



smileq

SMILEQ plugin

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This document is intended to give the user an overview of the SMILEQ plugin for JASON. It is assumed that JASON is running under the Windows operating system.

SMILEQ plugin

The SMILEQ plugin enables quantitative NMR analysis within JASON. It allows you to obtain the results of purity and molar concentration analysis of the analytes in a sample using an internal standard. The plugin is compatible with JASON ver1.3 or higher, and requires an additional SMILEQ license alongside the main JASON application.

In Chapter 1, how to activate the SMILEQ plugin and each tool in SMILEQ plugin will be explained.

1. Activate SMILEQ plugin

To get started with SMILEQ, you need to activate the plugin.

1.1. Start Plugin Manager

Select the Plugin Manager from the Main Menu, as shown in Figure 1.

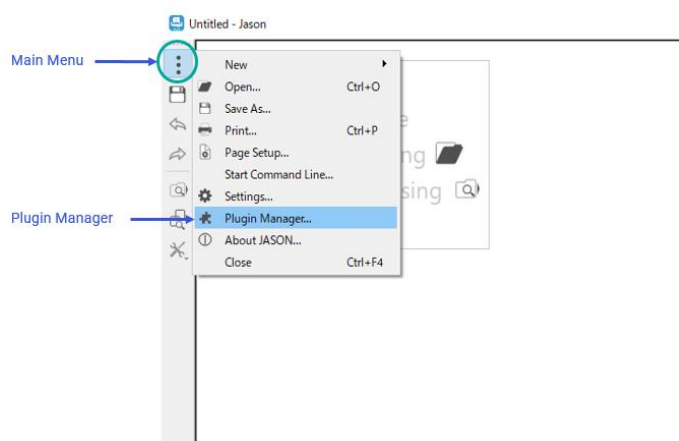


Figure 1: Start Plugin Manager

1.2. Install SMILEQ Plugin.

Check the SMILEQ Plugin checkbox and click the OK button, as shown in Figure 2.

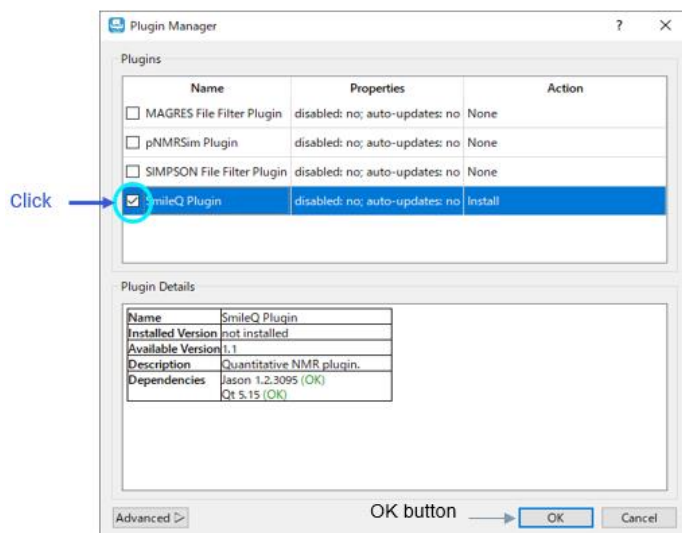


Figure 2: Start Plugin Manager

1.3. Accept Restart Dialog

After pressing the OK button, the dialog box shown below will appear. Click OK and then restart JASON

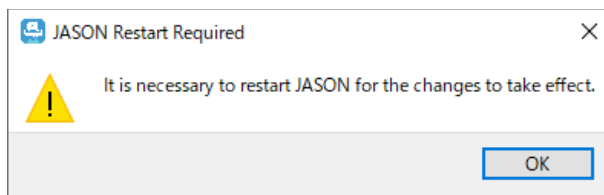


Figure 3: Restart Dialog

1.4. Activate SMILEQ License

The first time JASON is opened after installing the SMILEQ plugin, the JASON Plugin Licenses Dialog window will open, as shown in Figure 4.

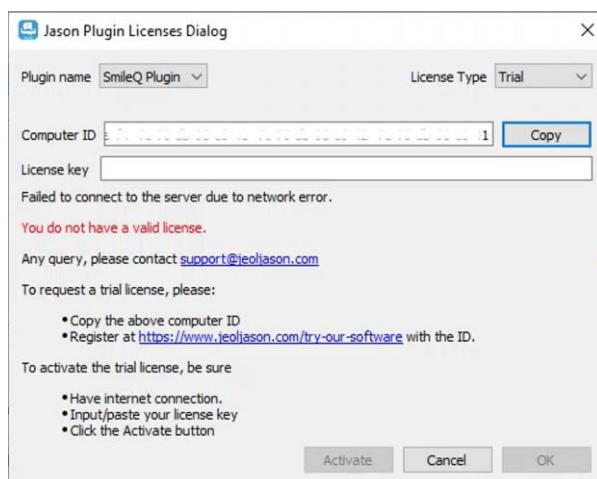


Figure 4: JASON Plugin Licenses Dialog (before the setting)

☞ If you have already received your license key, please proceed to step 3 in the procedure below.

1. Copy the Computer ID by clicking on the 'Copy' box.
2. Contact Support at support@jeoljason.com to request a SMILEQ license key quoting the Computer ID.
3. Once you receive the License Key, copy and paste it into the license key box.

☞ The Activate and OK buttons will become active once a valid license key has been entered.

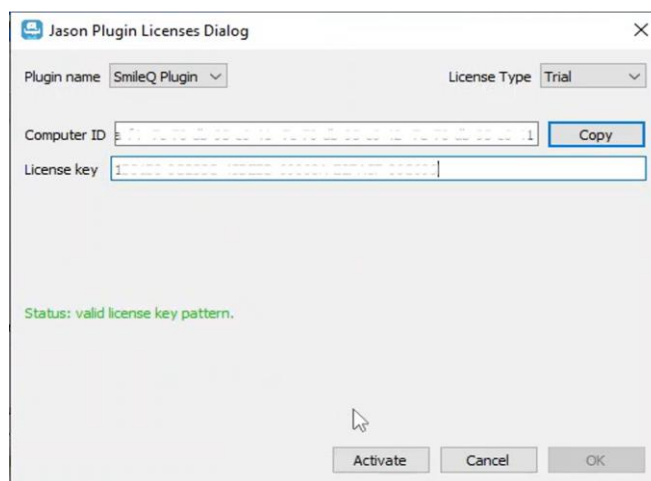


Figure 5: JASON Plugin License Dialog (after the setting)

4. Click the Activate button to activate the license key. The JASON software will open now.

1.5. Re-open JASON

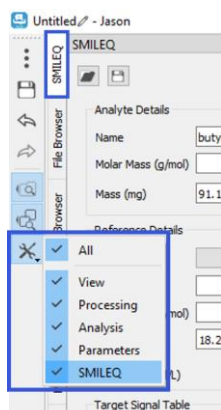


Figure 6: SMILEQ context menu and panel

After successfully registering the SMILEQ plugin, the functionality can be accessed using one of the context panels on the left-hand edge of the frame.

Like the other NMR tool widgets (e.g., View and Processing), the SMILEQ panel requires NMR data to be loaded onto the canvas. The SMILEQ widget will appear automatically when appropriate data is selected.

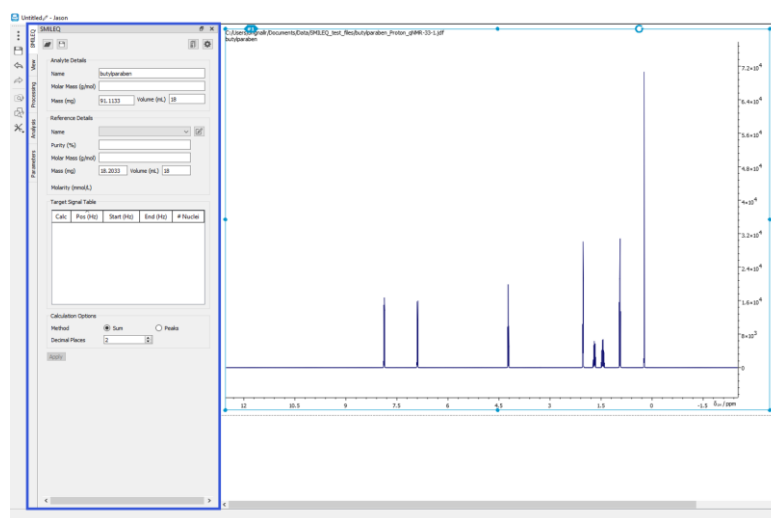


Figure 7: SMILEQ Widget

2. Overview of SMILEQ Widget

The SMILEQ widget is used to set the parameters for qNMR analysis, as is shown in Figure 8.

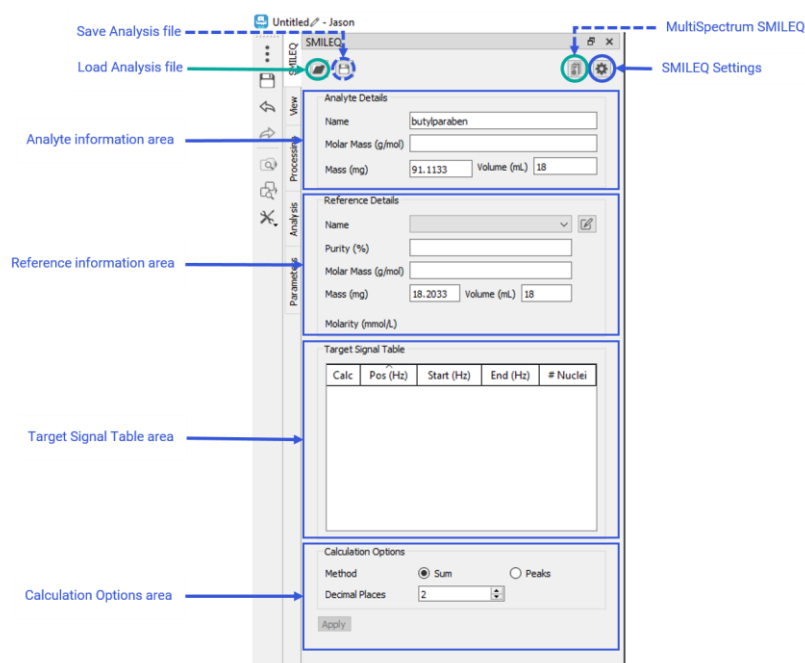


Figure 8: SMILEQ Widget

Analysis file Load/Save	Buttons to load/save analysis template files
SMILEQ Setting	The SMILEQ settings tab allows the user to define the paths where various configurations files are stored. The details will be explained in section 3.
Analyte Information Input Area	Area for defining the sample information required for quantitative analysis.
Reference Information Input Area	Area for defining the reference compound information required for quantitative analysis.
Target Signal Table Area	Area for entering the number of resonances in the spectrum and whether they should be used for quantitative analysis. These resonances can be defined as either Multiplets or Integrals. The multiplet and integral analysis tools available in JASON can be used to generate the entries in this table
Calculation Option Area	Determines whether SMILEQ uses the sum integral or deconvolution peak area for the quantitative calculation
MultiSpectrum SMILEQ	The MultiSpectrum button allows to perform the batch qNMR analysis across multiple analyte spectra.

2.1. Analyte Information Input Area

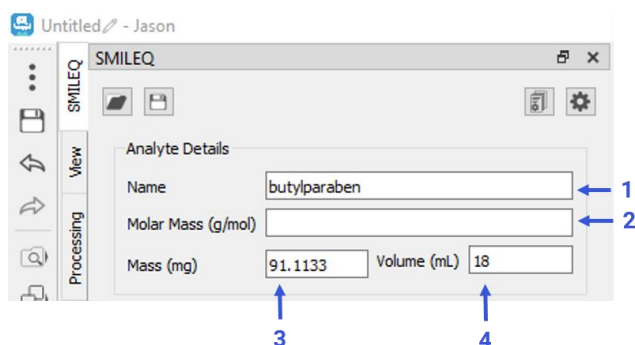


Figure 9: Setting of Analyte information Input Area

1. Enter a sample identifier in the “Name” text field.
2. Enter the molecular weight in the “Molar Mass” text field in units of g mol^{-1} .
3. Enter the weight of the sample in the “Mass” text field in units of mg.
4. Enter the volume of sample in the “Volume” text field in units of mL. This value is only required if molarity results are needed. The volume parameter entered here is the volume of solution in which the analyte sample was prepared, not the volume in the NMR tube.

The “**Name**”, “**Molar Mass**” and “” fields must have values entered in order to perform the quantitative analysis.

2.2. Reference Information Input Area

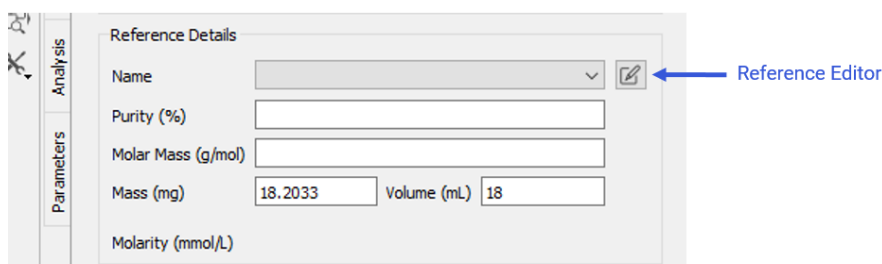


Figure 10: Setting of Reference Information Input Area

1. Select a Reference sample from the “Name” combo box.

This drop-down list can be edited using the Reference Editor button. See section 2.3, Reference Editor for details.

When a reference is selected, the values for purity (**Purity (%)**) of the reference compound and its molar mass (**Molar Mass**) are automatically loaded.

2. Enter the weight value of the reference in the "Mass" text field
3. Enter the volume of sample in the "Volume" text field, if molarity results are required.
Note, this is the volume of sample prepared, not the volume of the NMR sample itself.

☞ The **Name**, and **Volume** fields must be completed in order to perform the analysis.

2.3. Reference Editor

Each line in the reference editor contains all the information regarding a particular reference substance used for quantification.

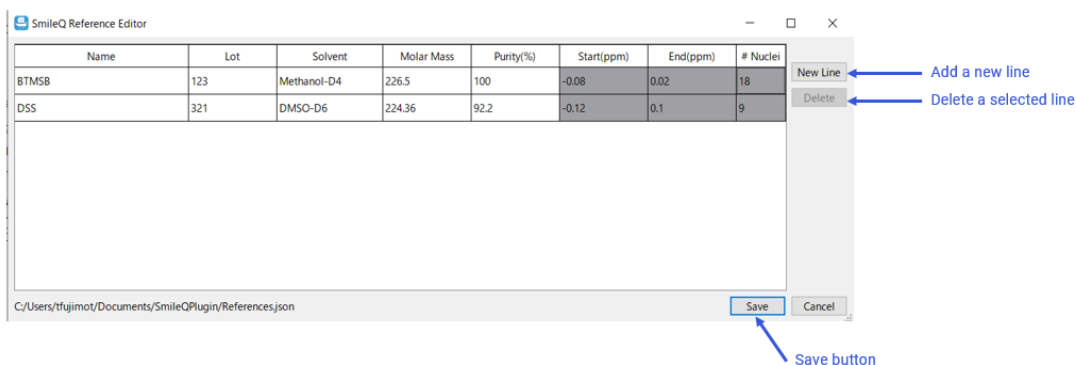


Figure 11: Editing of the Reference Editor Area

Click the "**New Line**" button to add a line to the table.

Clicking the "**Delete**" button will delete the selected line.

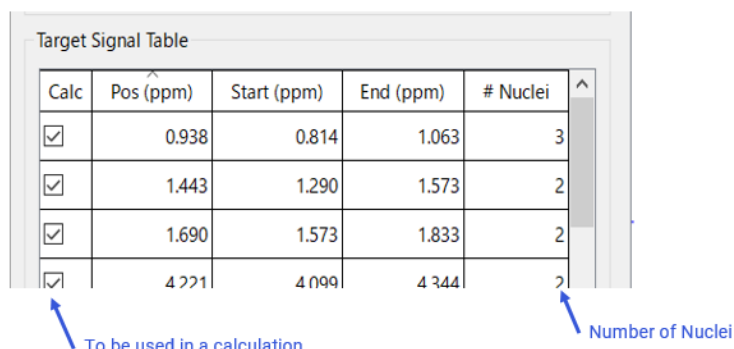
Click the "**Save**" button to save the reference information to a local file.

If any cells are red then this means that the cell contents are invalid or missing information. The "save" button cannot be pressed until these issues are rectified.

Pressing the Save button will save the changes made to the file which is defined in the.

2.4. Target Signal Table Area

All multiplets or integral regions defined on the spectrum will be shown in the Target Signal Table, see Figure 12.



Calc	Pos (ppm)	Start (ppm)	End (ppm)	# Nuclei
<input checked="" type="checkbox"/>	0.938	0.814	1.063	3
<input checked="" type="checkbox"/>	1.443	1.290	1.573	2
<input checked="" type="checkbox"/>	1.690	1.573	1.833	2
<input checked="" type="checkbox"/>	4.221	4.099	4.344	2

Figure 12: Editing of the Target Signal Table Area

1. A line will be added automatically if a new multiplet or integral region is defined.
2. The **Calc** checkbox is used to identify which multiplets or integral regions should be used in the quantification calculation.
3. Enter the number of identical nuclei associated with the multiplet or integral region in the column “# Nuclei”.

2.5. Calculation Options

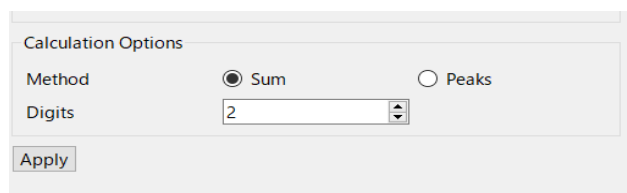


Figure 13: Setting of Calculation Options Area

1. The purity and/or concentration of each analyte can be calculated using two slightly different approaches.

Sum: This uses a point by point summation across the defined region to calculate the integral.

Peaks: This uses the sum of the peak areas in the region as determined by the peak deconvolution algorithm.

2. The number of digits shown in the results table is defined by the “Digits” box
3. Click the **“Apply”** button to perform the quantitative analysis, and create the quantitative results table and quantitative parameters report on the canvas.

☞ If the analysis results are already available, the **“Apply”** button will change to the **“Update”** button, and the results will be updated when the button is pressed.

2.6. MultiSpectrum SMILEQ

MultiSpectrum SMILEQ allows the analysis of multiple datasets loaded onto the canvas. Once an analysis file has been defined in the main SMILEQ settings (from the cogwheel button), pressing the MultiSpectrum SMILEQ button opens dialog shown in Figure 14.

No.	File Name	Processing File Name	Analysis File Name	Analyte Name	Analyte MolMass	Analyte Weight (mg)	Analyte Volume (mL)	Reference Weight (mg)	Reference Volume (mL)
#1	<input checked="" type="checkbox"/> butylparaben_1_Proton_qNMR-20-1...			butylparaben_1	91.1133			18.2033	
#2	<input checked="" type="checkbox"/> butylparaben_1_Proton_qNMR-21-1...			butylparaben_1	91.1133			18.2033	
#3	<input checked="" type="checkbox"/> butylparaben_1_Proton_qNMR-22-1...			butylparaben_1	91.1133			18.2033	

Figure 14: MultiSpectrum SMILEQ

1. Entries in the "Processing File Name" and "Analysis File Name" columns can be selected from the dropdown button which appears when you click on the appropriate cell in the table. You can then select the appropriate file from the list provided.
2. Other parameters in the table can be entered as necessary.

Click "" to run the quantitation calculation across the selected spectra. The results will be displayed on the Purity Result tab and/or Molarity Result as appropriate.

Checking the "Reports of result" box will generate the quantitative results table and quantitative parameters report for each individual spectrum selected for analysis.

3. Preparing for qNMR analysis

The SMILEQ plugin has two uses. Firstly, it be used to perform a manual quantification calculation and report the results for a given spectrum. These results can be purity and/or molarity. Secondly, using the relevant configurations files, automated quantification can be performed. These procedures will be explained chapter 4 Analysis Procedure.

This chapter describes how the various configuration files for SMILEQ are prepared in advance of the analysis.

3.1. Necessary Files

To use the automated analysis, SMILEQ needs a reference file and an analysis file before starting the analysis.

To perform a manual quantification calculation, it is not necessary to create the analysis file.

3.1.1. Creating a Reference File

The SMILEQ plugin allows the user to save reference substance information in the reference file.

1. Select the **"Reference Editor"** button in the reference information input area.
2. Add the new line and enter the following parameters:

Name:	name of the reference material
Lot:	lot number of reagents if available
Solvent:	Select solvent from the combo box
Molar Mass:	Molar Mass of reference substance (in g/mol)
Purity (%):	purity of the reagent
Start, End (ppm):	the integration range for the location of the reference signal
Nuclides:	number of nuclides corresponding to the reference signal

3. Press the Save button.

☞ "2.3 Reference Editor" is used to create and update the reference file.

3.1.2. Creating an Analysis File

The SMILEQ plugin also allows the user to create an analysis file for efficient, automated, qNMR analysis.

The parameters used in a calculation can be saved as an analysis template file as a Jason Quantitative Analysis File (.jqaf).

To create the file, first the desired data is prepared and then the analysis is conducted.

The parameters for analysis can be saved, except that for the "Mass" of the sample. The saved file can then be used to perform quantification on other NMR data acquired under the same conditions as those used for the saved analysis template.

☞ The procedure for analysis can be found in section 4.1, "Manual quantitation analysis".

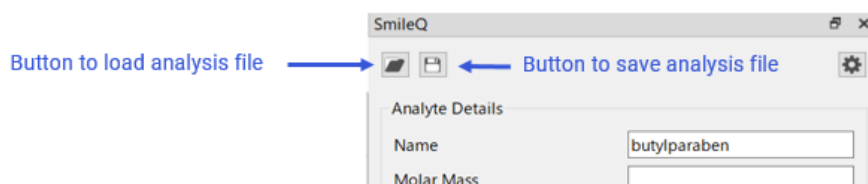


Figure 15: Button to save and load analysis template file

3.2. Necessary Settings

The SMILEQ settings tab allows the user to define the paths where various configurations files are stored.

3.2.1. Settings (General)

These settings are related to quantification with SMILEQ plugin.

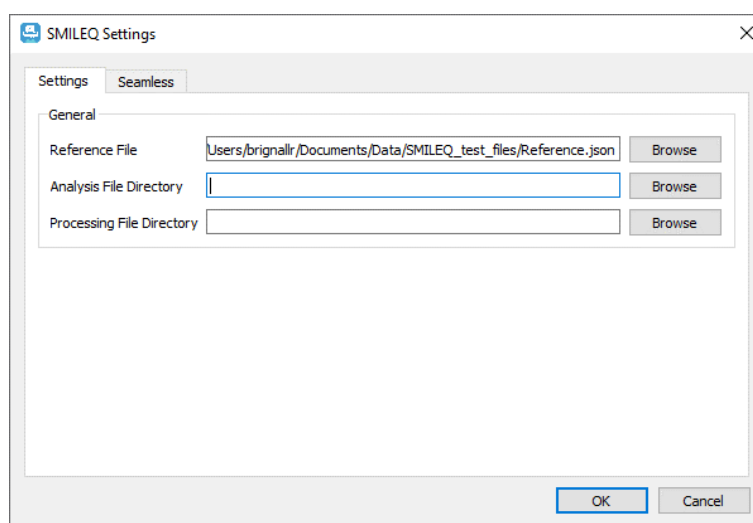


Figure 16: General Settings

Reference File: Location where reference file is saved. By default, this location is set to C:/Users/<user name>/json/plugins/smileq

Analysis File Directory: Location where you want to store analysis file that is Jason Quantitative Analysis File (.jqaf).

3.2.2. Seamless

Seamless is an automatic data acquisition, processing and reporting workflow for quantitative NMR (qNMR), which combines Delta (JEOL NMR standard software) and JASON in a single application. JASON receives the NMR data (FID) acquired using Delta and then conducts data processing, quantitation analysis and making reports the results using the SMILEQ plugin automatically.

This section describes settings related to SMILEQ for Seamless which means the automatic qNMR analysis by using Delta and SMILEQ plugin. If you do not need to conduct analysis using Seamless, these settings are not necessary.

In the case of Seamless, the details of the calculations can be configured using the JASON SMILEQ plugin and the details saved as a Jason Quantitative Configuration File (.jqcf) file. Using the tool below, found on the Seamless tab in the SMILEQ settings, click the Save button to create the configuration file used by SMILEQ.

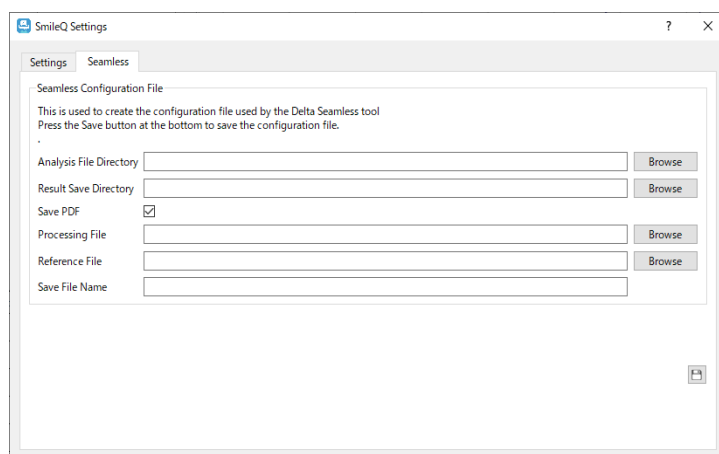


Figure 17: Configuration for SMILEQ setting

Analysis File Directory:	The directory where to store analysis file that is Jason Quantitative Analysis File (.jqaf) will be used SMILEQ.
Result Save Directory:	The directory where the results documents of SMILEQ will be saved.
Save PDF:	Option to create a PDF as results document.
Processing File:	The JASON processing file which contains the complete list of processing steps to be applied to the NMR data prior to the qNMR analysis.
Save File Name:	Name to use for the results document.
Reference File:	Reference file which contains the list of reference substance information for analysis.

- ☞ If "**Processing File**" is not set, the default processing list will be applied.
- ☞ If "**Save File Name**" is not set, the analysis document will be saved with the name "result".

The name of the analysis document will be given a date and time.

- ☞ Delta V6.1 is required to perform SMILEQ.
- ☞ For more detailed information, please refer to the Delta user's manual "SMILEQ – automated qNMR analysis"

3.3. Integration and Baseline correction

The guidelines relating to qNMR state that accurate phase- and baseline correction are necessary for high quality qNMR analysis. To ensure reproducible results, the baseline processing item should be included in the processing list, and the auto baseline setting in the Multiplets/Integrals section of JASON's global settings should be switched off. This option can be found under the settings tab, shown in Figure 18.

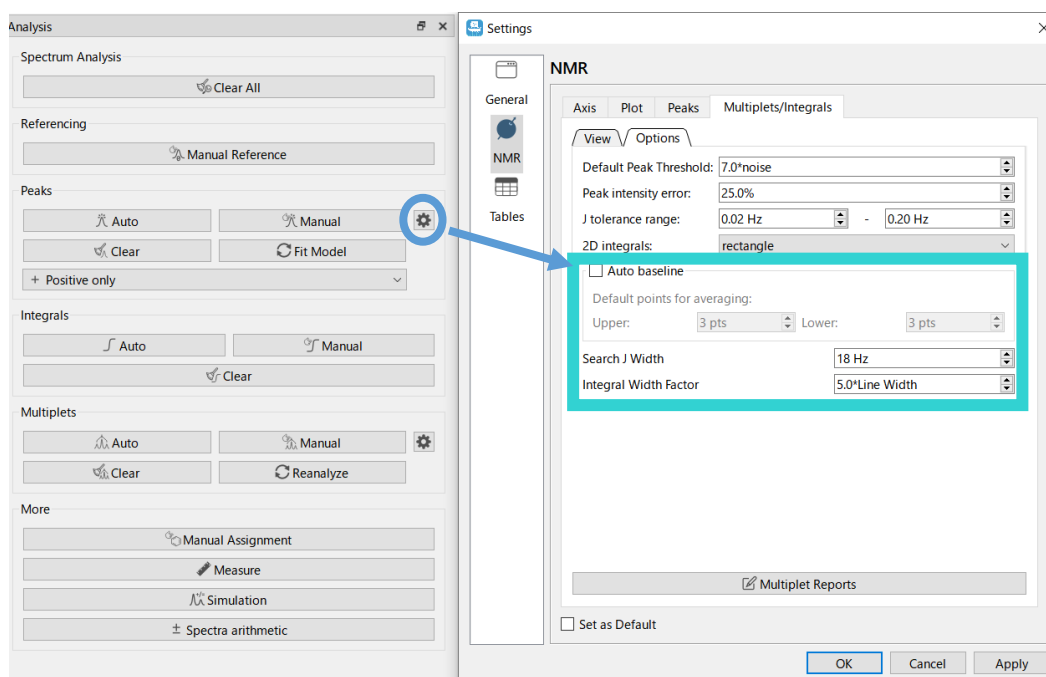


Figure 18 : Setting the auto baseline correction for integrals in JASON

If baseline correction is not included in the processing list, then ensure you active the auto baseline under the integration settings.

4. Analysis Procedure

SMILEQ plugin provides several analysis procedures. In this chapter, each procedure will be described.

4.1. Manual quantitation analysis

☞ The movie is available URL: <https://youtu.be/ZDecamUawH4>

This procedure is used to perform a manual quantification analysis and report the results for a given spectrum loaded onto the canvas.

Before undertaking any quantitative analysis using SMILEQ, ensure that all standard NMR data processing steps have been performed, including apodization, zero filling, FFT, phase and baseline correction and chemical shift referencing. Details on how to perform these operations can be found in the main JASON manual, and on the JEOL JASON website and YouTube channel.

1. Using the tools on the analysis panel, multiplet or integral regions can be set, either automatically or manually as desired.

☞ Signals such the reference substance, solvent(s) or impurities can be excluded at this stage. (The reference signal will be automatically integrated when the reference compound details are set later).

2. Select the SMILEQ widget and enter the required parameters in the sample information area (see section 2.1 for details of the necessary information and Figure 20).

☞ Where the raw data (FID) has parameters such as mass or volume stored, these values will be set automatically in the appropriate fields on the SMILEQ widget. It is only compatible data that acquired by "qNMR seamless" function. (see 3.2.2)

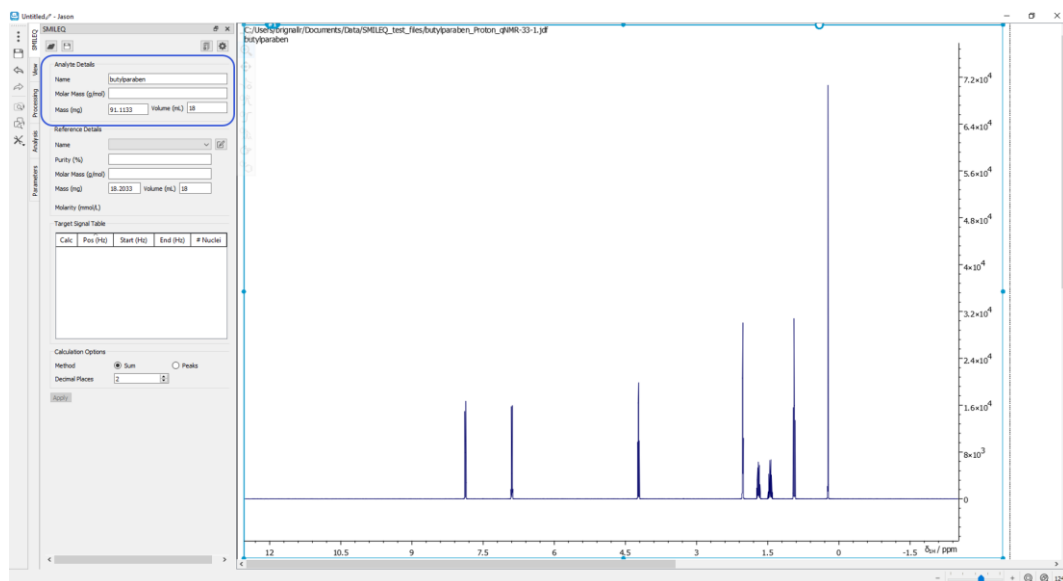


Figure 19: SMILEQ widget after conducting multiplet analysis

- Fill in the box at reference information area. Select the desired reference from the **"Name"** and enter the reference parameters required for the analysis (see section 2.2 and Figure 21.)

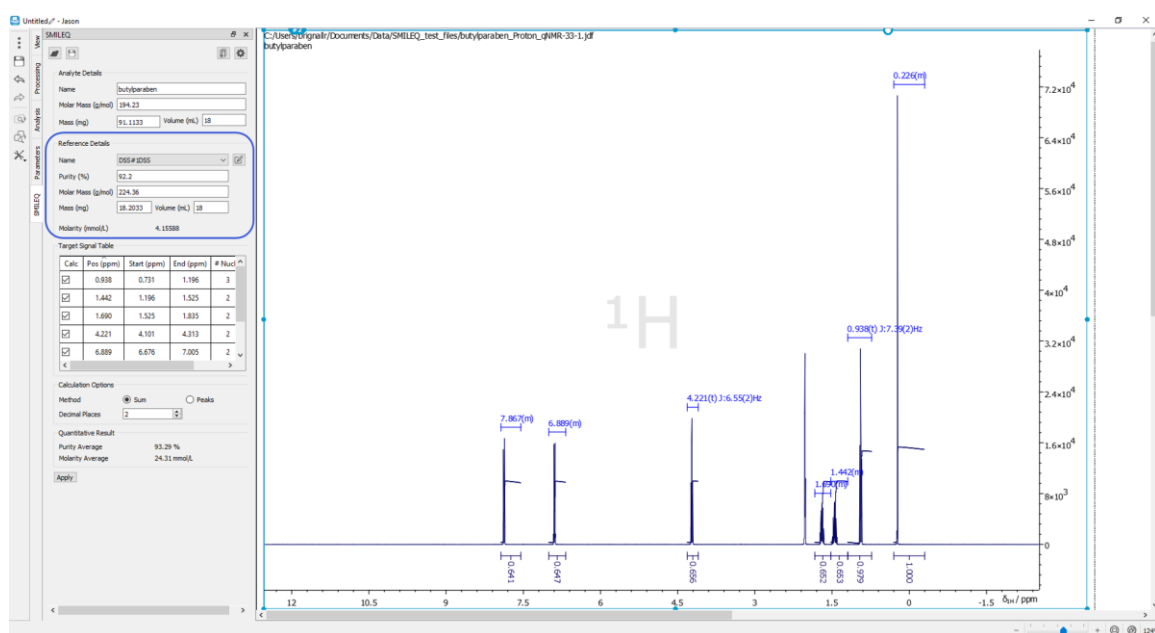


Figure 20: SMILEQ widget after selecting the reference

- Confirm the **"Target Signal Table"** details. If necessary, manually (de)select the signal for the analyte or enter the nuclides information as shown in Figure 22.

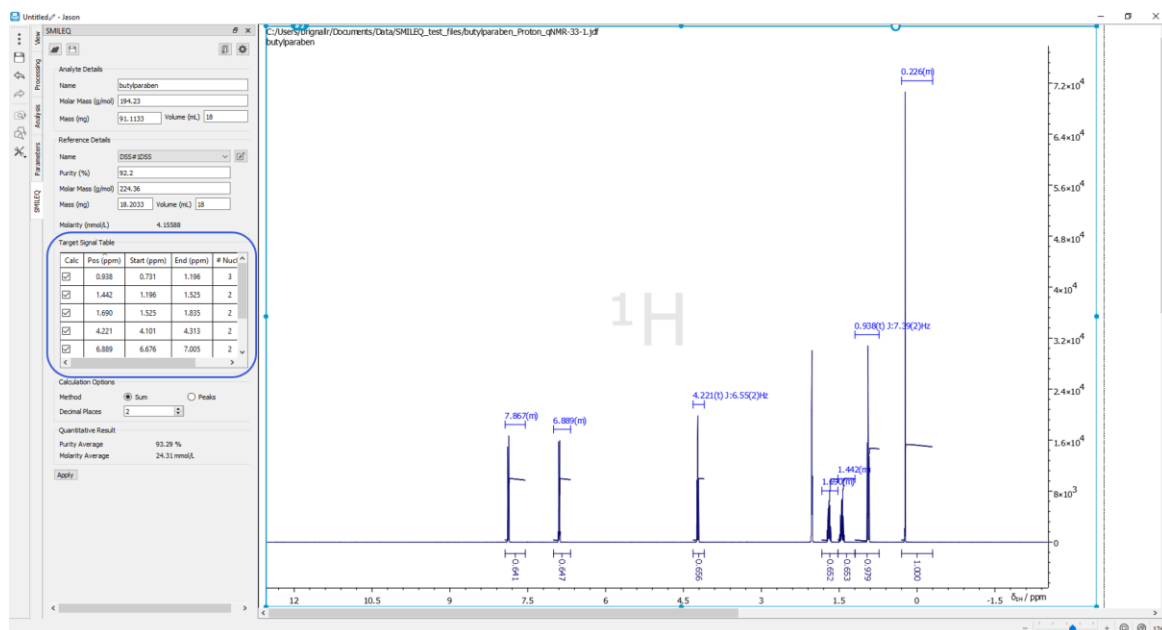


Figure 21: SMILEQ widget after confirming the target signal table

- After setting the calculation option, press the **“Apply”** button to view the result as shown in Figure 23.

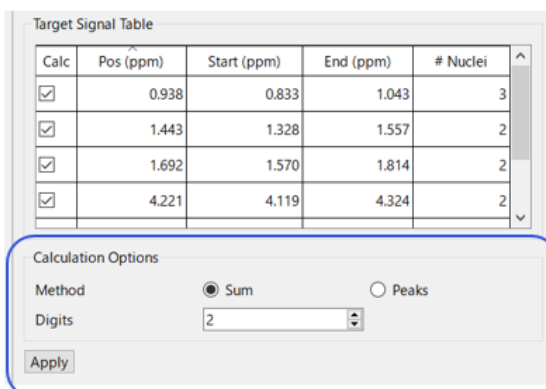


Figure 22: Calculation option on the SMILEQ widget

Figure 23 shows the result of a successful quantitative analysis, with the report and parameters tables being added to the canvas.

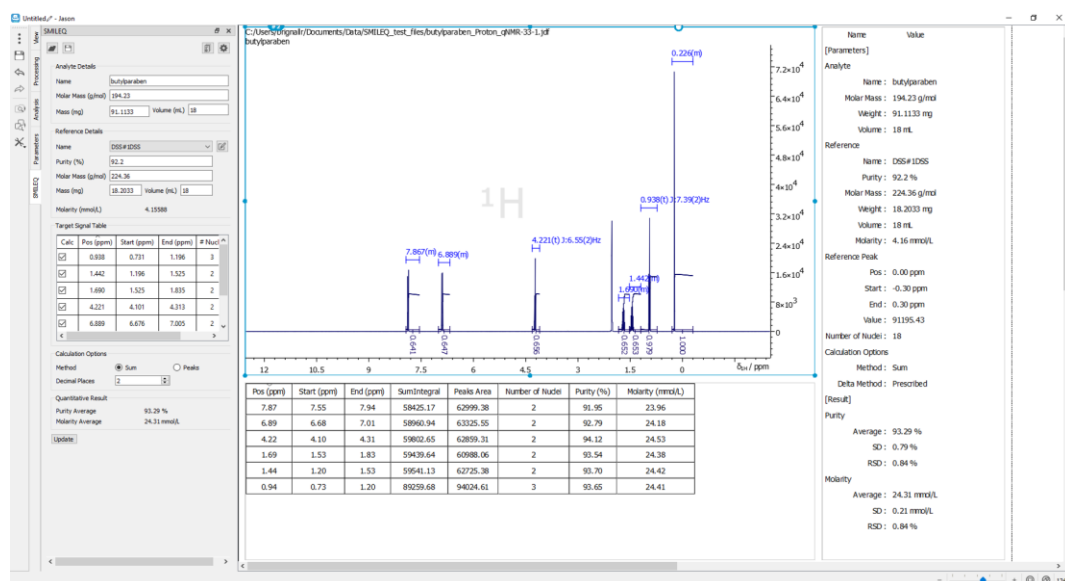


Figure 23: The result of quantification analysis

- ☞ The details of the analysis conditions used can be saved as an analysis file (.jqaf file). See Section 3.1.2 “Creating a Reference File”. This analysis file is necessary for the procedure described below in Section 4.2 “Automated quantitative analysis”.

4.2. Automated quantitative analysis

The movie is available URL: <https://youtu.be/qrk803hcQJw>

This procedure can be used to perform automatic analysis by using an analysis file that was configured for quantification analysis in advance (see section 4.2).

Before initiating the analysis using the SMILEQ widget, ensure that all processing of the NMR data (apodization, zero filling, FFT, phase and baseline correction and chemical shift referencing) has been performed.

The automated quantitative analysis is then performed as follows:

1. Select the SMILEQ widget and load the jqaf analysis file using the load button.

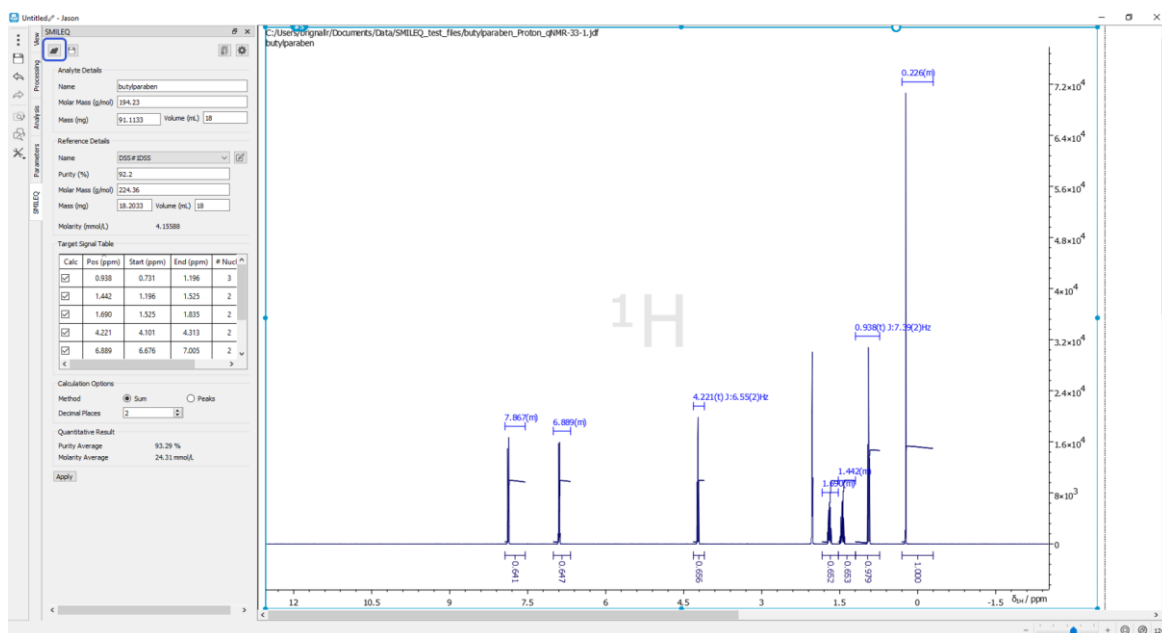


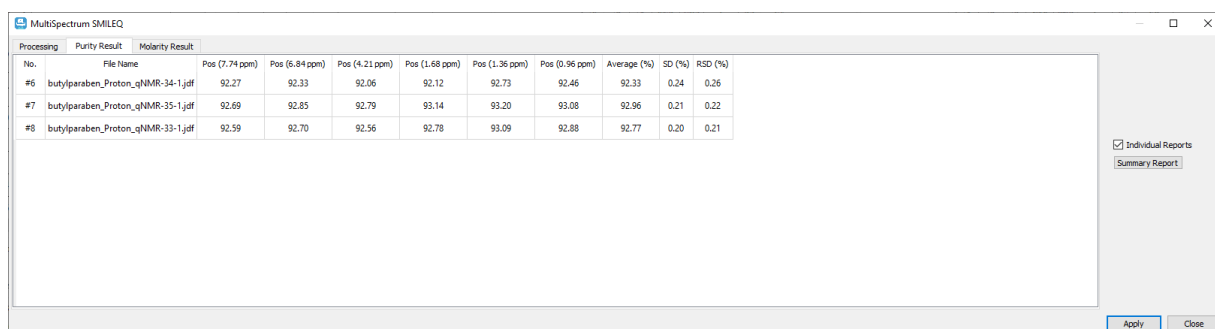
Figure 24: SMILEQ widget after loading the template file for analysis

2. Fill in any blank fields in the sample information area and reference information area if necessary.
3. Press the **"Apply"** button to view the result. The report and parameters tables will be added to the canvas.

4.3. Multi Spectrum Analysis

This procedure can be used to perform quantitative analysis across multiple spectra of the same analyte by using a processing file and an analysis file that was configured for quantification analysis in advance.

1. Load the datasets into canvas and ensure that all process of the NMR data has been performed.
2. Select the SMILEQ widget and open MultiSpectrum SMILEQ.
3. The column of "Processing File Name" and "Analysis File Name" is dropdown button, it is able to select the desired setting condition. Setting of other parameters will be set if necessary.
4. Click "Apply" to run the calculation and will be display the list of result on Purity Result tab and/or Molarity Result.



No.	File Name	Pos (7.74 ppm)	Pos (6.84 ppm)	Pos (4.21 ppm)	Pos (1.68 ppm)	Pos (1.36 ppm)	Pos (0.96 ppm)	Average (%)	SD (%)	RSD (%)
#6	butylparaben_Proton_qNMR-34-1.jdf	92.27	92.33	92.06	92.12	92.73	92.46	92.33	0.24	0.26
#7	butylparaben_Proton_qNMR-35-1.jdf	92.69	92.85	92.79	93.14	93.20	93.08	92.96	0.21	0.22
#8	butylparaben_Proton_qNMR-33-1.jdf	92.59	92.70	92.56	92.78	93.09	92.88	92.77	0.20	0.21

Figure 25: The table for Purity Result

5. When "Reports of result" check box is ON, the individual report will be generated.
6. When the "Summary Report" button is pressed, the summary report will be generated.

Summary Report (Purity)

Sample name	Pos (ppm)	Run 1	Run 2	Run 3	Ave (%)	SD (%)	RSD (%)	Ave (%)	SD (%)
butylparaben	7.87	92.27	92.69	92.59	92.52	0.22	0.24	92.68	0.34
	6.89	92.33	92.85	92.70	92.62	0.27	0.29		
	4.22	92.06	92.79	92.56	92.47	0.37	0.40		
	1.69	92.12	93.14	92.78	92.68	0.51	0.55		
	1.44	92.73	93.20	93.09	93.01	0.24	0.26		
	0.94	92.46	93.08	92.88	92.81	0.32	0.34		

Figure 26: The summary report

5. Results

In all cases, the output of SMILEQ will include; the spectrum and related parameters, and the analysis results. The mean, SD and RSD for the purity are calculated and displayed at the bottom of the results summary.

In Chapter 5.1, the report of results will be explained.

5.1. Report of Result Details

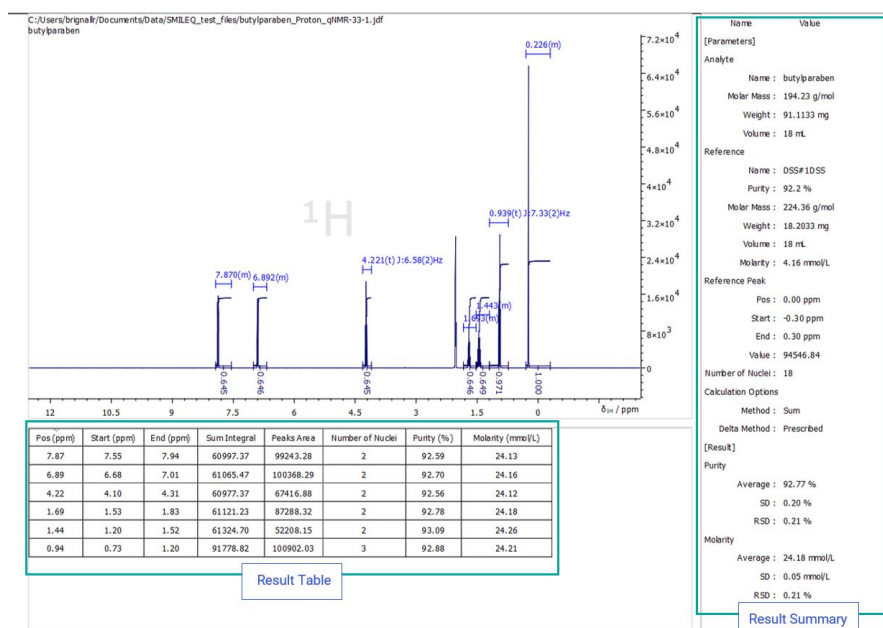


Figure 27: Example of the result report

Results Table: The purity or molarity of each of the region is reported in this table.

- ☞ If the regions of interest used in the quantification are defined using multiplet analysis in JASON, then the Peak Area estimate will be available. However, if the regions are defined as integrals only then it is not possible to use the peak area calculation.
- ☞ The result table can be changed to show or hide each parameter and display the color of the table by using the table tools panel that appears when the table is selected.
- ☞ The results table will only show the results for the regions defined in the multiplet details table that have the "calc" check box selected.

Results Summary: A summary of the results for the analyte is reported here.

5.2. Explanation of Parameters

Result Table	
Name of Parameters	Description
Pos	Signal position (ppm)
Start	Start position of multiplet or integral region (ppm)
End	End position of multiplet or integral region (ppm)
Sum integral	Integral value by direction summation
Peak Area	When using multiplet, the value of area from peak deconvolution. In the case when the option is "sum", this parameter is shown as "integral".
Number of Nuclei	Number of Nuclei, e.g. protons, responsible for the signal
Purity	Purity value (%)
Molarity	Molarity value (mmol/L)

Result Summary	
Name of Parameters	Description
Analyte	Target Analyte
Name	Analyte name or anything is able to write
Molar mass	Molar mass (g/mol)
Mass	Mass (mg)
Volume	Prepared sample solution volume (mL) / this parameter will be calculated molarity.
Reference	
Name	Reference name that is prepared in Reference Editor
Purity	Possessed purity value of reference material (%)
Molar mass	Molar mass (g/mol)
Mass	Mass (mg)
Volume	Prepared sample solution volume (mL) / this parameter will be calculated molarity.
Molarity	Molarity (mmol/L)
Reference Peak	
Pos	Signal position (ppm)
Start	Start position of multiplet or integral region (ppm)
End	End position of multiplet or integral region (ppm)
Sum integral	Integral value by direct summation
Number of Nuclei	Number of Nuclei, e.g. protons, responsible for the signal
Method	Calculation option whether using Sum integral or Peak area from deconvolution
Delta Method	When qNMR seamless, the parameters are shown. qNMR analysis method, "Prescribed "means using template file for analysis

[Result]	
Name of Parameters	Description
Purity	
Average	Average value of purity (%)
SD	Standard deviation of purity value for all signals

RSD	Relative standard deviation of purity value for all signals
Molarity	
Average	Average value of molarity (mmol/L)
SD	Standard deviation of purity value for all signals
RSD	Relative standard deviation of purity value for all signals