DVS Standard Analysis Suite
Version 7.6

User Manual

Revision 1

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2 Introduction

The DVS Standard Analysis Suite is designed to rapidly analyse and present raw DVS data.

Employing simple user interfaces, the software creates useful curves and reports, allowing the user to quickly and easily assess the results of a DVS experiment.

In addition to data plotting and analysis, the Analysis Suite includes several modules that enable the user to perform diagnostic tests upon the DVS to assess the system's performance.

This user manual is designed to help the user understand the basic operational principles of the Standard Analysis Suite, and how to use the system to gain efficient use of the software.

However, if some questions remain unanswered please do not hesitate to contact either your SMS representative or SMS directly. Also available for on-line customer support are the SMS application notes that can be found at: http://www.thesorptionsolution.com.

Note: The pictures below show the DVS Version Option Panel. When the s/w is initially installed and only one type of file format is being used the DVS Instrument Info Panel is shown instead.

3 General Functionality

The DVS Analysis Suite Standard Edition comprises the following modules:

DVS Plot Manager

DVS Isotherm Analysis

DVS Baseline Correction

DVS Vapour Content Offset

DVS Salt Validation Calibration

DVS Partial Pressure Check

DVS Drift and Noise Check

DVS Service Baseline Check

DVS Method Report

DVS Config

DVS Analysis Suite Info

The modules listed above are accessible via the Analysis Suite toolbar (Figure 3.1 below). The toolbar appears in Excel following the correct installation of the Analysis Suite (see Appendix A – Installation Instructions).

Figure 3.1: The DVS Analysis Suite (Standard) toolbar

The toolbar will appear in the Add-Ins section of the Office Ribbon - it cannot be dragged out of that section to float above any part of the spreadsheet or to any other area at the top or side of the screen.

The buttons on the toolbar represent the modules listed above.

To start a module, click the appropriate button on the toolbar. The main dialog screen for the module will be displayed. To close the module and return to the normal Excel environment, click on the **Exit** button on the main dialog screen:

When one of the plotting, calculation, 'file opening' or 'file importing' procedures is running, the cursor will change to an hour glass and the status bar will display "Plotting…", "Calculating…", "Opening file…" or "Importing file…". The

cursor and the status bar text will both be reset when the procedure is finished.

4 Common Functions

4.1 Introduction

While each module performs a unique task, they all share three common functions (Import DVS Data, Open Data File, DVS Version).

4.2 Importing DVS Data

From DVS Analysis Suite non-CFR (v4.0) onwards, raw data may be directly imported into a new Excel '.XLS' file provided the number of lines imported is less than 65536. From DVS Analysis Suite non-CFR (v6.0) onwards, raw data may be directly imported into a new Excel '.XLS' file provided the number of lines imported is less than 65536 otherwise raw data may be directly imported into a new Excel .XLSB or .XLSX file provided the number of lines imported is less than 1048576. From DVS Analysis Suite CFR (v4.0) onwards then raw data may be directly imported into a new Excel '.XLS' file provided the number of lines imported is less than 65536. From DVS Analysis Suite CFR (v7.1) onwards, raw data may be directly imported into a new Excel '.XLSX' file provided the number of lines imported is less than 1048576 (and provided the IWB supports it).

Note: Be aware that the fastest import (and open) format is XLS followed by XLSB (only non-CFR use) followed by XLSX.

Data from a DVS raw data file can be imported using any module by clicking the **Import Data File** button on the module's main dialog screen:

Upon clicking this button, the standard Excel 'file opening' dialog box will appear, entitled **Select DVS Raw data file to import**) and through this the DVS raw data file ('.DAT') can be selected:

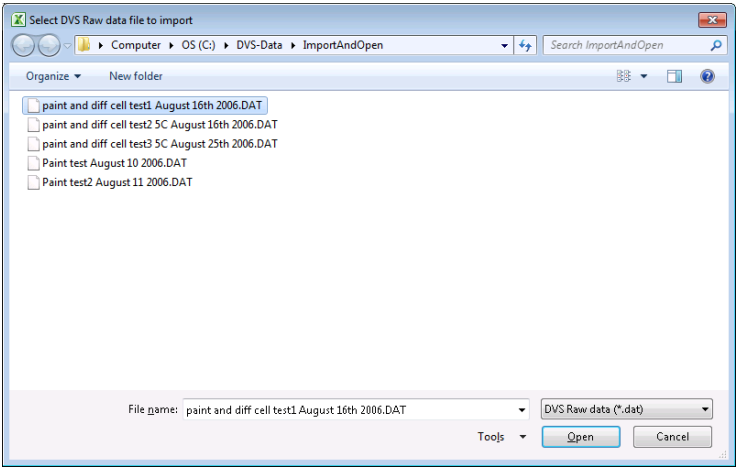


Figure 4.1: The **Select DVS Raw data file to import** dialog box

After selecting the desired .DAT file and clicking **Open**, the standard Excel "file save" dialog box will appear – entitled **Select DVS Analysis data file to save -** and through this the DVS data file name ('.XLS') can be specified:

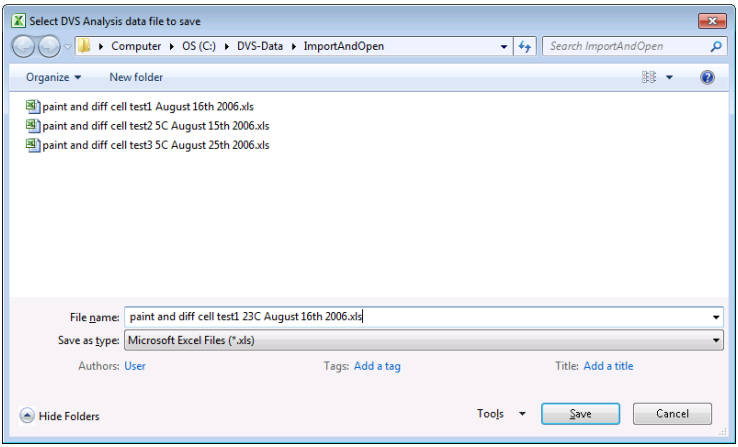


Figure 4.2: The **Select DVS Analysis data file to save** dialog box

While the file is being imported, the cursor will change to an hour glass and the status bar will display the message "Importing file…". The cursor and the status bar text will both be reset when the file is open.

A new Excel file containing the DVS data is created and opened automatically. An example of the data is given below.

Note that if the small toggle button next to the Import button is depressed the default adsorbate will be swapped (in other words if adsorbate A had a larger partial pressure variation than adsorbate B and would normally have been chosen as the default adsorbate depressing the toggle button would make adsorbate B the default adsorbate.

Note: For Excel 2003 or earlier files are always saved as XLS. For Excel 2007 or later in non-CFR situations files are saved as either Excel's default "Save As" format (or XLSB if it breaks the XLS limit) or as XLS if small enough. This can be overridden in the Config dialog. For CFR situations only default Excel formats of XLS and XLSX are valid.

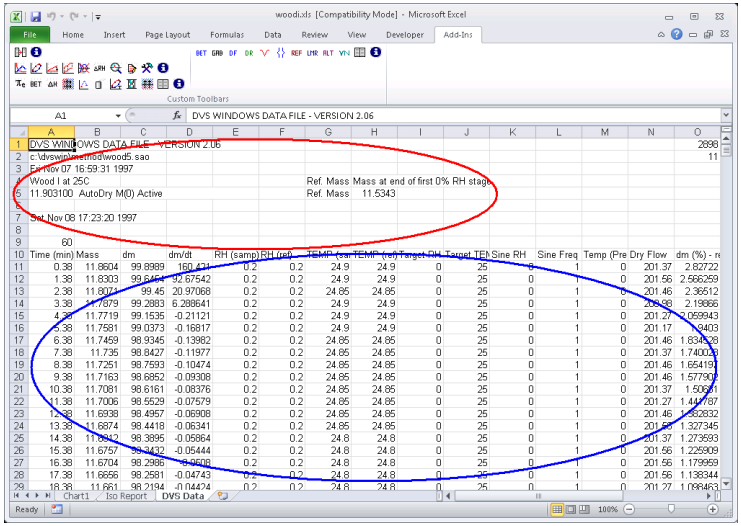


Figure 4.3: DVS data once imported into Excel. The information in the top left hand corner (Red Ellipse) describes the experimental set-up, and is called the DVS Header. The raw DVS data is presented in vertical columns (Blue Ellipse).

4.2.1 Multiple File Imports

More than one file may be imported at a time.

File import is handled differently depending on whether single or multiple file import is selected and also on whether CFR mode is active.

Note: Be aware that opening multiple large files (and using multiple large files when opened) can stretch Excel's resources (leading to crashes or hangs).

4.2.1.1 Non-CFR Mode

Single File Import

If a single file is imported using non-CFR mode software, the target output filename may be altered via the Excel "file save" dialog.

Multiple File Import

If multiple files are imported using non-CFR mode software, the target output filenames are fixed and cannot be altered via the Excel "file save" dialog.

Instead the filenames are given the name of the original .DAT imported file but the .DAT extension is changed to a .XLS extension.

If this target filename already exists in Excel then the characters _1, _2, … _9 are appended to the end of the filename before the .XLS extension to make the filename unique. The extra characters are appended to ALL the multiple files until a unique set is found. If a unique name cannot be found the import is abandoned.

4.2.1.2 CFR mode

In CFR mode both single-file import and multiple-file import are identical to the single file import in non-CFR mode.
The behaviour described above can be summarised by stating that target filenames during a multiple file import in non-CFR mode cannot be altered.

Note: If a DVS Advantage, DVS-HT, DVS Intrinsic, or DVS Adventure/Resolution instrument is being used, any data can be imported at any time - even data from a running experiment.

However, if a **DVS 1/1000 or DVS 2/2000** instrument is being used, it is not recommended to Import data from the running experiment until that experiment is completely finished. To analyse data while the experiment is running, please perform the following steps:

- Using DVSWin 3.XX: perform a Data File Carbon Copy operation and analyse the raw data file produced.

- Using DVSWin 2.XX: perform an Export Data operation and analyse the DVS Analysis file (.XLS) produced.

4.3 Opening DVS Data Files

DVS Analysis files ('.XLS' in the case of Excel 2003 and '.XLSB or .XLSX' in the case of Excel 2007 and higher) can be opened from any module by clicking the **Open Data File** button on the module's main dialog screen.

Upon clicking this button, the standard Excel 'file opening' dialog box (entitled **Open**) - will appear.

Through this the data file can be selected and opened in the same way as a normal Excel spreadsheet file – an individual file may be opened either by clicking on it once and clicking the **Open** button, or by double-clicking on the file.

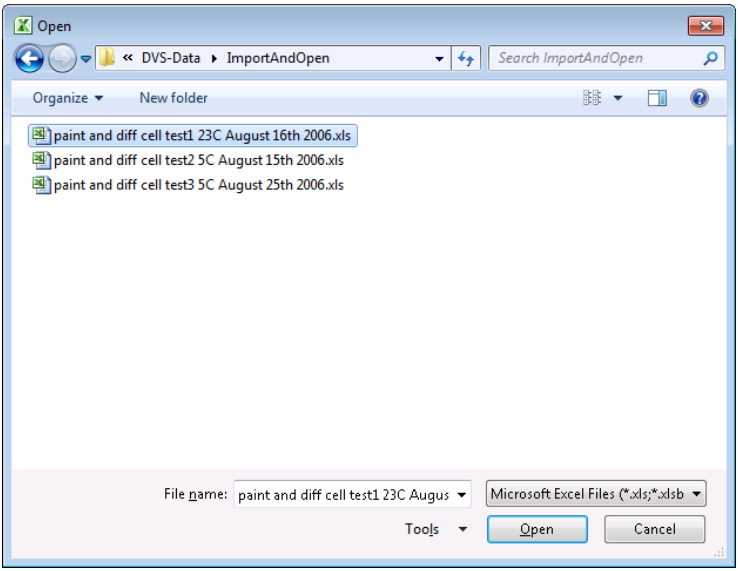


Figure 4.4: A standard Excel 'file opening' box appears upon clicking the DVS Open Data File button

While the file is being opened, the cursor will change to an hour glass and the status bar will display the message "Opening file…". The cursor and the status bar text will both be reset when the file is open.

4.3.1 Importance of using Open Data File

When a file is opened using the **Open Data File** button, the file's 'dm (%) - ref' data column is calculated and information is stored regarding the number of rows of data and where any drying or balance tare occurred (see also Section 5.3.10 on 'dm(%)-ref').

Therefore, for any subsequent analysis or plots to be produced from the data after opening, the file must be opened through an analysis suite module using the **Open Data File** button, rather than through the 'File' menu in Excel.

Because the correct unit of mass is required in the 'dm (%) - ref' column calculation, the DVS Version Number (Section 4.3.2) must be selected correctly on the main dialog screen before clicking the **Open Data File** button.

In addition the DVS Version Number is used to select whether a Legacy, Advantage or HT instrument is being used.

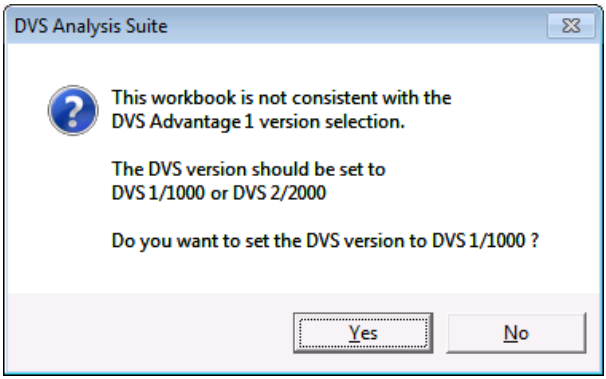
4.3.2 DVS Version (if options displayed)

In all modules the DVS version can be toggled between 'DVS 1/1000', 'DVS 2/2000', 'DVS Advantage 1', 'DVS Advantage 2', 'DVS-HT', 'DVS Intrinsic', 'DVS Adv. Plus' and 'DVS Vacuum' using the **DVS Version** option buttons on the main dialog screen (Figure 4.5 below).

Figure 4.5: The DVS Version option buttons, featured as part of every module in the Analysis Suite

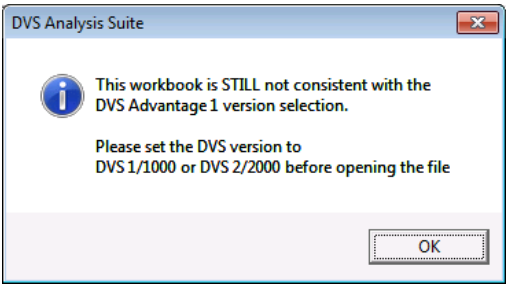
The choice of DVS Version will alter the mass unit used in plotting, calculation and 'file opening'/'file importing' procedures.

Normally, if the wrong DVS Version is selected upon opening or importing a file, a message of the form shown below will appear:



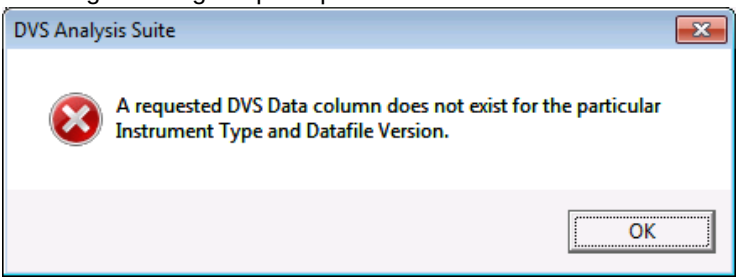
In this case, an attempt has been made to open a DVS 1/000 file with the DVS-HT mode selected. The panel above gives the user the chance to automatically select the suggested DVS Version – in this case the DVS 1/1000.

Upon clicking **Yes**, the file will be opened as a DVS 1/1000 data file and the DVS Analysis Suite will be switched to the **DVS Version** selection.

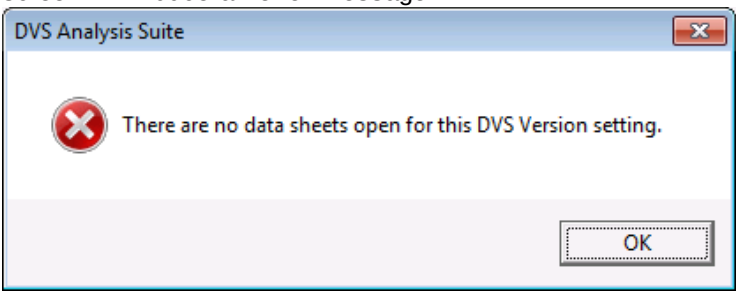


Upon clicking **No**, the above panel may re-appear suggesting another alternative DVS Version (eg; DVS 2/2000). If all of the suggested alternatives are exhausted by clicking **No** to each, then finally a panel of the sort shown below will appear:

Upon clicking **OK**, the desired file is opened, but with the following message superimposed:



Upon clicking **OK** to this message, the user will find that the Excel file is opened, but no analysis can be performed using the DVS Analysis Suite. Buttons pressed on any module screen will induce an error message:



As the above shows, the DVS version number is correctly set when a DVS data file is imported or opened.

However, in the case of Legacy DVS files, the setting may not be automatically corrected. Therefore, for DVS 1/1000 or DVS 2/2000 files it should be set correctly before any files are imported or opened.

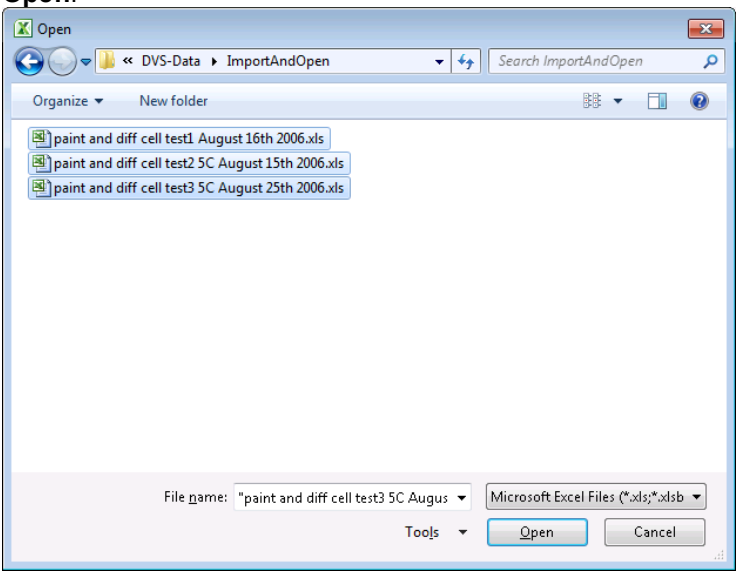
This is important, as the **DVS Version** setting determines the unit of mass used in the calculation of the 'dm (%) - ref' data column.

Thereafter any DVS Analysis Suite functions may be used on the imported or opened files.

4.3.3 Opening multiple files

Multiple files may be opened – either one at a time or simultaneously.

To open files simultaneously, each desired file must be selected while holding down the **Crtl** key before clicking **Open**:



Whether opened simultaneously or one at a time, the files still remain as individual Excel files.

4.3.4 Analysing Multiple Files

The DVS Analysis Suite has a multi-file selection capability. The following points should therefore be noted:

- Only files matching the instrument type, specified in the DVS Version Number, may be chosen for analysis;

- If DVS files are open but none are of the correct type then an analysis will not be possible.

4.4 Report and Chart Formatting

All reports and charts produced by the DVS Analysis Suite include presentation-quality titles and borders and a summary of experimental details. On computers connected to a printer, the page setup and print area are also set so that the report/chart is ready to print. All charts include chart and axis titles and a series legend.

4.5 Errors

In the event of an error occurring while running the analysis suite, please notify Surface Measurement Systems of the error. The preferable means of notification is by sending an e-mail which includes a description of the operation being attempted, the error message given and the data file being used, as an attachment. If e-mail is not possible notify Surface Measurement Systems by phone and send the file by floppy or CD disk (in '.DAT' format if the file is large).

5 DVS Plot Manager

5.1 What does this module do?

The **DVS Plot Manager** module produces various Excel charts from DVS data files – it includes six predefined plots as well as a custom plotting capability.

5.2 Why is this useful?

The Plot Manager module enables the user to know the 'story' of each DVS experiment.

It provides a graphical representation of vapour sorption and desorption by the sample (by producing a sample mass vs time graph), and thus allows the user to know quickly whether the experiment has been successful or not, and whether the data can be used to calculate desired parameters.

The Plot Manager also plots other parameters – instrument temperature and partial pressure, for example – which allow the user to quickly assess the performance of the instrument.

5.3 How to use this module

Clicking on the **DVS Plot Manager** button calls up the main dialog screen for the module as shown below in **Error! Reference source not found.**. The features differ slightly between DVS Versions, but the overall layout remains the same.

On the left hand side of the screen are the buttons to open/import a DVS data file, produce a predefined or custom

plot or exit from the module. Just below these buttons is the DVS Version selection area (Section 4.3.2).

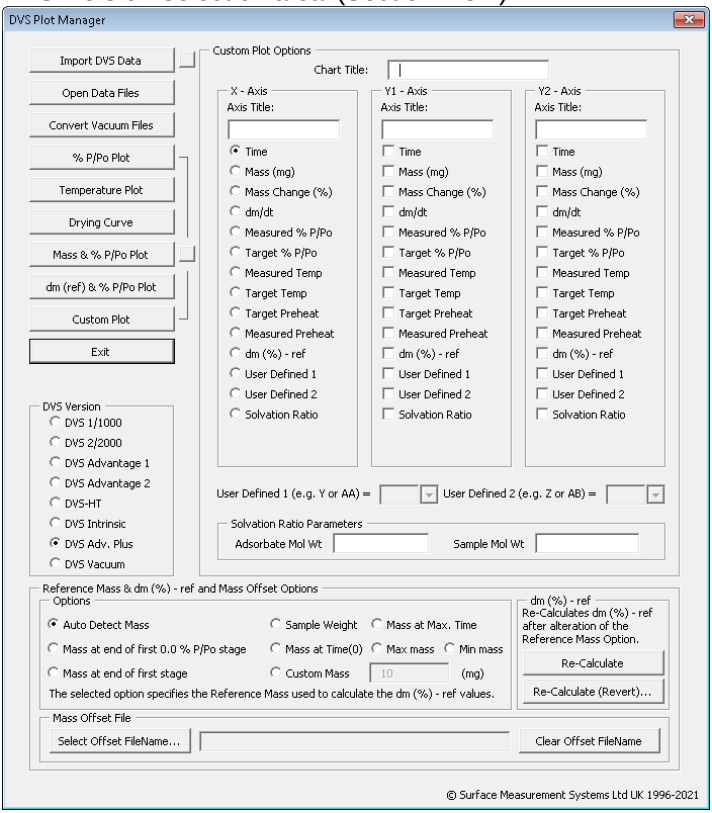


Figure 5.1a: The **DVS Plot Manager** main dialog screen (DVS Advantage Plus version)

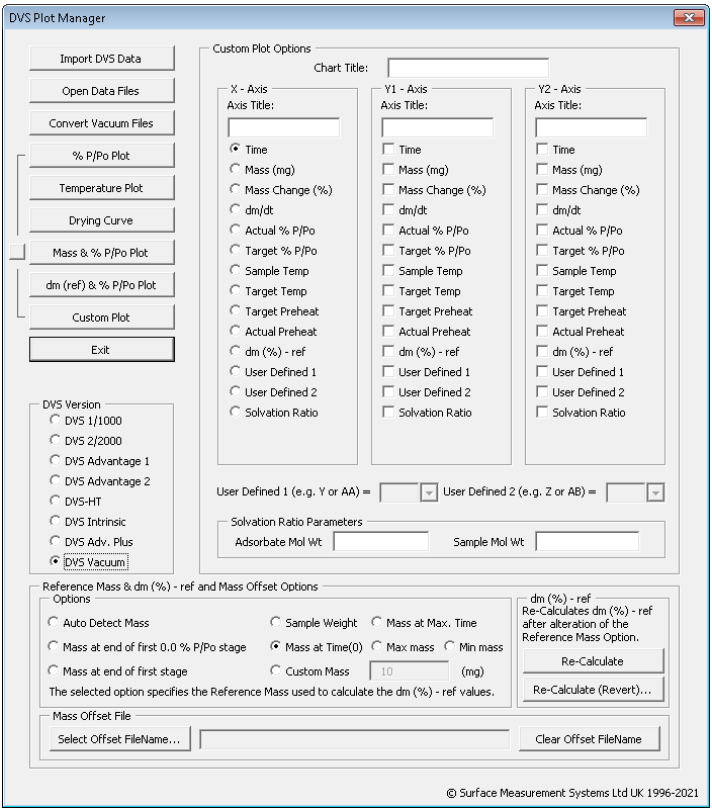


Figure 5.2b: The **DVS Plot Manager** main dialog screen (DVS Vacuum version)

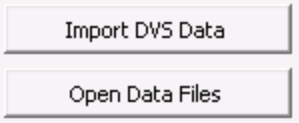
On the panel's right-hand side is the Custom Plot options selection area. The lower part of the screen contains the Open Data File Settings that are applied to files subsequently opened with the **Open Data File** button. These functions are also explained in detail in the sections to follow.

If the DVS Version is set to DVS Adv. Plus (Adventure, Resolution, Endeavour, Intrinsic Plus instruments) then there is a small toggle button to the right of the Plot buttons which can be used to exclude Solvent B from plots (if applicable).

If the DVS Version is set to DVS Vacuum then there is a small toggle button to the left of the Plot buttons which can be used to switch from Relative Pressure to absolute Pressure on plots. This feature is also available for Isotherm Analysis.

If the DVS Version is set to DVS 1/1000 then there is a small toggle button to the right of the Convert Vacuum Files button which can be used to toggle between Converting pre v2.1.6 raw data files to pre v2.1.6 Excel files or to Converting pre v2.1.6 Excel files to DVS Vacuum File Format files (DVS Data sheet only).

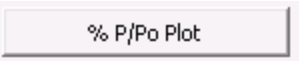
5.3.1 Import DVS Data & Open Data File



These functions are explained in detail in Sections 4.2 and 4.3, respectively.

Before any data plots may be constructed, a raw DVS data file must first be open in Excel using these buttons, which appear on every module main panel - the file(s) may be imported and opened from any module before using the plot functions.

5.3.2 % P/P_o Plot



Clicking the **% P/P_o Plot** button charts the partial pressure (%) generated by the DVS system and the target partial pressure (%) against time (minutes).

These plots provide a convenient means of checking the correlation between the target and actual partial pressures achieved in the instrument, as shown below in Figure 5.3:

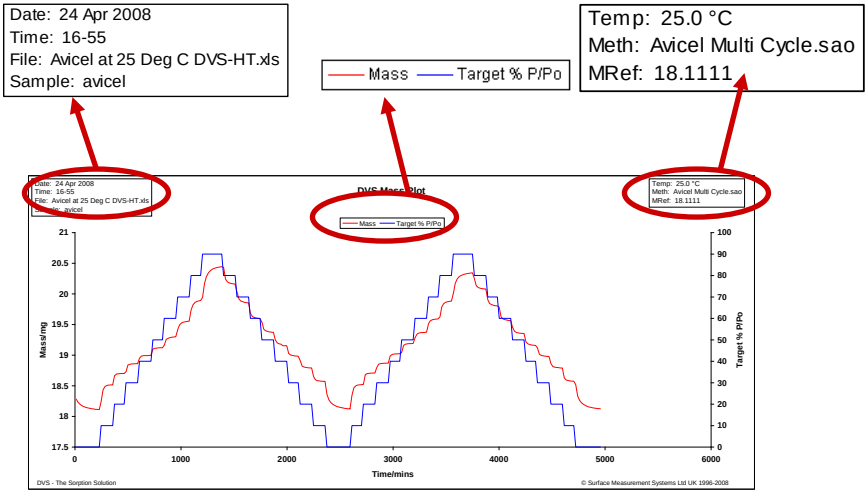


Figure 5.3: A **DVS Partial Pressure Plot** created from DVS Advantage results. The two lines show the system's target and sample $\%P/P_o$ have good correlation

Figure 5.3 also shows that additional information is contained within the plot area; in the top left-hand corner, a box showing the experimental date and time, data filename and sample name; in the top right-hand corner a box showing the experimental conditions of temperature, method file and sample M(0); in the centre is the chart legend.

This additional information is contained in all DVS Plots.

5.3.3 Temperature Plot

Temperature Plot

Clicking the **Temperature Plot** button produces a plot of the system's sample temperature (°C) against time (minutes).

This plot (Figure 5.4 below) allows the user to check the instrument's temperature in the sample chamber.

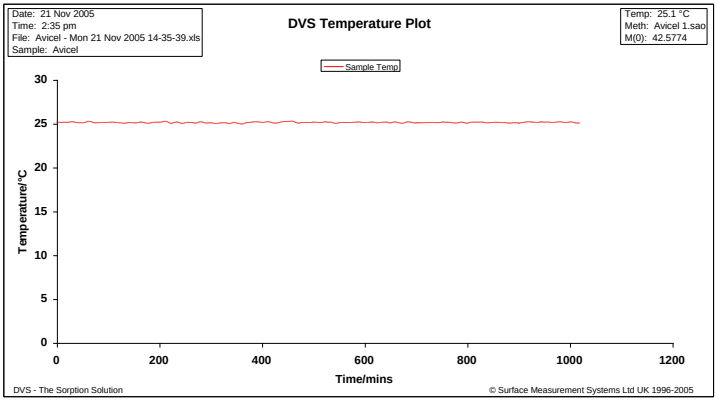


Figure 5.4: A DVS Temperature Plot showing the system's temperature stability in the sample chamber

5.3.4 Drying Curve

Drying Curve

Clicking the **Drying Curve** button produces a standard drying curve plot.

This will chart mass against time for the first 0% P/P_o stage of the experiment wherever that stage occurs in the experiment (refer Figure 5.5 below).

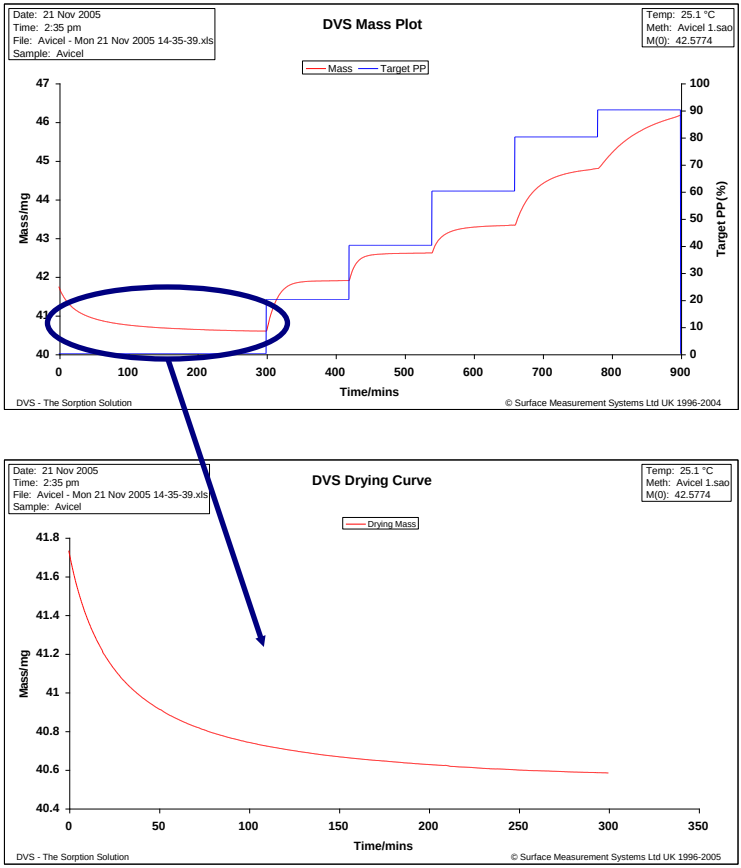


Figure 5.5: An entire DVS Mass Plot (top). Pressing the Drying Curve button produces a mass vs time graph of only the 'drying' (0% P/P_o) section of the experiment (bottom)

If there is no drying data contained in the DVS file and the Drying Curve button is pressed, the message shown below in

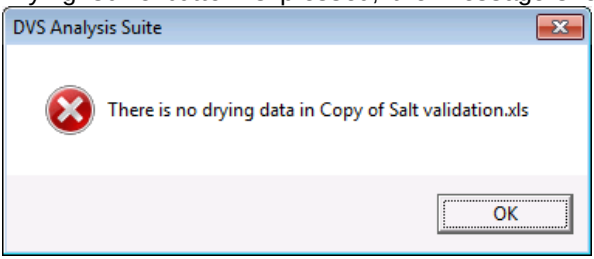


Figure **5.6** appears;

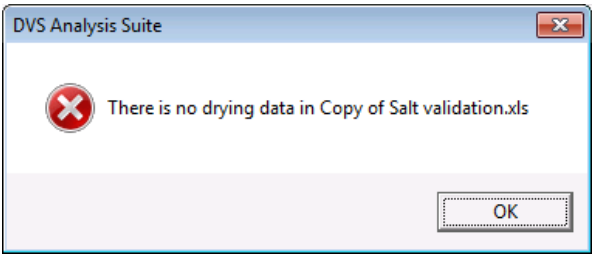
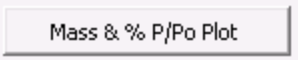


Figure 5.6: Error message given when there is no 'drying' to plot (the error message also specifies the filename in question – in this case 'Copy of Salt validation.xls'

5.3.5 Mass & % P/P_o Plot



Clicking the **Mass & % P/P_o Plot** button produces a plot of mass (mg or g) and target partial pressure (%) against time (minutes) (Figure 5.6 below).

If the balance was Auto Tared during the experiment, the plot will show only the post-tare data.

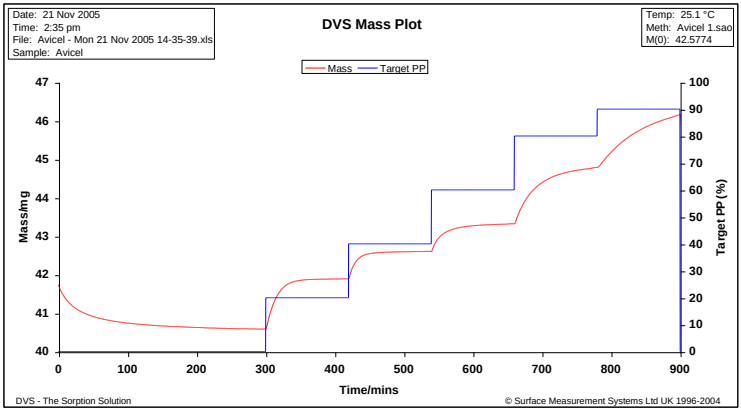
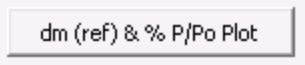


Figure 5.7: A **DVS Mass Plot**, showing the mass data (mg) in red and the target $\%P/P_o$ in blue vs time (mins).

5.3.6 dm (ref) & % P/P_o Plot



Clicking the **dm (ref) & % P/P_o Plot** button produces a plot of 'dm (%) – ref' and partial pressure (%) against time (minutes) (Figure 5.8 below).

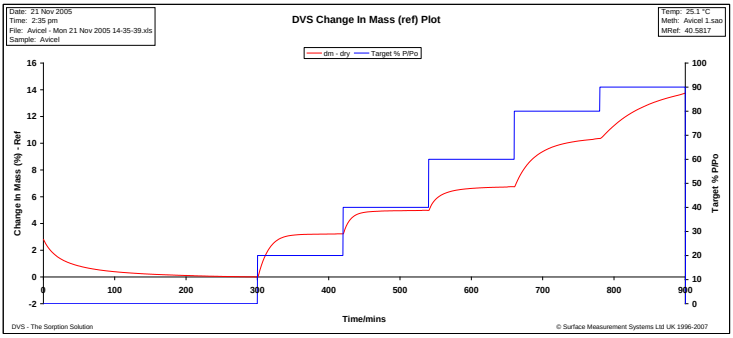


Figure 5.8: A DVS Change In Mass (ref) Plot.

5.3.7 Custom Plot

The custom plot procedure produces a plot that can include any combination of DVS data series, on a chart formatted to the same standard as the predefined plots described above.

5.3.7.1 Custom Plot Options

Before clicking on the **Custom Plot** button, the **Custom Plot Options** must be specified on the main dialog screen (Figure 5.9 below).

Note: the Custom Plot Options vary depending upon which DVS Version is selected in the DVS Version box on the main Plot Manager panel. However, the method of choosing the custom plot options is the same across all DVS Versions.

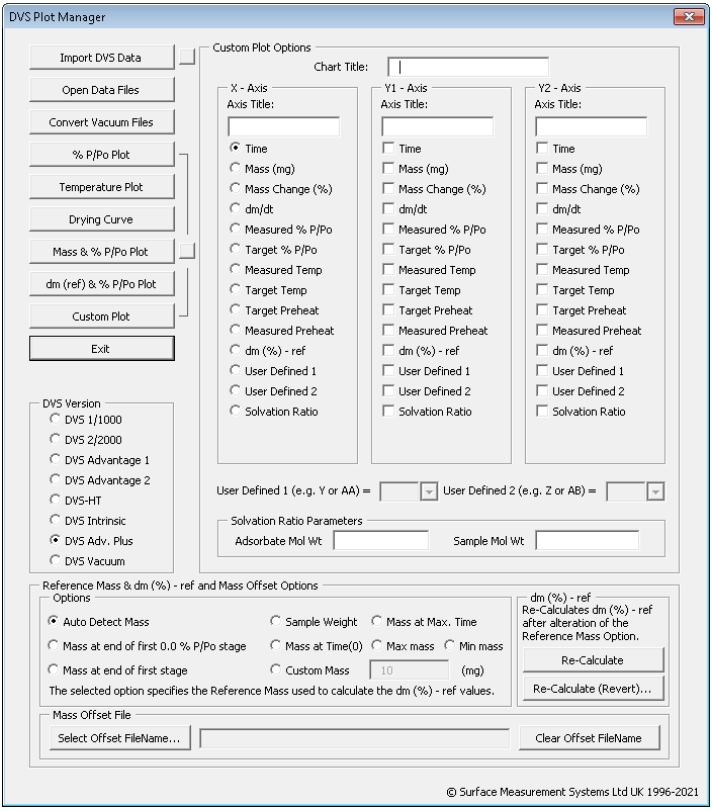
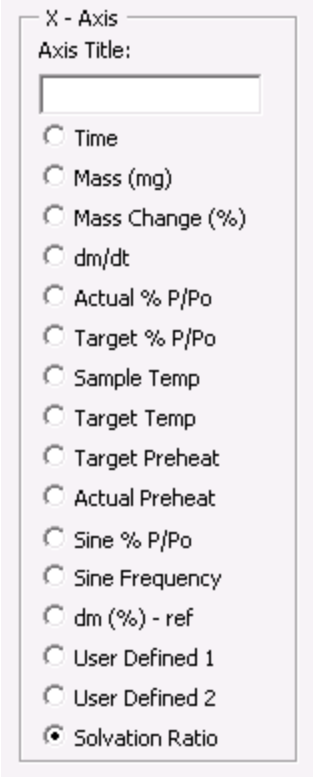


Figure 5.9: The Custom Plot Options selection area on the DVS Plot Manager main dialog screen.

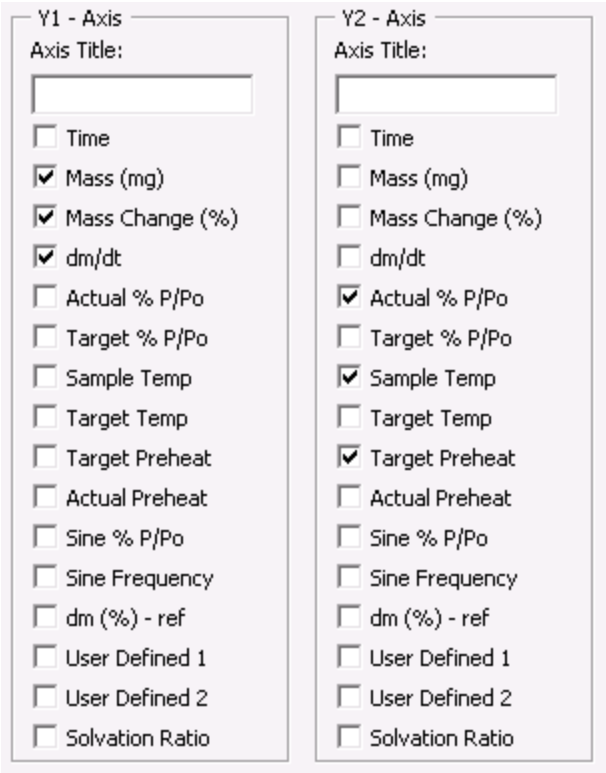
X-Axis



The X-Axis series is chosen by selecting one of the options in the **X-Axis** selection area, shown below:

The text entered in the **Axis Title** box in the X-Axis selection area will be displayed next to the X axis. If nothing is entered in this edit box, a default title will be displayed in the chart. If desired, this can be altered in Excel after the chart has been created.

Y-Axes



The y-axis series are chosen by ticking as many of the check boxes as required in the **Y1-Axis** and **Y2-Axis** selection areas:

The series selected in the **Y1-Axis** area will be plotted on the left hand axis of the chart and those selected in the **Y2-Axis** area will be plotted on the right hand axis.

As many series on each axis may be selected as is desirable as long as at least one Y1-Axis series is selected. It is possible to not plot any Y2-Axis series, if desired.

The text entered in the **Axis Title** boxes in the Y1-Axis and Y2-Axis selection areas will be displayed next to the Y1 and Y2 axes respectively. If nothing is entered in these edit boxes, default titles will be displayed in the chart. If desired, these can be altered in Excel after the chart has been created.

Chart Title

The text entered in the **Chart Title** edit box is displayed as the title at the top of the chart:

If nothing is entered in this edit box, a default title will be displayed in the chart. If desired, this can be altered in Excel after the chart has been created.

5.3.7.2 Column Edit Boxes

The custom option can be used in a slightly different way to plot DVS Data sheet columns which are NOT usually able to be plotted. Entering column letters in the Column Edit boxes overrides the normal pickup from the User Defined columns and instead picks up data from the columns entered. Alternatively pick from the drop down lists.

5.3.7.3 Custom Plot Example

In the example shown below in Figure 5.8**0**, the parameter chosen for the X-Axis is time, while the Y1-Axis (entitiled 'Various Parameters 1') is selected to display Mass, Mass

change, and dm/dt. The Y2-Axis (entitled 'Various Parameters 2') is selected to display Actual and Target % P/P_o, and Sample and Target Temp.

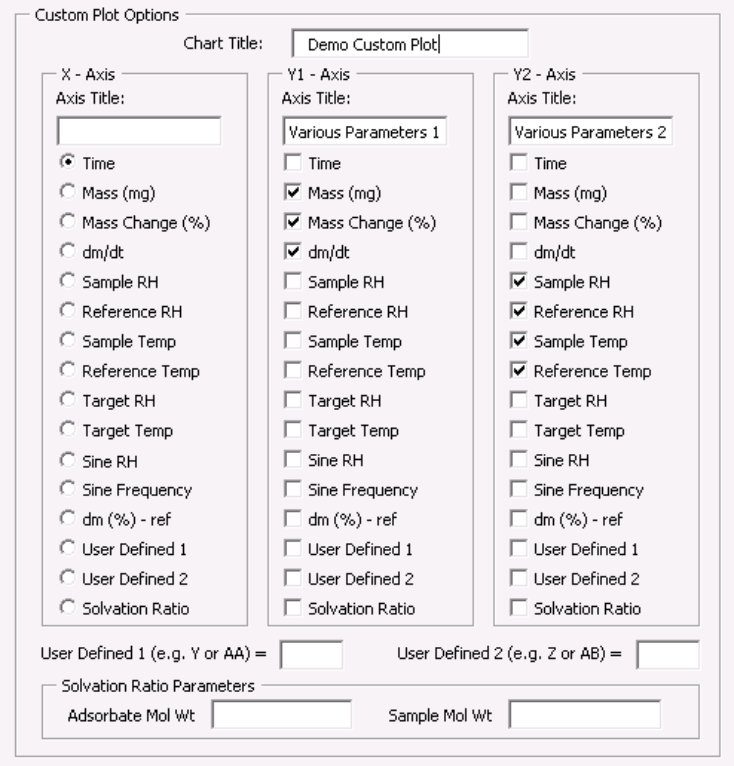


Figure 5.80: The Custom Plot Option parameters chosen for this example.

The resulting plot is shown in Figure 5.91.

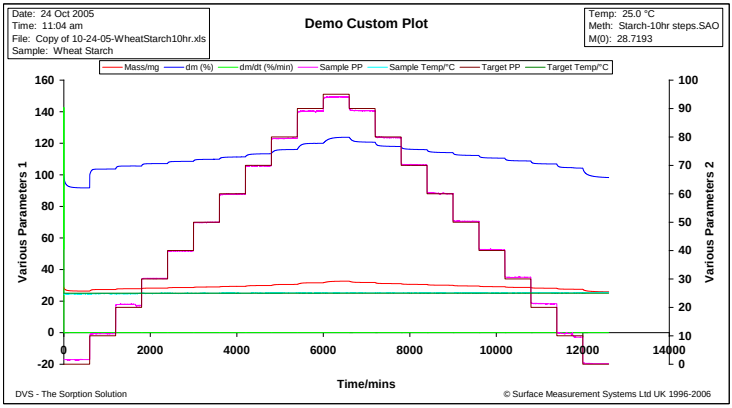


Figure 5.91: The resulting **Custom Plot** from the choice of parameters shown in Figure 5.8.

User Defined data

The series in the DVS raw data file labelled **User Defined 1** and **User Defined 2** (Figure 5.102 below) can be used to plot data not already covered by the listed plot options.

Temp (Preheat)	Dry Flow	dm (%) - ref	User Defined 1	User Defined 2
0	200	8.733881535		
0	199.71	8.379132879		
0	199.71	8.055908996		
0	199.9	7.76269062		
0	199.9	7.489222705		
0	199.8	7.237404334		
0	199.8	6.999639174		
0	199.71	6.77896576		

Figure 5.102: The User Defined columns at the very end of the DVS raw data file (circled).

If the **User Defined 1** or **User Defined 2** series are selected (Figure 5.113 below) the chart created will include the data in the User Defined 1 or User Defined 2 columns.

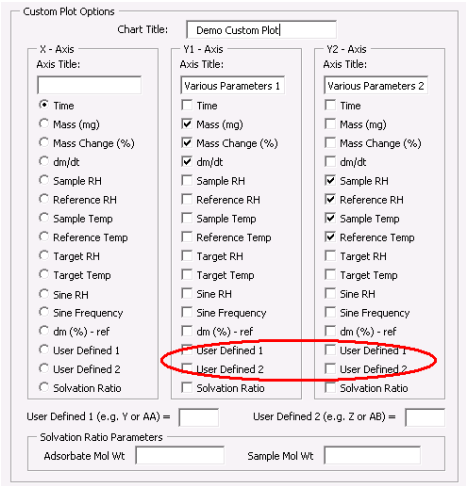


Figure 5.113: The custom plot User Defined options.

By default, a new data file contains no data in the User Defined columns, as Figure 5.102 above shows. Data must be put in these columns by the user in Excel.

For example, the user could fill the **User Defined 1** column with a subtraction of the data in the **Target Temp** column from the data in the **Sample Temp** column, and then construct a custom plot of **User Defined 1** against **Time**.

This would produce a plot of the difference in target and sample % P/P_o against time, as shown below in Figure **5.12** and Figure 5.135:

User Defined 1
0.3
0.2
0.2
0.2
0.2
0.2
0.2
0.2

Figure 5.124: The User Defined 1 column, calculated as Sample Temp minus Target Temp.

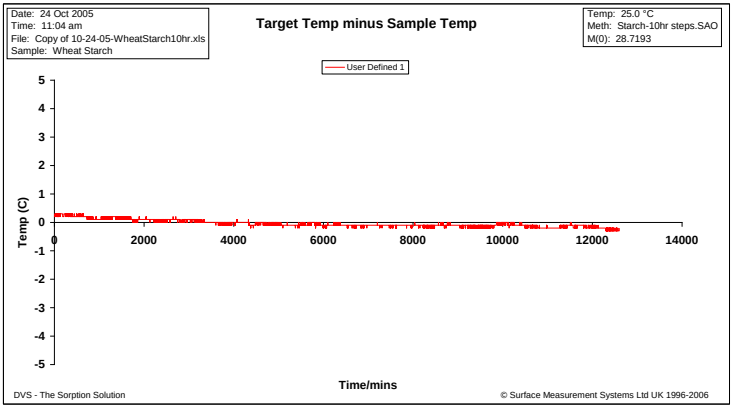
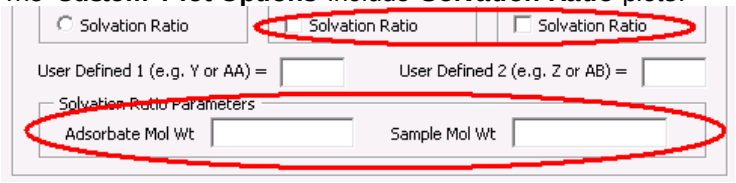


Figure 5.135: A **Custom Plot** of User Defined 1 data vs Time.

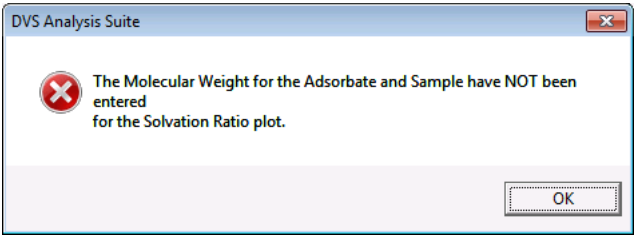
5.3.7.4 Plotting Solvation Ratio

The **Custom Plot Options** include **Solvation Ratio** plots:

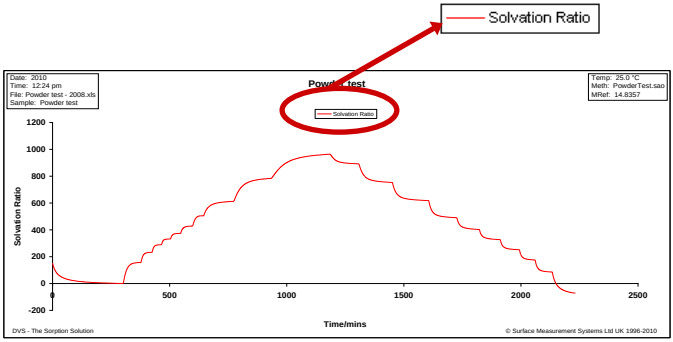


Solvation Ratio represents the molar ratio of sorbed vapour to initial sample mass, and therefore the molecular weights of both sample and adsorbate are required for the plot to be constructed.

If the Solvation Ratio option is chosen without providing the required molecular weights, and the **Custom Plot** button is clicked, the following warning will appear:



Once the molecular weight parameters have been provided, the creation of a **Solvation Ratio** plot will coincide with the addition of a new column of data in the **DVS Data** Excel sheet:



Molecular Weight of Adsorbate
18.01528
Molecular Weight of Sample
128000
Solvation Ratio
122.6029
121.7409
120.9746
120.1605
119.2505
118.5321
117.718
116.9517
116.2333
115.4671
114.8445
114.1261
113.3598
112.6893
111.8752

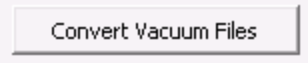
Chart1 | DVS Data

This column appears at the very end of the original body of DVS Data (ie; the column furthest to the right).

Note: Solvation Ratio curves may be included in Isotherm plots produced by the **Isotherm Analysis Manager** (see Section 6).

However, in order to do this, the Solvation Ratio plot must already have been produced using the **Custom Plot** option in **Plot Manager**.

5.3.8 Convert Vacuum Files

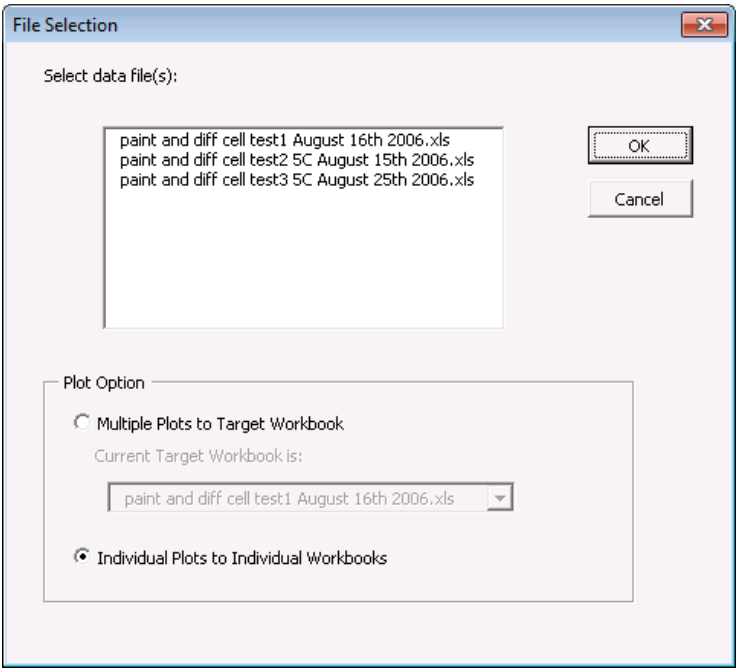


The convert Vacuum files function converts a DVS vacuum file to a DVS Data file format (DVS 1 format).

5.3.9 Multiple Plots

Plots can be constructed for several files at once. To do this, all the files of interest must first have been opened using the **Open Data File** button on any of the modules.

Then, upon clicking the Plot button (in this case, the **Mass & %P/P$_o$** button), the **File Selection** panel will appear:



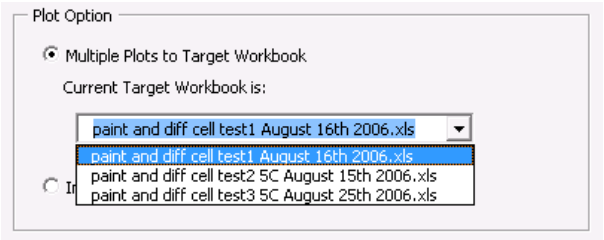
The **Select data files(s)** window presents a list of the currently open files that may be included in the multiple plot.

The user may select the files to be plotted by clicking on them – for multiple file selection, hold down the **Ctrl** key whilst clicking on the files.

The **Plot Option** box allows the user to determine where each plot will be saved:

Multiple Plots to Target Workbook
This option saves all of the plots to one of the files included in the multiple plot.

When this option is chosen, a drop-down menu is activated, allowing the user to choose the desired file:



Individual Plots to Individual Workbooks
This option saves each individual plot to its respective source file.

Clicking **OK** will then produce the multiple plots. Below is an example of multiple mass plots saved to the same file (Figure 5.146 below):

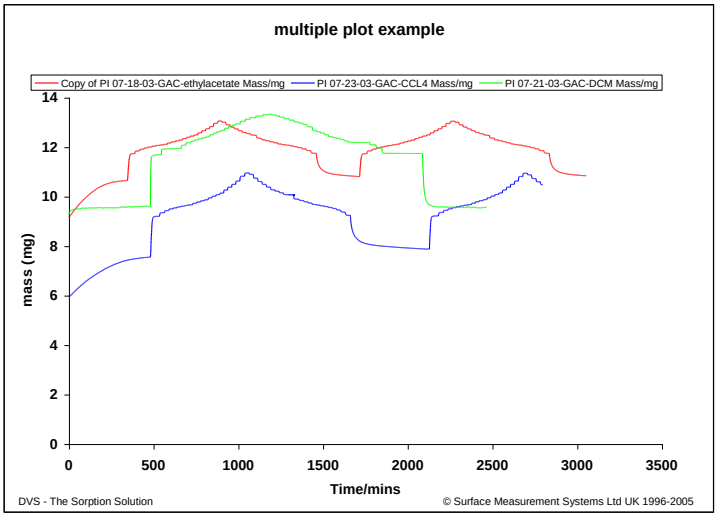
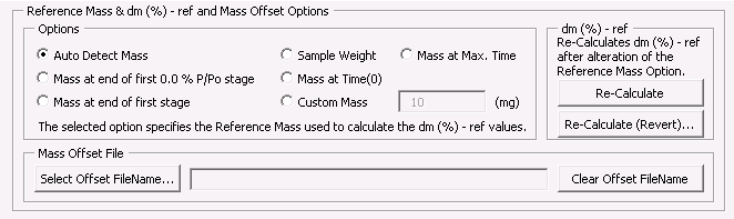


Figure 5.146: An example of multiple plots saved to a target workbook

5.3.10 Reference Mass & dm (%) - ref



These functions alter the way a file's **dm(%)-ref** data is calculated when it is opened. They do this by changing the value of 'reference mass' used in the dm(%)-ref calculation.

When a DVS data file is opened or imported through the Analysis Suite, a **dm(%)-ref** column is calculated and inserted into the file (see Figure 5.157below).

This data is used in the predefined **dm (ref) & % P/P$_o$ Plot** function (see Section 5.3.6) and all analysis modules that calculate isotherms.

Sine Freq	Temp (Preheat)	Dry Flow	dm (%) - ref	User Defined 1	User Defined 2
1	0	200	[illegible]		
1	0	199.71	8.379132879		
1	0	199.71	8.055908996		
1	0	199.9	7.76269062		
1	0	199.9	7.489222705		
1	0	199.8	7.237404334		
1	0	199.8	6.999639174		
1	0	199.71	6.77896576		
1	0	199.9	6.570446474		
1	0	200	6.372562053		

Figure 5.157: The dm(%)-ref column is inserted into the DVS Data Excel spreadsheet.

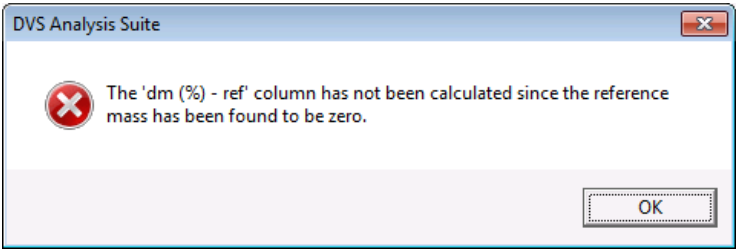
This column contains the sample's percentage change in mass with respect to a 'reference mass'; eg; the **dm(%)–ref** value for a mass value equal to the 'reference mass' is 0%:

$$dm(\%) - ref = \frac{(mass_value) - (ref_mass)}{ref_mass} \times 100$$

Equation 1

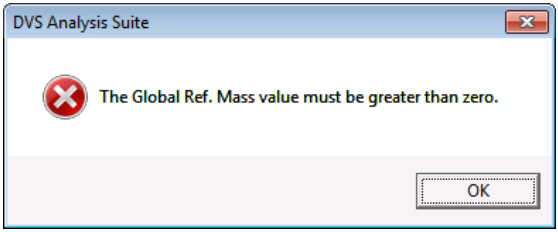
Zero 'reference mass'

Equation 1 shows that it is impossible to calculate **dm(%)-ref** with a 'reference mass' of zero. In the case of a zero 'reference mass' being selected, a message of the form shown below will appear:



In this case, the **dm(%)-ref** values previously calculated when the file was last opened will remain.

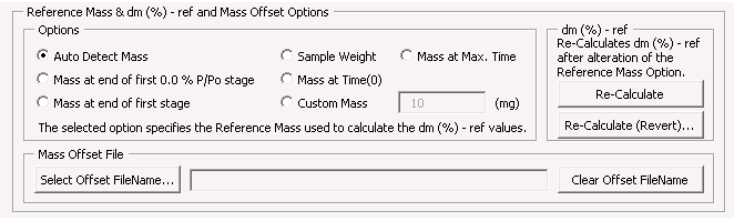
If importing the file for the first time and a 'reference mass' value of zero is detected, the following message appears:



Clickng **OK** then returns the DVS data file – the **dm(%)-ref** column of data will display all zeroes. The **dm(%)-ref** data will need to be re-calculated using a legitimate 'reference mass' value (see Section 5.3.17 for re-calculating **dm(%)-ref**).

Crucial to calculating **dm(%)-ref** is the way in which the 'reference mass' is defined – this varies depending upon which option is selected in the **Options** box explained below.

5.3.10.1 Auto Detect Mass



If the DVS data file is imported or opened with this option selected, the software automatically selects a 'reference mass' from the other available options to calculate **dm(%)-ref**.

Header Information

Use of **Auto Detect Mass** is indicated in the data file to the right of the header information, along with the 'reference mass' value used:

	A	B	C	D	E	F	G	H	I	J	K
1	DVS WINDOWS DATA FILE - VERSION 2.06										
2	c:\Program Files\DVS 2_18 Video\method\Starch-10hr steps.SAO										
3	Mon Oct 24 11:00:19 2005										
4	Wheat Starch						Ref. Mass	Mass at end of first 0.0 P/Po stage			
5		10	AutoDry M(0) Active				Ref. Mass	26.3285			
6	10 hr/step, 25C, H2O										
7	Mon Oct 24 11:04:03 2005										

In the case shown above, the **Auto Detect Mass** option was used, which selected the **Mass at end of first 0% P/P$_o$ stage** as the 'reference mass'. The **Mass at end of first 0% P/P$_o$ stage** may also be selected directly as the 'reference mass' by using another option (explained below in Section 5.3.11).

Highlighted Data

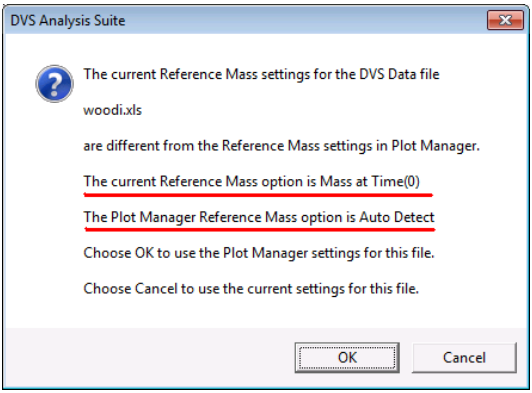
In addition to displaying the selected 'reference mass' in the data file header, the cell containing the 'reference mass' value is highlighted in mauve (see Figure 5.168 below):

10	Time (min)	Mass	dm	dm/dt	RH (samp)	RH (ref)	TEMP (sar	TEMP (ref)	Target RH
605	594.28	26.3289	91.67668	-0.00025	1.6	1.6	24.8	24.8	0
606	595.28	26.3289	91.67668	-0.00013	1.6	1.6	24.8	24.8	0
607	596.28	26.3288	91.67633	-7.4E-05	1.6	1.5	24.8	24.8	0
608	597.28	26.3286	91.67563	-0.00014	1.6	1.5	24.8	24.8	0
609	598.28	26.3285	91.67529	-0.00036	1.6	1.6	24.8	24.8	0
610	599.28	26.3285	91.67529	-0.00036	1.6	1.6	24.8	24.8	0
611	600.28	26.3285	100.0004	-0.00036	1.6	1.7	24.8	24.8	10
612	601.28	26.3471	100.071	0.005417	9.6	9.6	24.8	24.8	10

Figure 5.168: The chosen 'reference mass' – in this case the mass value at the end of the first 0% P/P$_o$ stage – is highlighted in the DVS data sheet.

Previously Defined 'reference mass'

If a file with a previously defined 'reference mass' is opened with a different 'reference mass' setting selected, the following message appears:



The highlighted text shows the current 'reference mass' file setting and the new setting selected in the Plot Manager.

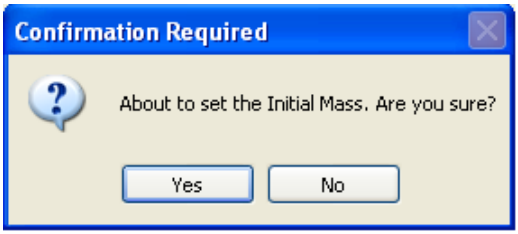
Clicking **OK** will use the new settings, while clicking **Cancel** will keep the file's original 'reference mass' setting.

Selection of 'reference mass'

The selection of which mass data to use as the 'reference mass' when using the **Auto Detect Mass** setting depends upon how the experiment was conducted – which is explained below:

a. Setting M(0)

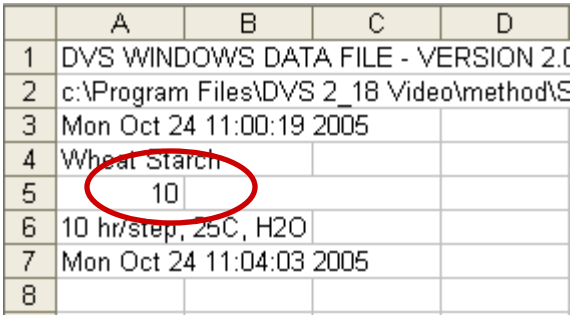
M(0) is referred to in the software as the 'Initial Mass'. For instance, when the Auto M(0) button is clicked in the DVS Control software, the following panel appears:



If an Initial Mass, or M(0), is set - either automatically or using Auto Dry M(0) - then it will appear in the DVS raw data header as the **Sample Weight**:

	A	B	C	D	E	F	G	H	I
1	DVS WINDOWS DATA FILE - VERSION 2.06								
2	c:\Program Files\DVS 2_18 Video\method\Starch-10hr steps.SAO								
3	Mon Oct 24 11:00:19 2005								
4	Wheat Starch						Ref. Mass	Mass at Time(0)	
5	28.7193	AutoDry M(0) Active					Ref. Mass	28.628	
6	10 hr/step, 25C, H2O								
7	Mon Oct 24 11:04:03 2005								
8									

Even when an M(0) is not set by the user, a default value of '10' is entered by the software into the header information:



	A	B	C	D
1	DVS WINDOWS DATA FILE - VERSION 2.0			
2	c:\Program Files\DVS 2_18 Video\method\S			
3	Mon Oct 24 11:00:19 2005			
4	Wheat Starch			
5	10			
6	10 hr/step, 25C, H2O			
7	Mon Oct 24 11:04:03 2005			
8				

When running a new experiment, if a new M(0) is not taken, then the M(0) from the last experiment will be used.

Note: It is important to understand that, despite its notation as 'Initial Mass', the M(0) does not necessarily constitute the 'reference mass' used in 'dm(%)-ref' calculations.

Whether M(0) is used in the 'reference mass' calculations depends on the set-up of the experiment and sometimes on the handling of the data after it has been imported into Excel.

This is explained further below.

b. Protocol for finding the 'reference mass'

When the **Auto Detect Mass** option is selected in **Plot Manager**, the software searches for a 'reference mass' value to calculate the 'dm(%) - ref' column.

The search for the 'reference mass' follows three steps:

I. End of drying stage

If the experiment contained a drying stage – a period of 0% P/P_o – then the mass reading at the end of this stage is taken as the 'reference mass' regardless of any tare or M(0) settings:

10	Time (min)	Mass	Target PP
605	594.28	26.3289	0
606	595.28	26.3289	0
607	596.28	26.3288	0
608	597.28	26.3286	0
609	598.28	26.3285	0
610	599.28	26.3285	0
611	600.28	26.3285	10
612	601.28	26.3471	10

Figure 5.19: DVS data at the end of the drying stage. The last mass value in the drying period (circled) is used as the 'reference mass' for **dm% - ref** calculations. In addition, the Analysis Suite software highlights the 'reference mass' in mauve.

The drying stage can be located anywhere in the experiment – the 'reference mass' will still be taken as the mass value accompanying the last 0% P/Po cell:

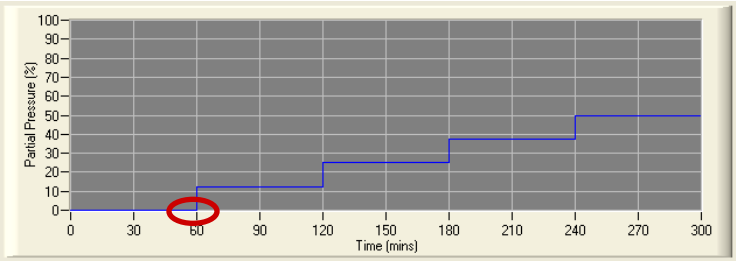


Figure 5.17: In the experiment shown above, the 'reference mass' would be taken from the point in the experiment circled.

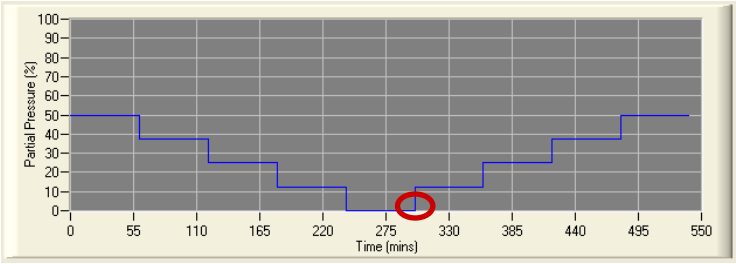


Figure 5.180: In this experiment, the 'reference mass' would be taken at the point circled.

It is also important to note that if the experiment contains several drying stages, the mass at the end of the first of these stages is taken as the 'reference mass':

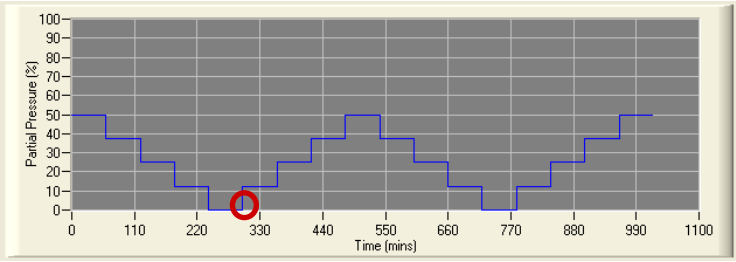


Figure 5.191: The 'reference mass' would be taken from the end of the first drying stage, circled above.

II. Auto Tare in the first stage

If there is no drying (0% P/P_o) stage in the entire experiment but there has been an **Auto Tare** operation (which takes place at the end of the first stage of the experiment), then the 'reference mass' will be taken as the last mass value of that stage.

Following this, the balance then takes approximately five seconds to perform the tare.

The resulting data will show the mass value going to zero very shortly after the first stage has finished (see Figure 5.202 below):

10	Time (min)	Mass	Target RH
60	16.7	137.1858	0
61	17.03	137.1857	0
62	17.37	137.1857	0
63	17.7	0	2.04
64	18.03	0	2.04
65	18.37	0	2.04
66	18.7	0.0003	2.04

1. 'reference mass' value taken at the end of the drying

2. balance is then tared

Figure 5.202: Auto Tare data showing the 'reference mass' value (mauve) and the balance tare (light blue). Whenever the Auto Tare function is used, the balance tare is highlighted along with the 'reference mass'. The balance completes the tare about five seconds after the drying stage. The appearance of the zero (tare) value will depend on the data saving rate used – in the example above, it takes around twenty seconds for the first 'zero' mass value to be saved and displayed.

III. No drying stage, no Auto Tare

If there is no drying stage or Auto Tare in the experiment, the **Sample Weight** recorded in the data file header is used as the 'reference mass' (Figure 5.213 below):

	A	B	C	D	E	F	G	H	I
1	DVS WINDOWS DATA FILE - VERSION 2.06								
2	c:\Program Files\DVS 2_18 Video\method\Starch-10hr steps.SAO								
3	Mon Oct 24 11:00:19 2005								
4	Wheat Starch						Ref. Mass	Mass at Time(0)	
5	28.7193	AutoDry M(0) Active					Ref. Mass	28.628	
6	10 hr/step, 25C, H2O								
7	Mon Oct 24 11:04:03 2005								
8									

Figure 5.213: The mass value recorded in the data file header (circled) is used as the 'reference mass' when there is no drying stage or Auto Tare in the experiment.

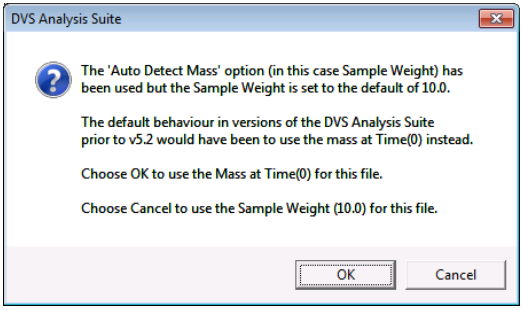
Correct M(0) value

Normally, the mass recorded in the header is the last recorded M(0). For this reason, it is important to ensure that M(0) has been recorded correctly – otherwise the 'dm(%) - ref' calculations will be based upon an incorrect mass value (in cases where there is no drying stage in the data).

It is therefore important to reset M(0) for each new experiment. Otherwise, the value recorded in a previous experiment will be saved to the data file header and used in the **dm%-ref** calculations.

Mass Header Value of '10'

Whenever the Sample Weight value in the mass header is '10' and the Sample Weight value is to be used as the 'reference mass', the following message will appear:



This is to ensure that the 'reference mass' is not being accidentally based upon the default value of '10' which appears in the **Sample Mass** cell when a value of M(0) is not set.

Clicking **OK** will ensure that the **Mass at Time(0)** option (see Section 5.3.14) is used instead of the **Sample Weight**, and clicking **Cancel** will allow the value of '10' to be used as the 'reference mass'.

Altered M(0) value

The header figure can be altered by the user. For instance, the user might open the data file, alter the header mass value, then save and close the file.

The next time the file is opened, the dm%-ref column will be re-calculated using this altered mass value as the 'reference mass'.

It is therefore recommended that the header mass value is not altered in any way.

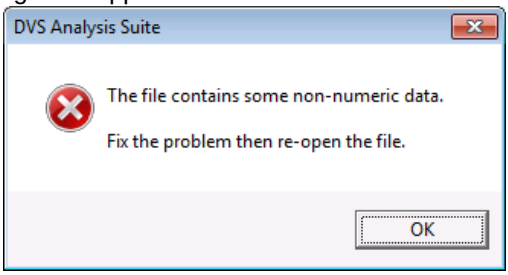
IV. Auto Dry M(0)

If **Auto Dry M(0)** is activated, then most DVS Control software versions will record a new M(0) value in the header at the end of the experiment's first stage. This, however, makes no difference to the dm(%)-ref column – taking an M(0) at the end of the first stage does not interfere with the 'reference mass' selection procedure outlined above.

It is also possible that the user might completely erase the header mass value – this instance is discussed below.

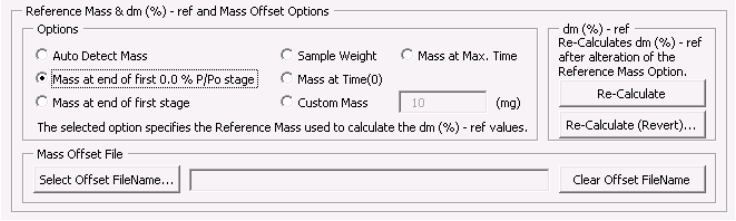
V. No drying stage, no Auto Tare and no header value

In the unlikely event that there is no drying stage, no Auto Tare and no M(0) value in the data file the software will not be able to find a 'reference mass' and the following message will appear:



Clicking **OK** returns the **Plot Manager** dialog.

5.3.11 Mass at end of first 0.0% P/P_0 stage



If the DVS data file is imported or opened with this option selected, the software selects the mass at the end of the experiment's first 0% P/Po stage as the 'reference mass' to calculate **dm(%)-ref**.

Header Information

The use of this option is indicated in the data file to the right of the header information, along with the 'reference mass' value used:

	A	B	C	D	E	F	G	H	I	J	K
1	DVS WINDOWS DATA FILE - VERSION 2.06										
2	c:\Program Files\DVS 2_18 Video\method\Starch-10hr steps.SAO										
3	Mon Oct 24 11:00:19 2005										
4	Wheat Starch						Ref. Mass	Mass at end of first 0.0 P/Po stage			
5	10	AutoDry M(0) Active					Ref. Mass	26.3285			
6	10 hr/step, 25C, H2O										
7	Mon Oct 24 11:04:03 2005										

Highlighted Data

In addition to displaying the selected 'reference mass' in the data file header, the cell containing the 'reference mass' value is highlighted in mauve (see below):

10	Time (min)	Mass	dm	dm/dt	RH (samp)	RH (ref)	TEMP (sar	TEMP (ref)	Target RH
605	594.28	26.3289	91.67668	-0.00025	1.6	1.6	24.8	24.8	0
606	595.28	26.3289	91.67668	-0.00013	1.6	1.6	24.8	24.8	0
607	596.28	26.3288	91.67633	-7.4E-05	1.6	1.5	24.8	24.8	0
608	597.28	26.3286	91.67563	-0.00014	1.6	1.5	24.8	24.8	0
609	598.28	26.3285	91.67529	-0.00028	1.6	1.6	24.8	24.8	0
610	599.28	26.3285	91.67529	-0.00036	1.6	1.6	24.8	24.8	0
611	600.28	26.3285	100.0004	-0.00036	1.6	1.7	24.8	24.8	10
612	601.28	26.3471	100.071	0.005417	9.6	9.6	24.8	24.8	10

Figure 5.224: The chosen 'reference mass' – the mass value at the end of the first 0% P/P_o stage – is highlighted in the DVS data sheet.

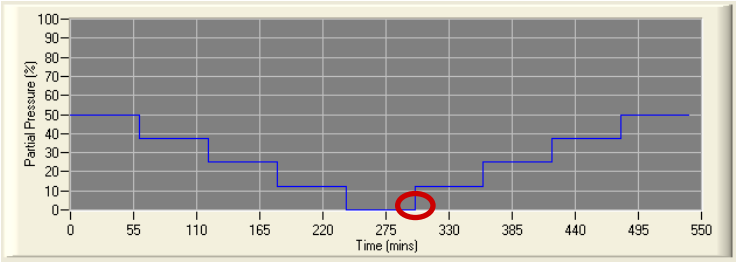


Figure 5.235: In this experiment, the 'reference mass' would be taken at the point circled.

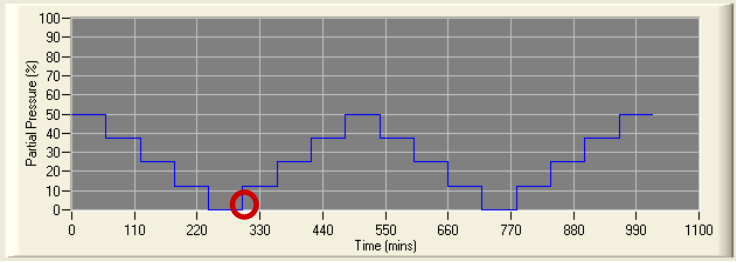
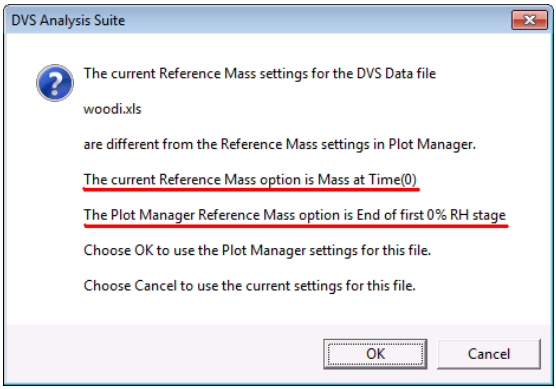


Figure 5.246: The 'reference mass' would be taken from the end of the first drying stage, circled above.

Previously Defined 'reference mass'

If a file with a previously defined 'reference mass' is opened with a different 'reference mass' setting selected, the following message appears:

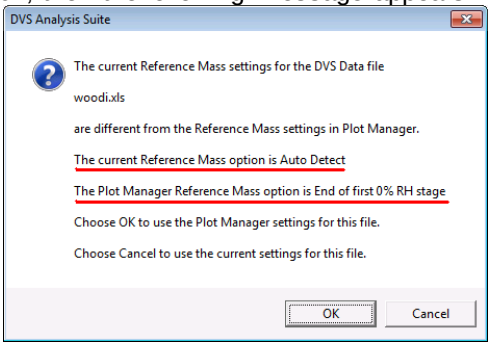


The highlighted text shows the current 'reference mass' file setting and the new setting selected in the Plot Manager.

Clicking **OK** will use the new settings, while clicking **Cancel** will keep the file's original 'reference mass' setting.

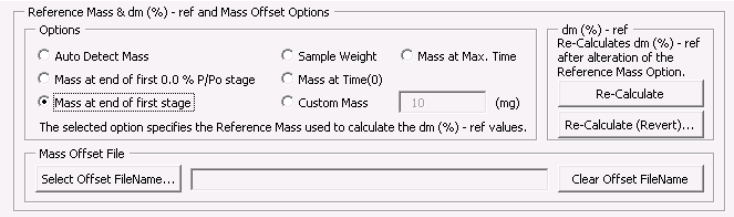
No 'reference mass' available

If there is no data in the file which fits the selected 'reference mass' option, then the following message appears:



Clicking **OK**, the file then opens, with the last saved mass value selected as the 'reference mass'

5.3.12 Mass at end of first stage



If the DVS data file is imported or opened with this option selected, the software selects the mass at the end of the experiment's first P/Po stage as the 'reference mass' to calculate **dm(%)-ref**.

Header Information

The use of this option is indicated in the data file to the right of the header information, along with the 'reference mass' value used:

	A	B	C	D	E	F	G	H	I	J
1	DVS WINDOWS DATA FILE - VERSION 2.06									
2	c:\Program Files\DVS 2_18 Video\method\Starch-10hr steps.SAO									
3	Mon Oct 24 11:00:19 2005									
4	Wheat Starch						Ref. Mass	Mass at end of first stage		
5	10	AutoDry M(0) Active					Ref. Mass	26.3285		
6	10 hr/step, 25C, H2O									
7	Mon Oct 24 11:04:03 2005									

data file header, the cell containing the 'reference mass' value is highlighted in mauve (see below):

10	Time (min)	Mass	dm	dm/dt	RH (samp)	RH (ref)	TEMP (sar	TEMP (ref)	Target RH
605	594.28	26.3289	91.67668	-0.00025	1.6	1.6	24.8	24.8	0
606	595.28	26.3289	91.67668	-0.00013	1.6	1.6	24.8	24.8	0
607	596.28	26.3288	91.67633	-7.4E-05	1.6	1.5	24.8	24.8	0
608	597.28	26.3286	91.67563	-0.00014	1.6	1.5	24.8	24.8	0
609	598.28	26.3285	91.67529	-0.00026	1.6	1.6	24.8	24.8	0
610	599.28	26.3285	91.67529	-0.00036	1.6	1.6	24.8	24.8	0
611	600.28	26.3285	100.0004	-0.00036	1.6	1.7	24.8	24.8	10
612	601.28	26.3471	100.071	0.005417	9.6	9.6	24.8	24.8	10

Figure 5.257: The chosen 'reference mass' – the mass value at the end of the first P/P_o method stage – is highlighted in the DVS data sheet. In this case, the first P/P_o method stage also happens to be set at 0%, but this is not relevant to the selection of the 'reference mass' in this case.

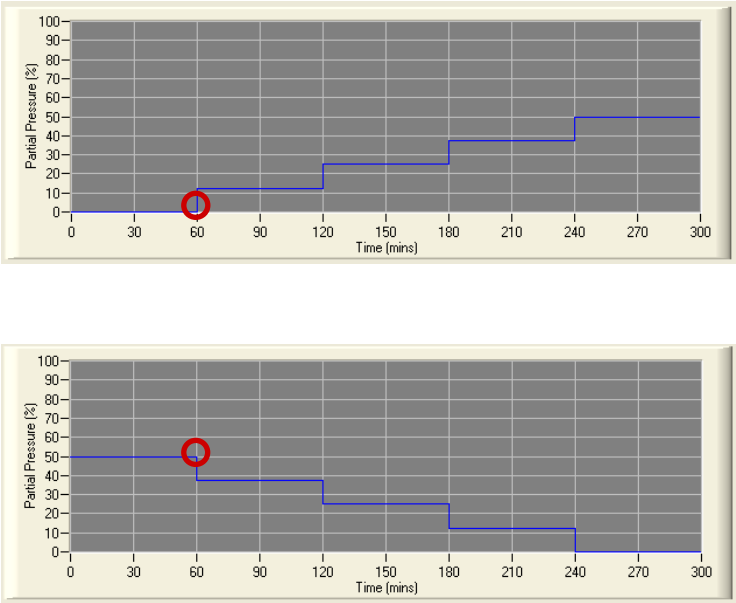
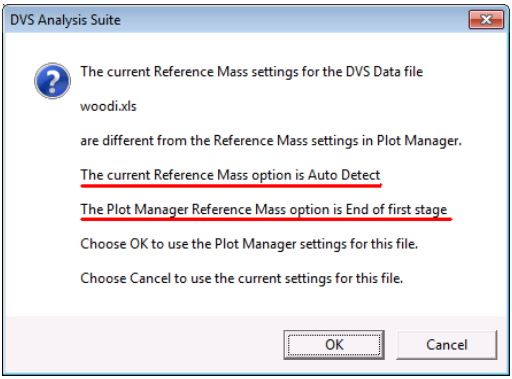


Figure 5.28: In the two examples shown above, the 'reference mass' would be taken from the points circled on each experiment. The value of the partial pressure of the first stage is irrelevant to the selection of the 'reference mass' – it is taken from the end of the fist stage regardless.

Previously Defined 'reference mass'

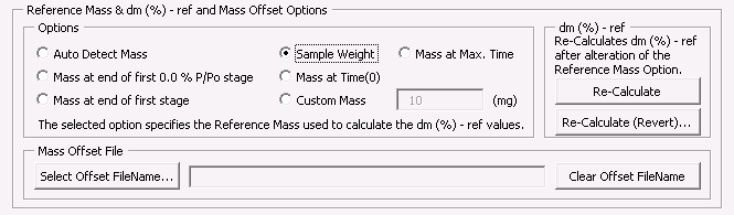
If a file with a previously defined 'reference mass' is opened with a different 'reference mass' setting selected, the following message appears:



The highlighted text shows the current 'reference mass' file setting and the new setting selected in the Plot Manager.

Clicking **OK** will use the new settings, while clicking **Cancel** will keep the file's original 'reference mass' setting.

5.3.13 Sample Weight



If the DVS data file is imported or opened with this option selected, the software selects the **Sample Weight** recorded in the header as the 'reference mass' to calculate **dm(%)-ref**.

For more information on recording the Sample Weight, please refer to Section 5.3.10.1.

Header Information & Highlighted Data

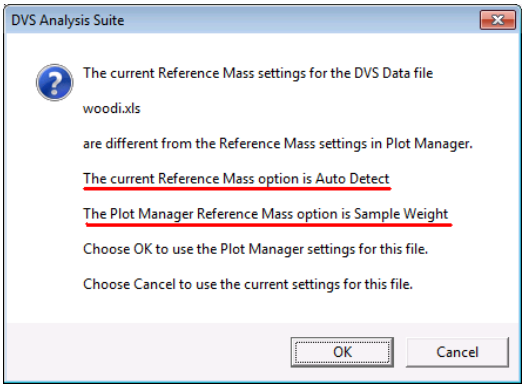
Use of this option is indicated in the data file to the right of the header information, along with the 'reference mass' value used:

	A	B	C	D	E	F	G	H	I
1	DVS WINDOWS DATA FILE - VERSION 2.06								
2	c:\Program Files\DVS 2_18 Video\method\Starch-10hr steps.SAO								
3	Mon Oct 24 11:00:19 2005								
4	Wheat Starch						Ref. Mass	Sample Weight	
5	28.7193	AutoDry M(0) Active					Ref. Mass	28.7193	
6	10 hr/step, 25C, H2O								
7	Mon Oct 24 11:04:03 2005								

In addition to displaying the selected 'reference mass' in the data file header, the cell containing the 'reference mass' value is highlighted in mauve (see above).

Previously Defined 'reference mass'

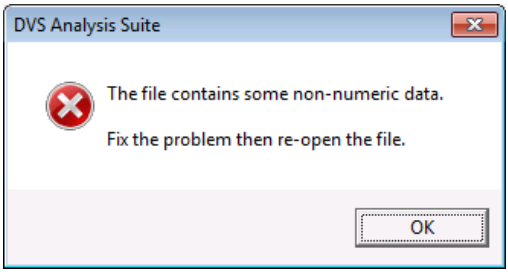
If a file with a previously defined 'reference mass' is opened with a different 'reference mass' setting selected, the following message appears:



The highlighted text shows the current 'reference mass' file setting and the new setting selected in the Plot Manager. Clicking **OK** will use the new settings, while clicking **Cancel** will keep the file's original 'reference mass' setting.

No 'reference mass' available

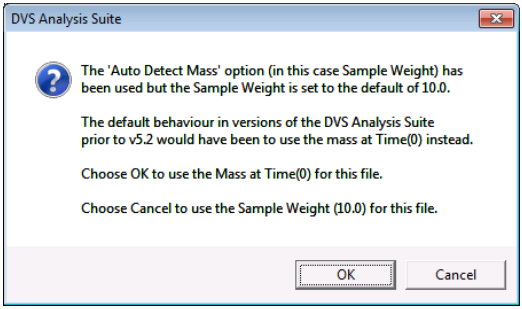
If for some reason there is no data in **Sample Weight** cell, then the following message appears:



In this case, the user will need to manually enter a Sample Weight value in the appropriate cell, or to choose another 'reference mass' option.

Sample Weight Value of '10'

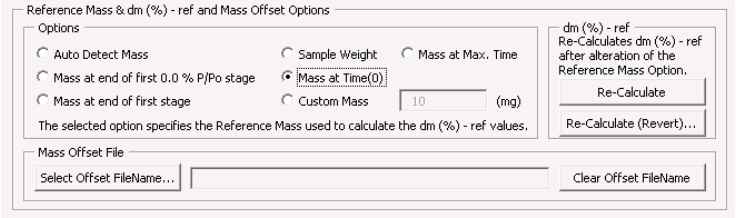
Whenever the **Sample Weight** value in the mass header is '10' and the **Sample Weight** value is to be used as the 'reference mass', the following message will appear:



This is to ensure that the 'reference mass' is not being accidentally based upon the default value of '10' which appears in the **Sample Mass** cell when a value of M(0) is not set.

Clicking **OK** will ensure that the **Mass at Time(0)** option is used instead of the **Sample Weight**, and clicking **Cancel** will allow the value of '10' to be used as the 'reference mass'.

5.3.14 Mass at Time(0)



If the DVS data file is imported or opened with this option selected, the software selects the **Mass at Time(0)** – ie; the first logged mass value - as the 'reference mass' to calculate **dm(%)-ref**.

Header Information

Use of this option is indicated in the data file to the right of the header information, along with the 'reference mass' value used:

	A	B	C	D	E	F	G	H	I
1	DVS WINDOWS DATA FILE - VERSION 2.06								
2	c:\Program Files\DVS 2_18 Video\method\Starch-10hr steps.SAO								
3	Mon Oct 24 11:00:19 2005								
4	Wheat Starch						Ref. Mass	Mass at Time(0)	
5	28.7193	AutoDry M(0) Active					Ref. Mass	28.628	
6	10 hr/step, 25C, H2O								
7	Mon Oct 24 11:04:03 2005								

Highlighted Data

In addition to displaying the selected 'reference mass' in the data file header, the cell containing the 'reference mass' value is highlighted in mauve (see Figure 5.29 below):

4	Wheat Starch							Ref. Mass	Mass at Time(0)	
5	28.7193	AutoDry M(0) Active						Ref. Mass	28.628	
6	10 hr/step, 25C, H2O									
7	Mon Oct 24 11:04:03 2005									
8										
9	60									
10	Time (min)	Mass	dm	dm/dt	RH (samp)	RH (ref)	TEMP (sar	TEMP (ref)	Target RH	
11	0.2	28.628	99.6821	52.91709	1.6	1.6	24.7	24.7	0	
12	1.28	28.5346	99.35688	142.7279	1.6	1.6	24.8	24.7	0	
13	2.28	28.4495	99.06056	142.1021	1.6	1.5	24.8	24.8	0	

Figure 5.29: The chosen 'reference mass' – the mass value at Time(0) – is highlighted in the DVS data sheet.

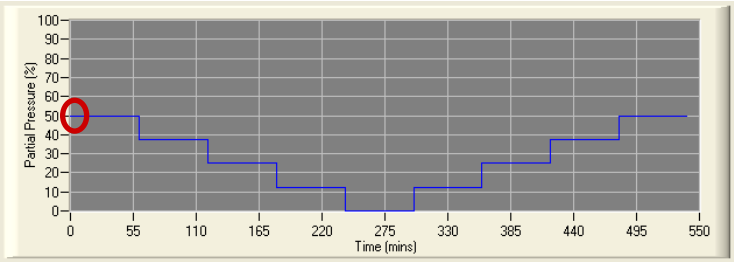
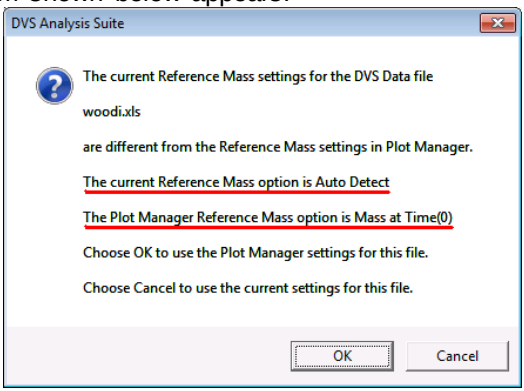


Figure 5.30: In the example shown above, the 'reference mass' would be taken from the circled point in the experiment.

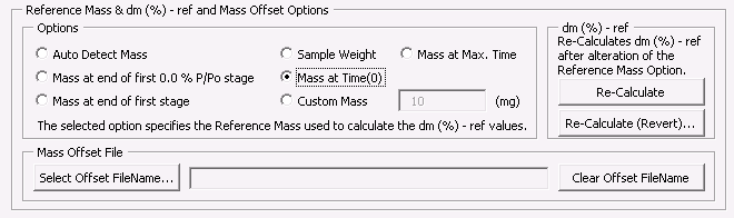
Previously Defined 'reference mass'

If a file with a previously defined 'reference mass' is opened with a different 'reference mass' setting selected, a message of the form shown below appears:



The highlighted text shows the current 'reference mass' file setting and the new setting selected in the Plot Manager.

Clicking **OK** will use the new settings, while clicking **Cancel** will keep the file's original 'reference mass' setting.



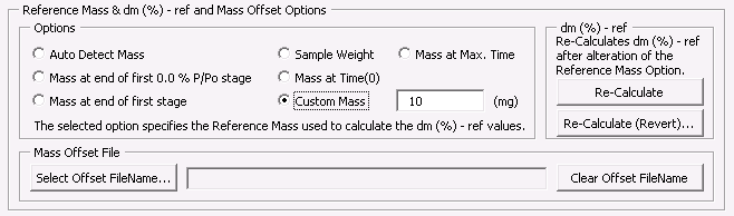
If the DVS data file is imported or opened with this option selected, the software selects the Mass at Max Time – ie; the last logged mass value - as the 'reference mass' to calculate dm(%)-ref.

Similar considerations as above apply to this option and therefore no further elaboration is necessary.

5.3.15 Mass at Max Time, Max & Min Mass

If the DVS data file is imported or opened with this option selected, the software respectively selects the Mass at Max Time, Mass at Max Mass and Mass at Min Mass.

5.3.16 Custom Mass



If the DVS data file is imported or opened with this option selected, the software selects the mass value entered by the

user in the data-entry box as the 'reference mass' to calculate **dm(%)-ref**.

When first activating this option, the data-entry box will appear with a default mass value of 10 mg, which may then be altered. Subsequently, the box will display the last entered value.

Header Information

Use of this option is indicated in the data file to the right of the header information, along with the 'reference mass' value used:

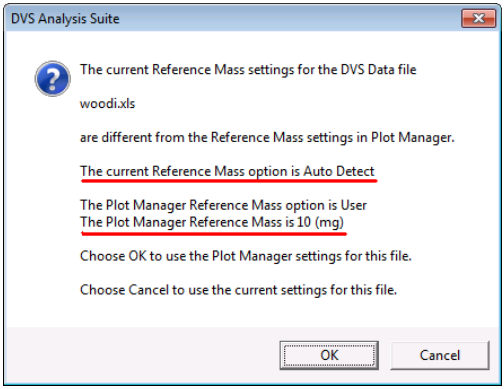
	A	B	C	D	E	F	G	H	I
1	DVS WINDOWS DATA FILE - VERSION 2.06								
2	c:\Program Files\DVS 2_18 Video\method\Starch-10hr steps.SAO								
3	Mon Oct 24 11:00:19 2005								
4	Wheat Starch						Ref. Mass	Custom Mass	
5	10	AutoDry M(0) Active					Ref. Mass	10	
6	10 hr/step, 25C, H2O								
7	Mon Oct 24 11:04:03 2005								
8									

Highlighted Data

As the user-entered **Custom Mass** value is not embedded in the DVS data file, it does not appear highlighted anywhere in the DVS data sheet upon being designated the 'reference mass'.

Previously Defined 'reference mass'

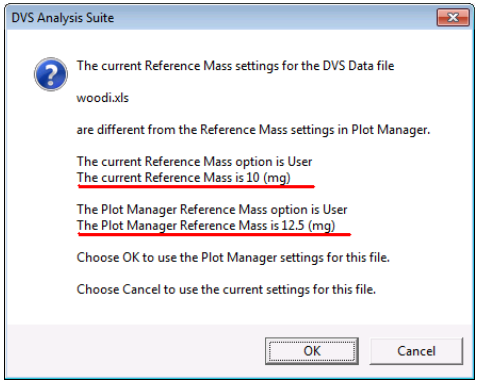
If a file with a previously defined 'reference mass' is opened with a different 'reference mass' setting selected, a message of the form shown below appears:



The highlighted text shows the current 'reference mass' file setting and the new setting selected in the Plot Manager.

Clicking **OK** will use the new settings, while clicking **Cancel** will keep the file's original 'reference mass' setting.

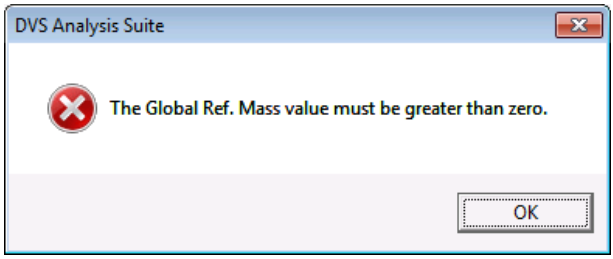
In addition, if the **Custom Mass** option is checked but the data file is opened with a different mass value in the Custom Mass box from the one used previously, the following message appears:



Clicking **OK** will use the new **Custom Mass** settings, while clicking **Cancel** will keep the file's original **Custom Mass** 'reference mass' value.

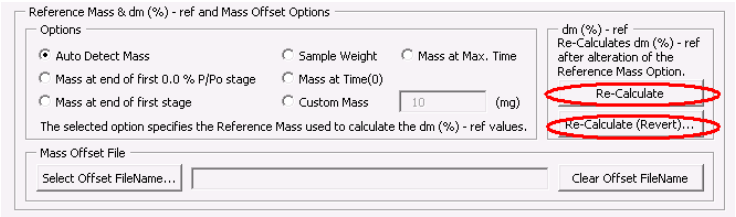
No 'reference mass' available

If there is no mass value contained in the **Custom Mass** data entry box when trying to import or open a file, the following message appears:



Clicking **OK** returns the Plot Manger dialog with the default **Custom Mass** value of 10 displayed in the entry box.

5.3.17 Re-Calculate dm(%)-ref



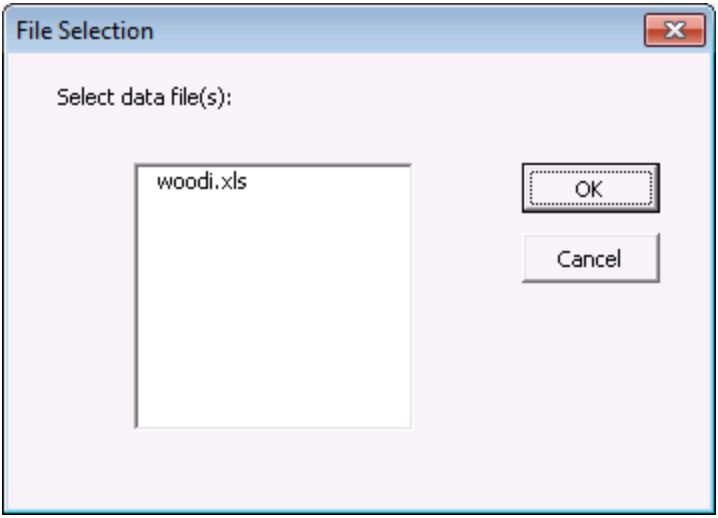
This option allows the user to re-calculate the **dm(%)-ref** column based upon a new 'reference mass' without having to close and re-open the DVS data file. Note the following:-

1) If a Mass Offset File is NOT specified then using Re-Calculate (Offset) will apply the chosen Reference Mass Option to the mass data in files selected for re-calculation
2) If a Mass Offset File IS specified then using Re-Calculate (Offset) will first subtract the Offset File masses and THEN will apply the chosen Reference

Mass Option to the mass data in files selected for re-calculation

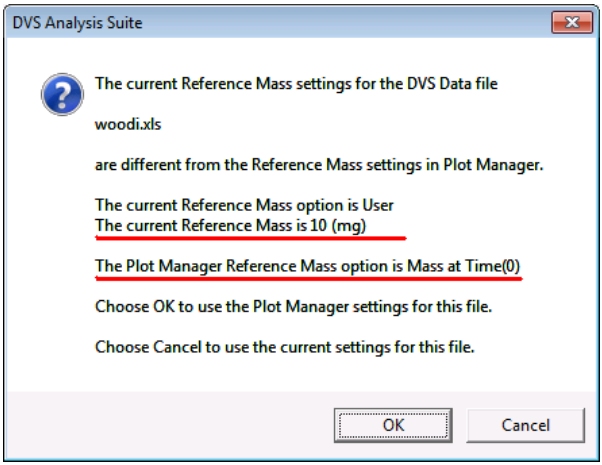
3) Irrespective of whether a Mass Offset File is specified then using Re-Calculate (Revert) will first re-instate the original masses (if any) and THEN will apply the chosen Reference Mass Option to the mass data in files selected for re-calculation

Upon opening the DVS data file and selecting the preferred 'reference mass' option, clicking the **Re-Calculate (Offset)** or **Re-Calculate (Revert)** button calls up the following window:



If several DVS data files are open, they will all appear on the list shown above.

Choosing the relevant file and clicking **OK** then returns the following window, asking the user for confirmation that a new 'reference mass' option is to be used:



Clicking **OK** then commences the re-calculation of **dm(%)-ref** based upon the newly chosen 'reference mass' option.

The DVS Plot Manager window is then returned, with the relevant DVS data file using the newly chosen 'reference mass' to calculate the **dm(%)-ref** data.

5.3.18 Altering dm %-ref data

The DVS Baseline Correction and DVS Vapour Content Offset modules can be used to modify the dm (%) - ref column in special circumstances (see Chapter 7 - DVS Baseline Correction and Chapter 8 - DVS Vapour Content Offset).

5.3.19 Altering default behavior

Next to the Plot Buttons there is a small toggle button. When depressed it stops the secondary adsorbate being plotted.

6 DVS Isotherm Analysis

6.1 What does this module do?

The **DVS Isotherm Analysis** module calculates and plots isotherms from raw DVS data.

An isotherm is a plot of the equilibrium amount of vapour sorbed by a sample versus the vapour's partial pressure – all measured at a constant temperature.

Figure 6.1 below shows an example of an isotherm measured at 25°C with a sample absorbing water vapour. Both sorption and desorption curves are shown in this case (explained below).

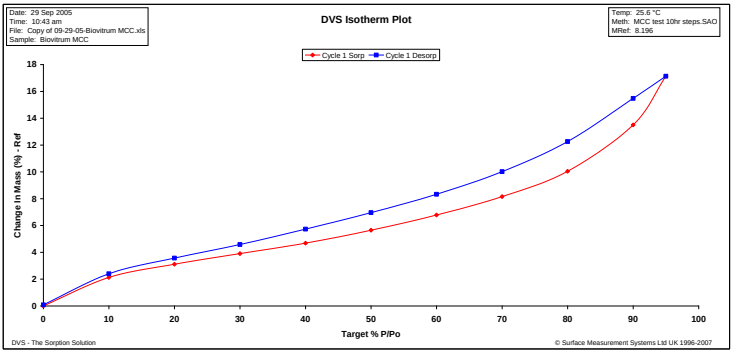


Figure 6.1: An isotherm of a solid sample absorbing water vapour at 25°C

In a DVS experiment, an isotherm is calculated based on the sample's equilibrium uptakes at each partial pressure step. Consequently for an isotherm to be accurate, the sample must reach equilibrium with the vapor at each $\%P/P_o$ step.

This will be visible in the data by a flat mass response (i.e. mass has reached a stable value at each partial pressure step).

Figure 6.2 below shows an example of DVS raw mass vs time data that could be used to construct an isotherm:

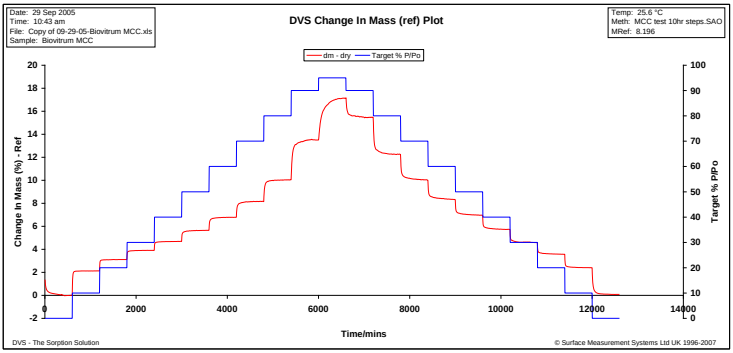


Figure 6.2: Raw DVS data that could be used to construct an isotherm

6.2 Why is this useful?

Isotherms are often the first step in determining any number of substrate surface chemical properties. They are important as their shapes also reveal much information about the sorption phenomena taking place between the sample and vapour.

There are typically 5 different shapes of isotherm (shown together in Figure 6.3 below), according to the BDDT (Brunauer, Deming, Deming, and Teller) nomenclature.

Examples of each isotherms are displayed below, including their descriptions and physical significance in each case.

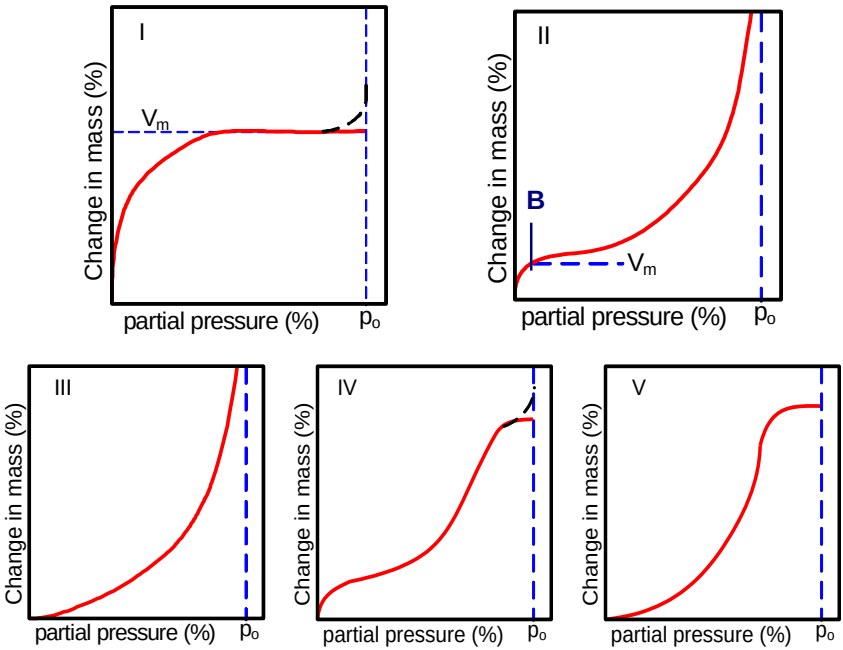
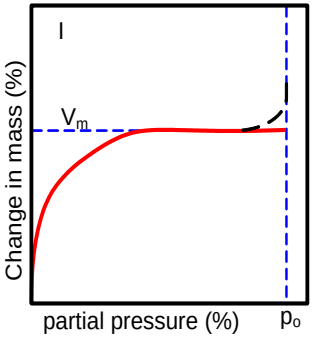


Figure 6.3: The five **BDDT isotherm classifications**

6.2.1 Type I isotherms

These show a fairly rapid rise in the amount of adsorption with increasing partial pressure up to a limiting value. They are referred to as Langmuir-type isotherms and are obtained when adsorption is restricted to a monolayer.



Chemisorption isotherms approximate to this shape. Also, microporous materials (i.e. activated carbons and molecular sieves) can yield a Type I isotherm.

Figure 6.4 below shows an example of a Type I isotherm calculated from an experiment with water vapour on Granular Activated Carbon (GAC) at 25°C. Figure 6.5 shows the raw data from which the isotherm was calculated

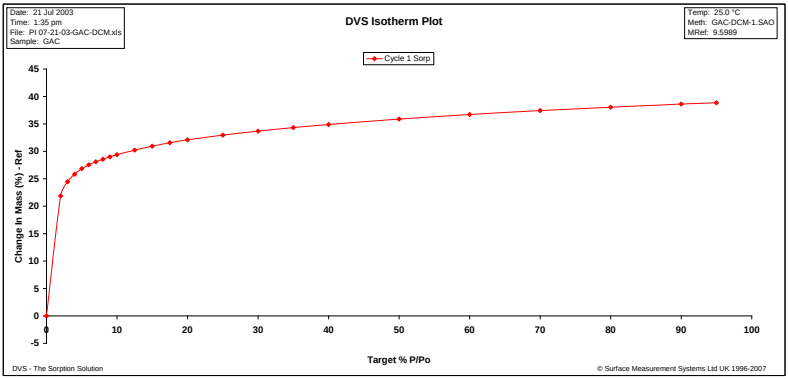


Figure 6.4: An experiment with water vapour on Granular Activated Carbon (GAC) at 25°C, showing a **Type I isotherm** Shape.

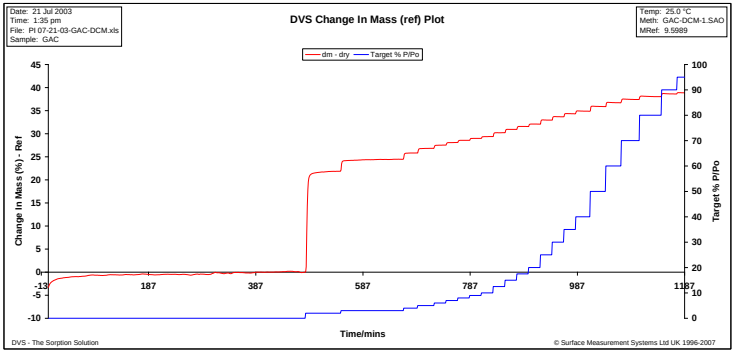
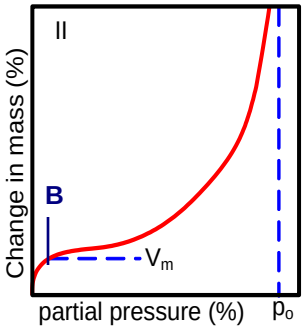


Figure 6.5: The raw DVS mass change (%) vs time data from which the isotherm was calculated (the blue line is target $\%P/P_o$).

6.2.2 Type II isotherms



Often referred to as sigmoid isotherms, they are frequently encountered and signify multilayer physical adsorption on nonporous solids.

For such solids, point B (refer picture, right, and Figure 6.3) represents the formation of an adsorbed monolayer. The remainder of the curve represents multilayer adsorption as for non-porous solids. For a Type II mechanism, the heat of adsorption is much higher than the heat of condensation (i.e. the molecules would rather interact with the surface than with each other). A Type II isotherm can be used to calculate a BET surface area, which can be done using the DVS Advanced Analysis Macros. For more information on the BET equation, see Chapter 6 of the Advanced Analysis Suite manual.

Figure 6.6 below shows an example of a Type II isotherm, obtained from an experiment using 1-butanol vapour on alpha lactose monohydrate at 25°C.

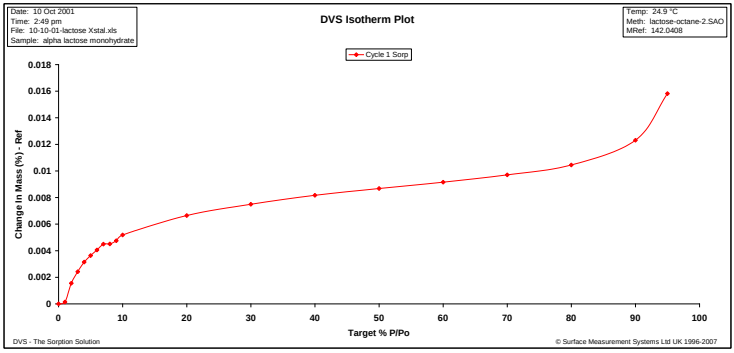
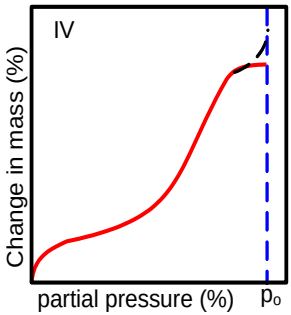


Figure 6.6: A Type II isotherm produced from 1-butanol vapour on alpha lactose monohydrate at 25°C.

6.2.3 Type IV isotherms



These level off near the saturation vapor pressure and are considered to reflect capillary condensation in porous solids - the effective pore diameters usually being between 2 nm and 20 nm.

The upper limit of adsorption is mainly governed by the total pore volume. A type IV isotherm will have a characteristic hysteresis loop at medium to high partial pressures.

As with Type II mechanisms, the heat of adsorption is much higher than the heat of condensation at low coverages and the isotherm can be used to calculate a BET surface area.

Figure 6.7 below shows an example of a Type IV isotherm, and Figure 6.8 shows the raw data from which the isotherm originated.

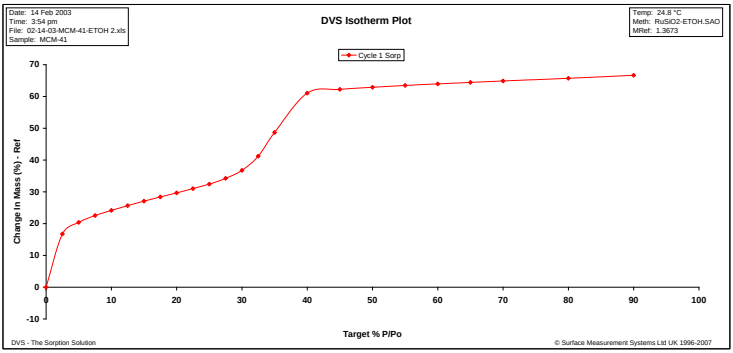


Figure 6.7: A Type IV isotherm produced from EtOH vapour on MCM-41 at 25°C.

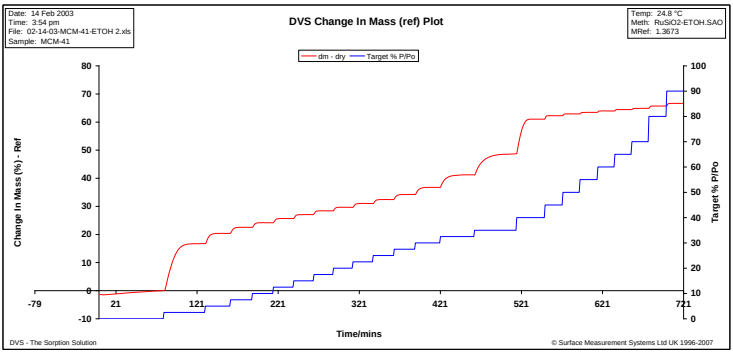


Figure 6.8: The raw mass change (%) vs time data from which the isotherm was calculated.

6.2.4 Type III and Type V isotherms

These show no rapid initial uptake of vapour, and occur when the forces of adsorption in the first monolayer are relatively small.

Water on hydrophilic, organic materials (i.e. starches, drugs, foods) often exhibits this behavior. For a Type III/V mechanism the heat of sorption is only slightly higher than the heat of condensation, so the thermodynamic incentive for surface sorption is low, leading to island or cluster sorption.

As a result, Type III/V isotherms *cannot* be used to calculate BET surface areas (even if the analysis yields an excellent correlation coefficient).

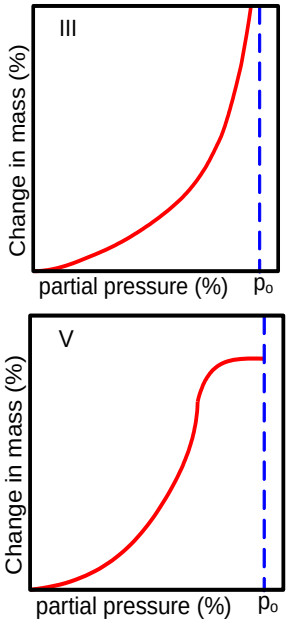


Figure 6.9 below shows an example of a type III isotherm calculated from a water vapour on caffeine experiment at 25°C.

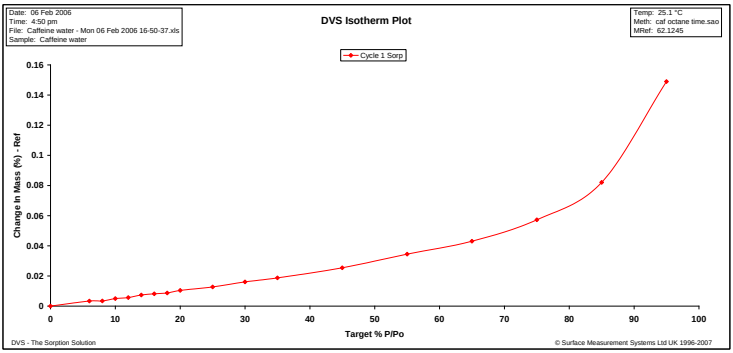


Figure 6.9: A Type III isotherm resulting from water vapour on caffeine at 25°C.

6.2.5 Hysteresis

When running a DVS experiment, it is possible to not only gradually increase the vapour pressure (adsorption phase), but also to decrease it (desorption phase). When running such an experiment, isotherms can reveal another important phenomenon known as hysteresis.

This term describes the difference in the amount adsorbed between the sorption and desorption phases of the isotherm.

The shape and location of the hysteresis can often yield important information about the sorption mechanism and sample structure. Below are some examples of different hysteresis gaps and their physical significance.

6.2.5.1 No hysteresis

When there is no hysteresis, this is typically indicative of a surface adsorption mechanism (Figure 6.10 below). There is most likely little to no bulk absorption and the sorption kinetics

will typically be relatively fast. This is a completely reversible sorption mechanism.

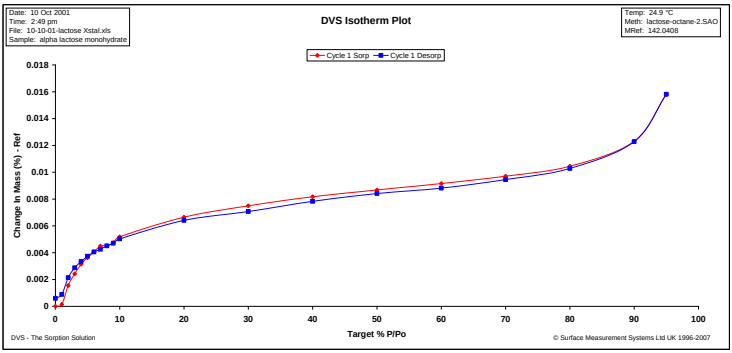


Figure 6.10: An example of no hysteresis.

6.2.5.2 Broad hysteresis gap

A broad hysteresis gap over the entire partial pressure range is often indicative of a bulk absorption mechanism. The vapor's path out of the bulk of the material is diffusion-limited.

In the case shown in Figure 6.11 below, bulk absorption becomes dominant at 70% P/P_0, as indicated by the sharp increase in vapor sorbed. The isotherm then shows that vapour desorption is significantly diffusion-limited across the entire desorption range.

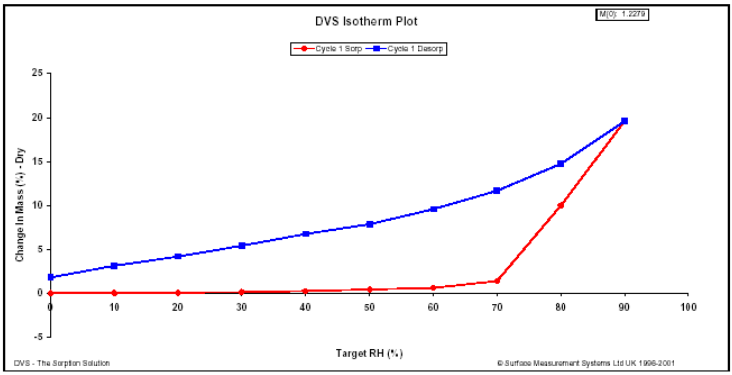


Figure 6.11: Bulk absorption indicated by an isotherm's broad hysteresis gap

6.2.5.3 Sharp hysteresis gap

A sharp hysteresis gap over a defined partial pressure range is often indicative of hydrate/solvate formation. In Figure 6.12 below, a reversible hydrate is formed at 45% P/P_o during the sorption stage and returns to its dehydrated state at 35% P/P_o during the desorption stage.

If the molecular weight of the starting material is known, then it is possible to calculate the hydrate stoichiometry based on the percentage mass uptake over the hysteresis gap.

To illustrate, consider an anhydrous material, Sample A, which forms a hydrated species at a particular vapour partial pressure. The stoichiometry, S, of the hydrate may be calculated according to Equation 2 below:

$$S = \frac{WG}{100\%} \times \frac{MW}{18.01 amu / watermolecule}$$

$$= HydrateStoichiometry$$

Equation 2

where: MW = molecular weigh of sample A;
WG = percentage weight gain of sample A at the partial pressure at which it forms the hydrate

The above equation assumes formation of a stoichiometrically, completely, hydrated species.

Similar analysis could be applied to solvate stoichiometries by simply replacing the molecular weight of water (18.01 amu) with the molecular weight of the vapor used in Equation 2.

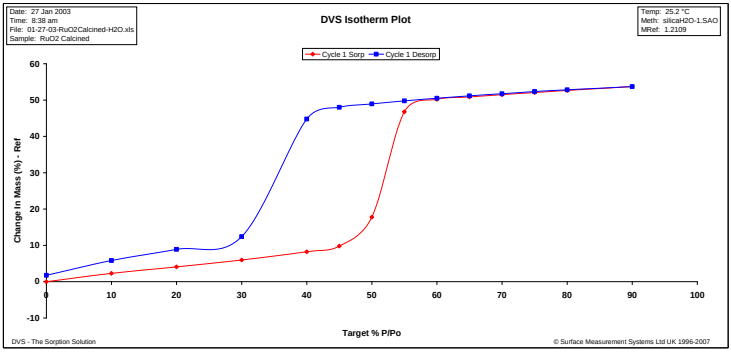


Figure 6.12: Hysteresis indicating hydrate formation

6.2.5.4 Hyseresis gap at medium to high $\%P/P_o$

A hysteresis gap at medium to high partial pressures is often indicative of a sample containing mesopores (2 < pore size < 50 nm).

Figure 6.13 below shows the results for octane sorption on a mesoporous alumina sample. The hysteresis could also be indicative of capillary condensation.

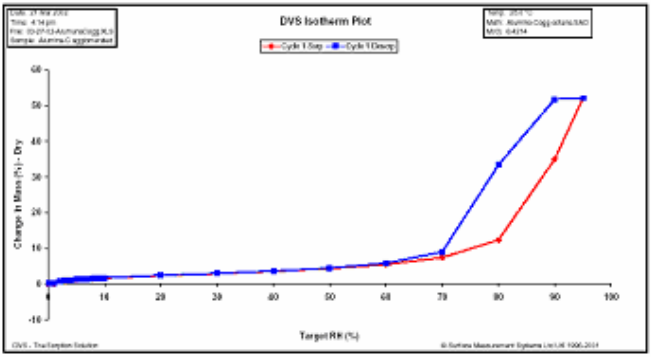


Figure 6.13: An isotherm produced by a mesoporous sample

6.2.5.5 Hysteresis gap at low partial pressures

A hysteresis gap at very low partial pressures can be indicative of a micoporous sample (pore size < 2 nm).

Often the isotherm will not return to the origin at the end of the desorption phase, because the vapor remains 'trapped' in the sample's micropores.

Figure 6.14 below shows an example of octane vapor sorption on a microporous zeolite sample. The octane is trapped in the zeolite's micropores; thus, the sample mass does not return to the dry value at the end of the desorption drying stage.

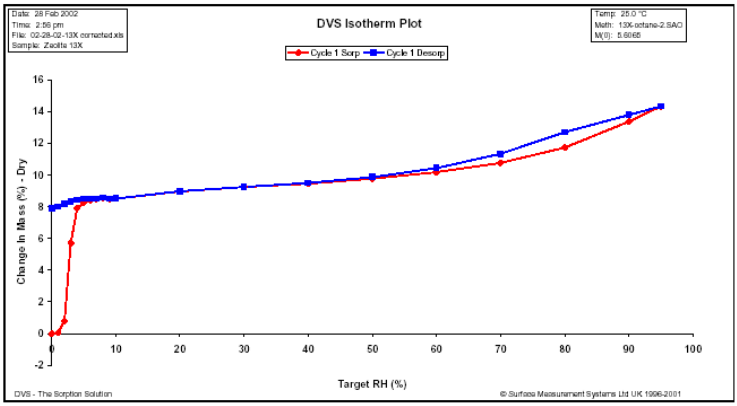


Figure 6.14: Low partial pressure hysteresis indicating the presence of micropores in zeolite

6.3 What experimental set-up is required to create isotherms?

As with most plotted scientific data, isotherms are improved with the number of points used to construct them.

Typically, an isotherm can be constructed by a minimum of 11 points – representing partial pressures of 0, 10, 20, 30, 40, 50, 60, 70, 80, 90, and 95% or 98% (the maximum partial pressure attainable using a DVS is 98%). More points can be added to the isotherm by simply adding more partial pressures into the experimental set-up.

The amount of time for which each $\%P/P_o$ stage should run will depend upon the nature of the sample and vapour, and the amount of sample. A single hour for each stage is suggested as a good first approximation, which can then be altered depending upon the results of the first experiment.

Also, a dm/dt criteria may be used instead of time – please refer to the Control Software manual for details.

Additionally, as the name 'iso-therm' suggests, the experiment is to be run at a single temperature. Different isotherms may be run at different temperatures for comparison, of course.

6.3.1 Sorption isotherms

For a simple sorption isotherm, therefore, a DVS experimental set-up could be as shown below in Figure 6.15:

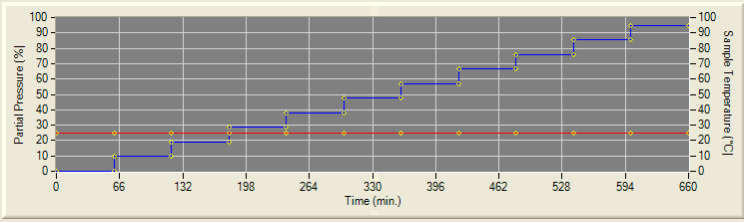


Figure 6.15: A standard DVS experimental set-up for producing a 'basic' sorption isotherm – the blue lines show the set-point partial pressures, and the red line the set temperature.

This type of experiment would produce a single-line isotherm as exemplified below:

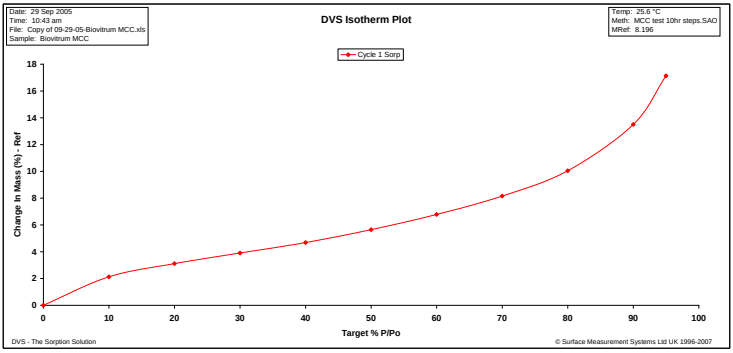


Figure 6.16: A 'basic' sorption isotherm.

6.3.2 Sorption/Desorption Isotherms

If the user wishes to see any hysteresis effects, then a desorption stage must be added to the experiment by running a full cycle (Figure 6.17 below).

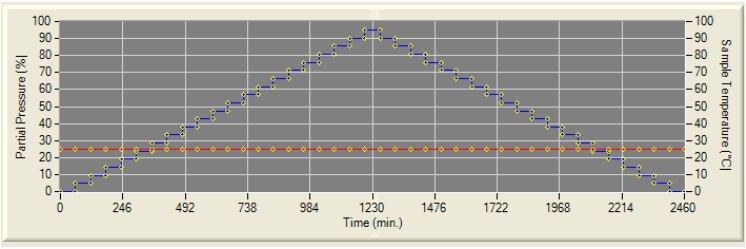


Figure 6.17: A full-cycle DVS experiment for producing an isotherm featuring a 'sorption' and 'desorption' curve

An isotherm of the type shown below would then result:

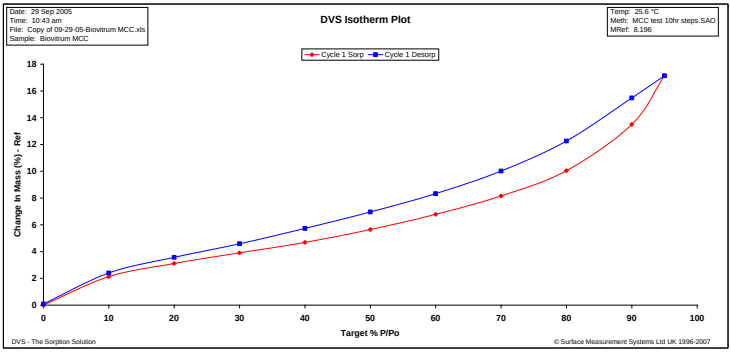


Figure 6.18: An isotherm showing sorption (red) and desorption (blue).

6.3.3 Multiple-cycle isotherms

Finally, if multiple sorption and desorption cycles are to be recorded, then a multiple-cycle experiment will be required (Figure 6.19 below):

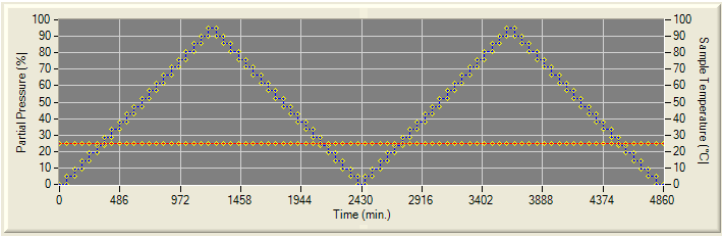


Figure 6.19: A multiple-cycle DVS experiment for producing a set of sorption-desorption isotherm pairs. Each cycle shown above would produce one such pair

Figure 6.20 below shows an example of the resulting isotherm plots:

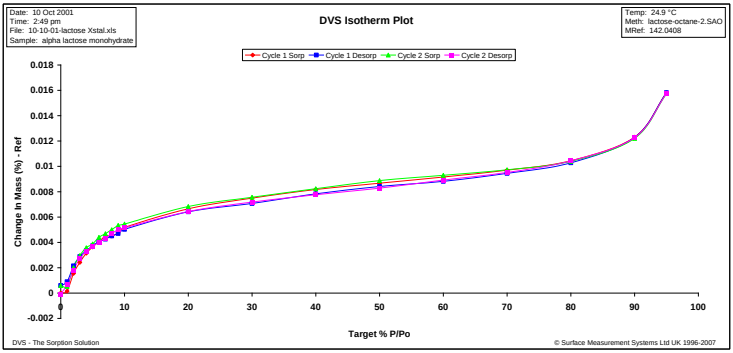


Figure 6.20: A multiple isotherm plot.

6.4 How to use this module

Clicking the **DVS Isotherm Manager** button calls up the main dialog screen for the module as shown in Figure 6.21a and Figure 6.22b below. On the left hand side of the screen are the calculation, plotting and 'file opening'/'file importing' buttons, as well as the DVS Version selection panel (see Section 4.3.2). On the right hand side are the selection areas for the calculation and plot options.

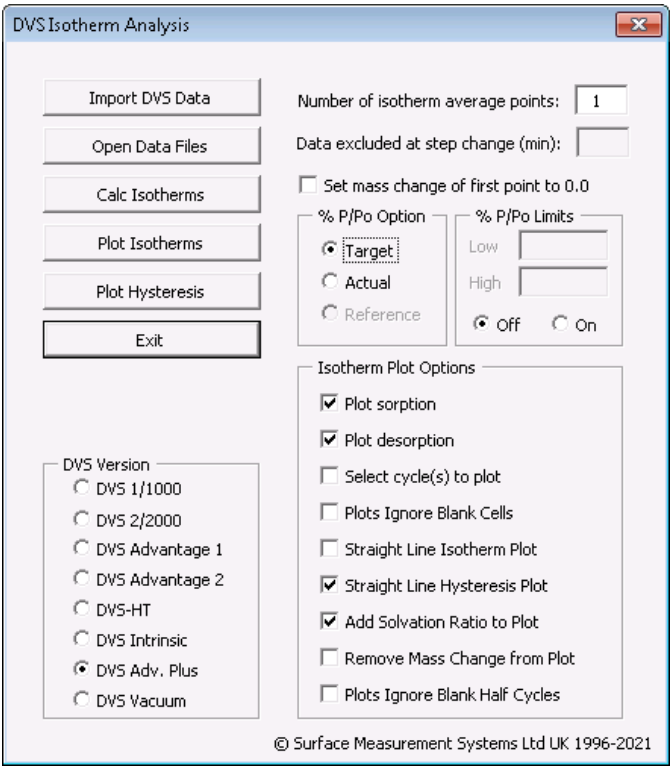


Figure 6.21a: The **DVS Isotherm Manager** main dialog screen (DVS Advantage Plus version)

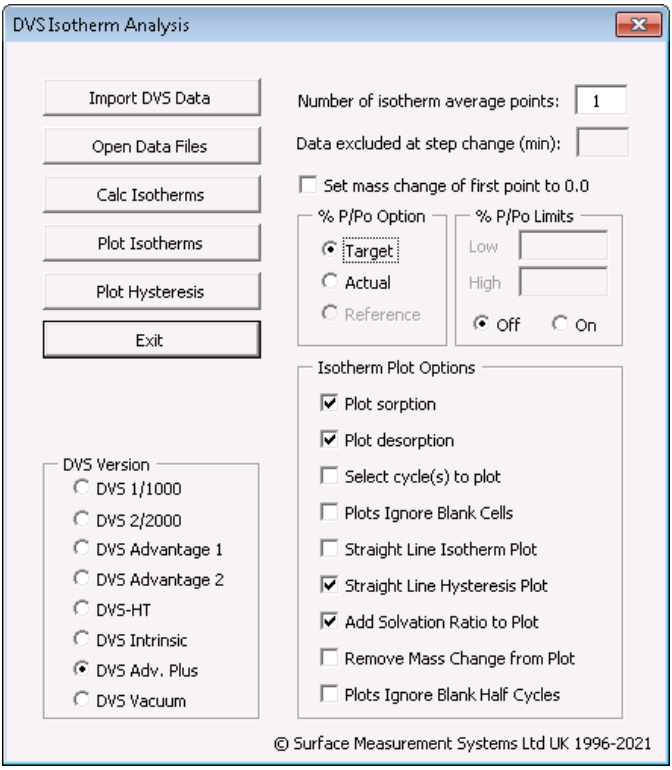
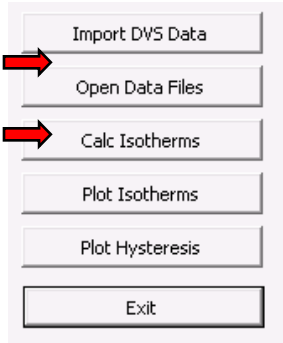


Figure 6.22b: The **DVS Isotherm Manager** main dialog screen (DVS Vacuum version)

6.4.1 Import DVS Data & Open Data File

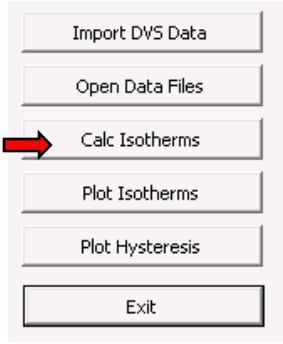
These functions are explained in detail in Sections 4.2 and 4.3, respectively.



Before any isotherm calculation or plotting may be carried out, a raw DVS data file must first be open in Excel using these buttons, which appear on every module main panel - the file(s) may be imported and opened from any module before using the isotherm functions.

If the DVS Version is set to DVS Vacuum then there is a small toggle button to the left of the Calc and Plot buttons which can be used to switch from Relative Pressure to absolute Pressure on plots. This feature is also available for Plot Manager.

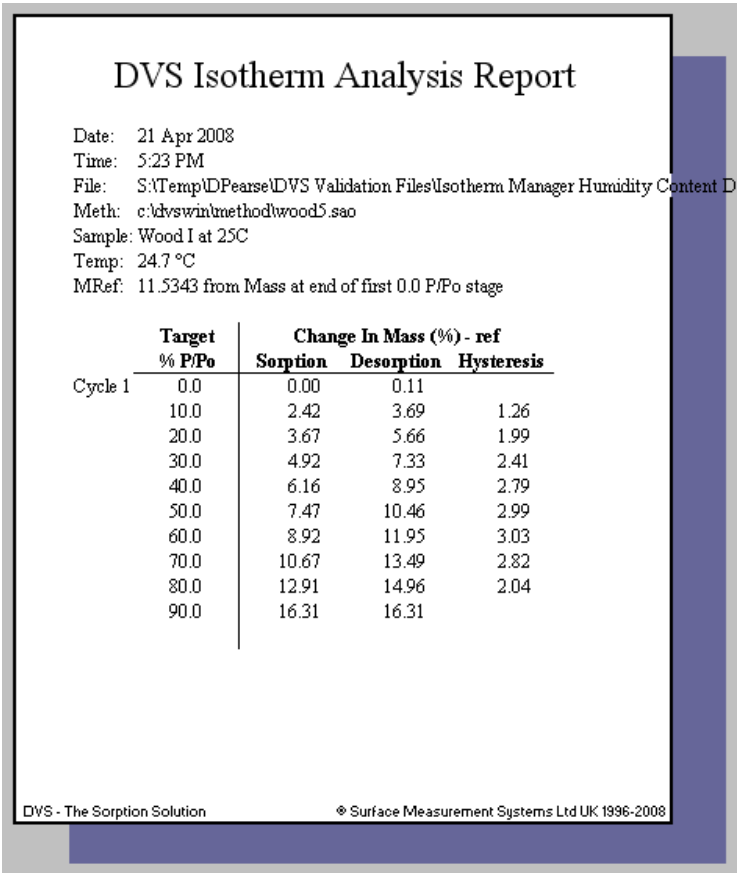
6.4.2 Calc Isotherms



Clicking the **Calc Isotherms** button produces an isotherm analysis report from an appropriate DVS data file.

The isotherm appears in the Excel DVS data file in a new sheet entitled **Iso Report**.

It tabulates 'dm (%) -ref' data with % P/P_o and breaks each isotherm cycle down into sorption, desorption and hysteresis (Figure 6.23 below).



DVS Isotherm Analysis Report

Date: 21 Apr 2008
Time: 5:23 PM
File: S:\Temp\DPearse\DVS Validation Files\Isotherm Manager Humidity Content D
Meth: c:\dvswin\method\wood5.sao
Sample: Wood I at 25C
Temp: 24.7 °C
MRef: 11.5343 from Mass at end of first 0.0 P/Po stage

	Target % P/Po	Change In Mass (%) - ref Sorption	Desorption	Hysteresis
Cycle 1	0.0	0.00	0.11	
	10.0	2.42	3.69	1.26
	20.0	3.67	5.66	1.99
	30.0	4.92	7.33	2.41
	40.0	6.16	8.95	2.79
	50.0	7.47	10.46	2.99
	60.0	8.92	11.95	3.03
	70.0	10.67	13.49	2.82
	80.0	12.91	14.96	2.04
	90.0	16.31	16.31	

DVS - The Sorption Solution

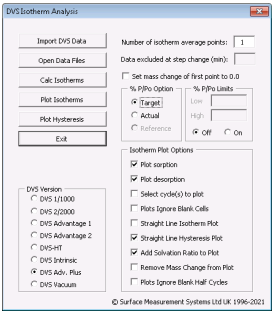
® Surface Measurement Systems Ltd UK 1996-2008

Figure 6.23: A **DVS Isotherm Analysis Report**.

The isotherm calculation can handle different target % P/P_o values in the sorption and desorption half cycles of an isotherm as well as irregular or incomplete cycles.

Before clicking the **Calc Isotherms** button, however, there are two sets of options that must be specified on the main dialogue screen; **Number of average isotherm points** and **Isotherm %P/P_o Option**.

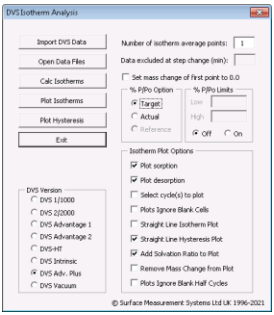
6.4.2.1 Number of isotherm average points



This box specifies the number of points at the end of each % P/P_o step over which the 'dm (%) – ref' figure is averaged to create a point on the isotherm; e.g. if '5' is entered in the edit box, each isotherm point will be an average of the last five 'dm (%) – ref' points of the % P/P_o step.

The recommended number of points to use is usually three to five.

6.4.2.2 Data excluded at step change (min)



This box specifies data excluded at the end of each % P/P_o step over which the 'dm (%) – ref' figure is NOT acquired to

create a point on the isotherm; e.g. if '5' is entered in the edit box, each isotherm point will NOT be taken less than 5 minutes prior to transition to the next % P/P_o step change.

This box is useful where a Raman instrument is also being used and the Raman use alters the data prior to a % P/Po step change.

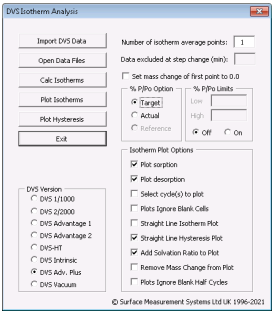
There is no recommended value for data excluded.

The box can only become active if the Config option (DataExcluded tab) which controls it is set to Exclude Data.

6.4.2.3 Set mass change of first point to 0.0

By selecting "Set mass change of first point to 0.0" when the report is written 0.0 will be written to the first isotherm point mass change if that row's partial pressure is 0.0.

6.4.2.4 Isotherm % P/P_o Option & % P/Po Limits



This box allows the user to specify the % P/P_o type - target, sample or reference (DVS 1/1000 & DVS 2/2000 only) - that is used in the isotherm calculation and report. It also allows a range to be used (optional).

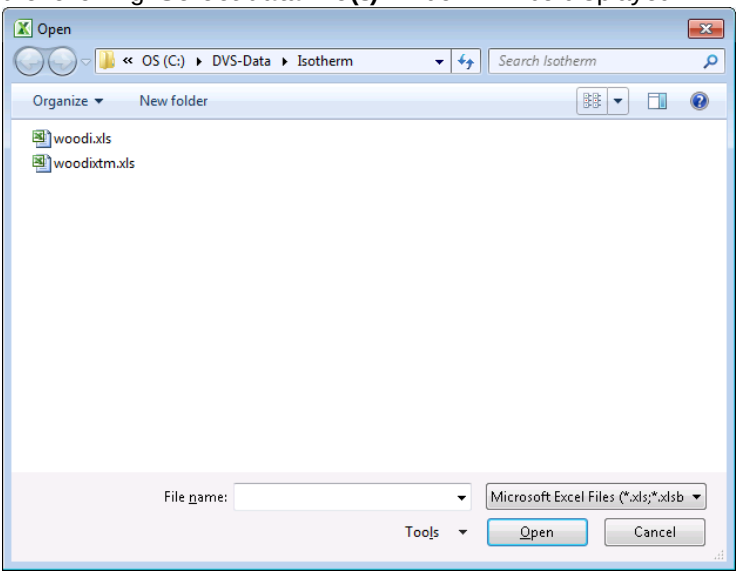
The default option is **Target** % P/P_o, however the commonly used option for isotherms is **Sample**. Selecting Target produces only one % P/P_o column in the Isotherm Report otherwise there is a separate sorption and desorption column.

6.4.2.5 Multiple isotherm reports

It is possible to calculate isotherm reports for several data files simultaneously.

This is achieved by firstly using the **Open Data File** button to open all the files for which an isotherm report is to be calculated.

Following this, the **Calc Isotherms** button must be clicked; the following **Select data file(s)** window will be displayed:



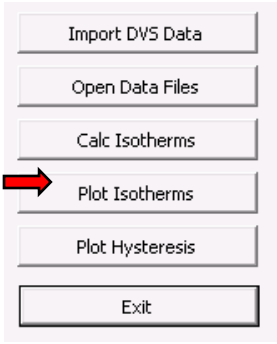
The DVS data files which have just been opened will appear in the list – each file can then be selected for isotherm calculation by clicking on it. When all the desired files have

been selected, press **OK** – an isotherm report like the one shown in Figure 6.23 will appear in each individual DVS data file.

Note that any entered parameters or options for the isotherm calculation are applied to ALL the files selected for isotherm calculation.

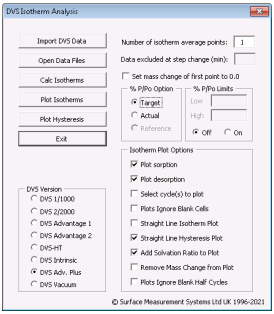
6.4.3 Plot Isotherms

Clicking the **Plot Isotherms** button produces an isotherm plot from a DVS data file for which an isotherm analysis report has already been calculated.



6.4.3.1 Isotherm Plot Options

Before creating a plot, the plot options must be set by ticking the required check boxes in the **Isotherm Plot Options** selection area on the Isotherm Analysis main panel:



At least one of the first two options - **Plot sorption** and **Plot desorption** – must be selected in order to plot an isotherm.

Plot sorption

With this option checked, only the sorption stage of the isotherm will be plotted, as shown below in Figure 6.24:

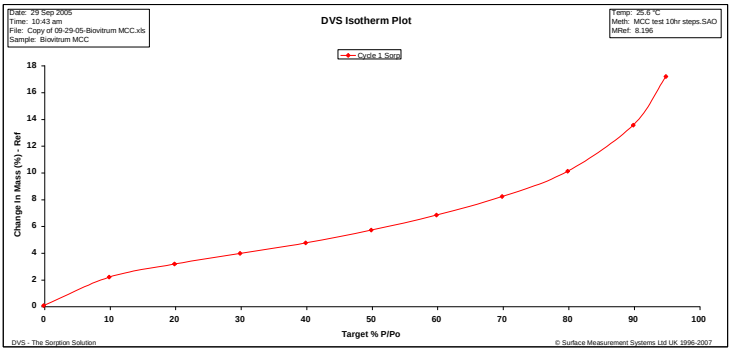


Figure 6.24: The sorption stage of an isotherm.

Plot desorption

Checking this option will produce a plot which includes any desorption isotherm data which has been calculated, as shown below in Figure 6.25:

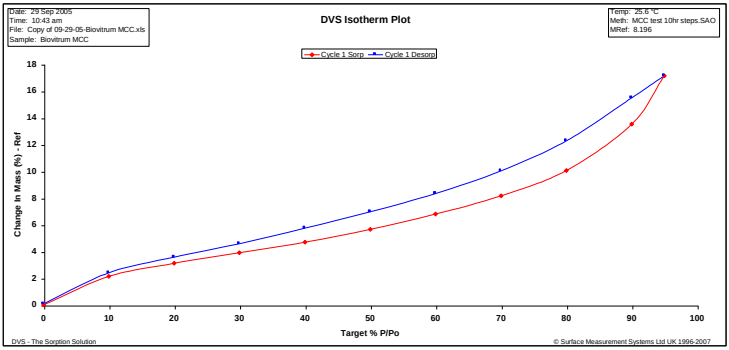
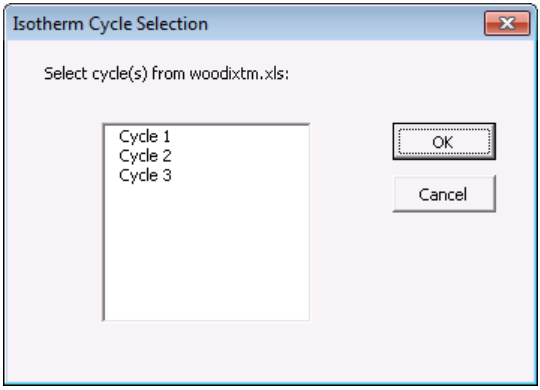


Figure 6.25: The desorption stage (blue) added to the isotherm.

Select cyles(s) to plot

This check box is relevant only to multiple-cycle data. It gives the user the option of selecting which cycle(s) of the data to include in the isotherm plot.

If this option is checked, then after clicking the **Plot Isotherms** button, the following panel will appear:



This Isotherm Cycle Selection panel allows the user to select what cycle(s) of a multiple-cycle experiment to include in the isotherm plot.

Cycles may be selected by clicking on them – multiple selections may be made by holding down the **Ctrl** key while clicking on the desired cycles.

After making the selection and clicking **OK**, the newly plotted isotherm(s) will appear on a new sheet in the DVS Excel file.

Figure 6.26 below shows an example of multiple cycles plotted on one graph.

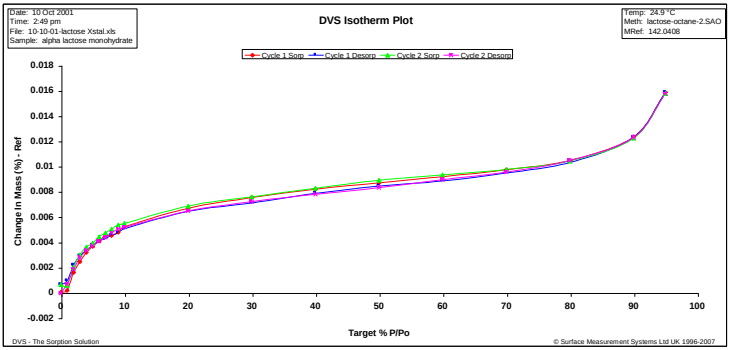


Figure 6.26: A multiple-cycle isotherm plot.

Note: If the **Select cycle(s) to plot** box is not ticked, all cycles of a multiple-cycle experiment will be plotted. This option has no effect on plots produced for single cycle experiments.

Plots Ignore Blank Cells/Plots Ignore Blank Half Cycle

On rare occasions the Isotherm Analysis Report will show a gap in its data – a cell may be left blank. Ticking the **Plots Ignore Blank Cells/Plots Ignore Blank Half Cycle** options ensures that a blank cell/half cycle does not translate into a gap/missed cycle in the plotted isotherm.

Straight Line Isotherm Plot/Straight Line Hysteresis Plot

There are two ways in which Isotherm and Hysteresis data may be presented in a graph;

Curved Plot: A curved line is fitted to the data points on the graph, as shown below in Figure 6.27:

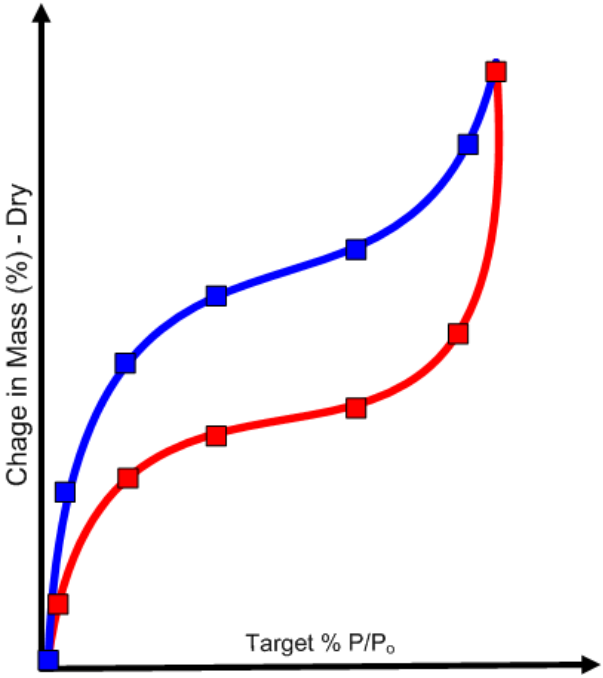


Figure 6.27: A representation of a curved isotherm plot.

This plot type is often favoured for presentational reasons, especially in cases where there are a lot of data points on the graph.

Straight Line Plot: A straight line is drawn between each point on the graph, as shown in Figure 6.28 below:

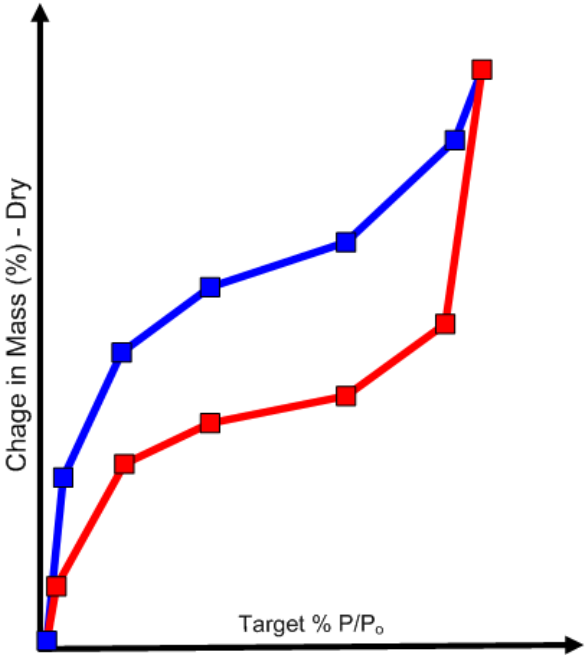
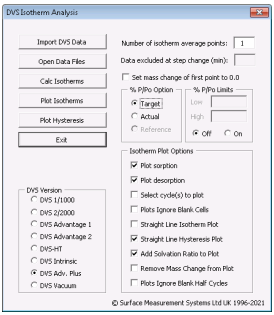


Figure 6.28: A representation of a straight line isotherm plot.

In the case of isotherms, straight line plots can sometimes make it easier to visualise the hysteresis gap between each pair of adsorption and desorption points.

The **Isotherm Plot Options** area allows the user to check or uncheck the Straight Line option for both isotherms and hysteresis plots:



Leaving the Straight Line option un-checked will result in a curved plot.

Examples of curved and straight line isotherm plots are shown below.

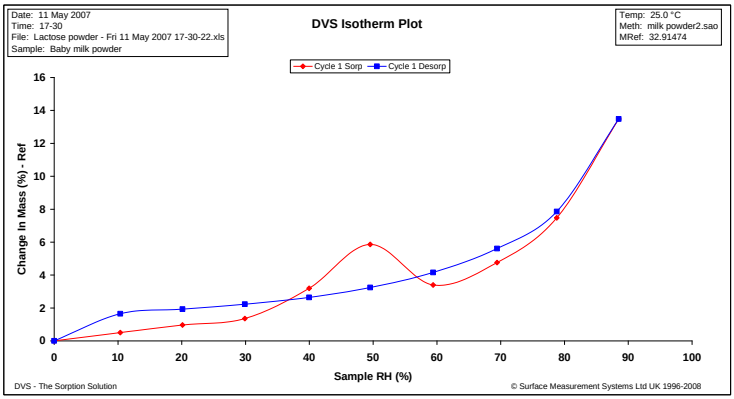


Figure 6.29: A curved isotherm (adsorption and desorption) plot.

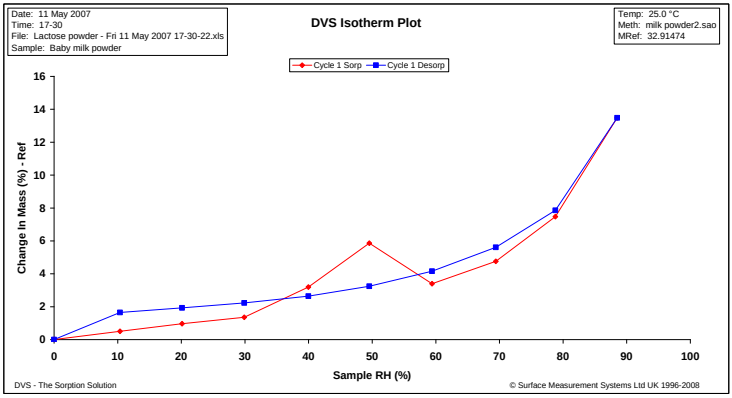


Figure 6.30: The same isotherm plotted using the **Straight Line Isotherm Plot** option.

Note: The **Straight Line Isotherm Plot** option is by default un-checked when the Analysis Suite software is first opened – the user's choice to select/deselect this option is then subsequently preserved.

Add Solvation Ratio to Plot

Clicking Add Solvation Ratio to Plot ensures that solvation ratio curves are added to isotherm or hysteresis plots:

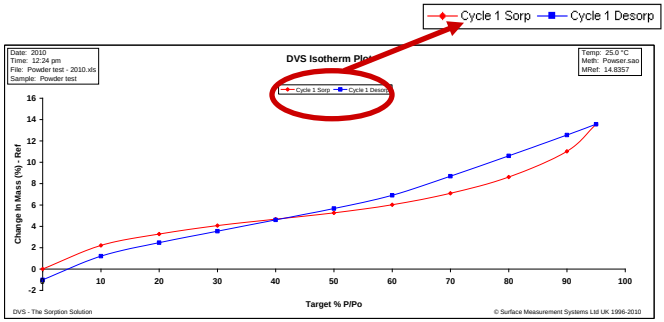


Figure 6.31: Sorption and Desorption lines plotted in an isotherm, without the Solvation Ratio added to the plot.

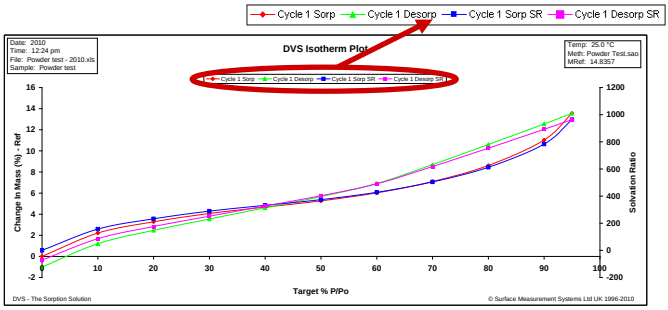
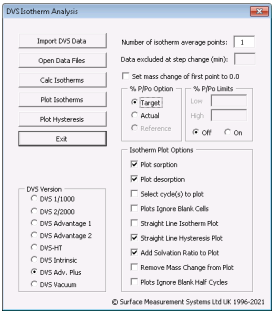


Figure 6.32: The same isotherm as above, but with the equivalent Solvation Ratio curves added.

Remove Mass Change from Plot

When the Add Solvation Ratio to Plot is ticked, it automatically activates the option directly below it, Remove Mass Change from Plot:



Using this option ensures that clicking the Plot Isotherm or Plot Hysteresis buttons will produce the Solvation Ratio curves only – the Mass Change curves will not be included:

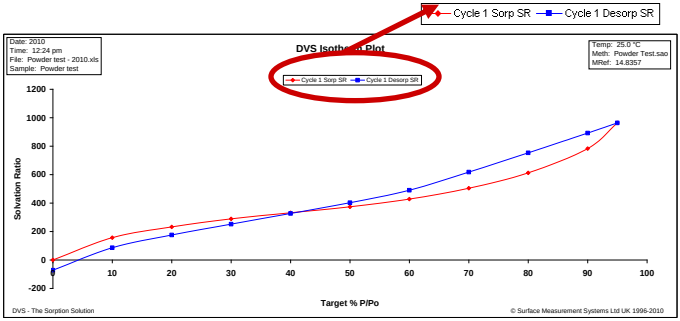
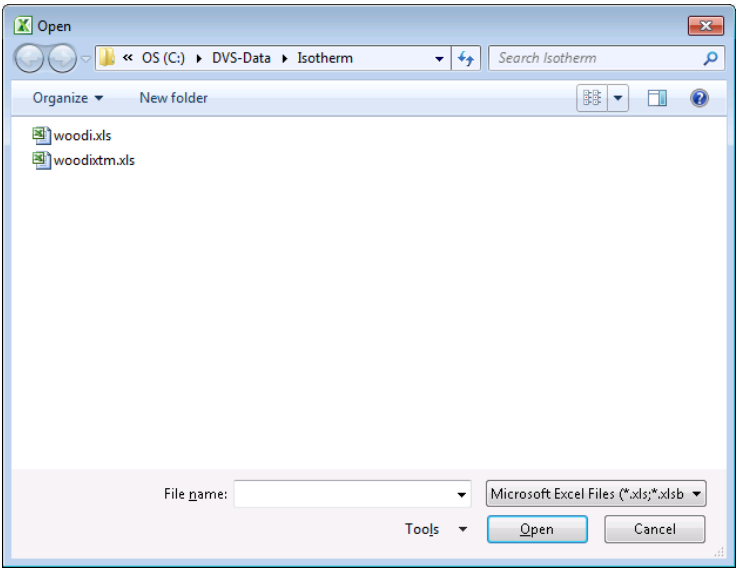


Figure 6.33: The Isotherm plot from Figure **6.31** and Figure 6.32, showing only the Solvation Ratio curves.

Note: Before Solvent Ratio curves can be added to isotherm plots, they must first be created using Plot Manager (Section 5).

6.4.3.2 Multiple Isotherm Plots

As with the plots produced by the **DVS Plot Manager** module, isotherms from more than one data file may be plotted on the same chart.



Before creating the plots, an isotherm report needs to have been created in each DVS data file to be included in the multiple plot (see Section 6.4.2). Note that any entered parameters or options are applied to ALL the files.

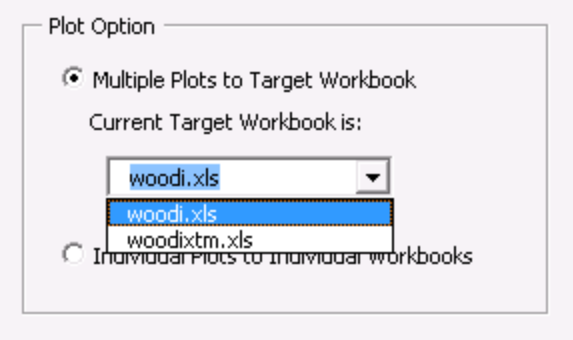
Also, the files to be included in the multiple plots need to all be open (see Section 4.3.3 for multiple file opening and Section 6.4.2.5 creating multiple isotherm reports).

Clicking the **Plot Isotherms** button will then display the following panel:

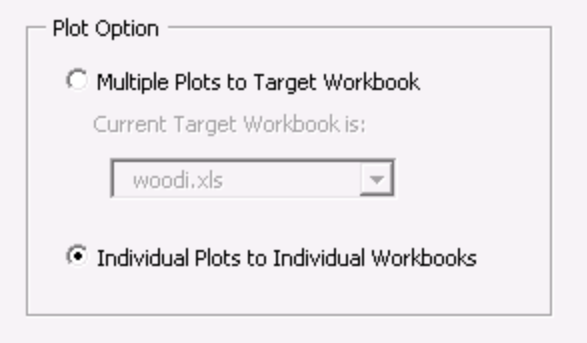
This **Select data file(s)** panel allows the user to select the files whose data will be included in the multiple isotherm plot. Only files currently open and with calculated isotherms will appear in the list.

At the bottom of the panel is the **Plot Option** box, which presents the user with two options;

Multiple Plots to Target Workbook, if selected, will ensure that the multiple isotherms will be displayed together on one plot in one of the open DVS files. The DVS file to display the multiple plot is selected in the drop-down menu in the Plot Option box;



Individual Plots to Individual Workbooks, the selection of which ensures that the isotherms will be plotted individually to their respective data files.



After choosing the desired options in the **Select data file(s)** panel and clicking **OK**, the multiple isotherms will appear – either in each of their respective files, or together on one plot in the DVS data file selected. An example of several isotherms plotted together is shown below in Figure 6.34:

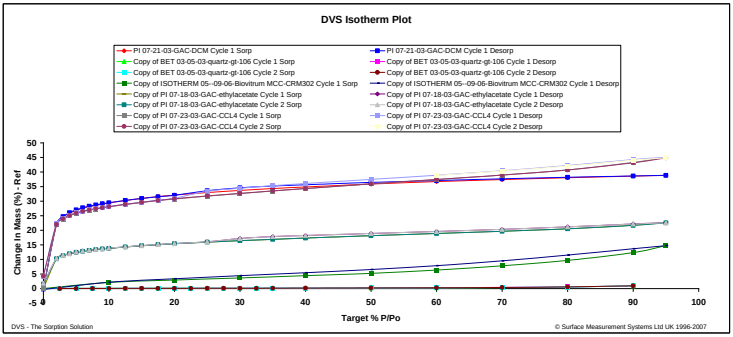
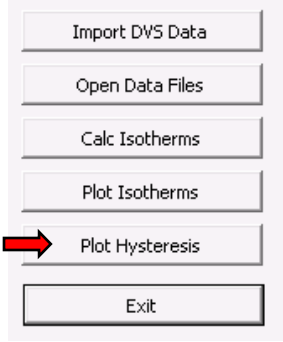


Figure 6.34: Multiple **DVS Isotherm Plots**.

6.4.4 Plot Hysteresis

Clicking the **Plot Hysteresis** button produces an isotherm hysteresis plot (ie; a plot of the difference between the sorption and desorption data) of a DVS data file. The plot is based on the data in the 'hysteresis' column in the Isotherm Analysis Report.



In order to produce such a plot, an Isotherm Analysis Report must first have been produced for the DVS data file (see Section 6.4.2).

In addition, the Plot Hysteresis option is only valid for those experiments with sorption and desorption stages – otherwise there would be no hysteresis to plot!

As the hysteresis plot is simply a plot of the difference between the 'dm(%) – ref' values for the sorption and desorption stages of the experiment, the **Plot sorption** and **Plot desorption** options in the **Isotherm Plot Options** box

are irrelevant and do not influence the hysteresis plot whether checked or unchecked.

The **Select cycle(s) to plot** option in the **Isotherm Plot Options box** may be used to produce hysteresis plots for multiple-cycle experiments in the same way as for isotherm plots, as described above on page 99.

An example of a hysteresis plot is shown below in Figure 6.35:

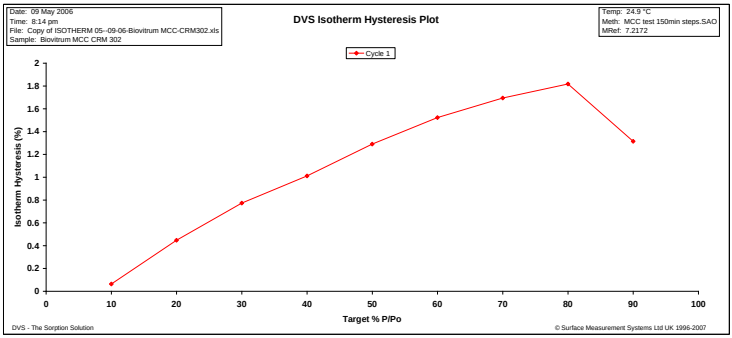
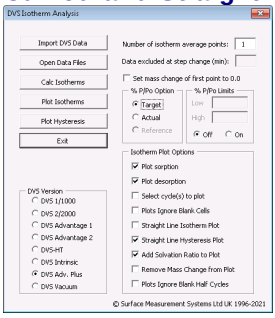


Figure 6.35: A **DVS Isotherm Hysteresis Plot**.

Curved and Straight Line Hysteresis Plots



The curved and straight line plot options in the **Isotherm Plot Options** area are explained in Section 6.4.3.1 above and apply to hysteresis plots also.

Figure 6.36 and Figure 6.37 below show examples of a straight-line and curved plot of the same hysteresis data:

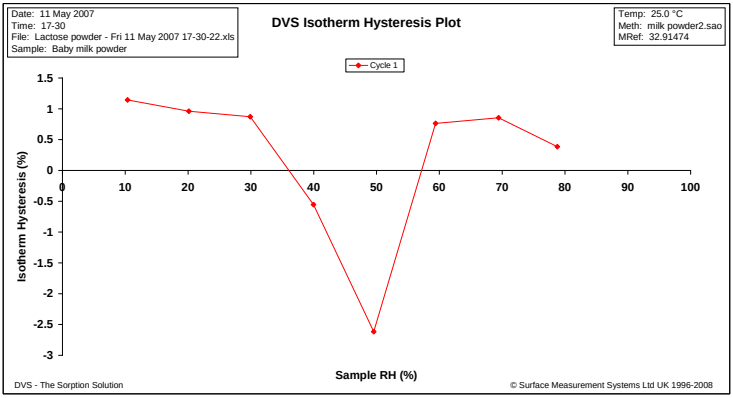
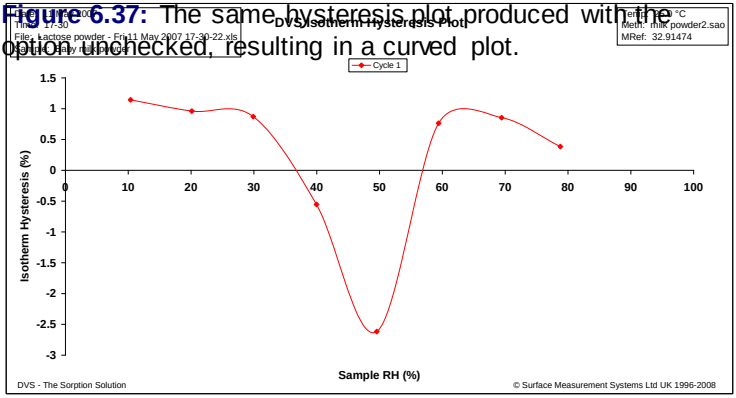


Figure 6.36: A hysteresis plot produced using the **Straight Line Hysteresis Plot** option.

Figure 6.37: The same hysteresis plot produced with the option unchecked, resulting in a curved plot.



Note: The **Straight Line Hysteresis Plot** option is by default checked when the Analysis Suite software is first opened – the user's choice to select/deselect this option is then subsequently preserved.

6.4.4.1 Multiple hysteresis plots

Multiple hysteresis curves may be plotted – either to individual data files or together on one chart – in the same way as described for isotherm plots. Please refer above to Section 6.4.3.2 for details.

7 DVS Baseline Correction

7.1 What does this module do?

The DVS Baseline Correction module allows the user to recalculate the 'dm (%) - ref' column in the DVS data file with a correction for baseline drift in a particular isotherm cycle.

7.2 Why is this useful?

Baseline drift can occur when, for example, a sample reaches its true dry mass at the end of an experiment despite the use of a drying period at the experiment's beginning. The true baseline is that recorded at the end of the experiment and not the false 'dry' baseline recorded at the beginning.

In such cases, the baseline correction can take this into account and calculate a more accurate baseline for the experiment data.

7.3 How to use this module

Clicking the **DVS Baseline Correction** button calls up the main dialog screen for the module, shown below in

On the top left hand side of the screen are the calculation and 'file opening'/'file importing' buttons. The DVS Version selection box (Section 4.3.2) is located in the bottom left-hand corner of the panel. On the rest of the screen are the selection areas for the calculation options.

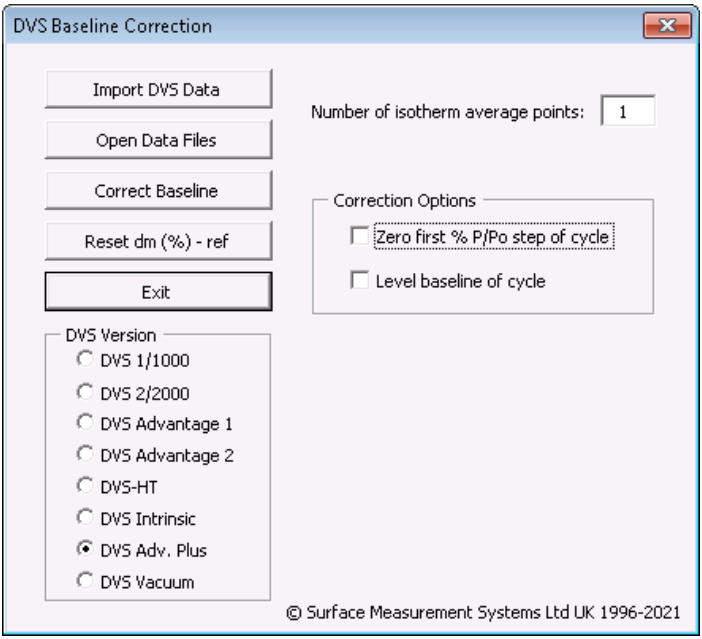
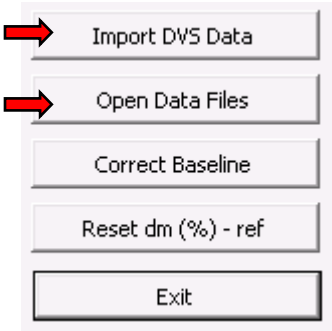


Figure 7.1: The DVS Baseline Correction main dialog screen.

7.3.1 Import DVS Data & Open Data File

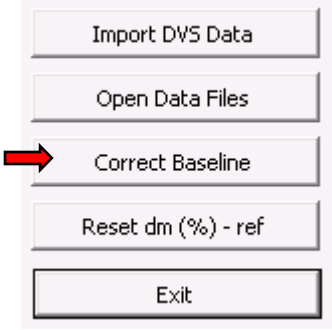
These functions are explained in detail in Section 4.2 and Section 4.3.



Before any baseline corrections may be carried out, a raw DVS data file must first be open in Excel using these buttons that appear on every module main panel - the file(s) may be imported and opened from any module before using the baseline correction functions.

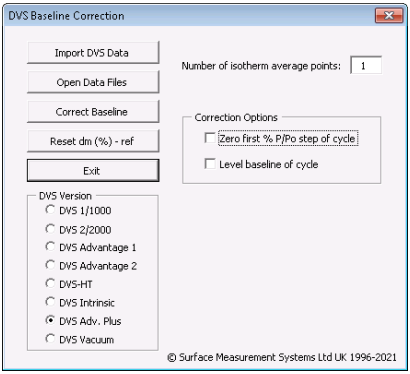
7.3.2 Correct Baseline

Clicking the **Correct Baseline** button performs a baseline correction on an isotherm cycle by recalculating the 'dm (%) - ref' column in the DVS data file.



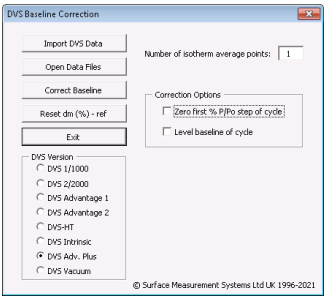
Before clicking the **Correct Baseline** button the desired **Number of isotherm average points** must be entered and the **Correction Options** must be specified:

7.3.2.1 Number of isotherm average points



This is explained in the DVS Isotherm Analysis Section 6.4.2.1.

7.3.2.2 Correction Options



This box features two options: Zero first %P/P_o step of cycle and Level baseline of cycle.

The **Zero first % P/P_o step of cycle** option recalculates the 'dm (%) - ref' column using the raw mass of the first isotherm point of the cycle as the dry mass. This makes the first isotherm point zero and all other points are referenced to it. If the first %P/P_o step in recording the isotherm was 0%, then using **Zero first % P/P_o step of cycle** is the equivalent of clicking **Auto Dry M(0)** in the experimental set-up.

The **Level baseline of cycle** option scales all the 'dm (%) - ref' column values to make the last isotherm point in the cycle equal to the first (see Figure 7.2 and Figure 7.3 below for an example):

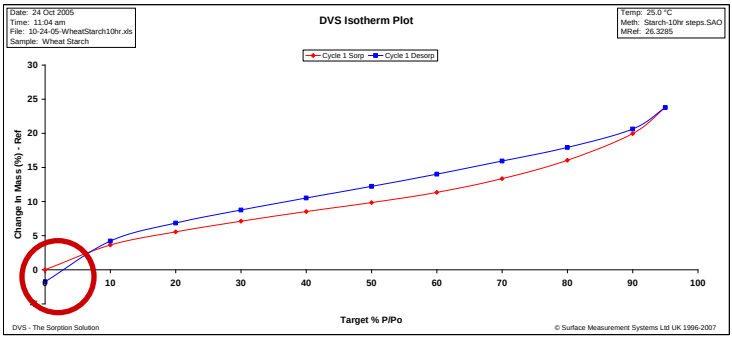


Figure 7.2: An isotherm before the Level baseline of cycle option has been used – the first and last points (circled) do not meet.

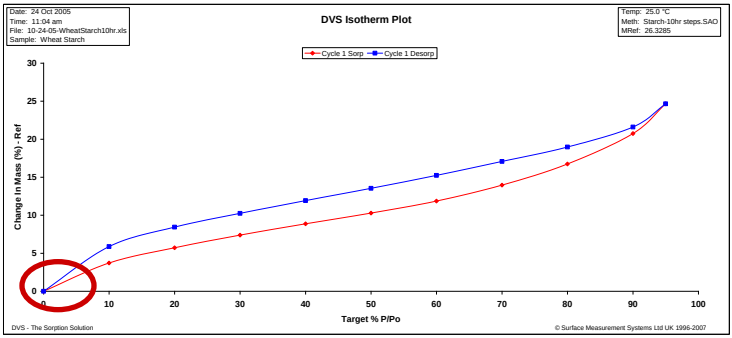


Figure 7.3: The same isotherm after using **Level baseline of cycle**, with the first and last points (circled) now equal.

Either or both of the corrections may be used. However, because the Zero first % P/P_o step of cycle correction recalculates the 'dm (%) - ref' column from the raw mass values, it will overwrite the results of any previous Level baseline of cycle corrections. The Level baseline of cycle option will need to be used again (after the 'Zero' operation) in order to make the 'ends' of the isotherm meet once more.

The **Level baseline of cycle** correction works from the existing 'dm (%) - ref' column – it therefore will not overwrite any previous modifications.

Along the same vein, any previous modifications made to the 'dm (%) - ref' column by the **DVS Vapour Content Offset** module will be overwritten by the **Zero first % P/P_o step of cycle** correction.

After setting the above parameters and clicking the **Correct Baseline** button, the module recalculates the 'dm (%) - ref' column from the raw mass values (see Figure 7.4):

Flow	dm (%) - ref	User
200	8.733881535	
199.71	8.379132879	
199.71	8.055908996	
199.9	7.762269862	
199.9	7.489222705	
199.8	7.237404334	
199.8	6.999639174	
199.71	6.77896576	

Flow	dm (%) - ref	User
200	8.646412269	
199.71	8.291818127	
199.71	7.968732759	
199.9	7.675888698	
199.9	7.402339497	
199.8	7.15066764	
199.8	6.913048996	
199.71	6.692522096	

Figure 7.4: A dm(%)-ref column before (left) and after (right) a baseline correction has been performed. Two equivalent pieces of data are circled to highlight the change that occurs in the values when using the **Correct Baseline** function.

7.3.2.3 Multiple isotherm cycle experiments

If the experiment was a multiple isotherm cycle experiment, then, upon clicking the **Correct Baseline** button, the user will be prompted to select the cycle for which the correction will be made.For example, for the experiment shown below in Figure 7.5 :

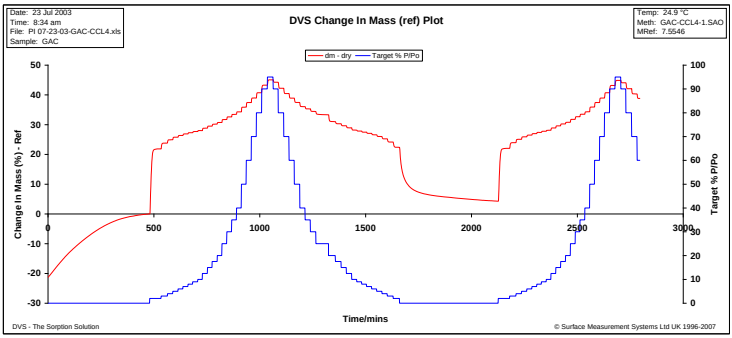
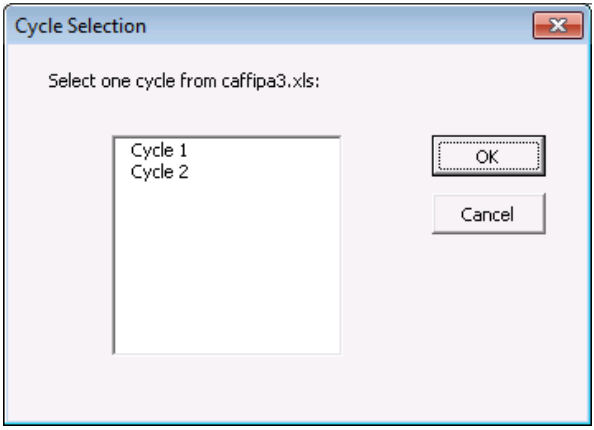


Figure 7.5: DVS multiple cycle data.

….clicking the **Correct Baseline** button will call up the following window:



Only one cycle may be chosen at a time for the baseline correction. After clicking on the desired cycle and clicking **OK**, the baseline correction will proceed.

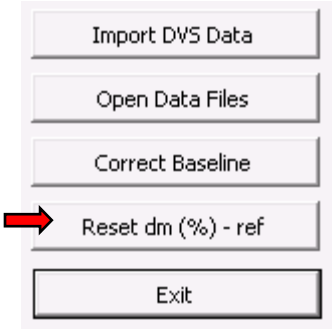
7.3.2.4 Multiple Baseline Corrections

If desired, the user can perform baseline corrections for more than one data file at a time. The procedure is the same as for producing multiple isotherm reports – please refer to Section 6.4.2.5).

Please note that any entered parameters or options are applied to ALL the files included in the multiple baseline correction.

7.3.3 Reset dm(%) - ref

Clicking the **Reset dm (%) - ref** button returns the 'dm (%) - ref' column in the DVS data file to its original, pre-Correct Baseline values.



It does not matter how many different Baseline Correction operations may have been performed upon the data – the Reset 'dm (%) – ref' function recalculates the column of data from the original mass values - in the same way as when a file is first opened. Thus the original 'dm(%)-ref' values may be retrieved at any time.

7.3.3.1 Reset dm(%)-ref for multiple DVS files

If desired, the user can reset the 'dm(%)-ref' column for more than one data file at a time.

The procedure is the same as for producing multiple isotherm reports – please refer to Section 6.4.2.5.

8 DVS Vapour Content Offset

8.1 What does this module do?

This module allows the user to incorporate into calculations any vapour (or 'non-dry' mass) that was present in the sample at the beginning of the experiment.

8.2 Why is this useful?

The 'dm(%) – ref' data which is calculated in each DVS file forms the backbone of any analysis that is carried out on the data. From simply plotting an isotherm to calculating complex parameters, the 'dm(%)-ref' data is crucial.

It is therefore equally crucial that this data is accurately calculated. If the sample is known to contain vapour which is not from the DVS system at any stage of an experiment, then the 'dm(%)-ref' data will be incorrect – it will be based upon a 'reference' sample mass that comprises dry sample mass *and* extra vapour molecules which were not present as part of the DVS experiment.

The **DVS Vapour Content Offset** module allows the Analysis Suite to take this extra 'vapour' into account by effectively subtracting it from the 'reference mass' before re-calculating the 'dm(%)-ref' column. The vapour which was subtracted from the 'reference mass' is incorporated into the 'dm(%)-ref' calculation as vapour.

It should be noted that in order for this module to be used, the user must know the initial vapour content and be able to quantify it as a percentage of the total sample mass (dry mass + vapour content).

8.3 How to use this module

Clicking the DVS Vapour Content Offset button on the Analysis Suite toolbar calls up the main dialog screen for the module, shown in Figure 8.1 below:

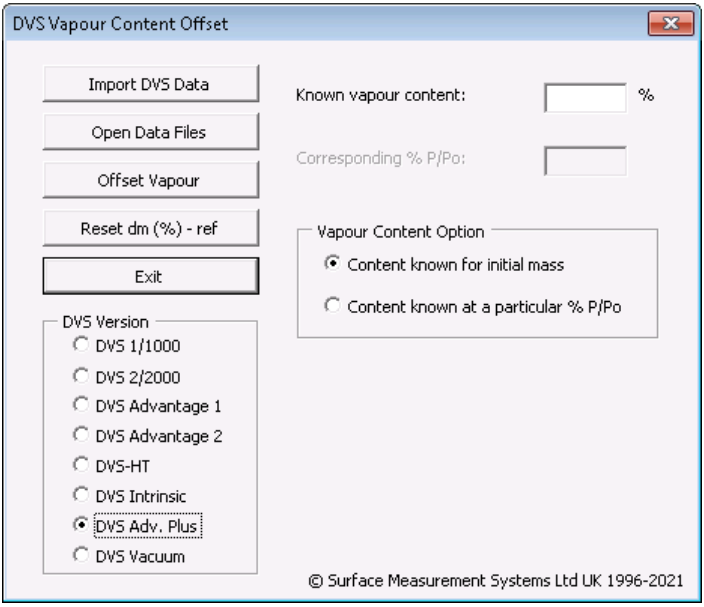


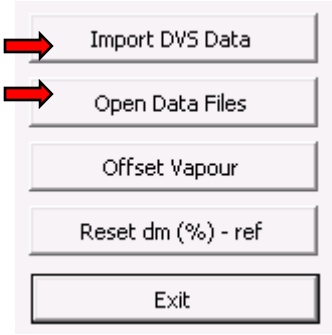
Figure 8.1: The **DVS Vapour Content Offset** main dialog screen.

On the top left hand side of the screen are the calculation and 'file opening'/'file importing' buttons and the DVS Version selection area. On the rest of the screen are the selection areas for the calculation options.

Note: The module will be labelled **Vapour** Content Offset or **Moisture** Content Offset depending upon the software configuration being used at the time (see Section 14).

8.3.1 Import DVS Data and Open Data File

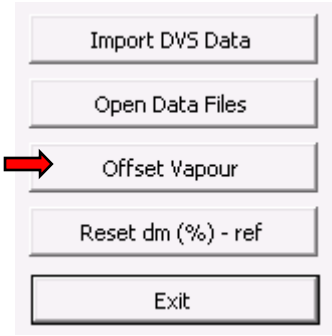
These functions are explained in detail in Section 4.2 and Section 4.3.



Before any vapour offset calculation may be carried out, a raw DVS data file must first be open in Excel using these buttons, which appear on every module main panel - the file(s) may be imported and opened from any module before using the vapour offset functions.

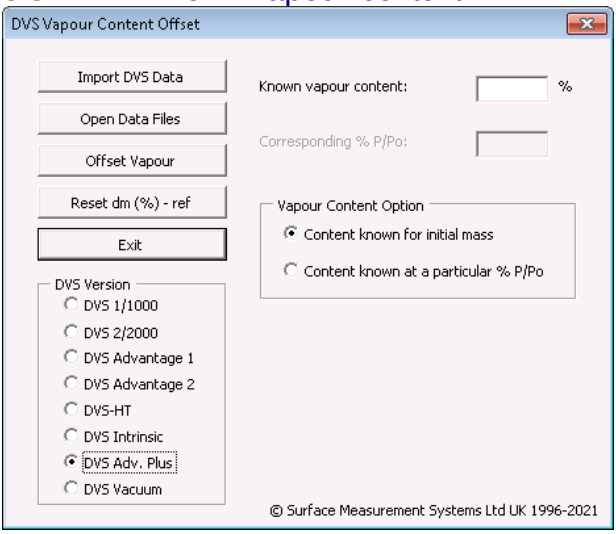
8.3.2 Offset Vapour

Clicking the **Offset Vapour** button performs a vapour content offset by recalculating the 'dm (%) - ref' column in the DVS data file.



Before clicking the **Offset Vapour** button, the **Known vapour content** must be specified and the appropriate **Vapour Content Option** selected:

8.3.2.1 Known vapour content



The **Known vapour content** value is the vapour content of the sample as a percentage of the total mass i.e;

$$\frac{mass(vapour)}{mass(dry+vapour)}$$

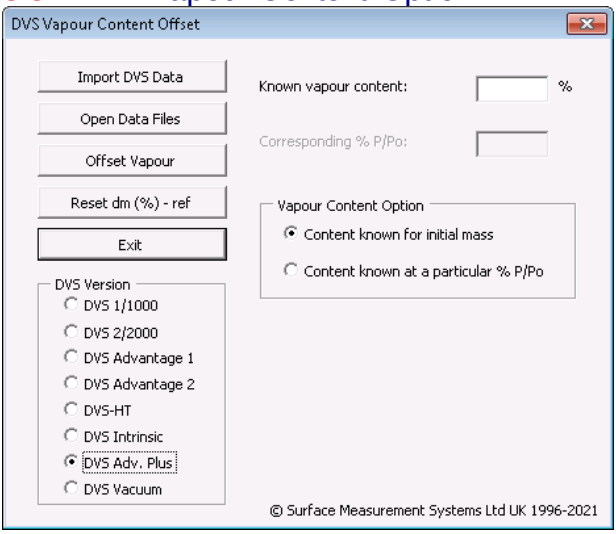
…whereas 'dm (%) – ref' values are calculated as a percentage of the dry mass i.e;

$$\frac{mass(vapour)}{mass(dry)}$$

If for example a known vapour content of 5% were entered, the 'dm (%) – ref' value (and hence the isotherm value) at that point would be calculated as 5.2632% (= 5/95).

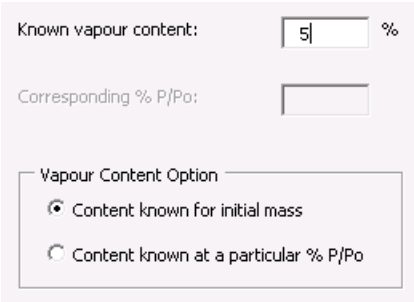
If the user wanted to force the 'dm (%) – ref' value to be 5% then it would be necessary to enter a known vapour content of 4.7619% (= 5/105).

8.3.2.2 Vapour Content Option



The **Vapour Content Option** allows the user specify whether this known vapour content applies to the sample's initial mass, or to the sample's mass at a particular $\%p/p_o$ during the experiment.

If the **Content known at initial mass** option is chosen, all the user has to specify is the **% Known vapour content** discussed in Section 8.3.2.1:



If, however, the **Content known at a particular $\%P/P_o$** option is chosen (Figure 8.2), then an extra box is enabled which

allows the user to specify the **Corresponding %P/P$_o$** to which this known vapour content applies.

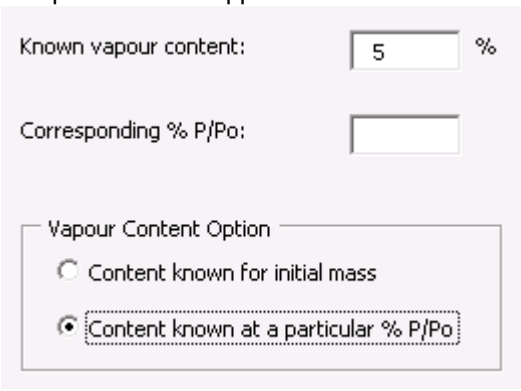


Figure 8.2: Selection of Content known at a particular %P/P$_o$ enables the Corresponding %P/P$_o$ box, which must be specified.

After setting the above parameters and clicking the **Offset Vapour** button, the module recalculates the 'dm (%) - ref' column from the raw mass values (Figure 8.3 below):

Flow	dm (%) - ref	User
200	8.733881535	
199.71	8.379132879	
199.71	8.055908996	
199.9	7.76269062	
199.8	7.489222705	
199.8	7.237404334	
199.8	6.999639174	

Flow	dm (%) - ref	User
200	5.263157895	
199.71	4.919732614	
199.71	4.606825853	
199.9	4.322966841	
199.8	4.058227867	
199.8	3.814447394	
199.8	3.584271563	

Figure 8.3: A **dm(%)-ref** column before (left) and after (right) vapour content offset has been performed (%5 vapour content in this example). Two equivalent pieces of data are circled to highlight the significant change that occurs in the values when using the Vapour Content Offset function.

8.3.2.3 Vapour content offset and baseline correction

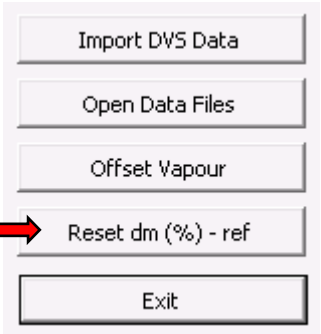
It should be noted that any previous modifications made to the 'dm (%) - ref' column by the baseline correction module (DVS Baseline Correction) will be overwritten.

8.3.2.4 Vapour content offsets for multiple DVS files

If desired, the user can perform vapour content offsets for more than one data file at a time. The procedure is the same as for producing multiple isotherm reports – please refer to Section 6.4.2.5.

Please note that any entered parameters or options are applied to ALL the files included in the multiple vapour content offset.

8.3.3 Reset dm (%) - ref



Clicking the **Reset dm (%) - ref** button returns the 'dm (%) - ref' column in the DVS data file to its original, pre-Vapour offset values.

It does not matter how many different vapour offset operations may have been performed upon the data – the Reset 'dm (%) – ref' function recalculates the column of data from the original mass values - in the same way as when a file is first opened. Thus the original 'dm(%)-ref' values may be retrieved at any time.

If desired, the user can reset the ‘dm(%)-ref’ column for more than one data file at a time. The procedure is the same as for producing multiple isotherm reports – please refer to Section 6.4.2.5.

9 DVS Salt Validation

9.1 What does this module do?

The **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 macro verifies that the partial pressure levels in the DVS are what the instrument says they are.

9.2 Why is this useful?

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.

9.3 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.

9.3.1 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.

9.3.2 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
$MgCl_2$	VI. 32.8	VII. 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 (L. Greenspan, Journal of Research of the National Bureau of Standards-A, vol. 81A (1), 1977, 89-96.)

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.

9.3.3 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.

9.3.4 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 9.1:

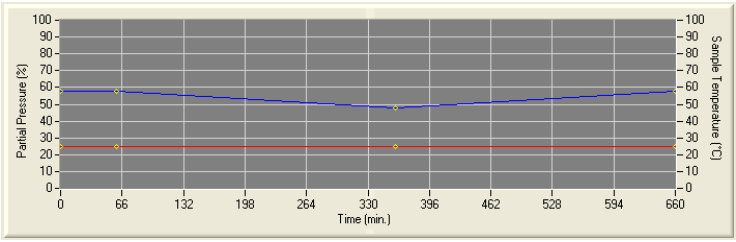


Figure 9.1: The proposed partial pressure profile for a salt validation using $MgNO_3$ at 25°C. The constant red line shows the temperature, while the blue line shows the partial pressure

Once this method has been run, the data may then be analysed using the **Salt Validation** module.

9.3.5 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 9.2 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.

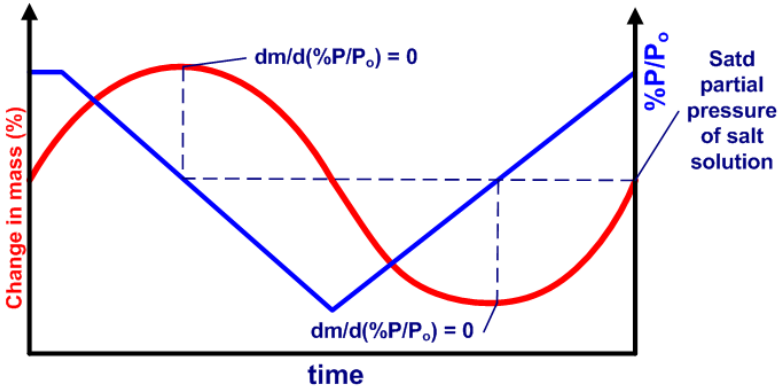


Figure 9.2: $dm/d(\%P/P_o)$ is zero at the saturated P/P_o of salt solution

9.4 How to use this module

Clicking the Salt Validation Calibration button will call up the main dialog screen for the module (shown below).

On the left hand side of the dialog screen are the calculation, plotting and 'file opening'/'file importing' buttons, and the DVS Version selection area. On the right hand side are the selection areas for the calculation and plot options.

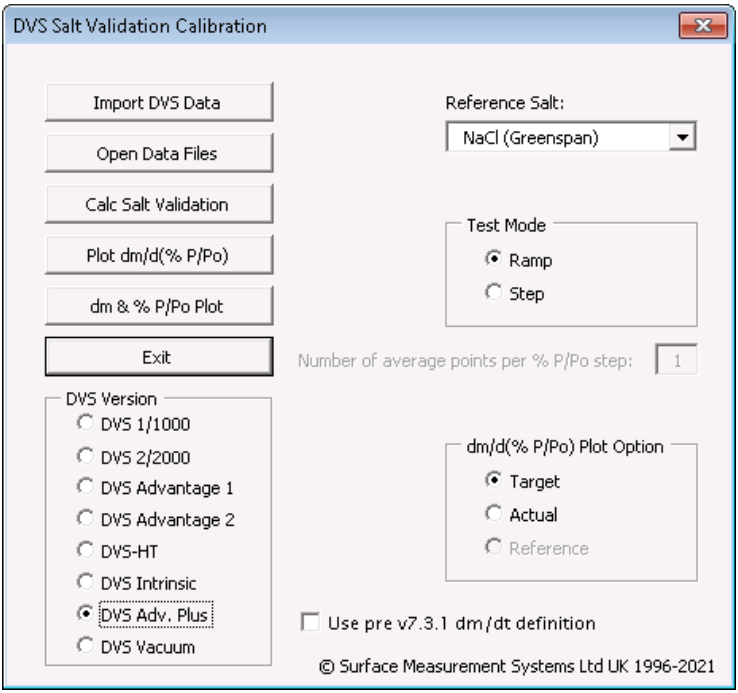
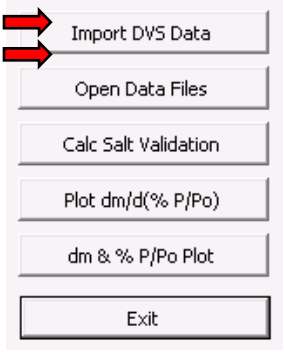


Figure 9.3: The DVS Salt Validation Calibration main dialog screen

9.4.1 Import DVS Data and Open Data File

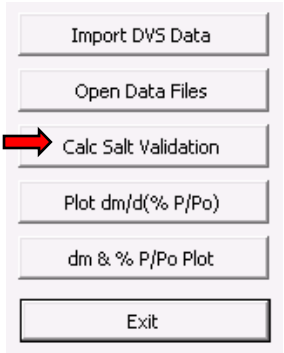
These functions are explained in detail in Section 4.2 and Section 4.3.



Before any salt validation calculations or plots may be carried out, a raw DVS data file must first be open in Excel using these buttons, which appear on every module main panel - the file(s) may be imported and opened from any module before using the salt validation functions.

9.4.2 Calc Salt Validation

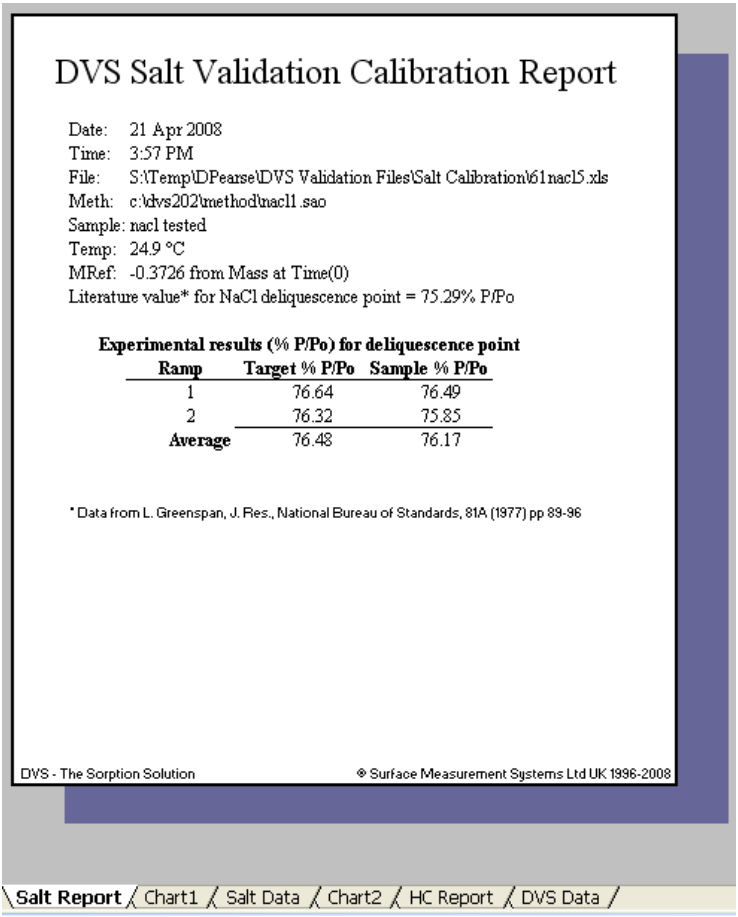
Clicking the **Calc Salt Validation** button produces a salt validation calibration report from an appropriate DVS data file (Figure 9.4 below). The report appears in the DVS Excel data file as a new sheet entitled **Salt Report**. Also appearing in the excel file is a new sheet entitled **Salt Data**, which contains the data used to calculate the Salt Report.



The report gives the experimental $\%P/P_o$ deliquescence point for each ramp (half cycle) of the experiment and breaks the results down by partial pressure category.

The calculation can handle either ramp or step mode experiments and also multiple cycle experiments.

Note: The option “Use pre v7.3.1 dm/dt definition” switches the definition of dm/dt to dm/dt * 100/M0 where M0 is the initial Sample Weight. This has no effect on the Salt Report but changes the dm/dt values in the Salt Data sheet.



DVS Salt Validation Calibration Report

Date: 21 Apr 2008
Time: 3:57 PM
File: S:\Temp\DPearse\DVS Validation Files\Salt Calibration\61nacl5.xls
Meth: c:\dvs202\method\nacl1.sao
Sample: nacl tested
Temp: 24.9 °C
MRef: -0.3726 from Mass at Time(0)
Literature value* for NaCl deliquescence point = 75.29% P/Po

Experimental results (% P/Po) for deliquescence point

Ramp	Target % P/Po	Sample % P/Po
1	76.64	76.49
2	76.32	75.85
Average	76.48	76.17

* Data from L. Greenspan, J. Res., National Bureau of Standards, 81A (1977) pp 89-96

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Salt Report | Chart1 | Salt Data | Chart2 | HC Report | DVS Data

Figure 9.4: A DVS Salt Validation Calibration Report

The report also gives an average for each $\%P/P_o$ category and a literature value for the salt type chosen (using data from L. Greenspan, J. Res., National Bureau of Standards, 81A (1977) pp 89-96 & H. Nyqvist, Int. J. Pharm. Tech. & Prod. Mfr., 4 (2), (1983) pp 47-48).

Prior to version v3.7, the DVS Analysis Suite used Nyqvist for Mg(NO3)2 and Greenspan for the remaining salt types. A choice is now given for the Mg(NO3)2 data ref.

Note: DVS Intrinsic Salt Report format: Note that a Salt Validation Calibration Report produced from a DVS Intrinsic data file will be of the format shown below, which is different from the Report shown in Figure 9.4:

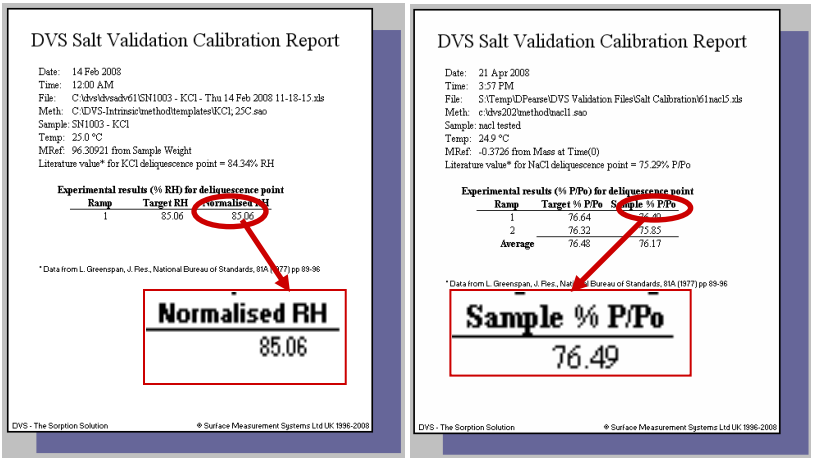


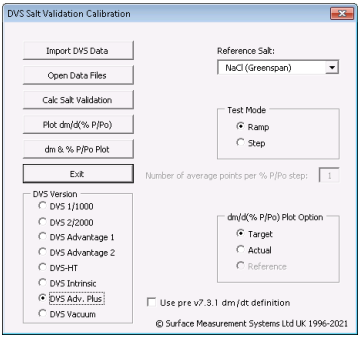
Figure 9.5: A DVS Intrinsic Salt Validation Calibration Report (left) and the Salt Reports produced for other DVS models (right).

The Intrinsic Salt Report features a data set labelled **Normalised RH** (all other DVS Salt Reports feature **Sample** data in this column).

The use of **Normalised RH** relates to how salt validations are performed on the DVS Intrinsic and is explained further in the **DVS Intrinsic Operation Manual**.

Before clicking the **Calc Salt Validation** button the **Reference Salt** and **Test Mode** must be selected on the main DVS Salt Validation Calibration panel:

9.4.2.1 Reference Salt



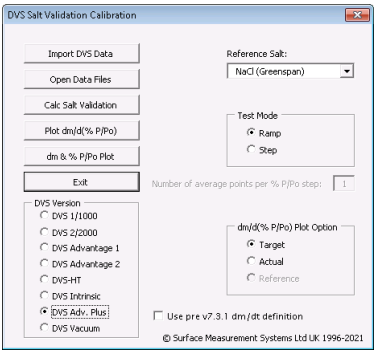
The Reference Salt selection consists of a drop-down list of salts for which the literature value of the % P/P_o deliquescence point can be calculated over a range of temperatures.

The source of each salt's literature value partial pressure is included in parentheses.

The salt validation report will show the literature value of the $\%P/P_o$ deliquescence point for the chosen salt at the average temperature of the experiment. To obtain a meaningful literature value in the report the salt selected in the drop-down list must be the same as the one used in the experiment.

9.4.2.2 Test Mode

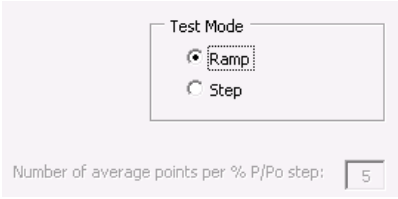
This selection allows the user to define the mode used to run the salt validation experiment - **Ramp** or **Step** mode.



The same mode as was used in the salt validation experiment must be chosen. If ramp mode is chosen and the experiment was a step experiment, the calculation will effectively calculate in step mode with the number of average points per step set to 1. If step mode is chosen and the experiment was a ramp experiment, a critical error will be displayed before calculation is complete.

If **Step** mode is chosen, another option is activated – **Number of average points per % P/P_o step.** This allows the user to specify the number of points at the end of each % P/P_o step over which the change in mass and dm/dt are averaged for each step in a step mode experiment; e.g. if "5" is entered in the edit box, each point will be an average of the last five points of the %P/P_o step.

This value is not required if ramp mode is chosen – in which case the **Number of average points per step** edit box will be disabled:



9.4.2.3 Multiple Salt Validation Calibration Reports

If desired, the user can calculate salt validation calibration reports for more than one data file at a time. The procedure is the same as for producing multiple isotherm reports – please refer to Section 6.4.2.5.

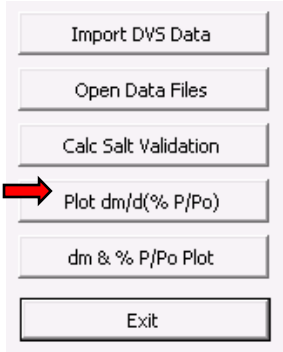
Please note that any entered salt calibration report parameters or options are applied to ALL the files included in the multiple report compilation.

Please also note that in the case of a DVS Advantage and DVS-HT instruments a calculation is only possible if the water is adsorbate is contained in **Reservoir A**.

9.4.3 Plot dm/d(% P/P_o)

Clicking the **Plot dm/d(% P/P_o)** button produces a salt validation calibration plot from a DVS data file for which a salt validation calibration report has been calculated.

This plot charts the change in mass (%) and dm/d(% P/P_o) against partial pressure (%) (Figure 9.6)



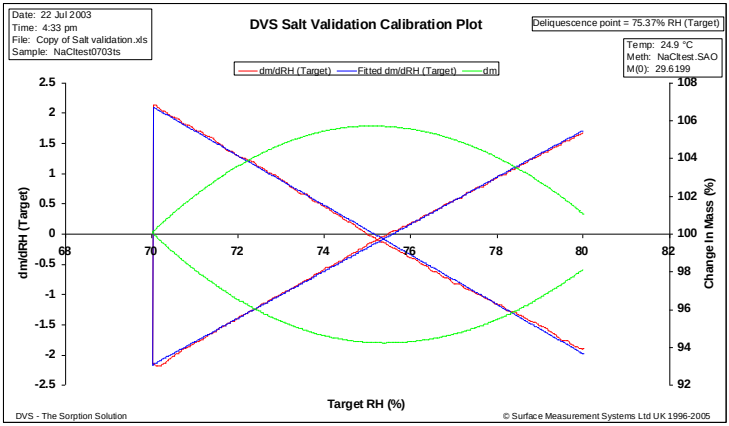
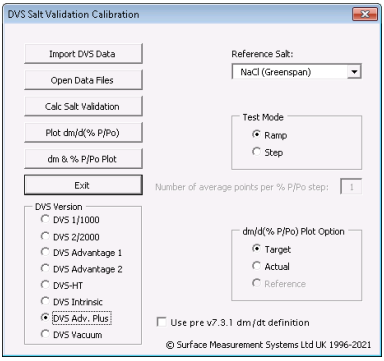


Figure 9.6: A DVS Salt Validation Calibration Plot

The curve plots makes it easy to discern the $\%P/P_o$ at which the salt solution's mass stops changing during the experiment. The $dm/d(\%P/P_o)$ data is also fitted with a line of best fit to aid clarity.

9.4.3.1 dm/d(% P/P_o) Plot Option

Before clicking the **Plot dm/d(% P/P_o)** button the % P/P_o type (target, sample or reference) to be used in the salt validation calibration plot must be set in the **dm/d(% P/P_o) Plot Option** selection area:



9.4.3.2 Multiple Plots

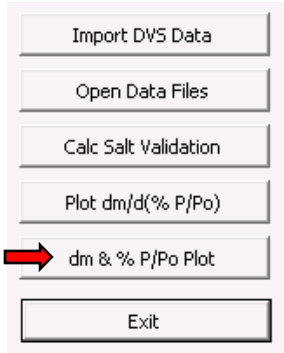
If desired, the user may plot salt validation data for more than one data file at a time.

This is achieved in the same fashion as for multiple isotherm plots. Please refer to Section 6.4.3.2 for details.

Note that any entered plot parameters or options are applied to ALL the files to be plotted.

9.4.4 dm & % P/P_o Plot

Clicking the **dm & % P/P_o Plot** button produces a standard change in mass and partial pressure plot.



This will chart change in mass (%) and target partial pressure (%) against time (minutes) (see Figure 9.7 below).

Please note that only target partial pressure is plotted, regardless of the options selected in the dm/d(%P/P_o) Plot Option box (see Section 9.4.3.1 above).

If the balance was auto tared during the experiment, the plot will show only the post tare data.

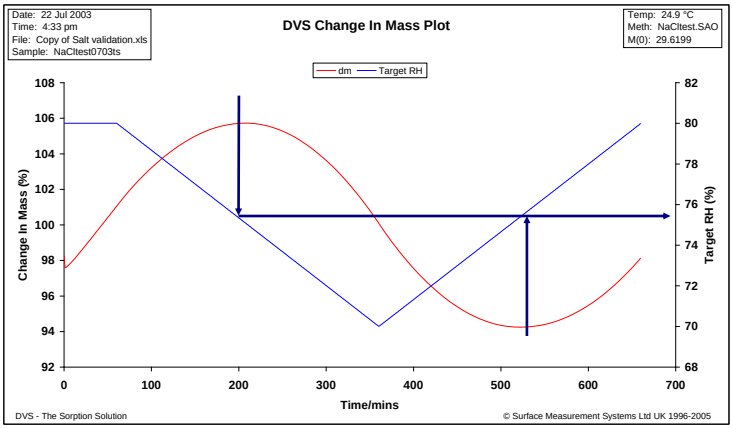


Figure 9.7: A DVS Change In Mass Plot.

The curve makes it easy to discern the points at which the saturated salt solution's mass stopped changing. The relative humidity at which this deliquescence occurred can then be read off the Target P/P_o (%) axis.

9.4.4.1 Multiple dm & $\%P/P_o$ Plot Plots

If desired, the user may plot salt validation data for more than one data file at a time.

This is achieved in the same fashion as for multiple isotherm plots. Please refer to Section 6.4.3.2 for details.

Note that any entered plot parameters or options are applied to ALL the files to be plotted.

10 DVS Partial Pressure Check

10.1 What does this module do?

The **DVS Partial Pressure Check** module is used to compare the actual partial pressure generated by the system against the target $\%P/P_o$ that has been set by the user.

The module also calculates the system partial pressure's variance with time.

10.2 Why is this useful?

It is important to verify that the DVS is reaching the target $\%P/P_o$ levels set in experiments in order for the system to generate meaningful data.

10.3 How to use this module

Clicking the **DVS Partial Pressure Check** button calls up the main dialog screen for the module as shown below.

On the top left hand-side of the dialog screen are the calculation, plotting and 'file opening'/'file importing' buttons. The bottom left-hand corner features the DVS Version selection box. On the right hand side are the selection areas for the calculation options.

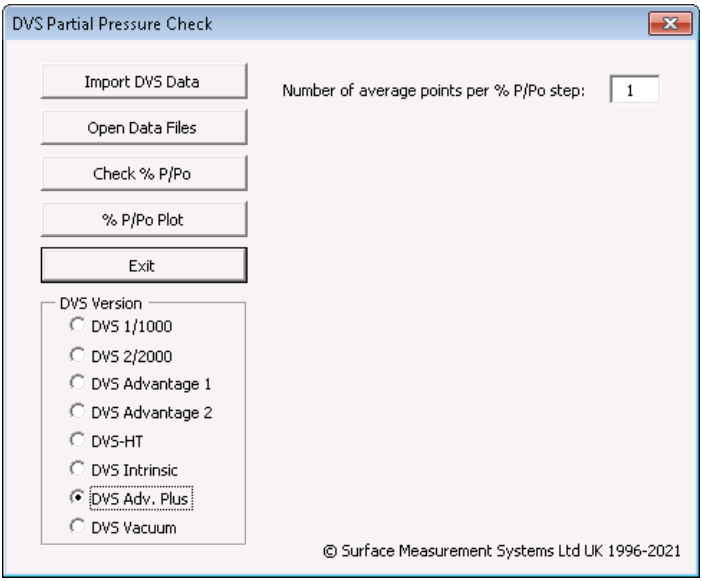
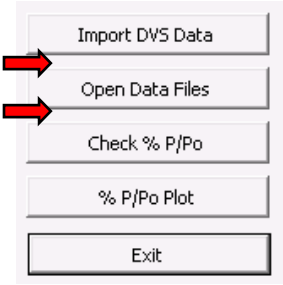


Figure 10.0.1: The **DVS Partial Pressure Check** main dialog screen.

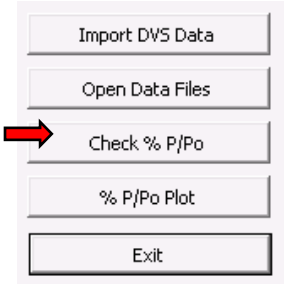
10.3.1 Import DVS Data & Open Data File



These functions are explained in detail in Section 4.2 and Section 4.3, respectively.

Before any partial pressure checks or plots may be carried out, a raw DVS data file must first be open in Excel using these buttons, which appear on every module main panel - the file(s) may be imported and opened from any module before using the partial pressure check functions.

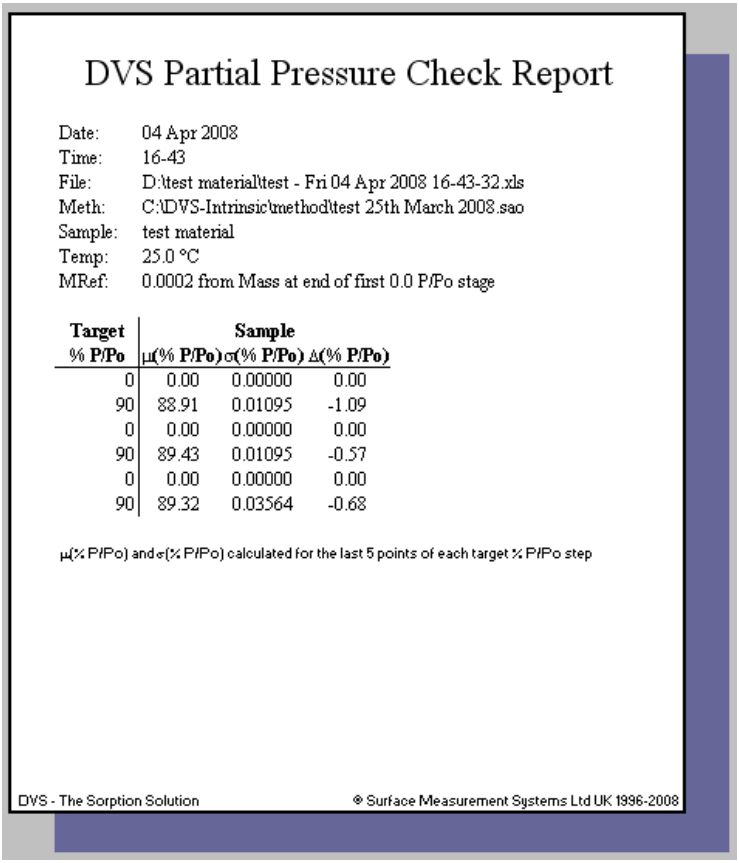
10.3.2 Check % P/P_o



Clicking the **Check % P/P_o** button produces a DVS Partial Pressure Check report from an appropriate DVS data file (see below).

The report appears on a new sheet entitled **HC Report**.

A partial pressure check report may only be calculated for one DVS data file at a time.



DVS Partial Pressure Check Report

Date: 04 Apr 2008
Time: 16-43
File: D:\test material\test - Fri 04 Apr 2008 16-43-32.xls
Meth: C:\DVS-Intrinsic\method\test 25th March 2008.sao
Sample: test material
Temp: 25.0 °C
MRef: 0.0002 from Mass at end of first 0.0 P/Po stage

Target	Sample		
% P/Po	μ(% P/Po)	σ(% P/Po)	Δ(% P/Po)
0	0.00	0.00000	0.00
90	88.91	0.01095	-1.09
0	0.00	0.00000	0.00
90	89.43	0.01095	-0.57
0	0.00	0.00000	0.00
90	89.32	0.03564	-0.68

μ(% P/Po) and σ(% P/Po) calculated for the last 5 points of each target % P/Po step

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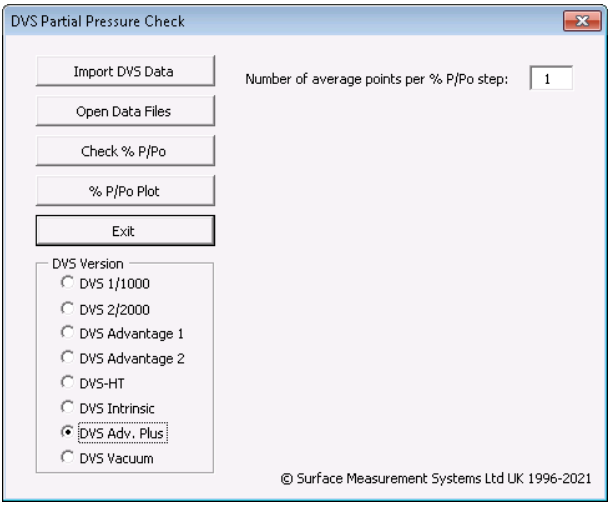
Figure 10.0.2: A **DVS Partial Pressure Check Report**

For each target % P/P_o step, the report gives the average (**μ(% P/P_o)**) and standard deviation (**σ(% P/P_o)**) of the system % P/Po values (over the specified number of points at the end target % P/Po (**Δ(% P/P_o)**).

10.3.2.1 Number of average points per %P/P_o step



Before clicking the **Check % P/P_o** button the user must first enter the **Number of average points per % P/P_o step**. This is the number of points at the end of each step for which the average and standard deviation of the % P/P_o is calculated.

The number of data points commonly used is three to five.

10.3.3 % P/P_o Plot

Clicking the **% P/P_o Plot** button produces a standard partial pressure plot.

This will chart the partial pressures (%) generated by the system, along with the target partial pressure (%)

against time (minutes) (see Figure 10.0.3 below).

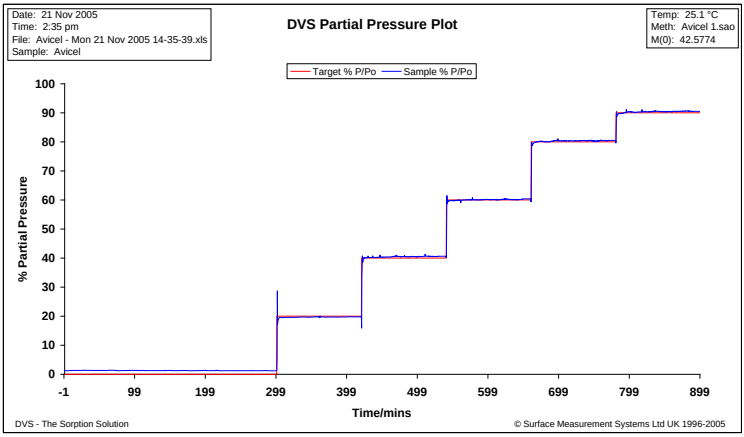


Figure 10.0.3: A DVS Partial Pressure Plot showing the partial pressures generated by the system alongside the target partial pressure plotted against time

10.3.3.1 Multiple %P/P_o plots

If desired, the user can create %P/P_o plots for more than one data file at a time. The procedure is the same as for producing multiple isotherm plots – but in this case, a %P/P_o check does not need to have been performed for any of the files. Please refer to Section 6.4.3.2.

11 DVS Drift and Noise Check

11.1 What does this module do?

The **DVS Drift and Noise Check** module is used to analyse the drift and noise in the mass data from an experiment with no sample hanging from the balance.

11.2 Why is this useful?

This module is important as it allows the user to run a quick diagnostic test of the instrument's baseline stability.
Stability of the instrument's mass baseline is obviously crucial to its ability to produce meaningful data.

11.3 What kind of experimental set-up is required for a drift and noise check?

In order to perform a drift and noise check, instrument data must be recorded with no sample present in the system.

Clean sample pans must be placed in the instrument and mass baseline data recorded at 0% P/P_o and one temperature.

It is recommended that the experiment be conducted overnight.

11.4 How to use this module

Clicking the DVS Drift Noise Check button calls up the main dialog screen for the module as shown below.

On the left hand side of the dialog screen are the calculation, plotting and 'file opening'/'file importing' buttons. In the bottom left-hand corner is the DVS Version selection box. On the right hand side are the selection areas for the analysis options.

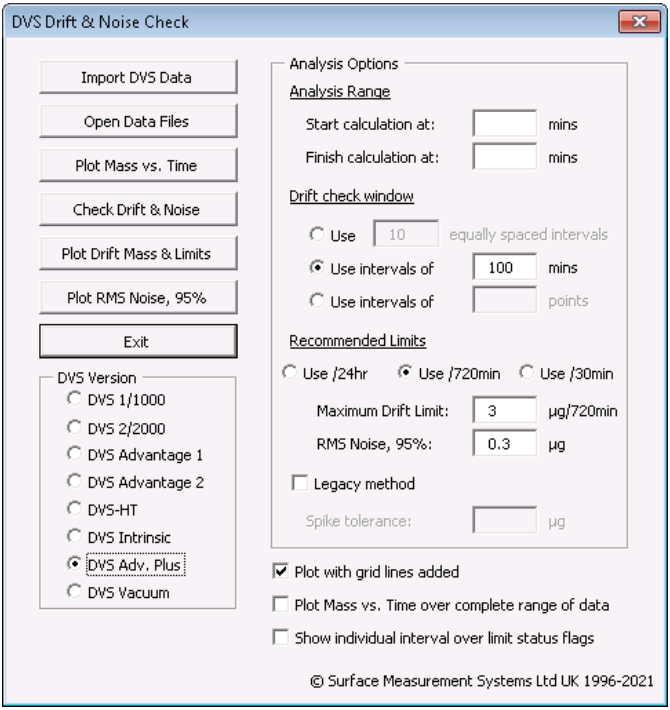
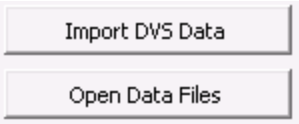


Figure 11.1: The DVS Drift and Noise Check main dialog screen.

11.4.1 Import DVS Data & Open Data File

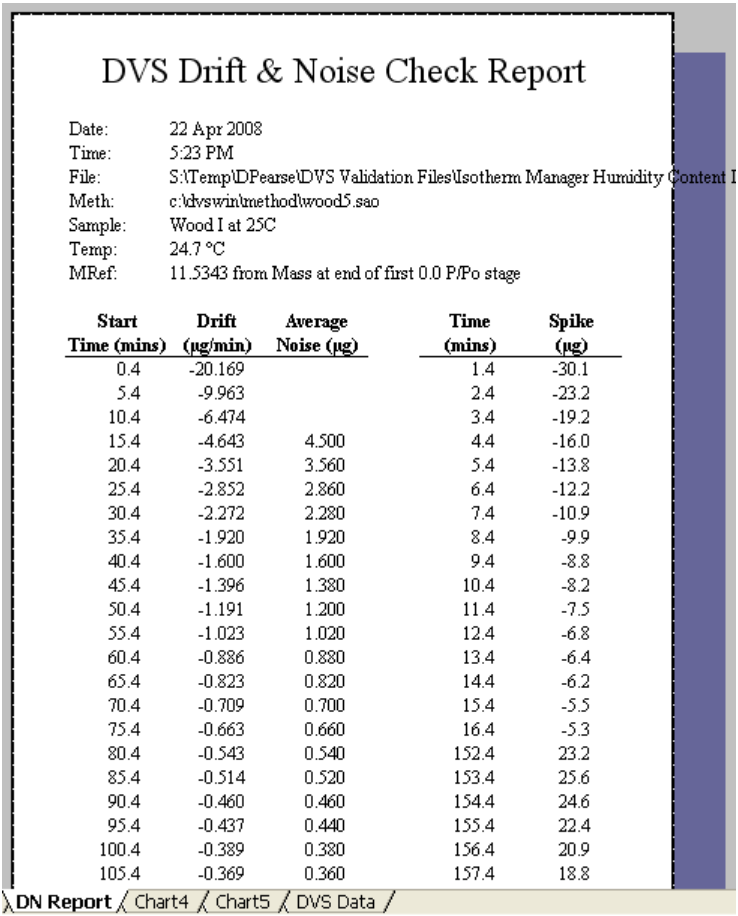


These functions are explained in detail in Section 4.2 and Section 4.3, respectively.

Before any partial drift and noise checks or plots may be carried out, a raw DVS data file must first be open in Excel using these buttons, which appear on every module main panel - the file(s) may be imported and opened from any module before using the drift and noise check functions.

11.4.2 Check Drift & Noise

Clicking the Check Drift & Noise button produces a DVS Drift and Noise Check report from an appropriate DVS data file (see Figure 11.2 below).



DVS Drift & Noise Check Report

Date: 22 Apr 2008
Time: 5:23 PM
File: S:\Temp\DPearse\DVS Validation Files\Isotherm Manager Humidity Content I
Meth: c:\dvswin\method\wood5.sao
Sample: Wood I at 25C
Temp: 24.7 °C
MRef: 11.5343 from Mass at end of first 0.0 P/Po stage

Start Time (mins)	Drift (µg/min)	Average Noise (µg)	Time (mins)	Spike (µg)
0.4	-20.169		1.4	-30.1
5.4	-9.963		2.4	-23.2
10.4	-6.474		3.4	-19.2
15.4	-4.643	4.500	4.4	-16.0
20.4	-3.551	3.560	5.4	-13.8
25.4	-2.852	2.860	6.4	-12.2
30.4	-2.272	2.280	7.4	-10.9
35.4	-1.920	1.920	8.4	-9.9
40.4	-1.600	1.600	9.4	-8.8
45.4	-1.396	1.380	10.4	-8.2
50.4	-1.191	1.200	11.4	-7.5
55.4	-1.023	1.020	12.4	-6.8
60.4	-0.886	0.880	13.4	-6.4
65.4	-0.823	0.820	14.4	-6.2
70.4	-0.709	0.700	15.4	-5.5
75.4	-0.663	0.660	16.4	-5.3
80.4	-0.543	0.540	152.4	23.2
85.4	-0.514	0.520	153.4	25.6
90.4	-0.460	0.460	154.4	24.6
95.4	-0.437	0.440	155.4	22.4
100.4	-0.389	0.380	156.4	20.9
105.4	-0.369	0.360	157.4	18.8

DN Report / Chart4 / Chart5 / DVS Data /

Figure 11.2: A **DVS Drift and Noise Check Report**

For each drift check time window in the mass data (see below), the report gives the Drift (µg/min) and the Average Noise (µg).

The Drift is calculated as the slope of a line fitted to the mass vs. time curve across the time window specified (explained below).

The Average Noise is calculated as the average change in mass between each successive mass value in the time window specified.

The average noise values are not affected by any spikes in the data. **Spikes** are ignored in the average noise calculation and listed in a table on the right-hand side of the report that gives the size (µg) of the spike and the time (minutes) at which it occurred (see Figure 11.3 below):

Start Time (mins)	Drift (µg/min)	Average Noise (µg)	Time (mins)	Spike (µg)
0.2	-118.472		1.2	-117.0
60.2	-10.870		2.2	-1077.0
120.2	-2.958	0.333	3.2	-119.0
180.2	-1.487	0.313	4.2	-866.0
240.2	-0.912	0.217	5.2	-109.0
300.2	-0.144	0.277	6.2	-114.0
360.2	0.494	0.167	7.2	-858.0
420.2	0.070	0.190	8.2	-115.0

Figure 11.3: Spike data in the Drift and Noise Check Report

At times, there may be too many spikes in a particular data window to allow an average noise calculation. In this case, the following dialogue box will appear (see Figure 11.4 below):

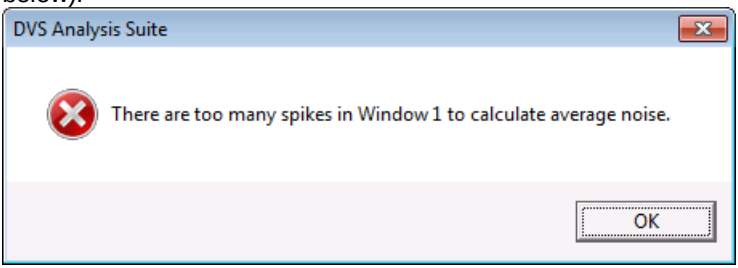


Figure 11.4: Drift and Noise Check error message

This may occur only once or several times in one report calculation. In any case, the user simply needs to click **OK** each time a box appears. The report will be compiled after all the boxes have been 'clicked' through.

At the bottom of the table of drifts and average noises are the average values for the data (see Figure 11.5 below). The value next to **Window average:** is the average of the absolute values of the drifts in each drift check time window.

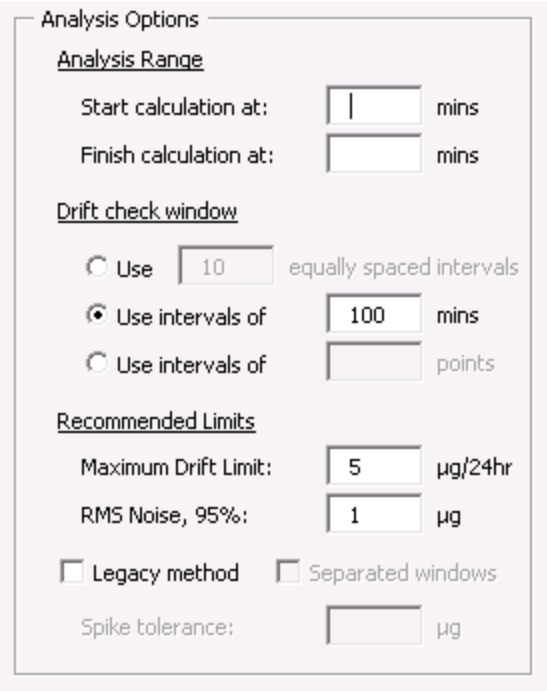
The values next to **File average:** are the average drift and noise over the whole file (these are not averages of the check window values).

Start Time (mins)	Drift (µg/min)	Average Noise (µg)
5525.0	0.014	0.027
5530.0	0.015	0.020
5535.0	0.014	0.013
5540.0	-0.036	0.020
5545.0	0.036	0.020
5550.0	-0.077	0.027
5555.0	0.023	0.020
5560.0	0.051	0.013
5565.0	-0.044	0.020
5570.0	0.049	0.013
5575.0	-0.021	0.007
5580.0	-0.029	0.011
Window average:	0.029	
File average:	0.000	0.022

Figure 11.5: The average values given at the bottom of the report – in this case, 'freeze panes' is activated in Excel, giving rise to the extra lines running through the report

11.4.2.1 Analysis Options

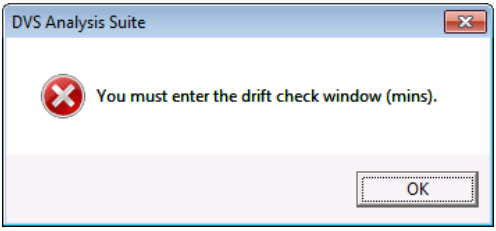
Before clicking the **Check Drift & Noise** button, the **Drift check window** and the **Spike tolerance** settings must be specified in the **Analysis Options** box:



The **Drift check window (mins)** specifies the length of the time steps over which drift and average noise values are calculated. If further advice is required on using this function, please contact Surface Measurement Systems.

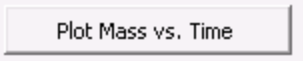
The **Spike tolerance (µg)** is the value above which the change in mass between two successive points is not included in the average noise calculation, but is added to the list of spikes in the data. If further advice is required on using this function, please contact Surface Measurement Systems.

If these parameters are not specified before clicking the Check Drift and Noise button, the following error message appears:



A Drift and Noise Check report may be calculated only for one DVS data file at a time.

11.4.3 Plot Mass vs. Time



Clicking the Plot Mass vs. Time button produces a plot of mass (mg or g) against time (minutes) for the Drift & Noise check experiment just conducted (see Figure 11.6 below).

The plot produced has the same format as those produced by the DVS Plot Manager.

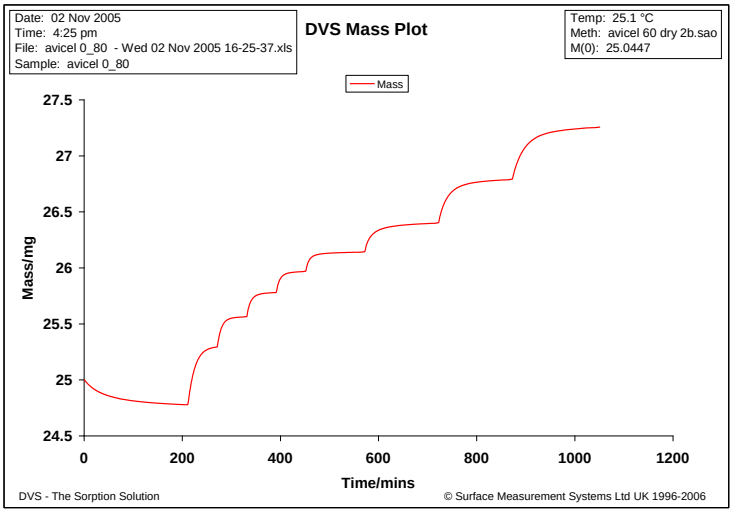


Figure 11.6: A **DVS Mass Plot**.

11.4.3.1 Multiple DVS Mass Plots

A Mass vs. Time plot may be calculated only for one DVS data file at a time.

11.4.4 Enhanced Calculation

A more rigorous calculation may be performed by utilizing the following settings.

Low Mass:-

a) Use intervals of 100 minutes
b) Use Maximum Drift Limit of 5ug/24hr
c) Use RMS Noise, 95% of 1ug

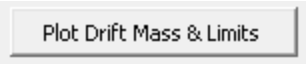
High Mass:-

a) Use intervals of 100 minutes
b) Use Maximum Drift Limit of 50ug/24hr
c) Use RMS Noise, 95% of 10ug

To undertake a calculation use the following procedure:-

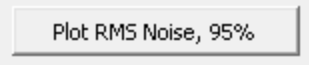
a) Click the Drift Noise Check Toolbar Button
b) Click 'Plot Mass vs. Time' and then Exit
c) Identify any linear range from the previous plot
d) Click the Drift Noise Check Toolbar Button
e) If a range has been identified enter the start and finish times (otherwise leave these unfilled)
f) Enter the Low/High Mass values from above
g) Click 'Check Drift and Noise'
h) Click 'Plot Drift Mass & Limits'
i) Click 'Plot RMS Noise, 95%' and the Exit
j) Inspect the report for any failures
k) The Actual Drift in the report should be less than 5ug for a Low Mass balance and less than 50ug for a High Mass balance (or less than whatever limits have been used)

11.4.5 Plot Drift Mass & Limits



Clicking the Plot Drift Mass & Limits button produces a plot of drift mass (µg) against time (minutes) for the Drift & Noise check experiment just conducted. This is only available for the enhanced calculation.

11.4.6 Plot RMS Noise, 95%



Clicking the Plot RMS Noise, 95% button produces a plot of noise (µg) against time (minutes) for the Drift & Noise check experiment just conducted. This is only available for the enhanced calculation.

If further advice is required on using this function, please contact Surface Measurement Systems.

12 DVS Service Baseline

12.1 What does this module do?

The **DVS Service Baseline** module is used to analyse the drift and noise in the mass data from an experiment with no sample hanging from the balance (using a simpler interface than the Drift Noise module).

12.2 Why is this useful?

This module is important as it allows the service engineer to run a quick diagnostic test of the instrument's baseline stability. Stability of the instrument's mass baseline is obviously crucial to its ability to produce meaningful data.

12.3 What kind of experimental set-up is required for a drift and noise check?

In order to perform a service baseline check, instrument data must be recorded with no sample present in the system.

Clean sample pans must be placed in the instrument and mass baseline data recorded at 0% P/P_o and one temperature.

12.4 How to use this module

Clicking the DVS Service Baseline button calls up the main dialog screen for the module as shown below.

On the left hand side of the dialog screen are the calculation, plotting and 'file opening'/'file importing' buttons. In the bottom left-hand corner is the DVS Version selection box. On the right hand side are the selection areas for the analysis options.

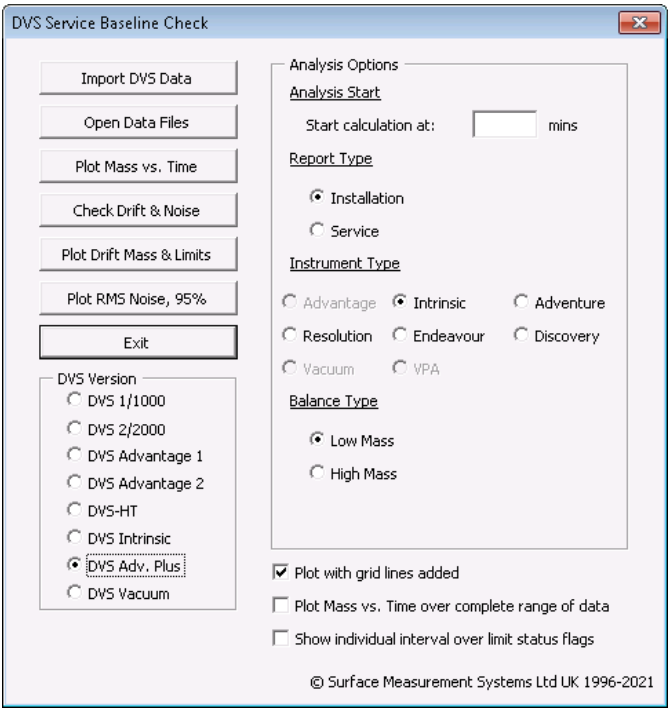
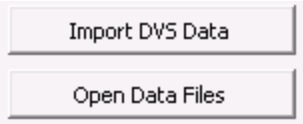


Figure 12.1: The DVS Service Baseline main dialog screen.

12.4.1 Import DVS Data & Open Data File

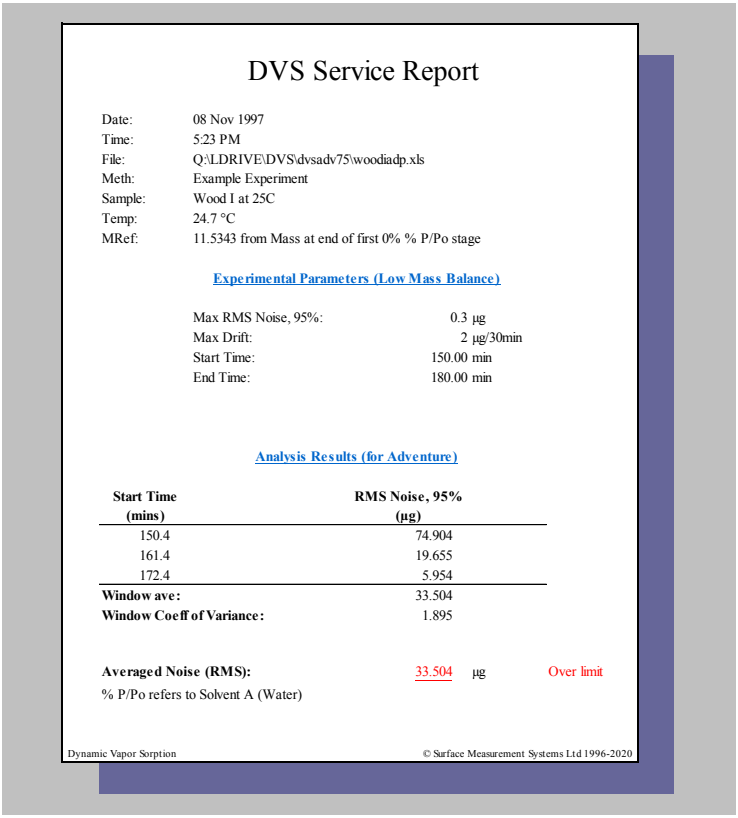


These functions are explained in detail in Section 4.2 and Section 4.3, respectively.

Before any partial drift and noise checks or plots may be carried out, a raw DVS data file must first be open in Excel using these buttons, which appear on every module main panel - the file(s) may be imported and opened from any module before using the drift and noise check functions.

12.4.2 Check Drift & Noise

Clicking the Service Baseline button produces a DVS Service Baseline report from an appropriate DVS data file (see Figure 12.2 below).



DVS Service Report

Date: 08 Nov 1997
Time: 5:23 PM
File: Q:\LDRIVE\DVS\dvsadv75\woodiadp.xls
Meth: Example Experiment
Sample: Wood I at 25C
Temp: 24.7 °C
MRef: 11.5343 from Mass at end of first 0% % P/Po stage

Experimental Parameters (Low Mass Balance)

Max RMS Noise, 95%:	0.3 µg
Max Drift:	2 µg/30min
Start Time:	150.00 min
End Time:	180.00 min

Analysis Results (for Adventure)

Start Time (mins)	RMS Noise, 95% (µg)
150.4	74.904
161.4	19.655
172.4	5.954
Window ave:	33.504
Window Coeff of Variance:	1.895

Averaged Noise (RMS): 33.504 µg Over limit

% P/Po refers to Solvent A (Water)

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Figure 12.2: A **DVS Service Report (for DVS Adventure)**

The report gives the Drift (µg/min) If an Installation Report is selected and the Average Noise (µg) if either an Installation or Service Report is selected (excludes Endeavour where Drift is always given).

The calculations are as for the Drift and Noise module but apart from the starting time all other options are pre-set.

The Drift, if displayed, is calculated as the slope of a line fitted to the mass vs. time curve across the time window specified (explained below).

The Average Noise is calculated as the average change in mass between each successive mass value in the pre-set time window.

12.4.2.1 Analysis Options

Before clicking the **Check Drift & Noise** button, the **Installation/Service Type, Instrument Type, Mass Type** and **Start Time** settings must be specified in the **Analysis Options** box:

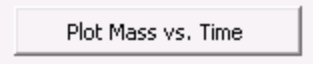
The **Installation/Service Type** specifies whether an Installation Report is produced or a Service Report is produced.

The **Instrument Type** specifies the type of instrument the report refers to.

The **Mass Type** specifies whether a low or high mass balance is being used.

A Service Baseline report may be calculated only for one DVS data file at a time.

12.4.3 Plot Mass vs. Time



Clicking the Plot Mass vs. Time button produces a plot of mass (mg or g) against time (minutes) for the Drift & Noise check experiment just conducted (see Figure 12.6 below).

The plot produced has the same format as those produced by the DVS Plot Manager.

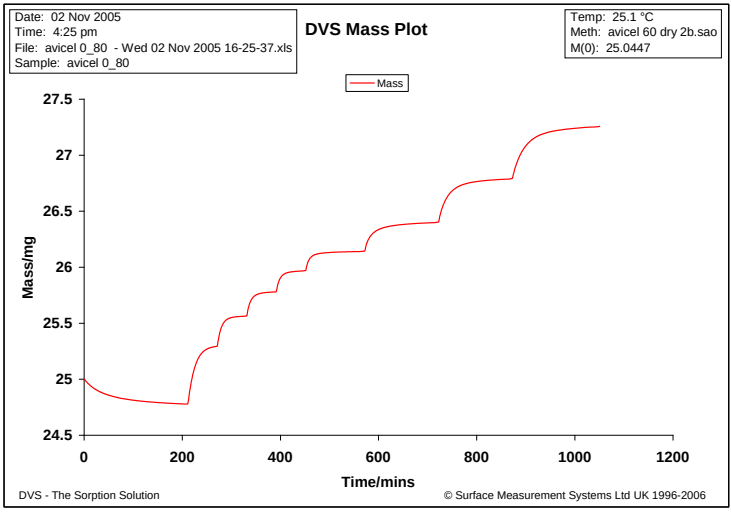
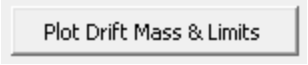


Figure 12.3: A **DVS Mass Plot**.

12.4.3.1 Multiple DVS Mass Plots

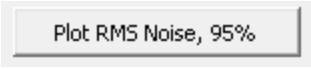
A Mass vs. Time plot may be calculated only for one DVS data file at a time.

12.4.4 Plot Drift Mass & Limits



Clicking the Plot Drift Mass & Limits button produces a plot of drift mass (µg) against time (minutes) for the Drift & Noise check experiment just conducted.

12.4.5 Plot RMS Noise, 95%



Clicking the Plot RMS Noise, 95% button produces a plot of noise (µg) against time (minutes) for the Drift & Noise check experiment just conducted.

13 DVS Method Report

13.1 What does this module do?

The **DVS Method Report** module is used to open a DVS method SAO/SEQ/PRE file and create a report from it adding presentation quality titles and borders.

On computers connected to a printer, the page setup and print area are also set so that the report is ready to print.

13.2 Why is this useful?

The **DVS Method Report** macro saves user time by instantly summarising key DVS experimental information in a presentation-quality format.

13.3 How to use this module

Clicking the DVS Method Report button calls up the main dialog screen for the module as shown below.

On the top left-hand side of the dialog screen are the Create Report and 'file opening'/'file importing' buttons. In the bottom left-hand corner is the DVS Version selection area. On the right hand side are the selection areas for the report creation options.

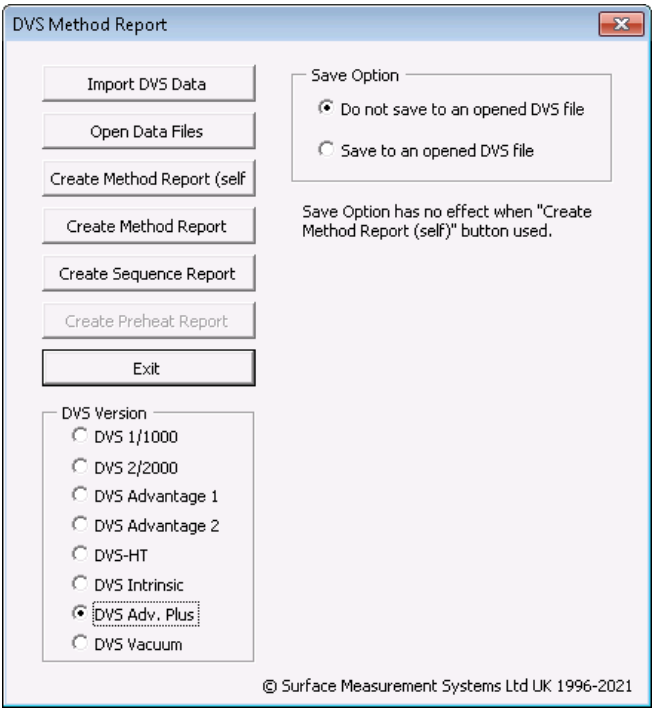
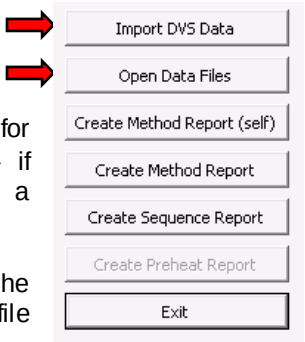


Figure 13.1: The **DVS Method Report** main dialog screen

13.3.1 Import DVS Data & Open Data File

These functions are explained in detail in Section 4.2 and Section 4.3, respectively.



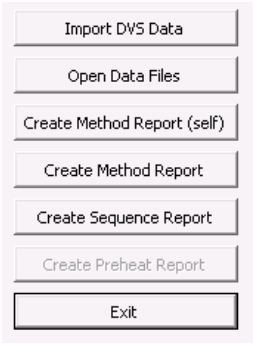
A DVS file does not need to be open for a method report to be compiled – if desired, the report can be saved to a new Excel file.

If, however, the user wishes to save the method report to a DVS data file, this file

must first be open in Excel using these buttons, which appear on every module main panel - the file(s) may be imported and opened from any module before method report functions.

13.3.2 Create Method Report

Clicking any one of the **Create Method/Sequence/Preheat Report** buttons displays an **Open** file selection dialog box for the DVS SAO/MTD/SEQ/PRE file from which the report information will be extracted (see Figure 13.2 below). Self indicates Report data is held in the DVS Data Sheet.



Note: If using the DVS 1/1000 or 2/2000, then only the Create Method Report button will be active – sequences and preheat are not used with these earlier DVS models.

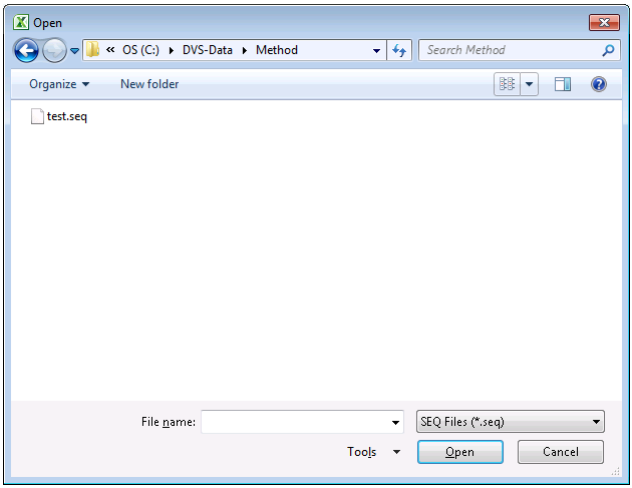


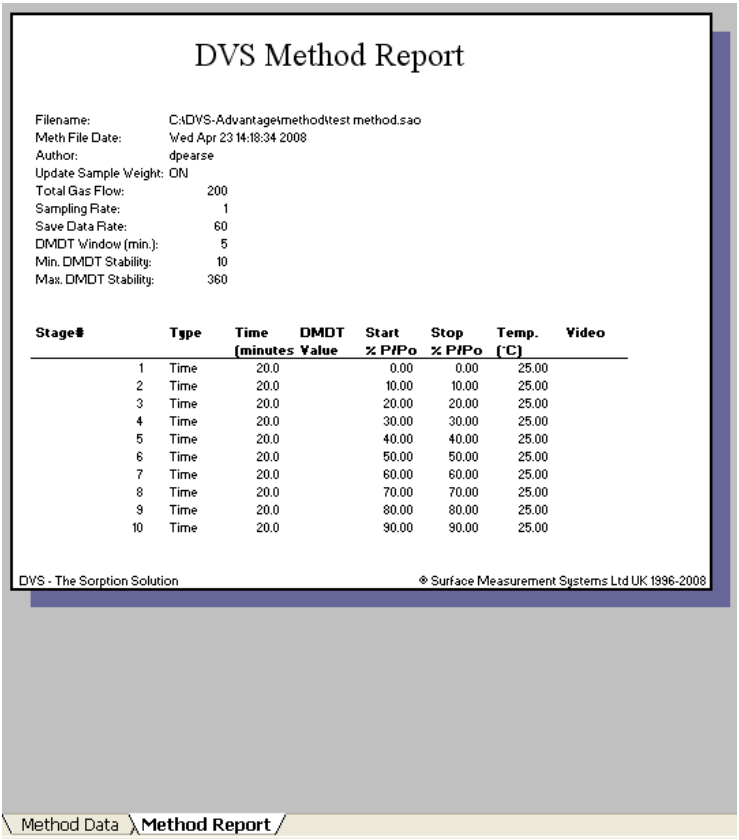
Figure 13.2: The **Open** file selection dialog box for selecting the file to be summarised in a Report

Upon selecting the desired file and clicking **Open**, a report is produced in Excel.

The reports produced are shown below:

13.3.2.1 Method Report

The Method Report summarises the details pertaining to each $\%P/P_o$ stage of a DVS experimental method:



DVS Method Report

Filename: C:\DVS-Advantage\method\test method.sao
Meth File Date: Wed Apr 23 14:18:34 2008
Author: dpearse
Update Sample Weight: ON
Total Gas Flow: 200
Sampling Rate: 1
Save Data Rate: 60
DMDT Window (min.): 5
Min. DMDT Stability: 10
Max. DMDT Stability: 360

Stage#	Type	Time (minutes	DMDT Value	Start % P/Po	Stop % P/Po	Temp. (°C)	Video
1	Time	20.0		0.00	0.00	25.00	
2	Time	20.0		10.00	10.00	25.00	
3	Time	20.0		20.00	20.00	25.00	
4	Time	20.0		30.00	30.00	25.00	
5	Time	20.0		40.00	40.00	25.00	
6	Time	20.0		50.00	50.00	25.00	
7	Time	20.0		60.00	60.00	25.00	
8	Time	20.0		70.00	70.00	25.00	
9	Time	20.0		80.00	80.00	25.00	
10	Time	20.0		90.00	90.00	25.00	

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Method Data | Method Report

Figure 13.3: A **DVS Method Report**.

A spreadsheet of the Report data also appears in the same Excel file on sheet entitled **Method Data:**

DVS ADVANTAGE PARTIAL PRESSURE METHOD FILE							
Version:	1.01						
Filename:	C:\DVS-Advantage\method\test method.sao						
Method Fil	Wed Apr 23 14:18:34 2008						
Author:	dpearse						
Update Sa	ON						
Total Gas	200						
Sampling F	1						
Save Data	60						
DMDT Wir	5						
Min. DMDT	10						
Max. DMD	360						
Stage#	Type	Time	DMDT Vali	Start PP(%	Stop PP(%	Temp.(C)	Video
1	Time	20		0	0	25	
2	Time	20		10	10	25	
3	Time	20		20	20	25	
4	Time	20		30	30	25	
5	Time	20		40	40	25	
6	Time	20		50	50	25	
7	Time	20		60	60	25	
8	Time	20		70	70	25	
9	Time	20		80	80	25	
10	Time	20		90	90	25	

Method Data | Method Report

This **Method Data** sheet appears in the Sequence and Preheat report Excel files also.

13.3.2.2 Sequence Report

The information summarised in a DVS Sequence Report is shown below:

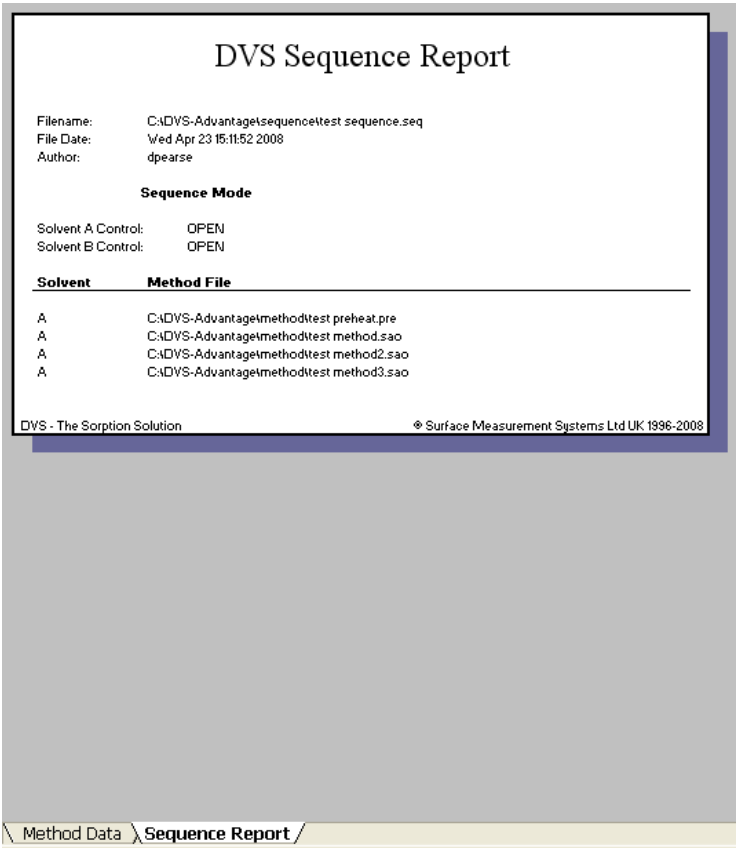


Figure 13.4: A **DVS Sequence Report**.

13.3.2.3 Preheat Report

The information summarised in the DVS Preheat Report is shown below:

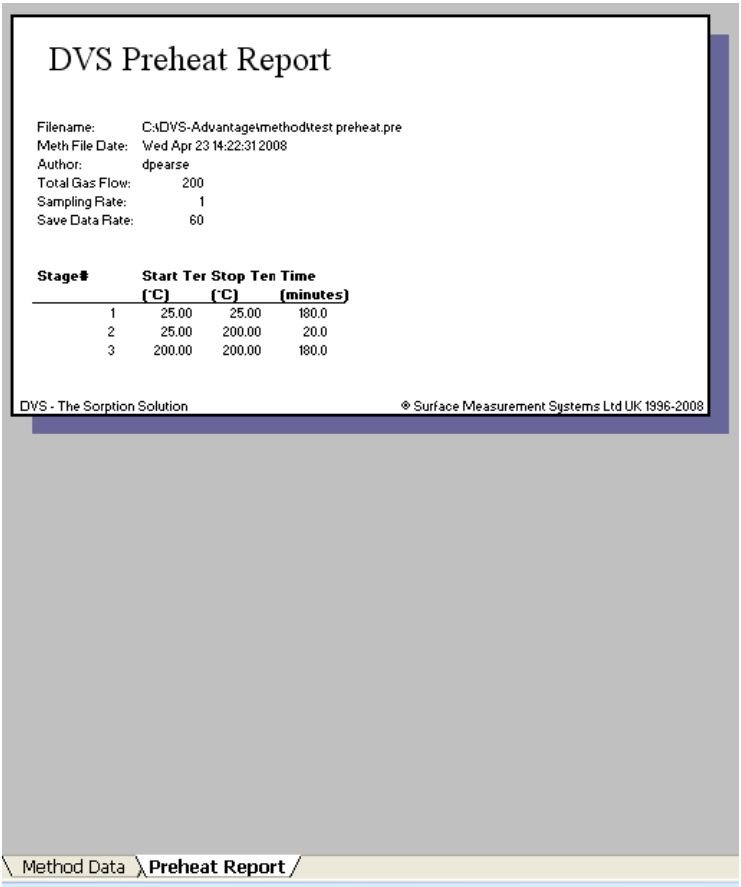
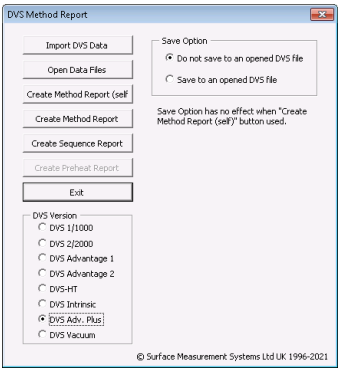


Figure 13.5: A **DVS Preheat Report.**

13.3.3 Save Option

Before any of the **Create Report** buttons are clicked, the user must specify where the report is to be saved using the **Save Option** selection area:



13.3.3.1 Do not save to opened DVS file

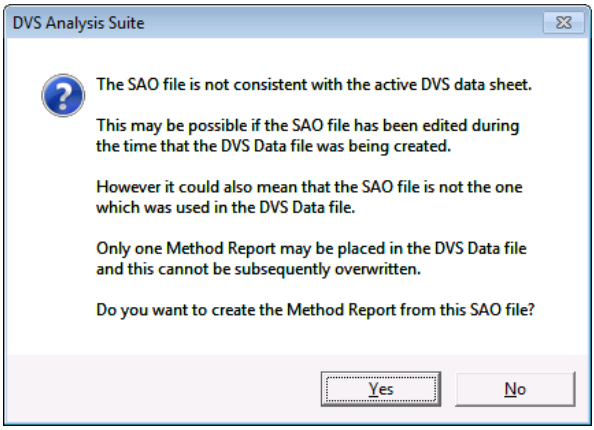
If this option is selected then the Method/Sequence/Preheat Report will be created in a new workbook which must be explicitly named after the DVS Method Report module has been exited if the data is to be saved.

13.3.3.2 Save to opened DVS file

If this option is selected then the Method/Sequence/Preheat Report will be created in the current active workbook.

If that workbook's SAO/SEQ/PRE file has the same name as the one being summarised in the Report, then the Report will be saved to the DVS file immediately.

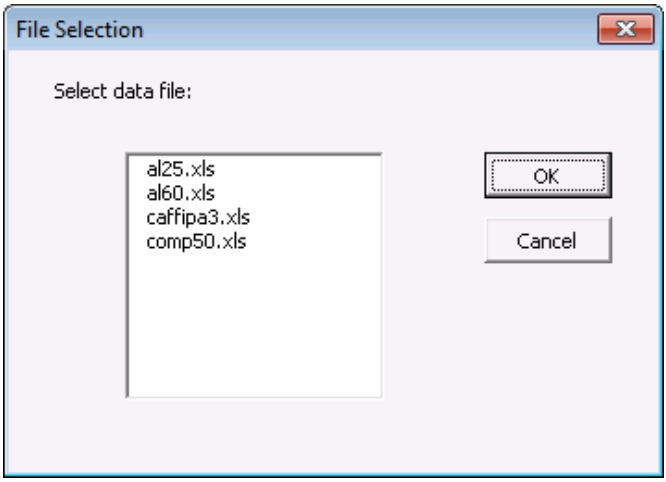
If the active workbook's SAO/SEQ/PRE file does not share the same name as the one being summarised in the Report, then the following message will appear:



Upon clicking **Yes**, the Report will appear in the active workbook.

Upon clicking **No**, no Report is compiled and the screen returns to the Method Report main dialog panel.

If several DVS files are open when any of the **Create Report** buttons are clicked, the open files are listed in the following panel:



The user must choose a destination file for the Report by clicking on one of the files listed and then clicking **OK** – only one file may be selected.

13.3.4 Multiple Reports

Only one Method/Sequence/Preheat Report may be calculated for one DVS data file at a time.

14 DVS Configuration

14.1 What does this module do?

This module allows the user to configure the display units of relative vapour pressure used by the DVS Analysis Suite software. It also allows configuration of other features.

14.2 Why is this useful?

The generic term 'relative vapour pressure' (abbreviated here as 'partial pressure') satisfactorily describes the amount of vapour being used in an experiment.

However, there are more specific terms which more accurately describe the vapour being used or the type of experiment being conducted.

For instance, the term Relative Humidity is a more appropriate term to use when water vapour is in use, and Water Activity is a more useful descriptor for experiments in the food industry.

Use of these more specific names for relative vapour pressure in the software interfaces and the plots and reports they produce aids clarity.

14.3 How to use this module

Clicking the DVS Config button calls up the main dialog screen for the module, as shown below.

On the left-hand side of the dialog screen are the **OK** and **Cancel** buttons. On the right hand side is the **RVP** (relative vapour pressure) **Display Unit** selection area, which features the following options; Relative Humidity, Partial Pressure (which both are expressed as percentages) and Water Activity (which expresses water relative vapour pressure as a decimal fraction). A Default option is also available, which is explained below in Section 14.3.2 below.

Whichever configuration is chosen, it remains selected until a change is made – even when the Analysis Software is shut down and re-started.

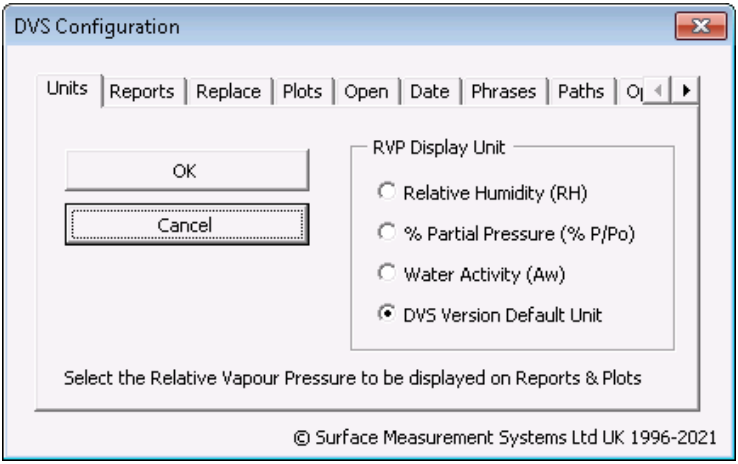


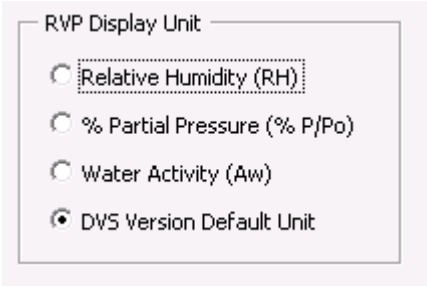
Figure 14.1: The **DVS Configuration** main dialog screen.

Note: It is important to ensure that the choice of RVP Display Unit is appropriate for the solvent in use in the DVS system – for instance, using Relative Humidity or Water Activity would be inappropriate if organic solvents were being used.

The software will not warn the user if the RVP Display Unit does not match the solvent in use.

14.3.1 Relative Humidity (RH)/% Partial Pressure (% P/P_o)/Water Activity (A_w)

Selection of any of these options followed by clicking **OK** will ensure that the amount of vapour used in an experiment will be expressed in the chosen units in the software interfaces, plots and reports.



14.3.1.1 Software Interfaces

As an example, the import/export and plotting buttons on the **DVS Plot Manager** module are shown below in Figure 14.2 when the **Relative Humidity**, **Partial Pressure**, and **Water Activity** configurations are chosen, respectively:

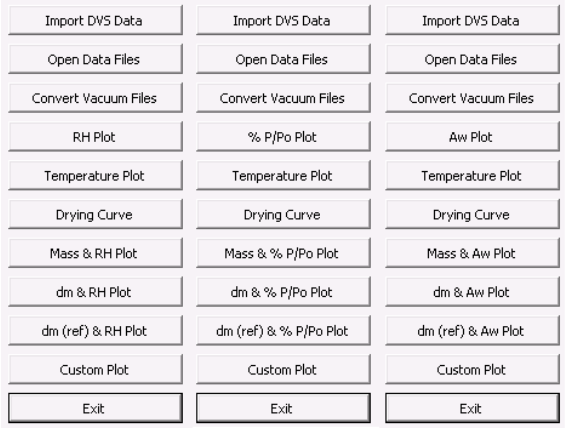


Figure 14.2: DVS Plot Manager buttons in their respective Relative Humidity, Partial Pressure and Water Activity configurations.

These changes in notation are applied to all of the Analysis Software modules. Also note that when the Relative Humidity and Water Activity configurations are in place, then the term **Moisture** is used; the Partial Pressure configuration uses the term **Vapour**.

14.3.1.2 Importing and Opening data files

When importing or opening DVS files, the Configuration setting will affect only those units (if any) displayed in the **Reference Mass Option** cell (refer Section 5.3.10) in the DVS data sheet.

All of the other relative vapour pressure units in the data sheet will be displayed in units of 'RH' or 'PP', depending upon the DVS Version selected (refer Sections 4.3.2 and 14.3.2) – see Figure 14.3 below:

)				
Ref. Mass Option	Mass at end of first 0.0 Aw method stage			
Ref. Mass	23.9693			
TEMP (samp)	TEMP (ref)	Target RH	Target TEM	Sine RH
24.9	24.7	95	25	0
24.9	24.7	95	25	0
24.9	24.7	95	25	0
24.9	24.8	95	25	0
24.9	24.7	95	25	0
24.9	24.8	95	25	0

Figure 14.3: Configuration units appear in the **Ref. Mass Option** cell, but nowhere else in a DVS data file.

If the Configuration settings are altered while a data file is open, the display units will not change. If the file is subsequently opened with the new Configuration settings in place, then the **Ref. Mass Option** cell will change to the new display units without a notification panel appearing.

14.3.1.3 Plots

The choice of configuration will affect the labeling of Plots produced by the Analysis software, as shown below in Figure 14.4 and Figure 14.5:

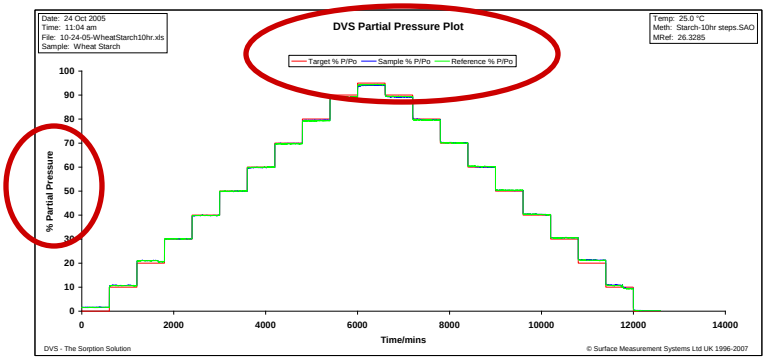


Figure 14.4: A DVS Plot featuring Partial Pressure labeling

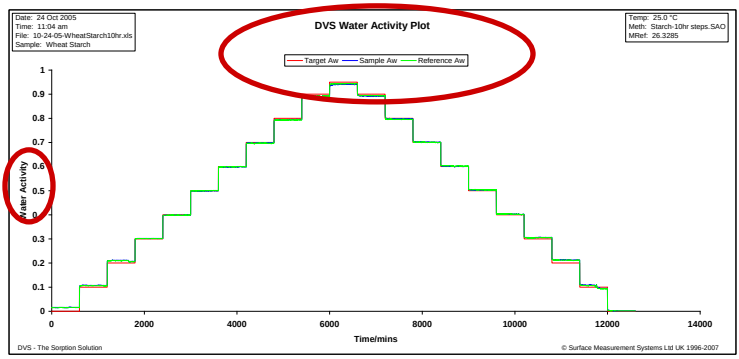


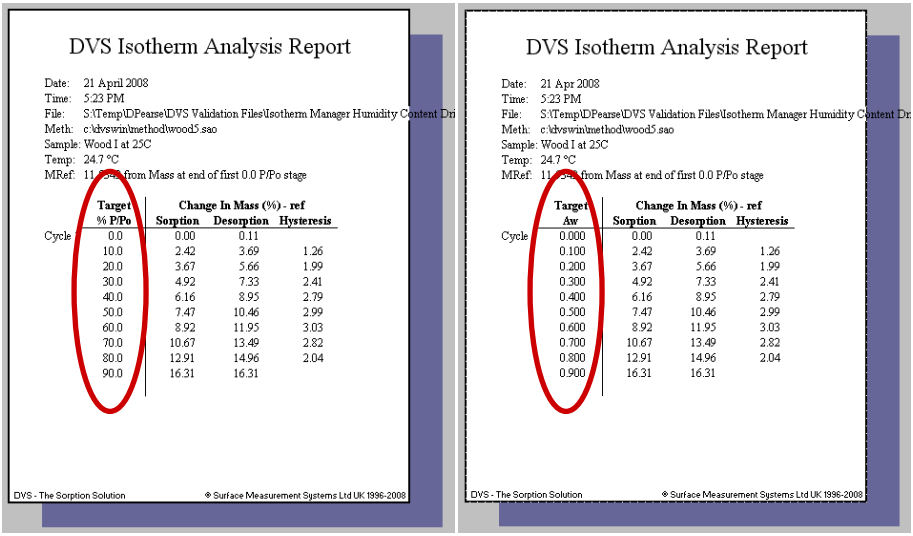
Figure 14.5: A DVS Plot featuring Water Activity labeling

The configuration can be altered with the file still open – the plots produced prior to the change will retain their original labelling, while all plots produced subsequently will feature the new configuration's notation. No plots are overwritten – a new sheet is created for each new plot. In this way, the same plot can be produced using different notations and saved in the one file.

If the file is closed and re-opened under a new configuration, the plots in the file will retain their original notations.

14.3.1.4 Reports

Reports produced by the Analysis software will also feature the notation of the chosen configuration Figure 14.6 below shows an Isotherm Report produced using the Water Activity and Partial Pressure configurations, respectively:



DVS Isotherm Analysis Report

Date: 21 April 2008
Time: 5:23 PM
File: S:\Temp\DPearse\DVS Validation Files\Isotherm Manager Humidity Content Dri
Meth: c:\dvswin\method\wood5.sao
Sample: Wood I at 25C
Temp: 24.7 °C
MRef: 11.[illegible] from Mass at end of first 0.0 P/Po stage

Cycle	Target % P/Po	Change In Mass (%) - ref Sorption	Desorption	Hysteresis
	0.0	0.00	0.11	
	10.0	2.42	3.69	1.26
	20.0	3.67	5.66	1.99
	30.0	4.92	7.33	2.41
	40.0	6.16	8.95	2.79
	50.0	7.47	10.46	2.99
	60.0	8.92	11.95	3.03
	70.0	10.67	13.49	2.82
	80.0	12.91	14.96	2.04
	90.0	16.31	16.31	

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DVS Isotherm Analysis Report

Date: 21 Apr 2008
Time: 5:23 PM
File: S:\Temp\DPearse\DVS Validation Files\Isotherm Manager Humidity Content Dr
Meth: c:\dvswin\method\wood5.sao
Sample: Wood I at 25C
Temp: 24.7 °C
MRef: 11.[illegible] from Mass at end of first 0.0 P/Po stage

Cycle	Target Aw	Change In Mass (%) - ref Sorption	Desorption	Hysteresis
	0.000	0.00	0.11	
	0.100	2.42	3.69	1.26
	0.200	3.67	5.66	1.99
	0.300	4.92	7.33	2.41
	0.400	6.16	8.95	2.79
	0.500	7.47	10.46	2.99
	0.600	8.92	11.95	3.03
	0.700	10.67	13.49	2.82
	0.800	12.91	14.96	2.04
	0.900	16.31	16.31	

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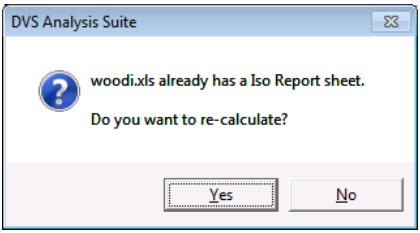
Figure 14.6: The same Isotherm Report produced under Partial Pressure and Water Activity configurations, respectively.

Unlike Plots, Analysis Suite Reports are overwritten each time a new Report is produced. If a Report already exists in a file and a new one is requested (whether under a new configuration or not), a message similar to that shown below will appear:



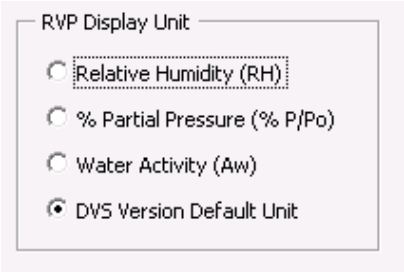
Clicking **Yes** will produce the new report and replace the old one – if the configuration has been changed or the file has

been opened under a new configuration, the report will feature the notation of the new configuration.

Also note, however, that if the file is closed and re-opened under a new configuration, the report in the file will retain its original notations.

14.3.2 DVS Version Default Unit

Selection of this option followed by clicking **OK** will ensure that the default configuration will be used, as determined by the DVS Version selected in the options box (see Section 4.3.2).



The default configurations are assigned to the various DVS Versions as follows:
DVS 1/1000 and DVS 2/2000 and Intrinsic: Display **RH** and **Moisture**

DVS Advantage 1, 2 and HT: Display **%P/P$_o$** and **Vapour**

Note that Water Activity is not a default option for any of the DVS Versions.

Note also that all of the Configuration rules which apply to file opening/importing, plotting and report creation which have been stated above also apply to the **DVS Version Default Unit** option.

14.3.3 Reports Tab (Not available if CFR)

Selection of this tab allows the report options to be changed.

The report can include the Path and File name or the File Name only.

The report can switch to Sample Description rather than use Sample Name.

14.3.4 Replace Tab

Selection of this tab allows the text Sample to be replaced with Actual in dialogs and reports etc. It also allows the text Actual to be replaced by Measured in dialogs and reports.

14.3.5 Plots Tab

Selection of this tab allows the Number of Points per Plot Series to be set (recommended 32000).

Be aware that any value greater than 32000 can only be used by Excel 2007 or later and that selecting Number of Points per Plot Series as the Excel max Limit may give issues when files are read back with Excel 2003 or earlier.

14.3.6 Open Tab

Selection of this tab allows fast opening of files. If the number of rows in files are larger than 30000 it is recommended that this is switched on.

14.3.7 Date Tab

Selection of this tab allows pre v7.1 date parsing behaviour.

14.3.8 Phrases Tab

Selection of this tab allows the Copyright text and Promotion text to be set. The text can either be set manually, set from internal example, reset to first install behaviour or cleared.

14.3.9 Paths Tab

Selection of this tab allows the paths that the Analysis s/w starts of with to be configured (separate Open and Import). The behaviour is either none set or the last folder used by the Analysis. In addition if folder is set it may also be fixed.

14.3.10 Opening Tab (Not available if CFR)

Selection of this tab allows exported text files to be opened by the Analysis s/w (if supported as an alternative to import).

14.3.11 HomeSheet Tab

Selection of this tab allows the resting sheet to be either the Report Sheet or whatever sheet was last created.

14.3.12 Redisplay Tab

Selection of this tab allows the form to be forced to be redisplayed dependant on Excel version.

14.3.13 CFRFixupPath Tab

Selection of this tab allows the behaviour of DenyDelete to be controlled when in CFR mode (not to be used unless advised

by SMS). It can only be changed when NOT in CFR mode but it ONLY affects operation in CFR mode (this is by design so that changes cannot be made when in CFR mode).

14.3.14 StartSheet Tab

Selection of this tab allows the Start Sheet to be set to DVS Data when files are opened (where this is allowed).

14.3.15 SaveAs Tab

Selection of this tab allows the default Save As behaviour to be controlled in non-CFR mode. Historically non-CFR Analysis s/w versions saved as xls provided the data could fit into 65535 rows (Excel 2003 row limit – 1). Using this tab allows this behaviour to be retained or overridden. However, note that when in CFR mode, the Excel default file Save As option is used provided the IWB is set up for both xls and xlsx files (otherwise only xls is used and that is limited to 65535 rows).

14.3.16 DataExcluded Tab

Selection of this tab allows the default Data Excluded behaviour to be controlled. Normally the data just prior to a step change is not excluded from Analysis. Using this tab allows the Data Excluded edit box to be enabled in the Isotherm, PiE, BET, HOS, Isobar and AC calculations. This is useful when data next to a step change is affected by, for example, Raman data acquisition.

14.3.17 Half Cycle Access Tab

Selection of this tab allows the pre v7.4 Advanced behaviour for Half Cycle Access of Isotherm data (not to be used unless advised by SMS).

14.3.18 No Chart Info Boxes Tab

Selection of this tab suppresses the creation of chart info boxes.

14.3.19 Repeat Warn Sys Separator Tab

Selection of this tab enables the System Separator Warning Check to be made every time the s/w starts.

14.3.20 Store AC Calib File Info Tab

Selection of this tab enables the ability to store previously used calib files details.

14.3.21 CFR User ID Tab (CFR Only)

Selection of this tab enables the ability to write the CFR User ID to reports and plots (requires CFR Admin Priv).

14.3.22 Plot Ranges Tab

Selection of this tab enables the ability for the Permeability Fit Plot Axes limits to be chopped so as to remove white space in the Permeability Fit chart.

14.3.23 Num Chars Read Tab

The analysis s/w needs to know what type of DVS file is imported/opened prior to it actually being opened. Selection of this tab enables the ability of the analysis s/w to extend the number of characters searched to determine the DVS file type.

14.3.24 Plot Drop Down Tab

Selection of this tab enables the ability to set items in the Plot Manager drop down lists so that only those items NOT explicitly mentioned in the options list are included (in the drop down list) or all are included.

14.3.25 Block CFR Analysis Tab (CFR Only)

Selection of this tab enables the ability to block CFR Analysis running when not in CFR mode (requires CFR Admin Priv).

14.3.26 Zoom Tab

Selection of this tab allows the Plot Manager form to be zoomed from 95% to 105% of its default value (recommended range is 98% to 102%).

14.3.27 DVS Any Tab

Selection of this tab allows Plot Manager and Isotherm analysis to analyse DVS Advantage 1, DVS-HT, DVS Intrinsic, DVS Advantage Plus and DVS Vacuum files at the same time (e.g. to combine plots from several different DVS file types into one plot). This applies ONLY to Plot Manager and Isotherm Analysis. If the feature is turned on a toggle

button is also added to Plot Manager and Isotherm Suite so that the option can be quickly turned on and off.

14.3.28 Version Buttons Tab

Selection of this tab allows the old style DVS Model Buttons to be displayed instead of the new DVS Info Panel.

14.3.29 Wrap Merge Tab

Selection of this tab allows the Reports to double the height and span cells for the File: parameter. If the feature is turned on a toggle button is also added so that the option can be quickly turned on and off.

14.3.30 Custom1 Tab

Selection of this tab allows the Salt and Isotherm dialog to be modified (not to be used unless advised by SMS).

14.3.31 Custom2 Tab

Selection of this tab allows the text 'PLOT:' to be prepended to the Report and Plot titles and intervening spaces to be replaced by underscores in the titles themselves (not to be used unless advised by SMS).

Note: Some tabs above are also used by the Isotherm Analysis Suite which is a separate suite to the DVS Std/Adv Analysis. The specific tabs are Phrases, CFR User ID, Block CFR Analysis, Wrap Merge and Custom 2.

15 DVS Analysis Suite Info

The **DVS Information** module is a simple feature that allows the user to determine which version of the Analysis Suite is installed.

Clicking the **DVS Information** module button calls up the dialogue screen shown below.

This shows which version of the Analysis Suite is installed and whether it is the Standard or Advanced edition.

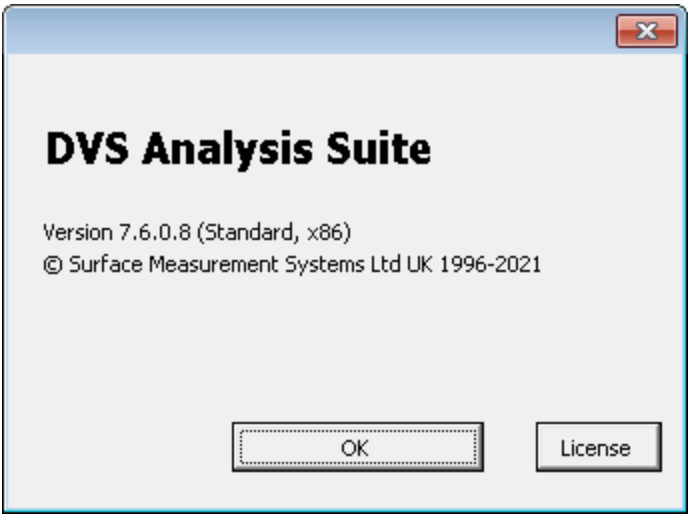


Figure 15.1: DVS Analysis Suite Info dialog screen.

When the DVS Analysis Suite is first installed, clicking **OK** on the Info dialog screen above may prompt further dialog screens to appear. If Windows Vista or later is being used some of these dialogs will NOT appear.

Clicking License allows the license information to be viewed.

Unlocking

Some of these additional screens allow the DVS Analysis Suite to be unlocked (released) after initial installation. In the case of Windows Vista or later there is a different procedure to be followed. Until the DVS Analysis Suite is released only the Info Button can be used.

In the case of Windows Vista or later a Release Key Application must be run. This can be found on the software distribution media and can also be launched from the SMS Launch Installer dialog.

The file **release.doc** describes this procedure, and can be found on the software installation CD.

Copyright and Promotion

The other additional screens allow the Copyright and Promotion string to be changed from the default SMS phrase. Note that this string, if altered, only applies to the Report and Plot spreadsheets – the Copyright message on the main DVS Information dialog screen remains unalterable. Note that clicking the **Close** button does not bring up the extra dialog screens.

Please also note that the Copyright and Promotion screens also will appear if the Analysis Suite has been re-installed after uninstallation.

If the **OK** button is clicked the dialog below is presented:

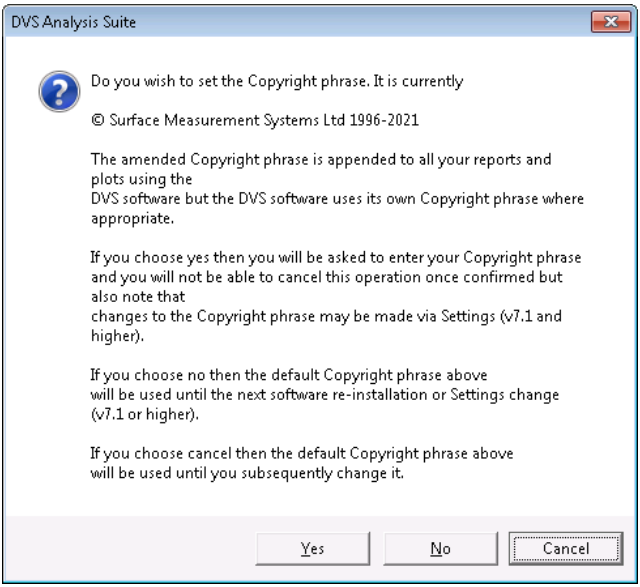


Figure 15.2: The DVS Analysis Suite Copyright dialog screen.

If the **No** button is pressed the default SMS Copyright message will become the permanent Report and Plot Copyright message. Only a re-installation of the software using the Uninstaller/Installer will allow it to be changed.

If the **Cancel** button is pressed the default SMS Copyright message will become the temporary Report and Plot Copyright message. If the user enters the **DVS Information** macro at a later stage the same opportunity to change the Copyright message will be presented.

If the **Yes** button is pressed the dialog screen shown below is presented which allows the Copyright message to be changed.

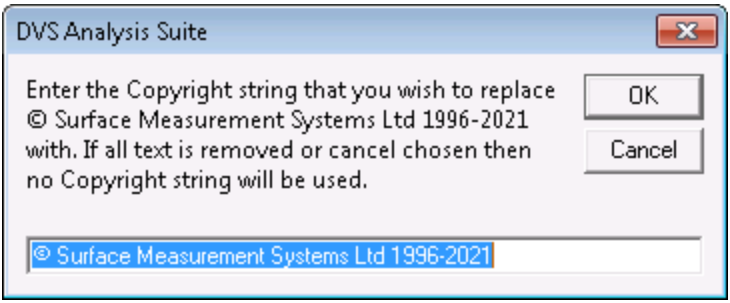


Figure 15.3: The DVS Analysis Suite Copyright input dialog screen.

By clicking the **OK** button, whatever is in the edit box becomes the permanent Copyright message; either the SMS default (if the user makes no change); or a different Copyright message (if the user makes a change); or nothing at all (if the user deletes the SMS default and enters nothing in its place).

Clicking the **Cancel** button will result in the process of changing the Copyright message being cancelled. This same action is initiated if the close button on the title bar is used. Being allowed to change the Copyright message in this manner is only available while the Copyright message remains at its default value. If changed from the default value then only by using the Settings dialog or re-installing using the Uninstaller/Installer can a subsequent change be made.

After pressing **OK** or **Cancel** or the **close** button the dialog below is presented:

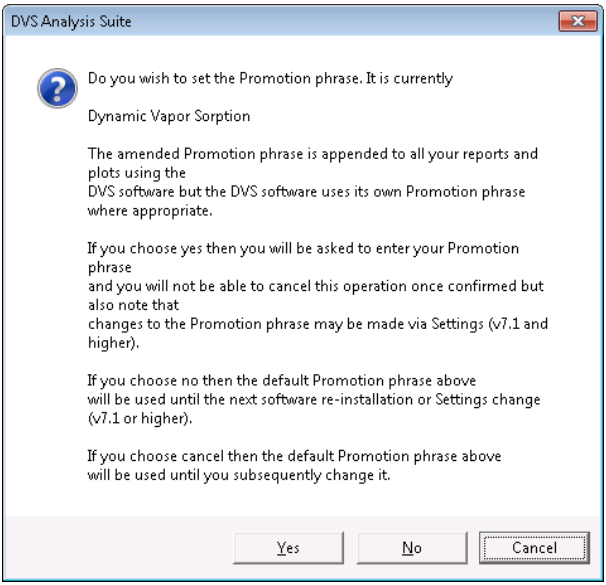


Figure 15.4: The **DVS Analysis Suite** Promotion dialog screen.

If the **No** button is clicked, the default SMS Promotion message will become the permanent Report and Plot Promotion message. Only a re-installation of the software using the Uninstaller/Installer will allow it to be changed.

If the **Cancel** button is pressed the default SMS Promotion message will become the temporary Report and Plot Promotion message. By subsequently entering the DVS Information macro, the same opportunity to change the Promotion message will be presented.

If the **Yes** button is pressed the dialog screen shown in Figure 14.5 below is presented which allows the Promotion message to be changed:

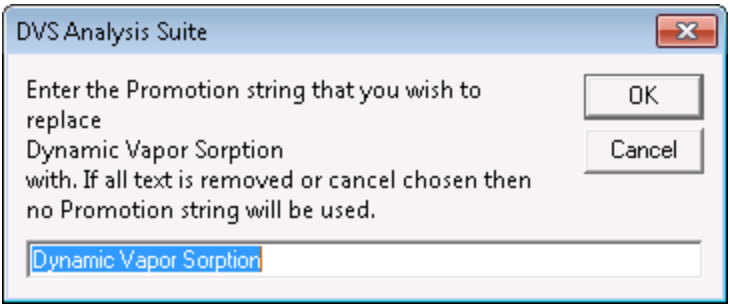


Figure 15.5: The DVS Analysis Suite Promotion input dialog screen.

By clicking the **OK** button, whatever is in the edit box becomes the permanent Promotional message; it will be either the SMS default (if the user makes no change); or a different Copyright message (if the user makes a change); or nothing at all (if the user deletes the SMS default and enters nothing in its place).

Clicking the **Cancel** button will result in the process of changing the Promotion message being cancelled. This same action is initiated if the close button on the title bar is used. Being allowed to change the Promotion message in this manner is only available while the Promotion message remains at its default value. If changed from the default value then only by using the Settings dialog or re-installing using the Uninstaller/Installer can a subsequent change be made.

16 Appendix A – Installation Instructions

The installation instructions for the DVS Analysis Suite differ depending on several factors. These factors include:

- The version of Windows and/or Excel being used
- If the user account that is being used with the DVS Analysis Suite has Administrator privileges
- Whether the auto-run procedure automatically starts up when the CD is inserted into the CD-ROM

The install instructions are divided up into the following sections:

- Installation Instructions for User Accounts with Administrator Privileges or any account on Vista
- Installation Instructions for Non-Administrator User Accounts
- DVS Analysis Suite Installation Instructions (XP or earlier)
- DVS Analysis Suite Installation Instructions (Vista or later)
- Important note for using Excel 2007

The DVS Analysis Suite is protected and instructions are listed below to UNLOCK this protection. Upgrades may require this process to be repeated

Note: The auto-run procedure, if present, launches the SMS Installer Menu dialog when the CD is inserted into the CD-ROM drive. This dialog provides access to the actual DVS Analysis Suite installer. The DVS Analysis Suite Installation Instructions section should be followed whether or not the auto-run procedure starts.

Note: Do NOT use RunAs to install the DVS Analysis Suite.

Note: Excel must not be run in Admin mode when installing.

Note: Excel must not be run with UAC off when installing.

Note: For Excel 2013 and higher release s/w before use.

16.1 Installation Instructions for User Accounts with Administrator Privileges or any Account on Vista

This case is applicable to Administrator logons on Win2000/XP when only the single Administrator logon will be used with the DVS Analysis Suite or any account on Vista.

When the DVS CD is placed in the CD drive the auto-run procedure should launch. Select “Launch Installer” and follow the steps in the DVS Analysis Suite Installation Instructions (XP or earlier) section below or in the case of Vista follow the steps in the DVS Analysis Suite Installation Instructions (Vista or later) section below.

16.2 Installation Instructions for Non-Administrator User Accounts

This case is applicable to non-administrator logons on Win2000/XP but initially requires an administrator to logon and install the DVS Analysis Suite software.

Note: Whenever Install is mentioned in the steps below follow the steps in the DVS Analysis Suite Installation Instructions (XP or earlier) section before returning to this section.

Although this manual deals with the Standard DVS Analysis Suite the instructions are given for both Standard and Advanced DVS Analysis Suites in case both are installed at the same time.

Step 1: Install on an admin account the DVS Standard and/or Advanced Analysis Suite.
Do NOT give a non-admin account admin privileges, use a proper admin account.
Choose NOT to install the Database if installing the DVS Advanced Analysis Suite.

Step 2: After step 1, both the DVS Analysis Suites MUST be UNLOCKED before they are subsequently removed from this account.
If only the DVS Standard Analysis Suite is installed then it must be unlocked. If both the DVS Standard and Advanced Analysis Suites are installed then both must be unlocked.

Step 3: If the DVS Analysis Suites are not to be used on the admin account they may now be uninstalled from that account.
The DVS Advanced Analysis Suite will issue a warning that there is no database to uninstall. This is normal and may be ignored.

Step 4: Install the DVS Analysis Suites on every non-Admin account that requires them.
The DVS Analysis Suites will issue a warning that a full install is not possible. This is normal and may be ignored.
Choose to install the Database for the DVS Advanced Analysis Suite.

Step 5 (Optional): If the DVS Standard Analysis Suite has been installed test the DVS Isotherm Manager to check that the DVS Standard Analysis Suite has been unlocked.

Step 6 (Optional): If the DVS Advanced Analysis Suite has been installed test the DVS Database Manager to check that the DVS Advanced

Analysis Suite has been unlocked and that the database can be accessed.

16.3 DVS Analysis Suite Installation Instructions (XP or earlier)

The DVS Analysis Suites can be installed from the installer disk by performing the following steps:

Step 1: Double click on 'Install.xls' if auto-run failed to start.
With Excel 97 or higher you may be prompted with a message similar to: "This workbook you are opening contains macros. Some macros may contain viruses that could be harmful to your computer". If this occurs click on 'Enable Macros' and continue with the installation at Step 2.

Step 2: Click on 'Install DVS Analysis Suite'.

With both the DVS Standard and Advanced Suite you will be asked to accept the licence agreement.

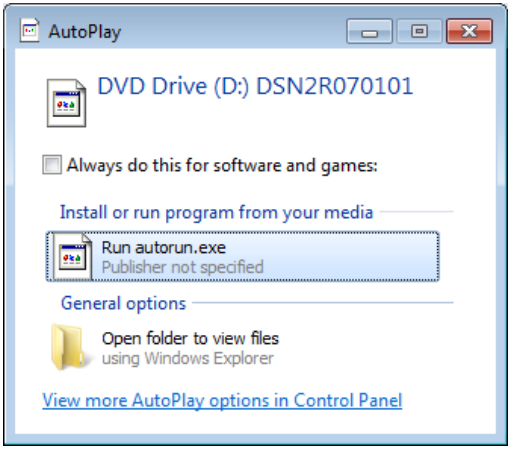
Step 3: When the installer informs you that "'Add-in name' was installed successfully" click on 'OK' to complete the installation.

Step 4: With both the DVS Standard and Advanced Suite you must send to SMS the Software Product Codes which will enable SMS to issue Software Release Codes. The Software Product Codes are obtained from the "Info" button. This is also where the Software Release codes should be used.

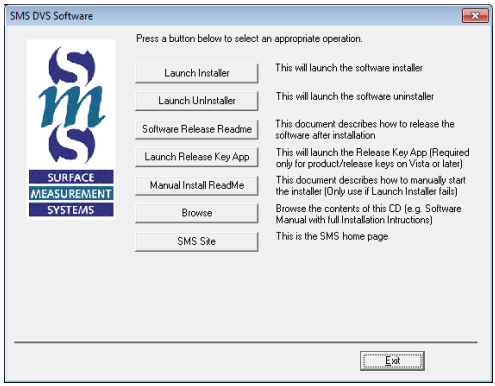
16.4 DVS Analysis Suite Installation Instructions (Win 7 or later)

The DVS Analysis Suites can be installed from the installer disk by performing the following steps (some steps are not offered during a re-installation):

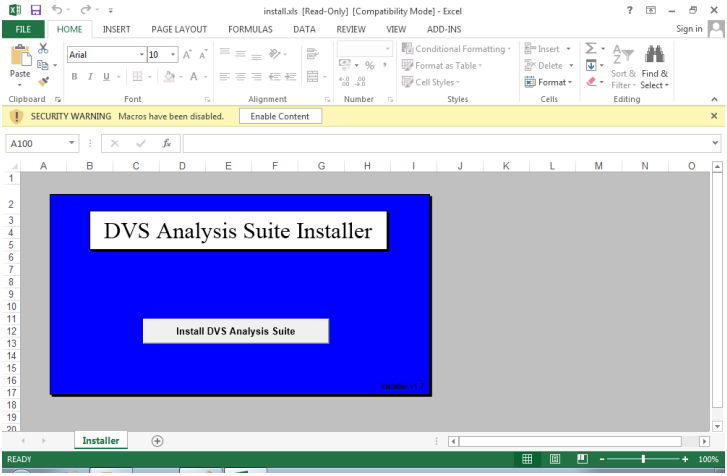
Step 1: After DVS Analysis CD is placed in CD drive click Run autorun.exe in the following dialog.



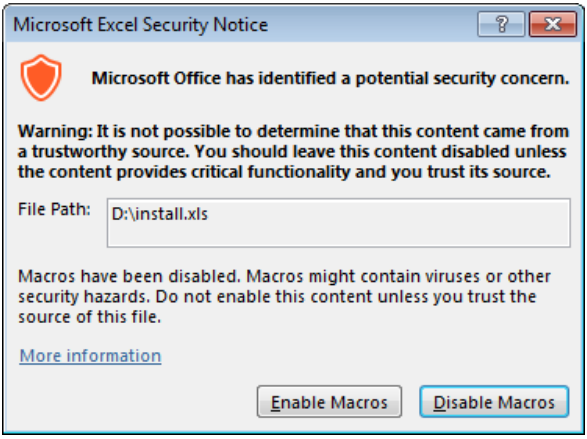
Step 2: Click Launch Installer in the following dialog.



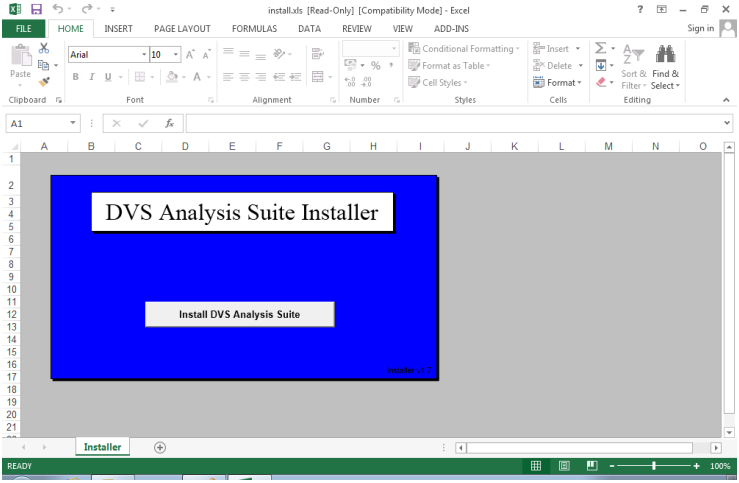
Step 3: When Excel is launched with Install.xls click Enable Content.



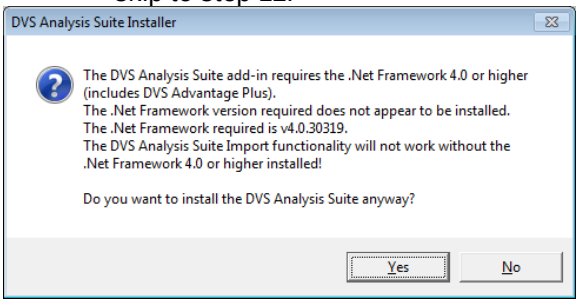
Step 4: The following dialog may be presented instead of the previous one and if this is the case then Enable Macros.



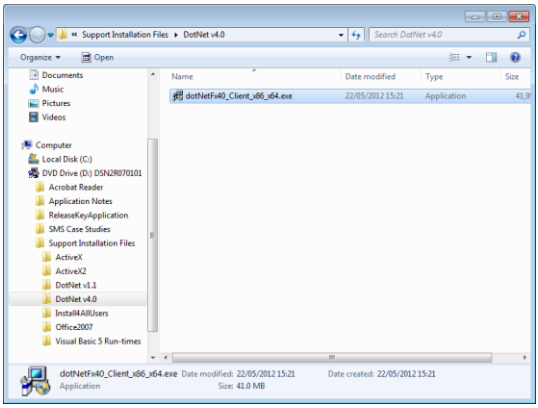
Step 5: Click Install DVS Analysis Suite.



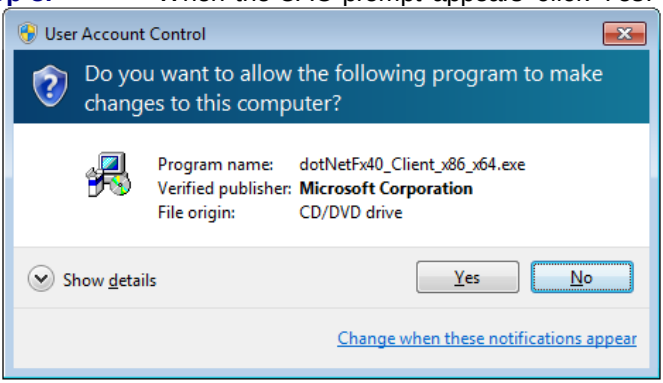
Step 6: If a dialog is presented stating that the .Net Framework is not installed Click No otherwise skip to step 12.



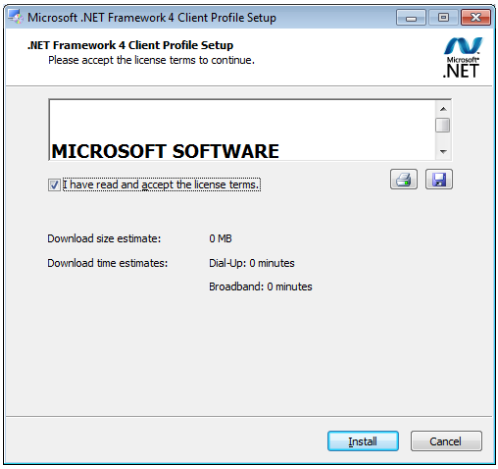
Step 7: Find dotNetFx40_Client_x86_x64.exe on the CD and double click to run (or DotNetFX.exe if .Net Framework 1.1 in dialog).



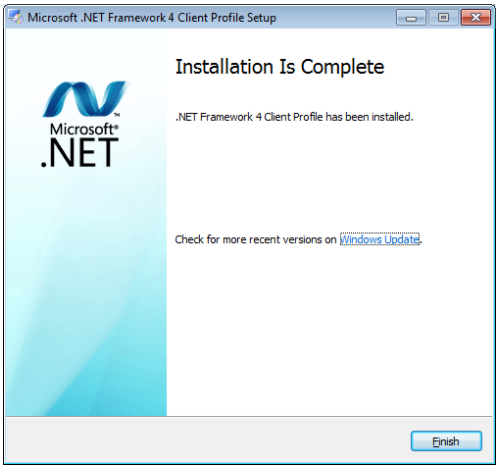
Step 8: When the UAC prompt appears click Yes.



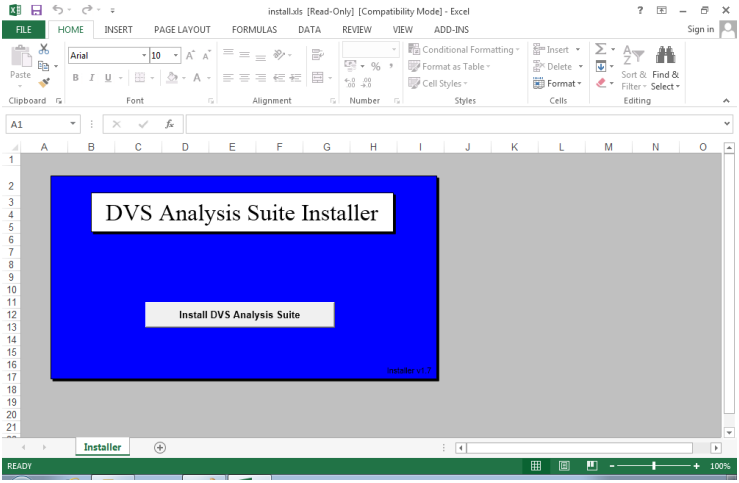
Step 9: Check license terms and click Install to Install the .Net Framework.



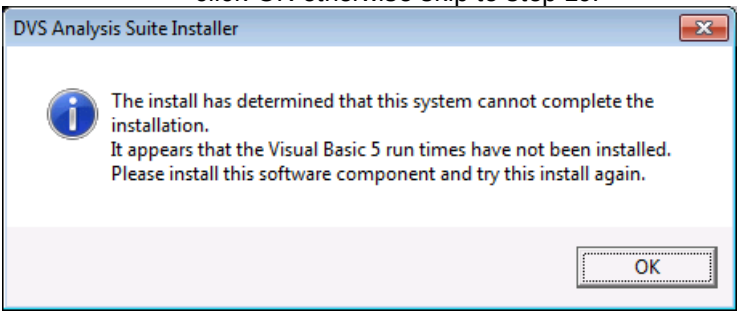
Step 10: When .Net Framework installation is complete click Finish.



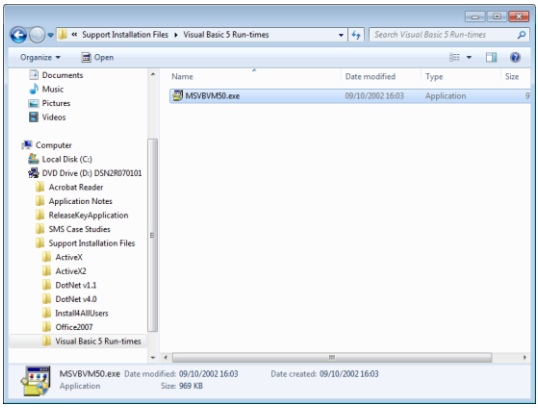
Step 11: Return to Excel and Click Install DVS Analysis Suite.



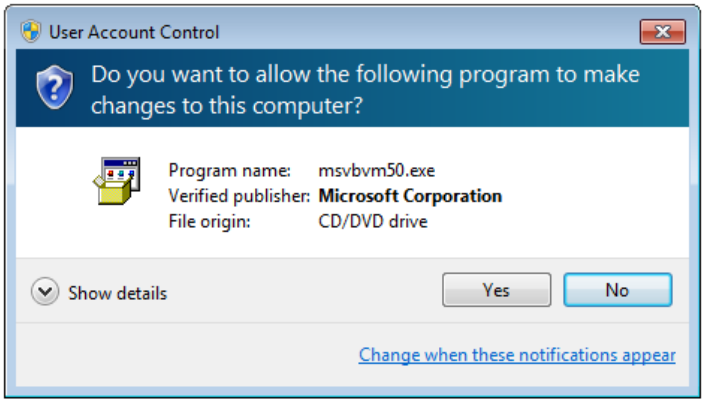
Step 12: If a message is displayed stating that the Visual Basic 5 Run times are not installed click OK otherwise skip to step 19.



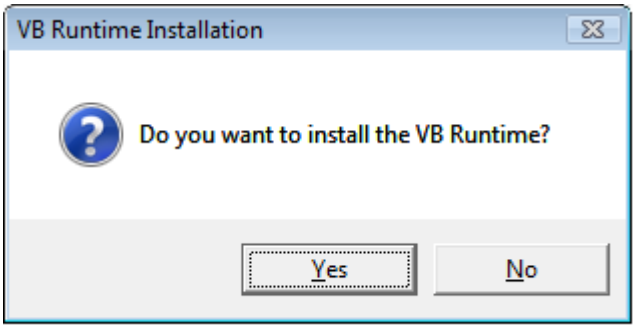
Step 13: Find MSVBVM50.exe on the CD and double click to run.



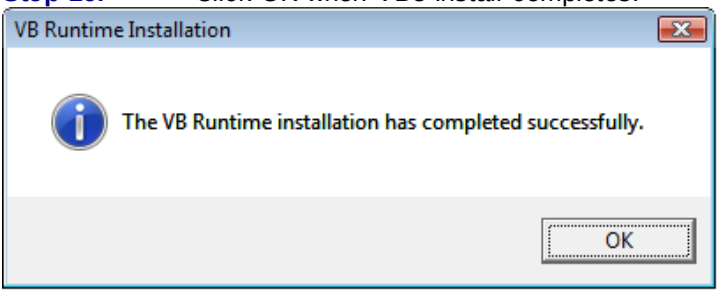
Step 14: When the UAC prompt appears click Yes.



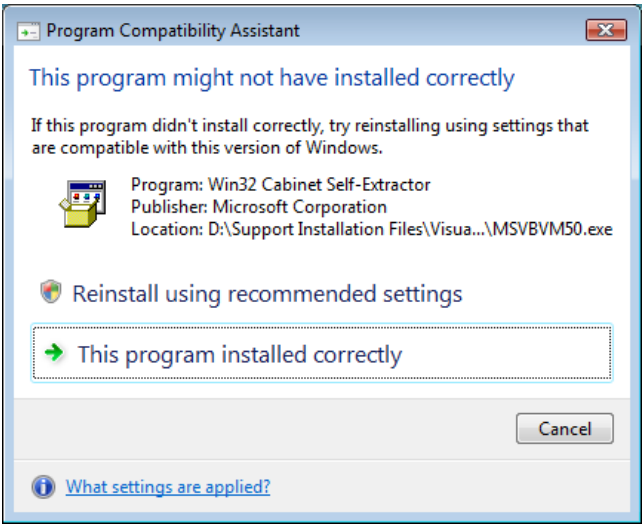
Step 15: Click Yes to install the VB5 Run times.



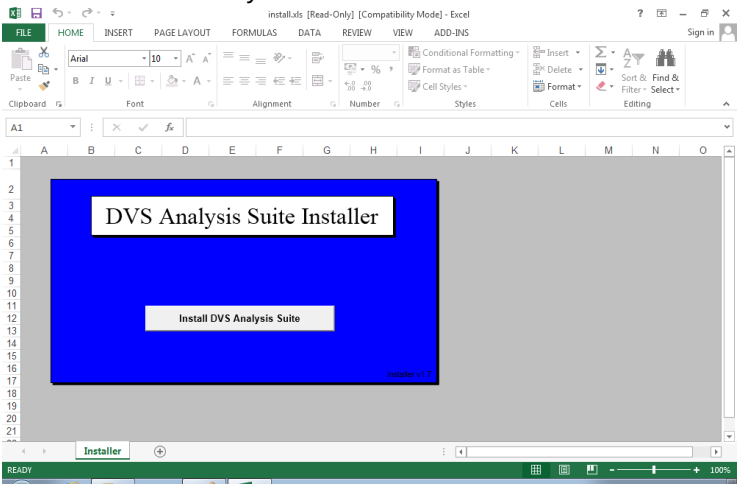
Step 16: Click OK when VB5 Install completes.



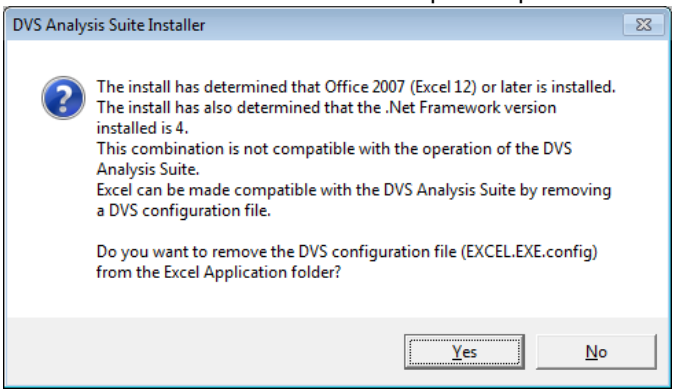
Step 17: Click This program installed correctly when the Program Compatibility Assistant is displayed.



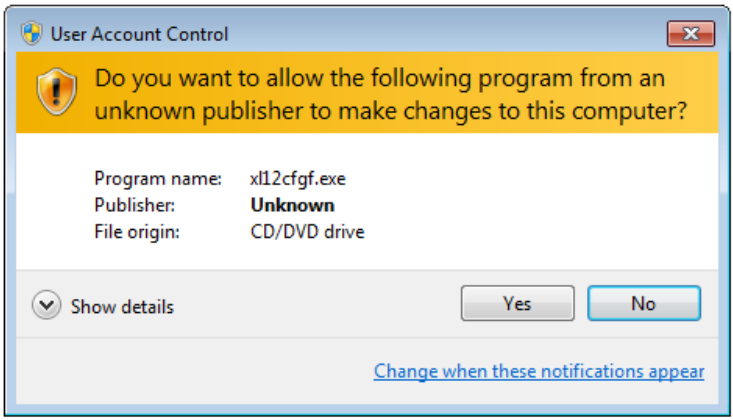
Step 18: Return to Excel and Click Install DVS Analysis Suite.



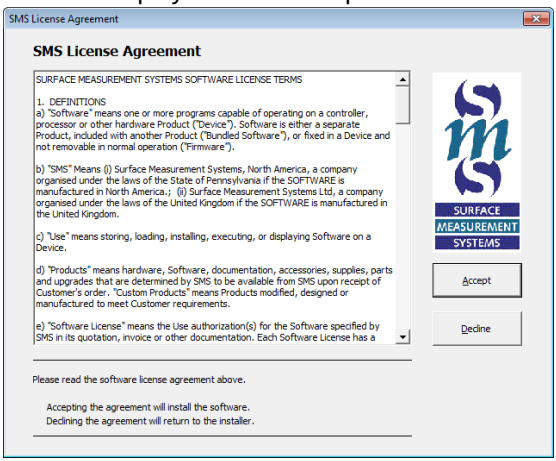
Step 19: If a DVS Analysis Installer dialog is displayed stating that Office 2007 (Office 12) or later requires an SMS EXCEL.EXE.config file to be deleted (or installed if .Net v1.1 is being used) click Yes otherwise skip to step 21.



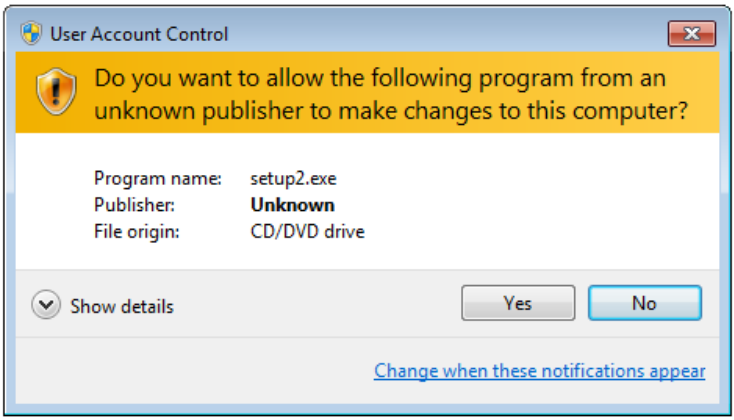
Step 20: When the UAC prompt appears click Yes.



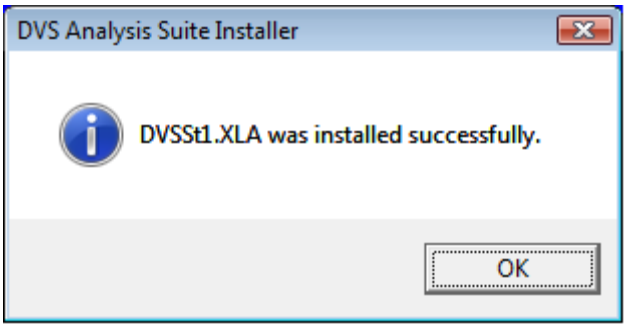
Step 21: When the SMS License agreement dialog is displayed click Accept.



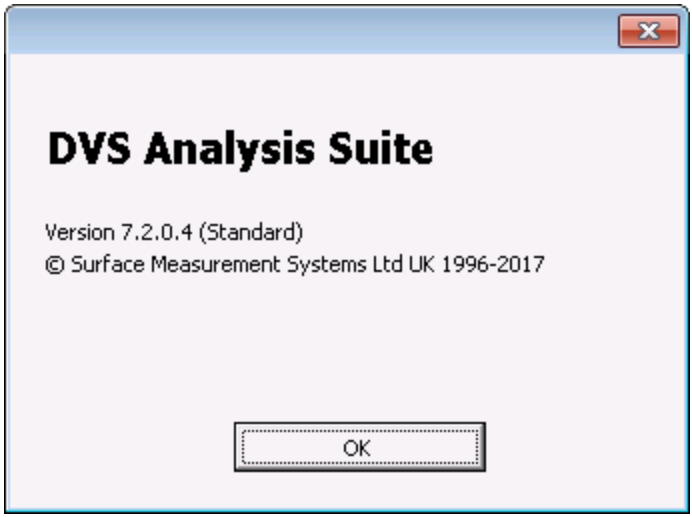
Step 22: If a UAC prompt appears click Yes.



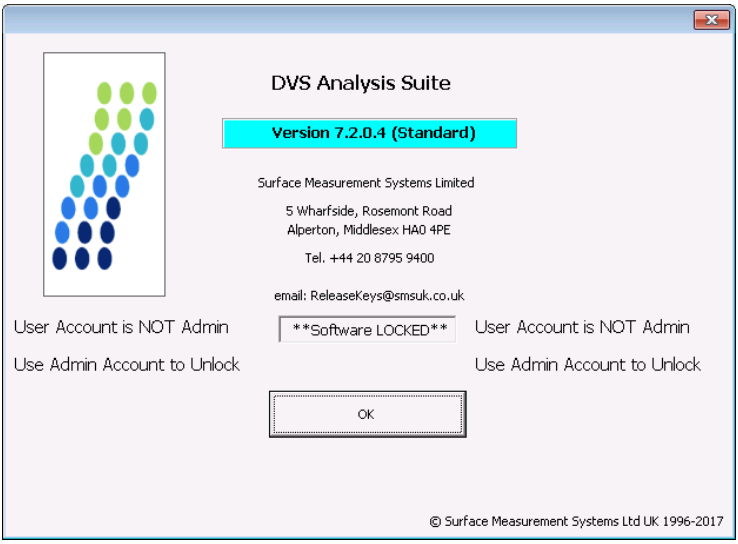
Step 23: When DVS Analysis Installer states that installation was successful click OK.



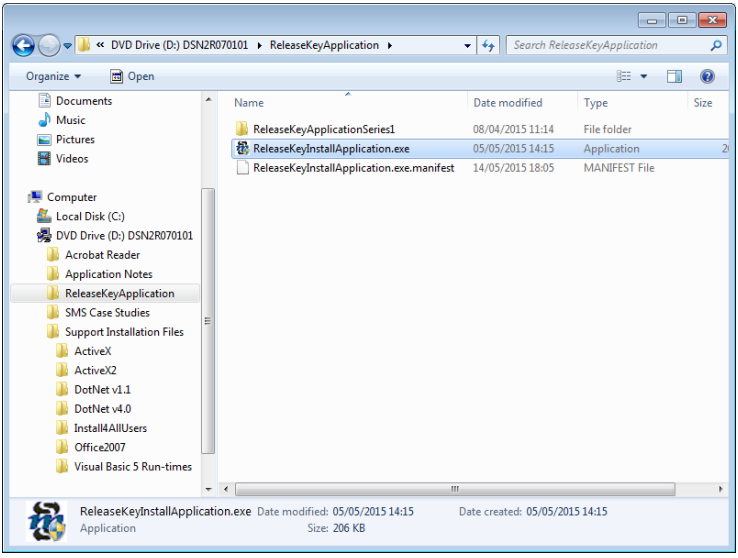
Step 24: Open Excel, click DVS Info button then click OK.



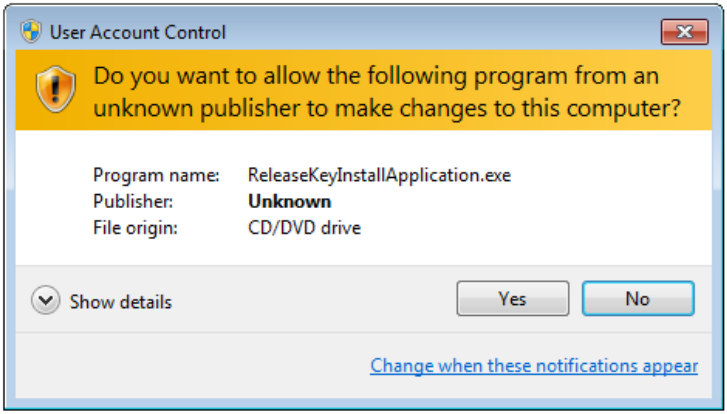
Step 25 If the software is locked click OK otherwise skip all further steps.



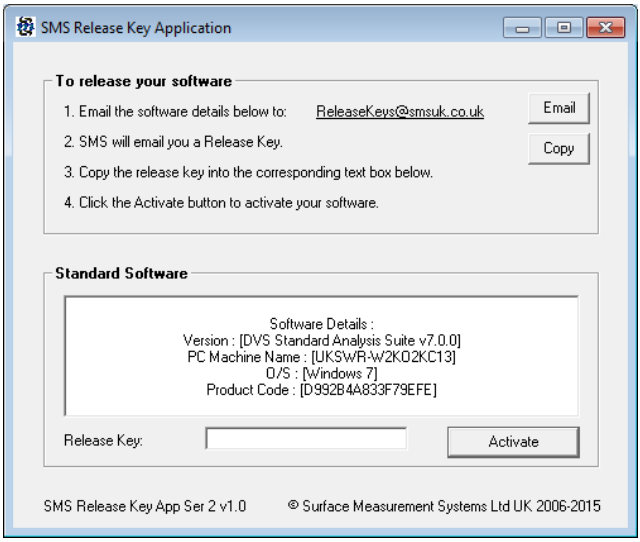
Step 26: Find ReleaseKeyInstallApplication.exe on the CD and double click to run.



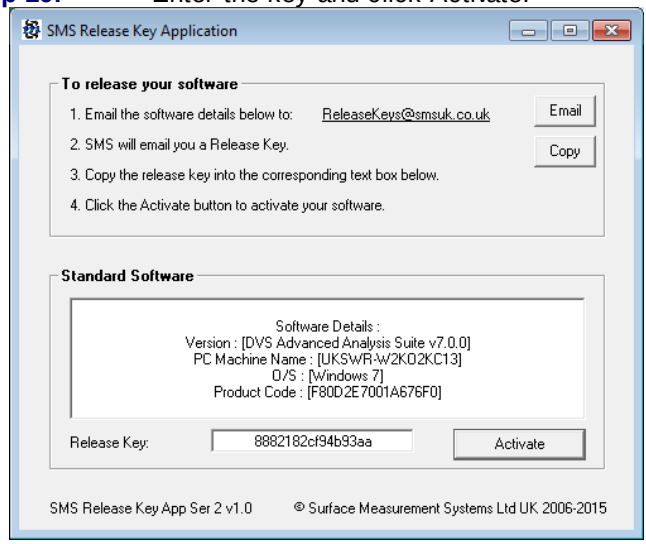
Step 27: When the UAC prompt appears click Allow.



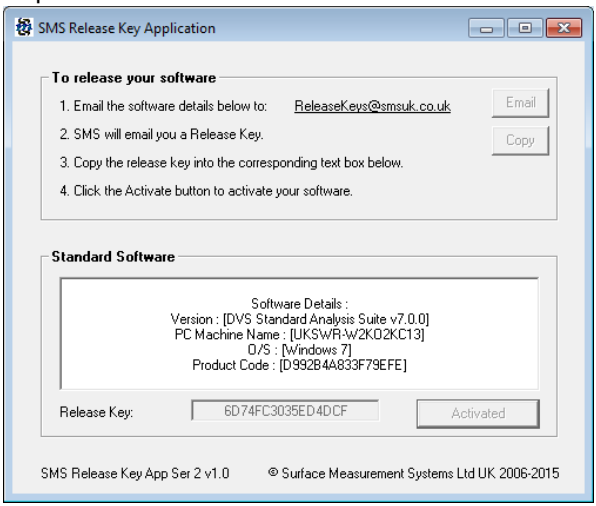
Step 28: When the ReleaseKeyInstallApplication launches send the Software Details to SMS. (Although the panel quotes v6.0.0 the one on the CD is valid for any v7).



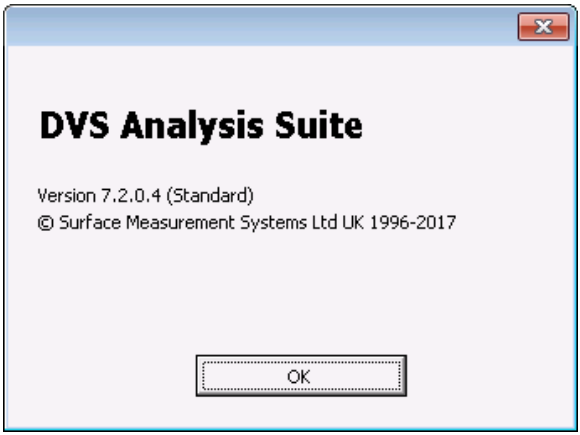
Step 29: Enter the key and click Activate.



Step 30: Close the ReleaseKeyInstallApplication panel then re-open it.

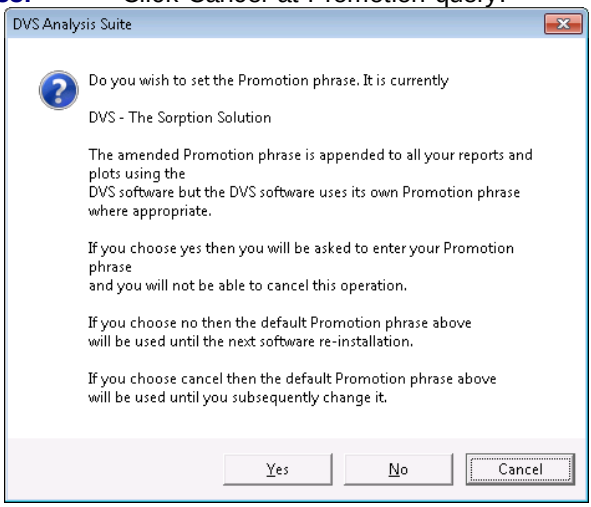


Step 31: Open Excel, click DVS Std Info button then click OK.



Step 32: Click Cancel at Copyright query.

Step 33: Click Cancel at Promotion query.



16.5 Important note for Excel 2007 or higher

When installing the **DVS Analysis Suite** on Excel 2007 or higher (and where the latest DVS support s/w is NOT available on the CD) there is a possibility that more than one DotNet Runtime will be installed.

In this case, when Excel is launched it will access the highest DotNet Runtime it can find and prevent access to all other lower versions. This means that DotNet 1.1 would not be loaded and the DVS Analysis Suite would NOT be able to perform DVS data (.DAT) Imports (all other functionality would work).

This can be overcome by placing a **config** file in the **Excel Application Sub-Folder** - when the Analysis Suite installer is launched and detects Excel 2007 it will offer to deploy the config file (EXCEL.EXE.config).

It is important to click **YES** to installing this config file to enable the Analysis Suite to import raw DVS data files!

However if the latest DVS support s/w is available on the CD AND the config file has been previously deployed the installer will prompt for this file to be deleted.

16.6 Important note about Excel File Formats

Excel 2003 or earlier uses the ".xls" file format which is very fast compared to the file formats of Excel 2007 or later.

The maximum number of rows of data that the ".xls" file format can handle is 65536 – 1 (65535 effective rows of data). In addition a small number of these rows are header rows.

It is highly recommended that if the 65535 limit is never broken that the ".xls" file format is used. This is especially true for the CFR version of the Analysis s/w where it can take roughly 10 minutes to save (or open) a file of this size. This is a current limitation of the s/w protecting files within Excel.

16.7 DVS Analysis Suite Installation Instructions for multiple Users (Win 7 or later)

Where multiple Windows Users will access the Analysis Suite an Install for all users is available in folder InstallForAllUsers within the Support Installation Files folder on the CD.

Double click the Setup.exe file and follow the instructions.

Notes:-

1) Apart from .Net v4.x any dependencies will be requested to be installed if they are not already installed
2) The Release Key is still a separate utility
3) If the distribution is copied from the CD the relative location of Setup.exe to the folders on the CD must be retained
4) If Setup.exe is moved from the CD but the folder structure is not retained it will request the folder in which the s/w in available
5) There is NO multiple user uninstall
6) Currently the feature is only available for non-CFR s/w

17 Appendix B – System Requirements

17.1 System Requirements

Hardware
Win 7/8/10: Dual Core 1.8 GHz or higher
Win 7/8/10: 2+ GB RAM

Software
Office (English) x32/x64: Office® 2010/13/16/19 (Not CFR).
Windows (English): Windows® 7/10.
.NET Framework v1.1 or 4.0 (latter used by default)
Internet Explorer v5.01 or later (depends on Windows version).

Note: There may be additional hardware or software requirements depending on the version of Windows or Office in use.

17.2 Tested Windows/Excel Combinations

This version of the DVS Analysis Suite has been tested using the following combinations of Windows (64 bit) and Excel:

MS Excel Version	MS Office Version	Win Version
Excel 2019 x32/x64	Office 2019 x32/x64	Win 10 Pro
Excel 2021 x32/x64	Office 2021 x32/x64	Win 10/11 Pro

Note: Although not listed in the table above this version should also be compatible with appropriate combinations of XP/Vista/7/8/10 and Office 2003/2007/2010/2013/2016/2019. While SMS no longer support the use of XP/Vista/Win 8 and Office 2003/2007 there are no known issues with using the analysis s/w on these obsolete Windows/Office combinations.

18 Appendix C – Customer Support

If you experience any problems with this product please contact your local supplier.

Contact SMS at the following locations –

United Kingdom
www.surfacemeasurementsystems.com
Surface Measurement Systems Limited
5 Wharfside, Rosemont Road
Alperton
Middlesex HA0 4PE
Phone: +44 020 8 795 9400
Fax: +44 020 8 795 9401
E-mail Support@SurfaceMeasurementSystems.com

USA and Canada
www.surfacemeasurementsystems.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@SurfaceMeasurementSystems.com