Thermo Scientific

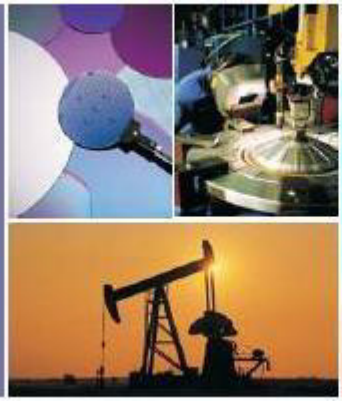
TriPlus

Automatic Sampling System

Standard Operating Procedures

PN 317 110 19, Revision April 2009

Home



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SECTION IV
TriPlus SPME

About This Manual

Overview

This manual is organized as follows:

Section **I**, *SOPs Overview*, contains a general description of the Standard Operating Procedures.

Section **II**, *TriPlus AS*, contains the procedure to test the TriPlus AS version combined with different injectors and detectors.

Section **III**, *TriPlus HS,* contains the procedure to test the TriPlus HS version combined with different injectors and detectors.

Section **IV**, *TriPlus SPME,* contains the procedure to test TriPlus SPME version combined with different injectors and detectors.

Appendix A, *Customer Communication*, contains contact information for Thermo Fisher Scientific offices worldwide. Use the *Reader Survey* in this section to give us feedback on this manual and help us improve the quality of our documentation.

The contains definitions of terms used in this guide and the help diskette. This also includes abbreviations, acronyms, metric prefixes, and symbols.

The *Index* contains an alphabetical list of key terms and topics in this guide, including cross references and the corresponding page numbers.

Conventions Used in This Manual

The following table lists symbols and typographical conventions. Only a few of them are used in this manual.

Bold	Bold text indicates names of windows, menus, dialog boxes, buttons, and fields.
Italic	Italic indicates cross references, first references to important terms defined in the glossary, and special emphasis.
`Monospace`	Monospace, or Courier, indicates filenames and filepaths, or to indicate text the user should enter with the keyboard.
`Monospace Bold`	Monospace Bold indicates messages or prompts displayed on the computer screen or on a digital display.
»	This symbol illustrates menu paths to select, such as **File»Open…**.
KEY NAME	Bold, uppercase sans serif font indicates the name of a key on a keyboard or keypad, such as **ENTER**.
 **CAUTION**	This symbol alerts you to an action or procedure that, if performed improperly, could damage the instrument.
 **NOTE**	This symbol alerts you to important information related to the text in the previous paragraph.
 **WARNING!**	This symbol alerts you to an action or procedure that, if performed improperly, could result in damage to the instrument or possible physical harm to the user. This symbol may be followed by icons indicating special precautions that should be taken to avoid injury.
	This symbol indicates electric shock hazard.

This symbol indicates danger from hazardous chemicals.

This symbol indicates danger from high temperature surfaces or substances.

This symbol indicates a fire hazard.

This symbol indicates an explosion hazard.

This symbol indicates a toxic hazard.

This symbol indicates the presence of flammable materials.

This symbol indicates the presence of radioactive material.

This symbol indicates an operation or procedure that must NOT be performed by the user. A Thermo Fisher Scientific authorized Customer Support Engineer must perform this procedure.

This symbol indicates all metal objects, such as watches, jewels, etc., must be taken off.

This symbol indicates an eye hazard. Eye protection must be worn.

This symbol indicates the user must wear a protective screen when performing the procedure.

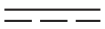
This symbol indicates the user must wear protective shoes when performing the procedure.

This symbol indicates the user must wear protective clothing when performing the procedure.

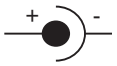
This symbol indicates the user must wear gloves when performing the procedure.

Instrument Markings and Symbols

The following table explains the symbols used on Thermo Fisher Scientific instruments. Only a few of them are used on the TriPlus sampler. See the asterisk.

	Symbol	Description
		Direct Current
*		Alternating Current
		Both direct and alternating current
		Three-phase alternating current
		Earth (ground) terminal
		Protective conductor terminal
		Frame or chassis terminal

	Symbol	Description
		Equipotentiality
*		On (Supply)
*		Off (Supply)
		Equipment protected throughout by DOUBLE INSULATION or REINFORCED INSULATION (Equivalent to Class II of IEC 536)
*		Instruction manual symbol affixed to product. Indicates that the user must refer to the manual for specific Warning or Caution information to avoid personal injury or damage to the product.
		Caution, risk of electric shock
*		Caution, hot surface
*		Caution, biohazard
		In-position of a bistable push control
		Out-position of a bistable push control

	Symbol	Description
*		Jack socket
*		Symbol in compliance to the Directive 2002/96/EC on Waste Electrical and Electronic Equipment (WEEE) placed on the european market after August, 13, 2005.

Abbreviations for Injectors and Detectors

Injector	Abbreviation
Split/splitless Injector	S/SL
Cold On-Column Injector	OCI
Programmable Temperature Vaporizing Injector	PTV
Large Volume Splitless	LVSL

Detector	Abbreviation
Flame Ionization Detector	FID
Electron Capture Detector	ECD
Nitrogen-Phosphorus Detector	NPD
Flame Photometric Detector	FPD
Thermal Conductivity Detector	TCD

SECTION I

SOPs Overview

This section contains a general description of the Standard Operating Procedures.

Chapter 1, *General Overview*, contains a guideline to apply correctly the SOPs.

1

General Overview

Chapter at a Glance...

Scope

The Standard Operating Procedures (SOP) described in this book are a series of instructions, operations and test criteria derived from our quality policy procedures used for final testing of the TriPlus sampler AS, HS and SPME version The SOPs have been developed to test and verify instrument complete analytical performance after the installation has been completed. This will help you as a guideline, to check if your TriPlus sampler continues to perform according to the original checkout testing specifications carried out in the factory premises. However, these tests alone cannot define if the instrument is not performing according to the original specifications.
The checkout is carried out injecting a standard solution into a test column under analytical conditions set according to the injector(s) and detector(s) hardware provided with the GC. Before starting the test checkout, refer to the Parts Referenced and the Analytical Condition required.

NOTE Each SOP has a proper Registration and Revision Number (e.g. TE P0578/01/E - 30 October 2006), according to our Quality Management policy.

For specific operating or maintenance questions, please refer to the following manuals:

- TriPlus Operating Manual PN 317 094 41
- TRACE GC Ultra Standard Operating Procedures PN 317 092 00
- FOCUS GC Instruction Manual PN 317 094 12

Parts Referenced

The SOPs require the following parts:

Table 1-1. SOPs Parts Referenced

Part	Description	Part Number
Test Column	Fused Silica Capillary Column TR-5; 7 m long; 0.32 mm ID; 0.25 µm film thickness.	260 800 01
Graphite Ferrule	Graphite ferrule for 0.32 mm ID column	290 134 87
Syringes	10 µl size; 50 mm needle length 10 µl size; 80 mm needle length	365 005 25 365 020 19
Test Mixtures	Test Mixture for FID checkout Test Mixture for ECD checkout Test Mixture for NPD checkout Test Mixture for FPD checkout Test Mixture for TCD checkout	338 190 20 338 190 11 338 190 06 338 190 06 338 190 16
Gases	Gas Chromatographic-grade purity *Carrier Gas* = Helium *Fuel Gases* = Hydrogen - Air *Make-up Gas* = Nitrogen *Reference Gas* = Helium	
Data Acquisition	Chrom-Card, ChromQuest, Xcalibur	

Getting Started

Before starting checkout, perform the following preliminary operations:

1. GC Installation
2. TriPlus Installation
3. SOP of the GC in use

SECTION II

TriPlus AS

This section, contains the procedures to test the TriPlus AS version combined with different injectors and detectors.

Chapter 2, *TriPlus AS Checkout Using S/SL Injector*

Chapter 3, *TriPlus AS Checkout Using OCI Injector*

Chapter 4, *TriPlus AS Checkout Using PTV Injector*

2

TriPlus AS Checkout Using S/SL Injector

Chapter at a Glance...

Operating Procedures

SOP Number: TE P0578/02/E - 02 May 2007

Scope

Use the following procedure to verify proper TriPlus **AS** Version operation combined with S/SL Injector and FID, ECD, NPD, FPD, TCD detectors.

Parts Referenced

Table 2-1. TriPlus AS - S/SL - Detectors Parts Referenced

Part	Description	Part Number
Test Column	Fused Silica Capillary Column TR-5; 7 m long; 0.32 mm ID; 0.25 µm film thickness.	260 800 01
Glass Liner	3 mm ID for splitless injection	453 200 32
Liner Seal	Graphite seal for glass liner	290 334 06
Graphite Ferrule	Graphite ferrule for 0.32 mm ID Column	290 134 87
Retaining Nut	M4 capillary column retaining nut	350 324 23
Septum	Standard septum for S/SL injector	313 032 11
Syringe	10 µL size; 50 mm needle length 10 µL size; 80 mm needle length	365 005 25 365 020 19
Vials, Seals and Caps	10 mL Crimp-top washing solvent vials 100 mL washing solvent bottle (requires P/N 386 060 74) 10 mL Crimp-top waste vial 2 mL sample vial (requires P/N 386 060 92)	190 046 47 240 140 85 190 505 50 240 140 21
Test Mixture for FID	Three components, Dodecane, Tetradecane, Hexadecane, in n-Hexane:	338 190 20
Test Mixture for TCD	Three components, Dodecane, Tetradecane, Hexadecane, in n-Hexane	338 190 16
Test Mixture for ECD	Two components Lindane, Aldrin, in Iso-octane	338 190 11
Test Mixture for NPD and FPD	Three components, Azobenzene, Octadecane, Parathion Methyl, in Iso-Octane	338 190 06
Interferential Filter	526 nm for phosphorus 394 nm for sulphur	281 071 00 281 070 00
Data Acquisition	Chrom-Card (10 V F.S.), ChromQuest, Xcalibur,	

Analytical Conditions

Set the parameters listed in the following Tables 2-2 and 2-3.

TriPlus AS

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Table 2-2. TriPlus AS - S/SL Analytical Conditions

Parameters Setting	
Analysis Type	Analysis Type = Single
Mode, Injector, Synch	Injection mode = Basic Injector port = Injector A (Split/Splitless) Start synch mode = Standard
Sampling Parameters	Sample volume = 1.0 µL Plunger strokes = 10 Air volume = 3.0 µL Filling volume = 3.5 µL
Vial Sample Depth	Vial Depth = Bottom
Injection Depth Mode	Pre-injection dwell time = 4.0 s Post-injection dwell time = 0.0 s Injection Depth = Max Injection Speed 100 µL/s
Viscosity	Sample pull-up speed = 1 µL/s Delay after bubble elimination = 4.0 s Viscosity delay = 1.0 s
Pre-injection Washes Parameters	Solvents wash sequence = A, - , - , - Solvent cycles = 1 Solvent Volume = 1.0 µL
Sample Washes Parameters	Rinses cycles = 1 Rinses volume = 1.5 µL
Post-injection Washes Parameters	Solvents wash sequence = A, - , - , - Solvent cycles = 1 Solvent Volume = 1.0 µL
Advanced Parameters	Wash solvent depth% = 100 Waste depth% = 15 Needle speed into vial = 10 mm/s Solvent filling speed = 20 µL/s Bubble elimination pulling speed = 10 µL/s Delay between strokes = 0.1 s

Gas Chromatograph

Table 2-3. Gas Chromatograph Analytical Conditions

Parameters	FID	ECD	NPD	FPD	TCD
Gases					
Carrier Gas: Helium = 30 kPa Constant Pressure					
Hydrogen (mL/min.)	35	---	2.3	90	---
Air (mL/min.)	350	---	60	115	---
Make-up Gas (mL/min.)	30	30	15	---	27.5
Reference Gas (mL/min.)					30
Oven Program					
Initial Temperature (°C)	50	70	70	70	50
Initial Time (min.)	1	1	1	1	1
Ramp 1 (°C/min.)	20	20	20	20	20
Final Temperature (°C)	200	220	230	230	190
Final Time (min.)	1	1	1	1	1
S/SL Injector					
Operating Mode = *Splitless* (SL)	SL	SL	SL	SL	SL
Temperature (°C)	230	230	230	230	230
Splitless Time (min.)	0.8	0.8	0.8	0.8	0.8
Split Flow (mL/min.)	60	60	60	60	60
Constant Septum Purge	Yes	Yes	Yes	Yes	Yes
Detector					
Base temperature (°C)	250	250	300	300	
Detector Temperature (°C)	---	300	---	150	
Detector Signal Range - See [***]	10^0	---	10^0	10^0	
Reference Current (nA)		1			
Pulse Amplitude (V)		50			
Pulse Width (µs)		1			
Source Current			[*]		
Polarizer Voltage (V)			3.5		

Table 2-3. Gas Chromatograph Analytical Conditions (Continued)

Parameters	FID	ECD	NPD	FPD	TCD
High Voltage Mode				No	
Block Temperature (°C)					200
Transfer Temperature (°C)					190
Constant Filament Temp. (°C)					No
Filament Voltage (V)					10
Filament Temperature Limit (°C) [**]					350
Gain					x 10
Negative Polarity					No

[*] Refer to the Source Ignition procedure reported in the SOP Manual
[**] In case of TCD with the polyimide coated filaments, set the filament temperature limit to 320 °C.
[***] In the case your GC is equipped with the previous non-fast FPD control card, labeled FPD, set Detector Signal Range to 10^{1}.

Data System

Table 2-4. Data System

Parameters Setting	
Digital Signal Output	Acquisition Frequency = 10 Hz

Recommended Initial Operations

Before starting perform the SOP related to the GC configuration used as described in the relevant SOP manual.

OPERATING PROCEDURE

TriPlus AS - S/SL - Detectors Checkout

1. Perform an off-line blank run of the column.
2. Prepare the sample vial introducing 1 mL of test mixture into the empty vial and closing it immediately with the related cap and septa.
3. Place the sample vial in a selectable position of the sampler tray.
4. Fill one of the solvent wash vials with hexane. Place the solvent in solvent wash position A.
5. Set-up the data system to perform the analysis of the sample.
6. According to the detector in use, refer to the resulting chromatogram and the acceptance criteria indicating the successful completation of the checkout:
   - *TriPlus AS - S/SL - FID Checkout*
   - *TriPlus AS - S/SL - ECD Checkout*
   - *TriPlus AS - S/SL - NPD Checkout*
   - *TriPlus AS - S/SL - FPD Checkout*
   - *TriPlus AS - S/SL - TCD Checkout*

TriPlus AS - S/SL - FID Checkout

1. The resulting chromatogram should look like the one shown in Figure 2-1.

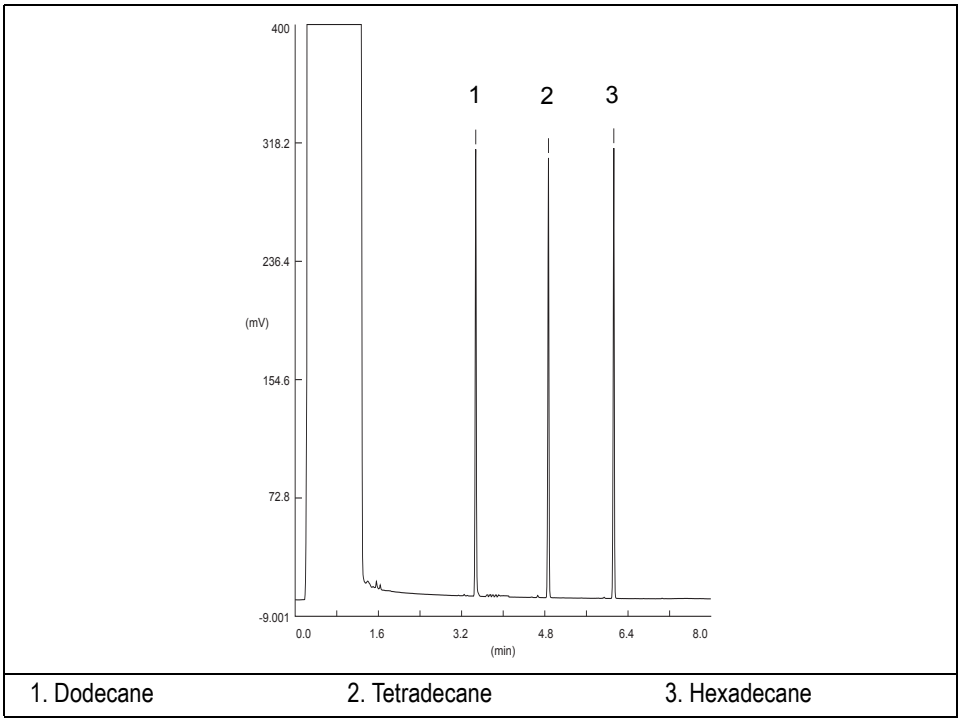


Figure 2-1. TriPlus AS - S/SL - FID Injection

2. The following criteria indicate successful completation of the checkout.

Table 2-5. TriPlus AS - S/SL - FID Acceptance Criteria

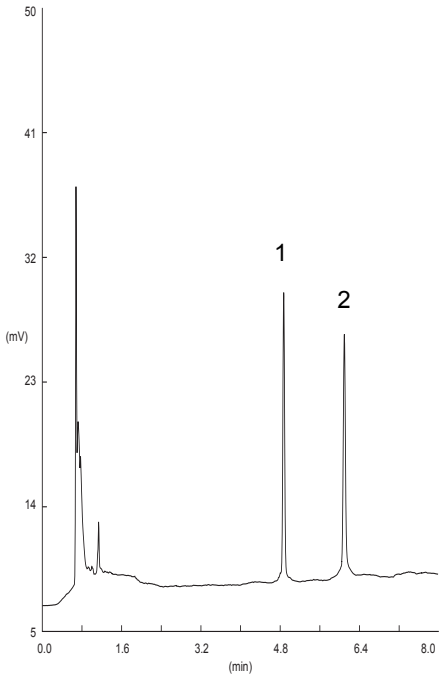
Analytical Results	
Area for each component	> 30 000 000

CAUTION **As default, the Signal Time in Chrom-Card WCC.INI is set to 0, then the acceptance values will result to be 10 times lower than the values reported in Table 2-5. To obtain the same values set Signal Time in Chrom-Card WCC.INI to 1.**

3. If these criteria are not met, repeat the test.

TriPlus AS - S/SL - ECD Checkout

1. The resulting chromatogram should look like the one shown in Figure 2-2.



1. Lindane 2. Aldrin

Figure 2-2. TriPlus AS - S/SL - ECD Injection

2. The following criteria indicate successful completation of the checkout.

Table 2-6. TriPlus AS - S/SL - ECD Acceptance Criteria

Analytical Results	
Lindane Signal-to-Noise Ratio	> 3 500
Aldrin Signal-to-Noise Ratio	> 3 500

3. If these criteria are not met, repeat the test.

TriPlus AS - S/SL - NPD Checkout

1. The resulting chromatogram should look like the one shown in Figure 2-3.

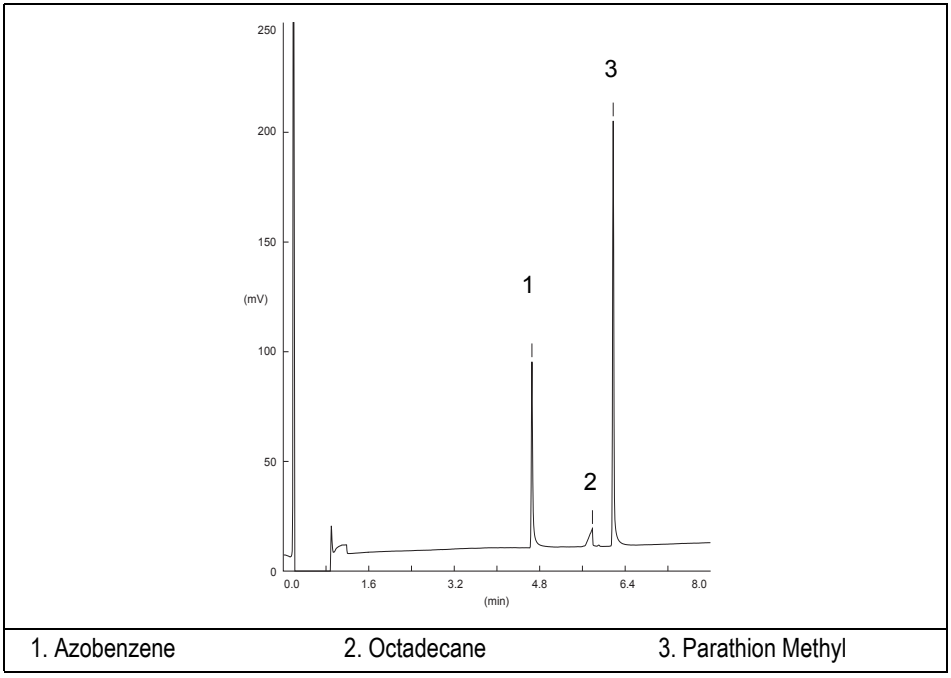


Figure 2-3. TriPlus AS - S/SL - NPD Injection

2. The following criteria indicate successful completation of the checkout.

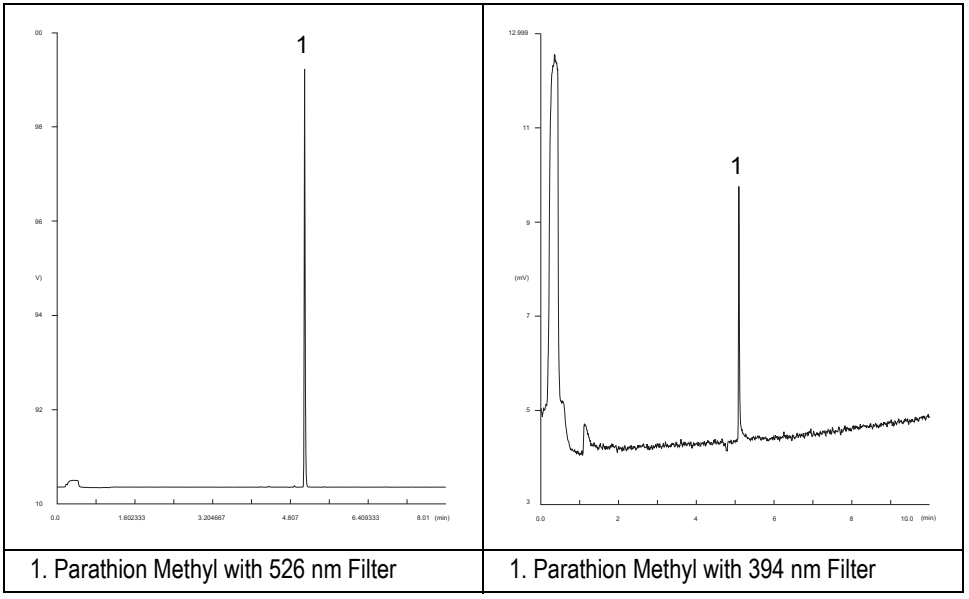
Table 2-7. TriPlus AS - S/SL - NPD Acceptance Criteria

Analytical Results	
Azobenzene Signal-to-Noise Ratio	650
Parathion Methyl Signal-to-Noise Ratio	> 1 800
Octadecane Signal-to-Noise Ratio	Negligible

3. If these criteria are not met, repeat the test.

TriPlus AS - S/SL - FPD Checkout

1. The resulting chromatogram should look like the one shown in Figure 2-4.



1. Parathion Methyl with 526 nm Filter | 1. Parathion Methyl with 394 nm Filter

Figure 2-4. TriPlus AS - S/SL - FPD Injection

2. The following criteria indicate successful completation of the checkout.

Table 2-8. TriPlus AS - S/SL - FPD Acceptance Criteria

Analytical Results		
Parathion Methyl Signal-to-Noise Ratio	**394 nm (S)**	**526 nm (P)**
	> 24	> 1 200

3. If these criteria are not met, repeat the test.

TriPlus AS - S/SL - TCD Checkout

1. The resulting chromatogram should look like the one shown in Figure 2-5.

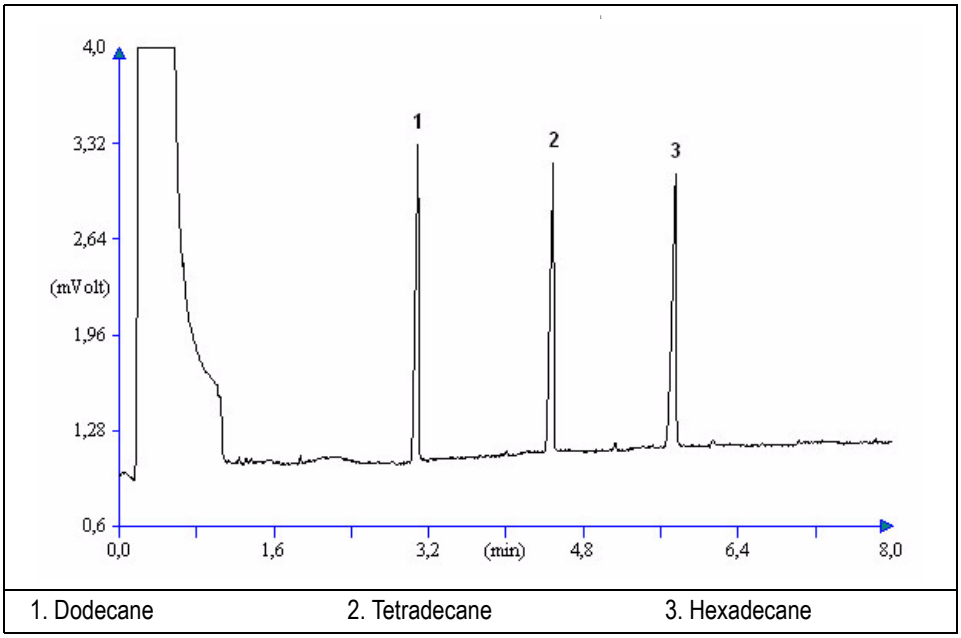


Figure 2-5. TriPlus AS - S/SL - TCD Injection

2. The following criteria indicate successful completation of the checkout.

Table 2-9. TriPlus AS - S/SL - TCD Acceptance Criteria

Analytical Results	
Area for each component	> 260 000

CAUTION **As default, the Signal Time in Chrom-Card WCC.INI is set to 0, then the acceptance values will result to be 10 times lower than the values reported in Table 2-9. To obtain the same values set Signal Time in Chrom-Card WCC.INI to 1.**

3. If these criteria are not met, repeat the test.

3

TriPlus AS Checkout Using OCI Injector

Chapter at a Glance...

Operating Procedures

SOP Number: TE P0579/01/E - 30 October 2006

Scope

Use the following procedure to verify proper TriPlus **AS** Version operation combined with OCI Injector and FID, ECD, NPD, FPD detectors.

Parts Referenced

Table 3-1. TriPlus AS - OCI - Detectors Parts Referenced

Part	Description	Part Number
Test Column	Fused Silica Capillary Column TR-5; 7 m long; 0.32 mm ID; 0.25 µm film thickness.	260 800 01
Pre-column	2 m long; 0.53 mm ID	260 603 75
Press-fit	Set of Press-fit connectors for TRACE OC	350 038 4
Graphite Ferrule	Graphite ferrule for 0.32 mm ID Column	290 134 87
Retaining Nut	M4 capillary column retaining nut	350 324 23
Syringe	10 µL size; 80 mm needle length	365 020 19
Vials, Seals and Caps	10 mL Crimp-top washing solvent vials 100 mL washing solvent bottle (requires P/N 386 060 74) 10 mL Crimp-top waste vial 2 mL sample vial (requires P/N 386 060 92)	190 046 47 240 140 85 190 505 50 240 140 21
Test Mixture for FID	Three components, Dodecane, Tetradecane, Hexadecane, in n-Hexane:	338 190 20
Test Mixture for ECD	Two components Lindane, Aldrin, in Iso-octane	338 190 11
Test Mixture for NPD and FPD	Three components, Azobenzene, Octadecane, Parathion Methyl, in Iso-Octane	338 190 06
Interferential Filter	526 nm for phosphorus 394 nm for sulphur	281 071 00 281 070 00
Data Acquisition	Chrom-Card (10 V F.S.), ChromQuest, Xcalibur,	

Analytical Conditions

Set the parameters listed in the following Tables 3-2 and 3-3.

TriPlus AS

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Table 3-2. TriPlus AS - OCI Analytical Conditions

Parameters Setting	
Analysis Type	Analysis Type = Single
Mode, Injector, Synch	Injection mode = Basic Injector port = Injector A (On-Column) Start synch mode = Standard
Sampling Parameters	Sample volume = 1.0 µL Plunger strokes = 10 Air volume = 3.0 µL Filling volume = 3.5 µL
Vial Sample Depth	Vial Depth = Bottom
Injection Depth Mode	Pre-injection dwell time = 1.0 s Post-injection dwell time = 2.0 s Injection Depth = Max Injection Speed = 100 µL/s
Viscosity	Sample pull-up speed = 1 µL/s Delay after bubble elimination = 4.0 s Viscosity delay = 1.0 s
Pre-injection Washes Parameters	Solvents wash sequence = A, - , - , - Solvent cycles = 1 Solvent Volume = 1.0 µL
Sample Washes Parameters	Rinses cycles = 1 Rinses volume = 1.5 µL
Post-injection Washes Parameters	Solvents wash sequence = A, - , - , - Solvent cycles = 1 Solvent Volume = 1.0 µL
Advanced Parameters	Wash solvent depth% = 100 Waste depth% = 15 Needle speed into vial = 10 mm/s Solvent filling speed = 20 µL/s Bubble elimination pulling speed = 10 µL/s Delay between strokes = 0.1 s

Gas Chromatograph

Table 3-3. Gas Chromatograph Analytical Conditions

Parameters	FID	ECD	NPD	FPD
Gases				
Carrier Gas: Helium = 30 kPa Constant Pressure				
Hydrogen (mL/min.)	35	---	2.3	90
Air (mL/min.)	350	---	60	115
Make-up Gas (mL/min.)	30	30	15	---
Oven Program				
Initial Temperature (°C)	70	85	85	85
Initial Time (min.)	1	1	1	1
Ramp 1 (°C/min.)	20	20	20	20
Final Temperature (°C)	200	220	230	230
Final Time (min.)	1	1	1	1
OCI Injector				
Secondary Cooling (min.)	0.2	3 s	0.2	10 s
Detector				
Base temperature (°C)	250	250	300	300
Detector Temperature (°C)	---	300	---	150
Detector Signal Range - See [**]	10^0	---	10^0	10^0
Reference Current (nA)		1		
Pulse Amplitude (V)		50		
Pulse Width (µs)		1		
Source Current			[*]	
Polarizer Voltage (V)			3.5	
High Voltage Mode				No

[*] Refer to the Source Ignition procedure reported in the SOP Manual
[[**] In the case your GC is equipped with the previous non-fast FPD control card, labeled FPD, set Detector Signal Range to 10^1.

Data System

Table 3-4. Data System

Parameters Setting	
Digital Signal Output	Acquisition Frequency = 10 Hz

Recommended Initial Operations

Before starting perform the SOP related to the GC configuration used as described in the relevant SOP manual.

OPERATING PROCEDURE

TriPlus AS - OCI - Detectors Checkout

1. Perform an off-line blank run of the column.
2. Prepare the sample vial introducing 1 mL of test mixture into the empty vial and closing it immediately with the related cap and septa.
3. Place the sample vial in a selectable position of the sampler tray.
4. Fill one of the solvent wash vials with hexane. Place the solvent in solvent wash position A.
5. Set-up the data system to perform the analysis of the sample.
6. According to the detector in use, refer to the resulting chromatogram and the acceptance criteria indicating the successful completation of the checkout:
   - *TriPlus AS - OCI - FID Checkout*
   - *TriPlus AS - OCI - ECD Checkout*
   - *TriPlus AS - OCI - NPD Checkout*
   - *TriPlus AS - OCI - FPD Checkout*

TriPlus AS - OCI - FID Checkout

1. The resulting chromatogram should look like the one shown in Figure 3-1.

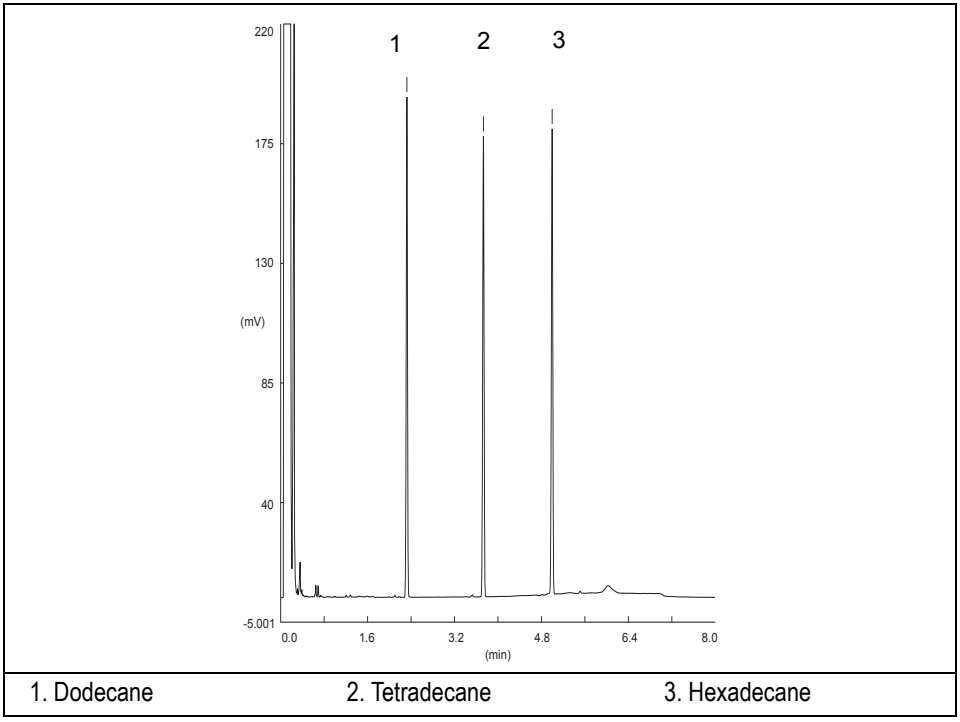


Figure 3-1. TriPlus AS - OCI - FID Injection

2. The following criteria indicate successful completation of the checkout.

Table 3-5. TriPlus AS - OCI - FID Acceptance Criteria

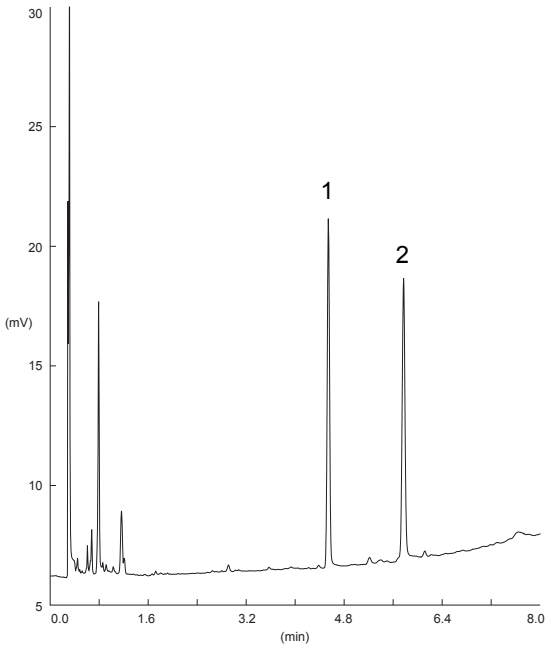
Analytical Results	
Area for each component	> 25 000 000

CAUTION **As default, the Signal Time in Chrom-Card WCC.INI is set to 0, then the acceptance values will result to be 10 times lower than the values reported in Table 3-5. To obtain the same values set Signal Time in Chrom-Card WCC.INI to 1.**

3. If these criteria are not met, repeat the test.

TriPlus AS - OCI - ECD Checkout

1. The resulting chromatogram should look like the one shown in Figure 3-2.



1. Lindane 2. Aldrin

Figure 3-2. TriPlus AS - OCI - ECD Injection

2. The following criteria indicate successful completation of the checkout.

Table 3-6. TriPlus AS - OCI - ECD Acceptance Criteria

Analytical Results	
Lindane Signal-to-Noise Ratio	> 3 000
Aldrin Signal-to-Noise Ratio	> 3 000

3. If these criteria are not met, repeat the test.

TriPlus AS - OCI - NPD Checkout

1. The resulting chromatogram should look like the one shown in Figure 3-3.

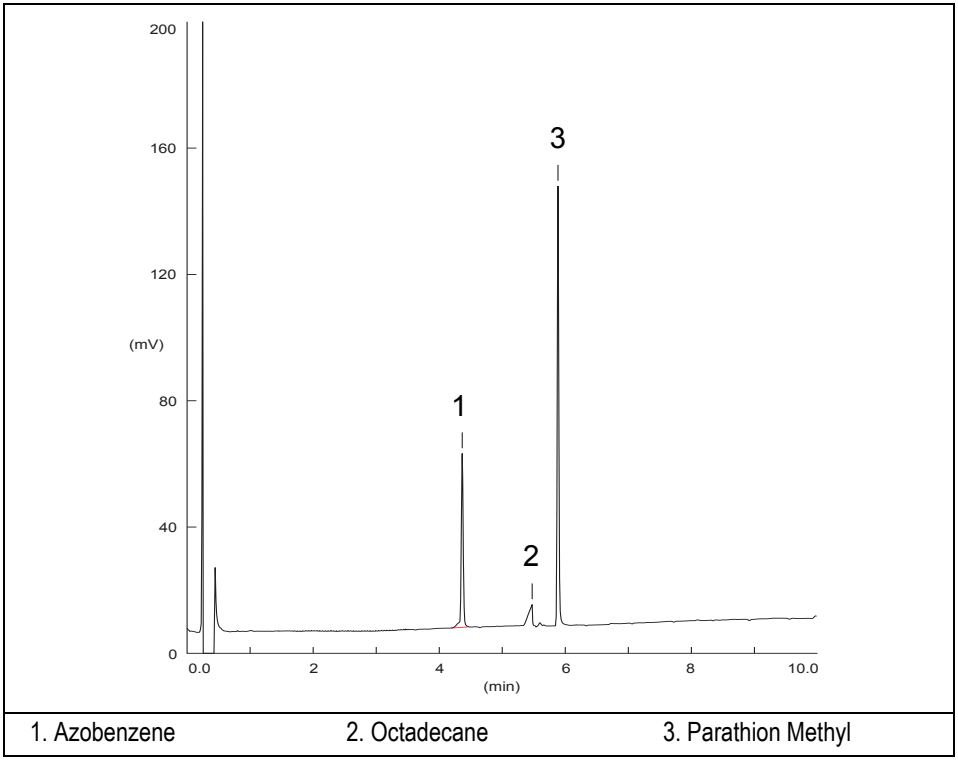


Figure 3-3. TriPlus AS - OCI - NPD Injection

2. The following criteria indicate successful completation of the checkout.

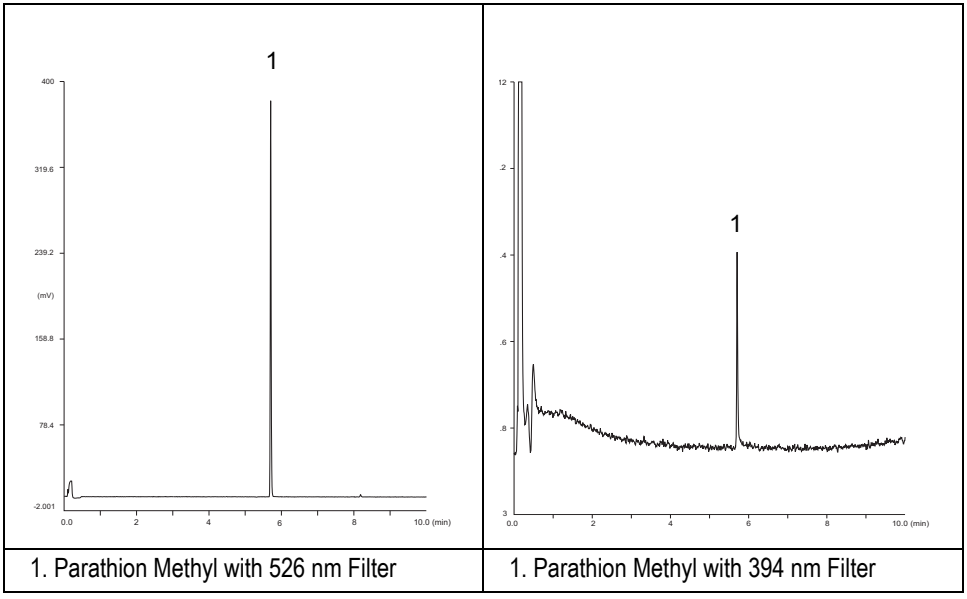
Table 3-7. TriPlus AS - OCI - NPD Acceptance Criteria

Analytical Results	
Azobenzene Signal-to-Noise Ratio	> 550
Parathion Methyl Signal-to-Noise Ratio	> 1 500
Octadecano Signal-to-Noise Ratio	Negligible

3. If these criteria are not met, repeat the test.

TriPlus AS - OCI - FPD Checkout

1. The resulting chromatogram should look like the one shown in Figure 3-4.



1. Parathion Methyl with 526 nm Filter | 1. Parathion Methyl with 394 nm Filter

Figure 3-4. TriPlus AS - OCI - FPD Injection

2. The following criteria indicate successful completation of the checkout.

Table 3-8. TriPlus AS - OCI - FPD Acceptance Criteria

Analytical Results		
Parathion Methyl Signal to Noise Ratio	394 nm (S) Filter	526 nm (P) Filter
	> 20	> 1 000

3. If these criteria are not met, repeat the test.

4

TriPlus AS Checkout Using PTV Injector

Chapter at a Glance...

Operating Procedures

SOP Number: TE P0580/01/E - 30 October 2006

Scope

Use the following procedure to verify proper TriPlus **AS** Version operation combined with PTV Injector and FID, ECD, NPD, FPD, detectors.

Parts Referenced

Table 4-1. TriPlus AS - PTV - Detectors Parts Referenced

Part	Description	Part Number
Test Column	Fused Silica Capillary Column TR-5; 7 m long; 0.32 mm ID; 0.25 µm film thickness.	260 800 01
Glass Liner	Silcosteel 2 mm ID	453 220 44
Liner Seal	Graphite seal for liner	290 034 17
Graphite Ferrule	Graphite ferrule for 0.32 mm ID Column	290 134 87
Retaining Nut	M4 capillary column retaining nut	350 324 23
Septum	Standard septum for PTV injector (set of 10)	313 132 25
Syringe	10 µL size; 50 mm needle length 10 µL size; 80 mm needle length	365 005 25 365 020 19
Vials, Seals and Caps	10 mL Crimp-top washing solvent vials 100 mL washing solvent bottle (requires P/N 386 060 74) 10 mL Crimp-top waste vial 2 mL sample vial (requires P/N 386 060 92)	190 046 47 240 140 85 190 505 50 240 140 21
Test Mixture for FID	Three components, Dodecane, Tetradecane, Hexadecane, in n-Hexane:	338 190 20
Test Mixture for TCD	Three components, Dodecane, Tetradecane, Hexadecane, in n-Hexane	338 190 16
Test Mixture for ECD	Two components Lindane, Aldrin, in Iso-octane	338 190 11
Test Mixture for NPD and FPD	Three components, Azobenzene, Octadecane, Parathion Methyl, in Iso-Octane	338 190 06
Interferential Filter	526 nm for phosphorus 394 nm for sulphur	281 071 00 281 070 00
Data Acquisition	Chrom-Card (10 V F.S.), ChromQuest, Xcalibur,	

Analytical Conditions

Set the parameters listed in the following Tables 4-2 and 4-3.

TriPlus AS

.

Table 4-2. TriPlus AS - PTV Analytical Conditions

Parameters Setting	
Analysis Type	Analysis Type = Single
Mode, Injector, Synch	Injection mode = Basic Injector port = Injector A (PTV) Start synch mode = Standard
Sampling Parameters	Sample volume = 1.0 µL Plunger strokes = 10 Air volume = 3.0 µL Filling volume = 3.5 µL
Vial Sample Depth	Vial Depth = Bottom
Injection Depth Mode	Pre-injection dwell time = 1.0 s Post-injection dwell time = 0.5 s Injection Depth = Max Injection Speed 100 µL/s
Viscosity	Sample pull-up speed = 1 µL/s Delay after bubble elimination = 4.0 s Viscosity delay = 1.0 s
Pre-injection Washes Parameters	Solvents wash sequence = A, - , - , - Solvent cycles = 1 Solvent Volume = 1.0 µL
Sample Washes Parameters	Rinses cycles = 1 Rinses volume = 1.5 µL
Post-injection Washes Parameters	Solvents wash sequence = A, - , - , - Solvent cycles = 1 Solvent Volume = 1.0 µL
Advanced Parameters	Wash solvent depth% = 100 Waste depth% = 15 Needle speed into vial = 10 mm/s Solvent filling speed = 20 µL/s Bubble elimination pulling speed = 10 µL/s Delay between strokes = 0.1 s

Gas Chromatograph

Table 4-3. Gas Chromatograph Analytical Conditions

Parameters	FID	ECD	NPD	FPD
Gases				
Carrier Gas: Helium = 30 kPa Constant Pressure				
Hydrogen (mL/min.)	35	---	2.3	90
Air (mL/min.)	350	---	60	115
Make-up Gas (mL/min.)	30	30	30	---
Oven Program				
Initial Temperature (°C)	50	70	70	70
Initial Time (min.)	1	1	1	1
Ramp 1 (°C/min.)	20	20	20	20
Final Temperature (°C)	200	220	230	230
Final Time (min.)	1	1	1	1
PTV Injector				
Operating Mode: = *PTV Splitless* (PTVSL)	PTVSL	PTVSL	PTVSL	PTVSL
Splitless Time (min.)	0.8	0.8	0.8	0.8
Split Flow (mL/min.)	50	60	50	50
Constant Septum Purge	Yes	Yes	Yes	Yes
Inject Temperature (°C)	50	50	50	50
Inject Time (min.)	0.1	0.1	0.1	0.1
Transfer Ramp (°C/s)	10	10	10	10
Transfer Temperature (°C)	260	260	260	260
Transfer Time (min.)	1	1	1	1
Detector				
Base temperature (°C)	250	250	300	300
Detector Temperature (°C)	---	300	---	150
Detector Signal Range - See [**]	10^0	---	10^0	10^0
Reference Current (nA)		1		
Pulse Amplitude (V)		50		

Table 4-3. Gas Chromatograph Analytical Conditions (Continued)

Parameters	FID	ECD	NPD	FPD
Pulse Width (µs)		1		
Source Current			[*]	
Polarizer Voltage (V)			3.5	
High Voltage Mode				No

[*] Refer to the Source Ignition procedure reported in the SOP Manual
[**] In the case your GC is equipped with the previous non-fast FPD control card, labeled FPD, set Detector Signal Range to 10^1.

Data System

Table 4-4. Data System

Parameters Setting	
Digital Signal Output	Acquisition Frequency = 10 Hz

Recommended Initial Operations

Before starting perform the SOP related to the GC configuration used as described in the relevant SOP manual.

OPERATING PROCEDURE

TriPlus AS - PTV - Detectors Checkout

1. Perform an off-line blank run of the column.
2. Prepare the sample vial introducing 1 mL of test mixture into the empty vial and closing it immediately with the related cap and septa.
3. Place the sample vial in a selectable position of the sampler tray.
4. Fill one of the solvent wash vials with hexane. Place the solvent in solvent wash position A.
5. Set-up the data system to perform the analysis of the sample.
6. According to the detector in use, refer to the resulting chromatogram and the acceptance criteria indicating the successful completation of the checkout:
   - *TriPlus AS - PTV - FID Checkout*
   - *TriPlus AS - PTV - ECD Checkout*
   - *TriPlus AS - PTV - NPD Checkout*
   - *TriPlus AS - PTV - FPD Checkout*

TriPlus AS - PTV - FID Checkout

1. The resulting chromatogram should look like the one shown in Figure 4-1.

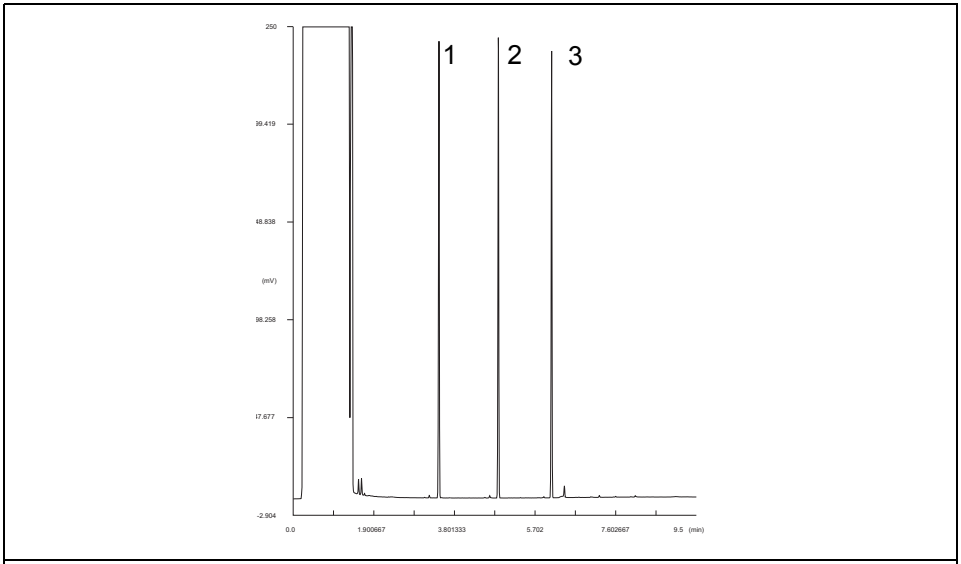


Figure 4-1. TriPlus AS - PTV - FID Injection

2. The following criteria indicate successful completation of the checkout.

Table 4-5. TriPlus AS - PTV - FID Acceptance Criteria

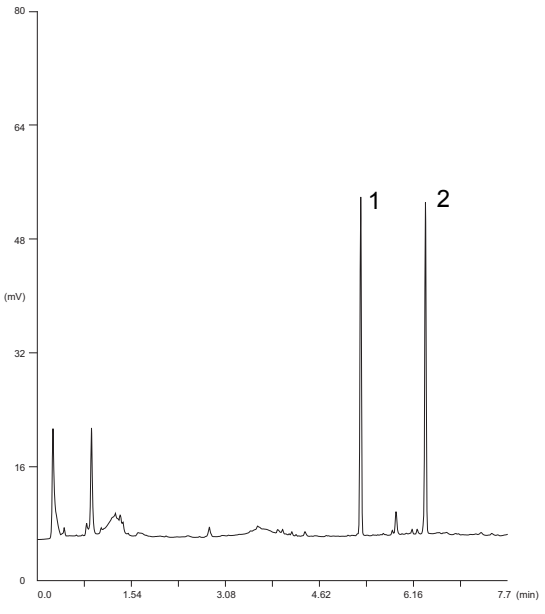
Analytical Results	
Area for each component	> 25 000 000

CAUTION **As default, the Signal Time in Chrom-Card WCC.INI is set to 0, then the acceptance values will result to be 10 times lower than the values reported in Table 4-5. To obtain the same values set Signal Time in Chrom-Card WCC.INI to 1.**

3. If these criteria are not met, repeat the test.

TriPlus AS - PTV - ECD Checkout

1. The resulting chromatogram should look like the one shown in Figure 4-2.



1. Lindane 2. Aldrin

Figure 4-2. TriPlus AS - PTV - ECD Injection

2. The following criteria indicate successful completation of the checkout.

Table 4-6. TriPlus AS - PTV - ECD Acceptance Criteria

Analytical Results	
Lindane Signal-to-Noise Ratio	> 3 000
Aldrin Signal-to-Noise Ratio	> 3 000

3. If these criteria are not met, repeat the test.

TriPlus AS - PTV - NPD Checkout

1. The resulting chromatogram should look like the one shown in Figure 4-3.

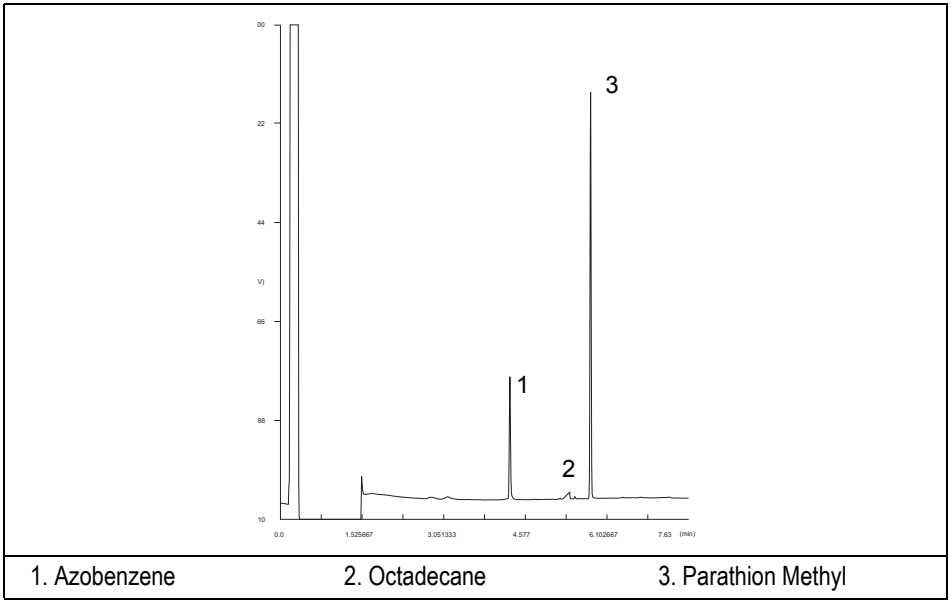


Figure 4-3. TriPlus AS - PTV - NPD Injection

2. The following criteria indicate successful completation of the checkout.

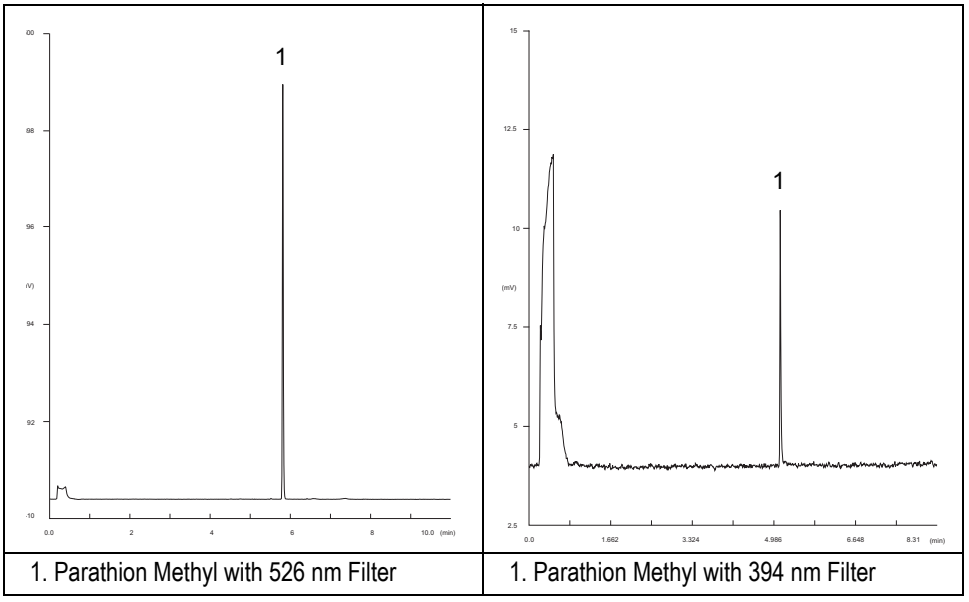
Table 4-7. TriPlus AS - PTV - NPD Acceptance Criteria

Analytical Results	
Azobenzene Signal-to-Noise Ratio	> 550
Parathion Methyl Signal-to-Noise Ratio	> 1 500
Octadecane Signal-to-Noise Ratio	Negligible

3. If these criteria are not met, repeat the test.

TriPlus AS - PTV - FPD Checkout

1. The resulting chromatogram should look like the one shown in Figure 4-4.



1. Parathion Methyl with 526 nm Filter	1. Parathion Methyl with 394 nm Filter

Figure 4-4. TriPlus AS - PTV - FPD Injection

2. The following criteria indicate successful completation of the checkout.

Table 4-8. TriPlus AS - PTV - FPD Acceptance Criteria

Analytical Results		
Parathion Methyl Signal-to-Noise Ratio	**394 nm (S) Filter**	**526 nm (P) Filter**
	> 20	> 1 000

3. If these criteria are not met, repeat the test.

SECTION III

TriPlus HS

This section, contains the procedures to test the TriPlus HS version combined with different injectors and detectors.

Chapter 5, *TriPlus HS Checkout Using S/SL Injector*

Chapter 6, *TriPlus HS Checkout Using PTV Injector*

5

TriPlus HS Checkout Using S/SL Injector

Chapter at a Glance...

Operating Procedures

SOP Number: TE P0581/01/E - 30 October 2006

Scope

Use the following procedure to verify proper TriPlus **HS** Version operation combined with S/SL Injector and FID, ECD detectors.

Parts Referenced

Table 5-1. TriPlus HS - S/SL - Detectors Parts Referenced

Part	Description	Part Number
Test Column	Fused Silica Capillary Column TR-5; 7 m long; 0.32 mm ID; 0.25 µm film thickness.	260 800 01
Glass Liner	3 mm ID for splitless injection	453 200 32
Liner Seal	Graphite seal for glass liner	290 334 06
Graphite Ferrule	Graphite ferrule for 0.32 mm ID Column	290 134 87
Retaining Nut	M4 capillary column retaining nut	350 324 23
Septum	Standard septum for S/SL injector	313 032 11
Syringe	2.5 mL syringe w/plunger	190 052 27
Vials, Seals and Caps	20 mL sample vial (requires P/N 386 036 00)	240 063 05
Test Mixture for FID	Three components, Dodecane, Tetradecane, Hexadecane, in n-Hexane:	338 190 20
Test Mixture for ECD	Two components Lindane, Aldrin, in Iso-octane	338 190 11
Data Acquisition	Chrom-Card (10 V F.S.), ChromQuest, Xcalibur,	

Analytical Conditions

Set the parameters listed in the following Tables 5-2 and 5-3.

TriPlus HS

.

Table 5-2. TriPlus HS - S/SL Analytical Conditions

Parameters Setting	
Analysis Type	Analysis Type = Single
Injector, Mode Enrichments	Injector port = Injector A (SSL) Incubation Mode = Constant Analysis time = 10 min. Sample draw = 1.0 mL Enrichments # = 0 Enrichments Delay = 0.1 min. Sampling depth in vial = Standard Sampling vial depth = 25 mm
Incubation Parameters	Agitator temperature = 70 °C Agitator On time = 15 s Agitator Off time = 5 s Incubation time = 1.0 min.
Syringe Parameters	Syringe temperature = 70 °C Enable prefilling = No Filling volume = 2 mL Filling counts # = 10 Filling delay = 0 s Pre-injection syringe flush = No
Speed Parameters	Filling speed = 100 mL/min. Injection speed = 20 mL/min.
Injection Parameters	Injection depth = 35 mm Pre-injection delay = 0 s Post-injection delay = 0 s
Post-Injection Syringe Washes Parameters	Solvents wash sequence = A, -, -, - Solvent cycles = 1 Solvent volume = 1.0 mL Dry time = 5 s Wash frequency = Never
Standby Parameters	St-by incubation temperature = 70 °C St-by syringe temperature = 70 °C St-by syringe flush = Off

Table 5-2. TriPlus HS - S/SL Analytical Conditions (Continued)

Parameters Setting	
Synch Mode	Start sync mode = Normal
Anticipated Sync Before End of Incubation Time	Anticipated time = 3.0 min.
Advanced Parameters	Needle speed into vial = 20 mm/s

Gas Chromatograph

Table 5-3. Gas Chromatograph Analytical Conditions

Parameters	FID	ECD
Gases		
Carrier Gas: Helium = 30 kPa Constant Pressure		
Hydrogen (mL/min.)	35	---
Air (mL/min.)	350	---
Make-up Gas (mL/min.)	30	30
Oven Program		
Initial Temperature (°C)	40	70
Initial Time (min.)	1	1
Ramp 1 (°C/min.)	20	20
Final Temperature (°C)	180	220
Final Time (min.)	0.5	1
PrepRun Time-out (min)	20	20
S/SL Injector		
Operating Mode = *Splitless* (SL)	SL	SL
Temperature (°C)	220	220
Splitless Time (min.)	0.8	0.8
Split Flow (mL/min.)	60	60
Constant Septum Purge	Yes	Yes
Detector		
Base temperature (°C)	250	250

Table 5-3. Gas Chromatograph Analytical Conditions (Continued)

Parameters	FID	ECD
Detector Temperature (°C)	---	250
Detector Signal Range	10[1]	---
Reference Current (nA)		1
Pulse Amplitude (V)		50
Pulse Width (µs)		1

Data System

Table 5-4. Data System

Parameters Setting	
Digital Signal Output	Acquisition Frequency = 10 Hz

Recommended Initial Operations

Before starting perform the SOP related to the GC configuration used as described in the relevant SOP manual.

OPERATING PROCEDURE

TriPlus HS - S/SL - Detectors Checkout

1. Perform an off-line blank run of the column.
2. Set-up the data system to perform the analysis of the sample.
3. Prepare the sample vial introducing 10 µL of test mixture into the empty vial and closing it immediately with the related cap and septa.
4. Place the sample vial in a selectable position of the sampler tray.

CAUTION **To meet the acceptance criteria, the analysis must be performed immediately after the sample vial preparation.**

5. According to the detector in use, refer to the resulting chromatogram and the acceptance criteria indicating the successful completation of the checkout:
   - *TriPlus HS - S/SL - FID Checkout*
   - *TriPlus HS - S/SL - ECD Checkout*

TriPlus HS - S/SL - FID Checkout

1. The resulting chromatogram should look like the one shown in Figure 5-1.

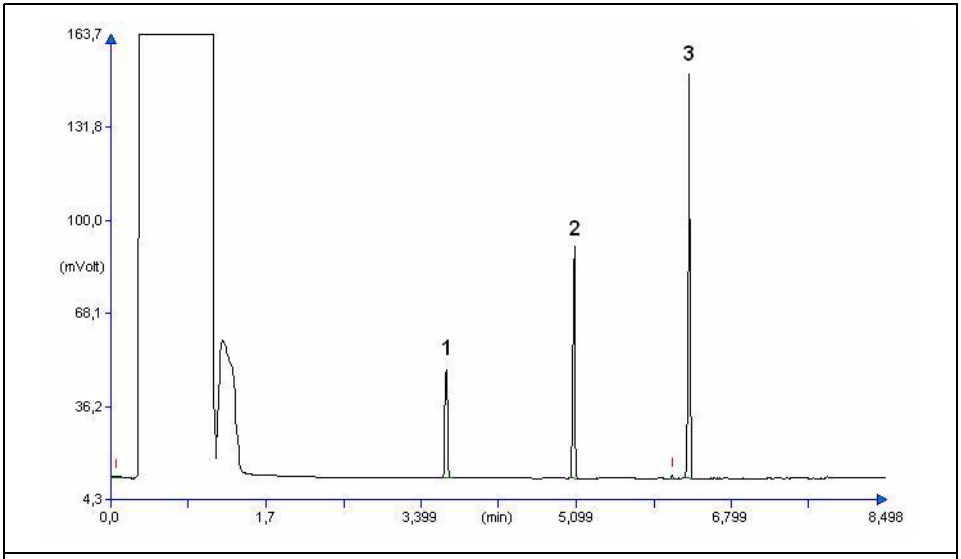


Figure 5-1. TriPlus HS - S/SL - FID Injection

2. The following criteria indicate successful completation of the checkout.

Table 5-5. TriPlus HS - S/SL - FID Acceptance Criteria

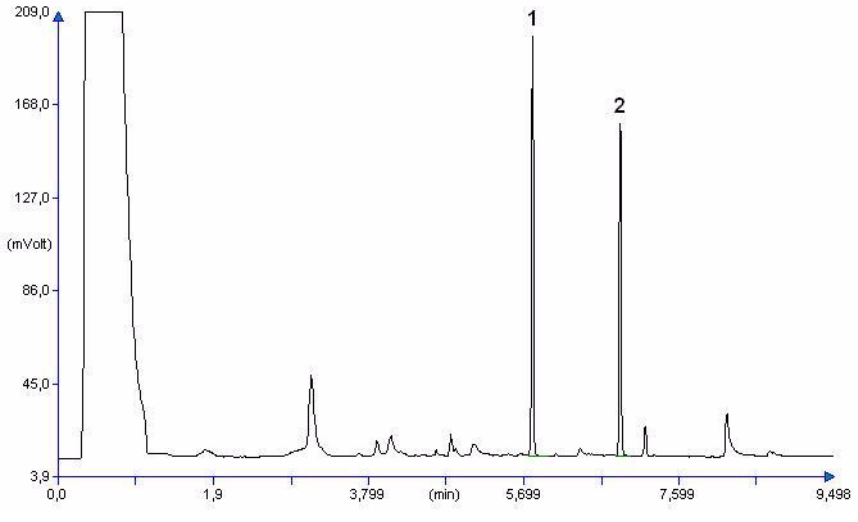
Analytical Results	
Area for Dodecane	> 5 000 000
Area for Tetradecane	> 10 000 000
Area for Hexadecane	> 15 000 000

CAUTION **As default, the Signal Time in Chrom-Card WCC.INI is set to 0, then the acceptance values will result to be 10 times lower than the values reported in Table 5-5. To obtain the same values set Signal Time in Chrom-Card WCC.INI to 1.**

3. If these criteria are not met, repeat the test.

TriPlus HS - S/SL - ECD Checkout

1. The resulting chromatogram should look like the one shown in Figure 5-2.



1. Lindane 2. Aldrin

Figure 5-2. TriPlus HS - S/SL - ECD Injection

2. The following criteria indicate successful completation of the checkout.

Table 5-6. TriPlus HS - S/SL - ECD Acceptance Criteria

Analytical Results	
Lindane Signal-to-Noise Ratio	> 1 000
Aldrin Signal-to-Noise Ratio	> 1 000

3. If these criteria are not met, repeat the test. +

6

TriPlus HS Checkout Using PTV Injector

Chapter at a Glance...

Operating Procedures

SOP Number: TE P0582/01/E - 30 October 2006

Scope

Use the following procedure to verify proper TriPlus **HS** Version operation combined with PTV Injector and FID, ECD detectors.

Parts Referenced

Table 6-1. TriPlus HS - PTV - Detectors Parts Referenced

Part	Description	Part Number
Test Column	Fused Silica Capillary Column TR-5; 7 m long; 0.32 mm ID; 0.25 µm film thickness.	260 800 01
Glass Liner	Silcosteel 2 mm ID	453 220 44
Liner Seal	Graphite seal for liner	290 034 17
Graphite Ferrule	Graphite ferrule for 0.32 mm ID Column	290 134 87
Retaining Nut	M4 capillary column retaining nut	350 324 23
Septum	Standard septum for PTV injector (set of 10)	313 132 25
Syringe	2.5 mL syringe w/plunger	190 052 27
Vials, Seals and Caps	20 mL sample vial (requires P/N 386 036 00)	240 063 05
Test Mixture for FID	Three components, Dodecane, Tetradecane, Hexadecane, in n-Hexane:	338 190 20
Test Mixture for ECD	Two components Lindane, Aldrin, in Iso-octane	338 190 11
Data Acquisition	Chrom-Card (10 V F.S.), ChromQuest, Xcalibur,	

Analytical Conditions

Set the parameters listed in the following Tables 6-2 and 6-3.

TriPlus HS

.

Table 6-2. TriPlus HS - PTV Analytical Conditions

Parameters Setting	
Analysis Type	Analysis Type = Single
Injector, Mode Enrichments	Injector port = Injector B (PTV) Incubation Mode = Constant Analysis time = 10 min. Sample draw = 1.0 mL Enrichments # = 0 Enrichments Delay = 0.1 min. Sampling depth in vial = Standard Sampling vial depth = 25 mm
Incubation Parameters	Agitator temperature = 70 °C Agitator On time = 15 s Agitator Off time = 5 s Incubation time = 1.0 min.
Syringe Parameters	Syringe temperature = 70 °C Enable prefilling = No Filling volume = 2 mL Filling counts # = 10 Filling delay = 0 s Pre-injection syringe flush = No
Speed Parameters	Filling speed = 100 mL/min. Injection speed = 4 mL/min.
Injection Parameters	Injection depth = 35 mm Pre-injection delay = 0 s Post-injection delay = 0 s
Post-Injection Syringe Washes Parameters	Solvents wash sequence = A, -, -, - Solvent cycles = 1 Solvent volume = 1.0 mL Dry time = 5 s Wash frequency = Never
Standby Parameters	St-by incubation temperature = 70 °C St-by syringe temperature = 70 °C St-by syringe flush = Off

Table 6-2. TriPlus HS - PTV Analytical Conditions (Continued)

Parameters Setting	
Synch Mode	Start sync mode = Normal
Anticipated Sync Before End of Incubation Time	Anticipated time = 3 min.
Advanced Parameters	Needle speed into vial = 20 mm/s

Gas Chromatograph

.

Table 6-3. Gas Chromatograph Analytical Condition

Parameters	FID	ECD
Gases		
Carrier Gas: Helium = 30 kPa Constant Pressure		
Hydrogen (mL/min.)	35	---
Air (mL/min.)	350	---
Make-up Gas (mL/min.)	30	30
Oven Program		
Initial Temperature (°C)	40	70
Initial Time (min.)	1	1
Ramp 1 (°C/min.)	20	20
Final Temperature (°C)	180	220
Final Time (min.)	0.5	1
PrepRun Time-out (min)	20	20
PTV Injector		
Operating Mode = *CT Splitless* (CTSL)	CTSL	CTSL
Splitless Time (min.)	0.8	0.8
Split Flow (mL/min.)	60	60
Constant Septum Purge	Yes	Yes
Inject Temperature (°C)	220	220
Detector		
Base temperature (°C)	250	250

Table 6-3. Gas Chromatograph Analytical Condition (Continued)

Parameters	FID	ECD
Detector Temperature (°C)	---	250
Detector Signal Range	10[1]	---
Reference Current (nA)		1
Pulse Amplitude (V)		50
Pulse Width (µs)		1

Data System

Table 6-4. Data System

Parameters Setting	
Digital Signal Output	Acquisition Frequency = 10 Hz

Recommended Initial Operations

Before starting perform the SOP related to the GC configuration used as described in the relevant SOP manual.

OPERATING PROCEDURE

TriPlus HS - PTV - Detectors Checkout

1. Perform an off-line blank run of the column.
2. Set-up the data system to perform the analysis of the sample.
3. Prepare the sample vial introducing 10 µL of test mixture into the empty vial and closing it immediately with the related cap and septa.
4. Place the sample vial in a selectable position of the sampler tray.

CAUTION **To meet the acceptance criteria, the analysis must be performed immediately after the sample vial preparation.**

5. According to the detector in use, refer to the resulting chromatogram and the acceptance criteria indicating the successful completation of the checkout:
   - *TriPlus HS - PTV - FID Checkout*
   - *TriPlus HS - PTV - ECD Checkout*

TriPlus HS - PTV - FID Checkout

1. The resulting chromatogram should look like the one shown in Figure 6-1.

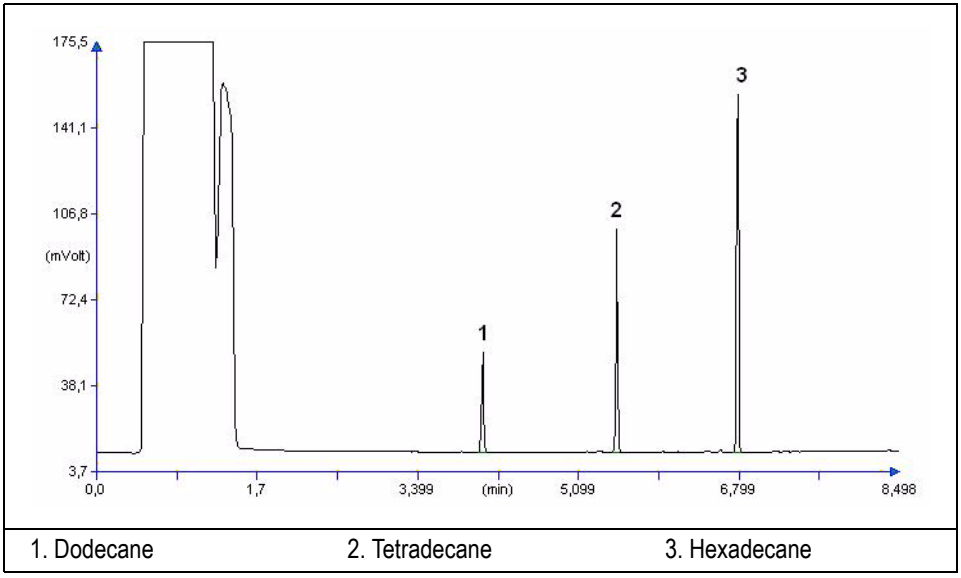


Figure 6-1. TriPlus HS - PTV - FID Injection

2. The following criteria indicate successful completation of the checkout.

Table 6-5. TriPlus HS - PTV - FID Acceptance Criteria

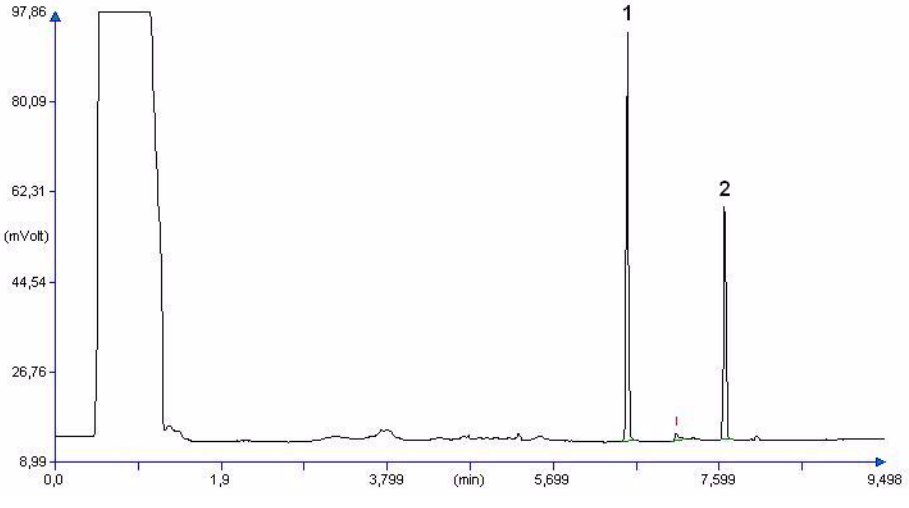
Analytical Results	
Area for Dodecane	> 5 000 000
Area for Tetradecane	> 10 000 000
Area for Hexadecane	> 15 000 000

CAUTION **As default, the Signal Time in Chrom-Card WCC.INI is set to 0, then the acceptance values will result to be 10 times lower than the values reported in Table 6-5. To obtain the same values set Signal Time in Chrom-Card WCC.INI to 1.**

3. If these criteria are not met, repeat the test.

TriPlus HS - PTV - ECD Checkout

1. The resulting chromatogram should look like the one shown in Figure 6-2.



1. Lindane 2. Aldrin

Figure 6-2. TriPlus HS - PTV - ECD Injection

2. The following criteria indicate successful completation of the checkout.

Table 6-6. TriPlus HS - PTV - ECD Acceptance Criteria

Analytical Results	
Lindane Signal-to-Noise Ratio	> 1 000
Aldrin Signal-to-Noise Ratio	> 1 000

3. If these criteria are not met, repeat the test.

SECTION IV

TriPlus SPME

This section, contains the procedures to test the TriPlus SPME version combined with different injectors and detectors.

7

TriPlus SPME Checkout Using S/SL Injector

Chapter at a Glance...

Operating Procedures

SOP Number: TE P0583/02/E - 01 October 2008

Scope

Use the following procedure to verify proper TriPlus **SPME** Version operation combined with S/SL injector and FID, ECD detectors.

Parts Referenced

Table 7-1. TriPlus SPME - S/SL Parts Referenced

Part	Description	Part Number
Test Column	Fused Silica Capillary Column TR-5; 7 mt long 0.32 mm ID; 0.25 µm film thickness.	260 800 01
Glass Liner	Liner 0.8 mm ID for S/SL	453 520 83
Liner Seal	Graphite seal for glass liner	290 334 06
Graphite Ferrule	Graphite ferrule for 0.32 mm ID Column	290 134 87
Retaining Nut	M4 capillary column retaining nut	350 324 23
Septum	Standard septum for S/SL injector	313 032 11
Septum Cap	Septum Cap for SPME Injection	347 500 04
Syringe	10 µl size; 70 mm needle length	365 001 03
Test Mixture for FID	Three components: Dodecane, Tetradecane, Hexadecane, in n-Hexane:	338 190 20
Test Mixture for ECD	Two components: Lindane, Aldrin, in Iso-octane	338 190 11
Data Acquisition	Chrom-Card, ChromQuest, Xcalibur	
SPME	Kit SPME for TriPlus containing: Syringe holder, Fiber holder, 100 µm PDMS fiber	
Conditioning Station	Fiber conditioning station (optional)	
Vials, Seals and Caps	20 mL sample vial (requires P/N 386 036 00)	240 063 05
Septa and Caps	Aluminium caps, 20 mm Silicon-Teflon septa	386 094 10
Crimper	Vial hand crimper	

Analytical Conditions

Set the parameters listed in the following Tables 7-2 and 7-3.

TriPlus SPME

.

Table 7-2. TriPlus SPME - S/SL Analytical Conditions

Parameters Setting	
General Parameters	Injector = S/SL Needle Speed In Vial = 20 mm/s Extraction Time = 10.0 min. Sampling Vial Depth = 25 mm Injection Depth = 35 mm Desorption Time = 1.0 min. Analysis Time = 0 minute
Incubation Parameters	Incubation Mode = Constant Agitator Temperature = 70 °C St-by Agitator Temperature = 70 °C Agitator ON Time = 600 s Incubation Time = 5 min. Agitator OFF Time = 0 s
Synch. Mode	GC Synch. Start Mode = When needle enters Inj.
Conditioning Station *(Fiber Conditioning Port if present or other injection port if available)*	Internal. Std Vial = None Conditioning Time = 8.0 min. Fiber Conditioning Port Temperature = 280 °C St-by Conditioning Port Temperature = 280 °C
SPME Fiber Holder Setup >> Fiber Zero Adjustment	Fiber Exposition = 21 mm

Gas Chromatograph

.

Table 7-3. Gas Chromatograph Analytical Condition

Parameters	FID	ECD
Gases		
Carrier Gas: Helium = 30 kPa Constant Pressure		
Hydrogen (mL/min.)	35	---
Air (mL/min.)	350	---
Make-up Gas (mL/min.)	30	30
Oven Program		
Initial Temperature (°C)	40	60
Initial Time (min.)	4	1
Ramp 1 (°C/min.)	40	20
Final Temperature (°C)	240	240
Final Time (min.)	0.1	1
PrepRun Time-out (min)	20	20
S/SL Injector		
Operating Mode = *Splitless* (SL)	SL	SL
Temperature (°C)	280	280
Splitless Time (min.)	1	1
Split Flow (mL/min.)	80	80
Stop Septum Purge (min.)	1	1
Detector		
Base temperature (°C)	250	250
Detector Temperature (°C)	---	300
Detector Signal Range	10^0	---
Reference Current (nA)		1
Pulse Amplitude (V)		50
Pulse Width (µs)		1

Data System

Table 7-4. Data System

Parameters Setting	
Digital Signal Output	Acquisition Frequency = 10 Hz

Recommended Initial Operations

Before starting perform the SOP related to the GC configuration used as described in the relevant SOP manual.

1. Replace the glass liner.
   The glass liner currently installed in your injector should be carefully removed and replaced with the 0.8 mm ID glass liner for SPME application, as required for the checkout, with the appropriate liner seal.

2. Replace the septum
   A new septum should be installed properly in your injector.

3. Replace the Septum Cap
   The appropriate septum cap for TriPlus SPME injection should be installed on your injector.

4. Perform the leak test.

5. Install the TriPlus SPME version, as described in the relevant chapter of the TriPlus user manual and connect it to the data handling.

6. Turn the sampler on and let the automatic recognition of the objects.

7. Set up the S/SL position as described in the relevant chapter of the TriPlus manual.

8. If the Fiber Conditioning Station is not present and another injector is present and can be used for the fiber bakeout, set also this injector.

9. Load the SOP method parameters for the TRACE GC and the TriPlus.

OPERATING PROCEDURE

TriPlus SPME - S/SL - Detectors Checkout

Fiber Conditioning, Sample Preparation and Analysis

Before start, let the new fiber condition at the temperature and for the time specified by the supplier.
If the Fiber conditioning station is present, it can be used for this purpose, otherwise a dedicated injector can be used for the conditioning.

1. Perform an off-line blank run of the column.
2. Set-up the data system to perform the analysis of the sample.
3. Prepare the sample vial introducing 10 µL of test mixture into the 20 mL empty vial and closing it immediately with the related cap and septa.
4. Place the sample vial in a selectable position of the sampler tray.

CAUTION **To meet the acceptance criteria, the analysis must be performed immediately after the sample vial preparation.**

5. According to the detector in use, refer to the resulting chromatogram and the acceptance criteria indicating the successful completation of the checkout:
   - *TriPlus SPME - S/SL - FID Checkout*
   - *TriPlus SPME - S/SL - ECD Checkout*

TriPlus SPME - S/SL - FID Checkout

1. The resulting chromatogram should look like the one shown in Figure 7-1.

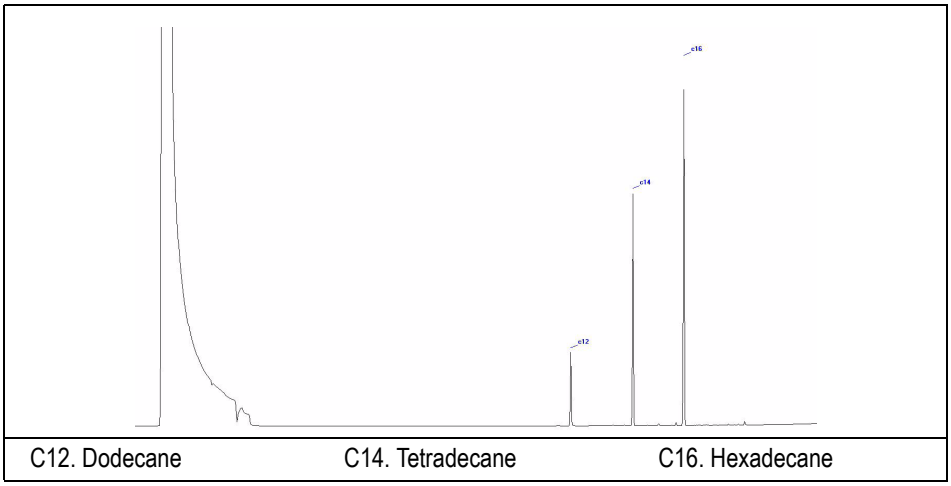


Figure 7-1. TriPlus SPME - S/SL - FID Injection

NOTE The difference of recovery, for the components analyzed, is due to different partitioning between the phase and the head space for the components at the temperature of equilibration.

2. The following criteria indicate successful completion of the checkout.

Table 7-5. TriPlus SPME - S/SL - FID Acceptance Criteria

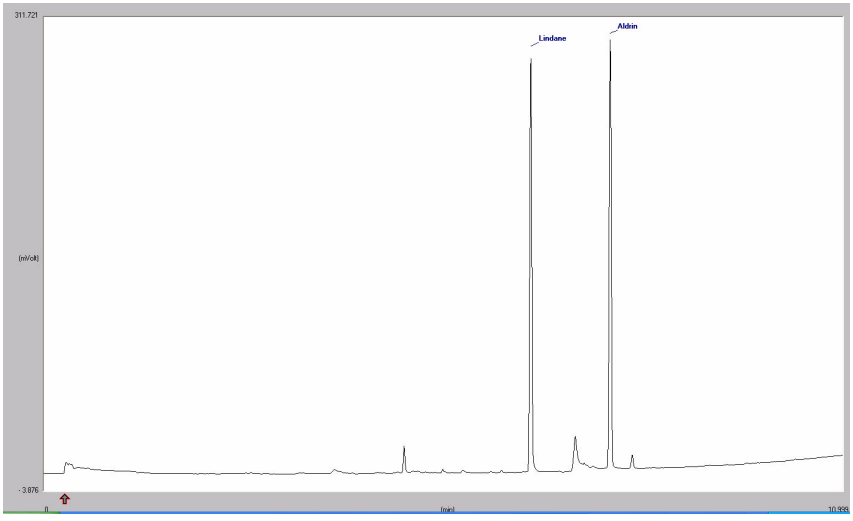
Analytical Results	
Dodecane Area	> 5 000 000
Tetradecane Area	> 10 000 000
Hexadecane Area	> 15 000 000

CAUTION **As default, the Signal Time in Chrom-Card WCC.INI is set to 0, then the acceptance values will result to be 10 times lower than the values reported in Table 7-5. To obtain the same values set Signal Time in Chrom-Card WCC.INI to 1.**

3. If these criteria are not met, repeat the test.

TriPlus SPME - S/SL - ECD Checkout

1. The resulting chromatogram should look like the one shown in Figure 7-2.



1. Lindane 2. Aldrin

Figure 7-2. TriPlus SPME - S/SL - ECD Injection

NOTE The difference of recovery, for the components analyzed, is due to different partitioning between the phase and the head space for the components at the temperature of equilibration.

2. The following criteria indicate successful completion of the checkout.

Table 7-6. TriPlus SPME - S/SL - ECD Acceptance Criteria

Analytical Results	
Lindane Signal-to-Noise Ratio	> 1 000
Aldrin Signal-to-Noise Ratio	> 1 000

3. If these criteria are not met, repeat the test.

8

TriPlus SPME Checkout Using PTV Injector

Chapter at a Glance...

Operating Procedures

SOP Number: TE P0584/02/E - 01 October 2008

Scope

Use the following procedure to verify proper TriPlus **SPME** Version operation combined with PTV injector and FID, ECD detectors.

Parts Referenced

Table 8-1. TriPlus SPME - PTV Parts Referenced

Part	Description	Part Number
Test Column	Fused Silica Capillary Column TR-5; 7 mt long 0.32 mm ID; 0.25 µm film thickness.	260 800 01
Glass Liner	Liner 1 mm ID for PTV	453 220 54
Liner Seal	Graphite seal for liner	290 334 06
Graphite Ferrule	Graphite ferrule for 0.32 mm ID Column	290 134 87
Retaining Nut	M4 capillary column retaining nut	350 324 23
Septum	Standard septum for PTV injector (set of 10)	313 132 25
Septum Cap	Septum Cap for SPME Injection	347 500 04
Syringe	10 µl size; 70 mm needle length	365 001 03
Test Mixture for FID	Three components: Dodecane, Tetradecane, Hexadecane, in n-Hexane:	338 190 20
Test Mixture for ECD	Two components: Lindane, Aldrin, in Iso-octane	338 190 11
Data Acquisition	Chrom-Card, ChromQuest, Xcalibur	
SPME	Kit SPME for TriPlus containing: Syringe holder, Fiber holder, 100 µm PDMS fiber	
Conditioning Station	Fiber conditioning station (optional)	
Vials, Seals and Caps	20 mL sample vial (requires P/N 386 036 00)	240 063 05
Septa and Caps	Aluminium caps, 20 mm Silicon-Teflon septa	386 094 10
Crimper	Vial hand crimper	

Analytical Conditions

Set the parameters listed in the following Tables 8-2 and 8-3.

TriPlus SPME

.

Table 8-2. TriPlus SPME - PTV Analytical Conditions

Parameters Setting	
General Parameters	Injector = S/SL Needle Speed In Vial = 20 mm/s Extraction Time = 10.0 min. Sampling Vial Depth = 25 mm Injection Depth = 35 mm Desorption Time = 1.0 min. Analysis Time = 0 minute
Incubation Parameters	Incubation Mode = Constant Agitator Temperature = 70 °C St-by Agitator Temperature = 70 °C Agitator ON Time = 600 s Incubation Time = 5 min. Agitator OFF Time = 0 s
Synch. Mode	GC Synch. Start Mode = When needle enters Inj.
Conditioning Station *(Fiber Conditioning Port if present or other injection port if available)*	Internal. Std Vial = None Conditioning Time = 8.0 min. Fiber Conditioning Port Temperature = 280 °C St-by Conditioning Port Temperature = 280 °C
SPME Fiber Holder Setup >> Fiber Zero Adjustment	Fiber Exposition = 21 mm

Gas Chromatograph

.

Table 8-3. Gas Chromatograph Analytical Condition

Parameters	FID	ECD
Gases		
Carrier Gas: Helium = 30 kPa Constant Pressure		
Hydrogen (mL/min.)	35	---
Air (mL/min.)	350	---
Make-up Gas (mL/min.)	30	30
Oven Program		
Initial Temperature (°C)	40	60
Initial Time (min.)	4	1
Ramp 1 (°C/min.)	40	20
Final Temperature (°C)	240	240
Final Time (min.)	0.1	1
PrepRun Time-out (min)	20	20
PTV Injector		
Operating Mode = *Constant Temperature Splitless* (CTSL)	CTSL	CTSL
Splitless Time (min.)	1	1
Split Flow (mL/min.)	80	80
Stop Septum Purge (min.)	1	1
Inject Temperature (°C)	280	280
Detector		
Base temperature (°C)	250	250
Detector Temperature (°C)	---	300
Detector Signal Range	10^0	---
Reference Current (nA)		1
Pulse Amplitude (V)		50
Pulse Width (µs)		1

Data System

Table 8-4. Data System

Parameters Setting	
Digital Signal Output	Acquisition Frequency = 10 Hz

Recommended Initial Operations

Before starting perform the SOP related to the GC configuration used as described in the relevant SOP manual.

1. Replace the glass liner.
   The glass liner currently installed in your injector should be carefully removed and replaced with the 1 mm ID liner for SPME application, as required for the checkout, with the appropriate liner seal.

2. Replace the septum
   A new septum should be installed properly in your injector.

3. Replace the Septum Cap
   The appropriate septum cap for TriPlus SPME injection should be installed on your injector.

4. Perform the leak test.

5. Install the TriPlus SPME version, as described in the relevant chapter of the TriPlus user manual and connect it to the data handling.

6. Turn the sampler on and let the automatic recognition of the objects.

7. Set up the PTV position as described in the relevant chapter of the TriPlus manual.

8. If the Fiber Conditioning Station is not present and another injector is present and can be used for the fiber bakeout, set also this injector.

9. Load the SOP method parameters for the TRACE GC and the TriPlus.

OPERATING PROCEDURE

TriPlus SPME - PTV - Detectors Checkout

Fiber Conditioning, Sample Preparation and Analysis

Before start, let the new fiber condition at the temperature and for the time specified by the supplier.
If the Fiber conditioning station is present, it can be used for this purpose, otherwise a dedicated injector can be used for the conditioning.

1. Perform an off-line blank run of the column.
2. Set-up the data system to perform the analysis of the sample vial.
3. Prepare the sample vial introducing 10 µL of test mixture into the 20 mL empty vial and closing it immediately with the related cap and septa.
4. Place the sample vial in a selectable position of the sampler tray.

CAUTION **To meet the acceptance criteria, the analysis must be performed immediately after the sample vial preparation.**

5. According to the detector in use, refer to the resulting chromatogram and the acceptance criteria indicating the successful completation of the checkout:
   - *TriPlus SPME - PTV - FID Checkout*
   - *TriPlus SPME - PTV - ECD Checkout*

TriPlus SPME - PTV - FID Checkout

1. The resulting chromatogram should look like the one shown in Figure 8-1.

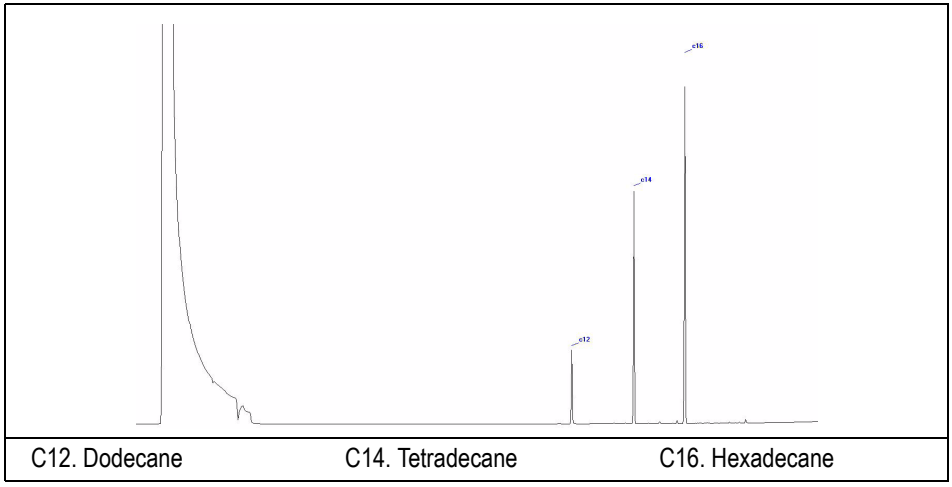


Figure 8-1. TriPlus SPME - PTV - FID Injection

NOTE The difference of recovery, for the components analyzed, is due to different partitioning between the phase and the head space for the components at the temperature of equilibration.

2. The following criteria indicate successful completion of the checkout.

Table 8-5. TriPlus SPME - PTV - FID Acceptance Criteria

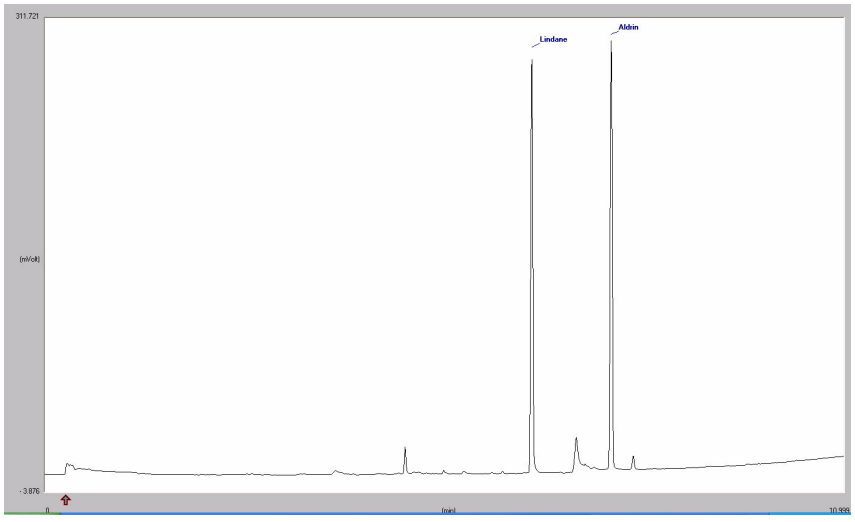
Analytical Results	
Dodecane Area	> 5 000 000
Tetradecane Area	> 10 000 000
Hexadecane Area	> 15 000 000

CAUTION **As default, the Signal Time in Chrom-Card WCC.INI is set to 0, then the acceptance values will result to be 10 times lower than the values reported in Table 8-5. To obtain the same values set Signal Time in Chrom-Card WCC.INI to 1.**

3. If these criteria are not met, repeat the test.

TriPlus SPME - PTV - ECD Checkout

1. The resulting chromatogram should look like the one shown in Figure 8-2.



1. Lindane 2. Aldrin

Figure 8-2. TriPlus SPME - PTV - ECD Injection

NOTE The difference of recovery, for the components analyzed, is due to different partitioning between the phase and the head space for the components at the temperature of equilibration.

2. The following criteria indicate successful completion of the checkout.

Table 8-6. TriPlus SPME - PTV - ECD Acceptance Criteria

Analytical Results	
Lindane Signal-to-Noise Ratio	> 1 000
Aldrin Signal-to-Noise Ratio	> 1 000

3. If these criteria are not met, repeat the test.

A

Customer Communication

Thermo Fisher Scientific provides comprehensive technical assistance worldwide and is dedicated to the quality of our customer relationships and services.

How To Contact Us

This appendix contains contact information for Thermo Fisher Scientific office. Use the list reported in *Customer Communication* to contact your local Thermo Fisher Scientific office or affiliate.

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This appendix also contains a one-page *Reader Survey*. Use this survey to give us feedback on this manual and help us improve the quality of our documentation

Reader Survey

Product: TriPlus
Manual: Standard Operating Procedures
Part No.: 317 110 19

Please help us improve the quality of our documentation by completing and returning this survey. Circle one number for each of the statements below.

	Strongly Agree	Agree	Neutral	Disagree	Strongly Disagree
The manual is well organized.	1	2	3	4	5
The manual is clearly written.	1	2	3	4	5
The manual contains all the information I need.	1	2	3	4	5
The instructions are easy to follow.	1	2	3	4	5
The instructions are complete.	1	2	3	4	5
The technical information is easy to understand.	1	2	3	4	5
Examples of operation are clear and useful.	1	2	3	4	5
The figures are helpful.	1	2	3	4	5
I was able to install the system using this manual.	1	2	3	4	5

If you would like to make additional comments, please do. (Attach additional sheets if necessary.)

_____

_____

_____

_____

Fax or mail this form to:
Thermo Fisher Scientific S.p.A.
Strada Rivoltana km 4
20090 Rodano (MI)
ITALY
Fax: 39 02 95059388

Glossary

This section contains an alphabetical list and descriptions of terms used in this guide and the help diskette. This also includes abbreviations, acronyms, metric prefixes, and symbols.

A

A	ampere
ac	alternating current
ADC	analog-to-digital converter

B

b	bit
B	byte (8 b)
baud rate	data transmission speed in events per second

C

ºC	Celsius
CIP	Carriage and Insurance Paid To
cm	centimeter
COC	Cold On-Column Injector
CPU	central processing unit (of a computer)
CSE	Customer Service Engineer
<Ctrl>	control key on the terminal keyboard

D

d	depth
DAC	digital-to-analog converter
dc	direct current
DS	data system

E

ECD	Electron Capture Detector
EMC	electromagnetic compatibility
ESD	electrostatic discharge

F

ºF	Fahrenheit
FID	Flame Ionization Detector
FOB	Free on Board
FPD	Flame Photometric Detector
ft	foot

G

g	gram
GC	gas chromatograph
GND	electrical ground

H

h	height
h	hour
harmonic distortion	A high-frequency disturbance that appears as distortion of the fundamental sine wave.
HV	high voltage
Hz	hertz (cycles per second)

I

IEC	International Electrotechnical Commission

impulse	See *transient*
in.	inch
I/O	input/output

K

k	kilo (10^3 or 1024)
K	Kelvin
kg	kilogram
kPa	kilopascal

L

l	length
l	liter
LAN	Local Area Network
lb	pound
LED	light-emitting diode
LVOCI	Large Volume On-Column Injector
LVSL	Large Volume Splitless

M

m	meter (or milli [10^{-3}])
M	mega (10^6)
µ	micro (10^{-6})
min	minute
mL	milliliter
mm	millimeter

m/z	mass-to-charge ratio

N

n	nano (10^{-9})
NPD	Nitrogen Phosphorous Detector

O

Ω	ohm

P

p	pico (10^{-12})
Pa	pascal
PCB	printed circuit board
PN	part number
psi	pounds per square inch

R

RAM	random access memory
<Return>	<Return> key on the keyboard
RF	radio frequency
ROM	read-only memory
RS-232	industry standard for serial communications

S

s	second
sag	See *surge*
slow average	A gradual, long-term change in average RMS voltage level, with typical durations greater than 2 s.

SOP	Standard Operating Procedure
SPME	Solid Phase Micro Extraction
surge	A sudden change in average RMS voltage level, with typical duration between 50 µs and 2 s.

T

TCD	Thermal Conductivity Detector
transient	A brief voltage surge of up to several thousand volts, with a duration of less than 50 µs.

U

V

V	volt
V ac	volts, alternating current
V dc	volts, direct current
VGA	Video Graphics Array

W

w	Width
W	Watt

NOTE The symbol for a compound unit that is a quotient (for example, degrees Celsius per minute or grams per liter) is written with a negative exponent with the denominator. For example:
°C min^{-1} instead of °C/min
g L^{-1} instead of g/L

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Operating Procedures

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