

# QUALITY BY DESIGN SOFTWARE SYSTEM

# GC METHOD DEVELOPMENT GUIDE – Empower CDS



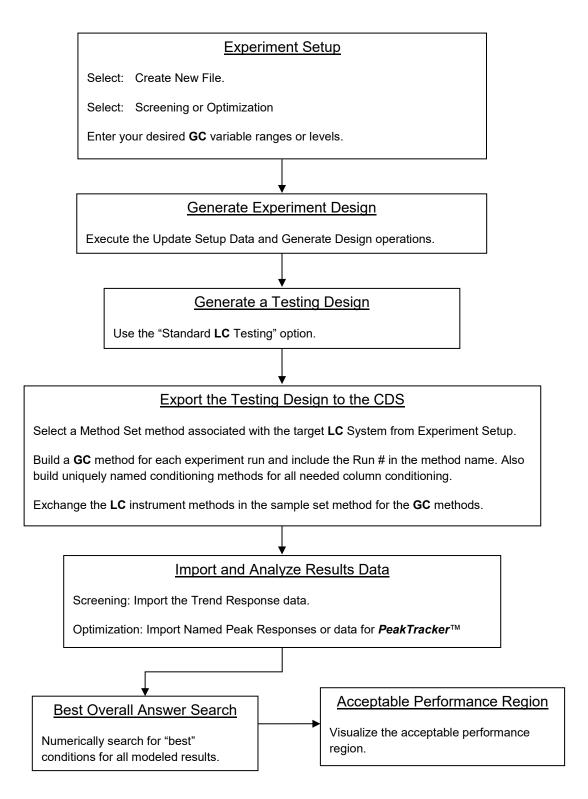
S-Matrix Corporation

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### **Overview of the Workflow**

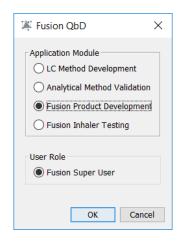
GC Method Development in Fusion QbD is done using the Fusion Product Development Module (FPD). Below is a general workflow.



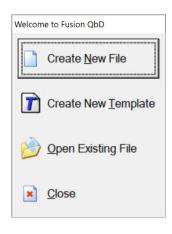
### **Create the GC Method Development Experiment Design**

The Fusion Product Development module is used for creating GC Method Development experiments. In this example, we will describe the process for creating a simple GC experiment using the initial hold time and the temperature programming rate.

1. Launch Fusion QbD. Select the 'Fusion Product Development' module.



2. Select the 'Create New File' option.



3. Set the 'No. of Mixture Variables' to zero (0).

Set the 'No. of Process Variables' to your desired number (2 in this example). Enter your variable settings

Initial Hold Time Continuous LB = 0.0 UB = 5.0
Temperature Ramp Rate Continuous LB = 10.0 UB = 30.0



Note – you can add parameters and set them as 'State=Constant' if you want to document the constant settings used in the experiment.

#### 4. Generate the Design.

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Edit Activity Tools Window He	lp		
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n of Experiments	Name: Administrator Company: S.Matrix Project: Project 1 (Us Date: 17 FEB 2020 09: Experiment Design	er Defined) 77:40 PST [UTC-08:00]	S-Matrix.
	Experiment Design Ma		
	Run No.	Initial Hold Time (Minutes)	Temperature Ramp Rate (Deg/Min)
	1	5.0	10.0
I			
	2	2.5	20.0
	3	2.5	20.0
	2 3 4		
	2 3 4 5	2.5	20.0
	4	2.5	20.0 30.0

# **Create the Companion Testing Design**

1. In the Data Entry View, click the '+ Create Testing Design' button on the Menu Bar.

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<u>File Edit Activity Tools Window H</u>	elp							
🗅 🖻 📂 🔛 🕲 👢 🍏 📰 🏏	Edit R	un No. Lab	els 🕡 Matrix Master 📑	Create/Edit Response Data	Show/Hide Responses	🕈 Create Testing Design	🚅 Export 📳 Sort Grid	0
Design of Experiments			1			L		
<ul> <li>Create a Design</li> </ul>		Run No.	Initial Hold Time	Temperature Ramp Rate				
<ul> <li>Design Reports</li> </ul>	1	1	5	10	1			
Data Entry / Analysis	2	2	2.5	20				
Data Entry	3	3	2.5	20				
<ul> <li>Data Analysis</li> </ul>	4	4	5	30				
BestAnswer Searches	5	5	0	30				
<ul> <li>Best Overall Answer</li> </ul>	6	6	2.5	20				
-      Acceptable Performance Region	7	7	0	10				
Point Predictions								
Visualization Graphics								

2. Select Standard LC from the 'Testing Design Type' list box.

Use the settings shown in the image below.

Kreate Testing Design				×
Testing Design Name Testing Design	(1)	Testing Design Type Standard LC	~	
		Descriptive Statistics Time Series Standard LC Inhaler Testing		
	Replication Scheme No. of Preparal No. of Test Repeats per			
The settings are valid.				
		Back	Finish	Cancel

3. Save the file with a unique and recognizable file name.

🐺 Fusion Product Development - GC E	xport.smae					-		×
Eile Edit Activity Tools Window H	lelp							
🗅 🖻 🖻 🗟 📕 🍏 🎫 +	Create Testing Design 😐 Delete Testing Design	👫 Create Response	Alt Response Reductions	💰 Export 🚡 Import Responses	0			
besign of Experiments	Response Name	Response Units			Testing Design Type Standard LC		fiew Testing	g Desigr
Reporting Toolkit								

# **Export the Testing Design to the CDS**

1. Access the Data Entry View and select the 'Testing Design (1)' tab.

Click the 'Export' button on the main menu bar.

Select on the 'Export to CDS' option on the 'Export Options' dialog.

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File Edit Activity Tools Window H	lelp					-	
🗅 🖻 🔗 📄 🕲 🖡 🏉 🔳 🔸	Create Testing Desig	n 😐 Delete Testing Design	👫 Create Response	It Response Reductions	🚅 Export	a Import Responses	0
Design of Experiments • Create a Design • Design Reports Data Entry / Analysis	Response Name		Response Units	Lower Limit	<= Respon	Upper Limit	Testing Desi Standard LC
Collin Adays     C	A 1 1 2 2 3 3 4 4 5 5 6 6 7 7 7						• 
	4						•
	Experiment Design	Testing Design (1)					
Ready							<b>d</b>
		Coptions Select Export Type C Export To Chromat C Export To File	ography Data System	Cancel			

2. Click 'Next' on the 1<sup>st</sup> Export wizard dialog pictured below.

Export		
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▼ : 1 ▼ to ▼ : 7 ▼ Export the	testing design by Column 💌	Update
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3 3		
4 4 5 5		
6 6 7 7		
7 7		
		-
		•
	<< Back Next >> Einish	Cancel
	Tuex >>	Cancel

3. Select a valid LC instrument system in the 2<sup>nd</sup> Export wizard dialog pictured below, and click Next.

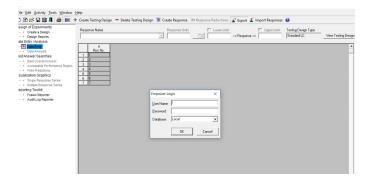
	Instrument Data System	^
Instrument Name	Waters Empower	
Aliance 1	Instrument Name     Alliance 1	
	Alliance 1	
Autosampler Configuration	Waters 2690/2695 Alliance	
Internal Autosampler	Waters 2998 PDA Detector	
Tray Name	External Switching Valves (contact closure)	
5-Carousel Tray - 120 2-mL vials		
First Vial Last Vial		
1 120 •	Pump:Quaternary Pump	~
Include Blank Injection(s) In each exported sequent     Include Suitability Injection(s) 5 - In each export		
Maximum number of injections per vial 50 -	a	

Note – Fusion QbD export automation only works directly with LC instrument systems and methods. Therefore, you must select a licensed LC instrument system. You will match this with the Method Set method you select in Step 6 below, which must be associated with the same LC instrument method and system.

 Select the 'None Selected' option in the Standards Strategy list box within the 3<sup>rd</sup> Export wizard dialog pictured below, and click 'Finish'.

Bracketing				
Strategy None Selected>		Cļear	<u>R</u> eset	

5. Log on to Empower using your Empower logon credentials.



6. In Empower, select the Project and Method Set method which will be used to build the Sample Set method, and click 'Next'.

Find     Filter     Res       Projects     Filter     Res       Projects     Constructions     Solution       Solution     Solution     Solution	set	Show Methods created by Fusion QbD  Method Set  Acquity 1 Base Method  Acquity 1 Class QD a 1 Base  Aliance 1  Information  Enable Shutdown Method  Acquity H Class QD a 1 Base  Aliance 1
er Types (logged in as 'Owner') Owner dy RTANT: Be sure the selected template method has the correct to to the on-line Help for detailed information on these requirement		nd properties required for the experiment design you are exporting.

7. Enter a recognizable name which will be used to name the Sample Set method, Instrument methods, and Method Set methods built as part of the export operation, then click 'Finish'.

Enter the Sample Set Name	×
Sample Set Name GC Testing Experiment 1	
Set Equilibration Time minutes	
Run Time 20 minutes	
Injection Volume 1 uL	
	<< Back

#### **IMPORTANT**

**Run Time** and **Injection Volume** – the level settings you enter here will be automatically written to the Sample Set method. You should review and edit these settings as needed in Empower for your experiment run conditions.

### **Construct the Required GC Methods within the CDS**

- 1. Log on to Empower using your Empower logon credentials.
- 2. Use the 'Browse Projects' option to navigate to the Empower project in which you exported your experiment design.
- 3. Open an existing GC instrument method which correctly operates the GC on which you will run your experiment design.
- Edit the method to contain the correct level settings for all GC parameters you did NOT include in your design in terms of your current sample and this experiment.
- 5. Execute the 'File | Save As...' operation to save the file with a recognizable name. In this example we will use the name "Fusion\_GC\_Base\_Method".
- 6. With Empower open, launch Fusion QbD, open your GC experiment design file, navigate to the 'Design Reports' View, and select the 'Experiment Design' report from the 'Design Reports list box, as pictured below.

Fusion Product Development - GC Study 1.sm	nae			
ile <u>E</u> dit <u>A</u> ctivity <u>T</u> ools <u>W</u> indow <u>H</u> elp				
🗅 📝 😂 🔛 🎒 👢 🎒 🥅 😾 Edit Ru	un No. Labels 💰 Export 🖂 Imp	ort Images 🕜		
- Create a Design	n Reports iment Design			
Acceptable Performance Region     Point Predictions Isualization Graphics     Single Response Series     Mutiple Response Series teporting Toolkit     Fusion Reporter     Audé Log Reporter	Name: Administrator Company: S-Matrix Project: Project 1 (User Defin Date: 17 FEB 2020 09:07:40 P Experiment Design Experiment Design Matrix		S-Matrix.	
		Initial Hold Time	Temperature Ramp Rate	
	Run No.	(Minutes)	(Deg/Min)	
	Run No.			
	Run No. 1 2	(Minutes)	(Deg/Min)	
	Run No. 1 2 3	(Minutes) 5.0	(Deg/Min) 10.0	
	Run No.           1           2           3           4	(Minutes) 5.0 2.5	(Deg/Min) 10.0 20.0	
	Run No.           1           2           3           4           5	(Minutes) 5.0 2.5 2.5	(Deg/Min) 10.0 20.0 20.0	
	1 2 3 4	(Minutes) 5.0 2.5 2.5 5.0	(Deg/Min) 100 200 200 300 300	

 Open the GC instrument method from Step 5 above. Referring to the Fusion QbD experiment design, edit the GC method to reflect the level settings of the variables in Run No. 1. 8. Execute the 'File | Save with Method Set...' operation to save the GC Instrument method, and its associated Method Set method, with a recognizable name and a Run 1 extension. In this example we will use the name "Fusion\_GC\_Study\_001".

				Inbox - Josep								
n	-	GC Method 001 in SMD2019 as System/Administrator - Instrument Method Editor										
F.	File	Edit View Help										
_2		New	Ctrl+N									
2	1	Open	Ctrl+O	2								
f E rea		Save	Ctrl+S	)6								
ea		Save As										
esi V		Save with Method Set		ature Solvents Channel								
ita	_	Exit										

Repeat Steps 7 and 8 above to generate the Instrument Methods and associated
 Method Set methods required for all the experiment design runs. It is best to do this in
 the sequential order of the experiment design – for example:

Fusion\_GC\_Study\_001 Fusion\_GC\_Study\_002 ... Fusion\_GC\_Study\_007

Note – doing this in sequential order minimizes mistakes, and supports ease of association of the methods into the associated Sample Set method constructed at the time of export from Fusion QbD.

 Select the Sample Set method which Fusion QbD constructed within your target Empower project, and open it for editing.

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<u>F</u> ile	Edit View Tools Database						
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Filter E	By: Default	• E	dit Vie <u>w</u> Update Max Row	vs: 1000			
• •	Sample Sets Injections Char	nels Methods Res	ult Sets Results Peaks Fra	actions	Offs Curve	s View Filters Custom Fields Audit Trails	
Ē	Method Name	Method Type	Method Date	Method id	Old Id		
1	GC Testing Experiment 1_1	Sample Set	2/16/2020 7:54:19 PM PST	1040			
2	Acquity H Class QDa 1 Base	Method Set	8/15/2019 3:31:00 PM EDT	1019			
3	Alliance 1	Method Set	8/15/2019 3:36:46 PM EDT	1031			
4	Acquity 1 Base Method	Method Set	8/15/2019 3:31:00 PM EDT	1022			
5	Acquity H Class QDa 1 Base	Instrument	8/15/2019 3:31:00 PM EDT	1000			
6	Alliance 1	Instrument	8/15/2019 3:36:31 PM EDT	1030			
-	Acquiby 1 Base Method	lostrument	8/15/2010 3:31:00 PM EDT	1004			

 For each experiment run (row) in the Sample Set method, use the Instrument method list boxes within the 'Method Set / Report or Export Method' column to exchange the LC instrument method for the GC method you constructed in the previous section.

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5	Vial	lnj Vol (ul.)	# of Injs	Label	SampleName	Level	Function	Method Set / Report or Export Method	Label Reference	Processing	Run Time (Minutes)	Data Start (Minutes)	Next Inj. Delay (Minutes)	MS Tune Method	MS Calibration Method	Column Position	A Add
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2	2	10.0	1	Unk-001-002	2		Inject Samples	Alliance 1 Base Method		Normal	20.00	0.00	0.00				
3	3	10.0	1	Unk-001-003	3		Inject Samples	Alliance 1 Base Method		Normal	20.00	0.00	0.00				
4	4	10.0	1	Unk-001-004	4		Inject Samples	Aliance 1 Base Method		Normal	20.00	0.00	0.00				
5	6	10.0	1	Unk-001-005	6		Inject Samples	Aliance 1 Base Method		Normal	20.00	0.00	0.00				Г
6	6	10.0	1	Unk-002-001	6		Inject Samples	Alliance 1 Base Method		Normal	20.00	0.00	0.00				Г
7	7	10.0	1	Unk-002-002	7		Inject Samples	Alliance 1 Base Method		Normal	20.00	0.00	0.00				
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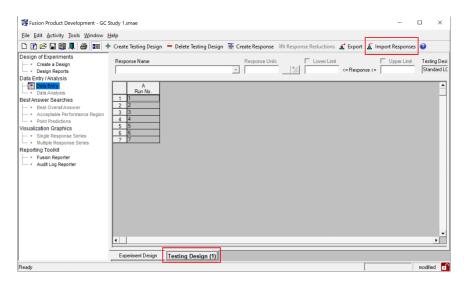
3. Confirm that the run times and injection volumes match the experiment method requirements, and your method times, and save the edited Sample Set method when the exchanges and edits are complete.

#### **IMPORTANT**

You should carry out a column conditioning run to bake your column prior to starting your experiment run. You will also need to manually insert rows column conditioning with appropriately constructed conditioning methods as required to support changes between experiment runs.

## Run your Experiment within the CDS and Import Your Results

- 1. Prepare your GC system. Load and execute the Sample Set in Empower.
- After running the experiment on the GC, process the completed Sample Set into a Results Set using an appropriate processing method. Review the processed results to correct any integration issues.
- 3. Launch Fusion QbD and access the Fusion Product Development module.
- 4. Open your experiment file, and access the Data Entry View.
- 5. Select the 'Testing Design (1)' tab, and import your experiment chromatogram results for analysis and visualization.



Note – refer to the Empower Data Exchange module under the Help menu in Fusion QbD for tips with integration and the Fusion QbD import operation.