Agilent SimDis System

Reference Manual

Agilent Technologies

Notices

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Manual Part Number

G2887-90010

Edition

First edition, April 2005

Printed in USA

Agilent Technologies, Inc.
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Wilmington, DE 19808-1610 USA

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In This Manual

This manual describes the Agilent Technologies SimDis system, and explains how to install it, set it up, and use it to collect and analyze data.

1 Introduction

Introduces the SimDis system, the ASTM standards, and the overall process needed to set up the system for collecting data.

2 Installation

Describes the installation process for the system.

3 SimDis Data Analysis Tutorial

Explains how to complete setup of the system using the data you have collected from the sample runs, and how to analyze a reference / checkout sample.

4 Sample Preparation

Explains the samples for each ASTM standard and how to prepare them.

5 Running Samples

Explains how to run the various samples you have prepared in order to prepare the system for use, calibrate the ChemStation Method, and collect data required for SIMDIS analysis.

6 Automated SimDis Operation

Describes how to set up the ChemStation software and SimDis software to automatically perform SIMDIS analyses.

7 Software Reference

Provides quick reference information for the SimDis system software. Lists menu commands and other parameters, and briefly describes them.

8 Maintenance

Describes the tasks you must perform to operate and maintain the SimDis system hardware.

A Calibration Sample Compositions

Lists the compositions of selected commercially available calibration standards.

B HT PTV Inlet Operation

Describes the operating and configuration parameters for the HT PTV inlet.

Contents

1 Introduction

This chapter introduces the Agilent SimDis system (Simulated Distillation). It also describes the hardware and software requirements.

The Agilent SimDis System

The Agilent SimDis System enables the use of gas chromatography to determine the boiling point range distribution of petroleum fractions.

It is a comprehensive system of hardware and software that provides:

- An easy-to-use interface
- Data treatments
- Sample sequencing
- Error handling
- Reporting

The system requirements are listed later in this chapter.

NOTE See Chapter 2, "Installation" for installation instructions.

ASTM Test Methods

You can set up each gas chromatograph (GC) to be used as an Agilent SimDis System to perform one of three ASTM (American Society for Testing and Materials) test methods. Table 1 compares each method.

Table 1 SimDis System-supported ASTM test methods

		Applicable to items with these boiling points...		
ASTM test method	**Determines the boiling range distribution of...**	**Initial****	**Final****	**Notes**
D 2887	Petroleum products and fractions	NA	≤538 °C (1000 °F)	Limited to samples having a boiling range greater than 55 °C (100 °F) and having vapor pressure sufficiently low to permit sampling at ambient temperature.
D 2887x (proposed)*	Petroleum products and fractions	>100 °C (212 °F)	<615 °C (1139 °F)	
D 6352	Petroleum distillate fractions	>174 °C (345 °F)	<700 °C (1292 °F)	(C10 to C90)

*As of print time for this manual, D 2887x is not an ASTM test method. It is under consideration by an ASTM technical committee. Throughout this manual, it is referred to "D 2887x."

**At atmospheric pressure as measured by the test method.

NA Not applicable

This manual assumes the reader is already familiar with these test methods. For more information, refer to the ASTM documentation.

System Requirements

Some SimDis system requirements are general, applying to all SimDis installations. Others are specific to the ASTM test method that you use.

General requirements

These items are always required no matter what configuration you choose.

- Agilent 6890N GC, firmware revision N.05.05

NOTE Visit the Agilent Web site (www.agilent.com / chem) to update your firmware, if needed.

- Flame Ionization Detector (FID)
- G2613A or G2913A Injector
- Agilent G2070BA ChemStation software (revision B.01.01 or greater) or G2070AA (revision A.10.03)
- Agilent G2887AA SimDis software

NOTE

- A Mass Selective Detector (MSD) is not supported.
- The GC can have up to two inlets and two detectors. The Agilent SimDis System only uses one of each, an inlet (described later) and the FID.
- Each GC used for SimDis must have its own licensed copy of the SimDis software. GCs using ChemStation alone do not need the SimDis license.
- A valve box is not supported.

Method-specific hardware and consumables

After you select an ASTM test method and an inlet, you can determine the GC hardware and consumables you will use. The choices you make also depend on the method you develop. Table 2, Table 3, and Table 4 explain further.

Table 2 D 2887 hardware and consumables

	HT PTV[1] inlet	Split/Splitless inlet
Liner	5188-5313	5183-4711 (Deactivated), 5183-4647 (Deactivated), or 19251-60540 (Nondeactivated) **Note:** Place the glass wool in the liner so it is at the top.
O-ring	0900-0028	5180-4182 (12 / pkg)
Septum	5183-4759 or 5183-4757	5183-4759 or 5183-4757
Column	125-10HB (DB-1)[2] 10 m x 0.53-mm id, 2.65-µm film	125-10HB (DB-1)[2] 10 m x 0.53-mm id, 2.65-µm film
Ferrule	5188-3515 (320 ferrule) 5188-5314 (530 ferrule)	5080-8773
Column nut	5188-5312	5181-8830
Syringe (see note below)	Autosampler, 0.5 µL (5182-9651) or 5 µL, (5181-1273)	Autosampler, 0.5 µL (5182-9651) or 5 µL, (5181-1273)

[1]High Temperature Programmable Temperature Vaporizer (see Appendix B, "HT PTV Inlet Operation.")
[2]125-2814, 10 m x 0.53-mm id, 3.00-µm film is an acceptable alternative.

Table 3 D 2887x hardware and consumables

	HT PTV inlet
Liner	5188-5313
O-ring	0900-0028

Table 3 D 2887x hardware and consumables (continued)

	HT PTV inlet
Septum	5183-4759
Column	190957-021 (HP-1) 10 m x 0.53-mm id, 0.88-µm film 19095Z-020 (HP-1) 5 m x 0.53-mm id, 0.88-µm film 145-1001 (DB-HT SIMDIS) 5 m x 0.53-mm id, 0.15-µm film
Ferrule	5188-3515 (320 ferrule) 5188-5314 (530 ferrule)
Column nut	5188-5312
Syringe (see note below)	Autosampler, 0.5 µL (5182-9651) or 5 µL, (5181-1273)

Table 4 D 6352 hardware and consumables

	HT PTV inlet
Liner	5188-5313
O-ring	0900-0028
Septum	5183-4759 or 5183-4757
Column	145-1001 (DB-HT SIMDIS) 5 m x 0.53-mm id, 0.15-µm film
Ferrule	5188-3515 (320 ferrule) 5188-5314 (530 ferrule)
Column nut	5188-5312
Syringe (see note below)	Autosampler, 0.5 µL (5182-9651) or 5 µL, (5181-1273)

NOTE We recommend that you use the 5-µL syringe initially. Depending on their dilution, some samples may require that you inject smaller volumes to prevent overload problems. If so, switch to the 0.5-µL syringe (both syringes are provided).

Optional components

G2914A or G2614A Automatic Liquid Sampler Tray

Getting Started with the SimDis System

To get started with the SimDis system:

1. Select an ASTM test method. This choice impacts the selection and installation of column, ferrules, septa, liner, and other hardware as well as the ChemStation Method parameters.
2. Install the system as described in Chapter 2, “Installation.”
3. Prepare the samples needed for setup, method calibration, and checkout. See Chapter 4, “Sample Preparation.”
4. Create the appropriate GC method. See Chapter 5, “Running Samples.”
5. Run solvent blank / system cleanliness samples. See Chapter 5, “Running Samples.”
6. Run calibration samples and calibrate the method. See Chapter 5, “Running Samples.”
7. Run reference / checkout samples to check system performance. See Chapter 5, “Running Samples.”

After successfully running the reference / checkout samples, the system is ready for general use.

2 Installation

This chapter explains how to install and set up the hardware and software for an Agilent SimDis system.

To Install the SimDis System

To install the SimDis system:

1. Select the D 2887, D 2887x, or D 6352 ASTM test method. See Chapter 1, “Introduction” for descriptions of each test method.
2. If needed, install a 6890N GC. See the GC documentation for more information.
3. Install any GC accessories. See each accessory’s installation documentation.

   See the HT PTV Inlet Installation Guide (part number G2888-90030) to learn how to install the HT PTV inlet.
4. Install any GC hardware required for the selected test method. See the 6890N GC documentation and Chapter 1, “Introduction” for more information.
5. If needed, install Agilent G2070BA ChemStation software (revision B.01.01 or greater) or G2070AA (revision A.10.03), as described in its documentation.

CAUTION Be sure to install the SimDis software in a location where its users have read/write privileges.

6. Install the SimDis software. A link to the installation program appears on the automatic startup page of the SimDis Software CD-ROM (part number G2887-90020).

NOTE One license covers up to **four** GCs. You can purchase multiple licenses. Also, after you activate the license, you cannot reset the details (for example, the number of covered GCs) with subsequent installations.

7. Refer to the instructions enclosed in the SimDis package to activate each software license. You must activate the license(s) within 10 days of the installation.

Verification

After installation, ChemStation and SimDis are linked, enabling them to work together, collecting and analyzing data.

To verify that the link was established, check that a new menu (SimDis) appears on the ChemStation menu bar in Data Analysis view. Figure 1 shows the new menu.

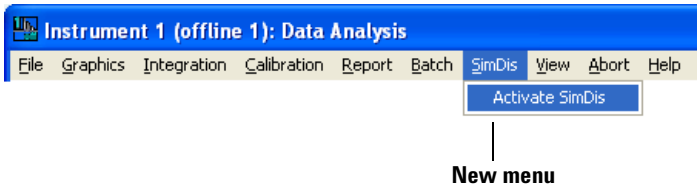


Figure 1 New ChemStation menu

Choose **Activate SimDis** from this menu to start the SimDis software from within ChemStation.

Multiple Users

If users with different Windows accounts will use the software:

- Use your Activation key to activate SimDis under each login.
- Make sure each user has read/write privileges in the ChemStation and SimDis program folders.

3
SimDis Data Analysis Tutorial

You may want to print this chapter to have it readily available while you follow described processes: the best way to follow the tutorial approach used here is to work simultaneously with both the SimDis application and with this chapter.

Overview and Initialization

In this chapter, you will walk through a tutorial process demonstrating typical use of the SimDis software application to manually analyze ChemStation data provided for ASTM Method D 2887.

Three types of files are typically expected to prepare SimDis for analysis of your samples: a solvent blank run, a calibration run, and a “checkout” sample run. It is this file set which will be used in the tutorial.

Initializing tutorial data file sets

Agilent provides a set of data files for use in this tutorial. If there is possibility this tutorial was done at a previous time, it is important to refresh folders and their contained files used in the exercises:

1 Using Windows Explorer, locate the home folder for the SimDis application, typically as —

C:\Program Files\Agilent SimDis

2 Within this folder, locate and *entirely delete* the sub-folder —

\TutorialDataFileSets

3 Insert your SimDis installation CD-ROM and entirely copy the folder found there, **< drive >\TutorialDataFileSets** , to the SimDis home folder.

4 Check **Properties** and **Attributes** of these copied folders and contained files: if they are ‘read-only’ (‘ R ‘), *uncheck* their respective **Read-only** checkboxes.

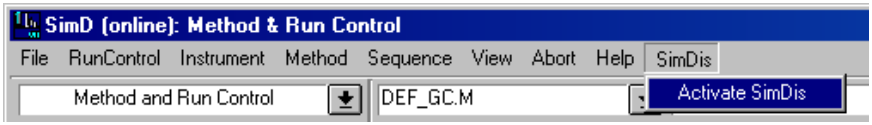
This process insures you will be working with a refreshed set of folders and files.

1. Starting SimDis

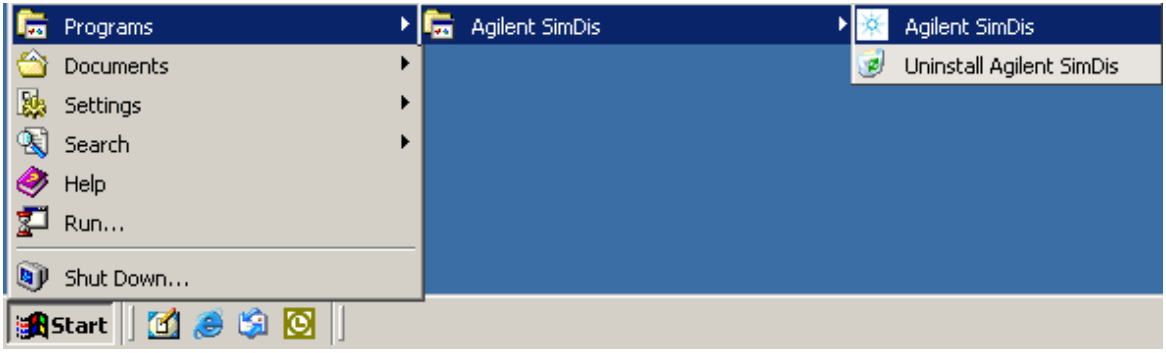
The SimDis application may be started in any one of three ways:

- By selecting its desktop icon:

- By selecting its ChemStation main menu item:



- Via ***Start***:



2. A Brief Orientation

From your viewpoint, consider SimDis to be primarily an interactive application whereby virtually all operations are accomplished through your selection of appropriate functions provided on various panels (windows). Wherever a choice affects displayed data, the result of that change is immediately seen allowing you to quickly fine-tune analysis parameters.

Further, graphical data presentations are also interactive with respect to both manipulating the presentation, and with respect to modifying analysis of the data itself.

Work flow is generally one of left-to-right tab selection, with main tabs being **Browse**, **Setup**, **SimDis**, **Report**, **Automation**, and **Licensing**. Each of these have additional tabs and/or other things (buttons, drop-down menus, entry fields, and so forth). We will define and use many as we move through the tutorial.

Items not used in this tutorial process are described in Chapter 7, “Software Reference”.

3. Main Menu Items

With virtually all functions handled on tab views, main menu items **File**, **Option**, and **Help** are intentionally limited in scope (Figure 2).

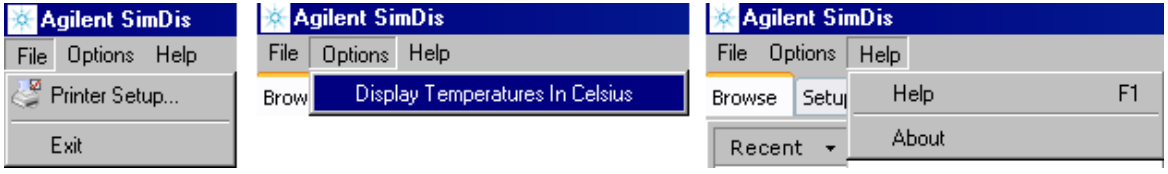


Figure 2 SimDis main menu items

- **File / Printer Setup** — opens your standard Print Setup panel allowing selection of a suitable printer and printer settings
- **File / Exit** — Closes the SimDis application

- **Options / Display Temperatures In Celsius** – switches from Fahrenheit to Celsius
- **Help / Help** – opens access to this document
- **Help / About** – provides current SimDis version and other information. Mouse-click the panel to close it, or use **Close** in its upper-right corner.

4. The Browse Tab

Assuming the application is properly licensed (see Chapter 2, “Installation”), the application opens in **Browse** mode (left-most **Browse** tab highlighted):

Browse provides primary means to search for and to view contents within ChemStation data folders (Figure 3).

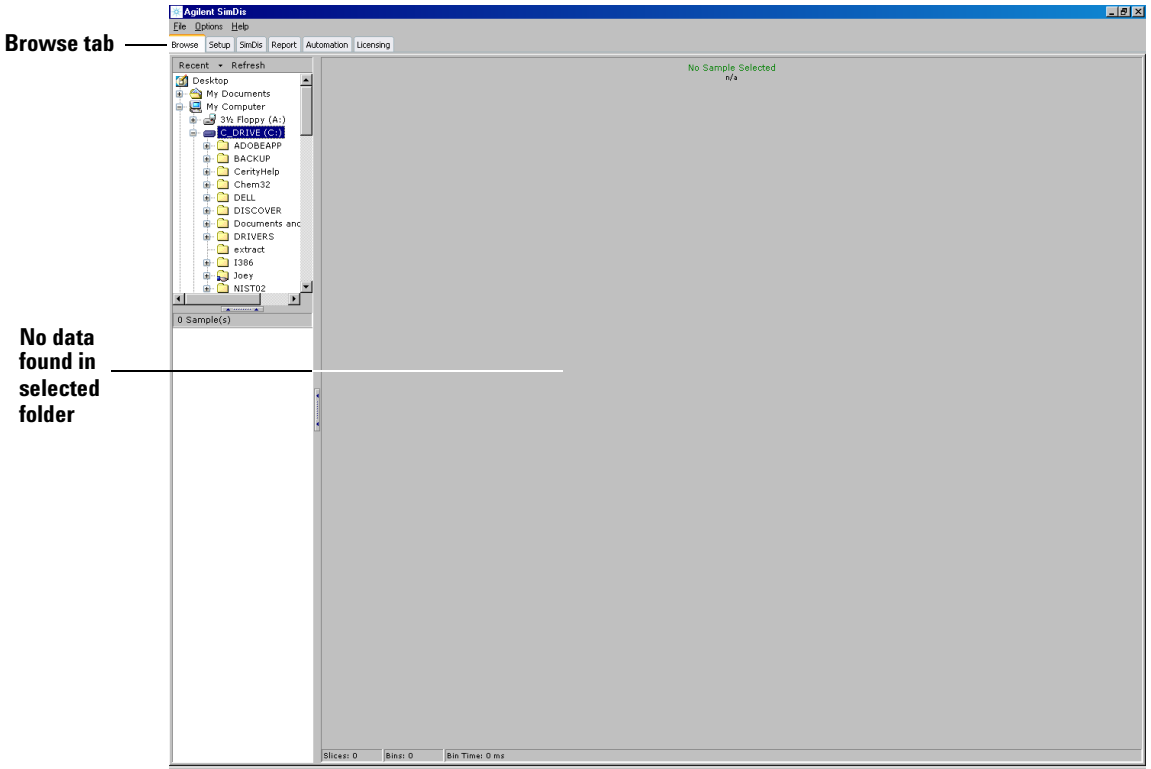


Figure 3 The **Browse** tab and listed ChemStation data folders

The data file viewed and its parent folder are selected for use on both **SimDis** and **Report** tabs to be described later.

Moving boundaries

Two aids are provided in moving boundaries between different parts of the view:

Appears in moving the mouse over a boundary. The boundary may then be dragged to a new location.

Located on boundaries. If selected, the boundary immediately hides the affected part of the view, or, selected again, restores the previous view.

Viewing and selecting files for analysis

The first step in the process requires selecting specific files to be analyzed. The **Browse** tab serves two purposes:

- It is the means by which you search for and view existing ChemStation data folders
- As we shall see, when a given data folder is selected and viewed, unless another is selected later in the process, this selection is carried forward into subsequent steps

To select a data folder:

1 Browse to the folder containing tutorial data files for Method D 2887. Typically, this will be:

C:\Program Files\Agilent SimDis\TutorialDataFileSets\D2887

2 Upon locating the folder, three ChemStation data folders appear in the lower half of the browsing portion of the view with the first listed data folder (the tutorial calibration sample file, **Calibration.D** in this case) is automatically opened such that its chromatogram is now displayed.

3 In turn, select each of the other two data folders to view their respective contents: the “checkout” sample (**RefGasOil.D**), and the solvent blank run (**SolventBlank.D**).

Note the two items, **Recent** and **Refresh**, located just above the folders list:

- **Recent** – as a convenience, lists recently selected data folders
- **Refresh** – updates the folders list such that it truly reflects your data folders source

Following are the three D 2887 data folders to be selected, each with its associated display (Figure 4, Figure 5, and Figure 6).

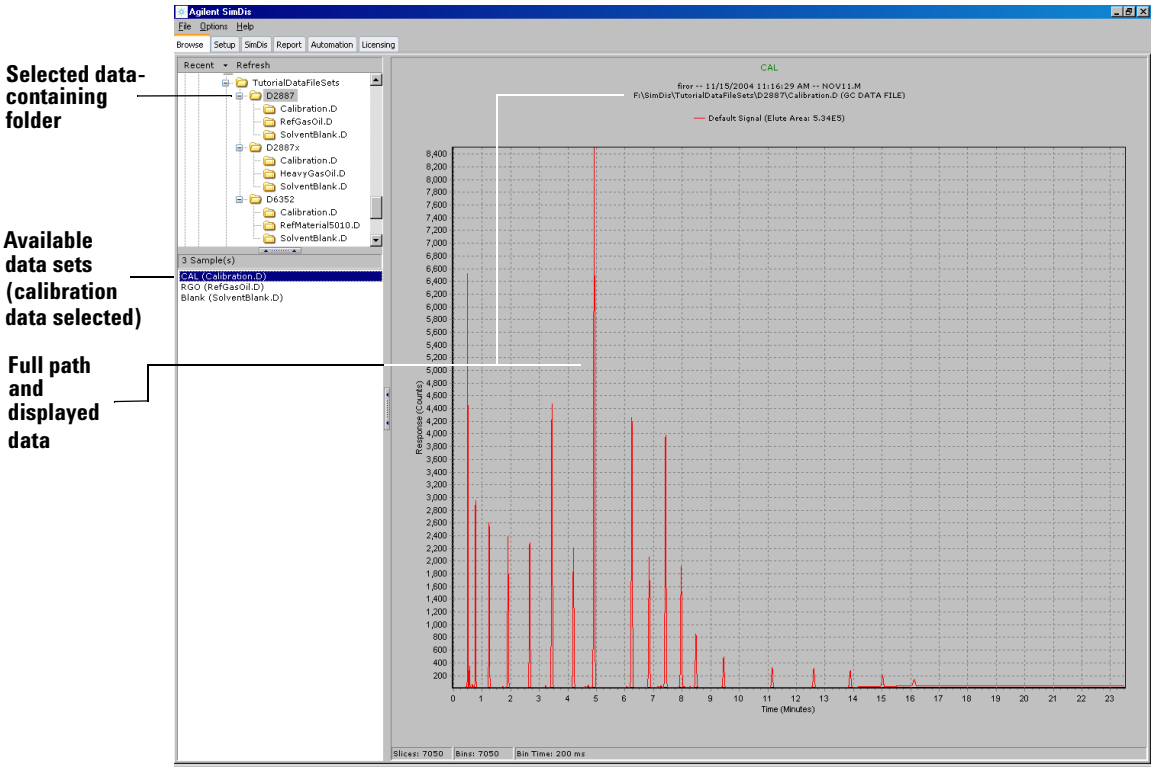


Figure 4 Data folder for D 2887 '**Calibration.D**'

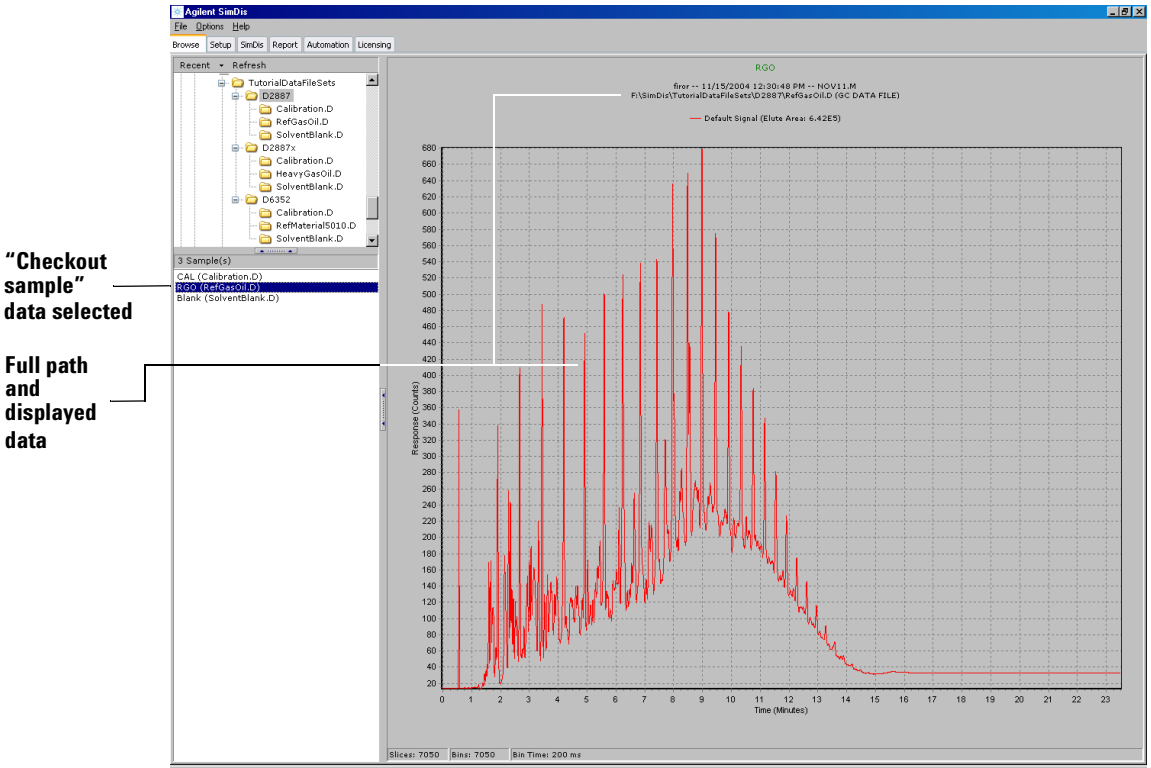


Figure 5 Data folder for D 2887 '**RefGasOil.D**'

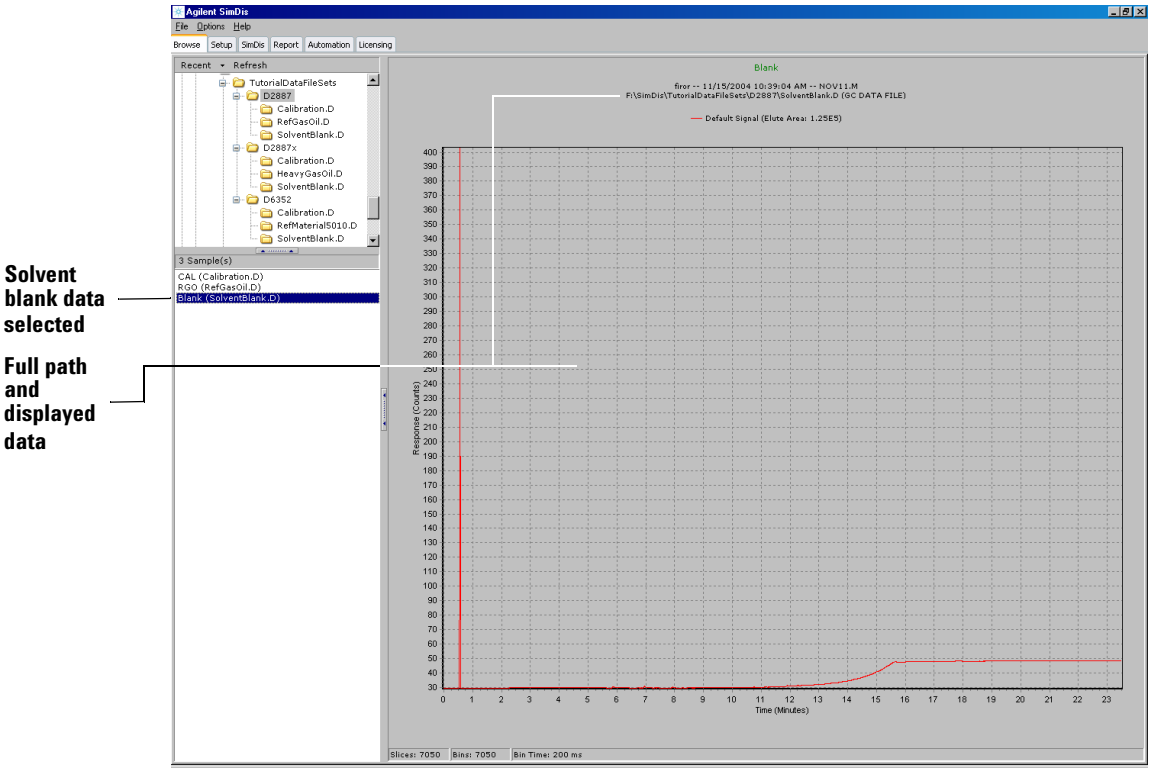
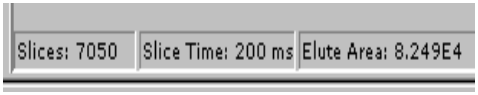


Figure 6 Data folder for D 2887 '**SolventBlank.D**'

In each case, check header and legend information located above the chromatographic display:

- Header information includes the full path to the selected data folder. Always use this as verification that you have indeed selected your intended ChemStation data folder
- The legend shows the data plotted in the associated color. This will be more important in later steps where two or more plots may be overlayed.

- Also note information about the displayed data at the lower-left corner of the graphical display area:



  - **Slices:** total number of area slices found in this data
  - **Slice Time:** time-width (in milliseconds) of each area slice
  - **Elute Area:** total area slice data accumulated below the signal curve

Before continuing, make sure that **RefGasOil.D** is selected.

Manipulating the chromatographic display

The displayed chromatographic plot is, by default, scaled to the tallest component peak (Response axis), and to the full original run time for the analysis. In many situations, you will want to move the plotted signal within the display area, and/or to inspect a portion of the displayed chromatographic signal in far greater detail than can be seen in the default view.

Interactive control is provided for both needs. In fact, these features are available for any SimDis graphical display.

To move the displayed chromatogram within the display area

1. Locate your mouse cursor anywhere within the display area.
2. Hold down your right mouse button and drag the display as desired.
3. Release the mouse button to lock the plot in its new location.

This process can be repeated as desired to further move what is displayed.

To zoom in (magnify) a selected chromatogram portion

Magnify a desired portion of the chromatographic display by creating a “box” around the portion of interest:

1 Place your mouse cursor at what will become the left side of a box created around your desired chromatographic portion of the display.

2 Hold down the left mouse button to set the starting point.

3 While continuing to hold down the left mouse button, drag the mouse cursor **right**, and up or down as necessary, to box your portion of interest (Figure 7).

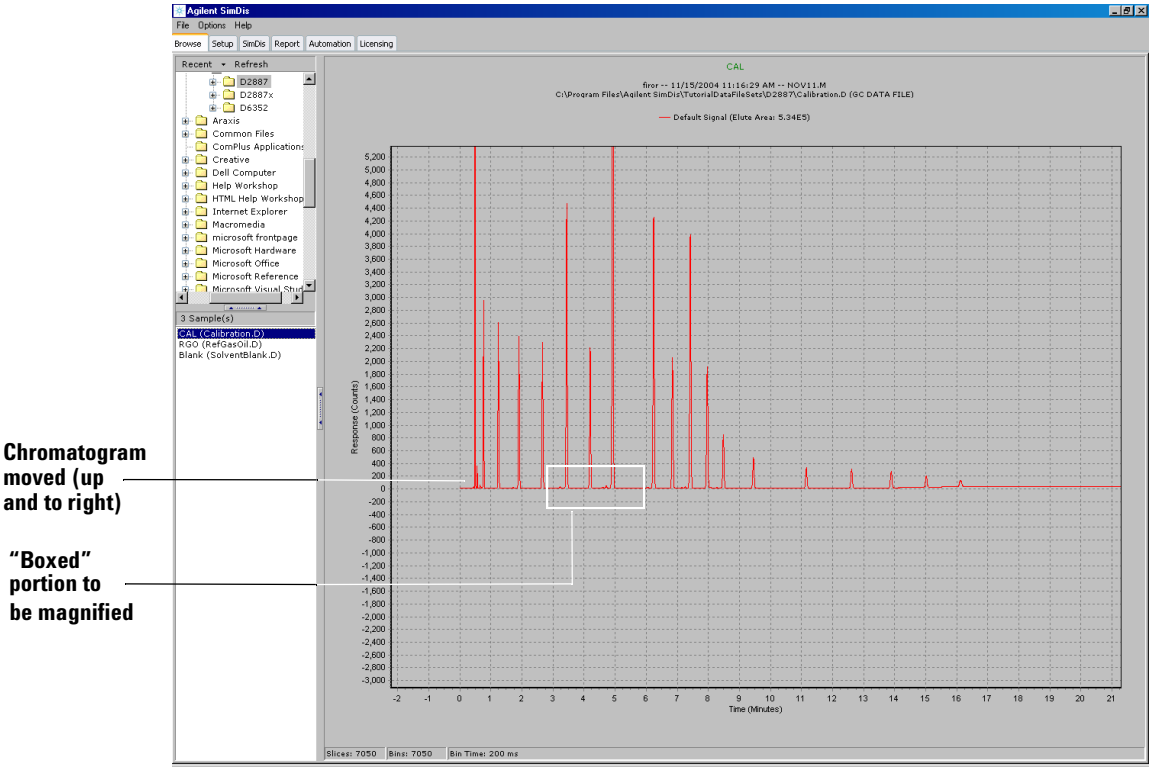


Figure 7 Displayed chromatogram moved and a portion “boxed” for magnification (“zoom in”)

4 Upon releasing the mouse button, the display immediately refreshes showing the now-zoomed portion (Figure 8).

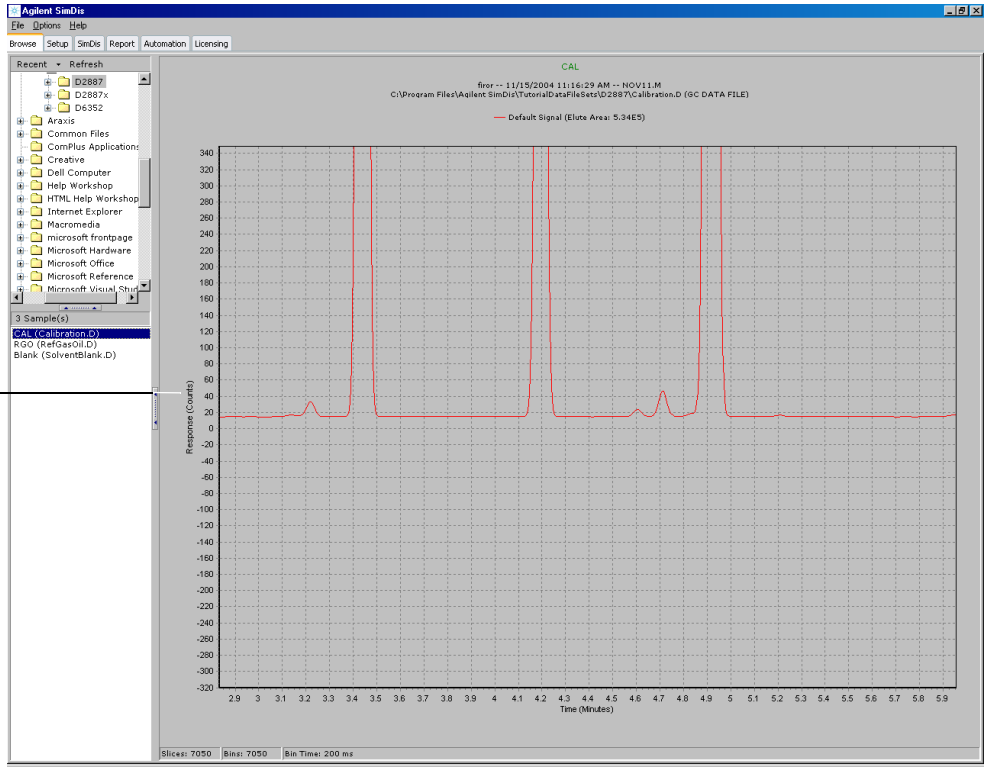


Figure 8 "Zoomed" (magnified) portion of the chromatogram

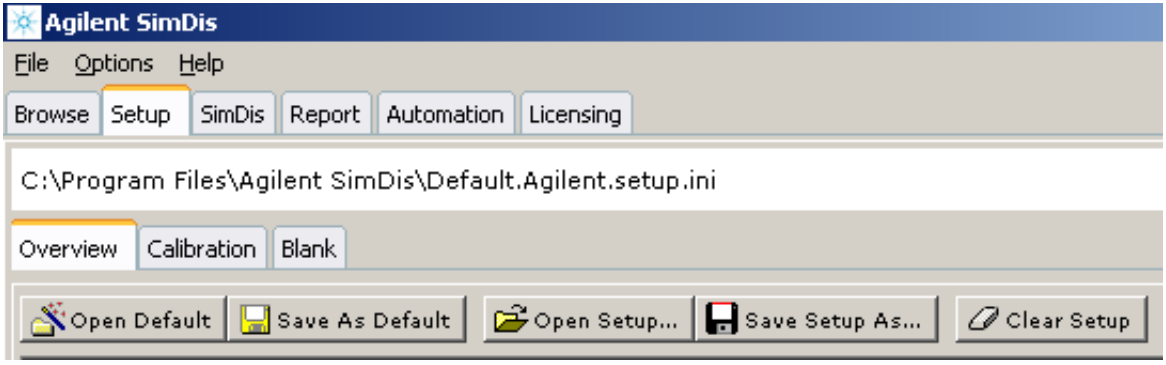
This process can be repeated as desired to further magnify any part of what is displayed.

To return to the original default view at any time

Hold down the mouse's left button anywhere within the graphical display area and drag the mouse cursor **left** and up or down to form a "box". Upon releasing the mouse button, the display immediately reverts to its original default position and scaling.

5. The Setup Tab

Having selected our three files to work with, we can now move to the next tab, **Setup**, which provides functions applied to both calibration and solvent blank data. The purpose is to optimize these data sources for subsequent use in analyzing both the "checkout" sample and, later, your own laboratory samples:



At this time, we will defer discussion of the path statement leading to an .ini file and five associated buttons, **Open Default** , **Save as Default**, **Open Setup . . .** , **Save Setup As . . .** , and **Clear Setup** to "Preserving your Setup tab settings" on page 57.

The panel includes three subtabs — **Overview** (the default opening view), **Calibration**, and **Blank** , and also a graphical display area (Figure 9).

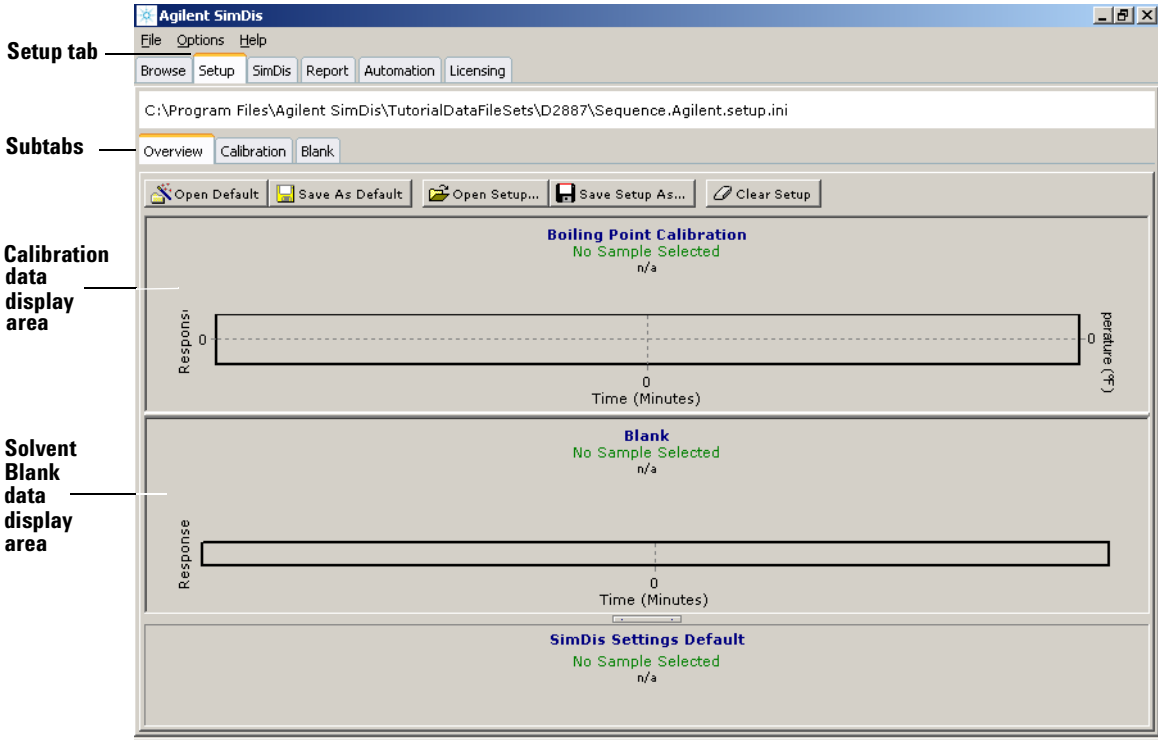


Figure 9 **Setup** / **Overview** panel

At this point, with respect to Figure 9, no Calibration or solvent Blank files have been selected. Selection is performed via Calibration and Blank subtabs described next and is based upon information derived from ChemStation report files produced and saved when chosen samples were originally run (more on this in Chapter 5, “Running Samples”).

The Calibration tab

Select the **Calibration** subtab. This provides three new subtabs, **Display** (the opening view), **Select**, and **Edit**:

- **Display —** allows display-only capability
- **Select —** provides means to select a data folder without having to return to **Browse**. Selecting ‘**(no calibration)**’ from the displayed list removes any previously-selected calibration.
- **Edit —** provides tools to alter interpretation of the calibration data

Use **Select** to choose the desired calibration data file (in this case, **Calibration.D**, typically found in **C:\Program Files\Agilent SimDis\TutorialDataFileSets\D2887\Calibration.D**).

This should result in a detailed graphical display of your selected calibration data (Figure 10).

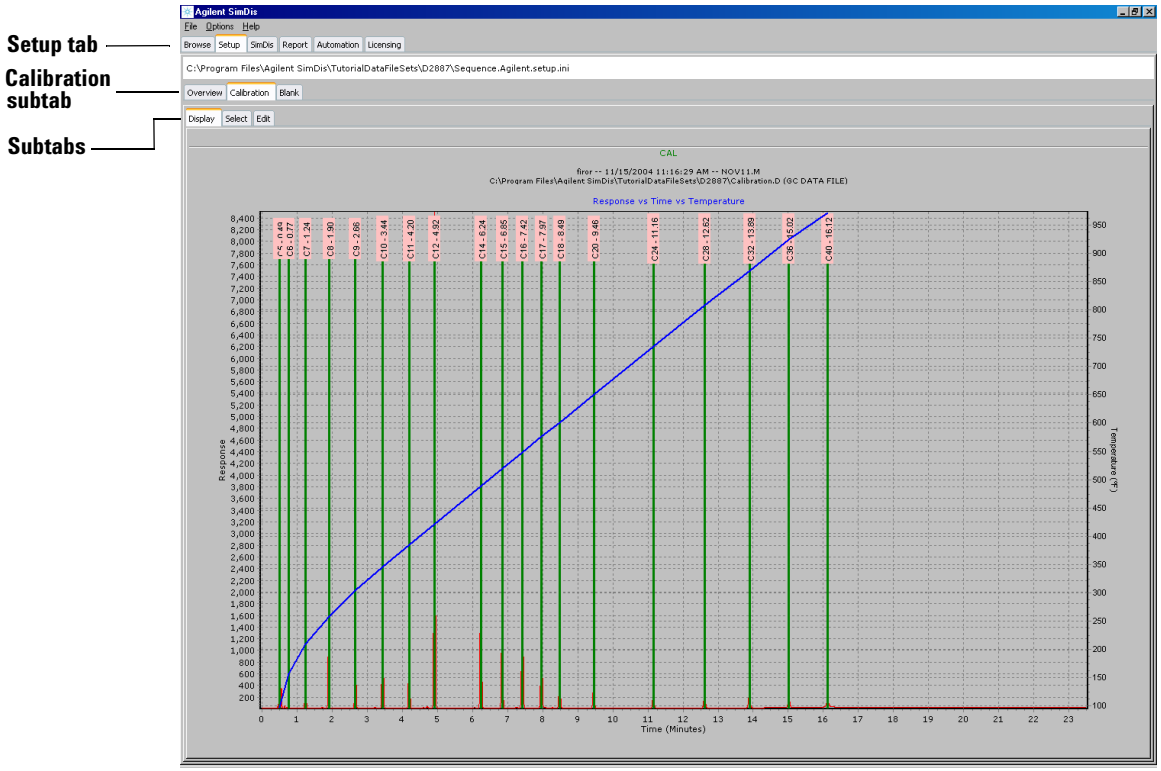


Figure 10 **Calibration** / **Display** panel

Verify the selected calibration file

Verify it is indeed the correct selection by checking its header path information shown in the displayed chromatogram.

In your own work, should this *not* be the intended choice, you must return to the **Browse** tab and select the correct folder.

We will now spend time using various available editing tools and features. It is recommended you try these manipulations yourself to become comfortable with them and to see their effects.

The Calibration / Edit tab

Various manipulations will be discussed in related groups of tools. Understand that nothing you do here affects original data generated by the ChemStation: you can always reselect the original data folder and start over.

Verifying the correct chromatogram

As a matter of habit, inspect header information above the display area to verify you have the correct, desired ChemStation data. If not, use the **Select** tab to search for and to select the correct data.

Changing peak-specific information

Note peak labels located at the top of each identified calibration peak (Figure 11). Each label contains both a carbon number assignment and the component's measured retention time. As an exercise, let us change the carbon number assigned to a chosen component peak:

1. Locate your mouse cursor at the lower right corner of the label associated with C24 eluting at 10.68 minutes.
2. Right mouse-click the label to obtain a peak-specific menu associated with the C24 peak.

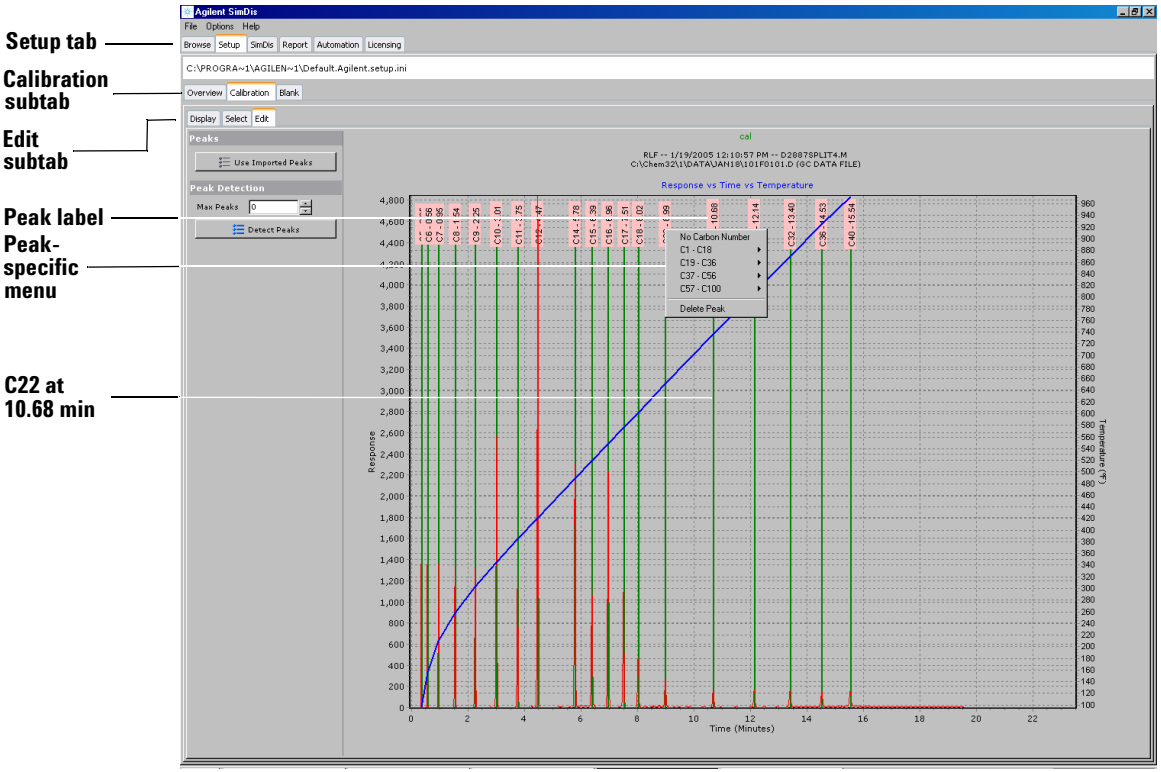
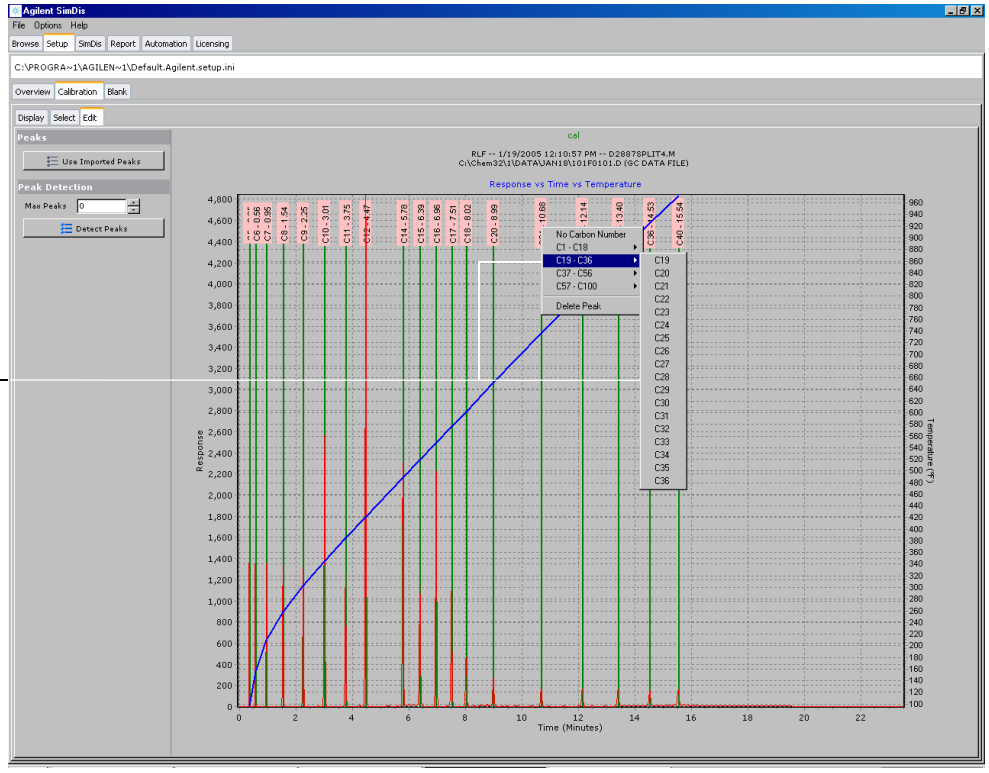


Figure 11 **Calibration** / **Edit** panel view with peak-specific menu opened (right mouse-click on peak label)

3 Next select **C19-C36** from the menu (Figure 12). A submenu list of carbon number choices appears. From this list, select **C22**, thereby changing the association for this component from its original “C24” to “C22”.



Carbon number selection

Figure 12 Reassigning the specific peak's carbon number

4 As the change is implemented, note the immediate impact upon the boiling point fraction distribution curve (Figure 13) it no longer exhibits the expected, typical shape originally observed.

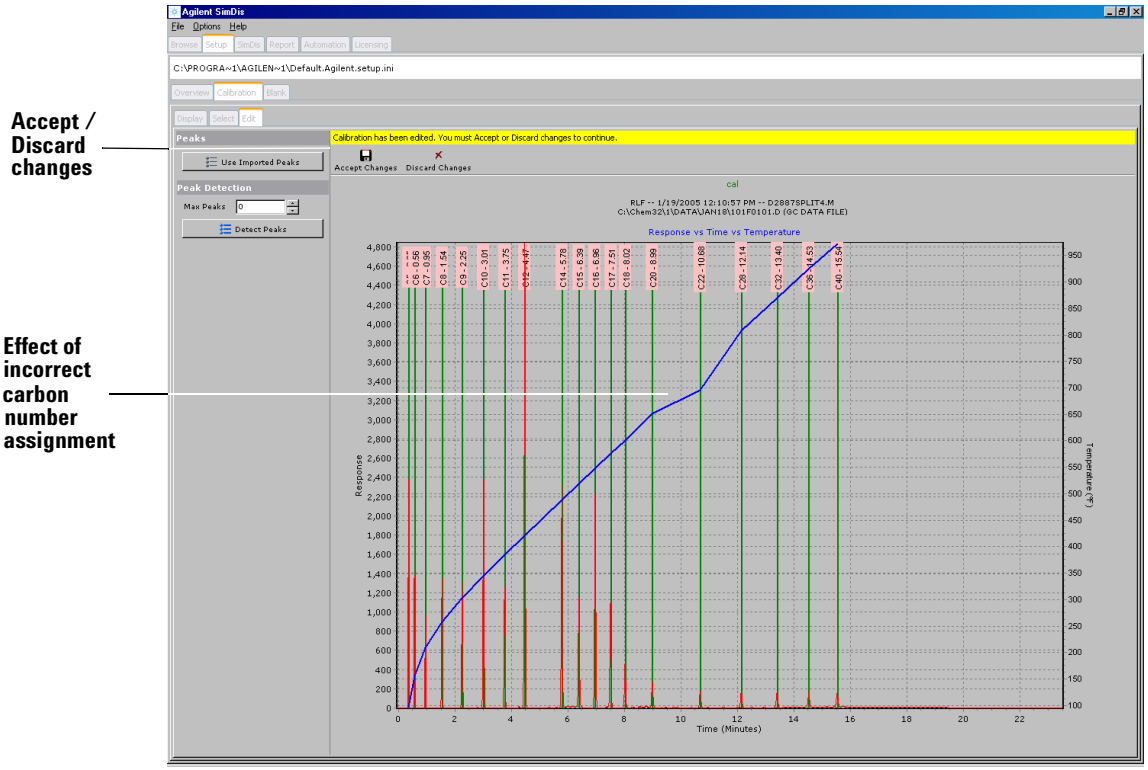


Figure 13 Effect of misassigned carbon number

5 Also note appearance of the highlighted text line "**Calibration has been edited. You must Accept or Discard changes to continue.**" along with two associated options, **Accept Changes** and **Discard Changes**:

- You can select **Discard Changes** to immediately restore the misassigned component peak back to its original carbon number, OR, perhaps better for purposes of this exercise,
- **Accept Changes**, then repeat the exercise steps to reassign the peak to its original carbon number, C24.

As a final thought for this exercise, recognize the immediate visual feedback you receive in making this change with respect to how the calibration data is affected. In general, immediate visual graphical responses to changes you make serve to quickly optimize how SimDis interprets your data.

Adding a peak

Next, we will learn how to include manually an originally unrecognized (unintegrated) component peak in our calibration analysis:

1 Zoom in on the region encompassing recognized component peaks "C24" (10.68 minutes) and "C28" (12.14 minutes) sufficiently to note the small peak at about 11.5 minutes (Figure 14).

2 Right-click on the top of this peak and select **Add Peak**.

- Be precise as possible in locating the top of a peak to be added to mark its best possible retention time
- The chosen top for the peak is released simply by clicking anywhere within the graphical display area allowing you to mark the top again, perhaps at greater magnification if desired
- Understand that the peak top you select visually may not necessarily be the top selected from the SimDis point of view: it chooses the nearest area slice boundary to your visual selection

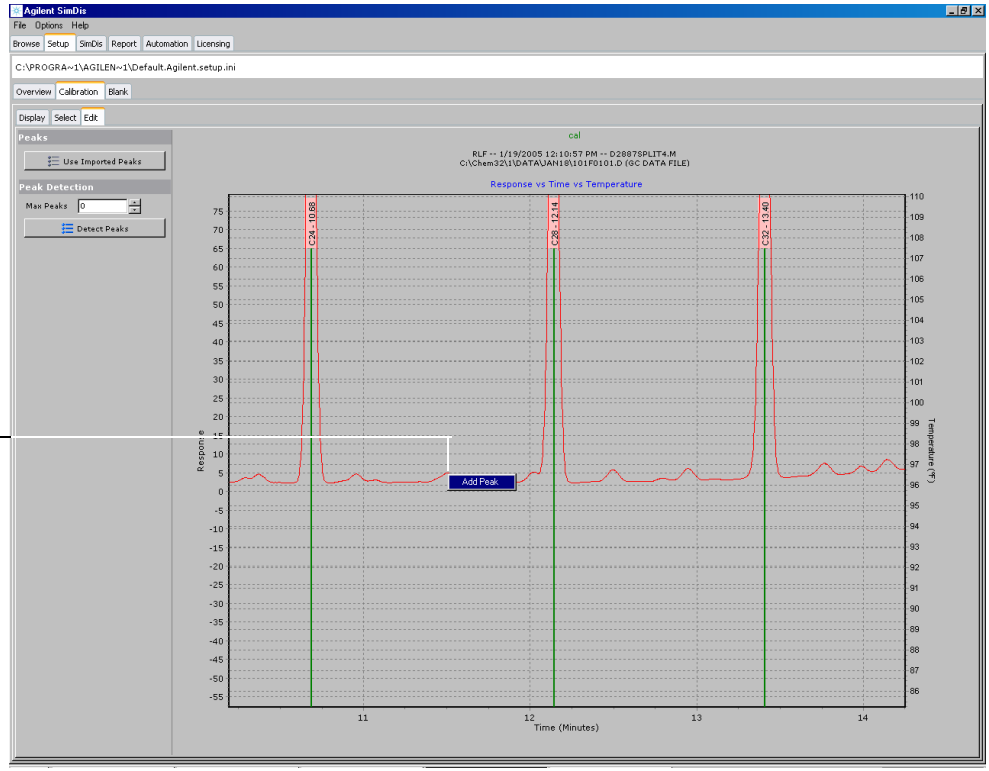


Figure 14 Marking the top of a component peak to be added

3 A green line and retention time label (approximately 11.5 minutes in this example) appear to confirm manual recognition (Figure 15).

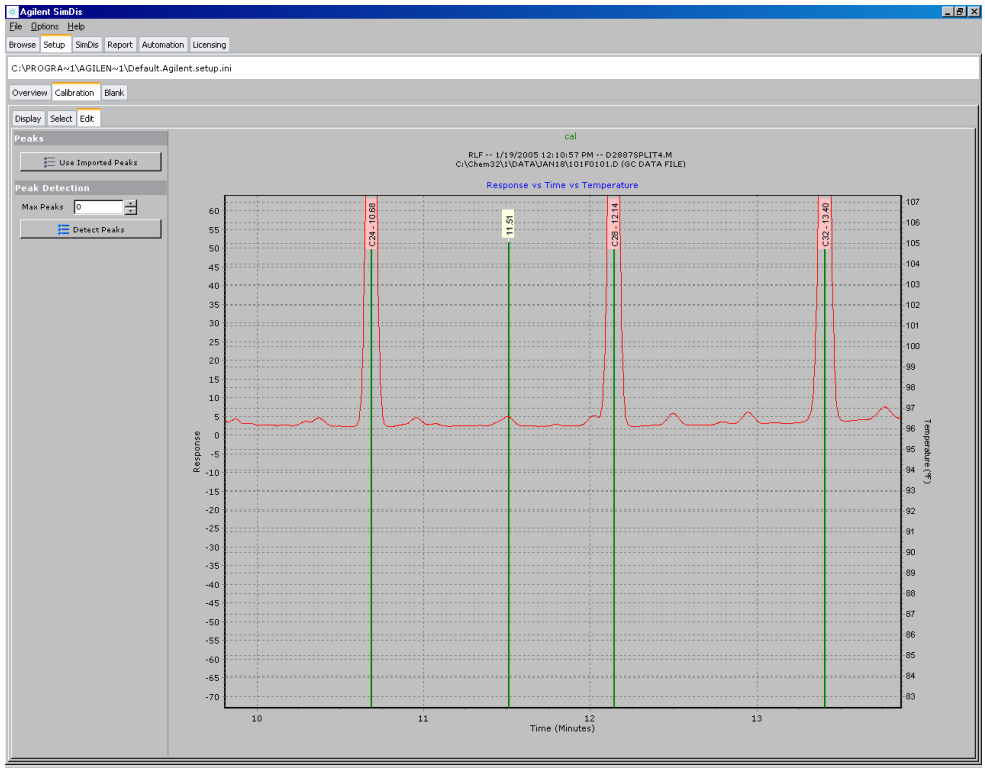


Figure 15 Manually added peak now included

4 Assign a carbon number to this added component as described previously. In this case, assign “C26” as it is a reasonable choice for a component whose retention time falls between that of “C24” and “C28” (Figure 16).

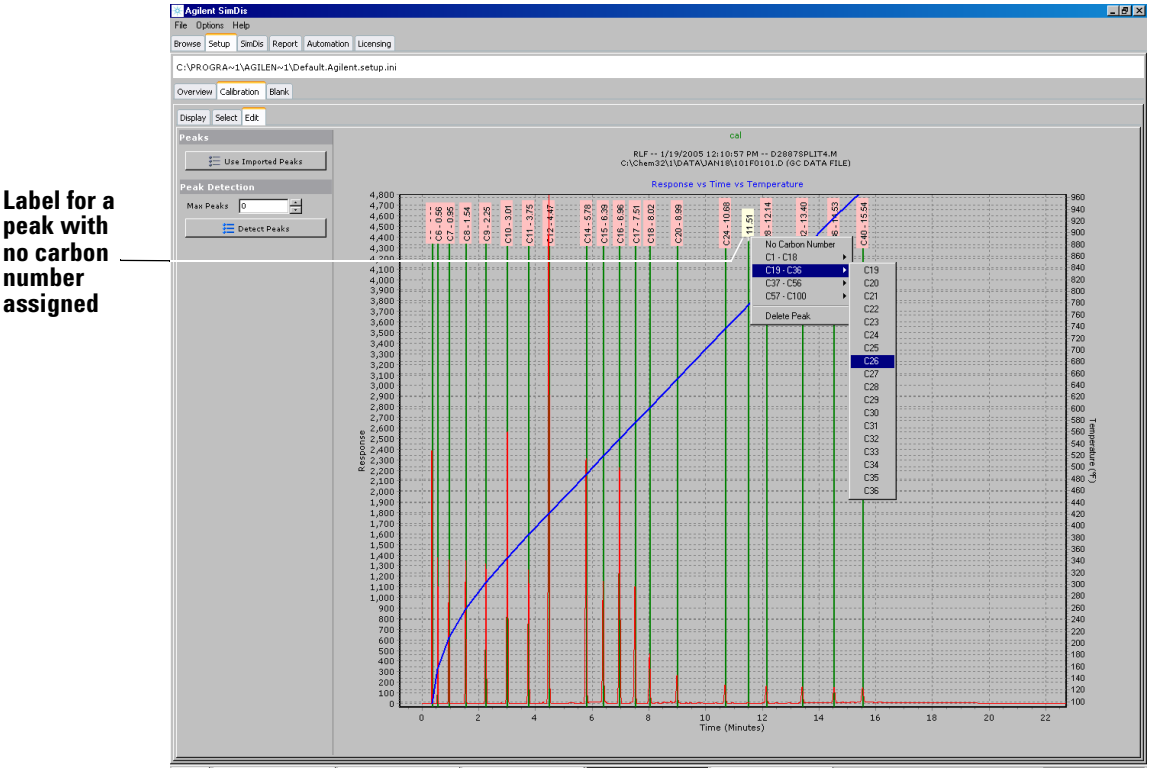


Figure 16 Carbon number assigned to added peak

5 Upon making the carbon number assignment, note that you must now either accept or discard this change before continuing. For our purposes here, **Accept** the change.

Note that addition of this originally-unrecognized component does not significantly alter appearance of the overlayed boiling point fraction distribution curve suggesting that the chosen retention time and assigned carbon number constitutes a reasonable association.

Deleting a peak

In this exercise, we will delete from consideration the component we just added (Figure 17).

1 Right-click the added component peak **label** (“C26”) to open the peak-specific menu.

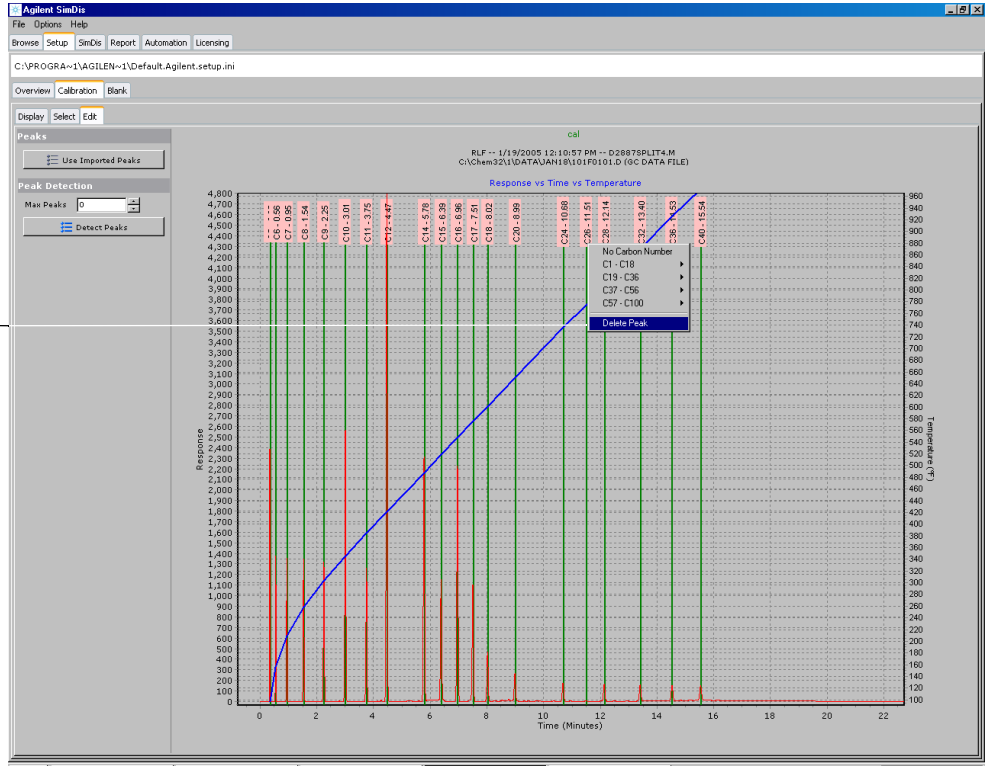


Figure 17 Deleting a component peak

2 Select **Delete Peak** from the menu.

- Upon making this selection, the green line and peak label disappear from the display indicating the component peak is no longer included in calculations
- The overlayed boiling point fraction distribution curve is also refreshed but, in this case, any change is too subtle to be seen

3 You must now either accept or discard this change before continuing. For our purposes here, **Discard** the change to restore the deleted component peak.

Finally, note that deleting a component peak does nothing to the underlying raw chromatographic data. Feel free, if desired, to again zoom in on the displayed chromatogram to verify the small component peak we used (at approximately 11.5 minutes) is both present and unchanged.

Unassigning a component peak carbon number

The carbon number assignment for any recognized component peak may be removed if desired (Figure 18).

1 Right-click the added component peak **label** (“C26”) to open the peak-specific menu.

2 Select **No Carbon Number** from the menu.

- Upon selection, the carbon number assignment “C26” disappears
- The component peak remains recognized but no longer is included in calculations. Carefully inspect the overlayed boiling point fraction distribution curve to determine effect of this change (if any)

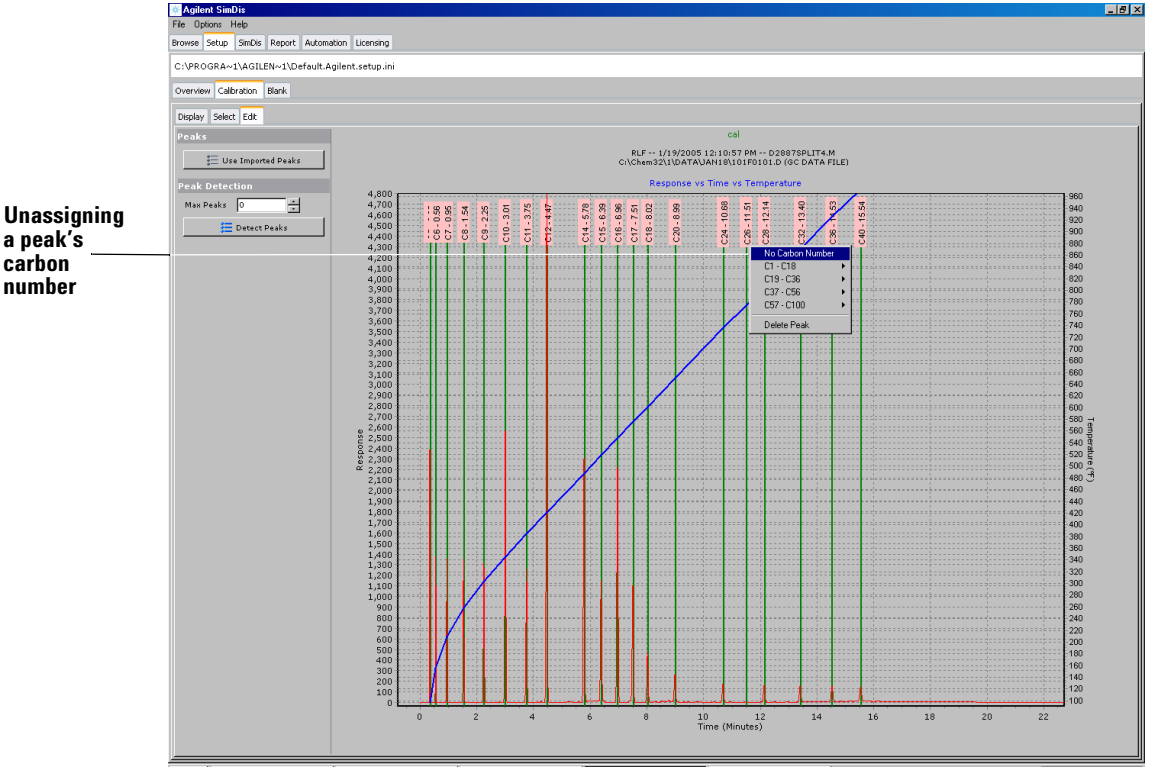
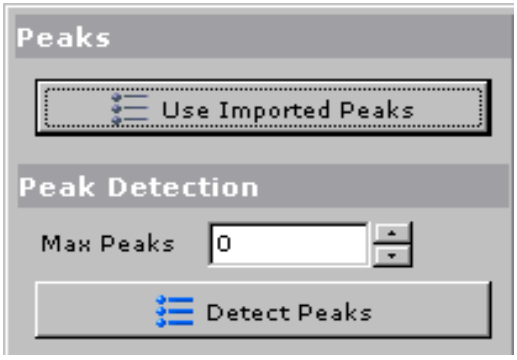


Figure 18 Unassigning a component peak's carbon number

3 You must now either accept or discard this change before continuing. For our purposes here, **Discard** the change to restore the component peak's carbon number assignment.

Additional features

Two additional features are **Peaks** and **Peak Detection**.



See Chapter 7, “Software Reference”, for more information.

The Blank tab

With your solvent blank data choice confirmed, select the **Blank** subtab. This provides two new subtabs, **Display** (the opening view) and **Select**, and also a graphical display of your selected solvent blank data (Figure 19).

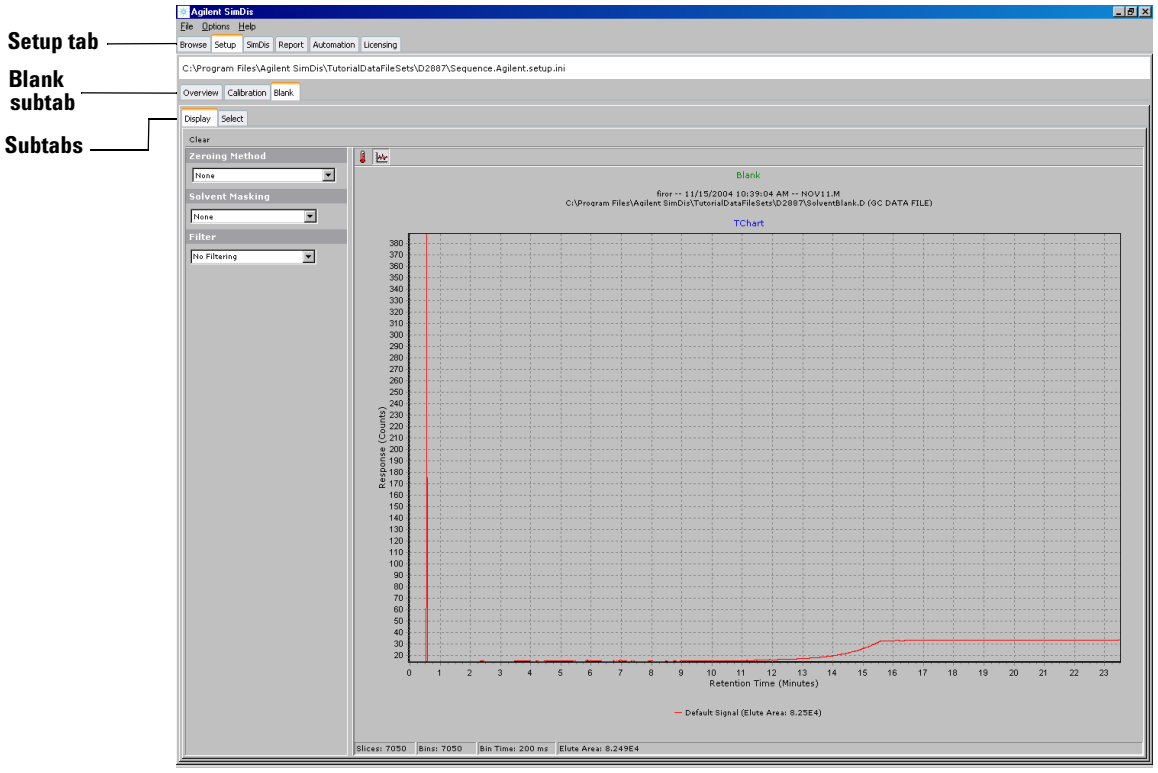


Figure 19 The selected solvent blank data displayed

In this tutorial context, we will discuss only some of the features available affecting SimDis interpretation and use of solvent blank data. See Chapter 7, “Software Reference”, for more information.

Verifying the correct chromatogram

As a matter of habit, inspect header information above the display area to verify you have the correct, desired ChemStation data. If not, switch to the **Select** tab to search for and to select the correct data file, or select '**(no blank)**' from the displayed list to remove any previously-selected solvent blank run (Figure 20).

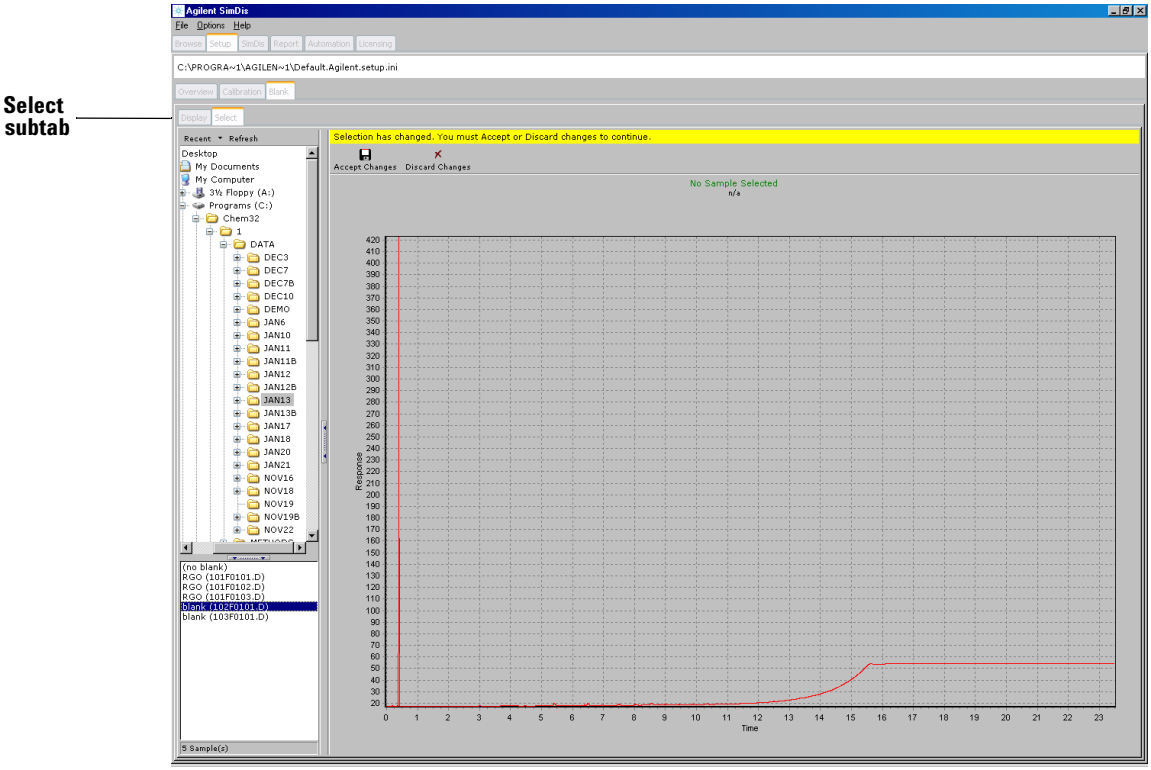


Figure 20 Use the **Select** tab to choose a different solvent blank

Upon selecting a different solvent blank data source, you must now either accept or discard this change before continuing. For our purposes here, **Discard** the change.

X-axis units: Temperature or Time

The button located at the upper-left corner of the graphical display area,

switches x-axis units between **Time** (minutes) or **Temperature** (Fahrenheit or Celsius, depending upon the choice made via the main menu item, **Options**). Display of Retention Times is normally recommended.

Choosing data to be displayed

To the right of **Time / Temperature** is a second button allowing choice between displaying only raw data or, additionally, the result of applying a selected filter to the data:

As an example, following are two displays: the first shows a highly magnified (zoom in) portion of our raw solvent blank data, and the second additionally shows effect of applying a filter to the data (Figure 21 and Figure 22).

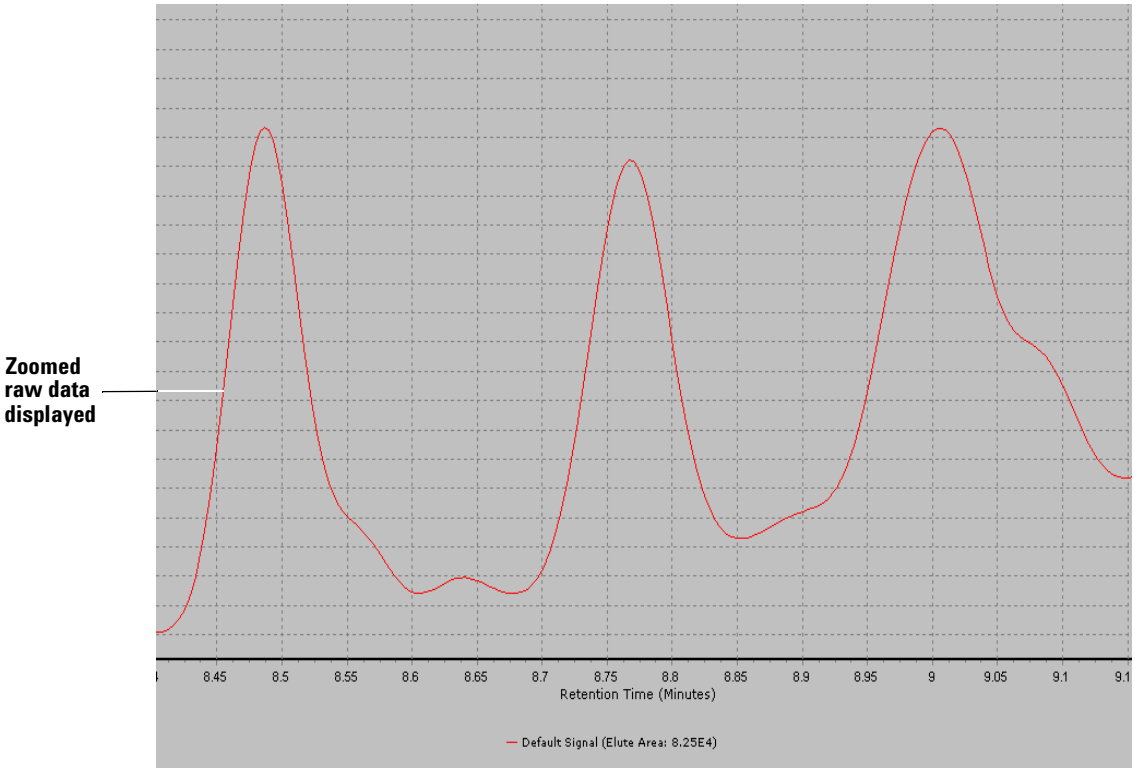


Figure 21 No filter effect displayed

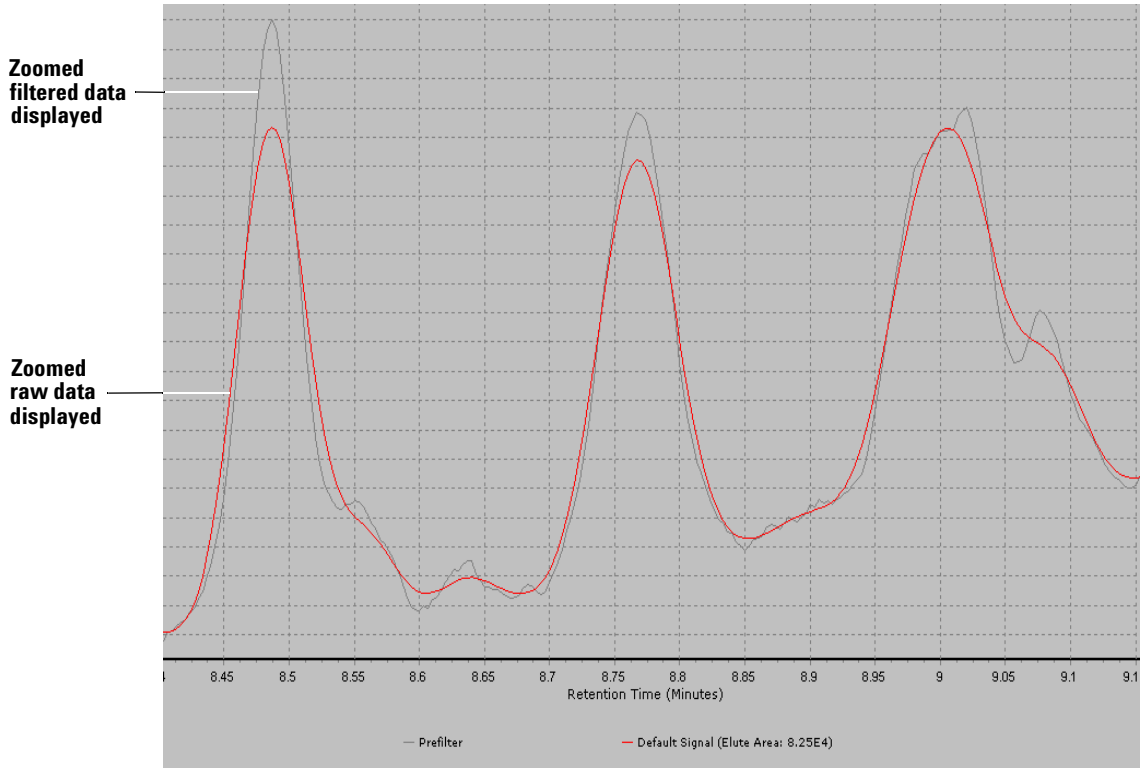


Figure 22 Effect of filter displayed

Two items of note:

- The effect of applying a filter to your data may be too subtle to see without significant magnification of the data
- The legend just below the graphical display area defines to color(s) used for displayed plot(s)

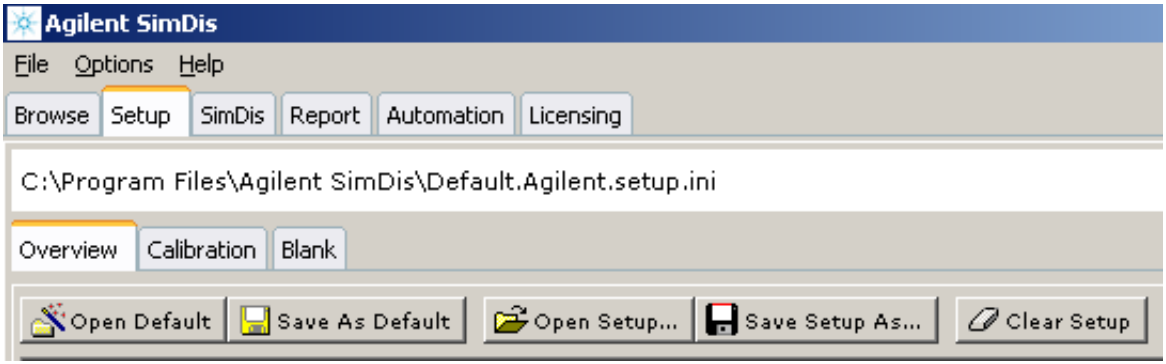
Additional features

Three additional drop-down menu features are **Zeroing Method**, **Solvent Masking**, and **Filter**:

Discussion of these is found in Chapter 7, “Software Reference”.

Preserving your Setup tab settings

At the beginning of discussion of the **Setup** tab (“5. The Setup Tab" on page 36) and its **Overview** subtab, we chose to ignore the displayed file path statement and five associated buttons, **Open Default**, **Save As Default**, **Open Setup . . .** , **Save Setup As . . .** , and **Clear Setup**. SimDis preserves your settings via these buttons for future use in analyzing compatible samples:



The four buttons, **Open Default**, **Save As Default**, **Open Setup . . .** , and **Save Setup As . . .** , files as means to save **Setup** tab settings:

- **Open Default** opens the SimDis default **.ini** file, **Default.Agilent.setup.ini** (located in the home SimDis folder, typically **C:\Program Files\Agilent SimDis**) and, in so doing, loads its contained specified settings, data file pointers, and so forth.
- **Save As Default** stores currently-specified settings, data file sets, and actions as the same file name, **Default.Agilent.setup.ini**, and thus overwrites previous contents.

- **Open Setup . . .** opens a browse view such that you can locate and select any user-named **.setup** file previously saved, in this case, one previously created as **D2887(050411).setup** (Figure 23).

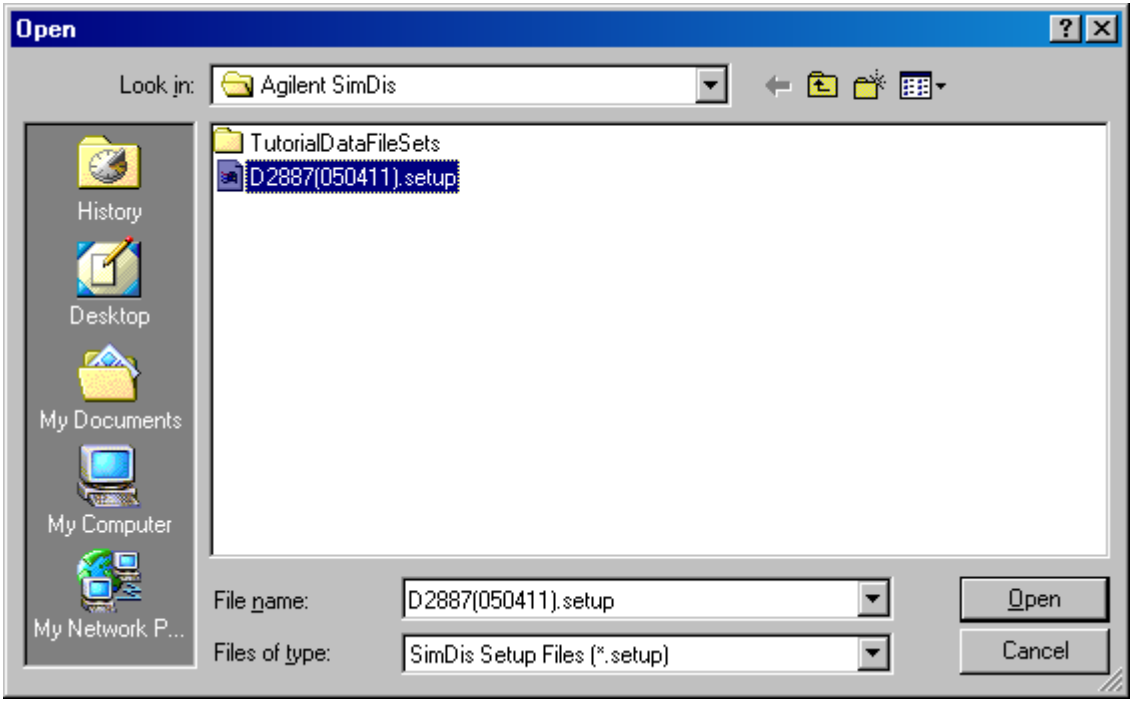


Figure 23 Locating a **.setup** file

- **Save Setup As . . .** opens a view such that you can specify and save a uniquely-named **.setup** file (in this example, one named **D2887(050411).setup** so as to include both the ASTM method employed and a date (yymmdd) in the name (Figure 24).

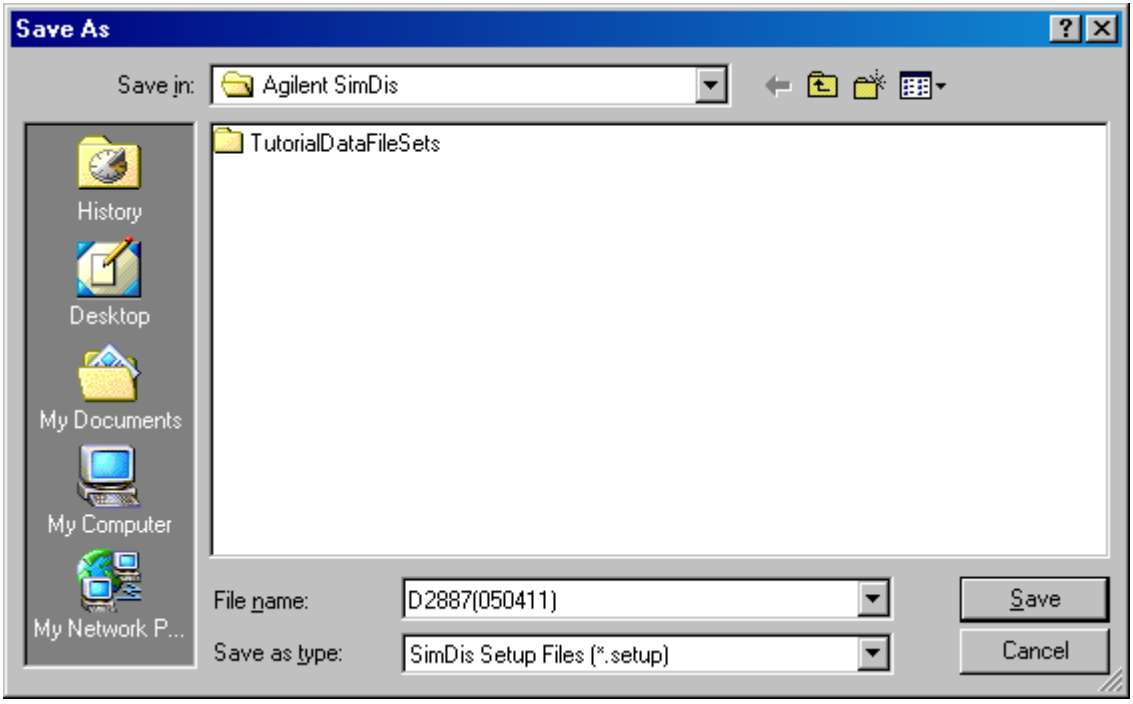


Figure 24 Creating and saving a uniquely-named **.setup** file

NOTE In specifying your own **.setup** file names, ONLY the first part of the name should be uniquely defined. That is, the name must adhere to the format: **< your file name >.setup**

Contents stored in, and usage of, a uniquely-named **.setup** file is no different than in the default **.ini** file. This alternative mode simply provides flexibility in maintaining multiple unique sets of **Setup** tab parameters. Note that an opened **.setup** file may be saved as the default **.ini** file, and, vice versa, the default **.ini** file may be saved at any time as a uniquely-named **.setup** file.

The fifth button, **Clear Setup**, re-initializes the SimDis default **.ini** file to a state where no **Calibration** and/or **Blank** sample files have been specified. Note that the button has no effect on saved **.setup** files.

As an exercise, save your present settings under a unique name (using **Save Setup As . . .)** and exit SimDis. Upon restarting SimDis and doing **Open Setup . . .** in **Setup** / **Overview**, select your uniquely-named **.setup** file to verify your data file sets, settings, and actions are as they were before.

6. The SimDis Tab

Having completed viewing and/or manipulating our calibration and solvent blank files, we can now move to the next tab, **SimDis**, which provides functions applied to our “checkout” sample file, in this case a ‘Reference Gas Oil’ analysis. The purpose is to optimize parameters and process as needed to achieve results expected under the given ASTM method, in this case, D 2887. With the checkout sample’s analysis conforming to expected ASTM results, one can presume your own laboratory samples will be treated properly:

The panel includes a considerable number of buttons, drop-down menus, and entry fields. As we use some of these features, remember that nothing we do within SimDis affects the raw ChemStation data file set: we can always start over if necessary.

Data displayed is the chosen **RefOilGas.D** data set you selected earlier via **Browse** (Figure 25).

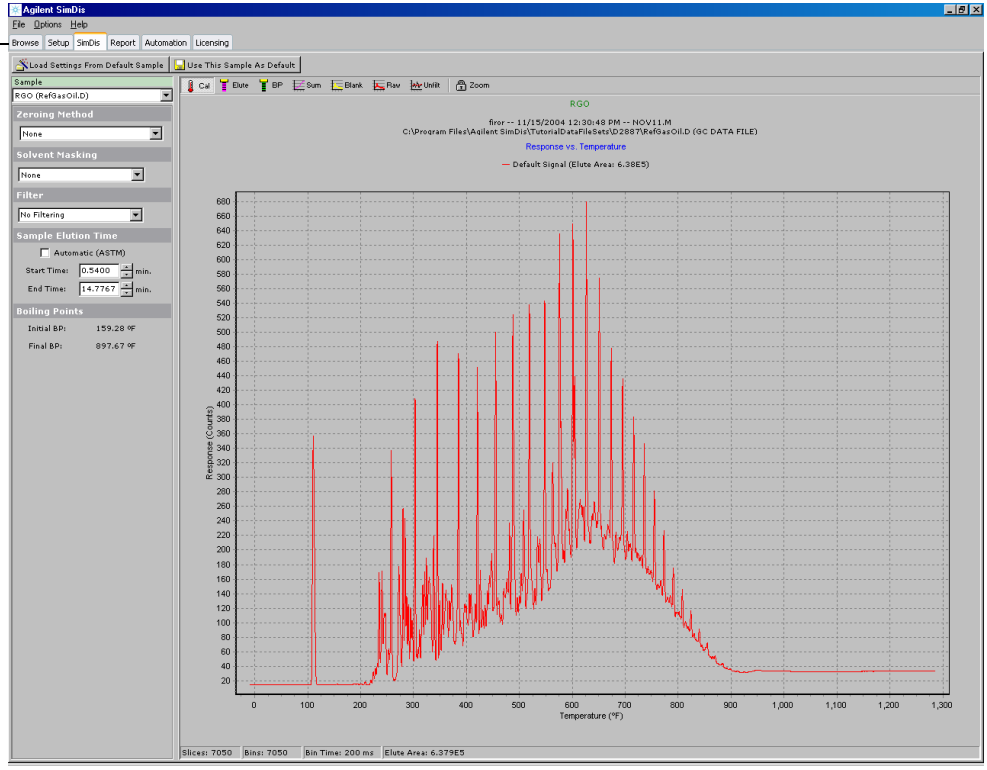


Figure 25 **SimDis** tab view

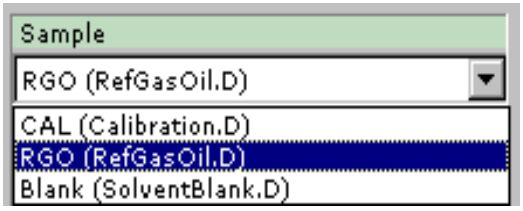
At this time, we will defer discussion of
Load Settings From Default Sample and **Use This Sample As Default**:

to “Default Sample buttons" on page 78.

Verifying the correct chromatogram

Again as a matter of habit, inspect header information above the display area to verify you have the correct, desired ChemStation data. If not, switch to the **Browse** tab to search for and to select the correct data if it is in an entirely different folder than the one originally chosen.

If the desired data set is in the **current** data folder, use the **Sample** drop-down list:



X-axis units: Temperature or Time

Note the **Cal** button, located left-most in the row of buttons above the graphical display:

Use this button to switch x-axis units between Time (minutes) or Temperature (Fahrenheit or Celsius, depending upon the choice made via the main menu item, **Options**). Display of Retention Times is normally recommended.

Zeroing Method drop-down menu

The **Zeroing Method** drop-down menu sets how to treat the sample's underlying chromatographic baseline:

We will look at two choices in the drop-down menu list, **None** and **Subtract Blank**. Other choices are described in Chapter 7, "Software Reference".

None selected

None does nothing to the sample's chromatographic baseline, thus giving a true view of the raw chromatographic signal (assuming no other signal-affecting functions are currently in use). In Figure 26, for example, note presence of the solvent peak, CS_2 , as the isolated left-most component peak:

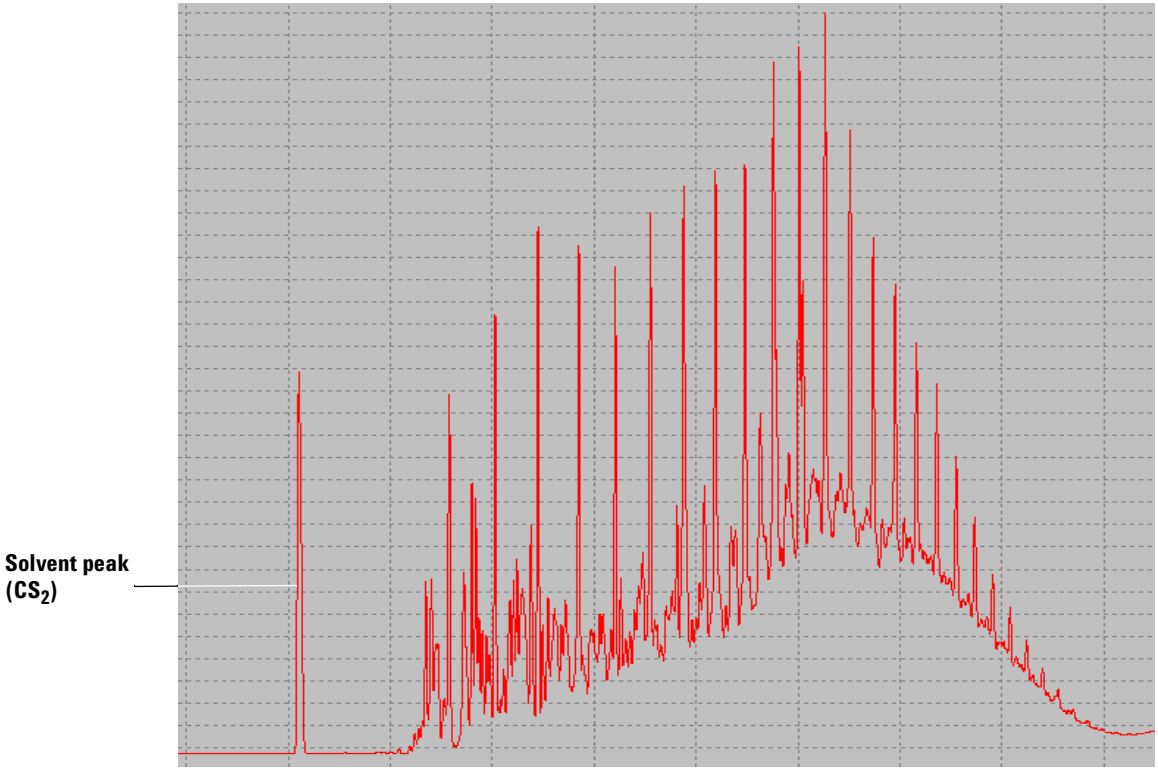


Figure 26 Uncorrected data displayed

Subtract Blank selected

Since we have a compatible solvent blank run already selected, we will now allow SimDis to subtract it from the sample's run by selecting **Subtract Blank** from the **Zeroing Method** drop-down list to yield the following net result (Figure 27).

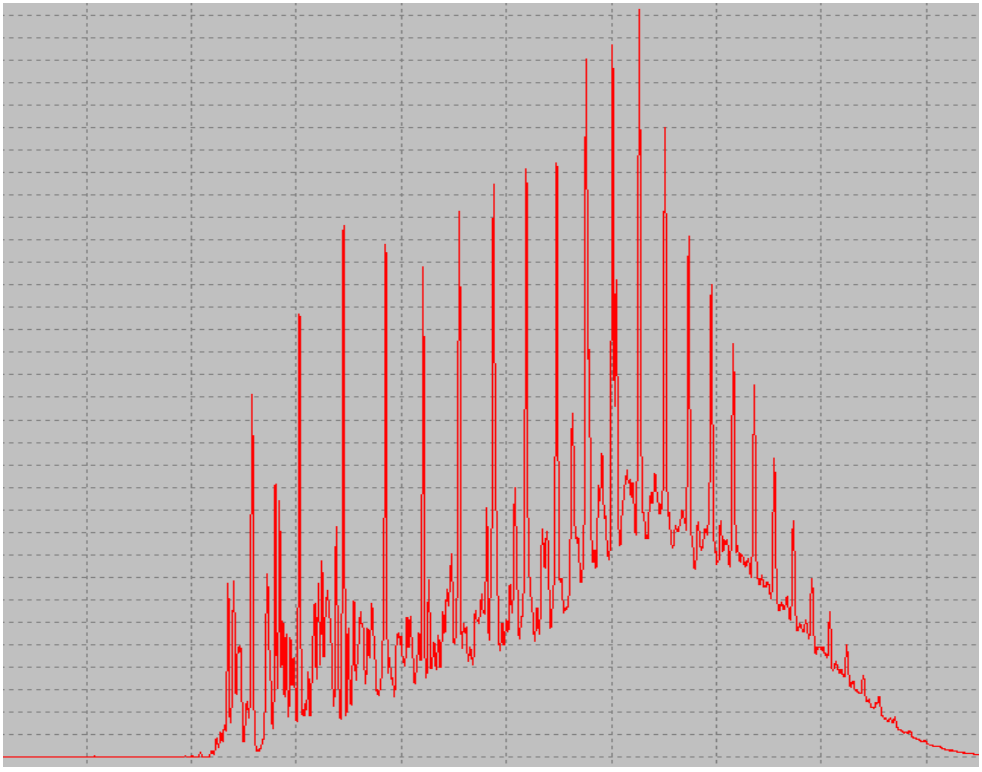


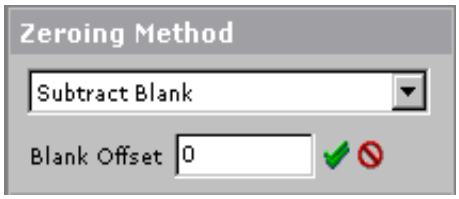
Figure 27 Corrected data displayed (solvent blank data subtracted)

Not only has the solvent peak been removed, but also any other potential chromatographic issues such as column bleed and small amounts of sample components carryover. This is the most common baseline treatment used for ASTM method D 2887.

The Blank button: As a convenience in viewing the relationship between sample versus solvent blank signals, note the **Blank** button located approximately midway in the row of buttons at the top of the graphical display area:

Use the button to switch between display of the sample chromatogram versus the sample chromatogram and an overlay display of the solvent blank chromatogram. The legend above the display area shows color used for each chromatogram display.

Blank Offset: In selecting **Subtract Blank** as the **Zeroing Method**, **Blank Offset** appears allowing you to manually adjust the response relationship between the sample's data and that of the solvent blank:



- An entered value adds or subtracts the specified response counts to the solvent blank run, thereby shifting solvent blank data relative to the sample's data values by the fixed amount:
  - Negative entry values decrease solvent blank data values and, after subtraction, thereby increase sample data values
  - Positive entry values increase solvent blank data values and, after subtraction, thereby decrease sample data values

To manually determine **Blank Offset**:

1 Use the **Blank** button to overlay the selected solvent blank run onto the displayed sample signal.

2 Enter **Blank Offset** values successively until sample and solvent blank signals superimpose at the starting time of the run.

Two buttons are also provided in choosing an offset value:

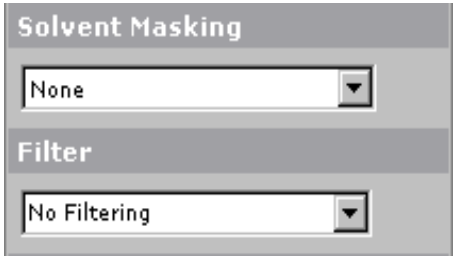
Recommended. If selected, SimDis determines a "best guess" offset value based upon the difference between the first data point for the solvent blank run versus that of the sample run. This is the recommended choice in most cases. More detail is available in Chapter 7, "Software Reference".

If selected, the offset is set to 0 (zero) whether appropriate to do so or not

In setting manual offset values, be careful, particularly with positive values, to not permit your sample's net resultant data to fall below zero on the Response (counts) axis as SimDis will then ignore it.

Solvent Masking and Filter drop-down menus

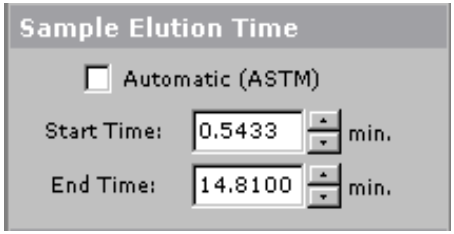
At this time, we will defer discussion of the two drop-down menus, **Solvent Masking** and **Filter**:



These menus are described in Chapter 7, “Software Reference”: “Solvent masking methods" on page 177 and “Filter methods" on page 178.

Sample Elution Time

Sample Elution Time entry fields let you set start and/or end times for SimDis analysis of the selected sample (our Reference Gas Oil “checkout” sample in this case):



Typically, the start time is anywhere after the solvent peak but before the beginning of any meaningful elution of components. The end is typically after elution of all meaningful components.

Automatic (ASTM) checkbox: Select (checkbox checked) to have SimDis employ the ASTM process for determining *initial boiling point (IPB)* and *final boiling point (FPB)* as described in the respective documents for ASTM Methods D 2887, D 2887x, and D 6352.

The Elute button: As a convenience in viewing start and end times, note the **Elute** button located second from the left in the row of buttons at the top of the graphical display area:

Use the button to switch on or off display of start / end markers overlayed onto the sample's chromatographic display (Figure 28).

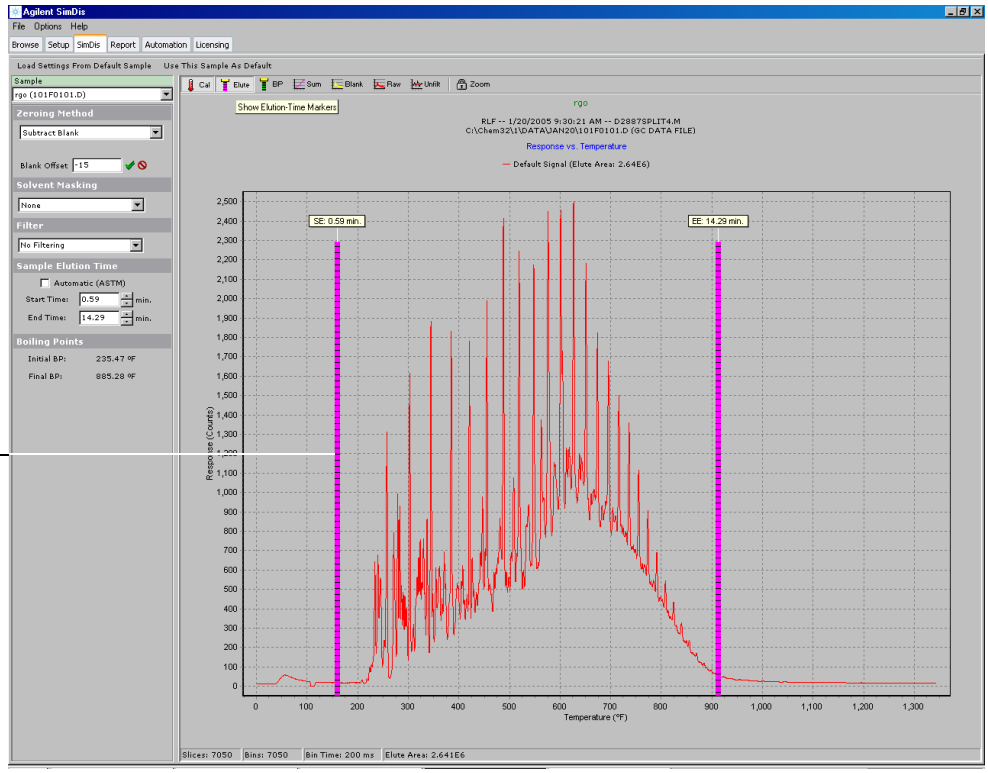


Figure 28 Start Elution (SE) and End Elution (EE) markers enabled

Markers are labeled "SE" (Start Elution) and "EE" (End Elution), and include their respective times (in minutes).

Moving Elution Markers: Manual placement of Elution markers may be necessary when, for example, use of the **Automatic (ASTM)** process causes the solvent peak to be included as part of the sample chromatographic data. Generally stated, **Start Elution** time should be set between the end of the solvent peak and beginning of the sample data; **End Elution** time should be somewhat after the last observable component peak in the sample data.

There are two ways to move elution markers:

- By direct entry of a numeric time value (in minutes) into the desired field (**Start Time:** or **End Time:**). Upon making a change, the affected elution marker (if displayed) will shift accordingly.
- By using an interactive graphics approach:

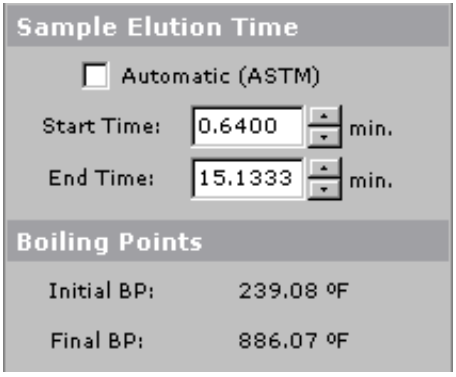
  1 Highlight the **Start Time:** or **End Time:** field by locating your mouse cursor within the field and doing a single mouse-click.

  2 Move your mouse cursor to the desired new time location along the displayed chromatogram (zoom first if desired) and double-click your mouse.

  The affected elution time marker shifts to the new location and the original entry field updates to reflect the new time value. Note that the actual time used is the nearest “end-of-slice” time so the value you enter may change slightly to reflect this.

Boiling Points

Initial (**Initial BP:**) and final (**Final BP:**) boiling point values are displayed below **Boiling Points** in association with **Sample Elution Time**:



Temperature units (Fahrenheit or Celsius) depend upon the choice made via the main menu item, **Options**.

This information is not interactive but is derived from **Sample Elution Time** values.

The BP button: Use this button to toggle display of the initial and final boiling point markers (Figure 29).

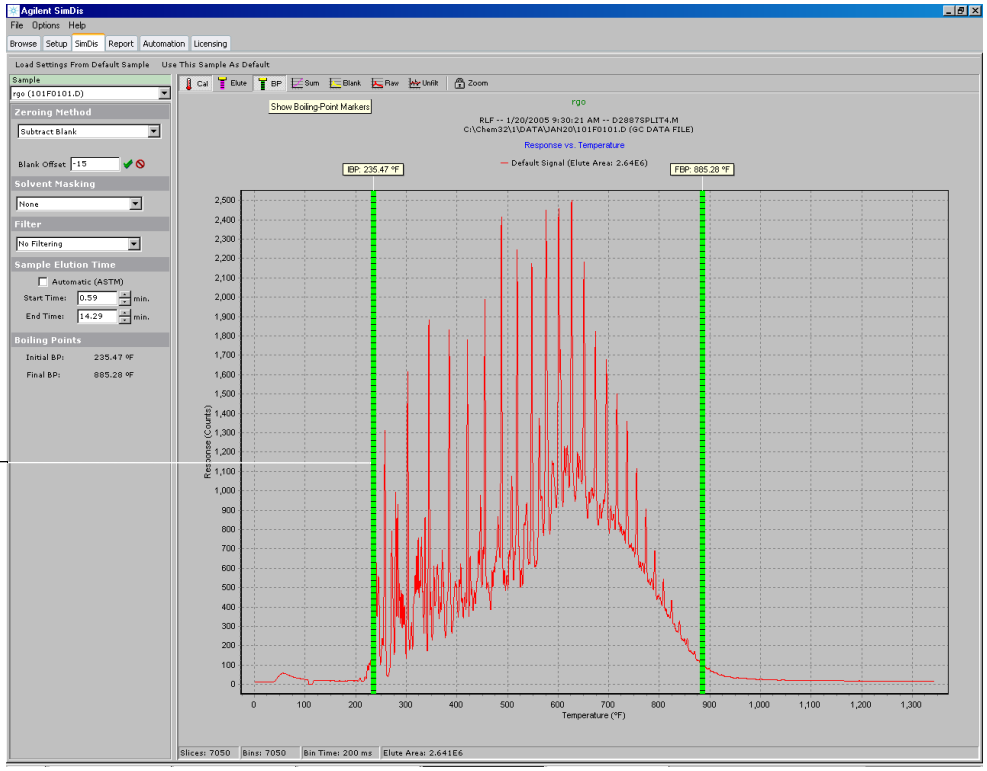


Figure 29 Initial boiling point (IBP) and final boiling point (FBP) markers enabled

Markers are labeled "**IBP**" (initial boiling point) and "**FBP**" (final boiling point), and include their respective temperatures (in the units selected via the main menu item, **Options**).

Display of boiling point markers is independent of display of elution time markers.

Remaining graphical display buttons

At this time, we will describe the remaining four buttons located above the graphical display area:

Sum (Show Percentage Yield): if enabled (Figure 30), the sample's chromatographic display is exchanged for a "Yield % Off" display. See Chapter 7, "Software Reference".

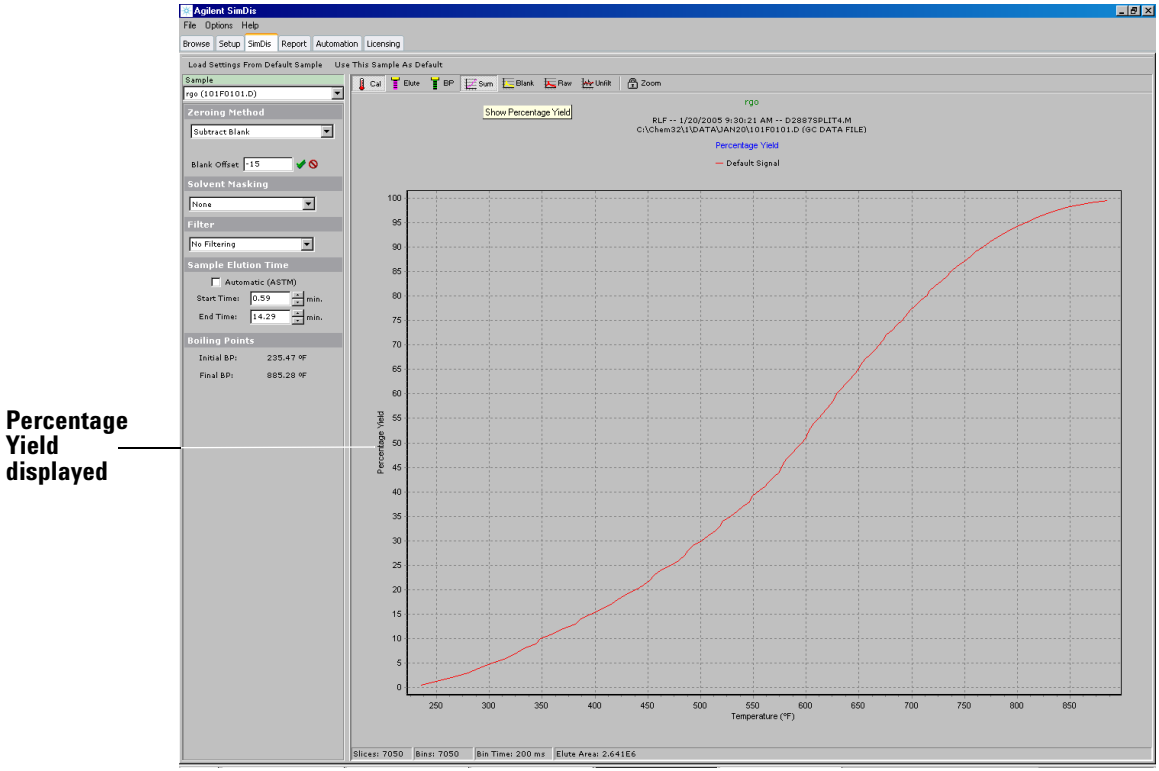


Figure 30 Alternative 'Yield % Off'

Raw (Show Uncorrected Signal): switches the display between showing the corrected chromatographic signal (in our case, a baseline subtraction of the solvent blank) or showing **both** the corrected chromatographic signal and the uncorrected (raw) chromatographic signal (Figure 31).

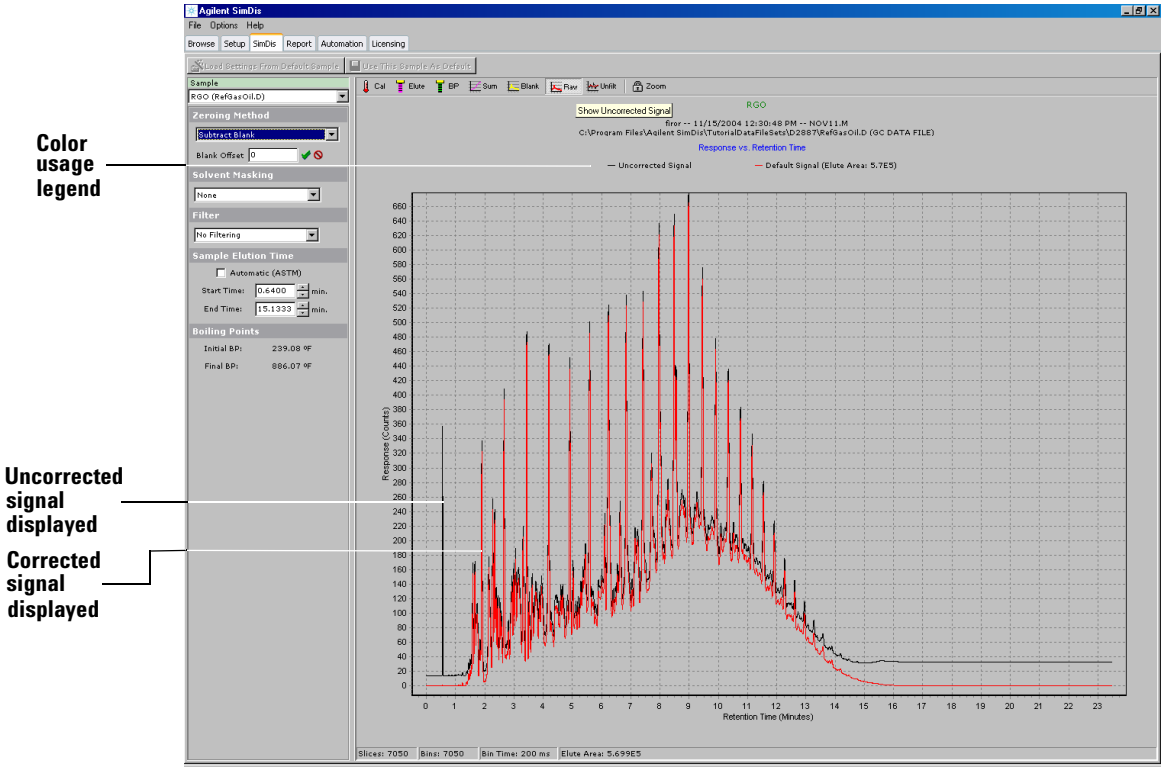


Figure 31 Corrected and uncorrected signals displayed

As before, when overlayed plots are displayed, the legend above the graphical display area shows color assignments. Also note the intentional large displacement between plotted signals to provide visual emphasis. Typically, they should essentially superimpose for a good solvent blank baseline subtraction situation.

Understand that, if no signal corrective measures are used, both display choices are identical (switch **Zeroing Method** to **None**, if desired, to see that the condition of **Raw** no longer matters).

Unflt (Show Unfiltered Data): similar in function to **Raw**, this button switches the display between showing the effect of use of a **Filter** to improve signal data versus also showing the data without use of the filter. Though **Filter** choices are described later in Chapter 7**, "Software Reference**", we can demonstrate effect upon the displayed signal via use of the **Unflt** button (Figure 32).

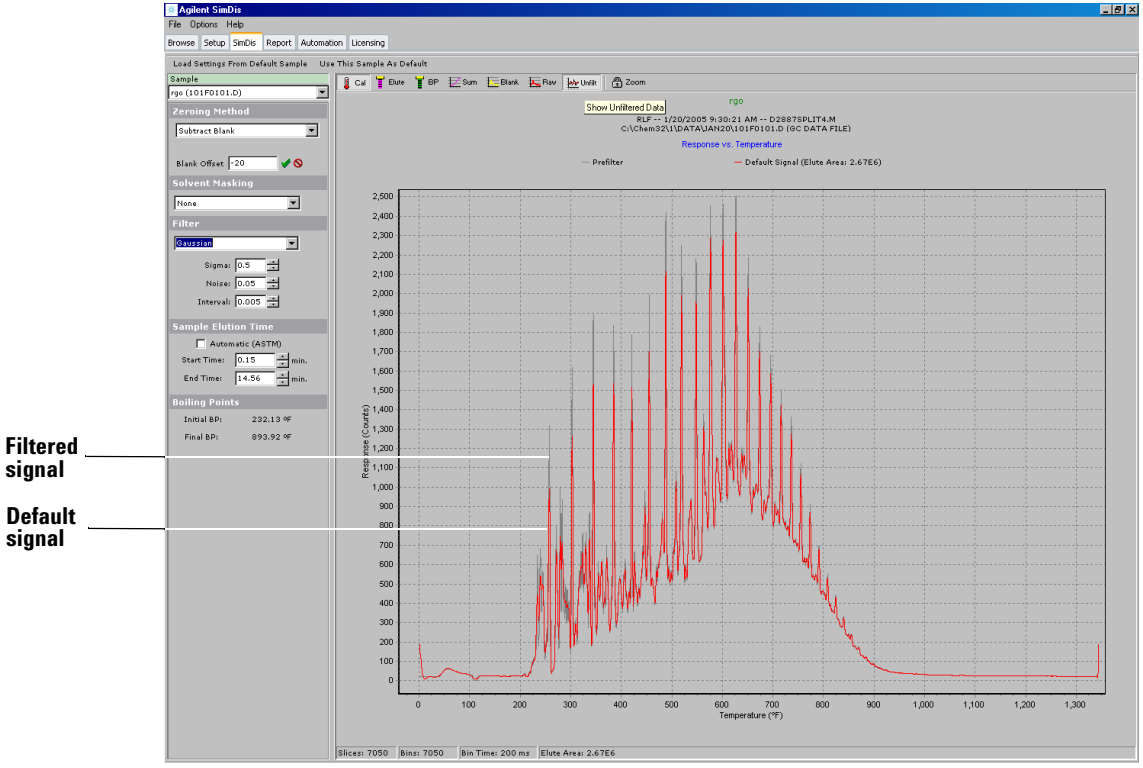


Figure 32 Example of a Gaussian filter on data

Note the following:

- To obtain this display, a **Gaussian Filter** was employed using its default parameter values
- As before, when overlayed plots are displayed, the legend above the graphical display area shows color assignments
- Finally, note absence of the solvent peak in either **Unflt** view choice: viewing the effect of a **Filter** uses corrected data as opposed to raw, uncorrected data.

Zoom (Retain zoom when switching samples / channels): if enabled, the current magnified (zoomed) display area is carried forward into graphical displays of other selected data files within the **SimDis** tab view, whether meaningful or not.

Finally, note that **all** buttons located above the graphical display area operate independently and may be used in any combination.

Default Sample buttons

At the beginning of our discussion, “6. The SimDis Tab" on page 61, we deferred discussion of **Load Settings From Default Sample** and **Use This Sample As Default** located immediately below the **SimDis** tab (and other tabs).

- All **SimDis** tab parameter settings, along with reference to the sample chosen in defining them, must be saved such that they can be applied to data from appropriate samples run later. Use:

  to save **SimDis** tab contents along with reference to the sample chosen to establish associated parameters.

  Once a given sample and its associated parameters are designated "**Default**" all future samples will be treated by SimDis using these conditions until such time that a new "**Default**" set is created.

- Use:

  to reset current **SimDis** tab conditions to most recent "**Default**" conditions.

No dialog is associated with either button and both become disabled ("greyed out") once "**Default**" conditions are established via **Use This Sample As Default**. The buttons become enabled when a new sample is chosen either from the **Sample** pull-down menu ("Verifying the correct chromatogram" on page 53), or by returning to the **Browse** tab (see "4. The Browse Tab" on page 27) to select a different ChemStation data-containing folder.

Finally, **SimDis** tab parameters are preserved locally with any selected sample data file, so it is up to you to specify the one to be the "**Default**" applied to other samples. This becomes important in making ChemStation runs where SimDis automatically treats the data generated from each run (see Chapter 6, "Automated SimDis Operation", for more about this).

7. The Report Tab

Before working with the **Report** tab, recall that printer selection and settings for SimDis reports are defined via main menu item **File / Printer Setup**. In practice, you may need to make choices there before defining **Report** tab parameters.

Having selected and prepared our three data file sets, **Calibration.D**, **RefGasOil.D**, and **SolventBlank.D**, we are now ready to move to the final operational step of reporting results via the **Report** tab:

Upon selecting the tab, the following view appears (Figure 33).

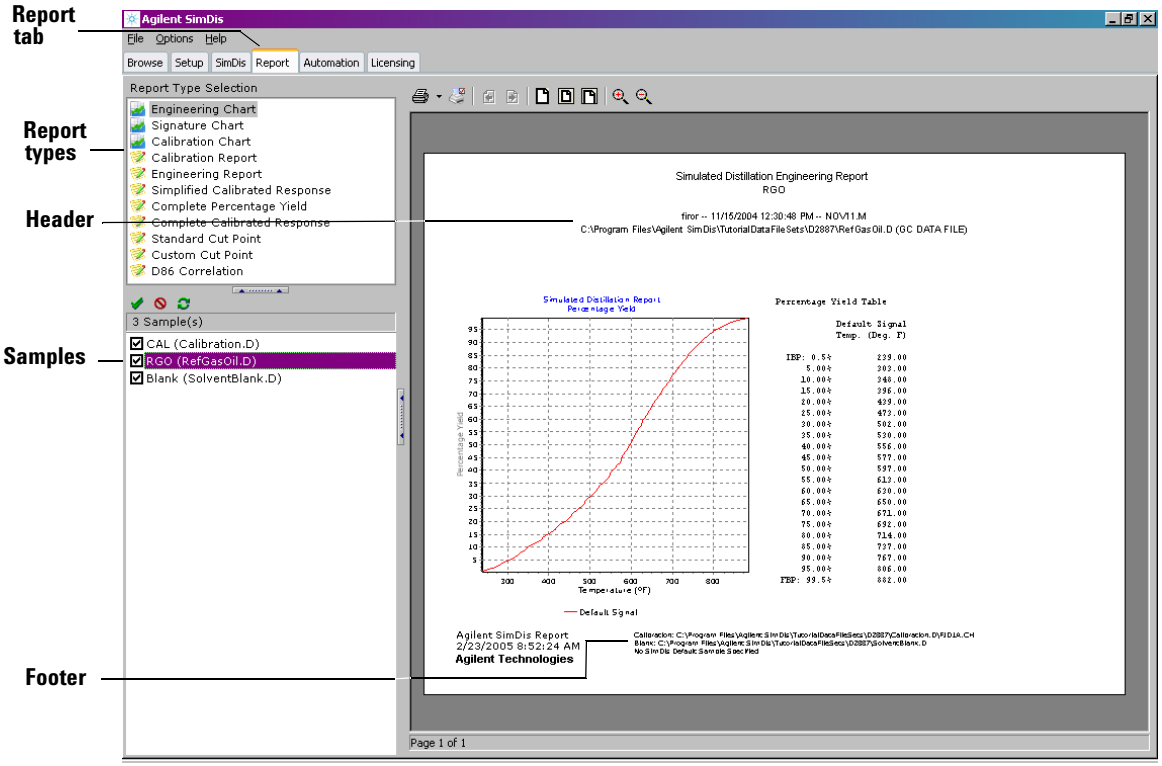
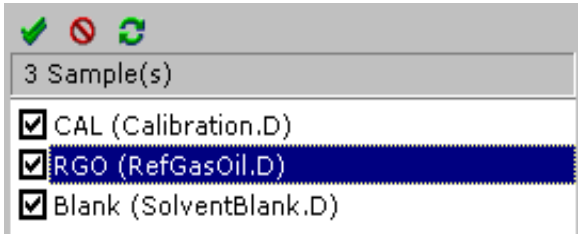


Figure 33 **Report** tab view

Review both header and footer information to verify the displayed report is derived from correct data file sets. In particular, verify that solvent blank and calibration files are as expected.

Sample(s) selection

Let us start by focusing on the sample selection area located below the row of tabs:



Listed are all sample data file sets found in the folder originally selected using **Browse**.

For our purposes here, in the context of obtaining a final sample results report, only **RefOilGas.D** has meaning. In your situation, there may be folders containing many suitable sample data file sets. The checkbox in front of each data file set controls whether or not to produce a report for that data file set:

- If checked, a report can be produced
- If unchecked, the data file set is skipped and no report will be produced
- Mouse-clicking a checkbox changes the reporting state for the associated data file set

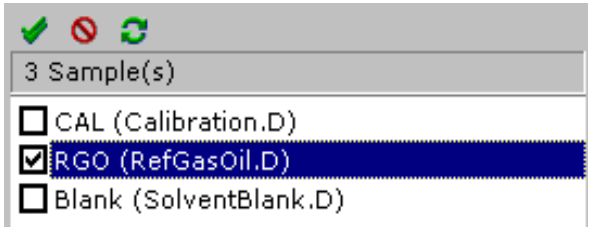
There are also three shortcut buttons available which are particularly useful when many data file sets are listed:

Check All Samples: checks all checkboxes setting all to be marked for “batch” processing / reporting.

Uncheck All Samples: unchecks all checkboxes such that none ar marked for reporting.

Invert All Sample Checkboxes: “flips” the check state of each checkbox to its opposite state.

For our purposes here, we only want **RefOilGas.D** to be checked for reporting:



Spend a few minutes checking and unchecking checkboxes, both individually and through use of the three shortcut tools.

Report Type selection

Two report styles are available:

If Report types containing a graphical chart

Report types containing text only

Either choice provides its own set of associated buttons. In a manner similar to graphical display situations we have seen in other views, the report display changes immediately with respect to choices made through use of the buttons. Thus, you have immediate feedback as to the effect of choice made. Feel free to try the following described functional items:

Report types containing graphical charts

Selecting a report containing a graphical chart provides a number of buttons affecting printed reports:

Moving from left to right:

These two buttons are involved in report printing:

- Select **Print** to immediately send the report to the designated printer

- If available, select **Batch Print** to stream reports to the designated printer for all selected (checkboxes checked) samples

Selecting these three buttons alters the report page format to be used when the report is printed. Note that the displayed report changes with the choice of button providing immediate feedback as to appearance of the printed report.

Select these “zoom” buttons to either magnify (zoom in, “+”) or reduce (zoom out, “–”) the displayed report. This function does not affect the printed report.

Report types containing text only

Similarly, selecting a text-only report provides a number of buttons affecting printing and/or saving of reports:

These three buttons are involved in report printing:

- Select **Print** to immediately send the report to the designated printer

- Select **Batch Print** to stream reports to the designated printer for all selected (checkboxes checked) samples

- Select to immediately send the report to a designated folder as a specified file name

Select **Save** from the drop-down menu to immediately send the report to a designated folder, or select **Batch Save** to stream reports to the designated folder for all selected (checkboxes checked) samples. Files are named as they appear in the samples list.

Select to include, or not include, header information in the report. Note that the displayed report changes with selection of header inclusion providing immediate feedback as to appearance of the printed and/or saved report.

Select one or the other of these buttons to format the file to be saved as either a “comma-separated variable” (“**csv**”) file or as a “tab-separated variable” (“**tab**”) separated file. The displayed report changes with selection of either button providing immediate feedback as to appearance of the printed and/or saved report.

Custom Cut Point Report – Use this text-only report type if you want to specify your own temperature "cut" points (Figure 34).

- A table is provided for entry of your specified **From** / **To** temperatures. Entered temperature values must match the unit chosen via the **Options** drop-down menu.
- Four buttons are associated with this report type:

Apply	Use this button to immediately display the resulting report using your currently entered Cut Point values
Load	Associated with **Save**, use this button to recall previously-saved Cut Point values
Save	Use this button to save current Cut Point values as a **.cut** file in any file name / folder of your choosing
Clear	Immediately erases all current Cut Point values in the entry table

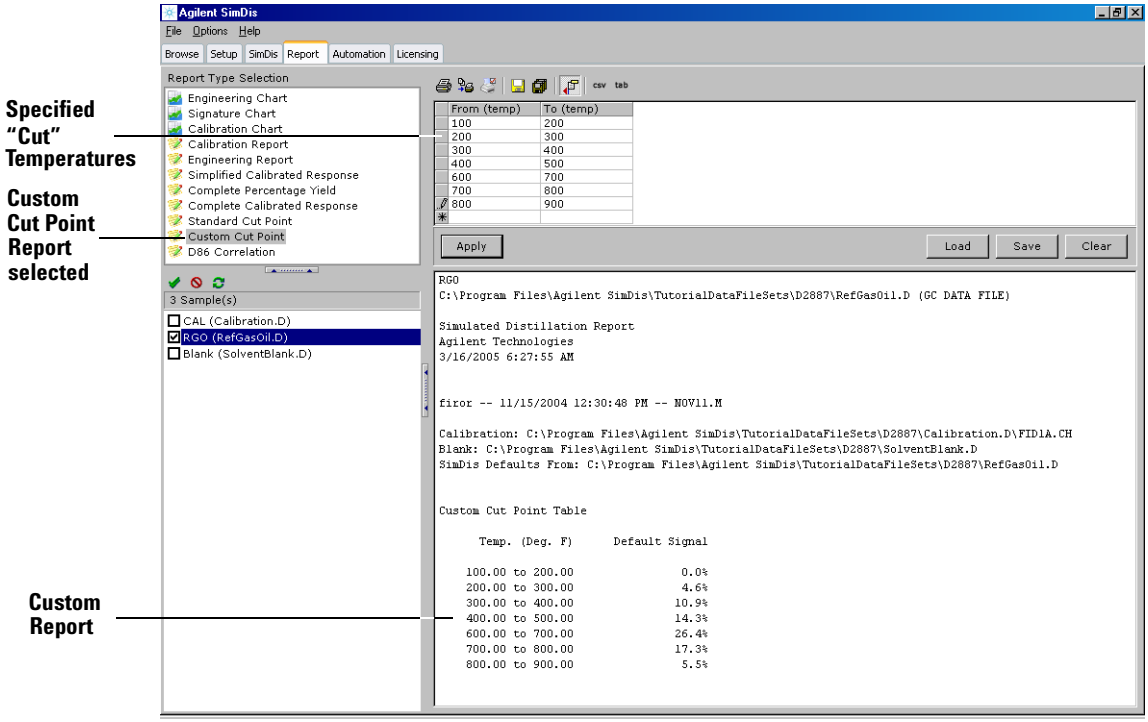


Figure 34 Custom Cut Point Report

8. Remaining Tabs

We have now completed our walk-through of the four operational tabs, **Browse**, **Setup**, **SimDis**, and **Report**. Two nonoperational tabs remain, **Automation** and **Licensing**:

See Chapter 6, "Automated SimDis Operation" and Chapter 2, "Installation": "To Install the SimDis System" on page 20 for more information.

Conclusion

Additional data file sets

Two additional tutorial data file sets are available for you to use with the SimDis application. Locations for you to **Browse** to in accessing these data file sets is, typically:

- For ASTM Method D 2887x –

  C:\Program Files\Agilent SimDis\TutorialDataFileSets\D2887x

- For ASTM Method D 6352 –

  C:\Program Files\Agilent SimDis\TutorialDataFileSets\D6352

If you use either of these methods in your laboratory, it is recommended that you repeat steps in this tutorial as needed using the appropriate data file set.

Moving on

With SimDis familiarization now completed, next steps involve your actual sample preparation, and the running of those samples to acquire ChemStation data in your laboratory. Proceed to Chapter 4, “Sample Preparation”.

4
Sample Preparation

This chapter describes preparation of your Solvent Blank, Calibration, and “checkout” samples for each one of the three ASTM methods, D 2887, D 2887x, or D 6352.

Types of Samples Needed

Four types of samples are typically required for each of the three ASTM standards, D 2887, D 2887x, and D 6352:

- A solvent-only sample for blank runs
- A calibration sample to establish relationships between component hydrocarbon retention times and their respective boiling points at atmospheric pressure
- A “checkout” sample to verify proper system behavior
- Your actual laboratory samples

WARNING Where Carbon Disulfide, CS_2, is used as solvent, note that this chemical is both toxic and highly flammable. Review its Material Safety Data Sheet (“MSDS”) for details in its safe handling and use.

NOTE In all cases, samples described are not to be considered quantitative: amounts specified are approximate.

NOTE Only general sample preparation procedures are included here. Typical example chromatograms and analytical conditions applicable to these samples are to be found in Chapter 5, “Running Samples.”

For clarity, these similar (but not identical) sample preparation processes are described separately for each of the three ASTM methods. Select the specific section(s) for the applicable method(s) you use.

Sample Preparation for ASTM D 2887

ASTM D 2887: applicable to petroleum products and fractions having an initial boiling point greater than 55 °C and a final boiling point less than 538 °C.

Solvent Blank sample

A Solvent Blank sample is used to determine system cleanliness and column bleed behavior before calibration and analysis of your own samples. It is simply a sample of the solvent used, CS_2 ("reagent grade" purity or better).

Calibration sample

Prepare the calibration sample from Agilent "*Boiling Point Calibration No. 1*" sample, part number 5080-8716, according to the following procedure:

1. Warm the source sample ampule to 50 to 60 °C and shake, as needed, to ensure no solids are visible.
2. Break the ampule and transfer its contents to a larger, sealable container.
3. Add CS_2: 5 to 10 parts CS_2 per 1 part of the sample.
4. Seal the container.
5. Refrigerate the CS_2-diluted sample container if not to be used immediately.
6. At time of use, if refrigerated, inspect for visible solids. If necessary, warm as needed to redissolve sample components.

Reference Gas Oil "checkout" sample

Based upon the specific inlet being used, the “checkout” sample is prepared from Agilent “*Reference Gas Oil Sample No. 1*”, part number 5060-9086, according to the following procedure:

HT PTV inlet

1. No source sample warming is necessary.
2. Break the ampule and transfer its contents to a larger, sealable container.
3. Add CS_2: 5 to 10 parts CS_2 per 1 part of the sample.
4. Seal the container.
5. Refrigerate the CS_2-diluted sample container if not to be used immediately.
6. At time of use, if refrigerated, inspect for visible solids. If necessary, warm as needed to redissolve sample components.

Agilent Split/Splitless Inlet (split mode)

1. No source sample warming is necessary.
2. Normally, no dilution is necessary. Instead, recommended, use a 5-µL syringe to inject 0.1 µL.
3. If dilution is required, break the ampule and transfer its contents to a larger, sealable container.
4. Add CS_2 in an amount appropriate for your split ratio.
5. Seal the container.
6. Refrigerate the CS_2-diluted sample container if not to be used immediately.
7. At time of use, if refrigerated, inspect for visible solids. If necessary, warm as needed to redissolve sample components.

Agilent Cool On-Column Inlet

1. No source sample warming is necessary.
2. The sample may be run without dilution for an 0.1 µL injection volume.
3. If dilution is required, break the ampule and transfer its contents to a larger, sealable container.
4. Add CS_2: 5 to 10 parts CS_2 per 1 part of the sample.
5. Seal the container.
6. Refrigerate the CS_2-diluted sample container if not to be used immediately.
7. At time of use, if refrigerated, inspect for visible solids. If necessary, warm as needed to redissolve sample components.

Your laboratory samples

To avoid sample overload problems, particularly for the HT PTV inlet, and/or for viscosity problems, dilute your samples as needed using CS_2. Dilution also may be necessary for columns with film thicknesses less than 1.0 micron (for example, Agilent column 19095Z-021, HP-1, 10-m, 0.53-mm O.D., 0.88-µm film thickness).

Sample Preparation for ASTM D 2887x

ASTM D 2887x: applicable to petroleum products and fractions having an initial boiling point greater than 100 °C and a final boiling point less than 615 °C.

Solvent Blank sample

A Solvent Blank sample is used to determine system cleanliness and column bleed behavior before calibration and analysis of your own samples. It is simply a sample of the solvent used, CS_2 (preferred), or cyclohexane as an alternative.

Calibration sample

Prepare the calibration sample from Agilent "*Polywax® 500 (neat)*" sample, part number 5188-5316, combined with Agilent "*Low Boiling Point Calibration No. 220*" sample, part number 5080-8768, according to the following procedure:

1. Dissolve the Polywax sample using CS_2, typically, 1 part sample to 100 parts CS_2.
2. Combine the diluted Polywax sample with the Agilent sample: 1 part Agilent sample to 20 parts diluted Polywax sample.
3. Recommended but not required: spike the sample with an additional amount of n-eicosane ("C20") to provide a known starting point for identification of components having higher carbon numbers.
4. Transfer this combined sample to a suitable sealable container.
5. Seal the container.
6. Refrigerate the container if not to be used immediately.
7. At time of use, if refrigerated, inspect for visible solids. If necessary, warm as needed to redissolve sample components.

Note that if your laboratory samples contain light-end components in the range of C5 through C18, you may optionally want to add these to the calibration mix. Similarly, you may optionally add components in the range C62 through C68 to validate C60, the highest-boiling *required* component.

Heavy Gas Oil "checkout" sample

If available, a "*Heavy Gas Oil*" or mid-range distillate of known boiling point distribution, can be used if diluted with CS_2 as needed, to avoid inlet / column overload problems, particularly for the HT PTV inlet.

Your laboratory samples

To avoid sample overload problems and/or for viscosity problems, dilute your samples as needed using CS_2 (preferred), or using cyclohexane as an alternative.

Sample Preparation for ASTM D 6352

ASTM D 6352 - 02: applicable to petroleum products and fractions having an initial boiling point greater than 174 °C and a final boiling point less than 700 °C.

Solvent Blank sample

A Solvent Blank sample is used to determine system cleanliness and column bleed behavior before calibration and analysis of your own samples. It is simply a sample of the solvent used (CS_2 in most cases).

Calibration sample

The calibration sample is prepared from Agilent "*Polywax® 655 (neat)*" sample, part number 5188-5317, combined with Agilent "*Low Boiling Point Calibration No. 220*" sample, part number 5080-8768, according to the following procedure:

1. Dissolve the Polywax sample using CS_2 to achieve a 1% (by weight) solution.

   If desired, toluene or p-xylene may be used in place of CS_2 (review their *Material Safety Data Sheets* with respect to health and safety information).
2. Combine the diluted Polywax sample with the Agilent sample: 1 part Agilent sample to 20 parts diluted Polywax sample. Note that addition of the low boiling C5 to C18 sample is not required if your samples have initial boiling points above C20 to C22.
3. Spike the sample with an additional amount of n-eicosane ("C20") to provide a known starting point for identification of other sample components.
4. Transfer this combined sample to a suitable sealable container.
5. Seal the container.
6. Refrigerate the container if not to be used immediately.

7 At time of use, if refrigerated, inspect for visible solids. If necessary, warm as needed to redissolve sample components.

Reference Material 5010 "checkout" sample

If available, "*Reference Material 5010*", described in ASTM Method D 6352, can be used if diluted with CS_2 as needed to avoid inlet / column overload problems.

Your laboratory samples

To avoid sample overload problems and/or for viscosity problems, dilute your samples as needed using CS_2.

Conclusion

At this time, with your samples prepared, you are ready to run the samples (solvent blank, calibration, and "checkout") using the GC ChemStation as described in Chapter 5, "Running Samples".

5
Running Samples

This chapter describes how to set up the ChemStation to produce and save data-containing files used by SimDis, and then to create and to save ChemStation Methods used to run samples for each of the ASTM methods. General knowledge of the GC, injector, and the ChemStation (its methods, sequences, reporting, and running of samples) is assumed. Further, your system should be correctly configured and ready for use (column installed, gases and power connected, and so forth) for the ASTM method to be employed.

It is also assumed that you have available solvent blank, calibration, and “checkout” samples for your chosen ASTM method. See Chapter 5, “Running Samples”, if not.

A Brief Discussion about the ChemStation

The ChemStation provides two modes for running samples:

- Samples run individually and separately through manual initiation
- Groups of samples run automatically and continuously ("Sequences"). Note that the number of sample runs included in a Sequence can be as few as one.

Note that ChemStation documentation referring to "blank" runs is in the context of running true blank runs (*no* sample injected) as opposed to *solvent blank* runs where sample (the solvent) is injected.

For simplicity, sample-related discussions following are in terms of running samples individually, one-by-one. Since you can use either ChemStation mode for this, refer to your ChemStation documentation as needed and familiarize yourself with use of both modes.

Mode 1– Samples run *outside* a ChemStation Sequence

This mode accommodates only the running of single, individual samples. For this mode, familiarize yourself with use of the following ChemStation view obtained by the main menu item **RunControl** followed by **Sample Info . . .** (Figure 35).

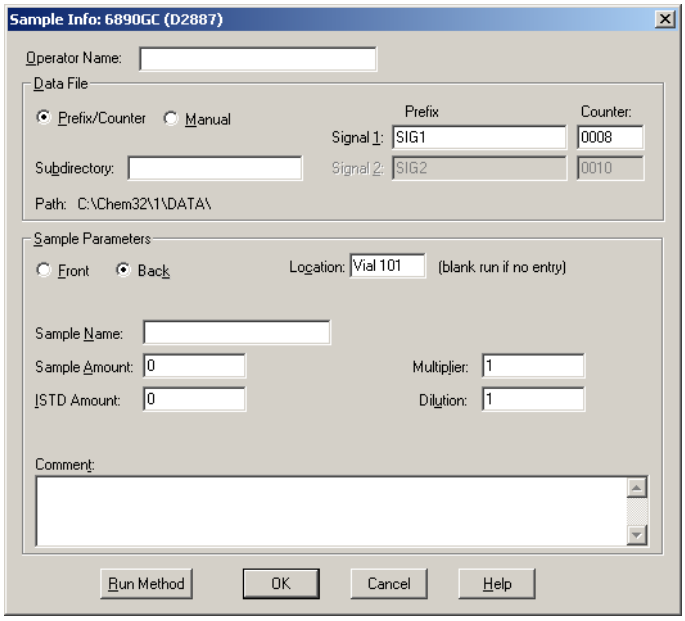


Figure 35 Single sample mode (nonSequence operation)

Mode 2– Samples run from within a ChemStation Sequence

This mode accommodates automated, continuous running of any number of samples from one to the capacity limit of your automatic sampler. For this, familiarize yourself with use of the following ChemStation views obtained by the main menu item **Sequence** followed separately by **Sequence Parameters . . .** and **Sequence Table . . .** (Figure 36 and Figure 37).

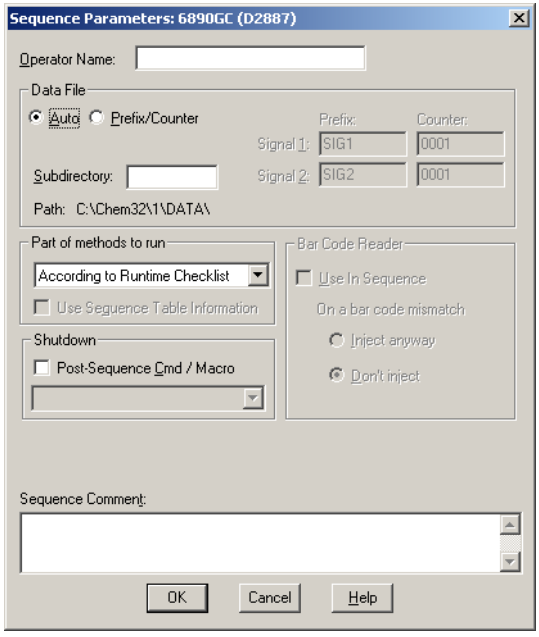


Figure 36 ChemStation Sequence Parameters

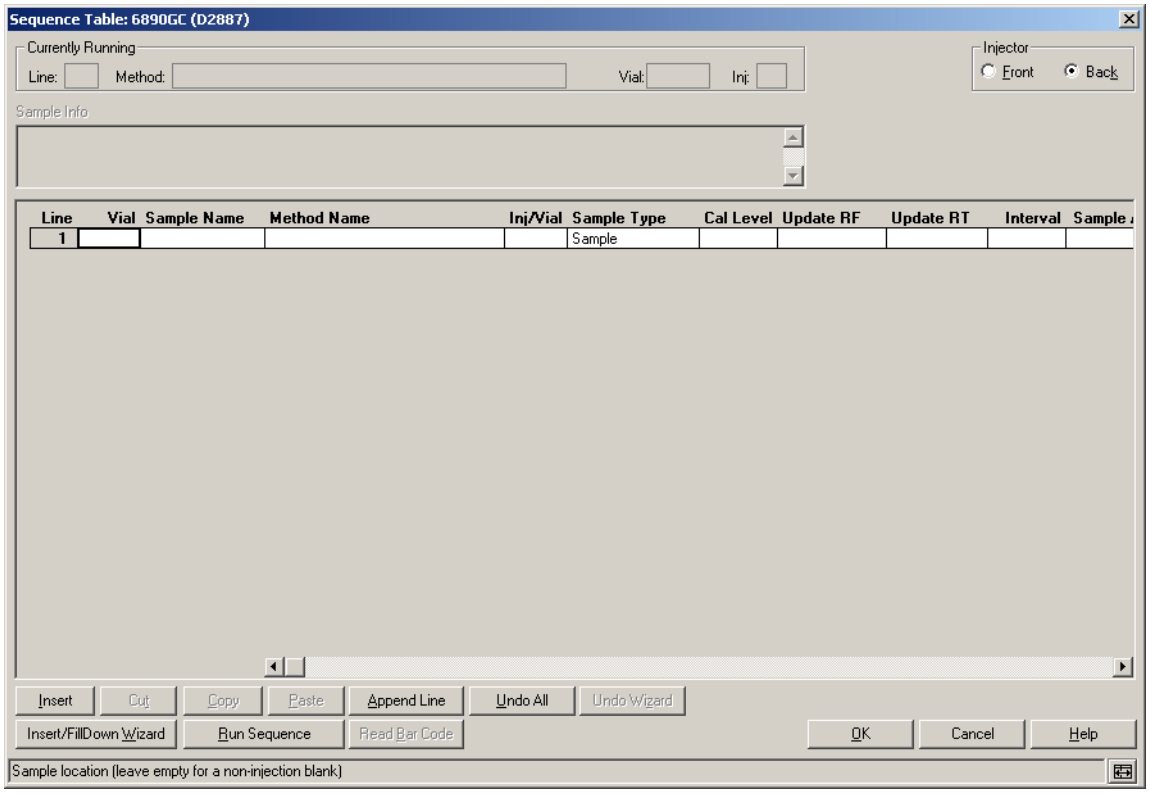


Figure 37 ChemStation Sequence Table

ChemStation organization of methods and sample data

This chapter treats running of samples in a very simplistic and linear manner as compared to your likely situation: many samples from different sources, arriving at different dates and times, and all competing for quick turnaround times. You also have periodic required system validations to perform (solvent blank, calibration, and “checkout” sample runs) which might cause small revisions to be made to your methods over time. To help in your organization, note that:

- The ChemStation offers a very flexible environment with respect to unique naming of both methods and, separately, data sets generated from your runs made using a selected method. Thus, your operations can be organized such that everything is readily recognized and traceable.
- A number of different opportunities are provided to add notes, for example into a given method, to document changes made or other significant items.
- One can have the ChemStation save a *copy* of the method within data-containing folders created in the running of samples under that method. This feature, described in Chapter 6, “Automated SimDis Operation” (see page 145), is useful in absolutely associating a given method with any sample data it generated.

If needed, review your ChemStation documentation with respect to using these features in your processes, both for discrete single runs, and for automated series of runs (“Sequences”).

Process Overview

Before you can collect data and analyze it using the SimDis application for any of the ASTM methods you intend to use (D 2887, D 2887x, or D 6352), you must:

1. For the given ASTM method, create a new ChemStation Method. As part of that method, enable production of report files required by the SimDis application.
2. Enter all necessary chromatographic setpoints (temperatures, gas flow rates, injection parameters, and so forth).

   Understand that the same GC parameters must be used for all sample types (solvent blank, calibration, “checkout”, and your laboratory samples) run under the given ASTM method to insure identical baseline behaviors. Non-GC parameters, such as injection parameters and analysis of data by the ChemStation, may be modified depending upon the sample type.
3. **Save** this ChemStation “base” method using an easily recognizable name.
4. With setpoints established and the GC given stabilization time, one or more Solvent Blank runs are made to verify system cleanliness, to capture column bleed behavior, to find retention time location for the solvent peak, and to ensure the system is ready for use with subsequent sample types.
5. Make one or more runs with the appropriate calibration sample to establish a calibrated method.
6. Make one or more runs with the appropriate “checkout” sample used to both verify system performance for the given ASTM method, and to separately set up SimDis parameters and actions to be applied to analyses of your own laboratory samples.
7. See Chapter 3, “SimDis Data Analysis Tutorial”, for processes involved in working with SimDis to ensure it will properly analyze your laboratory samples data.
8. Run your laboratory samples using SimDis to analyze their resulting data.

Each of these steps is described in following sections. Before continuing, make sure the GC and the ChemStation PC are turned on, then start the ChemStation program.

1. Set Up ChemStation Report Files for SimDis

For the new method, do the following to enable production of ChemStation output files required by SimDis:

1 Open the ChemStation **Specify Report** view via the following steps:

   a Switch to **Data Analysis** view if not already there. Use main menu item **View** and then **2 Data Analysis** to switch views if needed.

   b In Data Analysis view, use main menu item **Report** followed by **Specify Report . . .** to open the **Specify Report** view (Figure 38).

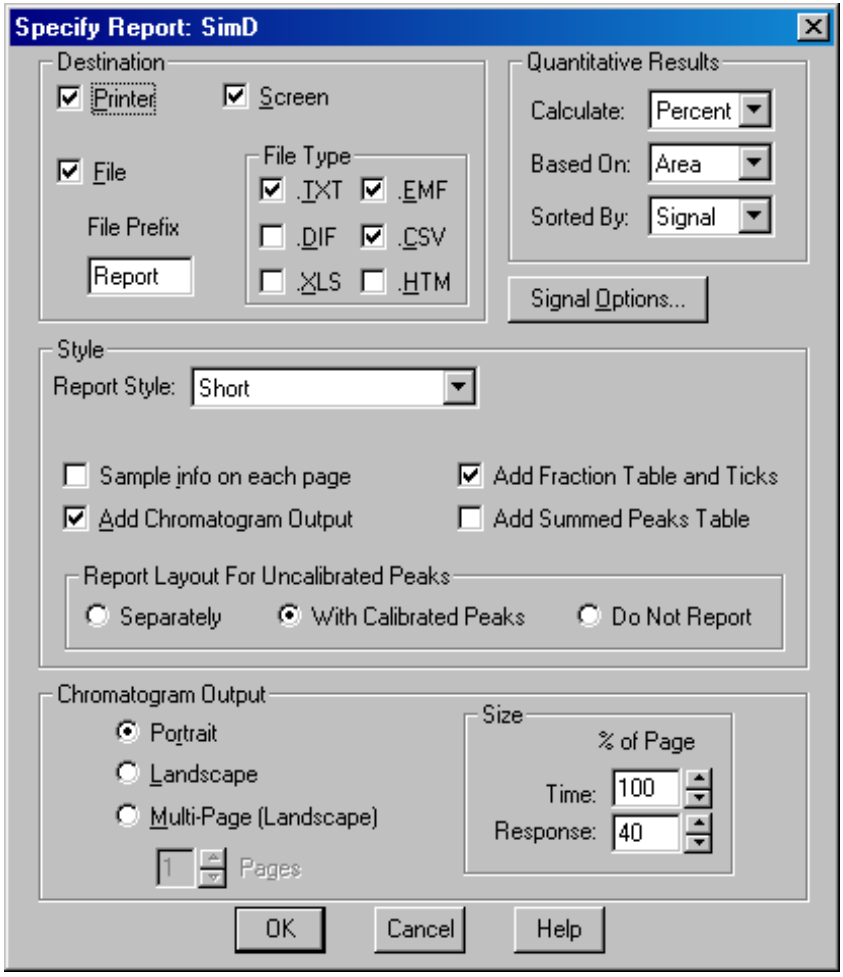


Figure 38 ChemStation Specify Report

2 In the **Destination** portion of the view (upper left corner), if not already so, enable (checkbox checked) all of the following:

- **Printer**
- **Screen**
- **File**

and, under **File Type**, enable (checkbox checked) all of the following:

- **.TXT**
- **.EMF**
- **.CSV**

File Prefix may remain as **Report** (default file name).

3 Verify, and if necessary change, **Quantitative Results** (upper right portion of the view) / **Calculate:** to **Percent** selected from the drop-down list of choices.

4 All other items may remain in their default states.

5 Close the view by selecting **OK**.

2. Add Appropriate Setpoints to the Method

Depending upon the particular ASTM method, choose the appropriate Table below and enter listed setpoints. For-

- ASTM D 2887, use Table 5
- ASTM D 2887x, use Table 6
- ASTM D 6352, use Table 7

If necessary, consult your ChemStation documentation with respect to where to locate and modify these many parameters.

NOTE Conditions listed here are to be considered only as reasonable starting points. You may need to adjust various parameters as necessary to "fine tune" your method.

Table 5 ASTM D 2887 ChemStation Method parameters

Parameter / Setting	Value
GC Conditions	
Oven	
Maximum temp	350 °C
Equilibration time	3.00 min
Initial temp	40 °C (On)
Initial time	0.00 min
Ramp 1	
Rate (1)	20.0 °C / min (on)
Final temp (1)	350 °C
Final time (1)	4.00 min
Run time	19.50 min
HT PTV Inlet	
Mode	Split
Initial temp	350 °C (On)

Table 5 ASTM D 2887 ChemStation Method parameters (continued)

Parameter / Setting	Value
Initial time	0.00 min
Ramp 1	
Rate (1)	0.0 °C / min (Off)
Cryo	Off
Cryo use temp	25 °C
Cryo timeout	30.00 min (On)
Cryo fault	On
Pressure	3.44 psi (On)
Split ratio	2:1
Split flow	28.0 mL / min
Total flow	44.8 mL / min
Gas saver	Off
Gas type	Helium
Split/Splitless Inlet (alternative choice)	
Mode	Split
Initial temp	325 °C (On)
Pressure	4.63 psi (On)
Split ratio	10:1
Split flow	130.0 mL / min
Total flow	144.9 mL / min
Gas saver	Off
Gas type	Helium
Column	
Capillary column	
Model number	Agilent 125-10HB DB-1
Max temperature	350 °C
Nominal length	10.0 m
Nominal diameter	530.00 µm

Table 5 ASTM D 2887 ChemStation Method parameters (continued)

Parameter / Setting	Value
Nominal film thickness	2.65 µm
Mode	Constant flow
Nominal init pressure	3.44 psi
Average velocity	95 cm / s
Inlet	Back Inlet
Outlet	Front ***or*** Back detector
Outlet pressure	Ambient
Detector	
Front ***or*** Back	FID
Temperature	375 °C (On)
Hydrogen flow rate	40 mL / min (On)
Air flow rate	450 mL / min (On)
Mode	Constant column + makeup flow rate
Makeup flow rate	45 mL / min (On)
Makeup gas type	Nitrogen
Flame	On
Electrometer	On
Lit offset	2.0
Signal	
Data rate	5 Hz
Type	Front ***or*** Back detector
Save data	On
Zero	0.0 (Off)
Range	0
Fast peaks	Off
Attenuation	0
Column compensation	derive from Front ***or*** Back detector

Table 5 ASTM D 2887 ChemStation Method parameters (continued)

Parameter / Setting	Value
Injector	
Sample Washes	5
Sample Pumps	3
* Injection Volume	0.1 µL
* Syringe Size	5 µL
PreInj Solvent A Washes	5
PreInj Solvent B Washes	5
PostInj Solvent A Washes	4
PostInj Solvent B Washes	4
† Viscosity Delay	0 s
Plunger Speed	Fast
PreInj Dwell	0.00 min
PostInj Dwell	0.00 min
ChemStation Conditions	
Run Time Checklist	
Pre-Run Cmd / Macro	Off
Data Acquisition	On
Standard Data Analysis	On
Customized Data Analysis	Off
Save GLP Data	Off
Post-Run Cmd / Macro	On
Name	DDEReport
Save Method with Data	Off
Injection Source and Location	
Injection Source	GC Injector
Injection Location	Back

Table 5 ASTM D 2887 ChemStation Method parameters (continued)

Parameter / Setting	Value
Integration Events	
Non signal specific Integration Events-	
Tangent Skim Mode	Standard
Default Integration Event Table "Event"-	
Initial Slope Sensitivity	1.000
Initial Peak Width	0.040
Initial Area Reject	1.000
Initial Height Reject	1.700
Initial Shoulders	Off
Detector Default Integration Event Table "Event_FID"-	
Initial Slope Sensitivity	50.000
Initial Peak Width	0.040
Initial Area Reject	100.000
Initial Height Reject	1.000
Initial Shoulders	Off
Apply Manual Integration Events	No
Calculation and Calibration	
Calculate	Area percent
Rel. Reference Window	3.000 %
Abs. Reference Window	0.000 min
Rel. Non-ref. Window	3.000 %
Abs. Non-ref. Window	0.000 min
Use Multiplier & Dilution Factor with ISTDs	
Uncalibrated Peaks	Not reported
Partial Calibration	Yes, identified peaks are recalibrated
Correct All Ret. Times	No, only for identified peaks
Curve Type	Linear
Origin	Included

Table 5 ASTM D 2887 ChemStation Method parameters (continued)

Parameter / Setting	Value
Weight	Equal
Recalibration Settings	
Average Response	Average all calibrations
Average Retention Time	Floating average new 75%
Calibration Report Options	
Printout of recalibrations within a sequence	Calibration table after recalibration
Normal Report after Recalibration	
If the sequence is done with bracketing	Results of first cycle (ending previous bracket)
Signal (1 ***or*** 2)	
FID1 (A ***or*** B)	
Peak Sum Table	No entries

* For calibration and/or laboratory samples, depending upon dilution, smaller injected amounts may be required, for example Injection Volume: 0.05 µL using the alternative 0.5-µL syringe provided.

† For samples run undiluted ("neat"), a greater setting may be necessary.

Table 6 ASTM D 2887x ChemStation Method parameters

Parameter / Setting	Value
GC Conditions	
Oven	
Maximum temp	450 °C
Equilibration time	0.50 min
Initial temp	45 °C (On)
Initial time	0.00 min
Ramp 1	
Rate (1)	15.0 °C / min (On)
Final temp (1):	430 °C
Final time (1)	4.00 min

Table 6 ASTM D 2887x ChemStation Method parameters (continued)

Parameter / Setting	Value
Run time	29.67 min
HT PTV Inlet	
Mode	Split
Initial temp:	75 °C (On)
Initial time:	0.00 min
Ramp	
Rate (1)	400.00 °C / min (On)
Final temp (1)	420 °C
Final time (1)	11.00 min
Ramp 2	
Rate (2)	200.00 °C / min (On)
Final temp (2)	75 °C
Final time (2)	0.00 min
Ramp 3	
Rate (3)	0.0 °C / min (Off)
Cryo	Off
Cryo use temp	25 °C
Cryo timeout	30.00 min (On)
Cryo fault	On
Pressure	4.6 psi (On)
Split ratio	1:1
Split flow	15.9 mL / min
Total flow	35.1 mL / min
Gas saver	
Gas type	Helium
Column	
Capillary column	
Model number	Agilent 145-simd DBHT-SIMD

Table 6 ASTM D 2887x ChemStation Method parameters (continued)

Parameter / Setting	Value
Max temperature	430 °C
Nominal length	5.0 m
Nominal diameter	530.00 µm
Nominal film thickness	0.15 µm
Mode	Constant flow
Initial flow	16 mL / min
Nominal init pressure	2.08 psi
Average velocity	122 cm / s
Inlet	Back Inlet
Outlet	Front ***or*** Back Detector
Outlet pressure	Ambient
Detector	
Front ***or*** Back	FID
Temperature	400 °C (On)
Hydrogen flow rate	40 mL / min (On)
Air flow rate	450 mL / min (On)
Mode	Constant column + makeup flow rate
Combined flow rate	45 mL / min (On)
Makeup gas type	Nitrogen
Flame	On
Electrometer	On
Lit offset	2.0
Signal	
Data rate	5 Hz
Type	Front ***or*** Back Detector
Save Data	On
Zero	0.0 (Off)

Table 6 ASTM D 2887x ChemStation Method parameters (continued)

Parameter / Setting	Value
Range	0
Fast Peaks	Off
Attenuation	0
Column Compensation	Derive from Front ***or*** Back Detector
Injector	
Sample Washes	5
Sample Pumps	3
* Injection Volume	0.1 µL
* Syringe Size	5 µL
PreInj Solvent A Washes	5
PreInj Solvent B Washes	5
PostInj Solvent A Washes	4
PostInj Solvent B Washes	4
† Viscosity Delay	0 s
Plunger Speed	Fast
PreInj Dwell	0.00 min
PostInj Dwell	0.00 min
ChemStation Conditions	
Run Time Checklist	
Pre-Run Cmd / Macro	Off
Data Acquisition	On
Standard Data Analysis	On
Customized Data Analysis	Off
Save GLP Data	Off
Post-Run Cmd / Macro	On
Name	DDEReport
Save Method with Data	Off

Table 6 ASTM D 2887x ChemStation Method parameters (continued)

Parameter / Setting	Value
Injection Source and Location	
Injection Source	GC Injector
Injection Location	Back
Integration Events	
Non signal specific Integration Events	
Tangent Skim Mode	Standard
Default Integration Event Table "Event"	
Initial Slope Sensitivity	5.000
Apply Manual Integration Events	No
Initial Peak Width	0.040
Initial Area Reject	10.000
Initial Height Reject	0.000
Initial Shoulders	Off
Detector Default Integration Event Table "Event_FID"	
Initial Slope Sensitivity	50.000
Initial Peak Width	0.040
Initial Area Reject	100.000
Initial Height Reject	1.000
Initial Shoulders	Off
Integration OFF at time 0.010 min	
Integration ON at time 6.600 min	
Calculation and calibration	
Calculate	Area percent
Rel. Reference Window	2.000 %
Abs. Reference Window	0.000 min
Rel. Non-ref. Window	1.500 %

Table 6 ASTM D 2887x ChemStation Method parameters (continued)

Parameter / Setting	Value
Abs. Non-ref. Window	0.000 min
Use Multiplier & Dilution Factor with ISTDs	
Uncalibrated Peaks	Not reported
Partial Calibration	Yes, identified peaks are recalibrated
Correct All Ret. Times	No, only for identified peaks
Curve Type	Linear
Origin	Included
Weight	Equal
Recalibration settings	
Average Response	Average all calibrations
Average Retention Time	Floating average new 75%
Calibration Report Options	
Printout of recalibrations within a sequence	Calibration Table after recalibration
Normal report after recalibration	
If the sequence is done with bracketing	Results of first cycle (ending previous bracket)
Signal (1 ***or*** 2)	
FID1 (A ***or*** B)	
Peak sum table	***No Entries in table***

* For calibration and/or laboratory samples, depending upon dilution, smaller injected amounts may be required, for example Injection Volume: 0.05 µL using the alternative 0.5-µL syringe provided.

† For samples run undiluted ("neat"), a greater setting may be necessary.

Table 7 ASTM D 6352 ChemStation Method parameters

Parameter / Setting	Value
GC Conditions	
Oven	
Maximum temp	450 °C
Equilibration time	0.50 min
Initial temp	45 °C (On)
Initial time	0.00 min
Ramp 1	
Rate (1)	15.0 °C / min (on)
Final temp (1)	440 °C
Final time (1)	4.00 min
Run time	30.33 min
HT PTV Inlet	
Mode	Split
Initial temp	60 °C (On)
Initial time	0.00 min
Ramp 1	
Rate (1)	400.00 °C / min (on)
Final temp (1)	425 °C
Final time (1)	11.00 min
Ramp 2	
Rate (2)	200.00 °C / min (on)
Final temp (2)	75 °C
Final time (2)	0.00 min
Ramp 3	
Rate (3)	0.0 °C / min (off)
Cryo	Off
Cryo use temp	25 °C
Cryo timeout	30.00 min (On)
Cryo fault	On

Table 7 ASTM D 6352 ChemStation Method parameters (continued)

Parameter / Setting	Value
Pressure	1.79 psi (On)
Split ratio	2:1
Split flow	28.0 mL / min
Total flow	44.8 mL / min
Gas saver	Off
Gas type	Helium
Column	
Capillary column	
Model number	Agilent 145-simd DBHT-SIMD
Max temperature	430 °C
Nominal length	5.0 m
Nominal diameter	530.00 µm
Nominal film thickness	0.15 µm
Mode	Constant flow
Nominal init pressure	2.1 psi
Average velocity	76 cm / s
Inlet	Back inlet
Outlet	Front ***or*** Back Detector
Outlet pressure	Ambient
Detector	
Front or Back	FID
Temperature	425 °C (On)
Hydrogen flow rate	40 mL / min (On)
Air flow rate	450 mL / min (On)
Mode	Constant column + makeup flow rate
Combined flow rate	45 mL / min (On)
Makeup Gas Type	Nitrogen
Flame	On

Table 7 ASTM D 6352 ChemStation Method parameters (continued)

Parameter / Setting	Value
Electrometer	On
Lit offset	2.0
Signal	
Data rate	5 Hz
Type	Front ***or*** Back Detector
Save Data	On
Zero	0.0 (Off)
Range	0
Fast Peaks	Off
Attenuation	0
Column Compensation	Derive from Front ***or*** Back Detector
Injector	
Sample Washes	5
Sample Pumps	3
* Injection Volume	0.1 µL
* Syringe Size	5 µL
PreInj Solvent A Washes	5
PreInj Solvent B Washes	5
PostInj Solvent A Washes	4
PostInj Solvent B Washes	4
† Viscosity Delay	0 s
Plunger Speed	Fast
PreInj Dwell	0.00 min
PostInj Dwell	0.00 min
ChemStation Conditions	
Run Time Checklist	
Pre-Run Cmd / Macro	Off

Table 7 ASTM D 6352 ChemStation Method parameters (continued)

Parameter / Setting	Value
Data Acquisition	On
Standard Data Analysis	On
Customized Data Analysis	Off
Save GLP Data	Off
Post-Run Cmd / Macro	On
Name	DDEReport
Save Method with Data	Off
Injection Source and Location	
Injection Source	GC Injector
Injection Location	Back
Integration Events	
Non signal specific Integration Events-	
Tangent Skim Mode	Standard
Default Integration Event Table "Event"-	
Initial Slope Sensitivity	5.000
Initial Peak Width	0.040
Initial Area Reject	7.000
Initial Height Reject	0.000
Initial Shoulders	Off
Detector Default Integration Event Table "Event_FID"-	
Initial Slope Sensitivity	50.000
Initial Peak Width	0.040
Initial Area Reject	100.000
Initial Height Reject	1.000
Initial Shoulders	Off
Integration	Off at time 0.010 min
Integration	On at time 6.400 min
Apply Manual Integration Events	No

Table 7 ASTM D 6352 ChemStation Method parameters (continued)

Parameter / Setting	Value
Calculation and Calibration	
Calculate	Area percent
Rel. Reference Window	0.500 %
Abs. Reference Window	0.000 min
Rel. Non-ref. Window	0.300 %
Abs. Non-ref. Window	0.000 min
Use Multiplier & Dilution Factor with ISTDs	
Uncalibrated Peaks	Not reported
Partial Calibration	Yes, identified peaks are recalibrated
Correct All Ret. Times	No, only for identified peaks
Curve Type	Linear
Origin	Included
Weight	Equal
Recalibration Settings	
Average Response	Average all calibrations
Average Retention Time	Floating average new 75%
Calibration Report Options	
Printout of recalibrations within a sequence	Calibration table after recalibration
Normal Report after Recalibration	
If the sequence is done with bracketing	Results of first cycle (ending previous bracket)
Signal (1 ***or*** 2)	
FID1 (A ***or*** B)	
Peak Sum Table:	***No entries in table***

* For calibration and/or laboratory samples, depending upon dilution, smaller injected amounts may be required, for example Injection Volume: 0.05 µL using the alternative 0.5-µL syringe provided.

Table 7 ASTM D 6352 ChemStation Method parameters (continued)

Parameter / Setting	Value
† For samples run undiluted ("neat"), a greater setting may be necessary.	

Ensure an initial uncalibrated method

Ensure that this new ChemStation Method you are building has no calibration table at this time and, if it does, delete it. From the ChemStation **Data Analysis** view:

1. Select its main menu item, **Calibration**.
2. From the drop-down list of choices, select **Delete Calibration Table**.
3. Select **Yes** to the verification message which may then appear.

   If no calibration table actually exists, you will simply get a message to that effect.

3. Save the “Base” Method

At this point you have a “base” method (no calibration information) to use as a starting point for making modified versions suitable for each different sample type run under your chosen ASTM method. Consider this to be a template from which you can generate copies as needed for the actual running of samples.

For that reason, we recommend that you **Save** this ChemStation Method as an easily recognizable name, for example as ‘**D2887.M**’, ‘**D2887(initial).M**’, or anything making sense in your environment.

Finally, note that you may find need to modify chromatographic parameters in the method in your own work versus startup values provided here. Make sure the “base” method is updated and saved accordingly to reflect such changes.

4. Run the Solvent Blank Sample

The CS_2 solvent blank sample is used in two different ways:

- To judge system cleanliness (visual baseline inspection only). This cleanliness check is done as required by laboratory protocol, as part of new column conditioning, and so forth.
- To create and store a baseline profile to be subtracted from other sample run data within the SimDis application.

The general procedure is to run the solvent blank sample repeatedly until the observed baseline is visually free of contaminant peaks. The last run of the set, typically the cleanest, is then separately selected and used within SimDis for baseline subtraction. Here are two typical solvent blank runs (Figure 39).

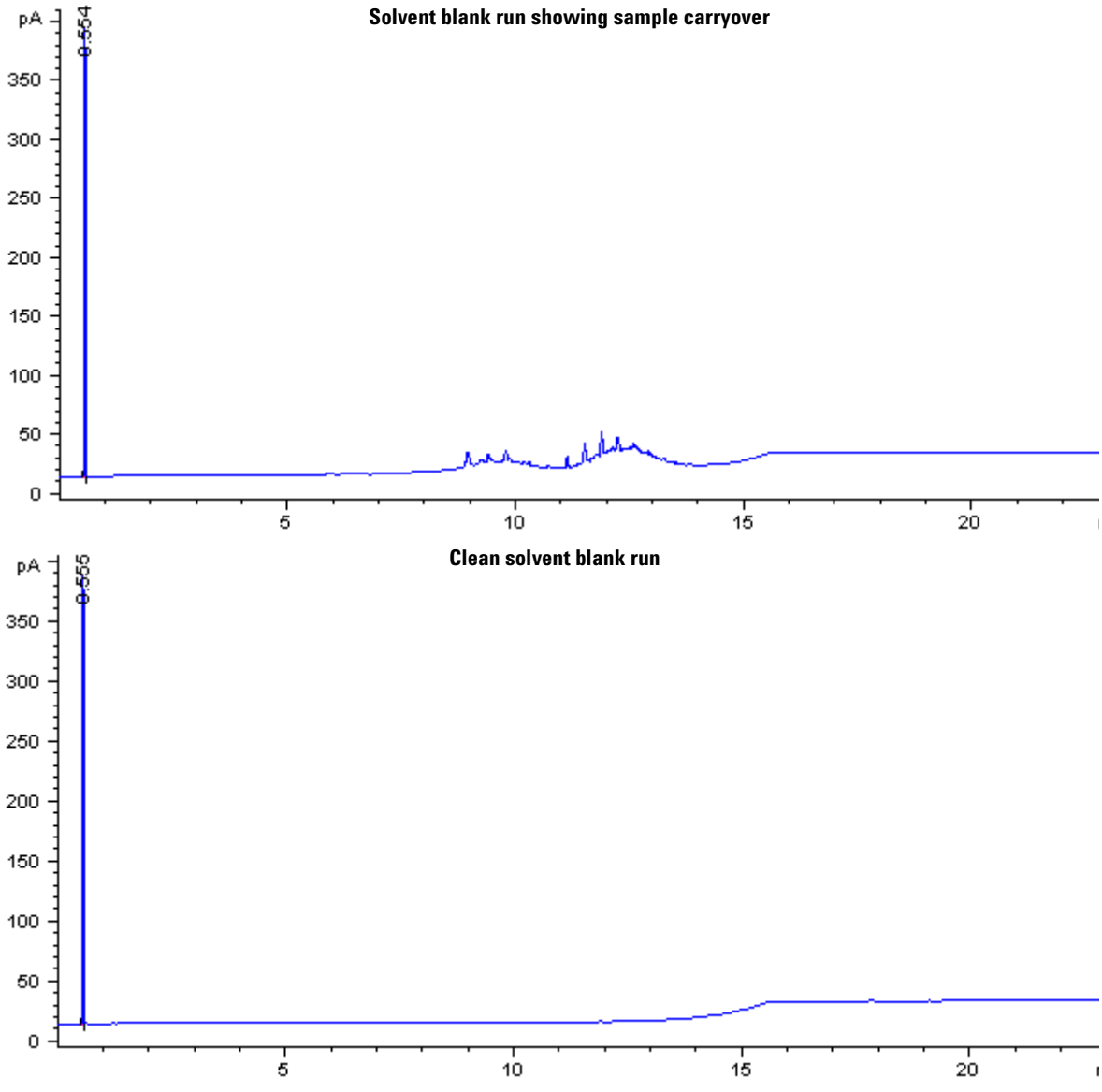


Figure 39 Example solvent blank runs

The first chromatogram in Figure 39 shows a typical contaminated solvent blank baseline profile: evidence of sample components remaining from previous analyse(s) are clearly visible.

The second chromatogram in Figure 39 shows a typical clean baseline profile suitable for use by SimDis in its analysis of samples.

If the system does not appear to clean itself out after repeated solvent blank runs, inlet maintenance may need to be performed. For the HT PTV inlet, refer to Chapter 8, “Maintenance”, for servicing information; refer to your GC user documentation for any other inlet type (for example, the Split/Splitless inlet).

Method modification- disabling ChemStation integration

Solvent blank runs need not be integrated as no component peaks need to be found and identified. To modify the method:

1 If not all ready done, select the ChemStation **Data Analysis** view.

2 Select **Edit / Set Integration Events Table**,

to bring the integration events table into view (Figure 40).

Large Initial Area Reject value

Integration Off at time 0

Time	Integration Events	Value
Initial	Slope Sensitivity	50
Initial	Peak Width	0.04
Initial	Area Reject	10000
Initial	Height Reject	1
Initial	Shoulders	OFF
0.000	Integration	OFF

Figure 40 ChemStation Integration Events Table

3 Edit the Integration Events Table such that either or both of the following are done:

- Set **Initial Area Reject** sufficiently high to exclude the solvent peak, and/or
- Switch off integration at the beginning of the run

If needed, consult your ChemStation documentation with respect to editing the Integration Events Table.

4 Given these modifications, you may now want to **Save** the method as an easily-recognized *new* name associated with running solvent blank samples in the future (for example, with the 32-bit ChemStation, as '**D2887(SolventBlank).M**').

Assuming GC, injector, and ChemStation are ready, you can now run solvent blank samples as needed under this method.

5. Run the Calibration Sample

With a presumably clean chromatographic system, the next stage is to run the calibration sample appropriate for the ASTM method being followed and to then modify the ChemStation Method by creating a calibration table associated with the sample:

1 Load the “base” method created and saved previously.

2 Assuming the calibration sample is ready (free of solids), run the sample in individual runs to “fine-tune” the method:

- If needed, adjust chromatographic parameters to optimize resultant chromatograms and Area% reports (all expected component peaks represented and well-resolved from each other and from the baseline)
- If needed, adjust injection volume and/or sample dilution ratio to increase areas of expected component peaks.

However, as you increase amounts injected, be very careful with regard to causing inlet / column overload, particularly for the HT PTV inlet, as serious long-term carryover contamination can occur.

CAUTION

As you increase amounts injected, be *very* careful with regard to causing inlet / column overload, particularly for the HT PTV inlet, as serious long-term carryover contamination can occur.

- Adjust **Initial Area Reject** as described in the previous section to exclude any small contaminant components (being careful to *not* exclude any desired component(s))
- As described in the previous section, you may switch off integration just before the solvent peak and return it to on just after to exclude its area contribution from the report

Ideally, your calibration sample Area% report should show *only* calibrant components provided in the sample.

NOTE Optimizing your chromatography using the calibration sample is worth the time spent: the better the condition of data available to the SimDis application, the less data manipulation necessary within SimDis.

3 With your chromatography optimized, use the Area% report from your last run to build a single-level calibration table as required by the SimDis application:

- If needed, refer to your ChemStation documentation for procedures in building a calibration table inside the method
- The SimDis application requires component peak naming to be of the following format:

  C< carbon number > with no spacing between 'C' and the carbon number.

  The format must be followed regardless of local language considerations.

See Table 8.

Table 8 Compound Naming, ChemStation Calibration Table

ASTM Method	ChemStation Calibration Table compound names
D 2887	C5, C6, C7, C8, C9, C10, C11, C12, C14, C15, C16, C17, C18, C20, C24, C26, C30, C36, and C40
D 2887x	C20, C22, C24, C26, C28, C30, C32, C34, C36, C38, C40, C42, C44, C46, C48, C50, C52, C54, C56, C58, and C60
D 6352	C20, C22, C24, C26, C28, C30, C32, C34, C36, C38, C40, C42, C44, C46, C48, C50, C52, C54, C56, C58, C60, C62, C64, C66, C68, C70, C72, C74, C76 C78, C80, C82, C84, C86, and C88

For ASTM Method D 2887x, C62 through C68, and C5 through C18 are optional components which may be added: see Chapter 4, "Sample Preparation", for more information.

Similarly, for ASTM Method D 6352, C5 through C18 are optional components which may be added: see Chapter 4, “Sample Preparation”, for more information. Also note that components C5 through C12, if added, are not resolved under conditions used here: they will elute as a single chromatographic peak.

- If you want to include one or more nontypical components, for example “C22” for ASTM Method D 2887, you must also supply the boiling point (in Fahrenheit) for each. The format is to have a space between the carbon number and its associated boiling temperature:

  C< carbon number > *space* < boiling temperature in °F >

- In building the table, give each component an amount of ‘ **1** ’ (unity)

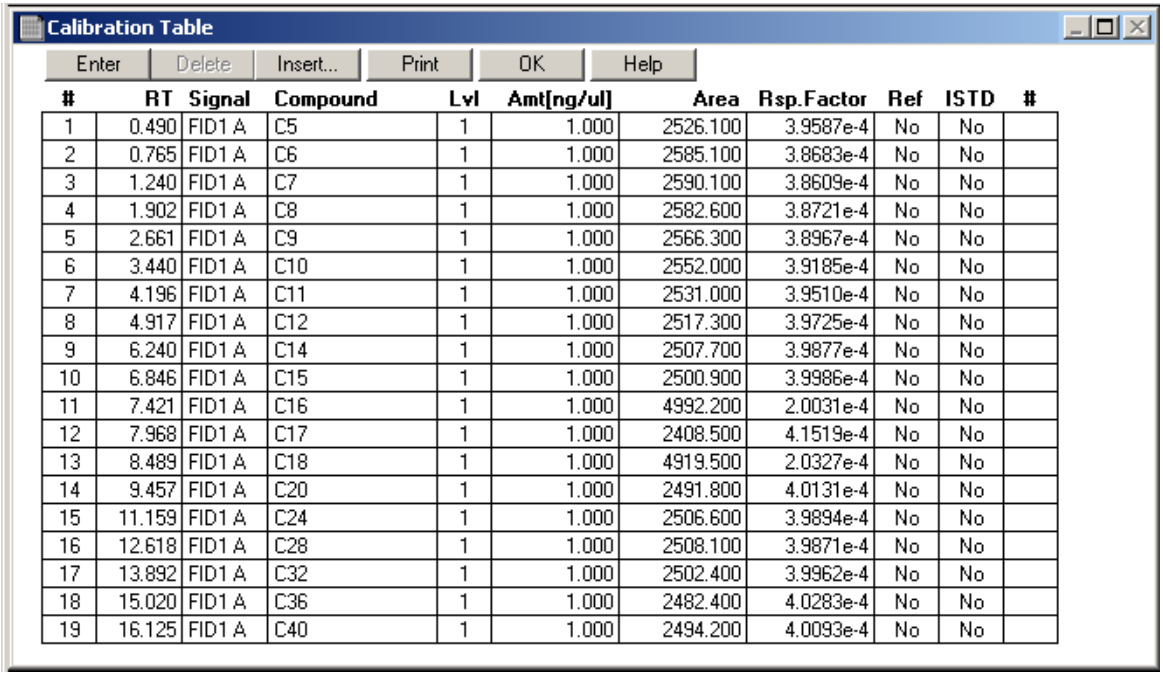
NOTE

Since integration processes are far more sophisticated within the ChemStation than within the SimDis application, it is strongly recommended that you build your calibration table inside the ChemStation as a matter of practice, as opposed to performing all your peak identifications inside SimDis. This is especially true for high temperature ASTM Methods D 2887x and D 6352 where the SimDis integrator may erroneously capture extra component peaks at the high-temperature end of the chromatogram.

Basically, consider the SimDis application to be a final editing tool to apply to your calibration runs only when necessary to repair minor problems.

If peaks *must* be identified entirely within SimDis, screening out unwanted high-temperature component peaks may be improved by doing a solvent blank subtraction from the calibration run via the **SimDis** tab.

Figure 41 is a typical example ChemStation calibration table created for ASTM Method D 2887:



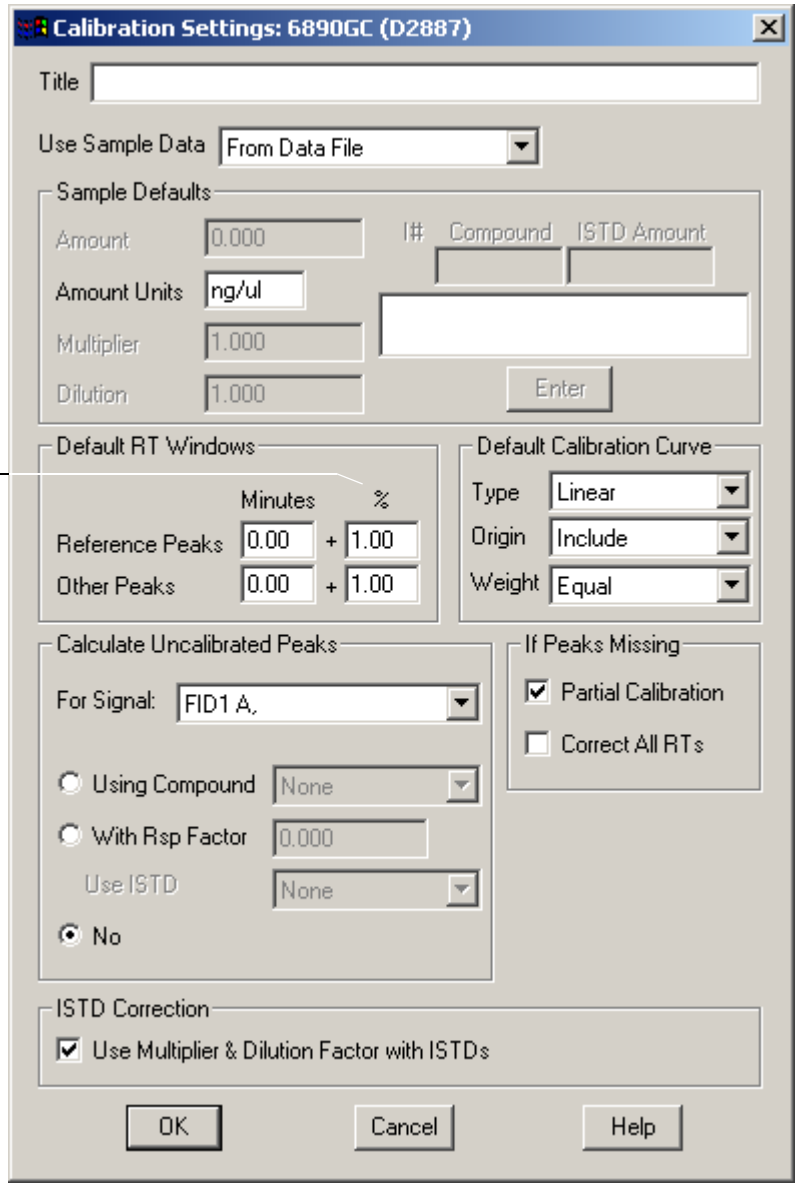
Calibration Table

Enter | Delete | Insert... | Print | OK | Help

#	RT	Signal	Compound	Lvl	Amt[ng/ul]	Area	Rsp.Factor	Ref	ISTD	#
1	0.490	FID1 A	C5	1	1.000	2526.100	3.9587e-4	No	No	
2	0.765	FID1 A	C6	1	1.000	2585.100	3.8683e-4	No	No	
3	1.240	FID1 A	C7	1	1.000	2590.100	3.8609e-4	No	No	
4	1.902	FID1 A	C8	1	1.000	2582.600	3.8721e-4	No	No	
5	2.661	FID1 A	C9	1	1.000	2566.300	3.8967e-4	No	No	
6	3.440	FID1 A	C10	1	1.000	2552.000	3.9185e-4	No	No	
7	4.196	FID1 A	C11	1	1.000	2531.000	3.9510e-4	No	No	
8	4.917	FID1 A	C12	1	1.000	2517.300	3.9725e-4	No	No	
9	6.240	FID1 A	C14	1	1.000	2507.700	3.9877e-4	No	No	
10	6.846	FID1 A	C15	1	1.000	2500.900	3.9986e-4	No	No	
11	7.421	FID1 A	C16	1	1.000	4992.200	2.0031e-4	No	No	
12	7.968	FID1 A	C17	1	1.000	2408.500	4.1519e-4	No	No	
13	8.489	FID1 A	C18	1	1.000	4919.500	2.0327e-4	No	No	
14	9.457	FID1 A	C20	1	1.000	2491.800	4.0131e-4	No	No	
15	11.159	FID1 A	C24	1	1.000	2506.600	3.9894e-4	No	No	
16	12.618	FID1 A	C28	1	1.000	2508.100	3.9871e-4	No	No	
17	13.892	FID1 A	C32	1	1.000	2502.400	3.9962e-4	No	No	
18	15.020	FID1 A	C36	1	1.000	2482.400	4.0283e-4	No	No	
19	16.125	FID1 A	C40	1	1.000	2494.200	4.0093e-4	No	No	

Figure 41 ChemStation Calibration Table (typical for ASTM Method D 2887)

4 Finally, as in Figure 42, also set ‘ **1%** ‘**Default RT Windows** (**Calibration / Calibration Settings . . .**) and select **OK** to accept the change:



1% Default RT Windows

Figure 42 ChemStation Calibration Settings (Default RT Windows)

5 Print a ChemStation report to confirm that all components are correctly identified.

6 Finally, given these modifications made to the ChemStation Method, **Save** the method as an easily-recognized *new* name associated with running calibration samples in the future (for example, with the 32-bit ChemStation, as '**D2887(Calibration).M**').

7 If you have modified setpoint(s) affecting the chromatography (temperatures, gas flow rates, and so forth), *and* if you have created separately a "base" method and/or solvent blank method, make sure you update them with these same revised setpoint(s) such that identical chromatographic behavior may be presumed for all methods created thus far.

6. Run the "Checkout" Sample

The "checkout" sample, prepared as described in Chapter 4, "Sample Preparation", serves two purposes:

- To establish suitable SimDis parameters for optimal analysis of your own laboratory samples under your chosen ASTM method
- To validate your system with respect to its capability to achieve results conforming to requirements of your chosen ASTM method

With respect to the ChemStation Method, since there is no need for discrete peak identification, there is therefore no need for either signal integration or for existence of a calibration table. To that end:

1 Load the method used for your solvent blank as integration is explicitly turned off (via an integration event) and no calibration table exists.

2 **Save** this method as an easily-recognized *new* name associated with running "checkout" samples in the future (for example, with the 32-bit ChemStation, as '**D2887(Checkout).M**').

As desired, make one or more runs of your “checkout” sample.

Carefully review your results to make sure they conform to the requirements for the ASTM Method you are using:

- For ASTM Method D 2887:

  see Table 9, extracted from “TABLE 3 Test Method D 2887 Reference Gas Oil No. 1” found in ASTM document *D 2887 - 97a, Standard Test Method for Boiling Range Distribution of Petroleum Fractions by Gas Chromatography*

- For ASTM Method D 2887x:

  at time of printing, there is no specific information available as to conformance standards for this proposed ASTM Method.

- For ASTM Method D 6352:

  see Table 10, extracted from “TABLE 2 Test Method D 6352 Reference Material 5010” found in ASTM document *D 6352 - 02, Standard Test Method for Boiling Range Distribution of Petroleum Distillates in Boiling Range from 174 to 700°C by Gas Chromatography*

Table 9 D 2887 Allowable Difference information from TABLE 3, ASTM D 2887 Method documentation

	Batch 2		Allowable Difference	
% Off	°C	°F	°C	°F
IBP	115	240	7.6	13.7
5	151	304	3.8	6.8
10	176	348	4.1	7.4
15	201	393	4.5	8.1
20	224	435	4.9	8.7
25	243	470		
30	259	499	4.7	8.4

Table 9 D 2887 Allowable Difference information from TABLE 3, ASTM D 2887 Method documentation (continued)

	Batch 2		Allowable Difference	
% Off	°C	°F	°C	°F
35	275	527		
40	289	552	4.3	7.7
45	302	576		
50	312	594	4.3	7.7
55	321	611		
60	332	629	4.3	7.7
65	343	649		
70	354	668	4.3	7.7
75	365	690		
80	378	712	4.3	7.7
85	391	736		
90	407	764	4.3	7.7
95	428	803	5.0	9.0
FBP	475	888	11.8	21.2

Table 10 D 6352 Allowable Difference information from TABLE 2, ASTM D 6352 Method documentation

% OFF	Average °F	95.5% CL, °F Allowable Difference	Average °C	95.5% CL*, °C Allowable Difference
IBP	801	16	428	9
5	891	5	477	3
10	918	5	493	3

Table 10 D 6352 Allowable Difference information from TABLE 2, ASTM D 6352 Method documentation (continued)

% OFF	Average °F	95.5% CL, °F Allowable Difference	Average °C	95.5% CL*, °C Allowable Difference
15	936	5	502	3
20	950	6	510	3
25	963	6	518	4
30	975	7	524	4
35	987	7	531	4
40	998	8	537	4
45	1008	8	543	4
50	1019	8	548	5
55	1030	8	554	4
60	1040	8	560	4
65	1051	8	566	4
70	1062	8	572	4
75	1073	9	578	5
80	1086	8	585	4
85	1099	7	593	4
90	1116	8	602	4
95	1140	7	616	4
FBP	1213	32	655	18

* CL Confidence Level

7. Establish Appropriate SimDis Parameters for Your Own Samples

With ChemStation solvent blank, calibration, and "checkout" runs now made, and following the general processes described in Chapter 3, "SimDis Data Analysis Tutorial", work within the SimDis application to set its parameters appropriately to insure your laboratory samples will be analyzed properly.

8. Run Your Laboratory Samples

The ChemStation Method used for your "checkout" sample is also appropriate for running your own laboratory samples under the chosen ASTM method. If desired, **Save** the "checkout" method as an easily-recognized *new* name associated with running your own samples in the future (for example, with the 32-bit ChemStation, as '**D2887(Samples).M**').

You are now ready to make one or more runs using your own laboratory samples and subsequently to analyze them using the SimDis application.

Automate Your Sample Analyses

Typically, you will want to run your laboratory samples such that SimDis analysis and reporting is performed automatically at the end of each ChemStation run. This is done by creating a ChemStation "Sequence" of sample runs. This topic is described separately in Chapter 6, "Automated SimDis Operation".

6
Automated SimDis Operation

This chapter describes how to set up ChemStation and SimDis applications to automatically perform SimDis analysis and reporting at the end of each run. Information here applies to all ChemStation Methods to be used with SimDis regardless of the specific ASTM method.

Overview

Automating the SimDis application requires “attaching” it to a ChemStation Method as a Post-Run application. With this done:

1. The ChemStation internally starts SimDis at the end of its own processes for each given run.
2. As SimDis opens, it finds the current run data, applies your previously-defined SimDis settings to it, prints the report previously selected, and closes itself.
3. With SimDis closed, control returns to the ChemStation allowing it to continue to the next run (within a Sequence), or to wait for you to manually initiate some activity.

From the ChemStation point-of-view, with SimDis attached to a given method as a Post-Run application, every run, whether done individually or from within a Sequence, will trigger SimDis to generate a report for the current run data.

Set Up the ChemStation Method

For method(s) where you want SimDis to automatically start at the end of a run, two things must be done: one to provide run information SimDis needs, and the other to automatically start SimDis at the end of the run:

1 Load the method to be modified.

2 To ensure SimDis receives necessary run information, you likely have already done this as a setup step described in section "1. Set Up ChemStation Report Files for SimDis" on page 106. Refer to that section to either verify this step was done, or to make the necessary changes.

3 To ensure SimDis automatically starts at the end of every run under the method, do the following (Figure 43).

   a In ChemStation **Method & Run Control** view, select main menu item **Method** followed by **Run Time Checklist**.

   b Enter **DDEReport** (no spaces and case sensitive: type it as shown) into the **Post-Run Command / Macro** field and enable (checkbox checked) the function.

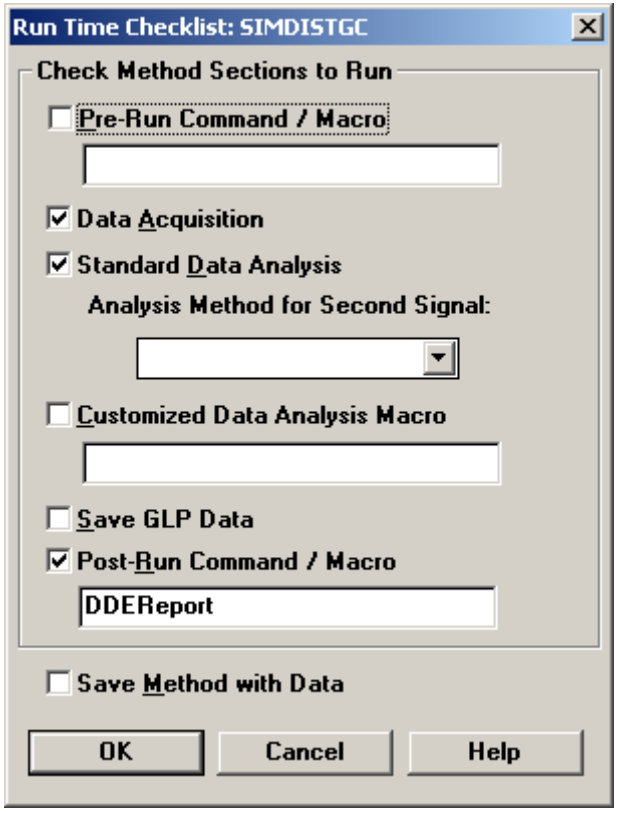


Figure 43 Setup for SimDis post-run processing

Other items above the **Post-Run Command / Macro** area should remain unchanged.

c Select **OK** to accept the modification.

4 **Save** the method.

Some notes:

- These modifications must be made to *every* method where you intend to have SimDis automatically start at the end of any run made by that method.
- As a convenience, the **Post-Run Command / Macro** checkbox may be unchecked (disabled) if you wish to "disconnect" SimDis such that it no longer starts automatically. **DDEReport** remains in the field. **Save** the method if you want this change in behavior to be kept.
- The checkbox and text, **Save Method with Data**, if enabled (checkbox checked), causes the ChemStation to save a *copy* of the method within data-containing folders created in the running of samples under that method. This feature is useful in absolutely associating a given method with any sample data it generates.

Associating SimDis to Correctly Analyze and Report Your Run Data

When SimDis is started by the ChemStation at the end of a run, it applies its current internal settings (via its **Setup**, **SimDis**, and **Report** tabs) to the ChemStation-generated run data. It is critically important, therefore, to be sure SimDis conditions are appropriate for the chosen ChemStation Method and samples to be run under that method. This is true regardless of the way in which the given run is made (inside or outside a Sequence).

There are two ways in which to establish the desired correct association:

- To select appropriate data files and set them as defaults from within SimDis and then, separately, set the ChemStation to use SimDis defaults, *OR*
- To explicitly select appropriate data files from within the ChemStation to be used by SimDis.

Path 1: Setting appropriate default data files within SimDis

This choice is essentially the process as described in Chapter 3, “SimDis Data Analysis Tutorial”: one of locating and selecting appropriate solvent blank, calibration, and “checkout” sample files within SimDis, setting SimDis parameters as needed, and then saving everything as defaults. Briefly, from within SimDis:

1. Use the **Browse** tab to locate and to view your desired solvent blank, calibration, and “checkout” data file sets.
2. Use the **Setup** tab to specifically select calibration and solvent blank data file sets:
   a. As needed, set parameters and/or modify their treatment by SimDis.
   b. Use the **Save As Default** button (**Overview** subtab) to store both paths and settings applied to these files.

3 Use the **SimDis** tab to specifically locate your appropriate "checkout" sample (see "Default Sample buttons" on page 78):

   a As needed, set parameters and/or modify treatment of the data by SimDis.

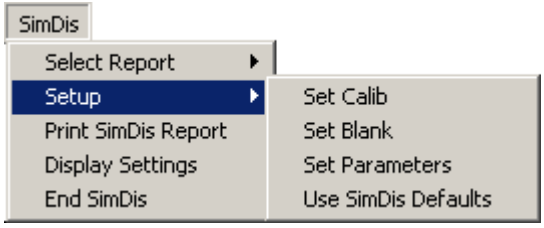
   b Use the **Use This Sample As Default** button to store both the path and settings applied to this file.

Having made your data file selections, set appropriate treatment parameters, and saved this collection of information as defaults, you can now turn to the ChemStation:

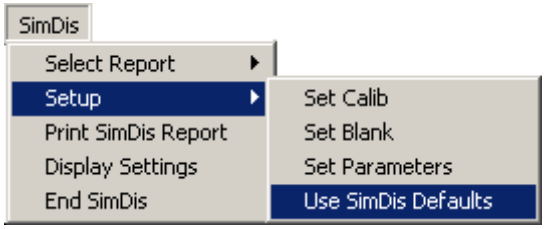
1 If needed, start the ChemStation, and switch to its **Data Analysis** view.

2 If needed, start SimDis from the ChemStation main menu item **SimDis**:

3 With SimDis running, return to ChemStation main menu item **SimDis** followed by **Setup**:



4 Finally, select **Use SimDis Defaults**:



NOTE For *single*-GC systems (most situations), this is the recommended path to follow in associating automated SimDis analysis and reporting with ChemStation run data.

NOTE From this point forward, *every* ChemStation run you make, *regardless* of ChemStation Method used, if SimDis is started by the method, resultant run data will be analyzed using the same SimDis default information unless or until it is subsequently changed and defaults updated.

Path 2: Setting appropriate default data files within the ChemStation

NOTE The following process does *not* bypass the requirement that you have already used SimDis to *separately* locate, select, and, if needed, modify SimDis treatments of the selected files. The assumption in this choice is that, at some previous time, you have already "touched" the files with SimDis which you will now select via the ChemStation.

Having at some time *separately* used SimDis to make your data file selections, and to set appropriate treatment parameters for them, you can use the ChemStation to select appropriate data files for SimDis to use:

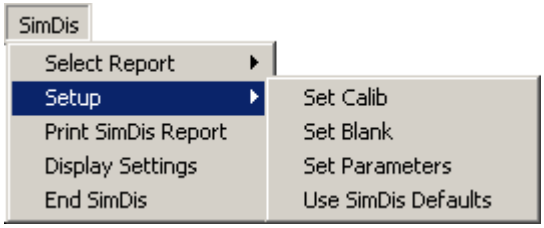
1 If needed, start the ChemStation, and switch to its **Data Analysis** view.

2 If needed, start SimDis from the ChemStation main menu item **SimDis**:

3 Select ChemStation main menu item **File** followed by **Load Signal . . . .**

4 Locate, select and load the calibration run data file you wish to use.

5 With SimDis running, return to ChemStation main menu item **SimDis** followed by **Setup**:



6 Select **Set Calib**. A confirming message appears showing the full path to the loaded file.

7 In turn, repeat steps **3** through **6** to load and set the solvent blank data file you wish to use (**Set Blank**), and then the "checkout" sample data file you wish to use (**Set Parameters**).

NOTE

This process *must* be used as part of setting up multi-GC ChemStation systems. See "Multi-GC ChemStation considerations" on page 151 for detail.

NOTE From this point forward, *every* ChemStation run you make, *regardless* of ChemStation Method used, if SimDis is started by the method, resultant run data will be analyzed using the same SimDis information unless or until it is subsequently changed by selecting different solvent blank, calibration, and/or "checkout" data file sets.

ChemStation Method considerations

SimDis **Setup** selections you make are not stored as part of a ChemStation Method. They are kept as part of the SimDis environment and applied only when SimDis is started, either manually or as a Post-Run application by a ChemStation Method.

ChemStation Sequence considerations

In setting up a ChemStation Sequence to run a group of samples sequentially, recognize that all samples chosen to be within the group will be analyzed and reported by SimDis using the same **Setup** selections you have made. There is no way, within a given sequence of samples, to switch SimDis **Setup** choices: for example, you cannot have a Sequence composed of both D 2887 and D 6352 samples.

Additionally, while your runs are in progress, do not make changes to parameters and/or saving of different values as defaults within SimDis which could alter SimDis analysis of your samples. Neither the ChemStation nor SimDis prevents you from making inappropriate changes "on the fly" while runs are in progress.

NOTE SimDis is not supported with respect to *offline* ChemStation operation.

Multi-GC ChemStation considerations

It is important to know that SimDis provides only *one default* set of sample information. Thus:

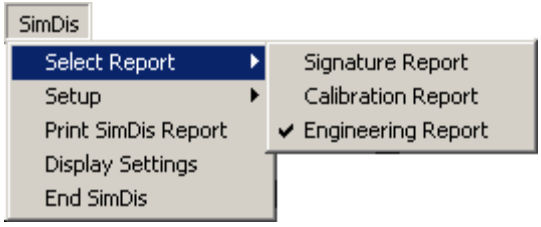
- For a multi-GC ChemStation situation, in *only one* ChemStation Instrument session, may you select **Use SimDis Defaults** (**SimDis / Setup** menu items)
- For other configured Instrument session(s), you *must separately* and *explicitly* select an appropriate file for **Set Calib, Set Blank**, and **Set Parameters** (**SimDis / Setup** menu items) for each Instrument session

Other ChemStation SimDis menu items

Remaining choices available via the ChemStation **SimDis** main menu item include:

Select Report –

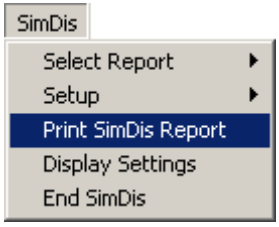
Use this to set the report type to be printed at the end of each run:



NOTE From this point forward, *every* ChemStation run you make, *regardless* of ChemStation Method used, if SimDis is started by the method, the resultant report will be of the selected type unless or until a different selection is made.

Print SimDis Report –

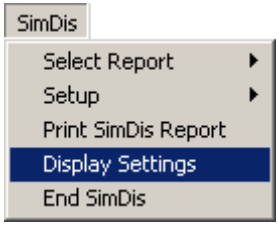
Immediately prints a SimDis report of the type chosen in **Select Report**:



NOTE Data files must be in the standard ChemStation folders, for example in **\HPCHEM\1\DATA** {16-bit ChemStation, Instrument '**1**'}, or in **\Chem32\1\DATA** {32-bit ChemStation, Instrument '**1**'}.

Display Settings –

Displays data file choices made via **Setup**:



- Figure 44 is an example of what is shown if **Use SimDis Defaults** is selected
- Figure 45 is an example of what is shown if **Set Calib**, **Set Blank**, and **Set Parameters** are used

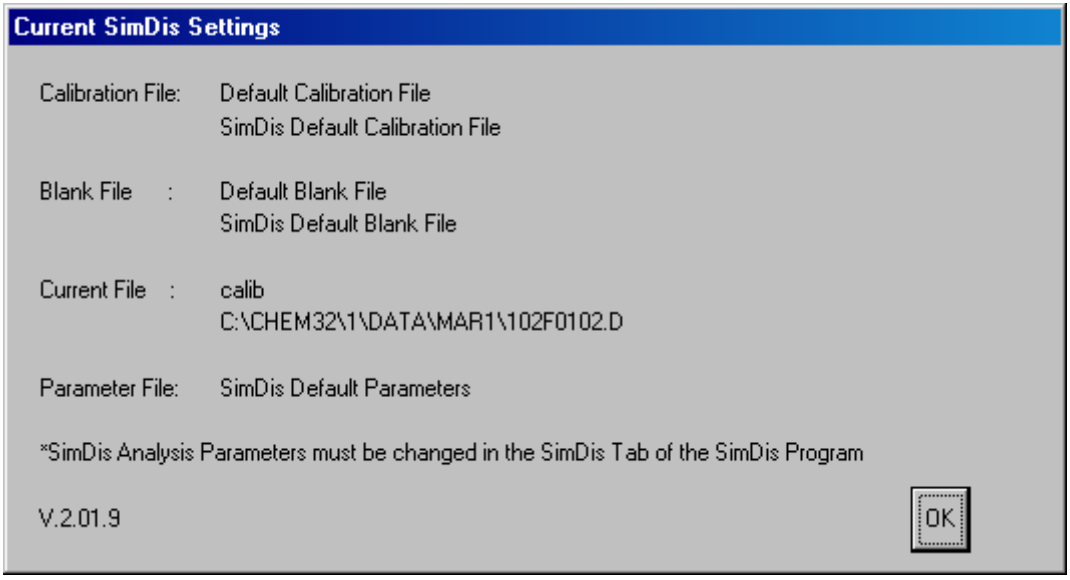


Figure 44 Typical **SimDis** / **Display Settings** (**Setup** / **Use SimDis Defaults** used)

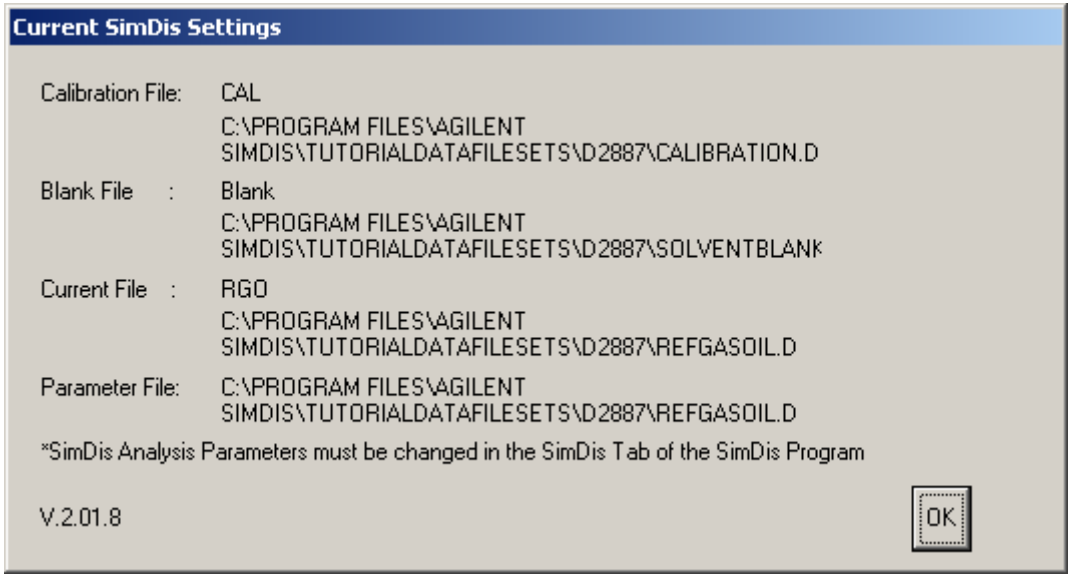
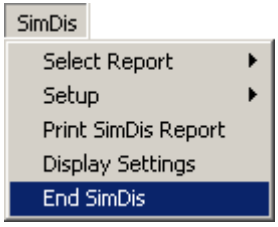


Figure 45 Typical **SimDis** / **Display Settings** (**Setup** / **Set Calib**, **Set Blank**, and **Set Parameters** used)

Display Settings provides opportunity to carefully review your choices and, if necessary, select different ones.

End SimDis –

Terminates SimDis:



ChemStation – SimDis Association Completed

This completes setting up association between runs to be made by the ChemStation and SimDis behavior applied automatically to ChemStation data for each of those runs.

Whether as individual ChemStation runs, or as groups of samples run in sequences, as each run finishes at the ChemStation, SimDis automatically starts and applies your chosen settings to the run's data to produce your selected SimDis report.

SimDis Automation Tab

The SimDis application provides a monitoring function, accessed via its **Automation** tab, useful when groups of samples are run from within ChemStation Sequences.

Anytime while a ChemStation Sequence is running, bring the SimDis application into view and select its **Automation** tab to display real-time information about the process:

Depending upon how your ChemStation is configured with respect to Instruments, and how you have made your **SimDis** / **Setup** choices, one of several possible styles will be seen:

- Figure 46 is an example of a single-Instrument system where **Use SimDis Defaults** has been selected
- Figure 47 is an example of a single-Instrument system where explicit **Set Calib**, **Set Blank**, and **Set Parameters** selections are used

- Figure 48 is an example of a multi-Instrument system where **Use SimDis Defaults** has been selected for one Instrument and explicit **Set Calib**, **Set Blank**, and **Set Parameters** selections are used for the second Instrument
- Figure 49 is an example of a multi-Instrument system where explicit **Set Calib**, **Set Blank**, and **Set Parameters** selections are used for *both* Instrument

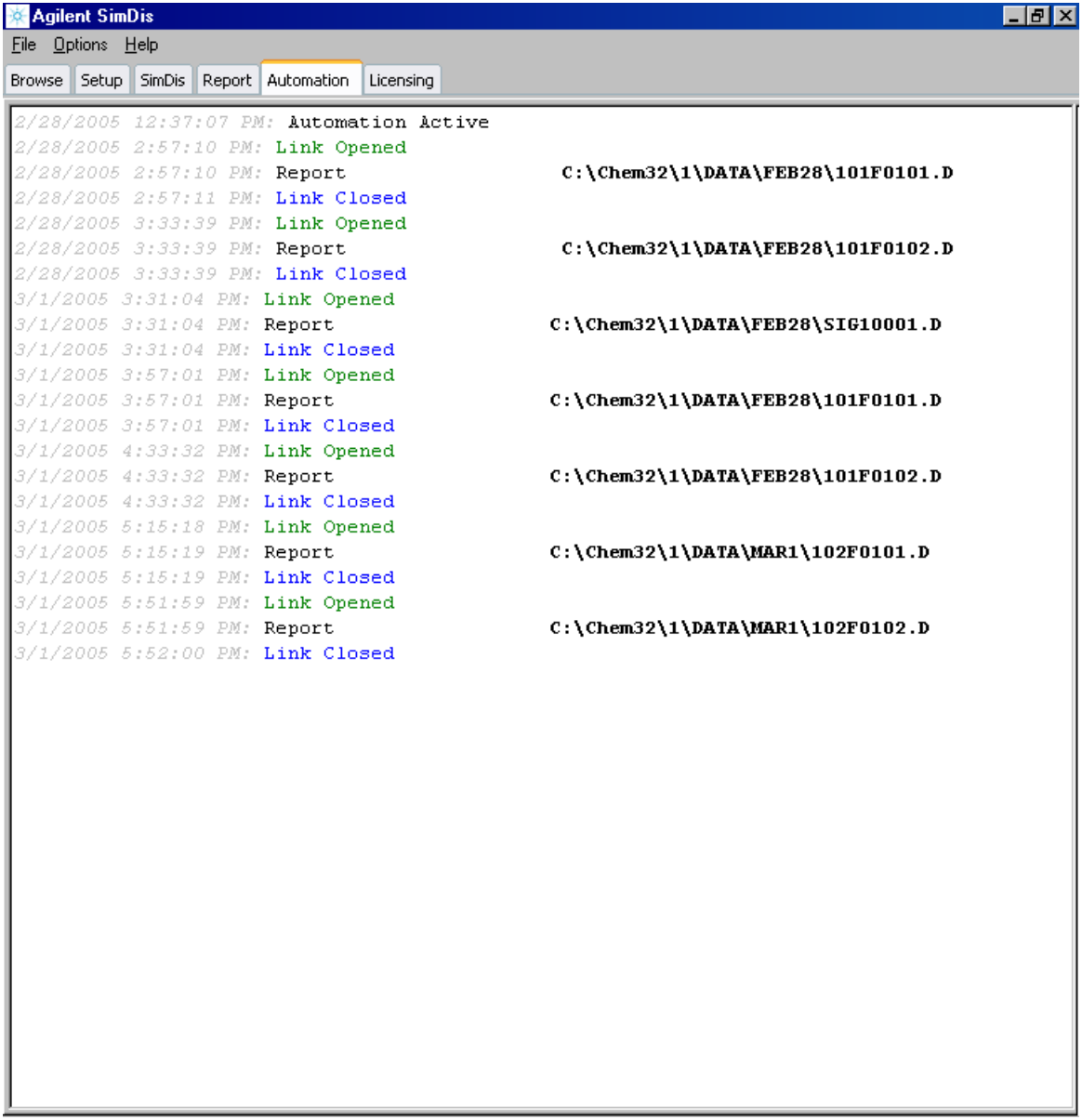


Figure 46 One Instrument configured and **Setup** / **Use SimDis Defaults** selected

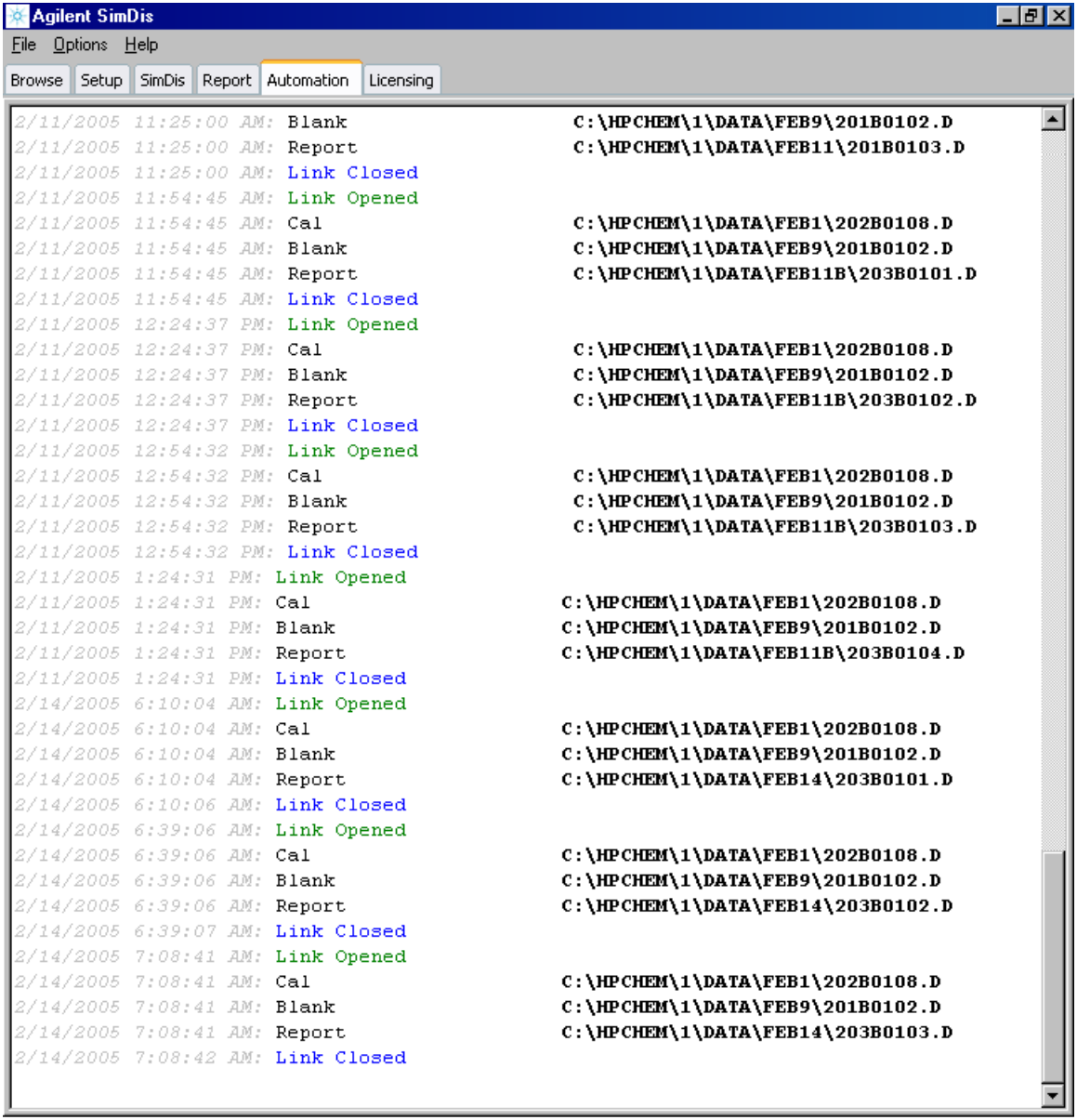


Figure 47 One Instrument configured and **Setup** / **Set Calib**, **Set Blank**, and **Set Parameters** explicitly selected

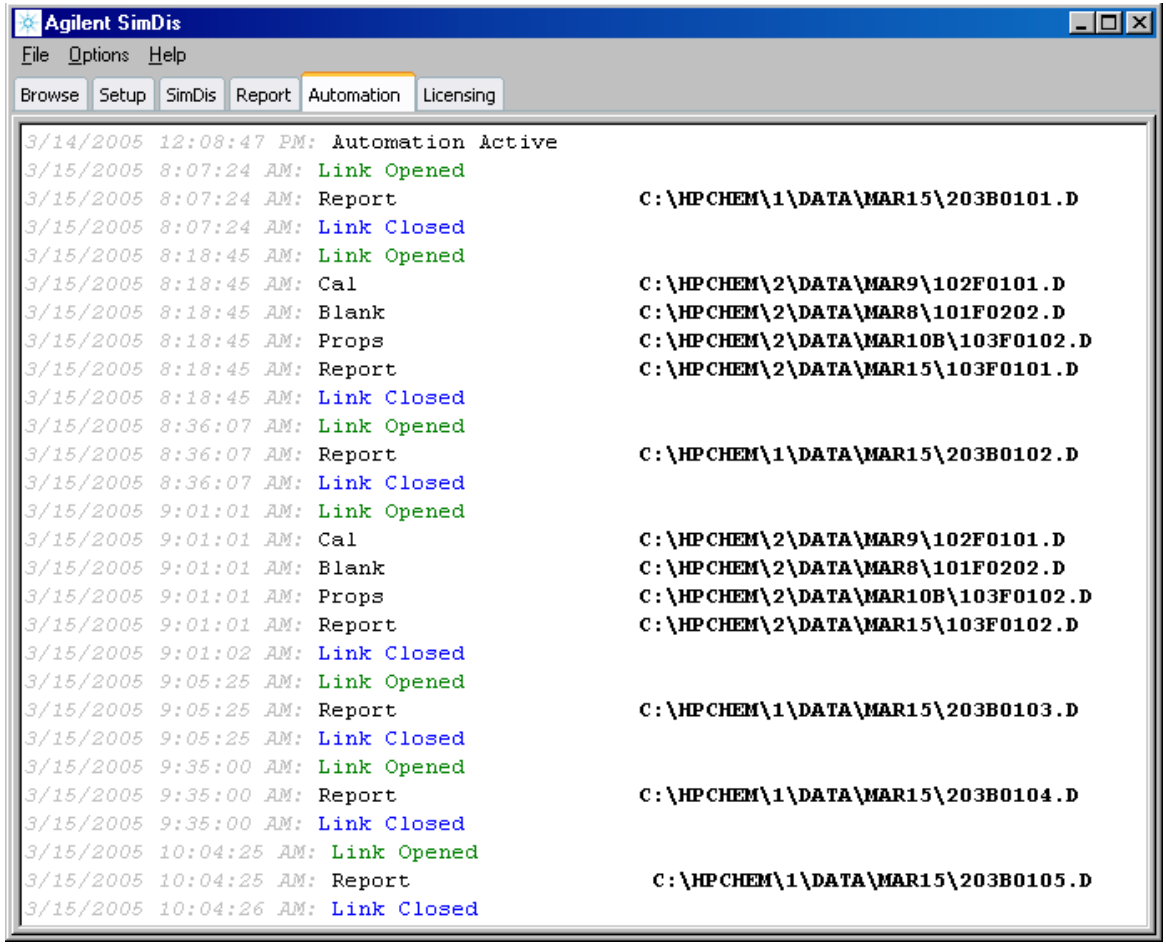


Figure 48 Two Instruments configured and **Setup** / **Use SimDis Defaults** selected for **Instrument 1** and **Set Calib**, **Set Blank**, and **Set Parameters** explicitly selected separately for **Instrument 2**

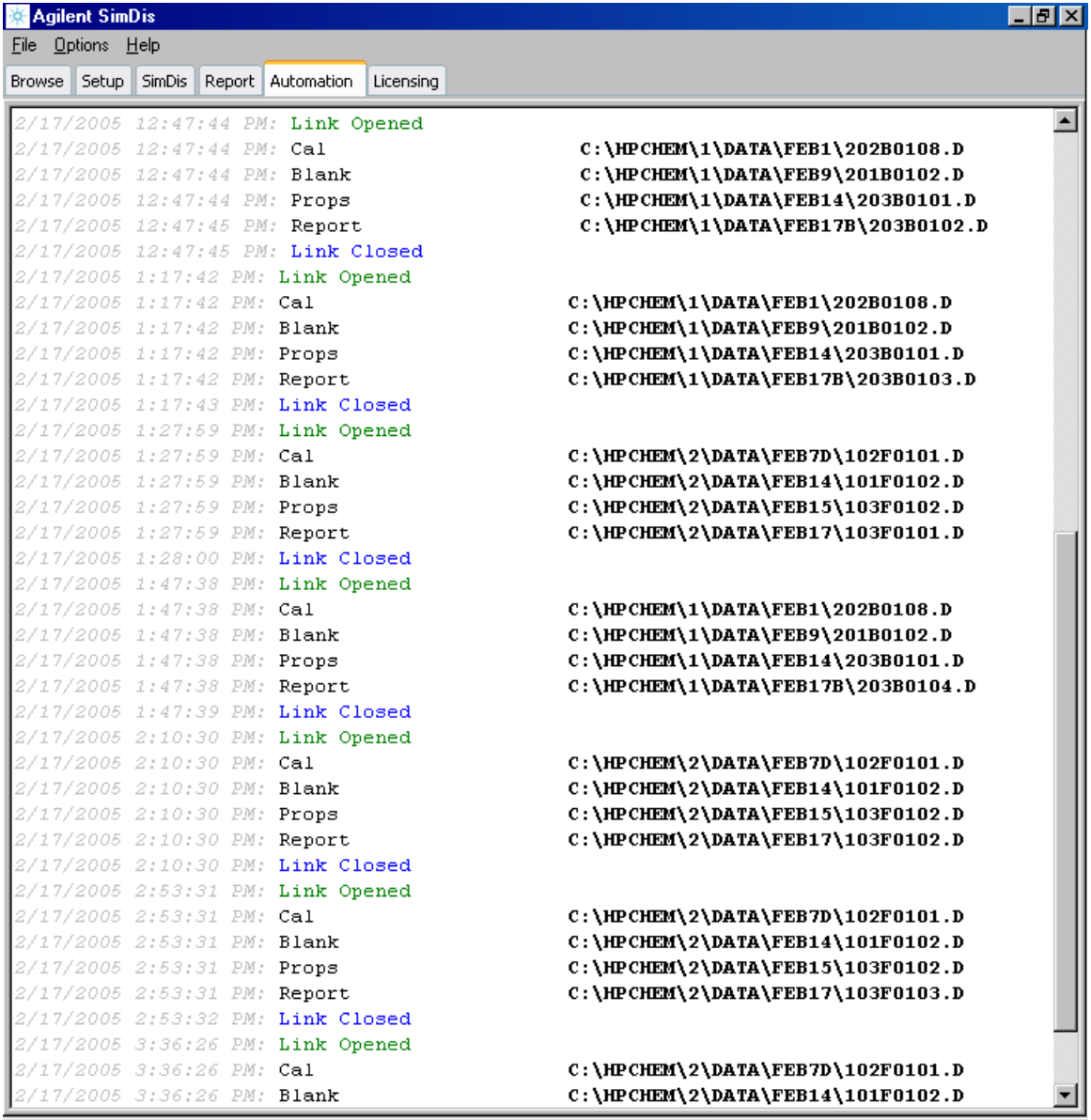


Figure 49 Two Instruments configured and **Setup** / **Set Calib**, **Set Blank**, and **Set Parameters** explicitly selected separately for each Instrument channel

In Figure 48 and Figure 49, note that there are two ChemStation Instrument sessions, “**\1**” and “**\2**”, both of which are running samples separately as part of their respective sequences.

Displayed information may be copied and pasted to, for example, an open word processing document but may not be printed directly.

For troubleshooting purposes, if ChemStation Sequence operation should halt prematurely, the displayed information will show you that and you should then review ChemStation logs for detail as to why the halt occurred.

7
Software Reference

This chapter summarizes common tasks and the functions available in the SimDis software.

Overview

The Agilent SimDis software user interface consists of a menu bar and six tab controls:

- Browse
- Setup
- SimDis
- Report
- Automation
- Licensing

Each of these is described later in this chapter.

Common Tasks

To make an entry

Many settings can be made by either direct typing, for example a retention time, or by selecting the entry field then double-clicking in the displayed chromatogram.

For example, from the SimDis pane, if using **Mask to Value** to eliminate a solvent peak appearing from 0.3 to 0.6 minutes, either:

- Select the **Mask End** field and enter a value of **0.6**, or
- Select the **Mask End** field, then double-click the chromatogram at a point just after the solvent peak ends. The retention time at that point is entered into the **Mask End** field.

This technique is particularly useful when displaying the calibrated temperatures instead of retention times.

To select a data file

When selecting a data file (whether for analysis as a sample or as a blank or calibration run), it immediately becomes active. This makes it very easy to analyze new data or test the impact of different blank or calibration runs on a sample file.

For sample data

1 Go to **Browse**, locate the sequence folder containing the run data, then click the sample data file in the file list.

Alternately, select the sample data from the Sample list on the **SimDis** tab.

2 Verify the correct settings are loaded. See the **Setup** tab.

- Samples which already have settings will use them instead of the current setup
- Samples with no settings will use the defaults

For calibration and blank data

1. Go to **Setup**, then click **Calibration** or **Blank**, as needed.
2. Click **Select**, then locate the sequence freeloader containing the run data.
3. Click the sample data file in the file list.

To add a calibrated peak

To add a peak to a calibration:

1. Go to **Setup** / **Calibration** / **Edit**.
2. Right-click the peak to add to the calibration and select **Add Peak**.

To delete a peak from a calibration

To delete a peak from a calibration:

1. Go to **Setup** / **Calibration** / **Edit**.
2. Right-click the calibrated peak **label** and select **Delete Peak**.

To change a peak's information

To add, change, or delete a peak's carbon number:

1. Go to **Setup** / **Calibration** / **Edit**.
2. Right-click the calibrated peak's **label**.
3. Select the carbon number to apply or select **No Carbon Number** to remove the current carbon number association.

Accept or rejecting a change

After making certain changes, SimDis prompts to accept or reject the change. Note that accepting a change means that the change will be used for processing, but does not also save it in any default or custom parameters file.

To find more information

For more information, see Chapter 3, “SimDis Data Analysis Tutorial”.

File and Options Menus

This section lists and briefly summarizes the commands available from the SimDis File and Options menus described in Table 11.

Table 11 File and Options menu commands

Command	Comment
File	
• Printer Setup	Use to select a printer.
• Exit	Exits SimDis, not ChemStation.
Options	
• Display Temperatures in Celsius	By default, temperatures are displayed in degrees Farenheit.
Help	
• Help	Displays help.
• About	Displays information about the software.

Browse Tab

Use **Browse** to locate ChemStation run data and display the chromatogram associated with it. Displaying a file selects it (and its parent folder) for processing. Until changed, the selection will be reflected in the SimDis pane. Associated files, such as calibration and blank files, will be displayed from the Setup Overview tab.

Browse displays three adjustable panes: a folder tree, a sample list, and a preview pane. See Figure 50.

- Select a subfolder (folders containing data runs) to display the list of samples within them
- Select a data folder from the folder tree or sample list to display its associated chromatogram in the preview pane

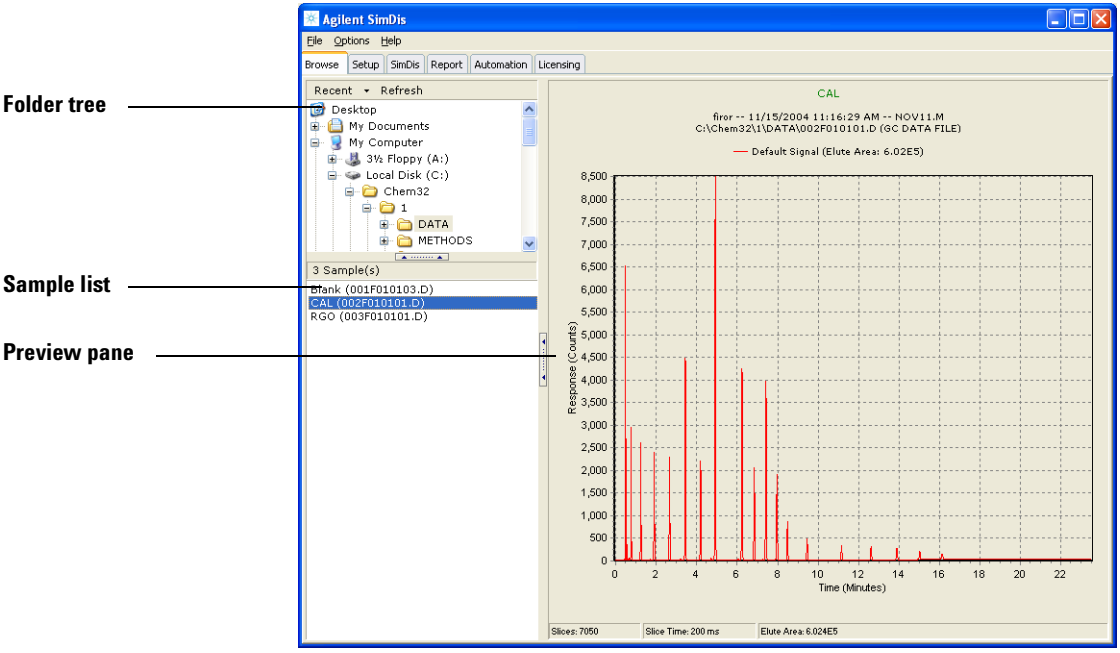


Figure 50 Browse panes

Browse contains two command buttons:

- **Recent**—Displays the most recently-accessed data files for quick reference.
- **Refresh**—Refreshes the folder tree display.

Setup Tab

Use **Setup** to:

- Select calibration and solvent blank data files for use in setting up a SimDis analysis
- Display the currently-selected calibration and solvent blank data file chromatograms
- Save a setup as the default
- Save setups for later re-use
- Edit the calibration data peaks

Setup contains three tab controls (See Figure 51).

Overview Use to display the selected chromatograms as they will be used and to save setup files.

Calibration Use to select, display, and edit a calibration file for use.

Blank Use to select, display, and edit a solvent blank run file for use.

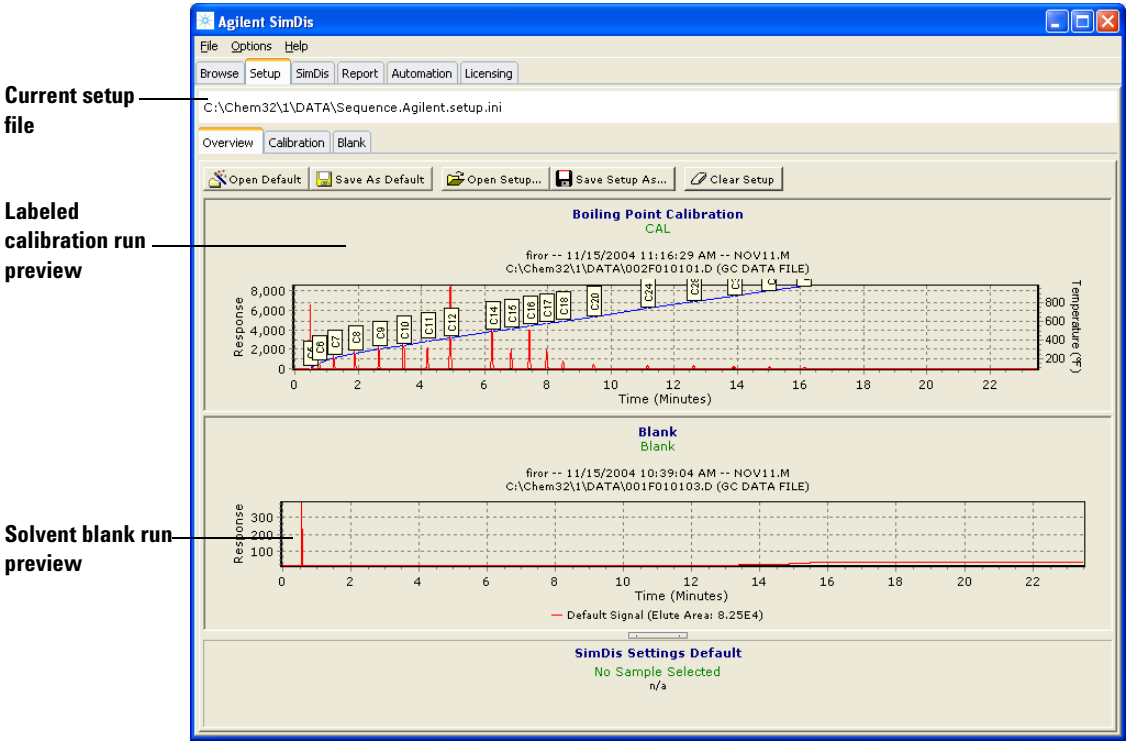


Figure 51 Example Setup, Overview tab

The Setup commands, parameters, and fields are described in Table 12 through Table 14.

Table 12 Setup Overview tab

Command button	Comment
Open Default	Open the saved default calibration and solvent blank settings for use.
Save As Default	Save the current calibration and solvent blank selections and settings as the default.

Table 12 Setup Overview tab (continued)

Command button	Comment
Open Setup...	Load calibration and solvent blank selection and settings from a saved setup file. Only files with extension **.setup** appear in the list.
Save Setup As...	Save the current calibration and solvent blank selections and settings. The file extension must be **.setup**.
Clear Setup	Disassociates calibration, blank, and sample selections and settings from all data files in the active subdirectory (the directory selected in the Browse tab). Note that selections made on the SimDis tab for an individual sample are not cleared.

Table 13 Setup Calibration tab

Command / Pane	Comment
Display	Use to show the current selections, including peak labels, against the data chromatogram and BP curve.
Select	Use to locate, select, and load a calibration file. A new file is immediately displayed using the existing settings.
• Recent	Displays the most recently-accessed data files for quick reference.
• Refresh	Refreshes the folder tree display.
Edit	Use to change calibration information.
• Peaks / Use Imported Peaks	Use the identified peak data from the ChemStation report.

Table 13 Setup Calibration tab (continued)

Command / Pane	Comment
• Peak Detection / Detect Peaks	Click to have the program search the data file for the number of peaks, in elution order, entered in the **Max Peaks** field.
• Peak Detection / Max Peaks	Limit the number of peaks detected by the program.

Table 14 Setup Blank tab

Command	Comment
Display	
• Show Calibrated Data (Show Temperatures)	Toggles response vs. calibrated boiling point temperatures or retention times.
• Show Prefiltering Data	Toggles display of the solvent blank chromatogram without filtering, if used.
Zeroing Method drop-down list	Set the **Zeroing Method** to use. See"Zeroing methods" on page 177.
Solvent Masking drop-down list	Set the **Solvent Masking** to use. See"Solvent masking methods" on page 177.
Filter drop-down list	Set the **Filter** Method to use. See"Filter methods" on page 178.
Select	These functions are the same as for Calibration / Select.

SimDis Tab

Use **SimDis** to:

- Select the sample to be analyzed (first, use the Browse tab to select a subdirectory)
- Set the **Zeroing Method**, **Solvent Masking** method, and **Filter** method for the sample data
- Adjust the sample elution times for the analysis
- Show or hide boiling point data, sample elution time data, and other items in the sample chromatogram

The SimDis commands, parameters, and fields are described in Table 15 and Table 16.

Table 15 SimDis tab command buttons

Button		Comment
Load Settings From Default Sample		Replace the current data treatments (for example, zeroing and solvent masking) with those of the default sample.
Use This Sample As Default		Saves this sample's data treatments as the default sample settings.
	Show Calibrated Data (Show Temperature)	Toggles response vs. calibrated boiling point temperatures or retention times.
	Show Elution-Time Markers	Toggles display of markers and values for start and end elution times.
	Show Boiling-Point Markers	Toggles display of initial and final boiling point markers.
	Show Percentage Yield	Toggles display of percentage yield and chromatogram peaks.
	Show Blank	Toggles an overlay of the current solvent blank run onto the sample data chromatogram.

Table 15 SimDis tab command buttons (continued)

Button		Comment
	Show Uncorrected Signal	Toggles display of the raw, uncorrected signal data, if zeroing and masking are used.
	Show Unfiltered Data	Toggles display of the unfiltered signal data, if filtering is used.
	Retain zoom when switching samples / channels	

Table 16 SimDis tab controls

Control	Comment
Sample drop-down list box	Select the sample to analyze. Alternately, select the sample using the **Browse** tab.
Zeroing Method drop-down list box	Select the **Zeroing Method** to use. See"Zeroing methods" on page 177.
Solvent Masking drop-down list box	Select the **Solvent Masking** to use. See"Solvent masking methods" on page 177.
Filter drop-down list box	Select the **Filter** Method to use. See"Filter methods" on page 178.
Sample Elution Time	
• Automatic ASTM, Start Time, and End Time	Select to use the ASTM algorithm to determine the sample elution times. Clear to manually set the start and end of elution.

Zeroing methods

Use the Zeroing methods to correct for baseline / offset column bleed.

None

A diagnostic tool. Use to examine the raw signal to determine its quality.

Zero to minimum

Determines the minimum value of the chromatogram and offsets the chromatogram by that amount to create a new zero.

D2887 Sampled baseline

When no acceptable solvent blank run exists, select **D2887 Sampled baseline** to use the ASTM D 2887 baseline algorithm.

Subtract Blank

Recommended. Subtract the selected solvent blank run from the current data to compensate for baseline upsets such as rise. This also sets any resultant negative peaks to zero, eliminating loss of area. If desired, enter a **Blank Offset** to add the entered value to all data points.

SE-EE Interpolated baseline

Draws a line between the start of elution (SE) and end of elution (EE), then zeroes the chromatogram to the line.

Solvent masking methods

Use the Solvent Masking methods to eliminate unwanted solvent peaks.

None

Keep all peaks for analysis. (Other tools and techniques may still be applied to unwanted peaks.)

Interpolated Mask

Replaces the signal from the **Mask Start** to the **Mask End** with a set of linearly interpolated values.

Mask to Zero

Replaces the signal from the **Mask Start** to the **Mask End** with a value of **0** (zero).

Mask to value

Replaces the signal from the **Mask Start** to the **Mask End** with the value entered in **Mask Value** field.

Filter methods

Filtering options allow smooth noisy signals and, in many cases, improves the signal to noise ratio. For references to publications giving details, see “Filter References" on page 188. Filtering modes include:

No Filtering

No signal filtering.

Savitzky-Golay

The Savitzky-Golay method is a commonly used tool for signal improvement in analytical chemistry. The method applies a polynomial least squares regression on the selected data with the general characteristic of maintaining peak height and width for typical chromatographic peaks. The following parameters are provided:

Past Points: The number of past data values to use in the filter

Future Points: The number of future data values to use in the filter

Derivative:	Derivative of the input the filter should be trying to estimate (0 (zero) or 1 (unity) are the most useful)
Polynomial Order:	Order of the underlying polynomial and the highest conserved moment (2 and 4 are the most useful)

For **Derivative** = 0 (zero) and **Polynomial Order** = 1 (unity), with n-past and n-future points, the Savitzky-Golay method becomes a [2n + 1]-point moving average.

Gaussian

The Gaussian method converts the signal to the frequency domain using the FFT (Fast Fourier Transform) and filters the signal with a Gaussian low-pass filter. The signal is then reconstructed. The following parameters are provided:

Sigma:	Defines standard deviation of the Gaussian filter. It can be thought of as a peak width parameter
Noise:	Controls amount of smoothing
Interval:	Parameter associated with time span of the filter

Wavelet

The Wavelet Filter uses a new approach to smoothing and de-noising signals based upon a family of mathematical functions commonly referred to as “wavelets”. The filter uses the Daubechies family of wavelet coefficients to perform transforms having the general property of preserving local time-based characteristics of the signal: information often lost in Gaussian or FFT (Fast Fourier Transform) filters. The following parameters are provided:

Level: Specifies how much high frequency data is discarded before reconstructing the signal

Filter: Specifies order of the wavelet transform used (between 2 and 10)

Report Tab

Use **Report** to:

- Display and print the results of analyses
- Set the desired report type
- Select report output

A sample Report pane is shown in Figure 52.

Table 17 lists the report types available.

Table 17 Report type selections

Report type	Description
Engineering Chart	Yield % Off graph and D 2887 percentage yield table.
Signature Chart	Sample temperature vs. response chromatogram.
Calibration Chart	Calibration sample chromatogram and labelled temperature calibration curve
Calibration Report	Time, temperature, and carbon numbers for the calibration sample
Engineering Report	Percentage yield table, in 5% increments
Simplified Calibration Response	Sampled listing of time, temperature, and signal value for the calibration sample
Complete Percentage Yield	Percentage yield table, in 1% increments
Complete Calibrated Response	Complete listing of time, temperature, and signal value for the calibration sample
Standard Cut Point	Tabulation of percentages vs. temperature, in 50 °F (or 27.78 °C) increments
Custom Cut Point	Create and use custom cut points to generate the tabulated cut point report. Enter the cut points in the From (temp) and To (temp) table fields. • Click **Apply** to see the results of the custom cut points • Click **Load** to load a saved custom cut point file • Click **Save** to save the custom cut points to a file • Click **Clear** to delete the current custom cut points
D86 Correlation	D 2887 to D 86 correlation (STP577)(yield vs. temperature).

To view or print a report:

1 Verify the correct data is selected from the Sample list.

2 Select the desired report from the **Report Type Selection** list.

When printing more than one file, all are printed using the selected report type.

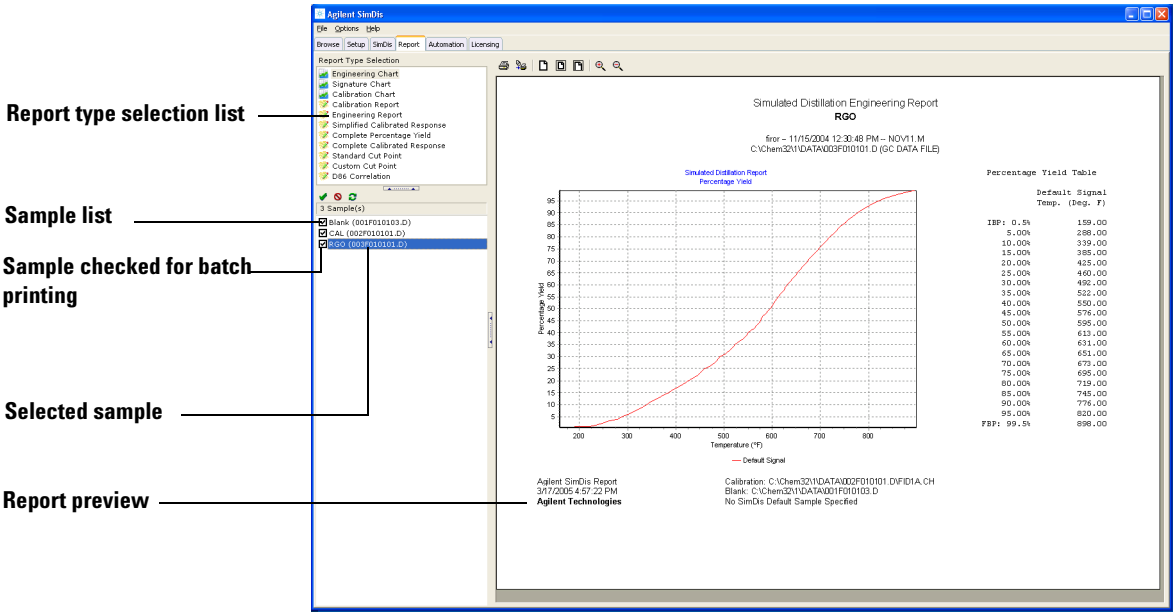


Figure 52 Example Report preview

The **Report** tab tools are described in Table 18.

Table 18 **Report** tab

Control	Comment
Sample list	
	Check (select) all samples in the sample list.

Table 18 **Report** tab (continued)

Control	Comment
	Uncheck all samples in the sample list.
	Invert selections in the sample list.
Report tools	
	Print the current report (graphical layout).
	Print reports for all samples currently selected in the sample list (graphical layout).
	Set up the printer (text reports only).
	Saves the current report as a text file (text reports only).
	Saves the all reports currently selected in the sample list as text files (text reports only).
	Show report at actual size (screen only).
	Fit report to page (screen only).
	Fit report to page width (screen only).
	Zoom in or out in the report pane.
	Includes header information in the report.

Table 18 **Report** tab (continued)

Control	Comment
csv	Formats the text report output file with comma separators.
tab	Formats the text report output file with tab-delimiters.

Automation Tab

Automation maintains a running list of the automated sample processing performed by the software and the ChemStation. This list is selectable for copying and pasting but is not printable.

Automation logs the following files and events:

- Automation start
- Activation (Link Opened) of the SimDis software from the ChemStation, when ChemStation executes the SimDis application and passes run control to the SimDis software
- Calibration, solvent blank, and properties (labeled “Prop,” this is the sample file containing the data treatments used, such as a Reference Gas Oil chromatogram) file names and paths
- Report file name and path—the sample file for which the report was generated
- Deactivation (Link Closed) of the SimDis software from the ChemStation when the SimDis software completes its analysis and returns run control to the ChemStation

Use this information to troubleshoot sequence problems. It can help isolate whether a sequence stopped due to a problem in the ChemStation or in the SimDis software. See also the ChemStation logs.

Also use this information to verify that correct settings are used for each sample run in a multi-instrument environment.

Licensing Tab

Use this tab to enter the unlock code during product activation, and to add new licenses. See Chapter 2, “Installation”.

Note that the trial period starts at the first installation, and cannot be reset with subsequent installations.

Agilent ChemStation SimDis Menu Items

At installation, the SimDis application adds a new **SimDis** menu item to the ChemStation menu bar. This menu provides easy access to the commonly-used commands listed in Table 19 below.

Table 19 ChemStation SimDis Menu Commands

Menu selection	Comment
Activate SimDis	Start the SimDis application.
Setup > Set Calib	Use the currently- loaded ChemStation data file as the calibration run in the SimDis software.
Setup > Set Blank	Use the currently-loaded ChemStation data file as the solvent blank run in the SimDis software.
Setup > Set Parameters	Locate a checkout (reference) sample. This sample's SimDis settings will be used for subsequent data processing using the software (until you change any SimDis settings).
Setup > Use SimDis defaults	Use the saved default settings for subsequent data processing (until you change any SimDis settings).
Select report	Choose the report template for reporting and printing.
Print SimDis Report	Print a SimDis report for the loaded data file using the current report selections for type and output. You can only print from standard ChemStation data paths.
Display Settings	Display the currently-selected calibration file, solvent blank file, sample file, and parameters file.
End SimDis	Exit the SimDis software (does not close ChemStation).

Filter References

Savitzky-Golay

A. Savitzky and M.J.E. Golay, "Smoothing and Differentiation of Data by Simplified Least Squares Procedures", *Analytical Chemistry* (1964), **36**, 1627–1639.

J. Steinier, Y. Termonia, and J. Deltour, "Comments on Smoothing and Differentiation of Data by Simplified Least Square Procedures", *Analytical Chemistry* (1972), **44**, 1906–1909.

H.H. Madden, "Comments on Savitzky-Golay Convolution Method for Least-Squares Fit, Smoothing and Differentiation of Digital Data", *Analytical Chemistry* (1978), **50**, 1383–1386.

P.A. Gorry, "General Least-Squares Smoothing and Differentiation by the Convolution (Savitzky-Golay) Method", *Analytical Chemistry* (1990), **62**, 570–573.

Gaussian

R. Gonzalez and R. Woods, Digital Image Processing, Addison-Wesley Publishing Company, 1992.

Mark Mackenzie and Kiet Tieu, ”Orthogonal Gaussian Filters for Signal Processing“, Proceedings, Workshop on the Internet, Telecommunications and Signal Processing, December 2002, 9–11.

Alle Meije Wink and Jos B.T.M. Roerdink, ”The Effect of Image Enhancement on the Statistical Analysis of Functional Neuroimages: Wavelet-Based Denoising and Gaussian Smoothing, Proceedings of SPIE - Volume 5032, Medical Image Processing, May 2003, (Milan Sonka and J. Michael Fitzpatrick, Editors), 1320–1330.

Wavelet

B. Walczak and D.L. Massart, “Tutorial - Noise suppression and signal compression using the wavelet packet transform”, Chemometrics and Intelligent Laboratory Systems (1997), **36** 81–94.

B. Walczak and D.L. Massart, “Wavelet packet transform applied to a set of signals: A new approach to the best-basis selection”, Chemometrics and Intelligent Laboratory Systems (1997), **38** 39–50.

C.R. Mittermayr, S.G. Nikolov, H. Hutter, and M. Grasserbauer, “Wavelet de-noising of Gaussian peaks, a comparative study”, Chemometrics and Intelligent Laboratory Systems (1996), **34** 187–202.

8 Maintenance

This chapter describes SimDis System-specific maintenance you will need to perform periodically on the 6890N GC.

Refer to the 6890N GC documentation for:

- Split/Splitless inlet information
- Column installation information not covered in this chapter
- General GC maintenance procedures

Replacing HT PTV Inlet Parts

This section describes how to replace specific parts in the HT PTV inlet.

Preparing for part replacement

Before working on the HT PTV inlet:

1. Turn off the GC and unplug the power cord.
2. Wait for it to cool.
3. Turn off the inlet, oven, and detector.
4. If you are using hydrogen carrier gas, turn it off at the source.

Septum

If a septum leaks, you will see symptoms such as longer or shifting retention times, loss of response, and/or loss of column head pressure. Signal noise will also increase.

The useful lifetime of a septum depends upon injection frequency and needle quality; burrs, sharp edges, rough surfaces, or a blunt end on the needle decrease septum lifetime. When the instrument is in steady use, replace the septum daily.

WARNING **Be careful! The oven and inlet may be hot enough to cause burns.**

To replace the septum:

1 Prepare the inlet as described in “Preparing for part replacement" on page 192.

2 Remove the injector and set it aside.

3 Unscrew the septum nut. See Figure 53.

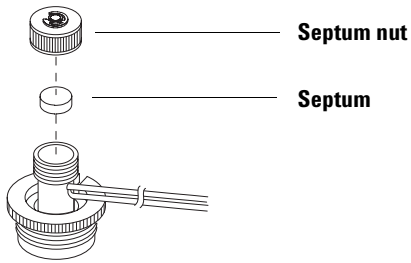


Figure 53 Inlet with the septum and septum nut removed

4 Remove the septum. If it sticks, use a sharp tool to remove it. Be sure to get all of it. Take care to avoid gouging or scratching the interior of the septum head.

5 If pieces of the septum are sticking, use a small piece of rolled-up steel wool and forceps or tweezers to scrub the residue from the septum nut and head. Use compressed air or nitrogen to blow away pieces of steel wool and septum.

6 Use forceps to insert a new septum (see “System Requirements" on page 14). Press it firmly into the fitting.

7 Replace the septum nut and tighten it finger-tight. Tightening the cap pushes the clip up approximately 1 mm from the surface of the nut. Do not over-tighten the nut. See Figure 54.

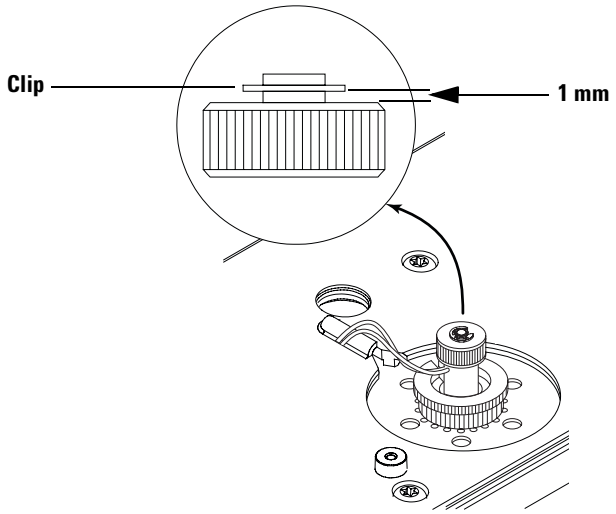


Figure 54 Replacing the septum nut

8 Replace the injector.

9 Restore gas flows and conditions.

Chemical trap

The chemical trap helps reduce the release of sample out of the split vent. It requires periodic replacement, depending on usage.

WARNING **The chemical trap may contain residual amounts of any samples or other chemicals you have run through the GC. Follow appropriate safety procedures for handling these types of substances while replacing the chemical trap.**

To replace the chemical trap:

1 Prepare the inlet as described in “Preparing for part replacement" on page 192.

2 Remove the pneumatics chassis cover by lifting up on each end to release the clips and remove the cover. See Figure 55.

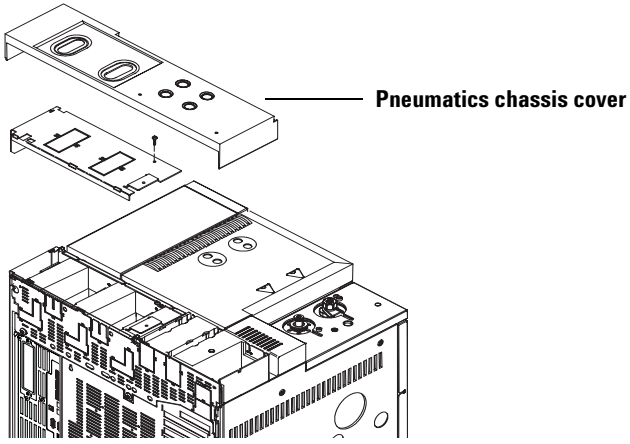


Figure 55 Pneumatics chassis cover

3 Unscrew the two sides of the filter housing. Move the housing to the left, with the line to the inlet still going through its center. See Figure 56.

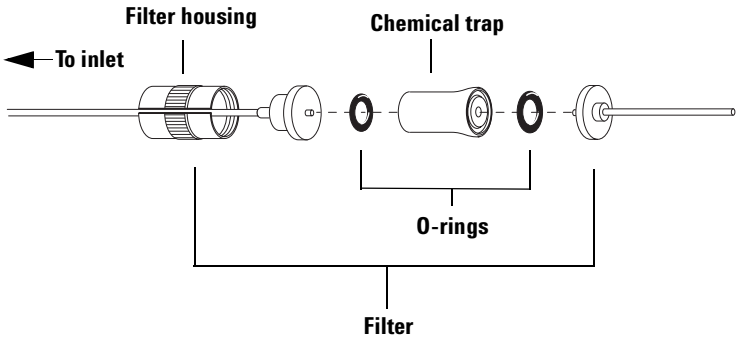


Figure 56 Filter (exploded view)

4 Remove the chemical trap from the filter housing.

5 Install O-rings in both ends of the new chemical trap.

6 Insert the new chemical trap into the filter housing. It should fit completely inside. If not, turn it around and re-insert it.

7 Reassemble the filter and finger-tighten it.

8 Replace the pneumatics chassis cover.

9 Replace the injector.

10 Restore flows and operating conditions.

11 Perform a leak test.

Liner and O-ring

You will need to change the O-ring when:

- you change the liner
- it wears out and becomes a source of leaks in the inlet

CAUTION Inspect the O-ring frequently when you use the inlet at temperatures above 300 °C (572 °F). You will need to replace the O-ring more often at such high temperatures.

O-rings contains plasticizers to give them elasticity. They seal the top of the inlet, the inlet base, and the liner. However, at high temperatures, the plasticizers bake out and the O-rings become hard and are no longer able to create a seal.

To change the liner and O-ring:

1 Prepare the inlet as described in "Preparing for part replacement" on page 192.

2 Remove the injector and set it aside.

3 Loosen the retaining weldment nut and lift the septum head from the inlet body. Be careful to avoid breaking the liner. See Figure 57.

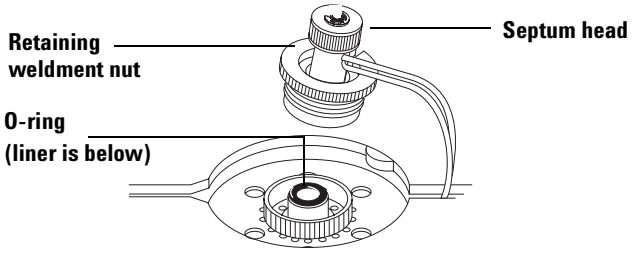


Figure 57 Exploded view of the upper part of the inlet

CAUTION Be careful when handling liners. They are very thin; you can easily break them.

4 **Gently** remove the liner from the inlet body. If necessary, use needle-nosed pliers.

5 Place a new O-ring on the new liner (a few millimeters down from the end without the baffles).

NOTE If you are using a **split/splitless** inlet with any of these liners:

- 5183-4711 (Deactivated)
- 5183-4647 (Deactivated)
- 19251-60540 (Non-deactivated)

place the glass wool on the liner so it is at the top of the inlet body.

This **does not** apply to the HT PTV inlet.

6 Install the liner (baffle-end down) and O-ring into the inlet body. The O-ring is pushed to the top of the liner.

7 Check that the O-ring is below the surface line of the inlet flange nut. Place the straight edge of an object across the inlet flange nut. If it rests on both sides of the ring, the

O-ring's position is correct. If necessary, push the liner and O-ring down further. See Figure 58.

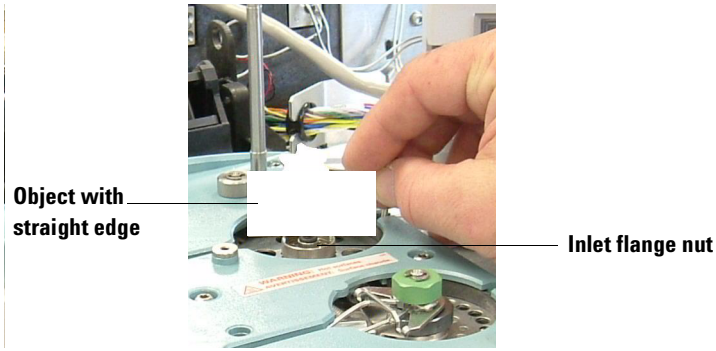


Figure 58 Checking the O-ring

8 Reassemble the inlet.

9 Reinstall the injector.

10 Restore operating conditions.

11 Perform a leak check.

Installing Columns

This section describes how to install the columns used with HT PTV inlet. See the 6890N GC documentation for information on column installation for split/splitless inlets.

Required tools

- Open-end wrenches (5 mm and 1/4 inch)
- Flat file
- Needle-nosed pliers (optional)
- Typewriter correction fluid
- Metric ruler

Installing fused silica capillary columns in an HT PTV inlet

DB-1 columns (used with the D 2887 test method) and the HP-1 columns (used with the D 2887x test method) are made of fused silica. To install them into an HT PTV inlet:

1. Turn off the GC and unplug the power cord.
2. Wait for the GC to cool.
3. Turn off gas flows to the inlet and detector.
4. Open the oven door.
5. Prepare the column as described in the 6890N GC documentation.

6 Slide a column nut and standard ferrule over the inlet end of the column. The inlet end of the ferrule should face up (toward the inlet). The bottom of the ferrule should be in front of the column nut. See Figure 59.

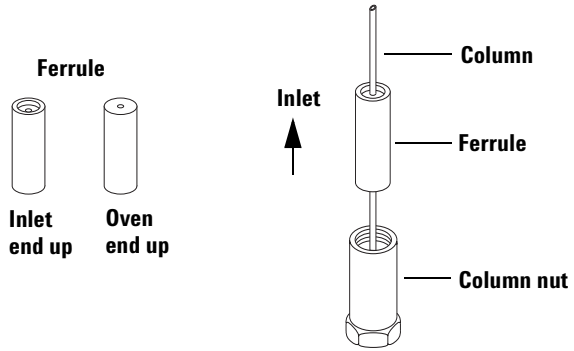


Figure 59 Placing the column nut and ferrule on the column

7 Score the column a few centimeters from the end, break it off, and wipe the column.

8 Using typewriter correction fluid, make a mark, 13 mm from the inlet end of the column. See Figure 60.

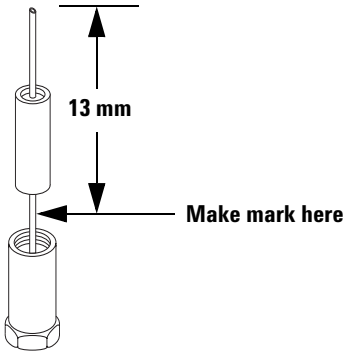


Figure 60 Marking the column

9 Insert the column in the inlet and slide the nut and ferrule up the column to the lower insulation cover. Finger-tighten the column nut until it starts to grab the column.

10 Adjust the column position so that the correction fluid mark on the column is even with the bottom of the nut. See Figure 61.

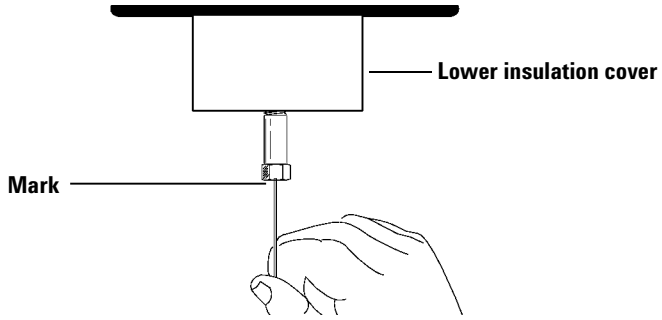


Figure 61 Adjusting the column

11 Tighten the column nut an additional 1/4 to 1/2 turn so that you cannot pull the column from the fitting with gentle pressure.

12 Hang the column on the hanger.

13 Install the column in the detector.

14 Close the oven door.

15 Restore power to the GC and return it to operating condition (including turning on the gas flows).

16 Perform a leak check.

17 Condition the column as recommended by the manufacturer.

Installing metal capillary columns in an HT PTV inlet

The DB-HT SIMDIS column is a metal capillary column, used with both the D 2887x and D 6352 standards.

To install it into an HT PTV inlet:

1 Turn off the GC and unplug the power cord.

2 Wait for the GC to cool.

3 Turn off gas flows to the inlet.

4 Open the oven door.

5 Prepare the column as described in the 6890N GC documentation.

CAUTION Be careful not to force the column into the ferrule. The ferrules contain graphite packing, and bits of the graphite could fall into the column.

CAUTION If you have a problem installing a ferrule onto the column or fitting the column into the inlet, check for burrs. If there are any, use a flat file to carefully remove any burrs from the column. Do not contaminate the column's open end with debris. Then try to install the column again.

6 Slide a column nut and standard ferrule over the inlet end of the column. The inlet end of the ferrule should face up (toward the inlet). The bottom of the ferrule should be in front of the column nut. See Figure 62.

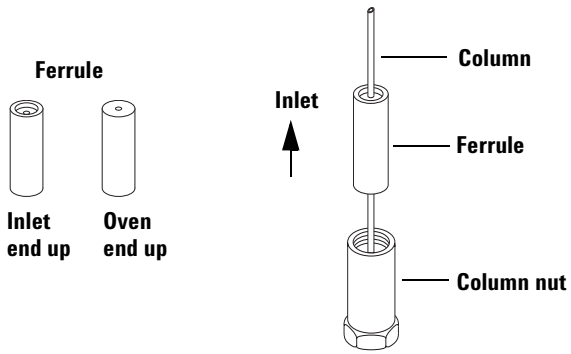


Figure 62 Placing the column nut and ferrule on the column

7 Score the column a few centimeters from the end, break it off, and wipe the column.

8 Using typewriter correction fluid, make a mark, 13 mm from the inlet end of the column. See Figure 63.

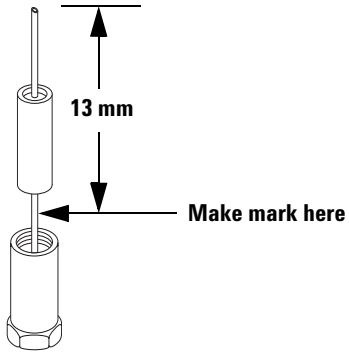


Figure 63 Marking the column

9 Insert the column in the inlet and slide the nut and ferrule up the column to the inlet base. Finger-tighten the column nut until it starts to grab the column.

10 Adjust the column position so that the correction fluid mark on the column is even with the bottom of the nut. See Figure 64.

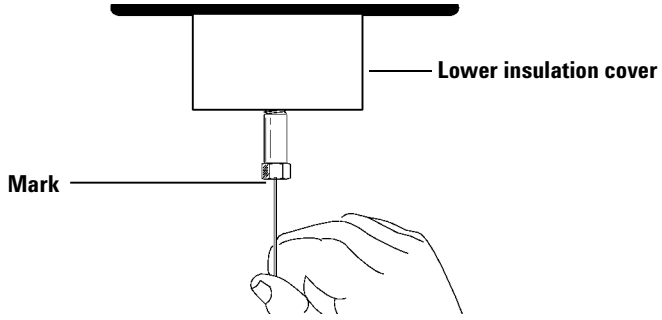


Figure 64 Adjusting the column

11 Tighten the column nut an additional 1/4 to 1/2 turn so that you cannot pull the column from the fitting with gentle pressure.

12 Hang the column on the hanger.

13 Install the column in the detector.

14 Close the oven door.

15 Restore power to the GC and return it to operating condition (including turning on the gas flows).

16 Perform a leak check.

17 Condition the column as recommended by the manufacturer.

Cleaning an HT PTV Inlet's Split Vent Line

An HT PTV inlet's split vent line may become contaminated with sample residue:

- After long use
- If the inlet overloads

To avoid having the residue affect chromatograms, clean the split vent line. You will need:

- Acetone
- Cotton swab
- Needle-nosed pliers (optional)

To clean the split vent line:

1. Turn off the GC and unplug the power cord.
2. Wait for the GC to cool.
3. Loosen the retaining weldment nut and lift the septum head off the inlet.
4. Carefully pull out the O-ring / liner. Use needle-nosed pliers, if necessary.
5. Use a cotton swab moistened with acetone to clean the exposed part of the inlet. See Figure 65.

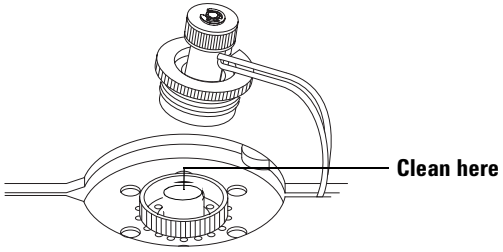


Figure 65 Exploded view of the upper part of the inlet

6. Install a new liner and O-ring. See "Liner and O-ring" on page 197.
7. Re-assemble the inlet.

8 Replace the injection port cover.

9 Do blank runs with an oven program running from 40 to 350 °C (104 to 662 °F). Set the inlet's temperature to 350 °C. The chromatograms should improve with each run.

Replacement Parts

Orderable replacement parts and consumables are listed in Table 20. The table only lists items specific to SimDis. For general GC replacement parts and consumables, refer to the 6890N GC documentation. Also, refer to the Agilent catalog for consumables and supplies. More parts information is also available on the Agilent Web site at www.agilent.com / chem.

Table 20 SimDis System replacement parts

Description	ASTM Standard	Part number
Inlet hardware		
Septum, 11-mm high temperature, low bleed, green (all inlets), 50 / pk	All	5183-4759
Liner, general purpose split, glass wool, taper, deactivated, 1 each **(Split/Splitless inlet only)**	D 2887	5183-4711
Liner, split, glass wool, non-deactivated, 1 each **(Split/Splitless inlet only)**	D 2887	19251-60540
Liner, split, low press, drop, glass wool, taper, deactivated, 1 each **(Split/Splitless inlet only)**	D2887	5183-4647
Liner, for HT PTV inlet, multiple restriction, glass-wool packed	All	JAS 90309L
Columns and related hardware		
DB-HT SIMDIS, 5 m x 0.53 mm 0.15 µm	D 2887x and D 6352	145-1001
HP-1, 10 m x 0.53 mm 0.88 µm	D 2887x	19095Z-021
HP-1, 5 m x 0.53 mm 0.88 µm	D 2887x	19095Z-020
DB-1, 10 m × 0.53 mm × 2.65 µm	D 2887	125-10HB
Ferrule, Graphpak 2M, 0.53 mm, for HT PTV inlet, 10 / pk	All	5182-9770
Ferrule, graphite, 1.0 mm id, for fused silica capillary column, 10 / pk **(Split/Splitless inlet only)**	D 2887	5080-8773
Ferrule, graphite, for 0.53 mm metal capillary column, 10 / pk **(Split/Splitless inlet only)**	All	JAS 9111153U
Other hardware		
Syringe, 5 µL	All	5181-1273

Table 20 SimDis System replacement parts (continued)

Description	ASTM Standard	Part number
Syringe, autosampler, 0.5 µL	all	5182-9651
Standards and samples		
Boiling Point Calibration No. 1	D 2887	5080-8716
Reference Gas Oil Sample No. 1 Batch 2	D 2887	5060-9086
Restek Polywax 500 (neat) sample	D 2887x and D 6352	5188-5316
Restek Polywax 655 sample	D 6352	5188-5317
Reference Material 5010	Refer to the ASTM D 6352 documentation.	
Low Boiling Point Calibration No. 220	D 2887x and D 6352	5080-8768
HT PTV inlet hardware		
Upper insulation		G2888-00020
Lower insulation		G2888-00030
Lower insulation cover		19243-00070
Column nut		5188-5312
Ferrule		G2888-20630
Cable assembly		G2888-60525
Mounting plate		G2888-00050
Filter housing		G2888-20590
Retaining nut weldment		G2888-20560
O-ring (for chemical trap)		0905-1291
Chemical trap		G1544-80540
Septum nut		G2888-60835
EPC flow module		G2888-60503
Inlet supply weldment		G2888-80500
Inlet flange nut		G2888-20550
O-ring (for liner)		5188-5311

Table 20 SimDis System replacement parts (continued)

Description	ASTM Standard	Part number
Liner		5183-4647
Thermocouple board		G2888-60010
Septum		G2888-20640
Heater sensor assembly		G2888-60520
Inlet weldment		G2888-60550

A
Calibration Sample Compositions

Compositions of two commercially available calibration standards are provided here for reference purposes. Unless otherwise noted, boiling points are from TABLE 2, *ASTM D 2887 - 97a, Standard Test Method for Boiling Range Distribution of Petroleum Fractions by Gas Chromatography.*

ASTM D 2887 Quantitative Calibration Solution

Table 21 lists the composition and boiling point data for the *ASTM D 2887 Quantitative Calibration Solution* available from Supelco, Inc. (Supelco Park, Bellefonte, PA 16823-0048, USA) as part number 500658:

Table 21 *ASTM D 2887 Quantitative Calibration Solution,* Supelco Part Number 500658

Analytes (all >98% pure)	µg / mL ±0.5%	B.P. (°C)	B.P. (°F)
n-Pentane (C5)	5000	36	97
n-Hexane (C6)	5000	69	156
n-Heptane (C7)	5001	98	208
n-Octane (C8)	5056	126	259
n-Nonane (C9)	5001	151	304
n-Decane (C10)	5000	174	345
n-Undecane (C11)	5001	196	385
n-Dodecane (C12)	5000	216	421
n-Tetradecane (C14)	5000	254	489
n-Pentadecane (C15)	5000	271	520
n-Hexadecane (C16)	10000	287	549
n-Heptadecane (C17)	5001	302	576
n-Octadecane (C18)	10000	316	601
n-Eicosane (C20)	5001	344	651
n-Tetracosane (C24)	5000	391	736
n-Hexacosane (C26)	5000	412	774
n-Triacontane (C30)	5000	449	840
n-Hexatriacontane (C36)	5087	496	925

Table 21 *ASTM D 2887 Quantitative Calibration Solution*, Supelco Part Number 500658 (continued)

Analytes (all >98% pure)	µg / mL ±0.5%	B.P. (°C)	B.P. (°F)
n-Tetracontane (C40)	5081	522	972
n-Tetratetracontane (C44)	5000	545	1013

Boiling Point Calibration Sample #1

Table 22 lists the composition and boiling point data for the Agilent *Boiling Point Calibration Sample #1*, part number 5080-8716:

Table 22 *Boiling Point Calibration Sample #1,* Agilent part number 5080-8716 (no solvent)

Analyte	% by weight	B.P. (°C)	B.P. (°F)
n-Pentane (C5)	8.32	36	97
n-Hexane (C6)	4.38	69	156
n-Heptane (C7)	4.55	98	208
n-Octane (C8)	4.67	126	259
n-Nonane (C9)	4.77	151	304
n-Decane (C10)	9.71	174	345
n-Undecane (C11)	4.92	196	385
n-Dodecane (C12)	19.91	216	421
n-Tetradecane (C14)	10.14	254	489
n-Pentadecane (C15)	5.11	271	520
n-Hexadecane (C16)	10.28	287	549
n-Heptadecane (C17)	5.17	302	576
n-Octadecane (C18)	2.21	316	601
n-Eicosane (C20)	1.30	344	651
n-Tetracosane (C24)	0.90	391	736
n-Octacosane (C28)	0.90	431	808
n-Dotriacontane (C32)	0.90	466	871
n-Hexatriacontane (C36)	0.91	496	925
n-Tetracontane (C40)	0.92	522	972

B
HT PTV Inlet Operation

This chapter describes the configuration and operation of the High Temperature Programmable Temperature Vaporizer (HT PTV) inlet.

NOTE You must always install the HT PTV inlet in the back inlet position of the 6890N GC.

Configuration

To configure an HT PTV inlet, press [**Config**] [**Back Inlet**]. Table 23 lists the settings you can make.

Table 23 Configuration settings.

Setting	SimDis selection	Options
Gas type	Helium	Helium Hydrogen Nitrogen Argon methane 5%
Cooling type	None	None Compressed air
Cryo	Off	
Use cryo temp	Not applicable	
Cryo time-out	Not applicable	
Cryo fault	Not applicable	

Operation

Operating modes and parameters

The HT PTV inlet is fully integrated into the 6890N GC and by extension, the ChemStation. Its operating modes and parameters are the same as the standard PTV inlet. The one exception is that cryo parameters do not apply because SimDis uses air cooling.

For details, refer to the 6890N GC user information.

Programming

For D 2887 simulated distillations, typically use a static temperature, although inlet temperature ramping can be used.

For D 2887x and D 6352 simulated distillations, a rapid ramp often works best. Run the inlet temperature up, hold, then cool back to the initial temperature. Figure 66 illustrates an example.

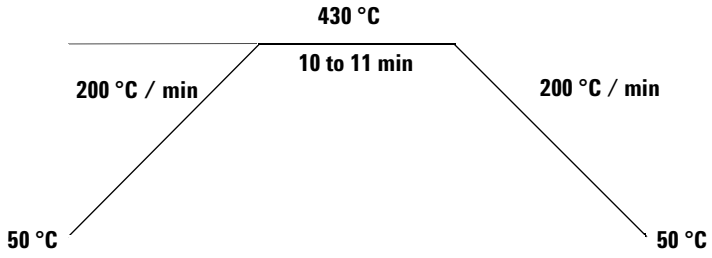


Figure 66 Example inlet temperature program for ASTM D 2887x or D 6352

NOTE Because the cool-down ramp is uncontrolled, the actual cool-down rate depends on the GC oven program.

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