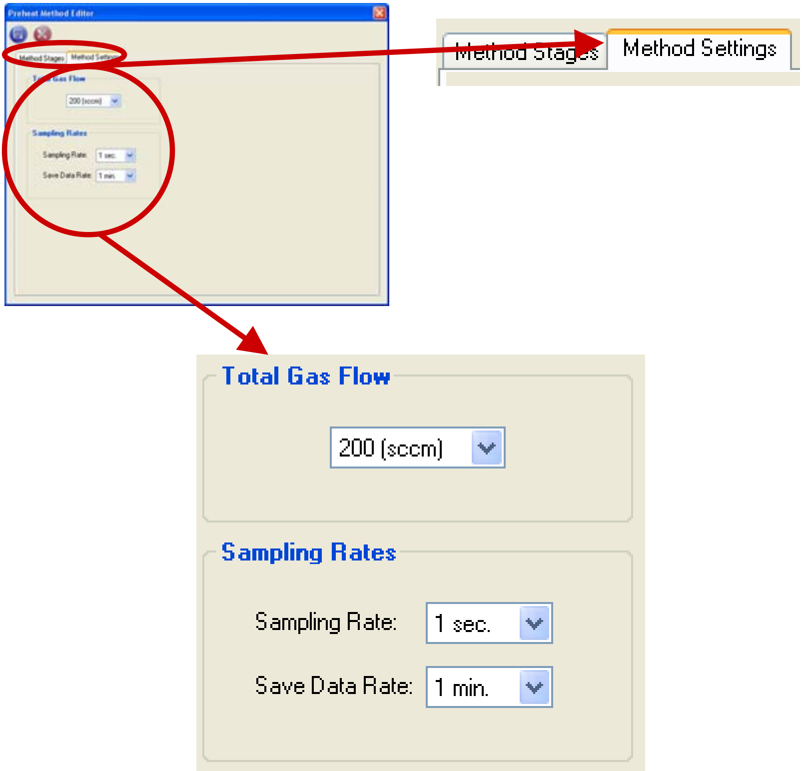
Temperature ramps are created by making the **Start** and **Stop Temp** setpoints different from each other:



	Stage#	Start Temp.(°C)	Stop Temp.(°C)	Time (min.)
▶	1	20.0	160.0	60.0

Note: Temperature steps and ramps can be included in the same Preheat Method by simply editing the Preheat file's Stages accordingly.

Method Settings Tab: Gas Flow and Sampling Rates

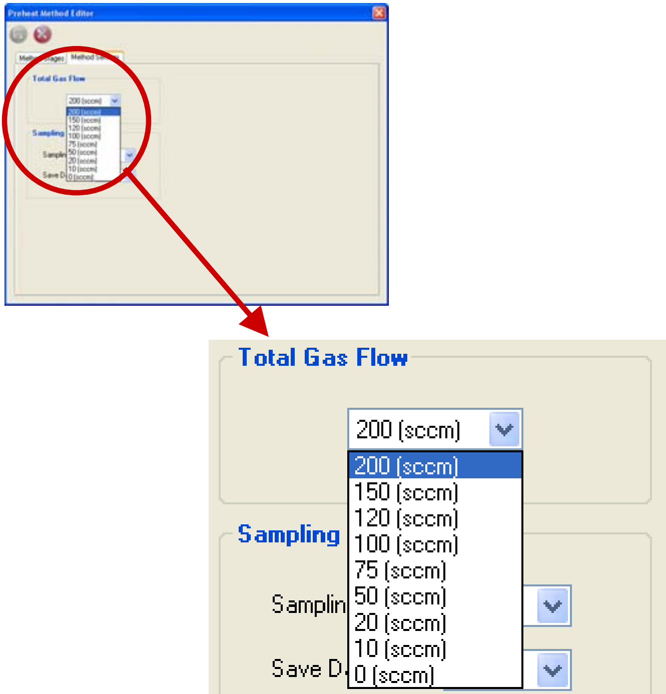


The Method Settings Tab is used to define the Preheat Method's Total Gas Flow and Sampling Rates, which are defined below.

Total Gas Flow

This box allows the user to specify the preheat flowrate from a set of gas flows in standard cubic centimetres per minute (sccm). The default setting for a DVS Advantage is 200 sccm.

Clicking on the edit box or the arrow to its right reveals the list of available flowrates:



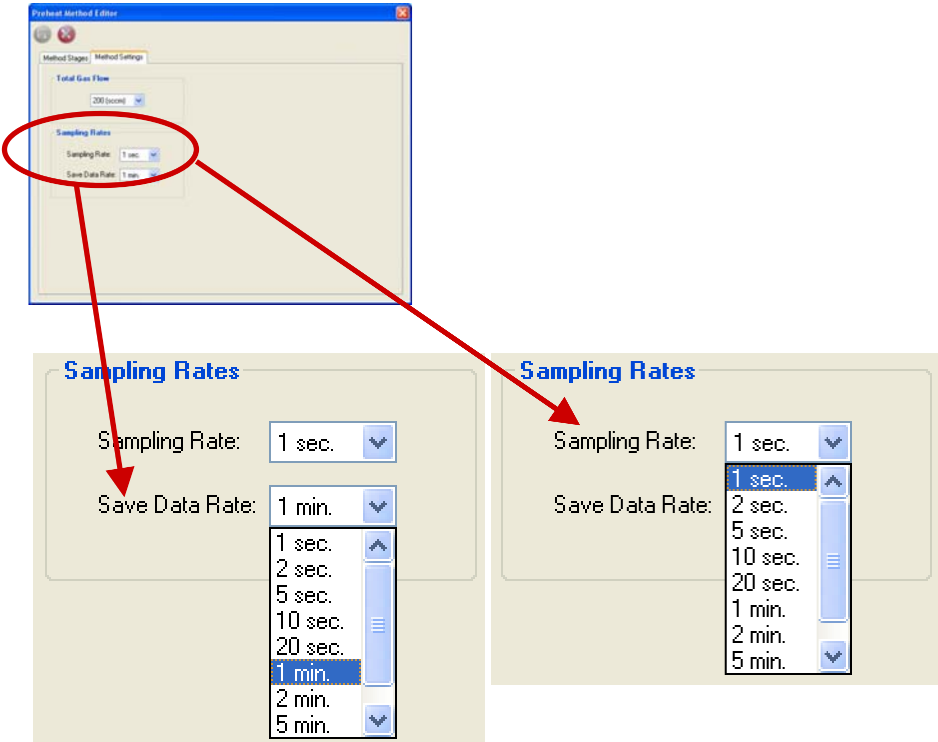
Sampling Rates

These drop-down lists allow the user to define the rate at which data is saved and displayed when running the Method. Both rates are set to the current **General Preference** settings by default (Section 9.2.1.1). Please also refer to Section 9.3.1.

Sampling Rate: The rate at which mass data is updated on the software interface.

	The recommended setting is 20 seconds.
Save Data Rate:	The rate at which data is saved to a DVS raw data file on the hard disk. The recommended setting is 1 minute.

Clicking on the edit boxes or the arrows to their right reveals the list of available flowrates:



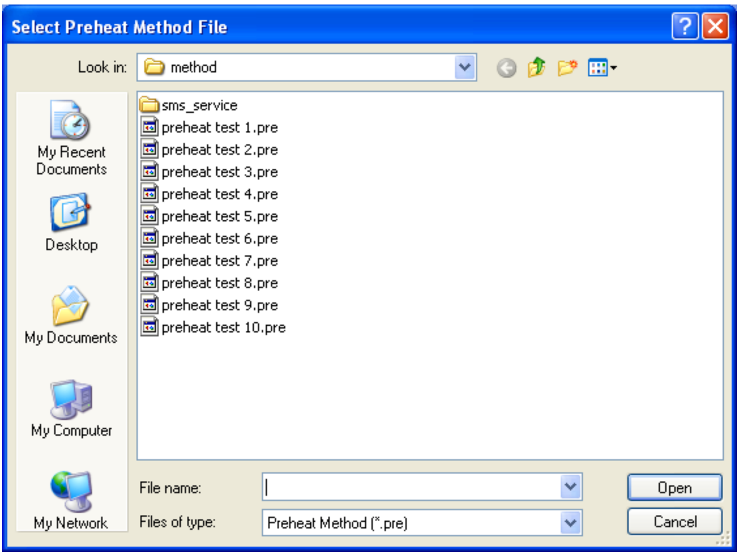
Note: The Sampling and Save Data Rates defined in **File→Preferences** will appear in the **Sampling Rates** drop-down boxes of the Method Settings tab when the Partial Pressure Method Editor is first opened.

These settings can then be altered and saved to the new Method file.

9.5.1.2 Edit Method

This option allows the user to edit previously saved Preheat Method files.

Clicking the **Edit Method** option calls up the **Select Preheat Method File** window which allows the user to select which Preheat Method file to edit:



By default, the software will immediately go to the directory specified in the Preferences option (Section 9.2.1.2) – another directory may be selected in the window above if desired.

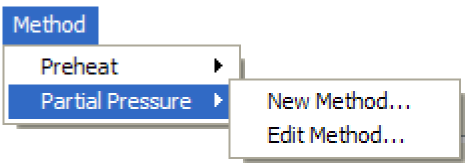
Upon choosing a file, clicking **Open** will open the Preheat Method file for editing.

The file may be edited and saved as described above in the **New Method** Section.

Note: If a Preheat Method is *active* (ie; running) as part of a running Sequence, then it may still be edited. While the Preheat Method is running, the **Method→Preheat→Edit Method** option may be used as described in Section 9.5.1.2 to do this.

The newly edited Method will need to be saved under a new name – but note that it is not automatically loaded into the running Sequence. To do this, the running Sequence will need to be edited to include the new Method as described in Section 9.6.1.3.

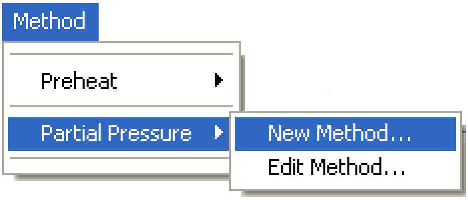
9.5.2 Partial Pressure



Partial Pressure Methods may be used to automatically control the vapour partial pressure that the DVS sample chamber receives over a defined period of time. This is achieved by associating multiple time periods (ie: Method Stages) that have individual vapour partial pressure settings.

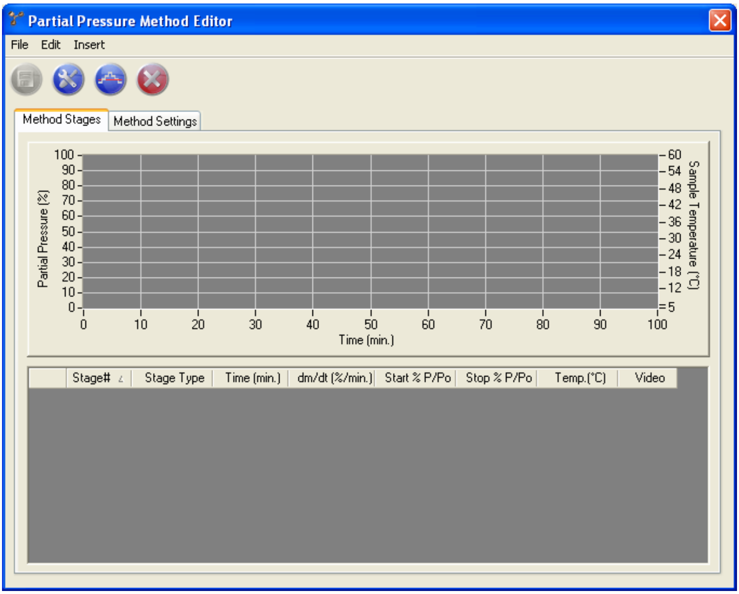
The method stages are run sequentially.

9.5.2.1 New Method



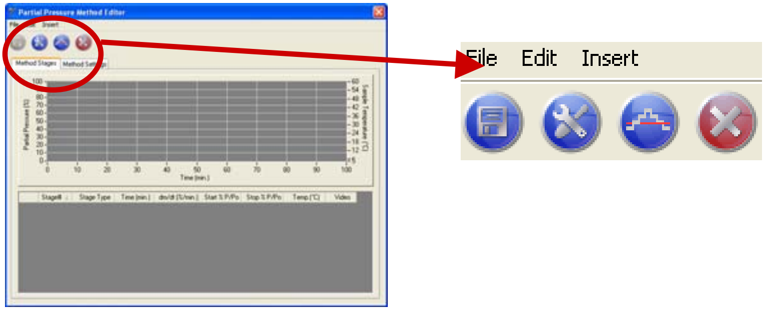
This option allows the user to create new Method files.

Clicking **Method→Partial Pressure→ New Method…** calls up the Partial Pressure **Method Editor** window:

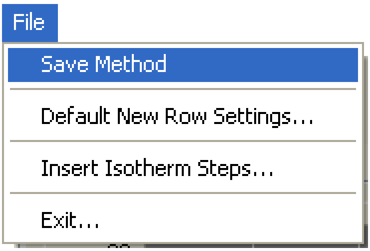


The **Partial Pressure Method Editor** window looks similar to the **Active Method** panel on the main DVS Advantage dialog screen (Section 10.6) – it similarly features a **Method Stages** tab and a **Method Settings** tab:

At the top of the window are the buttons and menu items which allow the user to build a new Partial Pressure Method file. These functions are explained in the sections which follow:

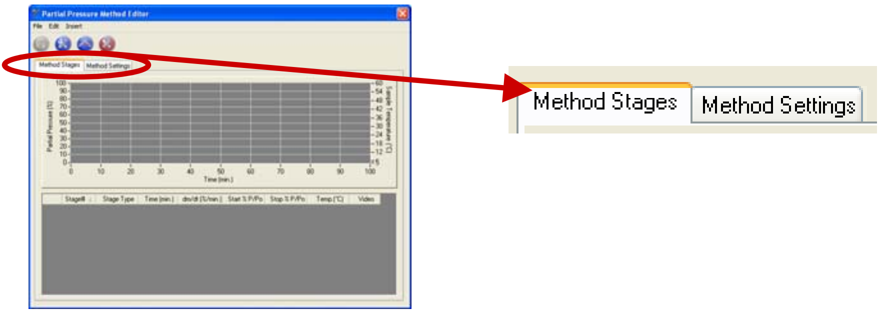


9.5.2.2 Save Method 

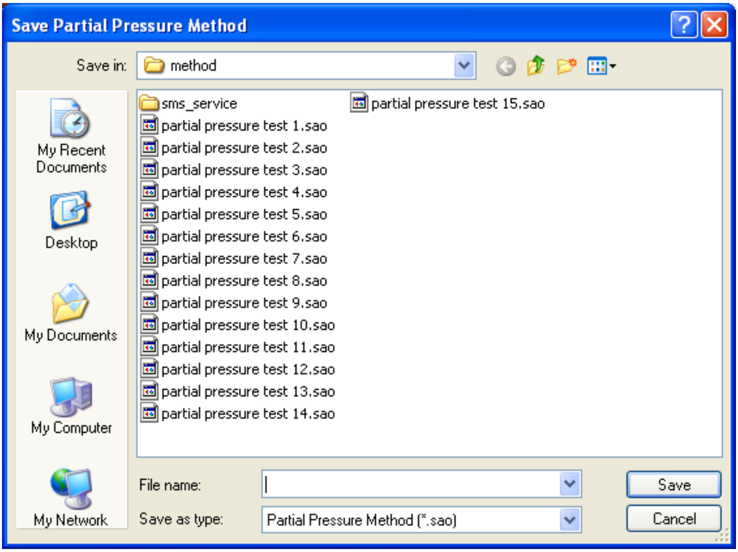


Clicking the **Save Method** button , or going to **File → Save Method** saves a Method to a file after it has been formulated in the **Partial Pressure Method Editor**.

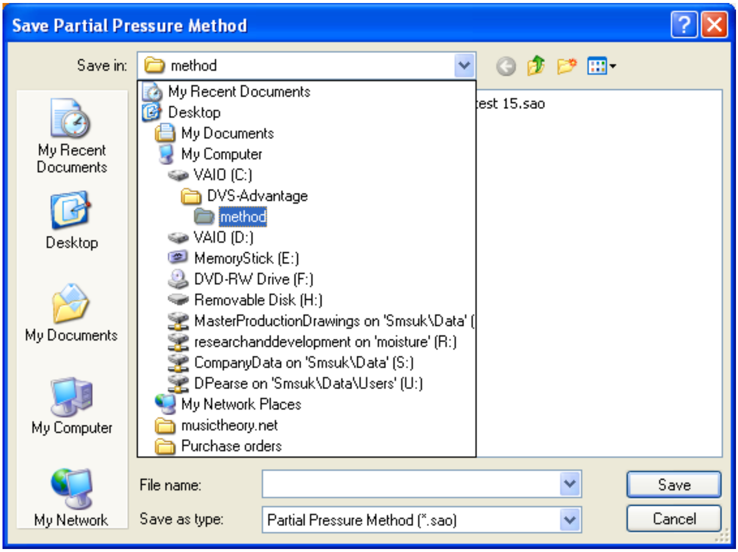
Note: The **Save Method** option only becomes active after a Method has been created (see sections below).



Once the Save Method function has been activated, a **Save Partial Pressure Method** window is opened, requiring the user to name the new Partial Pressure Method file.

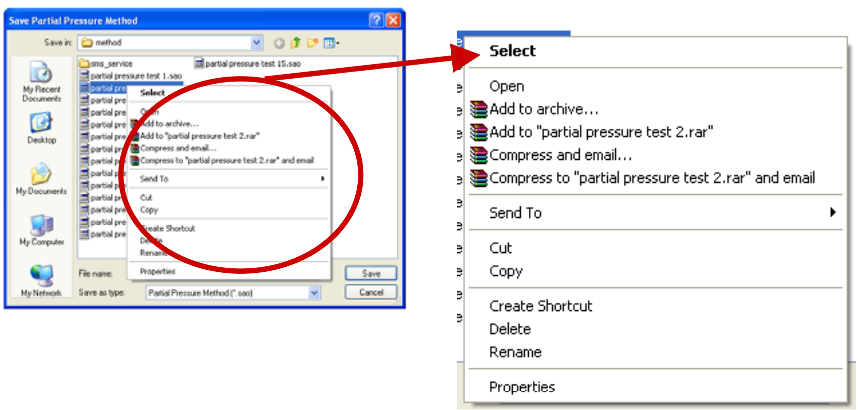


The **Save Partial Pressure Method File** window automatically navigates to the directory specified in **File → Preferences → Default Directories** (Section 9.2.1.2), but a different location may be specified using the **Save in** drop-down menu:



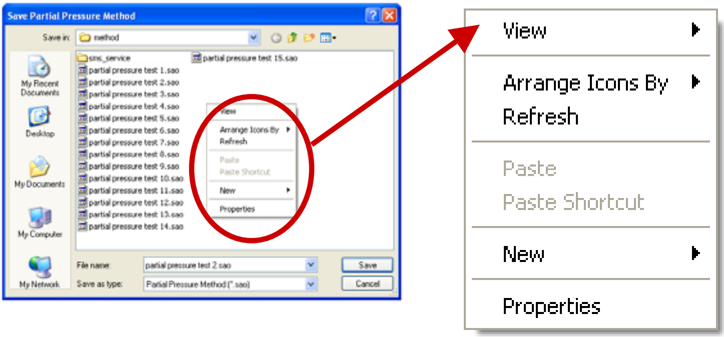
As the pictures above show, **Partial Pressure Method** files are saved with the extension **.sao** (**s**imple **a**utomated **o**peration).

Normal Windows functions are available in the **Save Partial Pressure Method File** window. Right-clicking on a pre-existing file in the window calls up the following menu:



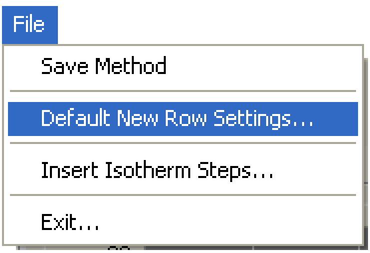
Files can be deleted, renamed, etc, before the new Method file is saved.

Right-clicking in a blank space in the window calls up the following menu:



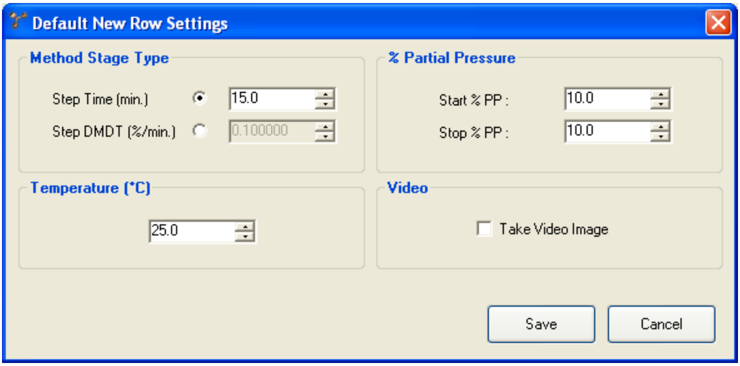
After the new Method file has been named, clicking **Save** then saves the new Method and returns to the main DVS Advantage dialog screen.

9.5.2.3 Default New Row Settings 



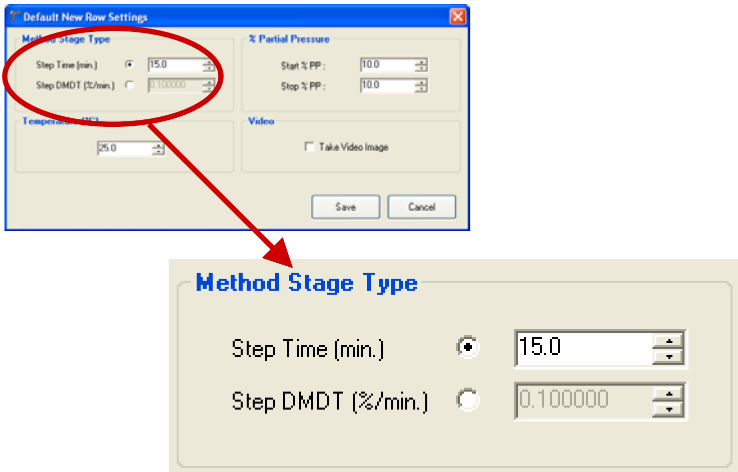
This function specifies default settings for any new Stage (or 'Row') inserted or appended to a Method.

Clicking the **Default New Row Settings** button , or going to **File → Default New Row Settings…** opens the following window:



The four Stage parameters to be set are explained below:

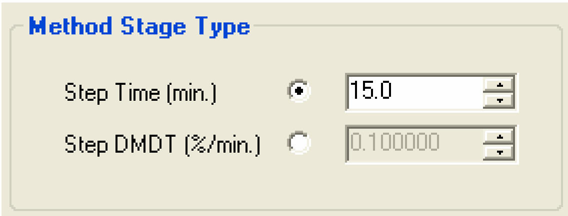
Method Stage Type



The **Method Stage Type** box allows the user to choose the Stage end determinant – **Time** or **dm/dt**:

Time: These Stages are time-based ie; the Stage will run for the specified period of time regardless of the state of the sample. This means that if the sample has not yet reached equilibrium at the end of the allotted Stage time, the instrument will move to the next Stage regardless.

Clicking the **Step Time (min)** option will activate the data entry box as shown below:

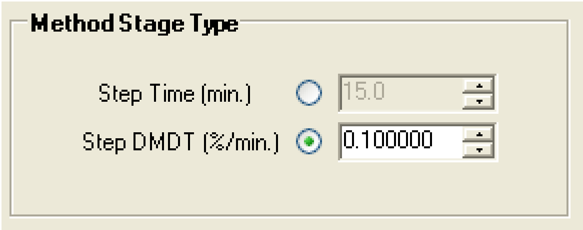


This box specifies the duration of each Stage in the Method in minutes. The figure may be entered directly, or by using the arrows at the side of the box. A maximum Stage time of 9999 minutes is allowable.

dm/dt: DMDT is a mode which specifies a rate of mass change as the criteria for moving on to the next Stage in a Method - rather than a Stage running for a specified period of time.

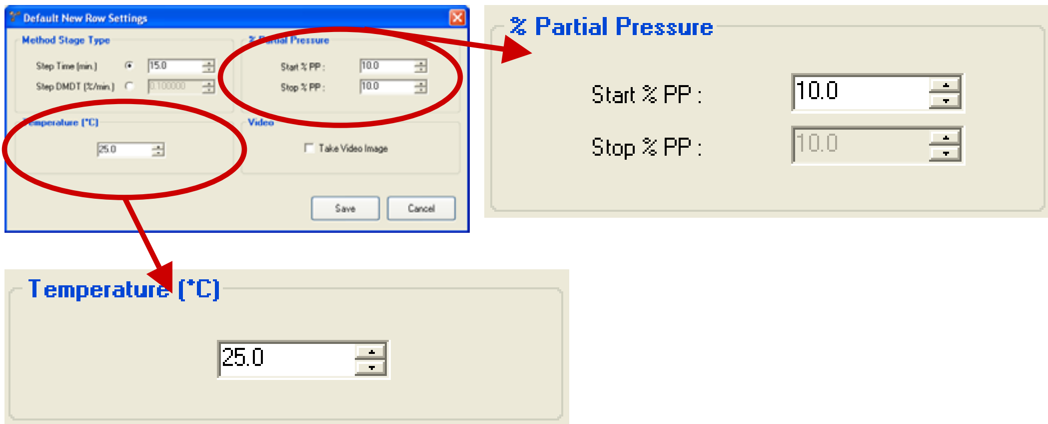
If running a Stage in DMDT mode, the value of dm/dt required to establish equilibrium for the Method Stage must be specified.

Clicking the **Step DMDT (%/min)** option enables the data entry box that specifies the required dm/dt (%/min):



Again, the figure may be entered directly, or by using the arrows at the side of the box. A maximum dm/dt of 9.99 % per minute (and a minimum of -9.99) are allowable. Also refer to earlier section in hardware for recommendations on suitable Step dm/dt values to use.

Temperature (°C) and % Partial Pressure

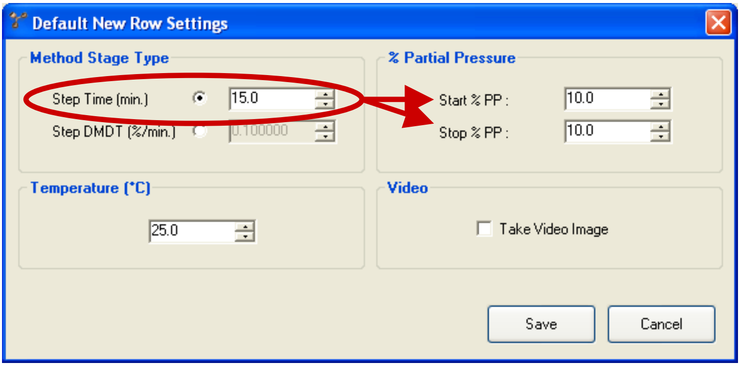


These boxes specify the default Temperatures and Partial Pressures to be used in new Stages.

The maximum allowable Temperature is **60°C** and the minimum is **5°C**. Partial Pressure may be set from 0% to 100% (although a maximum of around 98% p/p^o is actually attainable).

Time-based Method Stage

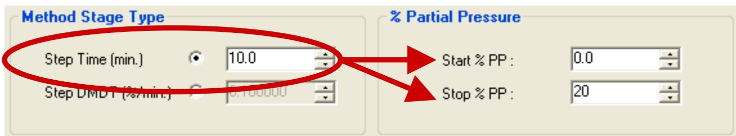
If **Time** is chosen as the Method Stage Type, then initial and final **Partial Pressures** may be specified:



If the user wishes to keep the partial pressure in the default Stage constant, then they can set their **Start** and **Stop** values equal.

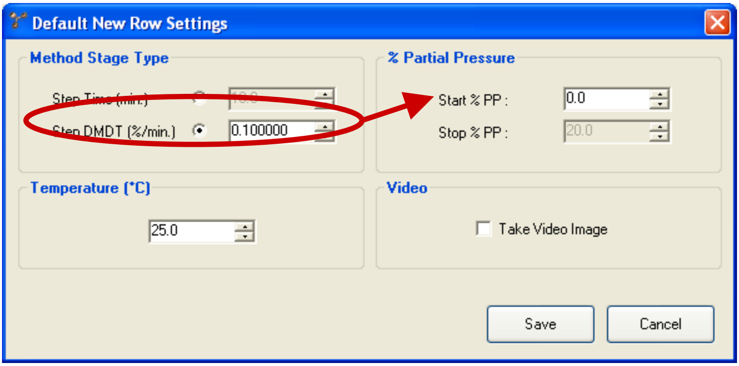
If it is desired to ramp the partial pressure, then the **Stop** value may be set above or below the **Start** value, depending on whether an upward or downward ramp is desired.

For example, the setting shown below would produce a default Stage that would ramp the partial pressure up from 0% to 20% across 10 minutes:

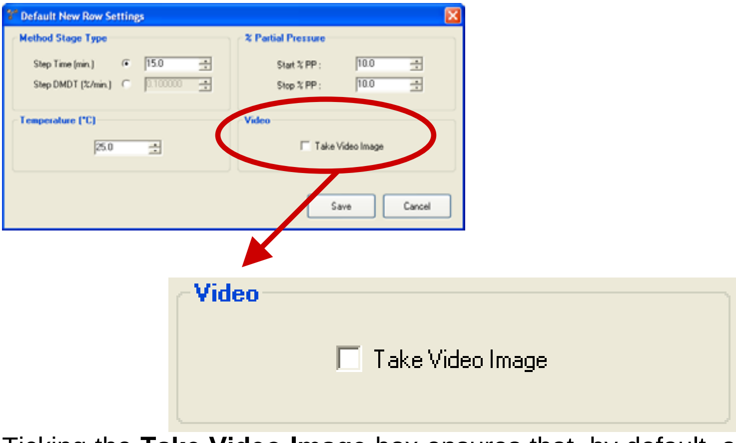


dm/dt-based Method Stage

If the **dm/dt** criterion is chosen, then only the initial partial pressure may be set:



Video



Ticking the **Take Video Image** box ensures that, by default, a sample snapshot will be taken at the end of any Stage which is inserted into the Method.

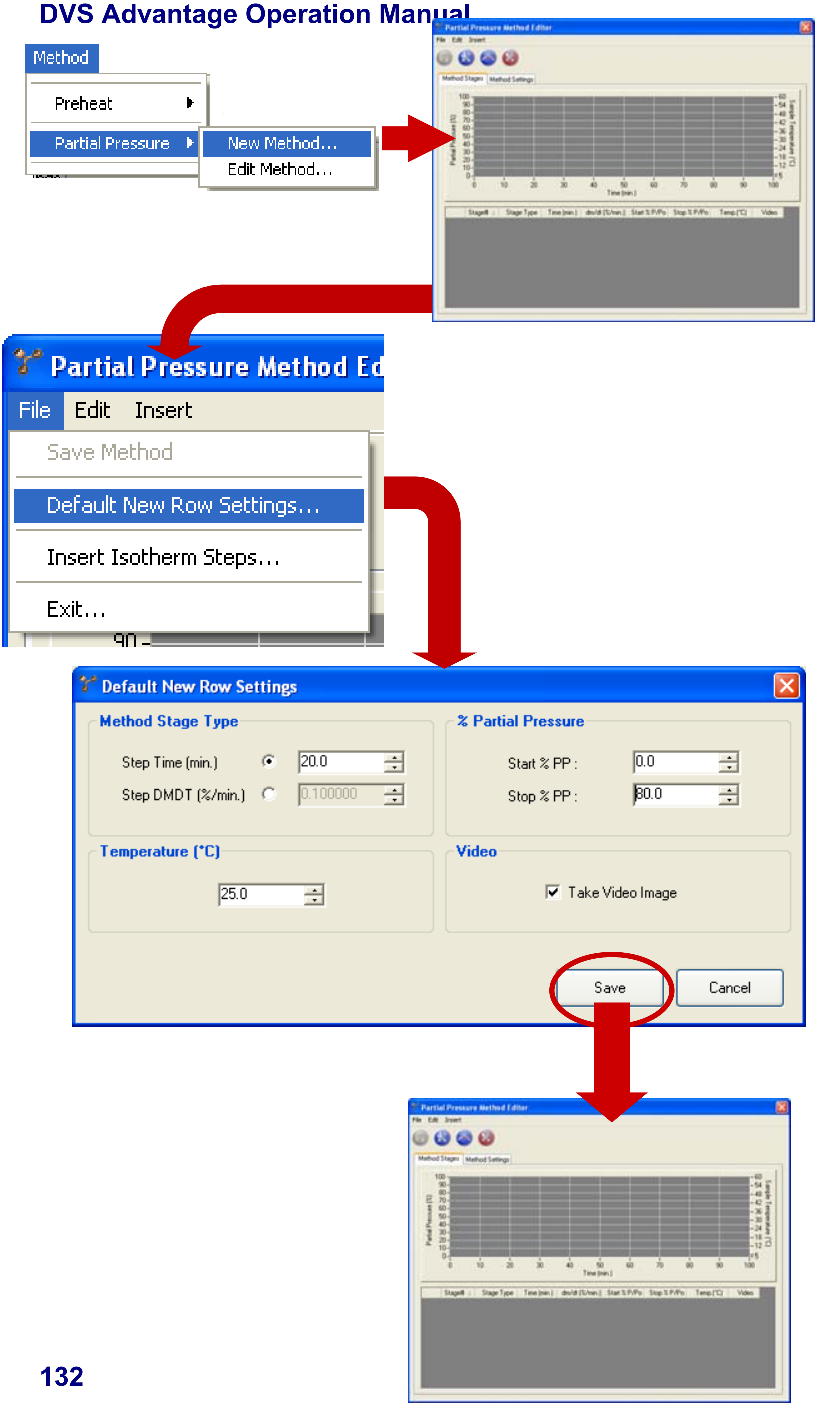
However, it is important to remember that this setting will only take effect if the **Video** preferences are set to follow the settings defined in the Method (refer to Section 9.2.1.3 for more information on setting the Video preferences).

Note: The Video option is not currently available with this version of the DVS Advantage Control software – this option being checked or unchecked therefore has no influence on the operation of the system.

Default New Row Settings flowing through to New Methods

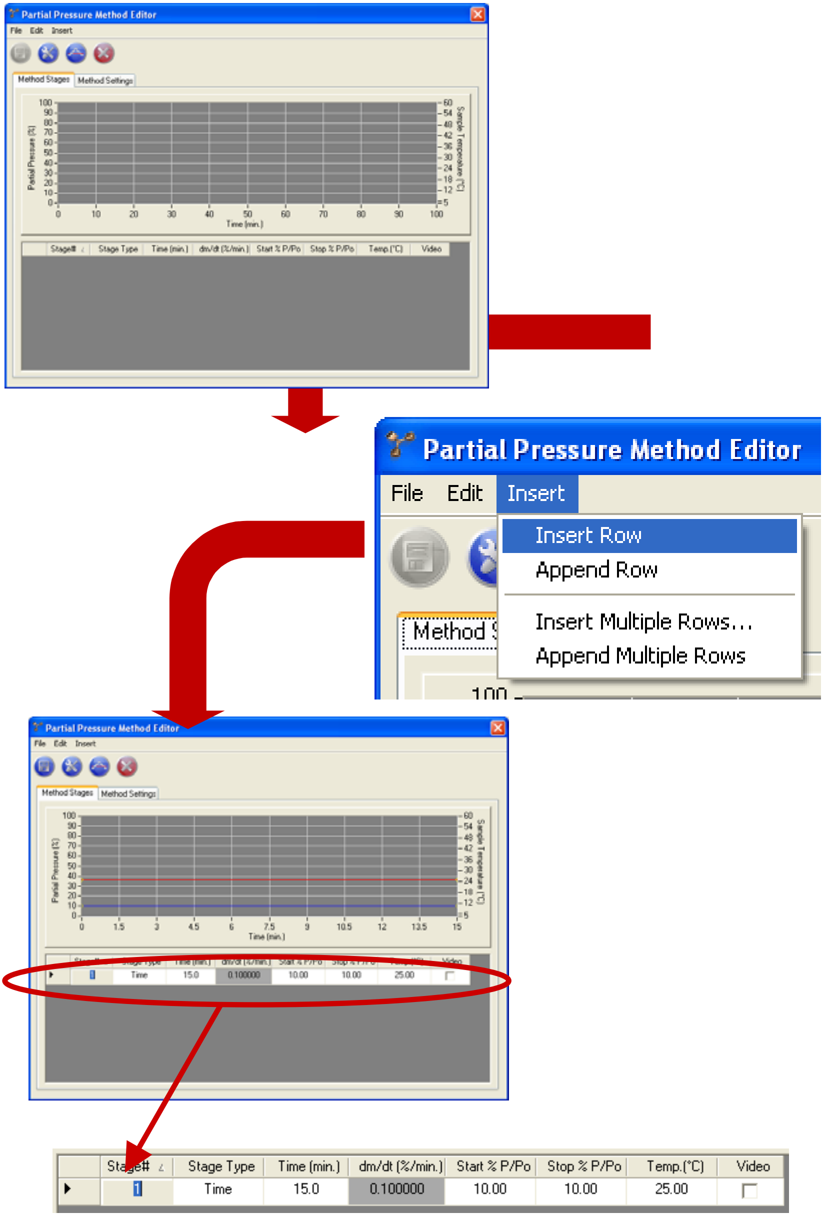
After all options are specified and the **Save** button is clicked, any new Stages inserted into a Method will display the chosen default settings.

For example, entering the **Default New Row Settings** shown below and clicking **Save** returns the Method Editor Window…..


Method
Preheat
Partial Pressure
New Method...
Edit Method...
Partial Pressure Method Ed
File
Edit
Insert
Save Method
Default New Row Settings...
Insert Isotherm Steps...
Exit...
Default New Row Settings
Method Stage Type
Step Time (min.)
20.0
Step DMDT (%/min.)
0.100000
% Partial Pressure
Start % PP :
0.0
Stop % PP :
80.0
Temperature (°C)
25.0
Video
Take Video Image
Save
Cancel

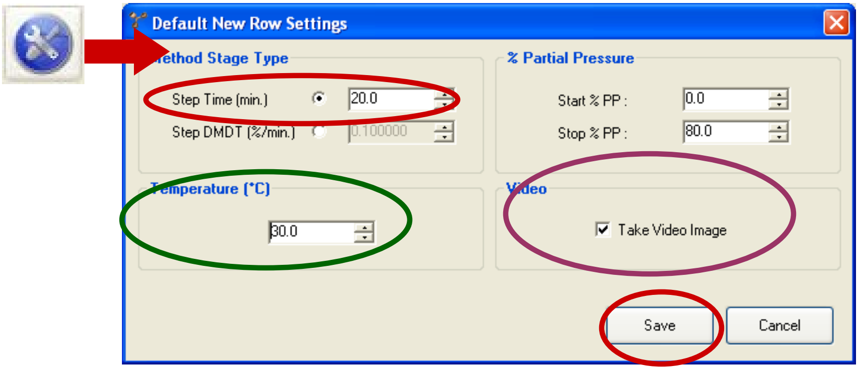
….then any Row(s) inserted into a new Method will display by default all of the parameters set in **Default New Row Settings**.

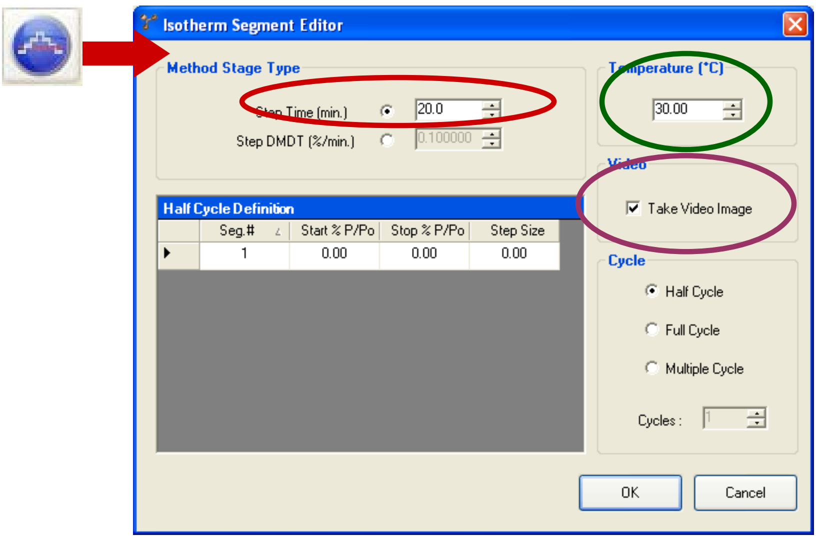
Notice in the example below that as the **Time Stage Type** is chosen, the **dm/dt** box is greyed out:


Partial Pressure Method Editor
File
Edit
Insert
Method Stages
Method Settings
Partial Pressure (%)
Sample Temperature (°C)
Time (min.)
Stage#
Stage Type
Time (min.)
dm/dt (%/min.)
Start % P/Po
Stop % P/Po
Temp.(°C)
Video
Partial Pressure Method Editor
File
Edit
Insert
Insert Row
Append Row
Insert Multiple Rows...
Append Multiple Rows
Method S
100
Partial Pressure Method Editor
File
Edit
Insert
Method Stages
Method Settings
Partial Pressure (%)
Sample Temperature (°C)
Time (min.)
Stage#
Stage Type
Time (min.)
dm/dt (%/min.)
Start % P/Po
Stop % P/Po
Temp.(°C)
Video
1
Time
15.0
0.100000
10.00
10.00
25.00

Default New Row Settings flowing through to Isotherm Segment Editor

When the **Isotherm Segment Editor (****)** is first opened the **Method Stage Type**, **Temperature** and **Video** settings will contain the default values specified in the **Default New Row Settings**:

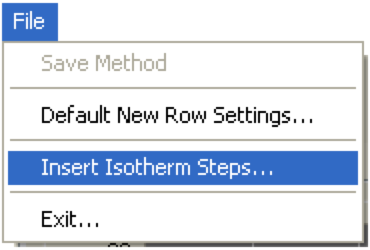




Re-starting

If the DVS Advantage Control software is re-started, the **Default New Row Settings** are preserved.

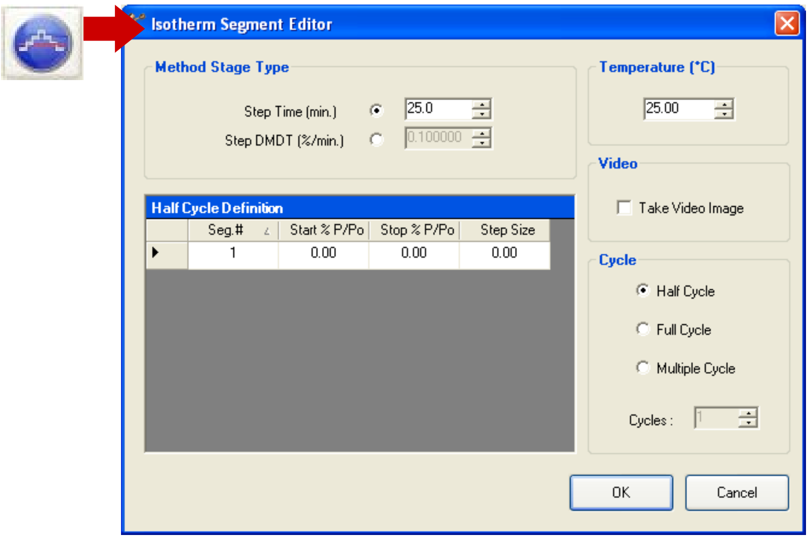
9.5.2.4 Insert Isotherm Steps 



This function allows the quick set-up of an Isotherm Method – an experiment in which <u>the temperature remains constant while the partial pressure is varied.</u>

Please refer to the **Standard Analysis Suite Manual** for more information on the science of isotherms.

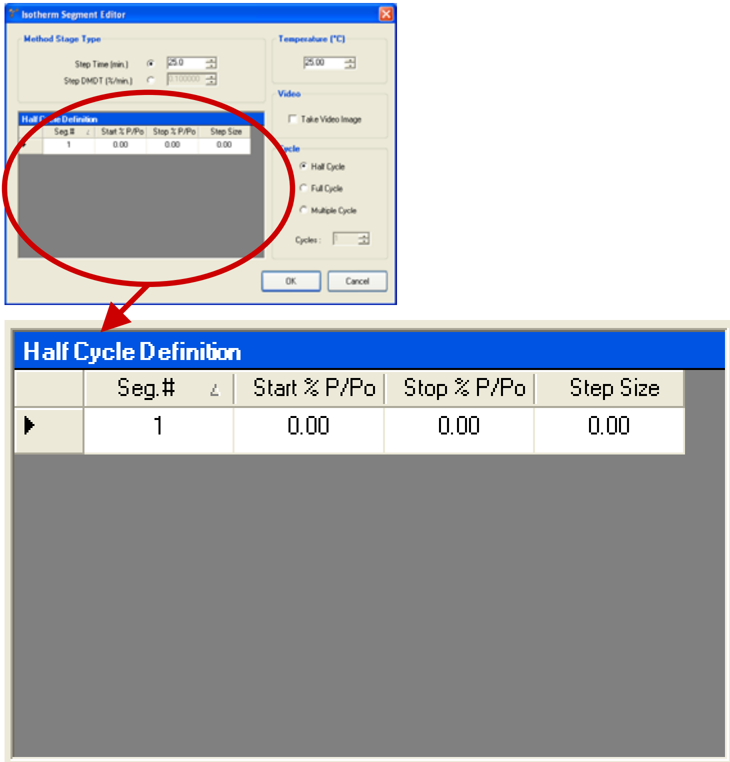
Clicking the **Insert Isotherm Steps** button  or going to **File → Insert Isotherm Steps…** calls up the **Isotherm Segment Editor**:



> **Note:** When the **Isothem Segment Editor** is first opened, the **Method Stage Type**, **Temperature**, and **Video** sections will contain the default settings specified in **Default New Row Settings** (Section 9.5.2.3).

Using the **Isotherm Segment Editor** panel, the following parameters can be specified for the isotherm experiment:

Half Cycle Definition



Methods may be defined in terms of **Half Cycles.** In the case of an Isotherm experiment, a **Half Cycle** is a series of Stages of specified RH all run at one temperature.

A Method may be constituted by one **Half Cycle**, or the Half Cycle's mirror image can be appended to its end to make a **Full Cycle**. Multiples of a Full Cycle may also be conjoined to form a **Multiple Cycle** Method.

These terms are explained further in the **Cycle** options outlined later in this Section.

The **Half Cycle Definition** box specifies the number of Stages - and their Relative Humidities - to be included in the **Isotherm Half Cycle** Method.

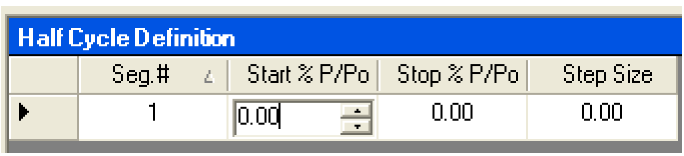
Seg.#

This denotes the number of the Stage and cannot be edited - new Stages are added or appended, the numbers will change automatically.

Start and Stop %P/P$_o$

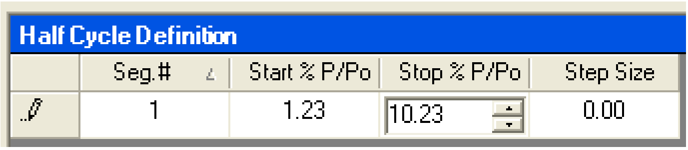
These values define the Relative Humidities at which the **Half Cycle** begins and ends.

Both the Start and Stop %P/P$_o$ values may be edited by clicking on them – this activates a data entry box:



The desired values may be entered either directly or by using the toggle arrows – the arrows change the values by 1.00 %P/P_o on each click.

The values entered do not need to be whole numbers – decimal values are also accepted:

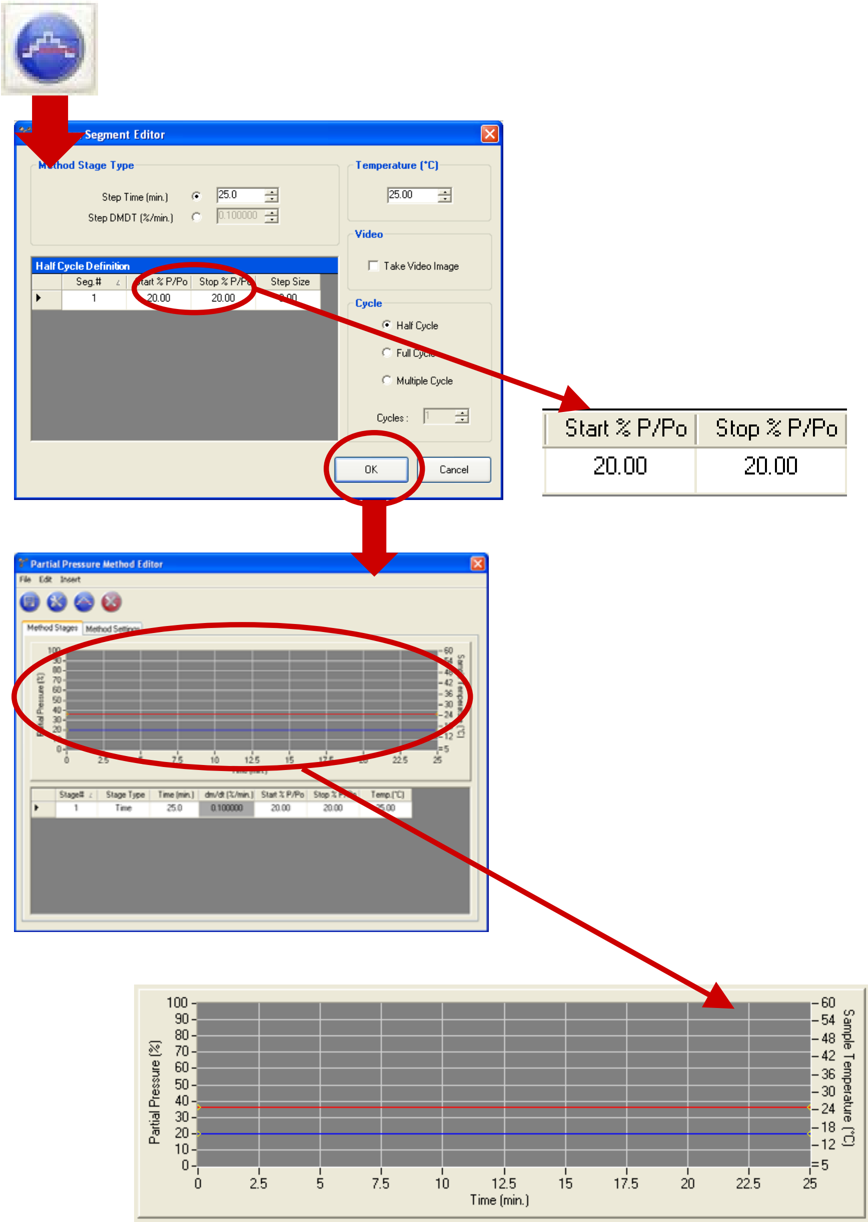


Step Size

The **Step Size** determines the size of the partial pressure steps used to get from the Start %P/P_o value to the Stop %P/P_o value.

When the Start and Stop values are the same, then clearly there is no step required, and so the Step Size can remain at the default value of zero.

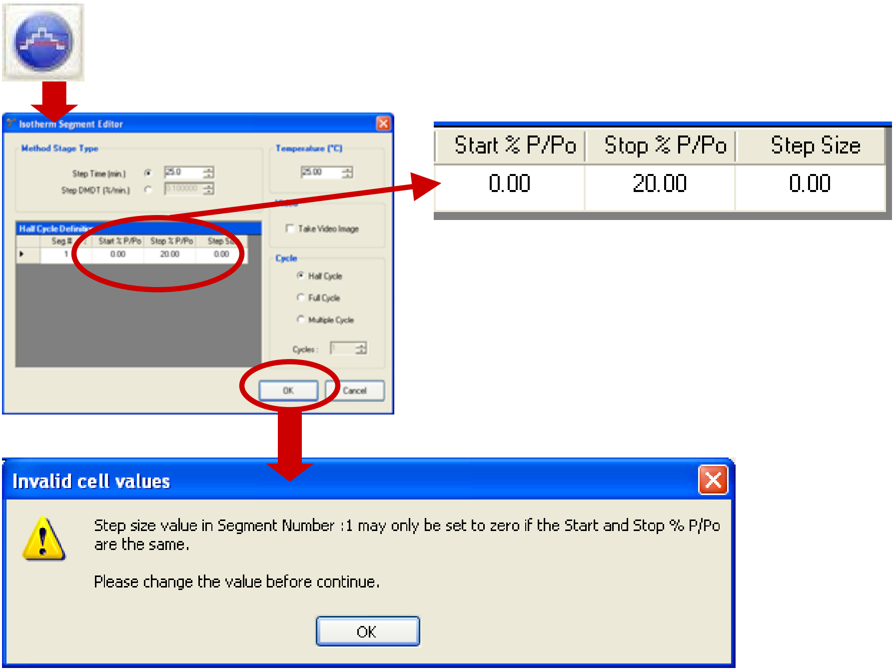
Having the Start and Stop %P/P_o values the same will produce a Half Cycle Method made up of one Stage at a constant RH, as shown below:


Segment Editor
Method Stage Type
Step Time (min.)
25.0
Step DMDT (%/min.)
0.100000
Temperature (°C)
25.00
Video
Take Video Image
Half Cycle Definition
Seg.#
Start % P/Po
Stop % P/Po
Step Size
1
20.00
20.00
Cycle
Half Cycle
Full Cycle
Multiple Cycle
Cycles :
OK
Cancel
Start % P/Po
Stop % P/Po
20.00
20.00
Partial Pressure Method Editor
File
Edit
Insert
Method Stages
Method Settings
Stage#
Stage Type
Time (min.)
dm/dt (%/min.)
Start % P/Po
Stop % P/Po
Temp.(°C)
1
Time
25.0
0.100000
20.00
20.00
25.00
Partial Pressure (%)
Sample Temperature (°C)
Time (min.)
100
90
80
70
60
50
40
30
20
10
0
60
54
48
42
36
30
24
18
12
5
0
2.5
5
7.5
10
12.5
15
17.5
20
22.5
25

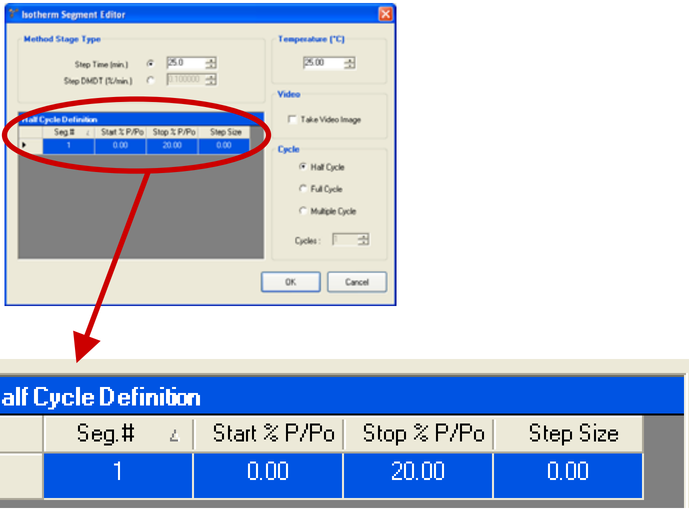
The blue line on the Method Stages graph shows the constant Relative Humidity level at 20% P/P_o, the red line shows the Method temperature.

When the Start and Stop values are different, a **Step Size** greater than zero must be specified.

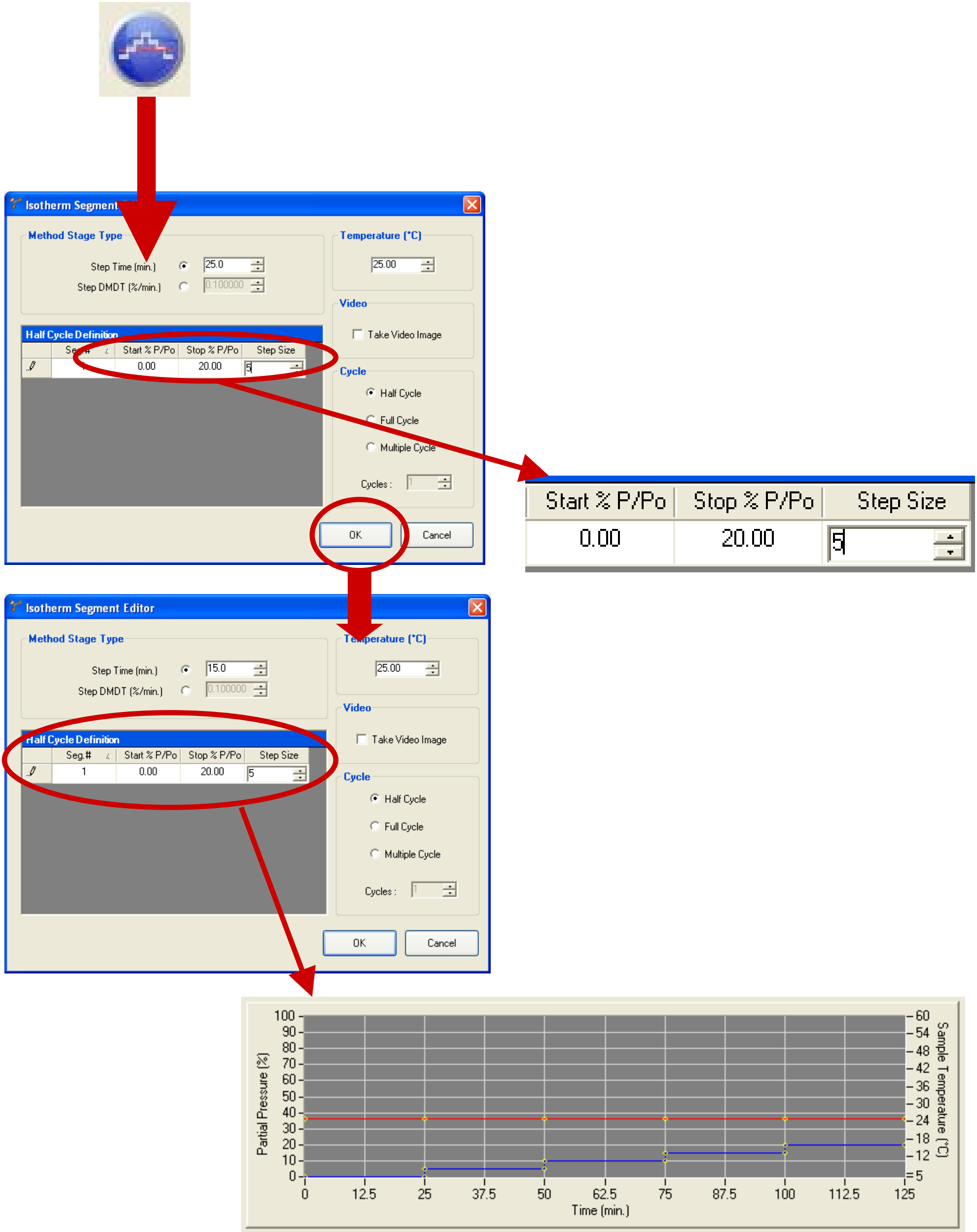
If this is not done, then upon clicking **OK** on the **Isotherm Segment Editor**, an error message appears as shown below:



Clicking **OK** will then return the **Isotherm Segment Editor** screen, with the Method Segment in question highlighted:



In the above example, if a Step Size of 5 were entered, then the partial pressure would rise from 0% %P/P$_o$ to 20% %P/P$_o$ in 5% %P/P$_o$ steps as shown below:

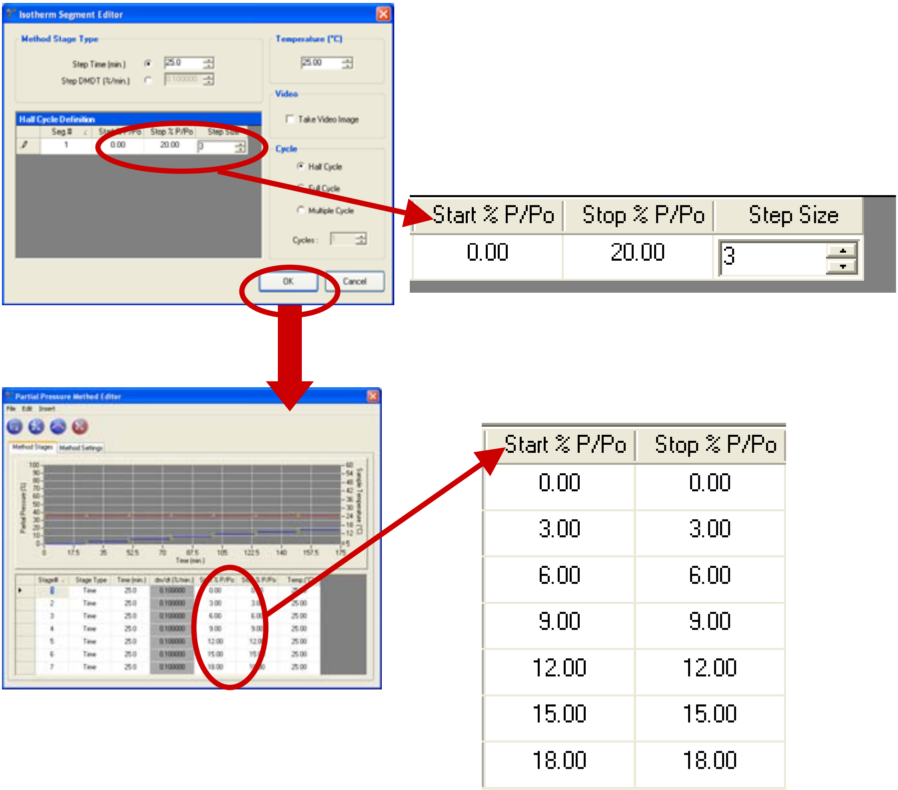

Isotherm Segment Editor
Method Stage Type
Step Time (min.)
25.0
Step DMDT (%/min.)
0.100000
Temperature (°C)
25.00
Video
Take Video Image
Half Cycle Definition
Seg.#
Start % P/Po
Stop % P/Po
Step Size
1
0.00
20.00
5
Cycle
Half Cycle
Full Cycle
Multiple Cycle
Cycles :
1
OK
Cancel
Start % P/Po
Stop % P/Po
Step Size
0.00
20.00
5
Isotherm Segment Editor
Method Stage Type
Step Time (min.)
15.0
Step DMDT (%/min.)
0.100000
Temperature (°C)
25.00
Video
Take Video Image
Half Cycle Definition
Seg.#
Start % P/Po
Stop % P/Po
Step Size
1
0.00
20.00
5
Cycle
Half Cycle
Full Cycle
Multiple Cycle
Cycles :
1
OK
Cancel
Partial Pressure (%)
100
90
80
70
60
50
40
30
20
10
0
0
12.5
25
37.5
50
62.5
75
87.5
100
112.5
125
Time (min.)
Sample Temperature (°C)
60
54
48
42
36
30
24
18
12
5

The Partial Pressure profile above shows $\%P/P_o$ steps of 5% from 0% to 20% P/P_o (blue line). The red line represents the experimental temperature.

Note: A 'ramp' (ie; a gradual change in $\%P/P_o$) may be created by using a step size of $1\%P/P_o$.

Step sizes may be entered which do not 'fit' into the $\%P/P_o$ range defined by the **Start** and **Stop RH** values. But in this case the **Stop $\%P/P_o$** value will be rounded down to the nearest Step value.

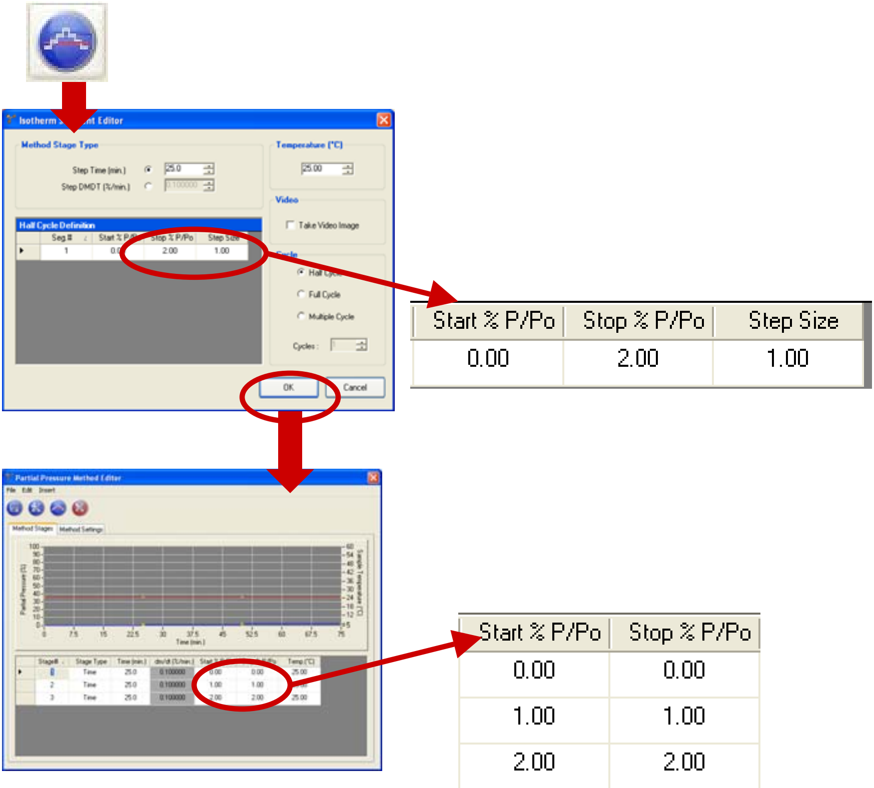
For instance, a step size of 3, with Start $\%P/P_o$ of zero and stop $\%P/P_o$ of 20 will produce a Method whose last stage is at $18\%P/P_o$ (ie; the last value below 20 which is divisible by 3):


Isotherm Segment Editor
Method Stage Type
Step Time (min.)
Step DMDT (%/min.)
Temperature (°C)
Video
Take Video Image
Half Cycle Definition
Cycle
Half Cycle
Full Cycle
Multiple Cycle
Cycles :
OK
Cancel
Start % P/Po
Stop % P/Po
Step Size
0.00
20.00
3
Partial Pressure Method Editor
Method Stages
Method Settings
Start % P/Po
Stop % P/Po
0.00
0.00
3.00
3.00
6.00
6.00
9.00
9.00
12.00
12.00
15.00
15.00
18.00
18.00

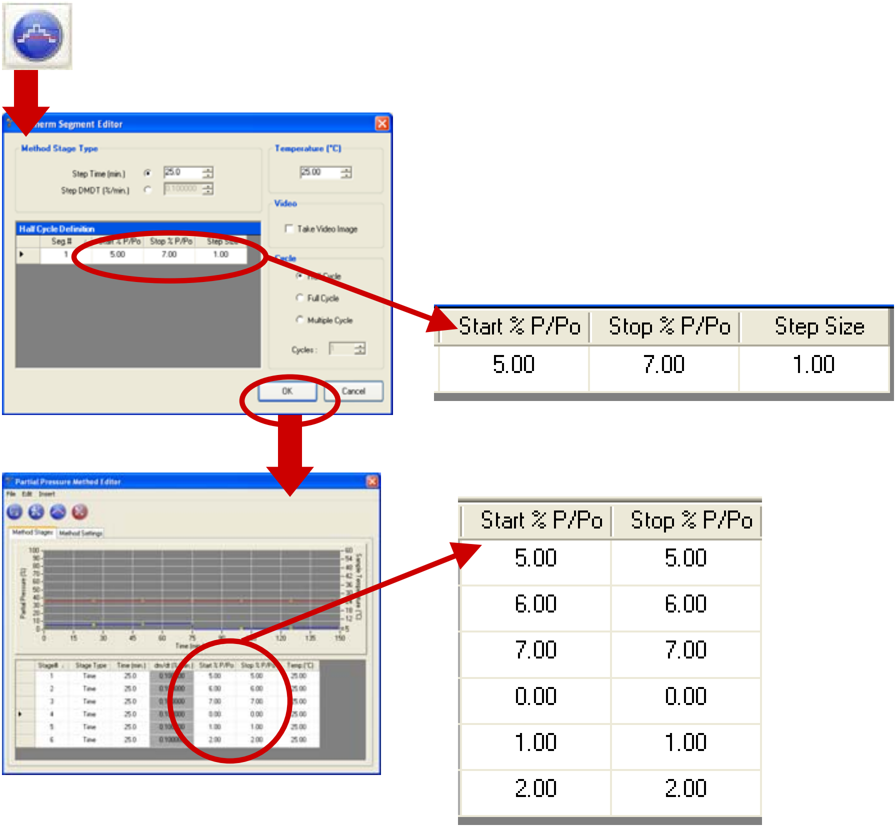
Inserting

If **Insert Isotherm Steps** has already been used to insert a Half Cycle into the **Method Editor**, returning to **Insert Isotherm Steps** and inserting another Half Cycle will not overwrite the original one – the new Half Cycle method stages will be inserted at the current row location of the Method Editor, as shown below:

Initially, a Half Cycle is added to a blank Method Editor screen:



Another Half Cycle is then added, at the current row location, and as the current row is the first row, all the existing rows are moved down:



Performing operations upon Rows

Current Rows

If any part of a segment is clicked (eg; to change a value), then it becomes the active segment. This is indicated by a small arrow that appears in the panel on the segment's left:

Half Cycle Definition

Seg.#	Start % P/Po	Stop % P/Po	Step Size
1	0.00	3.00	1.00
2	5.00	10.00	1.00
3	12.00	20.00	2.00

Highlighting Rows

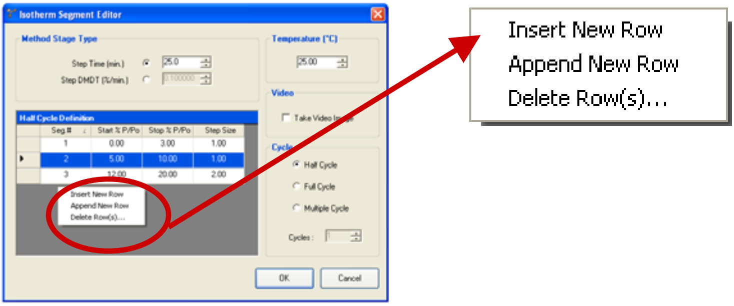
Clicking on the panel to the left of a Segment will highlight all of its contents:

Half Cycle Definition

Seg.#	Start % P/Po	Stop % P/Po	Step Size
1	0.00	3.00	1.00
2	5.00	10.00	1.00
3	12.00	20.00	2.00

Inserting, Appending, and Deleting Rows

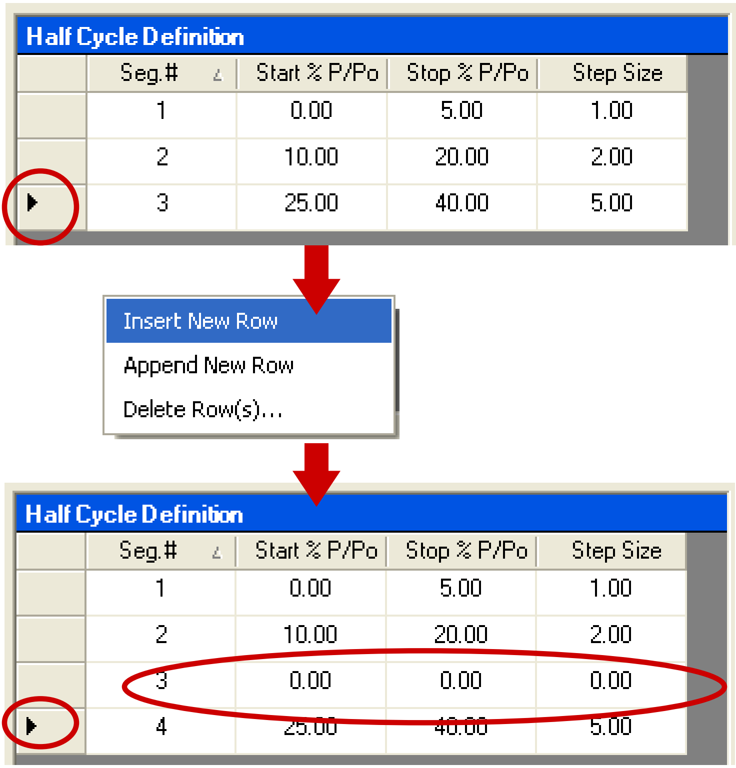
Right-clicking anywhere in the **Half Cycle Definition** panel activates the following options:



Insert New Row

This option inserts a row in the **Half Cycle Definition** panel with default values (zero for the **Start** and **Stop RH** values, and the **Step Size**).

The new Row is inserted above the selected Row (which is indicated with an arrow):



<u>Append New Row</u>
This function adds a new Row to the end of the current list, regardless of which Row is currently selected.

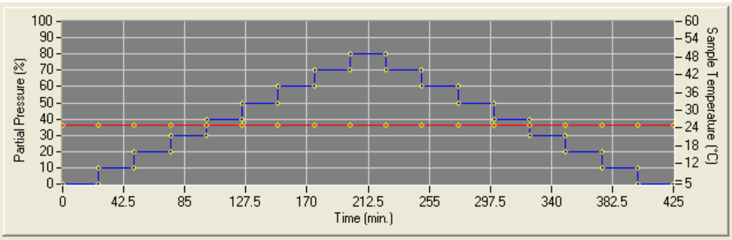
In this way, for instance, extra Partial Pressure steps may be appended to an already-created isotherm.

For example, an isotherm can be quickly constructed as shown below:

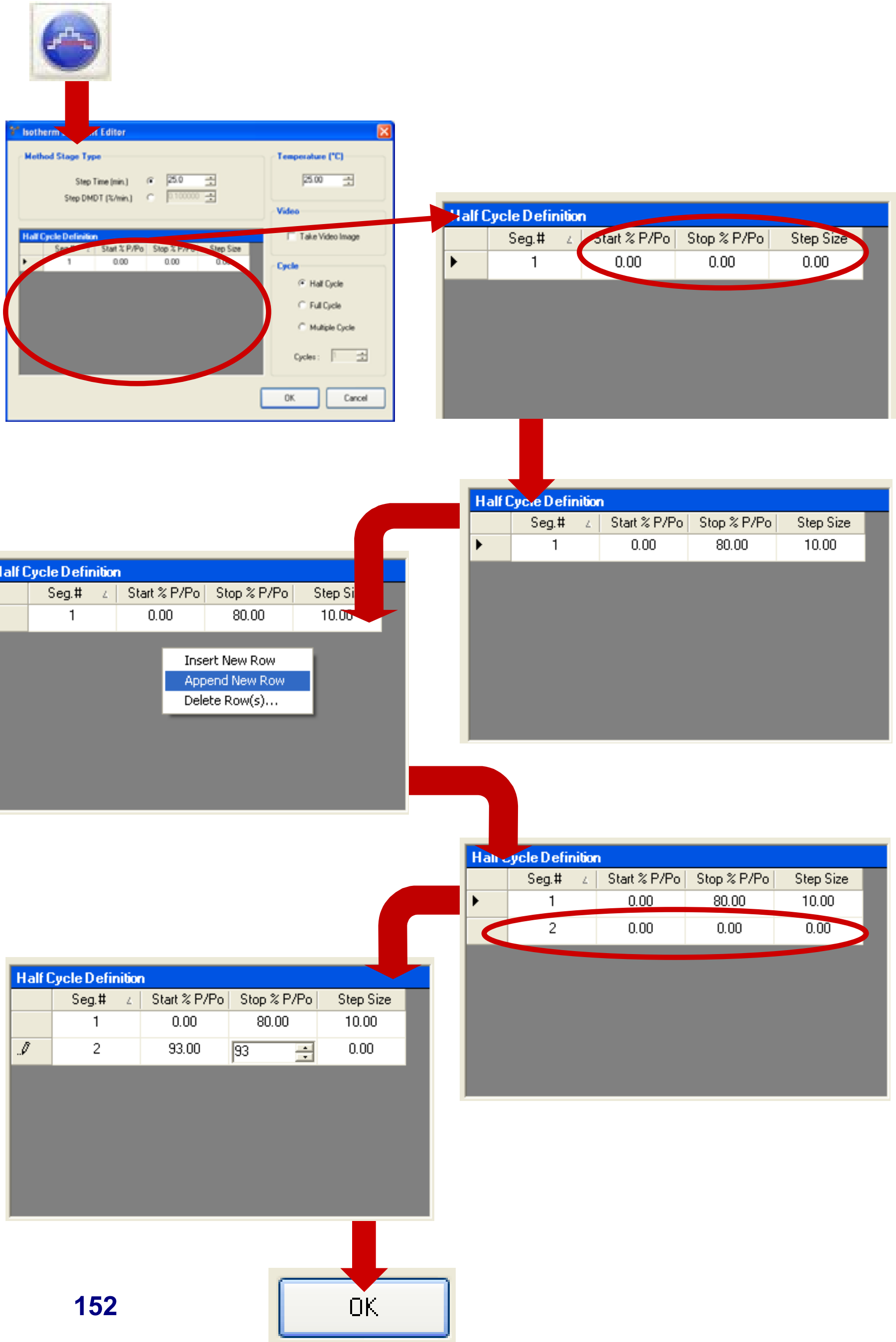
Half Cycle Definition

	Seg.#	Start % P/Po	Stop % P/Po	Step Size
▶	1	0.00	80.00	10.00

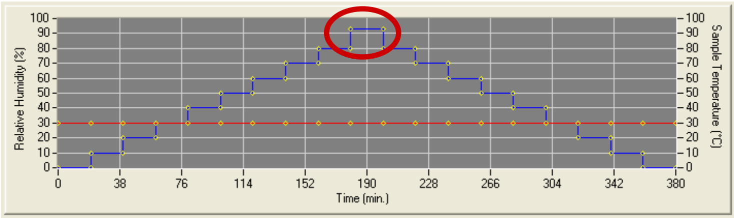
..which would produce the following Partial Pressure profile in the Method Editor window (when the Full Cycle option is used – see later in this Section):



If, however, an extra segment is required which does not fit into the 10% P/P_o steps used in the isotherm set-up above, the **Append Rows** function can be used:

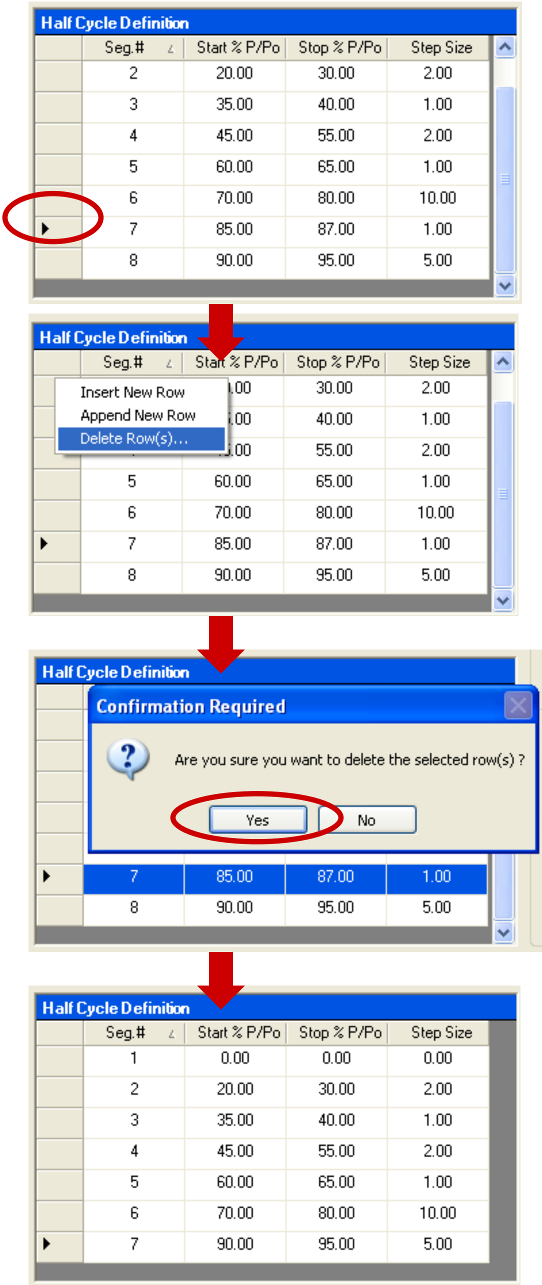

Isotherm Editor
Method Stage Type
Step Time (min.)
Step DMDT (%/min.)
Temperature (°C)
Video
Take Video Image
Cycle
Half Cycle
Full Cycle
Multiple Cycle
Cycles :
OK
Cancel
Half Cycle Definition
Seg.#
Start % P/Po
Stop % P/Po
Step Size
1
0.00
0.00
0.00
80.00
10.00
Insert New Row
Append New Row
Delete Row(s)...
2
93.00
93
OK

Clicking **OK** returns the **Method Editor** window, which displays the isotherm that includes the extra Stage required at 93% P/P_o (circled below):



Delete Row(s)…

This function will delete the Row which is selected with the arrow , or multiple Rows if they are highlighted. Deleting a Row selected with the arrow  takes place as follows:


Half Cycle Definition
Seg.# | Start % P/Po | Stop % P/Po | Step Size
2 | 20.00 | 30.00 | 2.00
3 | 35.00 | 40.00 | 1.00
4 | 45.00 | 55.00 | 2.00
5 | 60.00 | 65.00 | 1.00
6 | 70.00 | 80.00 | 10.00
7 | 85.00 | 87.00 | 1.00
8 | 90.00 | 95.00 | 5.00
Half Cycle Definition
Insert New Row
Append New Row
Delete Row(s)...
Half Cycle Definition
Confirmation Required
Are you sure you want to delete the selected row(s) ?
Yes
No
Half Cycle Definition
Seg.# | Start % P/Po | Stop % P/Po | Step Size
1 | 0.00 | 0.00 | 0.00
2 | 20.00 | 30.00 | 2.00
3 | 35.00 | 40.00 | 1.00
4 | 45.00 | 55.00 | 2.00
5 | 60.00 | 65.00 | 1.00
6 | 70.00 | 80.00 | 10.00
7 | 90.00 | 95.00 | 5.00

Multiple rows may be highlighted one at a time by holding down the **Ctrl** key while clicking the panels to their left, as shown below:

Half Cycle Definition

	Seg.#	Start % P/Po	Stop % P/Po	Step Size
	1	0.00	0.00	0.00
	2	0.00	0.00	0.00
	3	0.00	0.00	0.00
▶	4	0.00	0.00	0.00

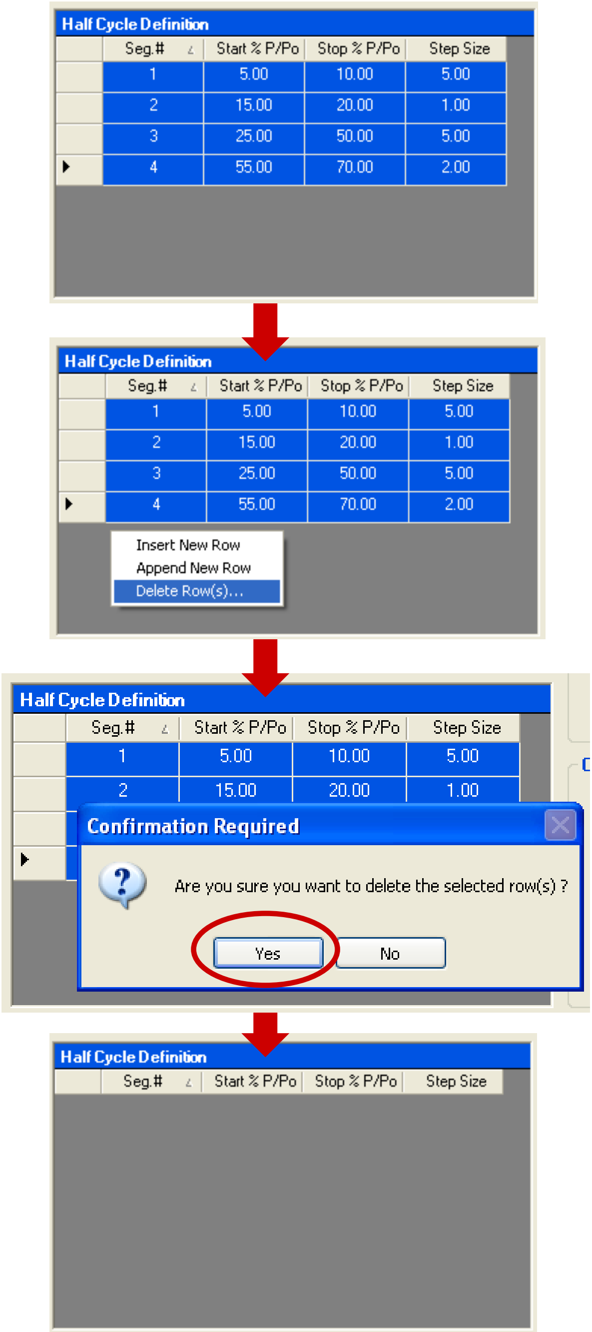
Alternatively, a group of rows may be selected at once by holding down the **Shift** key while clicking the first and last row in the group – all rows in between them will also be selected:

Half Cycle Definition

	Seg.#	Start % P/Po	Stop % P/Po	Step Size
	1	5.00	10.00	5.00
	2	15.00	20.00	1.00
	3	25.00	50.00	5.00
▶	4	55.00	70.00	2.00

Finally, a group of Rows can also be selected by highlighting one Row and dragging the cursor up or down along the Rows to be selected.

Once the multiple Rows have been selected the deletion process may take place as follows:


Half Cycle Definition
Seg.#
Start % P/Po
Stop % P/Po
Step Size
1 5.00 10.00 5.00
2 15.00 20.00 1.00
3 25.00 50.00 5.00
4 55.00 70.00 2.00
Half Cycle Definition
Seg.#
Start % P/Po
Stop % P/Po
Step Size
1 5.00 10.00 5.00
2 15.00 20.00 1.00
3 25.00 50.00 5.00
4 55.00 70.00 2.00
Insert New Row
Append New Row
Delete Row(s)...
Half Cycle Definition
Seg.#
Start % P/Po
Stop % P/Po
Step Size
1 5.00 10.00 5.00
2 15.00 20.00 1.00
Confirmation Required
Are you sure you want to delete the selected row(s) ?
Yes
No
Half Cycle Definition
Seg.#
Start % P/Po
Stop % P/Po
Step Size

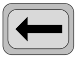
Other Operations

It is possible to move from one cell to another in the **Half Cycle Definition** panel by using the arrow keys on the control PC's keyboard (as happens in Microsoft Excel®).

Each cell is activated for editing when highlighted using the arrow key:

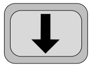
Half Cycle Definition

	Seg.#	Start % P/Po	Stop % P/Po	Step Size
	1	10.00	20.00	1.00
▶	2	30.00	40.00	1.00
	3	50.00	60.00	1.00



Half Cycle Definition

	Seg.#	Start % P/Po	Stop % P/Po	Step Size
	1	10.00	20.00	1.00
▶	2	30.00	40.00	1.00
	3	50.00	60.00	1.00



Half Cycle Definition

	Seg.#	Start % P/Po	Stop % P/Po	Step Size
	1	10.00	20.00	1.00
	2	30.00	40.00	1.00
▶	3	50.00	60.00	1.00

Other Errors

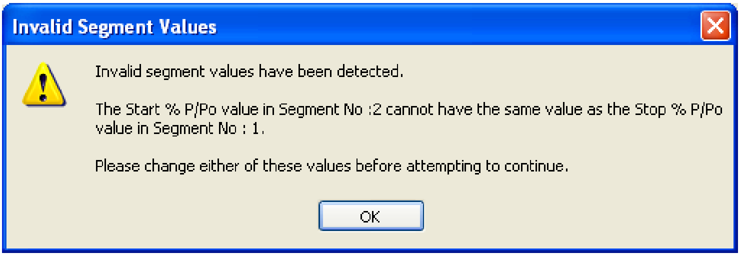
When a Half Cycle Definition contains multiple Rows, the Stop $\%P/P_o$ of a Row cannot be the same as the Start $\%P/P_o$ of the Row that immediately follows it.

For example, if two Rows were constructed as shown below:

Half Cycle Definition

	Seg.#	Start % P/Po	Stop % P/Po	Step Size
	1	0.00	10.00	1.00
▶	2	10.00	20.00	2.00

Clicking OK on the **Isotherm Segment Editor** window will call up the following error message:

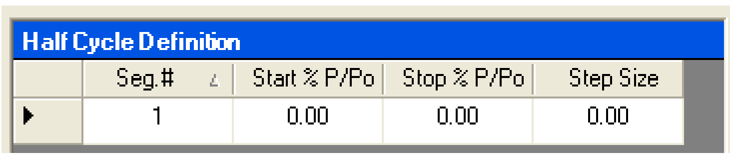


Clicking **OK** returns the **Isotherm Segment Editor** so that the Rows may be edited accordingly.

Closing the Isotherm Segment Editor

If data is entered in the **Half Cycle Definition** panel, and then the **Isotherm Segment Editor** is closed by clicking **Cancel** or the close window button (), the data will be lost.

The next time the **Insert Isotherm Steps** button () is clicked, it will open the **Isotherm Segment Editor** with a 'blank' **Half Cycle Definition**:

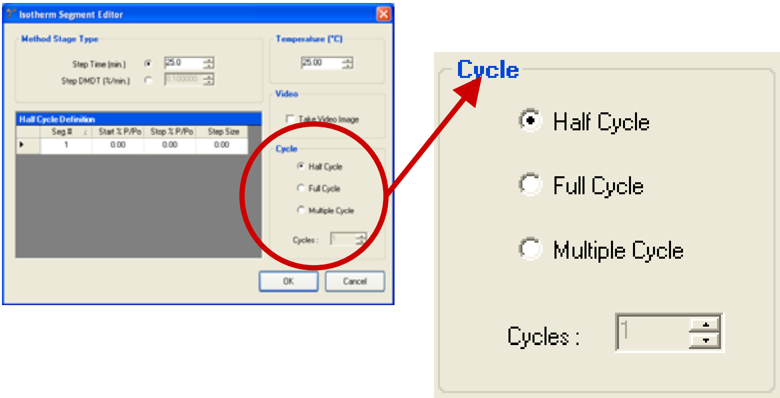


Half Cycle Definition

	Seg.#	Start % P/Po	Stop % P/Po	Step Size
▶	1	0.00	0.00	0.00

Cycle

This selection area determines whether the Isotherm Method is run in Half, Full, or Multiple Cycle mode:



Half Cycle

The **Half Cycle Definition** Panel creates, as the name suggests, a **Half Cycle**.

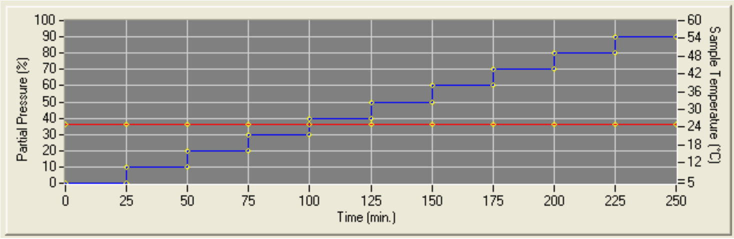
This means that whatever stages are specified in the panel, they constitute a **Half Cycle** experiment:

For example, if the following stages are created:

Half Cycle Definition

	Seg.#	Start % P/Po	Stop % P/Po	Step Size
▶	1	0.00	90.00	10.00

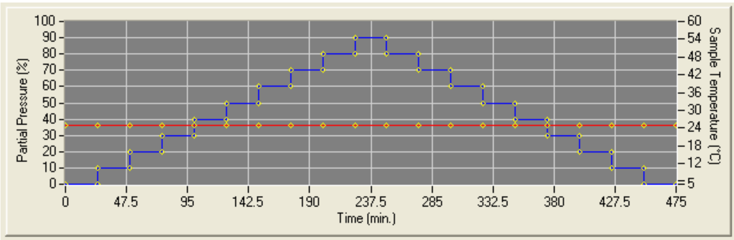
….then clicking **OK** on the **Isotherm Segment Editor** with the **Half Cycle** option selected will produce a Relative Humidity profile on the **Method Editor** window based on the Stages shown above with no additions or changes (ie; increasing Relative Humidity steps of 10% from 0% to 90%):



Full Cycle

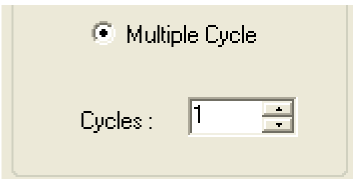
This option simply appends a mirror image of the original **Half Cycle** experiment to the Relative Humidity profile.

With the **Full Cycle** option selected on the **Isotherm Segment Editor**, the experiment shown above would appear on the Method Editor window as follows:

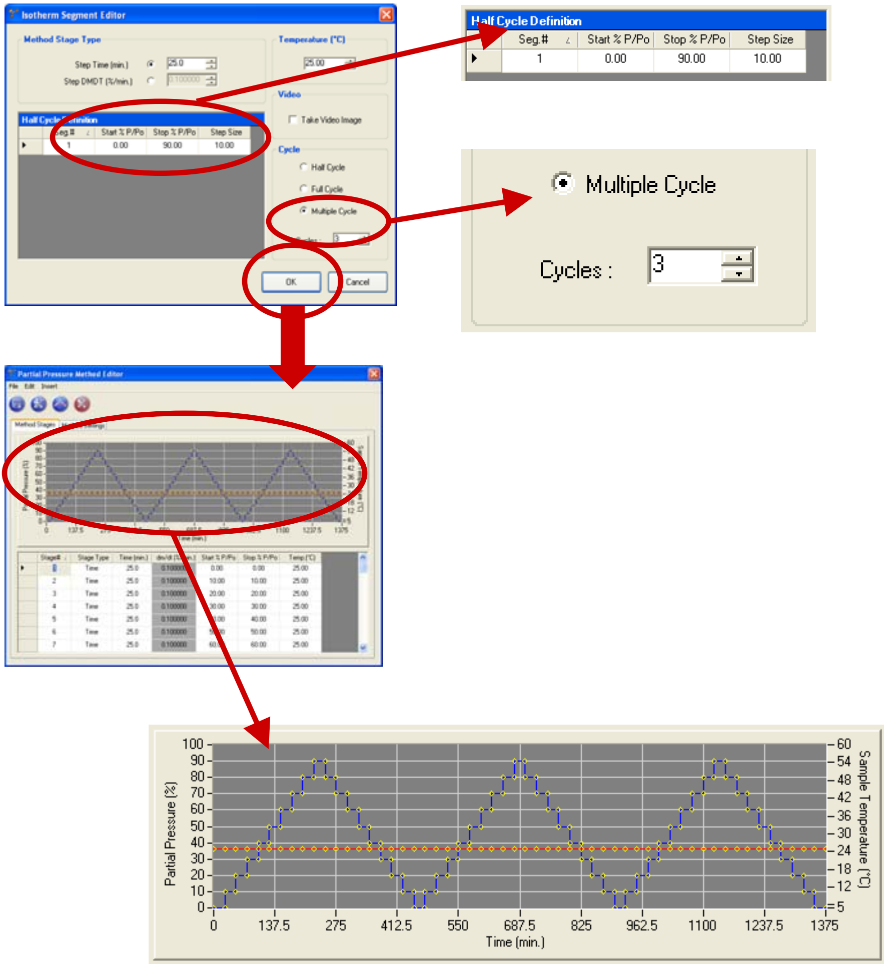


Multiple Cycles

This option creates several Full Cycles in sequence. The number of desired Full Cycles is specified in the data entry box that is activated when the **Multiple Cycle** option is selected:

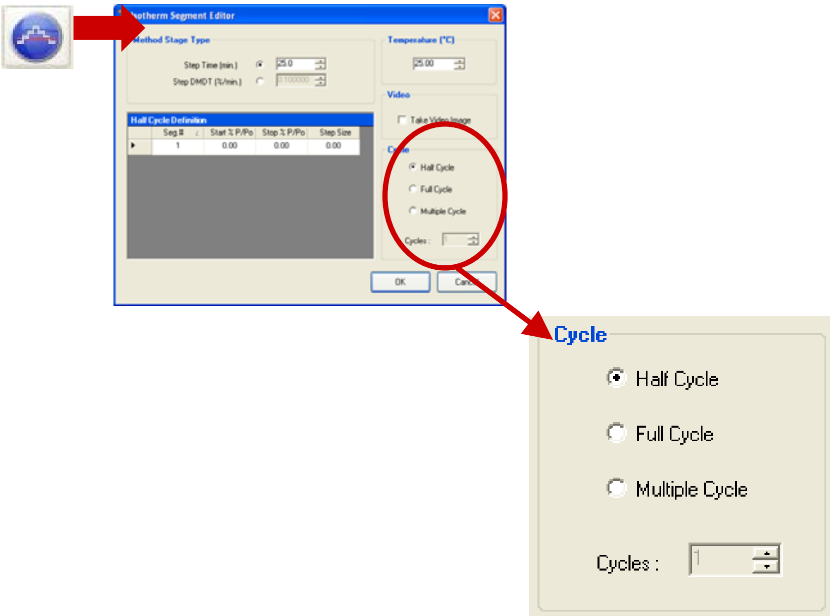


The number of cycles can be entered directly or by using the arrows on the side of the edit box. The maximum number of cycles that may be programmed is 10. As an example, selecting ‘3’ cycles as shown above for the current Half Cycle would produce the following Relative Humidity profile on the **Method Editor** window:



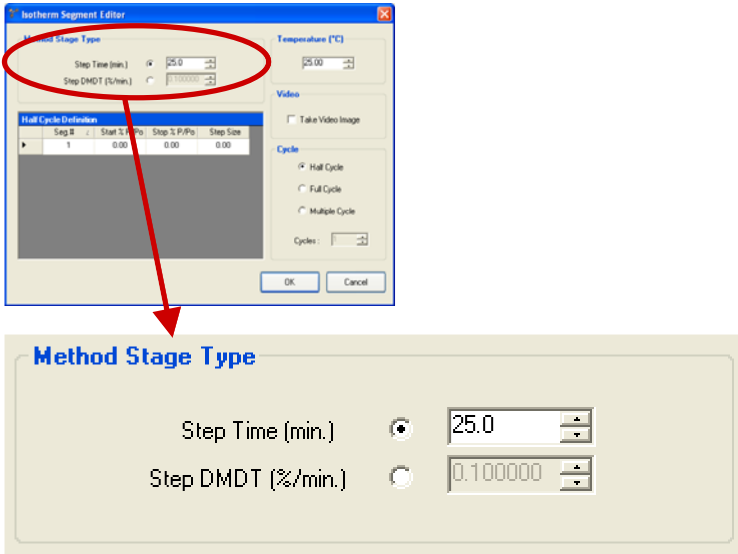
Closing the Isotherm Segment Editor

The next time the **Insert Isotherm Steps** button () is clicked, it will open the **Isotherm Segment Editor** with the default Cycle setting of Half Cycle:

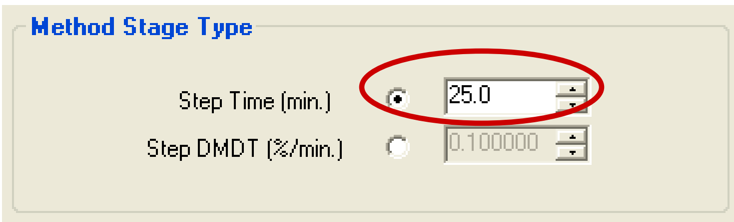


Method Stage Type

This box sets the stage end determinant to either Time or dm/dt (refer Sections 6.2.2 and 9.5.2.3 for an explanation of these parameters):

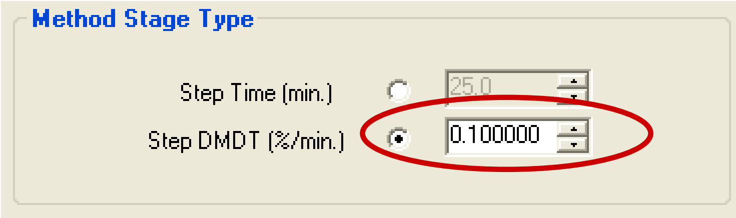


Clicking the **Step Time (min)** option will activate the data entry box as shown below:



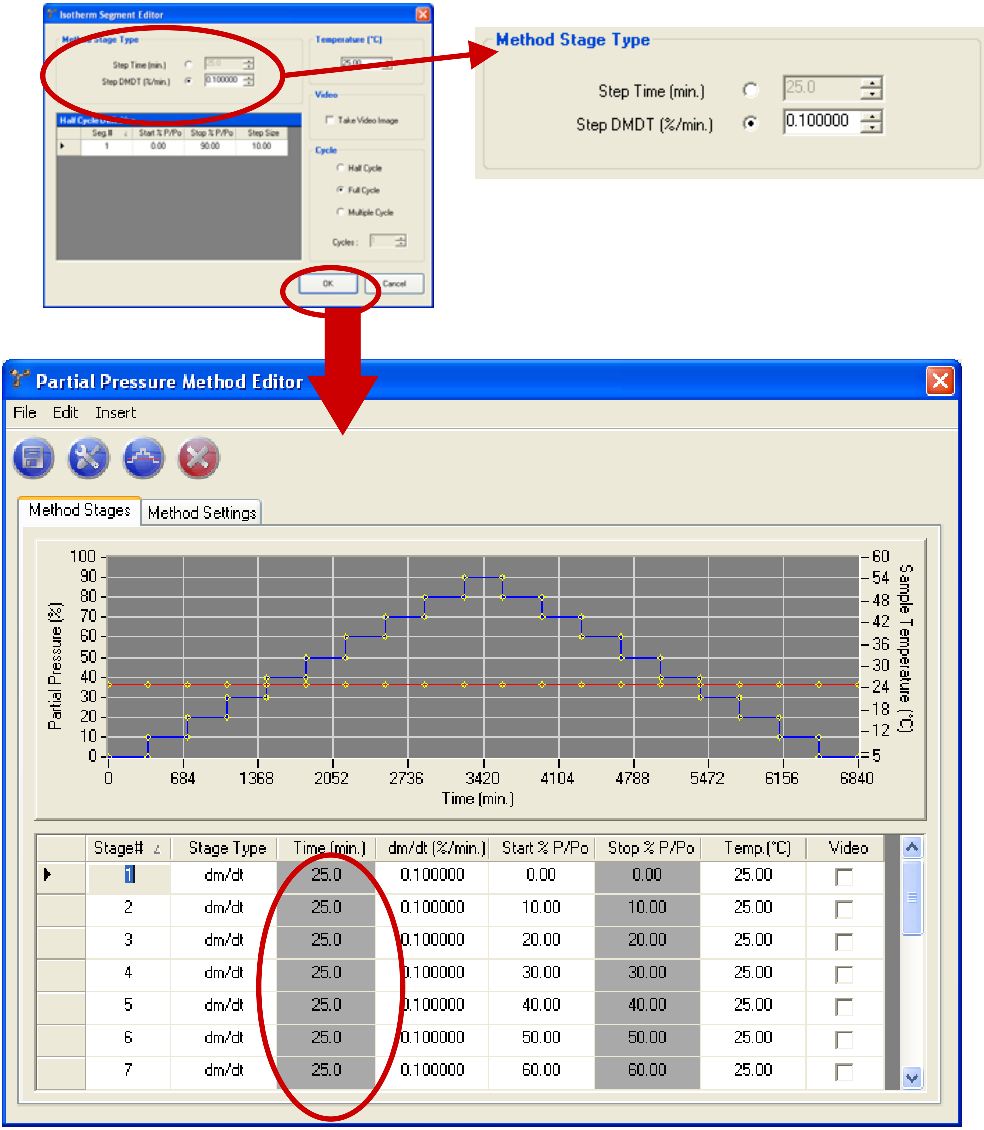
This box specifies the duration of each Stage in the Method in minutes. The figure may be entered directly, or by using the arrows at the side of the box. A maximum Stage time of 9999 minutes is allowable.

Clicking the **Step dm/dt (%/min)** option enables the data entry box that specifies the required dm/dt (%/min):



Again, the figure may be entered directly, or by using the arrows at the side of the box. A maximum DMDT of 9.99 % per minute (and a minimum of -9.99) is allowable. Also refer to earlier section in hardware for recommendations on suitable Step dm/dt values to use.

Once the desired **Method Stage Type** has been selected in the **Isotherm Segment Editor**, clicking **OK** returns the **Method Editor** window. The **Method Stages** tab displays the chosen **Method Stage Type** (the Method Stages Type which has not been chosen is greyed out):

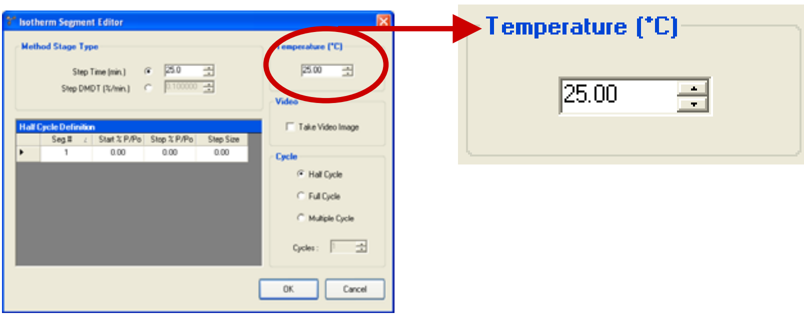

Isotherm Segment Editor
Temperature (°C)
Video
Take Video Image
Cycle
Half Cycle
Full Cycle
Multiple Cycle
Cycles :
OK
Cancel
Method Stage Type
Step Time (min.)
25.0
Step DMDT (%/min.)
0.100000
Partial Pressure Method Editor
File
Edit
Insert
Method Stages
Method Settings
Partial Pressure (%)
Sample Temperature (°C)
Time (min.)
Stage#
Stage Type
Time (min.)
dm/dt (%/min.)
Start % P/Po
Stop % P/Po
Temp.(°C)
Video
1 dm/dt 25.0 0.100000 0.00 0.00 25.00
2 dm/dt 25.0 0.100000 10.00 10.00 25.00
3 dm/dt 25.0 0.100000 20.00 20.00 25.00
4 dm/dt 25.0 0.100000 30.00 30.00 25.00
5 dm/dt 25.0 0.100000 40.00 40.00 25.00
6 dm/dt 25.0 0.100000 50.00 50.00 25.00
7 dm/dt 25.0 0.100000 60.00 60.00 25.00

Closing the Isotherm Segment Editor

Every time the **Insert Isotherm Steps** button  is clicked, the **Isotherm Segment Editor** window is opened featuring the **Method Stage Type** defined in the **Default New Row Settings** (Section 9.5.2.3).

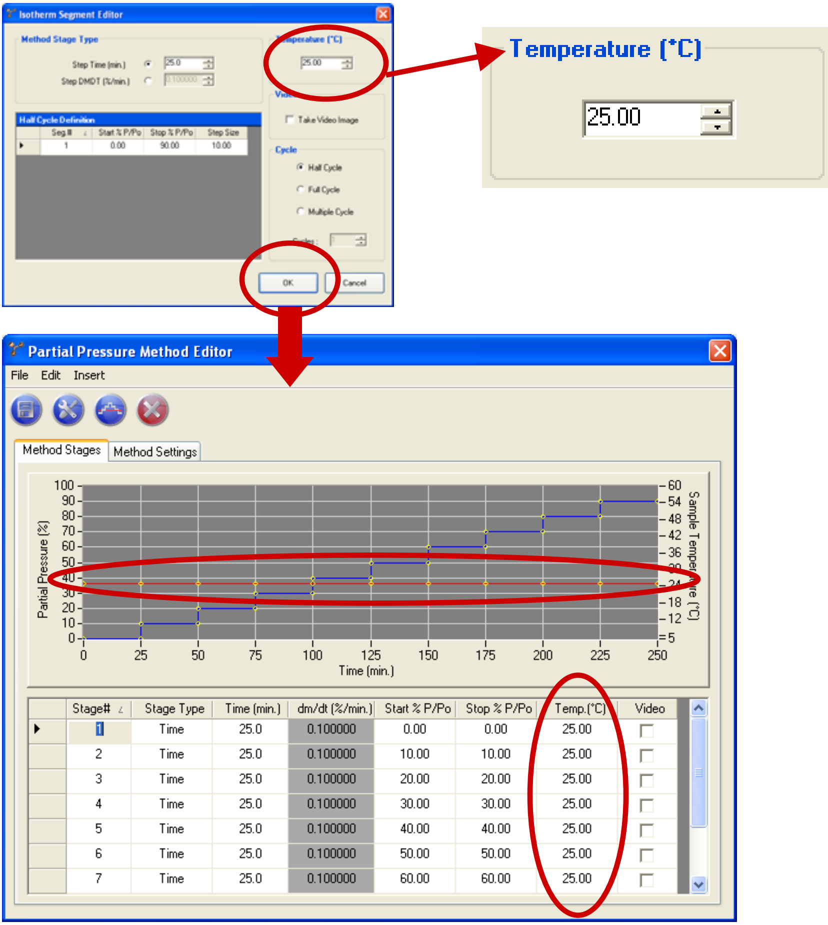
No matter what **Method Stage Type** had last been set in the **Isotherm Segment Editor**, the **Default New Row Settings** will appear whenever the Segment Editor is next opened.

Temperature(°C)
This selection area sets the constant isotherm temperature. The desired setting may be entered directly or by using the arrows to the right of the data entry box.



The maximum allowable Temperature is **60°C** and the minimum is **5°C**.

Once the Temperature is set and **OK** is clicked, the setting appears numerically in the **Method Editor**'s table, and graphically as a red line in its Relative Humidity profile:


Isotherm Segment Editor
Method Stage Type
Step Time (min.)
Step DMDT (%/min.)
Temperature (°C)
Take Video Image
Half Cycle Definition
Seg.# Start % P/Po Stop % P/Po Step Size
1 0.00 90.00 10.00
Cycle
Half Cycle
Full Cycle
Multiple Cycle
OK
Cancel
Temperature (°C)
25.00
Partial Pressure Method Editor
File Edit Insert
Method Stages
Method Settings
Partial Pressure (%)
Sample Temperature (°C)
Time (min.)
Stage# Stage Type Time (min.) dm/dt (%/min.) Start % P/Po Stop % P/Po Temp.(°C) Video
1 Time 25.0 0.100000 0.00 0.00 25.00
2 Time 25.0 0.100000 10.00 10.00 25.00
3 Time 25.0 0.100000 20.00 20.00 25.00
4 Time 25.0 0.100000 30.00 30.00 25.00
5 Time 25.0 0.100000 40.00 40.00 25.00
6 Time 25.0 0.100000 50.00 50.00 25.00
7 Time 25.0 0.100000 60.00 60.00 25.00

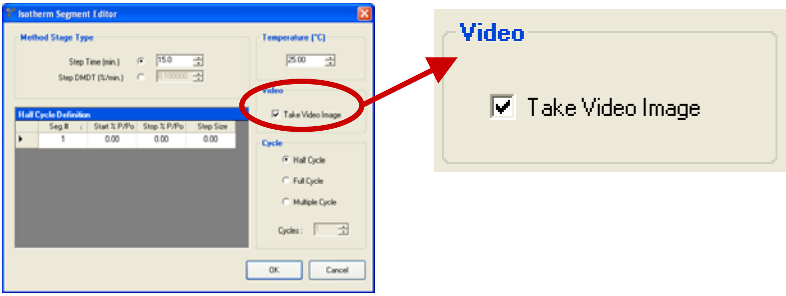
Closing the Isotherm Segment Editor

Every time the **Insert Isotherm Steps** button  is clicked, the **Isotherm Segment Editor** window is opened featuring the **Temperature** defined in the **Default New Row Settings** (Section 9.5.2.3).

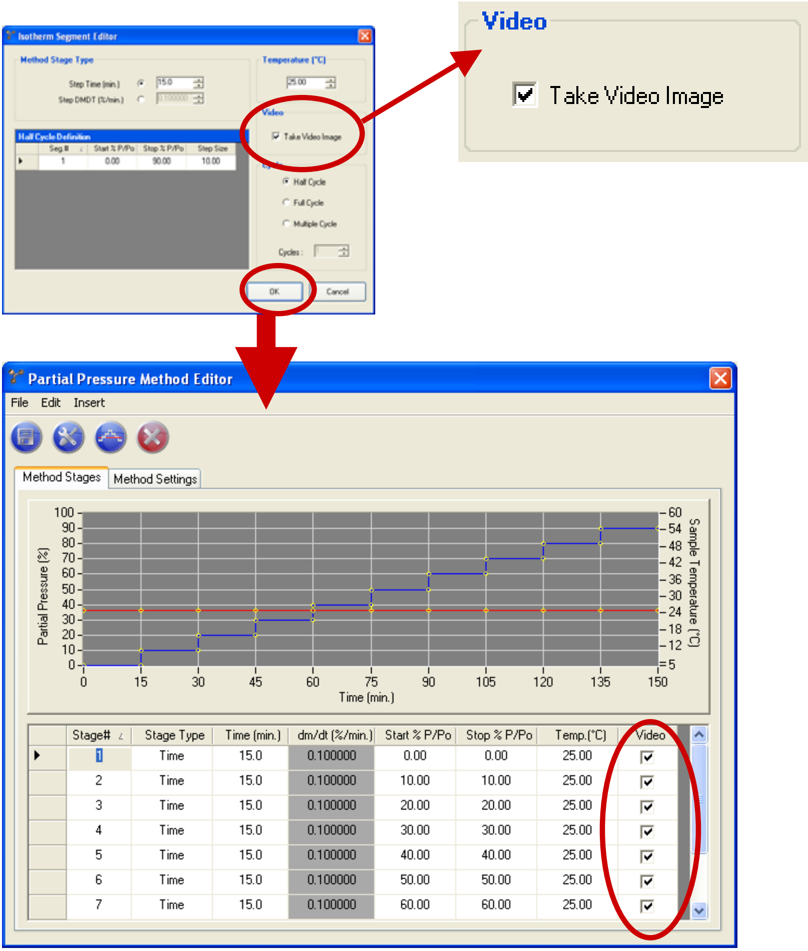
No matter what **Temperature** had last been set in the **Isotherm Segment Editor**, the **Default New Row Settings** will appear whenever the Segment Editor is next opened.

Video

Ticking this box ensures that a snapshot of the sample is taken at the end of each Stage of the Method (provided the Video preferences have been set to follow the Method settings – Section 9.2.1.3).



If the **Take Video Image** option has been ticked, clicking **OK** will return the **Method Editor** table; the **Video** box at the end of each Stage will be ticked:



In the **Method Editor** window, the Video boxes may be edited further if desired (ie; un-ticked, re-ticked).

Closing the Isotherm Segment Editor

Every time the **Insert Isotherm Steps** button  is clicked, the **Isotherm Segment Editor** window is opened featuring the **Video** setting defined in the **Default New Row Settings** (Section 9.5.2.3).

No matter what **Video** setting had last been specified in the **Isotherm Segment Editor**, the **Default New Row Settings** will appear whenever the Segment Editor is next opened.

Returning to the Method Editor main screen

After specifying all desired parameters in the **Isotherm Segment Editor**, clicking **OK** will return the **Partial Pressure Method Editor** main screen, with the newly created Method installed – see the example outlined in Figure 9.6 below:

Clicking the Insert Isotherm Steps button  or going to **File → Insert Isotherm Steps…** calls up the **Isotherm Segment Editor**:

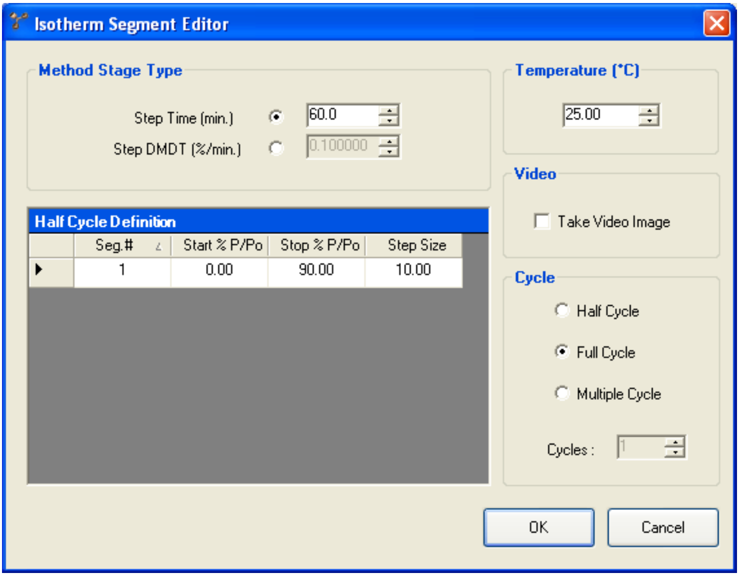


Figure 9.6: After the **Isotherm Segment Editor** is completed as shown above and OK is clicked….

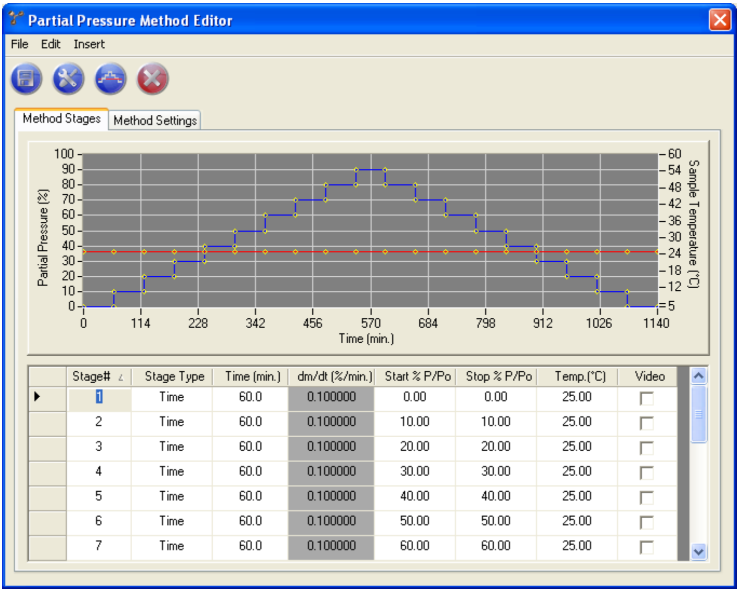


Figure 9.7:…the **Partial Pressure Method Editor** main screen is returned, displaying the details of the newly created Method

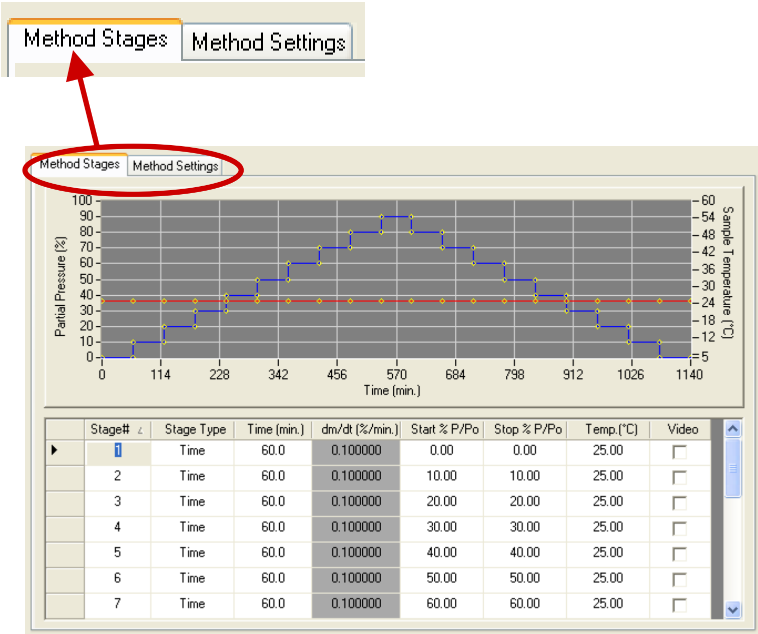
Note: If a Method was already contained in the Method Editor, then any new Method stages created using the Isotherm Segment Editor will be inserted into the Method originally displayed.

Changes flowing through from Default New Row Settings

When the **Isotherm Segment Editor** is first opened, the **Method Stage Type** and **Temperature** sections will contain the default values specified in the **Default New Row Settings** (see Section 9.5.2.3 above).

9.5.2.5 Method Stages Tab

The **Method Stages** tab displays the relative humidity/temperature profile of the Method, and also an editable table of the parameters of each Stage of the Method.



Stage#	Stage Type	Time (min.)	dm/dt (%/min.)	Start % P/Po	Stop % P/Po	Temp.(°C)	Video
1	Time	60.0	0.100000	0.00	0.00	25.00	
2	Time	60.0	0.100000	10.00	10.00	25.00	
3	Time	60.0	0.100000	20.00	20.00	25.00	
4	Time	60.0	0.100000	30.00	30.00	25.00	
5	Time	60.0	0.100000	40.00	40.00	25.00	
6	Time	60.0	0.100000	50.00	50.00	25.00	
7	Time	60.0	0.100000	60.00	60.00	25.00	

Methods may also be created and edited on the Method Stages Tab without using the Insert Isotherm Steps button  (Section 9.5.2.4 above) – however the Insert Isotherm Steps function provides a very fast way of establishing the broad features of a Method file. Alternatively, settings specified in the Isotherm Segment Editor may be altered in this table.

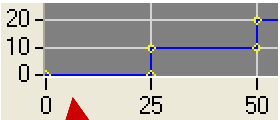
The partial pressure table includes a column for **Stage #**, which is not editable. The **Stage Type** column allows the user to select the stage-end determinant (Time or dm/dt). This is followed by editable **Time (min)** and **dm/dt (%/min)** columns (explained in the previous section). **Start** and **Stop %P/P$_o$** ,and **Temp (°C)** columns are also featured (again, explained in the previous section).

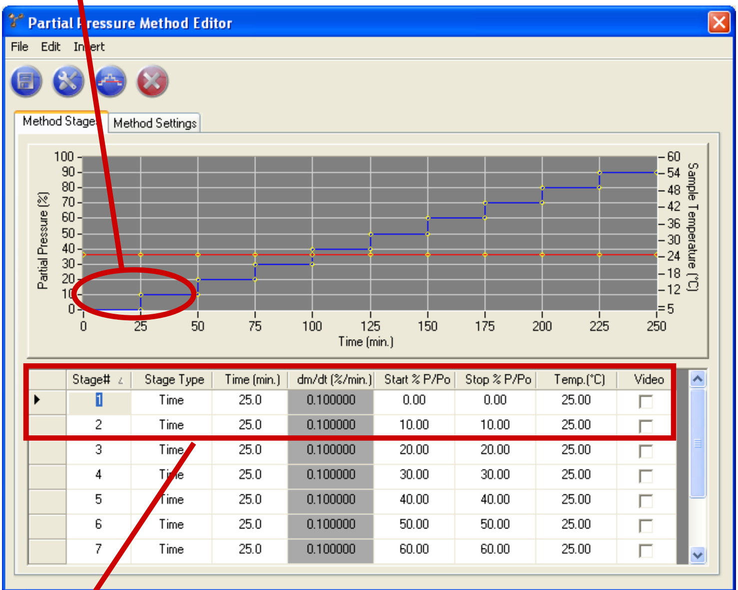
Creating 'ramps'

Any Row values may be altered by simply clicking on them, as described in Section 9.5.2.4.

A ramp may be created by simply making the Start %P/P$_o$ different from the Stop %P/P$_o$ in a Stage.

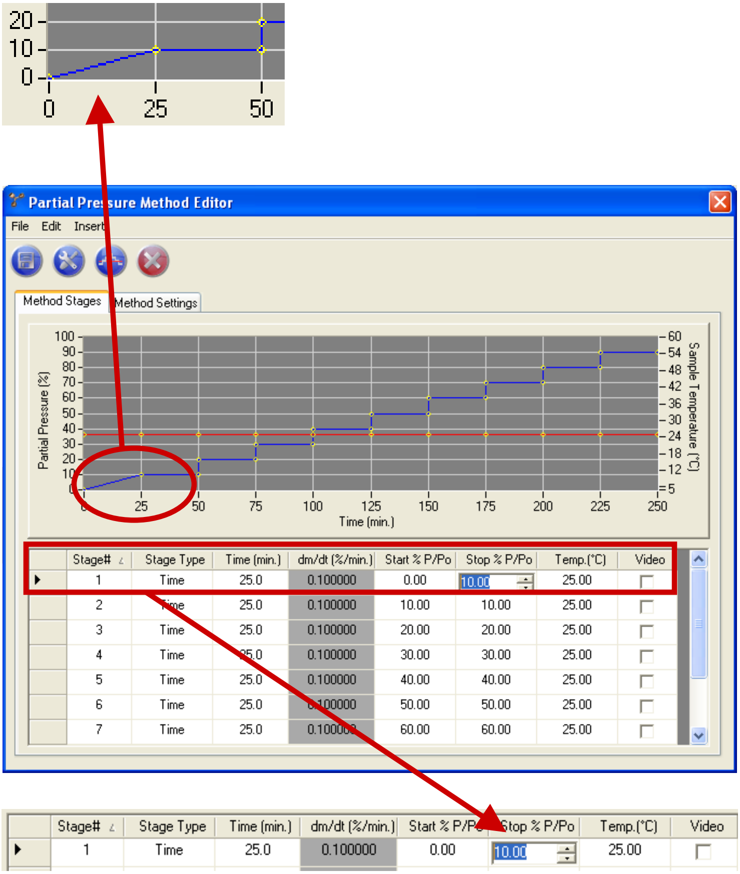
For example, if a Method is initially set up as shown:





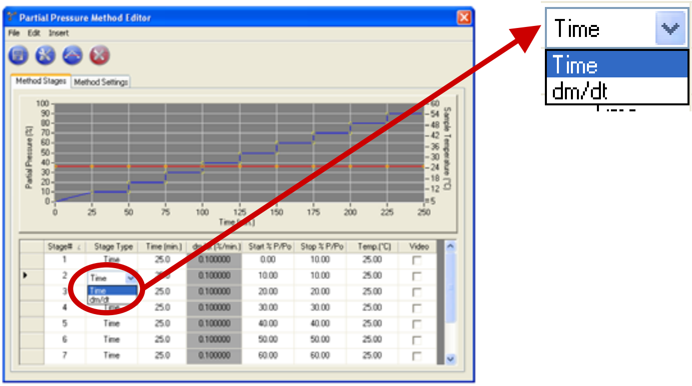
Stage#	Stage Type	Time (min.)	dm/dt (%/min.)	Start % P/Po	Stop % P/Po	Temp.(°C)	Video
1	Time	60.0	0.100000	0.00	0.00	25.00	☐
2	Time	60.0	0.100000	10.00	10.00	25.00	☐

Then changing the **Stop %P/P$_o$** in the first stage from 0%P/P$_o$ to 10% P/P$_o$ will create a ‘ramp’ from 0% to 10% P/P$_o$. The software automatically steps the ramp in increments of 1% P/P$_o$:



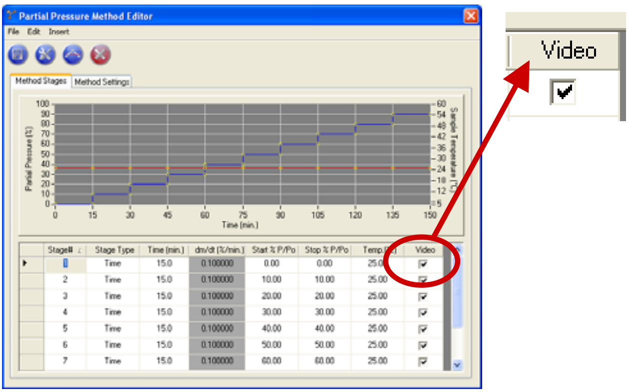
Stage End Determinant

The stage end determinant may be changed between time and dm/dt by using the drop-down menu:



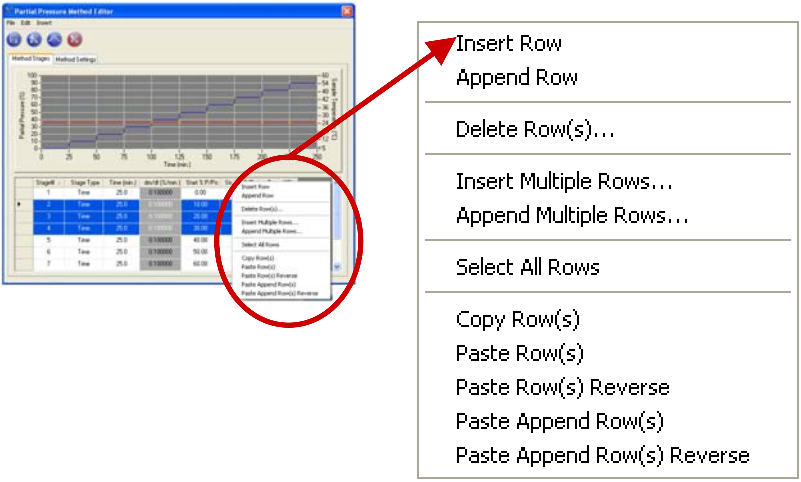
Video

As mentioned above, the **Video** check box may be ticked or un-ticked in each row, or Stage, depending upon whether the user wishes for a sample snapshot to be taken at the end of each stage.

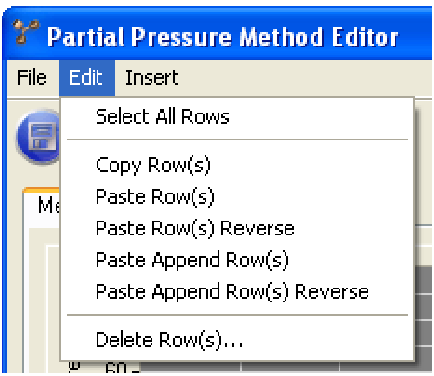


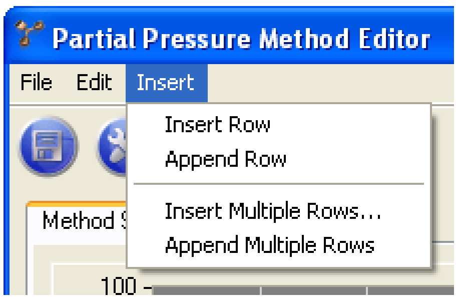
Performing Operations upon Rows:

Right-clicking the Stages table calls up the following shortcut menu:



Note: The functions listed above can also be found in the Edit and Insert drop-down menus on the Partial Pressure Method Editor window:





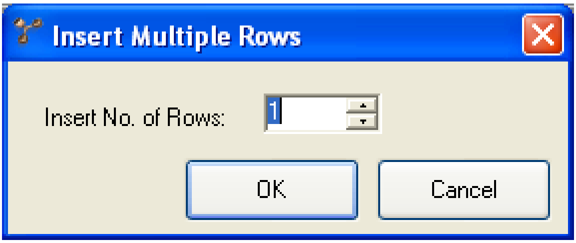
Insert Row, Append Row, Delete Row(s)

These items are explained in Section 9.5.2.4.

Note: Rows are inserted or appended with the values specified in the **Default New Row Settings** (Section 9.5.2.3).

Insert Multiple Rows & Append Multiple Rows

These options call up a window asking the user to specify how many Rows are to be inserted or appended. **The same panel appears for the Append Multiple Rows option**:



The number of rows to be inserted or appended may be entered directly into the data entry box, or by using the arrows to the right of the box. A maximum of 50 rows may be specified.

The new Rows are inserted or appended after clicking **OK.**

Select All Rows

Selects all rows in the table:

Stage#	Stage Type	Time (min.)	dm/dt (%/min.)	Start % P/Po	Stop % P/Po	Temp.(°C)	Video
1	Time	25.0	0.100000	0.00	10.00	25.00	
2	Time	25.0	0.100000	10.00	10.00	25.00	
3	Time	25.0	0.100000	20.00	20.00	25.00	
4	Time	25.0	0.100000	30.00	30.00	25.00	
5	Time	25.0	0.100000	40.00	40.00	25.00	
6	Time	25.0	0.100000	50.00	50.00	25.00	
7	Time	25.0	0.100000	60.00	60.00	25.00	

Copy Row(s)

This option copies the current or selected Row(s).

Paste Row(s)

This option pastes the copied Row(s) above the current Row.

The currently active or selected Row(s) will be those initially copied unless new Row(s) are selected before pasting.

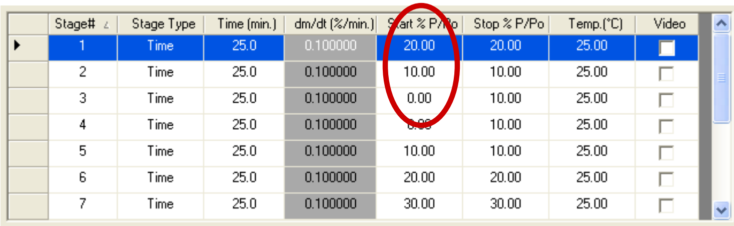
Paste Row(s) Reverse

If multiple rows have been copied, then this function pastes them in their reverse order (bottom to top).

For example, if the following rows are copied (going from 0% to 20% P/P_o top to bottom):

Stage#	Stage Type	Time (min.)	dm/dt (%/min.)	Start % P/Po	Stop % P/Po	Temp.(°C)	Video
1	Time	25.0	0.100000	0.00	10.00	25.00	
2	Time	25.0	0.100000	10.00	10.00	25.00	
3	Time	25.0	0.100000	20.00	20.00	25.00	
4	Time	25.0	0.100000	30.00	30.00	25.00	
5	Time	25.0	0.100000	40.00	40.00	25.00	
6	Time	25.0	0.100000	50.00	50.00	25.00	
7	Time	25.0	0.100000	60.00	60.00	25.00	

…then Paste Row(s) Reverse will place the copied Rows above the current row (in this case Row #1) in reverse order:



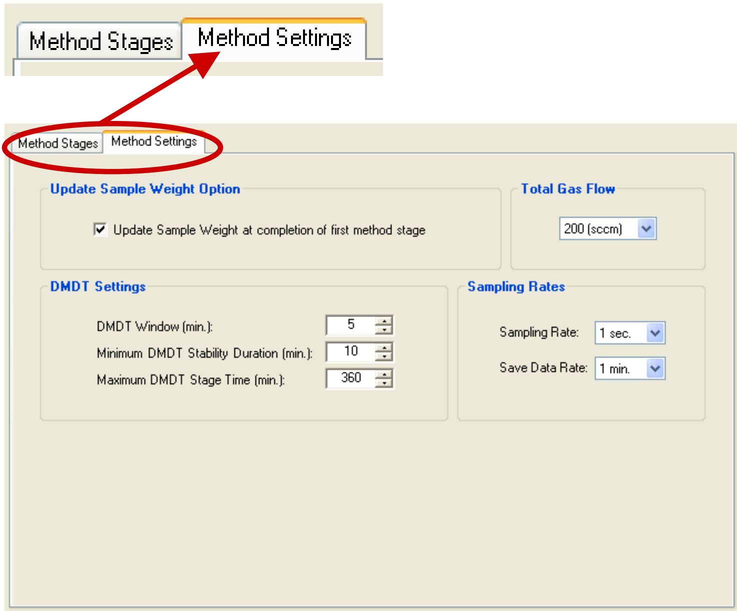
	Stage#	Stage Type	Time (min.)	dm/dt (%/min.)	Start % P/Po	Stop % P/Po	Temp.(°C)	Video
▶	1	Time	25.0	0.100000	20.00	20.00	25.00	☐
	2	Time	25.0	0.100000	10.00	10.00	25.00	☐
	3	Time	25.0	0.100000	0.00	10.00	25.00	☐
	4	Time	25.0	0.100000	0.00	10.00	25.00	☐
	5	Time	25.0	0.100000	10.00	10.00	25.00	☐
	6	Time	25.0	0.100000	20.00	20.00	25.00	☐
	7	Time	25.0	0.100000	30.00	30.00	25.00	☐

Paste Append Row(s) Reverse

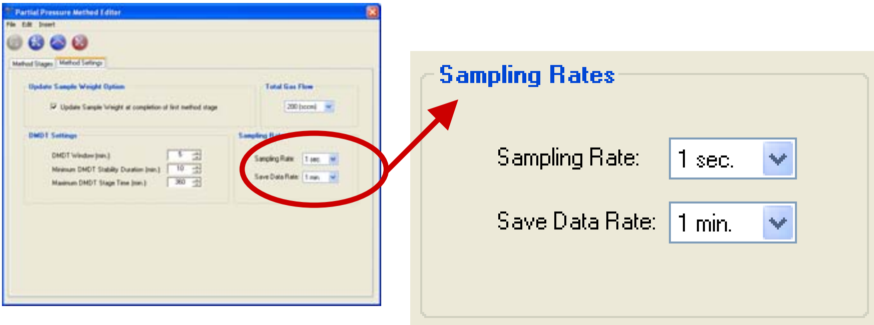
This performs the same action as Paste Row(s) Reverse – only the copied rows are pasted to the bottom of the table.

9.5.2.6 Method Settings tab

This window, shown below, sets the experimental conditions of the Method:



Sampling Rates

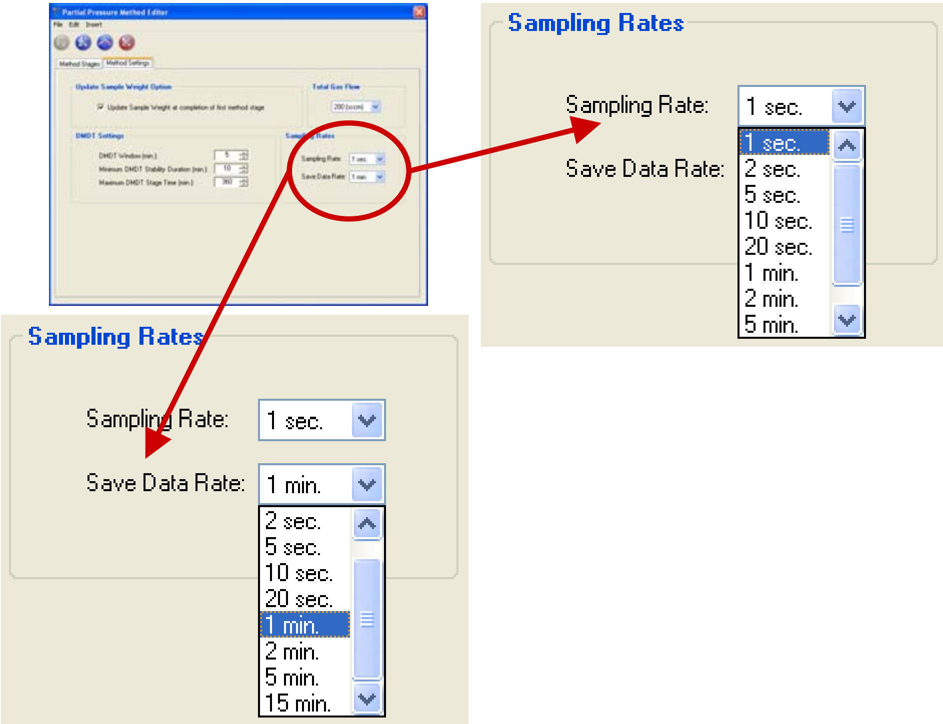


These drop-down lists allow the user to define the rate at which data is saved and displayed when running the Method. Both rates are set to the current **General Preference** settings by default (Section 9.2.1.1). Please also refer to Section 9.3.1.

Sampling Rate: The rate at which mass data is updated on the software interface. The recommended setting is 20 seconds.

Save Data Rate: The rate at which data is saved to a DVS raw data file on the hard disk. The recommended setting is 1 minute. More frequent data logging gives rise to larger file sizes.

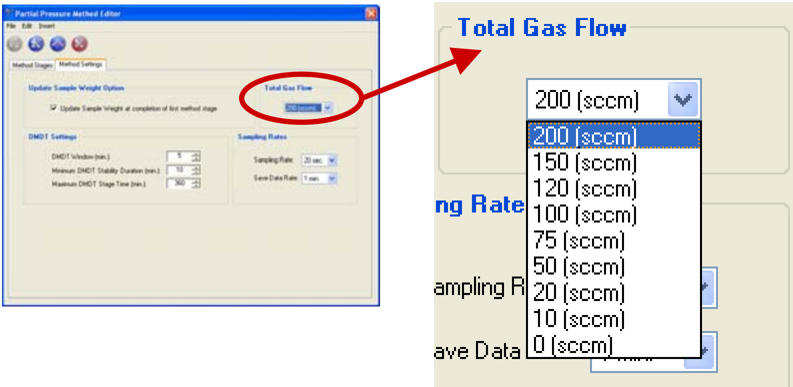
Clicking on the edit boxes or the arrows to their right reveals the list of available flowrates:



Note: The Sampling and Save Data Rates defined in **File→Preferences** will appear in the **Sampling Rates** drop-down boxes of the Method Settings tab when the Partial Pressure Method Editor is first opened.

These settings can then be altered and saved to the new Method file.

Total GasFlow



This determines the flow rate while the Method is running. Values are chosen using the drop-down menu as shown above, and will initially be set to the default value of 200sccm.

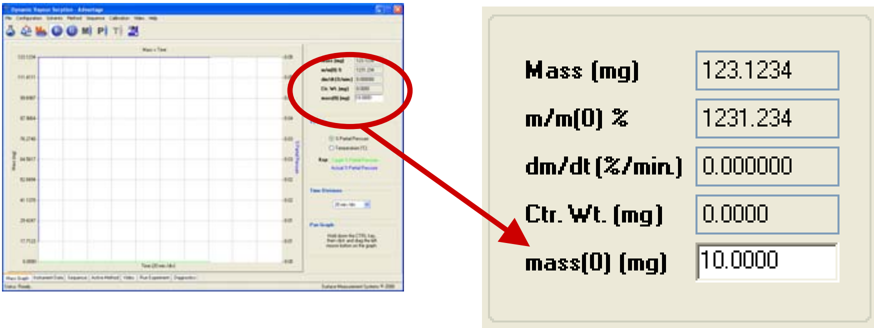
Note: The Flow Rate may also be set on the Instrument Data panel (Section 10.4), but that setting is independent of flowrates set for Method files.

It is important to make sure that the Flow Rate required for the Method is set correctly in the Method Settings tab, as the Instrument Data tab setting will have no bearing on the Method setting.
If the Method Settings tab is not used to alter the Method's Flow Rate, then the default value of 200sccm will be used automatically when the Method is run.

For most experiments, 200sccm will be appropriate.

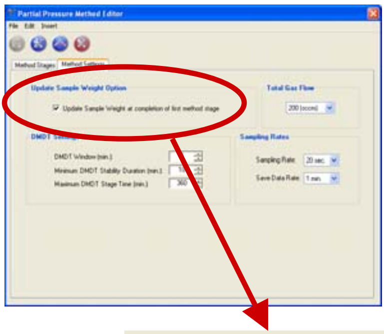
Update Sample Weight Option

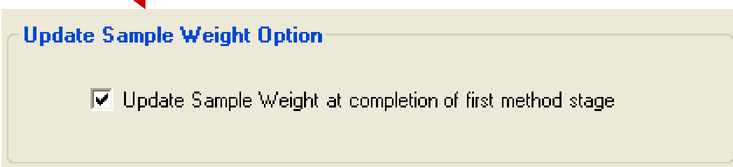
On the Control software's **Mass Graph** tab (Section 10.3) is displayed a sample weight:



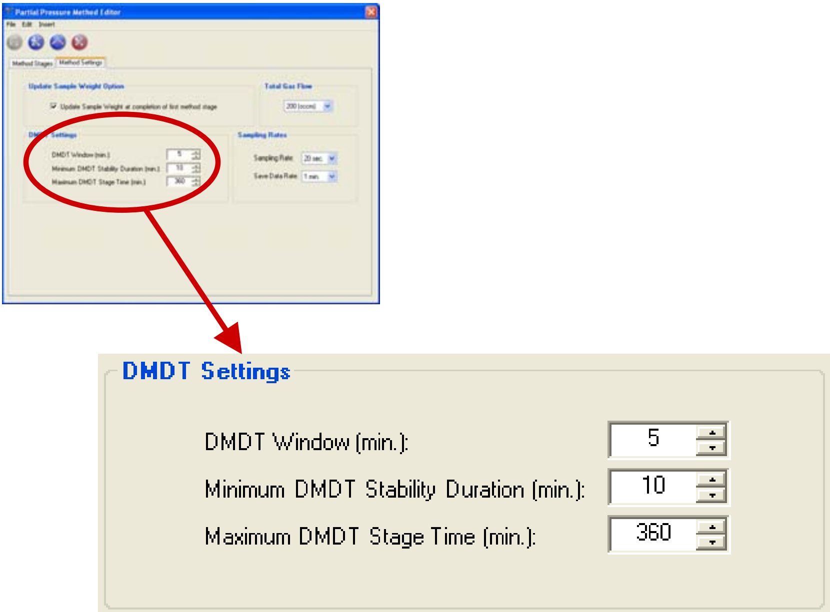
When the **Update Sample Weight Option** in the **Method Settings** tab is checked, the **mass(0) (mg)** value is set to the last mass value recorded at the end of the Method's first stage.

This option is checked by default - if the option is un-checked, then the **mass(0) (mg)** value will remain at the value to which it was last set.





dm/dt Settings



This area specifies the dm/dt settings if dm/dt has been chosen as the stage end determinant – the actual dm/dt value required to define sample 'stability' is set either in the **Partial Pressure Method Editor** window, or in **Insert Isotherm Steps**. Refer also Sections 9.5.2.1 and 9.5.2.3.

dm/dt Window (min)

This defines the time window (minutes) across which the sample's dm/dt is calculated.

The DVS Intrinsic software samples mass data every second (regardless of the Save Data Rate). This means that a **dm/dt Window** of 5 minutes would lead to the dm/dt value being calculated across 300 mass data points (5x60sec).

The dm/dt value is re-calculated every second (as each new mass point is sampled). If a dm/dt Window of 2min were used, then the dm/dt value would be re-calculated every second across a mass data range of 120 points.

The recommended value is 5 minutes, which is set as default, but a minimum of 1minute and maximum of 10 minutes may also be set.

Min dm/dt Stability Duration

This sets the minimum time for which the sample's mass change must satisfy the dm/dt requirement set in the Method Editor. Once the sample has satisfied the dm/dt requirement for this minimum period, the experiment moves on to the next Stage.

The recommended value is 10 minutes, which is set as default. The possible values range from 10 to 2000 minutes.

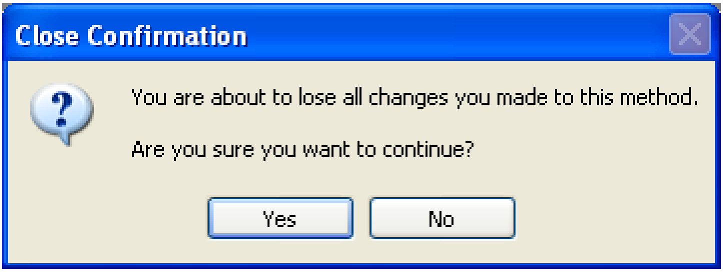
Max dm/dt Stage Time

This defines the maximum time that the instrument will run any Stage before moving automatically to the next stage, **irrespective** of whether the dm/dt criterion has been met.

The recommended default is 360 minutes. The permissible values range from 10 minutes to 2000 minutes.

9.5.2.7 Close Method Editor 

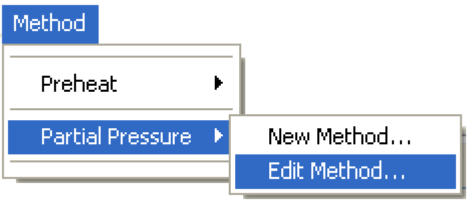
Clicking the **Close Method Editor** button will call up the following window:



Clicking **Yes** will exit the **Method Editor** and lose all changes made.

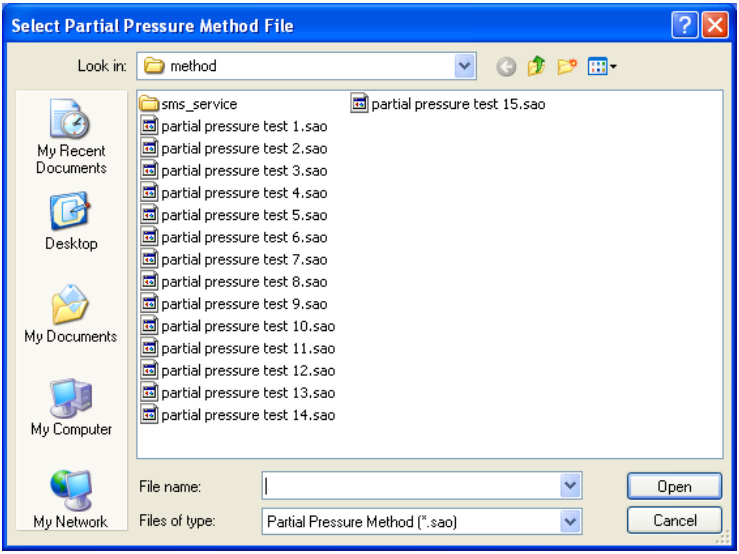
Clicking **No** will restore the Method Editor screen, allowing the Method to be saved if desired.

9.5.2.8 Edit Method



This option allows the user to edit previously saved Partial Pressure Method files.

Clicking the **Method→Partial Pressrue→Edit Method** option calls up the **Select Partial Pressure Method File** window which allows the user to select which Partial Pressure Method file to edit:



By default, the software will immediately go to the directory specified in the Preferences option (Section 9.2.1.2) – another directory may be selected in the window above if desired.

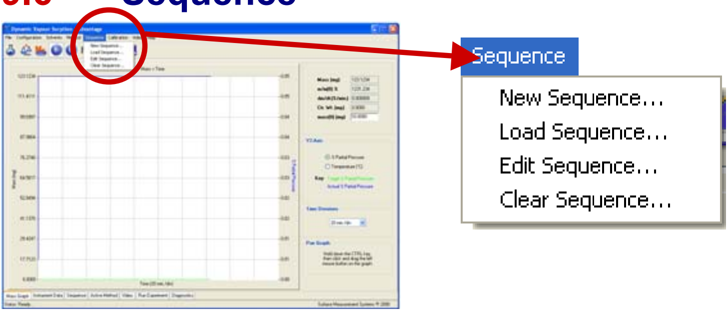
Upon choosing a file, clicking **Open** will open the Partial Pressure Method file for editing.

The file may be edited and saved as described above in the **New Method** Section.

Note: If a Partial Pressure Method is *active* (ie; running) as part of a running Sequence, then it may still be edited. While the Preheat Method is running, the **Method→Preheat→Edit Method** option may be used as described in Section 9.5.1.2 to do this.

The newly edited Method will need to be saved under a new name – but note that it is not automatically loaded into the running Sequence. To do this, the running Sequence will need to be edited to include the new Method as described in Section 9.6.1.3.

9.6 Sequence



A DVS Sequence is a series of up to ten DVS Methods (Preheat, Partial Pressure or DCC – refer Section 9.5) that are run consecutively.

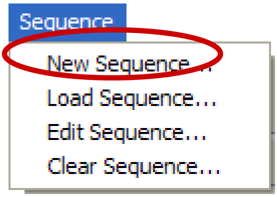
The Sequence menu allows the user to create a new Sequence, load an existing Sequence, edit an existing Sequence or to clear a previously loaded Sequence.

Note: A Sequence must first be loaded before the **Edit Sequence** and **Clear Sequence** menu options can become active.

Also, the entire Sequence menu is disabled while data is being saved. The data saving must be stopped in order to access the menu.

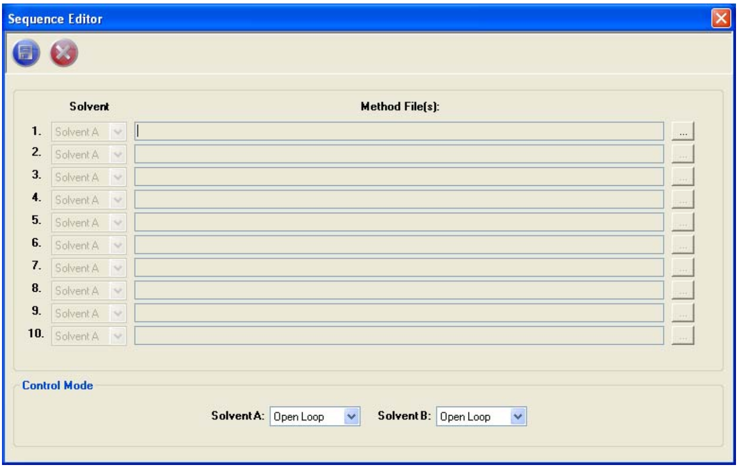
Note: The Sequence menu may also be called up by going to the **Sequence** panel and right-clicking anywhere on the tab (see Section 10.5.1).

9.6.1.1 New Sequence



This option allows the user to create a new Sequence.

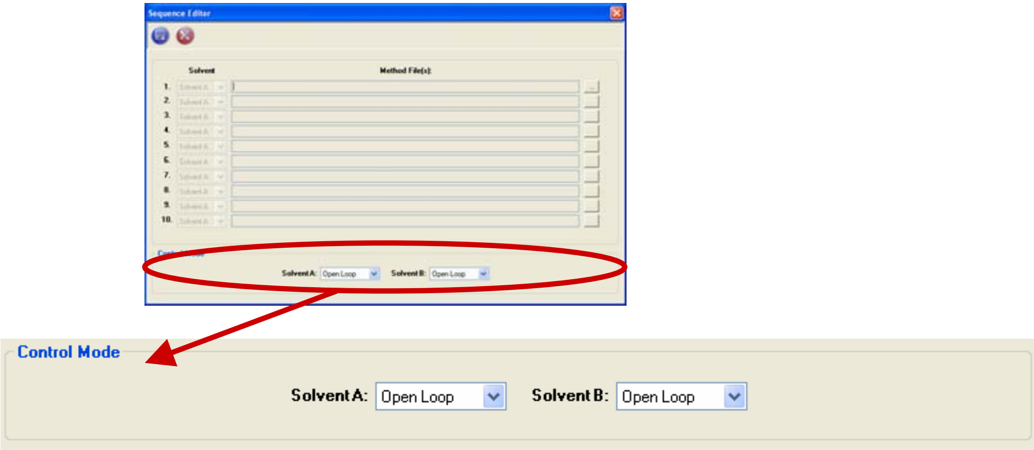
Clicking the **New Sequence** option calls up the following **Sequence Editor** window:



The Sequence Editor allows the user to load the Methods that are to be run and also configure which solvent is to be used for each particular Method.

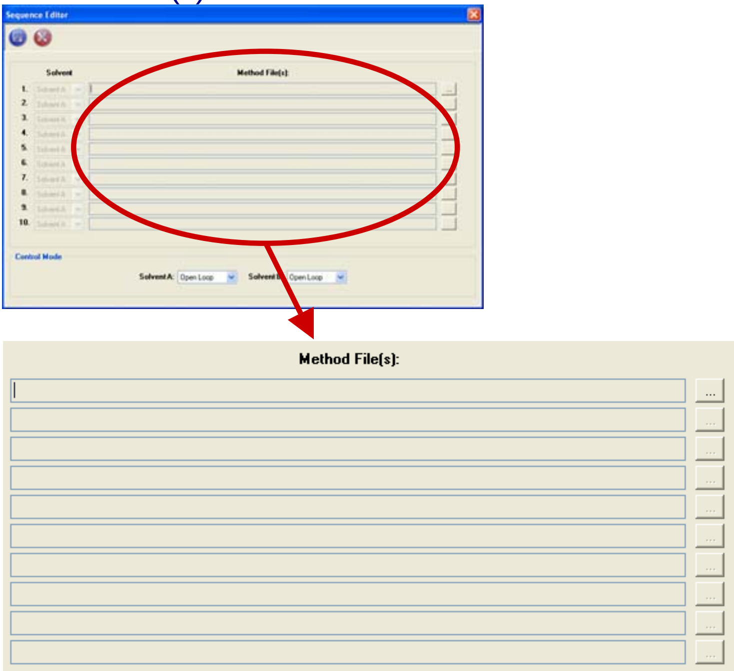
The settings displayed in the Sequence Editor are the same as those displayed in the Sequence Panel (Section 10.5) - the only difference being that the settings in the Sequence Editor may be changed.

Control Mode



These drop-down boxes allow the user to set the Control Mode to be used for Solvents A and B. Both solvents are set to the current General Preference settings by default (Section 9.2.1.1). Please refer to Section 9.3.4 for more detailed information on Control Modes.

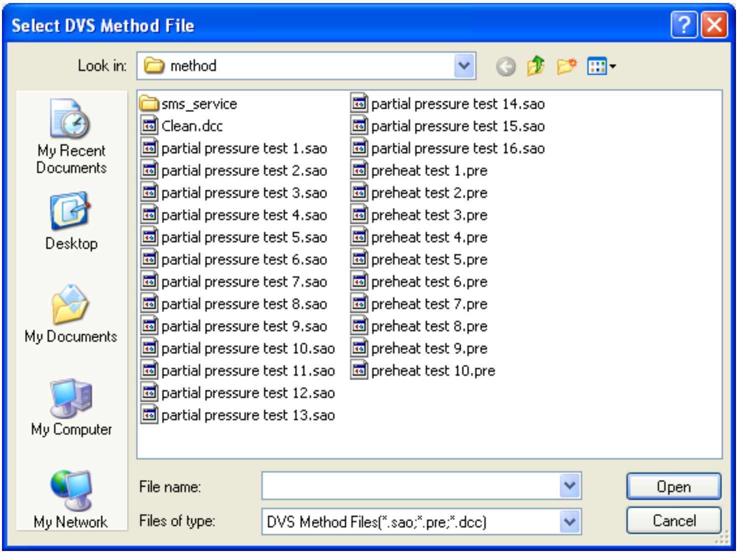
Method File(s)



This area allows the user to select the Method files which will constitute the Sequence – a maximum of ten Method files may be used. The selected Method files can be either Pre-heat Methods (.pre), Partial Pressure Methods (.sao) or the special sensor cleaning method “Clean.dcc”.

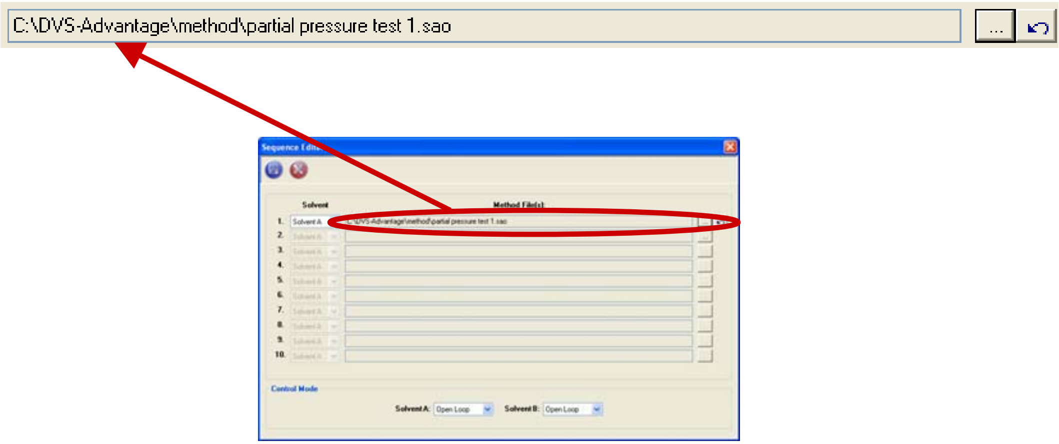
The list of Methods must be filled from the top down.

To insert a Method into the new Sequence, the **Load Method** button ... at the end of the highest empty box must be clicked – this will call up the **Select DVS Method File** window:



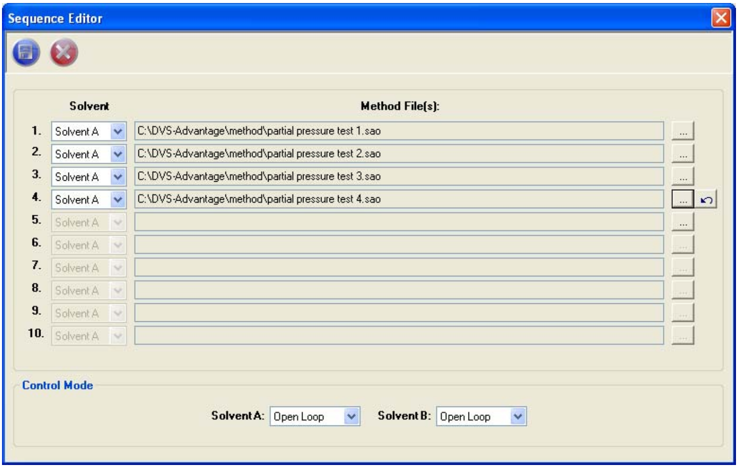
The Select DVS Method file window will automatically navigate to the default Method Files directory specified in Preferences (Section 9.2.1). From there, a different directory may be chosen if desired.

Upon selecting a Method file and clicking **Open**, the Sequence Editor window is returned featuring the Method File loaded (along with its directory location) at the top of the list (only one Method file may be selected at a time):



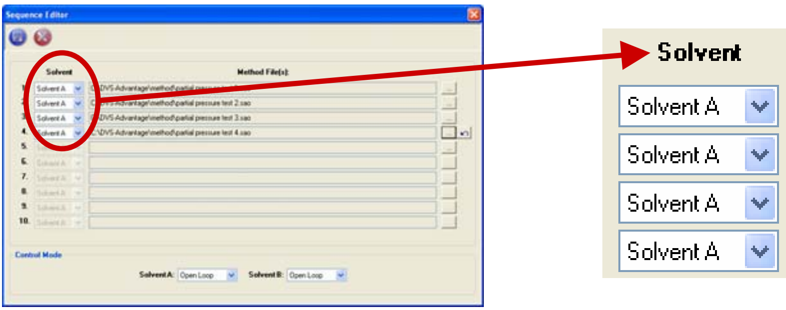
Note: After a Method file has been loaded, the Load Method button remains active in case a different Method file needs to be loaded in place of the original.

In this way, the Sequence may be populated with Method files one-by-one as desired:



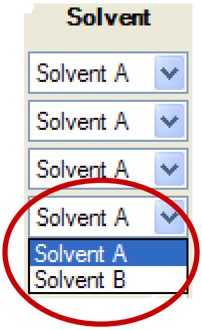
As the list is populated, the **Unload Method** button  appears next to the last populated box. Clicking this button removes the last Method file from the list:

Solvent

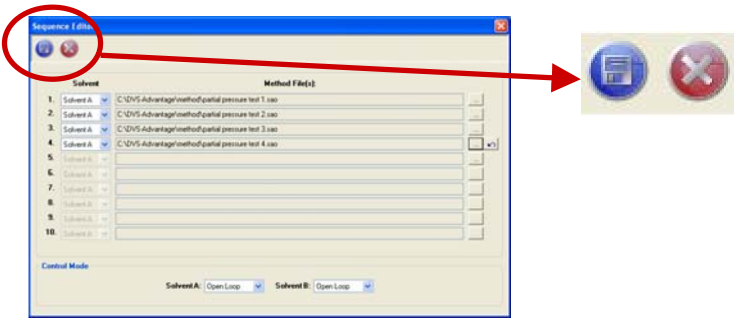


Once a Method file has been loaded the Solvent edit box to its left becomes active. This allows the user to select which solvent will be used with this Method in the Sequence.

The Sequence Editor appears with Solvent A (left-hand bottle) as default – clicking the edit box or the down arrow next to it will allow Solvent B (right-hand bottle) to be selected:

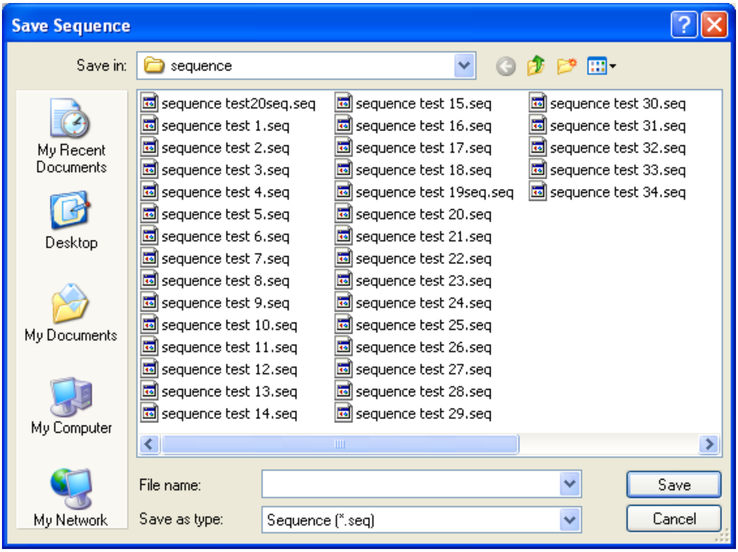


Save Sequence and Close Sequence Editor



Clicking the **Save Sequence** button  allows the user to save the newly created Sequence file.

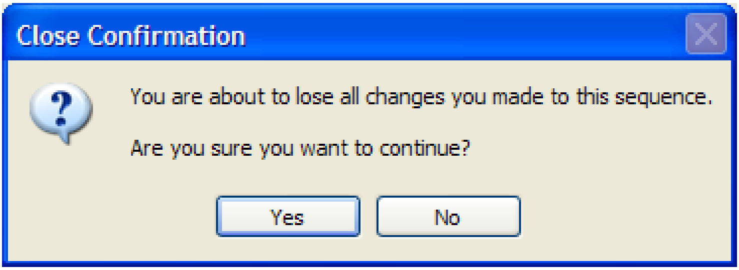
Clicking the button calls up the **Save Sequence** window:



By default, the software will immediately navigate to the directory specified in the Preferences option (Section 9.2.1.2) – another directory may be selected in the window above if desired.

Upon specifying the Sequence filename name, clicking **Save** will save the file and close the Save Sequence window and the Sequence Editor window, returning the DVS Advantage Software main panel. The newly saved Sequence will appear in the Sequence Panel (Section 10.5).

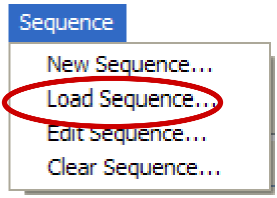
Clicking the **Close Sequence Editor** button  (or the **Close** button ) calls up the following window:



Clicking **Yes** closes the Sequence Editor window and returns the DVS Advantage Software main panel. The new Sequence file will not be saved.

Clicking **No** returns the Sequence Editor window.

9.6.1.2 Load Sequence



This option allows the user to load an existing Sequence file.

Clicking the **Load Sequence** option calls up the following window:

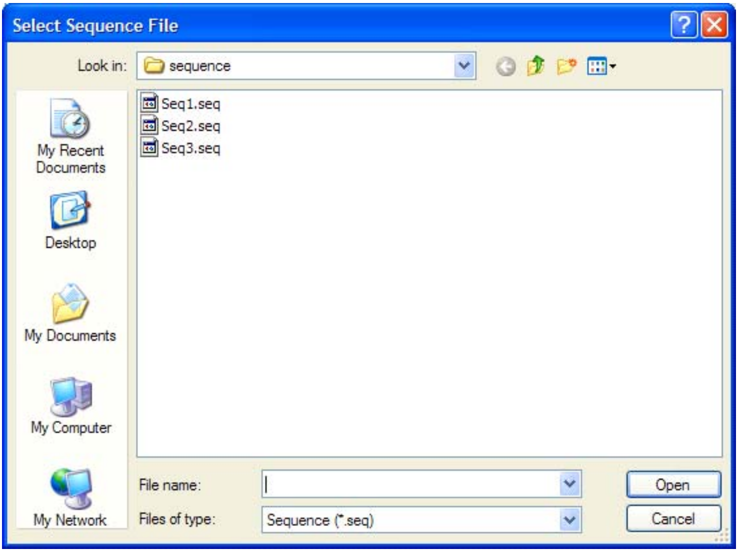
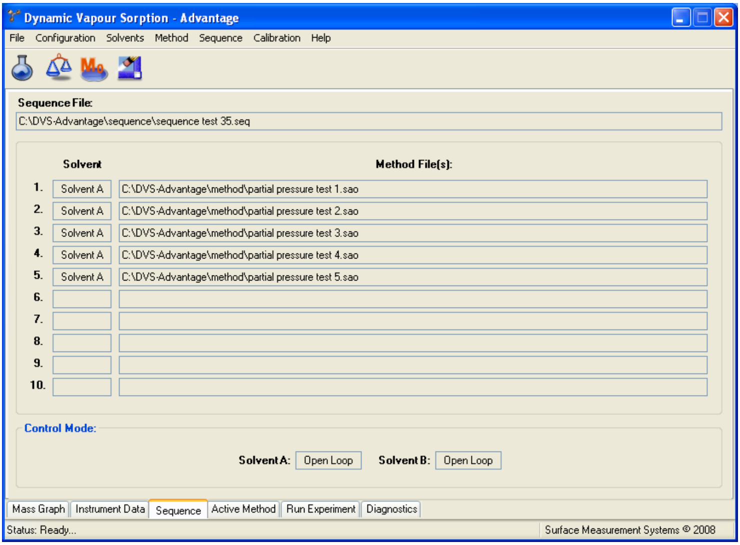


Figure 9.8: The **Select Sequence File** window

By default, the software will immediately navigate to the directory specified for Sequence files in the Preferences option (Section 9.2.1.2) – another directory may be selected in the window above if desired.

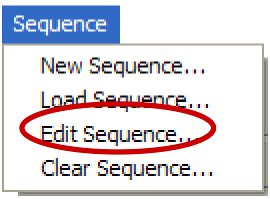
Upon selecting a Sequence file and clicking **Open**, the Sequence will appear in the DVS Advantage software Sequence panel (Section 10.5):



The Sequence is now loaded.

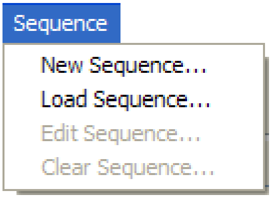
Note: The Sequence panel does not need to be cleared in order to load a Sequence. If a Sequence is already loaded, the Load Sequence option can be used to overwrite it with another Sequence. The overwritten Sequence file is not lost from the computer hard drive.

9.6.1.3 Edit Sequence



This option allows the user to edit an existing Sequence file which has been loaded (see Section 9.6.1.2 above).

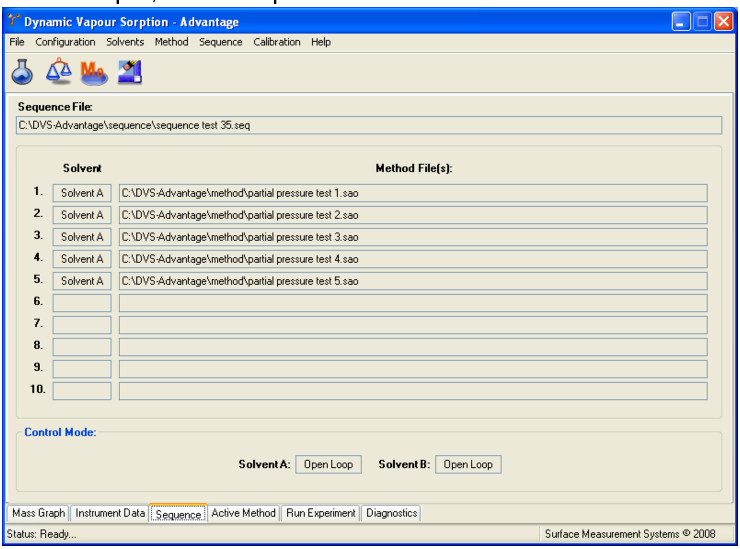
Note: If there is no Sequence loaded in the DVS Advantage Software, then the Edit Sequence option is disabled:



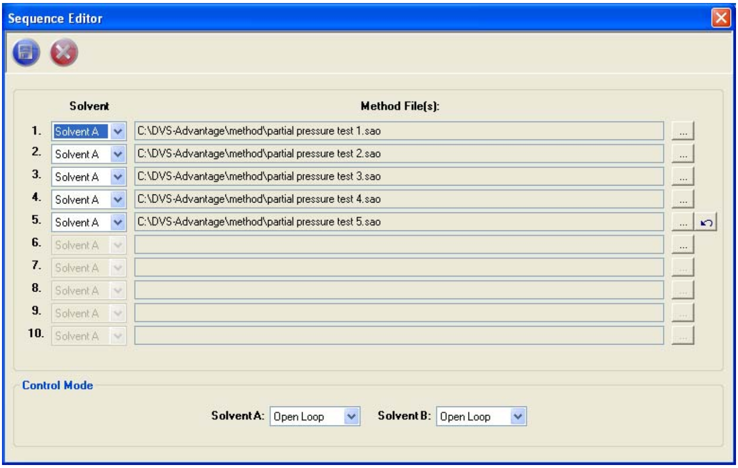
The Edit Sequence function applies only to a Sequence file that has been loaded.

Clicking the **Edit Sequence** option calls up the Sequence Editor window for the Sequence file that is currently loaded in the software.

For example, if the Sequence shown below is loaded:



Selecting the Edit Sequence option will display the loaded Sequence settings in the Sequence Editor panel:



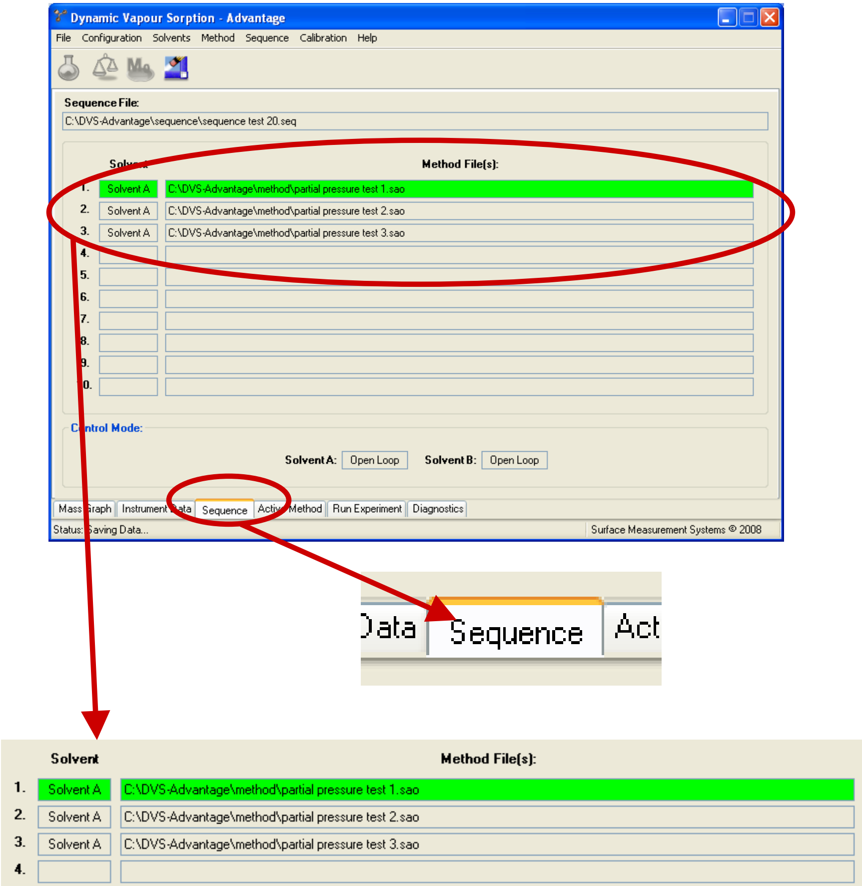
The various functions as outlined in Section 9.6.1.1 may then be used to edit the Sequence.

Upon completing the changes to the Sequence, the user may close the Sequence Editor, in which case all changes to the Sequence will be lost, or they may Save the edited Sequence under a new Sequence filename (refer Section 9.6.1.1 for details on closing and saving Sequence files).

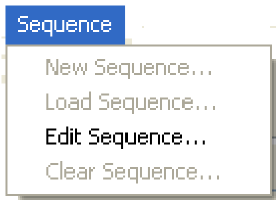
Upon **Saving** the edited Sequence, the DVS Advantage software interface will be returned, showing the newly edited Sequence in the Sequence panel.

Editing a running Sequence (and Method)

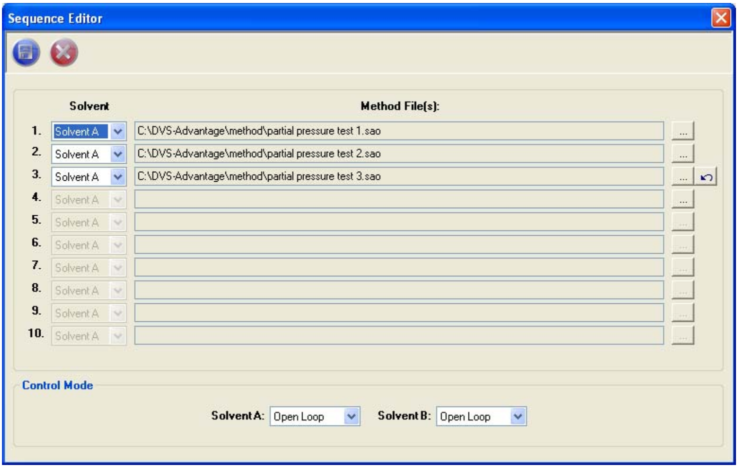
A Sequence (or Method) may be edited while it is running and data being saved. When a Sequence is running, the Sequence panel appears as shown below, displaying the active Method of the Sequence in green (see Section 10.5 for a more detailed explanation of the Sequence Panel):



With a Sequence running, the Sequence menu appears with all options deactivated except for **Edit Sequence….**



Clicking **Edit Sequence** calls up the **Sequence Editor** window. The running Sequence will be displayed (however the active Method will not be highlighted green):

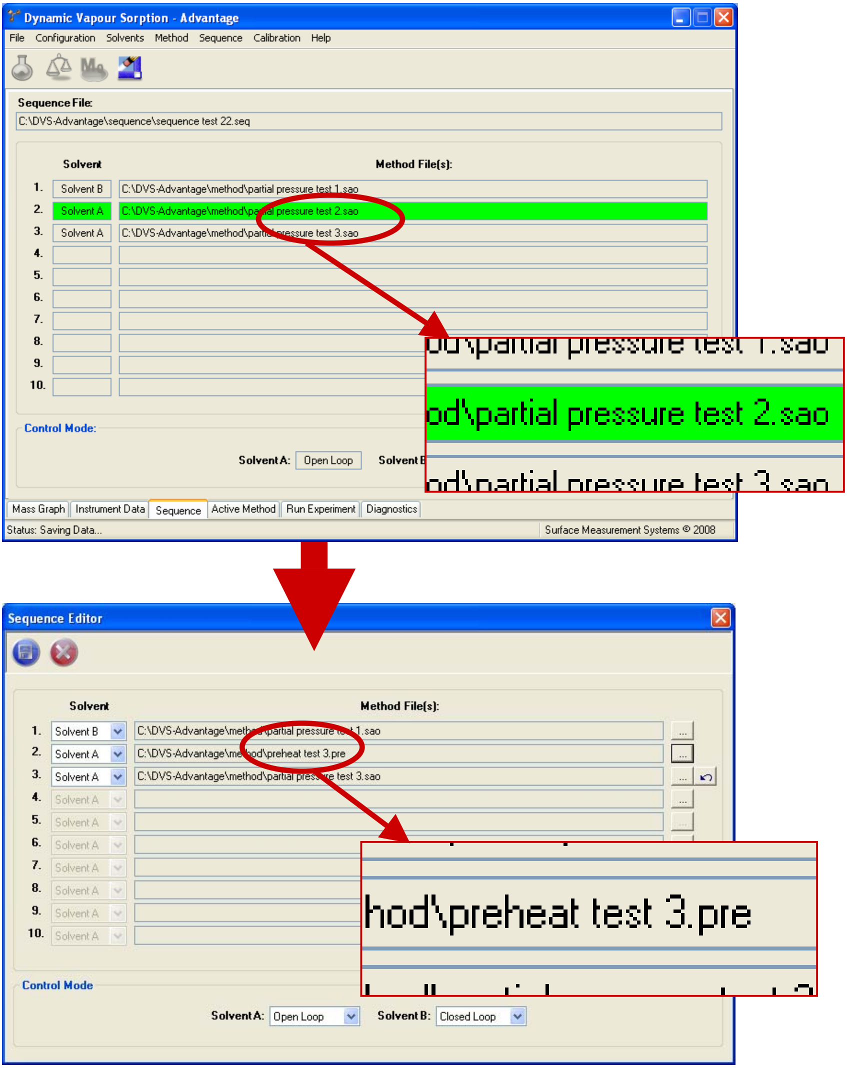


All of the Editing options function as described above in Section 9.6.1.1.

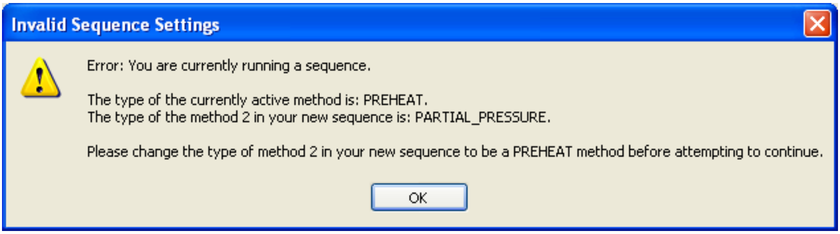
Editing the running Sequence

To edit a running Sequence, some points need to be remembered:

1. <u>Replace like-for-like Methods</u>
   The *active* Method in a running Sequence can only be replaced by a Method of the same type ie; a partial pressure Method must be replaced by a partial pressure Method, preheat by preheat, clean.dcc by clean.dcc:



If - as shown above - a different Method type is used to replace the active Method in the running Sequence, a message of the form shown below will appear when saving the new Sequence:



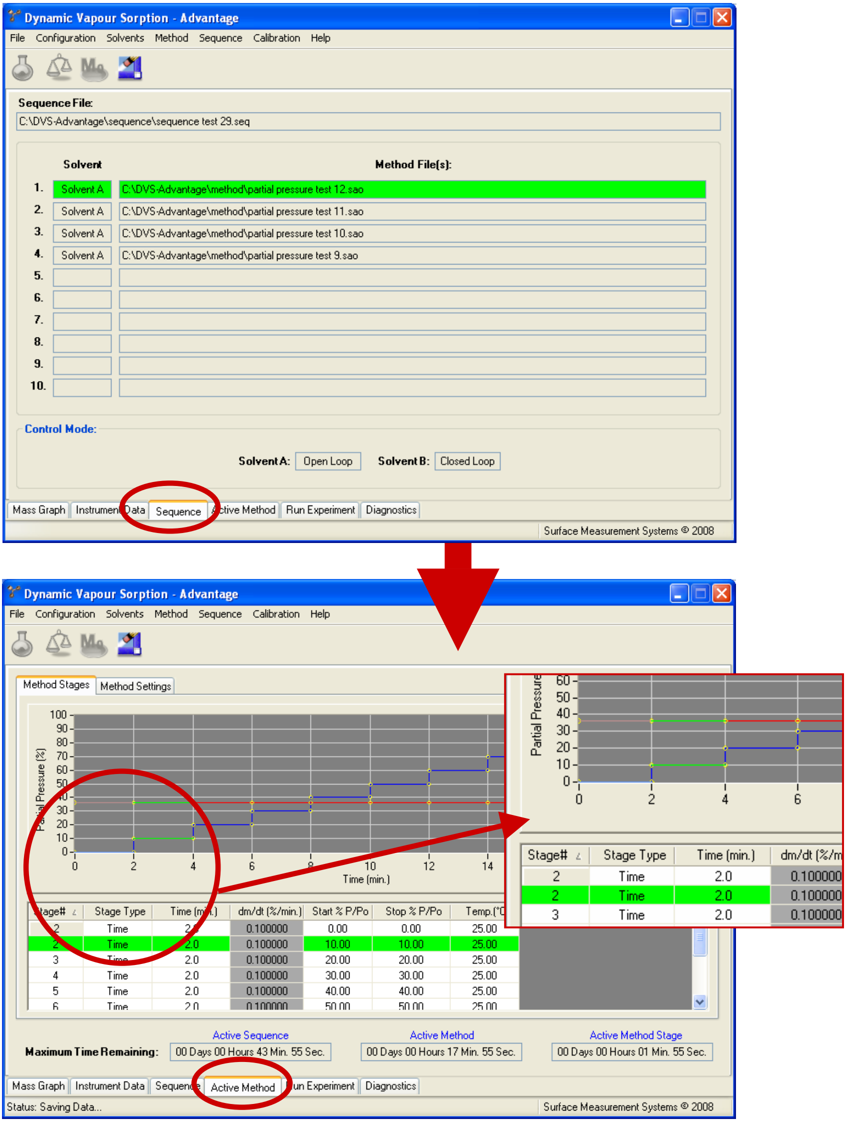
Clicking **OK** will return the Sequence Editor.

If a Method is not active, then it may be replaced by a Method of any type.

2. Number of Stages

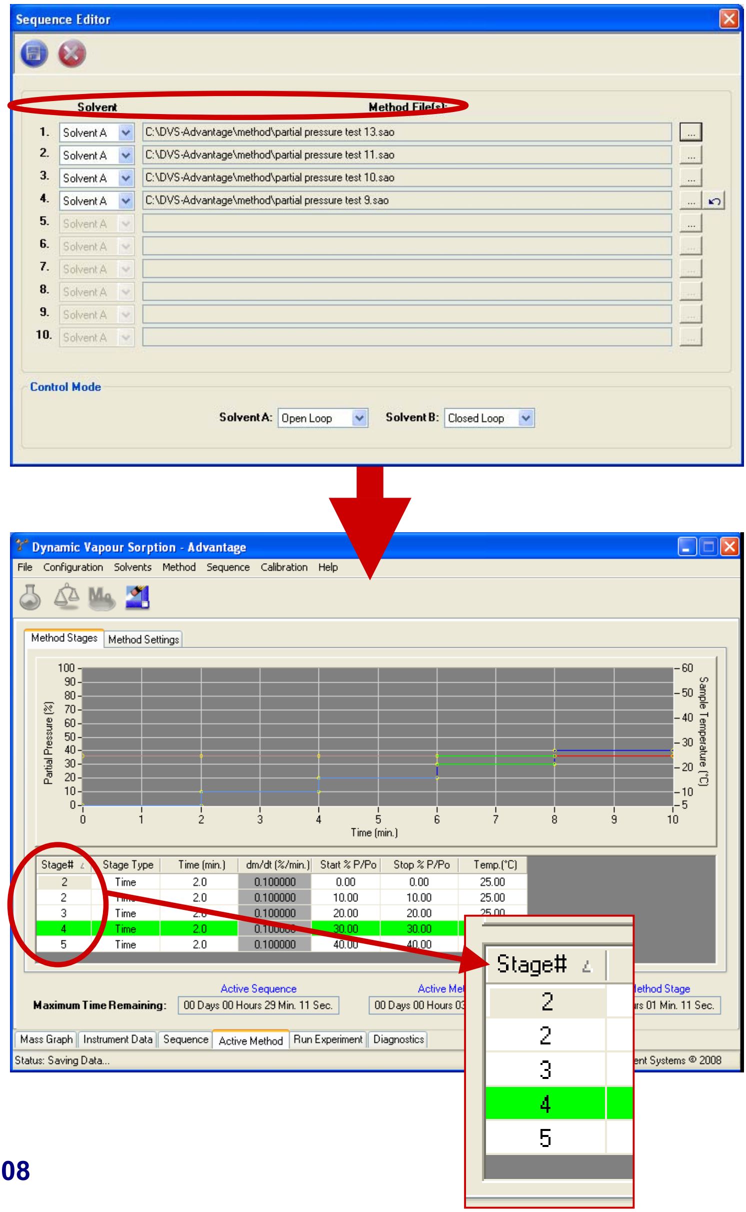
When replacing the active Method, the new Method must contain at least as many Stages as the number of the active Stage in the original Method.

For example, if the active Method of a running Sequence contained ten Stages, and Stage 2 was active when the Sequence was edited:



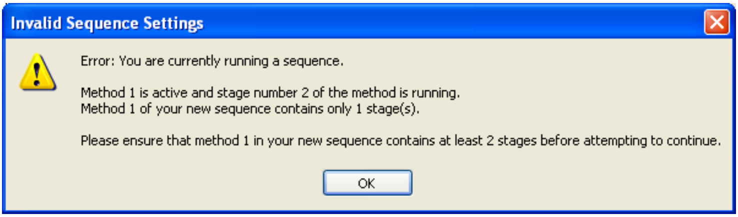
Dynamic Vapour Sorption - Advantage
File Configuration Solvents Method Sequence Calibration Help
Sequence File:
C:\DVS-Advantage\sequence\sequence test 29.seq
Solvent
Method File(s):
1. Solvent A C:\DVS-Advantage\method\partial pressure test 12.sao
2. Solvent A C:\DVS-Advantage\method\partial pressure test 11.sao
3. Solvent A C:\DVS-Advantage\method\partial pressure test 10.sao
4. Solvent A C:\DVS-Advantage\method\partial pressure test 9.sao
5.
6.
7.
8.
9.
10.
Control Mode:
Solvent A: Open Loop
Solvent B: Closed Loop
Mass Graph Instrument Data Sequence Active Method Run Experiment Diagnostics
Surface Measurement Systems © 2008
Dynamic Vapour Sorption - Advantage
File Configuration Solvents Method Sequence Calibration Help
Method Stages Method Settings
Partial Pressure (%)
Time (min.)
Stage# Stage Type Time (min.) dm/dt (%/min.) Start % P/Po Stop % P/Po Temp.(°C)
2 Time 2.0 0.100000 0.00 0.00 25.00
2 Time 2.0 0.100000 10.00 10.00 25.00
3 Time 2.0 0.100000 20.00 20.00 25.00
4 Time 2.0 0.100000 30.00 30.00 25.00
5 Time 2.0 0.100000 40.00 40.00 25.00
6 Time 2.0 0.100000 50.00 50.00 25.00
Partial Pressure
Stage# Stage Type Time (min.) dm/dt (%/m
2 Time 2.0 0.100000
2 Time 2.0 0.100000
3 Time 2.0 0.100000
Active Sequence
Active Method
Active Method Stage
Maximum Time Remaining: 00 Days 00 Hours 43 Min. 55 Sec. 00 Days 00 Hours 17 Min. 55 Sec. 00 Days 00 Hours 01 Min. 55 Sec.
Mass Graph Instrument Data Sequence Active Method Run Experiment Diagnostics
Status: Saving Data...
Surface Measurement Systems © 2008

…..then the newly edited Method would have to contain *at least* 2 stages:



This is because the newly installed Method in the new Sequence picks up from where the original Method was interrupted for editing (in this example, on the second stage) – *the new Method does not commence from the beginning.*

If the new Method contains fewer Stages than the number of the active Stage, then a message of the form shown below will appear when saving the new Sequence:



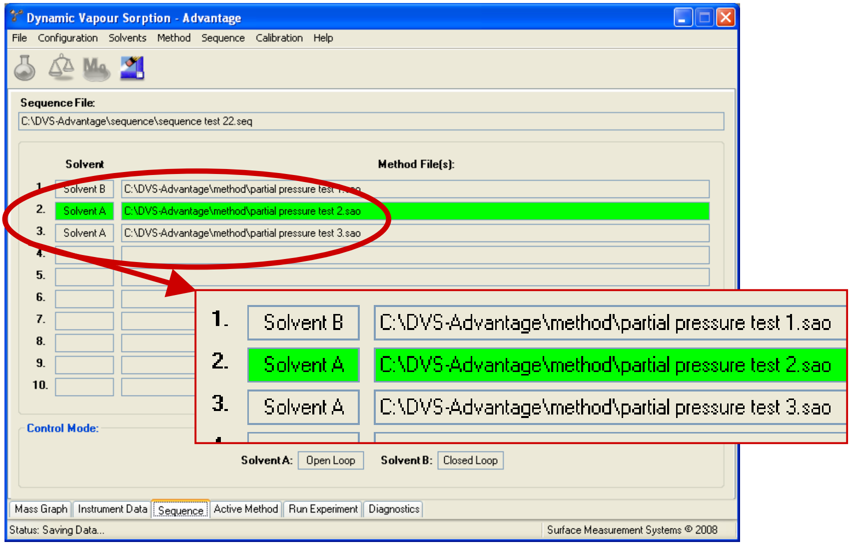
Clicking **OK** will return the Sequence Editor.

Note: The conditions described above apply both to Preheat and Partial Pressure Methods.

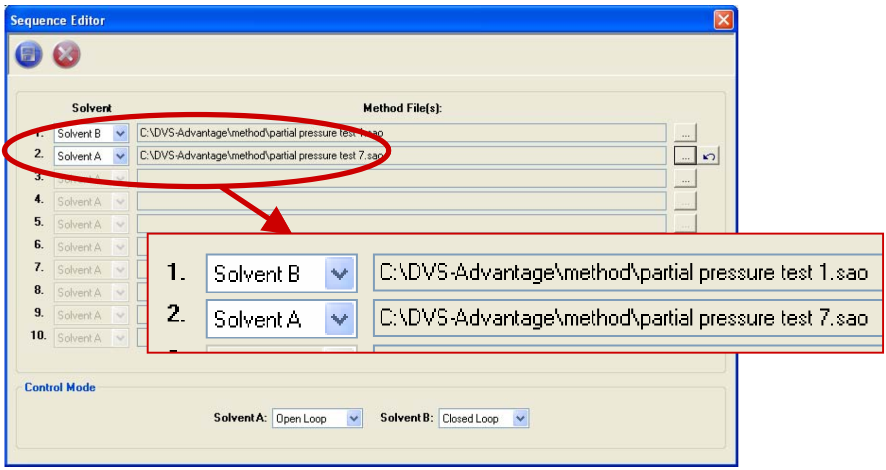
3. Number of Methods

The newly edited Sequence must contain at least as many Methods as the number of the active Method in the original Sequence.

For example, if the original Sequence contained three Methods, and Method 2 was active when the Sequence was edited:

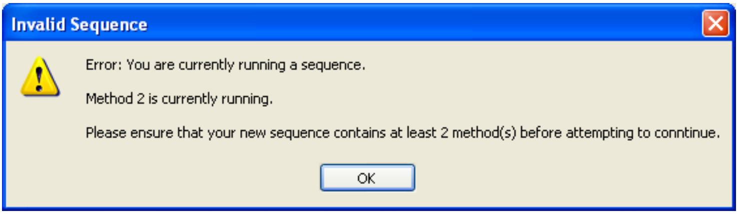


…..then the newly edited Sequence would have to contain *at least* 2 Methods:



This is because the newly edited Sequence picks up from where the original Sequence was interrupted for editing (in this example, on the second Method file) – *the new Sequence does not commence from the beginning.*

If the new Sequence contains fewer Methods than the number of the active Method, then a message of the form shown below will appear when saving the new Sequence:

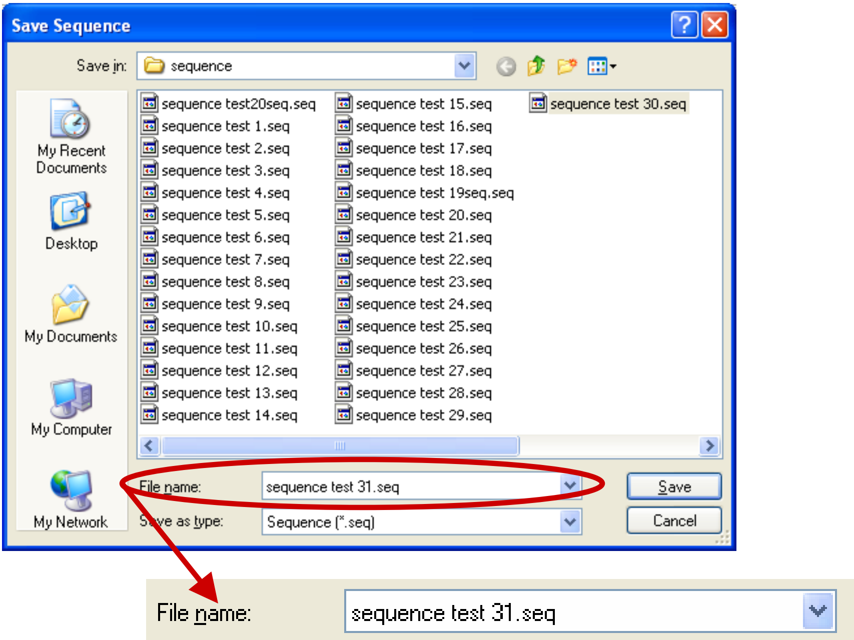


Clicking **OK** will return the Sequence Editor.

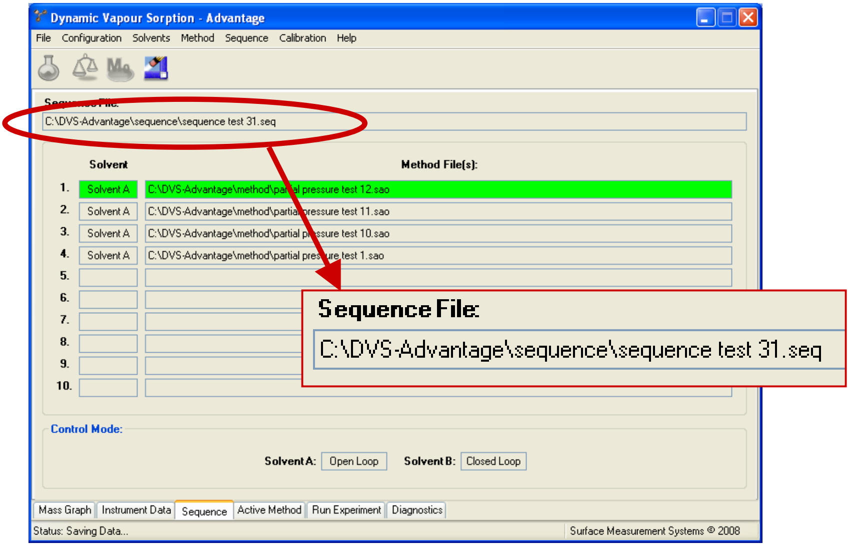
4. <u>Solvents and Control Modes</u>
   The Solvents and their Control Modes may be edited freely on a running Sequence.

5. <u>New Sequence automatically loaded and run</u>

Once the running Sequence has been edited according to the requirements above, clicking the **Save Sequence** button  calls up the **Save Sequence** window, which allows the user to name the newly edited Sequence:



Upon naming the new Sequence and clicking **Save**, the DVS Advantage Control software Sequence panel is returned, displaying the newly edited Sequence, which is now running in place of the original Sequence:

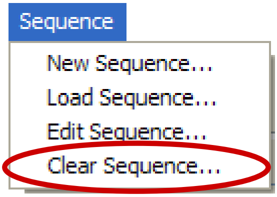


Note: The *active* (ie; running) Method of a Sequence may also be edited. While the Method is running, the **Edit Method** option may be used as described in Sections 9.5.1.2 and 9.5.2.8. This applies both to Preheat and Partial Pressure Methods.

The newly edited Method will need to be saved under a new name – but note that it is not automatically loaded into the running Sequence.

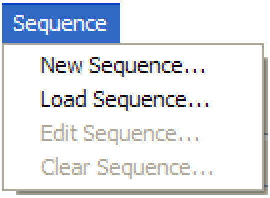
To do this, the running Sequence will need to be edited to include the new Method as described above.

9.6.1.4 Clear Sequence



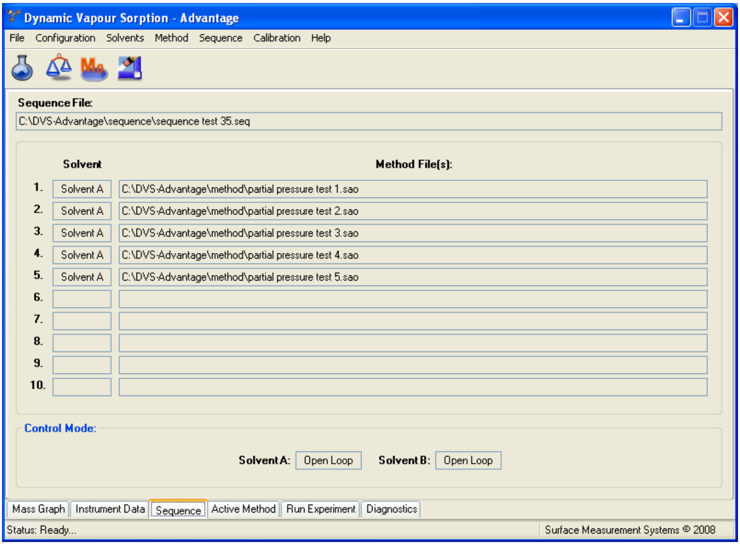
This option clears any Sequence that is loaded in the DVS Advantage Software.

Note: If there is no Sequence loaded in the DVS Advantage Software, then the Clear Sequence option is disabled:

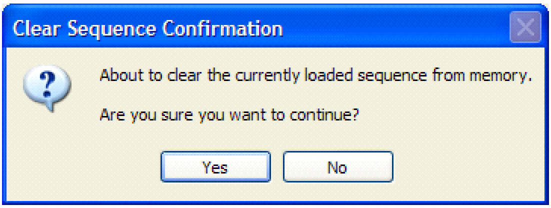


The Clear Sequence function applies only to a Sequence file that has been loaded.

For example, if the following Sequence were loaded:



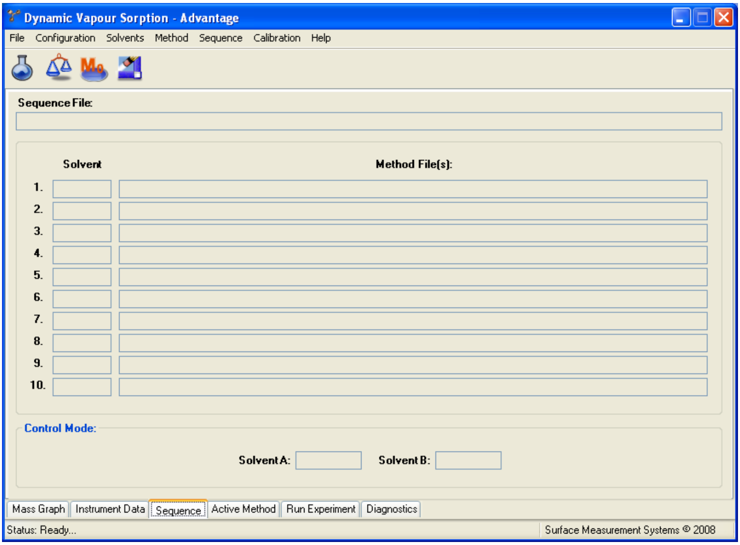
…then clicking the **Clear Sequence** option calls up the following window:



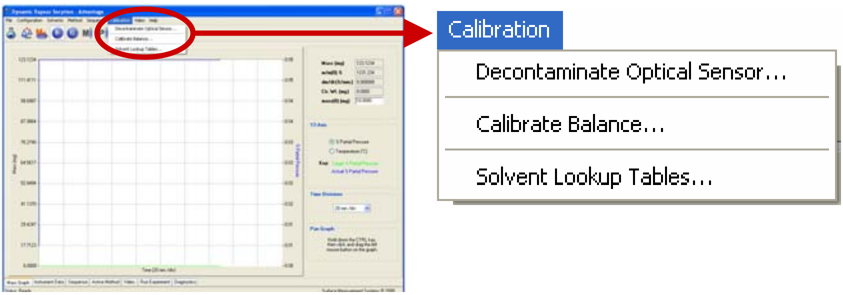
Note: The Sequence file being cleared is not cleared from the computer's hard drive. The file may still later be accessed and loaded for use in the DVS Advantage software.

When a Sequence file is cleared, it is merely cleared from the Sequence panel.

Upon clicking **Yes**, the DVS Advantage software interface is returned, with the Sequence panel cleared:

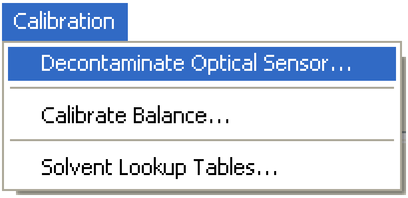


9.7 Calibration



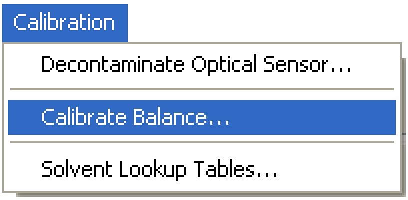
Note: The Calibration menu is disabled whenever data is being saved. Data saving must be stopped before the menu options may be used.

9.7.1.1 Decontaminate Optical Sensor

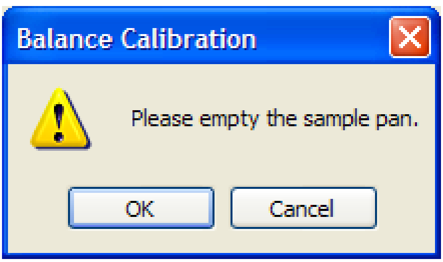


This menu item sends the Optical Sensor into Dynamic Contamination Correction mode (DCC). Please refer to Appendix E (Section 15) for more details on the Optical Sensor Dynamic Contamination Correction.

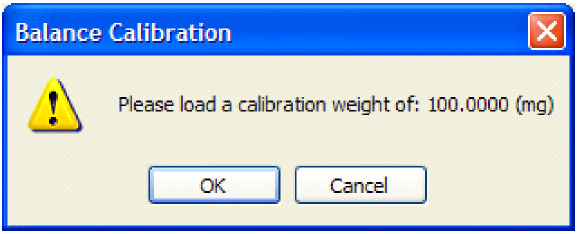
9.7.1.2 Calibrate Balance



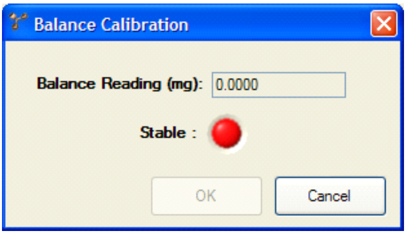
Upon clicking the **Calibrate Balance** option, the following window appears:



- At this point make sure the sample pan is empty and clean and wait for the balance to stabilise.
- Click **OK**. The following prompt will then appear:



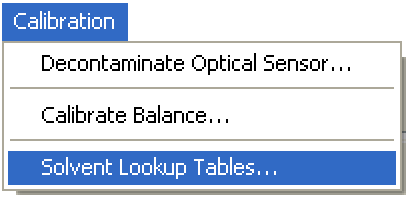
- Carefully place a certified calibration weight in the sample pan.
- Click **OK**. The following panel will then appear:



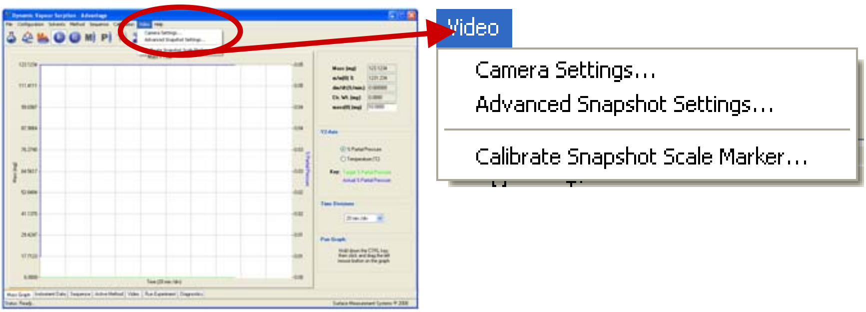
- The panel shown above then displays the current measured mass. The light below this value will be red, and will remain so until the balance reading becomes stable (this typically takes 5 minutes). When the mass reading is stable the light will turn green and the user can then click **OK** to register the system's new calibration constant.

9.7.1.3 Solvent Lookup Tables

Please refer to Appendix D (Section 14) for information on the operation of the Solvent Lookup Tables. Only a qualified SMS service engineer should edit these values.



9.8 Video



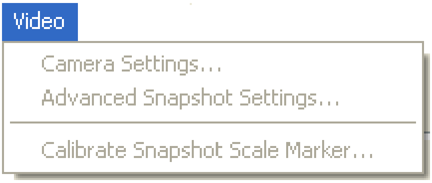
The DVS system's video allows the user to take manual or automated pictures of the sample; sample snapshots may be taken manually when not saving data, or at a pre-defined frequency when saving data or at the end of each Method Stage when running a Method.

Please also refer to Sections 9.2.1.3 and 10.7 for more information on the Video function.

The **Video** menu options allow the user to adjust various parameters of the system's **Dino-Lite Digital Microscope** video camera and to set various image parameters.

Note: The **Video** menu option will only appear if a DVS Advantage system with Video has been purchased.

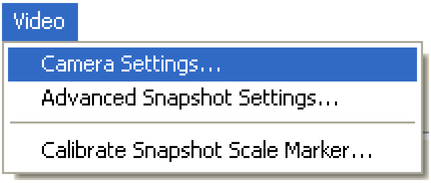
In addition, the Video menu option becomes inactive whenever data is being saved (ie; when running a Method or when simply saving data). Any desired parameters must therefore be set before data saving commences:



Additional Note: The **Dino-Lite Digital Microscope** video camera connects to the DVS control computer via a USB port. If the camera is not correctly connected at the time the control software is opened, the following message will appear:

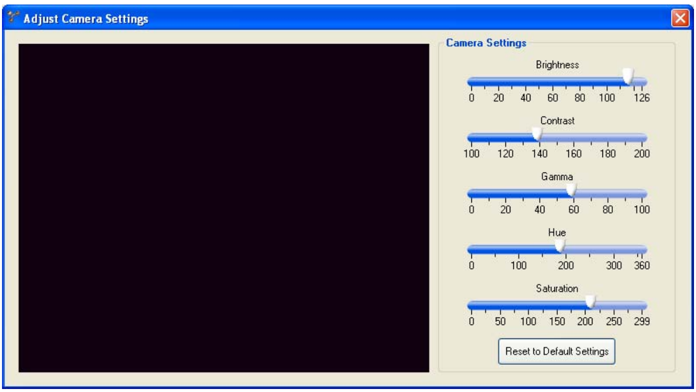
Similarly, if any of the Video menu options are clicked but the camera has become disconnected, the message below will appear:

9.8.1 Camera Settings



This option allows the user to adjust the picture from the video microscope to ensure optimum image quality of the sample snapshots.

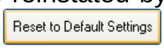
Clicking this option calls up the **Adjust Camera Settings** window as shown:



Note: The screen grab above shows no picture, but a live video picture will appear in the black box shown above when actually using the software.

This window provides a live video feed from the camera, and allows the user to adjust the camera to the settings required for the upcoming snapshots of the sample.

Once the desired **Brightness**, **Contrast**, **Gamma**, **Hue** and **Saturation** have been found, the settings may be saved by clicking the close button . These settings will be preserved through software re-starts.

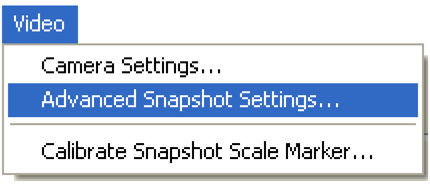
The factory default settings may be reinstated by clicking the **Reset to Default Settings** button 

, which are then saved by closing the window as above.

Note: The **Camera Settings** option also allows the user to observe a live video image of the sample once it is enclosed in the sample chamber. However, the Video function does not allow any live video footage to be recorded.

It is useful to know, however, that if data is being saved either by running a Method or by simply saving data, the **Camera Settings** option is disabled (as is the whole Video menu).

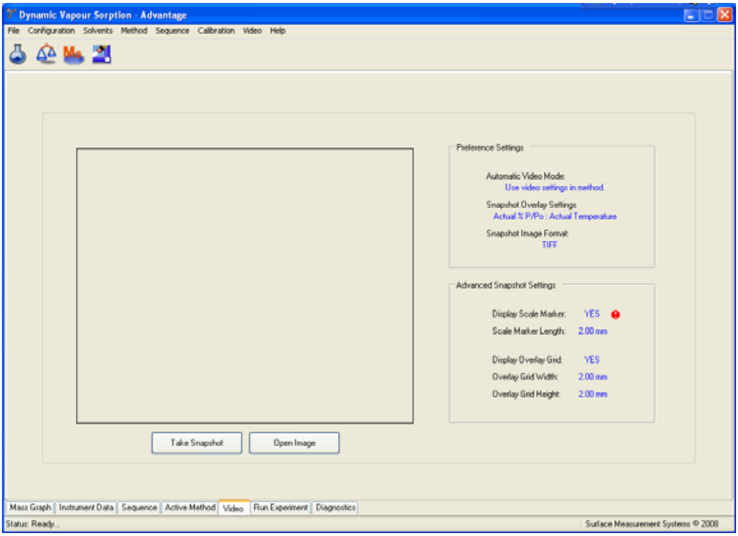
9.8.2 Advanced Snapshot Settings



This option allows the user to set parameters relating to the snapshots taken of the sample.

When the DVS

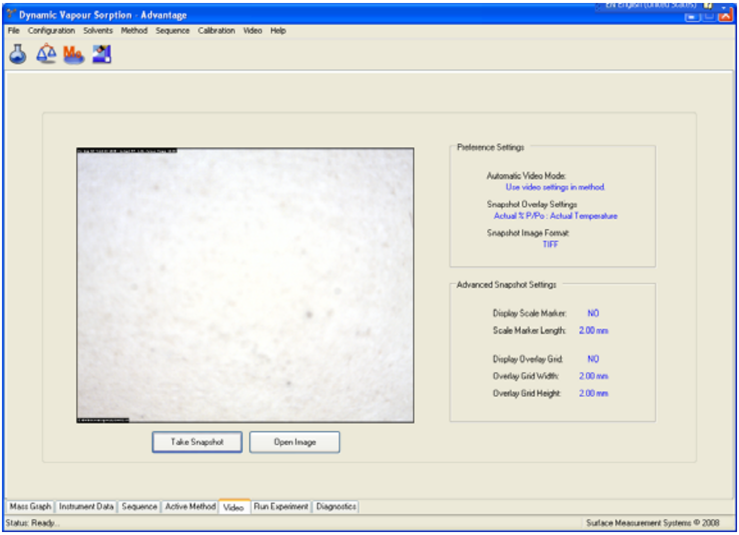
Advantage Control software is started up, the Video tab will display no snapshot (Refer to Section 10.7 for more details on the Video tab):



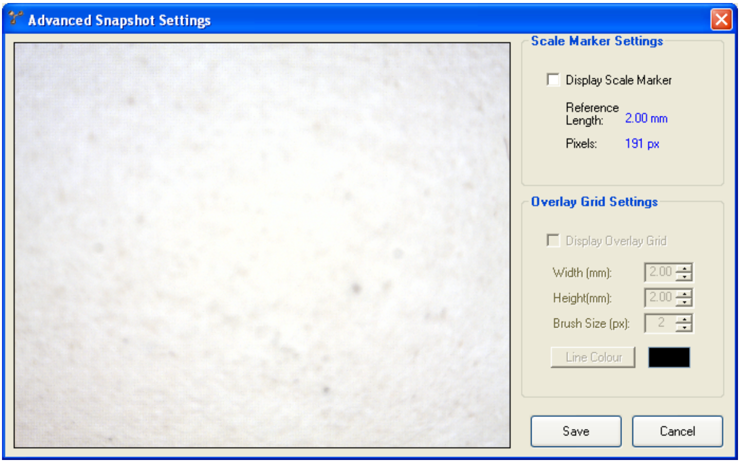
In this case, clicking the **Advanced Snapshot Settings** option will call up the following window……

…..which simply indicates that a snapshot needs to be taken before any settings may be adjusted. Clicking **OK** will return the Control software main window.

After a snapshot of the sample is taken (refer to Section 10.7.1), the image will appear in the **Video** tab as shown:

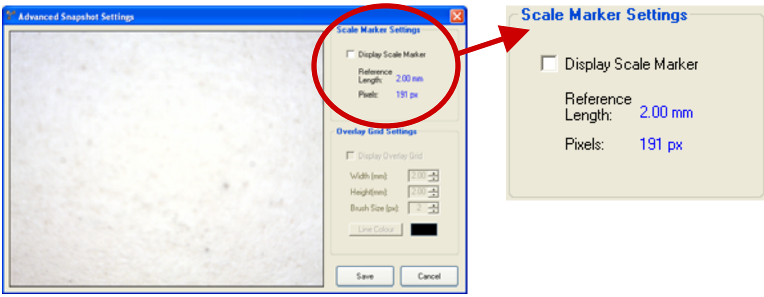


Clicking the Advanced Snapshot Settings option then calls up the following window:



As shown above, the window displays the current snapshot along with boxes which allow the adjustment of various settings.

9.8.2.1 Scale Marker Settings

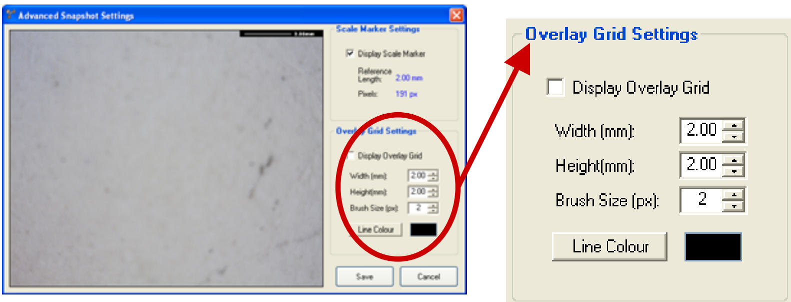


A line marking the scale of the snapshot may be displayed. Ticking the **Display Scale Marker** box displays the marker in the top, right-hand corner of the picture as shown:

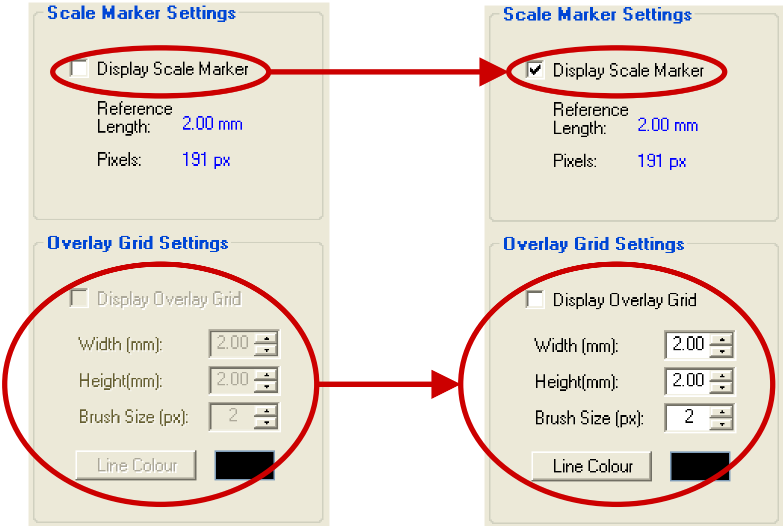
As shown above, the **Scale Marker Settings** box also displays the Reference length of the scale, and the number of pixels to which this length corresponds.

The Reference length may be set in the **Calibrate Snapshot Scale Marker** option (explained below in Section 9.8.3).

9.8.2.2 Overlay Grid Settings

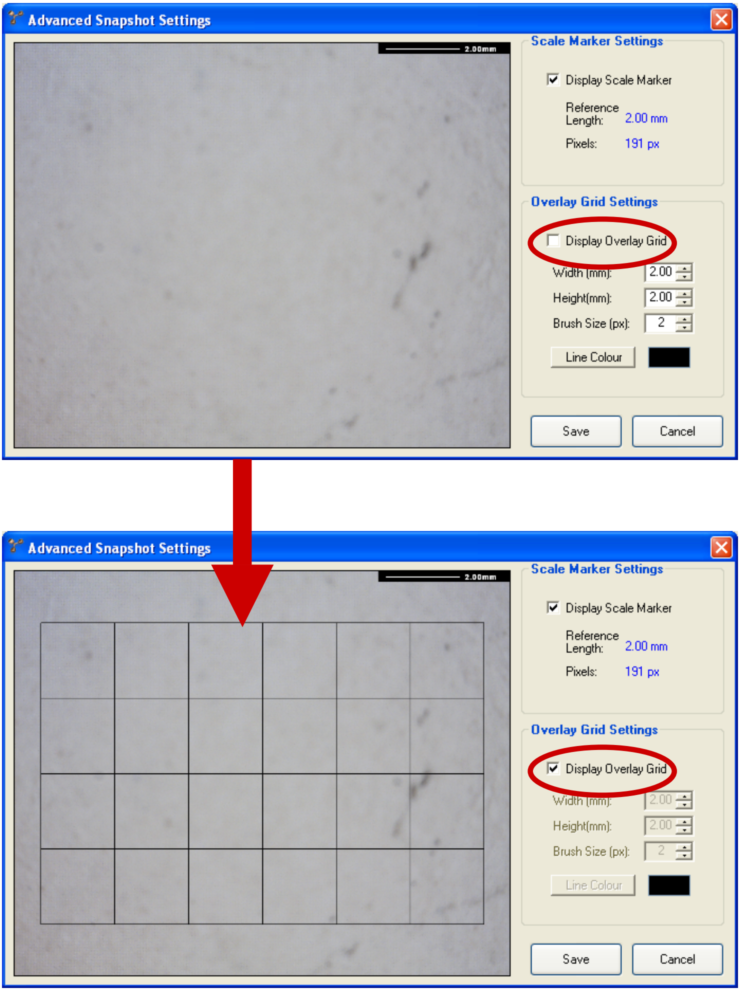


The **Overlay Grid Settings** box is activated only when the **Display Scale Marker** box is ticked:



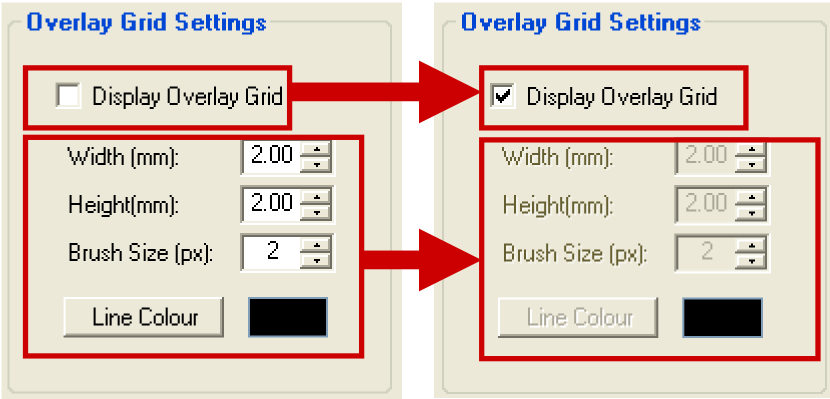
Once activated, the **Overlay Grid Settings** box allows the user to superimpose a grid over the snapshot images. This grid assists dimensional analysis of the sample snapshots. The **Overlay Grid Settings** box also allows for the grid to be edited.

Clicking the **Display Overlay Grid** box superimposes the grid on the latest snapshot as shown:



The settings that are in place (Width, Height, Brush Size and colour) when the **Display Overlay Grid** box is ticked will determine the features of the grid.

Once the **Display Overlay Grid** box has been ticked, however, these settings become inactive:



To edit the Overlay Grid, the **Display Overlay Grid** box must therefore be un-checked. The editable parameters are as follows:

Width (mm): Defines the horizontal distance between the vertical lines of the grid. The available settings run from 2mm to 10mm.

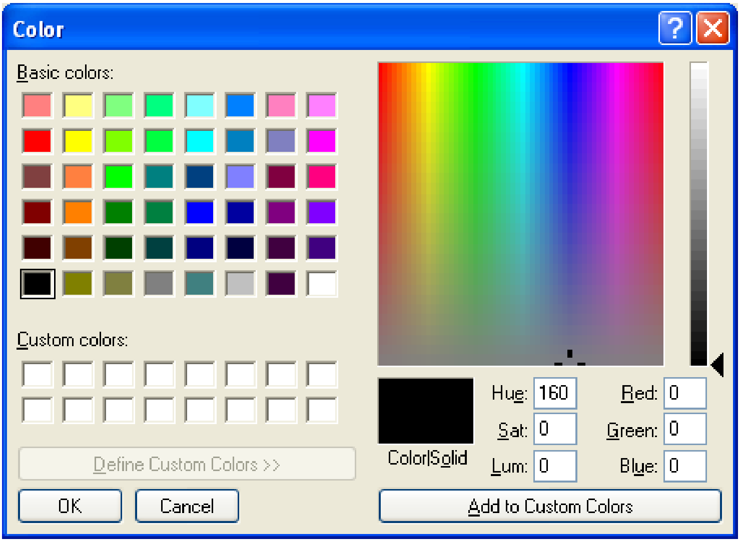
Height (mm): Defines the distance between the horizontal lines of the grid. The available settings run from 2mm to 10mm.

Brush Size (px): Defines the thickness of the grid lines in pixels. The available settings run from 1mm to 10mm.

The desired settings may be entered directly or by using the arrows at the right of each data entry box.

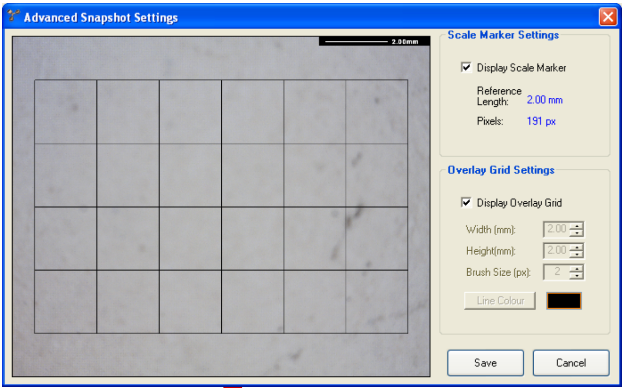
Line Colour: Clicking the **Line Colour** button calls up the following window, from which the desired grid colour may be selected:

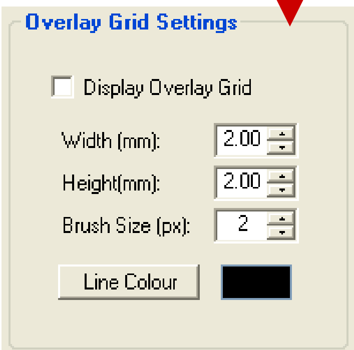
Clicking the **Define Custom Colours** button calls up the window shown below, which allows custom colours to be selected and saved:

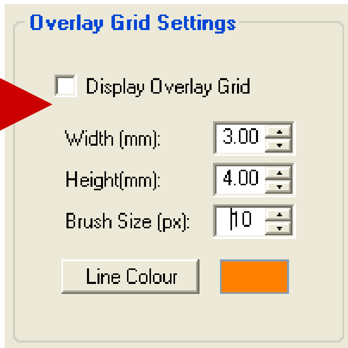


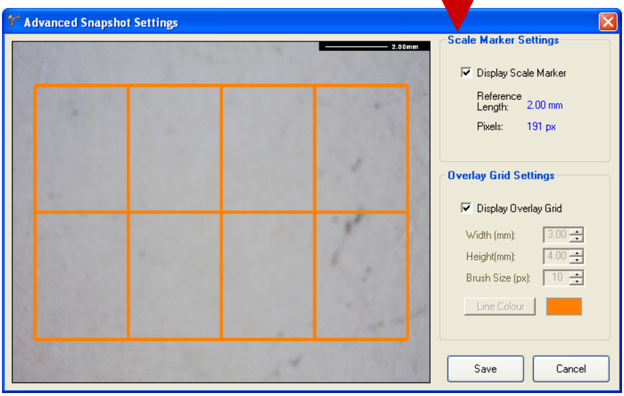
Clicking **OK** on the **Colour** window then returns the **Overlay Grid Settings** box with the newly selected colour in place.

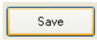
After setting the above parameters and clicking the **Display Overlay Grid** box once more, the grid will reappear in its newly edited form:


Advanced Snapshot Settings
2.00mm
Scale Marker Settings
Display Scale Marker
Reference Length: 2.00 mm
Pixels: 191 px
Overlay Grid Settings
Display Overlay Grid
Width (mm): 2.00
Height(mm): 2.00
Brush Size (px): 2
Line Colour
Save
Cancel


Overlay Grid Settings
Display Overlay Grid
Width (mm): 2.00
Height(mm): 2.00
Brush Size (px): 2
Line Colour


Overlay Grid Settings
Display Overlay Grid
Width (mm): 3.00
Height(mm): 4.00
Brush Size (px): 10
Line Colour


Advanced Snapshot Settings
2.00mm
Scale Marker Settings
Display Scale Marker
Reference Length: 2.00 mm
Pixels: 191 px
Overlay Grid Settings
Display Overlay Grid
Width (mm): 3.00
Height(mm): 4.00
Brush Size (px): 10
Line Colour
Save
Cancel

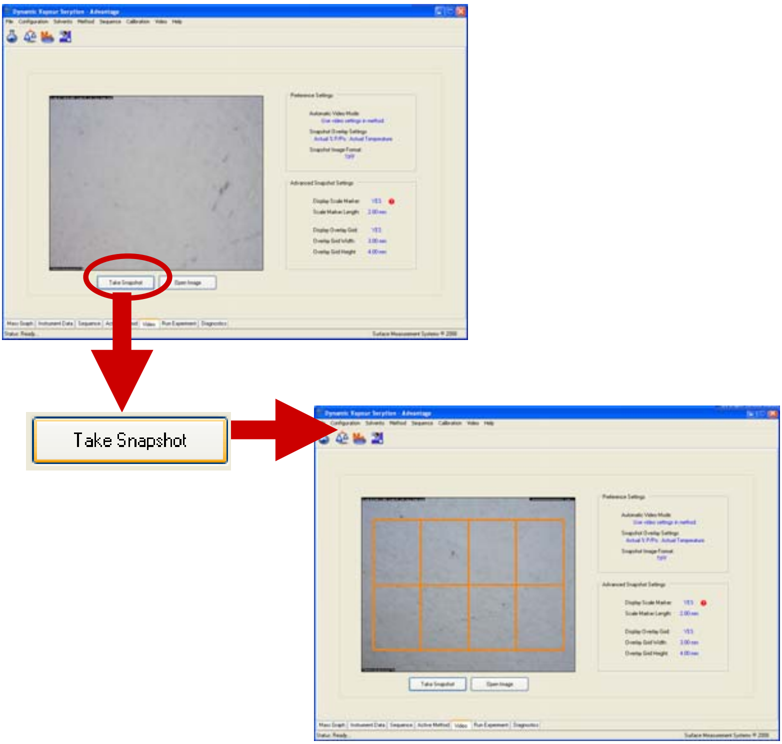
Clicking the **Save** button 

then saves all of the settings recorded in the **Advanced Snapshot Settings** window, and returns the DVS Advantage Control software main interface.

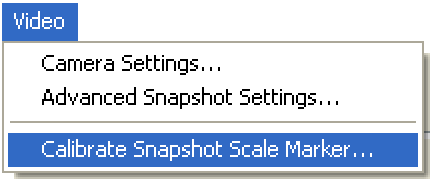
These newly saved settings will be applied to all sample snapshots taken subsequently, and will be retained through software re-starts.

For example, after saving the settings above and going to the **Video** tab, clicking the **Take Snapshot** button 

will result in the new image being recorded as shown (also refer to Section 10.7 for more details on taking snapshots and the Video tab):

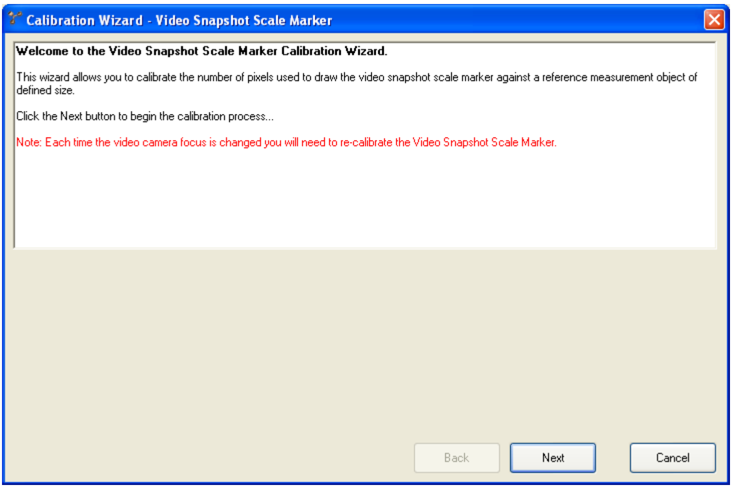


9.8.3 Calibrate Snapshot Scale Marker



The Scale Marker will need to be calibrated if the distance between the camera and sample changes, or if the camera's focus is altered. This will ensure that the distance represented by the Scale Marker on the sample snapshots is physically accurate.

Clicking the **Calibrate Snapshot Scale Marker** option calls up the following wizard which guides the user through the calibration process:



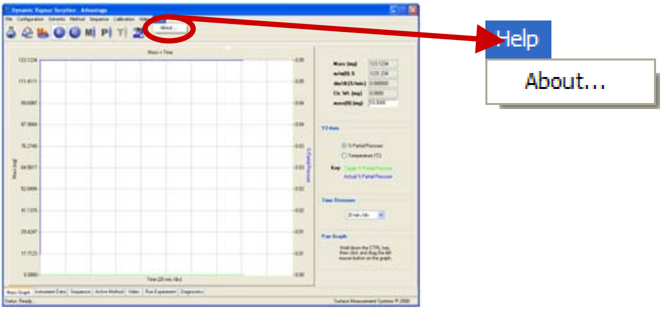
The wizard takes the user through a step-by-step process of taking a snapshot of a reference measurement object (eg; a piece of paper with 1mm markings), and drawing a line on this snapshot which defines a particular length.

The wizard is self-explanatory and as such it is not replicated in this manual.

However, it should be noted that the calibration must take place with the camera at the same focus as will be used in the experiment. In addition, the reference measurement object must be the same distance from the camera as the sample will be in the DVS Advantage system.

To that end, it is advised that the reference measurement object is placed in the sample pan and hung in the system in order to perform the calibration.

9.9 Help



9.9.1.1 About

Clicking this option calls up the following panel, which details the software's version number and other system details. A link to online help is also provided.

10 Software panels

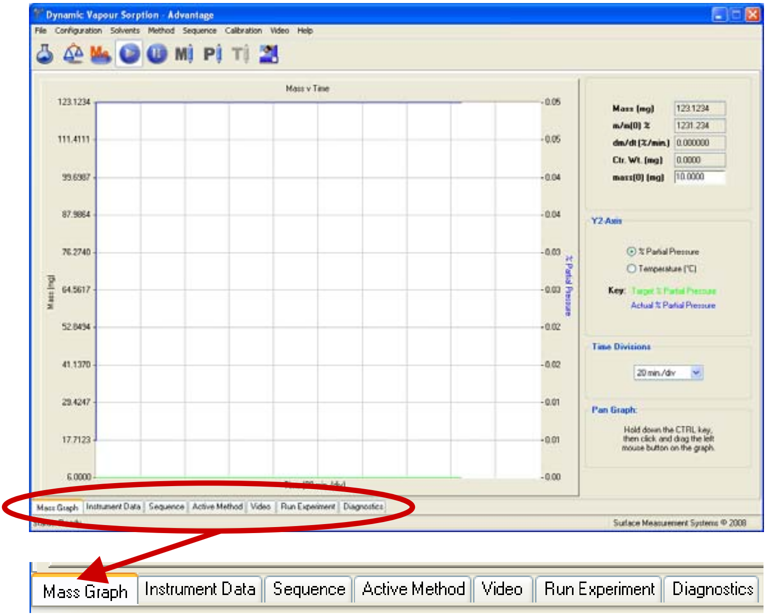
10.1 Introduction

The DVS Advantage Control software is composed of six panels, each of which serves a specific purpose relevant to setting up, running and monitoring an experiment.

The panels are entitled as follows:

- Mass Graph
- Instrument Data
- Sequence
- Active Method
- Run Experiment
- Diagnostics

These panels are accessed by clicking on tabs at the bottom of the control software interface, as shown below:



This section explains the functions and operations of each of these panels.

10.2 Common Functions

There are some buttons which appear across the top of all of the panels, each of which are explained below:

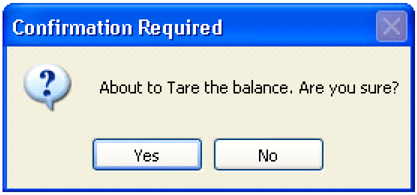
10.2.1 Select Solvents

This function is also accessible via the Solvents option on the software toolbar, which is outlined in Section 9.4.1.

10.2.2 Tare Balance

This function tares the balance ie; sets the current mass value to zero.

Clicking the button calls up the following window:



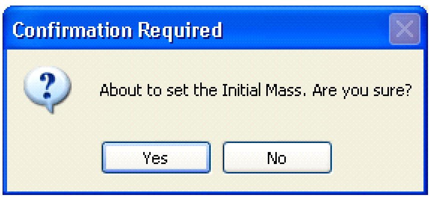
Clicking **No** will retain the current balance reading.

Clicking **Yes** will set the current mass reading to zero.

10.2.3 Set Initial Mass

This function defines the M(0) value for the experiment. Clicking this button sets the current mass reading as the M(0) (initial mass) value.

Upon clicking the button, the following window appears:

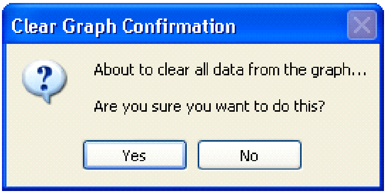


Upon clicking **Yes**, the newly defined Initial Mass will appear in the display on the Mass Graph panel (see Section 10.3 below).

10.2.4 Clear Graph

This function clears the graph on the Mass Graph panel of all data and re-commences the plot from the left hand side of the graph.

Clicking this button calls up the following window:

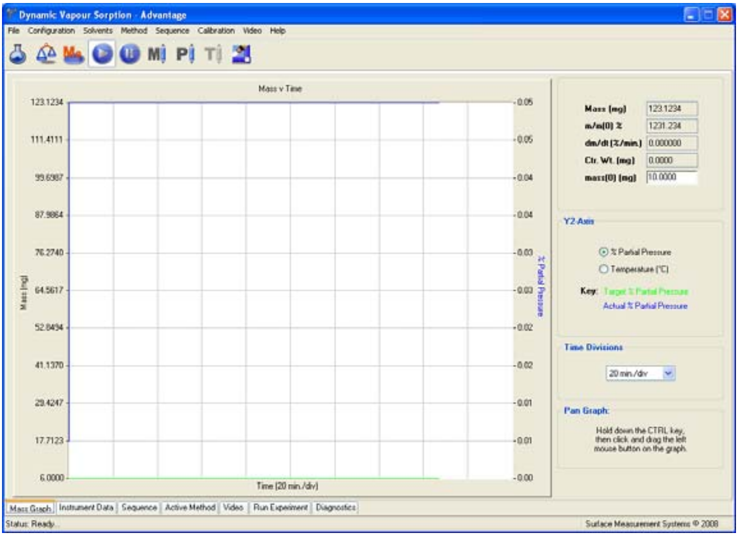


Upon clicking **Yes**, the Mass Graph is cleared of all displayed data (including partial pressure and temperature). The data will re-commence plotting from the left-hand side of the graph.

10.3 Mass Graph panel

The Mass Graph panel shown below charts the raw mass, partial pressure and temperature data measured by the instrument.

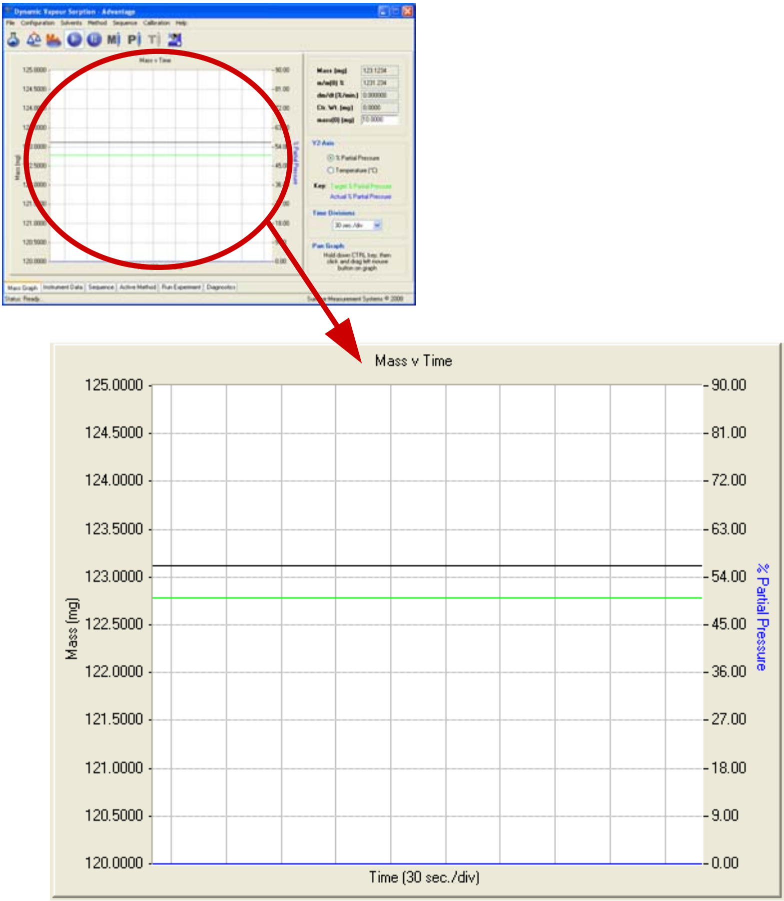
Also displayed on this panel are numerical mass data and controls for altering the format of the chart.



10.3.1 Mass vs Time graph

The **Mass vs Time** graph displays two parameters vs time. On the left-hand axis is permanently displayed the raw mass data (mg) measured by the system, while the right-hand axis displays either partial pressure (%) or temperature (°C).

At the top of the panel are located several buttons which control the manner in which the data is displayed:


Mass v Time
125.0000
124.5000
124.0000
123.5000
123.0000
122.5000
122.0000
121.5000
121.0000
120.5000
120.0000
Mass (mg)
90.00
81.00
72.00
63.00
54.00
45.00
36.00
27.00
18.00
9.00
0.00
% Partial Pressure
Time (30 sec./div)

10.3.1.1 Time Axis – Scrolling & Time Axis Paused

By default the **Time Axis – Scrolling** button is activated. This ensures that the time axis scrolls from right to left as the data is displayed. The plotted lines must first reach the right-hand end of the time axis before scrolling commences.

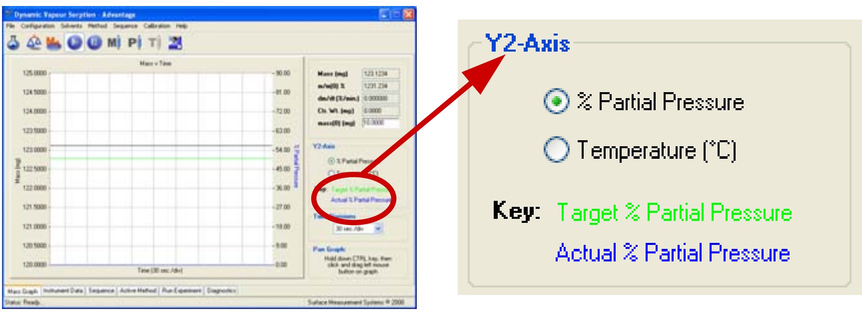
Clicking the **Time Axis – Paused** button freezes the time axis. The data lines continue to move left-to-right across the chart until they reach the right-hand end of the time axis – the data then continues to be plotted to the right-hand side, off screen.

10.3.1.2 Rescale Mass/Partial Pressure/Temperature Axis

These buttons allow the vertical axes displaying mass, partial pressure and temperature to be rescaled at any time before, during, or after an experiment.

These functions are also accessible via the Configuration toolbar option, which is detailed in Section 9.3.2.

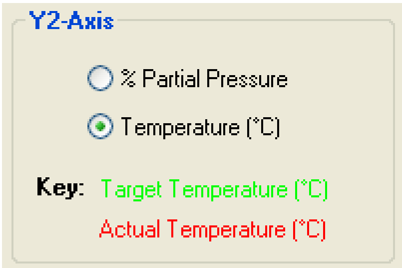
10.3.2 Y2 Axis



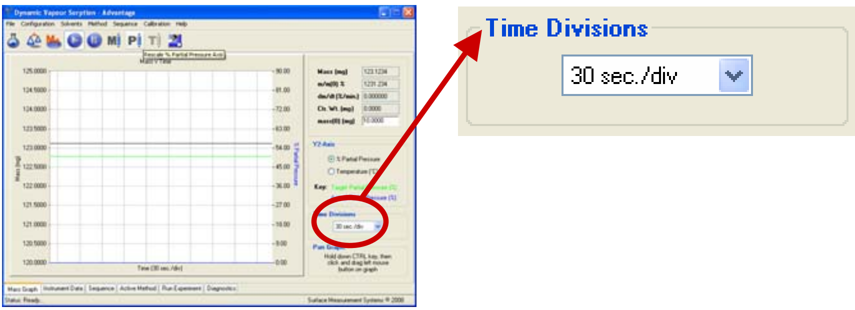
This box allows the user to switch between displaying **Partial Pressure (%)** and **Temperature (°C)** on the graph's secondary (right-hand) Y-axis.

Clicking the **% Partial Pressure** option will display the measured system partial pressure with a blue line. The target partial pressure is displayed by a green line, as indicated in the key shown above.

Clicking the **Temperature (°C)** option displays the actual and target sample temperature using red and green lines, respectively:

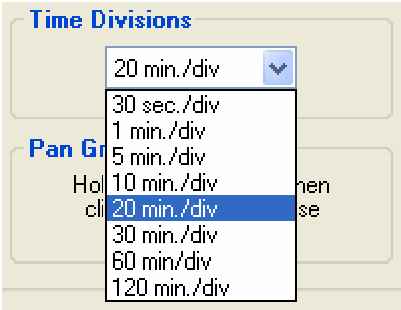


10.3.3 Time Divisions

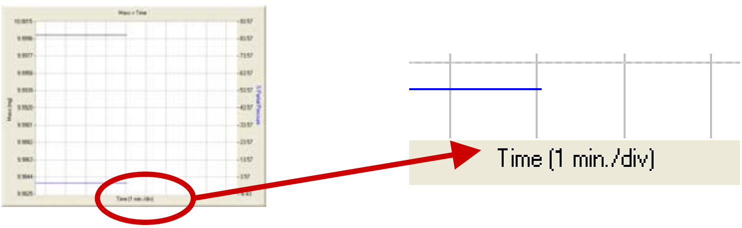


This box determines the scale of the graph's time axis.

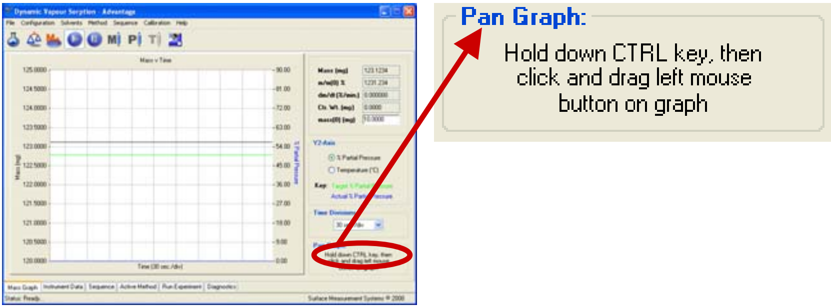
The default value is 20 minutes per division. Clicking in the edit box or the down arrow reveals a drop-down menu which displays the available options for rescaling the time axis:



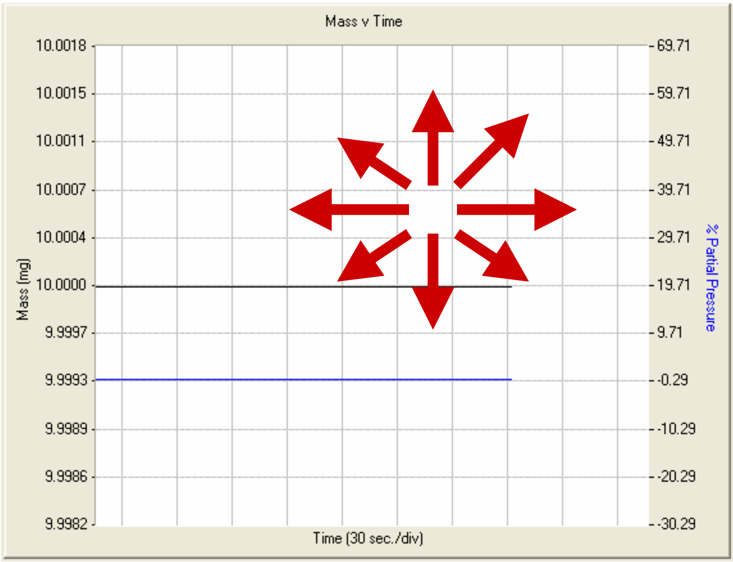
Upon selecting the desired scale by clicking on it, the Mass Graph will be displayed with its new time divisions. The selected time scale is also displayed on the horizontal axis label:



10.3.4 Pan Graph



The **Pan Graph** function is self-explanatory. Upon following the instructions displayed above, a hand icon will appear over the graph which allows the user to pan the graph in any direction:

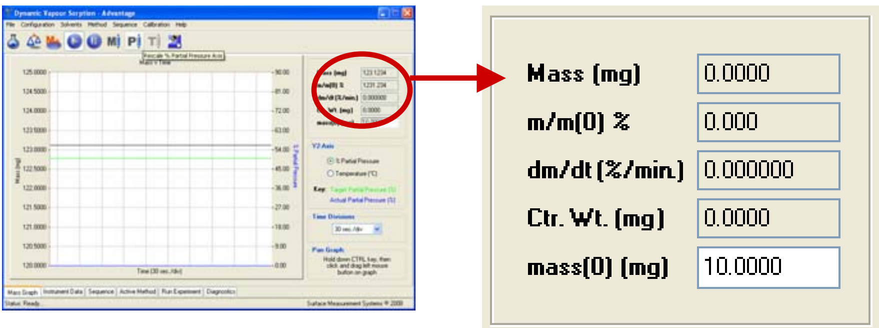


When panning the graph, the two data lines remain in the same positions in relation to each other.

As the lines are moved up and down on the graph, the values on the vertical axes change to follow their movement.

In addition, as the data lines are moved laterally, the vertical lines showing the time divisions move along with them.

10.3.5 Mass Data box



The mass data box continuously displays and updates the mass data being recorded and calculated by the system.

Mass (mg): The last measured value of the sample mass.

m/m(0)%: This parameter is the ratio of the current mass referenced to mass(0) as a percentage.

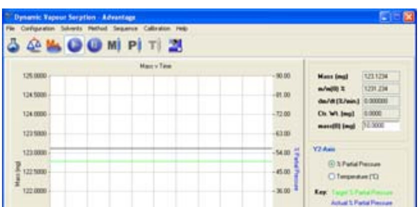
dm/dt (%/min): This function is the current slope of the mass data expressed as a percentage of the initial mass M(0). It is calculated by fitting a linear regression to a default time window of the last five minutes of data (Sections 6.2.2 and 9.6.1.1).

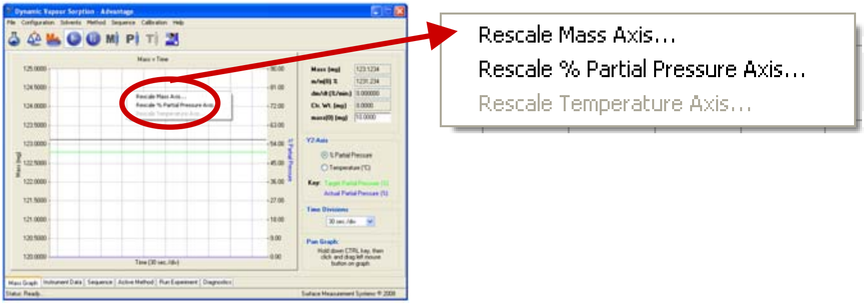
Crt. Wt (mg): Displays the current value of the counter weight being used. This value is added to all mass readings from the balance (Sections 6.1.4.1 and 9.3.3).

mass(0) (mg): Displays the recorded M(0) value

10.3.6 Right-clicking the Mass Graph panel

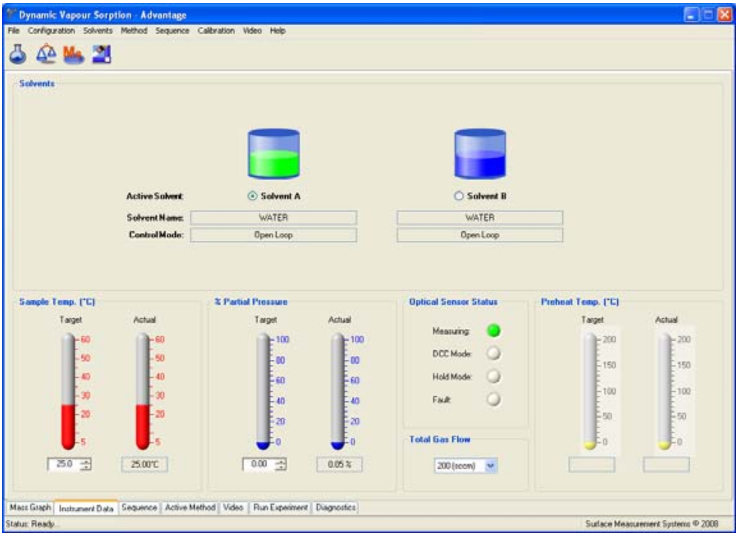
Right-clicking anywhere on the Mass Graph panel calls up the Rescale Axes options that appear in the Configuration menu (Section 9.3.2):





10.4 Instrument Data panel

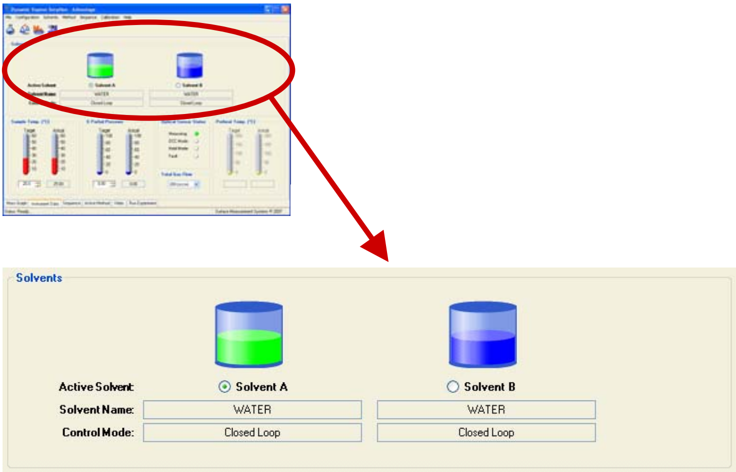
The Instrument Data Panel shown below displays measured instrument parameters as well as control switches for the main DVS functions.



When a Sequence is running (see Section 9.6), the Instrument Data panel serves only as a display of the system's parameters – none of the parameters it indicates may be altered by the user.

However, if there is no Sequence running, then the user may use the Instrument Data panel to input setpoints for almost all of the system parameters displayed.

10.4.1 Solvents Box



This box indicates the Control Mode in use for each solvent (see Sections 9.2.1.1 and 9.3.4), and which solvent has been designated Solvent A and Solvent B (see Section 9.4.1).

In addition the bottle icons indicate which solvent - A or B - is Active (in use) in the present experiment. The Active solvent is illuminated green as shown above.

If the user wishes to perform an experiment without loading a Sequence, then they may use this area to choose the Active Solvent by clicking on the desired label – Solvent A or Solvent B (see Figure 10.1 below):

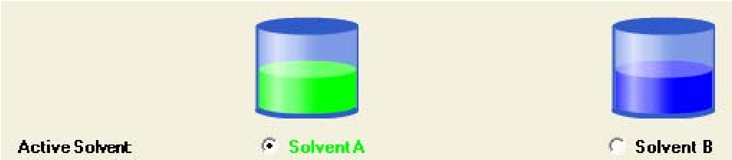
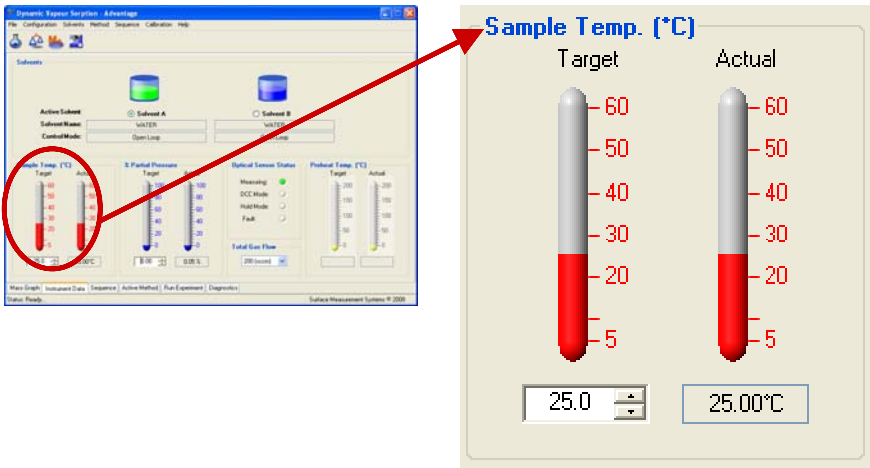


Figure 10.1: The **Active Solvent** may be toggled between Solvent A and Solvent B

Once a Sequence is running or Data is being saved, however, then the solvent selection function deactivates until the Sequence has finished or Save Data is switched off:

Figure 10.2: Active Solvent selection is inactive while a Sequence is running or Save Data is activated

10.4.2 Sample Temperature



This box allows the user to input and view the system's temperature setpoint and to read the measured system temperature.

The setpoint, or **Target** temperature, may be entered into the data entry box beneath the left-hand column labelled 'Target'. The desired temperature may be entered directly, or the up and down arrows can be used to alter the value in increments of 1°C. The column is coloured red to reflect the numeric value of the target temperature – remember that a maximum target temperature of 60°C is allowable in most cases

Note: In some cases, a higher sample temperature is attainable, depending upon the system incubator supplied with the DVS unit. In such cases, an SMS Service Engineer will be required to alter the system .dat file, and then the control software will display the appropriate sample temperature range.

The measured, or **Actual** temperature is indicated in the right-hand column. Again, the level of red-fill in the column reflects the figure displayed in the box below it.

This feature is useful for those situations in which the user wishes to run an experiment without employing a Sequence. The user may use this box to alter the system temperature at will.

If the Save Data function is activated (see Section 0), then the Target temperature may still be altered using the Sample Temp box.

If, however, a Sequence is running, then the setpoint entry box becomes inactive – the sample temperature setpoint will already have been defined in the Sequence itself (see Figure below). However, the Target column is still important in that it displays the target temperature being employed by the Sequence.

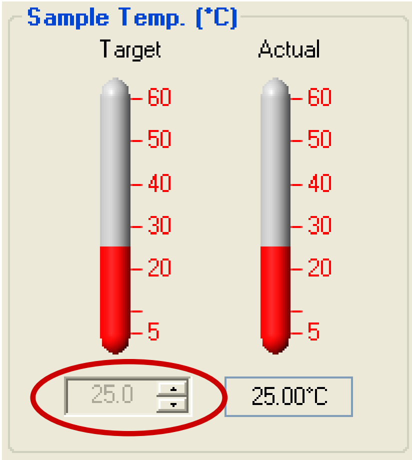
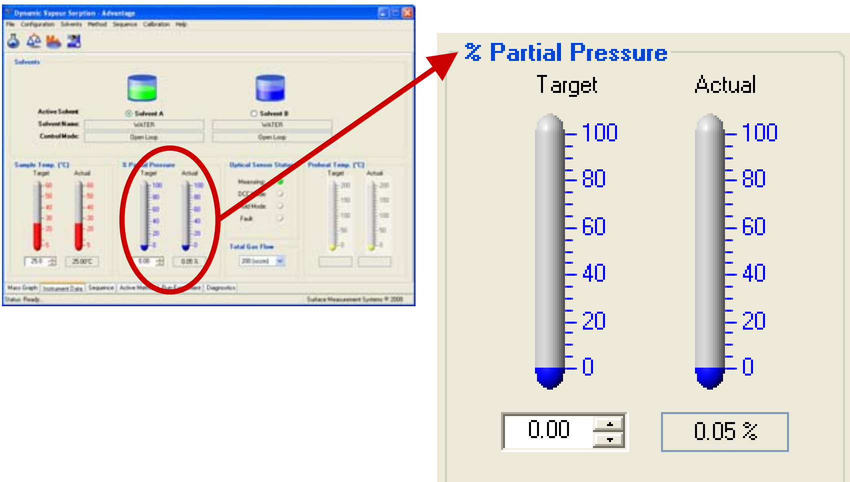


Figure 10.3: When a Sequence is running, the Target temperature setpoint data entry box becomes inactive.

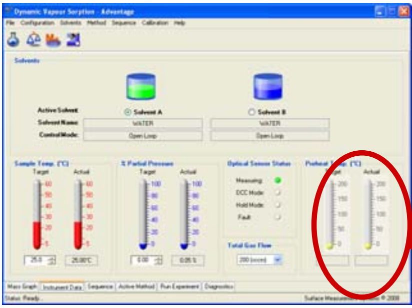
10.4.3 Partial Pressure

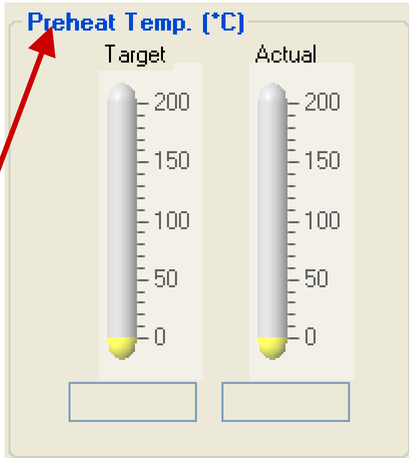


The Partial Pressure box operates in exactly the same way as the Sample Temperature box. Please refer to Section 10.4.2 for details.

Note: When the Optical Sensor is cleaning itself (ie: a DCC is active – Appendix E, Section 15), the Target and Actual PP (%) controls are disabled.

10.4.4 Preheat Temperature

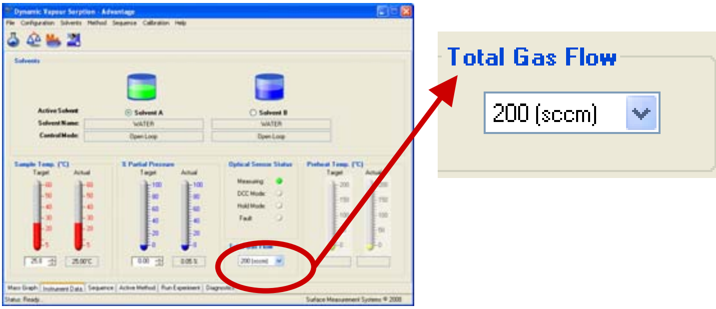




This box displays parameters only, and cannot be used to edit setpoints.

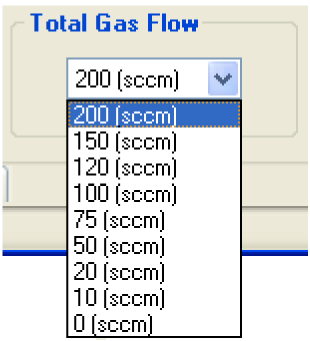
The **Preheat Temp** displays the **Target** and **Actual** sample preheater temperatures which are defined in the Preheat Method file (Section 9.5.1). This display only updates when the Preheater is running. The Actual preheat temperature is measured by a thermocouple located within 2cm of the sample pan.

10.4.5 Total Gas Flow



This drop-down menu allows the user to choose the total gas flow for the system in standard cubic centimetres per minute (sccm). The default setting for the DVS Advantage is 200 scccm.

The flowrate may be altered by clicking on the value in the edit box or the adjacent down arrow, which reveals the other available flowrates:

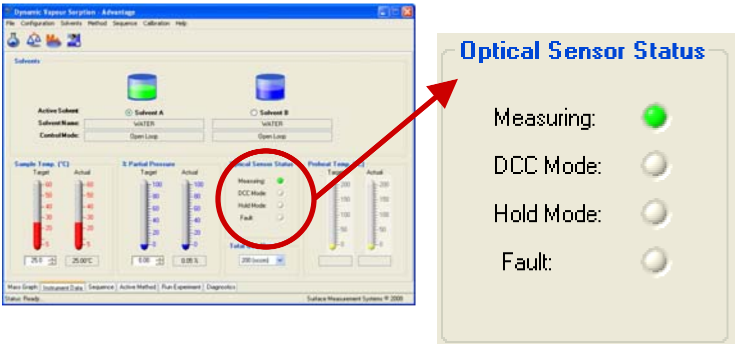


The Total Gas Flow box allows the flowrate to be set when there is no Sequence running. If the Save Data function is in use, the box is still active.

The box does become inactive once a Sequence is loaded, however. The flowrate is then defined by that specified in the Sequence, and is displayed in the Total Gas Flow box:

10.4.6 Optical Sensor Status

This box displays the current status of the Optical Sensor:



Measuring: Indicates that the Optical Sensor is online and actively measuring.

DCC: Indicates the Optical Sensor is in Dynamic Contamination Correction (DCC) mode. This mode is initiated automatically by the software or when the "*Decontaminate Sensor*" menu item is manually selected by the user. For more information regarding this please refer to Appendix E, Section 15 –

Optical Sensor Dynamic Contamination Correction.

Hold: Indicates the Optical Sensor is in Data Hold mode. This mode is entered once the DCC mode has completed. For more information regarding this please refer to Appendix E.

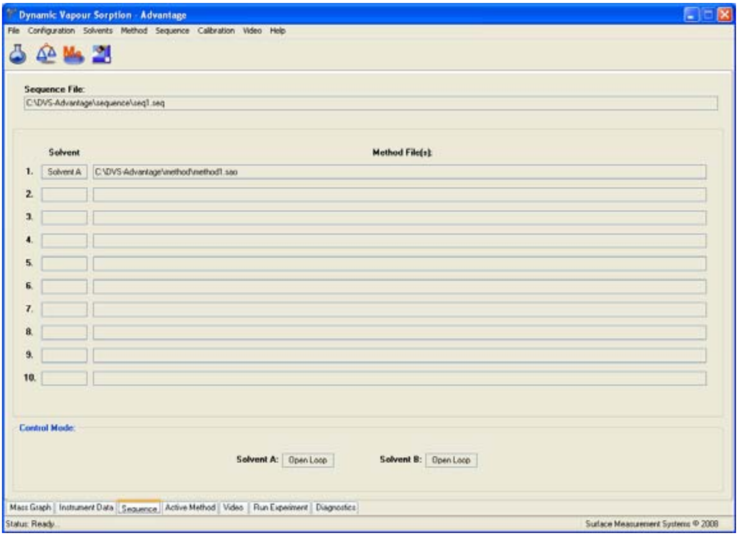
Fault: Indicates an optical fault with the Optical Sensor. This mode indicates that the Optical Sensor needs to be manually cleaned. For more information regarding this please refer to Appendix E.

Whenever a state is active, its ‘LED’ is illuminated – otherwise it remains greyed-out.

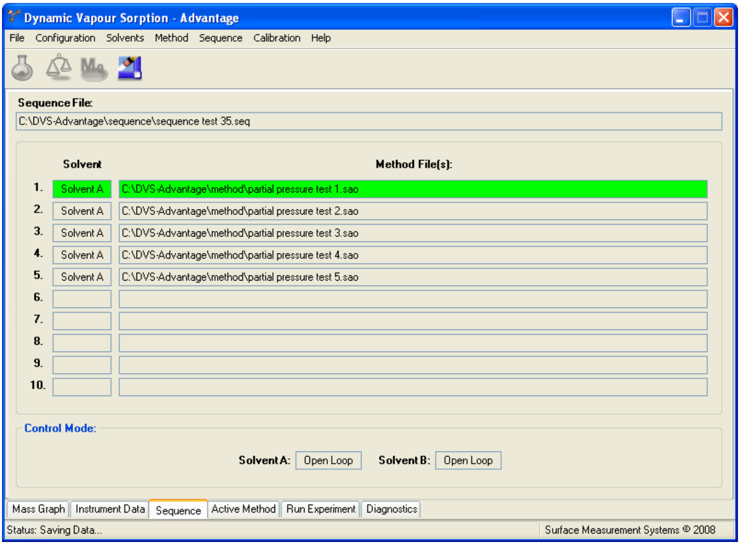
10.5 Sequence panel

The Sequence panel shown below shows the settings for the Sequence that is currently loaded. It displays the series of Method files that make up the Sequence along with the Control Mode, Sampling Rates, DMDT Values and Gas Flow settings that are also saved as part of the sequence file.

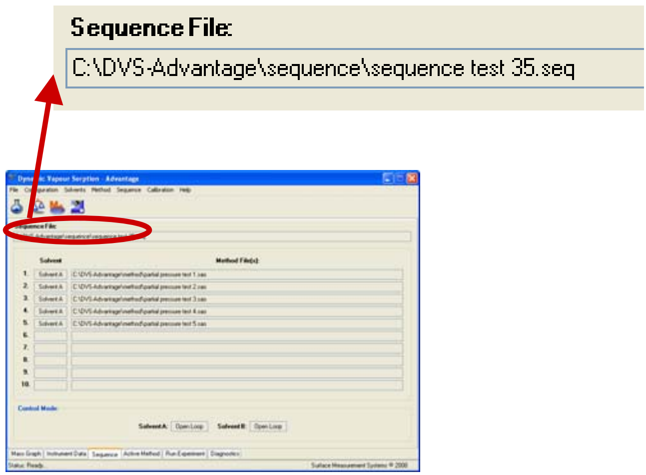
Please refer to Section 9.6 for details on creating, editing, and loading Sequence files.



Also note that when a Sequence is running, the active Method is highlighted green as shown below:

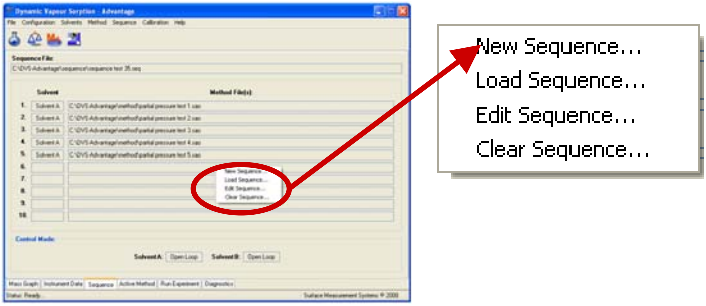


In addition, the name and location of the currently loaded Sequence file is displayed at the top of the panel in the box labelled **Sequence File**:

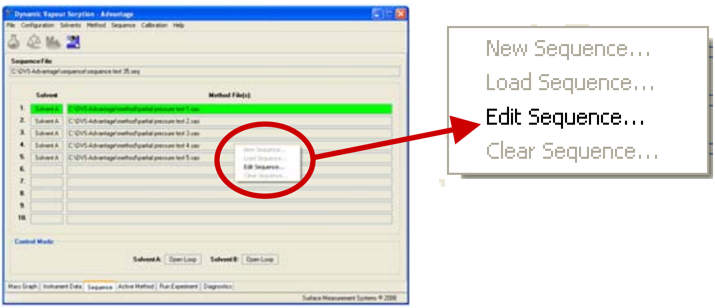


10.5.1 Right-clicking the Sequence panel

Right-clicking anywhere on the Sequence panel calls up the same list of functions as appears under the Sequence toolbar option (Section 9.6):



When a Sequence is running, however, these options become inactive, with the exception of **Edit Sequence** (see Section 9.6.1.3 for more details on editing running Sequences):



Note also that while a Sequence is running, the Select Solvents, Tare Balance and Set Initial Mass buttons at the top of the panel are rendered inactive – these functions are specified in the Sequence itself and cannot be ‘manually’ performed while a Sequence is running.

10.6 Active Method panel

The **Active Method** panel displays the details for the currently active method of a running Sequence.

The active Method may be either a **Preheat Method** (Figure 10.4), a **Partial Pressure Method** (Figure 10.5) or the special sensor cleaning method **Clean.dcc**. The display updates according to what type of method is active.

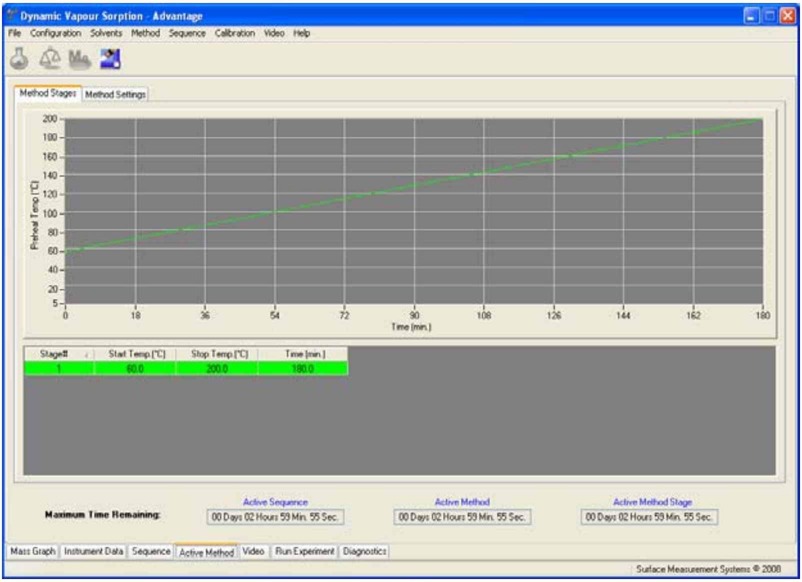


Figure 10.4: The DVS Control software **Active Method** panel showing an active Preheat Method running.

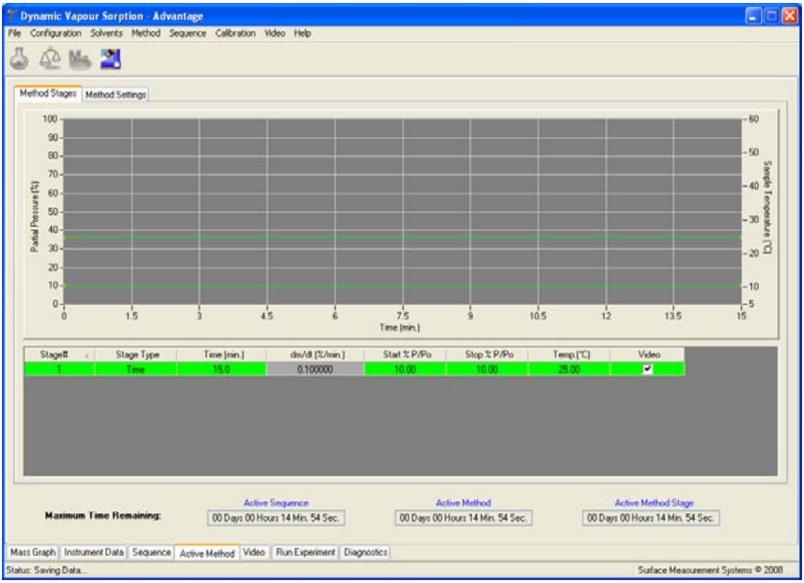


Figure 10.5: The DVS Control software **Active Method** panel showing an active Partial Pressure Method running.

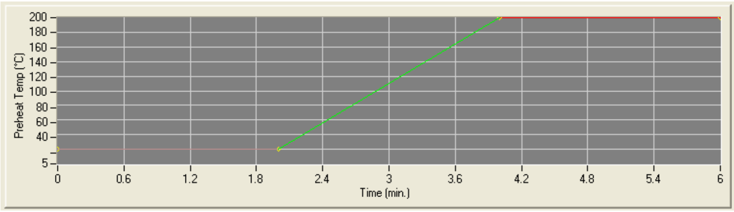
10.6.1 Active Method graph

The Active Method graph for Preheat, Partial Pressure and DCC Methods displays the profile of the current method as a function of time.

Each method stage is colour coded, to give a visual indication of how far the method has progressed.

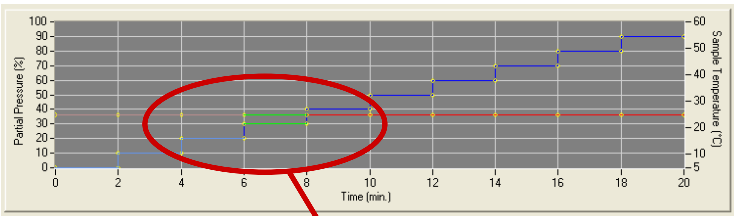
Preheat Methods

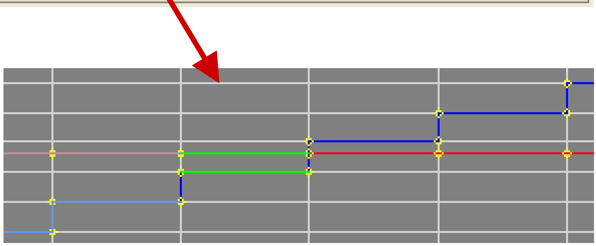
The current active stage is shown in green, future stages are shown in red and completed method stages are shown in light red, as shown below:



Partial Pressure Methods

The current active stage is shown in green, future stages are shown in dark blue and completed method stages are shown in light blue, as shown below:

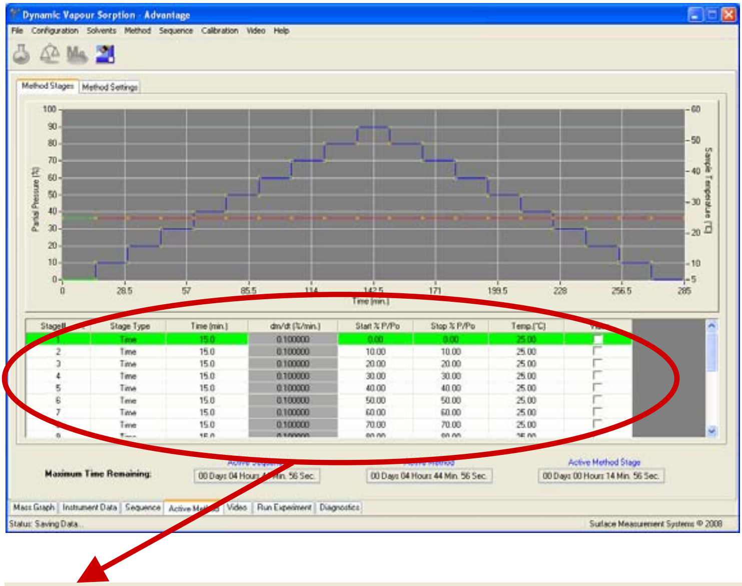




10.6.2 Active Method table

The Active Method table displays the details of the Method's Stages as specified in the New Method toolbar option (see Section 9.5.1.1).

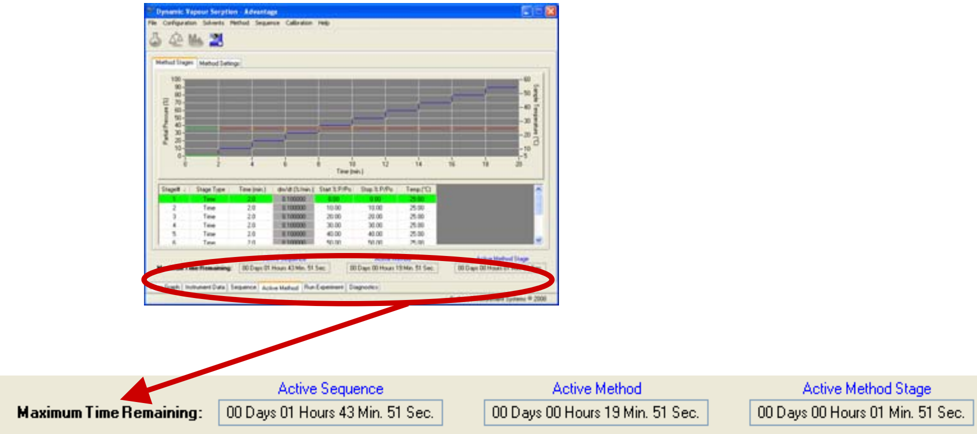
In addition, the active Stage of the Method is highlighted green, as shown below:



Stage#	Stage Type	Time (min.)	dm/dt (%/min.)	Start % P/Po	Stop % P/Po	Temp.(°C)	Video
1	Time	15.0	0.100000	0.00	0.00	25.00	☐
2	Time	15.0	0.100000	10.00	10.00	25.00	☐
3	Time	15.0	0.100000	20.00	20.00	25.00	☐
4	Time	15.0	0.100000	30.00	30.00	25.00	☐
5	Time	15.0	0.100000	40.00	40.00	25.00	☐
6	Time	15.0	0.100000	50.00	50.00	25.00	☐
7	Time	15.0	0.100000	60.00	60.00	25.00	☐
8	Time	15.0	0.100000	70.00	70.00	25.00	☐
9	Time	15.0	0.100000	80.00	80.00	25.00	☐

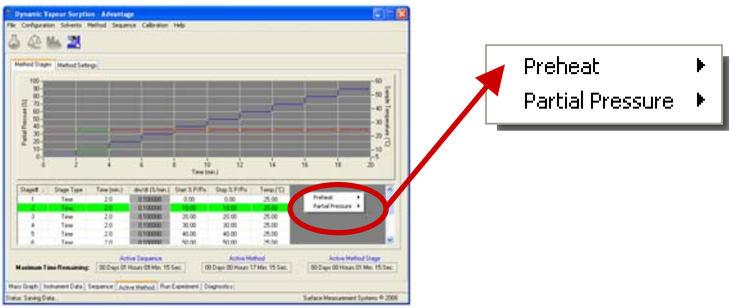
10.6.3 Maximum Time Remaining

These boxes indicate the time remaining until the end of the active Sequence, Method and Method Stage:



10.6.4 Right-clicking the Active Method panel

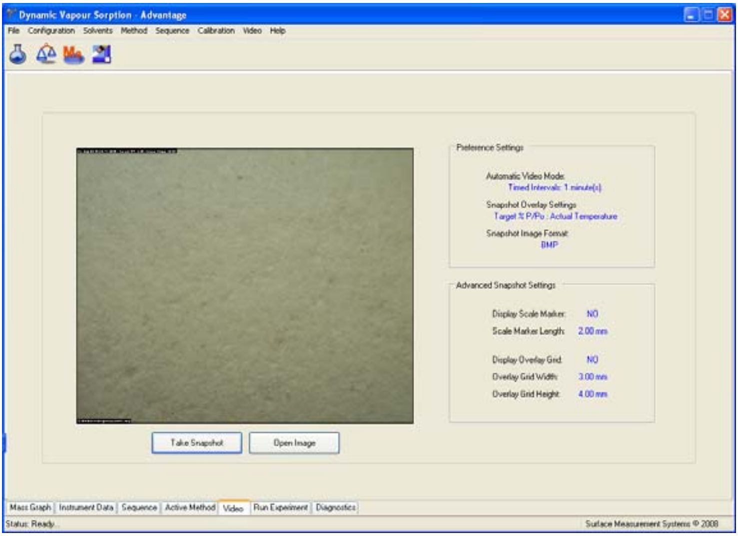
Right-clicking anywhere on the Active Method panel calls up the same list of functions as appears under the Method toolbar option (see Section 9.5):



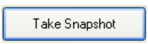
When a Sequence is running, these options remain active, as shown above.

10.7 Video Panel

The **Video** panel allows the user to take manual snapshots of the sample using the system's digital camera. The panel also displays the current snapshot and picture settings, as well as allowing the user to open previously saved pictures:

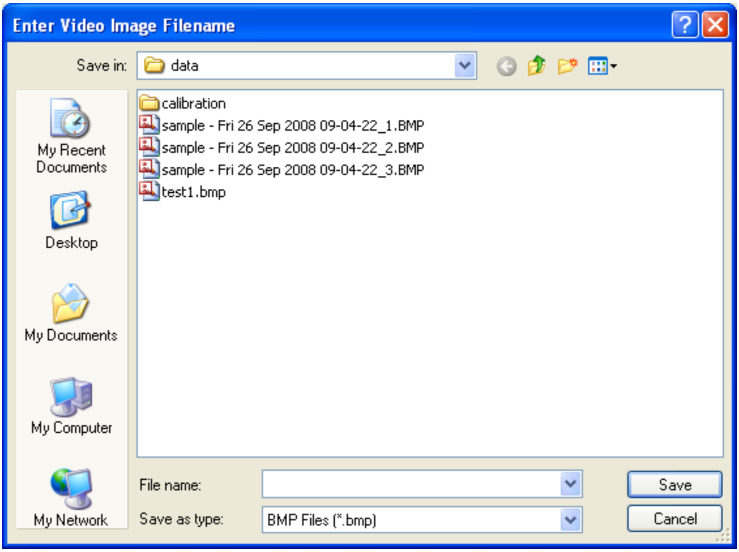


10.7.1 Take Snapshot

Clicking the **Take Snapshot** button 

allows the user to manually save a snapshot of the sample when data is not being saved.

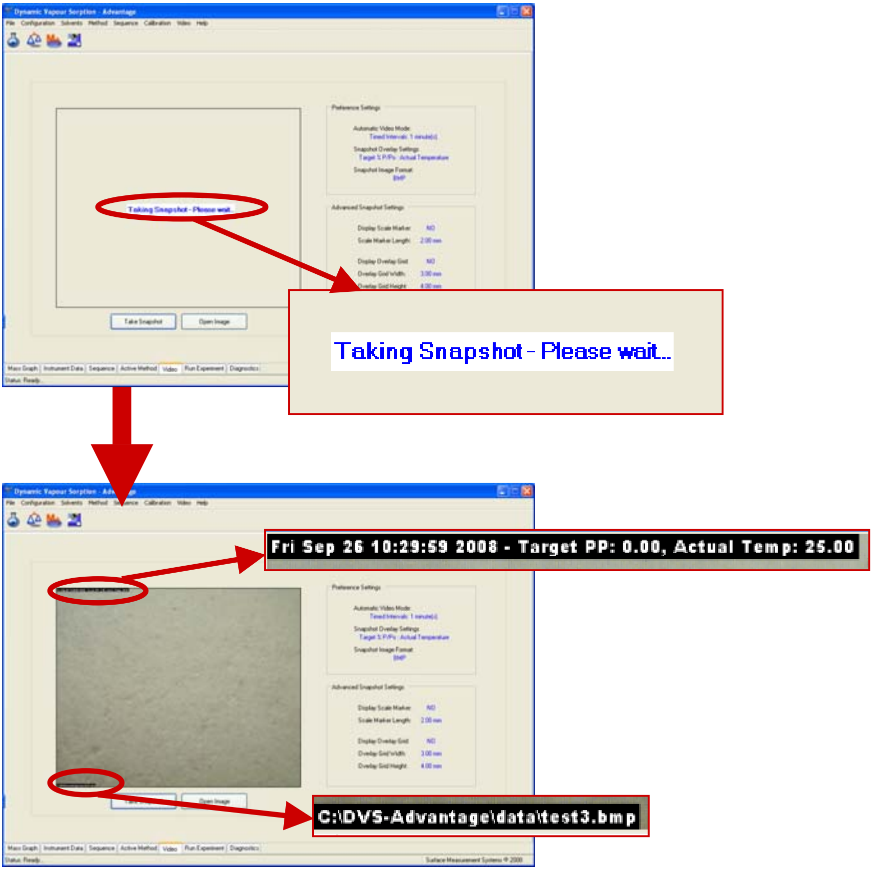
Clicking the button calls up the **Enter Video Image Filename** window, which prompts the user to save the image under the desired filename:



Note: The file will be saved in the format specified in **File** → **Preferences** → **Video**.

In addition, the **Enter Video Image Filename** window will automatically open at the same location as where the DVS data files are saved. The location of the picture files may of course be changed using the **Save In** drop-down menu at the top of the window.

Once a filename is entered and the **Save** button is clicked, the **Video** panel shows a 'please wait' message before displaying the newly recorded sample snapshot:



The recorded picture's top left-hand corner displays the date and experimental data according to set Preferences (see Section 9.2.1.3). In the bottom left-hand corner is displayed the directory location to which the picture was saved, and the filename (with extension).

This data appears to be very small when displayed on the **Video** panel. However, these images are high-resolution, and as such, are meant to be viewed 'up close' in a picture-viewing program. When the picture is viewed at a zoom of

100% (ie; the ‘normal’ picture size), the data is clearly displayed.

Note: Whenever the software is started up, the **Video** panel will be blank until a snapshot is taken. From that point on the panel will display the most recent snapshot until the DVS control software is shut down.

Additional Note: Whenever data is being saved (ie; when running a Method or when simply saving data), the **Take Snapshot** button becomes inactive.

If images are to be taken while saving data, it must be done through either the Method settings or according to a regular time interval. Which of these approaches is used may be selected in **File** → **Preferences** → **Video** → **Automatic Snapshot Operation Mode** (see Section 9.2.1.3).

10.7.2 Open Image

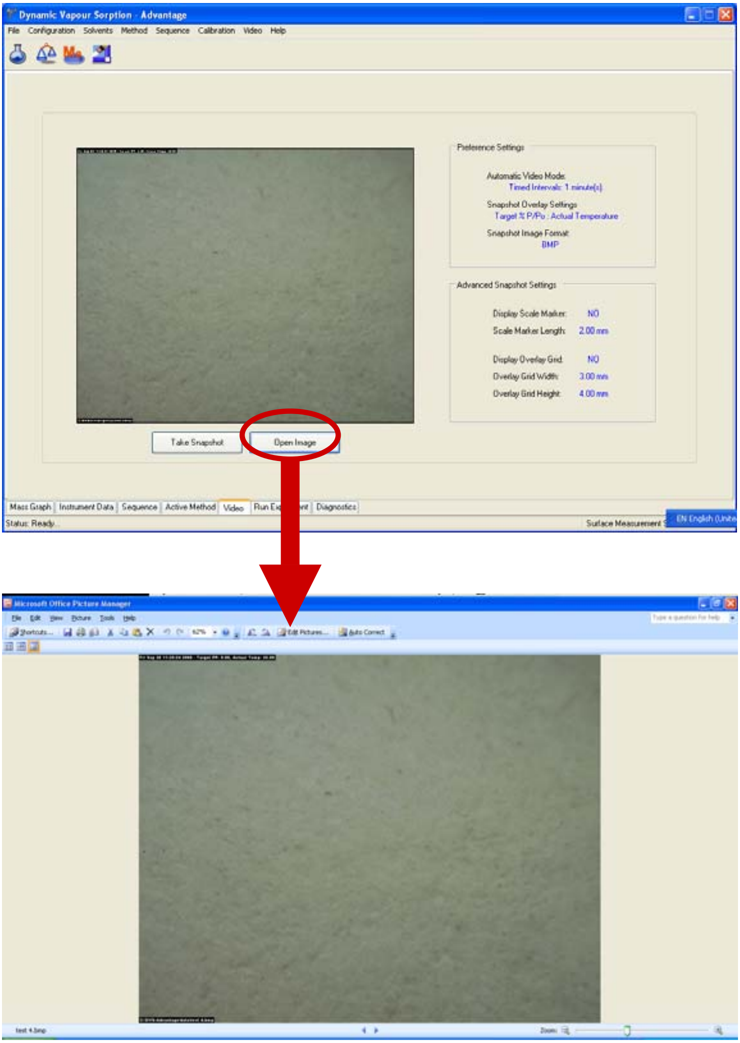
Clicking the **Open Image** button 

allows the user to open the image most recently recorded and displayed in the **Video** panel.

Opening the image allows the user to see the picture full-size, and to take advantage its high resolution – it is possible to zoom in close to the sample picture and discern a greater amount of detail.

Note: The image will be opened using the picture viewer set as default in the control computer’s windows operating system.

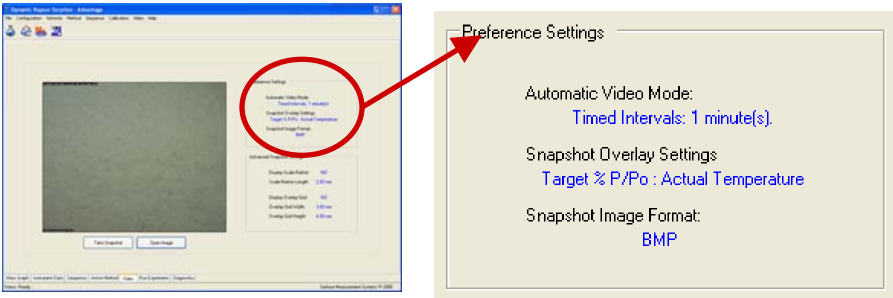
Clicking **Open Image** immediately calls up the image displayed in the Video panel:



The picture viewer may then be used to zoom in to see the file at 100% of its normal size (the magnification shown above is 62%). In the example shown above, the default picture viewer in use is Microsoft Office Picture Manager®.

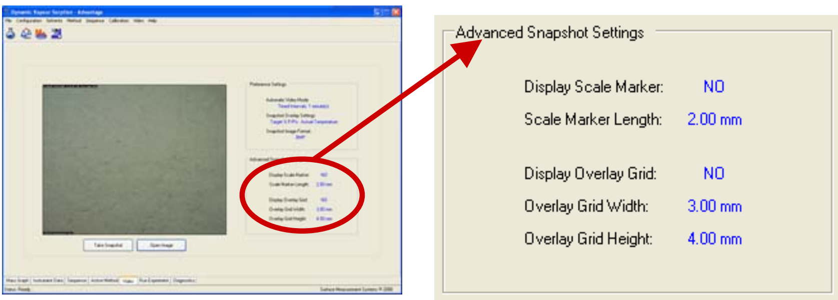
Note: The **Open Image** button remains active even when data is being saved so that images may be examined as they are taken throughout the data-saving process.

10.7.3 Preference Settings



The **Preference Settings** box on the **Video** panel displays the image preferences selected in **File** → **Preferences** → **Video** (see Section 9.2.1.3).

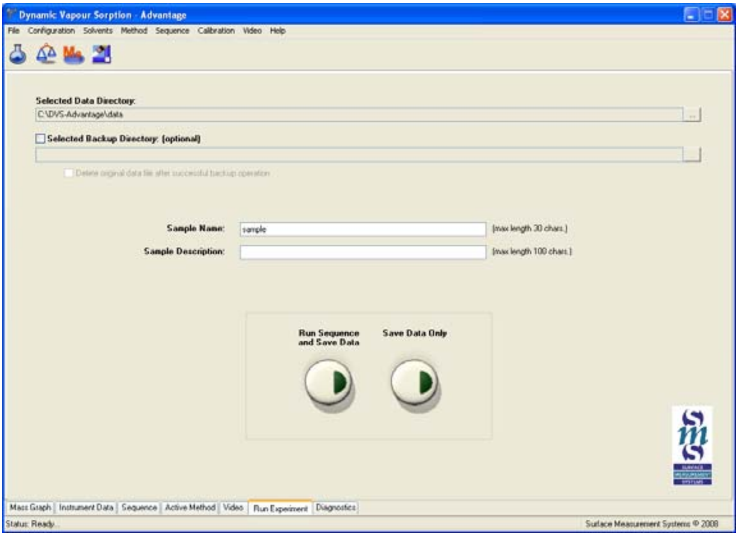
10.7.4 Advanced Snapshot Settings



The **Advanced Snapshot Settings** box on the **Video** panel displays the image settings selected in **Video** → **Advanced Snapshot Settings** (see Section 9.8.2).

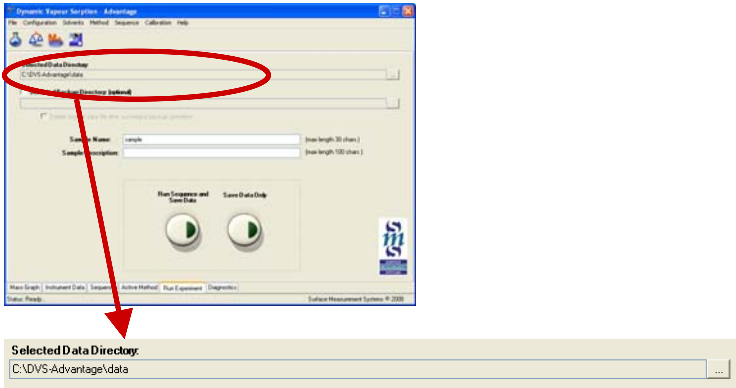
10.8 Run Experiment panel

The **Run Experiment** panel is used to configure a DVS experiment:



An experiment may be configured by following the steps outlined below:

Step 1: Select the Data Directory



This directory specifies the location to which the raw data files from the DVS experiment will be saved.

This should be set to a directory location on the local PC attached to the DVS instrument rather than a network directory so that network connection failures do not affect the running DVS experiment.

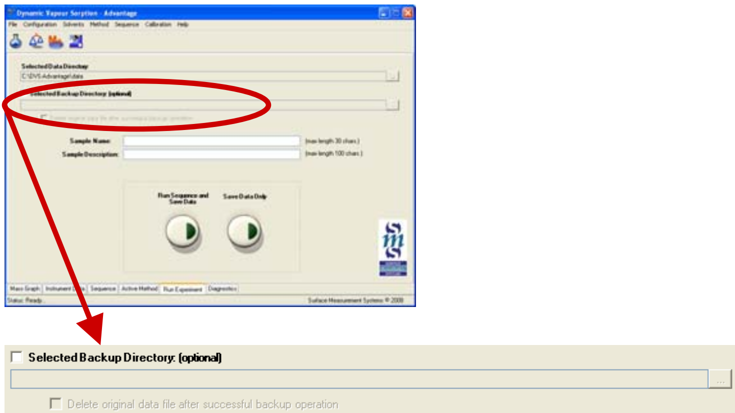
The directory displayed automatically is the one specified in the Preferences toolbar option (see Section 9.2.1.2). The software is supplied with the following Preference specified by default:

C:\DVS-Advantage\data

To choose a different directory, click on the ... button at the end of the Selected Data Directory box – the following window will appear:

Upon selecting the desired folder and clicking **OK**, the newly specified directory appears in the Selected Data Directory box.

Step 2: (Optional) Select the Backup Directory



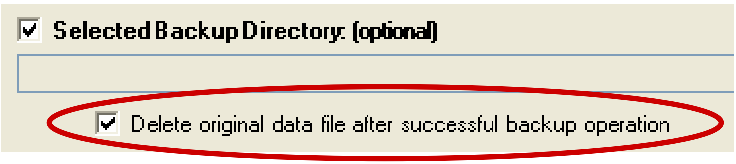
This option specifies the backup directory to which a copy of the raw data files will be stored once the DVS experiment has completed. This option should be set to a directory location on the network.

This option may be used to ensure that the DVS experiment is not interrupted by a network connection failure.

In order to activate the **Select Backup Directory** option, the tick-box next to the section header must be clicked:

Selection of the Backup Directory works in the same way as for the Data Directory, as shown above.

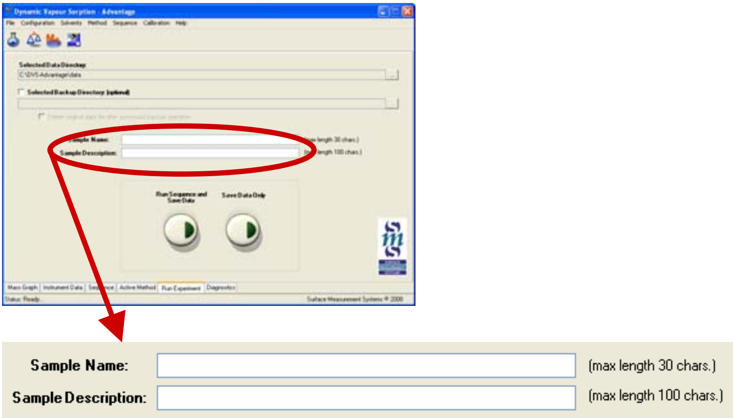
Delete original data file after successful backup operation: If the raw data file backup operation is successful and this option is selected, all raw data files produced by the current DVS experiment that were saved to the Data Directory (as specified in Step 1) are deleted.



This option is also activated using a tick-box:

Note: If the backup operation is unsuccessful, the raw data files will not be deleted from the Output Directory, so that the DVS data will never be lost.

Steps 3 & 4: Enter Sample Name & Enter Sample Description



Enter Sample Name

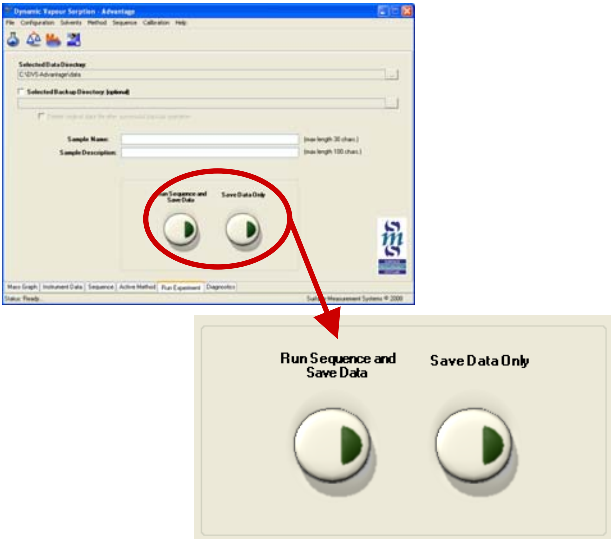
Enter the name of the sample being tested. The maximum length of the name that may be entered is 30 characters.

Enter Sample Description

A detailed description of the experiment may be entered in this field. The maximum length of the sample description is 100 characters.

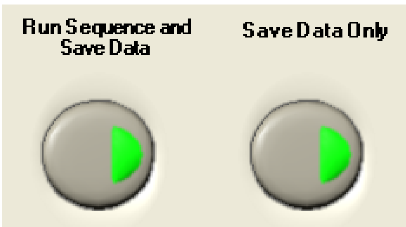
Step 6: Start the experiment

To start the experiment, simply click one of the following buttons:



Run Sequence and Save Data

Clicking this switch starts running the loaded sequence and saving DVS raw data to the Output directory. Clicking this option illuminates both buttons green:

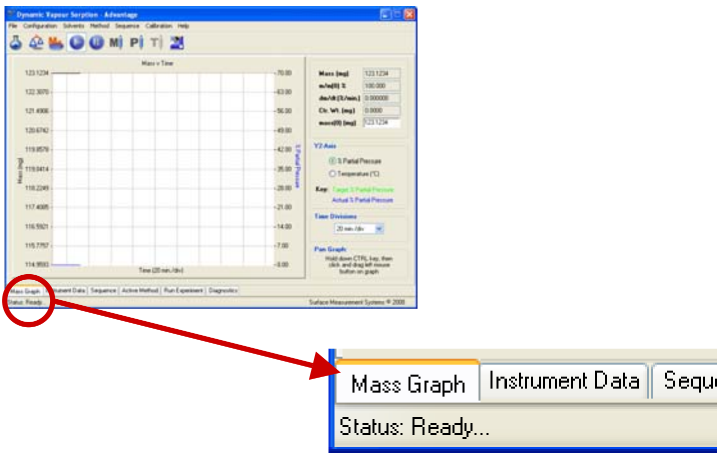


Save Data Only

Clicking this switch starts saving DVS raw data to the Output directory. This mode allows experimental conditions to be modified without running a Sequence.

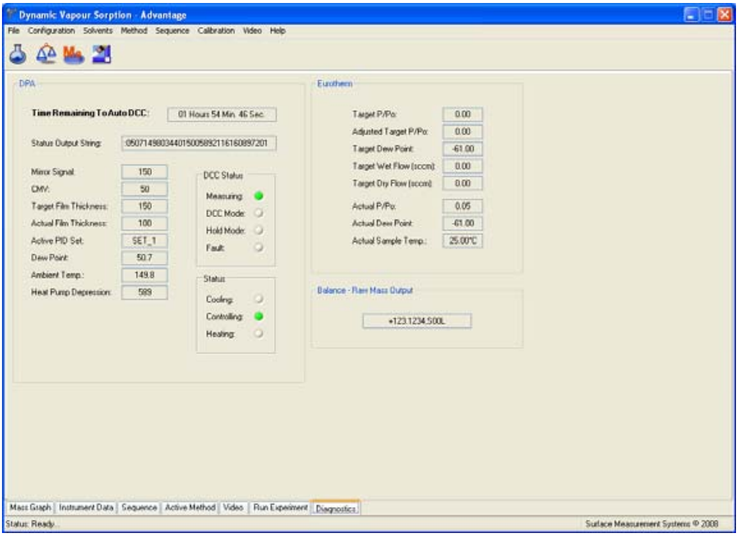
> **Note:** The filenames of the raw data files produced by a DVS experiment are automatically created by appending the date and time to the sample name.
>
> Eg: Sample Name = SampleX
>
> If a DVS experiment is started at 10:49:32am on Wednesday the 19th of November 2006, the DVS raw data file created will be named:
>
> **SampleX - Wed 19 Nov 2006 10-49-32.dat**

10.9 Status Bar



The status bar is located in the bottom left hand corner of the DVS Advantage interface. Once the system is saving data the status bar text will change from **Status: Ready** to **Status: Saving Data**. When saving data the text will also flash to indicate that saving data is active.

10.10 Diagnostics Panel

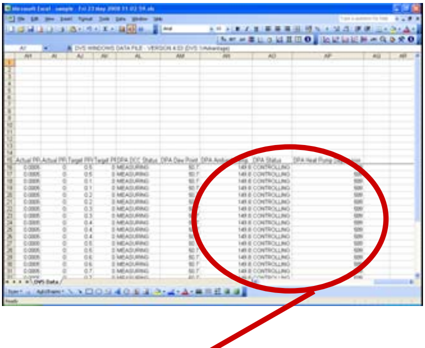


The Diagnostics Panel, shown above, displays various data relating to the system's Dew Point Analyser (which measures the system partial pressure), Eurotherm control computer, and the system balance itself.

This panel is designed to provide information required by SMS Service Engineers when working with the DVS Advantage.

Incidentally, some of the DPA data displayed on this panel is also saved into the DVS raw data file when data is saved:

Note: The Diagnostics panel is only visible if the menu option **Configuration→Diagnostics Tab Visible** has been checked. See Section 9.3.5.



DPA DCC Status	DPA Dew Point	DPA Ambient Temp.	DPA Status	DPA Heat Pump Depression
MEASURING	50.7	149.8	CONTROLLING	589
MEASURING	50.7	149.8	CONTROLLING	589
MEASURING	50.7	149.8	CONTROLLING	589
MEASURING	50.7	149.8	CONTROLLING	589
MEASURING	50.7	149.8	CONTROLLING	589
MEASURING	50.7	149.8	CONTROLLING	589
MEASURING	50.7	149.8	CONTROLLING	589
MEASURING	50.7	149.8	CONTROLLING	589
MEASURING	50.7	149.8	CONTROLLING	589

Again, this information is relevant for SMS Service Engineers when saving data to check the system's performance.

11 Appendix A – Instrument Specification

Model	DVS Advantage 1	DVS Advantage 2 Low Range	DVS Advantage 2 High Range
Sample capacity	1.5g	10g	100g
Dynamic range	150mg	1g	10g
Minimum sample weight	1mg	10mg	100mg
Sensitivity	0.1µg	1 µg	10 µg
Sample pre-heater temperature	25* - 200°C	25* - 200°C	25* - 200°C
Temperature range	5 - 60°C	5 - 60°C	5 - 60°C
Humidity range	0 – 98%RH	0 – 98%RH	0 – 98%RH
Humidity accuracy	±1.5%	±1.5%	±1.5%
Vapour concentration range	0 – 98% p/p	0 – 98% p/p	0 – 98% p/p
Vapour pressure accuracy	±1.5% p/p	±1.5% p/p	±1.5% p/p
Gas Flow Rates	0 – 200 sccm	0 – 200 sccm	0 – 200 sccm

* Current temperature setting for oven.

12 Appendix B – Salt Validation Calibration.

A DVS Salt Validation module allows the user to verify that the system's Mass Flow Controllers are accurately generating the desired partial pressures.

In short, the calibration verifies that the partial pressure levels in the DVS are what the instrument says they are.

Performing Salt Validation Calibrations is important as accurate measurement of partial pressure is absolutely essential to the working of the DVS instrument.

With no accurate idea of the partial pressures being generated in the DVS, any results generated by the instrument become meaningless.

Note: It is recommended that Salt Validation Calibrations are carried out at least every six months.

12.1 How to perform a Salt Validation on the DVS

The calibration is based upon the principle that the vapour pressure above a saturated salt solution in equilibrium with its surroundings is a constant at a particular temperature.

The procedure also involves using a module included in the DVS Standard Analysis Suite software. Please refer to the Standard Analysis Suite Manual for information on how to operate the relevant module for salt validation.

12.1.1 Salt solution

The first step in the procedure involves preparing a small, saturated salt sample. This is done by placing approximately 20-30mg of test salt in a sample pan adding drops of de-ionised water until a slush or paste is formed.

12.1.2 Choice of Salt

Several different salts can be used for the calibration; Table 1 below lists five salts commonly used to validate the DVS partial pressure generation:

Salt	Water %p/p_o at 25°C	Water %p/p_o at 40°C
LiCl	11.3	11.2
$MgCl_2$	32.8	31.6
$MgNO_3$	52.89	48.42
NaCl	75.3	74.7
KNO_3	93.7	89.0

Table 1: Salt p/p_o values for calibration Saturated salt deliquescence points [1]

It is recommended that at least three salts are used which cover the full range of partial pressures – this ensures a comprehensive system validation. For example, it would be advisable to use LiCl, $MgNO_3$, and KNO_3 to cover the full range of partial pressures.

12.1.3 Temperature

Next, the water vapour partial pressure above the solution must be measured. This can be done at 25°C or any other desired temperature between 10°C and 50°C. It is recommended that a temperature is chosen which is closest to the one commonly used in experiments.

12.1.4 Method

To execute the validation, a method must be set up which begins at a partial pressure approximately 5% above the salt solution partial pressure. For example, if using $MgNO_3$ at 25°C, the method would start at around 58% p/p_o (ie; 5% above 52.89%). This first stage would need to last for one hour.

Following this, the partial pressure needs to be ramped down to 5% below the salt partial pressure (ie; 10% below method's initial p/p_o). This ramp should take place across 300 minutes. In the case of $MgNO_3$ at 25°C, this would mean ramping down to 48% p/p_o.

Finally, the ramp needs to be reversed, going back up to the starting partial pressure. Again, this occurs across 300 minutes. In the case of $MgNO_3$ at 25°C, this would mean ramping back up to 58% p/p_o.

The partial pressure profile that could be used for $MgNO_3$ at 25°C is shown below in Figure 12.1:

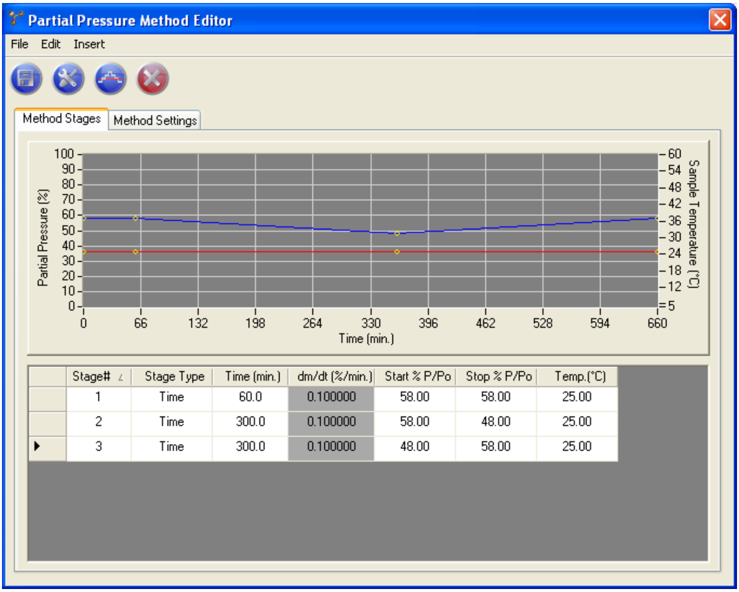


Figure 12.1: The proposed partial pressure profile for a salt validation using $MgNO_3$ at 25°C.

Once this method has been run, the data may then be analysed using the **Salt Validation** module.

12.1.5 Results

At the point where the DVS-generated $\%p/p_o$ is equal to the $\%p/p_o$ above the saturated salt solution, the rate of change of mass with partial pressure ($dm/d(\%p/p_o)$) of the salt solution is zero. At this point, the DVS-generated $\%p/p_o$ is the saturated salt literature value – irrespective of what the partial pressure sensors are reading.

By plotting the target $\%p/p_o$ vs. $dm/d(\%P/P_o)$ as shown in Figure 12.2 and finding the intercept where $dm/d(\%p/p_o) = 0$, the generated $\%p/p_o$ can be calibrated against the actual partial pressure above the saturated salt solution at this temperature - thus enabling the user to validate the accuracy of the instrument's partial pressure sensors.

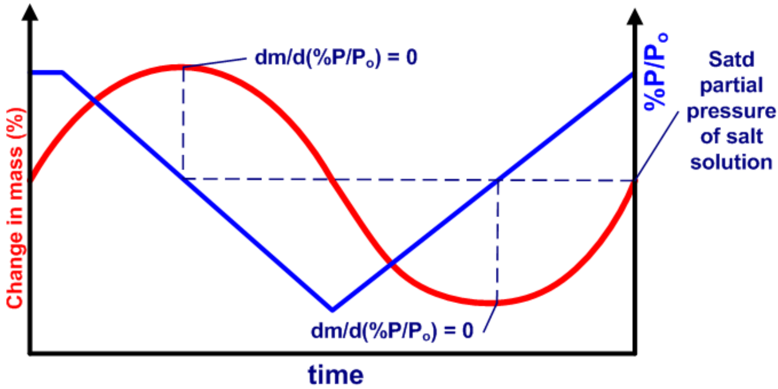


Figure 12.2: dm/d(%p/p_o) is zero at the saturated partial pressure of the salt solution

13 Appendix C – References

[1] L. Greenspan, Journal of Research of the National Bureau of Standards-A, vol. 81A (1), 1977, 89-96.

14 Appendix D – Solvent Lookup Table

Note: Only qualified service engineers should ever change the Solvent Lookup Table values.

Note: The Solvent Lookup table is only in effect when the DVS Advantage is running in OPEN LOOP mode.

Note: This information is provided for informational purposes only.

The DVS uses Mass Flow Controllers (MFC's) to flow gas through the DVS Advantage system to produce the desired Partial Pressure. In order to generate a certain partial pressure (PP), one of the MFC's flows a percentage of dry air (called the Dry Flow) into the DVS system while another MFC flows a certain percent of solvent into the system (called the Wet flow). The sum of the two flows adds up to 100%.

eg: Say the desired Partial Pressure is 40%. The Wet Flow MFC produces 40% of wet air and the Dry Flow MFC produces 60% of dry air to produce the desired 40% PP.

MFCs may slightly over- or under-generate the amount of gas flow they are requested to produce. This means that the system may be asked to produce 40%p/p_0 but the system actually produces 42%p/p_0.

The DVS system **automatically** overcomes this by using the Solvent Lookup Table to slightly adjust the requested gas flow rates of the MFCs to ensure correct generation of the partial pressure.

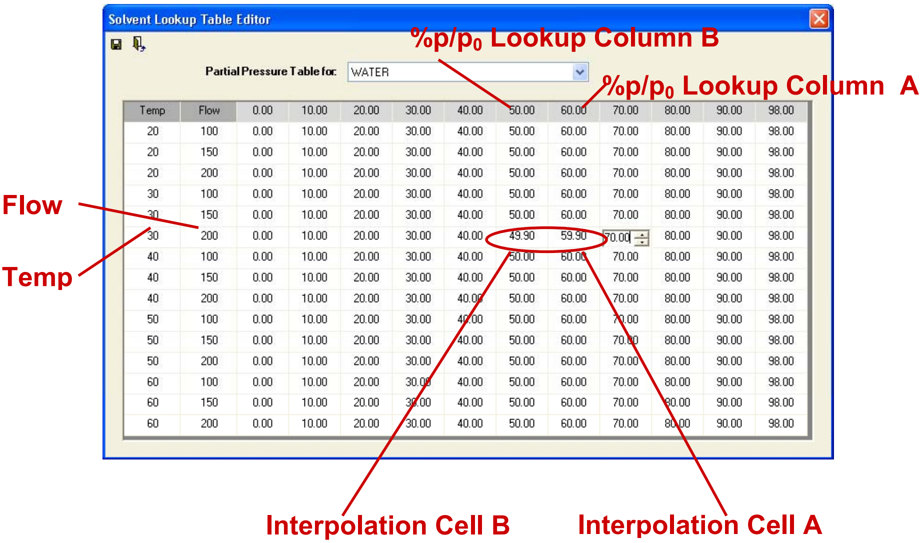
SMS service engineers alter the Solvent Lookup Table during the calibration process. In the DVS Advantage each solvent has its own partial pressure lookup table.

The lookup row used in the table is based upon the Sample Temperature and the Total Gas Flow setting.

Lookup Example:

Assume that the DVS Advantage system is operating under the conditions listed below:

- Requested Partial Pressure = 58%.
- Sample temperature = 25 ^{o}C
- Total Gas Flow = 150 sccm
- The Solvent Lookup Table has the settings in the figure below:



Temp	Flow	0.00	10.00	20.00	30.00	40.00	50.00	60.00	70.00	80.00	90.00	98.00
20	100	0.00	10.00	20.00	30.00	40.00	50.00	60.00	70.00	80.00	90.00	98.00
20	150	0.00	10.00	20.00	30.00	40.00	50.00	60.00	70.00	80.00	90.00	98.00
20	200	0.00	10.00	20.00	30.00	40.00	50.00	60.00	70.00	80.00	90.00	98.00
30	100	0.00	10.00	20.00	30.00	40.00	50.00	60.00	70.00	80.00	90.00	98.00
30	150	0.00	10.00	20.00	30.00	40.00	50.00	60.00	70.00	80.00	90.00	98.00
30	200	0.00	10.00	20.00	30.00	40.00	49.90	59.90	70.00	80.00	90.00	98.00
40	100	0.00	10.00	20.00	30.00	40.00	50.00	60.00	70.00	80.00	90.00	98.00
40	150	0.00	10.00	20.00	30.00	40.00	50.00	60.00	70.00	80.00	90.00	98.00
40	200	0.00	10.00	20.00	30.00	40.00	50.00	60.00	70.00	80.00	90.00	98.00
50	100	0.00	10.00	20.00	30.00	40.00	50.00	60.00	70.00	80.00	90.00	98.00
50	150	0.00	10.00	20.00	30.00	40.00	50.00	60.00	70.00	80.00	90.00	98.00
50	200	0.00	10.00	20.00	30.00	40.00	50.00	60.00	70.00	80.00	90.00	98.00
60	100	0.00	10.00	20.00	30.00	40.00	50.00	60.00	70.00	80.00	90.00	98.00
60	150	0.00	10.00	20.00	30.00	40.00	50.00	60.00	70.00	80.00	90.00	98.00
60	200	0.00	10.00	20.00	30.00	40.00	50.00	60.00	70.00	80.00	90.00	98.00

Figure 14.1: Solvent Table Editor panel

Step 1: Find the correct lookup row in the table:

This is a two stage process:

Locate the Temp:

Starting from the top row in the table, find the row with the first temperature value that is **greater than** the sample temperature.

> **Note:** If there is no Temp greater than the sample temperature the maximum temperature is used.

Result = 30 ^{o}C.

Locate the correct Flow (nb: The Temp is the same value found in step 1):

The lookup row is the row with the correct temperature found in step 1 and a flow value that is **greater than** the current Total Flow rate.

> **Note:** If there is no flow greater than 200sccm so the max flow is used.

Result = 200 sccm

We now have located the correct lookup row: Temp = 30^{o}C and Flow = 200.

Step 2: Identify the Partial Pressure Lookup Columns A and B

Using the lookup row found in step 1, PP Lookup Column A is identified by the Column Header that has a value **greater than** the Requested Partial Pressure.

PP Lookup Column B is the column to the immediate left of Column A.

Note: If no partial pressure column header is greater than the requested partial pressure then the very last partial pressure column is used as PP Lookup Column A.

Result = PP Lookup Column Header A value = 60.0
PP Lookup Column Header B value = 50.0

Step 3: Identify Interpolation cells A and B: (These values are used in later calculations)

- **If PP Lookup Column A <u>was</u> found in step 2 above.**

  Interpolation Cell A:
  Row: Temp = 30 ^{o}C, Flow = 200
  Column: PP Lookup Column A = 60.0
  Cell Value = 59.90

  Interpolation Cell B: located on the same row as Cell A, but is found in PP Lookup Column B.
  Row: Temp = 30 ^{o}C, Flow = 200
  Column: PP Lookup Column B = 50.0
  Cell Value = 49.90

- **If the PP Lookup Column was NOT found in step 2 and the <u>very last</u> partial pressure column is being used the Target Partial Pressure lookup cells are:**

  Target PP Lookup Cell A:
  Row: Temp = 30^{o}C, Flow = 200
  Column: PP Lookup Column A = (last column)
  Value = cell value at the location specified by the above row/column combination

  Target PP Lookup Cell B: *Not used*

Step 4: Calculate the Actual PP Delta value:

If the PP Lookup Column A was found in step 2, then the Actual PP Delta value can be calculated.

The Actual PP Delta value is the difference between the PP Lookup Column A header value (60.00) and the PP Lookup Column B header value (50.00).

> **Note:** If the Partial Pressure Lookup Column A was not found in step 3, the Actual PP Delta value is set to zero.

Result: 50.0 – 40.0
Actual PP Delta = 10.0

Step 5: Calculate the Requested PP Delta value

The Requested PP Delta value is the difference between the Requested Partial Pressure (58.0) and the PP Lookup Column B Header value (50.00).
Result = 58.0 – 50.0
Requested PP Delta = 8.0

Step 6: Perform Interpolation and Adjustment

Which calculation is performed here depends if the Requested Partial Pressure Column was located in step 3.

a. If Partial Pressure Lookup Column A **was** found in step 3:

   Calculate the **interpolation value**: the difference between Interpolation Cell A and Interpolation Cell B from step 3.

   Result = 59.90 - 49.90
   Interpolation value = 10.00

Calculate the adjusted partial pressure according to the following formula:

*Adjusted PP = Interpolation Cell B + (Requested PP Delta * Interpolation value) / Actual PP Delta*

Result = 49.90 + [(8.0 * 10.00) / 10.0]

Adjusted PP = 57.90

b. If the Requested Partial Pressure Column **<u>was not</u>** located in step 3:

Calculate the adjusted value according to the following formula:

Adjusted PP = Target Partial Pressure Lookup A + PP Delta

15 Appendix E – Optical Sensor Dynamic Contamination Correction

To alleviate the problems of measurement accuracy due to contamination the Optical Sensor has a contamination compensation process called Dynamic Contamination Correction (DCC).

A DCC consists of 2 stages:

1. DCC stage: During this stage the mirror surface is heated to remove contamination and the DCC LED on the DVS Advantage software interface is illuminated.
2. Hold stage: During this stage the system stabilises onto the active dew point and the HOLD LED is illuminated.

Note: The FAULT LED may be illuminated after a DCC has completed. This indicates that the mirror needs to be cleaned manually. See the Optical Sensor Maintenance section below

Optical Sensor Maintenance – Manual Cleaning

Clean the mirror surface and optics window using a cotton bud soaked in distilled water. If the sensor has been exposed to oil based contamination then use one of the following solvents instead of distilled water: (methanol, ethanol, or isopropyl alcohol).

When does a DCC occur?

- A DCC operation may be started manually by selecting the Calibration→Decontaminate Sensor menu item in the DVS Advantage software.

- A DCC will automatically occur whenever power to the DVS Advantage Electronics box instrument is switched on.
- A DCC will also automatically occur at regular intervals when the DVS Advantage software is running. The interval rate is defined in the DVSAdvantage.ini file by the AUTO_DCC_MINS value. This value should only be changed by a qualified SMS service engineer.
- You may also load a DCC method as part of a sequence:

  In order to keep the Optical Sensor reading accurate we strongly recommend that you perform a DCC cycle prior to running a method. To do this you can simply load the “Clean.dcc” method that can be found in your DVS Advantage method directory into your sequence before the method that you wish to run.

Note: It is STRONGLY recommended that a Clean.dcc Method be run before each new partial pressure method in a sequence.

16 Appendix F - Process for setting up new organic solvents in the DVS Advantage

16.1 Background and overview:

The DVS Advantage can work in two modes as far as vapour generation is concerned: closed or open loop. In closed loop, when the user asks for say, 30% Partial Pressure (pp), the mass flow controllers (MFCs) will initially provide 30% 'wet' flow, 70% 'dry' flow. The Dew Point Analyser (DPA) will then measure the actual value of the partial pressure generated, and feed this back into the software which will then automatically adjust the requests made to the MFCs, and the process repeated until 30% is measured by the DPA. In open loop mode, the process is the same, with the distinction that the DPA measured pp value is not fed back into the software for the purpose of making adjustments, such that asking for 30% pp will result in the MFCs generating 30% wet flow and 70% dry flow. The value is still recorded in the data file for the experiment, but no adjustments are made by the software based on its value.

In the open loop system, to compensate for variance from ideal mixing and the volume of the solvent vapour of high vapour pressure solvents, a partial pressure table is used by the software. In essence this acts as an offset function; at lower $\%p/p_0$ values where the generated $\%p/p_0$ is likely to be higher than the requested $\%p/p_0$, the offset will be negative; at higher $\%p/p_0$ where the generated $\%p/p_0$ is likely to be lower than the requested $\%p/p_0$, the offset will be positive. The value that the user inputs into the software is hereafter referred to as 'user requested value' the latter value from the partial pressure table is referred to as 'software requested value'.

Whilst for water, being a relatively low vapour pressure solvent, the offsets are relatively minor, for solvents with higher vapour pressures, such as Dichloromethane, the offset may be of the order of 5-10 %. i.e; at a requested %p/p_0 of, say, 10%, without use of this table the instrument will generate of the order of 15% depending on conditions. The %p/p_0 table would be set up such that when the user requests 10% (user requested value) the software in fact requests 5% (software requested value), with the end result that 10% is generated. More importantly than the size of the offset is that for new solvents, this table has to be set up.

It is also worth mentioning at this point the effect of flow rate on the absolute pp value. Although the influence of flow rate is likely to be subtle, it should however be considered if one intends to use a completely rigorous approach. Especially with high vapour pressure solvents, higher flow rates will increase the effect of evaporative cooling. This in turn will affect the saturated vapour pressure, as the temperature of the solvent will be slightly decreased, which in turn will affect the actual vapour pressure obtained.

The offset that is required in Open loop mode, and the pp feedback that occurs in closed loop mode both rely on the values of the pp that are obtained from the DPA. The DPA however, provides a temperature value, namely the dew point of the gas/solvent vapour mixture. The software calculates the pp from the dew point using the following equation:

$$\ln(p) = C_1 + C_2/T + C_3\ln(T) + C_4T^{C_5}$$

Where:

p = partial pressure in Pa,
T = dew point temperature in Kelvin, and
$C_{1\text{-}5}$ are 5 constants unique to the solvent.

The value of these constants can, for the most part be found in Table 2-6, Perry's Chemical Engineer's Handbook, 7th Edition, 1997, Eds: Robert H. Perry, Don W. Green, Pub: McGraw Hill.

In the event that your solvent is not listed within this book, you will need to obtain data points on the Temperature/saturated vapour pressure curve, and then fit this to the above equation to obtain values for the constants. Please contact the Application Science department of SMS in the UK office if you require help with this curve fitting.

16.2 Procedure

The procedure for setting up the partial pressure table for new solvents is as follows, note that this is only required if working in Open Loop mode:

1. Obtain the 5 constants either from curve fitting software and an appropriate data set of temperature/saturated vapour pressure, or from Perry's handbook.

2. (Refer Section 9.4.2)
   Bring up the **Edit Solvents** panel by clicking on **Solvents → Edit Solvents**. Click on the **New Solvent** button (the left of the two buttons) and complete the name and the 5 constants fields. Click **Done**.

3. Set up and run an experiment using that solvent. This is not absolutely necessary but will provide an idea of the magnitude of the changes to be made, and also serve as a check that the set of 5 constants used are giving sensible results.

4. When the experiment is finished, plot a graph of Time versus Actual and Target partial pressure, using the **Custom Plot** function of the isotherm macros within the DVS Analysis Suite.

5. Open the partial pressure table by clicking on **Calibration → Solvent Lookup Table Editor**.

6. The partial pressure table is constructed with user requested values of the partial pressure along the top row, in increments of 10%, Temperature in the first column from the left, in increments of 10°C, and flow rate in the second column from the left in increments of 50 sccm (see Figure 16.1). The main values of the table correspond to the temperature and flow rate as the first two columns to the left; the remaining numbers correspond to the actual values requested by the software when the values at the top of the column are input as target $\%p/p_0$. In order to make effective changes to this table, values need to be entered for both the temperature below and temperature above that at which you intend to work, and also at the different flow rates the you are likely to use.

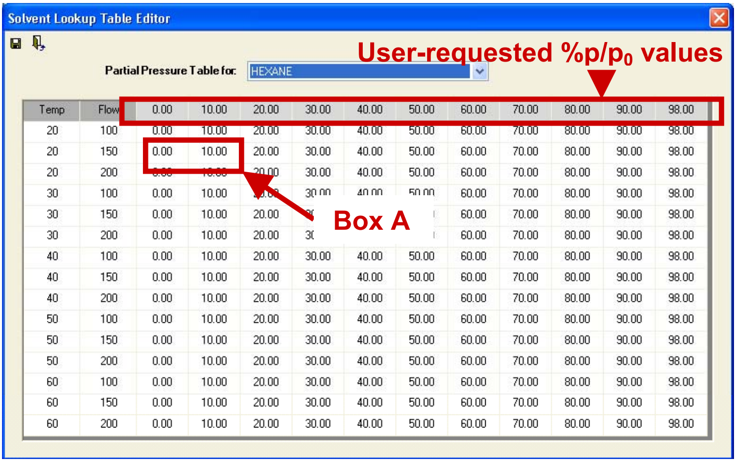


Figure 16.1: The **Solvent Lookup Table Editor**

For example if you were working at 25°C, at a flow rate of 200 sccm, you would need to ensure that the correct values were entered at both 20 and 30°C and at the flow rate of 200 sccm. At the very least changes made at one temperature should be mirrored at the other temperatures.

7. It is possible to measure the changes in real time, rather than shutting down and restarting the system to see the effect of any changes. This speeds up the process considerably.

8. This step refers to changing the numbers within the cells of the table. The cells in box A correspond respectively to the software requested values corresponding to a user requested values of 0%, and 10% at 20°C and a flow rate of 150 sccm. Obviously for the value for 0%, the instrument will never be able to generate less than that value, so this value in the left cell of Box A will always remain at 0%. To correct the value next to it, currently set at 10 %, refer to the

value obtained in the actual/target PP plot for a target $\%p/p_0$ of 10%. Subtract the target $\%p/p_0$ from the actual $\%p/p_0$ value to give the difference between the two values; subtract this difference from 10 and enter this value into the cell. For example, if in your experiment when the target pp was 10%, the actual $\%p/p_0$ value was 13%, the number to enter into the cell within the table is 7.

9. Close the Solvent Lookup Table Editor, saving any changes if prompted, and return to the real time measurement window. Give the instrument a few minutes for the new values to take effect and reach equilibrium, then take note of the new value and use that to fine tune the value input into the partial pressure table- a further iteration may be required.

10. Repeat this process for all cells within the range 10-98. It may not be possible to quite reach the maximum value of the pp depending on the solvent in question due to evaporative cooling.

11. Set up and run another experiment using the solvent, to act as a check for the values entered.

Your table should now be set up and your new solvent values entered correctly.

17 Appendix G - Software Installation/Removal

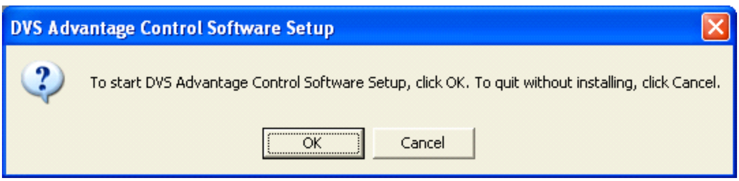
17.1 Software Installation

Prior to installation, the user must ensure that the computer system meets the minimum system requirements (Section 5).

The software installation procedure consists of the 2 steps listed below:

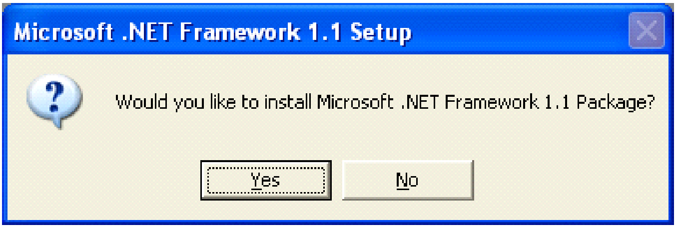
17.1.1 Installing the DVS Advantage Software

1. Insert the DVS Advantage Software CD into the CD-Drive.
2. Browse to the **Control Software** folder on the installation CD.
3. Double click the **Setup.exe** file and the following prompt will appear:



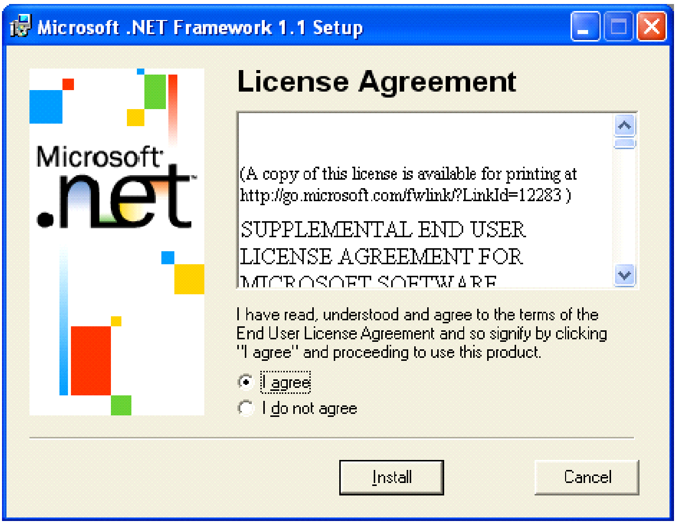
4. Click the **OK** button to start installation of the DVS Advantage Control Software.
5. The installation wizard will then perform a check to see if Microsoft .NET Framework v1.1 is installed.

6. If the installation wizard detects that Microsoft .NET Framework v1.1 is installed then the process will immediately proceed to step 13. If the installation wizard detects the Microsoft .NET Framework v1.1 is NOT installed the following steps will occur:



7. Click the **Yes** button.

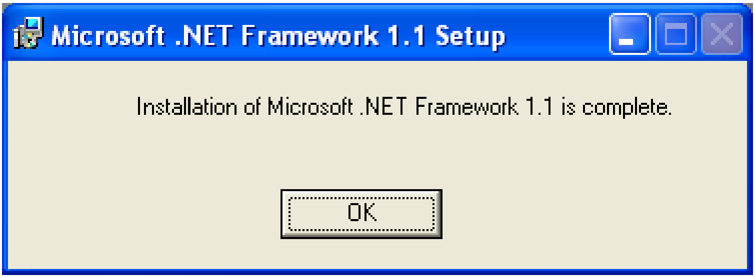
8. The Microsoft .NET Framework v1.1 License Agreement confirmation panel will then appear:



9. Select the **"I agree"** option and click the **Install** button.

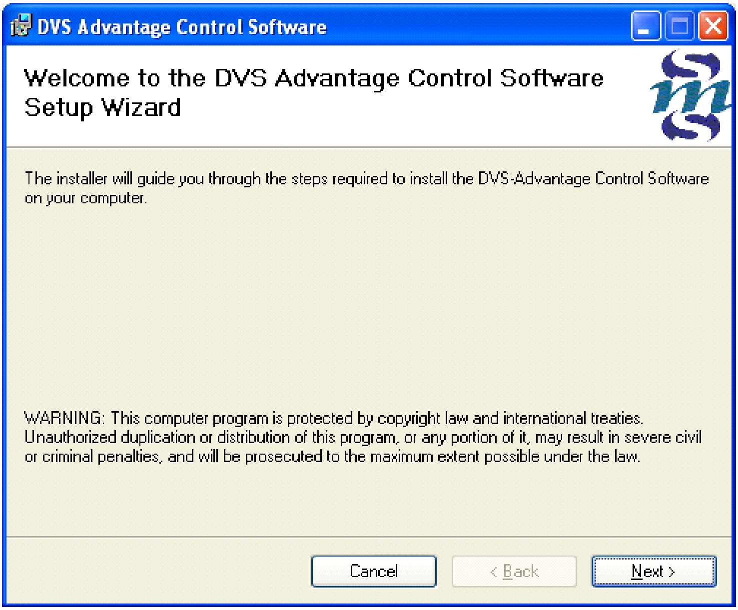
10. The installation wizard will then install the .NET framework.

11. Once the installation of the .NET Framework has completed the following notification will appear:

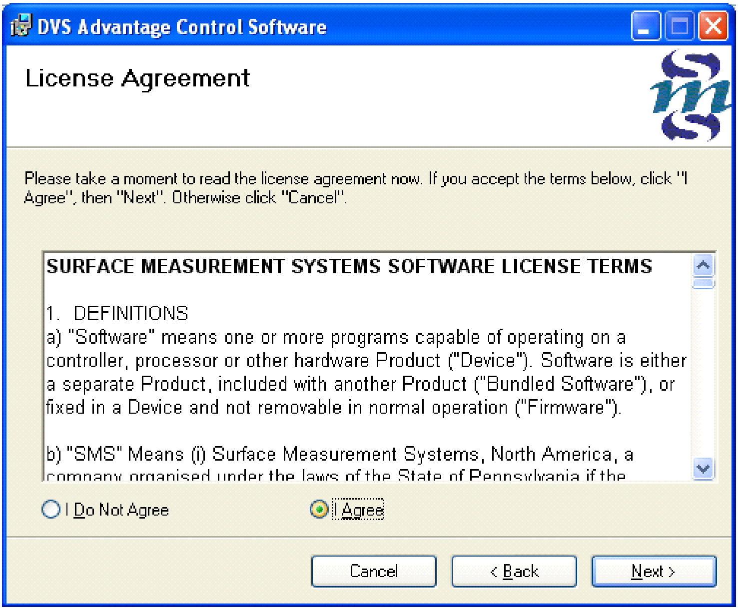


12. Click the **OK** button.

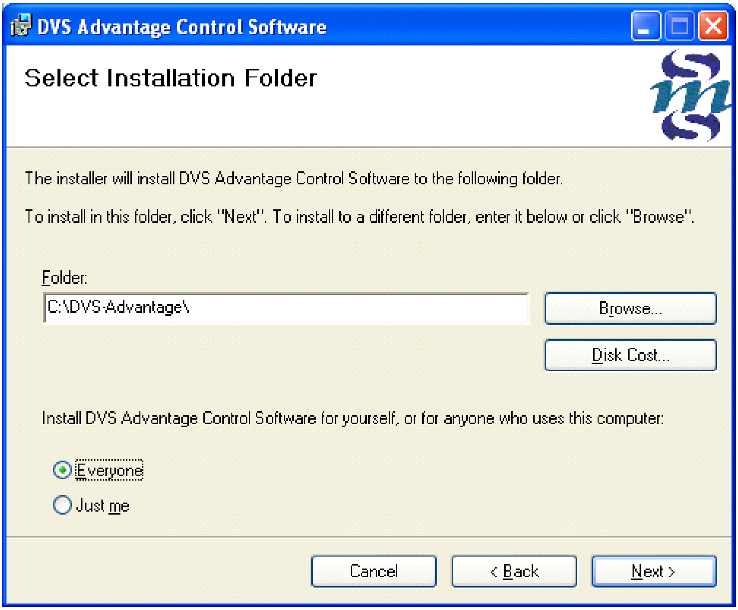
13. The DVS Advantage Control Software Setup Wizard will then appear:



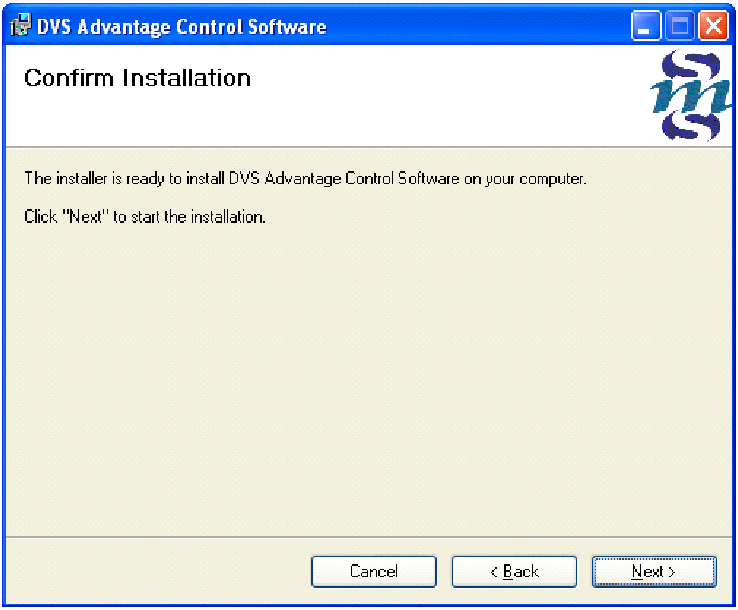
14. Click the **Next** button.
15. The **DVS Advantage Software License Agreement** confirmation screen will then appear:



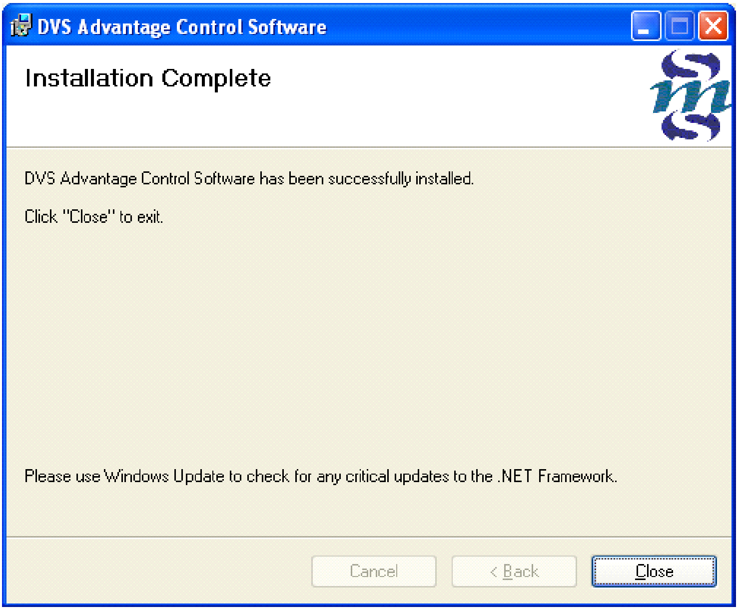
16. Select the **I Agree** option and then click the **Next** button.
17. The **Select Installation Folder** screen will then appear:



18. Ensure the **Everyone** option is selected.
19. The default installation folder is: “C:\DVS-Advantage” and it is strongly suggested that this is not changed.
20. Click the **Next** button.
21. The **Confirm Install** screen will then appear:



22. Click the **Next** button to install the software.
23. Once the software has completely installed the following screen will be shown:



24. Click the **Close** button.
25. A final success confirmation message will then appear:

26. Click **OK**.
27. The installation of the DVS Advantage Control Software is now complete.

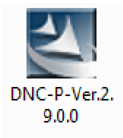
17.1.2 Installing the Dino-Lite USB camera

Note: Please ensure the Dino-Lite Digital Microscope USB cable is not connected to the PC USB port before starting the installation process.

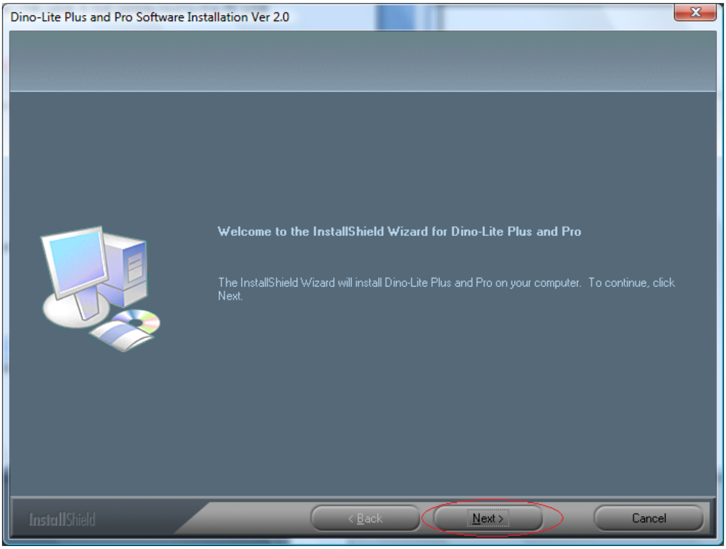
17.1.2.1 Microsoft Windows Vista™

1. Switch on the PC and insert the DVS Advantage Software CD is in the CD-drive.

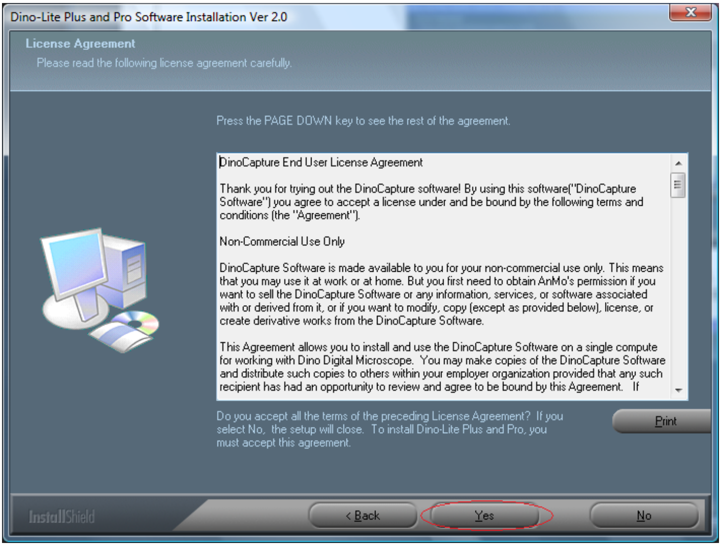
2. Browse to the **Support Installation Files\Dino-Lite Driver 2.9.0** folder on the installation CD. Double click the **DNC-P-Ver.2.9.0.0.exe** program to start the installation process.



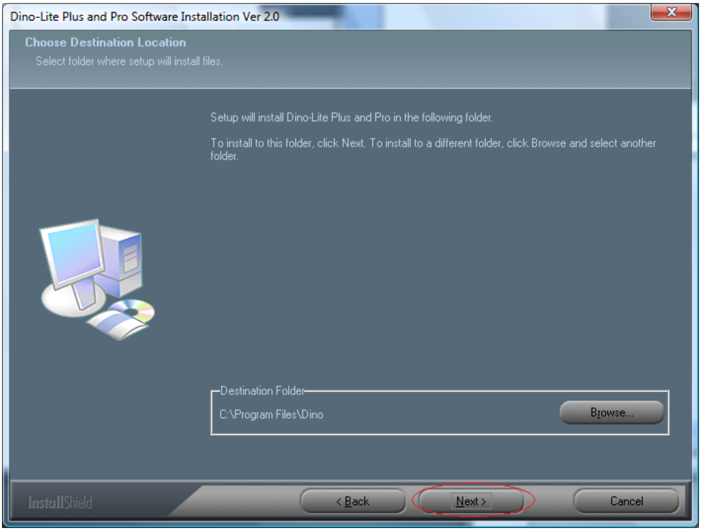
3. The Installation Wizard will appear. Click the **Next** button at the bottom of the dialog.



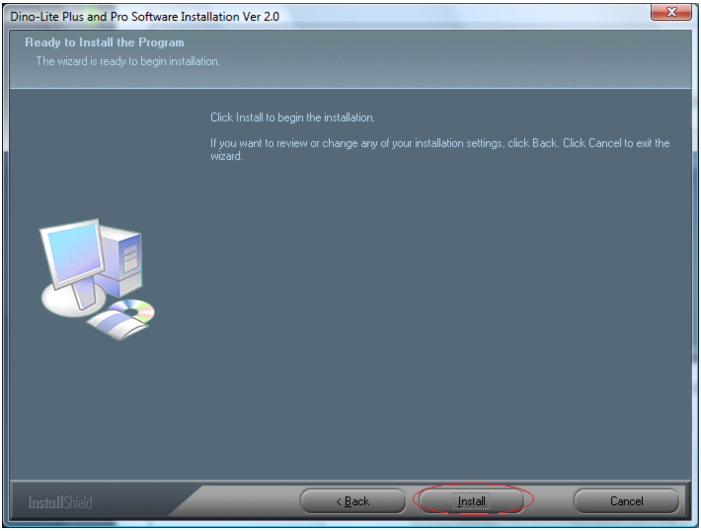
4. Please read and accept the **DinoCapture End User License Agreement** by clicking the **Yes** button.



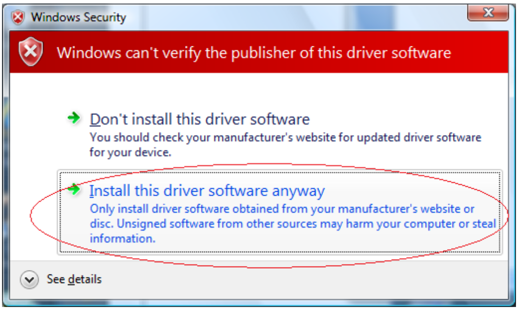
5. In the **Choose Destination Location** windows, click the **Browse** button to the select your own destination folder or click the **Next** button to continue.



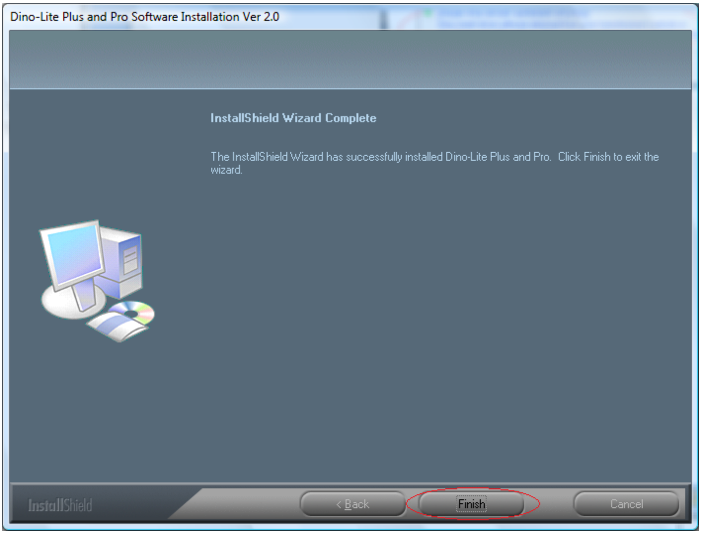
6. In the **Ready to Install the Program** windows, click the **Install** button to start the installation process.



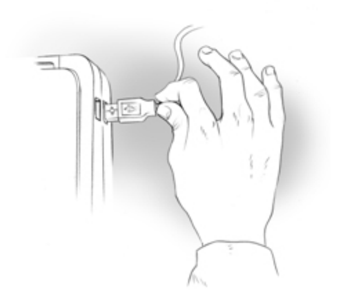
7. When the **Windows Security** warning dialog appears, click the **Install this driver software anyway** option to continue.



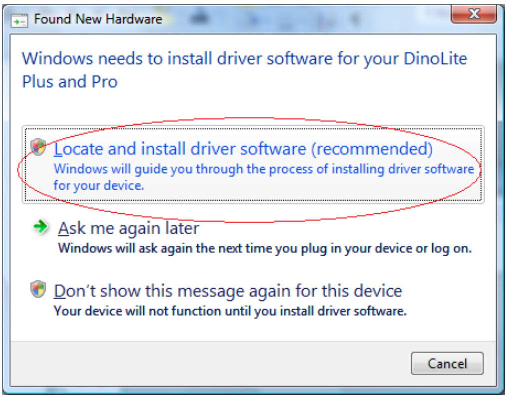
8. Once the installation process is completed, the **InstallShield Wizard Complete** windows will appear. Click the **Finish** button to continue.


Dino-Lite Plus and Pro Software Installation Ver 2.0
InstallShield Wizard Complete
The InstallShield Wizard has successfully installed Dino-Lite Plus and Pro. Click Finish to exit the wizard.
InstallShield
< Back
Finish
Cancel

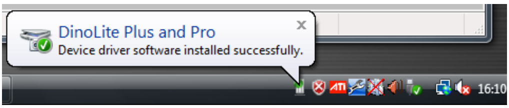
9. Connect the Dino-Lite Digital Microscope USB cable to the PC USB port.



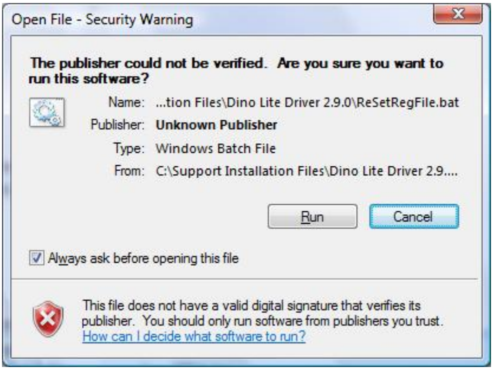
10. The **Found New Hardware** windows will appear. Click on the **Locate and install driver software (recommended)** option.



11. When the hardware is successfully installed the tooltip message will appear at the bottom of the screen



12. Ensure that the DVS Advantage Software CD is in the CD-drive. Browse to the **Support Installation Files\Dino-Lite Driver 2.9.0** folder on the installation CD. Double click the **ReSetRegFile.bat** file and the following dialog will appear:



13. Click the **Run** button to complete the installation process.

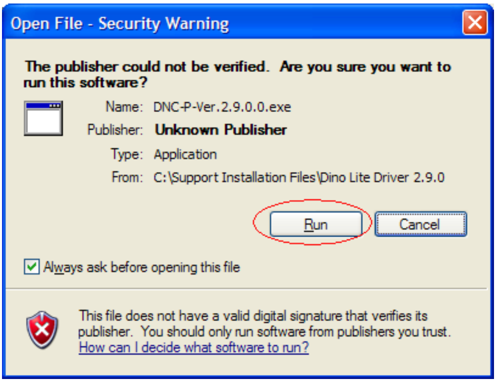
Note: *If the Windows Vista User Account Control appears during the installation process, enter the username and click* ***OK*** *or if the current user is an administrator then click* ***Continue****.*

17.1.2.2 Microsoft Windows XP

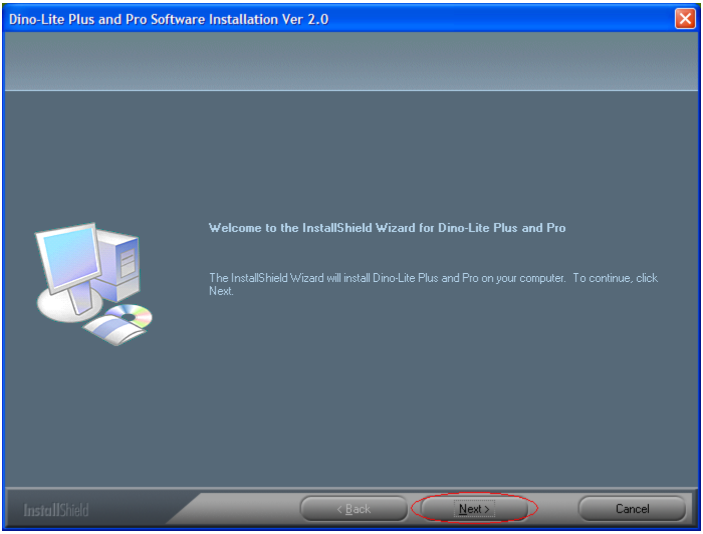
1. Switch on the PC and insert the DVS Advantage Software CD is in the CD-drive.

2. Browse to the **Support Installation Files\Dino-Lite Driver 2.9.0** folder on the installation CD. Double click the **DNC-P-Ver.2.9.0.0.exe** program to start the installation process.

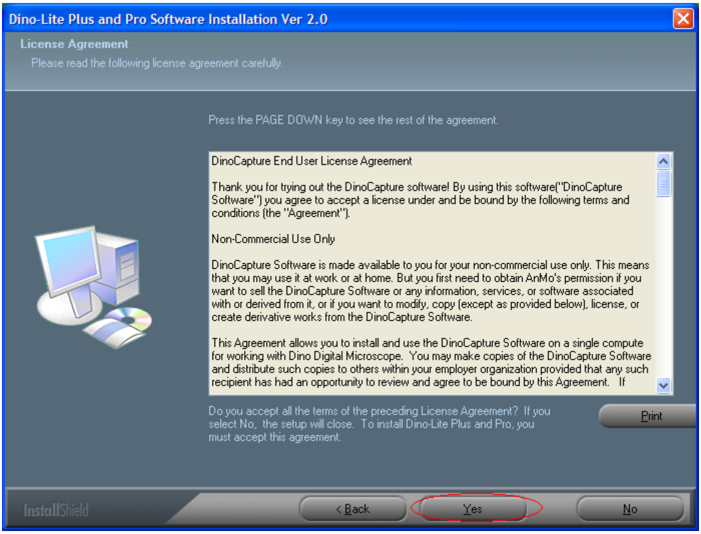
3. In the **Open File – Security Warning** dialog, click the **Run** button to proceed.



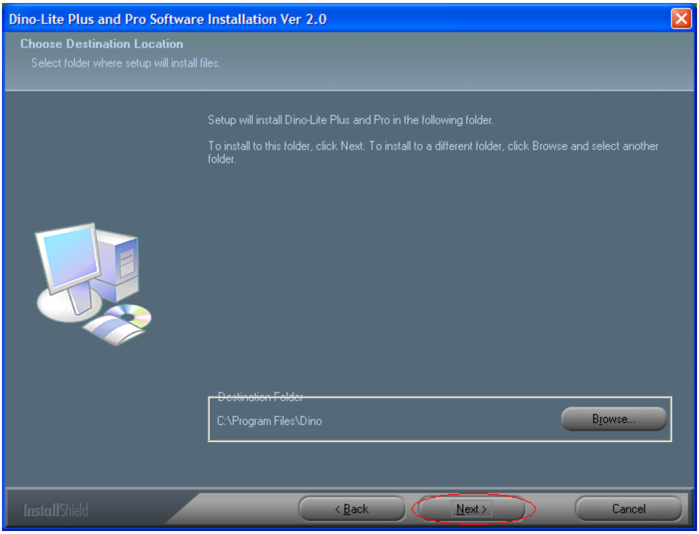
4. The Installation Wizard will appear. Click the **Next** button at the bottom of the dialog.



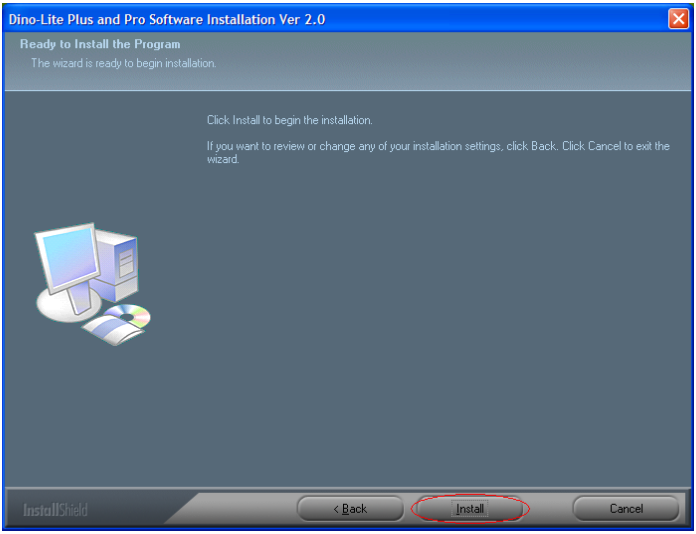
5. Please read and accept the **DinoCapture End User License Agreement** by clicking the **Yes** button.



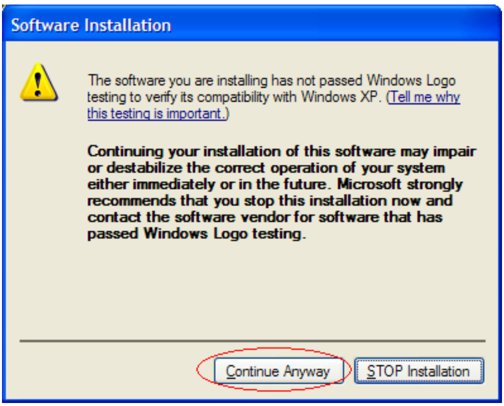
6. In the **Choose Destination Location** windows, click the **Browse** button to the select your own destination folder or click the **Next** button to continue.



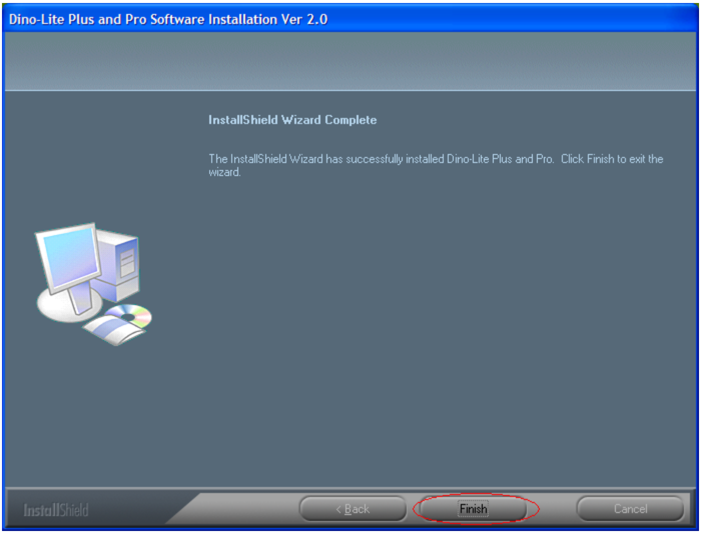
7. In the **Ready to Install the Program** windows, click the **Install** button to start the installation process.



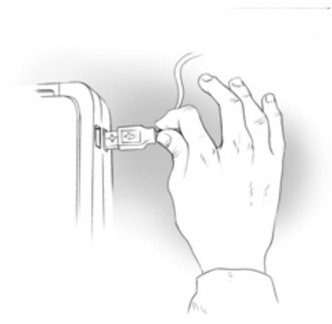
8. When the **Software Installation** warning dialog appears, click the **Continue Anyway** button to continue.



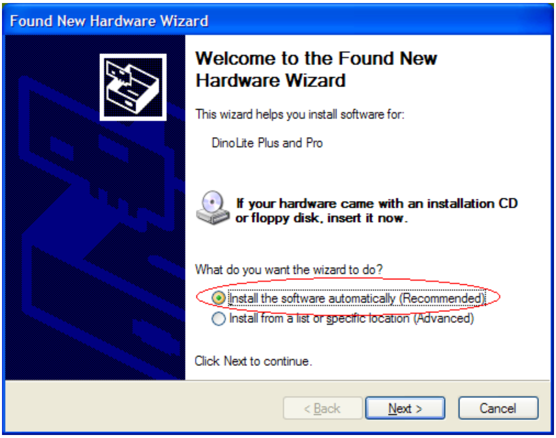
9. Once the installation process is completed, the **InstallShield Wizard Complete** windows will appear. Click the **Finish** button to continue.



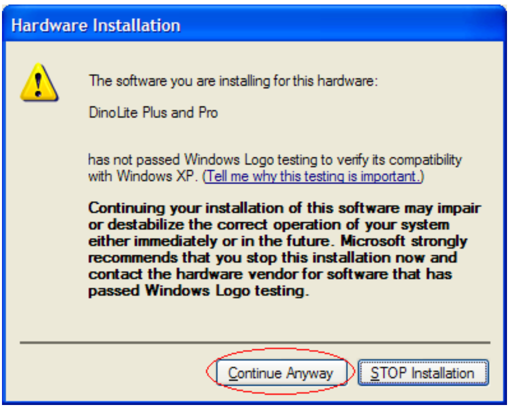
10. Connect the Dino-Lite Digital Microscope USB cable to the PC USB port.



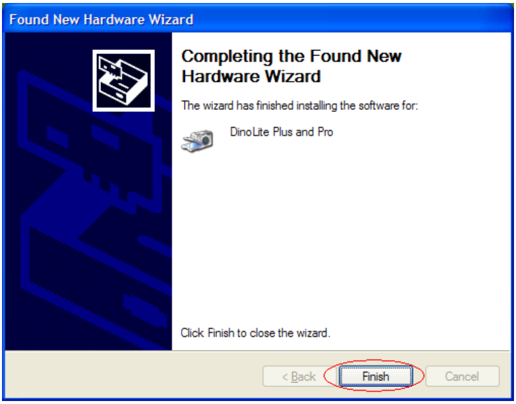
11. The **Found New Hardware Wizard** will appear. Check the **Install the software automatically (Recommended)** option. Click the **Next** button to continue.



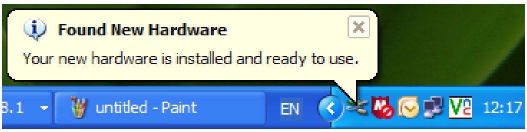
12. When the **Hardware Installation** warning dialog appears, click the **Continue Anyway** button to continue.



13. When the hardware is successfully installed the **Found New Hardware Wizard** will appear. Click the **Finish** button to complete the installation process.



14. A tooltip message will appear at the bottom of the screen to acknowledge a successful hardware installation.



17.1.3 Configure the Dino-Lite camera

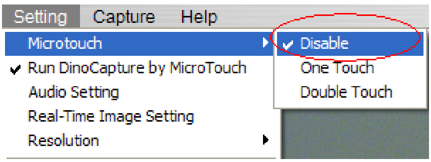
This section only applies to the Dino-Lite *Microtouch* version of the camera.

1. Switch on the PC and ensure that the Dino-Lite Digital Microscope USB cable is connected to the PC USB port.

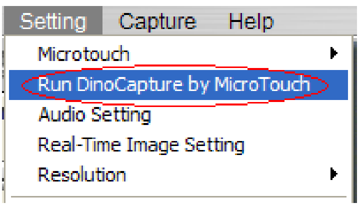
2. On the Windows Desktop, double click the DinoCapture icon to open the **Dino-Lite Capure** application.

3. On the Dino-Lite camera, press the **Microtouch** button at the bottom of the camera.

4. On the DinoCapture application, select **Setting → Microtouch → Disable** to disable the Microtouch feature.



5. On the main menu again, select **Setting → Run DinoCapture by MicroTouch** to disable this feature.



17.1.4 Additional notes for IT Administrators for Windows Vista computers

Once the DVS Advantage Control Software has been installed and configured, users can log in to the Windows Operating System using their personal Windows user account, however there are several issues that IT Administrators may need to address:

17.1.4.1 Running the DVS Advantage software for the first time

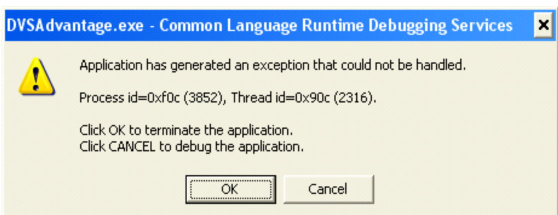
In some cases when the DVS Advantage Control Software is run for the first time on a Windows user account the DVS Advantage Control Software Installation Wizard will start to run and may request the installation CD to be inserted into the CD-ROM.

In this case the user account requires Local Administrative rights to the DVS Advantage computer to allow the Installation Wizard to install missing components from the installation CD. Once the Installation Wizard has completed, the Local Administrative rights to the DVS Advantage computer are no longer required and may be removed.

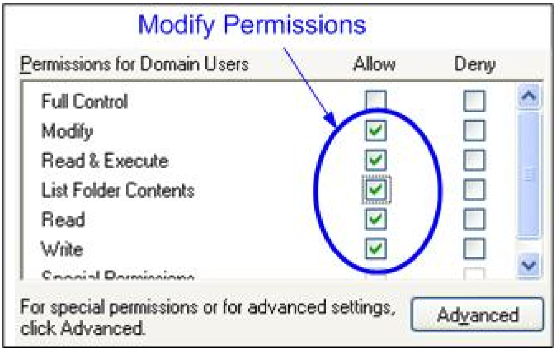
17.1.4.2 User Security Permissions

All Windows user accounts used to run the DVS Advantage Control Software require **Modify** permissions on the installation directory and all files and folders within it.

Note: If users do NOT have the required security permissions the following error message will appear:



Permissions for all users should be configured on the installation directory as shown below:



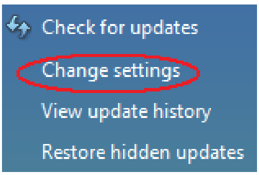
17.1.4.3 Disable Automatic Updates

The installation of software downloaded by the Windows Automatic Update feature can interfere with the operation of the DVS Advantage control software.

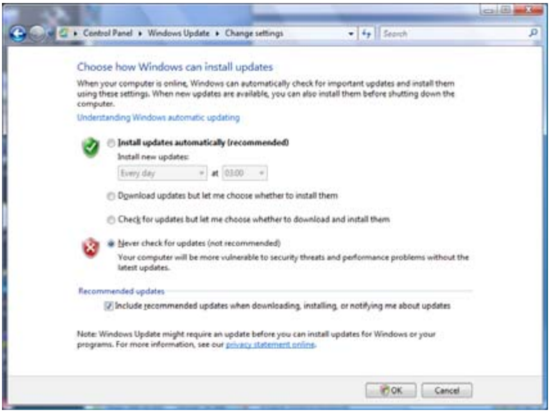
We suggest that the Automatic Updates are turned off and that any updates are performed when the DVS Advantage control software is not running.

To turn off Automatic Updates follow these steps:

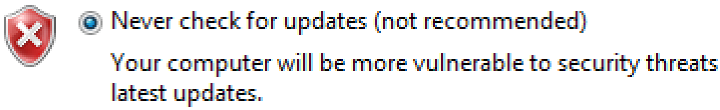
Open the Windows Control Panel and double-click on the **Windows Update** option. In the **Windows Update** dialog box, click on the Change settings option on the left panel.



The following dialog box will appear:



In the Windows Update dialog box, please ensure that the **Never check for updates** option is enabled.



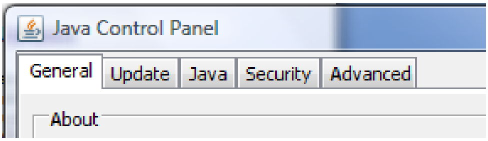
Click on the **OK** button to accept the changes. (*If the Windows Vista User Account Control appears, enter the administrator's detail and click **OK** or if the current user is an administrator then click **Continue**.)*

17.1.4.4 Disable Java Updates

Similar to Automatic Updates, Java updates can also interfere with the operation of the DVS Advantage control software. SMS suggests that Java updates are turned off and any desired updates are applied when the DVS Advantage control software is not running.

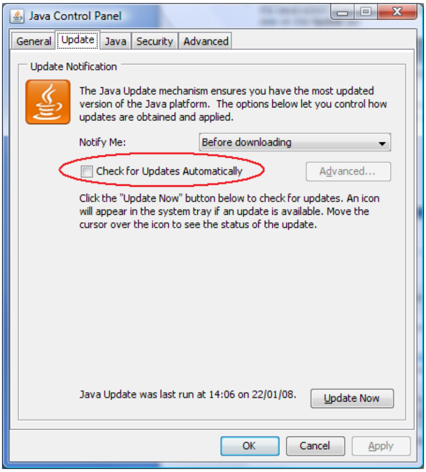
To switch off Java updates following these steps:

Open the Windows Control Panel and double-click on the **Java** option. In the Java Control Panel dialog box, click on the **Update** tab.

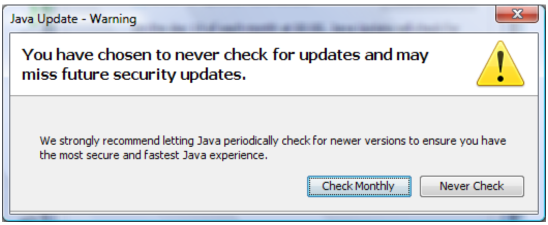


In the Java Control Panel dialog box, click on the **Update** tab.

In the Update option, please ensure that the **Check for Updates Automatically** option is unchecked.



Click the **Never Check** button option in the following dialog box:



Click on the **OK** button to accept the changes.

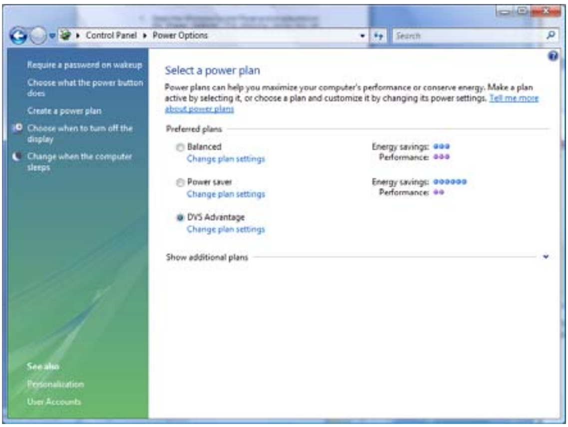
17.1.4.5 Update Power Options Features

Hibernation of the computer prevents the DVS Advantage control software from communicating with the DVS Advantage instrument.

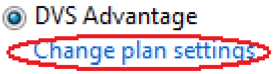
If hard disks are switched off at any point it will mean that the DVS Advantage control software cannot save experimental data to the hard disk.

You must ensure that the **each Windows user account** that will be used is updated so that it has the settings listed below:

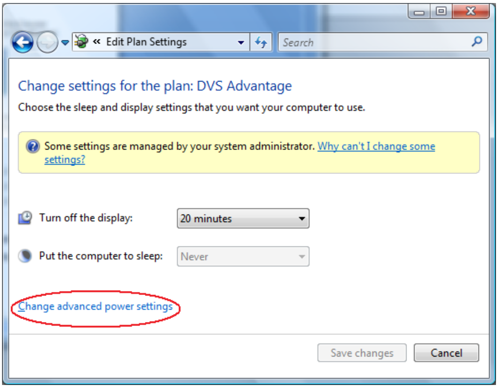
Open the Windows Control Panel and double-click on the **Power Options**. The following dialog box will appear:



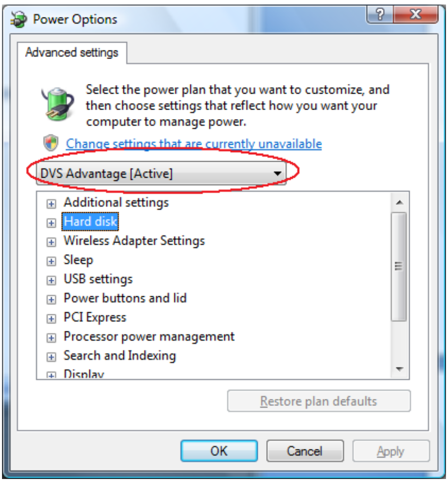
Click the on **DVS Advantage Change plan settings** option.



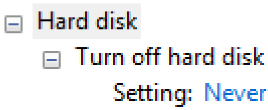
In the Edit Plan Settings window, click on the **Change advanced power settings** option.



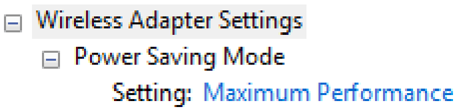
In the Power Options window, ensure that the DVS Advantage setting is active.



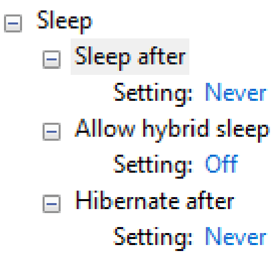
In the **Hard disk** selection, ensure that all the **Turn off hard disk after** options are set to **0** i.e. **Never**.



In the **Wireless Adapter Settings**, ensure that all the **Power Saving Mode** options are set to **Maximum Performance**.



Click on the **Sleep** settings and set all the **Sleep after** options to **Never**. Set all the **Allow hybrid sleep** options to **Off** and set all the **Hibernate after** options to **Never**.

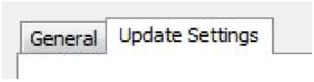


17.1.4.6 InstallShield Update Manager

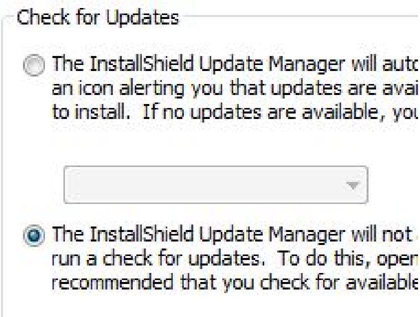
If this application is installed on the computer, you will need to ensure that updates for the program do not affect the operation of the DVS Advantage control software by ensuring the following settings are used:

Open the Windows Control Panel and double-click on the **Program Updates** options (if it exists).

In the Update Manager dialog box, click on the **Update Settings** tab.



Ensure that the automatically check for updates option is disabled.

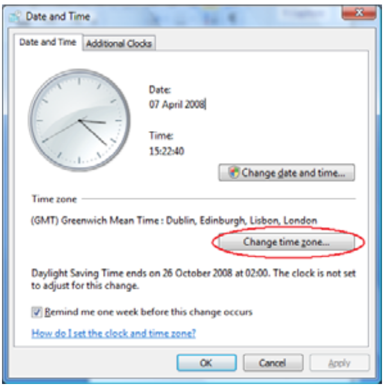


Click the **OK** button.

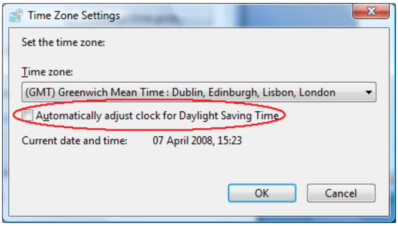
17.1.4.7 Date and Time Settings

An error will occur if the date or time is changed whilst the DVS Advantage control software is running.

In the Control Panel, select the **Date and Time** options. Click on the **Change time zone** button.

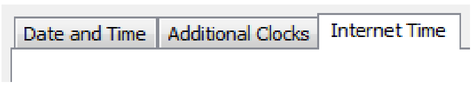


In the Time Zone settings, ensure that the **Automatically adjust clock for Daylight Saving Time** option is unchecked.



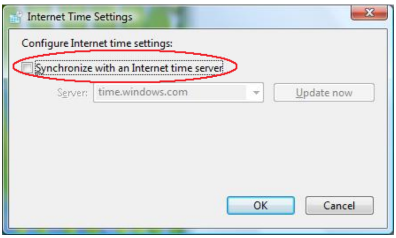
1. Click the **OK** button to continue.

2. Select the **Internet Time** tab (if visible)



5. Click on the **Change settings** button. (*If the Windows Vista User Account Control appears, enter the administrator's detail and click **OK** or if the current user is an administrator then click **Continue**.)*

6. Ensure that the **Synchronize with an Internet time server** option is unchecked.



7. Click the **OK** button to exit.

17.1.5 Additional notes for IT Administrators for Windows XP computers

Once the DVS Advantage Control Software has been installed and configured, users can log in to the Windows Operating System using their personal Windows user account, however there are several issues that IT Administrators may need to address:

17.1.5.1 Running the DVS Advantage software for the first time

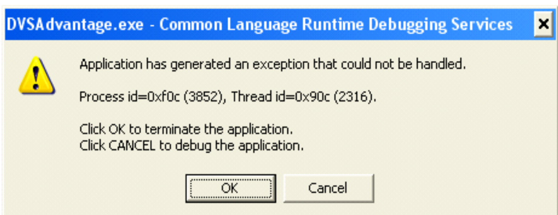
In some cases when the DVS Advantage Control Software is run for the first time on a Windows user account the DVS Advantage Control Software Installation Wizard will start to run and may request the installation CD to be inserted into the CD-ROM.

In this case the user account requires Local Administrative rights to the DVS Advantage computer to allow the Installation Wizard to install missing components from the installation CD. Once the Installation Wizard has completed, the Local Administrative rights to the DVS Advantage computer are no longer required and may be removed.

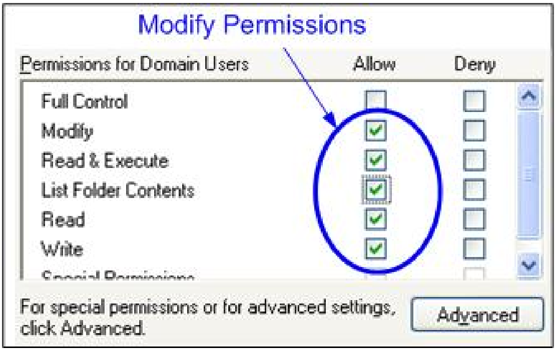
17.1.5.2 User Security Permissions

All Windows user accounts used to run the DVS Advantage Control Software require **Modify** permissions on the installation directory and all files and folders within it.

Note: If users do NOT have the required security permissions the following error message will appear:



Permissions for all users should be configured on the installation directory as shown below:



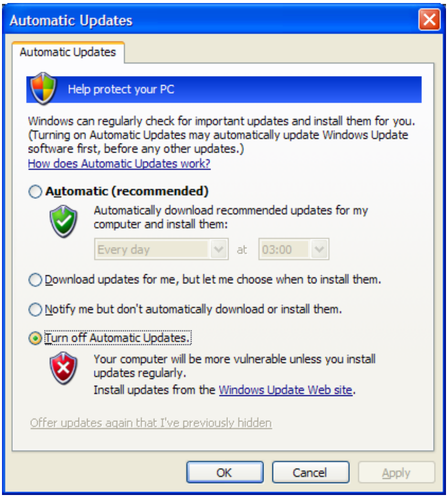
17.1.5.3 Disable Automatic Updates

The installation of software downloaded by the Windows Automatic Update feature can interfere with the operation of the DVS Advantage control software.

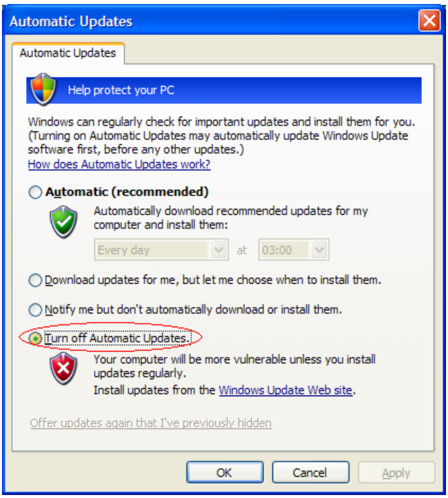
We suggest that the Automatic Updates are turned off and that any updates are performed when the DVS Advantage control software is not running.

To turn off Automatic Updates follow these steps:

Open the Windows Control Panel and double-click on the **Automatic Updates** option. The following dialog box will appear:



In the Automatic Updates dialog box, please ensure that the **Turn off Automatic Updates** option is enabled.



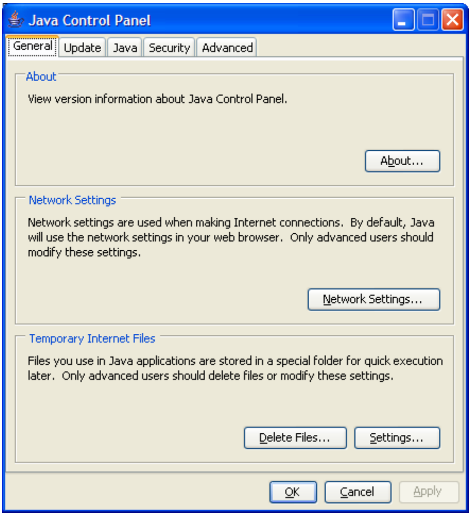
Click on the **OK** button to accept the changes.

17.1.5.4 Disable Java Updates

Similar to Automatic Updates, Java updates can also interfere with the operation of the DVS Advantage control software **if it is installed**. SMS suggests that Java updates are turned off and any desired updates are applied when the DVS Advantage control software is not running.

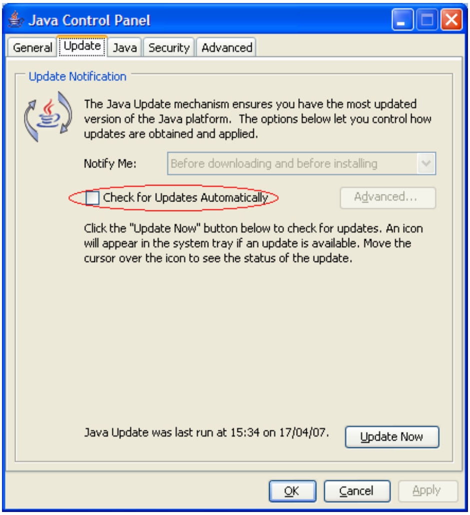
To switch off Java updates following these steps:

Open the Windows Control Panel and double-click on the **Java** option (if it exists). The following dialog box will appear:



In the Java Control Panel dialog box, click on the **Update** tab.

In the Update option, please ensure that the **Check for Updates Automatically** option is unchecked.



Click on the **OK** button to accept the changes.

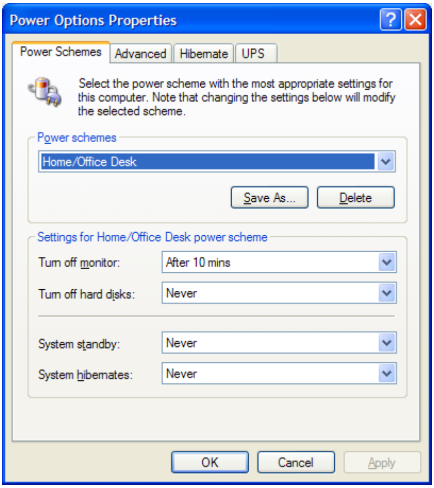
17.1.5.5 Update Power Options Features

Hibernation of the computer prevents the DVS Advantage control software from communicating with the DVS Advantage instrument.

If hard disks are switched off at any point it will mean that the DVS Advantage control software cannot save experimental data to the hard disk.

You must ensure that the **each Windows user account** that will be used is updated so that it has the settings listed below:

Open the Windows Control Panel and double-click on the **Power Options**. The following dialog box will appear:

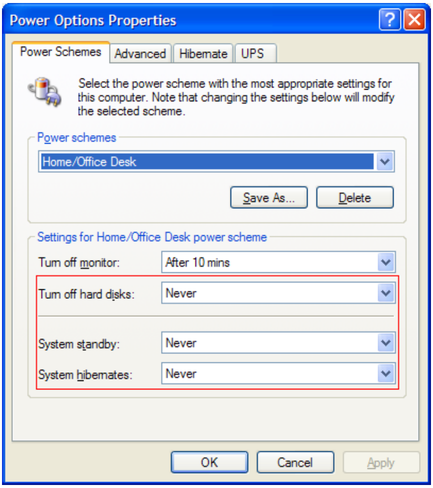


The **Power Schemes** tab should be active. Ensure that the Power schemes are set to the following configuration:

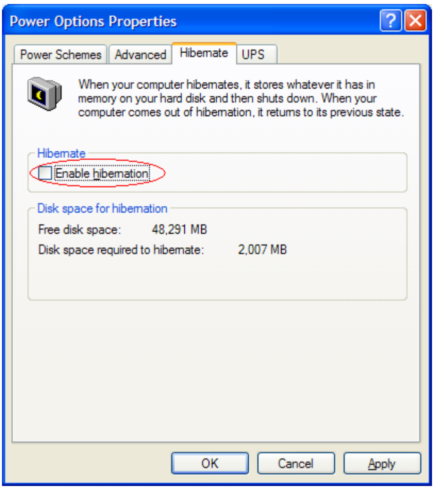
Turn off hard disks: *Never*

System standby: *Never*

System hibernates: *Never*



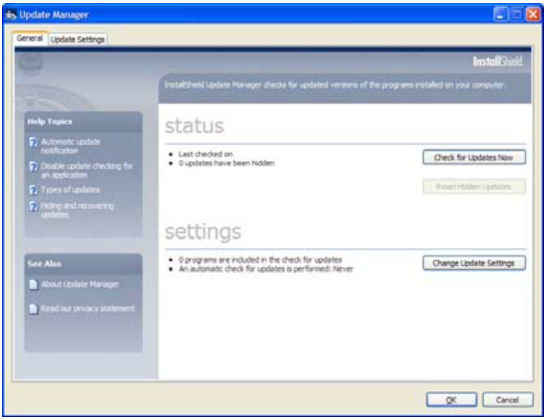
Now select the **Hibernate** tab and ensure that the **Enable hibernation** option is unchecked.



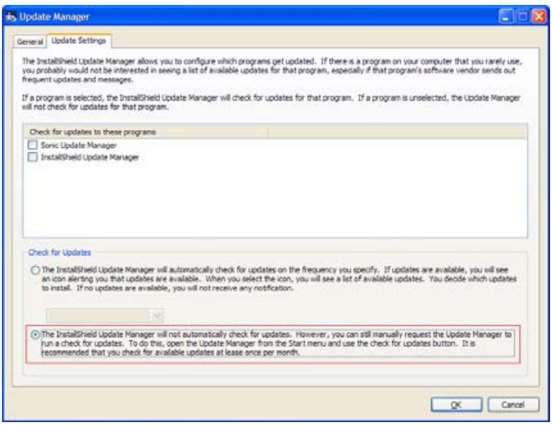
17.1.5.6 InstallShield Update Manager

If InstallShield is installed on the PC, ensure that updates for InstallShield do not affect the operation of the DVS Advantage control software by ensuring the following settings are used:

Open the Windows Control Panel and double-click on the **Program Updates** options (if it exists). The following dialog box will appear:



In the Update Manager dialog box, click on the **Update Settings** tab and ensure that the automatically check for updates option is disabled



17.1.5.7 Daylight Savings

An error will occur if the date or time is changed whilst the DVS Advantage control software is running.

Ensure the **"Automatically adjust clock for daylight saving changes"** setting of the computer is unchecked.

17.1.5.8 SMTP Email Configuration

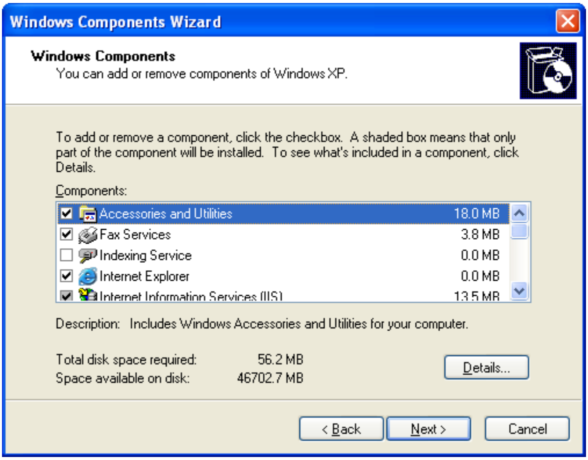
Note: The Email Settings feature is not available in 21 CFR Part 11 versions of DVS Advantage software.

To allow users of the DVS Advantage Control Software to use the SMTP email functionality in the DVS Advantage software, the following steps need to be performed:

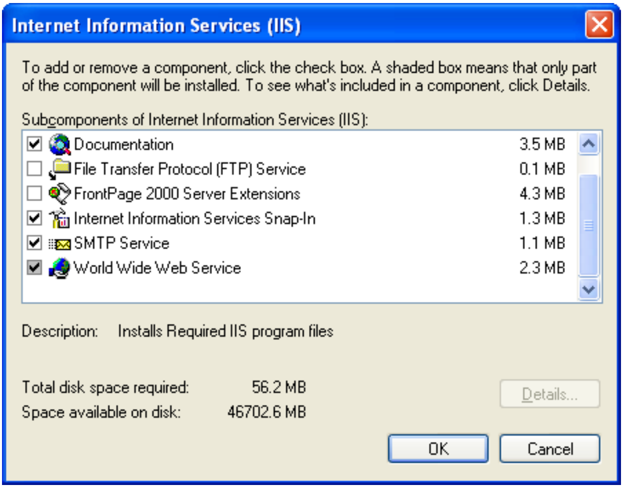
Ensure the SMTP Service is installed

1. Open the Windows **Control Panel**, double-click the **Add or Remove Programs** icon.

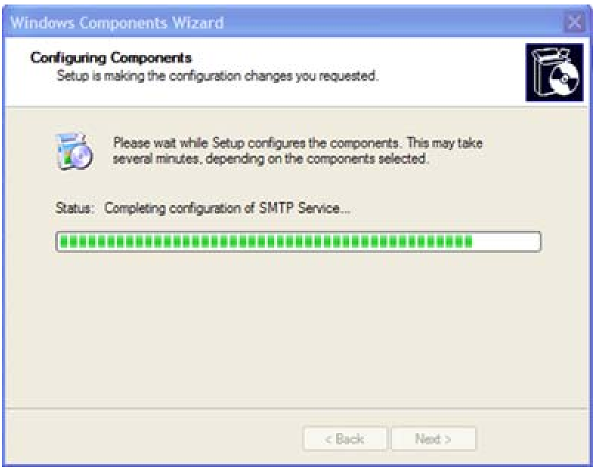
2. In the Add or Remove Programs panel, click **Add/Remove Windows Components** at the left side bar menu option. The following dialog will appear:



3. In the Windows Components Wizard panel, ensure the **Internet Information Services (IIS)** option is checked.
4. Click the **Details…** button and the following dialog will appear:



5. In the Internet Information Services (IIS) panel, ensure the **SMTP Service** option is checked.
6. Click the **OK** button to confirm selection.
7. In the Windows Components Wizard, click the **Next** button to proceed.
8. Insert the Window XP Professional CD if prompted.

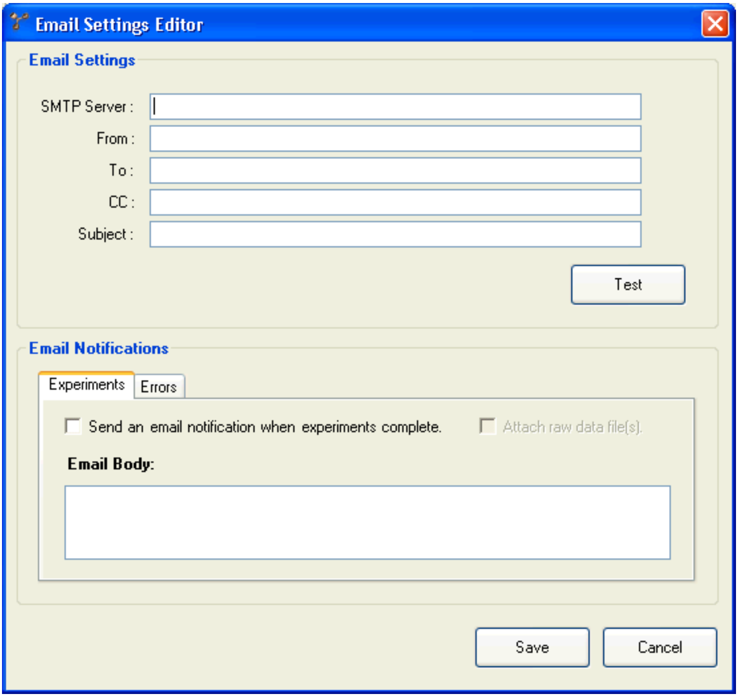


9. Click the **Finish** button when the Windows Components Wizard successfully completed the installation process.

Configure the SMTP Server name

1. To configure the SMTP Server name start up the DVS Advantage Control software and select the File→Preferences→Email Settings… menu item.

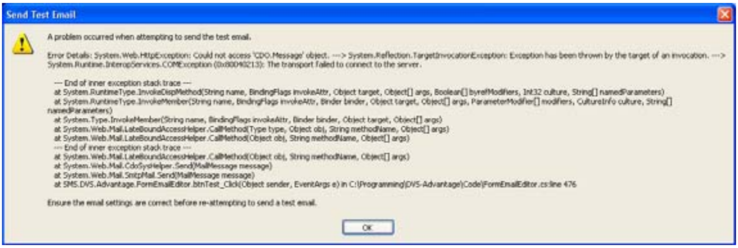
2. The dialog below will then appear:



SMTP Server: This needs to be set to the name of the e-mail relay server to be used. If the **SMTP Server** field is left blank, the name of the local SMTP server is used.

3. A test email can be sent by clicking on the **Test** button.

4. If the SMTP Server is not detected the warning message below will appear when a Test email is sent:



5. Upon correctly setting the SMTP Server name, click the Save button to save this name for all users of the DVS-Advantage instrument.

6. Shutdown the DVS Advantage software.

For information on the use of the Email function, please refer to the related section in this manual.

Remarks

For information about configuring the local SMTP server, see the articles "Manage Your Company's E-mail with the Windows 2000 SMTP Service" and "Using SMTP for Outgoing Messages" in the MSDN library at http://msdn.microsoft.com.

17.2 Uninstalling the DVS Advantage Software

When removing the DVS Advantage Control Software, please follow the 2 procedures listed below:

17.2.1 Backup Important Files

1. Backup important DVS Advantage system files.

Before uninstalling the DVS–Advantage software, make sure **a backup copy of the following folders and all the files they contain is made**:

   a. C:\DVS-Advantage\config

   b. C:\DVS-Advantage\database

These folders contain both the system and calibration settings for the DVS instrument.

If these files are lost then the DVS Advantage instrument will need to be re-calibrated by a SMS service engineer!!

2. Backup existing Sequence, Method and Data files stored in the following directories:

   a. C:\DVS-Advantage\method

   b. C:\DVS-Advantage\sequence

   c. C:\DVS-Advantage\data

> **Note:** DVS-Advantage sequence, method (.sao & .pre) and data files (.dat and .xls) are often reusable in new versions of the DVS–Advantage software. This means that if a new version of DVS–Advantage software is installed, these files may later be copied to the sequence, method and data folders of the new DVS Advantage software version.

17.2.2 Uninstall the DVS–Advantage software

1. Make sure that the **DVS Advantage system files** listed in the section above **have been backed up**.
2. Ensure that you are logged into the computer as an Administrator.
3. Start the Windows Control Panel.
4. Double click the **Add/Remove Programs** icon.
5. Select the **DVS Advantage Control Software** application.
6. Click the **Remove** button.
7. Click the **YES** button when prompted for Removal confirmation.

Once the Removal is complete, using Windows Explorer, browse to the installation folder and delete the folder if it still exists.

18 Appendix H – Customer Support

If you experience any problems with this product please contact your local supplier.

Contact SMS at the following locations –

United Kingdom (Worldwide Office)
www.smsuk.co.uk
Surface Measurement Systems Ltd UK
5 Wharfside
Rosemont Road
Alperton HA0 4PE
Phone: +44 020 8 795 9400
Fax: +44 020 8 795 9401
E-mail support@smsuk.co.uk

North America
www.smsna.com
Surface Measurement Systems NA
2125 28th Street SW
Suite 1
Allentown PA 18103
Phone: (610) 798 8299
Fax: (610) 798 0334
E-mail support@smsna.com