Agilent Technologies

Automated Data Acquisition and Processing with the Aromatics in Gasoline Analysis (RFG) Software

MS ChemStation G1701CA/G1701DA

Shown below is the Aromatics in Gasoline Analysis Software top level. To initiate a sequence of runs, choose **Sequence** and **Edit Sample Table Log**. This will enable the operator to enter individual sample and internal standard weights, sample density, and QC information (Sample Type). These values are then used to calculate weight and volume percent numbers for samples.
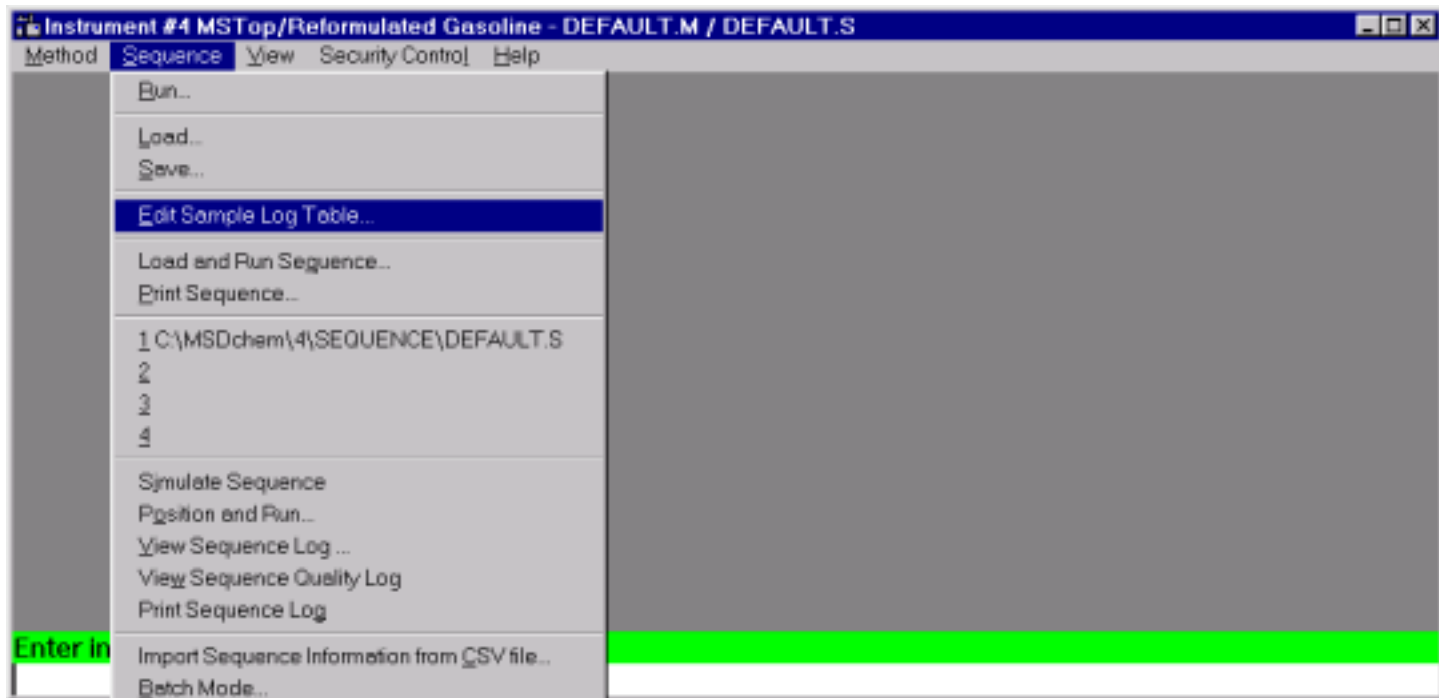
Sample Log Table for 26MAR02.S (on line 1 of 7)

Line	Type	Vial	Data File	Method	Sample Name
1)	Sample	1	DATA01	GAS04	Sample 1
2)	Sample	2	DATA02	GAS04	Sample 2
3)	Sample	3	DATA03	GAS04	Sample 03
4)	Sample	1	DATA04	GAS04	Sample 01-2
5)	QC	2	QCDATA01	GAS04	QC Sample
6)	Sample	3	DATA05	GAS04	Sample 03-2
7)	Sample	4	DATA06	GAS04	Sample 04-2

Type

Sample

Vial: 1

Data File: DATA01

Method: GAS04

Sample Name: Sample 1

Miscellaneous Information

Expected Barcode

Sample Amt: 0

Multiplier: 1

Gas Smpl Wt: 10.2344

Gas Density: 0.8888

Gas ISTD Wt: 0.2345

[Repeat] [Cut] [Copy] [Paste] [Read] [OK] [Cancel] [Help] [More>>]

Use the arrow keys to select entry

The sequence panel where individual sample information is entered. All weight information is entered in grams. You may choose an individual method for each

sample, although typically the same method will be used for all samples in a given sequence. A '?' entered in the method box will display a listing of the available methods for the instrument. If you want, you can update the default method box with the method of your choice and each sample will automatically have that method specified. Using the Repeat button will cause the vial number to increment, the method box to be filled out.   Weight information and Gas Density is entered on the lower panel as each line in the sequence table is selected. All information can be changed at a later time if desired. If you want to specify a new sequence file name to be used, select Sequence and Save. Enter the new file name. When you Exit, the new name will be used.

To initiate data collection, select Run from the Sequence menu. Notice the data file path has been set for you - indicating the date and hour the sequence file was set up. This will enable you to keep your files in order on the disk. Enter your Operator Name, and select Run Sequence. That's it! All data will be collected and processed, and a final weight and volume percent report printed out.

Data Processing with the Aromatics in Gasoline Analysis (RFG) Software

MS ChemStation G1701CA/G1701DA

The Aromatics Gasoline Analysis methods require some specialized handling of the standard Agilent Technologies software package where sample information entry and quantitation is concerned. The Automated Data Acquisition document describes how one enters individual sample weight, internal standard weight, and sample density. This document will outline how to enter the necessary information into the Data Analysis method to enable weight and volume percent numbers to be calculated and reported automatically.

There are basically three groups of compounds in the proposed ASTM method this software was designed to accommodate:

- Internal standards,
- Calibrated compounds
- Uncalibrated compounds

Each compound has some common information entries required in the method quantitation database such as retention time, ion to be used for quantitation, etc., as well as some unique assignments. Before beginning analysis, the supplied method must be verified to contain the correct information for all compounds.

To begin, start a Data Analysis session using the supplied GAS04.M method. Then, from the **Calibrate** menu option **Edit Compounds**, check each compound for the correct information.

Below is the compound panel page 1 (in this case, for an internal standard). Here, retention times, m/z values for quantitation, and compound names are specified. If the compound is an internal standard, you MUST place a value of 1 in the 'Sample ISTD Concentration' box for RFG calculations to be performed correctly. Don't worry about correct retention times yet - RFG software allows easy updates using mouse actions after you have run your first standard.

Compound #1: benzene-d6 (Page 2)

Special compound attributes

CAS #: 1076-43-3   Compound Type: I

Special reporting parameters

Surrogate amount: 0.000000   Matrix spike amount: 0.000000

	LOW	HIGH	% Dev	MDL
Matrix A (conc)	0.000000	0.000000	0.000000	0.000000
Matrix B (conc)	0.000000	0.000000	0.000000	0.000000

Signal level Minimum: 0.000000   Maximum: 0.000000

MS Database Name: _____   Entry #: 0

User defined items

A1	_____	N4	0.000000	N7	0.020000
A2	_____	N5	0.884500	N8	0.000000
A3	_____	N6	0.395900	N9	0.000000

[Prev] [Next] [Page 1] [Page 3] [OK] [Cancel] [Help]

This is the compound panel page 2 for an internal standard.
Important numbers here are the following:
N5 - Specific Gravity of compound
N6 - percentage of this internal standard in the ISTD mix
    used to spike samples.
N7 - Ws/Wg ratio for y-intercept test.
    Compound Type - I (for Internal Standard)

Remember there is only one area in the data acquisition setup to enter an internal standard weight. N6 is used to calculate the actual weight of this one internal standard compound. When an internal standard mix is replaced, these numbers must be updated accordingly.

Compound #4: Toluene (Page 2)

Special compound attributes

CAS #: [   ]   Compound Type: [   ]

Special reporting parameters

Surrogate amount: [0.000000]   Matrix spike amount: [0.000000]

	LOW	HIGH	% Dev	MDL
Matrix A (conc)	0.000000	0.000000	0.000000	0.000000
Matrix B (conc)	0.000000	0.000000	0.000000	0.000000

Signal level Minimum: [0.000000]   Maximum: [0.000000]

MS Database Name: [   ]   Entry #: [0]

User defined items

A1	[ ]	N4	0.000000	N7	0.000000
A2	[ ]	N5	0.871900	N8	0.000000
A3	[ ]	N6	0.000000	N9	0.000000

[Prev] [Next] [Page 1] [Page 3]   [OK] [Cancel] [Help]

This is the compound panel page 2 for a calibrated compound. Important numbers here are the following:

N5 - Specific Gravity of compound
N6 - percentage of this compound in a standard mix if standards are prepared from a common stock. This allows easy calibrated amount updates in the method.
N7 - N/A

Compound Type - N/A

Compound #30: C12 benzenes (Page 2)

Special compound attributes

CAS # [   ]   Compound Type [UN]

Special reporting parameters

Surrogate amount [0.000000]   Matrix spike amount [0.000000]

	LOW	HIGH	% Dev	MDL
Matrix A (conc)	0.000000	0.000000	0.000000	0.000000
Matrix B (conc)	0.000000	0.000000	0.000000	0.000000

Signal level Minimum [0.000000]   Maximum [0.000000]

MS Database Name [   ]   Entry # [0]

User defined items

A1 [ ]	N4 [25.000000]	N7 [0.000000]
A2 [ ]	N5 [0.920400]	N8 [0.000000]
A3 [ ]	N6 [0.000000]	N9 [0.000000]

[Prev] [Next] [Page 1] [Page 3] [OK] [Cancel] [Help]

This is the compound panel page 2 for an uncalibrated compound. Important numbers here are the following:

N4 - Compound Number of reference calibrated compound.
N5 - Specific Gravity
N6 - N/A
N7 - N/A

Compound Type - UN

Uncalibrated compounds have their response factors taken from a specified reference compound (N4). During calibration update, the calibration information for the reference compound - responses and amounts - is copied into the calibration table for the uncalibrated compound. The retention time window for a type UN compound (page 1) is set to encompass the range for elution one expects these compounds to have on the given chromatographic column.

Compound #28 -- Page 1   ☒

Name `C10 benzenes`   Concentration Units `mg`

Retention Time Information

Ret Time `26.800`   RRT 0.893

Extract signals from

`-` `5.650` `+` `1.183`   ⦿ Min ○ %

This is 21.150   to 27.983   min

Ref Spec Name `                    `

Signals to Be Used for Quantitation

Quant signal `Target Ion ▼`

	m/z	Relative Response	% Uncert `Re ▼`
Tgt	`134.00`	100.0	
Q1	`120.00`	`244.80`	`20.00`
Q2	`91.00`	`0.00`	`20.00`
Q3	`    `	`    `	`    `

Quantitation Parameters

Quant type `Target compound      ▼`

Measure response by `Area            ▼`

Ident by `Meets qualifiers, Best RT ▼`

Maximum number of hits `1`

Subtraction Meth. `Extend Area Quant ▼`

Calibration Information

Curve Fit `Linear Regr, Force (0,0)  ▼`

Weight `Equal weighting        ▼`

Linear term: 3.02e-001

Coef of Det (r^2):   0.998030

[Prev] [Next] [Plot] [Page 2] [Page 3] [OK] [Cancel] [Help]

This is page 1 for an Uncalibrated Aromatic (UN) compound. Note the extremely wide range that will be used to extract the signals - 5.16 to 28.496 minutes. Throughout this range, the extracted ion chromatogram of mass 134 will be integrated, and if any of the calibrated peaks with mass 134 are found, their area will be subtracted from the total area. This way the area for the uncalibrated peak is representative of the uncalibrated compound.

Each processed data file will have a 'petroint.txt' file located within the data file directory listing the integration summation and subtraction steps for troubleshooting purposes.

Calibration concentrations for each compound go on page 3 of the compound form. You may enter each compound amount in milligrams by hand, or use the **InitCal** menu option **Update Aromatic Amounts** and have them filled in automatically.

There are two ways the software will evaluate numeric entries and update the page 3 compound concentrations:

- Standard levels prepared from a common stock (Serial Dilution; requires N6 field entry),
- Individually weighed standard levels (requires text file in specific format - supplied by standards vendor).

Depending on your standard prep type, choose the appropriate option.
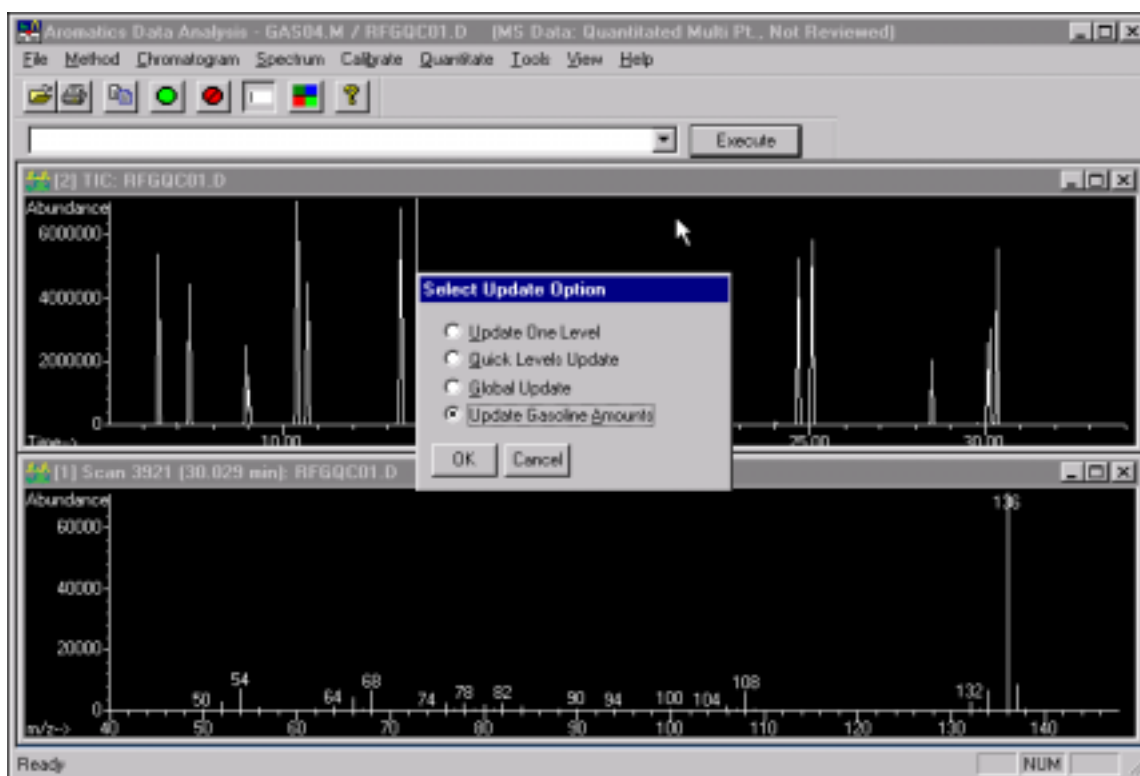
Select the appropriate Update Option:

Select Standards Preparation Type

○ Individual Level Weights
◉ Serial Dilution

[OK] [Cancel]

Selecting **'Serial Dilution'** will cause prompts for individual level TOTAL weights to be displayed. The N6 field will be used to calculated the actual weight of each individual component for each level.

Enter Total Wt of Analytes in mgs

Level ID: A1

Level ID: A2

Level ID: A3

Level ID: A4

Level ID: A5

[Save] [Cancel]

You will be prompted for Internal Standard weights after entry of Calibrated Compound values has been completed.

Selecting **'Individual Level Weights'** will bring up an input panel for specifying the text file containing all compound information. This file (typically "a:\calamts.txt") is to be provided by the standards vendor on a floppy disk. You should make sure the disk you are using corresponds to the lot number of the standards you analyzed, because each lot will have small differences in the actual weights. If you would like to create your own calamts.txt, please consult with your Agilent Technologies Analytical consultant for the required format of this file.

THE CALIBRATION AMOUNTS NEED ONLY BE CHANGED WHEN NEW STANDARDS ARE PREPARED AND A NEW CALIBRATION CURVE MUST BE CREATED.

This is true for ALL calibration and internal standard solutions. If a new internal standard mix is prepared for samples only (not calibration standards), you need only change the N6 field for the internal standards to correctly reflect their percentage values.

The method should now contain correct values for calibration amounts, reference compounds, and ISTD fractions. What is needed at this time is correct retention times, and for that you need to collect some data to allow review.

Save the method (File menu option Save Method), and use the Sequence menu (Top Level) option Make/View/Edit sequence to run one standard. Be sure to specify the correct weight, ISTD amount, etc., and then run the sequence. After the acquisition is complete, load the run just made in Data Analysis for viewing.

With the file loaded and chromatogram displayed, use the Tools menu option EasyID - you can now select the correct peak for each compound by performing a click/drag with the right mouse button under the chromatographic trace, then moving to the next compound in the list. If you have selected an internal standard, you may optionally elect to update all target compound RT's automatically. This is a useful feature for preexisting, in-house methods where some chromatographic maintenance has been performed; for initial setup it is recommended that you manually review EACH peak.
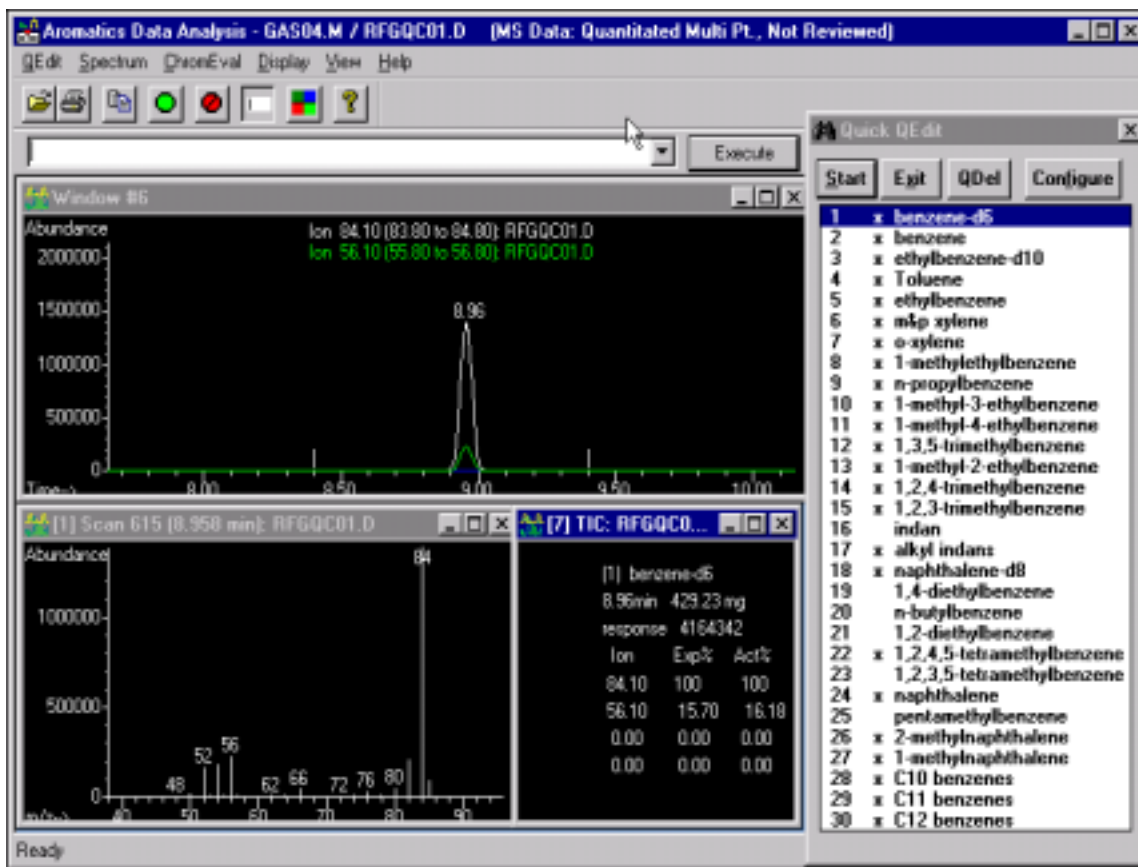
Here is an example of an EasyID process where the internal standard peak is not within the qualitative window set in the Data Analysis method. Performing a click/drag with the right mouse button under the peak in the chromatographic trace will update the method with the retention time of the peak selected. You will do this for each compound in the method; typically, this is done once during method setup.

Now that the retention times have been corrected, you will need to perform a quantitation to verify retention time entries, integration parameters, etc. Use the **Quant** menu option **Calculate and Generate Report**, and specify **Summary to Screen**. When the report appears, pay attention to listed retention times, not weight and volume percent numbers. Remember, the method is not calibrated to your standards, so calculated amounts at this point are not real. If all peaks have been located, save the method (**File … Save Method**), and run the remaining standards. You may get some printouts during the sequence; remember that the calibration is not yet valid. Again, pay no attention to calculated amounts, just compound recognition.

To update the calibration using your standards, you will need to do the following for EACH standard in a Data Analysis session with GAS04.M loaded up:

1. Load the data file (File menu option Load Data File)
2. Update appropriate level (**Calibrate** menu option **Update One Level**)
3. Select **Recalibrate**, select correct **Level ID** and **Replace** responses.

Now, you can view the results of your calibration by first requantitating any standard, then selecting from the **Calibrate** menu option **List** … **Select Calibration Report**. At this point, you can save your method to disk and begin running samples.

NOTE: If you are using two standard 'group' mixes and therefore have 10 levels, you MUST ensure that there are no false positive results in the quantitative results file. To do this, load the data file and run the QEDIT process from the Quant menu. If a false positive is detected (i.e. toluene reported in a run that is not supposed to have any toluene present) you must use the Qedit menu option QDEL to delete the false entry BEFORE the calibration update.