Thermo Scientific

TRACE GC Ultra

Gas Chromatograph

Standard Operating Procedures

PN 317 092 00, Revision May 2010_B

Home

Part of Thermo Fisher Scientific

Thermo SCIENTIFIC

TRACE™ GC Ultra - Standard Operating Procedures
May 2010 Edition
Part Number 31709200

Printed in Italy

Published by Thermo Fisher Scientific S.p.A., Strada Rivoltana, 20090 Rodano - Milan - Italy
Tel: +39 02 95059355 Fax: +39 02 95059388

Printing History: First Edition, released June 1998.
Second Edition, released November 1998.
Third Edition, released June 1999.
Fourth Edition, released May 2001
Fifth Edition, released January 2002
Sixth Edition, released, May 2003
Seventh Edition, released April 2004
Eighth Edition, released January 2005
Ninth Edition, released June 2005
Tenth Edition, released December 2005
Eleventh Edition, released June 2006
Twelfth Edition, released January 2007
Thirteenth Edition, released May 2007
Fourteenth Edition, released February 2008
Fifteenth Edition, released April 2009
Sixteenth Edition, released September 2009
Seventeenth Edition, released May 2010_B

Disclaimer
Technical Information contained in this publication is for reference purposes only and is subject to change without notice. Every effort has been made to supply complete and accurate information; however, Thermo Fisher Scientific assumes no responsibility and will not be liable for any errors, omissions, damage, or loss that might result from any use of this manual or the information contained therein (even if this information is properly followed and problems still arise).

This publication is not part of the Agreement of Sale between Thermo Fisher Scientific and the purchaser of a TRACE™ GC Ultra system. In the event of any conflict between the provisions of this document and those contained in Thermo Fisher Scientific's Terms and Conditions, the provisions of the Terms and Conditions shall govern.

Reference to System Configurations and Specifications supercede all previous information and are subject to change without notice.

Trademarks

Contents

SECTION VI
SOPs Using PID

SECTION VII
SOPs Using TCD

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**, *SOPs Using FID*, contains the procedure to test the TRACE GC Ultra with the Flame Ionization Detector (FID) using different injectors.

Section **III**, *SOPs Using ECD,* contains the procedure to test the TRACE GC Ultra with the Electron Capture Detector (ECD) using different injectors.

Section **IV**, *SOPs Using NPD,* contains the procedure to test the TRACE GC Ultra with the Nitrogen Phosphorus Detector (NPD) using different injectors.

Section **V**, *SOPs Using FPD*,contains the procedure to test the TRACE GC Ultra with the Flame Photometric Detector (FPD) using different injectors.

Section **VI**, *SOPs Using PID*,contains the procedure to test the TRACE GC Ultra with the Photoionization Detector (PID) using different injectors.

Section **VII**, *SOPs Using TCD*, contains the procedure to test the TRACE GC Ultra with the Thermal Conductivity Detector (TCD) using different injectors

Section **VIII**, *SOPs Using PDD*, contains the procedure to test the TRACE GC Ultra with the Pulsed Discharge Detector (PDD) using different injectors.

Section **IX**, *SOPs Using FID-NPD-FPD in Stacked Configuration*, contains the procedures to test the TRACE GC Ultra with the Flame Ionization Detector (FID), Nitrogen-Phosphorus Detector (NPD) or Flame Photometric Detector (FPD) in series (stacked configuration) with the Electron Capture Detector ECD using different injectors.

Section **X**, *SOPs for Large Volume Applications*, contains the procedures to test the TRACE GC Ultra for large volume application by using different injectors.

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 *Glossay* 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 symbols and typographical conventions are used throughout 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 danger from high temperature surfaces or substances.
	This symbol indicates an explosion hazard.
	This symbol indicates the presence of radioactive material.

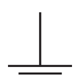
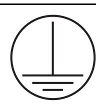
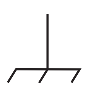
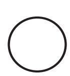
Abbreviations for Injectors and Detectors

	Abbreviation
Split/splitless Injector	S/SL
Large Volume Splitless	LVSL
Cold On-Column Injector	OCI
Packed Column Injector	PKD
Packed Column with Septum Purge	PPKD
Programmable Temperature Vaporizing Injector	PTV

Detector	Abbreviation
Flame Ionization Detector	FID
Electron Capture Detector	ECD
Nitrogen-Phosphorus Detector	NPD
Flame Photometric Detector	FPD
Photoionization Detector	PID
Thermal Conductivity Detector	TCD
Pulsed Discharge Detector	PDD

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 TRACE GC Ultra gas chromatograph.

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
	Equipotentiality
	On (Supply)
	Off (Supply)

Symbol	Description
	Equipment protected throughout by DOUBLE INSULATION or REINFORCED INSULATION (Equivalent to Class II of IEC 536)
	Caution, risk of electric shock
	Caution, hot surface
	Caution (refer to accompanying documents)
	In-position of a bistable push control
	Out-position of a bistable push control
	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.

Safety Information

Precaution for Gases

Before using gases, carefully read the indications of hazard and the warning reported in the Safety sheet supplied by the manufacturer with reference to the relevant CAS number (Chemical Abstract Service).

WARNING! **Hydrogen is a harmful gas that mixed with air may give rise to an explosion hazard. The use of hydrogen requires the operator's extreme caution and the recourse to special precautions due to the risk involved.**

Precaution for the Electron Capture Detector

WARNING! **The Electron Capture Detector (ECD) contains a 63Ni beta-emitting radioactive source of 370 MBq (10 mCi). For no reason should the detector be opened or handled by the operator. Any maintenance or service operations involving even partial disassembling of the instrument must be performed ONLY by qualified personnel at the laboratory expressly authorized by Thermo Fisher Scientific and specially licensed to handle radioactive material.**

Using the TRACE GC Ultra Document Set

The TRACE GC Ultra Document Set (CD-Rom PN 317 095 00) includes all manuals in electronic format, and serves as your library for information about the TRACE GC Ultra hardware and software.
The TRACE GC Ultra Document Set (PN 317 093 00) as paper copy is also available Furthermore, Thermo Fisher Scientific part numbers (PN) for the paper copy manuals are provided for each book title.

Site Preparation and Installation Manual (PN 317 091 90)
This manual and diskette describes how to set up a workspace for the TRACE GC Ultra and how to connect the TRACE GC Ultra to the gas supplies and peripheral devices.

Acceptance Package (PN 317 092 20)
This folder contains required shipping documents and quality report forms.

Getting Started (PN 317 092 30)
This guide contains sequences for checking configuration, installing detectors, and making a first analysis with the TRACE GC Ultra.

Operating Manual (PN 317 091 70)
This manual provides descriptions of the TRACE GC Ultra hardware and software and instructions for their use.

UFM Ultra Fast Module Device (PN 317 093 98)
This manual provides descriptions of the TRACE GC Ultra equipped with the UFM device. and instructions for its use. The relevant *Standard Operating Procedure* is provided in a separated document PN 317 094 09.

Quick Reference Card (PN 317 092 40)
This reference card contains guidelines for carrier gas use and injection sequences.

K-Factor Quick Reference (P/N 317 092 41)
This card indicates the theoretical K-Factors related to the carrier gas and the column in use.

Preventive Maintenance Schedule (PN 317 092 80)
This document provides a list of recommended scheduled maintenance and a year-long log book to record maintenance, observations, supply lists, and service records.

Maintenance and Troubleshooting Manual (PN 317 091 80)
This manual contains instructions for diagnosing and resolving operational problems.

Standard Operating Procedures (PN 317 092 00)
This manual contains instructions, operating sequences, and test criteria for final testing of the TRACE GC Ultra.

Spare Parts Catalog (PN 317 092 10)
This catalog contains a list of spare parts for the TRACE GC Ultra.

SECTION I

SOPs Overview

The *SOPs Overview* 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...

Operating Procedures

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 TRACE GC Ultra. 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 TRACE GC Ultra 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. P0292/01/E - 12 June 1998), according to our Quality Management policy.

If your GC is equipped with the Ultra Fast Module, please refer to the relevant SOP (PN 317 094 09).

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

- TRACE GC Ultra Operating Manual PN 317 091 70
- TRACE GC Ultra Maintenance and Troubleshooting Manual PN 317 091 80
- TRACE GC Ultra Site Preparation and Installation Manual PN 317 091 90
- TRACE GC Ultra Getting Started Manual PN 317 092 30

Parts Referenced

The SOPs require the following parts:

Table 1-1. SOPs Parts Referenced

	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 for 0.32 mm ID column	290 134 87
Syringes	10 µl size; 70 mm needle length for S/SL injections	365 001 03
	10 µl size; 75 mm needle length for OC injections	365 020 07
	10 µl size; 50 mm needle length for PTV, PKD and PPKD injections	365 005 25
Test Mixtures	Test Mixture for FID checkout	338 190 20
	Test Mixture for ECD checkout	338 190 11
	Test Mixture for NPD checkout	338 190 06
	Test Mixture for FPD checkout	338 190 06
	Test Mixture for PID checkout	338 190 06
	Test Mixture for TCD checkout	338 190 16
	Test Mixture for PDD checkout	338 190 32
Gases	Gas Chromatographic-grade purity *Carrier Gas* = Helium *Fuel Gases* = Hydrogen - Air *Make-up Gas* = Nitrogen *Discharge gas for PDD* = Helium ultrapure (At least 99.999% of purity)	
Data Acquisition	Chrom-Card, ChromQuest, Atlas, Xcalibur, Computing-integrator	

Getting Started

Before starting checkout, perform the following preliminary operations sequentially:

1. **Gas Supply Connections**
   Connect the gas supplies following the instructions reported in Chapter 4 of the TRACE GC Ultra Site Preparation and Installation Manual

2. **Data Handling Connections**
   Connect your data handling following the instructions reported in Chapter 5 of the TRACE GC Ultra Site Preparation and Installation Manual

3. **Column Installation**
   Install the test column according to the injector installed in your GC following the **Operating Sequences** reported in Chapter 14 "*Columns*" of the TRACE GC *Ultra Operating Manual*.

   - *Preparing a Capillary Column*
   - *Connecting a Capillary Column to a Split/Splitless Injecto*r
   - *Connecting a Capillary Column to an Cold On-Column Injector*
   - *Connecting a Capillary Column to an Packed Column Injector*
   - *Connecting a Capillary Column to an Purged Packed Injector*
   - *Connect a Capillary Column to an Programmable Temperature Vaporizing Injector*

4. **Glass Liner and Septum Installation**
   Install the glass liner following the **Operating Sequences** "*Install a Liner and Septum*" reported in the TRACE GC Ultra Operating Manual.

   - Chapter 5: *Split/Splitless Injector*
   - Chapter 9: *Packed Column Injector*

- Chapter 10: *Purged Packed Injector*
- Chapter 11: *Programmable Temperature Vaporizing Injector*

5. **Column Leak Test**
   Perform the column leak check following the **Operating Sequence** "*Performing a Leak Check*" reported in Chapter 14 "*Columns*" of the TRACE GC Ultra Operating Manual.

6. **Column Evaluation**
   Set column length, nominal ID and film thickness to calculate the column K factor. It is also possible to manually set the carrier gas flow measured at the end of the column to obtain the effective K factor.

   Perform column evaluation following the **Operating Sequence** "*Performing a Column Evaluation*" reported in Chapter 14 "*Columns*" of the TRACE GC Ultra Operating Manual.

7. **Column Conditioning**

CAUTION **When performing column conditioning, the column should be connected only to the injector leaving the column outlet disconnected to avoid the possibility of contamination of the detector base body.**

Column conditioning consists of passing a flow of carrier gas through the column and heating it to a temperature of 20-50 °C above the maximum temperature that will be used for running the analysis. If the Cold On-Column Injector is used, ensure that the injection valve is closed. Refer to Table 1-2 for the parameter setting and to the **Operating Procedure** "*Test Column Conditioning*" on page 31, to perform the operation.

8. **Detector Connections**
   This operation should be carried out at the end of the column conditioning. Connect the test column to the detector following the **Operating Sequences** reported in Chapter 14 "*Columns*" of the TRACE GC Ultra Operating Manual.

- *Connecting a Capillary Column to an FID, FPD or NPD Detector*
- *Connecting a Capillary Column to an ECD Detector*

- *Connect a Capillary Column to a PID Detector*
- *Connecting a Capillary Column to a TCD Detector*
- *Connecting a Capillary Column to a PDD Detector*

OPERATING PROCEDURE

Test Column Conditioning

Refer to Table 1-2 for the parameters setting:

Table 1-2. Column Conditioning Parameters

Gases	Carrier Gas: Helium = 30 kPa Constant Pressure
Oven Program	Initial Temperature = 50 °C Initial Time = 1 min. Ramp 1 = 20 °C/min. Final Temperature = 250 °C Final Time = 30 min.
Injector	Temperature = according to the injector in use

Before beginning, press **CONFIG** to verify the GC configuration:

Left inlet or Right inlet	S/SL, OCI, PKD, PPKD or PTV
Left carrier or Right carrier	He (helium)

1. Use **LEFT CARRIER** or **RIGHT CARRIER** to display the appropriate Carrier Gas Control Table. Verify to operate in constant pressure mode. If not, scroll to `Pressure mode`, press **MODE/TYPE** to access the selection menu, then select `Constant pressure`. Scroll to `Pressure` and set the pressure value to have the required carrier gas flow rate `Col.flow`.

```
         LEFT CARRIER[1]
Pressure          30.0    30.0
Col.flow          3.00
Lin. veloc.              (60.9)<
```

1. These settings could also be for a right carrier.

2. Using **OVEN**, entry the Column Oven Control Table and set the oven temperature and the Oven Program required.

```
               OVEN

Temp                 50.0   50.0
Initial Time                1.00
Ramp 1                      20.0
Final temp                   250
Final time 1                30.0<
Ramp 2                       Off
```

3. Using **LEFT INLET** or **RIGHT INLET**, entry the appropriate Injector Control Table and set the required temperature setpoint `Temp`. In the case of Cold On-Column Injector, this step is not required.

```
        LEFT INLET (S/SL)[1]

Temp                  230    230<
Pressure             30.0   30.0
Mode:                  Splitless
```

1. These settings could also be for a right inlet.

or

```
        LEFT INLET (OCI)[1]

Pressure             30.0   30.0<
Sec. Cool time               Off
```

1. These settings could also be for a right inlet.

or

```
        LEFT INLET (PKD)[1]

Temp                  200    200
Pressure             30.0   30.0
```

1. These settings could also be for a right inlet.

or

```
     LEFT INLET (PPKD)¹
Temp                200   200
Pressure           30.0  30.0
Mode:               Wide bore<
Constant sept purge?       Y<
```

1. These settings could also be for a right inlet.

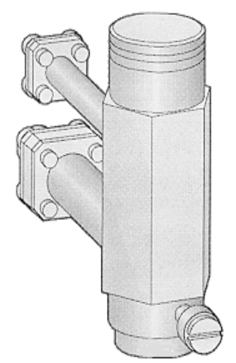
or

```
     RIGHT INLET (PTV)
Temp                  70    70
Pressure            30.0  30.0
Mode:            PTV Splitless
```

Press **PREP RUN** then **START** on the GC to begin the column conditioning.

SECTION II

SOPs Using FID



The *SOPs Using FID* section, contains the procedures to test the TRACE GC Ultra with the Flame Ionization Detector (FID) using different injectors.

2

Checkout Using FID with S/SL Injector

Chapter at a Glance...

Operating Procedures

SOP Number: P0292/07/E - 01 September 2009

Scope

Use the following procedure to verify proper FID operation with the Split/Splitless Injector.

Parts Referenced

Table 2-1. FID-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	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; 70 mm needle length	365 001 03
Test Mixture	Three components in n-Hexane: *Component* *Concentration* Dodecane 20 µg/ml Tetradecane 20 µg/ml Hexadecane 20 µg/ml	338 190 20
Gases	Chromatographic-grade purity	
Data Acquisition	Chrom-Card, ChromQuest, Atlas, Xcalibur, Computing-integrator	

Analytical Conditions Required for Splitless Injection

Table 2-2. FID-S/SL Analytical Conditions

Parameters	Setting
Gases	Carrier Gas: Helium = 30 kPa Constant Pressure Hydrogen = 35 ml/min Air = 350 ml/min Make-up Gas: Nitrogen = 30 ml/min
Oven Program	Initial Temperature = 50 °C Initial Time = 1 minute Ramp 1 = 20 °C/minute Final Temperature = 200 °C Final Time = 1 minute
Injector	Operating Mode = Splitless Temperature = 230 °C Splitless Time = 0.8 minutes Split Flow = 60 ml/min Constant Septum Purge = Yes
Detector	Base Temperature = 250 °C Detector Signal Range = 10^0
Injected Volume	1 µl + needle of Test Mixture
Analog Signal Output	Chrom-Card Acquisition Frequency = Medium
Digital Signal Output	Chrom-Card, ChromQuest, Atlas, Xcalibur Acquisition Frequency = 10 Hz

Recommended Initial Operations

Before starting the checkout, the following operations should be carried out:

1. Replace the glass liner.
   The glass liner currently installed in your injector should be carefully removed and replaced with the 3 mm ID glass liner for splitless 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. Connect the required gas lines
   Verify the required gas supplies are properly connected to your GC.

4. Install the test column.
   The column currently installed should be carefully removed and replaced with the required test column.

5. Perform Column Evaluation and Leak Test.

6. Connect your data handling.
   Verify that your data handling is properly connected to your GC system.

OPERATING PROCEDURE

FID-S/SL Checkout in Splitless Mode

Before beginning, press **CONFIG** to verify the GC configuration:

Left inlet or Right inlet	S/SL
Left carrier or Right carrier	He (helium)
Left detector or Right detector	FID

1. Use **LEFT CARRIER** or **RIGHT CARRIER** to display the appropriate Carrier Gas Control Table. Verify to operate in constant pressure mode. If not, scroll to `Flow mode`, press **MODE/TYPE** to access the selection menu, then select `con pres`. Scrool to `Pressure` and set the pressure value to have the required carrier gas flow rate `Col.flow`.

```
        LEFT CARRIER[1]

Pressure          30.0   30.0
Col.flow          3.00
Lin. veloc.             (60.9)<
```

1. These settings could also be for a right carrier.

2. Use **OVEN** to display the Column Oven Control Table. Set the oven temperature and the Oven Program required.

```
             OVEN

Temp              50.0   50.0
Initial Time             1.00
Ramp 1                   20.0
Final temp                200
Final time 1             1.00<
Ramp 2                    Off
```

3. Use **LEFT INLET** or **RIGHT INLET** to display the appropriate Split/Splitless Injector Control Table. Set the required temperature *Temp* setpoint. Verify to

operate in **Splitless** mode. If not, scroll to `Mode`, press **MODE/TYPE** to access the selection menu, then select `Splitless`. Scroll to `Splitless time` to set the required setpoint.

LEFT INLET (S/SL)[1]		
Temp	230	230
Pressure	30.0	30.0
Mode:		Splitless
Total flow		(63.0)
Split Flow	60.0	60.0
Splitless time	0.80	0.80
Constant sept purge?		Y<

1. These settings could also be for a right inlet.

4. Use **LEFT DETECTOR** or **RIGHT DETECTOR** to display the appropriate FID Detector Control Table. Set the required temperature `Base Temp` and the detector gases `H2`, `Air` and `Mkup` required setpoints.

LEFT DETECTOR (FID)[1]		
Flame		Off
Base temp	250	250
Signal pA		(5.5)
Ign.thresh		2.0
Flameout retry		Off
H2	35	35
Air	350	350
Mkup N2	30	30<

1. These settings could also be for a right detector.

5. Ignite the FID flame scrolling to `Flame` and pressing **ON**.

6. Use **LEFT SIGNAL** or **RIGHT SIGNAL** to display the appropriate FID Detector Signal Control Table. Observe the FID flame signal at the display. This is the flame-on background offset. Scroll to `Range` and set the electrometer amplifier input range required.

LEFT SIGNAL (FID)[1]	
Output	(1000)
Offset	100
Auto zero?	Y/N
Range 10^(0..3)	0<
Analog filter	Off
Baseline Comp	Off

1. These settings could also be for a right signal.

7. Activate your Data System and set the parameters required for the checkout.

8. In the FID Detector Signal Control Table, scroll to `Auto zero?` and turn it **YES**.

9. Perform a blank analysis injecting pure hexane and press **START** on the GC to begin the checkout run.

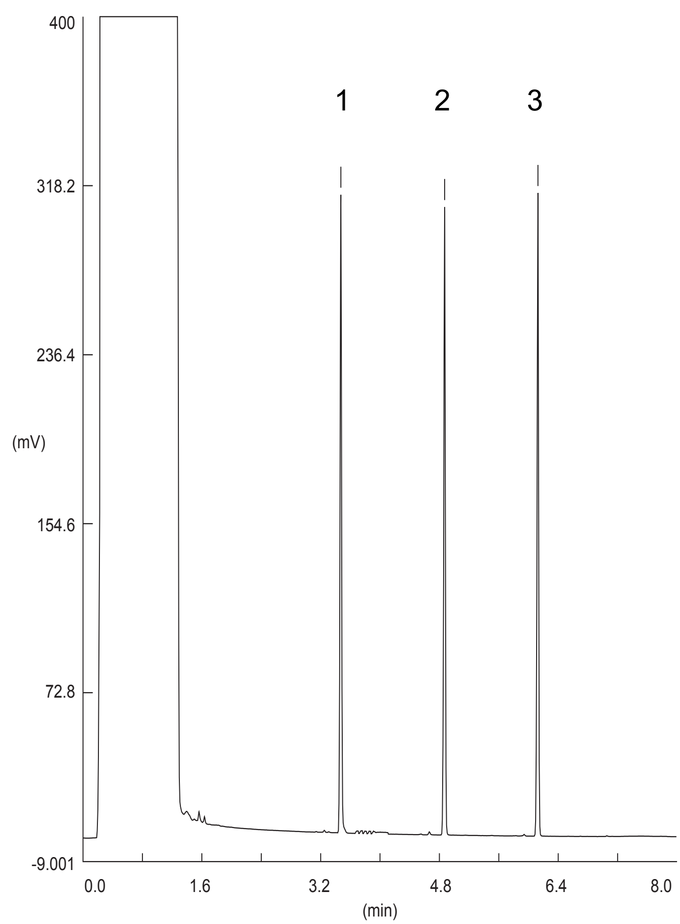
Baseline Acquisition and Analysis

NOTE Refer to the Acceptance Values reported in the Table 2-3 according to the data handling in use.

10. With the GC in Stand-by/Prep Run condition, activate the data system for 10 minutes to evaluate your baseline in isothermal condition.

11. After the baseline evaluation has been completed, set-up the data system to acquire a single run.

12. Inject the test mixture and press **START** on the GC to begin the checkout run. The resulting chromatogram should look like the one shown in *Figure 2.1*.



1. Dodecane 2. Tetradecane 3. Hexadecane

Figure 2-1. FID-Splitless Injection

13. The following acceptance criteria indicate successful completion of FID-S/SL checkout according to the data handling in use.

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

Table 2-3. FID-S/SL Acceptance Criteria

Acceptance Values	CHROM-CARD		
	Baseline Parameters	**Analog** *(1V Full Scale)*	**Digital** *(10V Full Scale)*
	Noise (µV)	< 30	< 300
	Wander (µV)	< 50	< 500
	Drift (µV/h)	< 100	< 1 000
	Analytical Results	**Analog** *(1V Full Scale)* *Area Counts (0.1 µVs)*	**Digital** *(10V Full Scale)* *Area Counts (0.1 µVs)*
	Components	> 4 000 000 for each component	> 40 000 000 for each component
	Area Count Ratio Calculated as C12/C16	1 ± 0.1	1 ± 0.1
⚠			

Computing-integrator (e.g. ChromJet)	
⚠	

Acceptance Values	CHROMQUEST	
	Baseline Parameters *(1V Full Scale)*	
	Noise (µV)	< 30
	Wander (µV)	< 50
	Drift (µV/h)	< 100
	Analytical Results *(1V Full Scale) - Area Counts (0.01 µVs)*	
	Components	> 40 000 000 for each component
	Area Count Ratio Calculated as C12/C16	1 ± 0.1
⚠		

ATLAS	
Baseline Parameters *(10V Full Scale)*	
Noise (µV)	< 300
Wander (µV)	< 500
Drift (µV/h)	< 1 000
Analytical Results *(10V Full Scale) - Area Counts (µVs)*	
Components	> 4 000 000 for each component
Area Count Ratio Calculated as C12/C16	1 ± 0.1

Acceptance Values	XCALIBUR	
	Baseline Parameters *(Acquisition Frequency = 10 Hz)*	
	Noise (Counts)	< 3 000
	Wander Counts)	< 5 000
	Drift (Counts/h)	< 10 000
	Analytical Results *Area Counts (Cts*s)*	
	Components	> 40 000 000 for each component
	Area Count Ratio Calculated as C12/C16	1 ± 0.1

Analytical Acceptance Comments	
1	When the make-up gas is not used, the acceptance values will result to be 2.5 times lower than the values reported in Table 2-3.
2	When helium is used as make-up gas, the acceptance values will result to be 10 times lower than the values reported in Table 2-3.

3

Checkout Using FID with OC Injector

Chapter at a Glance...

Operating Procedures

SOP Number: P0293/08/E - 01 September 2009

Scope

Use the following procedure to verify proper FID operation with the Cold On-Column Injector.

Parts Referenced

Table 3-1. FID-OCI 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
Vespel Ferrule	Vespel Ferrule for 0.32 mm ID Column	290 134 60
Retaining Nut	M4 capillary column retaining nut	350 324 23
Syringe	10 µl size; 75 mm needle length	365 020 07
Test Mixture	Three components in n-Hexane: *Component* *Concentration* Dodecane 20 µg/ml Tetradecane 20 µg/ml Hexadecane 20 µg/ml	338 190 20
Gases	Chromatographic-grade purity	
Data Acquisition	Chrom-Card, ChromQuest, Atlas, Xcalibur, Computing-integrator	
Syringe	10 µl size; 80 mm needle length	365 020 19
Pre-column	2 m long; 0.53 mm ID	260 603 75
Press-fit set	Set of Press-fir connectors for TRACE OC	350 038 45

Analytical Conditions Required for On-Column Injection

Table 3-2. FID-OCI Analytical Conditions

Parameters Setting	
Gases	Carrier Gas: Helium = 30 kPa Constant Pressure Hydrogen = 35 ml/min Air = 350 ml/min Make-up Gas: Nitrogen = 30 ml/min
Oven Program	Initial Temperature = 70 °C Initial Time = 1 minute Ramp 1 = 20 °C/minute Final Temperature = 200 °C Final Time = 1 minute
Injector	Secondary Cooling = 0.2 minutes
Detector	Base Temperature = 250 °C Detector Signal Range = 10^0
Injected Volume	1 µl of Test Mixture
Analog Signal Output	Chrom-Card Acquisition Frequency = Medium
Digital Signal Output	Chrom-Card, ChromQuest, Atlas, Xcalibur Acquisition Frequency = 10 Hz

Recommended Initial Operations

Before starting the checkout, the following operations should be carried out:

1. Connect the required gas lines
   Verify the required gas supplies are properly connected to your GC.

2. Install the test column.
   The column currently installed should be carefully removed and replaced with the required test column.
   In case of automatic On-column for TriPlus sampler, install the pre-column and connect it to the test column by press-fit connector.

3. Install and connect the TriPlus sampler and its components.

4. Perform Column Evaluation and Leak Test.

5. Connect your data handling.
   Verify that your data handling is properly connected to your GC system.

6. Verify the opening/closing of the OC injector actuator by using the proper commands.

7. Verify the alignment of the syringe on the OC injector.

OPERATING PROCEDURE

FID-OCI Checkout in On-Column Mode

Before beginning, press **CONFIG** to verify the GC configuration:

Left inlet or Right inlet	OCI
Left carrier or Right carrier	He (helium)
Left detector or Right detector	FID

1. Use **LEFT CARRIER** or **RIGHT CARRIER** to display the appropriate Carrier Gas Control Table. Verify to operate in constant pressure mode. If not, scroll to `Flow mode`, press **MODE/TYPE** to access the selection menu, then select `con pres`. Scrool to `Pressure` and set the pressure value to have the required carrier gas flow rate `Col.flow`.

```
         LEFT CARRIER[1]
Pressure         30.0   30.0
Col.flow         3.00
Lin. veloc.           (60.9)<
```

1. These settings could also be for a right carrier

2. Use **OVEN** to display the Column Oven Control Table. Set the oven temperature *Temp* and the Oven Program required.

```
              OVEN
Temp             70.0   70.0
Initial Time            1.00
Ramp 1                  20.0
Final temp               200
Final time 1            1.00<
Ramp 2                   Off
```

3. Use **LEFT INLET** or **RIGHT INLET** to display the appropriate Cold On-Column Injector Control Table. Scroll to `Sec. cool time` and set the required secondary cooling time.

LEFT INLET (OCI)[1]		
Pressure	30.0	30.0
Sec. Cool Time		0.2<

1. These settings could also be for a right inlet.

4. Use **LEFT DETECTOR** or **RIGHT DETECTOR** to display the appropriate FID Detector Control Table. Set the required temperature `Base Temp` and the detector gases `H2`, `Air` and `Mkup` (if available) required setpoints.

LEFT DETECTOR (FID)[1]		
Flame		Off
Base temp	250	250
Signal pA		(5.5)
Ign. thresh		2.0
Flameout retry		Off
H2	35	35
Air	350	350
Mkup N2	30	30<

1. These settings could also be for a right detector.

5. Ignite the FID flame scrolling to `Flame` and pressing **ON**.

6. Use **LEFT SIGNAL** or **RIGHT SIGNAL** to display the appropriate FID Detector Signal Control Table. Observe the FID flame signal at the display. This is the flame-on background offset. Scroll to `Range` and set the electrometer amplifier input range required.

LEFT SIGNAL (FID)[1]	
Output	(1000)
Offset	100
Auto zero?	Y/N
Range 10^(0..3)	0<
Analog filter	Off
Baseline comp	Off

1. These settings could also be for a right signal.

7. Activate your Data System and set the parameters required for the checkout.

8. In the FID Detector Signal Control Table scroll to `Auto zero?` and turn it **YES**.

9. Perform a blank analysis injecting pure hexane and press **START** on the GC to begin the checkout run.

Baseline Acquisition and Analysis

NOTE Refer to the Acceptance Values reported in the Table 3-3 according to the data handling in use.

10. With the GC in Stand-by/Prep Run condition, activate the data system for 10 minutes to evaluate your baseline in isothermal condition.

11. After the baseline evaluation has been completed, set-up the data system to acquire a single run.

12. Perform the analysis

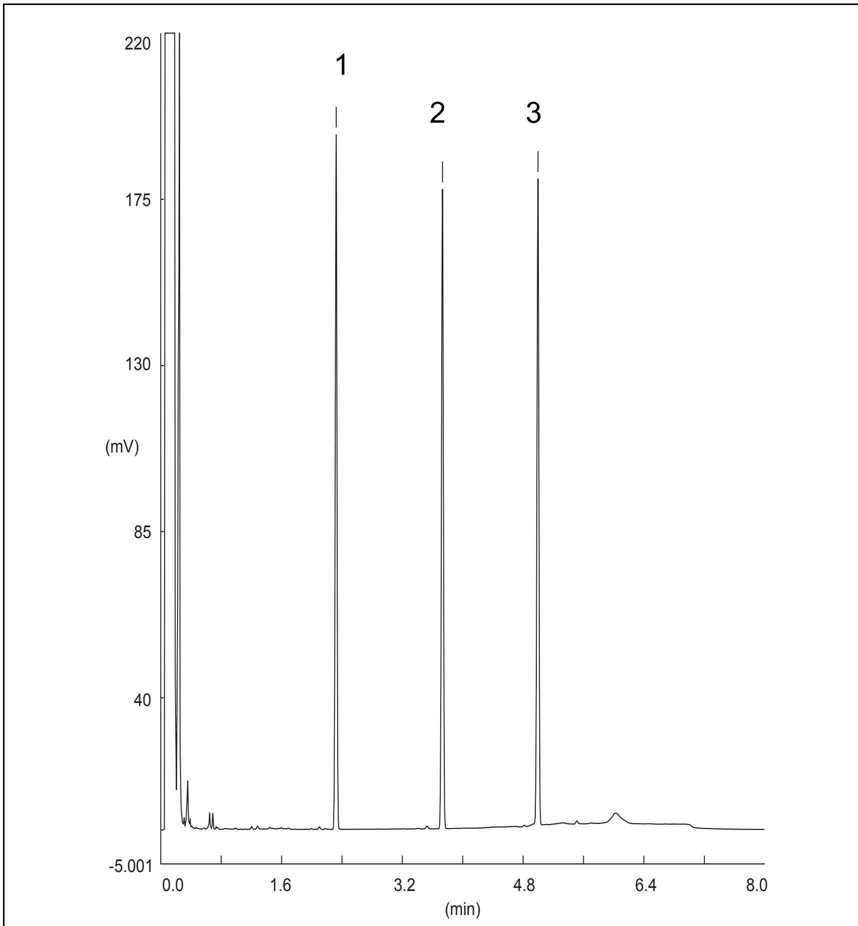
    Manual injection

    - Inject the test mixture and press **START** on the GC to begin the checkout .

    Automatic injection with TriPlus sampler

    - Fill a vial with the standard mix and place that vial in the sample tray.
    - Load the method for OC and perform the sampling.

The resulting chromatogram should look like the one shown in *Figure 3.1*.



1. Dodecane	2. Tetradecane	3. Hexadecane

Figure 3-1. FID-On-Column Injection

13. The following criteria indicate successful completion of FID-OCI checkout.

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

Table 3-3. FID-OCI Acceptance Criteria

Acceptance Values

CHROM-CARD		
Baseline Parameters	**Analog** *(1V Full Scale)*	**Digital** *(10V Full Scale)*
Noise (μV)	< 30	< 300
Wander (μV)	< 50	< 500
Drift (μV/h)	< 100	< 1 000
Analytical Results	**Analog** *(1V Full Scale)* *Area Counts (0.1 μVs)*	**Digital** *(10V Full Scale)* *Area Counts (0.1 μVs)*
Components	> 2 500 000 for each component	> 25 000 000 for each component
Area Count Ratio Calculated as C12/C16	1 ± 0.1	1 ± 0.1

Computing-integrator (e.g. ChromJet)

Acceptance Values

CHROMQUEST	
Baseline Parameters *(1V Full Scale)*	
Noise (μV)	< 30
Wander (μV)	< 50
Drift (μV/h)	< 100
Analytical Results *(1V Full Scale) - Area Counts (0.01 μVs)*	
Components	> 25 000 000 for each component
Area Count Ratio Calculated as C12/C16	1 ± 0.1

Acceptance Values	ATLAS	
	Baseline Parameters *(10V Full Scale)*	
	Noise (µV)	< 300
	Wander (µV)	< 500
	Drift (µV/h)	< 1 000
	Analytical Results *(10V Full Scale) - Area Counts (µVs)*	
	Components	> 2 500 000 for each component
	Area Count Ratio Calculated as C12/C16	1 ± 0.1
⚠		

Acceptance Values	XCALIBUR	
	Baseline Parameters *(Acquisition Frequency = 10 Hz)*	
	Noise (Counts)	< 3 000
	Wander Counts)	< 5 000
	Drift (Counts/h)	< 10 000
	Analytical Results *Area Counts (Cts*s)*	
	Components	> 25 000 000 for each component
	Area Count Ratio Calculated as C12/C16	1 ± 0.1
⚠		

Analytical Acceptance Comments	
1	When the make-up gas is not used, the acceptance values will result to be 2.5 times lower than the values reported in Table 3-3.
2	When helium is used as make-up gas, the acceptance values will result to be 10 times lower than the values reported in Table 3-3.

4

Checkout Using FID with PKD Injector

Chapter at a Glance...

Operating Procedures

SOP Number: P0307/07/E - 01 September 2009

Scope

Use the following procedure to verify proper FID operation with Packed Injector.

Parts Referenced

Table 4-1. FID-PKD 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	2 mm ID tapered	453 220 50
Liner Seal	Graphite seal for glass liner	290 334 05
Pre-column	Fused Silica Capillary Column 0.5 mt long 0.53 mm ID	260 603 75
Retaining Nut	M4 capillary column retaining nut	350 324 23
Press-fit connections	For columns 0.53/0.32 mm ID	350 438 16
Injection side adapter	For Wide bore column	347 003 03
Retaining Nut	For Injection side adapter	350 024 04
Detector side adapter	For Wide bore column	347 103 04
Ferrule	6 mm ID double brass ferrule	290 341 37
Nut	1/4” G-6 mm ID nut	350 201 18
Graphite Ferrule	Graphite ferrule for 0.53 mm ID Column Graphite ferrule for 0.32 mm ID Column	290 134 86 290 134 87
Septum	Standard septum for Packed Injector	313 032 26
Syringe	10 µl size; 50 mm needle length	365 005 25

Table 4-1. FID-PKD Parts Referenced (Continued)

Part	Description	Part Number
Test Mixture	Three components in n-Hexane: *Component* *Concentration* Dodecane 20 µg/ml Tetradecane 20 µg/ml Hexadecane 20 µg/ml	338 190 20
Gases	Chromatographic-grade purity	
Data Acquisition	Chrom-Card, ChromQuest, Atlas, Xcalibur, Computing-integrator	

Analytical Conditions Required for Packed Injector

Table 4-2. FID-PKD Analytical Conditions

Parameters	Setting
Gases	Carrier Gas: Helium = 30 kPa Constant Pressure Hydrogen = 35 ml/min Air = 350 ml/min Make-up Gas: Nitrogen = 30 ml/min
Oven Program	Iso Temperature = 50 °C Initial Time = 1 minute Ramp 1 = 20 °C/minute Final Temperature = 200 °C Final Time = 1 minute
Injector	Operating Mode = Packed Temperature = 200 °C
Detector	Base Temperature = 250 °C Detector Signal Range = 10^0
Injected Volume	1 µl + needle of Test Mixture
Analog Signal Output	Chrom-Card Acquisition Frequency = Medium
Digital Signal Output	Chrom-Card, ChromQuest, Atlas, Xcalibur Acquisition Frequency = 10 Hz

Recommended Initial Operations

Before starting the checkout, the following operations should be carried out:

1. Insert the glass liner.
   Remove the column and insert the 2 mm ID tapered glass liner, as required for the checkout, from the bottom of the injector with the appropriate liner seal. Fix the liner by using the appropriate adapter for capillary column.
   Refer to the TRACE GC Ultra Maintenance and Troubleshooting Manual.

2. Mount the adapter for capillary column on the detector base body.

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

4. Connect the required gas lines
   Verify the required gas supplies are properly connected to your GC.

5. Install the precolumn.
   Connect the precolumn to the injector.

6. Install the test column
   Connect the test column to the precolumn by using the press fit connections provided.

7. Perform the manual leak check following the Operating Procedure "*Perforing a Leak Check* " in Chapter 14 of the TRACE GC Ultra Operating Manual.

8. Connect the other end of the test column to the detector base body.

9. Connect your data handling.
   Verify that your data handling is properly connected to your GC system.

OPERATING PROCEDURE

FID-PKD Checkout

Before beginning, press **CONFIG** to verify the GC configuration:

Left inlet or Right inlet	PKD
Left carrier or Right carrier	He (helium)
Left detector or Right detector	FID

1. Use **LEFT CARRIER** or **RIGHT CARRIER** to display the appropriate Carrier Gas Control Table. Verify to operate in constant pressure mode. If not, scroll to `Flow mode`, press **MODE/TYPE** to access the selection menu, then select `con pres`. Scrool to `Pressure` and set the pressure value to have the required carrier gas flow rate `Col.flow`.

```
            LEFT CARRIER[1]

Pressure          30.0    30.0
Col.flow          3.00
Lin. veloc.              (60.9)<
```

1. These settings could also be for a right carrier.

2. Use **OVEN** to display the Column Oven Control Table. Set the oven temperature and the Oven Program required.

```
                OVEN

Temp              50.0    50.0
Initial Time              1.00
Ramp 1                    20.0
Final temp                 200
Final time 1              1.00<
Ramp 2                     Off
```

3. Use **LEFT INLET** or **RIGHT INLET** to display the appropriate PKD Injector Control Table. Set the required temperature setpoint `Temp`.Verify to operate in

Packed mode. If not, scroll to `Mode`, press **MODE/TYPE** to access the selection menu, then select `Packed`.

LEFT INLET (PKD)[1]		
Temp	200	200
Pressure	30.0	30.0
Mode:		Packed

1. These settings could also be for a right inlet.

4. Use **LEFT DETECTOR** or **RIGHT DETECTOR**, entry the appropriate FID Detector Control Table. Set the required temperature `Base Temp` and the detector gases `H2`, `Air` and `Mkup` required setpoints.

LEFT DETECTOR (FID)[1]		
Flame		Off
Base temp	250	250
Signal pA		(5.5)
Ign.thresh		2.0
Flameout retry		Off
H2	35	35
Air	350	350
Mkup N2	30	30<

1. These settings could also be for a right detector.

5. Ignite the FID flame scrolling to `Flame` and pressing **ON**.

6. Use **LEFT SIGNAL** or **RIGHT SIGNAL** to display the appropriate FID Detector Signal Control Table. Observe the FID flame signal at the display. This is the flame-on background offset. Scroll to `Range` and set the electrometer amplifier input range required.

7. Activate your Data System and set the parameters required for the checkout.

8. In the FID Detector Signal Control Table, scroll to `Auto zero?` and turn it **YES**.

LEFT SIGNAL (FID)[1]	
Output	(1000)
Offset	100
Auto zero?	Y/N
Range 10^(0..3)	0<
Analog filter	Off
Baseline comp	Off

1. These settings could also be for a right signal.

9. Perform a blank analysis injecting pure hexane and press **START** on the GC to begin the checkout run.

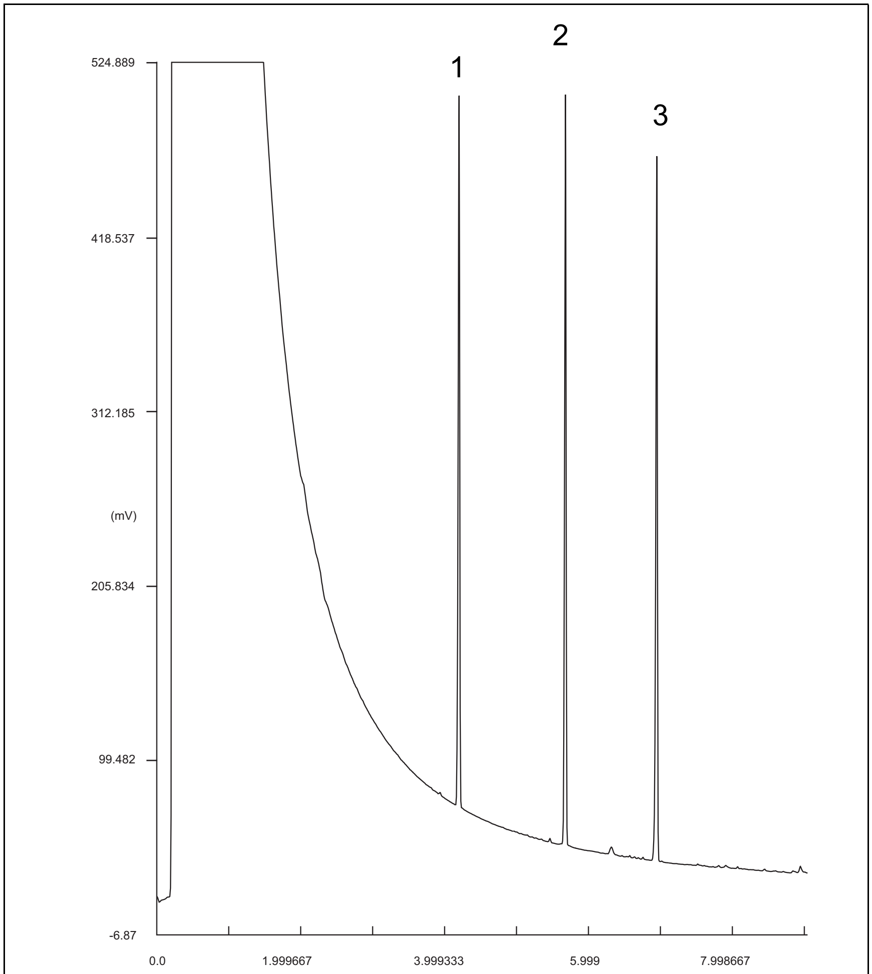
Baseline Acquisition and Analysis

NOTE Refer to the Acceptance Values reported in the Table 4-3 according to the data handling in use.

10. With the GC in Stand-by/Prep Run condition, activate the data system for 10 minutes to evaluate your baseline in isothermal condition.

11. After the baseline evaluation has been completed, set-up the data system to acquire a single run.

12. Inject the test mixture and press **START** on the GC to begin the checkout run. The resulting chromatogram should look like the one shown in *Figure 4.1*



1. Dodecane 2. Tetradecane 3. Hexadecane

Figure 4-1. FID-Packed Injection

13. The following criteria indicate successful completion of FID-PKD checkout.

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

Table 4-3. FID-PKD Acceptance Criteria

Acceptance Values	CHROM-CARD		
	Baseline Parameters	**Analog** *(1V Full Scale)*	**Digital** *(10V Full Scale)*
	Noise (µV)	< 30	< 300
	Wander (µV)	< 50	< 500
	Drift (µV/h)	< 100	< 1 000
	Analytical Results	**Analog** *(1V Full Scale)* *Area Counts (0.1 µVs)*	**Digital** *(10V Full Scale)* *Area Counts (0.1 µVs)*
	Components	> 3 600 000 for each component	> 36 000 000 for each component
	Area Count Ratio Calculated as C12/C16	1 ± 0.1	1 ± 0.1
⚠			

Computing-integrator (e.g. ChromJet)	
⚠	

Acceptance Values	CHROMQUEST	
	Baseline Parameters *(1V Full Scale)*	
	Noise (µV)	< 30
	Wander (µV)	< 50
	Drift (µV/h)	< 100
	Analytical Results *(1V Full Scale) - Area Counts (0.01 µVs)*	
	Components	> 36 000 000 for each component
	Area Count Ratio Calculated as C12/C16	1 ± 0.1
⚠		

Acceptance Values

ATLAS	
Baseline Parameters *(10V Full Scale)*	
Noise (μV)	< 300
Wander (μV)	< 500
Drift (μV/h)	< 1 000
Analytical Results *(10V Full Scale) - Area Counts (μVs)*	
Components	> 3 600 000 for each component
Area Count Ratio Calculated as C12/C16	1 ± 0.1

XCALIBUR	
Baseline Parameters *(Acquisition Frequency = 10 Hz)*	
Noise (Counts)	< 3 000
Wander Counts)	< 5 000
Drift (Counts/h)	< 10 000
Analytical Results *Area Counts (Cts*s)*	
Components	> 36 000 000 for each component
Area Count Ratio Calculated as C12/C16	1 ± 0.1

Analytical Acceptance Comments	
1	When the make-up gas is not used, the acceptance values will result to be 2.5 times lower than the values reported in Table 4-3.
2	When helium is used as make-up gas, the acceptance values will result to be 10 times lower than the values reported in Table 4-3.

5

Checkout Using FID with PPKD Injector

Chapter at a Glance...

Operating Procedures

SOP Number: P0308/07/E - 01 September 2009

Scope

Use the following procedure to verify proper FID operation with Purged Packed Injector.

Parts Referenced

Table 5-1. FID-PPKD 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	2 mm ID tapered	453 220 50
Liner Seal	Graphite seal for glass liner	290 334 05
Pre-column	Fused Silica Capillary Column 0.5 mt long 0.53 mm ID	260 603 75
Retaining Nut	M4 capillary column retaining nut	350 324 23
Press-fit connections	For columns 0.53/0.32 mm ID	350 438 16
Injection side adapter	For Wide bore column	347 003 03
Retaining Nut	For Injection side adapter	350 024 04
Detector side adapter	For Wide bore column	347 103 04
Ferrule	6 mm ID double brass ferrule	290 341 37
Nut	1/4” G-6 mm ID nut	350 201 18
Graphite Ferrule	Graphite ferrule for 0.53 mm ID Column Graphite ferrule for 0.32 mm ID Column	290 134 86 290 134 87
Septum	Standard septum for Purged Packed Injector	313 032 26
Syringe	10 µl size; 50 mm needle length	365 005 25

Table 5-1. FID-PPKD Parts Referenced (Continued)

Part	Description	Part Number
Test Mixture	Three components in n-Hexane: *Component* *Concentration* Dodecane 20 µg/ml Tetradecane 20 µg/ml Hexadecane 20 µg/ml	338 190 20
Gases	Chromatographic-grade purity	
Data Acquisition	Chrom-Card, ChromQuest, Atlas, Xcalibur, Computing-integrator	

Analytical Conditions Required for Purged Packed Injector

Table 5-2. FID-PPKD Analytical Conditions

Parameters	Setting
Gases	Carrier Gas: Helium = 30 kPa Constant Pressure Hydrogen = 35 ml/min Air = 350 ml/min Make-up Gas: Nitrogen = 30 ml/min
Oven Program	Initial Temperature = 50 °C Initial Time = 1 minute Ramp 1 = 20 °C/minute Final Temperature = 200 °C Final Time = 1 minute
Injector	Operating Mode = Wide bore Temperature = 200 °C
Detector	Base Temperature = 250 °C Detector Signal Range = 10^0
Injected Volume	1 µl + needle of Test Mixture
Analog Signal Output	Chrom-Card Acquisition Frequency = Medium
Digital Signal Output	Chrom-Card, ChromQuest, Atlas, Xcalibur Acquisition Frequency = 10 Hz

Recommended Initial Operations

Before starting the checkout, the following operations should be carried out:

1. Insert the glass liner.
   Remove the column and insert the 2 mm ID tapered glass liner, as required for the checkout, from the bottom of the injector with the appropriate liner seal. Fix the liner by using the appropriate adapter for capillary column.
   Refer to the TRACE GC Ultra Maintenance and Troubleshooting Manual.

2. Mount the adapter for capillary column on the detector base body.

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

4. Connect the required gas lines
   Verify the required gas supplies are properly connected to your GC.

5. Install the precolumn.
   Connect the precolumn to the injector.

6. Install the test column
   Connect the test column to the precolumn by using the press fit connections provided.

7. Perform the manual leak check following the Operating Procedure "*Perforing a Leak Check* " in Chapter 14 of the TRACE GC Ultra Operating Manual.

8. Connect the other end of the test column to the detector base body.

9. Perform Column Evaluation.

10. Connect your data handling.
    Verify that your data handling is properly connected to your GC system.

OPERATING PROCEDURE

FID-PPKD Checkout

Before beginning, press **CONFIG** to verify the GC configuration:

Left inlet or Right inlet	PPKD
Left carrier or Right carrier	He (helium)
Left detector or Right detector	FID

1. Use **LEFT CARRIER** or **RIGHT CARRIER** to display the appropriate Carrier Gas Control Table. Verify to operate in constant pressure mode. If not, scroll to `Flow mode`, press **MODE/TYPE** to access the selection menu, then select `con pres`. Scrool to `Pressure` and set the pressure value to have the required carrier gas flow rate `Col.flow`.

```
          LEFT CARRIER[1]

Pressure          30.0   30.0
Col.flow          3.00
Lin. veloc.             (60.9)<
```

1. These settings could also be for a right carrier.

2. Use **OVEN** to display the Column Oven Control Table. Set the oven temperature and the Oven Program required.

```
               OVEN

Temp              50.0   50.0
Initial Time             1.00
Ramp 1                   20.0
Final temp                200
Final time 1             1.00<
Ramp 2                    Off
```

3. Use **LEFT INLET** or **RIGHT INLET** to display the appropriate PPKD Injector Control Table. Set the required temperature setpoint `Temp`.Verify to operate in

Wide bore mode. If not, scroll to `Mode`, press **MODE/TYPE** to access the selection menu, then select `Widebore`.

LEFT INLET (PPKD)[1]		
Temp	200	200
Pressure	30.0	30.0
Mode:		Wide bore<
Constant sept purge?		Y<

1. These settings could also be for a right inlet.

4. Use **LEFT DETECTOR** or **RIGHT DETECTOR** to display the appropriate FID Detector Control Table. Set the required temperature `Base Temp`, and the detector gases `H2`, `Air` and `Mkup` required setpoints.

LEFT DETECTOR (FID)[1]		
Flame		Off
Base temp	250	250
Signal pA		(5.5)
Ign.thresh		2.0
Flameout retry		Off
H2	35	35
Air	350	350
Mkup N2	30	30<

1. These settings could also be for a right detector.

5. Ignite the FID flame scrolling to `Flame` and pressing **ON**.

6. Use **LEFT SIGNAL** or **RIGHT SIGNAL** to display the appropriate FID Detector Signal Control Table. Observe the FID flame signal at the display. This is the flame-on background offset. Scroll to `Range` and set the electrometer amplifier input range required.

LEFT SIGNAL (FID)[1]	
Output	(1000)
Offset	100
Auto zero?	Y/N
Range 10^(0..3)	0<
Analog filter	Off
Baseline comp	Off

1. These settings could also be for a right signal.

7. Activate your Data System and set the parameters required for the checkout.

8. In the FID Detector Signal Control Table, scroll to `Auto zero?` and turn it **YES**.

9. Perform a blank analysis injecting pure hexane and press **START** on the GC to begin the checkout run.

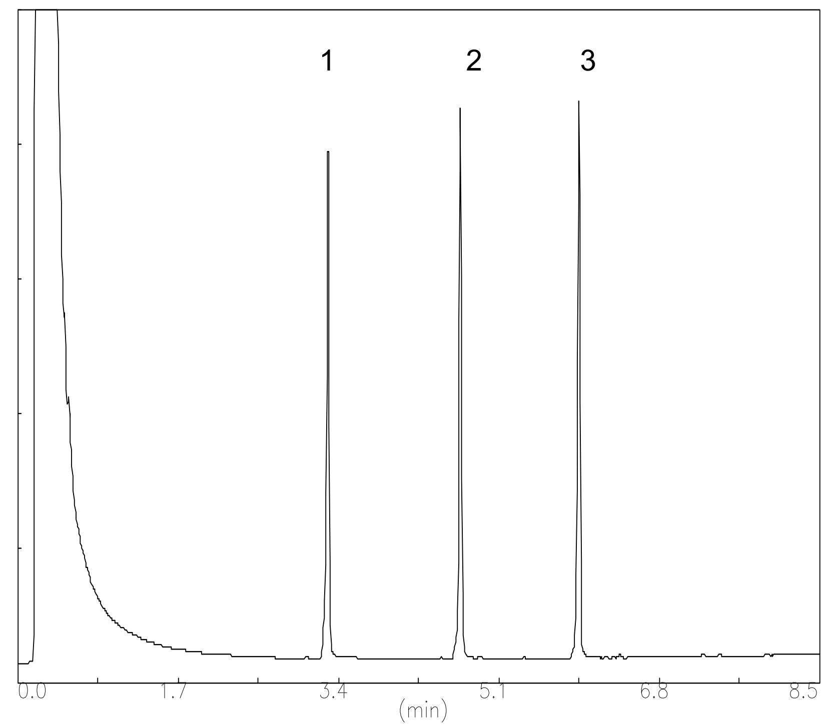
Baseline Acquisition and Analysis

NOTE Refer to the Acceptance Values reported in the Table 5-3 according to the data handling in use.

10. With the GC in Stand-by/Prep Run condition, activate the data system for 10 minutes to evaluate your baseline in isothermal condition.

11. After the baseline evaluation has been completed, set-up the data system to acquire a single run.

12. Inject the test mixture and press **START** on the GC to begin the checkout run. The resulting chromatogram should look like the one shown in *Figure 5.1*



1. Dodecane	2. Tetradecane	3. Hexadecane

Figure 5-1. FID-PPKD Injection

13. The following criteria indicate successful completion of FID-PPKD checkout.

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

Table 5-3. FID-PPKD Acceptance Criteria

Acceptance Values	CHROM-CARD		
	Baseline Parameters	**Analog** *(1V Full Scale)*	**Digital** *(10V Full Scale)*
	Noise (µV)	< 30	< 300
	Wander (µV)	< 50	< 500
	Drift (µV/h)	< 100	< 1 000
	Analytical Results	**Analog** *(1V Full Scale)* *Area Counts (0.1 µVs)*	**Digital** *(10V Full Scale)* *Area Counts (0.1 µVs)*
	Components	> 3 600 000 for each component	> 36 000 000 for each component
	Area Count Ratio Calculated as C12/C16	1 ± 0.1	1 ± 0.1
⚠			

Computing-integrator (e.g. ChromJet)	
⚠	

Acceptance Values	CHROMQUEST	
	Baseline Parameters *(1V Full Scale)*	
	Noise (µV)	< 30
	Wander (µV)	< 50
	Drift (µV/h)	< 100
	Analytical Results *(1V Full Scale) - Area Counts (0.01 µVs)*	
	Components	> 36 000 000 for each component
	Area Count Ratio Calculated as C12/C16	1 ± 0.1
⚠		

	ATLAS	
	Baseline Parameters *(10V Full Scale)*	
	Noise (µV)	< 300
	Wander (µV)	< 500
	Drift (µV/h)	< 1 000
	Analytical Results *(10V Full Scale) - Area Counts (µVs)*	
	Components	> 3 600 000 for each component
	Area Count Ratio Calculated as C12/C16	1 ± 0.1
⚠		

Acceptance Values	**XCALIBUR**	
	Baseline Parameters *(Acquisition Frequency = 10 Hz)*	
	Noise (Counts)	< 3 000
	Wander Counts)	< 5 000
	Drift (Counts/h)	< 10 000
	Analytical Results *Area Counts (Cts*s)*	
	Components	> 36 000 000 for each component
	Area Count Ratio Calculated as C12/C16	1 ± 0.1
⚠		

Analytical Acceptance Comments	
1	When the make-up gas is not used, the acceptance values will result to be 2.5 times lower than the values reported in Table 5-3.
2	When helium is used as make-up gas, the acceptance values will result to be 10 times lower than the values reported in Table 5-3.

6

Checkout Using FID with PTV Injector

Chapter at a Glance...

Operating Procedures

SOP Number: P0309/07/E - 01 September 2009

Scope

Use the following procedure to verify proper FID operation with the Programmable Temperature Vaporizing Injector.

Parts Referenced

Table 6-1. FID-PTV 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
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	365 005 25
Test Mixture	Three components in n-Hexane: *Component* *Concentration* Dodecane 20 µg/ml Tetradecane 20 µg/ml Hexadecane 20 µg/ml	338 190 20
Gases	Chromatographic-grade purity	
Data Acquisition	Chrom-Card, ChromQuest, Atlas, Xcalibur, Computing-integrator	

Analytical Conditions Required for PTV Splitless Injection

Table 6-2. FID-PTV Analytical Conditions

Gases	Carrier Gas: Helium = 30 kPa Constant Pressure Hydrogen = 35 ml/min Air = 350 ml/min Make-up Gas: Nitrogen = 30 ml/min
Oven Program	Initial Temperature = 50 °C Initial Time = 1 minute Ramp 1 = 20 °C/minute Final Temperature = 200 °C Final Time = 1 minute
Injector	Operating Mode = PTV splitless Splitless Time = 0.8 minutes Split Flow = 50 ml/min Constant Septum Purge = Yes Inject Temp = 50 °C Inject Time = 0.1 minute Transfer ramp = 10 °C/sec Transfer Temperature = 260 °C Transfer time = 1 minute
Detector	Base Temperature = 250 °C Detector Signal Range = 10^0
Injected Volume	1 µl of Test Mixture
Analog Signal Output	Chrom-Card Acquisition Frequency = Medium
Digital Signal Output	Chrom-Card, ChromQuest, Atlas, Xcalibur Acquisition Frequency = 10 Hz

Recommended Initial Operations

Before starting the checkout, the following operations should be carried out:

1. Replace the liner.
   The liner currently installed in your injector should be carefully removed and replaced with the 2 mm ID Silcosteel liner, 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. Connect the required gas lines
   Verify the required gas supplies are properly connected to your GC.

4. Install the test column.
   The column currently installed should be carefully removed and replaced with the required test column.

5. Perform Column Evaluation and Leak Test.

6. Connect your data handling.
   Verify that your data handling is properly connected to your GC system.

OPERATING PROCEDURE

FID-PTV Checkout in PTV Splitless Mode

Before beginning, press **CONFIG** to verify the GC configuration:

Right inlet	PTV
Right carrier	He (helium)
Right detector	FID

1. Use **RIGHT CARRIER** to display the appropriate Carrier Gas Control Table. Verify to operate in constant pressure mode. If not, scroll to `Flow mode`, press **MODE/TYPE** to access the selection menu, then select `con pres`. Scrool to `Pressure` and set the pressure value to have the required carrier gas flow rate `Col.flow`.

```
        RIGHT CARRIER¹

Pressure          30.0   30.0
Col.flow          3.00
Lin. veloc.             (60.9)<
```

2. Use **OVEN** to display the Column Oven Control Table. Set the oven temperature and the Oven Program required.

```
           OVEN

Temp               50.0   50.0
Initial Time              1.00
Ramp 1                    20.0
Final temp                 200
Final time 1              1.00<
Ramp 2                     Off
```

3. Use **RIGHT INLET** to display the appropriate Programmable Temperature Vaporizing Injector Control Table. Set the required temperature setpoint `Temp`. Verify to operate in **PTV splitless** mode. If not, scroll to `Mode`, press **MODE/TYPE** to access the selection menu, then select `PTV splitless`. Scroll to `Splitless time` to set the required setpoint.

```
        RIGHT INLET (PTV)

Temp                     50    50
Pressure               30.0  30.0
Mode:             PTV Splitless
Total flow               (53.0)
Split Flow             50.0  50.0
Splitless time         0.80  0.80
Constant sept purge?          Y
Inject phase menu:            Y<
```

4. Scroll to `Inject phase menu`. Press **MODE/TYPE** to enter the **PTV Phase Menu**.

```
          PTV PHASE MENU

Ramped pressure?              N
Inject temp                  50
Inject time                 0.1
Transfer ramp                10
Transfer temp               260
Transfer time              1.00<
```

5. Select `Ramped pressure?` **NO**. Set the required `Inject temp` and *Inject time* setpoints as required. Then, set the `Transfer ramp`, the `Transfer temp` and the `Transfer time` required setpoints.

6. Use **RIGHT DETECTOR** to display the appropriate FID Detector Control Table. Set the required temperature `Base Temp`. and the detector gases `H2`, `Air` and `Mkup` required setpoints.

7. Ignite the FID flame scrolling to `Flame` and pressing **ON**.

```
   RIGHT DETECTOR (FID)

Flame                      Off
Base temp          250     250
Signal pA                 (5.5)
Ign.thresh                 2.0
Flameout retry             Off
H2                  35      35
Air                350     350
Mkup N2             30      30<
```

8. Use **RIGHT SIGNAL** to display the appropriate FID Detector Signal Control Table. Observe the FID flame signal at the display. This is the flame-on background offset. Scroll to `Range` and set the electrometer amplifier input range required.

```
    RIGHT SIGNAL (FID)

Output                   (1000)
Offset                    100
Auto zero?                Y/N
Range 10^(0..3)             0<
Analog filter             Off
Baseline comp             Off
```

9. Activate your Data System and set the parameters required for the checkout.

10. In the FID Detector Signal Control Table, scroll to `Auto zero?` and turn it **ON**.

11. Activate your Data System and set the parameters required for the checkout.

12. In the FID Detector Signal Control Table, scroll to `Auto zero?` and turn it **YES**.

13. Perform a blank analysis injecting pure hexane and press **START** on the GC to begin the checkout run.

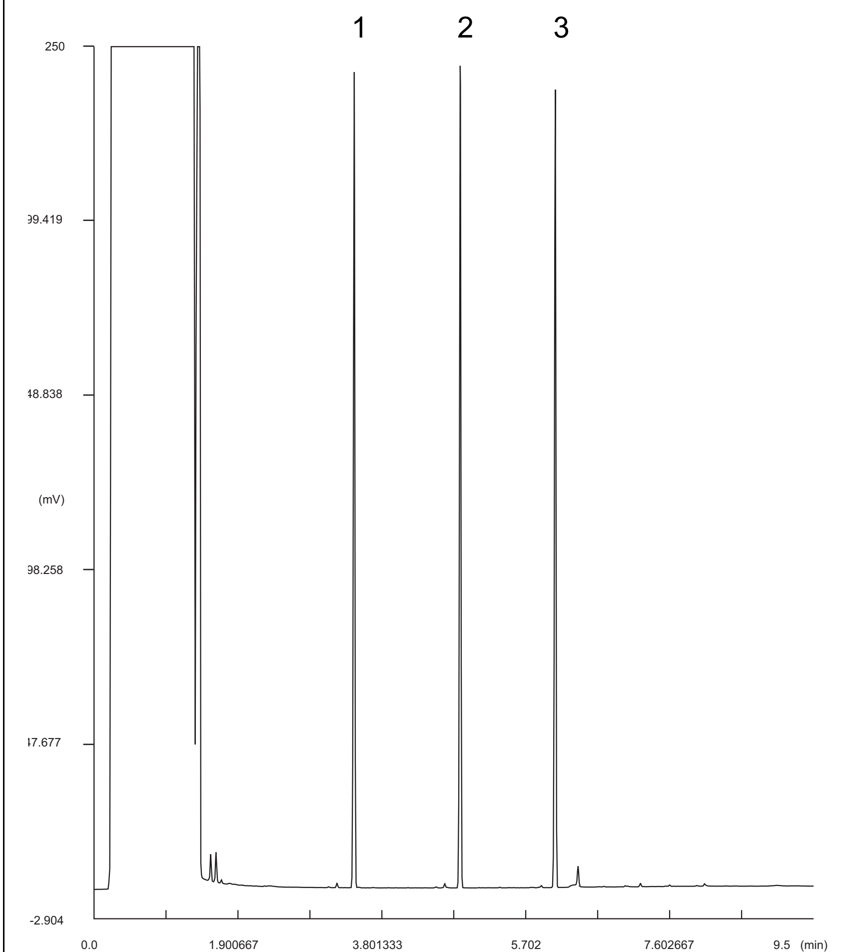
Baseline Acquisition and Analysis

NOTE Refer to the Acceptance Values reported in the Table 6-3 according to the data handling in use.

14. With the GC in Stand-by/Prep Run condition, activate the data system for 10 minutes to evaluate your baseline in isothermal condition.

15. After the baseline evaluation has been completed, set-up the data system to acquire a single run.

16. Inject the test mixture and press **START** on the GC to begin the checkout run. The resulting chromatogram should look like the one shown in *Figure 6.1*



1. Dodecane 2. Tetradecane 3. Hexadecane

Figure 6-1. FID-PTV Injection

17. The following criteria indicate successful completion of FID-PTV checkout.

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

Table 6-3. FID-PPKD Acceptance Criteria

Acceptance Values	CHROM-CARD		
	Baseline Parameters	**Analog** *(1V Full Scale)*	**Digital** *(10V Full Scale)*
	Noise (µV)	< 30	< 300
	Wander (µV)	< 50	< 500
	Drift (µV/h)	< 100	< 1 000
	Analytical Results	**Analog** *(1V Full Scale)* *Area Counts (0.1 µVs)*	**Digital** *(10V Full Scale)* *Area Counts (0.1 µVs)*
	Components	> 2 500 000 for each component	> 25 000 000 for each component
	Area Count Ratio Calculated as C12/C16	1 ± 0.1	1 ± 0.1

Computing-integrator (e.g. ChromJet)	

Acceptance Values	CHROMQUEST	
	Baseline Parameters *(1V Full Scale)*	
	Noise (µV)	< 30
	Wander (µV)	< 50
	Drift (µV/h)	< 100
	Analytical Results *(1V Full Scale) - Area Counts (0.01 µVs)*	
	Components	> 25 000 000 for each component
	Area Count Ratio Calculated as C12/C16	1 ± 0.1

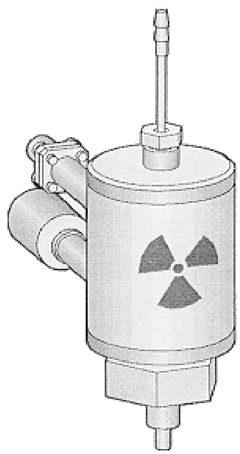
	ATLAS	
	Baseline Parameters *(10V Full Scale)*	
	Noise (µV)	< 300
	Wander (µV)	< 500
	Drift (µV/h)	< 1 000
	Analytical Results *(10V Full Scale) - Area Counts (µVs)*	
	Components	> 2 500 000 for each component
	Area Count Ratio Calculated as C12/C16	1 ± 0.1
⚠		

Acceptance Values	XCALIBUR	
	Baseline Parameters *(Acquisition Frequency = 10 Hz)*	
	Noise (Counts)	< 3 000
	Wander Counts)	< 5 000
	Drift (Counts/h)	< 10 000
	Analytical Results *Area Counts (Cts*s)*	
	Components	> 25 000 000 for each component
	Area Count Ratio Calculated as C12/C16	1 ± 0.1
⚠		

Analytical Acceptance Comments	
1	When the make-up gas is not used, the acceptance values will result to be 2.5 times lower than the values reported in Table 6-3.
2	When helium is used as make-up gas, the acceptance values will result to be 10 times lower than the values reported in Table 6-3.

SECTION III

SOPs Using ECD



The *SOPs Using ECD* section, contains the procedures to test the TRACE GC Ultra with the Electron Capture Detector (ECD) using different injectors.

7

Checkout Using ECD with S/SL Injector

Chapter at a Glance…

Operating Procedures

SOP Number: P0294/07/E - 01 September 2009

Scope

Use the following procedure to verify proper ECD operation with the Split/Splitless Injector.

Parts Referenced

Table 7-1. ECD-S/SL 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 injections	453 200 32
Liner Seal	Graphite seal 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; 70 mm needle length	365 001 03
Test Mixture	Two components in Iso-octane *Component* *Concentration* Lindane 0.030 µg/ml Aldrin 0.030 µg/ml	338 190 11
Gases	Chromatographic-grade purity	
Data Acquisition	Chrom-Card, ChromQuest, Atlas, Xcalibur, Computing-integrator	

Analytical Conditions Required for Splitless Injection

Table 7-2. ECD-S/SL Analytical Conditions

Parameters Setting	
Gases	Carrier Gas: Helium = 30 kPa Constant Pressure Make-up: Nitrogen = 30 ml/min
Oven Program	Initial Temperature = 70 °C Initial Time = 1 minute Ramp 1 = 20 °C/minute Final Temperature = 220 °C Final Time = 1 minute
Injector	Operating Mode = Splitless Temperature = 230 °C Splitless Time = 0.8 minutes Split Flow = 60 ml/min Constant Septum Purge = Yes
Detector	Base Temperature = 250 °C ECD temperature = 300 °C Reference Current = 1 nA Pulse Amplitude = 50 V Pulse Width = 1 µs
Injected Volume	1 µl + needle of Test Mixture
Analog Signal Output	Chrom-Card Acquisition Frequency = Medium
Digital Signal Output	Chrom-Card, ChromQuest, Atlas, Xcalibur Acquisition Frequency = 10 Hz

Recommended Initial Operations

Before starting the checkout, the following operations should be carried out:

1. Replace the glass liner.
   The glass liner currently installed in your injector should be carefully removed and replaced with the 3 mm ID glass liner for splitless 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. Connect the required gas lines
   Verify the required gas supplies are properly connected to your GC.

4. Install the test column.
   The column currently installed should be carefully removed and replaced with the required test column.

5. Perform Column Evaluation and Leak Test.

6. Connect your data handling.
   Verify that your data handling is properly connected to your GC system.

OPERATING PROCEDURE

ECD-S/SL Checkout in Splitless Mode

Before beginning, press **CONFIG** to verify the GC configuration:

Left inlet or Right inlet	S/SL
Left carrier or Right carrier	He (helium)
Left detector or Right detector	ECD

1. Use **LEFT CARRIER** or **RIGHT CARRIER** to display the appropriate Carrier Gas Control Table. Verify to operate in constant pressure mode. If not, scroll to `Flow mode`, press **MODE/TYPE** to access the selection menu, then select `con pres`. Scrool to `Pressure` and set the pressure value to have the required carrier gas flow rate `Col.flow`.

```
          LEFT CARRIER[1]
Pressure          30.0    30.0
Col.flow          3.00
Lin. veloc.             (60.9)<
```

1. These settings could also be for a right carrier.

2. Use **OVEN** to display the Column Oven Control Table. Set the oven temperature and the Oven Program required.

```
               OVEN
Temp              70.0   70.0
Initial Time             1.00
Ramp 1                   20.0
Final temp                220
Final time 1             1.00<
Ramp 2                    Off
```

3. Use **LEFT INLET** or **RIGHT INLET** to display the appropriate Split/Splitless Injector Control Table. Set the required temperature setpoint *Temp*. Verify to

operate in **Splitless** mode. If not, scroll to `Mode`, press **MODE/TYPE** to access the selection menu, then select `Splitless`. Scroll to `Splitless time` to set the required setpoint.

LEFT INLET (S/SL)[1]		
Temp	230	230
Pressure	30.0	30.0
Mode:		Splitless
Total flow		(63.0)
Split Flow	60.0	60.0
Splitless time	0.80	0.80
Constant sept purge?		Y<

1. These settings could also be for a right inlet.

4. Use **LEFT DETECTOR** or **RIGHT DETECTOR** to display the appropriate ECD Detector Control Table. Set the required temperature `Base Temp`. and the `Mkup` gas required setpoints.

LEFT DETECTOR (ECD)[1]		
Base temp	250	250
ECD Temp	300	300
Ref current nA		1.0
Freq kHz		(2.20)
Pulse amp V		50
Pulse width µs		1.0
Mkup (N2)	30	30<

1. These settings could also be for a right detector.

5. Set the `Reference Current` to 1.0 nA.

6. Set the `Pulse Amplitude` to 50 V.

7. Scroll to `Pulse Width` and press **ENTER** to open the menu selection. Select the pulse width to 1µs then press **ENTER**.

8. Observe the ECD frequency value at the display. A base frequency value between 1 kHz and 3 kHz should be displayed.

   If the ECD frequency value is less than 1kHz, reduce gradually the pulse amplitude till the base frequency value is about 1 kHz (the pulse amplitude value should not be less than 20V). Then, if necessary modify the reference current in order to have a base frequency of about 1-1,5 kHz. Let the detector signal to stabilize.

9. Activate your Data System and set the parameters required for the checkout.

10. Use **LEFT SIGNAL** or **RIGHT SIGNAL** to display the ECD Detector Signal Control Table. Scroll to `Auto zero?` and turn it **YES**.

LEFT SIGNAL (ECD)[1]	
Output	(1000)
Offset	100<
Auto zero?	Y/N
Baseline comp?	Off

1. These settings could also be for a right signal.

11. Perform a blank analysis injecting pure iso-octane and press **START** on the GC to begin the checkout run.

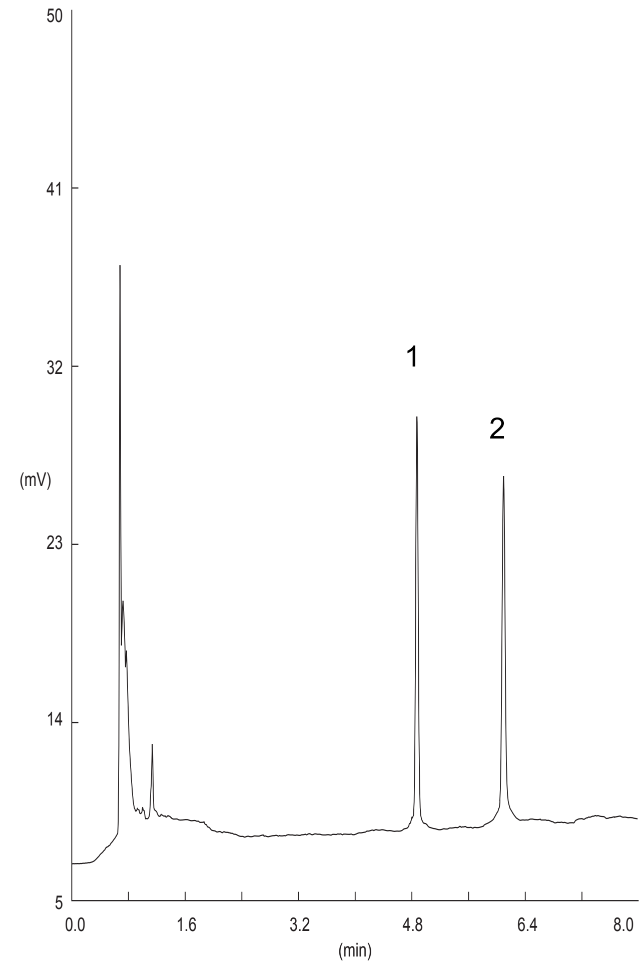
Baseline Acquisition and Analysis

NOTE Refer to the Acceptance Values reported in the Table 7-3 according to the data handling in use.

12. With the GC in Stand-by/Prep Run condition, activate the data system for 10 minutes to evaluate your baseline in isothermal condition.

13. After the baseline evaluation has been completed, set-up the data system to acquire a single run.

14. When the GC is ready, inject the test mixture and press **START** on the GC to begin the checkout run. The resulting chromatogram should look like the one shown in *Figure 7.1*.



1. Lindane 2. Aldrin

Figure 7-1. ECD-Splitless Injection

15. Establish the integration parameters and the peak table identifying the test mix components.

16. Set up the data system to calculate the signal-to-noise ratio.

Using Chrom-Card Data System

Operate as follows:

- Open the Method Editor and include the signal-to-noise Report into the Report Parameters Page.
- By clicking on the side icon, open the signal-to-noise calculation and set Lindane component as signal peak ID.
- Choose a part of baseline without peaks or interference signals and calculate the noise for 0.1 min. (Verify that the noise value is comparable with the one of the previous *Baseline Acquisition and Analysis*).
- Generate a report showing the chromatogram, peak area and signal-to-noise information for Lindane.
- Repeat the procedure to calculate the signal-to-noise ratio also for Aldrin.
- Generate a report showing the chromatogram, peak area and signal-to-noise information for Aldrin.

NOTE If it is not possible to find a suitable part of baseline, to have a complete Chrom-Card final report proceed as follows:
Start the acquisition on the data system. Let the system to acquire for about 1 minute and then inject the test mixture. The noise can be calculated during the first minute of acquisition. Since the retention time will shift by a delayed time, the Component Table has to be updated.

Using non-Chrom-Card Data System

Operate as follows:

- Set the parameters to calculate the signal-to-noise ratio according to the instruction reported in the relevant data system manual.

CAUTION **Choose a part of baseline without peaks or interference signals and calculate the noise for 0.1 min. (Verify that the noise value is comparable with the one of the previous *Baseline Acquisition and Analysis*).**

17. The following criteria indicate successful completion of ECD-S/SL checkout.

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

Table 7-3. ECD-S/SL Acceptance Criteria

Acceptance Values	CHROM-CARD		
	Baseline Parameters	**Analog** *(1V Full Scale)*	**Digital** *(10V Full Scale)*
	Noise (µV)	< 40	< 400
	Wander (µV)	< 80	< 800
	Drift (µV/h)	< 200	< 2 000
	Analytical Results		
	Lindane Signal-to-Noise Ratio		> 4 000
	Aldrin Signal-to-Noise Ratio		> 4 000
⚠			

Acceptance Values	CHROMQUEST	
	Baseline Parameters *(1V Full Scale)*	
	Noise (µV)	< 40
	Wander (µV)	< 80
	Drift (µV/h)	< 200
	Analytical Results	
	Lindane Signal-to-Noise Ratio	> 4 000
	Aldrin Signal-to-Noise Ratio	> 4 000
⚠		

Acceptance Values	ATLAS	
	Baseline Parameters *(10V Full Scale)*	
	Noise (µV)	< 400
	Wander (µV)	< 800
	Drift (µV/h)	< 2 000
	Analytical Results	
	Lindane Signal-to-Noise Ratio	> 4 000
	Aldrin Signal-to-Noise Ratio	> 4 000
⚠		

Acceptance Values	XCALIBUR	
	Baseline Parameters *(Acquisition Frequency = 10 Hz)*	
	Noise (Counts)	< 4 000
	Wander (Counts)	< 8 000
	Drift (Counts/h)	< 20 000
	Analytical Results	
	Lindane Signal-to-Noise Ratio	> 4 000
	Aldrin Signal-to-Noise Ratio	> 4 000
⚠		

Analytical Acceptance Comments	
1	Using Chrom-Card, set the signal-to-noise report parameters as described in the current procedure.
2	Using ChromQuest, Atlas, Xcalibur or a Computing integrator (e.g. ChromJet), calculate the S/N ratio as *Peak Height (counts)/noise (counts)*.

8

Checkout Using ECD with OC Injector

Chapter at a Glance…

Operating Procedures

SOP Number: P0295/08/E - 01 September 2009

Scope

Use the following procedure to verify proper ECD operation with the On-Column Injector.

Parts Referenced

Table 8-1. ECD-OCI Parts Referenced

	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
Retaining Nut	M4 capillary column retaining nut	350 324 23
Vespel Ferrule	Vespel ferrule for 0.32 mm ID Column	290 134 60
Syringe	10 µl size; 75 mm needle length	365 020 07
Test Mixture	Two components in Iso-octane *Component* *Concentration* Lindane 0.030 µg/ml Aldrin 0.030 µg/ml	338 190 11
Gases	Chromatographic-grade purity	
Data Acquisition	Chrom-Card, ChromQuest, Atlas, Xcalibur, Computing-integrator	
Syringe	10 µl size; 80 mm needle length	365 020 19
Pre-column	2 m long; 0.53 mm ID	260 603 75
Press-fit set	Set of Press-fir connectors for TRACE OC	350 038 45

Analytical Conditions Required for On-Column Injection

Table 8-2. ECD-OCI Analytical Conditions

Gases	Carrier Gas: Helium = 30 kPa Constant Pressure Make-up: Nitrogen = 30 ml/min
Oven Program	Initial Temperature = 85 °C Initial Time = 1 minute Ramp 1 = 20 °C/minute Final Temperature = 220 °C Final Time = 1 minute
Injector	Secondary Cooling = 3 seconds
Detector	Base Temperature = 250 °C ECD Temperature = 300 °C Reference Current = 1 nA Pulse Amplitude = 50 V Pulse Width = 1µs
Injected Volume	1 µl of Test Mixture
Analog Signal Output	Chrom-Card Acquisition Frequency = Medium
Digital Signal Output	Chrom-Card, ChromQuest, Atlas, Xcalibur Acquisition Frequency = 10 Hz

Recommended Initial Operations

Before starting the checkout, the following operations should be carried out:

1. Connect the required gas lines.
   Verify the required gas supplies are properly connected to your GC.

2. Install the test column.
   The column currently installed should be carefully removed and replaced with the required test column.
   In case of automatic On-column for TriPlus sampler, install the pre-column and connect it to the test column by press-fit connector.

3. Install and connect the TriPlus sampler and its components.

4. Perform Column Evaluation and Leak Test.

5. Connect your data handling.
   Verify that your data handling is properly connected to your GC system.

6. Verify the opening/closing of the OC injector actuator by using the proper commands.

7. Verify the alignment of the syringe on the OC injector.

OPERATING PROCEDURE

ECD-OCI Checkout in On-Column Mode

Before beginning, press **CONFIG** to verify the GC configuration:

Left inlet or Right inlet	OCI
Left carrier or Right carrier	He (helium)
Left detector or Right detector	ECD

1. Use **LEFT CARRIER** or **RIGHT CARRIER** to display the appropriate Carrier Gas Control Table. Verify to operate in constant pressure mode. If not, scroll to `Flow mode`, press **MODE/TYPE** to access the selection menu, then select `con pres`. Scrool to `Pressure` and set the pressure value to have the required carrier gas flow rate `Col.flow`.

```
        LEFT CARRIER[1]

Pressure         30.0   30.0
Col.flow         3.00
Lin. veloc.            (60.9)<
```

1. These settings could also be for a right carrier.

2. Use **OVEN** to display the Column Oven Control Table. Set the oven temperature and the Oven Program required.

```
             OVEN

Temp             85.0   85.0
Initial Time            1.00
Ramp 1                  20.0
Final temp               220
Final time 1            1.00<
Ramp 2                   Off
```

3. Use **LEFT INLET** or **RIGHT INLET** to display the appropriate Cold On-Column Injector Control Table. Scroll to `Sec. cool time` and set the required secondary cooling time.

```
      LEFT INLET (OCI)¹

Pressure          30.0  30.0
Sec. Cool Time          3.00<
```

1. These settings could also be for a right inlet.

4. Use **LEFT DETECTOR** or **RIGHT DETECTOR** to display the appropriate ECD Detector Control Table. Set the required temperature `Base Temp`. and the `Mkup` gas required setpoints.

```
    LEFT DETECTOR (ECD)¹

Base temp          250   250
ECD Temp           300   300
Ref current nA           1.0
Freq kHz              (2.20)
Pulse amp V               50
Pulse width µs           1.0
Mkup (N2)            30  30<
```

1. These settings could also be for a right detector.

5. Set the `Reference Current` to 1.0 nA.

6. Set the `Pulse Amplitude` to 50 V.

7. Scroll to `Pulse Width` and press **ENTER** to open the menu selection. Select the pulse width to 1µs then press **ENTER**.

8. Observe the ECD frequency value at the display. A base frequency value between 1 kHz and 3 kHz should be displayed. If the ECD frequency value is less than 1kHz, reduce gradually the pulse amplitude till the base frequency value is about 1 kHz (the pulse amplitude value should not be less than 20V). Then, if necessary modify the reference current in order to have a base frequency of about 1-1,5 kHz. Let the detector signal to stabilize.

9. Activate your Data System and set the parameters required for the checkout.

10. Use **LEFT SIGNAL** or **RIGHT SIGNAL** to display the appropriate ECD Detector Signal Control Table. Scroll to `Auto zero?` and turn it **YES**.

```
       LEFT SIGNAL (ECD)¹

Output                  (1000)
Offset                    100<
Auto zero?                Y/N
Baseline comp?            Off
```

1. These settings could also be for a right signal.

11. Perform a blank analysis injecting pure iso-octane and press **START** on the GC to begin the checkout run.

Baseline Acquisition and Analysis

NOTE Refer to the Acceptance Values reported in the Table 8-3 according to the data handling in use.

12. With the GC in Stand-by/Prep Run condition, activate the data system for 10 minutes to evaluate your baseline in isothermal condition.

13. After the baseline evaluation has been completed, set-up the data system to acquire a single run.

14. Perform the analysis

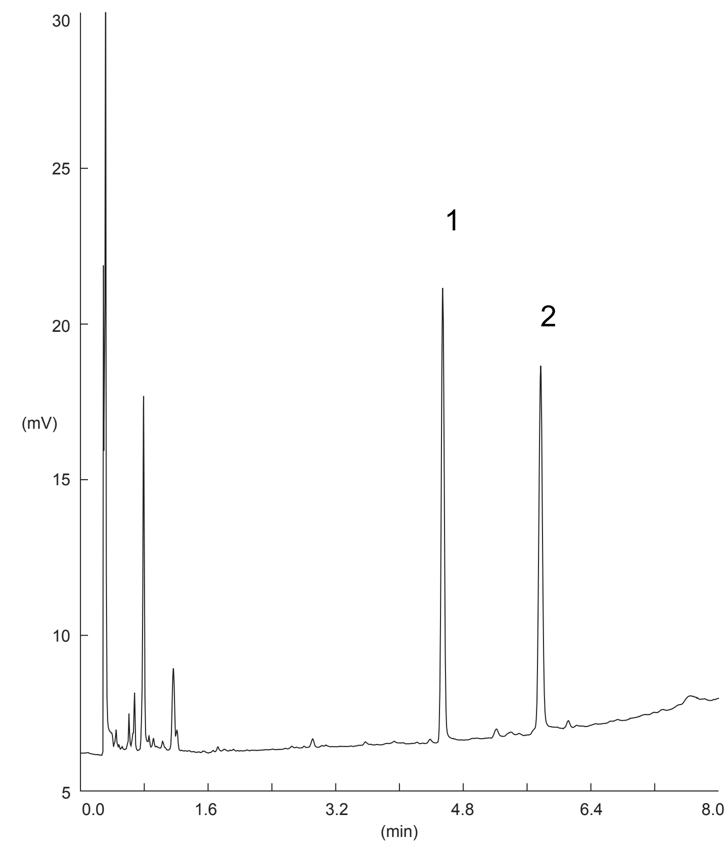
    Manual injection

    - Inject the test mixture and press **START** on the GC to begin the checkout run.

    *Automatic injection with TriPlus sample*r

    - Fill a vial with the standard mix and place that vial in the sample tray.
    - Load the method for OC and perform the sampling.

    The resulting chromatogram should look like the one shown in *Figure 8.1*.



1. Lindane	2. Aldrin

Figure 8-1. ECD-On-Column Injection

15. Establish the integration parameters and the peak table identifying the test mix components.

16. Set up the data system to calculate the signal-to-noise ratio.

Using Chrom-Card Data System

Operate as follows:

- Open the Method Editor and include the signal-to-noise Report into the Report Parameters Page.
- By clicking on the side icon, open the signal-to-noise calculation and set Lindane component as signal peak ID.
- Choose a part of baseline without peaks or interference signals and calculate the noise for 0.1 min. (Verify that the noise value is comparable with the one of the previous *Baseline Acquisition and Analysis*).
- Generate a report showing the chromatogram, peak area and signal-to-noise information for Lindane.
- Repeat the procedure to calculate the signal-to-noise ratio also for Aldrin.
- Generate a report showing the chromatogram, peak area and signal-to-noise information for Aldrin.

NOTE If it is not possible to find a suitable part of baseline, to have a complete Chrom-Card final report proceed as follows:
Start the acquisition on the data system. Let the system to acquire for about 1 minute and then inject the test mixture. The noise can be calculated during the first minute of acquisition. Since the retention time will shift by a delayed time, the Component Table has to be updated.

Using non-Chrom-Card Data System

Operate as follows:

- Set the parameters to calculate the signal-to-noise ratio according to the instruction reported in the relevant data system manual.

CAUTION **Choose a part of baseline without peaks or interference signals and calculate the noise for 0.1 min. (Verify that the noise value is comparable with the one of the previous *Baseline Acquisition and Analysis*).**

17. The following criteria indicate successful completion of ECD-OCI checkout.
18. If these criteria are not met, repeat the test.

Table 8-3. ECD-OCI Acceptance Criteria

Acceptance Values

CHROM-CARD		
Baseline Parameters	**Analog** *(1V Full Scale)*	**Digital** *(10V Full Scale)*
Noise (µV)	< 40	< 400
Wander (µV)	< 80	< 800
Drift (µV/h)	< 200	< 2 000
Analytical Results		
Lindane Signal-to-Noise Ratio		> 3 000
Aldrin Signal-to-Noise Ratio		> 3 000

Acceptance Values

CHROMQUEST	
Baseline Parameters *(1V Full Scale)*	
Noise (µV)	< 40
Wander (µV)	< 80
Drift (µV/h)	< 200
Analytical Results	
Lindane Signal-to-Noise Ratio	> 3 000
Aldrin Signal-to-Noise Ratio	> 3 000

Acceptance Values	**ATLAS**	
	Baseline Parameters *(10V Full Scale)*	
	Noise (µV)	< 400
	Wander (µV)	< 800
	Drift (µV/h)	< 2 000
	Analytical Results	
	Lindane Signal-to-Noise Ratio	> 3 000
	Aldrin Signal-to-Noise Ratio	> 3 000
⚠		

Acceptance Values	**XCALIBUR**	
	Baseline Parameters *(Acquisition Frequency = 10 Hz)*	
	Noise (Counts)	< 4 000
	Wander (Counts)	< 8 000
	Drift (Counts/h)	< 20 000
	Analytical Results	
	Lindane Signal-to-Noise Ratio	> 3 000
	Aldrin Signal-to-Noise Ratio	> 3 000
⚠		

Analytical Acceptance Comments	
1	Using Chrom-Card, set the signal-to-noise report parameters as described in the current procedure.
2	Using ChromQuest, Atlas, Xcalibur or a Computing integrator (e.g. ChromJet), calculate the S/N ratio as *Peak Height (counts)/noise (counts)*.

9

Checkout Using ECD with PKD Injector

Chapter at a Glance...

Operating Procedures

SOP Number: P0310/07/E - 01 September 2009

Scope

Use the following procedure to verify proper ECD operation with Packed Injector.

Parts Referenced

Table 9-1. ECD-PKD 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	2 mm ID tapered	453 220 50
Liner Seal	Graphite seal for glass liner	290 334 05
Pre-column	Fused Silica Capillary Column 0.5 mt long 0.53 mm ID	260 603 75
Retaining Nut	M4 capillary column retaining nut	350 324 23
Press-fit connections	For columns 0.53/0.32 mm ID	350 438 16
Injection side adapter	For Wide bore column	347 003 03
Retaining Nut	For Injection side adapter	350 024 04
Detector side adapter	For Wide bore column	347 103 04
Ferrule	6 mm ID double brass ferrule	290 341 37
Nut	1/4" G-6 mm ID nut	350 201 18
Graphite Ferrule	Graphite ferrule for 0.53 mm ID Column Graphite ferrule for 0.32 mm ID Column	290 134 86 290 134 87
Septum	Standard septum for Packed Injector	313 032 26
Syringe	10 µl size; 50 mm needle length	365 005 25

Table 9-1. ECD-PKD Parts Referenced (Continued)

Part	Description	Part Number
Test Mixture	Two components in Iso-octane *Component* *Concentration* Lindane 0.030 µg/ml Aldrin 0.030 µg/ml	338 190 11
Gases	Chromatographic-grade purity	
Data Acquisition	Chrom-Card, ChromQuest, Atlas, Xcalibur, Computing-integrator	

Analytical Conditions Required for Packed Injector

Table 9-2. ECD-PKD Analytical Conditions

Parameters Setting	
Gases	Carrier Gas: Helium = 30 kPa Constant Pressure Make-up Gas: Nitrogen = 30 ml/min
Oven Program	IsoTemperature = 140 °C Initial Time = 10 minute
Injector	Operating Mode = Packed Temperature = 180 °C
Detector	Base Temperature = 250 °C ECD temperature = 300 °C Reference Current = 1 nA Pulse Amplitude = 50 V Pulse Width = 1µs
Injected Volume	1 µl + needle of Test Mixture
Analog Signal Output	Chrom-Card Acquisition Frequency = Medium
Digital Signal Output	Chrom-Card, ChromQuest, Atlas, Xcalibur Acquisition Frequency = 10 Hz

Recommended Initial Operations

Before starting the checkout, the following operations should be carried out:

1. Insert the glass liner.
   Remove the column and insert the 2 mm ID tapered glass liner, as required for the checkout, from the bottom of the injector with the appropriate liner seal. Fix the liner by using the appropriate adapter for capillary column.
   Refer to the TRACE GC Ultra Maintenance and Troubleshooting Manual.

2. Mount the adapter for capillary column on the detector base body.

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

4. Connect the required gas lines
   Verify the required gas supplies are properly connected to your GC.

5. Install the precolumn.
   Connect the precolumn to the injector.

6. Install the test column
   Connect the test column to the precolumn by using the press fit connections provided.

7. Perform the manual leak check following the Operating Procedure "*Perforing a Leak Check* " in Chapter 14 of the TRACE GC Ultra Operating Manual.

8. Connect the other end of the test column to the detector base body.

9. Connect your data handling.
   Verify that your data handling is properly connected to your GC system.

OPERATING PROCEDURE

ECD-PKD Checkout

Before beginning, press **CONFIG** to verify the GC configuration:

Left inlet or Right inlet	PKD
Left carrier or Right carrier	He (helium)
Left detector or Right detector	ECD

1. Use **LEFT CARRIER** or **RIGHT CARRIER** to display the appropriate Carrier Gas Control Table. Verify to operate in constant pressure mode. If not, scroll to `Flow mode`, press **MODE/TYPE** to access the selection menu, then select `con pres`. Scrool to `Pressure` and set the pressure value to have the required carrier gas flow rate `Col.flow`.

```
            LEFT CARRIER[1]

 Pressure          30.0   30.0
 Col.flow          3.00
 Lin. veloc.             (60.9)<
```

1. These settings could also be for a right carrier.

2. Use **OVEN** to display the Column Oven Control Table. Set the oven temperature and the Oven Program required.

```
                 OVEN

 Temp                140    140
 Initial Time              10.0
 Ramp 1                     Off
```

3. Use **LEFT INLET** or **RIGHT INLET** to display the appropriate PKD Injector Control Table. Set the required temperature setpoint `Temp`.Verify to operate in **Packed** mode. If not, scroll to `Mode`, press **MODE/TYPE** to access the selection menu, then select `Packed`.

```
     LEFT INLET (PKD)¹

Temp                 180   180
Pressure            30.0  30.0
Mode:                   Packed
```

1. These settings could also be for a right inlet.

4. Use **LEFT DETECTOR** or **RIGHT DETECTOR** to display the appropriate ECD Detector Control Table. Set the required temperature `Base Temp` and the `Mkup` gas required setpoints.

```
    LEFT DETECTOR (ECD)¹

Base temp            250   250
ECD Temp             300   300
Ref current nA             1.0
Freq kHz                 (2.20)
Pulse amp V                 50
Pulse width µs             1.0
Mkup (N2)              30   30<
```

1. These settings could also be for a right detector.

5. Set the `Reference Current` to 1.0 nA.

6. Set the `Pulse Amplitude` to 50 V.

7. Scroll to `Pulse Width` and press **ENTER** to open the menu selection. Select the pulse width to 1µs then press **ENTER**.

8. Observe the ECD frequency value at the display. A base frequency value between 1 kHz and 3 kHz should be displayed. If the ECD frequency value is less than 1kHz, reduce gradually the pulse amplitude till the base frequency value is about 1 kHz (the pulse amplitude value should not be less than 20V). Then, if necessary modify the reference current in order to have a base frequency of about 1-1,5 kHz. Let the detector signal to stabilize.

9. Activate your Data System and set the parameters required for the checkout.

10. Use **LEFT SIGNAL** or **RIGHT SIGNAL** to display the ECD Detector Signal Control Table. Scroll to `Auto zero?` and turn it **YES**.

```
     LEFT SIGNAL (ECD)[1]

Output                     (1000)
Offset                       100<
Auto zero?                   Y/N
Baseline comp?               Off
```

1. These settings could also be for a right signal.

11. Perform a blank analysis injecting pure iso-octane and press **START** on the GC to begin the checkout run.

Baseline Acquisition and Analysis

NOTE Refer to the Acceptance Values reported in the Table 9-3 according to the data handling in use.

12. With the GC in Stand-by/Prep Run condition, activate the data system for 10 minutes to evaluate your baseline in isothermal condition.

13. After the baseline evaluation has been completed, set-up the data system to acquire a single run.

14. When the GC is ready, inject the test mixture and press **START** on the GC to begin the checkout run. The resulting chromatogram should look like the one shown in *Figure 9.1*.

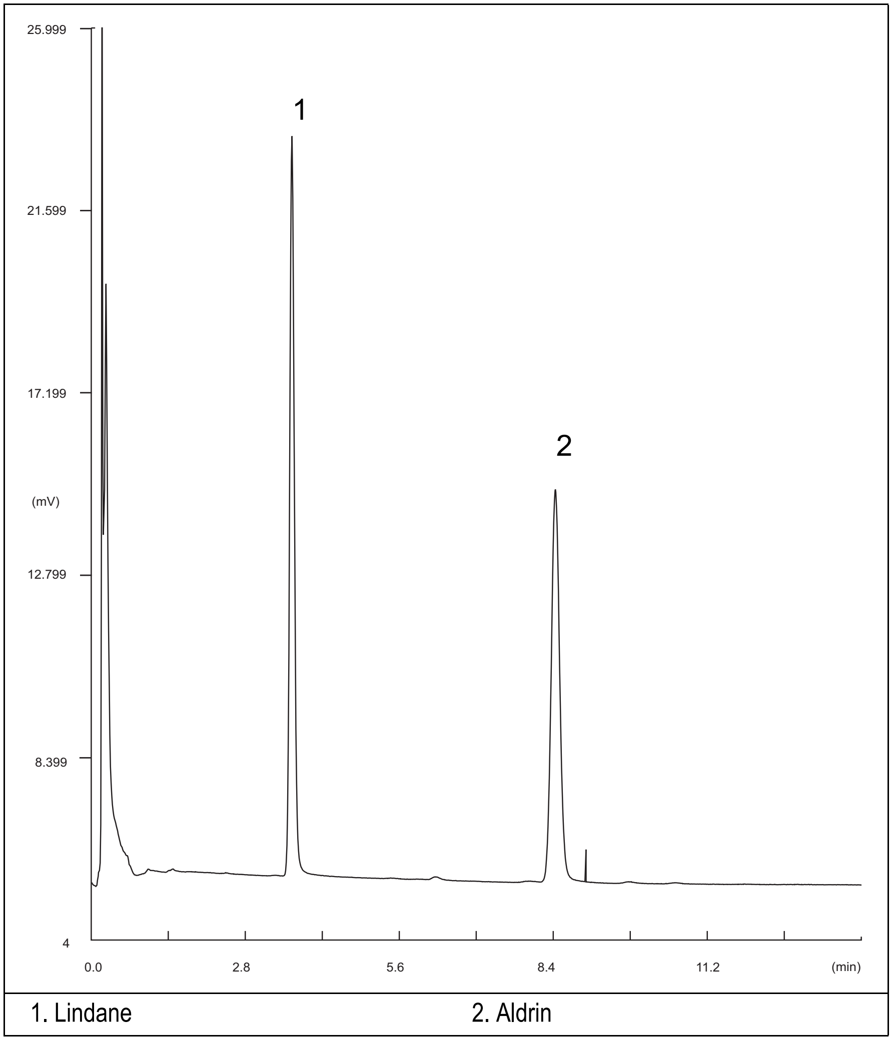


Figure 9-1. ECD-PKD Injection

15. Establish the integration parameters and the peak table identifying the test mix components.

16. Set up the data system to calculate the signal-to-noise ratio.

Using Chrom-Card Data System

Operate as follows:

- Open the Method Editor and include the signal-to-noise Report into the Report Parameters Page.
- By clicking on the side icon, open the signal-to-noise calculation and set Lindane component as signal peak ID.
- Choose a part of baseline without peaks or interference signals and calculate the noise for 0.1 min. (Verify that the noise value is comparable with the one of the previous *Baseline Acquisition and Analysis*).
- Generate a report showing the chromatogram, peak area and signal-to-noise information for Lindane.
- Repeat the procedure to calculate the signal-to-noise ratio also for Aldrin.
- Generate a report showing the chromatogram, peak area and signal-to-noise information for Aldrin.

NOTE If it is not possible to find a suitable part of baseline, to have a complete Chrom-Card final report proceed as follows:
Start the acquisition on the data system. Let the system to acquire for about 1 minute and then inject the test mixture. The noise can be calculated during the first minute of acquisition. Since the retention time will shift by a delayed time, the Component Table has to be updated.

Using non-Chrom-Card Data System

Operate as follows:

- Set the parameters to calculate the signal-to-noise ratio according to the instruction reported in the relevant data system manual.

CAUTION **Choose a part of baseline without peaks or interference signals and calculate the noise for 0.1 min. (Verify that the noise value is comparable with the one of the previous *Baseline Acquisition and Analysis*).**

17. The following criteria indicate successful completion of ECD-PKD checkout.

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

Table 9-3. ECD-PKD Acceptance Criteria

Acceptance Values	CHROM-CARD		
	Baseline Parameters	**Analog** *(1V Full Scale)*	**Digital** *(10V Full Scale)*
	Noise (µV)	< 40	< 400
	Wander (µV)	< 80	< 800
	Drift (µV/h)	< 200	< 2000
	Analytical Results		
	Lindane Signal-to-Noise Ratio		> 2 000
	Aldrin Signal-to-Noise Ratio		> 1 000

Acceptance Values	CHROMQUEST	
	Baseline Parameters *(1V Full Scale)*	
	Noise (µV)	< 40
	Wander (µV)	< 80
	Drift (µV/h)	< 200
	Analytical Results	
	Lindane Signal-to-Noise Ratio	> 2 000
	Aldrin Signal-to-Noise Ratio	> 1 000

	ATLAS	
	Baseline Parameters *(10V Full Scale)*	
	Noise (µV)	< 400
	Wander (µV)	< 800
	Drift (µV/h)	< 2 000
	Analytical Results	
	Lindane Signal-to-Noise Ratio	> 2 000
	Aldrin Signal-to-Noise Ratio	> 1 000
⚠		

Acceptance Values	**XCALIBUR**	
	Baseline Parameters *(Acquisition Frequency = 10 Hz)*	
	Noise (Counts)	< 4 000
	Wander (Counts)	< 8 000
	Drift (Counts/h)	< 20 000
	Analytical Results	
	Lindane Signal-to-Noise Ratio	> 2 000
	Aldrin Signal-to-Noise Ratio	> 1 000
⚠		

Analytical Acceptance Comments	
1	Using Chrom-Card, set the signal-to-noise report parameters as described in the current procedure.
2	Using ChromQuest, Atlas, Xcalibur or a Computing integrator (e.g. ChromJet), calculate the S/N ratio as *Peak Height (counts)/noise (counts)*.

10

Checkout Using ECD with PPKD Injector

Chapter at a Glance...

Operating Procedures

SOP Number: P0311/07/E - 01 September 2009

Scope

Use the following procedure to verify proper ECD operation with Purged Packed Injector.

Parts Referenced

Table 10-1. ECD-PPKD 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	2 mm ID tapered	453 220 50
Liner Seal	Graphite seal for glass liner	290 334 05
Pre-column	Fused Silica Capillary Column 0.5 mt long 0.53 mm ID	260 603 75
Retaining Nut	M4 capillary column retaining nut	350 324 23
Press-fit connections	For columns 0.53/0.32 mm ID	350 438 16
Injection side adapter	For Wide bore column	347 003 03
Retaining Nut	For Injection side adapter	350 024 04
Detector side adapter	For Wide bore column	347 103 04
Ferrule	6 mm ID double brass ferrule	290 341 37
Nut	1/4” G-6 mm ID nut	350 201 18
Graphite Ferrule	Graphite ferrule for 0.53 mm ID Column Graphite ferrule for 0.32 mm ID Column	290 134 86 290 134 87
Septum	Standard septum for Purged Packed Injector	313 032 26
Syringe	10 µl size; 50 mm needle length	365 005 25

Table 10-1. ECD-PPKD Parts Referenced (Continued)

Part	Description	Part Number
Test Mixture	Two components in Iso-octane *Component* *Concentration* Lindane 0.030 µg/ml Aldrin 0.030 µg/ml	338 190 11
Gases	Chromatographic-grade purity	
Data Acquisition	Chrom-Card, ChromQuest, Atlas, Xcalibur, Computing-integrator	

Analytical Conditions Required for Purged Packed Injector

Table 10-2. ECD-PPKD Analytical Conditions

Parameters Setting	
Gases	Carrier Gas: Helium = 30 kPa Constant Pressure Make-up Gas: Nitrogen = 30 ml/min
Oven Program	Initial Temperature = 70 °C Initial Time = 1 minute Ramp 1 = 20 °C/minute Final Temperature = 220 °C Final Time = 1 minute
Injector	Operating Mode = Wide bore Temperature = 200 °C
Detector	Base Temperature = 250 °C ECD temperature = 300 °C Reference Current = 1 nA Pulse Amplitude = 50 V Pulse Width = 1 µs
Injected Volume	1 µl + needle of Test Mixture
Analog Signal Output	Chrom-Card Acquisition Frequency = Medium
Digital Signal Output	Chrom-Card, ChromQuest, Atlas, Xcalibur Acquisition Frequency = 10 Hz

Recommended Initial Operations

Before starting the checkout, the following operations should be carried out:

1. Insert the glass liner.
   Remove the column and insert the 2 mm ID tapered glass liner, as required for the checkout, from the bottom of the injector with the appropriate liner seal. Fix the liner by using the appropriate adapter for capillary column.
   Refer to the TRACE GC Maintenance and Troubleshooting Manual.

2. Mount the adapter for capillary column on the detector base body.

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

4. Connect the required gas lines
   Verify the required gas supplies are properly connected to your GC.

5. Install the precolumn.
   Connect the precolumn to the injector.

6. Install the test column
   Connect the test column to the precolumn by using the press fit connections provided.

7. Perform the manual leak check following the Operating Procedure "*Perforing a Leak Check* " in Chapter 14 of the TRACE GC Ultra Operating Manual.

8. Connect the other end of the test column to the detector base body.

9. Perform Column Evaluation.

10. Connect your data handling.
    Verify that your data handling is properly connected to your GC system.

OPERATING PROCEDURE

ECD-PPKD Checkout

Before beginning, press **CONFIG** to verify the GC configuration:

Left inlet or Right inlet	PPKD
Left carrier or Right carrier	He (helium)
Left detector or Right detector	ECD

1. Use **LEFT CARRIER** or **RIGHT CARRIER** to display the appropriate Carrier Gas Control Table. Verify to operate in constant pressure mode. If not, scroll to `Flow mode`, press **MODE/TYPE** to access the selection menu, then select `con pres`. Scrool to `Pressure` and set the pressure value to have the required carrier gas flow rate `Col.flow`.

```
          LEFT CARRIER[1]

Pressure          30.0   30.0
Col.flow          3.00
Lin. veloc.            (60.9)<
```

1. These settings could also be for a right carrier.

2. Use **OVEN** to display the Column Oven Control Table. Set the oven temperature and the Oven Program required.

```
              OVEN

Temp              70.0  70.0
Initial Time            1.00
Ramp 1                  20.0
Final temp               220
Final time 1            1.00<
Ramp 2                   Off
```

3. Use **LEFT INLET** or **RIGHT INLET** to display the appropriate PPKD Injector Control Table. Set the required temperature setpoint `Temp`. Verify to operate

in **Wide bore** mode. If not, scroll to `Mode`, press **MODE/TYPE** to access the selection menu, then select `Wide bore`.

```
     LEFT INLET (PPKD)[1]

Temp                200   200
Pressure           30.0  30.0
Mode:               Wide bore<
Constant sept purge?        Y<
```

1. These settings could also be for a right inlet.

4. Use **LEFT DETECTOR** or **RIGHT DETECTOR** to display the appropriate ECD Detector Control Table. Set the required temperature `Base Temp` and the `Mkup` gas required setpoints.

```
    LEFT DETECTOR (ECD)[1]

Base temp           250   250
ECD Temp            300   300
Ref current nA            1.0
Freq kHz               (2.20)
Pulse amp V                50
Pulse width µs            1.0
Mkup (N2)             30   30<
```

1. These settings could also be for a right detector.

5. Set the `Reference Current` to 1.0 nA.

6. Set the `Pulse Amplitude` to 50 V.

7. Scroll to `Pulse Width` and press **ENTER** to open the menu selection. Select the pulse width to 1µs then press **ENTER**.

8. Observe the ECD frequency value at the display. A base frequency value between 1 kHz and 3 kHz should be displayed.
   If the ECD frequency value is less than 1kHz, reduce gradually the pulse amplitude till the base frequency value is about 1 kHz (the pulse amplitude value should not be less than 20V). Then, if necessary modify the reference current in order to have a base frequency of about 1-1,5 kHz. Let the detector signal to stabilize.

9. Activate your Data System and set the parameters required for the checkout.

10. Use **LEFT SIGNAL** or **RIGHT SIGNAL** to display the ECD Detector Signal Control Table. Scroll to `Auto zero?` and turn it **YES**.

```
      LEFT SIGNAL (ECD)[1]

Output                  (1000)
Offset                    100<
Auto zero?                Y/N
Baseline comp             Off
```

1. These settings could also be for a right signal.

11. Perform a blank analysis injecting pure iso-octane and press **START** on the GC to begin the checkout run.

Baseline Acquisition and Analysis

NOTE Refer to the Acceptance Values reported in the Table 10-3 according to the data handling in use.

12. With the GC in Stand-by/Prep Run condition, activate the data system for 10 minutes to evaluate your baseline in isothermal condition.

13. After the baseline evaluation has been completed, set-up the data system to acquire a single run.

14. When the GC is ready, inject the test mixture and press **START** on the GC to begin the checkout run. The resulting chromatogram should look like the one shown in *Figure 10.1*.

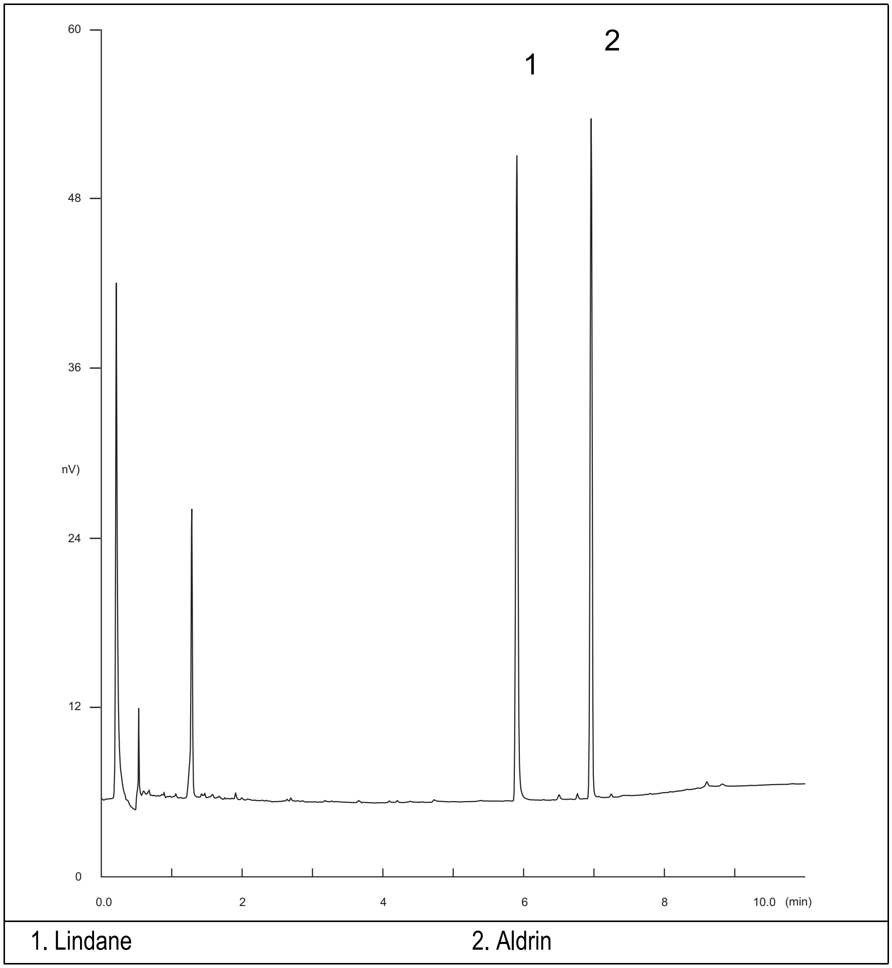


Figure 10-1. ECD-PPKD Injection

15. Establish the integration parameters and the peak table identifying the test mix components.

16. Set up the data system to calculate the signal-to-noise ratio.

Using Chrom-Card Data System

Operate as follows:

- Open the Method Editor and include the signal-to-noise Report into the Report Parameters Page.
- By clicking on the side icon, open the signal-to-noise calculation and set Lindane component as signal peak ID.
- Choose a part of baseline without peaks or interference signals and calculate the noise for 0.1 min. (Verify that the noise value is comparable with the one of the previous *Baseline Acquisition and Analysis*).
- Generate a report showing the chromatogram, peak area and signal-to-noise information for Lindane.
- Repeat the procedure to calculate the signal-to-noise ratio also for Aldrin.
- Generate a report showing the chromatogram, peak area and signal-to-noise information for Aldrin.

NOTE If it is not possible to find a suitable part of baseline, to have a complete Chrom-Card final report proceed as follows:
Start the acquisition on the data system. Let the system to acquire for about 1 minute and then inject the test mixture. The noise can be calculated during the first minute of acquisition. Since the retention time will shift by a delayed time, the Component Table has to be updated.

Using non-Chrom-Card Data System

Operate as follows:

- Set the parameters to calculate the signal-to-noise ratio according to the instruction reported in the relevant data system manual.

CAUTION **Choose a part of baseline without peaks or interference signals and calculate the noise for 0.1 min. (Verify that the noise value is comparable with the one of the previous *Baseline Acquisition and Analysis*).**

17. The following criteria indicate successful completion of ECD-PPKD checkout.

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

Table 10-3. ECD-PPKD Acceptance Criteria

Acceptance Values	CHROM-CARD		
	Baseline Parameters	**Analog** *(1V Full Scale)*	**Digital** *(10V Full Scale)*
	Noise (µV)	< 40	< 400
	Wander (µV)	< 80	< 800
	Drift (µV/h)	< 200	< 2 000
	Analytical Results		
	Lindane Signal-to-Noise Ratio		> 3 000
	Aldrin Signal-to-Noise Ratio		> 3 000

Acceptance Values	CHROMQUEST	
	Baseline Parameters *(1V Full Scale)*	
	Noise (µV)	< 40
	Wander (µV)	< 80
	Drift (µV/h)	< 200
	Analytical Results	
	Lindane Signal-to-Noise Ratio	> 3 000
	Aldrin Signal-to-Noise Ratio	> 3 000

Acceptance Values	ATLAS	
	Baseline Parameters *(10V Full Scale)*	
	Noise (µV)	< 400
	Wander (µV)	< 800
	Drift (µV/h)	< 2 000
	Analytical Results	
	Lindane Signal-to-Noise Ratio	> 3 000
	Aldrin Signal-to-Noise Ratio	> 3 000
⚠		

Acceptance Values	XCALIBUR	
	Baseline Parameters *(Acquisition Frequency = 10 Hz)*	
	Noise (Counts)	< 4 000
	Wander (Counts)	< 8 000
	Drift (Counts/h)	< 20 000
	Analytical Results	
	Lindane Signal-to-Noise Ratio	> 3 000
	Aldrin Signal-to-Noise Ratio	> 3 000
⚠		

Analytical Acceptance Comments	
1	Using Chrom-Card, set the signal-to-noise report parameters as described in the current procedure.
2	Using ChromQuest, Atlas, Xcalibur or a Computing integrator (e.g. ChromJet), calculate the S/N ratio as *Peak Height (counts)/noise (counts)*.

11

Checkout Using ECD with PTV Injector

Chapter at a Glance...

Operating Procedures

SOP Number: P0312/07/E - 01 September 2009

Scope

Use the following procedure to verify proper ECD operation with the Programmable Temperature Vaporizing Injector.

Parts Referenced

Table 11-1. ECD-PTV Parts Referenced

Part	Description	Part Number
Test Column	Fused Silica Capillary ColumnTR-5; 7 m long 0.32 mm ID; 0.25 µm film thickness.	260 800 01
Liner	Silcosteel 2 mm ID (set of 2)	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	365 005 25
Test Mixture	Two components in Iso-octane *Component* *Concentration* Lindane 0.030 µg/ml Aldrin 0.030 µg/ml	338 190 11
Gases	Chromatographic-grade purity	
Data Acquisition	Chrom-Card, ChromQuest, Atlas, Xcalibur, Computing-integrator	

Analytical Conditions Required for PTV Splitless Injection

Table 11-2 ECD-PTV Analytical Conditions

Gases	Carrier Gas: Helium = 30 kPa Constant Pressure Make-up Gas: Nitrogen = 30 ml/min
Oven Program	Initial Temperature = 70 °C Initial Time = 1 minute Ramp 1 = 20 °C/minute Final Temperature = 220 °C Final Time = 1 minute
Injector	Operating Mode = PTV splitless Splitless Time = 0.8 minutes Split Flow = 50 ml/min Constant Septum Purge = Yes Inject Temp = 50 °C Inject Time = 0.1 minute Transfer ramp = 10 °C/sec Transfer Temperature = 260 °C Transfer time = 1 minute
Detector	Base Temperature = 250 °C ECD temperature = 300 °C Reference Current = 1 nA Pulse Amplitude = 50 V Pulse Width = 1µs
Injected Volume	1 µl of Test Mixture
Analog Signal Output	Chrom-Card Acquisition Frequency = Medium
Digital Signal Output	Chrom-Card, ChromQuest, Atlas, Xcalibur Acquisition Frequency = 10 Hz

Recommended Initial Operations

Before starting the checkout, the following operations should be carried out:

1. Replace the liner.
   The liner currently installed in your injector should be carefully removed and replaced with the 2 mm ID Silcosteel liner, 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. Connect the required gas lines
   Verify the required gas supplies are properly connected to your GC.

4. Install the test column.
   The column currently installed should be carefully removed and replaced with the required test column.

5. Perform Column Evaluation and Leak Test.

6. Connect your data handling.
   Verify that your data handling is properly connected to your GC system.

OPERATING PROCEDURE

ECD-PTV Checkout in PTV Splitless Mode

Before beginning, press **CONFIG** to verify the GC configuration:

Right inlet	PTV
Right carrier	He (helium)
Right detector	ECD

1. Use **RIGHT CARRIER** to display the appropriate Carrier Gas Control Table. Verify to operate in constant pressure mode. If not, scroll to `Flow mode`, press **MODE/TYPE** to access the selection menu, then select `con pres`. Scrool to `Pressure` and set the pressure value to have the required carrier gas flow rate `Col.flow`.

```
           RIGHT CARRIER1

Pressure          30.0   30.0
Col.flow          3.00
Lin. veloc.            (60.9)<
```

2. Use **OVEN** to display the Column Oven Control Table. Set the oven temperature and the Oven Program required.

```
              OVEN

Temp              70.0   70.0
Initial Time             1.00
Ramp 1                   20.0
Final temp                220
Final time 1             1.00<
Ramp 2                    Off
```

3. Use **RIGHT INLET** to display the appropriate Programmable Temperature Vaporizing Injector Control Table. Set the required temperature setpoint `Temp`. Verify to operate in **PTV splitless** mode. If not, scroll to `Mode`, press

MODE/TYPE to access the selection menu, then select `PTV splitless`. Scroll to `Splitless time` to set the required setpoint.

```
      RIGHT INLET (PTV)

Temp                   70   70
Pressure             30.0  30.0
Mode:             PTV Splitless
Total flow            (53.0)
Split Flow           50.0  50.0
Splitless time       0.80  0.80
Constant sept purge?          Y
Inject phase menu:            Y<
```

4. Scroll to `Inject phase menu`. Press **MODE/TYPE** to enter the **PTV Phase Menu**.

```
        PTV PHASE MENU

Ramped pressure?              N
Inject temp                  50
Inject time                 0.1
Transfer ramp                10
Transfer temp               260
Transfer time              1.00<
```

5. Select `Ramped pressure?` **NO**.
   Set the required `Inject temp` and `Inject time` setpoints as required. Then, set the `Transfer ramp`, the `Transfer temp` and the `Transfer time` required setpoints.

6. Use **RIGHT DETECTOR** to display the appropriate ECD Detector Control Table. Set the required temperature `Base Temp` and the `Mkup` gas required setpoints.

```
   RIGHT DETECTOR (ECD)

Base temp            250   250
ECD Temp             300   300
Ref current nA             1.0
Freq kHz                 (2.20)
Pulse amp V                 50
Pulse width µs             1.0
Mkup (N2)             30   30<
```

7. Set the `Reference Current` to 1.0 nA.

8. Set the `Pulse Amplitude` to 50 V.

9. Scroll to `Pulse Width` and press **ENTER** to open the menu selection. Select the pulse width to 1µs then press **ENTER**.

10. Observe the ECD frequency value at the display. A base frequency value between 1 kHz and 3 kHz should be displayed.
    If the ECD frequency value is less than 1kHz, reduce gradually the pulse amplitude till the base frequency value is about 1 kHz (the pulse amplitude value should not be less than 20V). Then, if necessary modify the reference current in order to have a base frequency of about 1-1,5 kHz. Let the detector signal to stabilize.

11. Activate your Data System and set the parameters required for the checkout.

12. Use **RIGHT SIGNAL** to display the ECD Detector Signal Control Table. Scroll to `Auto zero?` and turn it **YES**.

```
    RIGHT SIGNAL (ECD)

Output                 (1000)
Offset                   100<
Auto zero?               Y/N
Baseline com’            Off
```

13. Perform a blank analysis injecting pure iso-octane and press **START** on the GC to begin the checkout run.

Baseline Acquisition and Analysis

NOTE Refer to the Acceptance Values reported in the Table 11-3 according to the data handling in use.

14. With the GC in Stand-by/Prep Run condition, activate the data system for 10 minutes to evaluate your baseline in isothermal condition.

15. After the baseline evaluation has been completed, set-up the data system to acquire a single run.

16. When the GC is ready, inject the test mixture and press **START** on the GC to begin the checkout run. The resulting chromatogram should look like the one shown in *Figure 11.1*.

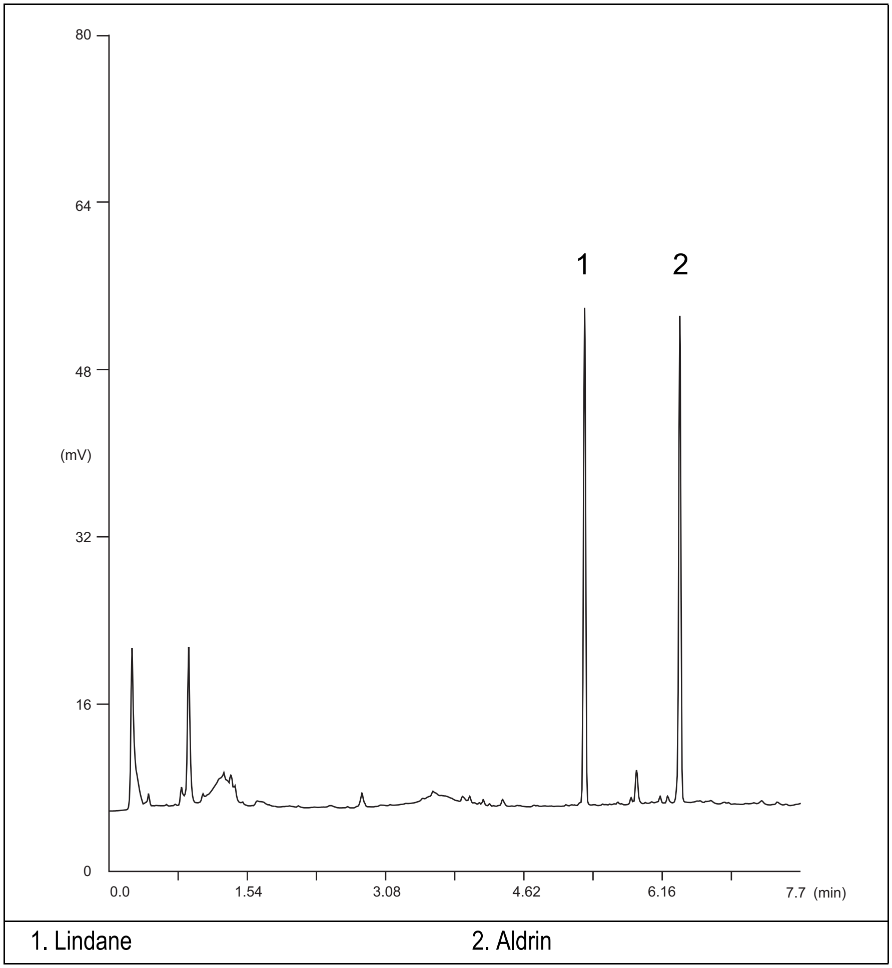


Figure 11-1. ECD-PTV Injection

17. Establish the integration parameters and the peak table identifying the test mix components.

18. Set up the data system to calculate the signal-to-noise ratio.

Using Chrom-Card Data System

Operate as follows:

- Open the Method Editor and include the signal-to-noise Report into the Report Parameters Page.
- By clicking on the side icon, open the signal-to-noise calculation and set Lindane component as signal peak ID.
- Choose a part of baseline without peaks or interference signals and calculate the noise for 0.1 min. (Verify that the noise value is comparable with the one of the previous *Baseline Acquisition and Analysis*).
- Generate a report showing the chromatogram, peak area and signal-to-noise information for Lindane.
- Repeat the procedure to calculate the signal-to-noise ratio also for Aldrin.
- Generate a report showing the chromatogram, peak area and signal-to-noise information for Aldrin.

NOTE If it is not possible to find a suitable part of baseline, to have a complete Chrom-Card final report proceed as follows:
Start the acquisition on the data system. Let the system to acquire for about 1 minute and then inject the test mixture. The noise can be calculated during the first minute of acquisition. Since the retention time will shift by a delayed time, the Component Table has to be updated.

Using non-Chrom-Card Data System

Operate as follows:

- Set the parameters to calculate the signal-to-noise ratio according to the instruction reported in the relevant data system manual.

CAUTION **Choose a part of baseline without peaks or interference signals and calculate the noise for 0.1 min. (Verify that the noise value is comparable with the one of the previous *Baseline Acquisition and Analysis*).**

19. The following criteria indicate successful completion of ECD-PPKD checkout.

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

Table 11-3. ECD-PTV Acceptance Criteria

Acceptance Values	CHROM-CARD		
	Baseline Parameters	**Analog** *(1V Full Scale)*	**Digital** *(10V Full Scale)*
	Noise (µV)	< 40	< 400
	Wander (µV)	< 80	< 800
	Drift (µV/h)	< 200	< 2000
	Analytical Results		
	Lindane Signal-to-Noise Ratio		> 3 000
	Aldrin Signal-to-Noise Ratio		> 3 000

Acceptance Values	CHROMQUEST	
	Baseline Parameters *(1V Full Scale)*	
	Noise (µV)	< 40
	Wander (µV)	< 80
	Drift (µV/h)	< 200
	Analytical Results	
	Lindane Signal-to-Noise Ratio	> 3 000
	Aldrin Signal-to-Noise Ratio	> 3 000

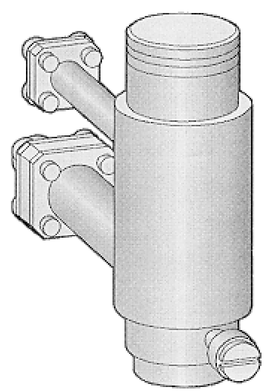
	ATLAS	
	Baseline Parameters *(10V Full Scale)*	
	Noise (µV)	< 400
	Wander (µV)	< 800
	Drift (µV/h)	< 2 000
	Analytical Results	
	Lindane Signal-to-Noise Ratio	> 3 000
	Aldrin Signal-to-Noise Ratio	> 3 000
⚠		

Acceptance Values	XCALIBUR	
	Baseline Parameters *(Acquisition Frequency = 10 Hz)*	
	Noise (Counts)	< 4 000
	Wander (Counts)	< 8 000
	Drift (Counts/h)	< 20 000
	Analytical Results	
	Lindane Signal-to-Noise Ratio	> 3 000
	Aldrin Signal-to-Noise Ratio	> 3 000
⚠		

Analytical Acceptance Comments	
1	Using Chrom-Card, set the signal-to-noise report parameters as described in the current procedure.
2	Using ChromQuest, Atlas, Xcalibur or a Computing integrator (e.g. ChromJet), calculate the S/N ratio as *Peak Height (counts)/noise (counts)*.

SECTION IV

SOPs Using NPD



The *SOPs Using NPD* section, contains the procedures to test the TRACE GC with the Nitrogen Phosphorus Detector (NPD) using different injectors.

12

Checkout Using NPD with S/SL Injector

Chapter at a Glance...

Operating Procedures

SOP Number: P0296/08/E - 01 September 2009

Scope

Use the following procedure to verify proper NPD operation with the Split/Splitless Injector.

Parts Referenced

Table 12-1. NPD-S/SL 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; 70 mm needle length	365 001 03
Test Mixture	Three components in Iso-Octane: *Component* *Concentration* Azobenzene 1 µg/ml Octadecane 1000 µg/ml Parathion methyl 1 µg/ml	338 190 06
Gases	Chromatographic-grade purity	
Data Acquisition	Chrom-Card, ChromQuest, Atlas, Xcalibur, Computing-integrator	

Analytical Conditions Required for Splitless Injection

Table 12-2. NPD-S/SL Analytical Conditions

Parameters Setting	
Gases	Carrier Gas: Helium = 30 kPa Constant Pressure Hydrogen = 2.3 ml/min Air = 60 ml/min Make-up: Nitrogen = 15 ml/min
Oven Program	Initial Temperature = 70 °C Initial Time = 1 minute Ramp 1 = 20 °C/minute Final Temperature = 230 °C Final Time = 1 minute
Injector	Operating Mode = Splitless Temperature = 230 °C Splitless Time = 0.8 minutes Split Flow = 60 ml/min Constant Septum Purge = Yes
Detector	Base Temperature = 300 °C Source Current = *Refer to Source Ignition* Polarizer voltage = 3.5 V Detector Signal Range = 10^0
Injected Volume	1 µl + needle of Test Mixture
Analog Signal Output	Chrom-Card Acquisition Frequency = Medium
Digital Signal Output	Chrom-Card, ChromQuest, Atlas, Xcalibur Acquisition Frequency = 10 Hz

Recommended Initial Operations

Before starting the checkout, the following operations should be carried out:

1. Replace the glass liner.
   The glass liner currently installed in your injector should be carefully removed and replaced with the 3 mm ID glass liner for splitless application, as required for the checkout, with the appropriate graphite seal.

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

3. Connect the required gas lines
   Verify the required gas supplies are properly connected to your GC.

4. Install the test column.
   The column currently installed should be carefully removed and replaced with the required test column.

5. Perform Column Evaluation and Leak Test

6. Connect your data handling.
   Verify that your data handling is properly connected to your GC system.

OPERATING PROCEDURE

NPD-S/SL Checkout in Splitless Mode

Before beginning, press **CONFIG** to verify the GC configuration:

Left inlet or Right inlet	S/SL
Left carrier or Right carrier	He (helium)
Left detector or Right detector	NPD

1. Use **LEFT CARRIER** or **RIGHT CARRIER** to display the appropriate Carrier Gas Control Table. Verify to operate in constant pressure mode. If not, scroll to `Flow mode`, press **MODE/TYPE** to access the selection menu, then select `con pres`. Scrool to `Pressure` and set the pressure value to have the required carrier gas flow rate `Col.flow`.

```
        LEFT CARRIER[1]

Pressure        30.0    30.0
Col.flow        3.00
Lin. veloc.            (60.9)<
```

1. These settings could also be for a right carrier.

2. Use **OVEN** to display the Column Oven Control Table. Set the oven temperature and the Oven Program required.

```
              OVEN

Temp              70.0   70.0
Initial Time             1.00
Ramp 1                   20.0
Final temp                230
Final time 1             1.00<
Ramp 2                    Off
```

3. Use **LEFT INLET** or **RIGHT INLET** to display the appropriate Split/Splitless Injector Control Table and set the required temperature setpoint *Temp*. Verify to operate in **Splitless** mode. If not, scroll to `Mode`, press **MODE/TYPE** to access the selection menu, then select `Splitless`. Scroll to `Splitless time` to set the required setpoint.

LEFT INLET (S/SL)[1]		
Temp	230	230
Pressure	30.0	30.0
Mode:		Splitless
Total flow		(63.0)
Split Flow	60.0	60.0
Splitless time	0.80	0.80
Constant sept purge?		Y<

1. These settings could also be for a right inlet.

4. Use **LEFT DETECTOR** or **RIGHT DETECTOR** to display the appropriate NPD Detector Control Table. .

LEFT DETECTOR (NPD)[1]		
Source cur,A		OFF
Base temp	300	300
Signal pA		(10.4)
Target curr. pA		(X.XX)
Auto adjust		No
Polarizer V		3.5
H2 delay time		Off
H2	2.3	2.3
Air	60	60
Mkup N2	15	15

1. These settings could also be for a right detector.

5. Scroll to `Polarizer V` and set 3.5 V.

Source Ignition

6. Switch on the source operating as follows:

   a. Open the detector gases `H2`, `Air` and `Mkup` and set the gas flow rates as follows:

      - `H2` = 2.3 ml/min
      - `Air` = 60 ml/min
      - `Mkup N2` = 15 ml/min)

   b. Increase the `Base Temp` to 300°C and wait that the NPD cell reaches the correct set temperature.

   c. Be sure that the backoff signal is between 0 and 0.5 pA.

   d. Switch on the source with an initial current of 2.50 A. The backoff signal can slightly increase, but should remain within 0 and 1.5 pA.

   e. Monitor the signal through the keypad or through the data system, increase the current value by steps of 0.002 A, until an immediate and strong increase of the signal is observed.

   f. Wait five minutes to let the source stabilizes.

   g. Check that source is correctly switched on decreasing hydrogen flow to 0.5 ml/min until signal decreases down to zero, then increase again to original value.

      - If the signal remains around zero, it means that the source is not switched on and it is necessary to increase further the current, accordingly to the procedure just described.
      - If the signal rises back to original value, it means that source is correctly switched on

   h. Increase the current value of 2% of the actual ignition current. Let the signal stabilizes until its level drops below 20 pA.

7. Use **LEFT SIGNAL** or **RIGHT SIGNAL** to display the appropriate NPD Detector Signal Control Table. Scroll to `Range` and set the electrometer amplifier input range required.

LEFT SIGNAL (NPD)[1]	
Output	(1000)
Offset	100
Auto zero?	Y/N
Range 10^(0..3)	0<
Analog filter	Off
Baseline comp	Off

1. These settings could also be for a right signal.

8. Activate your Data System and set the parameters required for the checkout.

9. In the NPD Detector Signal Control Table scroll to `Auto zero?` and turn it **YES**.

10. Perform a blank analysis injecting pure iso-octane and press **START** on the GC to begin the checkout run.

Baseline Acquisition and Analysis

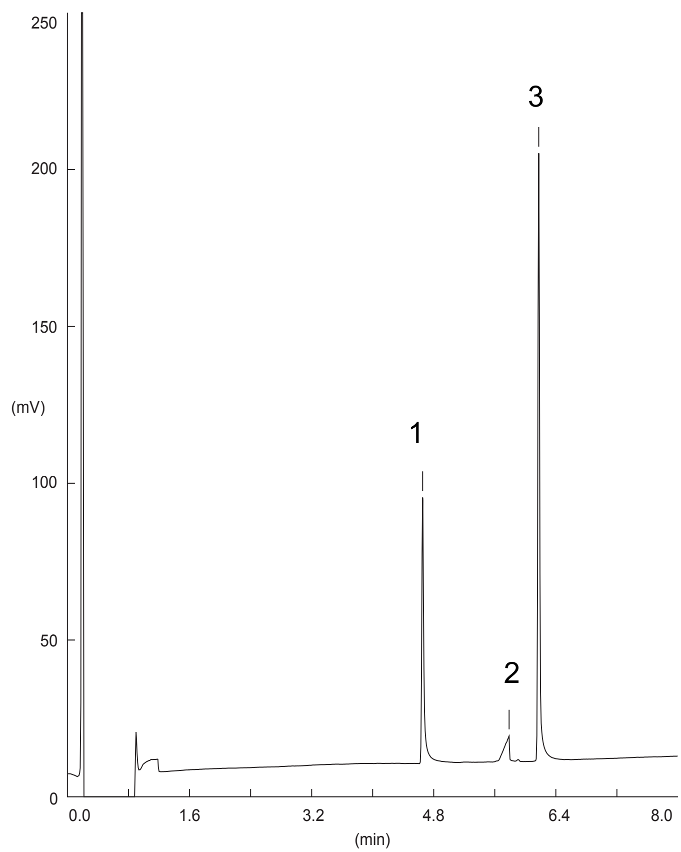
NOTE Refer to the Acceptance Values reported in the Table 12-3 according to the data handling in use.

11. With the GC in Stand-by/Prep Run condition, activate the data system for 10 minutes to evaluate your baseline in isothermal condition.

12. After the baseline evaluation has been completed, set-up the data system to acquire a single run.

13. Inject the test mixture and press **START** on the GC to begin the checkout run. The resulting chromatogram should look like the one shown in *Figure 12.1*.

CAUTION **Verify the peak shape. If any peak distortion is visible, change the analytical test column.**



1. Azobenzene 2. Octadecane 3. Parathion Methyl

Figure 12-1. NPD-Splitless Injection

14. Establish the integration parameters and the peak table identifying the test mix components.

15. Set up the data system to calculate the signal-to-noise ratio.

Using Chrom-Card Data System

Operate as follows:

- Open the Method Editor and include the signal-to-noise Report into the Report Parameters Page.
- By clicking on the side icon, open the signal-to-noise calculation and set Azobenzene component as signal peak ID.
- Choose a part of baseline without peaks or interference signals and calculate the noise for 0.1 min. (Verify that the noise value is comparable with the one of the previous *Baseline Acquisition and Analysis*).
- Generate a report showing the chromatogram, peak area and signal-to-noise information for Azobenzene.
- Repeat the procedure to calculate the signal-to-noise ratio also for Parathion Methyl.
- Generate a report showing the chromatogram, peak area and signal-to-noise information for Parathion Methyl.

NOTE If it is not possible to find a suitable part of baseline, to have a complete Chrom-Card final report proceed as follows:
Start the acquisition on the data system. Let the system to acquire for about 1 minute and then inject the test mixture. The noise can be calculated during the first minute of acquisition. Since the retention time will shift by a delayed time, the Component Table has to be updated.

Using non-Chrom-Card Data System

Operate as follows:

- Set the parameters to calculate the signal-to-noise ratio according to the instruction reported in the relevant data system manual.

CAUTION **Choose a part of baseline without peaks or interference signals and calculate the noise for 0.1 min. (Verify that the noise value is comparable with the one of the previous *Baseline Acquisition and Analysis*).**

16. The following criteria indicate successful completion of NPD-S/SL checkout.

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

Table 12-3. NPD-S/SL Acceptance Criteria

Acceptance Values	CHROM-CARD		
	Baseline Parameters	**Analog** *(1V Full Scale)*	**Digital** *(10V Full Scale)*
	Noise (µV)	< 40	< 400
	Wander (µV)	< 100	< 1 000
	Drift (µV/h)	< 300	<3 000
	Analytical Results		
	Azobenzene Signal-to-Noise Ratio		> 800
	Parathion Methyl Signal-to-Noise Ratio		> 2 500
	Octadecane Signal-to-Noise Ratio		Negligible

Computing-integrator (e.g. ChromJet)	

Acceptance Values	CHROMQUEST	
	Baseline Parameters *(1V Full Scale)*	
	Noise (µV)	< 40
	Wander (µV)	< 100
	Drift (µV/h)	< 300
	Analytical Results	
	Azobenzene Signal-to-Noise Ratio	> 800
	Parathion Methyl Signal-to-Noise Ratio	> 2 500
	Octadecano Signal-to-Noise Ratio	Negligible

ATLAS	
Baseline Parameters *(10V Full Scale)*	
Noise (µV)	< 400
Wander (µV)	< 1 000
Drift (µV/h)	< 3 000
Analytical Results	
Azobenzene Signal-to-Noise Ratio	> 800
Parathion Methyl Signal-to-Noise Ratio	> 2 500
Octadecano Signal-to-Noise Ratio	Negligible

Acceptance Values

XCALIBUR	
Baseline Parameters *(Acquisition Frequency = 10 Hz)*	
Noise (Counts)	< 4 000
Wander Counts)	< 10 000
Drift (Counts/h)	< 30 000
Analytical Results	
Azobenzene Signal-to-Noise Ratio	> 800
Parathion Methyl Signal-to-Noise Ratio	> 2 500
Octadecano Signal-to-Noise Ratio	Negligible

13

Checkout Using NPD with OC Injector

Chapter at a Glance...

Operating Procedures

SOP Number: P0297/08/E - 01 September 2009

Scope

Use the following procedure to verify proper NPD operation with the On-Column Injector.

Parts Referenced

Table 13-1. NPD-OCI 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
Vespel Ferrule	Vespel Ferrule for 0.32 mm ID Column	290 134 60
Retaining Nut	M4 capillary column retaining nut	350 324 23
Syringe	10 µl size; 75 mm needle length	365 020 07
Test Mixture	Three components in Iso-Octane: *Component* *Concentration* Azobenzene 1 µg/ml Octadecane 1000 µg/ml Parathion methyl 1 µg/ml	338 190 06
Gases	Chromatographic-grade purity	
Data Acquisition	Chrom-Card, ChromQuest, Atlas, Xcalibur, Computing-integrator	
Syringe	10 µl size; 80 mm needle length	365 020 19
Pre-column	2 m long; 0.53 mm ID	260 603 75
Press-fit set	Set of Press-fir connectors for TRACE OC	350 038 45

Analytical Conditions Required for On-Column Injection

Table 13-2. NPD-OCI Analytical Conditions

Gases	Carrier Gas: Helium = 30 kPa Constant Pressure Hydrogen = 2.3 ml/min Air = 60 ml/min Make-up: Nitrogen = 15 ml/min
Oven Program	Initial Temperature = 85 °C Initial Time = 1 minute Ramp 1 = 20 °C/minute Final Temperature = 230 °C Final Time = 1 minute
Injector	Secondary cooling = 0.2 minutes
Detector	Base Temperature = 300 °C Source Current = *Refer to Source Ignition* Polarizer voltage = 3.5 V Detector Signal Range = 10^0
Injected Volume	1 µl of Test Mixture
Analog Signal Output	Chrom-Card Acquisition Frequency = Medium
Digital Signal Output	Chrom-Card, ChromQuest, Atlas, Xcalibur Acquisition Frequency = 10 Hz

Recommended Initial Operations

Before starting the checkout, the following operations should be carried out:

1. Connect the required gas lines
   Verify the required gas supplies are properly connected to your GC.

2. Install the test column.
   The column currently installed should be carefully removed and replaced with the required test column.
   In case of automatic On-column for TriPlus sampler, install the pre-column and connect it to the test column by press-fit connector.

3. Install and connect the TriPlus sampler and its components.

4. Perform Column Evaluation and Leak Test.

5. Connect your data handling.
   Verify that your data handling is properly connected to your GC system.

6. Verify the opening/closing of the OC injector actuator by using the proper commands.

7. Verify the alignment of the syringe on the OC injector.

OPERATING PROCEDURE

NPD-OCI Checkout in On-Column Mode

Before beginning, press **CONFIG** to verify the GC configuration:

Left inlet or Right inlet	OCI
Left carrier or Right carrier	He (helium)
Left detector or Right detector	NPD

1. Use **LEFT CARRIER** or **RIGHT CARRIER** to display the appropriate Carrier Gas Control Table. Verify to operate in constant pressure mode. If not, scroll to `Flow mode`, press **MODE/TYPE** to access the selection menu, then select `con pres`. Scrool to `Pressure` and set the pressure value to have the required carrier gas flow rate `Col.flow`.

```
        LEFT CARRIER[1]

Pressure          30.0   30.0
Col.flow          3.00
Lin. veloc.             (60.9)<
```

1. These settings could also be for a right carrier.

2. Use **OVEN** to display the Column Oven Control Table. Set the oven temperature and the Oven Program required.

```
              OVEN

Temp              85.0   85.0
Initial Time             1.00
Ramp 1                   20.0
Final temp                230
Final time 1             1.00<
Ramp 2                    Off
```

3. Use **LEFT INLET** or **RIGHT INLET** to display the appropriate On-Column Injector Control Table. Scroll to `Sec.cool time` and set the required secondary cooling time.

LEFT INLET (OCI)[1]		
Pressure	30.0	30.0
Sec. Cool Time		0.2<

1. These settings could also be for a right inlet.

4. Use **LEFT DETECTOR** or **RIGHT DETECTOR** to display the appropriate NPD Detector Control Table. Set the required temperature `Base Temp` and the detector gases `H2`, `Air` and `Mkup` required setpoints.

LEFT DETECTOR (NPD)[1]		
Source cur,A		Off
Base temp	300	300
Signal pA		(10.4)
Target curr. pA		(X.XX)
Auto adjust		No
Polarizer V		3.5
H2 delay time		Off
H2	2.3	2.3
Air	60	60
Mkup N2	15	15

1. These settings could also be for a right detector.

5. Scroll to `Polarizer V` and set 3.5 V.

Source Ignition

6. Switch on the source operating as follows:

   a. Open the detector gases `H2`, `Air` and `Mkup` and set the gas flow rates as follows:

- `H2` = 2.3 ml/min
- `Air` = 60 ml/min
- `Mkup N2` = 15 ml/min

b. Increase the `Base Temp` to 300°C and wait that the NPD cell reaches the correct set temperature.

c. Be sure that the backoff signal is between 0 and 0.5 pA.

d. Switch on the source with an initial current of 2.50 A. The backoff signal can slightly increase, but should remain within 0 and 1.5 pA.

e. Monitor the signal through the keypad or through the data system, increase the current value by steps of 0.002 A, until an immediate and strong increase of the signal is observed.

f. Wait five minutes to let the source stabilizes.

g. Check that source is correctly switched on decreasing hydrogen flow to 0.5 ml/min until signal decreases down to zero, then increase again to original value.

  - If the signal remains around zero, it means that the source is not switched on and it is necessary to increase further the current, accordingly to the procedure just described.
  - If the signal rises back to original value, it means that source is correctly switched on

h. Increase the current value of 2% of the actual ignition current. Let the signal stabilizes until its level drops below 20 pA.

7. Use **LEFT SIGNAL** or **RIGHT SIGNAL** to display the appropriate NPD Detector Signal Control Table. Scroll to `Range` and set the electrometer amplifier input range required.

LEFT SIGNAL (NPD)[1]	
Output	(1000)
Offset	100
Autozero?	Y/N
Range 10^(0..3)	0<
Analog filter	Off
Baseline comp	Off

1. These settings could also be for a right signal.

8. Activate your Data System and set the parameters required for the checkout.

9. In the NPD Detector Signal Control Table scroll to `Auto zero?` and turn it **YES**.

10. Perform a blank analysis injecting pure iso-octane and press **START** on the GC to begin the checkout run.

Baseline Acquisition and Analysis

NOTE Refer to the Acceptance Values reported in the Table 13-3 according to the data handling in use.

11. With the GC in Stand-by/Prep Run condition, activate the data system for 10 minutes to evaluate your baseline in isothermal condition.

12. After the baseline evaluation has been completed, set-up the data system to acquire a single run.

13. Perform the analysis.

    Manual injection

    - Inject the test mixture and press **START** on the GC to begin the checkout run.

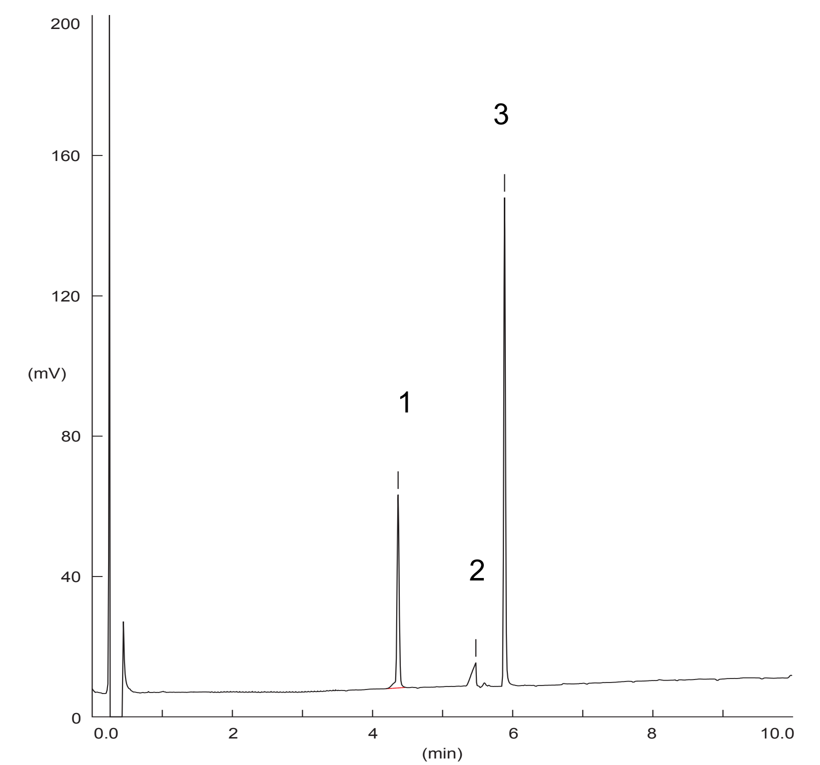
    Automatic injection with TriPlus sampler

    - Fill a vial with the standard mix and place that vial in the sample tray.

- Load the method for OC and perform the sampling.

The resulting chromatogram should look like the one shown in *Figure 7.1*.

CAUTION **Verify the peak shape. If any peak distortion is visible, change the analytical test column.**



1. Azobenzene 2. Octadecane 3. Parathion Methyl

Figure 13-1. NPD-On-Column Injection

14. Establish the integration parameters and the peak table identifying the test mix components.

15. Set up the data system to calculate the signal-to-noise ratio.

Using Chrom-Card Data System

Operate as follows:

- Open the Method Editor and include the signal-to-noise Report into the Report Parameters Page.
- By clicking on the side icon, open the signal-to-noise calculation and set Azobenzene component as signal peak ID.
- Choose a part of baseline without peaks or interference signals and calculate the noise for 0.1 min. (Verify that the noise value is comparable with the one of the previous *Baseline Acquisition and Analysis*).
- Generate a report showing the chromatogram, peak area and signal-to-noise information for Azobenzene.
- Repeat the procedure to calculate the signal-to-noise ratio also for Parathion Methyl.
- Generate a report showing the chromatogram, peak area and signal-to-noise information for Parathion Methyl.

NOTE If it is not possible to find a suitable part of baseline, to have a complete Chrom-Card final report proceed as follows:
Start the acquisition on the data system. Let the system to acquire for about 1 minute and then inject the test mixture. The noise can be calculated during the first minute of acquisition. Since the retention time will shift by a delayed time, the Component Table has to be updated.

Using non-Chrom-Card Data System

Operate as follows:

- Set the parameters to calculate the signal-to-noise ratio according to the instruction reported in the relevant data system manual.

CAUTION **Choose a part of baseline without peaks or interference signals and calculate the noise for 0.1 min. (Verify that the noise value is comparable with the one of the previous *Baseline Acquisition and Analysis*).**

16. The following criteria indicate successful completion of NPD-OCI checkout. If these criteria are not met, repeat the test.

Table 13-3. NPD-OCI Acceptance Criteria

	CHROM-CARD		
Acceptance Values	**Baseline Parameters**	**Analog** *(1V Full Scale)*	**Digital** *(10V Full Scale)*
	Noise (µV)	< 40	< 400
	Wander (µV)	< 100	< 1 000
	Drift (µV/h)	< 300	<3 000
	Analytical Results		
	Azobenzene Signal-to-noise ratio		> 550
	Parathion Methyl Signal-to-noise ratio		> 1 500
	Octadecane Signal-to-noise ratio		Negligible
⚠			

Computing-integrator (e.g. ChromJet)	
⚠	

	CHROMQUEST	
Acceptance Values	**Baseline Parameters** *(1V Full Scale)*	
	Noise (µV)	< 40
	Wander (µV)	< 100
	Drift (µV/h)	< 300
	Analytical Results	
	Azobenzene Signal-to-noise ratio	> 550
	Parathion Methyl Signal-to-noise ratio	> 1 500
	Octadecane Signal-to-noise ratio	Negligible
⚠		

ATLAS	
Baseline Parameters *(10V Full Scale)*	
Noise (µV)	< 400
Wander (µV)	< 1 000
Drift (µV/h)	< 3 000
Analytical Results	
Azobenzene Signal-to-noise ratio	> 550
Parathion Methyl Signal-to-noise ratio	> 1 500
Octadecane Signal-to-noise ratio	Negligible
⚠	

Acceptance Values

XCALIBUR	
Baseline Parameters *(Acquisition Frequency = 10 Hz)*	
Noise (Counts)	< 4 000
Wander Counts)	< 10 000
Drift (Counts/h)	< 30 000
Analytical Results	
Azobenzene	> 550
Parathion Methyl	> 1 500
Octadecane	Negligible
⚠	

14

Checkout Using NPD with PKD Injector

Chapter at a Glance…

Operating Procedures

SOP Number: P0313/08/E - 01 September 2009

Scope

Use the following procedure to verify proper NPD operation with Packed Injector.

Parts Referenced

Table 14-1. NPD-PKD 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	2 mm ID tapered	453 220 50
Liner Seal	Graphite seal for glass liner	290 334 05
Pre-column	Fused Silica Capillary Column 0.5 mt long 0.53 mm ID	260 603 75
Retaining Nut	M4 capillary column retaining nut	350 324 23
Press-fit connections	For columns 0.53/0.32 mm ID	350 438 16
Injection side adapter	For Wide bore column	347 003 03
Retaining Nut	For Injection side adapter	350 024 04
Detector side adapter	For Wide bore column	347 103 04
Ferrule	6 mm ID double brass ferrule	290 341 37
Nut	1/4” G-6 mm ID nut	350 201 18
Graphite Ferrule	Graphite ferrule for 0.53 mm ID Column Graphite ferrule for 0.32 mm ID Column	290 134 86 290 134 87
Septum	Standard septum for Packed Injector	313 032 26
Syringe	10 µl size; 50 mm needle length	365 005 25

Table 14-1. NPD-PKD Parts Referenced (Continued)

Part	Description	Part Number
Test Mixture	Three components in Iso-Octane: *Component* *Concentration* Azobenzene 1 µg/ml Octadecane 1000 µg/ml Parathion methyl 1 µg/ml	338 190 06
Gases	Chromatographic-grade purity	
Data Acquisition	Chrom-Card, ChromQuest, Atlas, Xcalibur, Computing-integrator	

Analytical Conditions Required for Packed Injector

Table 14-2. NPD-PKD Analytical Conditions

Parameters Setting	
Gases	Carrier Gas: Helium = 30 kPa Constant Pressure Hydrogen = 2.3 ml/min Air = 60 ml/min Make-up Gas: Nitrogen = 15 ml/min
Oven Program	IsoTemperature = 50 °C Initial Time = 1 minute Ramp 1 = 20 °C/minute Final Temperature = 200 °C Final Time = 1 minute
Injector	Operating Mode = Packed Temperature = 200 °C
Detector	Base Temperature = 300 °C Source Current = *Refer to Source Ignition* Polarizer voltage = 3.5 V Detector Signal Range = 10^0
Injected Volume	1 µl + needle of Test Mixture
Analog Signal Output	Chrom-Card Acquisition Frequency = Medium
Digital Signal Output	Chrom-Card, ChromQuest, Atlas, Xcalibur Acquisition Frequency = 10 Hz

Recommended Initial Operations

Before starting the checkout, the following operations should be carried out:

1. Insert the glass liner.
   Remove the column and insert the 2 mm ID tapered glass liner, as required for the checkout, from the bottom of the injector with the appropriate liner seal. Fix the liner by using the appropriate adapter for capillary column. Refer to the TRACE GC Ultra Maintenance and Troubleshooting Manual.

2. Mount the adapter for capillary column on the detector base body.

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

4. Connect the required gas lines
   Verify the required gas supplies are properly connected to your GC.

5. Install the precolumn.
   Connect the precolumn to the injector.

6. Install the test column
   Connect the test column to the precolumn by using the press fit connections provided.

7. Perform the manual leak check following the Operating Procedure “*Perforing a Leak Check* ” in Chapter 14 of the TRACE GC Ultra Operating Manual.

8. Connect the other end of the test column to the detector base body.

9. Connect your data handling.
   Verify that your data handling is properly connected to your GC system.

OPERATING PROCEDURE

NPD-PKD Checkout

Before beginning, press **CONFIG** to verify the GC configuration:

Left inlet or Right inlet	PKD
Left carrier or Right carrier	He (helium)
Left detector or Right detector	NPD

1. Use **LEFT CARRIER** or **RIGHT CARRIER** to display the appropriate Carrier Gas Control Table. Verify to operate in constant pressure mode. If not, scroll to `Flow mode`, press **MODE/TYPE** to access the selection menu, then select `con pres`. Scrool to `Pressure` and set the pressure value to have the required carrier gas flow rate `Col.flow`.

```
          LEFT CARRIER[1]

  Pressure        30.0   30.0
  Col.flow        3.00
  Lin. veloc.           (60.9)<
```

1. These settings could also be for a right carrier.

2. Use **OVEN** to display the Column Oven Control Table. Set the oven temperature and the Oven Program required.

```
               OVEN

  Temp            50.0  50.0
  Initial Time          1.00
  Ramp 1                20.0
  Final temp             200
  Final time 1          1.00<
  Ramp 2                 Off
```

3. Use **LEFT INLET** or **RIGHT INLET** to display the appropriate PKD Injector Control Table. Set the required temperature setpoint `Temp`. Verify to operate

in **Packed** mode. If not, scroll to `Mode`, press **MODE/TYPE** to access the selection menu, then select `Packed`.

LEFT INLET (PKD)[1]		
Temp	200	200
Pressure	30.0	30.0
Mode:		Packed

1. These settings could also be for a right inlet.

4. Use **LEFT DETECTOR** or **RIGHT DETECTOR** to display the appropriate NPD Detector Control Table. Set the required temperature `Base Temp` and the detector gases `H2`, `Air` and `Mkup` required setpoints.

LEFT DETECTOR (NPD)[1]		
Source cur,A		Off
Base temp	300	300
Signal pA		(10.4)
Target curr. pA		(X.XX)
Auto adjust		No
Polarizer V		3.5
H2 delay time		Off
H2	2.3	2.3
Air	60	60
Mkup N2	15	15

1. These settings could also be for a right detector.

5. Scroll to `Polarizer V` and set 3.5 V.

Source Ignition

6. Switch on the source operating as follows:

   a. Open the detector gases `H2`, `Air` and `Mkup` and set the gas flow rates as follows:

- `H2` = 2.3 ml/min
- `Air` = 60 ml/min
- `Mkup N2` = 15 ml/min)

b. Increase the `Base Temp` to 300°C and wait that the NPD cell reaches the correct set temperature.

c. Be sure that the backoff signal is between 0 and 0.5 pA.

d. Switch on the source with an initial current of 2.50 A. The backoff signal can slightly increase, but should remain within 0 and 1.5 pA.

e. Monitor the signal through the keypad or through the data system, increase the current value by steps of 0.002 A, until an immediate and strong increase of the signal is observed.

f. Wait five minutes to let the source stabilizes.

g. Check that source is correctly switched on decreasing hydrogen flow to 0.5 ml/min until signal decreases down to zero, then increase again to original value.

- If the signal remains around zero, it means that the source is not switched on and it is necessary to increase further the current, accordingly to the procedure just described.
- If the signal rises back to original value, it means that source is correctly switched on

h. Increase the current value of 2% of the actual ignition current. Let the signal stabilizes until its level drops below 20 pA.

7. Use **LEFT SIGNAL** or **RIGHT SIGNAL** to display the appropriate NPD Detector Signal Control Table. Scroll to `Range` and set the electrometer amplifier input range required.

LEFT SIGNAL (NPD)[1]	
Output	(1000)
Offset	100
Auto zero?	Y/N
Range 10^(0..3)	0<
Analog filter	Off
Baseline comp	Off

1. These settings could also be for a right signal.

8. Activate your Data System and set the parameters required for the checkout.

9. In the NPD Detector Signal Control Table scroll to `Auto zero?` and turn it **YES**.

10. Perform a blank analysis injecting pure iso-octane and press **START** on the GC to begin the checkout run.

Baseline Acquisition and Analysis

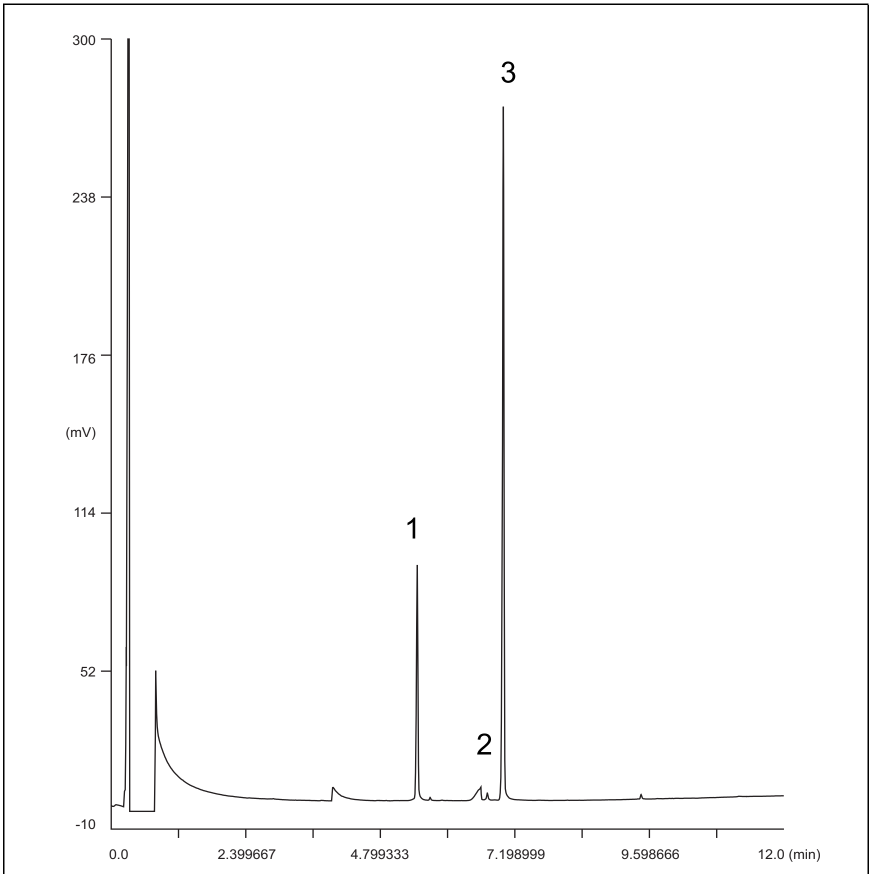
NOTE Refer to the Acceptance Values reported in the Table 14-3 according to the data handling in use.

11. With the GC in Stand-by/Prep Run condition, activate the data system for 10 minutes to evaluate your baseline in isothermal condition.

12. After the baseline evaluation has been completed, set-up the data system to acquire a single run.

13. Inject the test mixture and press **START** on the GC to begin the checkout run. The resulting chromatogram should look like the one shown in *Figure 14.1*.

CAUTION **Verify the peak shape. If any peak distortion is visible, change the analytical test column.**



1. Azobenzene 2. Octadecane 3. Parathion Methyl

Figure 14-1. NPD-Packed Injection

14. Establish the integration parameters and the peak table identifying the test mix components.

15. Set up the data system to calculate the signal-to-noise ratio.

Using Chrom-Card Data System

Operate as follows:

- Open the Method Editor and include the signal-to-noise Report into the Report Parameters Page.
- By clicking on the side icon, open the signal-to-noise calculation and set Azobenzene component as signal peak ID.
- Choose a part of baseline without peaks or interference signals and calculate the noise for 0.1 min. (Verify that the noise value is comparable with the one of the previous *Baseline Acquisition and Analysis*).
- Generate a report showing the chromatogram, peak area and signal-to-noise information for Azobenzene.
- Repeat the procedure to calculate the signal-to-noise ratio also for Parathion Methyl.
- Generate a report showing the chromatogram, peak area and signal-to-noise information for Parathion Methyl.

NOTE If it is not possible to find a suitable part of baseline, to have a complete Chrom-Card final report proceed as follows:
Start the acquisition on the data system. Let the system to acquire for about 1 minute and then inject the test mixture. The noise can be calculated during the first minute of acquisition. Since the retention time will shift by a delayed time, the Component Table has to be updated.

Using non-Chrom-Card Data System

Operate as follows:

- Set the parameters to calculate the signal-to-noise ratio according to the instruction reported in the relevant data system manual.

CAUTION **Choose a part of baseline without peaks or interference signals and calculate the noise for 0.1 min. (Verify that the noise value is comparable with the one of the previous *Baseline Acquisition and Analysis*).**

16. The following criteria indicate successful completion of NPD-PKD checkout.

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

Table 14-3. NPD-PKD Acceptance Criteria

CHROM-CARD		
Baseline Parameters	**Analog** *(1V Full Scale)*	**Digital** *(10V Full Scale)*
Noise (μV)	< 40	< 400
Wander (μV)	< 100	< 1 000
Drift (μV/h)	< 300	<3 000
Analytical Results		
Azobenzene Signal-to-noise ratio		600
Parathion Methyl Signal-to-noise ratio		1 700
Octadecano Signal-to-noise ratio		Negligible

(Acceptance Values)

Computing-integrator (e.g. ChromJet)

CHROMQUEST	
Baseline Parameters *(1V Full Scale)*	
Noise (μV)	< 40
Wander (μV)	< 100
Drift (μV/h)	< 300
Analytical Results	
Azobenzene Signal-to-noise ratio	> 600
Parathion Methyl Signal-to-noise ratio	> 1700
Octadecane Signal-to-noise ratio	Negligible

(Acceptance Values)

	ATLAS	
	Baseline Parameters *(10V Full Scale)*	
	Noise (µV)	< 400
	Wander (µV)	< 1 000
	Drift (µV/h)	< 3 000
	Analytical Results	
	Azobenzene Signal-to-noise ratio	> 600
	Parathion Methyl Signal-to-noise ratio	> 1 700
	Octadecane Signal-to-noise ratio	Negligible
⚠		

Acceptance Values	XCALIBUR	
	Baseline Parameters *(Acquisition Frequency = 10 Hz)*	
	Noise (Counts)	< 4 000
	Wander Counts)	< 10 000
	Drift (Counts/h)	< 30 000
	Analytical Results	
	Azobenzene Signal-to-noise ratio	> 600
	Parathion Methyl Signal-to-noise ratio	> 1 700
	Octadecane Signal-to-noise ratio	Negligible
⚠		

15

Checkout Using NPD with PPKD Injector

Operating Procedures

SOP Number: P0314/08/E - 01 September

Scope

Use the following procedure to verify proper NPD operation with Purged Packed Injector.

Parts Referenced

Table 15-1. NPD-PPKD 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	2 mm ID tapered	453 220 50
Liner Seal	Graphite seal for glass liner	290 334 05
Pre-column	Fused Silica Capillary Column 0.5 mt long 0.53 mm ID	260 603 75
Retaining Nut	M4 capillary column retaining nut	350 324 23
Press-fit connections	For columns 0.53/0.32 mm ID	350 438 16
Injection side adapter	For Wide bore column	347 003 03
Retaining Nut	For Injection side adapter	350 024 04
Detector side adapter	For Wide bore column	347 103 04
Ferrule	6 mm ID double brass ferrule	290 341 37
Nut	1/4” G-6 mm ID nut	350 201 18
Graphite Ferrule	Graphite ferrule for 0.53 mm ID Column Graphite ferrule for 0.32 mm ID Column	290 134 86 290 134 87
Septum	Standard septum for Purged Packed Injector	313 032 26
Syringe	10 µl size; 50 mm needle length	365 005 25

Table 15-1. NPD-PPKD Parts Referenced (Continued)

Part	Description	Part Number
Test Mixture	Three components in Iso-Octane: *Component* *Concentration* Azobenzene 1 µg/ml Octadecane 1000 µg/ml Parathion methyl 1 µg/ml	338 190 06
Gases	Chromatographic-grade purity	
Data Acquisition	Chrom-Card, ChromQuest, Atlas, Xcalibur, Computing-integrator	

Analytical Conditions Required for Purged Packed Injector

Table 15-2. NPD-PPKD Analytical Conditions

Parameters Setting	
Gases	Carrier Gas: Helium = 30 kPa Constant Pressure Hydrogen = 2.3 ml/min Air = 60 ml/min Make-up Gas: Nitrogen = 15 ml/min
Oven Program	Initial Temperature = 50 °C Initial Time = 1 minute Ramp 1 = 20 °C/minute Final Temperature = 200 °C Final Time = 1 minute
Injector	Operating Mode = Wide bore Temperature = 200 °C
Detector	Base Temperature = 300 °C Source Current = *Refer to Source Ignition* Polarizer voltage = 3.5 V Detector Signal Range = 10^0
Injected Volume	1 µl + needle of Test Mixture
Analog Signal Output	Chrom-Card Acquisition Frequency = Medium
Digital Signal Output	Chrom-Card, ChromQuest, Atlas, Xcalibur Acquisition Frequency = 10 Hz

Recommended Initial Operations

Before starting the checkout, the following operations should be carried out:

1. Insert the glass liner.
   Remove the column and insert the 2 mm ID tapered glass liner, as required for the checkout, from the bottom of the injector with the appropriate liner seal. Fix the liner by using the appropriate adapter for capillary column.
   Refer to the TRACE GC Ultra Maintenance and Troubleshooting Manual.

2. Mount the adapter for capillary column on the detector base body.

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

4. Connect the required gas lines
   Verify the required gas supplies are properly connected to your GC.

5. Install the precolumn.
   Connect the precolumn to the injector.

6. Install the test column
   Connect the test column to the precolumn by using the press fit connections provided.

7. Perform the manual leak check following the Operating Procedure "*Perforing a Leak Check* " in Chapter 14 of the TRACE GC Ultra Operating Manual.

8. Connect the other end of the test column to the detector base body.

9. Perform Column Evaluation.

10. Connect your data handling.
    Verify that your data handling is properly connected to your GC system.

OPERATING PROCEDURE

NPD-PPKD Checkout

Before beginning, press **CONFIG** to verify the GC configuration:

Left inlet or Right inlet	PPKD
Left carrier or Right carrier	He (helium)
Left detector or Right detector	NPD

1. Use **LEFT CARRIER** or **RIGHT CARRIER** to display the appropriate Carrier Gas Control Table. Verify to operate in constant pressure mode. If not, scroll to `Flow mode`, press **MODE/TYPE** to access the selection menu, then select `con pres`. Scrool to `Pressure` and set the pressure value to have the required carrier gas flow rate `Col.flow`.

```
          LEFT CARRIER[1]

Pressure          30.0   30.0
Col.flow          3.00
Lin. veloc.             (60.9)<
```

1. These settings could also be for a right carrier.

2. Use **OVEN** to display the Column Oven Control Table. Set the oven temperature and the Oven Program required.

```
               OVEN

Temp              50.0   50.0
Initial Time             1.00
Ramp 1                   20.0
Final temp                200
Final time 1             1.00<
Ramp 2                    Off
```

3. Use **LEFT INLET** or **RIGHT INLET** to display the appropriate PPKD Injector Control Table. Set the required temperature setpoint `Temp`.Verify to operate in

Wide bore mode. If not, scroll to `Mode`, press **MODE/TYPE** to access the selection menu, then select `Widebore`.

LEFT INLET (PPKD)[1]		
Temp	200	200
Pressure	30.0	30.0
Mode:		Wide bore<
Constant sept purge?		Y<

1. These settings could also be for a right inlet.

4. Use **LEFT DETECTOR** or **RIGHT DETECTOR** to display the appropriate NPD Detector Control Table. Set the required temperature `Base Temp` and the detector gases `H2`, `Air` and `Mkup` required setpoints.

LEFT DETECTOR (NPD)[1]		
Source cur, A		Off
Base temp	300	300
Signal pA		(10.4)
Target curr. pA		(X.XX)
Auto adjust		No
Polarizer V		3.5
H2 delay time		Off
H2	2.3	2.3
Air	60	60
Mkup N2	15	15

1. These settings could also be for a right detector.

5. Scroll to `Polarizer V` and set 3.5 V.

Source Ignition

6. Switch on the source operating as follows:

   a. Open the detector gases `H2`, `Air` and `Mkup` and set the gas flow rates as follows:

- `H2` = 2.3 ml/min
- `Air` = 60 ml/min
- `Mkup N2` = 15 ml/min)

b. Increase the `Base Temp` to 300°C and wait that the NPD cell reaches the correct set temperature.

c. Be sure that the backoff signal is between 0 and 0.5 pA.

d. Switch on the source with an initial current of 2.50 A. The backoff signal can slightly increase, but should remain within 0 and 1.5 pA.

e. Monitor the signal through the keypad or through the data system, increase the current value by steps of 0.002 A, until an immediate and strong increase of the signal is observed.

f. Wait five minutes to let the source stabilizes.

g. Check that source is correctly switched on decreasing hydrogen flow to 0.5 ml/min until signal decreases down to zero, then increase again to original value.

- If the signal remains around zero, it means that the source is not switched on and it is necessary to increase further the current, accordingly to the procedure just described.
- If the signal rises back to original value, it means that source is correctly switched on

h. Increase the current value of 2% of the actual ignition current. Let the signal stabilizes until its level drops below 20 pA.

7. Use **LEFT SIGNAL** or **RIGHT SIGNAL** to display the appropriate NPD Detector Signal Control Table. Scroll to `Range` and set the electrometer amplifier input range required.

LEFT SIGNAL (NPD)[1]	
Output	(1000)
Offset	100
Auto zero?	Y/N
Range 10^(0..3)	0<
Analog filter	Off
Baseline comp	Off

1. These settings could also be for a right signal.

8. Activate your Data System and set the parameters required for the checkout.

9. In the NPD Detector Signal Control Table scroll to `Auto zero?` and turn it **YES**.

10. Perform a blank analysis injecting pure iso-octane and press **START** on the GC to begin the checkout run.

Baseline Acquisition and Analysis

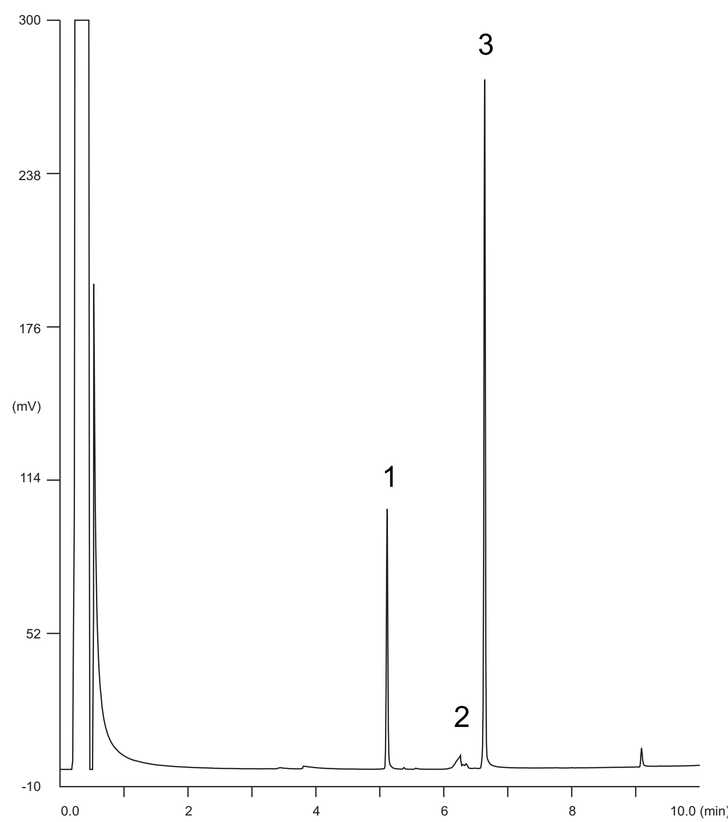
NOTE Refer to the Acceptance Values reported in the Table 15-3 according to the data handling in use.

11. With the GC in Stand-by/Prep Run condition, activate the data system for 10 minutes to evaluate your baseline in isothermal condition.

12. After the baseline evaluation has been completed, set-up the data system to acquire a single run.

13. Inject the test mixture and press **START** on the GC to begin the checkout run. The resulting chromatogram should look like the one shown in *Figure 15.1*.

CAUTION **Verify the peak shape. If any peak distortion is visible, change the analytical test column.**



1. Azobenzene	2. Octadecane	3. Parathion Methyl

Figure 15-1. NPD-PPKD Injection

14. Establish the integration parameters and the peak table identifying the test mix components.

15. Set up the data system to calculate the signal-to-noise ratio.

Using Chrom-Card Data System

Operate as follows:

- Open the Method Editor and include the signal-to-noise Report into the Report Parameters Page.
- By clicking on the side icon, open the signal-to-noise calculation and set Azobenzene component as signal peak ID.
- Choose a part of baseline without peaks or interference signals and calculate the noise for 0.1 min. (Verify that the noise value is comparable with the one of the previous *Baseline Acquisition and Analysis*).
- Generate a report showing the chromatogram, peak area and signal-to-noise information for Azobenzene.
- Repeat the procedure to calculate the signal-to-noise ratio also for Parathion Methyl.
- Generate a report showing the chromatogram, peak area and signal-to-noise information for Parathion Methyl.

NOTE If it is not possible to find a suitable part of baseline, to have a complete Chrom-Card final report proceed as follows: Start the acquisition on the data system. Let the system to acquire for about 1 minute and then inject the test mixture. The noise can be calculated during the first minute of acquisition. Since the retention time will shift by a delayed time, the Component Table has to be updated.

Using non-Chrom-Card Data System

Operate as follows:

- Set the parameters to calculate the signal-to-noise ratio according to the instruction reported in the relevant data system manual.

CAUTION **Choose a part of baseline without peaks or interference signals and calculate the noise for 0.1 min. (Verify that the noise value is comparable with the one of the previous *Baseline Acquisition and Analysis*).**

16. The following criteria indicate successful completion of NPD-PPKD checkout.
17. If these criteria are not met, repeat the test.

Table 15-3. NPD-PPKD Acceptance Criteria

Acceptance Values	CHROM-CARD		
	Baseline Parameters	**Analog** *(1V Full Scale)*	**Digital** *(10V Full Scale)*
	Noise (µV)	< 40	< 400
	Wander (µV)	< 100	< 1 000
	Drift (µV/h)	< 300	<3 000
	Analytical Results		
	Azobenzene Signal-to-noise ratio		600
	Parathion Methyl Signal-to-noise ratio		1 700
	Octadecane Signal-to-noise ratio		Negligible
⚠			

Computing-integrator (e.g. ChromJet)	
⚠	

Acceptance Values	CHROMQUEST	
	Baseline Parameters *(1V Full Scale)*	
	Noise (µV)	< 40
	Wander (µV)	< 100
	Drift (µV/h)	< 300
	Analytical Results	
	Azobenzene Signal-to-noise ratio	600
	Parathion Methyl Signal-to-noise ratio	1 700
	Octadecane Signal-to-noise ratio	Negligible
⚠		

ATLAS	
Baseline Parameters *(10V Full Scale)*	
Noise (µV)	< 400
Wander (µV)	< 1 000
Drift (µV/h)	< 3 000
Analytical Results	
Azobenzene Signal-to-noise ratio	> 600
Parathion Methyl Signal-to-noise ratio	> 1 700
Octadecane Signal-to-noise ratio	Negligible

Acceptance Values

XCALIBUR	
Baseline Parameters *(Acquisition Frequency = 10 Hz)*	
Noise (Counts)	< 4 000
Wander Counts)	< 10 000
Drift (Counts/h)	< 30 000
Analytical Results	
Azobenzene Signal-to-noise ratio	> 600
Parathion Methyl Signal-to-noise ratio	> 1 700
Octadecane Signal-to-noise ratio	Negligible

16

Checkout Using NPD with PTV Injector

Chapter at a Glance...

Operating Procedures

SOP Number: P0315/08/E - 01 September 2009

Scope

Use the following procedure to verify proper NPD operation with the Programmable Temperature Vaporizing Injector.

Parts Referenced

Table 16-1. NPD-PTV 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
Liner	Silcosteel 2 mm ID (set of 2)	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	365 005 25
Test Mixture	Three components in Iso-Octane: *Component* *Concentration* Azobenzene 1 µg/ml Octadecane 1000 µg/ml Parathion methyl 1 µg/ml	338 190 06
Gases	Chromatographic-grade purity	
Data Acquisition	Chrom-Card, ChromQuest, Atlas, Xcalibur, Computing-integrator	

Analytical Conditions Required for PTV Splitless Injection

Table 16-2. NPD-PTV Analytical Conditions

Parameters Setting	
Gases	Carrier Gas: Helium = 30 kPa Constant Pressure Hydrogen = 2.3 ml/min Air = 60 ml/min Make-up Gas: Nitrogen = 30 ml/min
Oven Program	Initial Temperature = 70 °C Initial Time = 1 minute Ramp 1 = 20 °C/minute Final Temperature = 230 °C Final Time = 1 minute
Injector	Operating Mode = PTV Splitless Splitless Time = 0.8 minutes Split Flow = 50 ml/min Constant Septum Purge = Yes Inject Temp = 50 °C Inject Time = 0.1 minute Transfer ramp = 10 °C/sec Transfer Temperature = 260 °C Transfer time = 1 minutes
Detector	Base Temperature = 300 °C Source Current = *Refer to Source Ignition* Polarizer voltage = 3.5 V Detector Signal Range = 10^0
Injected Volume	1 µl of Test Mixture

Table 16-2. NPD-PTV Analytical Conditions (Continued)

Parameters Setting	
Analog Signal Output	Chrom-Card Acquisition Frequency = Medium
Digital Signal Output	Chrom-Card, ChromQuest, Atlas, Xcalibur Acquisition Frequency = 10 Hz

Recommended Initial Operations

Before starting the checkout, the following operations should be carried out:

1. Replace the liner.
   The liner currently installed in your injector should be carefully removed and replaced with the 2 mm ID Silcosteel glass liner, 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. Connect the required gas lines
   Verify the required gas supplies are properly connected to your GC.

4. Install the test column.
   The column currently installed should be carefully removed and replaced with the required test column.

5. Perform Column Evaluation and Leak Test.

6. Connect your data handling.
   Verify that your data handling is properly connected to your GC system.

OPERATING PROCEDURE

NPD-PTV Checkout in PTV Splitless Mode

Before beginning, press **CONFIG** to verify the GC configuration:

Right inlet	PTV
Right carrier	He (helium)
Right detector	NPD

1. Use **RIGHT CARRIER** to display the appropriate Carrier Gas Control Table. Verify to operate in constant pressure mode. If not, scroll to `Flow mode`, press **MODE/TYPE** to access the selection menu, then select `con pres`. Scroll to `Pressure` and set the pressure value to have the required carrier gas flow rate `Col.flow`.

```
        RIGHT CARRIER¹
Pressure         30.0   30.0
Col.flow         3.00
Lin. veloc.           (60.9)<
```

2. Use **OVEN** to display the Column Oven Control Table. Set the oven temperature and the Oven Program required.

```
             OVEN
Temp              70.0  70.0
Initial Time            1.00
Ramp 1                  20.0
Final temp               230
Final time 1            1.00<
Ramp 2                   Off
```

3. Use **RIGHT INLET** to display the appropriate Programmable Temperature Vaporizing Injector Control Table. Set the required temperature setpoint

`Temp`. Verify to operate in **PTV splitless** mode. If not, scroll to `Mode`, press **MODE/TYPE** to access the selection menu, then select `PTV splitless`. Scroll to `Splitless time` to set the required setpoint.

```
     RIGHT INLET (PTV)

Temp                  70    70
Pressure            30.0  30.0
Mode:            PTV Splitless
Total flow            (53.0)
Split Flow          50.0  50.0
Splitless time      0.80  0.80
Constant sept purge?         Y
Inject phase menu:          Y<
```

4. Scroll to `Inject phase menu`. Press **MODE/TYPE** to enter the **PTV Phase Menu**.

```
      PTV PHASE MENU

Ramped pressure?             N
Inject temp                 50
Inject time                0.1
Transfer ramp               10
Transfer temp              260
Transfer time            1.00<
```

5. Select `Ramped pressure?` **NO**. Set the required `Inject temp` and *Inject time* setpoints as required. Then, set the `Transfer ramp`, the `Transfer temp` and the `Transfer time` required setpoints.

6. Use **RIGHT DETECTOR** to display the appropriate NPD Detector Control Table. Set the required temperature `Base Temp` and the detector gases `H2`, `Air` and `Mkup` required setpoints.

RIGHT DETECTOR (NPD)		
Source cur, A		Off
Base temp	300	300
Signal pA		(10.4)
Target curr. pA		(X.XX)
Auto adjust		No
Polarizer V		3.5
H2 delay time		Off
H2	2.3	2.3
Air	60	60
Mkup N2	15	15

7. Scroll to `Polarizer V` and set 3.5 V.

Source Ignition

8. Switch on the source operating as follows:

   a. Open the detector gases `H2`, `Air` and `Mkup` and set the gas flow rates as follows:

      - `H2` = 2.3 ml/min
      - `Air` = 60 ml/min
      - `Mkup N2` = 15 ml/min)

   b. Increase the `Base Temp` to 300°C and wait that the NPD cell reaches the correct set temperature.

   c. Be sure that the backoff signal is between 0 and 0.5 pA.

   d. Switch on the source with an initial current of 2.50 A. The backoff signal can slightly increase, but should remain within 0 and 1.5 pA.

e. Monitor the signal through the keypad or through the data system, increase the current value by steps of 0.002 A, until an immediate and strong increase of the signal is observed.

f. Wait five minutes to let the source stabilizes.

g. Check that source is correctly switched on decreasing hydrogen flow to 0.5 ml/min until signal decreases down to zero, then increase again to original value.

- If the signal remains around zero, it means that the source is not switched on and it is necessary to increase further the current, accordingly to the procedure just described.
- If the signal rises back to original value, it means that source is correctly switched on

h. Increase the current value of 2% of the actual ignition current. Let the signal stabilizes until its level drops below 20 pA.

9. Use **RIGHT SIGNAL** to display the appropriate NPD Detector Signal Control Table. Scroll to `Range` and set the electrometer amplifier input range required.

```
    RIGHT SIGNAL (NPD)

Output                  (1000)
Offset                    100
Auto zero?                Y/N
Range 10^(0..3)           0<
Analog filter             Off
Baseline comp             Off
```

10. Activate your Data System and set the parameters required for the checkout.

11. In the NPD Detector Signal Control Table scroll to `Auto zero?` and turn it **YES**.

12. Perform a blank analysis injecting pure iso-octane and press **START** on the GC to begin the checkout run.

Baseline Acquisition and Analysis

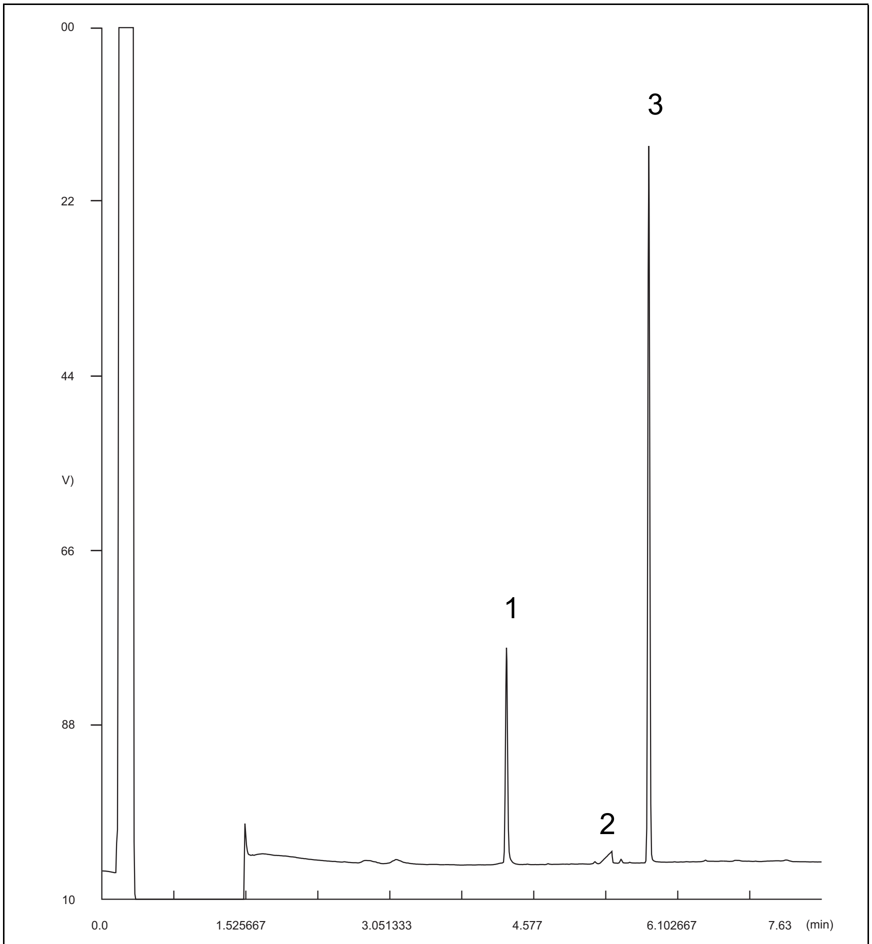
NOTE Refer to the Acceptance Values reported in the Table 16-3 according to the data handling in use.

13. With the GC in Stand-by/Prep Run condition, activate the data system for 10 minutes to evaluate your baseline in isothermal condition.

14. After the baseline evaluation has been completed, set-up the data system to acquire a single run.

15. Inject the test mixture and press **START** on the GC to begin the checkout run. The resulting chromatogram should look like the one shown in *Figure 16.1*.

CAUTION **Verify the peak shape. If any peak distortion is visible, change the analytical test column.**



1. Azobenzene	2. Octadecane	3. Parathion Methyl

Figure 16-1. NPD-PTV Injection

16. Establish the integration parameters and the peak table identifying the test mix components.

17. Set up the data system to calculate the signal-to-noise ratio.

Using Chrom-Card Data System

Operate as follows:

- Open the Method Editor and include the signal-to-noise Report into the Report Parameters Page.
- By clicking on the side icon, open the signal-to-noise calculation and set Azobenzene component as signal peak ID.
- Choose a part of baseline without peaks or interference signals and calculate the noise for 0.1 min. (Verify that the noise value is comparable with the one of the previous *Baseline Acquisition and Analysis*).
- Generate a report showing the chromatogram, peak area and signal-to-noise information for Azobenzene.
- Repeat the procedure to calculate the signal-to-noise ratio also for Parathion Methyl.
- Generate a report showing the chromatogram, peak area and signal-to-noise information for Parathion Methyl.

NOTE If it is not possible to find a suitable part of baseline, to have a complete Chrom-Card final report proceed as follows: Start the acquisition on the data system. Let the system to acquire for about 1 minute and then inject the test mixture. The noise can be calculated during the first minute of acquisition. Since the retention time will shift by a delayed time, the Component Table has to be updated.

Using non-Chrom-Card Data System

Operate as follows:

- Set the parameters to calculate the signal-to-noise ratio according to the instruction reported in the relevant data system manual.

CAUTION **Choose a part of baseline without peaks or interference signals and calculate the noise for 0.1 min. (Verify that the noise value is comparable with the one of the previous *Baseline Acquisition and Analysis*).**

18. The following criteria indicate successful completion of NPD-PTV checkout.

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

Table 16-3. NPD-PTV Acceptance Criteria

Acceptance Values

CHROM-CARD		
Baseline Parameters	**Analog** *(1V Full Scale)*	**Digital** *(10V Full Scale)*
Noise (µV)	< 40	< 400
Wander (µV)	< 100	< 1 000
Drift (µV/h)	< 300	<3 000
Analytical Results		
Azobenzene Signal-to-noise ratio		> 550
Parathion Methyl Signal-to-noise ratio		> 1500
Octadecane Signal-to-noise ratio		Negligible

Computing-integrator (e.g. ChromJet)

Acceptance Values

CHROMQUEST	
Baseline Parameters *(1V Full Scale)*	
Noise (µV)	< 40
Wander (µV)	< 100
Drift (µV/h)	< 300
Analytical Results	
Azobenzene Signal-to-noise ratio	> 550
Parathion Methyl Signal-to-noise ratio	> 1500
Octadecane Signal-to-noise ratio	Negligible

ATLAS	
Baseline Parameters *(10V Full Scale)*	
Noise (µV)	< 400
Wander (µV)	< 1 000
Drift (µV/h)	< 3 000
Analytical Results	
Azobenzene Signal-to-noise ratio	> 550
Parathion Methyl Signal-to-noise ratio	> 1500
Octadecane Signal-to-noise ratio	Negligible

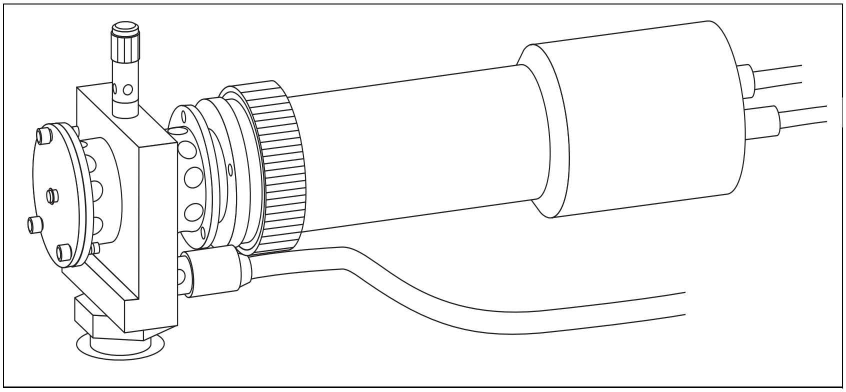
Acceptance Values

XCALIBUR	
Baseline Parameters *(Acquisition Frequency = 10 Hz)*	
Noise (Counts)	< 4 000
Wander Counts)	< 10 000
Drift (Counts/h)	< 30 000
Analytical Results	
Azobenzene Signal-to-noise ratio	> 550
Parathion Methyl Signal-to-noise ratio	> 1 500
Octadecane Signal-to-noise ratio	Negligible

SECTION

V

SOPs Using FPD



The *SOPs Using Fast FPD* section, contains the procedures to test the TRACE GC Ultra with the fast Flame Photometric Detector (FPD) using different injectors.

17

Checkout Using FPD with S/SL Injector

Chapter at a Glance...

Operating Procedures

SOP Number: P0316/08/E - 01 September 2009

Scope

Use the following procedure to verify proper FPD operation with the Split/Splitless Injector. This SOP is applicable both for the control card labeled **FPD** and for the control card labeled **FPD/F**.

Parts Referenced

Table 17-1. FPD-S/SL 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; 70 mm needle length	365 001 03
Interferential Filter	526 nm for phosphorus 394 nm for sulphur	281 071 00 281 070 00
Test Mixture	Three components in Iso-Octane: *Component* *Concentration* Azobenzene 1 µg/ml Octadecane 1000 µg/ml Parathion methyl 1 µg/ml	338 190 06
Gases	Chromatographic-grade purity	
Data Acquisition	Chrom-Card, ChromQuest, Atlas, Xcalibur, Computing-integrator	

Analytical Conditions Required for Splitless Injection

Table 17-2. FPD-S/SL Analytical Conditions

Parameters Setting	
Gases	Carrier Gas: Helium = 30 kPa Constant Pressure Hydrogen = 90 ml/min Air = 115 ml/min
Oven Program	Initial Temperature = 70 °C Initial Time = 1 minute Ramp 1 = 20 °C/minute Final Temperature = 230 °C Final Time = 1 minute
Injector	Operating Mode = Splitless Temperature = 230 °C Splitless Time = 0.8 minutes Split Flow = 60 ml/min Constant Septum Purge = Yes
Detector	Base Temperature = 300 °C FPD Temperature = 150 °C High voltage mode = No Detector Signal Range = 10^0 (See Note)
Injected Volume	1 µl + needle of Test Mixture
Analog Signal Output	Chrom-Card Acquisition Frequency = Medium
Digital Signal Output	Chrom-Card, ChromQuest, Atlas, Xcalibur Acquisition Frequency = 10 Hz

NOTE In the case your GC is equipped with the previous non-fast FPD control card, labeled **FPD**, set Detector Signal Range to 10^1.

Recommended Initial Operations

Before starting the checkout, the following operations should be carried out:

1. Replace the glass liner.
   The glass liner currently installed in your injector should be carefully removed and replaced with the 3 mm ID glass liner for splitless application, as required for the checkout, with the appropriate graphite seal.

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

3. Connect the required gas lines
   Verify the required gas supplies are properly connected to your GC.

4. Install the test column.
   The column currently installed should be carefully removed and replaced with the required test column.

5. Perform Column Evaluation and Leak Test

6. Connect your data handling.
   Verify that your data handling is properly connected to your GC system.

OPERATING PROCEDURE

FPD-S/SL Checkout in Splitless Mode

Before beginning, press **CONFIG** to verify the GC configuration:

Left inlet or Right inlet	S/SL
Left carrier or Right carrier	He (helium)
Left detector or Right detector	FPD

1. Use **LEFT CARRIER** or **RIGHT CARRIER** to display the appropriate Carrier Gas Control Table. Verify to operate in constant pressure mode. If not, scroll to `Flow mode`, press **MODE/TYPE** to access the selection menu, then select `con pres`. Scrool to `Pressure` and set the pressure value to have the required carrier gas flow rate `Col.flow`.

```
          LEFT CARRIER[1]

Pressure          30.0    30.0
Col.flow          3.00
Lin. veloc.             (60.9)<
```

1. These settings could also be for a right carrier.

2. Use **OVEN** to display the Column Oven Control Table. Set the oven temperature and the Oven Program required.

```
               OVEN

Temp              70.0   70.0
Initial Time             1.00
Ramp 1                   20.0
Final temp                230
Final time 1             1.00<
Ramp 2                    Off
```

3. Use **LEFT INLET** or **RIGHT INLET** to display the appropriate Split/Splitless Injector Control Table and set the required temperature setpoint `Temp`.

Verify to operate in **Splitless** mode. If not, scroll to `Mode`, press **MODE/TYPE** to access the selection menu, then select `Splitless`. Scroll to `Splitless time` to set the required setpoint.

LEFT INLET (S/SL)[1]		
Temp	230	230
Pressure	30.0	30.0
Mode:		Splitless
Total flow		(63.0)
Split Flow	60.0	60.0
Splitless time	0.80	0.80
Constant sept purge?		Y<

1. These settings could also be for a right inlet.

4. Use **LEFT DETECTOR** or **RIGHT DETECTOR** to display the appropriate FPD Detector Control Table. Set the required temperatures `Base Temp` and `FPD Temp`. Then, set the detector gases `H2` and `Air` required setpoints.

LEFT DETECTOR (FPD)[1]		
Flame		Off
Base temp	300	300
FPD temp	150	150
Signal pA		(1.4)
High voltage mode?		N
H2	90	90
Air	115	115
Mkup N2	00	00

1. These settings could also be for a right detector.

5. Verify that `High voltage mode` is set to **NO**.

6. Scroll to `Flame` and press **ON**. This start the ignition sequence. When ignition is confirmed, the photomultiplier tube is energized.

The baseline level `Signal pA`, will suddenly increase meaning that the flame is lit inside the detector. After a few seconds, the baseline should stabilize to the standing current of the system.

7. Use **LEFT SIGNAL** or **RIGHT SIGNAL** to display the appropriate FPD Detector Signal Control Table. Scroll to `Range` and set the electrometer amplifier input range required.

LEFT SIGNAL (FPD)[1]	
Output	(1000)
Offset	100
Auto zero?	Y/N
Range 10^(0..3)	0<
Baseline comp	Off

1. These settings could also be for a right signal.

8. Activate your Data System and set the parameters required for the checkout.

9. In the FPD Detector Signal Control Table scroll to `Auto zero?` and turn it **YES**.

10. Perform a blank analysis injecting pure iso-octane and press **START** on the GC to begin the checkout run.

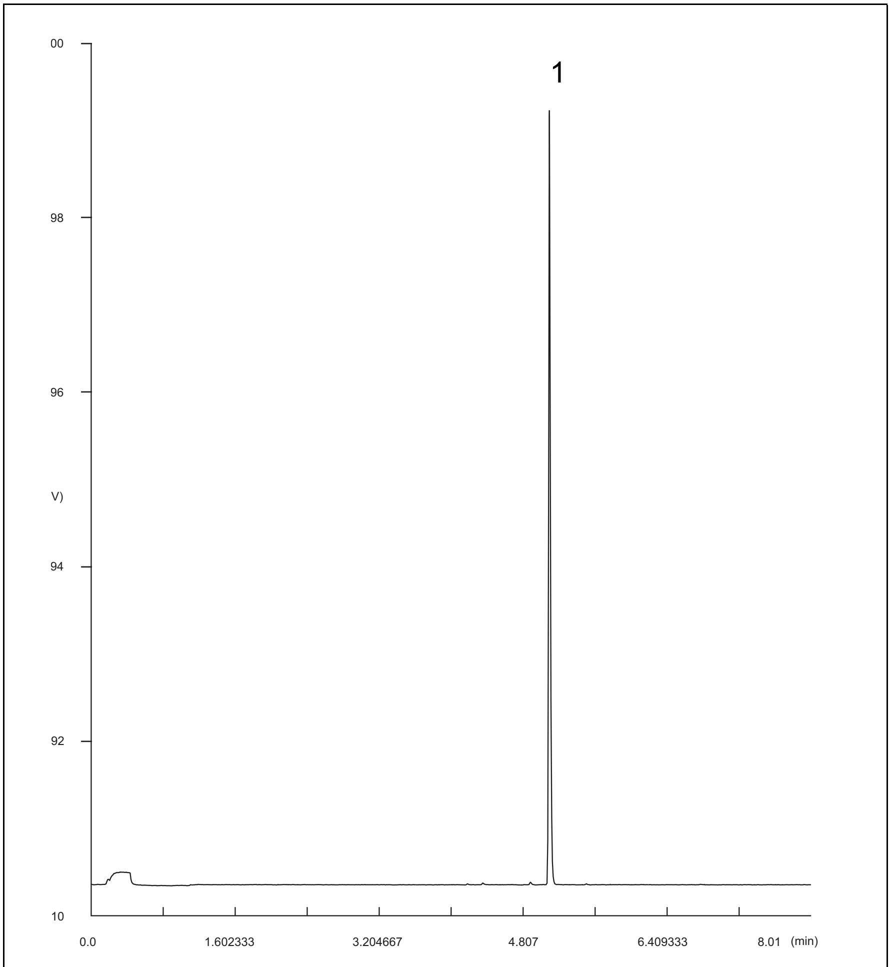
Baseline Acquisition and Analysis

NOTE Refer to the Acceptance Values reported in the Table 17-3 according to the data handling in use.

11. With the GC in Stand-by/Prep Run condition, activate the data system for 10 minutes to evaluate your baseline in isothermal condition.

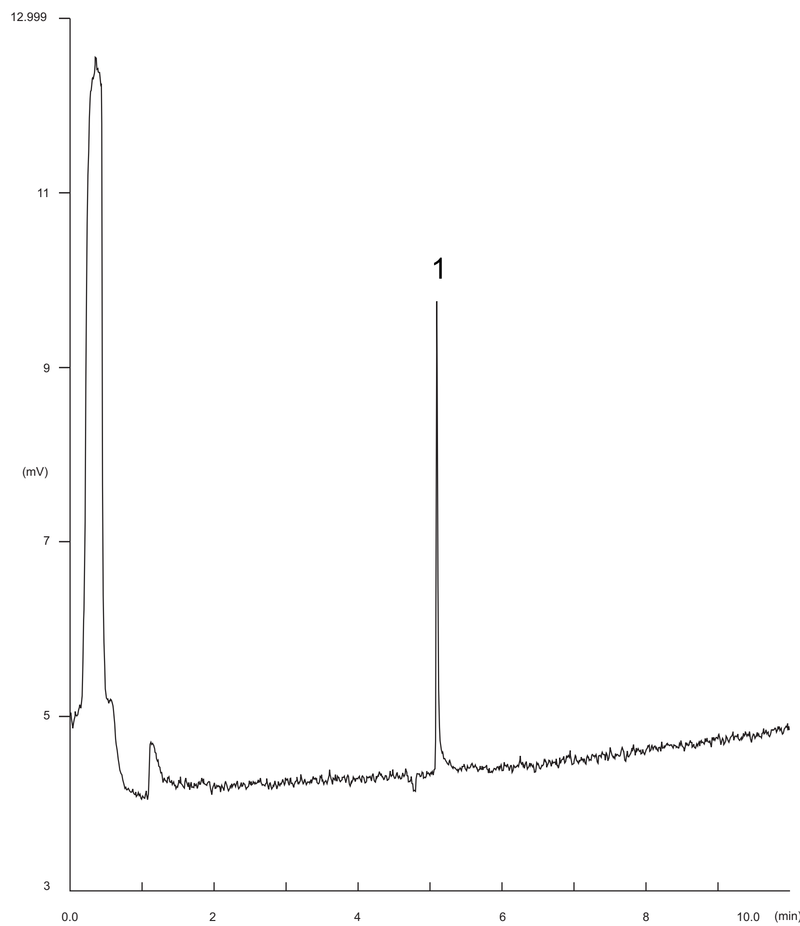
12. After the baseline evaluation has been completed, set-up the data system to acquire a single run.

13. When the GC is ready, inject the test mixture and press **START** on the GC to begin the checkout run. The resulting chromatogram should look like the one shown in *Figure 17.1* or *17.2*.



1. Parathion Methyl

Figure 17-1. FPD-Splitless Injection with 526 nm filter



1. Parathion Methyl

Figure 17-2. FPD-Splitless Injection with 394 nm

14. Establish the integration parameters and the peak table identifying the test mix components.

15. Set up the data system to calculate the signal-to-noise ratio.

Using Chrom-Card Data System

Operate as follows:

- Open the Method Editor and include the signal-to-noise Report into the Report Parameters Page.
- By clicking on the side icon, open the signal-to-noise calculation and set Parathion Methyl component as signal peak ID.
- Choose a part of baseline without peaks or interference signals and calculate the noise for 0.1 min. (Verify that the noise value is comparable with the one of the previous *Baseline Acquisition and Analysis*).
- Generate a report showing the chromatogram, peak area and signal-to-noise information for the component.

NOTE If it is not possible to find a suitable part of baseline, to have a complete Chrom-Card final report proceed as follows: Start the acquisition on the data system. Let the system to acquire for about 1 minute and then inject the test mixture. The noise can be calculated during the first minute of acquisition. Since the retention time will shift by a delayed time, the Component Table has to be updated.

Using non-Chrom-Card Data System

Operate as follows:

- Set the parameters to calculate the signal-to-noise ratio according to the instruction reported in the relevant data system manual.

CAUTION **Choose a part of baseline without peaks or interference signals and calculate the noise for 0.1 min. (Verify that the noise value is comparable with the one of the previous *Baseline Acquisition and Analysis*).**

16. The following criteria indicate successful completion of FPD-S/SL checkout.

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

Table 17-3. FPD-S/SL Acceptance Criteria

Acceptance Values

CHROM-CARD				
Baseline Parameters	**Analog** *(1V Full Scale)*		**Digital** *(10V Full Scale)*	
	394 nm (S) Filter	**526 nm (P) Filter**	**394 nm (S) Filter**	**526 nm (P) Filter**
Noise (μV)	< 50	< 50	< 500	< 500
Wander (μV)	< 100	< 100	< 1 000	< 1 000
Drift (μV/h)	< 100	< 100	< 1 000	< 1 000
Analytical Results				
Parathion Methyl Signal to Noise Ratio			**394 nm (S) Filter**	**526 nm (P) Filter**
			> 40	> 2 000

Acceptance Values

CHROMQUEST		
Baseline Parameters *(1V Full Scale)*		
		526 nm (P) Filter
Noise (μV)	< 50	< 50
Wander (μV)	< 100	< 100
Drift (μV/h)	< 100	< 100
Analytical Results		
Parathion Methyl Signal to Noise Ratio	**394 nm (S) Filter**	**526 nm (P) Filter**
	> 40	> 2 000

ATLAS		
Baseline Parameters (*10V Full Scale*)		
		526 nm (P) Filter
Noise (µV)	< 500	< 500
Wander (µV)	< 1 000	< 1 000
Drift (µV/h)	< 1 000	< 1 000
Analytical Results		
Parathion Methyl Signal to Noise Ratio	**394 nm (S) Filter**	**526 nm (P) Filter**
	> 40	> 2 000
⚠		

Acceptance Values

XCALIBUR		
Baseline Parameters (*Acquisition Frequency = 10 Hz*)		
		526 nm (P) Filter
Noise (Counts)	< 5 000	< 5 000
Wander (Counts)	< 10 000	< 10 000
Drift (Counts/h)	< 10 000	< 10 000
Analytical Results		
Parathion Methyl Signal to Noise Ratio	**394 nm (S) Filter**	**526 nm (P) Filter**
	> 40	> 2 000
⚠		

Analytical Acceptance Comments	
1	Using Chrom-Card, set the signal-to-noise report parameters as described in the current procedure.
2	Using ChromQuest, Atlas, Xcalibur or a Computing integrator (e.g. ChromJet), calculate the S/N ratio as *Peak Height (counts)/noise (counts)*.

18

Checkout Using FPD with OC Injector

Chapter at a Glance...

Operating Procedures

SOP Number: P0317/09/E - 01 September 2009

Scope

Use the following procedure to verify proper FPD operation with the On-Column Injector. This SOP is applicable both for the control card labeled **FPD** and for the control card labeled **FPD/F**.

Parts Referenced

Table 18-1. FPD-OCI 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
Vespel Ferrule	Vespel Ferrule for 0.32 mm ID Column	290 134 60
Retaining Nut	M4 capillary column retaining nut	350 324 23
Syringe	10 µl size; 75 mm needle length	365 020 07
Interferential Filter	526 nm for phosphorus 394 nm for sulphur	281 071 00 281 070 00
Test Mixture	Three components in Iso-Octane: *Component* *Concentration* Azobenzene 1 µg/ml Octadecane 1000 µg/ml Parathion methyl 1 µg/ml	338 190 06
Gases	Chromatographic-grade purity	
Data Acquisition	Chrom-Card, ChromQuest, Atlas, Xcalibur, Computing-integrator	
In case of automatic On-Column for TriPlus Sampler AS		
Syringe	10 µl size; 80 mm needle length	365 020 19

Table 18-1. FPD-OCI Parts Referenced (Continued)

Part	Description	Part Number
Pre-column	2 m long; 0.53 mm ID	260 603 75
Press-fit set	Set of Press-fir connectors for TRACE OC	350 038 45

Analytical Conditions Required for On-Column Injection

Table 18-2. FPD-OCI Analytical Conditions

Gases	Carrier Gas: Helium = 30 kPa Constant Pressure Hydrogen = 90 ml/min Air = 115 ml/min
Oven Program	Initial Temperature = 85 °C Initial Time = 1 minute Ramp 1 = 20 °C/minute Final Temperature = 230 °C Final Time = 1 minute
Injector	Secondary cooling = 10 seconds
Detector	Base Temperature = 300 °C FPD Temperature = 150 °C High voltage mode = No Detector Signal Range = 10^0 (see Note)
Injected Volume	1 µl of Test Mixture
Analog Signal Output	Chrom-Card Acquisition Frequency = Medium
Digital Signal Output	Chrom-Card, ChromQuest, Atlas, Xcalibur Acquisition Frequency = 10 Hz

NOTE In the case your GC is equipped with the previous non-fast FPD control card, labeled **FPD**, set Detector Signal Range to 10^1.

Recommended Initial Operations

Before starting the checkout, the following operations should be carried out:

1. Connect the required gas lines
   Verify the required gas supplies are properly connected to your GC.

2. Install the test column.
   The column currently installed should be carefully removed and replaced with the required test column.
   In case of automatic On-column for TriPlus sampler, install the pre-column and connect it to the test column by press-fit connector.

3. Install and connect the TriPlus sampler and its components.

4. Perform Column Evaluation and Leak Test.

5. Connect your data handling.
   Verify that your data handling is properly connected to your GC system.

6. Verify the opening/closing of the OC injector actuator by using the proper commands.

7. Verify the alignment of the syringe on the OC injector.

OPERATING PROCEDURE

FPD-OCI Checkout in On-Column Mode

Before beginning, press **CONFIG** to verify the GC configuration:

Left inlet or Right inlet	OCI
Left carrier or Right carrier	He (helium)
Left detector or Right detector	FPD

1. Use **LEFT CARRIER** or **RIGHT CARRIER** to display the appropriate Carrier Gas Control Table. Verify to operate in constant pressure mode. If not, scroll to `Flow mode`, press **MODE/TYPE** to access the selection menu, then select `con pres`. Scrool to `Pressure` and set the pressure value to have the required carrier gas flow rate `Col.flow`.

```
        LEFT CARRIER[1]
Pressure          30.0    30.0
Col.flow          3.00
Lin. veloc.             (60.9)<
```

1. These settings could also be for a right carrier.

2. Use **OVEN** to display the Column Oven Control Table. Set the oven temperature and the Oven Program required.

```
              OVEN
Temp              85.0   85.0
Initial Time             1.00
Ramp 1                   20.0
Final temp                230
Final time 1             1.00<
Ramp 2                    Off
```

3. Use **LEFT INLET** or **RIGHT INLET** to display the appropriate On-Column Injector Control Table. Scroll to `Sec. cool time` and set the required secondary cooling time.

```
      LEFT INLET (OCI)1

Pressure          30.0  30.0
Sec. Cool Time         10.00<
```

1. These settings could also be for a right inlet.

4. Use **LEFT DETECTOR** or **RIGHT DETECTOR** to display the appropriate FPD Detector Control Table. Set the required temperatures `Base Temp` and `FPD Temp`. Then, set the detector gases `H2` and `Air` required setpoints.

```
     LEFT DETECTOR (FPD)1

Flame                    Off
Base temp          300   300
FPD temp           150   150
Signal pA              (1.4)
High voltage mode?         N
H2                  90    90
Air                115   115
Mkup N2             00    00
```

1. These settings could also be for a right detector.

5. Verify that `High voltage mode` is set to **NO**.

6. Scroll to `Flame` and press **ON**. This start the ignition sequence. When ignition is confirmed, the photomultiplier tube is energized. The baseline level `Signal pA`, will suddenly increase meaning that the flame is lit inside the detector. After a few seconds, the baseline should stabilize to the standing current of the system.

7. Use **LEFT SIGNAL** or **RIGHT SIGNAL** to display the appropriate FPD Detector Signal Control Table. Scroll to *Range* and set the electrometer amplifier input range required.

LEFT SIGNAL (FPD)[1]	
Output	(1000)
Offset	100
Autozero?	Y/N
Range 10^(0..3)	0<
Baseline comp	Off

1. These settings could also be for a right signal.

8. Activate your Data System and set the parameters required for the checkout.

9. In the FPD Detector Signal Control Table scroll to `Auto zero?` and turn it **YES**.

10. Perform a blank analysis injecting pure iso-octane and press **START** on the GC to begin the checkout run.

Baseline Acquisition and Analysis

NOTE Refer to the Acceptance Values reported in the Table 18-3 according to the data handling in use.

11. With the GC in Stand-by/Prep Run condition, activate the data system for 10 minutes to evaluate your baseline in isothermal condition.

12. After the baseline evaluation has been completed, set-up the data system to acquire a single run.

13. Perform the analysis.

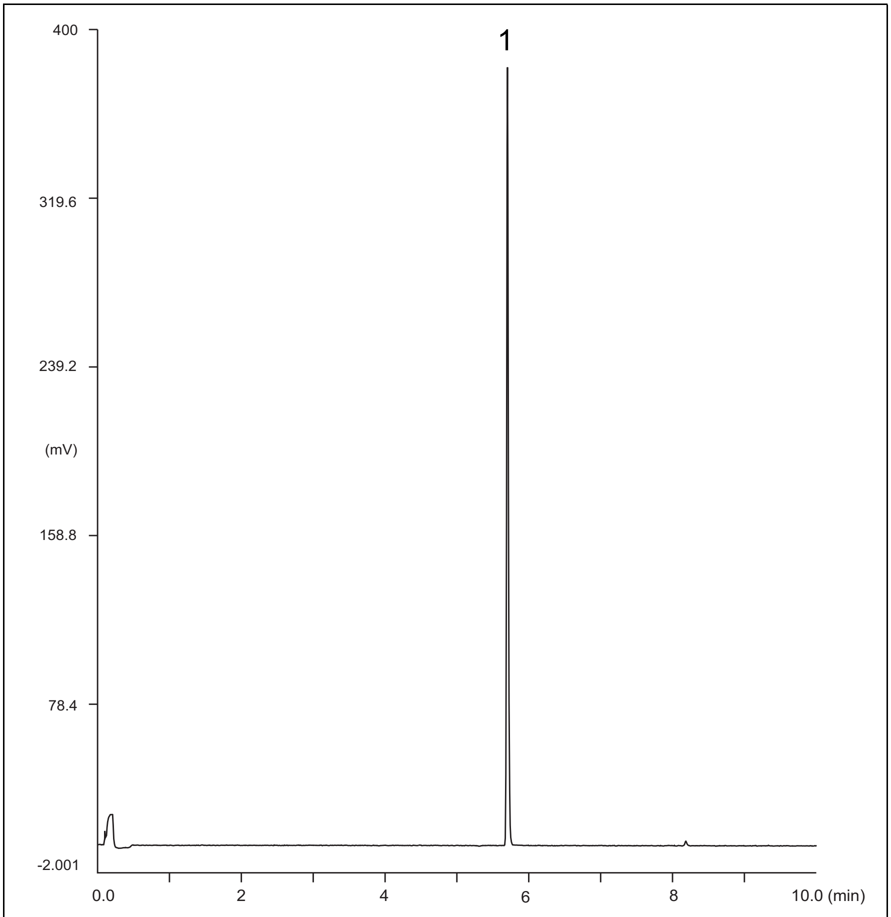
    Manual injection

    - Inject the test mixture and press **START** on the GC to begin the checkout run.

    *Automatic injection with TriPlus sample*r

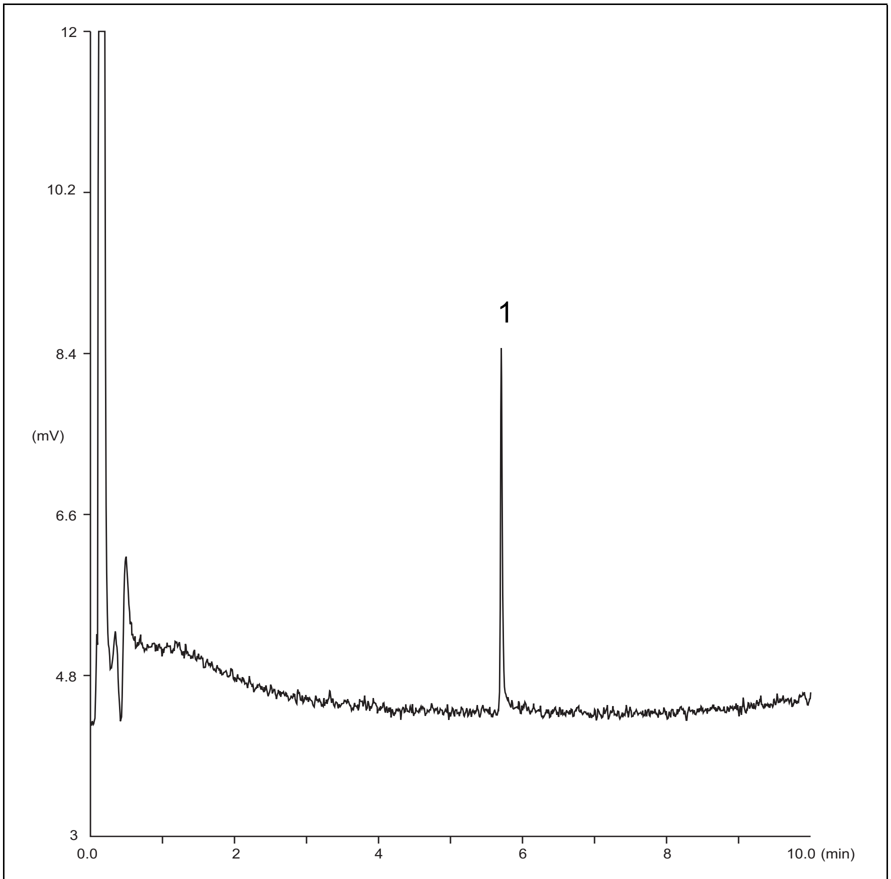
    - Fill a vial with the standard mix and place that vial in the sample tray.
    - Load the method for OC and perform the sampling..

The resulting chromatogram should look like the one shown in *Figure 18.1* or *18.2*.



1. Parathion Methyl

Figure 18-1. FPD-On-Column Injection with 526 nm filter



1. Parathion Methyl

Figure 18-2. FPD-On-Column Injection with 394 nm

14. Establish the integration parameters and the peak table identifying the test mix components.

15. Set up the data system to calculate the signal-to-noise ratio.

Using Chrom-Card Data System

Operate as follows:

- Open the Method Editor and include the signal-to-noise Report into the Report Parameters Page.
- By clicking on the side icon, open the signal-to-noise calculation and set Methylparathion component as signal peak ID.
- Choose a part of baseline without peaks or interference signals and calculate the noise for 0.1 min. (Verify that the noise value is comparable with the one of the previous *Baseline Acquisition and Analysis*).
- Generate a report showing the chromatogram, peak area and signal-to-noise information for the component.

NOTE If it is not possible to find a suitable part of baseline, to have a complete Chrom-Card final report proceed as follows: Start the acquisition on the data system. Let the system to acquire for about 1 minute and then inject the test mixture. The noise can be calculated during the first minute of acquisition. Since the retention time will shift by a delayed time, the Component Table has to be updated.

Using non-Chrom-Card Data System

Operate as follows:

- Set the parameters to calculate the signal-to-noise ratio according to the instruction reported in the relevant data system manual.

CAUTION **Choose a part of baseline without peaks or interference signals and calculate the noise for 0.1 min. (Verify that the noise value is comparable with the one of the previous *Baseline Acquisition and Analysis*).**

16. The following criteria indicate successful completion of FPD-OCI checkout.

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

Table 18-3. FPD-OCI Acceptance Criteria

Acceptance Values

CHROM-CARD				
Baseline Parameters	**Analog** *(1V Full Scale)*		**Digital** *(10V Full Scale)*	
	394 nm (S) Filter	**526 nm (P) Filter**	**394 nm (S) Filter**	**526 nm (P) Filter**
Noise (µV)	< 50	< 50	< 500	< 500
Wander (µV)	< 100	< 100	< 1 000	< 1 000
Drift (µV/h)	< 100	< 100	< 1 000	< 1 000
Analytical Results				
Parathion Methyl Signal to Noise Ratio			**394 nm (S) Filter**	**526 nm (P) Filter**
			> 20	> 1 000

Acceptance Values

CHROMQUEST		
Baseline Parameters *(1V Full Scale)*		
		526 nm (P) Filter
Noise (µV)	< 50	< 50
Wander (µV)	< 100	< 100
Drift (µV/h)	< 100	< 100
Analytical Results		
Parathion Methyl Signal to Noise Ratio	**394 nm (S) Filter**	**526 nm (P) Filter**
	> 20	> 1 000

Acceptance Values

ATLAS		
Baseline Parameters (*10V Full Scale*)		
		526 nm (P) Filter
Noise (µV)	< 500	< 500
Wander (µV)	< 1 000	< 1 000
Drift (µV/h)	< 1 000	< 1 000
Analytical Results		
Parathion Methyl Signal to Noise Ratio	**394 nm (S) Filter**	**526 nm (P) Filter**
	> 20	> 1 000
⚠		

XCALIBUR		
Baseline Parameters (*Acquisition Frequency = 10 Hz)*		
		526 nm (P) Filter
Noise (Counts)	< 5 000	< 5 000
Wander (Counts)	< 10 000	< 10 000
Drift (Counts/h)	< 10 000	< 10 000
Analytical Results		
Parathion Methyl Signal to Noise Ratio	**394 nm (S) Filter**	**526 nm (P) Filter**
	> 20	> 1 000
⚠		

Analytical Acceptance Comments	
1	Using Chrom-Card, set the signal-to-noise report parameters as described in the current procedure.
2	Using ChromQuest, Atlas, Xcalibur or a Computing integrator (e.g. ChromJet), calculate the S/N ratio as *Peak Height (counts)/noise (counts)*.

19

Checkout Using FPD with PKD Injector

Chapter at a Glance...

Operating Procedures

SOP Number: P0318/08/E - 01 September 2009

Scope

Use the following procedure to verify proper FPD operation with Packed Injector. This SOP is applicable both for the control card labeled **FPD** and for the control card labeled **FPD/F**.

Parts Referenced

Table 19-1. FPD-PKD 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	2 mm ID tapered	453 220 50
Liner Seal	Graphite seal for glass liner	290 334 05
Pre-column	Fused Silica Capillary Column 0.5 mt long 0.53 mm ID	260 603 75
Retaining Nut	M4 capillary column retaining nut	350 324 23
Press-fit connections	For columns 0.53/0.32 mm ID	350 438 16
Injection side adapter	For Wide bore column	347 003 03
Retaining Nut	For Injection side adapter	350 024 04
Detector side adapter	For Wide bore column	347 103 04
Ferrule	6 mm ID double brass ferrule	290 341 37
Nut	1/4" G-6 mm ID nut	350 201 18
Graphite Ferrule	Graphite ferrule for 0.53 mm ID Column Graphite ferrule for 0.32 mm ID Column	290 134 86 290 134 87
Septum	Standard septum for Packed Injector	313 032 26
Syringe	10 µl size; 50 mm needle length	365 005 25

Table 19-1. FPD-PKD Parts Referenced (Continued)

Part	Description	Part Number
Interferential Filter	526 nm for phosphorus 394 nm for sulphur	281 071 00 281 070 00
Test Mixture	Three components in Iso-Octane: *Component* *Concentration* Azobenzene 1 µg/ml Octadecane 1000 µg/ml Parathion methyl 1 µg/ml	338 190 06
Gases	Chromatographic-grade purity	
Data Acquisition	Chrom-Card, ChromQuest, Atlas, Xcalibur, Computing-integrator	

Analytical Conditions Required for Packed Injector

Table 19-2. FPD-PKD Analytical Conditions

Parameters	Setting
Gases	Carrier Gas: Helium = 30 kPa Constant Pressure Hydrogen = 90 ml/min Air = 115 ml/min
Oven Program	IsoTemperature = 70 °C Initial Time = 1 minute Ramp 1 = 20 °C/minute Final Temperature = 230 °C Final Time = 1 minute
Injector	Operating Mode = Packed Temperature = 200 °C
Detector	Base Temperature = 300 °C FPD Temperature = 150 °C High voltage mode = No Detector Signal Range = 10^0 (see Note)
Injected Volume	1 µl + needle of Test Mixture
Analog Signal Output	Chrom-Card Acquisition Frequency = Medium
Digital Signal Output	Chrom-Card, ChromQuest, Atlas, Xcalibur Acquisition Frequency = 10 Hz

NOTE In the case your GC is equipped with the previous non-fast FPD control card, labeled **FPD**, set Detector Signal Range to 10^1.

Recommended Initial Operations

Before starting the checkout, the following operations should be carried out:

1. Insert the glass liner.
   Remove the column and insert the 2 mm ID tapered glass liner, as required for the checkout, from the bottom of the injector with the appropriate liner seal. Fix the liner by using the appropriate adapter for capillary column.
   Refer to the TRACE GC Ultra Maintenance and Troubleshooting Manual.

2. Mount the adapter for capillary column on the detector base body.

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

4. Connect the required gas lines
   Verify the required gas supplies are properly connected to your GC.

5. Install the precolumn.
   Connect the precolumn to the injector.

6. Install the test column
   Connect the test column to the precolumn by using the press fit connections provided.

7. Perform the manual leak check following the Operating Procedure "*Perforing a Leak Check* " in Chapter 14 of the TRACE GC Ultra Operating Manual.

8. Connect the other end of the test column to the detector base body.

9. Connect your data handling.
   Verify that your data handling is properly connected to your GC system.

OPERATING PROCEDURE

FPD-PKD Checkout

Before beginning, press **CONFIG** to verify the GC configuration:

Left inlet or Right inlet	PKD
Left carrier or Right carrier	He (helium)
Left detector or Right detector	FPD

1. Use **LEFT CARRIER** or **RIGHT CARRIER** to display the appropriate Carrier Gas Control Table. Verify to operate in constant pressure mode. If not, scroll to `Flow mode`, press **MODE/TYPE** to access the selection menu, then select `con pres`. Scrool to `Pressure` and set the pressure value to have the required carrier gas flow rate `Col.flow`.

```
        LEFT CARRIER[1]

Pressure        30.0   30.0
Col.flow        3.00
Lin. veloc.           (60.9)<
```

1. These settings could also be for a right carrier.

2. Use **OVEN** to display the Column Oven Control Table. Set the oven temperature and the Oven Program required.

```
              OVEN

Temp               70.0   70.0
Initial Time              1.00
Ramp 1                    20.0
Final temp                 230
Final time 1              1.00<
Ramp 2                     Off
```

3. Use **LEFT INLET** or **RIGHT INLET** to display the appropriate PKD Injector Control Table. Set the required temperature setpoint `Temp`.Verify to operate in

Packed mode. If not, scroll to `Mode`, press **MODE/TYPE** to access the selection menu, then select `Packed`.

LEFT INLET (PKD)[1]		
Temp	200	200
Pressure	30.0	30.0
Mode:		Packed

1. These settings could also be for a right inlet.

4. Use **LEFT DETECTOR** or **RIGHT DETECTOR** to display the appropriate FPD Detector Control Table. Set the required temperatures `Base Temp` and `FPD Temp`. Then, set the detector gases `H2` and `Air` required setpoints.

LEFT DETECTOR (FPD)[1]		
Flame		Off
Base temp	300	300
FPD temp	150	150
Signal pA		(1.4)
High voltage mode?		N
H2	90	90
Air	115	115
Mkup N2	00	00

1. These settings could also be for a right detector.

5. Verify that `High voltage` mode is set to **NO**.

6. Scroll to `Flame` and press **ON**. This start the ignition sequence. When ignition is confirmed, the photomultiplier tube is energized. The baseline level `Signal pA`, will suddenly increase meaning that the flame is lit inside the detector. After a few seconds, the baseline should stabilize to the standing current of the system.

7. Use **LEFT SIGNAL** or **RIGHT SIGNAL** to display the appropriate FPD Detector Signal Control Table. Scroll to `Range` and set the electrometer amplifier input range required.

LEFT SIGNAL (FPD)[1]	
Output	(1000)
Offset	100
Auto zero?	Y/N
Range 10^(0..3)	0<
Baseline comp	Off

1. These settings could also be for a right signal.

8. Activate your Data System and set the parameters required for the checkout.

9. In the FPD Detector Signal Control Table scroll to `Auto zero?` and turn it **YES**.

10. Perform a blank analysis injecting pure iso-octane and press **START** on the GC to begin the checkout run.

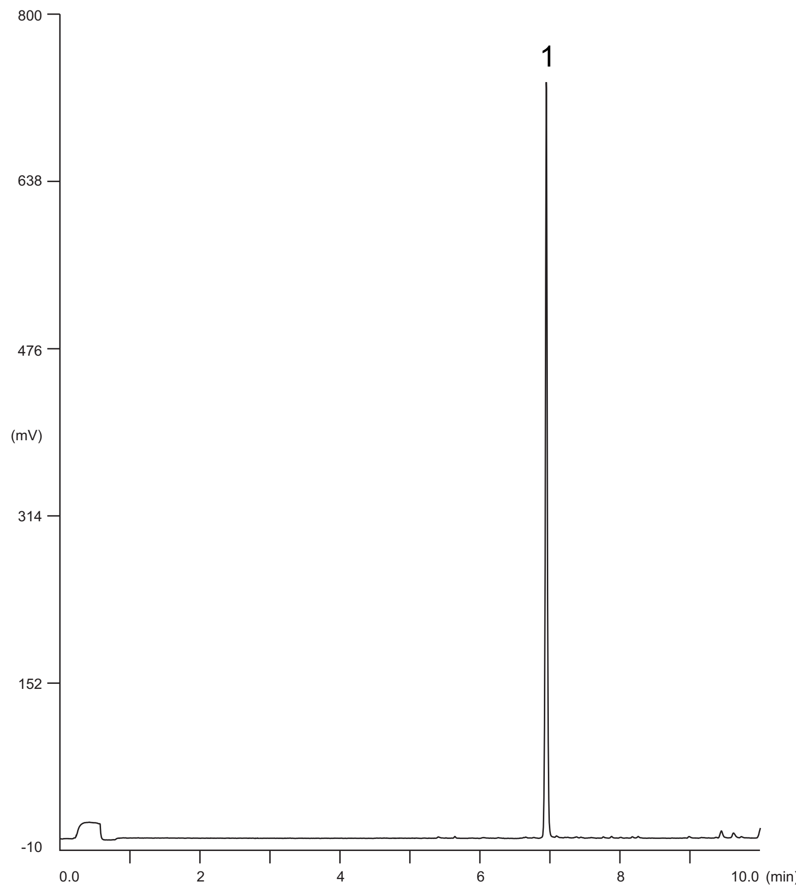
Baseline Acquisition and Analysis

NOTE Refer to the Acceptance Values reported in the Table 19-3 according to the data handling in use.

11. With the GC in Stand-by/Prep Run condition, activate the data system for 10 minutes to evaluate your baseline in isothermal condition.

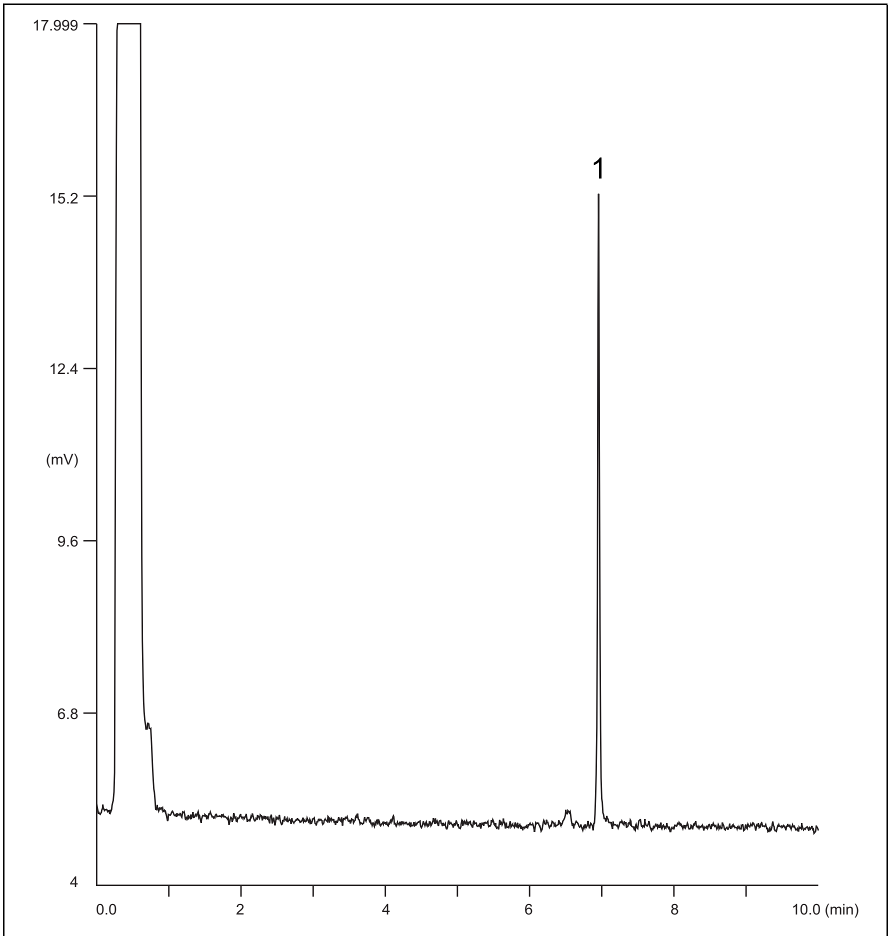
12. After the baseline evaluation has been completed, set-up the data system to acquire a single run.

13. When the GC is ready, inject the test mixture and press **START** on the GC to begin the checkout run. The resulting chromatogram should look like the one shown in *Figure 19.1* or *19.2*.



1. Parathion Methyl

Figure 19-1. FPD-PKD Injection with 526 nm filter



1. Parathion Methyl

Figure 19-2. FPD-PKD Injection with 394 nm

14. Establish the integration parameters and the peak table identifying the test mix components.

15. Set up the data system to calculate the signal-to-noise ratio.

Using Chrom-Card Data System

Operate as follows:

- Open the Method Editor and include the signal-to-noise Report into the Report Parameters Page.
- By clicking on the side icon, open the signal-to-noise calculation and set Methylparathion component as signal peak ID.
- Choose a part of baseline without peaks or interference signals and calculate the noise for 0.1 min. (Verify that the noise value is comparable with the one of the previous *Baseline Acquisition and Analysis*).
- Generate a report showing the chromatogram, peak area and signal-to-noise information for the component.

NOTE If it is not possible to find a suitable part of baseline, to have a complete Chrom-Card final report proceed as follows: Start the acquisition on the data system. Let the system to acquire for about 1 minute and then inject the test mixture. The noise can be calculated during the first minute of acquisition. Since the retention time will shift by a delayed time, the Component Table has to be updated.

Using non-Chrom-Card Data System

Operate as follows:

- Set the parameters to calculate the signal-to-noise ratio according to the instruction reported in the relevant data system manual.

CAUTION **Choose a part of baseline without peaks or interference signals and calculate the noise for 0.1 min. (Verify that the noise value is comparable with the one of the previous *Baseline Acquisition and Analysis*).**

16. The following criteria indicate successful completion of FPD-PKD checkout.

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

Table 19-3. FPD-PKD Acceptance Criteria

Acceptance Values

CHROM-CARD				
Baseline Parameters	**Analog** *(1V Full Scale)*		**Digital** *(10V Full Scale)*	
	394 nm (S) Filter	**526 nm (P) Filter**	**394 nm (S) Filter**	**526 nm (P) Filter**
Noise (µV)	< 50	< 50	< 500	< 500
Wander (µV)	< 100	< 100	< 1 000	< 1 000
Drift (µV/h)	< 100	< 100	< 1 000	< 1 000
Analytical Results				
Parathion Methyl Signal to Noise Ratio			**394 nm (S) Filter**	**526 nm (P) Filter**
			> 30	> 1 500

Acceptance Values

CHROMQUEST		
Baseline Parameters *(1V Full Scale)*		
		526 nm (P) Filter
Noise (µV)	< 50	< 50
Wander (µV)	< 100	< 100
Drift (µV/h)	< 100	< 100
Analytical Results		
Parathion Methyl Signal to Noise Ratio	**394 nm (S) Filter**	**526 nm (P) Filter**
	> 30	> 1 500

Acceptance Values

ATLAS		
Baseline Parameters *(10V Full Scale)*		
		526 nm (P) Filter
Noise (µV)	< 500	< 500
Wander (µV)	< 1 000	< 1 000
Drift (µV/h)	< 1 000	< 1 000
Analytical Results		
Parathion Methyl Signal to Noise Ratio	**394 nm (S) Filter**	**526 nm (P) Filter**
	> 30	> 1 500

XCALIBUR		
Baseline Parameters *(Acquisition Frequency = 10 Hz)*		
		526 nm (P) Filter
Noise (Counts)	< 5 000	< 5 000
Wander (Counts)	< 10 000	< 10 000
Drift (Counts/h)	< 10 000	< 10 000
Analytical Results		
Parathion Methyl Signal to Noise Ratio	**394 nm (S) Filter**	**526 nm (P) Filter**
	> 30	> 1 500

Analytical Acceptance Comments	
1	Using Chrom-Card, set the signal-to-noise report parameters as described in the current procedure.
2	Using ChromQuest, Atlas, Xcalibur or a Computing integrator (e.g. ChromJet), calculate the S/N ratio as *Peak Height (counts)/noise (counts)*.

20

Checkout Using FPD with PPKD Injector

Chapter at a Glance...

Operating Procedures

SOP Number: P0319/08/E - 01 September 2009

Scope

Use the following procedure to verify proper FPD operation with Purged Packed Injector. This SOP is applicable both for the control card labeled **FPD** and for the control card labeled **FPD/F**.

Parts Referenced

Table 20-1. FPD-PPKD 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	2 mm ID tapered	453 220 50
Liner Seal	Graphite seal for glass liner	290 334 05
Pre-column	Fused Silica Capillary Column 0.5 mt long 0.53 mm ID	260 603 75
Retaining Nut	M4 capillary column retaining nut	350 324 23
Press-fit connections	For columns 0.53/0.32 mm ID	350 438 16
Injection side adapter	For Wide bore column	347 003 03
Retaining Nut	For Injection side adapter	350 024 04
Detector side adapter	For Wide bore column	347 103 04
Ferrule	6 mm ID double brass ferrule	290 341 37
Nut	1/4” G-6 mm ID nut	350 201 18
Graphite Ferrule	Graphite ferrule for 0.53 mm ID Column Graphite ferrule for 0.32 mm ID Column	290 134 86 290 134 87
Septum	Standard septum for Purged Packed Injector	313 032 26
Syringe	10 µl size; 50 mm needle length	365 005 25

Table 20-1. FPD-PPKD Parts Referenced (Continued)

Part	Description	Part Number
Interferential Filter	526 nm for phosphorus 394 nm for sulphur	281 071 00 281 070 00
Test Mixture	Three components in Iso-Octane: *Component* *Concentration* Azobenzene 1 µg/ml Octadecane 1000 µg/ml Parathion methyl 1 µg/ml	338 190 06
Gases	Chromatographic-grade purity	
Data Acquisition	Chrom-Card, ChromQuest, Atlas, Xcalibur, Computing-integrator	

Analytical Conditions Required for Purged Packed Injector

Table 20-2. FPD-PPKD Analytical Conditions

Parameters Setting	
Gases	Carrier Gas: Helium = 30 kPa Constant Pressure Hydrogen = 90 ml/min Air = 115 ml/min
Oven Program	Initial Temperature = 50 °C Initial Time = 1 minute Ramp 1 = 20 °C/minute Final Temperature = 200 °C Final Time = 1 minute
Injector	Operating Mode = Wide bore Temperature = 200 °C
Detector	Base Temperature = 300 °C FPD Temperature = 150 °C High voltage mode = No Detector Signal Range = 10^0 (see Note)
Injected Volume	1 µl + needle of Test Mixture
Analog Signal Output	Chrom-Card Acquisition Frequency = Medium
Digital Signal Output	Chrom-Card, ChromQuest, Atlas, Xcalibur Acquisition Frequency = 10 Hz

NOTE In the case your GC is equipped with the previous non-fast FPD control card, labeled **FPD**, set Detector Signal Range to 10^1.

Recommended Initial Operations

Before starting the checkout, the following operations should be carried out:

1. Insert the glass liner.
   Remove the column and insert the 2 mm ID tapered glass liner, as required for the checkout, from the bottom of the injector with the appropriate liner seal. Fix the liner by using the appropriate adapter for capillary column.
   Refer to the TRACE GC Ultra Maintenance and Troubleshooting Manual.

2. Mount the adapter for capillary column on the detector base body.

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

4. Connect the required gas lines
   Verify the required gas supplies are properly connected to your GC.

5. Install the precolumn.
   Connect the precolumn to the injector.

6. Install the test column
   Connect the test column to the precolumn by using the press fit connections provided.

7. Perform the manual leak check following the Operating Procedure "*Perforing a Leak Check* " in Chapter 14 of the TRACE GC Ultra Operating Manual.

8. Connect the other end of the test column to the detector base body.

9. Perform Column Evaluation.

10. Connect your data handling.
    Verify that your data handling is properly connected to your GC system.

OPERATING PROCEDURE

FPD-PPKD Checkout

Before beginning, press **CONFIG** to verify the GC configuration:

Left inlet or Right inlet	PPKD
Left carrier or Right carrier	He (helium)
Left detector or Right detector	FPD

1. Use **LEFT CARRIER** or **RIGHT CARRIER** to display the appropriate Carrier Gas Control Table. Verify to operate in constant pressure mode. If not, scroll to `Flow mode`, press **MODE/TYPE** to access the selection menu, then select `con pres`. Scrool to `Pressure` and set the pressure value to have the required carrier gas flow rate `Col.flow`.

```
        LEFT CARRIER[1]

Pressure          30.0   30.0
Col.flow          3.00
Lin. veloc.             (60.9)<
```

[1] These settings could also be for a right carrier.

2. Use **OVEN** to display the Column Oven Control Table. Set the oven temperature and the Oven Program required.

```
             OVEN

Temp              70.0   70.0
Initial Time             1.00
Ramp 1                   20.0
Final temp                250
Final time 1             1.00<
Ramp 2                    Off
```

3. Use **LEFT INLET** or **RIGHT INLET** to display the appropriate PPKD Injector Control Table. Set the required temperature setpoint `Temp`.Verify to operate in

Widebore mode. If not, scroll to `Mode`, press **MODE/TYPE** to access the selection menu, then select `Widebore`.

```
     LEFT INLET (PPKD)[1]
Temp                  200   200
Pressure             30.0  30.0
Mode:                 Wide bore<
Constant sept purge?         Y<
```

These settings could also be for a right inlet.

4. Use **LEFT DETECTOR** or **RIGHT DETECTOR** to display the appropriate FPD Detector Control Table. Set the required temperatures `Base Temp` and `FPD Temp`. Then, set the detector gases `H2` and `Air` required setpoints.

```
    LEFT DETECTOR (FPD)[1]
Flame                       Off
Base temp             300   300
FPD temp              150   150
Signal pA                 (1.4)
High voltage mode?            N
H2                     90    90
Air                   115   115
Mkup N2                00    00
```

These settings could also be for a right detector.

5. Verify that `High voltage mode` is set to **NO**.

6. Scroll to `Flame` and press **ON**. This start the ignition sequence.
   When ignition is confirmed, the photomultiplier tube is energized. The baseline level `Signal pA`, will suddenly increase meaning that the flame is lit inside the detector. After a few seconds, the baseline should stabilize to the standing current of the system.

7. Use **LEFT SIGNAL** or **RIGHT SIGNAL** to display the appropriate FPD Detector Signal Control Table. Scroll to `Range` and set the electrometer amplifier input range required.

LEFT SIGNAL (FPD)[1]	
Output	(1000)
Offset	100
Auto zero?	Y/N
Range 10^(0..3)	0<
Baseline comp	Off

These settings could also be for a right signal.

8. Activate your Data System and set the parameters required for the checkout.

9. In the FPD Detector Signal Control Table scroll to `Auto zero?` and turn it **YES**.

10. Perform a blank analysis injecting pure iso-octane and press **START** on the GC to begin the checkout run.

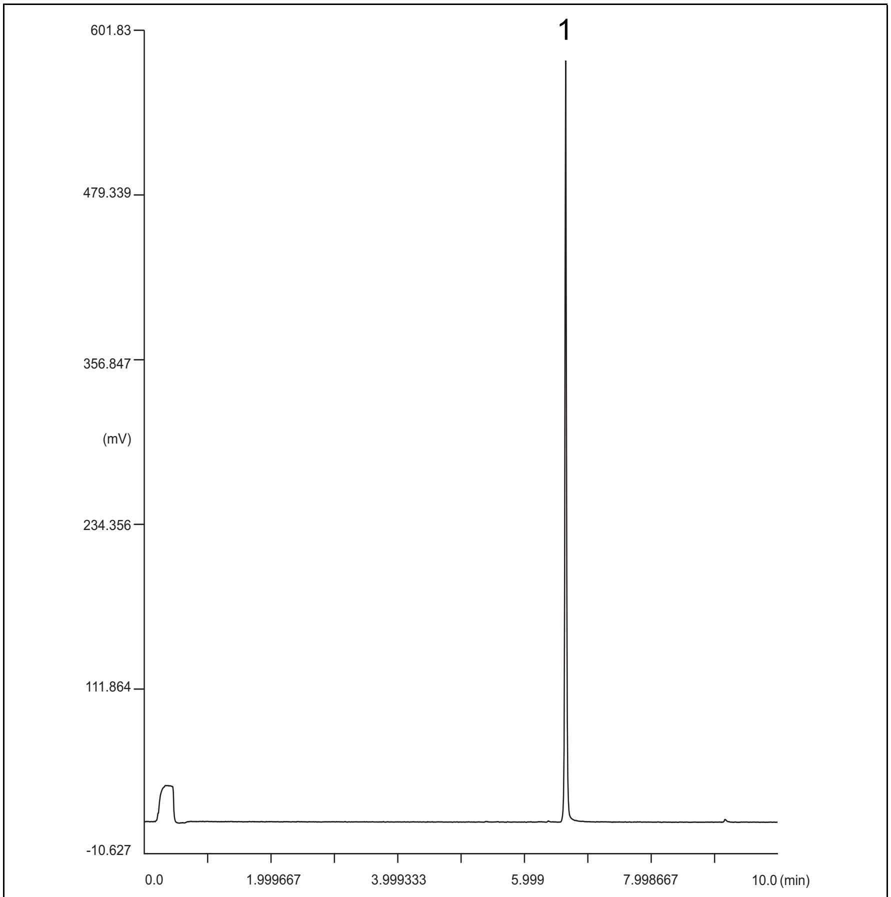
Baseline Acquisition and Analysis

NOTE Refer to the Acceptance Values reported in the Table 20-3 according to the data handling in use.

11. With the GC in Stand-by/Prep Run condition, activate the data system for 10 minutes to evaluate your baseline in isothermal condition.

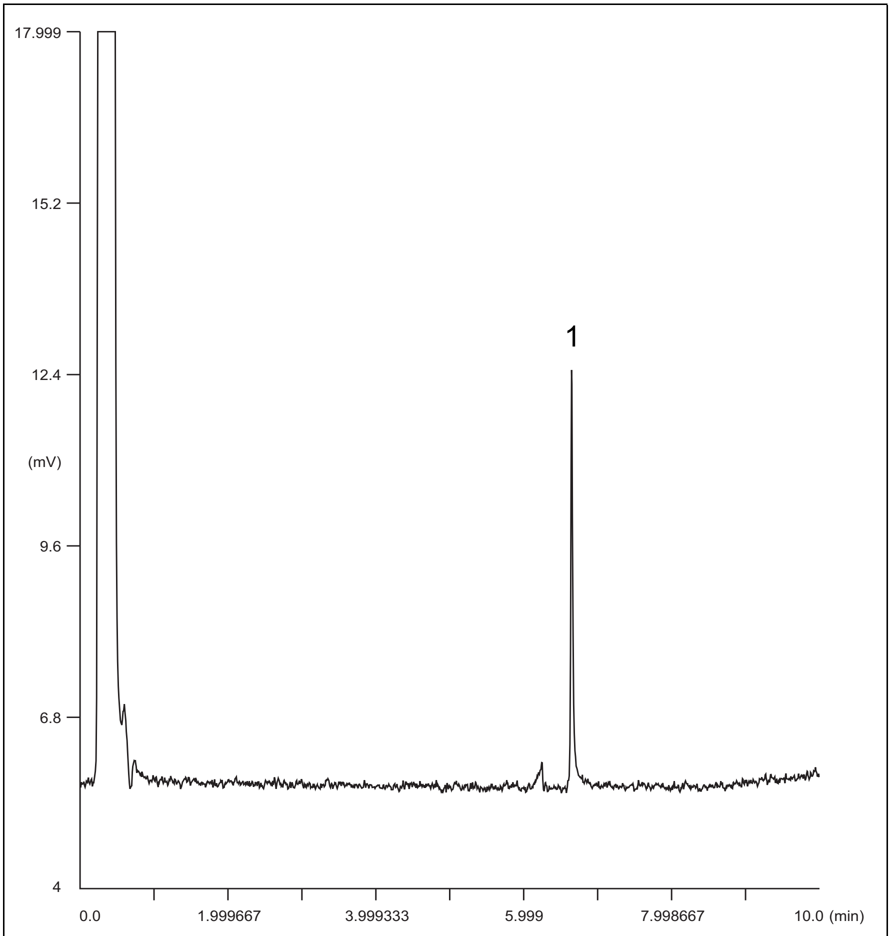
12. After the baseline evaluation has been completed, set-up the data system to acquire a single run.

13. When the GC is ready, inject the test mixture and press **START** on the GC to begin the checkout run. The resulting chromatogram should look like the one shown in *Figure 20.1* or *20.2*.



1. Parathion Methyl

Figure 20-1. FPD-PPKD Injection with 526 nm filter



1. Parathion Methyl

Figure 20-2. FPD-PPKD Injection with 394 nm

14. Establish the integration parameters and the peak table identifying the test mix components.

15. Set up the data system to calculate the signal-to-noise ratio.

Using Chrom-Card Data System

Operate as follows:

- Open the Method Editor and include the signal-to-noise Report into the Report Parameters Page.
- By clicking on the side icon, open the signal-to-noise calculation and set Methylparathion component as signal peak ID.
- Choose a part of baseline without peaks or interference signals and calculate the noise for 0.1 min. (Verify that the noise value is comparable with the one of the previous *Baseline Acquisition and Analysis*).
- Generate a report showing the chromatogram, peak area and signal-to-noise information for the component.

NOTE If it is not possible to find a suitable part of baseline, to have a complete Chrom-Card final report proceed as follows: Start the acquisition on the data system. Let the system to acquire for about 1 minute and then inject the test mixture. The noise can be calculated during the first minute of acquisition. Since the retention time will shift by a delayed time, the Component Table has to be updated.

Using non-Chrom-Card Data System

Operate as follows:

- Set the parameters to calculate the signal-to-noise ratio according to the instruction reported in the relevant data system manual.

CAUTION **Choose a part of baseline without peaks or interference signals and calculate the noise for 0.1 min. (Verify that the noise value is comparable with the one of the previous *Baseline Acquisition and Analysis*).**

16. The following criteria indicate successful completion of FPD-PPKD checkout.

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

Table 20-3. FPD-PPKD Acceptance Criteria

Acceptance Values	CHROM-CARD				
	Baseline Parameters	**Analog** *(1V Full Scale)*		**Digital** *(10V Full Scale)*	
		394 nm (S) Filter	**526 nm (P) Filter**	**394 nm (S) Filter**	**526 nm (P) Filter**
	Noise (μV)	< 50	< 50	< 500	< 500
	Wander (μV)	< 100	< 100	< 1 000	< 1 000
	Drift (μV/h)	< 100	< 100	< 1 000	< 1 000
	Analytical Results				
	Parathion Methyl Signal to Noise Ratio			**394 nm (S) Filter**	**526 nm (P) Filter**
				> 30	> 1 500

Acceptance Values	CHROMQUEST		
	Baseline Parameters *(1V Full Scale)*		
			526 nm (P) Filter
	Noise (μV)	< 50	< 50
	Wander (μV)	< 100	< 100
	Drift (μV/h)	< 100	< 100
	Analytical Results		
	Parathion Methyl Signal to Noise Ratio	**394 nm (S) Filter**	**526 nm (P) Filter**
		> 30	> 1 500

ATLAS		
Baseline Parameters *(10V Full Scale)*		
		526 nm (P) Filter
Noise (µV)	< 500	< 500
Wander (µV)	< 1 000	< 1 000
Drift (µV/h)	< 1 000	< 1 000
Analytical Results		
Parathion Methyl Signal to Noise Ratio	**394 nm (S) Filter**	**526 nm (P) Filter**
	> 30	> 1 500

Acceptance Values

XCALIBUR		
Baseline Parameters *(Acquisition Frequency = 10 Hz)*		
		526 nm (P) Filter
Noise (Counts)	< 5 000	< 5 000
Wander (Counts)	< 10 000	< 10 000
Drift (Counts/h)	< 10 000	< 10 000
Analytical Results		
Parathion Methyl Signal to Noise Ratio	**394 nm (S) Filter**	**526 nm (P) Filter**
	> 30	> 1 500

Analytical Acceptance Comments	
1	Using Chrom-Card, set the signal-to-noise report parameters as described in the current procedure.
2	Using ChromQuest, Atlas, Xcalibur or a Computing integrator (e.g. ChromJet), calculate the S/N ratio as *Peak Height (counts)/noise (counts)*.

21

Checkout Using FPD with PTV Injector

Chapter at a Glance…

Operating Procedures

SOP Number: P0320/08/E - 01 September 2009

Scope

Use the following procedure to verify proper FPD operation with the Programmable Temperature Vaporizing Injector. This SOP is applicable both for the control card labeled **FPD** and for the control card labeled **FPD/F**.

Parts Referenced

Table 21-1. FPD-PTV 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
Liner	Silcosteel 2 mm ID (set of 2)	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	365 005 25
Interferential Filter	526 nm for phosphorus 394 nm for sulphur	281 071 00 281 070 00
Test Mixture	Three components in Iso-Octane: *Component* *Concentration* Azobenzene 1 µg/ml Octadecane 1000 µg/ml Parathion methyl 1 µg/ml	338 190 06
Gases	Chromatographic-grade purity	
Data Acquisition	Chrom-Card, ChromQuest, Atlas, Xcalibur, Computing-integrator	

Analytical Conditions Required for PTV Splitless Injection

Table 21-2. FPD-PTV Analytical Conditions

Parameters	Setting
Gases	Carrier Gas: Helium = 30 kPa Constant Pressure Hydrogen = 90 ml/min Air = 115 ml/min
Oven Program	Initial Temperature = 70 °C Initial Time = 1 minute Ramp 1 = 20 °C/minute Final Temperature = 230 °C Final Time = 1 minute
Injector	Operating Mode = PTV splitless Splitless Time = 0.8 minutes Split Flow = 50 ml/min Constant Septum Purge = Yes Inject Temp = 50 °C Inject Time = 0.1 minute Transfer ramp = 10 °C/sec Transfer Temperature = 260 °C Transfer time = 1 minute
Detector	Base Temperature = 300 °C FPD Temperature = 150 °C High voltage mode = No Detector Signal Range = 10^0 (see Note)
Injected Volume	1 µl of Test Mixture
Analog Signal Output	Chrom-Card Acquisition Frequency = Medium
Digital Signal Output	Chrom-Card, ChromQuest, Atlas, Xcalibur Acquisition Frequency = 10 Hz

NOTE In the case your GC is equipped with the previous non-fast FPD control card, labeled **FPD**, set Detector Signal Range to 10^1.

Recommended Initial Operations

Before starting the checkout, the following operations should be carried out:

1. Replace the liner.
   The liner currently installed in your injector should be carefully removed and replaced with the 2 mm ID Silcolsteel liner, 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. Connect the required gas lines
   Verify the required gas supplies are properly connected to your GC.

4. Install the test column.
   The column currently installed should be carefully removed and replaced with the required test column.

5. Perform Column Evaluation and Leak Test.

6. Connect your data handling.
   Verify that your data handling is properly connected to your GC system.

OPERATING PROCEDURE

FPD-PTV Checkout in PTV Splitless Mode

Before beginning, press **CONFIG** to verify the GC configuration:

Right inlet	PTV
Right carrier	He (helium)
Right detector	FPD

1. Use **RIGHT CARRIER** to display the appropriate Carrier Gas Control Table. Verify to operate in constant pressure mode. If not, scroll to `Flow mode`, press **MODE/TYPE** to access the selection menu, then select `con pres`. Scrool to `Pressure` and set the pressure value to have the required carrier gas flow rate `Col.flow`.

```
        RIGHT CARRIER¹
Pressure          30.0   30.0
Col.flow          3.00
Lin. veloc.            (60.9)<
```

2. Use **OVEN** to display the Column Oven Control Table. Set the oven temperature and the Oven Program required.

```
              OVEN
Temp              70.0   70.0
Initial Time             1.00
Ramp 1                   20.0
Final temp                230
Final time 1             1.00<
Ramp 2                    Off
```

3. Use **RIGHT INLET** to display the appropriate Programmable Temperature Vaporizing Injector Control Table. Set the required temperature setpoint

`Temp`. Verify to operate in **PTV splitless** mode. If not, scroll to `Mode`, press **MODE/TYPE** to access the selection menu, then select `PTV splitless`. Scroll to `Splitless time` to set the required setpoint.

```
      RIGHT INLET (PTV)

Temp                  70   70
Pressure            30.0  30.0
Mode:            PTV Splitless
Total flow           (53.0)
Split Flow          50.0  50.0
Splitless time      0.80  0.80
Constant sept purge?        Y
Inject phase menu:          Y<
```

4. Scroll to `Inject phase menu`. Press **MODE/TYPE** to enter the **PTV Phase Menu**.

```
       PTV PHASE MENU

Ramped pressure?            N
Inject temp                50
Inject time               0.1
Transfer ramp              10
Transfer temp             260
Transfer time            1.00<
```

5. Select `Ramped pressure?` **NO**. Set the required `Inject temp` and *Inject time* setpoints as required. Then, set the `Transfer ramp`, the `Transfer temp` and the `Transfer time` required setpoints.

6. Use **RIGHT DETECTOR** to display the appropriate FPD Detector Control Table. Set the required temperatures `Base Temp` and `FPD Temp`. Then, set the detector gases `H2` and `Air` required setpoints.

RIGHT DETECTOR (FPD)		
Flame		Off
Base temp	300	300
FPD temp	150	150
Signal pA		(1.4)
High voltage mode?		N
H2	90	90
Air	115	115
Mkup N2	00	00

7. Verify that `High voltage mode` is set to **NO**.

8. Scroll to `Flame` and press **ON**. This start the ignition sequence.
   When ignition is confirmed, the photomultiplier tube is energized. The baseline level `Signal pA`, will suddenly increase meaning that the flame is lit inside the detector. After a few seconds, the baseline should stabilize to the standing current of the system.

9. Use **RIGHT SIGNAL** to display the appropriate FPD Detector Signal Control Table. Scroll to `Range` and set the electrometer amplifier input range required.

RIGHT SIGNAL (FPD)	
Output	(1000)
Offset	100
Auto zero?	Y/N
Range 10^(0..3)	0<
Baseline comp	Off

10. Activate your Data System and set the parameters required for the checkout.

11. In the FPD Detector Signal Control Table scroll to `Auto zero?` and turn it **YES**.

12. Perform a blank analysis injecting pure iso-octane and press **START** on the GC to begin the checkout run.

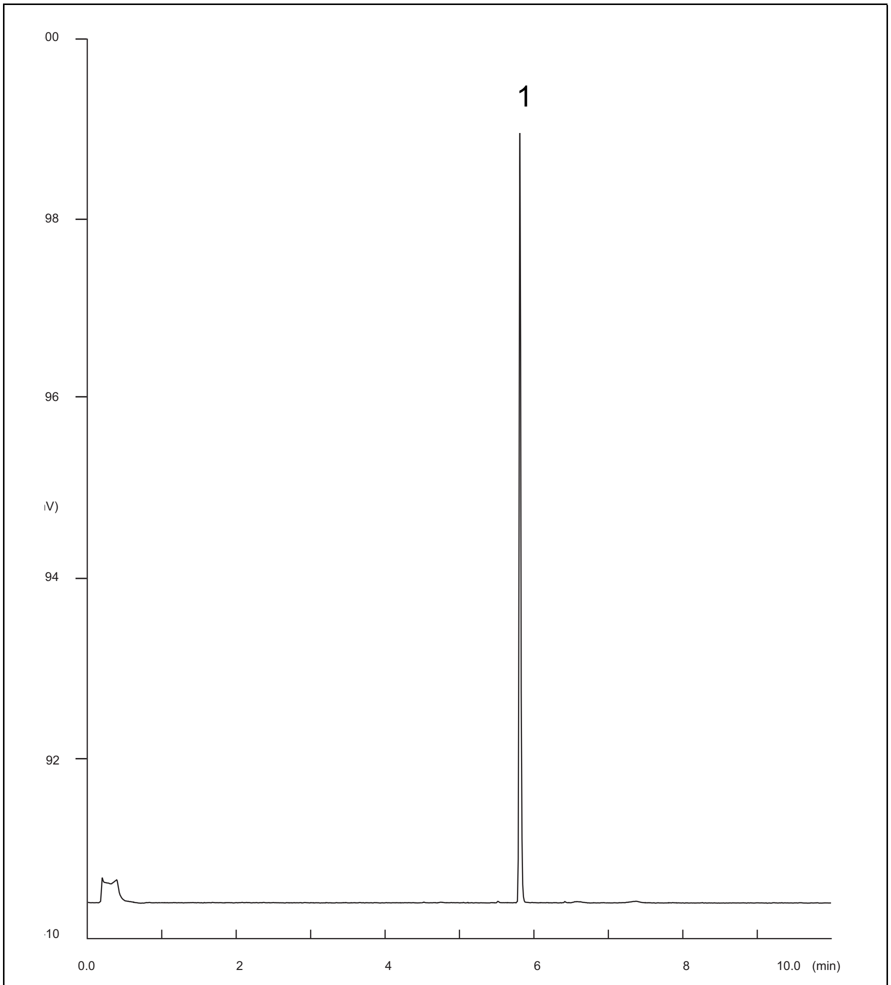
Baseline Acquisition and Analysis

NOTE Refer to the Acceptance Values reported in the Table 21-3 according to the data handling in use.

13. With the GC in Stand-by/Prep Run condition, activate the data system for 10 minutes to evaluate your baseline in isothermal condition.

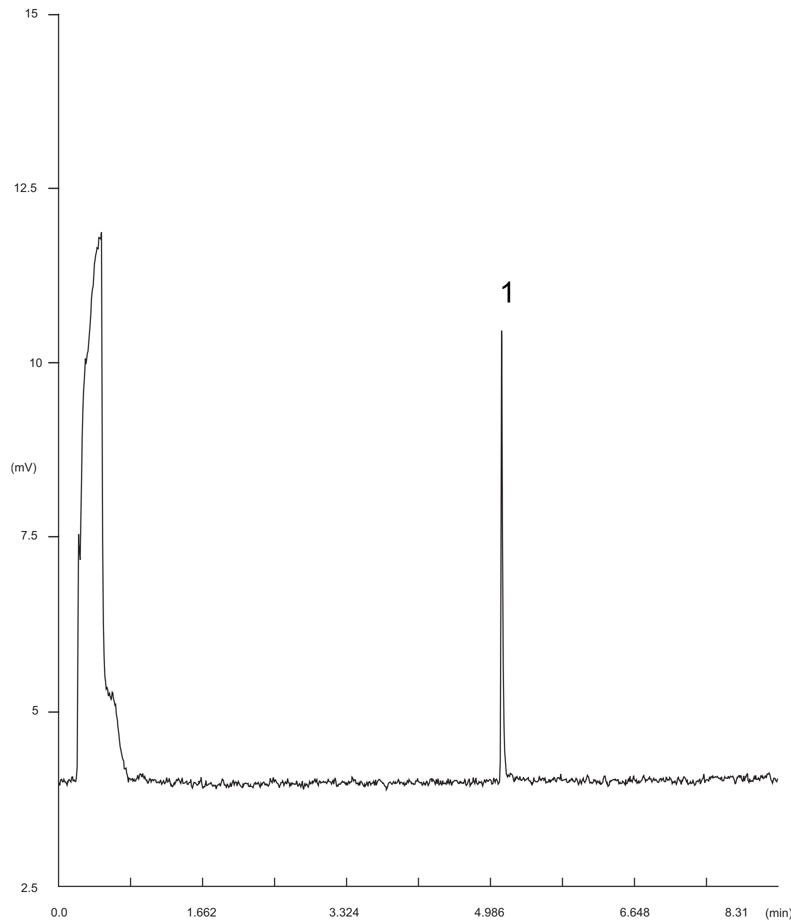
14. After the baseline evaluation has been completed, set-up the data system to acquire a single run.

15. When the GC is ready, inject the test mixture and press **START** on the GC to begin the checkout run. The resulting chromatogram should look like the one shown in *Figure 21.1* or *21.2*.



1. Parathion Methyl

Figure 21-1. FPD-PTV Injection with 526 nm filter



1. Parathion Methyl

Figure 21-2. FPD-PTV Injection with 394 nm filter

16. Establish the integration parameters and the peak table identifying the test mix components.

17. Set up the data system to calculate the signal-to-noise ratio.

Using Chrom-Card Data System

Operate as follows:

- Open the Method Editor and include the signal-to-noise Report into the Report Parameters Page.
- By clicking on the side icon, open the signal-to-noise calculation and set Methylparathion component as signal peak ID.
- Choose a part of baseline without peaks or interference signals and calculate the noise for 0.1 min. (Verify that the noise value is comparable with the one of the previous *Baseline Acquisition and Analysis*).
- Generate a report showing the chromatogram, peak area and signal-to-noise information for the component.

NOTE If it is not possible to find a suitable part of baseline, to have a complete Chrom-Card final report proceed as follows: Start the acquisition on the data system. Let the system to acquire for about 1 minute and then inject the test mixture. The noise can be calculated during the first minute of acquisition. Since the retention time will shift by a delayed time, the Component Table has to be updated.

Using non-Chrom-Card Data System

Operate as follows:

- Set the parameters to calculate the signal-to-noise ratio according to the instruction reported in the relevant data system manual.

CAUTION **Choose a part of baseline without peaks or interference signals and calculate the noise for 0.1 min. (Verify that the noise value is comparable with the one of the previous *Baseline Acquisition and Analysis*).**

18. The following criteria indicate successful completion of FPD-PTV checkout.

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

Table 21-3. FPD-PTV Acceptance Criteria

Acceptance Values	CHROM-CARD				
	Baseline Parameters	**Analog** *(1V Full Scale)*		**Digital** *(10V Full Scale)*	
		394 nm (S) Filter	**526 nm (P) Filter**	**394 nm (S) Filter**	**526 nm (P) Filter**
	Noise (µV)	< 50	< 50	< 500	< 500
	Wander (µV)	< 100	< 100	< 1 000	< 1 000
	Drift (µV/h)	< 100	< 100	< 1 000	< 1 000
	Analytical Results				
	Parathion Methyl Signal to Noise Ratio			**394 nm (S) Filter**	**526 nm (P) Filter**
				> 20	> 1 000
⚠					

Acceptance Values	CHROMQUEST		
	Baseline Parameters *(1V Full Scale)*		
			526 nm (P) Filter
	Noise (µV)	< 50	< 50
	Wander (µV)	< 100	< 100
	Drift (µV/h)	< 100	< 100
	Analytical Results		
	Parathion Methyl Signal to Noise Ratio	**394 nm (S) Filter**	**526 nm (P) Filter**
		> 20	> 1 000
⚠			

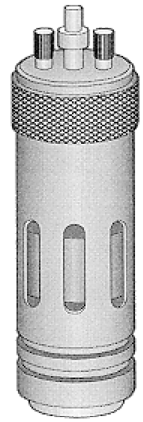
Acceptance Values	ATLAS		
	Baseline Parameters *(10V Full Scale)*		
			526 nm (P) Filter
	Noise (µV)	< 500	< 500
	Wander (µV)	< 1 000	< 1 000
	Drift (µV/h)	< 1 000	< 1 000
	Analytical Results		
	Parathion Methyl Signal to Noise Ratio	**394 nm (S) Filter**	**526 nm (P) Filter**
		> 20	> 1 000
⚠			

Acceptance Values	XCALIBUR		
	Baseline Parameters *(Acquisition Frequency = 10 Hz))*		
			526 nm (P) Filter
	Noise (Counts)	< 5 000	< 5 000
	Wander (Counts)	< 10 000	< 10 000
	Drift (Counts/h)	< 10 000	< 10 000
	Analytical Results		
	Parathion Methyl Signal to Noise Ratio	**394 nm (S) Filter**	**526 nm (P) Filter**
		> 20	> 1 000
⚠			

Analytical Acceptance Comments	
1	Using Chrom-Card, set the signal-to-noise report parameters as described in the current procedure.
2	Using ChromQuest, Atlas, Xcalibur or a Computing integrator (e.g. ChromJet), calculate the S/N ratio as *Peak Height (counts)/noise (counts)*.

SECTION VI

SOPs Using PID



The *SOPs Using PID* section, contains the procedures to test the TRACE GC Ultra with the Photoionization Detector (PID) using different injectors.

22

Checkout Using PID with S/SL Injector

Chapter at a Glance...

Operating Procedures

SOP Number: P0321/09/E - 01 September 2009

Scope

Use the following procedure to verify proper PID operation with the Split/Splitless Injector.

Parts Referenced

Table 22-1. PID-S/SL 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; 70 mm needle length	365 001 03
Test Mixture	Three components in Iso-Octane: *Component* *Concentration* Azobenzene 1 µg/ml Octadecane 1000 µg/ml Parathion Methyl 1 µg/ml	338 190 06
Detector UV Lamp	8.4 eV 9.6 eV 10.6 eV 11.8 eV	305 030 13 305 030 14 305 030 15 305 030 16
Gases	Chromatographic-grade purity	

Table 22-1. PID-S/SL Parts Referenced (Continued)

Part	Description	Part Number
Data Acquisition	Chrom-Card, ChromQuest, Atlas, Xcalibur, Computing-integrator	

Analytical Conditions Required for Splitless Injection

Table 22-2. PID-S/SL Analytical Conditions

Parameters Setting	
Gases	Carrier Gas: Helium = 30 kPa Constant Pressure Make-up: Nitrogen = 7 ml/min Sheath Gas: Nitrogen = 40 ml/min
Oven Program	Initial Temperature = 70 °C Initial Time = 1 minute Ramp 1 = 20 °C/minute Final Temperature = 230 °C Final Time = 1 minute
Injector	Operating Mode = Splitless Temperature = 230 °C Splitless Time = 0.8 minutes Split Flow = 60 ml/min Constant Septum Purge = Yes
Detector	Base Temperature = 230 °C High Current = No Detector Signal Range = 10^0
Injected Volume	1 µl + needle of Test Mixture
Analog Signal Output	Chrom-Card Acquisition Frequency = Medium
Digital Signal Output	Chrom-Card, ChromQuest, Atlas, Xcalibur Acquisition Frequency = 10 Hz

Recommended Initial Operations

Before starting the checkout, the following operations should be carried out:

1. Replace the glass liner.
   The glass liner currently installed in your injector should be carefully removed and replaced with the 3 mm ID glass liner for splitless application, as required for the checkout, with the appropriate graphite seal.

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

3. Connect the required gas lines
   Verify the required gas supplies are properly connected to your GC.

4. Install the test column.
   The column currently installed should be carefully removed and replaced with the required test column.

5. Perform Column Evaluation and Leak Test

6. Connect your data handling.
   Verify that your data handling is properly connected to your GC system.

OPERATING PROCEDURE

PID-S/SL Checkout in Splitless Mode

Before beginning, press **CONFIG** to verify the GC configuration:

Left inlet or Right inlet	S/SL
Left carrier or Right carrier	He (helium)
Left detector or Right detector	PID

1. Use **LEFT CARRIER** or **RIGHT CARRIER** to display the appropriate Carrier Gas Control Table. Verify to operate in constant pressure mode. If not, scroll to `Flow mode`, press **MODE/TYPE** to access the selection menu, then select `con pres`. Scrool to `Pressure` and set the pressure value to have the required carrier gas flow rate `Col.flow`.

```
          LEFT CARRIER[1]

Pressure          30.0   30.0
Col.flow          3.00
Lin. veloc.             (60.9)<
```

1. These settings could also be for a right carrier.

2. Use **OVEN** to display the Column Oven Control Table. Set the oven temperature and the Oven Program required.

```
              OVEN

Temp              70.0  70.0
Initial Time            1.00
Ramp 1                  20.0
Final temp               230
Final time 1            1.00<
Ramp 2                   Off
```

3. Use **LEFT INLET** or **RIGHT INLET** to display the appropriate Split/Splitless Injector Control Table and set the required temperature setpoint *Temp*. Verify

to operate in **Splitless** mode. If not, scroll to `Mode`, press **MODE/TYPE** to access the selection menu, then select `Splitless`. Scroll to `Splitless time` to set the required setpoint.

LEFT INLET (S/SL)[1]		
Temp	230	230
Pressure	30.0	30.0
Mode:		Splitless
Total flow		(63.0)
Split Flow	60.0	60.0
Splitless time	0.80	0.80
Constant sept purge?		Y<

1. These settings could also be for a right inlet.

4. Use **LEFT DETECTOR** or **RIGHT DETECTOR** to display the appropriate PID Detector Control Table. Set the required temperature *Base Temp* and the detector gases `Mkup` and `Sheath Gas` required setpoints.

LEFT DETECTOR (PID)[1]		
Lamp		On
Base temp	230	230
High current		N
Signal pA		(15.4)
Mkup N2	7.0	7.0
Sheath Gas	40	40

1. These settings could also be for a right detector.

5. Verify that `High current` is set to **NO**.

6. Scroll to `Lamp` and press **ON**. This start the ignition sequence. The baseline level `Signal pA`, will suddenly increase meaning that the lamp is lit inside the detector. A short period of conditioning is required in order to obtain a stable baseline.

7. Use **LEFT SIGNAL** or **RIGHT SIGNAL** to display the appropriate PID Detector Signal Control Table. Scroll to `Range` and set the electrometer amplifier input range required.

LEFT SIGNAL (PID)[1]	
Output	(1000)
Offset	100
Auto zero?	Y/N
Range 10^(0..3)	0<
Analog filter	Off
Baseline comp	Off

1. These settings could also be for a right signal.

8. Activate your Data System and set the parameters required for the checkout.

9. In the PID Detector Signal Control Table scroll to `Auto zero?` and turn it **YES**.

10. Perform a blank analysis injecting pure iso-octane and press **START** on the GC to begin the checkout run.

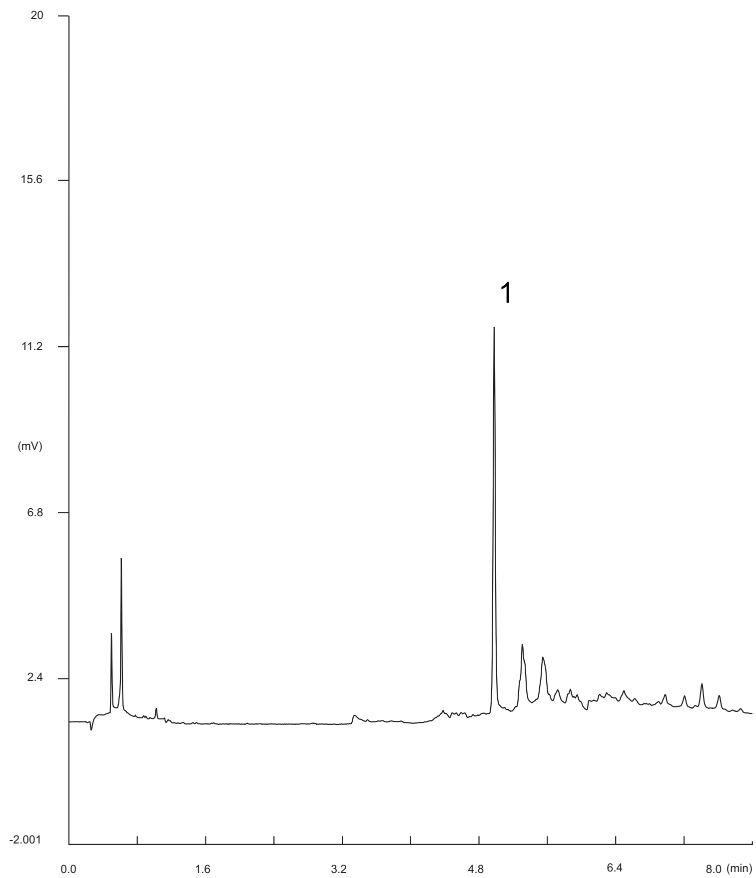
Baseline Acquisition and Analysis

NOTE Refer to the Acceptance Values reported in the Table 22-3 according to the data handling in use.

11. With the GC in Stand-by/Prep Run condition, activate the data system for 10 minutes to evaluate your baseline in isothermal condition.

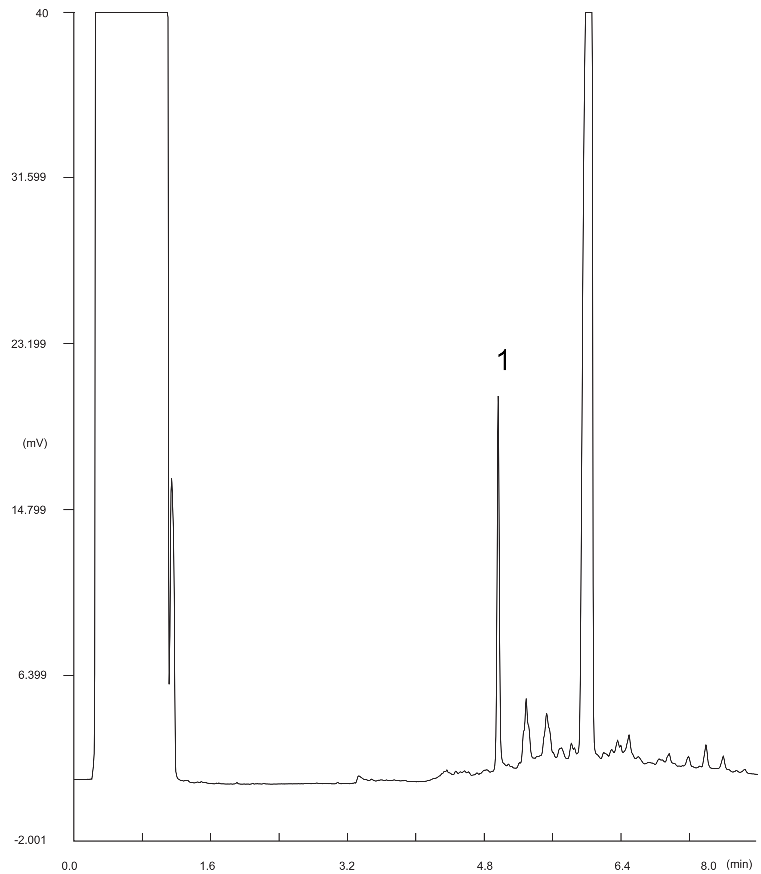
12. After the baseline the baseline evaluation has been completed, set-up the data system to acquire a single run.

13. Inject the test mixture and press **START** on the GC to begin the checkout run. The resulting chromatograms should look like the one shown in *Figures 22.1*, *22.2*, *22.3 or 22.4*.



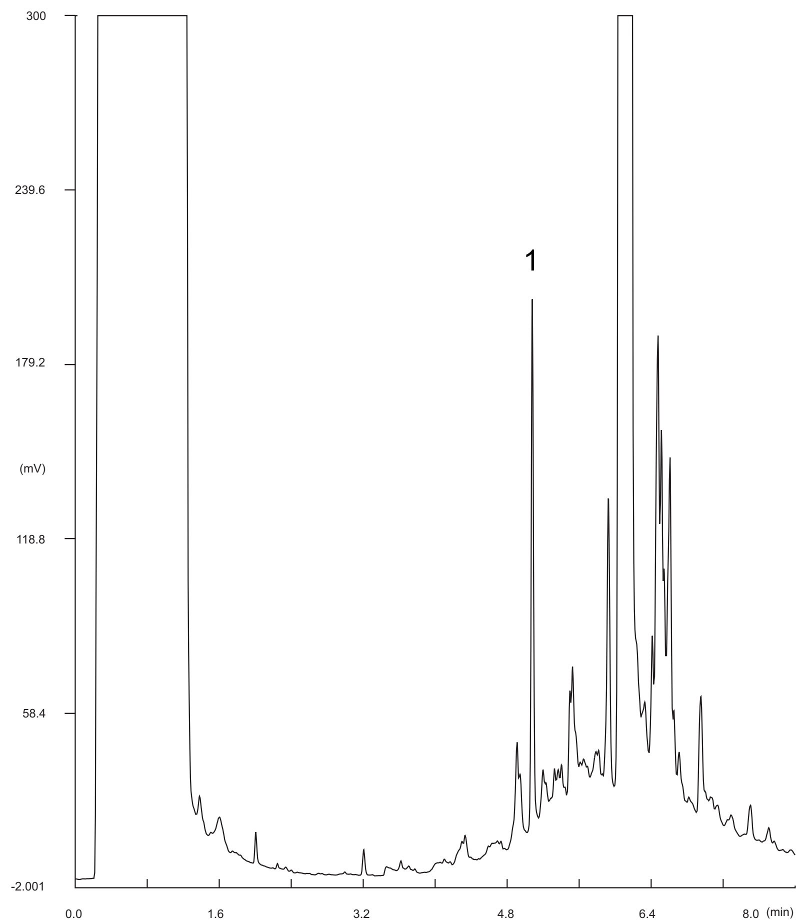
1. Azobenzene

Figure 22-1. PID-Splitless Injection with 8.4 eV UV Lamp



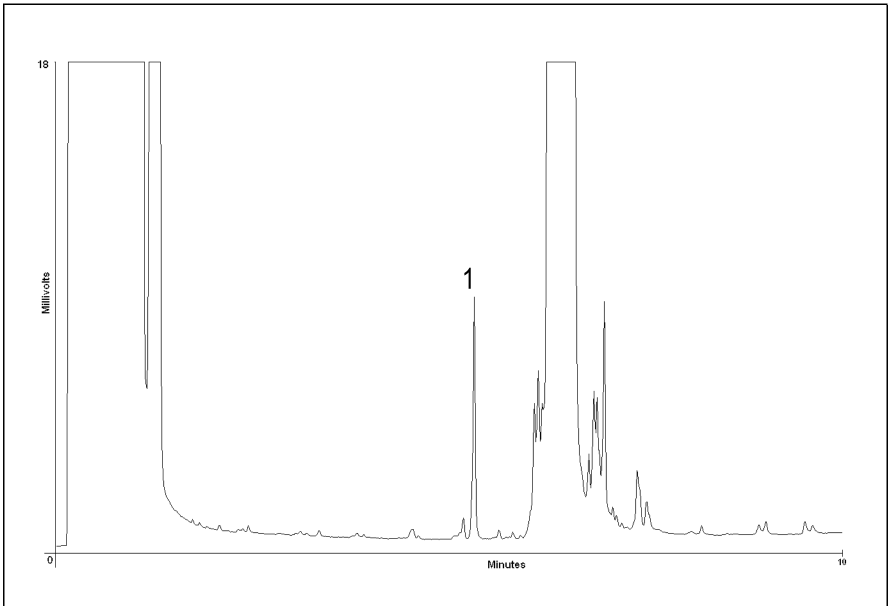
1. Azobenzene

Figure 22-2. PID-Splitless Injection with 9.6 eV UV Lamp



1. Azobenzene

Figure 22-3. PID-Splitless Injection with 10.6 eV UV Lamp



1. Azobenzene

Figure 22-4. PID-Splitless Injection with 11.8 eV UV Lamp

14. The following criteria indicate successful completion of PID-S/SL checkout.

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

Table 22-3. PID-S/SL Acceptance Criteria

Acceptance Values

CHROM-CARD								
Baseline Parameters	**Analog** *(1V Full Scale)*				**Digital** *(10V Full Scale)*			
Lamp (eV)	**8.4**	**9.6**	**10.6**	**11.8**	**8.4**	**9.6**	**10.6**	**11.8**
Noise (µV)	< 100	< 50	< 150	< 150	< 1 000	< 500	< 1 500	< 1 500
Wander (µV)	< 150	< 100	< 100	< 100	< 1 500	< 1 000	< 1 000	< 1 000
Drift (µV/h)	< 100	< 100	< 100	< 100	< 1 000	< 1 000	< 1 000	< 1 000
Analytical Results	**Analog** *(1V Full Scale) Area Counts (0.1 µVs)*				**Digital** *(10V Full Scale) Area Counts (0.1 µVs)*			
Lamp (eV)	**8.4**	**9.6**	**10.6**	**11.8**	**8.4**	**9.6**	**10.6**	**11.8**
Azobenzene	> 100*	> 160*	> 2 000*	> 40*	> 1 000*	> 1 600*	> 20 000*	> 400*

Computing-integrator (e.g. ChromJet)

Acceptance Values

CHROMQUEST				
Baseline Parameters *(1V Full Scale)*				
Lamp (eV)	**8.4**	**9.6**	**10.6**	**11.8**
Noise (µV)	< 100	< 50	< 150	< 150
Wander (µV)	< 150	< 100	< 100	< 100
Drift (µV/h)	< 100	< 100	< 100	< 100
Analytical Results *(1V Full Scale) - Area Counts (0.01 µVs)*				
Lamp (eV)	**8.4**	**9.6**	**10.6**	**11.8**
Azobenzene	> 1 000 000	> 1 600 000	> 20 000 000	> 400 000

Acceptance Values

ATLAS				
Baseline Parameters *(10V Full Scale)*				
Lamp (eV)	**8.4**	**9.6**	**10.6**	**11.8**
Noise (µV)	< 1 000	< 500	< 1 500	< 1 500
Wander (µV)	< 1 500	< 1 000	< 1 000	< 1 000
Drift (µV/h)	< 1 000	< 1 000	< 1 000	< 1 000
Analytical Results *(10V Full Scale) - Area Counts (µVs)*				
Lamp (eV)	**8.4**	**9.6**	**10.6**	**11.8**
Azobenzene	> 100 000	> 160 000	> 2 000 000	> 40 000

XCALIBUR				
Baseline Parameters *(Acquisition Frequency = 10 Hz)*				
Lamp (eV)	**8.4**	**9.6**	**10.6**	**11.8**
Noise (Counts)	< 10 000	< 5 000	< 15 000	< 15 000
Wander Counts)	< 15 000	< 10 000	< 10 000	< 10 000
Drift (Counts/h)	< 10 000	< 10 000	< 10 000	< 10 000
Analytical Results *Area Counts (Cts*s)*				
Lamp (eV)	**8.4**	**9.6**	**10.6**	**11.8**
Azobenzene	> 1 000 000	> 1 600 000	> 20 000 000	> 400 000

23

Checkout Using PID with OC Injector

Chapter at a Glance...

Operating Procedures

SOP Number: P0322/09/E - 01 September 2009

Scope

Use the following procedure to verify proper PID operation with the On-Column Injector.

Parts Referenced

Table 23-1. PID-OCI 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
Retaining Nut	M4 capillary column retaining nut	350 324 23
Vespel Ferrule	Vespel Ferrule for 0.32 mm ID Column	290 134 60
Syringe	10 µl size; 75 mm needle length	365 020 07
Test Mixture	Three components in Iso-Octane: *Component* *Concentration* Azobenzene 1 µg/ml Octadecane 1000 µg/ml Parathion methyl 1 µg/ml	338 190 06
Detector UV Lamp	8.4 eV 9.6 eV 10.6 eV 11.8 eV	305 030 13 305 030 14 305 030 15 305 030 16
Gases	Chromatographic-grade purity	
Data Acquisition	Chrom-Card, ChromQuest, Atlas, Xcalibur, Computing-integrator	

Table 23-1. PID-OCI Parts Referenced (Continued)

Part	Description	Part Number
In case of automatic On-Column for TriPlus Sampler AS		
Syringe	10 µl size; 80 mm needle length	365 020 19
Pre-column	2 m long; 0.53 mm ID	260 603 75
Press-fit set	Set of Press-fir connectors for TRACE OC	350 038 45

Analytical Conditions Required for On-Column Injection

Table 23-2. PID-OCI Analytical Conditions

Gases	Carrier Gas: Helium = 30 kPa Constant Pressure Make-up: Nitrogen = 7 ml/min Sheath Gas: Nitrogen = 40 ml/min
Oven Program	Initial Temperature = 85 °C Initial Time = 1 minute Ramp 1 = 20 °C/minute Final Temperature = 230 °C Final Time = 1 minute
Injector	Secondary cooling = 0.2 minutes
Detector	Base Temperature = 230 °C High Current = No Detector Signal Range = 10^0
Injected Volume	1 µl of Test Mixture
Analog Signal Output	Chrom-Card Acquisition Frequency = Medium
Digital Signal Output	Chrom-Card, ChromQuest, Atlas, Xcalibur Acquisition Frequency = 10 Hz

Recommended Initial Operations

Before starting the checkout, the following operations should be carried out:

1. Connect the required gas lines
   Verify the required gas supplies are properly connected to your GC.

2. Install the test column.
   The column currently installed should be carefully removed and replaced with the required test column.
   In case of automatic On-column for TriPlus sampler, install the pre-column and connect it to the test column by press-fit connector.

3. Install and connect the TriPlus sampler and its components.

4. Perform Column Evaluation and Leak Test.

5. Connect your data handling.
   Verify that your data handling is properly connected to your GC system.

6. Verify the opening/closing of the OC injector actuator by using the proper commands.

7. Verify the alignment of the syringe on the OC injector.

OPERATING PROCEDURE

PID-OCI Checkout in On-Column Mode

Before beginning, press **CONFIG** to verify the GC configuration:

Left inlet or Right inlet	OCI
Left carrier or Right carrier	He (helium)
Left detector or Right detector	PID

1. Use **LEFT CARRIER** or **RIGHT CARRIER** to display the appropriate Carrier Gas Control Table. Verify to operate in constant pressure mode. If not, scroll to `Flow mode`, press **MODE/TYPE** to access the selection menu, then select `con pres`. Scrool to `Pressure` and set the pressure value to have the required carrier gas flow rate `Col.flow`.

```
        LEFT CARRIER[1]
Pressure          30.0   30.0
Col.flow          3.00
Lin. veloc.            (60.9)<
```

1. These settings could also be for a right carrier.

2. Use **OVEN** to display the Column Oven Control Table. Set the oven temperature and the Oven Program required.

```
              OVEN
Temp              85.0   85.0
Initial Time             1.00
Ramp 1                   20.0
Final temp                230
Final time 1             1.00<
Ramp 2                    Off
```

3. Use **LEFT INLET** or **RIGHT INLET** to display the appropriate On-Column Injector Control Table. Scroll to `Sec. cool time` and set the required secondary cooling time.

```
      LEFT INLET (OCI)¹

Pressure         30.0  30.0
Sec. Cool Time        10.00<
```

1. These settings could also be for a right inlet.

4. Use **LEFT DETECTOR** or **RIGHT DETECTOR** to display the appropriate PID Detector Control Table. Set the required temperatures `Base Temp` and the detector gases `Mkup` and `Sheath Gas` required setpoints.

```
     LEFT DETECTOR (PID)¹

Lamp                       On
Base temp         230   230
High current                N
Signal pA              (15.4)
Mkup N2            7.0  7.0
Sheath Gas         40    40
```

1. These settings could also be for a right detector.

5. Verify that `High current` is set to **NO**.

6. Scroll to `Lamp` and press **ON**. This start the ignition sequence. The baseline level `Signal pA`, will suddenly increase meaning that the lamp is lit inside the detector. A short period of conditioning is required in order to obtain a stable baseline.

7. Use **LEFT SIGNAL** or **RIGHT SIGNAL** to display the appropriate PID Detector Signal Control Table. Scroll to `Range` and set the electrometer amplifier input range required.

LEFT SIGNAL (PID)[1]	
Output	(1000)
Offset	100
Autozero?	Y/N
Range 10^(0..3)	0<
Analog filter	Off
Baseline comp	Off

1. These settings could also be for a right signal.

8. Activate your Data System and set the parameters required for the checkout.

9. In the PID Detector Signal Control Table scroll to *Auto zero?* and turn it **YES**.

10. Perform a blank analysis injecting pure iso-octane and press **START** on the GC to begin the checkout run.

Baseline Acquisition and Analysis

NOTE Refer to the Acceptance Values reported in the Table 23-3 according to the data handling in use.

11. With the GC in Stand-by/Prep Run condition, activate the data system for 10 minutes to evaluate your baseline in isothermal condition.

12. After the baseline evaluation has been completed, set-up the data system to acquire a single run.

13. Perform the analysis.

    Manual injection

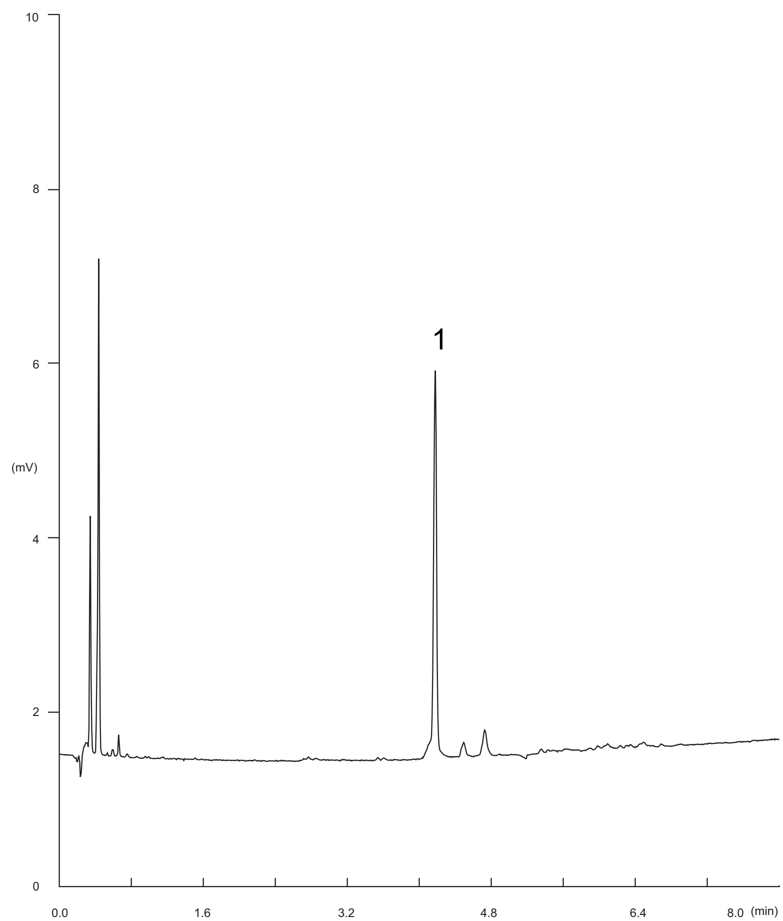
    - Inject the test mixture and press **START** on the GC to begin the checkout run.

    Automatic injection with TriPlus sampler

    - Fill a vial with the standard mix and place that vial in the sample tray.

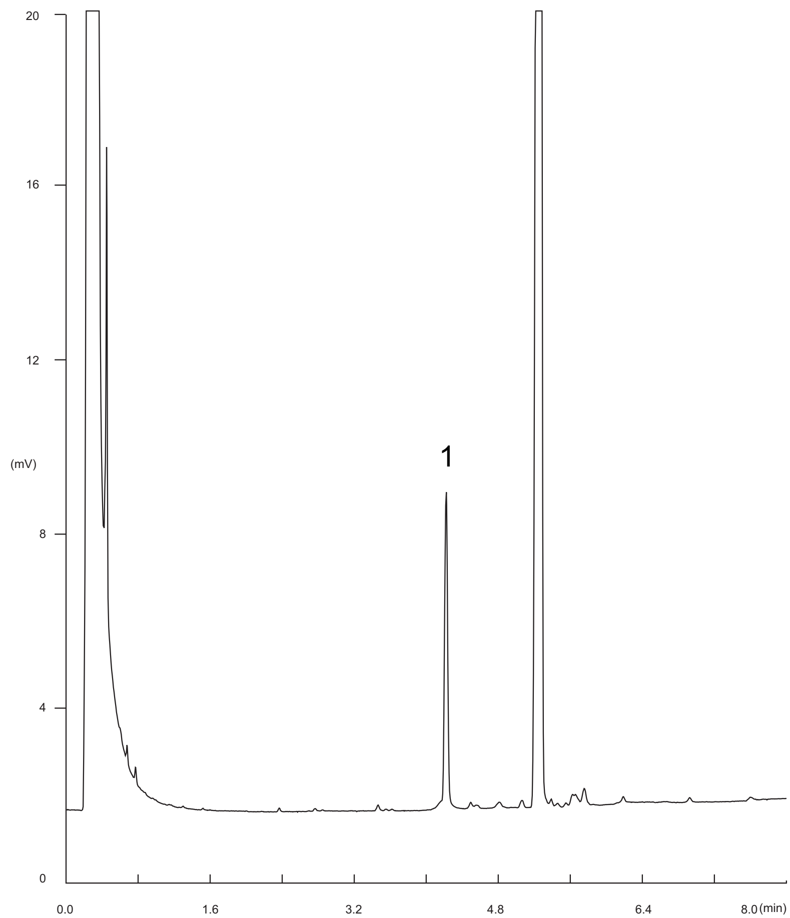
- Load the method for OC and perform the sampling.

The resulting chromatograms should look like the one shown in *Figures 23.1*, *23.2*, *23.3 or 23.4*.



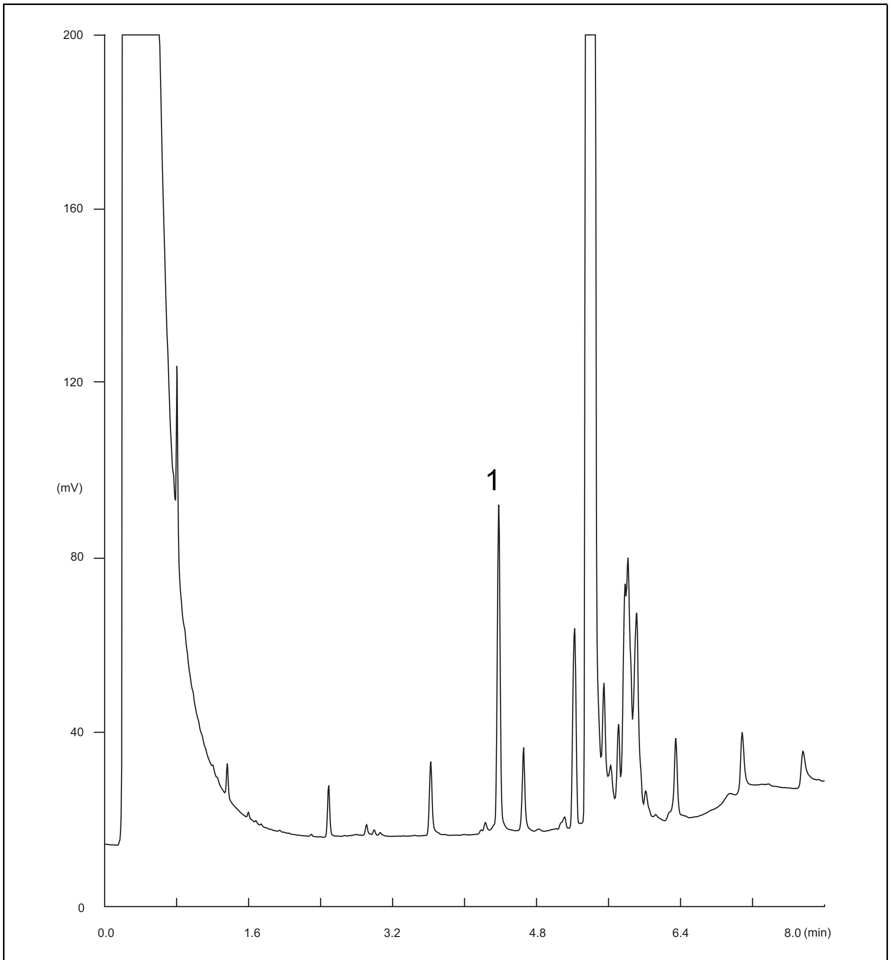
1. Azobenzene

Figure 23-1. PID-On-Column Injection with 8.4 eV UV Lamp



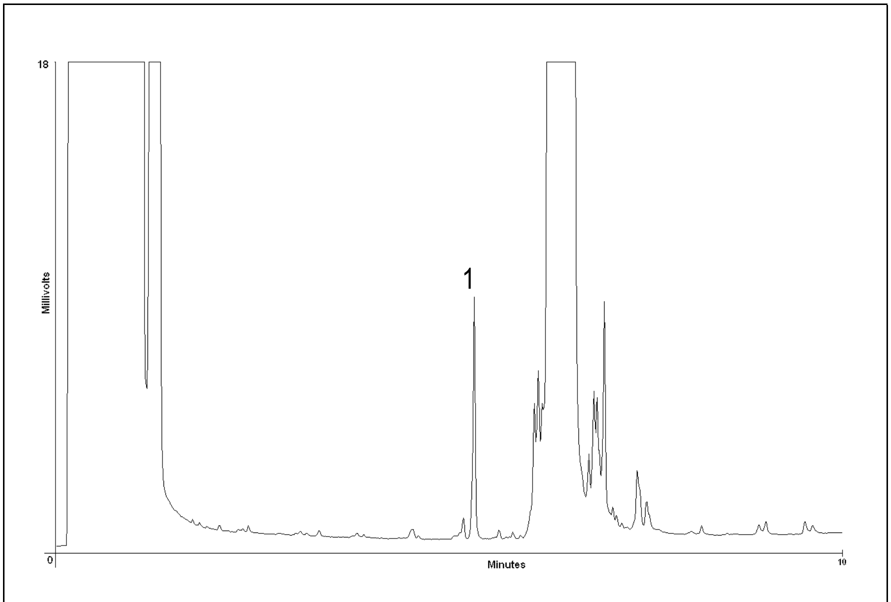
1. Azobenzene

Figure 23-2. PID-On-Column Injection with 9.6 eV UV Lamp



1. Azobenzene

Figure 23-3. PID-On-Column Injection with 10.6 eV UV Lamp



1. Azobenzene

Figure 23-4. PID-On-Column Injection with 11.8 eV UV Lamp

14. The following criteria indicate successful completion of PID-OCI checkout.

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

Table 23-3. PID-OCI Acceptance Criteria

Acceptance Values

CHROM-CARD								
Baseline Parameters	**Analog** *(1V Full Scale)*				**Digital** *(10V Full Scale)*			
Lamp (eV)	**8.4**	**9.6**	**10.6**	**11.8**	**8.4**	**9.6**	**10.6**	**11.8**
Noise (µV)	< 100	< 50	< 150	< 150	< 1 000	< 500	< 1 500	< 1 500
Wander (µV)	< 150	< 100	< 100	< 100	< 1 500	< 1 000	< 1 000	< 1 000
Drift (µV/h)	< 100	< 100	< 100	< 100	< 1 000	< 1 000	< 1 000	< 1 000
Analytical Results	**Analog** *(1V Full Scale) Area Counts (0.1 µVs)*				**Digital** *(10V Full Scale) Area Counts (0.1 µVs)*			
Lamp (eV)	**8.4**	**9.6**	**10.6**	**11.8**	**8.4**	**9.6**	**10.6**	**11.8**
Azobenzene	> 60*	> 110*	> 1 300*	> 24*	> 600*	> 1 100*	> 13 000*	> 240*

Computing-integrator (e.g. ChromJet)

Acceptance Values

CHROMQUEST				
Baseline Parameters *(1V Full Scale)*				
Lamp (eV)	**8.4**	**9.6**	**10.6**	**11.8**
Noise (µV)	< 100	< 50	< 150	< 150
Wander (µV)	< 150	< 100	< 100	< 100
Drift (µV/h)	< 100	< 100	< 100	< 100
Analytical Results *(1V Full Scale) - Area Counts (0.01 µVs)*				
Lamp (eV)	**8.4**	**9.6**	**10.6**	**11.8**
Azobenzene	> 600 000	> 1 100 000	> 13 000 000	> 240 000

Acceptance Values

ATLAS				
Baseline Parameters *(10V Full Scale)*				
Lamp (eV)	**8.4**	**9.6**	**10.6**	**11.8**
Noise (µV)	< 1 000	< 500	< 1 500	< 1 500
Wander (µV)	< 1 500	< 1 000	< 1 000	< 1 000
Drift (µV/h)	< 1 000	< 1 000	< 1 000	< 1 000
Analytical Results *(10V Full Scale) - Area Counts (µVs)*				
Lamp (eV)	**8.4**	**9.6**	**10.6**	**11.8**
Azobenzene	> 60 000	> 110 000	> 1 300 000	> 24 000

XCALIBUR				
Baseline Parameters *(Acquisition Frequency = 10 Hz)*				
Lamp (eV)	**8.4**	**9.6**	**10.6**	**11.8**
Noise (Counts)	< 10 000	< 5 000	< 15 000	< 15 000
Wander Counts)	< 15 000	< 10 000	< 10 000	< 10 000
Drift (Counts/h)	< 10 000	< 10 000	< 10 000	< 10 000
Analytical Results *Area Counts (Cts*s)*				
Lamp (eV)	**8.4**	**9.6**	**10.6**	**11.8**
Azobenzene	> 600 000	> 1 100 000	> 13 000 000	> 240 000

24

Checkout Using PID with PTV Injector

Chapter at a Glance...

Operating Procedures

SOP Number: P0325/08/E - 01 September 2009

Scope

Use the following procedure to verify proper PID operation with the Programmable Temperature Vaporizing Injector.

Parts Referenced

Table 24-1. PID-PTV 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
Liner	Silcosteel 2 mm ID (set of 2)	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	365 005 25
Test Mixture	Three components in Iso-Octane: *Component* *Concentration* Azobenzene 1 µg/ml Octadecane 1000 µg/ml Parathion methyl 1 µg/ml	338 190 06
Detector UV Lamp	8.4 eV 9.6 eV 10.6 eV 11.8 eV	305 030 13 305 030 14 305 030 15 305 030 16
Gases	Chromatographic-grade purity	

Table 24-1. PID-PTV Parts Referenced (Continued)

Part	Description	Part Number
Data Acquisition	Chrom-Card, ChromQuest, Atlas, Xcalibur, Computing-integrator	

Analytical Conditions Required for PTV Splitless Injection

Table 24-2. PID-PTV Analytical Conditions

Gases	Carrier Gas: Helium = 30 kPa Constant Pressure Make-up: Nitrogen = 7 ml/min Sheath Gas: Nitrogen = 40 ml/min
Oven Program	Initial Temperature = 70 °C Initial Time = 1 minute Ramp 1 = 20 °C/minute Final Temperature = 230 °C Final Time = 1 minute
Injector	Operating Mode = PTV splitless Splitless Time = 0.8 minutes Split Flow = 50 ml/min Constant Septum Purge = Yes Inject Temp = 50 °C Inject Time = 0.1 minute Transfer ramp = 10 °C/sec Transfer Temperature = 260 °C Transfer time = 1 minute
Detector	Base Temperature = 230 °C High Current = No Detector Signal Range = 10^0
Injected Volume	1 µl of Test Mixture
Analog Signal Output	Chrom-Card Acquisition Frequency = Medium
Digital Signal Output	Chrom-Card, ChromQuest, Atlas, Xcalibur Acquisition Frequency = 10 Hz

Recommended Initial Operations

Before starting the checkout, the following operations should be carried out:

1. Replace the liner.
   The liner currently installed in your injector should be carefully removed and replaced with the 2 mm ID Silcosteel liner, 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. Connect the required gas lines
   Verify the required gas supplies are properly connected to your GC.

4. Install the test column.
   The column currently installed should be carefully removed and replaced with the required test column.

5. Perform Column Evaluation and Leak Test.

6. Connect your data handling.
   Verify that your data handling is properly connected to your GC system.

OPERATING PROCEDURE

PID-PTV Checkout in PTV Splitless Mode

Before beginning, press **CONFIG** to verify the GC configuration:

Right inlet	PTV
Right carrier	He (helium)
Right detector	PID

1. Use **RIGHT CARRIER** to display the appropriate Carrier Gas Control Table. Verify to operate in constant pressure mode. If not, scroll to `Flow mode`, press **MODE/TYPE** to access the selection menu, then select `con pres`. Scrool to `Pressure` and set the pressure value to have the required carrier gas flow rate `Col.flow`.

```
          RIGHT CARRIER[1]

Pressure          30.0    30.0
Col.flow          3.00
Lin. veloc.              (60.9)<
```

2. Use **OVEN** to display the Column Oven Control Table. Set the oven temperature and the Oven Program required.

```
               OVEN

Temp              70.0   70.0
Initial Time             1.00
Ramp 1                   20.0
Final temp                230
Final time 1             1.00<
Ramp 2                    Off
```

3. Use **RIGHT INLET** to display the appropriate Programmable Temperature Vaporizing Injector Control Table. Set the required temperature setpoint

Temp. Verify to operate in **PTV splitless** mode. If not, scroll to Mode, press **MODE/TYPE** to access the selection menu, then select PTV splitless. Scroll to Splitless time to set the required setpoint.

```
      RIGHT INLET (PTV)
Temp                    70    70
Pressure              30.0  30.0
Mode:             PTV Splitless
Total flow              (53.0)
Split Flow            50.0  50.0
Splitless time        0.80  0.80
Constant sept purge?         Y
Inject phase menu:           Y<
```

4. Scroll to Inject phase menu. Press **MODE/TYPE** to enter the **PTV Phase Menu**.

```
       PTV PHASE MENU
Ramped pressure?             N
Inject temp                 50
Inject time                0.1
Transfer ramp               10
Transfer temp              260
Transfer time            1.00<
```

5. Select Ramped pressure? **NO**. Set the required Inject temp and *Inject time* setpoints as required. Then, set the Transfer ramp, the Transfer temp and the Transfer time required setpoints.

6. Use **RIGHT DETECTOR** to display the appropriate PID Detector Control Table. Set the required temperature Base Temp and the detector gases Mkup and Sheath Gas required setpoints.

RIGHT DETECTOR (PID)		
Lamp		On
Base temp	230	230
Lamp current		low
Signal pA		(15.4)
Mkup N2	7.0	7.0
Sheath Gas	40	40

7. Verify that `High current` is set to **NO**.

8. Scroll to `Lamp` and press **ON**. This start the ignition sequence. The baseline level `Signal pA`, will suddenly increase meaning that the lamp is lit inside the detector. A short period of conditioning is required in order to obtain a stable baseline.

9. Use **RIGHT SIGNAL** to display the appropriate PID Detector Signal Control Table. Scroll to *Range* and set the electrometer amplifier input range required.

RIGHT SIGNAL (PID)	
Output	(1000)
Offset	100
Autozero?	Y/N
Range 10^(0..3)	0<
Analog filter	Off
Analog filter	Off

10. Activate your Data System and set the parameters required for the checkout.

11. In the PID Detector Signal Control Table scroll to `Auto zero?` and turn it **YES**.

12. Perform a blank analysis injecting pure iso-octane and press **START** on the GC to begin the checkout run.

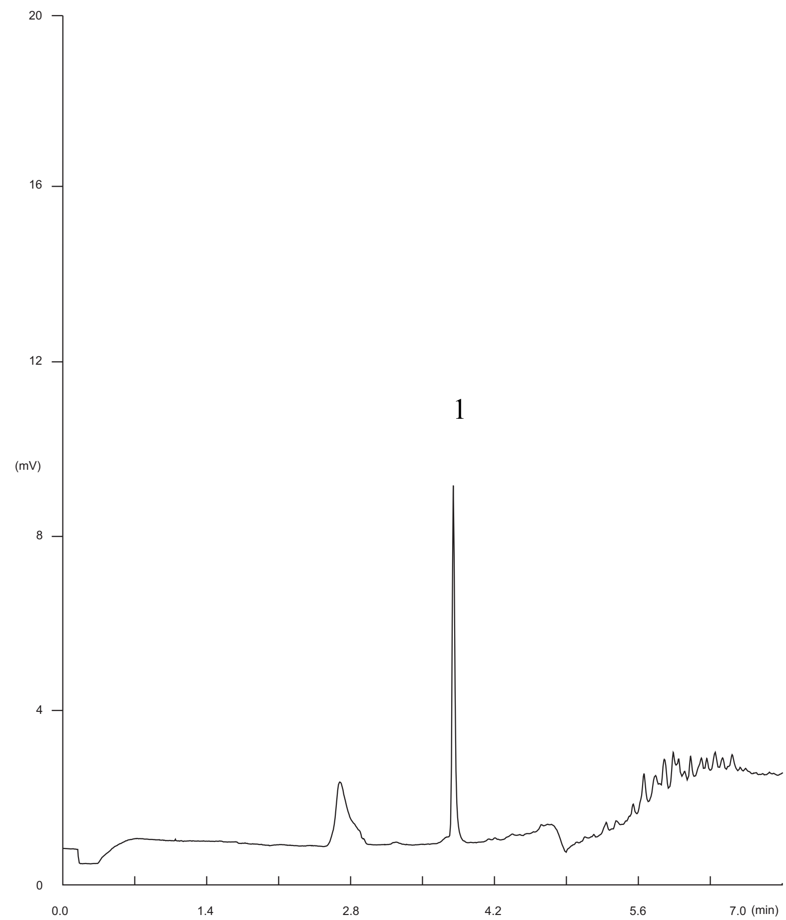
Baseline Acquisition and Analysis

NOTE Refer to the Acceptance Values reported in the Table 24-3 according to the data handling in use.

13. With the GC in Stand-by/Prep Run condition, activate the data system for 10 minutes to evaluate your baseline in isothermal condition.

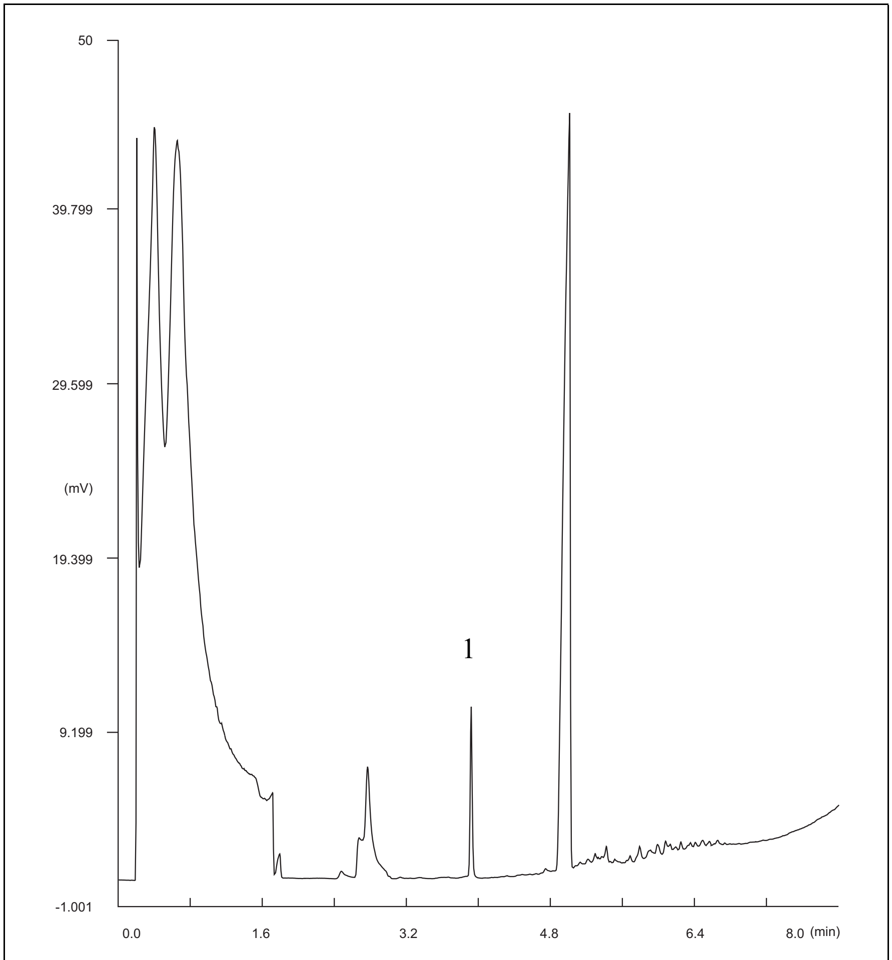
14. After the baseline evaluation has been completed, set-up the data system to acquire a single run.

15. Inject the test mixture and press **START** on the GC to begin the checkout run. The resulting chromatograms should look like the one shown in *Figure 24.1, 24.2*, *24.3 or 24.4.*



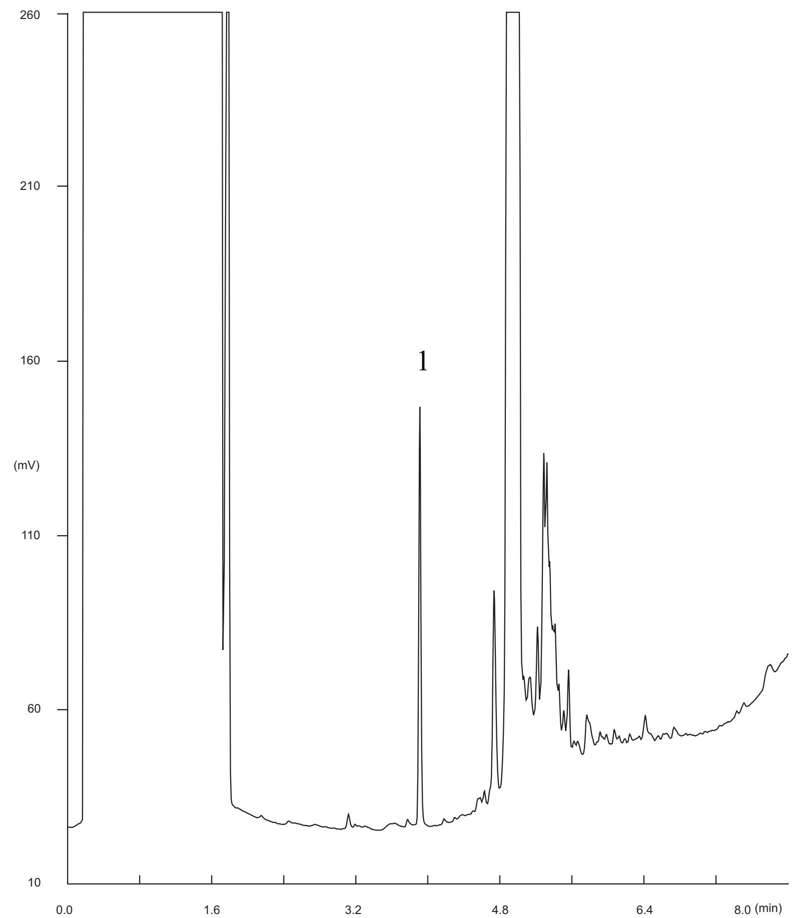
1. Azobenzene

Figure 24-1. PID-PTV Injection with 8.4 eV UV Lamp



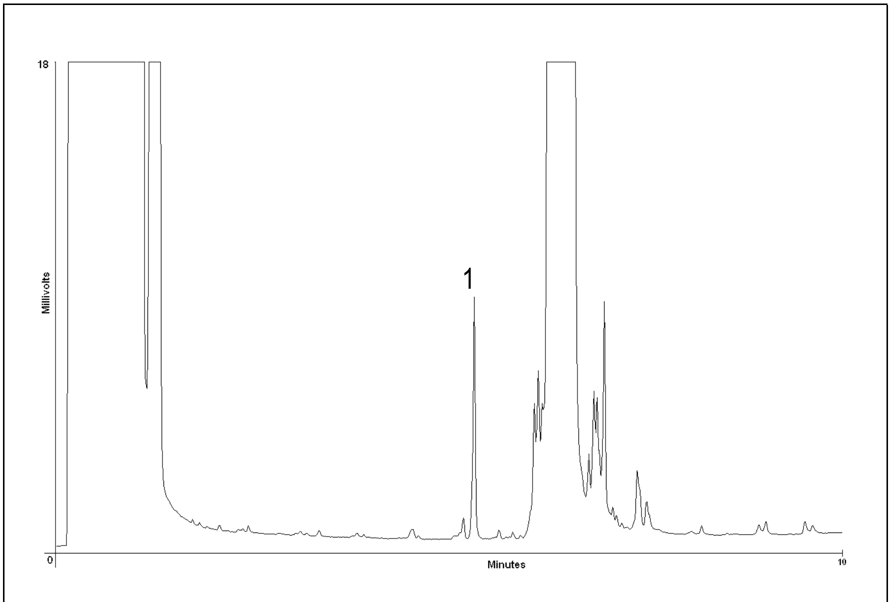
1. Azobenzene

Figure 24-2. PID-PTV Injection with 9.6 eV UV Lamp



1. Azobenzene

Figure 24-3. PID-PTV Injection with 10.6 eV UV Lamp



1. Azobenzene

Figure 24-4. PID-PTV Injection with 11.8 eV UV Lamp

16. The following criteria indicate successful completion of PID-PTV checkout.

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

Table 24-3. PID-PTV Acceptance Criteria

Acceptance Values

CHROM-CARD								
Baseline Parameters	**Analog** *(1V Full Scale)*				**Digital** *(10V Full Scale)*			
Lamp (eV)	**8.4**	**9.6**	**10.6**	**11.8**	**8.4**	**9.6**	**10.6**	**11.8**
Noise (µV)	< 100	< 50	< 150	< 150	< 1 000	< 500	< 1 500	< 1 500
Wander (µV)	< 150	< 100	< 100	< 100	< 1 500	< 1 000	< 1 000	< 1 000
Drift (µV/h)	< 100	< 100	< 100	< 100	< 1 000	< 1 000	< 1 000	< 1 000
Analytical Results	**Analog** *(1V Full Scale) Area Counts (0.1 µVs)*				**Digital** *(10V Full Scale) Area Counts (0.1 µVs)*			
Lamp (eV)	**8.4**	**9.6**	**10.6**	**11.8**	**8.4**	**9.6**	**10.6**	**11.8**
Azobenzene	> 60*	> 110*	> 1 300*	> 24*	> 600*	> 1 100*	> 13 000*	> 240*

Computing-integrator (e.g. ChromJet)

Acceptance Values

CHROMQUEST				
Baseline Parameters *(1V Full Scale)*				
Lamp (eV)	**8.4**	**9.6**	**10.6**	**11.8**
Noise (µV)	< 100	< 50	< 150	< 150
Wander (µV)	< 150	< 100	< 100	< 100
Drift (µV/h)	< 100	< 100	< 100	< 100
Analytical Results *(1V Full Scale) - Area Counts (0.01 µVs)*				
Lamp (eV)	**8.4**	**9.6**	**10.6**	**11.8**
Azobenzene	> 600 000	> 1 100 000	> 13 000 000	> 240 000

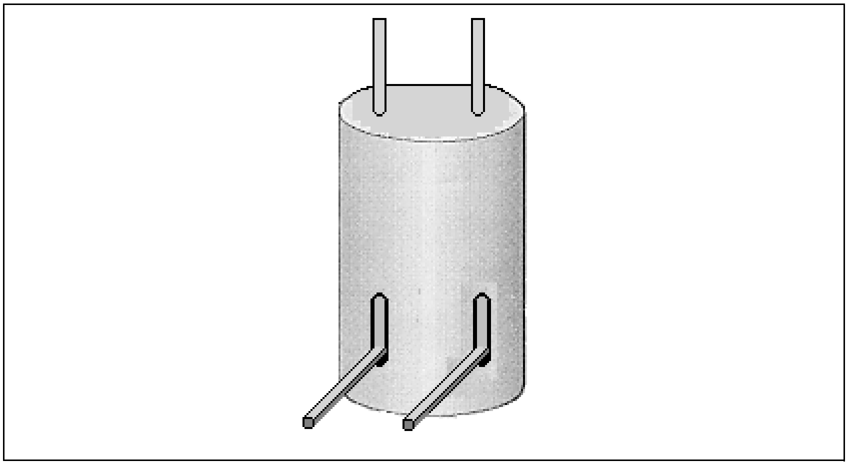
Acceptance Values

ATLAS				
Baseline Parameters *(10V Full Scale)*				
Lamp (eV)	**8.4**	**9.6**	**10.6**	**11.8**
Noise (µV)	< 1 000	< 500	< 1 500	< 1 500
Wander (µV)	< 1 500	< 1 000	< 1 000	< 1 000
Drift (µV/h)	< 1 000	< 1 000	< 1 000	< 1 000
Analytical Results *(10V Full Scale) - Area Counts (µVs)*				
Lamp (eV)	**8.4**	**9.6**	**10.6**	**11.8**
Azobenzene	> 60 000	> 110 000	> 1 300 000	> 24 000

XCALIBUR				
Baseline Parameters *(Acquisition Frequency = 10 Hz)*				
Lamp (eV)	**8.4**	**9.6**	**10.6**	**11.8**
Noise (Counts)	< 10 000	< 5 000	< 15 000	< 15 000
Wander Counts)	< 15 000	< 10 000	< 10 000	< 10 000
Drift (Counts/h)	< 10 000	< 10 000	< 10 000	< 10 000
Analytical Results *Area Counts (Cts*s)*				
Lamp (eV)	**8.4**	**9.6**	**10.6**	**11.8**
Azobenzene	> 600 000	> 1 100 000	> 13 000 000	> 240 000

SECTION VII

SOPs Using TCD



The *SOPs Using TCD* section, contains the procedures to test the TRACE GC Ultra with the Thermal Conductivity Detector (TCD) using different injectors.

25

Checkout Using TCD with S/SL Injector

Chapter at a Glance...

Operating Procedures

SOP Number: P0326/11/E - 01 September 2009

Scope

Use the following procedure to verify proper TCD operation with the Split/ Splitless Injector.

Parts Referenced

Table 25-1. TCD-S/SL 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
Retaining Nut	M4 capillary column retaining nut	350 324 23
Graphite Ferrule	Graphite ferrule for 0.32 mm ID Column	290 134 87
Septum	Standard septum for S/SL injector	313 032 11
Syringe	10 µl size; 70 mm needle length	365 001 03
Test Mixture	Three components in n-Hexane: *Component* *Concentration* Dodecane 200 µg/ml Tetradecane 200 µg/ml Hexadecane 200 µg/ml	338 190 16
Gases	Chromatographic-grade purity	
Data Acquisition	Chrom-Card, ChromQuest, Atlas, Xcalibur, Computing-integrator	

Analytical Conditions Required for Splitless Injection

Table 25-2. TCD-S/SL Analytical Conditions

Gases	Carrier Gas: Helium = 30 kPa Constant Pressure Make-up: Helium = 27.5 ml/min Reference Gas: Helium = 30 ml/min
Oven Program	Initial Temperature = 50 °C Initial Time = 1 minute Ramp 1 = 20 °C/minute Final Temperature = 190 °C Final Time = 1 minute
Injector	Operating Mode = Splitless Temperature = 200 °C Splitless Time = 0.8 minutes Split Flow = 60 ml/min Constant Septum Purge = Yes
Detector	Block Temperature = 200 °C Transfer Temperature = 190 °C Constant Filament Temperature = No Filament Voltage = 10V Filament Temperature limit = 350 °C (*) Gain = x 10 Negative Polarity = No
Injected Volume	1 µl + needle of Test Mixture
Analog Signal Output	Chrom-Card Acquisition Frequency = Medium
Digital Signal Output	Chrom-Card, ChromQuest, Atlas, Xcalibur Acquisition Frequency = 10 Hz

(*) In case of TCD with the polyimide coated filaments, set the filament temperature limit to 320 °C.

Recommended Initial Operations

Before starting the checkout, the following operations should be carried out:

1. Replace the glass liner.
   The glass liner currently installed in your injector should be carefully removed and replaced with the 3 mm ID glass liner for splitless application, as required for the checkout, with the appropriate graphite seal.

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

3. Connect the required gas lines
   Verify the required gas supplies are properly connected to your GC.

4. Install the test column.
   The column currently installed should be carefully removed and replaced with the required test column.

5. Perform Column Evaluation and Leak Test

6. Connect your data handling.
   Verify that your data handling is properly connected to your GC system.

OPERATING PROCEDURE

TCD-S/SL Checkout in Splitless Mode

Before beginning, press **CONFIG** to verify the GC configuration:

Left inlet or Right inlet	S/SL
Left carrier or Right carrier	He (helium)
Left detector or Right detector	TCD

1. Use **LEFT CARRIER** or **RIGHT CARRIER** to display the appropriate Carrier Gas Control Table. Verify to operate in constant pressure mode. If not, scroll to `Flow mode`, press **MODE/TYPE** to access the selection menu, then select `con pres`. Scrool to `Pressure` and set the pressure value to have the required carrier gas flow rate `Col.flow`.

```
        LEFT CARRIER[1]

Pressure         30.0   30.0
Col.flow         3.00
Lin. veloc.            (60.9)<
```

1. These settings could also be for a right carrier.

2. Use **OVEN** to display the Column Oven Control Table. Set the oven temperature and the Oven Program required.

```
             OVEN

Temp             50.0   50.0
Initial Time            1.00
Ramp 1                  20.0
Final temp               190
Final time 1            1.00<
Ramp 2                   Off
```

3. Use **LEFT INLET** or **RIGHT INLET** to display the appropriate Split/Splitless Injector Control Table and set the required temperature setpoint *Temp*. Verify

to operate in **Splitless** mode. If not, scroll to `Mode`, press **MODE/TYPE** to access the selection menu, then select `Splitless`. Scroll to `Splitless time` to set the required setpoint.

LEFT INLET (S/SL)[1]		
Temp	200	200
Pressure	30.0	30.0
Mode:		Splitless
Total flow		(63.0)
Split Flow	60.0	60.0
Splitless time	0.80	0.80
Constant sept purge?		Y<

1. These settings could also be for a right inlet.

4. Use **LEFT DETECTOR** or **RIGHT DETECTOR** to display the appropriate TCD Detector Control Table. Sequentially scroll to `Block Temp`, `Transf Temp`, `Ref flow` and `mkup flow` and set the required values.

LEFT DETECTOR (TCD)[1]		
Filament power		Off
Fil status		(not rdy)
Block temp	200	200
Transf temp	190	190
Const fil temp		Y/N
Fil volts (CV)		10
Fil temp limit[2]		350
Ref flow	30.0	30.0
Mkup flow	27.5	27.5<

1. These settings could also be for a right detector.
2. In case of TCD with the polyimide coated filaments, the temperature limit is 320 °C

5. Scroll to `Const fil temp` and select it **NO**.

6. Scroll to `Fil volts (CV)` and set the filament voltage. Scroll to `Fil temp limit` and set the required limit temperature setpoint.

7. Scroll to `Filament power` and turn it **ON**. After a few seconds the `ready` is displayed on `Fil status` line.

8. Use **LEFT SIGNAL** or **RIGHT SIGNAL** to display the appropriate TCD Detector Signal Control Table. Scroll to `Gain` and set the desired value.

LEFT SIGNAL (TCD)[1]	
Output	(1000)
Offset	100
Auto zero?	Y/N
Gain (x1..x10)	10<
Neg polarity?	N
Baseline comp	Off

1. These settings could also be for a right signal.

9. Activate your Data System and set the parameters required for the checkout.

10. In the TCD Detector Signal Control Table scroll to `Auto zero?` and turn it **YES**.

11. Perform a blank analysis injecting pure iso-octane and press **START** on the GC to begin the checkout run.

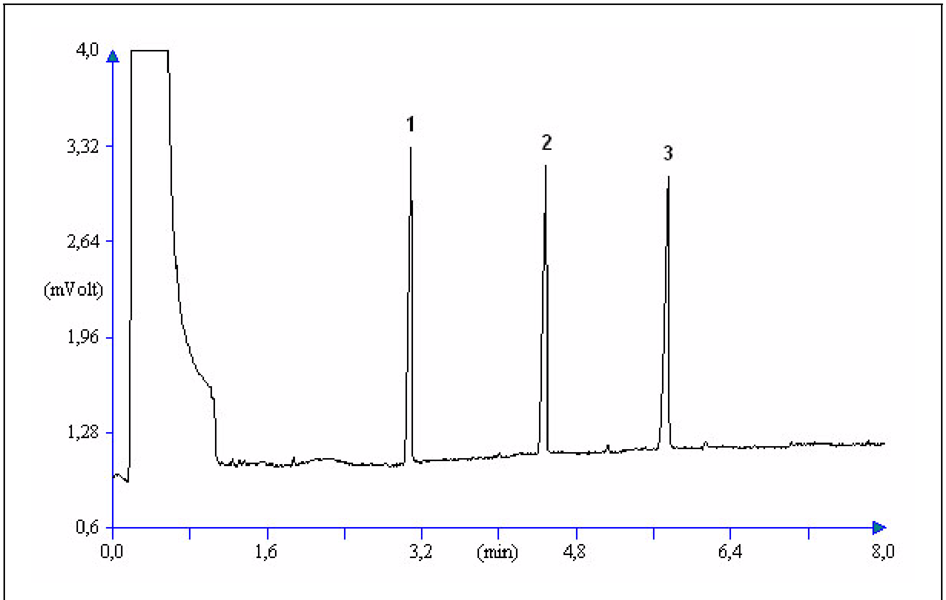
Baseline Acquisition and Analysis

NOTE Refer to the Acceptance Values reported in the Table 25-3 according to the data handling in use.

12. With the GC in Stand-by/Prep Run condition, activate the data system for 10 minutes to evaluate your baseline in isothermal condition.

13. After the baseline the baseline evaluation has been completed, set-up the data system to acquire a single run.

14. Inject the test mixture and press **START** on the GC to begin the checkout run. The resulting chromatogram should look like the one shown in *Figure 25.1*.



1. Dodecane 2. Tetradecane 3. Hexadecane

Figure 25-1. TCD-Splitless Injection

15. The following criteria indicate successful completion of TCD-S/SL checkout.

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

Table 25-3. TCD-S/SL Acceptance Criteria

	CHROM-CARD		
	Baseline Parameters	**Analog** *(1V Full Scale)*	**Digital** *(10V Full Scale)*
Acceptance Values	Noise (µV)	< 30	< 300
	Wander (µV)	< 140	< 1 400
	Drift (µV/h)	< 200	< 2 000
	Analytical Results	**Analog** *(1V Full Scale)* *Area Counts (0.1 µVs)*	**Digital** *(10V Full Scale)* *Area Counts (0.1 µVs)*
	Components	> 33 000 for each component	> 330 000 for each component

Computing-integrator (e.g. ChromJet)

	CHROMQUEST	
	Baseline Parameters *(1V Full Scale)*	
Acceptance Values	Noise (µV)	< 30
	Wander (µV)	< 140
	Drift (µV/h)	< 200
	Analytical Results *(1V Full Scale) - Area Counts (0.01 µVs)*	
	Components	> 330 000 for each component

	ATLAS	
	Baseline Parameters *(10V Full Scale)*	
	Noise (μV)	< 300
	Wander (μV)	< 1 400
	Drift (μV/h)	< 2 000
	Analytical Results *(10V Full Scale) - Area Counts (μVs)*	
	Components	> 33 000 for each component
⚠		

Acceptance Values	XCALIBUR	
	Baseline Parameters *(Acquisition Frequency = 10 Hz)*	
	Noise (Counts)	< 3 000
	Wander Counts)	< 14 000
	Drift (Counts/h)	< 20 000
	Analytical Results *Area Counts (Cts*s)*	
	Components	> 330 000 for each component
⚠		

Analytical Acceptance Comments	
1	In case of TCD equipped with the polyimide coated filaments, the acceptance values of the Components Area will result to be 4 times lower than the values reported in Table 25-3.

26

Checkout Using TCD with PKD Injector

Chapter at a Glance...

Operating Procedures

SOP Number: P0328/11/E - 01 September 2009

Scope

Use the following procedure to verify proper TCD operation with Packed Injector.

Parts Referenced

Table 26-1. TCD-PKD 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	2 mm ID tapered	453 220 50
Liner Seal	Graphite seal for glass liner	290 334 05
Pre-column	Fused Silica Capillary Column 0.5 mt long 0.53 mm ID	260 603 75
Retaining Nut	M4 capillary column retaining nut	350 324 23
Press-fit connections	For columns 0.53/0.32 mm ID	350 438 16
Injection side adapter	For Wide bore column	347 003 03
Retaining Nut	For Injection side adapter	350 024 04
Detector side adapter	For Wide bore column	347 103 04
Ferrule	6 mm ID double brass ferrule	290 341 37
Nut	1/4” G-6 mm ID nut	350 201 18
Graphite Ferrule	Graphite ferrule for 0.53 mm ID Column Graphite ferrule for 0.32 mm ID Column	290 134 86 290 134 87
Septum	Standard septum for Packed Injector	313 032 26
Syringe	10 µl size; 50 mm needle length	365 005 25

Table 26-1. TCD-PKD Parts Referenced (Continued)

Part	Description	Part Number
Test Mixture	Three components in n-Hexane: *Component* *Concentration* Dodecane 200 µg/ml Tetradecane 200 µg/ml Hexadecane 200 µg/ml	338 190 16
Gases	Chromatographic-grade purity	
Data Acquisition	Chrom-Card, ChromQuest, Atlas, Xcalibur, Computing-integrator	

Analytical Conditions Required for Packed Injector

Table 26-2. TCD-PKD Analytical Conditions

Parameters Setting	
Gases	Carrier Gas: Helium = 30 kPa Constant Pressure Make-up: Helium = 27.5 ml/min Reference Gas: Helium = 30 ml/min
Oven Program	Iso Temperature = 50 °C Initial Time = 1 minute Ramp 1 = 20 °C/minute Final Temperature = 190 °C Final Time = 1 minute
Injector	Operating Mode = Packed Temperature = 180 °C
Detector	Block Temperature = 200 °C Transfer Temperature = 190 °C Constant Filament Temperature = No Filament Voltage = 10V Filament Temperature limit = 350 °C (*) Gain = x 10 Negative Polarity = No
Injected Volume	1 µl + needle of Test Mixture
Analog Signal Output	Chrom-Card Acquisition Frequency = Medium
Digital Signal Output	Chrom-Card, ChromQuest, Atlas, Xcalibur Acquisition Frequency = 10 Hz

(*) In case of TCD with the polyimide coated filaments, set the filament temperature limit to 320 °C.

Recommended Initial Operations

Before starting the checkout, the following operations should be carried out:

1. Insert the glass liner.
   Remove the column and insert the 2 mm ID tapered glass liner, as required for the checkout, from the bottom of the injector with the appropriate liner seal. Fix the liner by using the appropriate adapter for capillary column.
   Refer to the TRACE GC Ultra Maintenance and Troubleshooting Manual.

2. Mount the adapter for capillary column on the detector base body.

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

4. Connect the required gas lines
   Verify the required gas supplies are properly connected to your GC.

5. Install the precolumn.
   Connect the precolumn to the injector.

6. Install the test column
   Connect the test column to the precolumn by using the press fit connections provided.

7. Perform the manual leak check following the Operating Procedure “*Perforing a Leak Check* ” in Chapter 14 of the TRACE GC Ultra Operating Manual.

8. Connect the other end of the test column to the detector base body.

9. Connect your data handling.
   Verify that your data handling is properly connected to your GC system

OPERATING PROCEDURE

TCD-PKD Checkout

Before beginning, press **CONFIG** to verify the GC configuration:

Left inlet or Right inlet	PKD
Left carrier or Right carrier	He (helium)
Left detector or Right detector	TCD

1. Use **LEFT CARRIER** or **RIGHT CARRIER** to display the appropriate Carrier Gas Control Table. Verify to operate in constant pressure mode. If not, scroll to `Flow mode`, press **MODE/TYPE** to access the selection menu, then select `con pres`. Scrool to `Pressure` and set the pressure value to have the required carrier gas flow rate `Col.flow`.

```
        LEFT CARRIER[1]

Pressure         30.0   30.0
Col.flow         3.00
Lin. veloc.           (60.9)<
```

1. These settings could also be for a right carrier.

2. Use **OVEN** to display the Column Oven Control Table. Set the oven temperature and the Oven Program required.

```
            OVEN

Temp             50.0   50.0
Initial Time            1.00
Ramp 1                  20.0
Final temp               190
Final time 1            1.00<
Ramp 2                   Off
```

3. Use **LEFT INLET** or **RIGHT INLET** to display the appropriate PKD Injector Control Table. Set the required temperature setpoint `Temp`.Verify to operate in

`Packed` mode. If not, scroll to `Mode`, press **MODE/TYPE** to access the selection menu, then select `Packed`.

LEFT INLET (PKD)[1]		
Temp	180	180
Pressure	30.0	30.0
Mode:		Packed

1. These settings could also be for a right inlet.

4. Use **LEFT DETECTOR** or **RIGHT DETECTOR** to display the appropriate TCD Detector Control Table. Sequentially scroll to `Block Temp`, `Transf Temp`, `Ref flow` and `mkup flow` and set the required values.

LEFT DETECTOR (TCD)[1]		
Filament power		Off
Fil status		(not rdy)
Block temp	200	200
Transf temp	190	190
Const fil temp		Y/N
Fil volts (CV)		10
Fil temp limit[2]		350
Ref flow	30.0	30.0
Mkup flow	27.5	27.5<

1. These settings could also be for a right detector.
2. In case of TCD with the polyimide coated filaments, the temperature limit is 320 °C

5. Scroll to `Const fil temp` and select it **NO**.

6. Scroll to `Fil volts (CV)` and set the filament voltage. Scroll to `Fil temp limit` and set the required limit temperature setpoint.

7. Scroll to `Filament power` and turn it **ON**. After a few seconds the `ready` is displayed on `Fil status` line.

8. Use **LEFT SIGNAL** or **RIGHT SIGNAL** to display the appropriate TCD Detector Signal Control Table. Scroll to `Gain` and set the desired value.

```
      LEFT SIGNAL (TCD)[1]
Output                  (1000)
Offset                    100
Auto zero?                Y/N
Gain (x1..x10)            10<
Neg polarity?               N
Baseline comp             Off
```

1. These settings could also be for a right signal.

9. Activate your Data System and set the parameters required for the checkout.

10. In the TCD Detector Signal Control Table scroll to `Auto zero?` and turn it **YES**.

11. Perform a blank analysis injecting pure iso-octane and press **START** on the GC to begin the checkout run.

Baseline Acquisition and Analysis

NOTE Refer to the Acceptance Values reported in the Table 26-3 according to the data handling in use.

12. With the GC in Stand-by/Prep Run condition, activate the data system for 10 minutes to evaluate your baseline in isothermal condition.

13. After the baseline evaluation has been completed, set-up the data system to acquire a single run.

14. Inject the test mixture and press **START** on the GC to begin the checkout run. The resulting chromatogram should look like the one shown in *Figure 26.1*

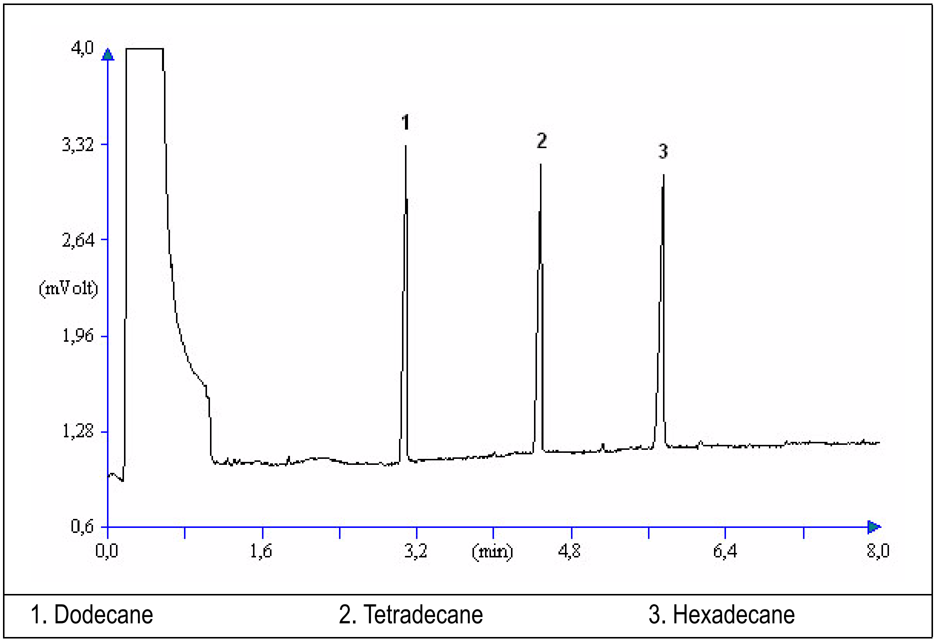


Figure 26-1. TCD-Packed Injection

15. The following criteria indicate successful completion of TCD-PKD checkout.

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

Table 26-3. TCD-PKD Acceptance Criteria

Acceptance Values	CHROM-CARD		
	Baseline Parameters	**Analog** *(1V Full Scale)*	**Digital** *(10V Full Scale)*
	Noise (µV)	< 30	< 300
	Wander (µV)	< 140	< 1 400
	Drift (µV/h)	< 200	< 2 000
	Analytical Results	**Analog** *(1V Full Scale)* *Area Counts (0.1 µVs)*	**Digital** *(10V Full Scale)* *Area Counts (0.1 µVs)*
	Components	> 30 000 for each component	> 300 000 for each component
⚠			

Computing-integrator (e.g. ChromJet)	
⚠	

Acceptance Values	CHROMQUEST	
	Baseline Parameters *(1V Full Scale)*	
	Noise (µV)	< 30
	Wander (µV)	< 140
	Drift (µV/h)	< 200
	Analytical Results *(1V Full Scale) - Area Counts (0.01 µVs)*	
	Components	> 300 000 for each component
⚠		

Acceptance Values	ATLAS	
	Baseline Parameters *(10V Full Scale)*	
	Noise (µV)	< 300
	Wander (µV)	< 1 400
	Drift (µV/h)	< 2 000
	Analytical Results *(10V Full Scale) - Area Counts (µVs)*	
	Components	> 30 000 for each component
⚠		

Acceptance Values	XCALIBUR	
	Baseline Parameters *(Acquisition Frequency = 10 Hz)*	
	Noise (Counts)	< 3 000
	Wander Counts)	< 14 000
	Drift (Counts/h)	< 20 000
	Analytical Results *Area Counts (Cts*s)*	
	Components	> 300 000 for each component
⚠		

Analytical Acceptance Comments	
1	In case of TCD equipped with the polyimide coated filaments, the acceptance values of the Components Area will result to be 4 times lower than the values reported in Table 26-3.

27

Checkout Using TCD with PPKD Injector

Chapter at a Glance…

Operating Procedures

SOP Number: P0329/11/E - 01 September 2009

Scope

Use the following procedure to verify proper TCD operation with Purged Packed Injector.

Parts Referenced

Table 27-1. TCD-PPKD 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	2 mm ID tapered	453 220 50
Liner Seal	Graphite seal for glass liner	290 334 05
Pre-column	Fused Silica Capillary Column 0.5 mt long 0.53 mm ID	260 603 75
Retaining Nut	M4 capillary column retaining nut	350 324 23
Press-fit connections	For columns 0.53/0.32 mm ID	350 438 16
Injection side adapter	For Wide bore column	347 003 03
Retaining Nut	For Injection side adapter	350 024 04
Detector side adapter	For Wide bore column	347 103 04
Ferrule	6 mm ID double brass ferrule	290 341 37
Nut	1/4” G-6 mm ID nut	350 201 18
Graphite Ferrule	Graphite ferrule for 0.53 mm ID Column Graphite ferrule for 0.32 mm ID Column	290 134 86 290 134 87
Septum	Standard septum for Purged Packed Injector	313 032 26
Syringe	10 µl size; 50 mm needle length	365 005 25

Table 27-1. TCD-PPKD Parts Referenced (Continued)

Part	Description	Part Number
Test Mixture	Three components in n-Hexane: *Component* *Concentration* Dodecane 200 µg/ml Tetradecane 200 µg/ml Hexadecane 200 µg/ml	338 190 16
Gases	Chromatographic-grade purity	
Data Acquisition	Chrom-Card, ChromQuest, Atlas, Xcalibur, Computing-integrator	

Analytical Conditions Required for Purged Packed Injector

Table 27-2. TCD-PPKD Analytical Conditions

Parameters Setting	
Gases	Carrier Gas: Helium = 30 kPa Constant Pressure Make-up: Helium = 27.5 ml/min Reference Gas: Helium = 30 ml/min
Oven Program	Initial Temperature = 50 °C Initial Time = 1 minute Ramp 1 = 20 °C/minute Final Temperature =190 °C Final Time = 1 minute
Injector	Operating Mode = Wide bore Septum Purge = Yes Temperature = 180 °C
Detector	Block Temperature = 200 °C Transfer Temperature = 190 °C Constant Filament Temperature = No Filament Voltage = 10V Filament Temperature limit = 350 °C (*) Gain = x 10 Negative Polarity = No
Injected Volume	1 µl + needle of Test Mixture
Analog Signal Output	Chrom-Card Acquisition Frequency = Medium
Digital Signal Output	Chrom-Card, ChromQuest, Atlas, Xcalibur Acquisition Frequency = 10 Hz

(*) In case of TCD with the polyimide coated filaments, set the filament temperature limit to 320 °C.

Recommended Initial Operations

Before starting the checkout, the following operations should be carried out:

1. Insert the glass liner.
   Remove the column and insert the 2 mm ID tapered glass liner, as required for the checkout, from the bottom of the injector with the appropriate liner seal. Fix the liner by using the appropriate adapter for capillary column.
   Refer to the TRACE GC Ultra Maintenance and Troubleshooting Manual.

2. Mount the adapter for capillary column on the detector base body.

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

4. Connect the required gas lines
   Verify the required gas supplies are properly connected to your GC.

5. Install the precolumn.
   Connect the precolumn to the injector.

6. Install the test column
   Connect the test column to the precolumn by using the press fit connections provided.

7.

8. Connect the other end of the test column to the detector base body.

9. Perform Column Evaluation.

10. Connect your data handling.
    Verify that your data handling is properly connected to your GC system.

OPERATING PROCEDURE

TCD-PPKD Checkout

Before beginning, press **CONFIG** to verify the GC configuration:

Left inlet or Right inlet	PPKD
Left carrier or Right carrier	He (helium)
Left detector or Right detector	TCD

1. Use **LEFT CARRIER** or **RIGHT CARRIER** to display the appropriate Carrier Gas Control Table. Verify to operate in constant pressure mode. If not, scroll to `Flow mode`, press **MODE/TYPE** to access the selection menu, then select `con pres`. Scrool to `Pressure` and set the pressure value to have the required carrier gas flow rate `Col.flow`.

```
        LEFT CARRIER[1]

Pressure          30.0    30.0
Col.flow          3.00
Lin. veloc.            (60.9)<
```

1. These settings could also be for a right carrier.

2. Use **OVEN** to display the Column Oven Control Table. Set the oven temperature and the Oven Program required.

```
            OVEN

Temp              50.0   50.0
Initial Time             1.00
Ramp 1                   20.0
Final temp                200
Final time 1             1.00<
Ramp 2                    Off
```

3. Use **LEFT INLET** or **RIGHT INLET** to display the appropriate PPKD Injector Control Table. Set the required temperature setpoint `Temp`.Verify to operate in

`Wide bore` mode. If not, scroll to `Mode`, press **MODE/TYPE** to access the selection menu, then select `Wide bore`.

```
      LEFT INLET (PPKD)[1]

Temp                 180   180
Pressure            30.0  30.0
Mode:                Wide bore<
Constant sept purge?         Y<
```

1. These settings could also be for a right inlet.

4. Use **LEFT DETECTOR** or **RIGHT DETECTOR** to display the appropriate TCD Detector Control Table. Sequentially scroll to `Block Temp`, `Transf Temp`, `Ref flow` and `mkup flow` and set the required values.

```
     LEFT DETECTOR (TCD)[1]

Filament power              Off
Fil status            (not rdy)
Block temp            200   200
Transf temp           190   190
Const fil temp              Y/N
Fil volts (CV)               10
Fil temp limit[1]            350
Ref flow             30.0  30.0
Mkup flow            27.5  27.5<
```

1. These settings could also be

2. In case of TCD with the polyimide coated filaments, the temperature limit is 320 °C

5. Scroll to `Const fil temp` and select it **NO**.

6. Scroll to `Fil volts (CV)` and set the filament voltage. Scroll to `Fil temp limit` and set the required limit temperature setpoint.

7. Scroll to `Filament power` and turn it **ON**. After a few seconds the `ready` is displayed on `Fil status` line.

8. Use **LEFT SIGNAL** or **RIGHT SIGNAL** to display the appropriate TCD Detector Signal Control Table. Scroll to `Gain` and set the desired value.

LEFT SIGNAL (TCD)[1]	
Output	(1000)
Offset	100
Auto zero?	Y/N
Gain (x1..x10)	10<
Neg polarity?	N
Baseline comp	Off

1. These settings could also be for a right signal.

9. Activate your Data System and set the parameters required for the checkout.

10. In the TCD Detector Signal Control Table scroll to `Auto zero?` and turn it **YES**.

11. Perform a blank analysis injecting pure iso-octane and press **START** on the GC to begin the checkout run.

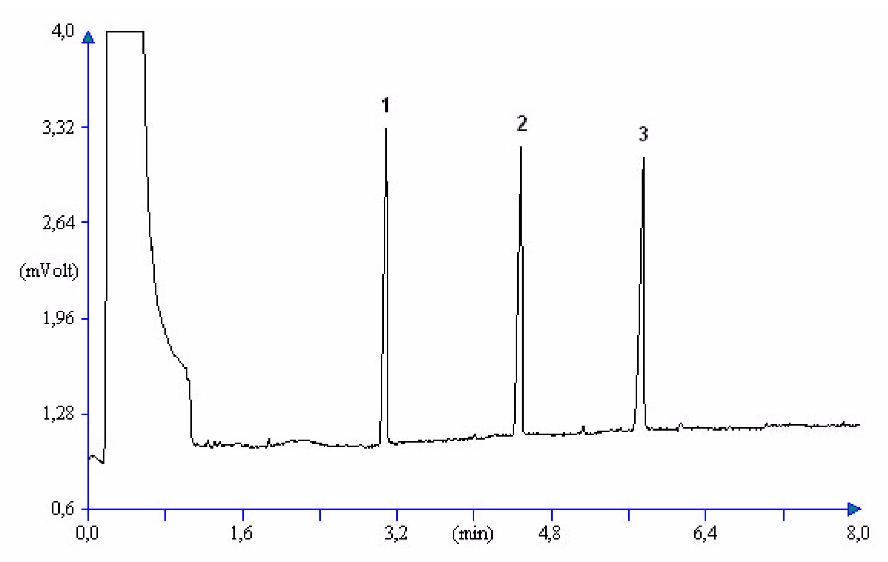
Baseline Acquisition and Analysis

NOTE Refer to the Acceptance Values reported in the Table 27-3 according to the data handling in use.

12. With the GC in Stand-by/Prep Run condition, activate the data system for 10 minutes to evaluate your baseline in isothermal condition.

13. After the baseline evaluation has been completed, set-up the data system to acquire a single run.

14. Inject the test mixture and press **START** on the GC to begin the checkout run. The resulting chromatogram should look like the one shown in *Figure 27.1*



1. Dodecane	2. Tetradecane	3. Hexadecane

Figure 27-1. TCD-PPKD Injection

15. The following criteria indicate successful completion of TCD-PPKD checkout.

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

Table 27-3. TCD-PPKD Acceptance Criteria

	CHROM-CARD		
Acceptance Values	**Baseline Parameters**	**Analog** *(1V Full Scale)*	**Digital** *(10V Full Scale)*
	Noise (μV)	< 30	< 300
	Wander (μV)	< 140	< 1 400
	Drift (μV/h)	< 200	< 2 000
	Analytical Results	**Analog** *(1V Full Scale)* *Area Counts (0.1 μVs)*	**Digital** *(10V Full Scale)* *Area Counts (0.1 μVs)*
	Components	> 30 000 for each component	> 300 000 for each component
⚠			

	Computing-integrator (e.g. ChromJet)
⚠	

	CHROMQUEST	
Acceptance Values	**Baseline Parameters** *(1V Full Scale)*	
	Noise (μV)	< 30
	Wander (μV)	< 140
	Drift (μV/h)	< 200
	Analytical Results *(1V Full Scale) - Area Counts (0.01 μVs)*	
	Components	> 300 000 for each component
⚠		

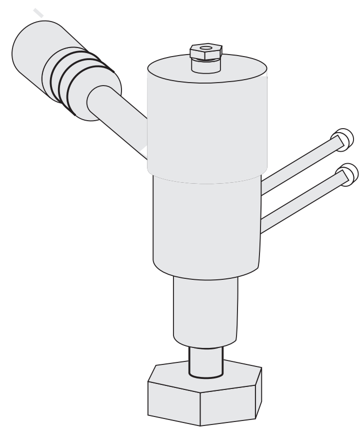
	ATLAS	
	Baseline Parameters *(10V Full Scale)*	
	Noise (µV)	< 300
	Wander (µV)	< 1 400
	Drift (µV/h)	< 2 000
	Analytical Results *(10V Full Scale) - Area Counts (µVs)*	
	Components	> 30 000 for each component
⚠		

Acceptance Values	**XCALIBUR**	
	Baseline Parameters *(Acquisition Frequency = 10 Hz)*	
	Noise (Counts)	< 3 000
	Wander Counts)	< 14 000
	Drift (Counts/h)	< 20 000
	Analytical Results *Area Counts (Cts*s)*	
	Components	> 300 000 for each component
⚠		

Analytical Acceptance Comments	
1	In case of TCD equipped with the polyimide coated filaments, the acceptance values of the Components Area will result to be 4 times lower than the values reported in Table 27-3.

SECTION VIII

SOPs Using PDD



The *SOPs Using PDD* section, contains the procedures to test the TRACE GC Ultra with the Pulsed Discharge Detector (PDD) using different injectors.

28

Checkout Using PDD with S/SL Injector

Chapter at a Glance…

Operating Procedures

SOP Number: P0381/05/E - 01 September 2009

Scope

Use the following procedure to verify proper PDD operation with the Split/Splitless Injector.

Parts Referenced

Table 28-1. PDD-S/SL 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; 70 mm needle length	365 001 03
Test Mixture	Three components in n-Hexane: *Component* *Concentration* Dodecane 1 µg/ml Tetradecane 1 µg/ml Hexadecane 1 µg/ml	338 190 32
Gases	Helium Chromatographic high grade purity (99.999%)	
Helium Purifier	Helium Purifier (VALCO)	432 100 76
Data Acquisition	Chrom-Card, ChromQuest, Atlas, Xcalibur, Computing-integrator	

Analytical Conditions Required for Splitless Injection

Table 28-2. PDD-S/SL Analytical Conditions

Parameters	Setting
Gases	Carrier Gas: Helium = 30 kPa Constant Pressure Discharge Gas: Helium = 30 ml/min (fixed value)
Oven Program	Initial Temperature = 60 °C Initial Time = 1 minute Ramp 1 = 20 °C/minute Final Temperature = 160 °C Final Time = 1 minute
Injector	Operating Mode = Splitless Temperature = 250 °C Splitless Time = 0.5 minutes Split Flow = 60 ml/min Constant Septum Purge = Yes
Detector	Base Temperature = 280 °C Detector Signal Range = 10^0
Injected Volume	1 µl + needle of Test Mixture
Analog Signal Output	Chrom-Card Acquisition Frequency = Medium
Digital Signal Output	Chrom-Card, ChromQuest, Atlas, Xcalibur Acquisition Frequency = 10 Hz

Recommended Initial Operations

Before starting the checkout, the following operations should be carried out:

1. Replace the glass liner.
   The glass liner currently installed in your injector should be carefully removed and replaced with the 3 mm ID glass liner for splitless 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. Connect the required gas lines
   Verify the required gas supplies are properly connected to your GC.

4. Verify that the helium purifier has been properly installed and purged.

5. Install the test column.
   The column currently installed should be carefully removed and replaced with the required test column.

6. Perform Column Evaluation and Leak Test.

7. Connect your data handling.
   Verify that your data handling is properly connected to your GC system.

CAUTION **When leak check has been successfully carried out, power the helium purifier On. Before starting checkout, wait about 2.5 hours to reach the complete activation of the helium purifier.**

OPERATING PROCEDURE

PDD-S/SL Checkout in Splitless Mode

Before beginning, press **CONFIG** to verify the GC configuration:

Left inlet or Right inlet	S/SL
Left carrier or Right carrier	He (helium)
Left detector or Right detector	PDD

1. Use **LEFT CARRIER** or **RIGHT CARRIER** to display the appropriate Carrier Gas Control Table. Verify to operate in constant pressure mode. If not, scroll to `Flow mode`, press **MODE/TYPE** to access the selection menu, then select `con pres`. Scrool to `Pressure` and set the pressure value to have the required carrier gas flow rate `Col.flow`.

```
        LEFT CARRIER[1]
Pressure         30.0    30.0
Col.flow         3.00
Lin. veloc.             (60.9)<
```

1. These settings could also be for a right carrier.

2. Use **OVEN** to display the Column Oven Control Table. Set the oven temperature and the Oven Program required.

```
            OVEN
Temp              60.0   60.0
Initial Time             1.00
Ramp 1                   20.0
Final temp                160
Final time 1             1.00<
Ramp 2                    Off
```

3. Use **LEFT INLET** or **RIGHT INLET** to display the appropriate Split/Splitless Injector Control Table. Set the required temperature *Temp* setpoint. Verify to

operate in **Splitless** mode. If not, scroll to `Mode`, press **MODE/TYPE** to access the selection menu, then select `Splitless`. Scroll to `Splitless time` to set the required setpoint.

LEFT INLET (S/SL)[1]		
Temp	250	250
Pressure	30.0	30.0
Mode:		Splitless
Total flow		(63.0)
Split Flow	60.0	60.0
Splitless time	0.50	0.50
Constant sept purge?		Y<

1. These settings could also be for a right inlet.

4. Use **LEFT DETECTOR** or **RIGHT DETECTOR** to display the appropriate PDD Detector Control Table. Set the required temperature `Base Temp` then turn the `Pulse generator` **ON**.

LEFT DETECTOR (PDD)[1]		
Pulse generator		On
Base temp	250	250
Signal pA		(1900.0)

1. These settings could also be for a right detector.

5. Use **LEFT SIGNAL** or **RIGHT SIGNAL** to display the appropriate PDD Detector Signal Control Table. Scroll to `Range` and set the electrometer amplifier input range required.

LEFT SIGNAL (PDD)[1]	
Output	(1000)
Offset	100
Auto zero?	Y/N
Range 10^(0..3)	0<
Analog filter	Off
Baseline Comp	Off

1. These settings could also be for a right signal.

6. Activate your Data System and set the parameters required for the checkout.

7. In the PDD Detector Signal Control Table, scroll to `Auto zero?` and turn it **YES**.

8. Perform a blank analysis injecting pure hexane and press **START** on the GC to begin the checkout run.

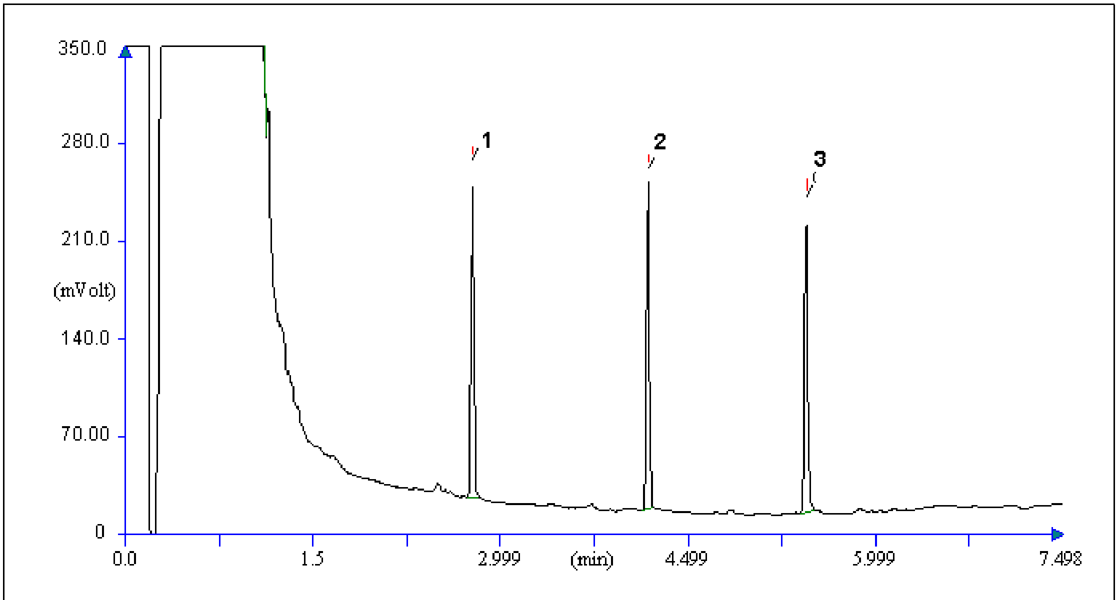
Baseline Acquisition and Analysis

NOTE Refer to the Acceptance Values reported in the Table 28-3 according to the data handling in use.

9. With the GC in Stand-by/Prep Run condition, activate the data system for 10 minutes to evaluate your baseline in isothermal condition.

10. After the baseline evaluation has been completed, set-up the data system to acquire a single run.

11. Inject the test mixture and press **START** on the GC to begin the checkout run. The resulting chromatogram should look like the one shown in *Figure 28.1*.



1. Dodecane 2. Tetradecane 3. Hexadecane

Figure 28-1. PDD-Splitless Injection

12. The following criteria indicate successful completion of PDD-S/SL checkout.

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

Table 28-3. PDD-S/SL Acceptance Criteria

Acceptance Values	CHROM-CARD		
	Baseline Parameters	**Analog** *(1V Full Scale)*	**Digital** *(10V Full Scale)*
	Noise (µV)	< 50	< 500
	Wander (µV)	< 500	< 5 000
	Drift (µV/h)	< 500	< 5 000
	Analytical Results	**Analog** *(1V Full Scale)* *Area Counts (0.1 µVs)*	**Digital** *(10V Full Scale)* *Area Counts (0.1 µVs)*
	Components	> 2 000 000 for each component	> 20 000 000 for each component
	Area Count Ratio Calculated as C12/C16	1 ± 0.1	1 ± 0.1
⚠			

Computing-integrator (e.g. ChromJet)	
⚠	

Acceptance Values	CHROMQUEST	
	Baseline Parameters *(1V Full Scale)*	
	Noise (µV)	< 50
	Wander (µV)	< 500
	Drift (µV/h)	< 500
	Analytical Results *(1V Full Scale) - Area Counts (0.01 µVs)*	
	Components	> 20 000 000 for each component
	Area Count Ratio Calculated as C12/C16	1 ± 0.1
⚠		

Acceptance Values

ATLAS	
Baseline Parameters *(10V Full Scale)*	
Noise (µV)	< 500
Wander (µV)	< 5 000
Drift (µV/h)	< 5 000
Analytical Results *(10V Full Scale) - Area Counts (µVs)*	
Components	> 2 000 000 for each component
Area Count Ratio Calculated as C12/C16	1 ± 0.1

XCALIBUR	
Baseline Parameters *(Acquisition Frequency = 10 Hz)*	
Noise (Counts)	< 5 000
Wander Counts)	< 50 000
Drift (Counts/h)	< 50 000
Analytical Results *Area Counts (Cts*s)*	
Components	> 20 000 000 for each component
Area Count Ratio Calculated as C12/C16	1 ± 0.1

29

Checkout Using PDD with OC Injector

Chapter at a Glance...

Operating Procedures

SOP Number: P0382/06/E - 01 September 2009

Scope

Use the following procedure to verify proper PDD operation with the Cold On-Column Injector.

Parts Referenced

Table 29-1. PDD-OCI 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
Retaining Nut	M4 capillary column retaining nut	350 324 23
Vespel Ferrule	Vespel Ferrule for 0.32 mm ID Column	290 134 60
Syringe	10 µl size; 75 mm needle length	365 020 07
Test Mixture	Three components in n-Hexane: *Component* *Concentration* Dodecane 1 µg/ml Tetradecane 1 µg/ml Hexadecane 1 µg/ml	338 190 32
Gases	Helium Chromatographic high grade purity (99.999%)	
Helium Purifier	Helium Purifier (VALCO)	432 100 76
Data Acquisition	Chrom-Card, ChromQuest, Atlas, Xcalibur, Computing-integrator	
Syringe	10 µl size; 80 mm needle length	365 020 19
Pre-column	2 m long; 0.53 mm ID	260 603 75
Press-fit set	Set of Press-fir connectors for TRACE OC	350 038 45

Analytical Conditions Required for On-Column Injection

Table 29-2. PDD-OCI Analytical Conditions

Parameters Setting	
Gases	Carrier Gas: Helium = 30 kPa Constant Pressure Discharge Gas: Helium = 30 ml/min (fixed value)
Oven Program	Initial Temperature = 70 °C Initial Time = 1 minute Ramp 1 = 20 °C/minute Final Temperature = 160 °C Final Time = 1 minute
Injector	Secondary Cooling = 0.2 minutes
Detector	Base Temperature = 280 °C Detector Signal Range = 10^0
Injected Volume	1 µl of Test Mixture
Analog Signal Output	Chrom-Card Acquisition Frequency = Medium
Digital Signal Output	Chrom-Card, ChromQuest, Atlas, Xcalibur Acquisition Frequency = 10 Hz

Recommended Initial Operations

Before starting the checkout, the following operations should be carried out:

1. Connect the required gas lines
   Verify the required gas supplies are properly connected to your GC.

2. Install the test column.
   The column currently installed should be carefully removed and replaced with the required test column.
   In case of automatic On-column for TriPlus sampler, install the pre-column and connect it to the test column by press-fit connector.

3. Install and connect the TriPlus sampler and its components.

4. Perform Column Evaluation and Leak Test.

5. Connect your data handling.
   Verify that your data handling is properly connected to your GC system.

6. Verify the opening/closing of the OC injector actuator by using the proper commands.

7. Verify the alignment of the syringe on the OC injector.

CAUTION **When leak check has been successfully carried out, power the helium purifier On. Before starting checkout, wait about 2.5 hours to reach the complete activation of the helium purifier.**

OPERATING PROCEDURE

PDD-OCI Checkout in On-Column Mode

Before beginning, press **CONFIG** to verify the GC configuration:

Left inlet or Right inlet	OCI
Left carrier or Right carrier	He (helium)
Left detector or Right detector	PDD

1. Use **LEFT CARRIER** or **RIGHT CARRIER** to display the appropriate Carrier Gas Control Table. Verify to operate in constant pressure mode. If not, scroll to `Flow mode`, press **MODE/TYPE** to access the selection menu, then select `con pres`. Scrool to `Pressure` and set the pressure value to have the required carrier gas flow rate `Col.flow`.

```
          LEFT CARRIER[1]

Pressure          30.0   30.0
Col.flow          3.00
Lin. veloc.           (60.9)<
```

1. These settings could also be for a right carrier

2. Use **OVEN** to display the Column Oven Control Table. Set the oven temperature *Temp* and the Oven Program required.

```
              OVEN

Temp              70.0   70.0
Initial Time             1.00
Ramp 1                   20.0
Final temp                160
Final time 1             1.00<
Ramp 2                    Off
```

3. Use **LEFT INLET** or **RIGHT INLET** to display the appropriate Cold On-Column Injector Control Table. Scroll to `Sec. cool time` and set the required secondary cooling time.

LEFT INLET (OCI)[1]		
Pressure	30.0	30.0
Sec. Cool Time		0.2<

1. These settings could also be for a right inlet.

4. Use **LEFT DETECTOR** or **RIGHT DETECTOR** to display the appropriate PDD Detector Control Table. Set the required temperature `Base Temp` then turn the `Pulse generator` **ON**.

LEFT DETECTOR (PDD)[1]		
Pulse generator		On
Base temp	250	250
Signal pA		(1900.0)

1. These settings could also be for a right detector.

5. Use **LEFT SIGNAL** or **RIGHT SIGNAL** to display the appropriate PDD Detector Signal Control Table. Scroll to `Range` and set the electrometer amplifier input range required.

LEFT SIGNAL (PDD)[1]	
Output	(1000)
Offset	100
Auto zero?	Y/N
Range 10^(0..3)	0<
Analog filter	Off
Baseline comp	Off

1. These settings could also be for a right signal.

6. Activate your Data System and set the parameters required for the checkout.

7. In the PDD Detector Signal Control Table scroll to `Auto zero?` and turn it **YES**.

8. Perform a blank analysis injecting pure hexane and press **START** on the GC to begin the checkout run.

Baseline Acquisition and Analysis

NOTE Refer to the Acceptance Values reported in the Table 29-3 according to the data handling in use.

9. With the GC in Stand-by/Prep Run condition, activate the data system for 10 minutes to evaluate your baseline in isothermal condition.

10. After the baseline evaluation has been completed, set-up the data system to acquire a single run.

11. Perform the analysis.

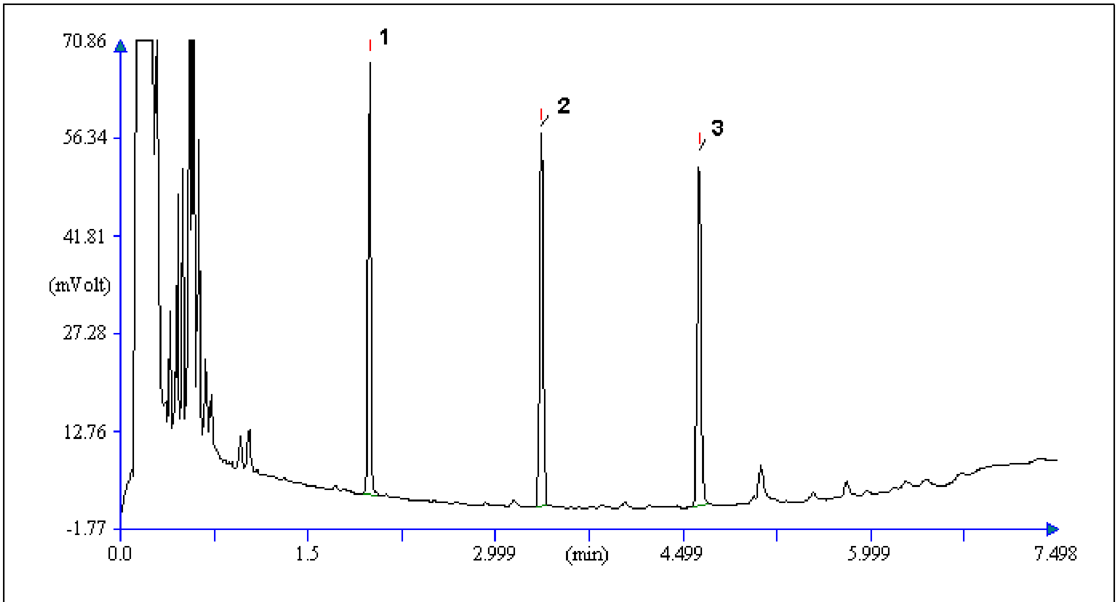
    Manual injection

    - Inject the test mixture and press **START** on the GC to begin the checkout run.

    *Automatic injection with TriPlus sample*r

    - Fill a vial with the standard mix and place that vial in the sample tray.
    - Load the method for OC and perform the sampling.

    The resulting chromatogram should look like the one shown in *Figure 29.1*.



1. Dodecane	2. Tetradecane	3. Hexadecane

Figure 29-1. PDD-On-Column Injection

12. The following criteria indicate successful completion of PDD-OCI checkout.

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

Table 29-3. PDD-OCI Acceptance Criteria

Acceptance Values	CHROM-CARD		
	Baseline Parameters	**Analog** *(1V Full Scale)*	**Digital** *(10V Full Scale)*
	Noise (μV)	< 50	< 500
	Wander (μV)	< 500	< 5 000
	Drift (μV/h)	< 500	< 5 000
	Analytical Results	**Analog** *(1V Full Scale)* *Area Counts (0.1 μVs)*	**Digital** *(10V Full Scale)* *Area Counts (0.1 μVs)*
	Components	> 1 000 000 for each component	> 10 000 000 for each component
	Area Count Ratio Calculated as C12/C16	1 ± 0.1	1 ± 0.1
⚠			

Computing-integrator (e.g. ChromJet)	
⚠	

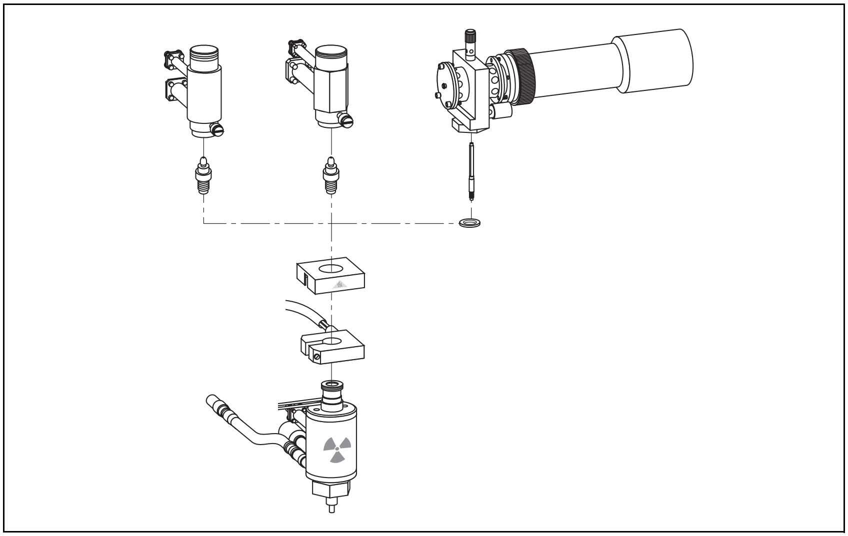
Acceptance Values	CHROMQUEST	
	Baseline Parameters *(1V Full Scale)*	
	Noise (μV)	< 50
	Wander (μV)	< 500
	Drift (μV/h)	< 500
	Analytical Results *(1V Full Scale) - Area Counts (0.01 μVs)*	
	Components	> 10 000 000 for each component
	Area Count Ratio Calculated as C12/C16	1 ± 0.1
⚠		

Acceptance Values	ATLAS	
	Baseline Parameters *(10V Full Scale)*	
	Noise (µV)	< 500
	Wander (µV)	< 5 000
	Drift (µV/h)	< 5 000
	Analytical Results *(10V Full Scale) - Area Counts (µVs)*	
	Components	> 1 000 000 for each component
	Area Count Ratio Calculated as C12/C16	1 ± 0.1
⚠		

Acceptance Values	XCALIBUR	
	Baseline Parameters *(Acquisition Frequency = 10 Hz)*	
	Noise (Counts)	< 5 000
	Wander Counts)	< 50 000
	Drift (Counts/h)	< 50 000
	Analytical Results *Area Counts (Cts*s)*	
	Components	> 10 000 000 for each component
	Area Count Ratio Calculated as C12/C16	1 ± 0.1
⚠		

SECTION IX

SOPs Using FID-NPD-FPD in Stacked Configuration



The *SOPs Using FID-NPD-FPD in Stacked Configuration* section, contains the procedures to test the TRACE GC Ultra with the Flame Ionization Detector (FID), Nitrogen-Phosphorus Detector (NPD) or Flame Photometric Detector (FPD) in series (stacked configuration) with the Electron Capture Detector ECD using different injectors.

30

Checkout Using Tandem FID

Chapter at a Glance…

Operating Procedures

SOP Number: P0383/05/E - 01 September 2009

Scope

Use the following procedure to verify proper Flame Ionization Detector (FID) installed in series (stacked configuration, see Figure 30-1) with the non-destructive Electron Capture Detector ECD using different injectors.

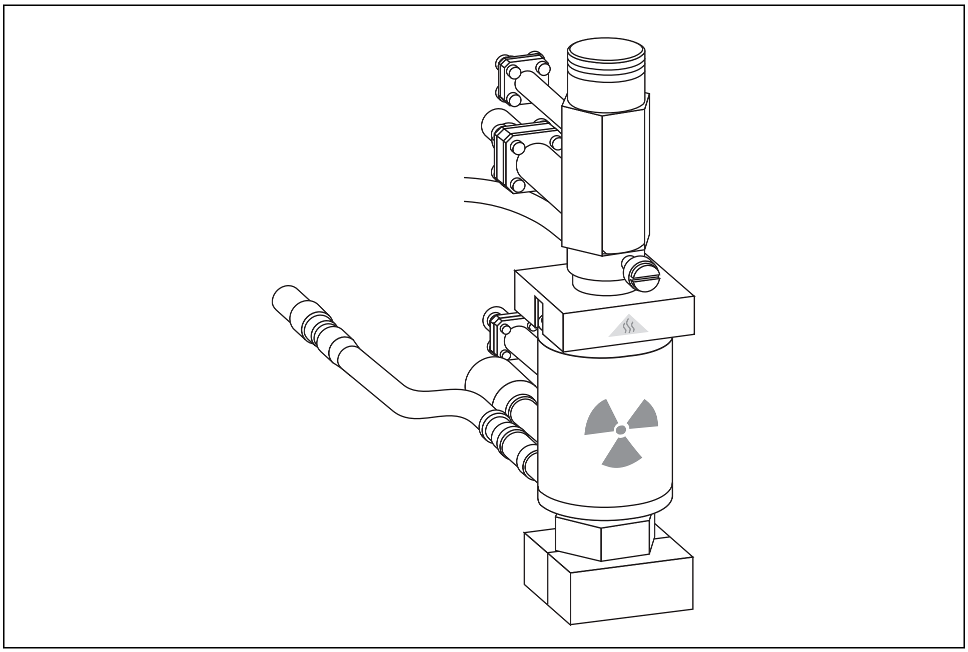


Figure 30-1. FID Tandem

Checkout Overview

The checkout must be carried out for each single ECD and FID detector, according to the injector used, referring to the relevant SOPs as reported in the following table.

Table 30-1. FID-ECD SOPs Reference

Detector	Refer To:
ECD	*Checkout Using ECD with S/SL Injector* on page 95.
	Checkout Using ECD with OC Injector on page 107.
	Checkout Using ECD with PKD Injector on page 119.
	Checkout Using ECD with PPKD Injector on page 131.
	Checkout Using ECD with PTV Injector on page 143.
FID	*Checkout Using FID with S/SL Injector* on page 37.
	Checkout Using FID with OC Injector on page 47.
	Checkout Using FID with PKD Injector on page 57.
	Checkout Using FID with PPKD Injector on page 69.
	Checkout Using FID with PTV Injector on page 81.

WARNING! To perform ECD checkout, refer to the relevant operating procedures reporting in Section III of this manual.

Before starting FID checkout procedures, it is strongly recommended to read the paragraph *Important Considerations* on page 400.

Important Considerations

This paragraph details the differences for FID checkout respect to the standard one reported in Section II of this manual.

FID Gas Required

In FID Tandem (stacked) configuration the FID only requires air and hydrogen as fuel gas to supply the flame.
The make-up gas is supplied by ECD.

Column Installation

When performing the checkout of the FID in stacked configuration it is not necessary any adjustment of the test column insertion depth. The test column remains connected to the ECD with the column insertion depth defined for this detector (109 mm measured from the bottom of the ferrule).

FID Detector and Signal Menus

When in stacked configuration, the FID is configured as **Auxiliary Detector**, then **AUX DETECTOR** and **AUX SIGNAL** instead of **LEFT/RIGHT DETECTOR** and **LEFT/RIGHT SIGNAL** must be pressed to access the relevant detector and signal menus.

The *Example of FID Tandem Checkout* operating procedure, on page 401, details the different procedure points respect to the standard FID checkout procedures reported in Section II of this manual.

OPERATING PROCEDURE

Example of FID Tandem Checkout

This procedure reports the different sequence points respect to the standard operating procedures reported in Section II. In the example, the S/SL injector is considered.

Before beginning, press **CONFIG** to verify the GC configuration:

Left inlet or Right inlet	**According to injector in use**
Left carrier or Right carrier	He (helium)
Aux detector	FID

1. Use **LEFT CARRIER** or **RIGHT CARRIER** to display the appropriate Carrier Gas Control Table. Verify to operate in constant pressure mode. If not, scroll to `Flow mode`, press **MODE/TYPE** to access the selection menu, then select `con pres`. Scrool to `Pressure` and set the pressure value to have the required carrier gas flow rate `Col.flow`.

```
        LEFT CARRIER[1]

Pressure         30.0   30.0
Col.flow         3.00
Lin. veloc.            (60.9)<
```

1. These settings could also be for a right carrier.

2. Use **OVEN** to display the Column Oven Control Table. Set the oven temperature and the Oven Program required.

```
             OVEN

Temp             50.0  50.0
Initial Time           1.00
Ramp 1                 20.0
Final temp              200
Final time 1           1.00<
Ramp 2                  Off
```

3. Use **LEFT INLET** or **RIGHT INLET** to display the appropriate Split/Splitless Injector Control Table. Set the required temperature *Temp* setpoint. Verify to operate in **Splitless** mode. If not, scroll to `Mode`, press **MODE/TYPE** to access the selection menu, then select `Splitless`. Scroll to `Splitless time` to set the required setpoint.

LEFT INLET (S/SL)[1]		
Temp	230	230
Pressure	30.0	30.0
Mode:		Splitless
Total flow		(63.0)
Split Flow	60.0	60.0
Splitless time	0.80	0.80
Constant sept purge?		Y<

1. These settings could also be for a right inlet.

4. Use **AUX DETECTOR** to display the appropriate FID Detector Control Table. Set the required temperature **`Base Temp`** and the detector gases **`H2`** and **`Air`** required setpoints.

AUX DETECTOR (FID)		
Flame		Off
Base temp	250	250
Signal pA		(5.5)
Ign.thresh		2.0
Flameout retry		Off
H2	35	35
Air	350	350<

5. Since the make-up gas is supplied by ECD detector, set the value into the ECD detector Control Table.

1. These settings could also be for a right detector.

6. Ignite the FID flame scrolling to `Flame` and pressing **ON**.

7. Use **AUX SIGNAL** to display the appropriate FID Detector Signal Control Table. Observe the FID flame signal at the display. This is the flame-on background offset. Scroll to `Range` and set the electrometer amplifier input range required.

AUX SIGNAL (FID)	
Output	(1000)
Offset	100
Auto zero?	Y/N
Range 10^(0..3)	0<
Analog filter	Off
Baseline Comp	Off

8. Activate your Data System and set the parameters required for the checkout.

9. In the Aux Detector Signal Control Table, scroll to `Auto zero?` and turn it **YES**.

10. Perform a blank analysis injecting pure hexane and press **START** on the GC to begin the checkout run.

Continue with Baseline Acquisition and Analysis.

31

Checkout Using Tandem NPD

Chapter at a Glance...

Operating Procedures

SOP Number: P0384/06/E - 01 September 2009

Scope

Use the following procedure to verify proper Nitrogen Phosphorus Detector (NPD) installed in series (stacked configuration, see Figure 31-1) with the non-destructive Electron Capture Detector ECD using different injectors.

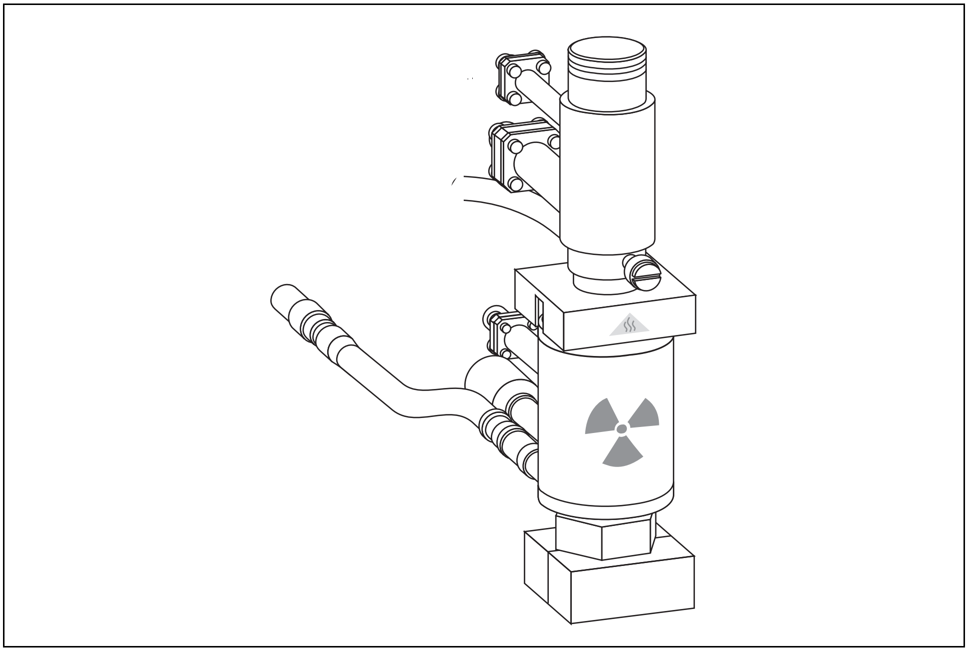


Figure 31-1. NPD Tandem

Checkout Overview

The checkout must be carried out for each single ECD and NPD detector, according to the injector used, referring to the relevant SOPs as reported in the following table.

Table 31-1. NPD-ECD SOPs Reference

Detector	Refer To:
ECD	*Checkout Using ECD with S/SL Injector* on page 95.
	Checkout Using ECD with OC Injector on page 107.
	Checkout Using ECD with PKD Injector on page 119.
	Checkout Using ECD with PPKD Injector on page 131.
	Checkout Using ECD with PTV Injector on page 143.
NPD	*Checkout Using NPD with S/SL Injector* on page 157.
	Checkout Using NPD with OC Injector on page 169.
	Checkout Using NPD with PKD Injector on page 181.
	Checkout Using NPD with PPKD Injector on page 195.
	Checkout Using NPD with PTV Injector on page 209.

WARNING! To perform ECD checkout, refer to the relevant operating procedures reporting in Section III of this manual.

Before starting NPD checkout procedures, it is strongly recommended to read the paragraph *Important Considerations* on page 408.

Important Considerations

This paragraph details the differences for NPD checkout respect to the standard one reported in Section IV of this manual.

NPD Gas Required

In NPD Tandem (stacked) configuration the NPD only requires air and hydrogen as fuel gas to supply the flame.
The make-up gas is supplied by ECD.

Column Installation

When performing the checkout of the NPD in stacked configuration it is not necessary any adjustment of the test column insertion depth. The test column remains connected to the ECD with the column insertion depth defined for this detector (109 mm measured from the bottom of the ferrule). For that reason, it is strongly recommended the use of the silcosteeled jet instead of the standard one.

NPD Detector and Signal Menus

When in stacked configuration, the NPD is configured as **Auxiliary Detector**, then **AUX DETECTOR** and **AUX SIGNAL** instead of **LEFT/RIGHT DETECTOR** and **LEFT/RIGHT SIGNAL** must be pressed to access the relevant detector and signal menus.

The *Example of NPD Tandem Checkout* operating procedure, on page 409, details the different procedure points respect to the standard NPD checkout procedures reported in Section IV of this manual.

OPERATING PROCEDURE

Example of NPD Tandem Checkout

This procedure reports the different sequence points respect to the standard operating procedures reported in Section IV. In the example, the S/SL injector is considered.

Before beginning, press **CONFIG** to verify the GC configuration:

Left inlet or Right inlet	**According to injector in use**
Left carrier or Right carrier	He (helium)
Aux detector	NPD

1. Use **LEFT CARRIER** or **RIGHT CARRIER** to display the appropriate Carrier Gas Control Table. Verify to operate in constant pressure mode. If not, scroll to `Flow mode`, press **MODE/TYPE** to access the selection menu, then select `con pres`. Scrool to `Pressure` and set the pressure value to have the required carrier gas flow rate `Col.flow`.

```
         LEFT CARRIER[1]

Pressure          30.0   30.0
Col.flow          3.00
Lin. veloc.            (60.9)<
```

1. These settings could also be for a right carrier.

2. Use **OVEN** to display the Column Oven Control Table. Set the oven temperature and the Oven Program required.

```
              OVEN

Temp              70.0  70.0
Initial Time            1.00
Ramp 1                  20.0
Final temp               230
Final time 1            1.00<
Ramp 2                   Off
```

3. Use **LEFT INLET** or **RIGHT INLET** to display the appropriate Split/Splitless Injector Control Table. Set the required temperature *Temp* setpoint. Verify to operate in **Splitless** mode. If not, scroll to `Mode`, press **MODE/TYPE** to access the selection menu, then select `Splitless`. Scroll to `Splitless time` to set the required setpoint.

LEFT INLET (S/SL)[1]		
Temp	230	230
Pressure	30.0	30.0
Mode:		Splitless
Total flow		(63.0)
Split Flow	60.0	60.0
Splitless time	0.80	0.80
Constant sept purge?		Y<

1. These settings could also be for a right inlet.

4. **Use AUX DETECTOR** to display the appropriate NPD Detector Control Table. Set the required temperature **`Base Temp`** and the detector gases **`H2`** and **`Air`** required setpoints.

AUX DETECTOR (NPD)		
Source cur,A		Off
Base temp	300	300
Signal pA		(10.4)
Target curr. pA		(X.XX)
Auto adjust		No
Polarizer V		3.5
H2 delay time		Off
H2	2.3	2.3
Air	60	60

5. Since the make-up gas is supplied by ECD detector, set the value into the ECD detector Control Table.

1. These settings could also be for a right detector.

6. Scroll to `Polarizer V` and set the required setpoint (3.5 V).

7. Scroll to `Source cur,A` and turn on the source as described in the relevant *Checkout Using NPD* procedure.

8. Use AUX Signal to display the appropriate NPD Detector Signal Control Table. Scroll to `Range` and set the electrometer amplifier input range required.

AUX SIGNAL (NPD)	
Output	**(1000)**
Offset	**100**
Auto zero?	**Y/N**
Range 10^(0..3)	**0<**
Analog filter	**Off**
Baseline Comp	**Off**

9. Activate your Data System and set the parameters required for the checkout.

10. In the Aux Detector Signal Control Table, scroll to `Auto zero?` and turn it **YES**.

11. Perform a blank analysis injecting pure hexane and press **START** on the GC to begin the checkout run.

Continue with Baseline Acquisition and Analysis.

32

Checkout Using Tandem FPD

Chapter at a Glance…

Operating Procedures

SOP Number: P0385/06/E - 01 September 2009

Scope

Use the following procedure to verify proper Flame Photometric Detector (FPD) installed in series (stacked configuration, see Figure 32-1) with the non-destructive Electron Capture Detector ECD using different injectors.

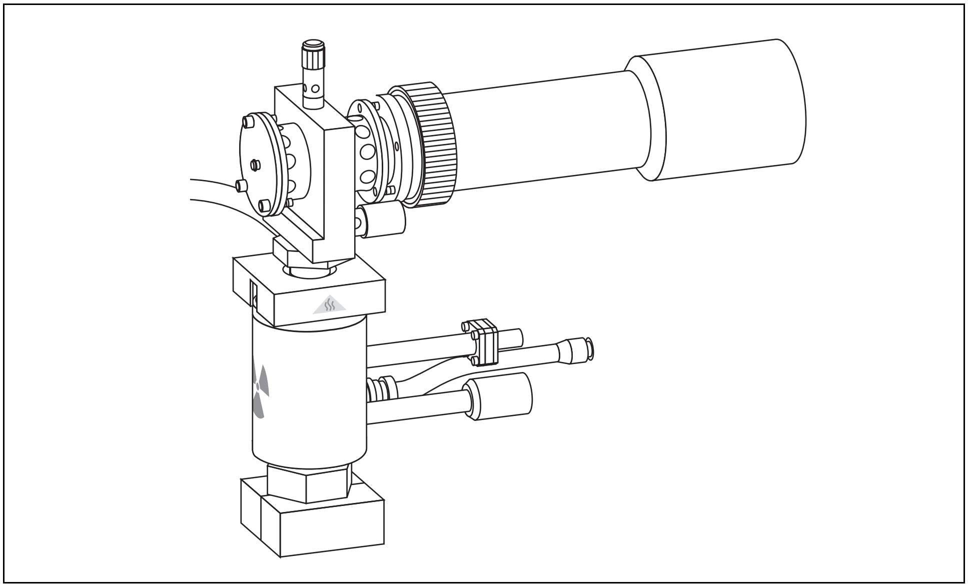


Figure 32-1. FPD Tandem

Checkout Overview

The checkout must be carried out for each single ECD and FPD detector, according to the injector used, referring to the relevant SOPs as reported in the following table.

Table 32-1. FPD-ECD SOPs Reference

Detector	Refer To:
ECD	*Checkout Using ECD with S/SL Injector* on page 95.
	Checkout Using ECD with OC Injector on page 107.
	Checkout Using ECD with PKD Injector on page 119.
	Checkout Using ECD with PPKD Injector on page 131.
	Checkout Using ECD with PTV Injector on page 143.
FPD	*Checkout Using FPD with S/SL Injector* on page 225.
	Checkout Using FPD with OC Injector on page 237.
	Checkout Using FPD with PKD Injector on page 249.
	Checkout Using FPD with PPKD Injector on page 263.
	Checkout Using FPD with PTV Injector on page 277.

WARNING! **To perform ECD checkout, refer to the relevant operating procedures reporting in Section III of this manual.**

Before starting FPD checkout procedures, it is strongly recommended to read the paragraph *Important Considerations* on page 416.

Important Considerations

This paragraph details the differences for FPD checkout respect to the standard one reported in Section V of this manual.

FPD Gas Required

In FPD Tandem (stacked) configuration the FPD only requires air and hydrogen as fuel gas to supply the flame.
The make-up gas supplied by ECD has to be maintained.

Column Installation

When performing the checkout of the FPD in stacked configuration it is not necessary any adjustment of the test column insertion depth. The test column remains connected to the ECD with the column insertion depth defined for this detector (109 mm measured from the bottom of the ferrule). For that reason, it is strongly recommended the use of the silcosteeled jet instead of the standard one.

FPD Detector and Signal Menus

When in stacked configuration, the FPD is configured as **Auxiliary Detector**, then **AUX DETECTOR** and **AUX SIGNAL** instead of **LEFT/RIGHT DETECTOR** and **LEFT/RIGHT SIGNAL** must be pressed to access the relevant detector and signal menus.

The *Example of FPD Tandem Checkout* operating procedure, on page 417, details the different procedure points respect to the standard FPD checkout procedures reported in Section V of this manual.

OPERATING PROCEDURE

Example of FPD Tandem Checkout

This procedure reports the different sequence points respect to the standard operating procedures reported in Section V. In the example, the S/SL injector is considered.

Before beginning, press **CONFIG** to verify the GC configuration:

Left inlet or Right inlet	**According to injector in use**
Left carrier or Right carrier	He (helium)
Aux detector	FPD

1. Use **LEFT CARRIER** or **RIGHT CARRIER** to display the appropriate Carrier Gas Control Table. Verify to operate in constant pressure mode. If not, scroll to `Flow mode`, press **MODE/TYPE** to access the selection menu, then select `con pres`. Scrool to `Pressure` and set the pressure value to have the required carrier gas flow rate `Col.flow`.

```
        LEFT CARRIER[1]

Pressure         30.0    30.0
Col.flow         3.00
Lin. veloc.             (60.9)<
```

1. These settings could also be for a right carrier.

2. Use **OVEN** to display the Column Oven Control Table. Set the oven temperature and the Oven Program required.

```
              OVEN

Temp             70.0   70.0
Initial Time            1.00
Ramp 1                  20.0
Final temp               230
Final time 1            1.00<
Ramp 2                   Off
```

3. Use **LEFT INLET** or **RIGHT INLET** to display the appropriate Split/Splitless Injector Control Table. Set the required temperature *Temp* setpoint. Verify to operate in **Splitless** mode. If not, scroll to `Mode`, press **MODE/TYPE** to access the selection menu, then select `Splitless`. Scroll to `Splitless time` to set the required setpoint.

```
    LEFT INLET (S/SL)¹

Temp                 230   230
Pressure            30.0  30.0
Mode:                Splitless
Total flow              (63.0)
Split Flow          60.0  60.0
Splitless time      0.80  0.80
Constant sept purge?        Y<
```

1. These settings could also be for a right inlet.

4. Use **AUX DETECTOR** to display the appropriate FPD Detector Control Table. Set the required temperature **`FPD Temp`** and the detector gases **`H2`** and **`Air`** required setpoints.

```
    AUX DETECTOR (FPD)

Flame                      Off
FPD temp             150   150
Signal pA                (1.4)
High voltage mode?           N
H2                    90    90
Air                  115   115
```

5. Since the make-up gas is supplied by ECD detector, set the value into the ECD detector Control Table.

Base temp	250	250
ECD Temp	300	300
Ref current nA		1.0
Freq kHz		(2.20)
Pulse amp V		50
Pulse width µs		1.0
Mkup (N2)	**30**	**30<**

1. These settings could also be for a right detector.

6. Verify that `High voltage mode` is set to **NO**.

7. Scroll to `Flame` and press **ON**. This start the ignition sequence.
   When ignition is confirmed, the photomultiplier tube is energized.
   The baseline level `Signal pA`, will suddenly increase meaning that the flame is lit inside the detector. After a few seconds, the baseline should stabilize to the standing current of the system.

8. Use AUX Signal to display the appropriate FPD Detector Signal Control Table. Scroll to `Range` and set the electrometer amplifier input range required.

AUX SIGNAL (FPD)	
Output	**(1000)**
Offset	**100**
Auto zero?	**Y/N**
Range 10^(0..3)	**0<**
Baseline Comp	**Off**

9. Activate your Data System and set the parameters required for the checkout.

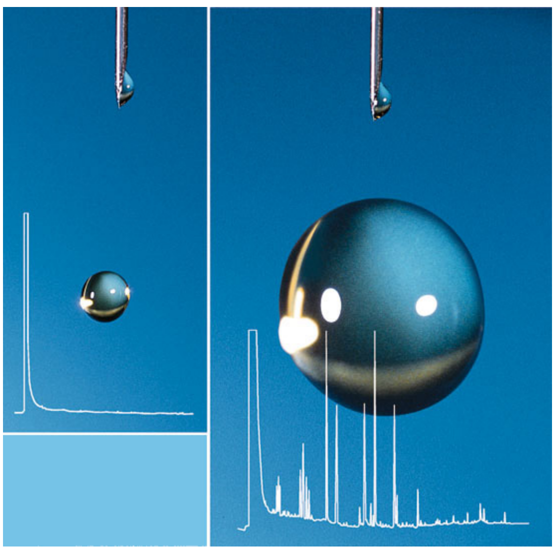
10. In the Aux Detector Signal Control Table, scroll to `Auto zero?` and turn it **YES**.

11. Perform a blank analysis injecting pure hexane and press **START** on the GC to begin the checkout run.

Continue with Baseline Acquisition and Analysis.

SECTION X

SOPs for Large Volume Applications



The *SOPs for Large Volume Applications* section, contains the procedures to test the TRACE GC Ultra for large volume application by using different injectors.

33

Checkout Using FID with LVSL Injector

Chapter at a Glance…

Operating Procedures

SOP Number: P0430/04/E - 01 September 2009

Scope

Use the following procedure to verify proper FID operation with the LVSL Injector.

Parts Referenced

Table 33-1. FID-LVSL 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	Retention gap DPTMDS deactivated; 5 m long 0.32 mm ID	260 800 10
Press fit	Deactivated universal press-tight connector (set of 5)	350 038 50
Glass Liner	5 mm ID for LVSL injection	453 020 65
Graphite Ferrule	Graphite ferrule for 0.32 mm ID Column	290 134 87
Retaining Nut	M4 capillary column retaining nut	350 324 23
LVSL Adapter	LVSL adapter for 9 mm septa	347 093 46
Vespel seal	Vespel seal for LVSL adapter	356 034 50
Septum Cap	Cap for LVSL adapter	350 010 55
Septum holder	Metallic holder for 9 mm septa	233 030 20
Septum	9 mm septa for LVSL injector (set of 10)	313 032 41
Syringe	50 µl size; 50 mm needle length, 0.63C, conic tip	365 030 15

Table 33-1. FID-LVSL Parts Referenced

Part	Description	Part Number
Test Mixture	Three components in n-Hexane: *Component* *Concentration* Dodecane 1 µg/ml Tetradecane 1 µg/ml Hexadecane 1 µg/ml	338 190 32
Gases	Chromatographic-grade purity	
Data Acquisition	Chrom-Card, ChromQuest, Atlas, Xcalibur, Computing-integrator	

Analytical Conditions Required for LVSL Injection

Table 33-2. FID-LVSL Analytical Conditions

Parameters	Setting
Gases	Carrier Gas: Helium = 30 kPa Constant Pressure Hydrogen = 35 ml/min Air = 350 ml/min Make-up Gas: Nitrogen = 30 ml/min
Oven Program	Initial Temperature = 55 °C Initial Time = 2 minutes Ramp 1 = 25 °C/minute Final Temperature = 200 °C Final Time = 1 minute
Injector	Operating Mode = Splitless Temperature = 230 °C Splitless Time = 0.8 minutes Split Flow = 60 ml/min Constant Septum Purge = Off Stop Purge for = 0.8 minutes
Detector	Base Temperature = 250 °C Detector Signal Range = 10^0
Injected Volume	20 µl + needle of Test Mixture
Analog Signal Output	Chrom-Card Acquisition Frequency = Medium
Digital Signal Output	Chrom-Card, ChromQuest, Atlas, Xcalibur Acquisition Frequency = 10 Hz

Recommended Initial Operations

Before starting the checkout, the following operations should be carried out:

1. Verify a “DPFC 2001” pneumatic module is installed.

2. Verify the LVSL kit is correctly installed (LVSL adapter, LV liner packed with glass wool).

3. Replace the 9-mm septum.
   A new septum should be installed properly in your injector.

4. Connect the required gas lines
   Verify the required gas supplies are properly connected to your GC.

5. Install the pre-column and connect the test column by means of the press fit.
   The column currently installed should be carefully removed and replaced with the required test column.

6. Perform Column Evaluation and Leak Test.

7. Connect your data handling.
   Verify that your data handling is properly connected to your GC system.

OPERATING PROCEDURE

FID-LVSL Checkout in Splitless Mode

Before beginning, press **CONFIG** to verify the GC configuration:

Left inlet or Right inlet	S/SL
Left carrier or Right carrier	He (helium)
Left detector or Right detector	FID

1. Use **LEFT CARRIER** or **RIGHT CARRIER** to display the appropriate Carrier Gas Control Table. Verify to operate in constant pressure mode. If not, scroll to `Flow mode`, press **MODE/TYPE** to access the selection menu, then select `con pres`. Scrool to `Pressure` and set the pressure value to have the required carrier gas flow rate `Col.flow`.

```
           LEFT CARRIER[1]

Pressure           30.0    30.0
Col.flow           3.00
Lin. veloc.               (60.9)<
```

1. These settings could also be for a right carrier.

2. Use **OVEN** to display the Column Oven Control Table. Set the oven temperature and the Oven Program required.

```
                OVEN

Temp               55.0   55.0
Initial Time              2.00
Ramp 1                    25.0
Final temp                 200
Final time 1              1.00<
Ramp 2                     Off
```

3. Use **LEFT INLET** or **RIGHT INLET** to display the appropriate Split/Splitless Injector Control Table. Set the required temperature *Temp* setpoint. Verify to

operate in **Splitless** mode. If not, scroll to `Mode`, press **MODE/TYPE** to access the selection menu, then select `Splitless`. Scroll to `Splitless time` to set the required setpoint.

LEFT INLET (S/SL)[1]		
Temp	230	230
Pressure	30.0	30.0
Mode:		Splitless
Total flow		(63.0)
Split Flow	60.0	60.0
Splitless time	0.80	0.80
Constant sept purge?		N
Stop purge for		0.8

1. These settings could also be for a right inlet.

4. Use **LEFT DETECTOR** or **RIGHT DETECTOR** to display the appropriate FID Detector Control Table. Set the required temperature `Base Temp` and the detector gases `H2`, `Air` and `Mkup` required setpoints.

LEFT DETECTOR (FID)[1]		
Flame		Off
Base temp	250	250
Signal pA		(5.5)
Ign.thresh		2.0
Flameout retry		Off
H2	35	35
Air	350	350
Mkup N2	30	30<

1. These settings could also be for a right detector.

5. Ignite the FID flame scrolling to `Flame` and pressing **ON**.

6. Use **LEFT SIGNAL** or **RIGHT SIGNAL** to display the appropriate FID Detector Signal Control Table. Observe the FID flame signal at the display. This is the

flame-on background offset. Scroll to `Range` and set the electrometer amplifier input range required.

LEFT SIGNAL (FID)[1]	
Output	(1000)
Offset	100
Auto zero?	Y/N
Range 10^(0..3)	0<
Analog filter	Off
Baseline Comp	Off

1. These settings could also be for a right signal.

7. Activate your Data System and set the parameters required for the checkout.

8. In the FID Detector Signal Control Table, scroll to `Auto zero?` and turn it **YES**.

9. Perform a blank analysis injecting pure hexane and press **START** on the GC to begin the checkout run.

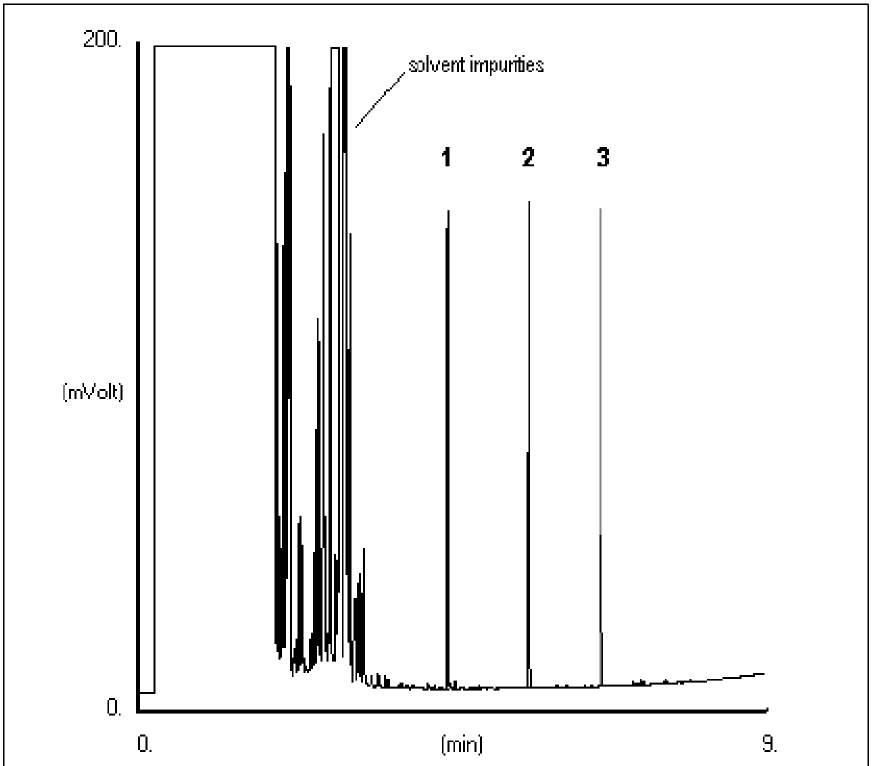
Baseline Acquisition and Analysis

NOTE Refer to the Acceptance Values reported in the Table 33-3 according to the data handling in use.

10. With the GC in Stand-by/Prep Run condition, activate the data system for 10 minutes to evaluate your baseline in isothermal condition.

11. After the baseline evaluation has been completed, set-up the data system to acquire a single run.

12. Inject the test mixture and press **START** on the GC to begin the checkout run. The resulting chromatogram should look like the one shown in *Figure 2.1.*



1. Dodecane 2. Tetradecane 3. Hexadecane

Figure 33-1. FID-LV Splitless Injection

13. The following criteria indicate successful completion of FID-LVSL checkout.

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

Table 33-3. FID-LVSL Acceptance Criteria

Acceptance Values	CHROM-CARD		
	Baseline Parameters	**Analog** *(1V Full Scale)*	**Digital** *(10V Full Scale)*
	Noise (μV)	< 30	< 300
	Wander (μV)	< 50	< 500
	Drift (μV/h)	< 100	< 1 000
	Analytical Results	**Analog** *(1V Full Scale)* *Area Counts (0.1 μVs)*	**Digital** *(10V Full Scale)* *Area Counts (0.1 μVs)*
	Components	> 1 500 000 for each component	> 15 000 000 for each component
	Area Count Ratio Calculated as C12/C16	1 ± 0.1	1 ± 0.1
⚠			

Acceptance Values	CHROMQUEST	
	Baseline Parameters *(1V Full Scale)*	
	Noise (μV)	< 30
	Wander (μV)	< 50
	Drift (μV/h)	< 100
	Analytical Results *(1V Full Scale) - Area Counts (0.01 μVs)*	
	Components	> 15 000 000 for each component
	Area Count Ratio Calculated as C12/C16	1 ± 0.1
⚠		

Acceptance Values	**ATLAS**	
	Baseline Parameters *(10V Full Scale)*	
	Noise (µV)	< 300
	Wander (µV)	< 500
	Drift (µV/h)	< 1 000
	Analytical Results *(10V Full Scale) - Area Counts (µVs)*	
	Components	> 1 500 000 for each component
	Area Count Ratio Calculated as C12/C16	1 ± 0.1
⚠		

Acceptance Values	**XCALIBUR**	
	Baseline Parameters *(Acquisition Frequency = 10 Hz)*	
	Noise (Counts)	< 3 000
	Wander Counts)	< 5 000
	Drift (Counts/h)	< 10 000
	Analytical Results *Area Counts (Cts*s)*	
	Components	> 15 000 000 for each component
	Area Count Ratio Calculated as C12/C16	1 ± 0.1
⚠		

Analytical Acceptance Comments	
1	When the make-up gas is not used, the acceptance values will result to be 2.5 times lower than the values reported in Table 33-3.
2	When helium is used as make-up gas, the acceptance values will result to be 10 times lower than the values reported in Table 33-3.

34

Checkout Using FID with LVOC Injector

Chapter at a Glance...

Operating Procedures

SOP Number: TE P0609/02/E-01 September 2009

Scope

Use the following procedure to verify proper FID operation with the LVOC Injector.

Parts Referenced

Table 34-1. FID-LVOC 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	UNCORET column, 15 m, 3 m coated with SE 54, 0.25 µm f.t.	260 604 27
Restrictor	Restrictor for SVE valve, 25 cm x 25µm ID	260 603 88
Connector	T-shaped connector	347 084 48
Vespel Ferrule	Vespel ferrule 0.9 mm hole (for connecting T-piece to SVE)	290 334 98
	Vespel ferrule for for 0.53 mm ID Column	290 134 71
Graphite Ferrule	Graphite ferrule for 0.32 mm ID Column	290 134 87
	Graphite ferrule for 0.53 mm ID Column	290 134 86
Retaining Nut	M4 capillary column retaining nut	350 324 23
	Column retaining nut and backwasher for OC	452 100 01
Seal	O-ring	290 113 02
Syringe	250 µl size; removable needle	365 004 90
Needle	(0,47/80 cone) for syringe p/n 365 004 90	365 664 80
Vial	2 ml screw-top vials	240 140 21
Cap	Screw-top cap for 2 ml vials p/n 240 140 21	386 060 92

Table 34-1. FID-LVOC Parts Referenced (Continued)

Part	Description	Part Number
Test Mixture	Three components in n-Hexane: *Component* *Concentration* Dodecane 1 µg/ml Tetradecane 1 µg/ml Hexadecane 1 µg/ml	338 190 32
Sampler	TriPlus autosampler	
Gases	Chromatographic-grade purity	
Data Acquisition	Chrom-Card, ChromQuest, Atlas, Xcalibur	

Analytical Conditions Required for LVOC Injection

Analytical Condition for the GC

Table 34-2. FID-LVOC Analytical Conditions for the GC

GC Parameters Setting	
Gases	Carrier Gas: Helium = 60 kPa Constant Pressure SVE flow = > 10 ml/min Sweep flow = ≥ 0,05 ml/min Hydrogen = 35 ml/min Air = 350 ml/min Make-up Gas: Nitrogen = 30 ml/min
Oven Program	Initial Temperature = 72 °C Initial Time = 2.5 minutes Ramp 1 = 50 °C/minute Final Temperature = 220 °C Final Time = 1 minute
Injector	Operating Mode = LVOC Secondary cooling = 6 s SVE Temperature = 150 °C SVE Duration = 12 s
Detector	Base Temperature = 250 °C Detector Signal Range = 10^0
Injected Volume	100 µl of Test Mixture
Digital Signal Output	Chrom-Card, ChromQuest, Atlas, Xcalibur Acquisition Frequency = 10 Hz

Analytical Condition for the TriPLus Autosampler

Table 34-3. FID-LVOC Analytical Conditions for the TriPlus Autosampler

TriPlus Parameters Setting	
Sampling	Sampling vol (µL) = 100 Plunger strokes = 0 Air volume (µL) = 10 Filling volume (µL) = 110
Injection depth mode	Pre-injection dwell time (s) = 0,2 Post-injection dwell time (s) = 0,3 Injection depth (mm) = 71 Injection speed (µL/s) = 10
Sampling depth in vial	Sampling vial depth % = 100
Sample viscosity	Sample pull up speed (µL/s) = 5 Delay after bubble elimination (s) = 1 Viscosity delay (s) = 0.3
GC syncro start	Synchro type = Delayed
Advanced parameters	Wash solvent depth % = 96 Waste depth % = 20 Needle speed into vial (mm/s) = 100 Solvent filling pull-up speed (µL/s) =10 Bubble elimination pull-up speed (µL/s) =10 Delay between strokes (s) = 0.1

Recommended Initial Operations

Before starting the checkout, the following operations should be carried out:

1. Connect the required gas lines.
   Verify the required gas supplies are properly connected to your GC.

2. Verify a “DPFC 2001” pneumatic module is installed.

3. Verify the LVOC actuator for TriPlus is correctly installed.

4. Replace the O-ring.
   A new O-ring should be installed properly in your injector

5. Installation of the pre-column and test column
   The column currently installed should be carefully removed and replaced with the required test column.
   Install the pre-column and connect it to the test column and the SVE by using the T-shaped connector. Insert the column into the precolumn for about two cm.

6. Install and connect the TriPlus sampler and its components.

7. Verify the opening/closing of the OC injector actuator by using the proper commands.

8. Verify the alignment of the sirynge on the OC injector.

9. Perform Column Evaluation and Leak Test.

10. Check the sweep flow at the outlet of the restrictor.
    It should be $\geq$ 0.05 ml/min.

11. Check the SVE flow. It should be > 10 ml/min.

12. Connect your data handling.
    Verify that your data handling is properly connected to your GC system.

OPERATING PROCEDURE

FID-LVOC Checkout in LVOC Mode

1. Set the GC parameters required to perform a LVOC injection listed in Table 34-2 on page 438.

2. Set the TriPlus parameters required to perform a LVOC injection listed in Table 34-3 on page 439

3. In the FID Detector Signal Control Table, scroll to `Auto zero?` and turn it **YES**.

4. Perform a blank analysis injecting pure hexane and press **START** on the GC to begin the checkout run.

Baseline Acquisition and Analysis

NOTE Refer to the Acceptance Values reported in the Table 34-4 according to the data handling in use.

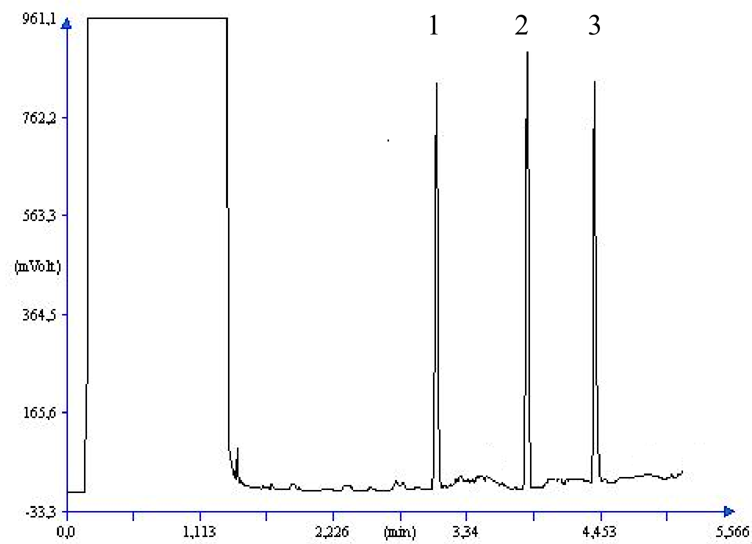
5. With the GC in Stand-by/Prep Run condition, activate the data system for 10 minutes to evaluate your baseline in isothermal condition.

6. After the baseline evaluation has been completed, set-up the data system to acquire a single run.

7. Fill one 2 ml vial with the sample and cap. Install the vial into the autosampler tray position 1.

8. Set up in the data system a sequence of three runs.

9. Perform the sequence. The resulting chromatogram should look like the one shown in *Figure 34-1.*



1. Dodecane	2. Tetradecane	3. Hexadecane

Figure 34-1. FID-LVOC Injection

10. The following criteria indicate successful completion of FID-LVOC checkout.

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

Table 34-4. FID-LVOC Acceptance Criteria

Acceptance Values	CHROM-CARD		
	Baseline Parameters	**Analog** *(1V Full Scale)*	**Digital** *(10V Full Scale)*
	Noise (μV)	< 30	< 300
	Wander (μV)	< 50	< 500
	Drift (μV/h)	< 100	< 1 000
	Analytical Results	**Analog** *(1V Full Scale)* *Area Counts (0.1 μVs)*	**Digital** *(10V Full Scale)* *Area Counts (0.1 μVs)*
	Components	> 10 000 000 for each component	> 100 000 000 for each component
	Area Count Ratio Calculated as C12/C16	1 ± 0.1	1 ± 0.1
⚠			

Acceptance Values	CHROMQUEST	
	Baseline Parameters *(1V Full Scale)*	
	Noise (μV)	< 30
	Wander (μV)	< 50
	Drift (μV/h)	< 100
	Analytical Results *(1V Full Scale) - Area Counts (0.01 μVs)*	
	Components	> 100 000 000 for each component
	Area Count Ratio Calculated as C12/C16	1 ± 0.1
⚠		

Acceptance Values	ATLAS	
	Baseline Parameters *(10V Full Scale)*	
	Noise (µV)	< 300
	Wander (µV)	< 500
	Drift (µV/h)	< 1 000
	Analytical Results *(10V Full Scale) - Area Counts (µVs)*	
	Components	> 10 000 000 for each component
	Area Count Ratio Calculated as C12/C16	1 ± 0.1
⚠		

Acceptance Values	XCALIBUR	
	Baseline Parameters *(Acquisition Frequency = 10 Hz)*	
	Noise (Counts)	< 3 000
	Wander Counts)	< 5 000
	Drift (Counts/h)	< 10 000
	Analytical Results *Area Counts (Cts*s)*	
	Components	> 100 000 000 for each component
	Area Count Ratio Calculated as C12/C16	1 ± 0.1
⚠		

Analytical Acceptance Comments	
1	When the make-up gas is not used, the acceptance values will result to be 2.5 times lower than the values reported in Table 34-4.
2	When helium is used as make-up gas, the acceptance values will result to be 10 times lower than the values reported in Table 34-4.

A

Customer Communication

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

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

How To Contact Us

Use http://www.thermo.com/com/cda/resources/resource_detail/1,,12512,00.html address for products information.

Use http://www.gc-gcms-customersupport.com/WebPage/Share/Default.aspx address to contact your local Thermo Fisher Scientific office or affiliate GC-GC/MS Customer Support.

Reader Survey

Product: TRACE GC Ultra
Manual: Standard Operating Procedures Manual
Part No.: 317 092 00

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

Glossay

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
PDD	Pulsed Discharge Detector
PID	Photo Ionization Detector
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
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

UFM	Ultra Fast Module

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

Index

Operating Procedures

Figures

Tables