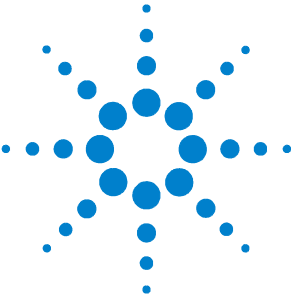
Agilent G2887AA
SIMDIS Software



ASTM D 2887 Quick Start Guide

Agilent Technologies

Notices

Manual Part Number

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Setting Up for ASTM D2887

This manual describes how to set up an Agilent SimDis system and optimize it for performing analyses in accordance with ASTM standard D 2887*. The system consists of:

- A 6890N gas chromatograph (GC), a 7683 automatic liquid sampler, a 5- or 0.5-µL syringe, and appropriate hardware
- A computer with the Agilent GC ChemStation (ChemStation, version A.10.03, or B.01.01 or greater)
- Agilent G2887AA SimDis (SimDis) software

This system quickly determines the boiling range distribution of petroleum fractions. It creates a relationship of retention times to boiling points for a calibration sample of known n-Paraffins, and then applies this relationship to an unknown feedstock sample's retention time data to determine the boiling range distribution and cut point information of the feedstock.

The SimDis software uses the following:

* ASTM D 2887–97A, Standard Test Method for Boiling Range Distribution of Petroleum Fractions by Gas Chromatography

- Solvent blank run data, to provide baseline compensation. (Refer to the online Reference Manual for the software's alternatives that work in accordance with ASTM D 2887.)
- Calibration sample run data, to provide the relationship of retention times to boiling points
- Reference gas oil (RGO) run data, to provide verification

The process described in this setup guide walks through the initial software setup and initial collection of the required data.

Before You Begin

The steps described here assume that the hardware and software needed for ASTM D 2887 analyses are installed and ready for use. Before beginning, make sure that you have:

- Installed the 6890N GC
- Installed an eight-vial turret in the 7683 injector (If using a sample tray, use the appropriate turret and adjust sample vial locations throughout this manual accordingly.)
- Installed the required column for ASTM D 2887 analysis
- Installed the ChemStation and have established GC control
- Installed and activated the SimDis software

Refer to the Agilent SimDis online *Reference Manual* found in the **Documents** folder on your SimDis software CD-ROM, and also available from the Help menu in the SimDis software for configuration details, installation instructions, and part numbers.

Supplies summary

Table 1 lists the primary samples and hardware needed for this analysis. Refer to the Agilent SimDis online *Reference Manual* for a complete listing.

Table 1 Supplies and hardware needed

Item	Part number
Boiling Point Calibration No. 1	5080-8716
Reference Gas Oil Sample No. 1 Batch 2	5060-9086
DB-1, 10 m × 0.53 mm × 2.65 µm	125-10HB
Liner, for HT PTV inlet, multiple restriction, glass-wool packed	5188-5313
O-ring, liner	0900-0028
Ferrule, column, for 0.53 mm column	5188-5314
Septum, green, 11-mm, advanced, 50/pk	5183-4759
Syringe, autosampler, 5 µL	5181-1273
CS_2 ("reagent grade" purity or better)	—

Overview of Setup Steps

Agilent SimDis setup for ASTM D 2887 involves the following steps:

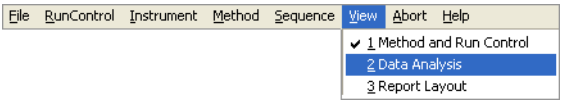
1 Create a Solvent Blank ChemStation Method

To create the method needed for running the ASTM solvent blanks:

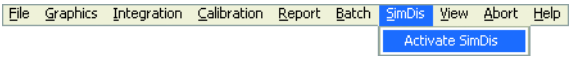
1 Open the ChemStation online instrument.

2 Activate SimDis for later use.

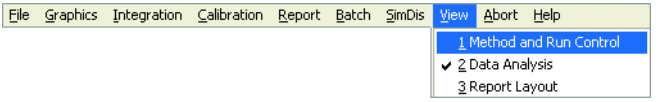
a Select View / Data Analysis



b Select SimDis / Activate SimDis



c Select View / Method and Run Control



3 Starting from **DEF_GC.M**, set the following column, oven, and inlet parameters. This example uses the back inlet and front detector.

Column: 1 / 2
Mode: Const Flow
Inlet: Back
Detector: Front
Outlet psi: Ambient

Installed Column

Manufacturer's Specifications
Model No: Agilent 125-10HB 350°C Max
D2887 SIMDIS column
Capillary 10.0 m × 530 µm × 2.65 µm nominal

He Flow

	Setpoint	Actual	
Pressure:	3.44		psi
Flow:	13.3		ml/min
Average Velocity:	99		cm/sec

Flow	ml/min²	ml/min	Hold min	Run Time
Initial		13.3	0.00	999.99
Ramp 1	0.00	0.0	0.00	
Ramp 2	0.00	0.0	0.00	
Ramp 3	0.00	0.0	0.00	
Post Run			0.00	999.99

Oven
On
Setpoint °C: 40
Actual °C:

Oven Configuration
Maximum °C: 350
Equilibration min: 3.00

Oven Ramp	°C/min	Next °C	Hold min	Run Time
Initial		40	0.00	0.00
Ramp 1	20.00	350	4.00	19.50
Ramp 2	0.00	0	0.00	
Ramp 3	0.00	0	0.00	
Ramp 4	0.00	0	0.00	
Ramp 5	0.00	0	0.00	
Ramp 6	0.00	0	0.00	
Post Run		50	0.00	19.50

Cryo Configuration
Cryo On
Quick Cooling On
°C, Ambient
Timeout Detection On
min
Fault Detection On

Back: HT PTV
Cryo Config...

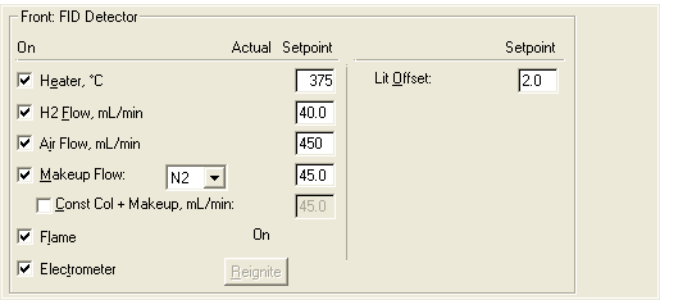
On	Actual	Setpoint
Heater, °C		350
Pressure, psi		3.44
Total Flow, mL/min		44.8

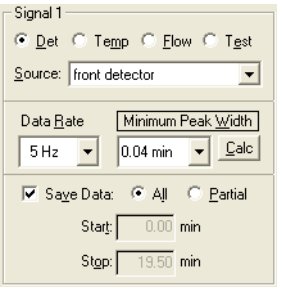
Mode: Split
Gas: He

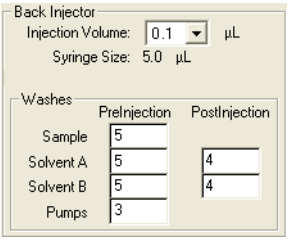
Ramps	°C/min	Next °C	Hold min
Initial		350	0.00
Ramp 1	0.00	0	0.00
Ramp 2	0.00	0	0.00
Ramp 3	0.00	0	0.00

Split Ratio: 2.0 :1
Split Flow: 28.0 mL/min
GasSaver 20.0 mL/min @ 2.00 min

4 Set the following detector, signal, and injector parameters.

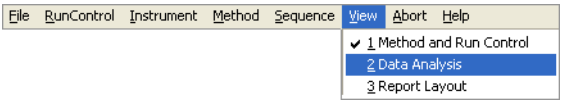




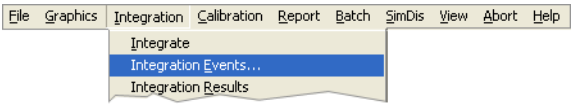


5 Set the following integration events and report options.

a



b



c

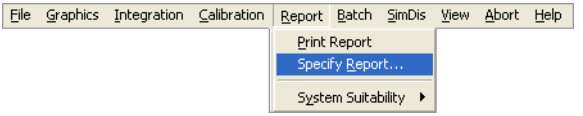
Events Table: FID Default

Time	Integration Events	Value
Initial	Slope Sensitivity	5
Initial	Peak Width	0.04
Initial	Area Reject	100000
Initial	Height Reject	5
Initial	Shoulders	TAN

Keep Area Reject high to prevent integration. Integration is not needed.

d Click  (**Exit and save events to the method**).

e



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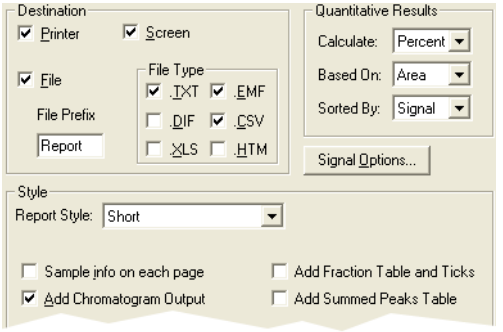


6 For this example, save the changes as **D2887_BLANK.M**.

2 Prepare the Samples

Prepare the sample and solvent vials listed in Table 2 and load them into the appropriate injector turret locations. For details about sample preparation, refer to the SimDis *Reference Manual*.

Table 2 Sample, solvent wash, and waste bottle preparation

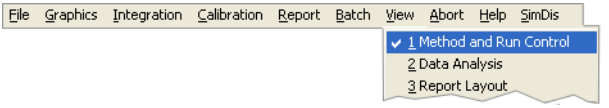
Bottle	Contents	Turret position
Solvent blank	CS_2 (reagent grade or better)	1
Calibration sample	1 part Boiling Point Calibration No. 1 to 5–10 parts CS_2	2
Reference Gas Oil	15 % by vol. Reference Gas Oil Sample No. 1 Batch 2, balance CS_2	3
Wash (2)	CS_2 (reagent grade or better)	A and B
Unknown samples		4–8
Waste	None	WA

3 Condition the Column

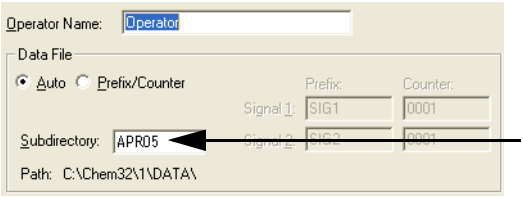
Run 4–5 solvent blanks to condition the column. This is the minimum number of solvent blanks runs typically needed to ensure system cleanliness and stable bleed. If the chromatograms still show impurities or shifting, keep running solvent blanks until the baseline is stable.

Create and run a sequence as shown below:

1



2 **Select Sequence / Sequence Parameters**

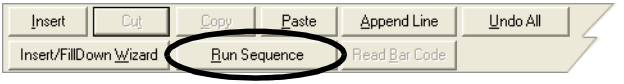


Enter a subdirectory name, for example, APR05. Use this subdirectory for all sequences in this document.

3 **Select Sequence / Sequence Table**

Line	Vial	Sample Name	Method Name	Inj/Vial	Sample Type
1	Vial 201	Solvent blank	D2887_BLANK	5	Sample

2

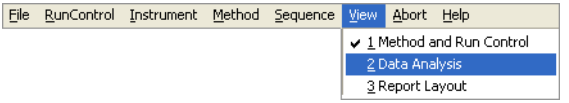


The final (most representative) solvent blank run will be used as part of the SimDis analysis in later steps.

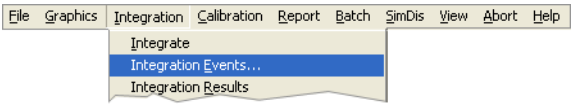
4 Create a Calibration Sample Method

Starting from **D2887_BLANK.M**, create a method to use for running calibration samples.

1



2



3

Events Table: FID Default

Time	Integration Events	Value
Initial	Slope Sensitivity	5
Initial	Peak Width	0.04
Initial	Area Reject	300
Initial	Height Reject	5
Initial	Shoulders	TAN

Adjust and rerun as needed to isolate the 19 calibration peaks

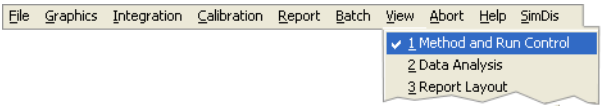
4 Click  (**Exit and save events to the method**).

5 For this example, save this method as **D2887_CAL.M**.

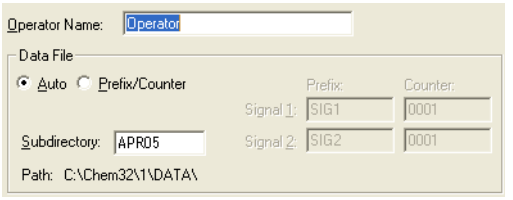
5 Run the Calibration Sample

Using the saved calibration method (D2887_CAL.M), create a sequence and run the calibration sample.

1



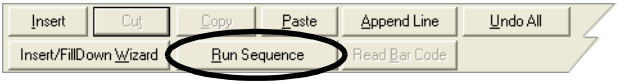
2 Select Sequence / Sequence Parameters



3 Select Sequence / Sequence Table

Line	Vial	Sample Name	Method Name	Inj/Vial	Sample Type
1	Vial 202	BP Calibration	D2887_CAL	1	Sample

4



6 Edit the Calibration Sample Method

Verify chromatographic performance

Examine the report output for the calibration sample. If needed, adjust the chromatographic conditions and rerun the calibration sample until all 19 expected calibration peaks are isolated and have significant area amounts. Adjust **Area Reject** as needed to eliminate noise peaks or to include small calibration peaks. Figure 1 shows an example acceptable calibration chromatogram.

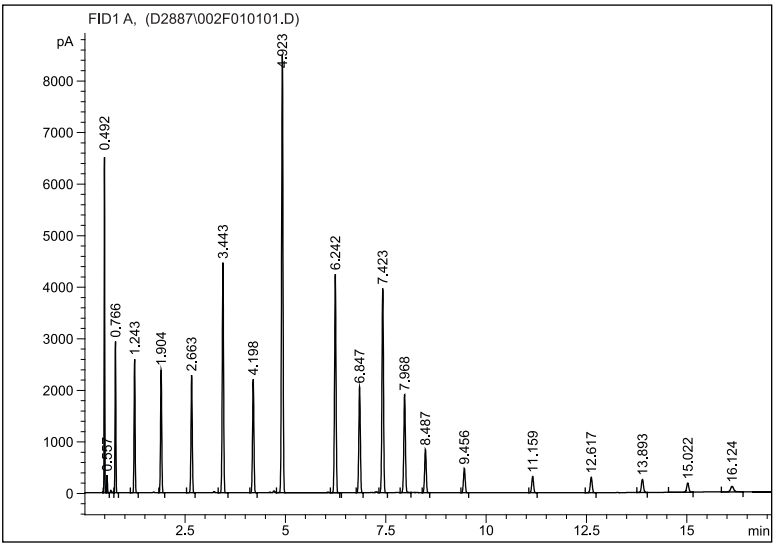


Figure 1 Example calibration sample chromatogram

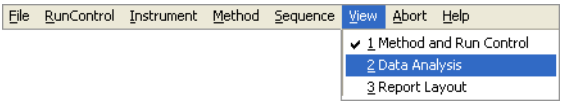
NOTE Remember that the same chromatographic conditions must be used for the solvent blank, calibration, reference gas oil, and sample runs. The solvent blank method and sample data will need to be changed/re-run after changing any chromatographic conditions. See "Correct All Methods and Rerun Samples as Needed" on page 17.

Calibrate the method

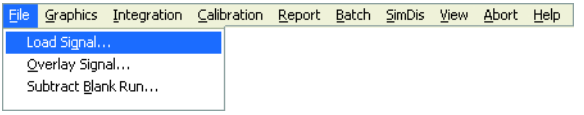
After the chromatography is acceptable, calibrate the method to identify and label the component peaks of interest.

- The data files listed assume default, automatic file naming.

1

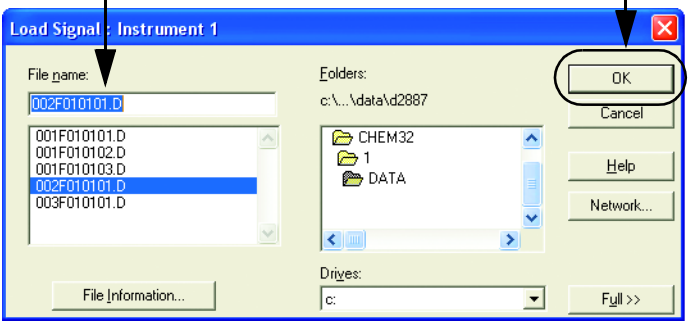


2

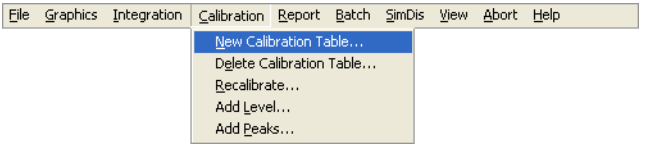


3

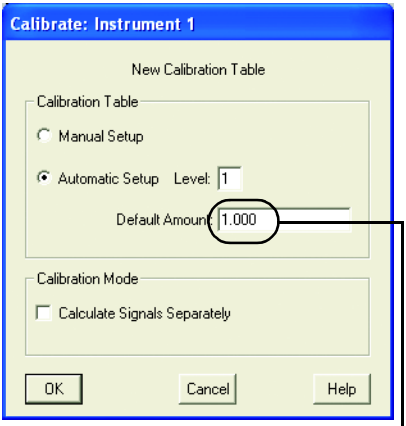
4



5



6



7 **Input the compound names exactly as shown**

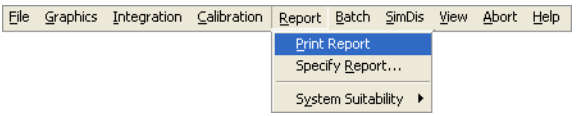
Enter | Delete | Insert... | Print | OK | Help

#	RT	Signal	Compound	Lvl	Amt[ng/ul]	Area	Rsp.Factor	Ref
1	0.492	FID1 A	C5	1	1.000	6824.800	1.4652e-4	No
2	0.557	FID1 A		1	1.000	416.450	2.4012e-3	No
3	0.766	FID1 A	C6	1	1.000	4157.200	2.4055e-4	No
4	1.243	FID1 A	C7	1	1.000	4663.700	2.1442e-4	No
5	1.904	FID1 A	C8	1	1.000	4953.300	2.0188e-4	No
6	2.663	FID1 A	C9	1	1.000	5138.700	1.9460e-4	Yes
7	3.443	FID1 A	C10	1	1.000	10431.000	9.5866e-5	No
8	4.198	FID1 A	C11	1	1.000	5318.500	1.8802e-4	No
9	4.923	FID1 A	C12	1	1.000	21332.000	4.6877e-5	No
10	6.242	FID1 A	C14	1	1.000	10902.000	9.1729e-5	No
11	6.847	FID1 A	C15	1	1.000	5503.400	1.8171e-4	No
12	7.423	FID1 A	C16	1	1.000	10801.000	9.2588e-5	No
13	7.968	FID1 A	C17	1	1.000	5391.100	1.8549e-4	No
14	8.487	FID1 A	C18	1	1.000	2394.000	4.1772e-4	Yes
15	9.456	FID1 A	C20	1	1.000	1418.700	7.0485e-4	No
16	11.159	FID1 A	C24	1	1.000	994.270	1.0058e-3	No
17	12.617	FID1 A	C28	1	1.000	993.980	1.0061e-3	No
18	13.893	FID1 A	C32	1	1.000	915.100	1.0928e-3	No
19	15.022	FID1 A	C36	1	1.000	676.990	1.4771e-3	No
20	16.124	FID1 A	C40	1	1.000	620.200	1.6124e-3	No

8 If desired, assign a few reference peaks, such as C9 and C18.

9 After making the changes, save the method. This is the method that you will use to run all SimDis calibration samples used.

10 Verify that the final calibration run's data file is loaded, generate a report, and write the output files to disk by selecting:

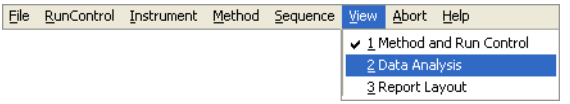


7 Create a Reference Gas Oil ChemStation Method

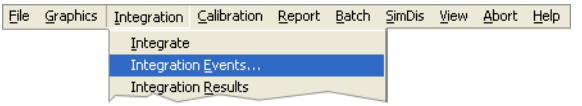
Now that you have a calibrated method, the next step is to edit this method for use on the reference gas oil standard. Running this sample confirms the suitability of the system for use, and generates the last piece of information needed for the SimDis software.

Starting from the calibration method (D2887_CAL.M), make the following changes:

1



2



3

Events Table — FID Default

Time	Integration Events	Value
Initial	Slope Sensitivity	5
Initial	Peak Width	0.04
Initial	Area Reject	100000
Initial	Height Reject	5
Initial	Shoulders	TAN

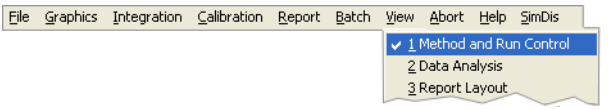
High Area Reject prevents integration. Instead, the SimDis software performs integration per the ASTM.

4 For this example, save this method as **D2887_RGO.M**. You will use this method to run all future reference gas oil samples.

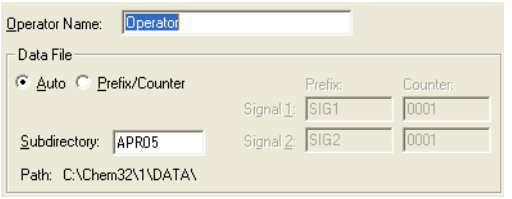
8 Run the Reference Gas Oil Sample

Using the saved RGO method (**D2887_RGO.M**), create a sequence and run the reference gas oil sample.

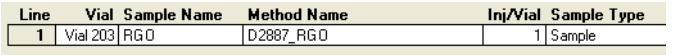
1



2 Select Sequence / Sequence Parameters

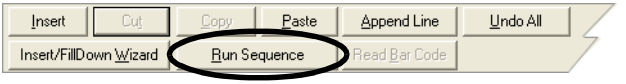


3 Select Sequence / Sequence Table



Line	Vial	Sample Name	Method Name	Inj/Vial	Sample Type
1	Vial 203	RGO	D2887_RGO	1	Sample

4



You will use the generated data files from this run to prepare the SimDis software to analyze your experimental samples for this day.

A typical reference gas oil chromatogram is shown in Figure 2 below.

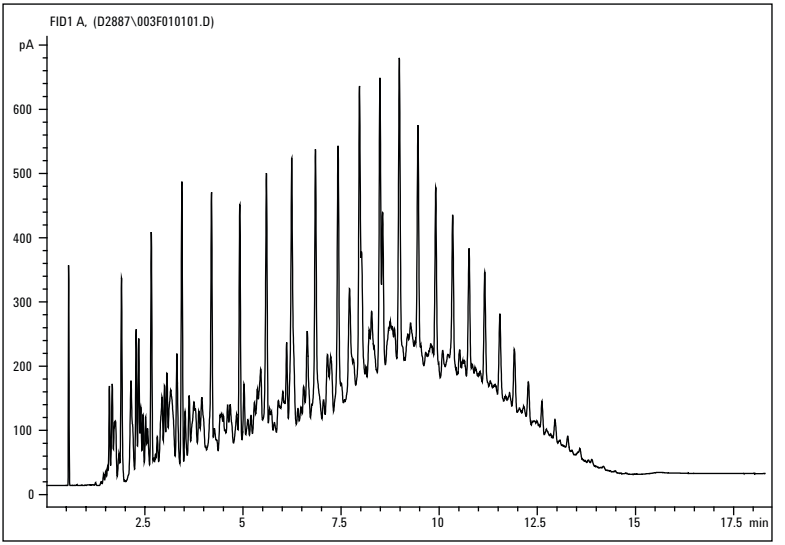


Figure 2 A typical reference gas oil chromatogram

9 Correct All Methods and Rerun Samples as Needed

All of the methods must use the same chromatographic conditions, and all samples must be run using these same conditions. If any chromatographic conditions changed between the solvent blank and calibration runs, or between the calibration and the reference gas oil runs, go back now and:

1. Save the most recent method (calibration or reference gas oil).
2. Load the solvent blank method and bring it up to date.
3. If needed, update the chromatographic conditions for the calibration method.
4. Rerun the solvent blank sample.
5. If needed, rerun the calibration sample and regenerate the report.

What Has Been Done to this Point

Up to this point you have:

- Created a solvent blank method (D2887_BLANK.M) and run solvent blanks to verify system cleanliness.
- Created solvent blank data file “001F010103.D”. (The example file name assumes default, automatic file naming and that only three solvent blanks were run.)
- Created a calibration method (D2887_CAL.M), run a calibration sample, adjusted the chromatographic conditions as needed, rerun the calibration sample, and calibrated the method.
- Created calibration data file “002F010101.D.” (The example file name assumes default, automatic file naming and that only one calibration sample was run.)
- Created a reference gas oil method (D2887_RGO.M) and run a reference gas oil sample.
- Created reference gas oil data file “003F010101.D.” (The example file name assumes default, automatic file naming and that only one reference gas oil sample was run.)
- If GC setpoints changed, updated the solvent blank method (D2887_BLANK.M) and rerun the sample.
- If GC setpoints changed, updated the calibration sample method (D2887_CAL.M) and rerun the sample.

This has created all of the data needed for the SimDis software to analyze an unknown sample and determine its boiling point range distribution.

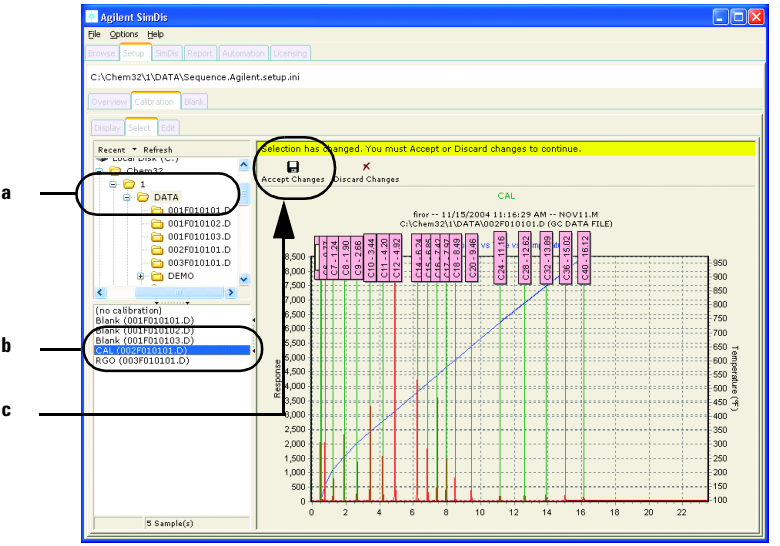
10 Set Up the SimDis Software for D 2887 Analysis

This step prepares the SimDis software to analyze unknown samples by using the data generated from the solvent blank, calibration, and reference gas oil samples.

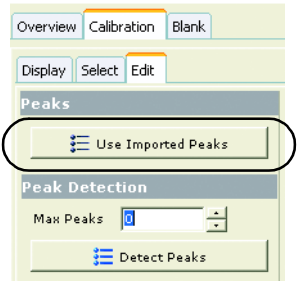
CAUTION Be sure to select the **most recent** solvent blank, calibration, and reference gas oil data files. Use of out-of-date data can cause inaccuracies due to retention time drift, system cleanliness changes, and similar phenomena.

1 Switch to the SimDis application. It should be shown in your Windows task bar. (If needed, launch it from its desktop icon or from the Start menu.)

2 Go to **Setup / Calibration / Select**. Locate the data folder for the calibration sequence and select the calibration file. For this example, browse to **..\Chem32\1\DATA** and select **002F010101.D**.



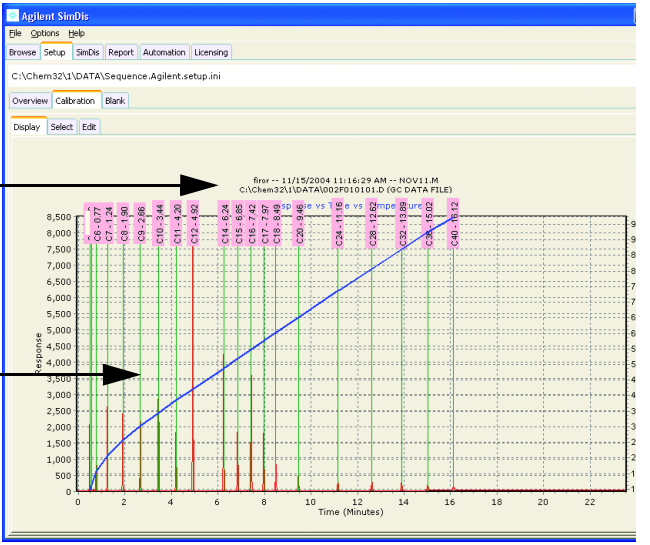
3 Go to **Setup / Calibration / Edit**. Click **Use Imported Peaks**.



4 Go to **Setup** / **Calibration** / **Display**. View the displayed chromatogram and verify the correct data file is loaded and that the chromatogram appears correct.

Verify correct data file.

19 calibrated peaks shown.

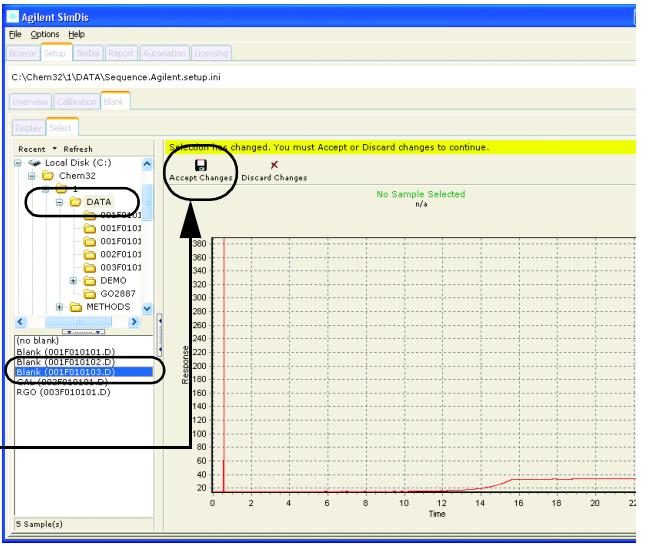


5 Go to **Setup** / **Blank**. Locate the data (sequence) folder containing the solvent blank data, then select the most recent (appropriate and acceptable) solvent blank data. Accept the changes. In this example, locate **..\Chem32\1\DATA**.

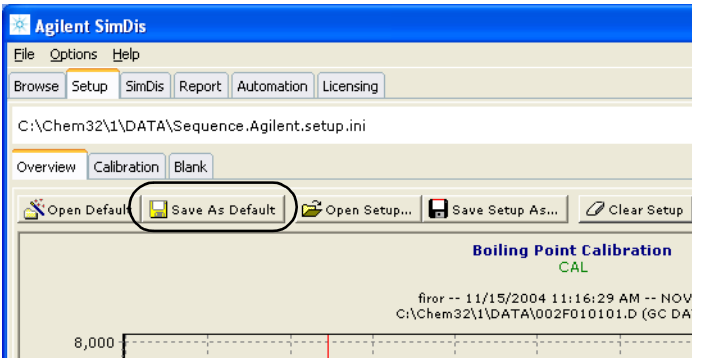
a

b

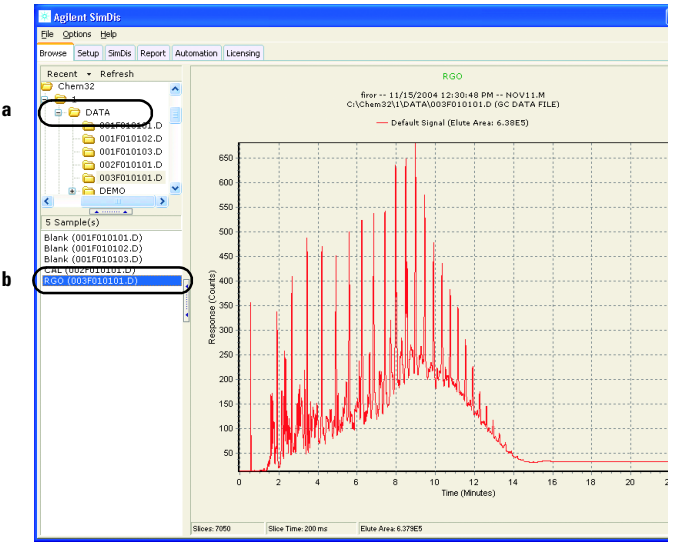
c



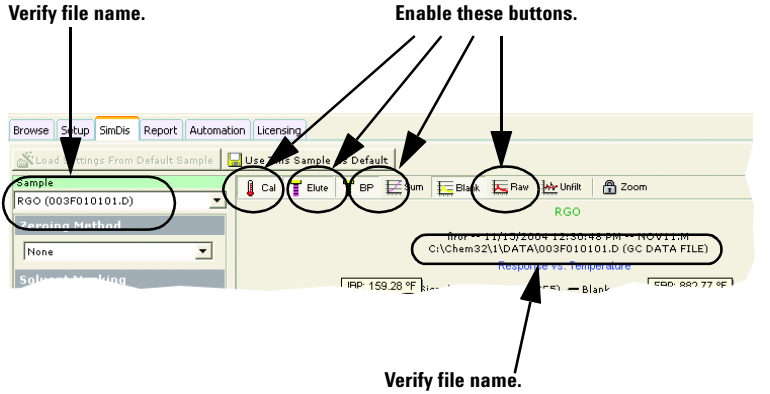
6 Go to **Setup** / **Overview** and click **Save As Default**.



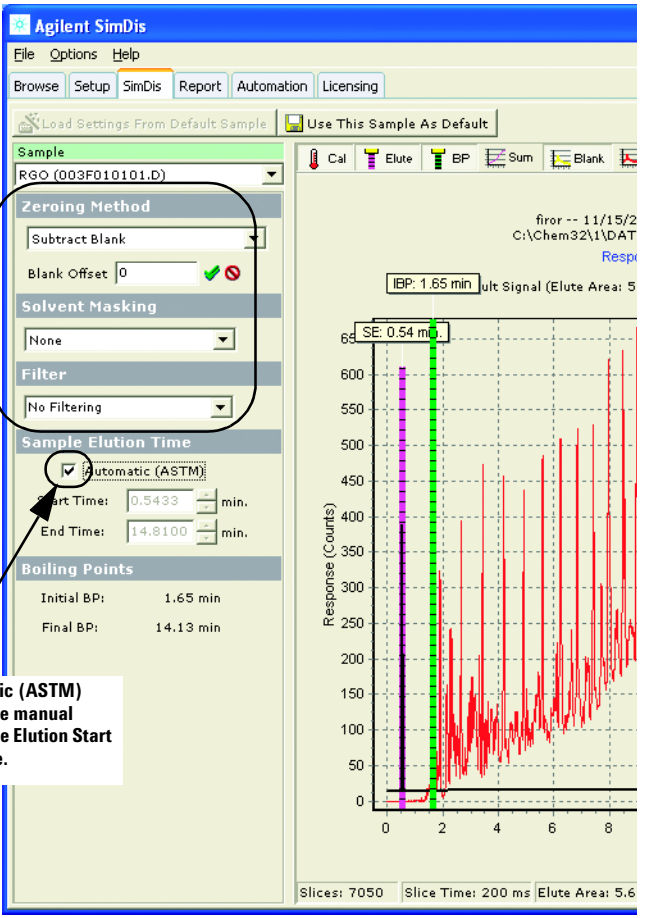
7 Go to the **Browse** tab. Locate the reference gas oil sequence folder, then select the reference gas oil (in this example, **RGO 003F010101.D**) sample data.



8 Go to the **SimDis** tab. Verify that the correct file data name is shown. Click to enable **Cal**, **Elute**, **BP**, and **Blank**.

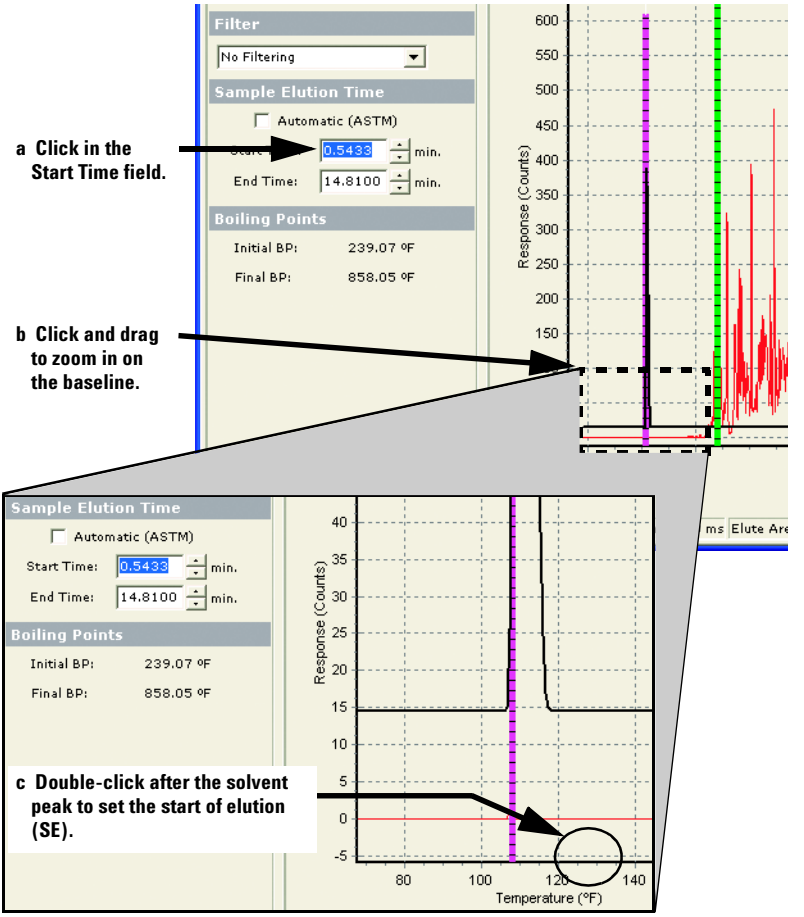


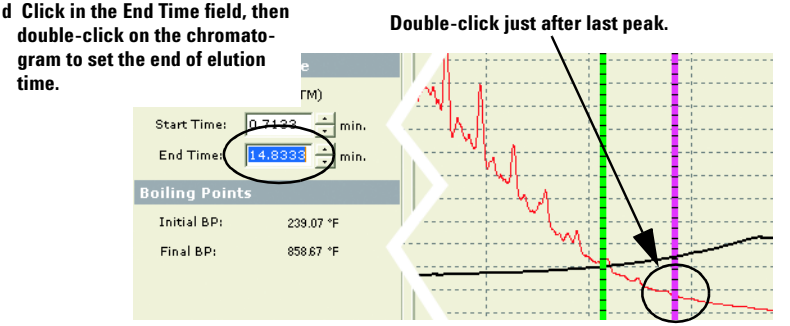
9 Set **Zeroing Method**, **Solvent Masking**, and **Filtering** as shown below. Clear the **Automatic ASTM** checkbox.



Clear the Automatic (ASTM) checkbox to enable manual selection of Sample Elution Start Time and End Time.

10 Set the sample elution times.



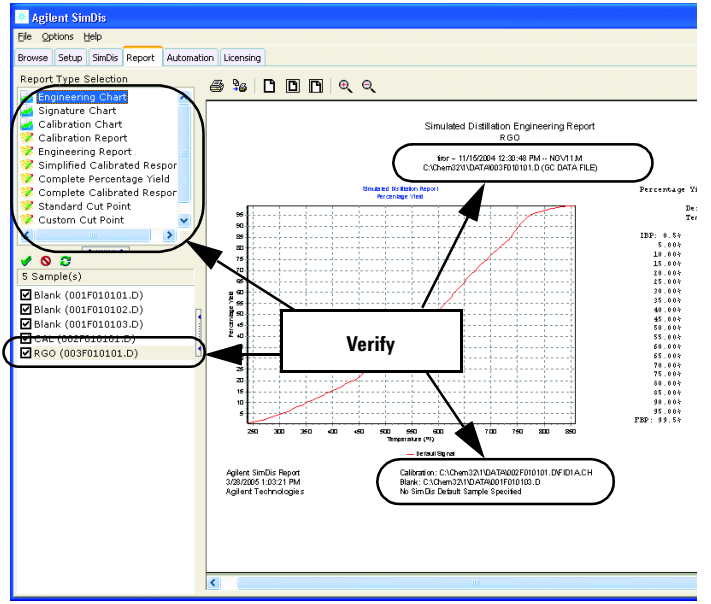


11 Go to the **Report** tab. The default report type is **Engineering Report**. Verify that:

- The correct data files are in use
- The desired report is selected
- The RGO sample is selected for printing

If not, repeat steps 2–10.

If satisfied with the results, return to the **SimDis** tab and click **Use This Sample As Default**.



12 Go to **Setup / Overview** and click **Save As Default** to save the current settings.

This completes SimDis software preparation. The software is now ready for automation using ChemStation sequences.

11 Create a D 2887 ChemStation Method for Automated SimDis Analysis

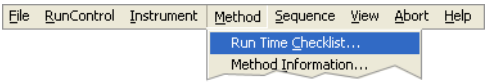
Now that the SimDis software is set up using the most recent, appropriate data, the next step is to create a ChemStation method suitable for running unknown samples.

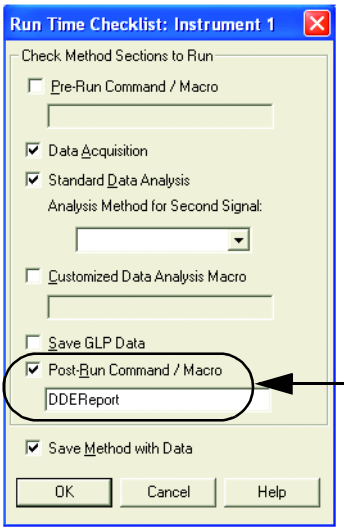
1 Go to the ChemStation online session.

2 Go to the **Method and Run Control View**.

3 Verify that the reference method (in this example, **D2887_RGO.M**) is loaded. If not, load it.

4 Set up for automation as shown below:





Select Post-Run Command/Macro and enter "DDEReport" exactly as shown.

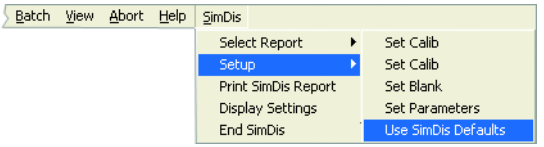
5 Save this method. For this example, save as **D2887.M**. This is the method to use for laboratory ASTM D 2887 SimDis samples. When run for a sample, by itself or in a sequence, it will now run the saved SimDis analysis automatically as a post-run event.

12 Select the ChemStation SimDis Report and Setup Options

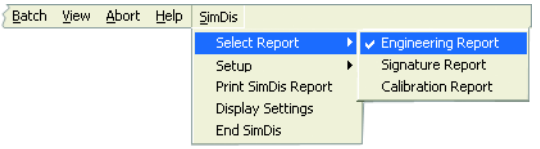
Now that the ASTM D 2887 method is ready, the last step is to choose setup and report options.

1 From the ChemStation, go to the **Data Analysis** view.

2 From the SimDis menu, select **Setup / Use SimDis Defaults**. This makes the SimDis software analyze new sample data using the blank, calibration, and SimDis parameters (from the RGO data file) that you just saved.



3 From the **SimDis / Select Report** submenu, select the type of report desired. The default is an Engineering report, which presents displays a Yield % Off graph and D 2887 percentage yield table.



The SimDis software is now set to automatically analyze laboratory samples and output the selected report type upon running a ChemStation sequence.

13 Run Laboratory Samples

To run the laboratory samples, create a sequence and use the final ASTM D 2887 method (in this example, **D2887.M**) for the samples. Start the sequence. As each sample run completes, the ChemStation will automatically invoke the SimDis software, which will generate the selected report. The ChemStation will then continue with the next sample.

Summary

Initial setup and the first set of runs are complete. The SimDis software has generated the requested reports. New reports can be generated from the existing data at any time. However, note that any changes made in the SimDis software **Setup** tab **immediately** take effect on the sample run files in the selected data subdirectory. Agilent strongly recommends reading the SimDis online manual to become more familiar with the operation of the software.

Also, remember to keep the chromatographic conditions of all four methods the same. The solvent blank, calibration, RGO, and unknown samples must be run under the same conditions.

Each laboratory should determine an appropriate frequency to perform system verification as outlined below:

1 Run the solvent blank.

2 When acceptable, run the calibration sample to verify the chromatographic and integration settings. Generate a report.

3 If needed, adjust the chromatographic conditions and rerun a solvent blank.

4 Run the RGO sample (or other known quality control sample) to verify overall system performance. Generate a report.

5 Use this collected run data to adjust SimDis data analysis settings, as needed, to accommodate current system performance. Save the changes as defaults.

6 Run the laboratory samples.