

# **Quick Reference Guide**

# OPTIC 3 - PTV on Shimadzu GC-2010 Gas Chromatograph



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This Quick Reference Guide is an addendum to the OPTIC 3-PTV Installation Guide onto Shimadzu GC-2010 OEM (Doc. ID SM-042). Please, read both documents attentively before installing the system.

## This guide is made to understand:

- 1. How to operate **GC/MS Solutions** and **Evolution Workstation** software with OPTIC 3-PTV Injection system installed on Shimadzu GC/MS QP2010.
- 2. How to use GC-2010 **AFC unit** for carrier gas control with OPTIC 3-PTV injector.
- 3. How to configure '**dummy injector**' on GC/MS QP2010 system in case no Shimadzu injector is installed.
- 4. How to create independent method using **Evolution Workstation software** in order to make temperature program and set the control parameters for venting process if using Large Volume Injection mode.
- 5. In case if OPTIC 3-PTV is combined with LINEX and CDC options, these options must be controlled by CTC Analytics **CycleComposer** software running outside GC/MS Solution.

# 1. Installation of OPTIC 3-PTV

### 1.1 GC/MS QP2010 system

If OPTIC 3-PTV is the only injection port installed on the system, a '**dummy**' SPL injector should be installed using a 'dummy' connector for GC-2010 Injector Heater port. This 'dummy' connector is a part of the Shimadzu 223-57164-91 AFC addition kit for GC- 2010. The 'dummy' SPL Injector should be configured as it is shown in the example screens of GC-2010 in Fig. 1.



Figure 1. Configuration 'dummy' SPL injector.

### **GC/MS Solutions Configuration**

### 1.1.1 Instrument / System Configuration

This configuration is similar to standard SPL injector, since SPL is used as dummy injector for OPTIC 3-PTV (see Fig. 2).

System Configuration			×
<u>A</u> vailable Modules	1	Modules <u>U</u> sed for Analysis	
Instrument GC-2010 Handbook Handbook Column Handbook Detectors Others	Properties	Instrument1 GC-2010 GC-2010 GC-2010 GC-2010 SPL1 Column MS MS Additional Heater	
Aug	įtTrail <u>S</u> et	Cancel <u>Print</u> <u>H</u> elp	

Figure 2. Instrument configuration

### 1.1.2 Instrument Parameters

Set the parameters as it is shown in Fig. 3.

🏭 GCM5 Real	Tim	e Analysis (Admin) <mark>- [Acc</mark>	quisition - Me	thod_test02.	.qgm, 30_60 me	sh glass beads pest	ticides grob <mark>19.qgd(L</mark>	ine1), 20080
🔬 Eile Edit	⊻iew	Method Instrument Ac	quisition <u>D</u> ata	a <u>T</u> ools <u>W</u> ind	low <u>H</u> elp			
	8		<b></b> €♦ 💻	? 🐰			J.	
Acquisition		🐻 GC 🚘 MS						
1		Inj. Port : SPL1	Inj. I	Heat Port :	INJ1			
Тор		Column Oven Temp. :	50.0	°C kPa	+			1
		Injection Temp. :	30.0	_ °C	+			
		Injection Mode	Splitless 💌		. I			
Wizard		Sampling Tim	1.00	min	0.0	5.0	10.0 mi	т in
		Carrier Gas : He Prim.	Piess 300-3	00	Program :	Pressure	•	
Sample Login		Flow Control Mode :	Pressure	-		, I. 5. 15		
		Pressure :	100.0	kPa	Hate	100.0	0.00	-
Ö		Total Flow :	7.5	mL/min	1 0.0	0.0	0.00	
Standby		Column <u>F</u> low :	4.55	mL/min	3 0.0	0.0	0.00	<b>T</b>
		Linear Velocity :	77.4	cm/sec	Total Program	Time: 0.0	0 min	_
Start		Purge Flow :	3.0	mL/min	Column			
		Split <u>R</u> atio :	-1.0		Name InertCa	ap 5MS/Sil Thickne	ss: 0.25 um 0.22	1
			. I		Length: 30.0	m Diamete	r: 0.32 mm	
Stop		Detail of Injection	n Port		Rea <u>d</u> y Check			
<b></b>		High Press. Injection	Carrier G	ias Save				
Method Detail		Splitter Hold	Fan		GC Program	L		
		Split Ratio Program			Prerun Program	n Tir	ne Program	
1. Line								

Figure 3. Instrument Parameters Window.

In order to avoid the temperature "ready check" for dummy injector (SPL1), SPL1 check box in the Ready Check dialog window should be unchecked (Fig. 4).

Ready Check		×
Heat Unit :	Injection Flow :	ОК
✓Column	SPL1 Carrier	
SPL1	SPL1 Purge	Cancel
<b>1</b> 110		Help
	APC Flow :	
Detector(FTD):		
	J	
	Detector APC Flow :	
, Baseline Drift :		
🔲 External Wait		
Equilibrium Time :	0 min	



# 2. Setting up OPTIC 3-PTV methods

### 2.1. Large Volume Injection Method

Set the parameters as it is shown in Fig. 5.

🐫 GCMS Real T	ime Analysis (Admin) - [Acquisition - Method_test0;	2.qgm, 30_60 mesh glass beads pesticides grob19.qgd(Line1),
<u>بالله E</u> ile <u>E</u> dit <u>V</u>	jew <u>M</u> ethod Instrument <u>A</u> cquisition <u>D</u> ata Iools <u>W</u> ir	ndow <u>H</u> elp
	5 L 💵 🎫 🖬 🖉 🖉	₩₩₩₩₩₩₩₩₩₩₩₩₩₩₩₩₩₩₩₩₩₩₩₩₩₩₩₩₩₩₩₩₩₩₩₩₩₩₩
<u> </u>		
Acquisition	🔯 GC 🗃 MS	
	Inj. Port : SPL1 Inj. Heat Port :	INJ1
Тор	Column <u>O</u> ven Temp. : 50.0 °C •C	250
<u> </u>	Injection Temp. 30.0	230
	Injection M de : Split 💌	
Wizard	Sempling Time 1.00 min	0.0 5.0 10.0 min
	Carrier Gas : He Prim. Press. : 500-900	Program : Column Oven Temperature
Sample Login	Flow Control Mode : Linear Velocity	
	Pressure : 14.3 kPa	Rate Final Temperature Hold Time
$\bigcirc$	Total Flow : 154.5 mL/min	1 25.00 325.0 2.00
Standhu	Cohene Floure 150 and Jain	2 0.00 0.0 0.00
o tanaby		3 0.00 0.0 0.00
	Linear Velocity : 44.4 cm/sec	Total Program Time : 15.00 min
Start	Purge Flow : 3.0 mL/min	Column
otait	Split <u>R</u> atio : 100.0	Name InertCap 5MS/Sil Thickness: 0.25 um
$\odot$		Length: 30.0 m Diameter: 0.32 mm 5gt
Stop	Detail of Injection Port	Rea <u>d</u> y Check
	High Press. Injection Carrier Gas Saver	
Method Detail	Splitter Hold Fan	GC Program
	Split Ratio Program	Time Program

Figure 5. Setting up LVI Method

Create a GC time program as it is shown in Fig. 6.



Figure 6. GC Temperature Program

Large Volume Injection requires eliminating solvent before injector is heated up. This process should programmed as a pre-running process (see example of the OPTIC 3 LVI method programmed within Evolution Workstation in Fig. 7). However GC-2010 does not allow this kind of pre-run programs.



Figure 7. Example of LVI method within Evolution Workstation

The LVI method for OPTIC 3-PTV should be made as a '**Split method**' within GC Solutions because the split valve must be opened during the solvent eliminating. During the method run, after solvent is vented, OPTIC -3 sends start signal to GC-2010, the split valve is closed and GC program is started.

NOTE: If this method is cancelled during the GC run, the split valve cannot be initialized. In this case, the split valve should be opened manually from the GC keypad.

### 2.2. LINEX Method

LINEX method is very complex because the injector pressure must be released before opening the LINEX head. This is needed in order to avoid jumping up the liner out of injector. Also solid material powder-like packing material or sample introduced into liner for the direct thermal desorption can pusher out of the liner if pressure is not released. Fig. 8 below shows an exampled of the LINEX method.

	no Apolycic (Admin) [Acquisition Method tost01	ann confirmation and/Linc1) 20090510 ant
	w Method Instrument Acquisition Data Tools Wind	aw Help
	<u> 19 28 19 18 18 18 28 28 28 28 28 28 28 28 28 28 28 28 28</u>	
r**** 💼		Det. 1.40 KV Set
Acquisition		
	Inj. Port : SPL1 Inj. Heat Port :	INJT
Тор	Column Oven Temp. : 50.0 °C kPa	
20.0	Injection Te <u>m</u> p. : 30.0 °C	
	Injection Mode : Split	0
Wizard	Sampling Time : 1.00 min	0.0 2.5 5.0 7.5 10.0 min
	Carrier Gas : He Prim. Press. : 500-900	Brazer
4	Flow Control Mode : Pressure	Program. Pressule
Sample Login	Pressure : 12.0 kPa	Rate Final Pressure Hold Time
$\bigcirc$	Total Flow : 1146.1 ml /min	
Standby		2 10.0 250.0 0.00
		3 0.0 0.0 0.00
		Total Program Time : 12.46 min
Start	Purge Flow : 0.0 mL/min	
	Split <u>B</u> atio : 544.3	Length: 60.0 m Diameter: 0.25 mm Set
	Detail of Inication Part	
Stop		Rea <u>dy</u> Check
	High Press. Injection Carrier Gas Saver	
Mathead Datail	Splitter Hold Fan	GC Program
Method Detail	Solit Batio Program	Prerun Program Time Program

Figure 8. Example of LINEX method within GC Solutions.

For the reasons mentioned above, Injection Mode should be set to **Split**, Flow Control Mode should be set to **Pressure** and the initial pressure should be set to a **minimum possible value** (12 kPa, for instance). At the same time, Total Flow should be set to a high value (more then 100 ml/min) in order to flush the interior of the injector after the LINEX head is closed. The "flush-time" should be set through the Evolution Workstation using LINEX-TD or LINEX-DMI method. Initial **Septum Purge Flow** should be set to **ZERO** because GC-2010 gives an error status if the LINEX head is opened for long time. Since it is very difficult to control ZERO Septum Purge Flow, its control parameters should be optimized (see Section 3).

When LINEX head is closed and the GC method is started, the injector pressure should be built up to required pressure immediately. Purge Flow should also be built up to a value of 3 ml/min or so. For this, Pressure program and Purge Flow program created be created according to the required analytical parameters (see example in Fig. 9).

W covo p l		Malle Landor	- C 1/1 ! 1 000	
GLM5 Real	line Analysis (Admin) - [Acquisition	- Method_testU1.qgm, ca	nrirmation.qgd(Line1), 200	sosia'dät]
			P Pladie (plate)	
		🗶 🗶 📲 🛲 📶		
(100) T		De	t. 1.40 kV Set	
	1			
Acquisition				
	Inj. Port : SPL1	Inj. Heat Port : INJ1		
Top	Column Oven Temp. : 50.0	°C mL/min		
	Injection Temp. : 30.0			
	Injection Mode : Split	<b>v</b> 0.0		
Wizard	Sampling Time : 1.00	0.0 F	0 5.0	10.0 .
all be	Carrier Gas : He Prim. Press. : 5	00-900		min
42	Elow Control Mode - Pressu	re V	ram : Purge Flow	
Sample Login	Pressure : 120		Rate Final Flow	Hold Time 🔺
			· 0.0	0.00
	Lotal Flow : 146.1	mL/min 1	300.00 3.0	0.00
Standby	Column Elow : 0.27	mL/min 3	0.00 0.0	0.00
	Linear Velocity : 13.3	cm/sec Tota	al Program Time : 15.0"	1 min
Start	Purge Flow : 0.0	mL/min Colu	mn	
Stdit	Split <u>R</u> atio : 544.3	Nam	e test Thicknes	ss: 0.25 um
$\odot$		Leng	yth: 60.0 m Diameter	: 0.25 mm
Stop	Detail of Injection Port	Rea	ady Check	
	High Press, Injection Car	rier Gas Saver		
Method Detail	Splitter Hold Far		C Program	
10	Split Ratio Program	Pren	un Program Tin	ne Program

Figure 9. Example of a GC method

In case of Splitless method, the split valve must be closed immediately by GC program started at the same moment as the Large Volume Injection method. Example of the program is shown in Fig. 10.



Figure 10. Example of GC Time Program

NOTE: If this method is cancelled during the GC run, the split valve cannot be initialized. In this case, the split valve should be opened manually from the GC keypad.

### 2.3. Settings for OPTIC 3 LINEX-TD Method

Example of the LINEX-TD method is shown in Fig. 11.

¥4	TAS	Evolu	ution \	<b>Vorks</b> tat	ion - [(	OPTIC N	Method	l: Met	hod5 [L	INEX-TO	)]]
	<u>F</u> ile	<u>E</u> dit	⊻iew	<u>M</u> ethod	<u>S</u> eque	nce <u>C</u> i	onfigura	tion	<u>W</u> indow	Help	
] [	2	Ē		6	Ē	Ľ	En	E,	E		ę
Ξ	Ger	neral									
	Meth	hod N-	ame		Metho	od5					L 1
	Equi	libratio	on Time	e (sec)	5						L 1
	End	Time	(sec)		600						L 1
	TD 9	Splitles	88		No						L 1
😑 Injector Temperature						L 1					
	Initia	l Tem	peratur	e (°C)	35						L 1
	Ramp Rate (*C/sec)		5.0						L 1		
	Final Temperature (°C)		200						L 1		
	Hold Time (sec)		480						L 1		
	Temperature Control		Кеер	Until En	ıd				L 1		
Ξ	Sol	vent '	Ventin	g							L 1
	Sam	ple Sv			60						L 1
											L 1
											L 1

Figure 11. Example of LINEX-TD Method

Please, note that Sample Sweep Time is the time needed to flush the injector after the LINEX head is closed. When Sample Sweep Time is elapsed, injector and GC programs are started.

### 2.4. Settings for OPTIC 3 LINEX-DMI Method

In generally, DMI method is used for liquid injections and requires solvent elimination (similar to Large Volume Injection method). The solvent elimination time should include the injector flush time (see description for LINEX-TD method above). An example of the LINEX-DMI method is shown in Fig. 12.

، 🖌	ATAS Evolution Workstati	on - [OPTIC Method: Method6 [			
	<u>File E</u> dit <u>V</u> iew <u>M</u> ethod	Sequence Configuration Window			
[	) 🖻 🔒 🖨				
$\square$	General				
	Method Name	Method6			
	Equilibration Time (sec)	5			
	End Time (sec)	600			
	DMI Splitless	No			
$\square$	Injector Temperature				
	Initial Temperature (°C)	35			
	Ramp Rate (*C/sec)	5.0			
	Final Temperature (*C)	200			
	Hold Time (sec)	480			
	Temperature Control	Keep Until End			
$\square$	Solvent Venting				
	Vent Mode	Threshold	Ŀ	Solvent Venting	
	Vent Time (sec)	30		Vent Mode Fixed T	ime
	Solvent Monitor Level (%)	10		Vent Time (sec) 60	
	Solvent Monitor Threshold	25		Solvent Monitor Level (%)	
	$\backslash$			Solvent Monitor Threshold	
			-		
	<u> </u>	Using so	olve	nt monitor	Using Fixed Vent Tir

Figure 12. Example of LINEX-DMI Method

## 3. Optimizing control parameters for purge flow

The optimization settings mentioned here only for the reference purposes. Please, consult the service manual of the GC-2010 gas chromatograph or contact Shimadzu service department.

In case the septum purge flow should be set to zero, offset in the list the control parameters for Purge Pressure should be optimized (see Fig. 13).

SPIT	Flow	Press
21	1 1000	44011
P mode 1 term	A Contraction	
Purge Pi	eastme	
Actual	0.0	0.0
Valve Voltage	-	0.0
Offset	C	33535
Gain		5555
I time		10
P term		50
I term		100
Primary I	ressure	Э
Actual		502.0
Offset		32700
		Ą

Figure 13. Offset Optimization

If the value of the purge flow should be set to some value (3 ml/min, for example), gain in the list the control parameters for Purge Pressure should be optimized (see Fig. 14).



Figure 13. Gain Optimization

Even with above described optimization of the offset and gain, the error message can be delayed for a maximum of 2 minutes. For this reason, in order to avoid the error message, the time between opening and closing the LINEX head must be shorter then 2 minutes.

In some cases, 2 minutes is not long enough to perform all the required manipulation with the sample. The only solution is then upgrading the OPTIC 3-PTV to OPTIC 3-S version. This version contains a dedicated Electronic Gas Control designed to work with LINEX and CDC Station.

p/no <b>H400062</b>	Upgrade kit OPTIC 3-PTV to OPTIC 3-S
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## 4. Connecting LINEX Head to GC-2010 AFC

To install OPTIC 3-PTV with LINEX option, perform the following steps:

- 1. Cut the ends of the PEEK lines connected to the LINEX Head as it is shown in Fig. 15.
- Connect the carrier line (blue marker) to AFC carrier line using the 1/8" union and the 1/8" to1/16" reducing ferule.



3. Connect the second line to the AFC purge flow line using the 1/8" union and 1/8" to 1/16" reducing flow.

Please contact ATAS GL International BV if you have any questions. <u>info@atasgl.com</u> http:// www.atasgl.com