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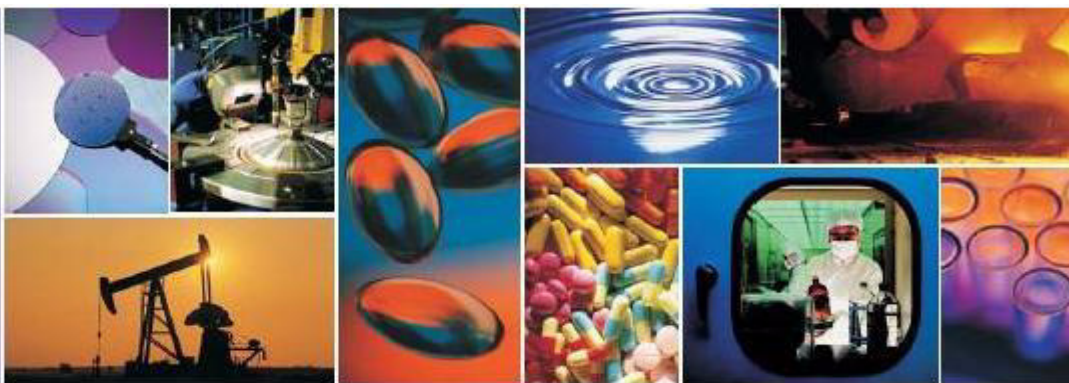
TRACE GC Ultra

Gas Chromatograph

Standard Operating Procedures

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[Home](#)



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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 *Glossary* 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. |
| <i>Italic</i> | 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.




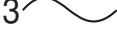






Abbreviations for Injectors and Detectors








| | 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 ^{63}Ni 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.

General Overview

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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 <i>Carrier Gas</i> = Helium <i>Fuel Gases</i> = Hydrogen - Air <i>Make-up Gas</i> = Nitrogen <i>Discharge gas for PDD</i> = 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 Injector*
- *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

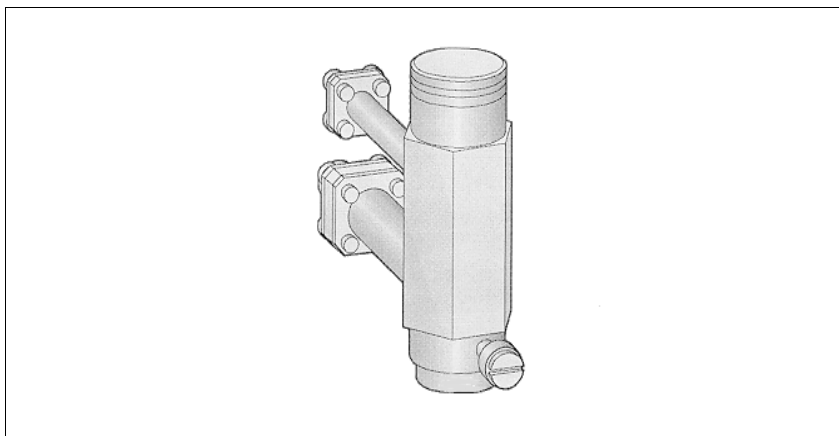
| RIGHT INLET (PTV) | | |
|-------------------|---------------|------|
| Temp | 70 | 70 |
| Pressure | 30.0 | 30.0 |
| Mode: | PTV Splitless | |

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.

Chapter 2, *Checkout Using FID with S/SL Injector*.

Chapter 3, *Checkout Using FID with OC Injector*.

Chapter 4, *Checkout Using FID with PKD Injector*.

Chapter 5, *Checkout Using FID with PPKD Injector*.

Chapter 6, *Checkout Using FID with PTV Injector*.

Checkout Using FID with S/SL Injector

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| FID-S/SL Checkout in Splitless Mode | 41 |
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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: <i>Component</i> <i>Concentration</i> 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 | |
| H ₂ | 35 | 35 |
| Air | 350 | 350 |
| Mkup N ₂ | 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*.

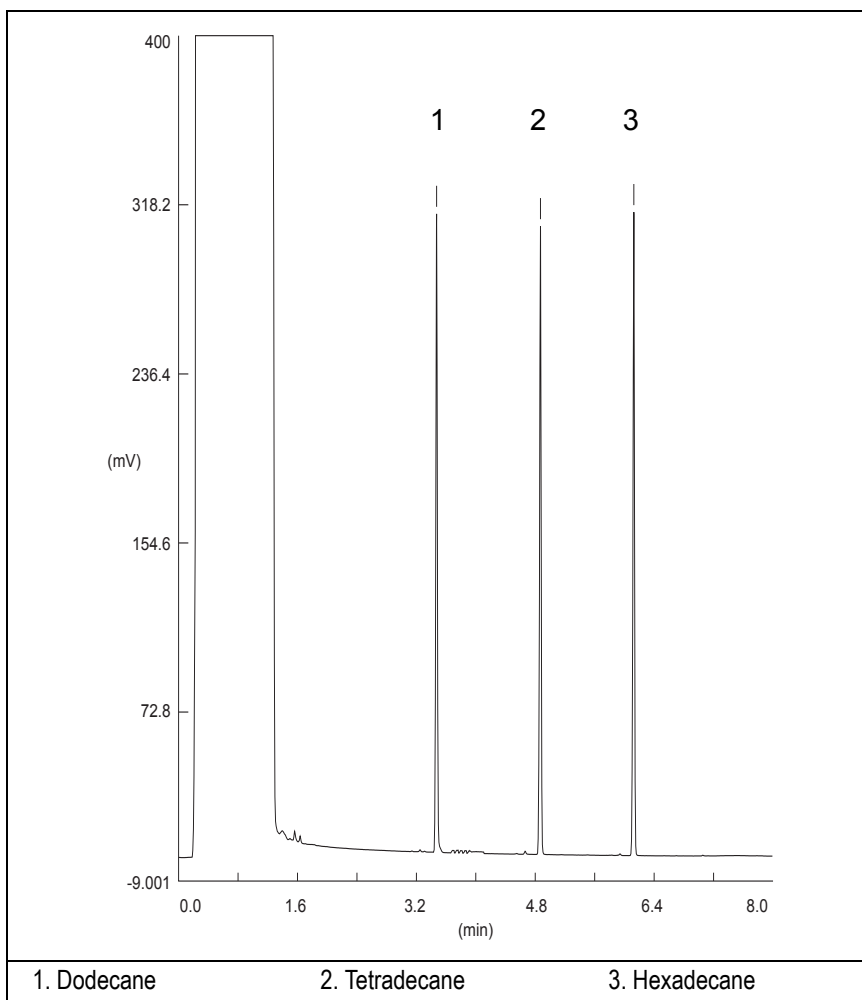





Figure 2-1. FID-Splitless Injection


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 ($\mu\text{V/h}$) | < 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 ($\mu\text{V/h}$) | < 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 ($\mu\text{V/h}$) | < 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. |

Checkout Using FID with OC Injector

Chapter at a Glance...

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| SOP Number: P0293/08/E - 01 September 2009 | 48 |
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| FID-OCI Checkout in On-Column Mode..... | 51 |
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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: <i>Component</i> <i>Concentration</i> 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*.

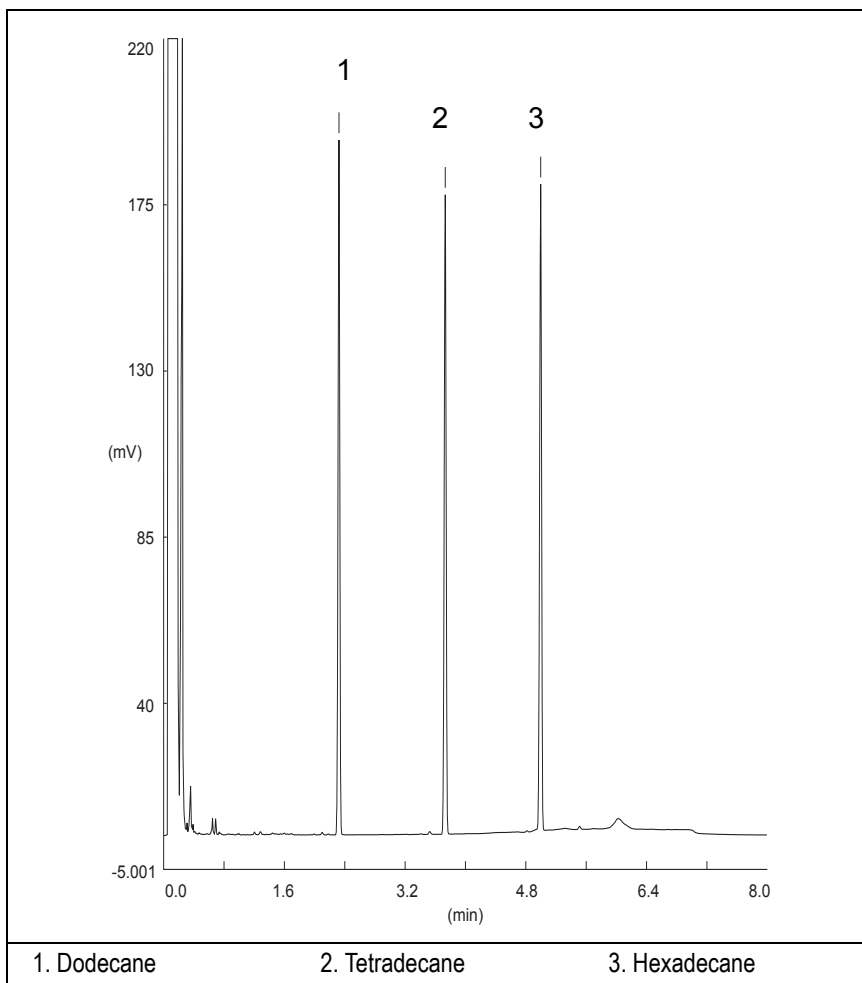





Figure 3-1. FID-On-Column Injection


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 ($\mu\text{V/h}$) | < 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 ($\mu\text{V/h}$) | < 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 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. |

Checkout Using FID with PKD Injector

Chapter at a Glance...

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Operating Procedures

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| FID-PKD Checkout | 62 |
|------------------------|----|

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 | 290 134 86 |
| | Graphite ferrule for 0.32 mm ID Column | 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: | 338 190 20 |
| | <i>Component</i> <i>Concentration</i> | |
| | Dodecane 20 µg/ml | |
| | Tetradecane 20 µg/ml | |
| | Hexadecane 20 µg/ml | |
| 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 “*Performing 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.** 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. 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 |
| H ₂ | 35 | 35 |
| Air | 350 | 350 |
| Mkup N ₂ | 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*

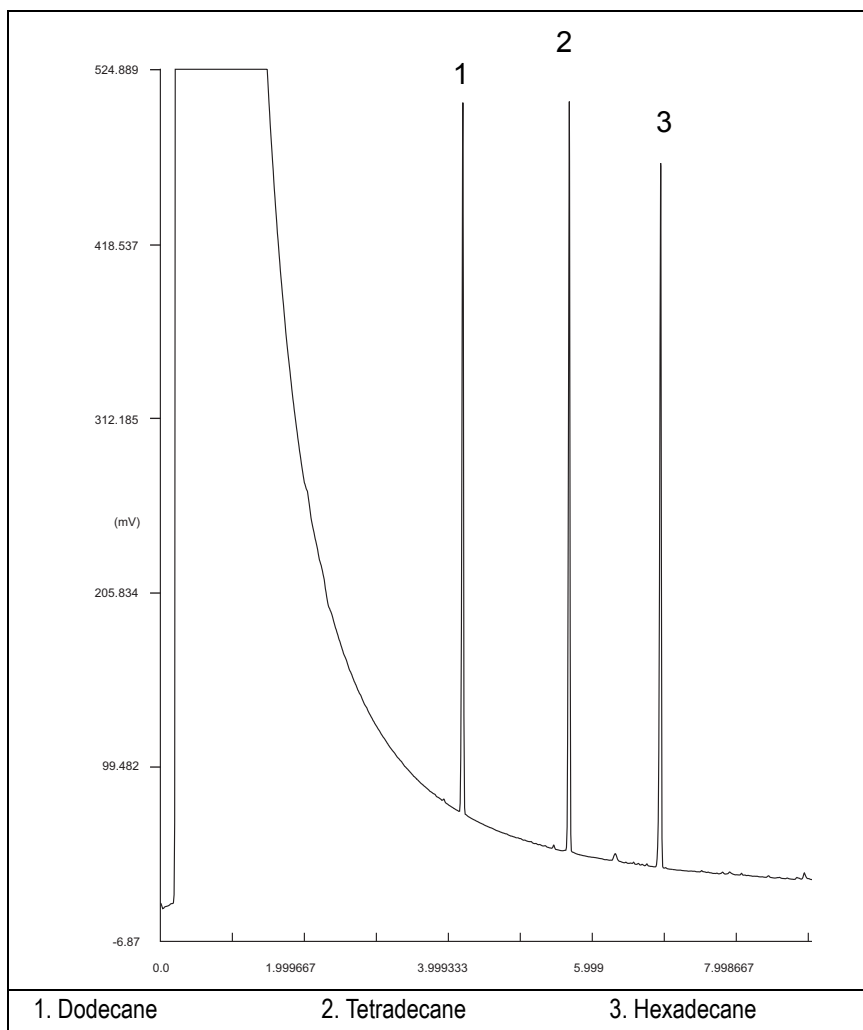





Figure 4-1. FID-Packed Injection


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 ($\mu\text{V/h}$) | < 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 ($\mu\text{V/h}$) | < 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 ($\mu\text{V/h}$) | < 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 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. |

Checkout Using FID with PPKD Injector

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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 | 290 134 86 |
| | Graphite ferrule for 0.32 mm ID Column | 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: | 338 190 20 |
| | <i>Component</i> <i>Concentration</i> | |
| | Dodecane 20 µg/ml | |
| | Tetradecane 20 µg/ml | |
| | Hexadecane 20 µg/ml | |
| 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 “*Performing 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.** 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. 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 |
| H ₂ | 35 | 35 |
| Air | 350 | 350 |
| Mkup N ₂ | 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*

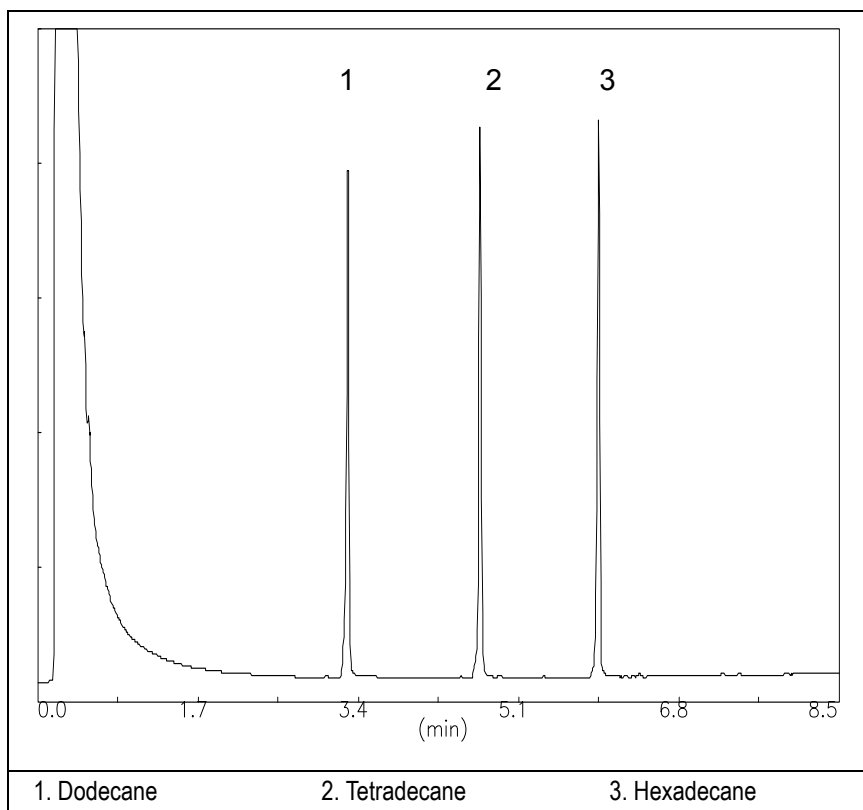





Figure 5-1. FID-PPKD Injection


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 ($\mu\text{V/h}$) | < 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 ($\mu\text{V/h}$) | < 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 ($\mu\text{V/h}$) | < 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 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. |

Checkout Using FID with PTV Injector

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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: <i>Component</i> <i>Concentration</i> 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**. Scroll 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 | 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*

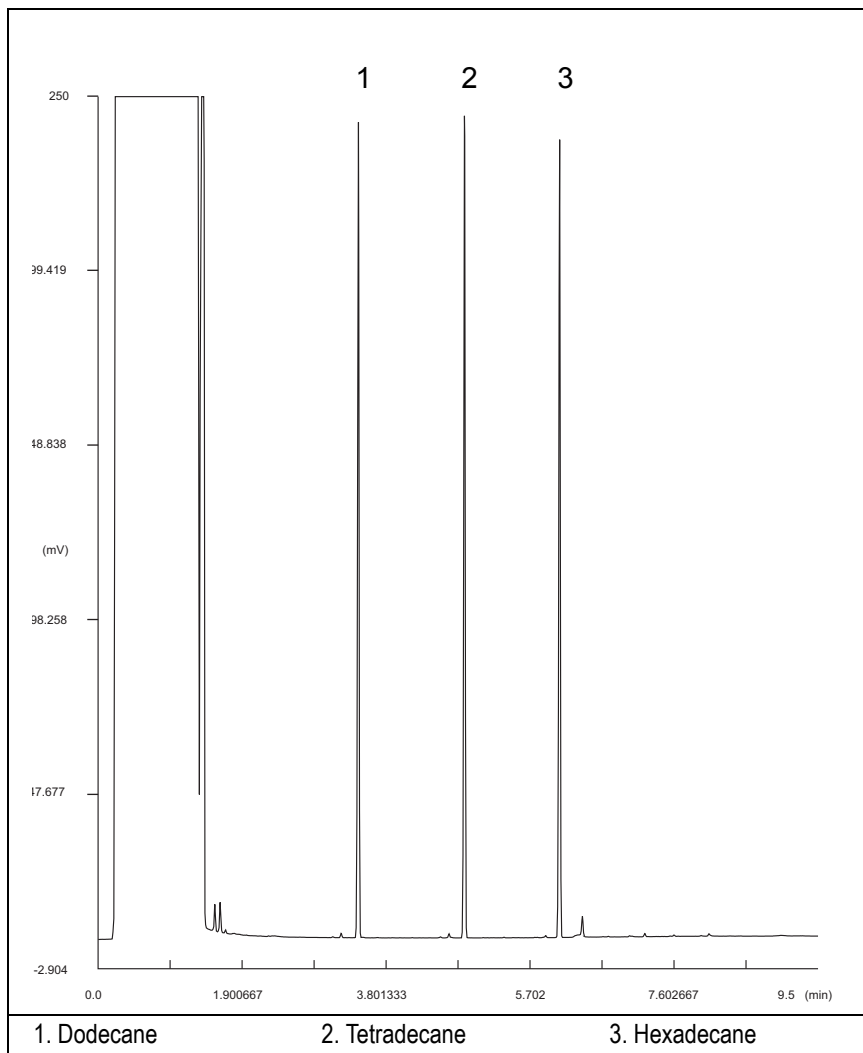





Figure 6-1. FID-PTV Injection


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 ($\mu\text{V/h}$) | < 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) | |
|---|--|
|  | |

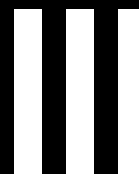
| Acceptance Values | CHROMQUEST | |
|---|---|---------------------------------|
| | Baseline Parameters (1V Full Scale) | |
| | Noise (μV) | < 30 |
| | Wander (μV) | < 50 |
| | Drift ($\mu\text{V/h}$) | < 100 |
| | Analytical Results (1V Full Scale) - Area Counts (0.01 μVs) | |
| | Components | > 25 000 000 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 ($\mu\text{V/h}$) | < 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 |
|  | |

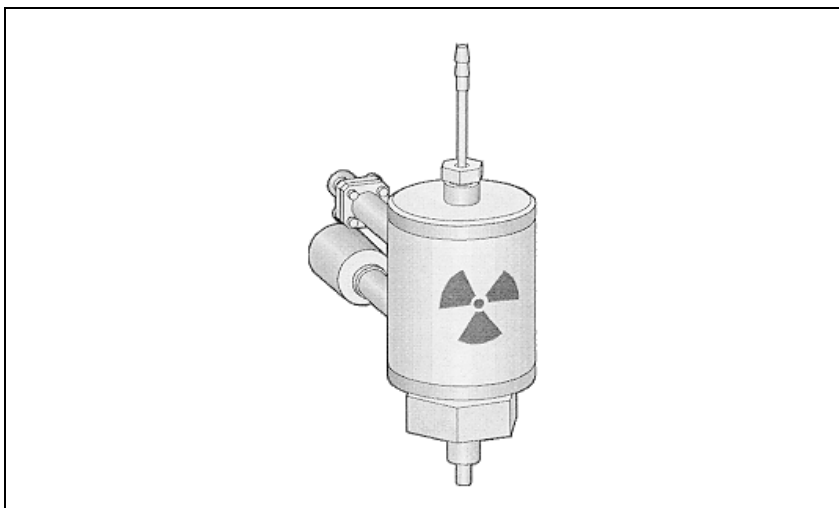
| Acceptance Values | XCALIBUR | |
|---|---|---------------------------------|
| | Baseline Parameters (Acquisition Frequency = 10 Hz) | |
| | Noise (Counts) | < 3 000 |
| | Wander Counts) | < 5 000 |
| | Drift (Counts/h) | < 10 000 |
| | Analytical Results Area Counts (Cts*s) | |
| | Components | > 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



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.

Chapter 7 *Checkout Using ECD with S/SL Injector.*

Chapter 8 *Checkout Using ECD with OC Injector.*

Chapter 9 *Checkout Using ECD with PKD Injector*

Chapter 10 *Checkout Using ECD with PPKD Injector.*

Chapter 11 *Checkout Using ECD with PTV Injector.*



Checkout Using ECD with S/SL Injector

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| ECD-S/SL Checkout in Splitless Mode | 99 |
|---|----|

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 <i>Component Concentration</i> 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 = 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. 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.** 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. 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 μ s | 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 1 μ s 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*.

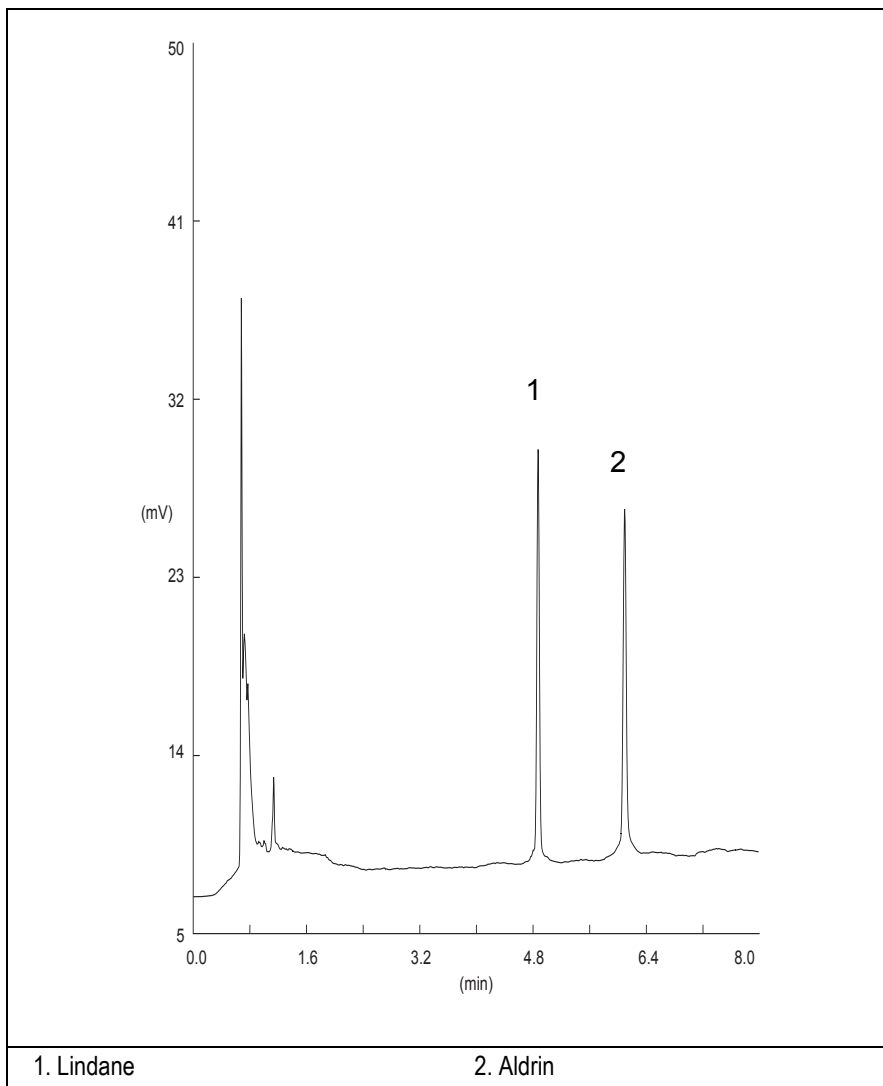


Figure 7-1. ECD-Splitless Injection

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 ($\mu\text{V/h}$) | < 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 ($\mu\text{V/h}$) | < 200 |
| | Analytical Results | |
| | Lindane Signal-to-Noise Ratio | > 4 000 |
| | Aldrin Signal-to-Noise Ratio | > 4 000 |
| |  | |

| ATLAS | |
|---|---------|
| Baseline Parameters (10V Full Scale) | |
| Noise (μV) | < 400 |
| Wander (μV) | < 800 |
| Drift ($\mu\text{V/h}$) | < 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 <i>Peak Height (counts)/noise (counts)</i> . |

Checkout Using ECD with OC Injector

Chapter at a Glance...

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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 <i>Component Concentration</i> 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-fit 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 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. 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 μ s | | 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 1 μ s 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 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 8.1*.

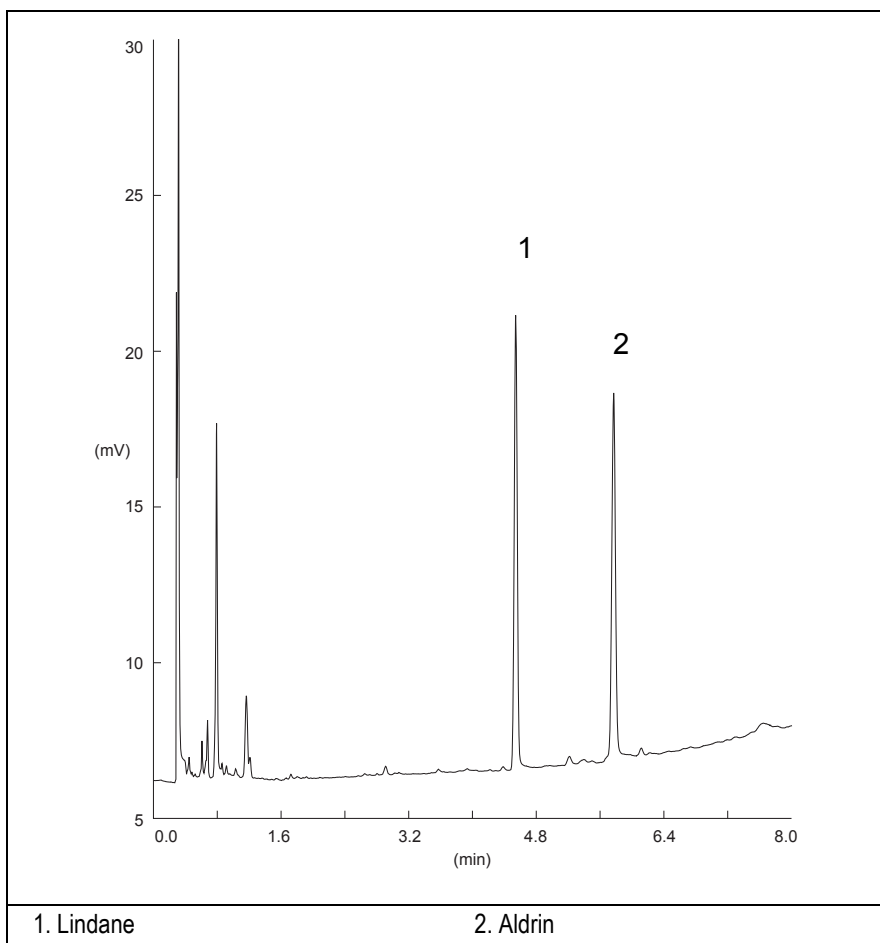


Figure 8-1. ECD-On-Column Injection

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 ($\mu\text{V/h}$) | < 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 ($\mu\text{V/h}$) | < 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 ($\mu\text{V/h}$) | < 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 <i>Peak Height (counts)/noise (counts)</i> . |

Checkout Using ECD with PKD Injector

Chapter at a Glance...

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| Scope | 120 |
| Parts Referenced | 120 |
| Analytical Conditions Required for Packed Injector | 122 |
| Recommended Initial Operations | 123 |

Operating Procedures

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| ECD-PKD Checkout | 124 |
|------------------------|-----|

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 | 290 134 86 |
| | Graphite ferrule for 0.32 mm ID Column | 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 | 338 190 11 |
| | <i>Component</i> <i>Concentration</i> | |
| | Lindane 0.030 µg/ml | |
| | Aldrin 0.030 µg/ml | |
| 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 “*Performing 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.** 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. 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 μ s | | 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 1 μ s 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*.

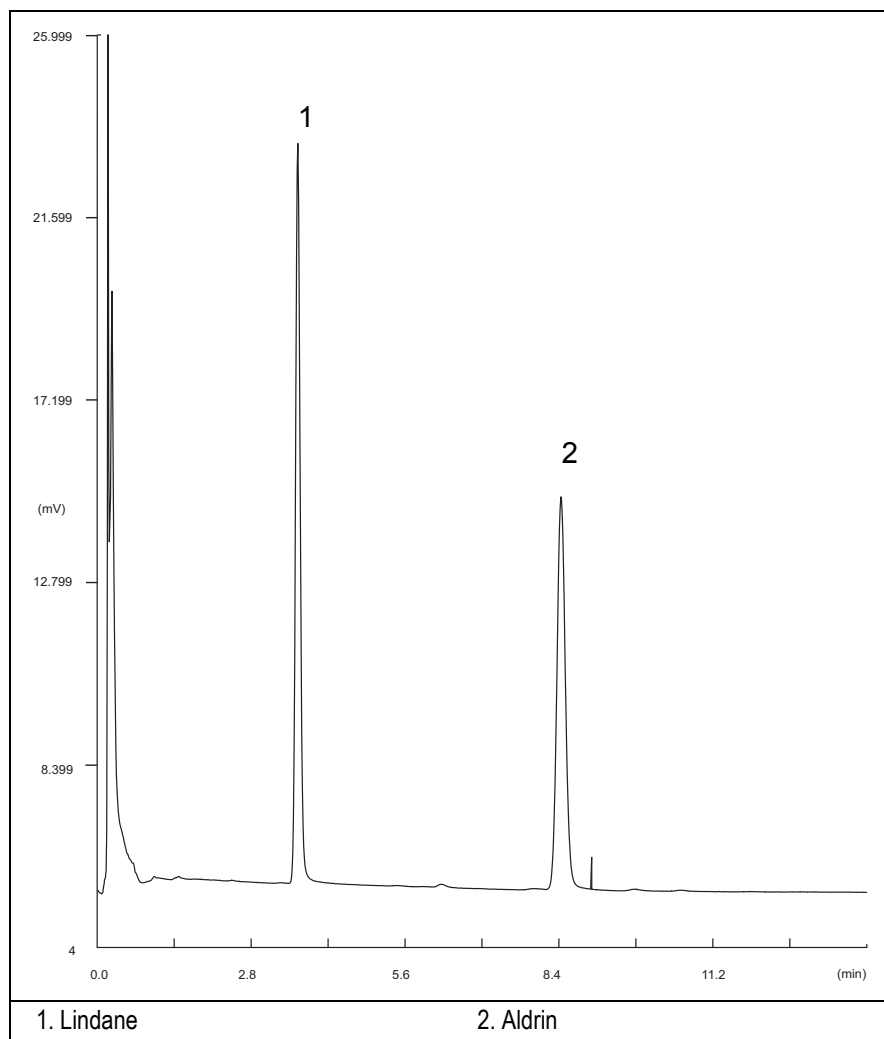


Figure 9-1. ECD-PKD Injection

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 ($\mu\text{V/h}$) | < 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 ($\mu\text{V/h}$) | < 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 ($\mu\text{V/h}$) | < 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 <i>Peak Height (counts)/noise (counts)</i> . |

Checkout Using ECD with PPKD Injector

Chapter at a Glance...

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| ECD-PPKD Checkout | 136 |
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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 | 290 134 86 |
| | Graphite ferrule for 0.32 mm ID Column | 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 | 338 190 11 |
| | <i>Component</i> <i>Concentration</i> | |
| | Lindane 0.030 µg/ml | |
| | Aldrin 0.030 µg/ml | |
| 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 = 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 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 “*Performing 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 μ s | | 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 1 μ s 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*.

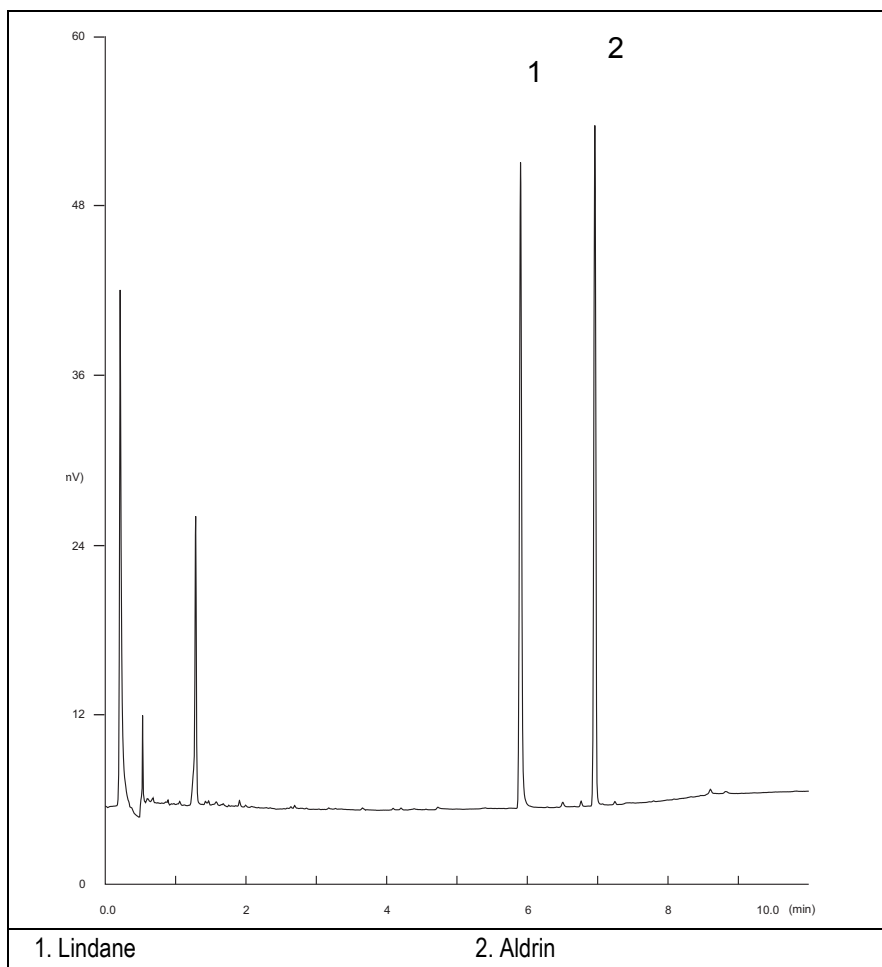


Figure 10-1. ECD-PPKD Injection

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 ($\mu\text{V/h}$) | < 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 ($\mu\text{V/h}$) | < 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 ($\mu\text{V/h}$) | < 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 <i>Peak Height (counts)/noise (counts)</i> . |

Checkout Using ECD with PTV Injector

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| ECD-PTV Checkout in PTV Splitless Mode | 147 |
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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 <i>Component Concentration</i> 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**. Scroll 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 | | 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 μ s | | 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 1 μ s 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.



NOTE

Baseline Acquisition and Analysis

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*.

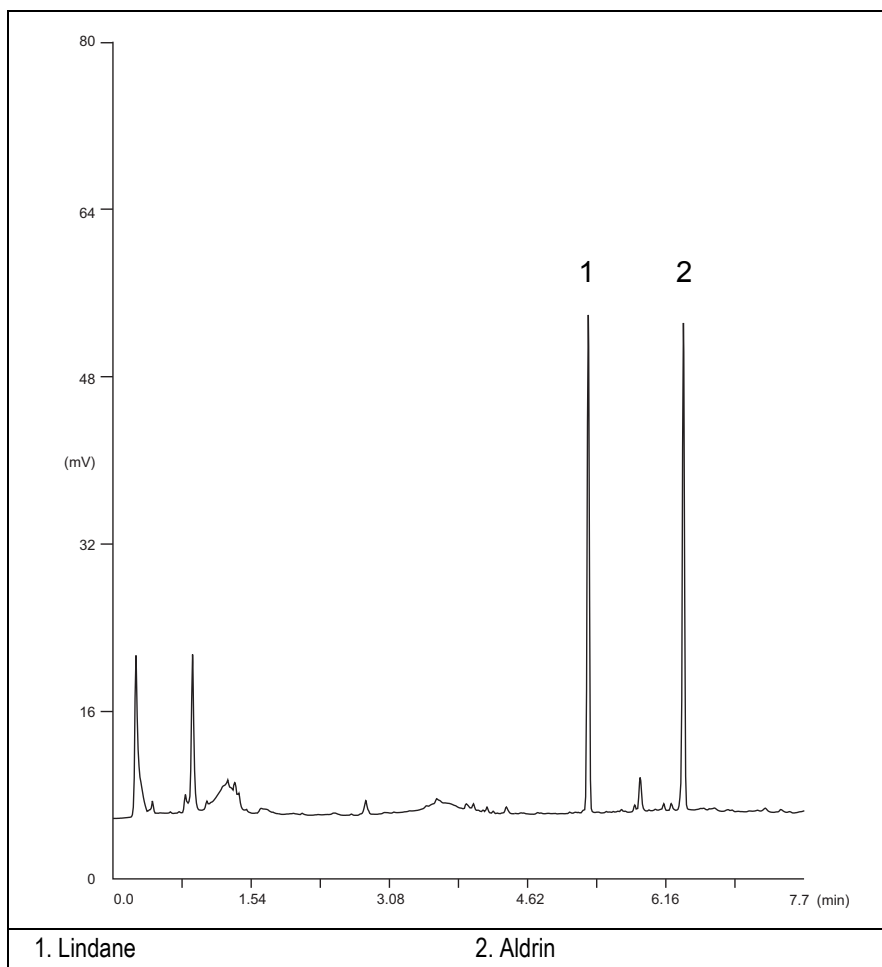


Figure 11-1. ECD-PTV Injection

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 ($\mu\text{V/h}$) | < 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 ($\mu\text{V/h}$) | < 200 |
| | Analytical Results | |
| | Lindane Signal-to-Noise Ratio | > 3 000 |
| | Aldrin Signal-to-Noise Ratio | > 3 000 |
|  | | |

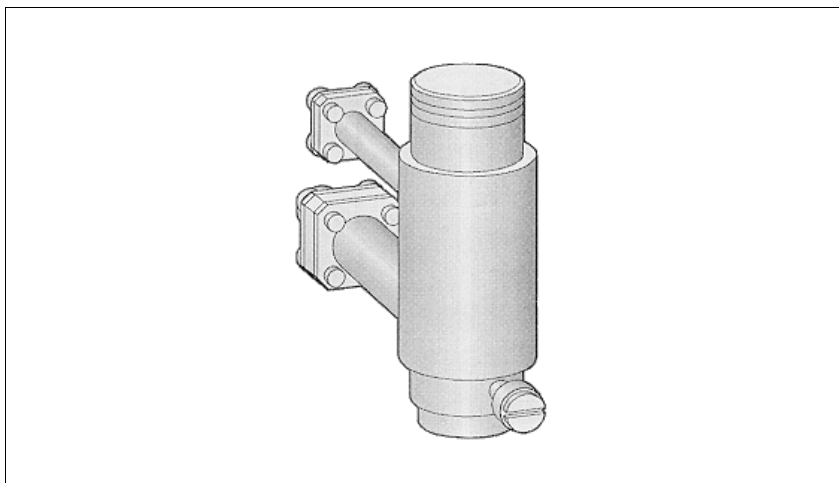
| | ATLAS | |
|---|--------------------------------------|---------|
| | Baseline Parameters (10V Full Scale) | |
| | Noise (μV) | < 400 |
| | Wander (μV) | < 800 |
| | Drift ($\mu\text{V/h}$) | < 2 000 |
| | Analytical Results | |
| | Lindane Signal-to-Noise Ratio | > 3 000 |
| | Aldrin Signal-to-Noise 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 <i>Peak Height (counts)/noise (counts)</i> . |

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.

Chapter 12, *Checkout Using NPD with S/SL Injector*.

Chapter 13, *Checkout Using NPD with OC Injector*.

Chapter 14, *Checkout Using NPD with PKD Injector*.

Chapter 15, *Checkout Using NPD with PPKD Injector*.

Chapter 16, *Checkout Using NPD with PTV Injector*.

Checkout Using NPD with S/SL Injector

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| NPD-S/SL Checkout in Splitless Mode | 161 |
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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: <div> <div>Component</div> <div>Concentration</div> <div>Azobenzene</div> <div>1 µg/ml</div> <div>Octadecane</div> <div>1000 µg/ml</div> <div>Parathion methyl</div> <div>1 µg/ml</div> </div> | 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 = <i>Refer to Source Ignition</i> 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.

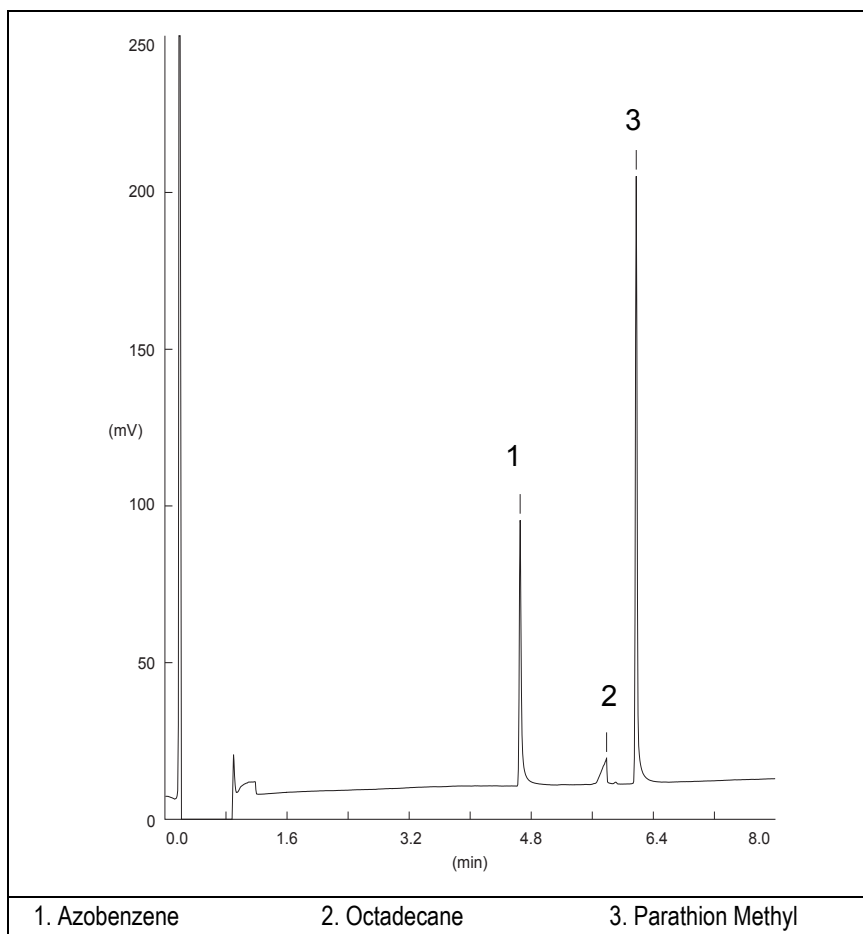


Figure 12-1. NPD-Splitless Injection

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 ($\mu\text{V/h}$) | < 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 ($\mu\text{V/h}$) | < 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 ($\mu\text{V/h}$) | < 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 |
|  | | |

Checkout Using NPD with OC Injector

Chapter at a Glance...

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| NPD-OCI Checkout in On-Column Mode | 173 |
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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: <i>Component</i> <i>Concentration</i> 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-fit 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 = <i>Refer to Source Ignition</i> 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:

- $H_2 = 2.3 \text{ ml/min}$
 - $Air = 60 \text{ ml/min}$
 - $Mkup N_2 = 15 \text{ ml/min}$
- 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.

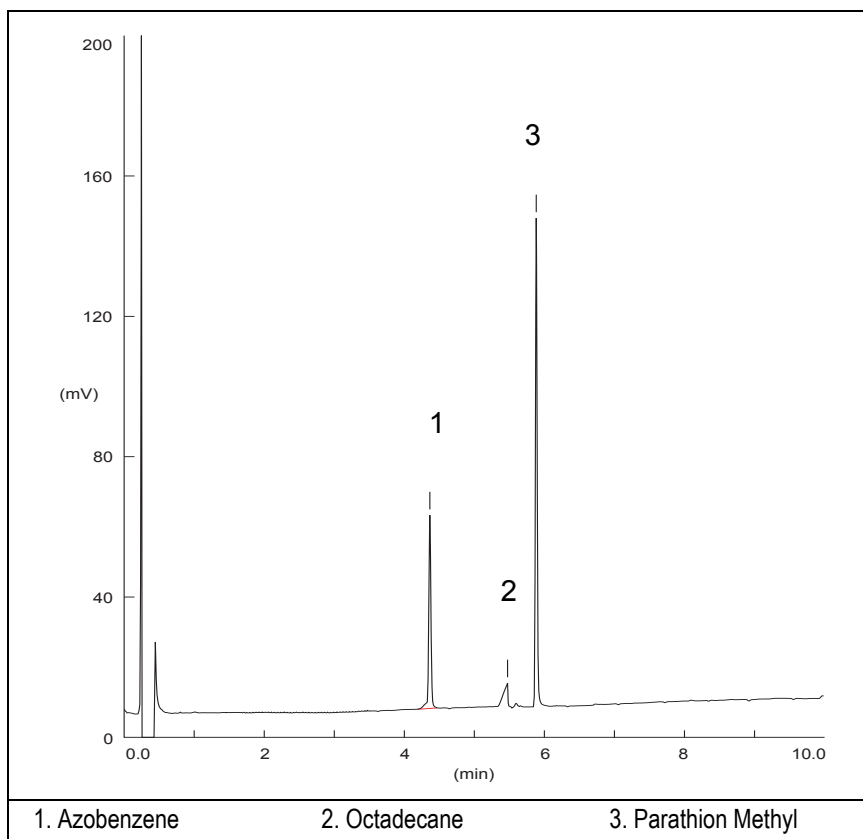


Figure 13-1. NPD-On-Column Injection

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

| Acceptance Values | CHROM-CARD | | |
|-------------------|---|------------------------|--------------------------|
| | Baseline Parameters | Analog (1V Full Scale) | Digital (10V Full Scale) |
| | Noise (μV) | < 40 | < 400 |
| | Wander (μV) | < 100 | < 1 000 |
| | Drift ($\mu\text{V/h}$) | < 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) | |
|---|--|
|  | |

| Acceptance Values | CHROMQUEST | |
|-------------------|---|------------|
| | Baseline Parameters (1V Full Scale) | |
| | Noise (μV) | < 40 |
| | Wander (μV) | < 100 |
| | Drift ($\mu\text{V/h}$) | < 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 ($\mu\text{V/h}$) | < 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 |
|  | | |

Checkout Using NPD with PKD Injector

Chapter at a Glance...

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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 | 290 134 86 |
| | Graphite ferrule for 0.32 mm ID Column | 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: <i>Component</i> <i>Concentration</i> 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 = <i>Refer to Source Ignition</i> 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 “*Performing 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 |
| H ₂ delay time | | Off |
| H ₂ | 2.3 | 2.3 |
| Air | 60 | 60 |
| Mkup N ₂ | 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:

- $H_2 = 2.3 \text{ ml/min}$
 - $Air = 60 \text{ ml/min}$
 - $Mkup N_2 = 15 \text{ ml/min}$
- 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.

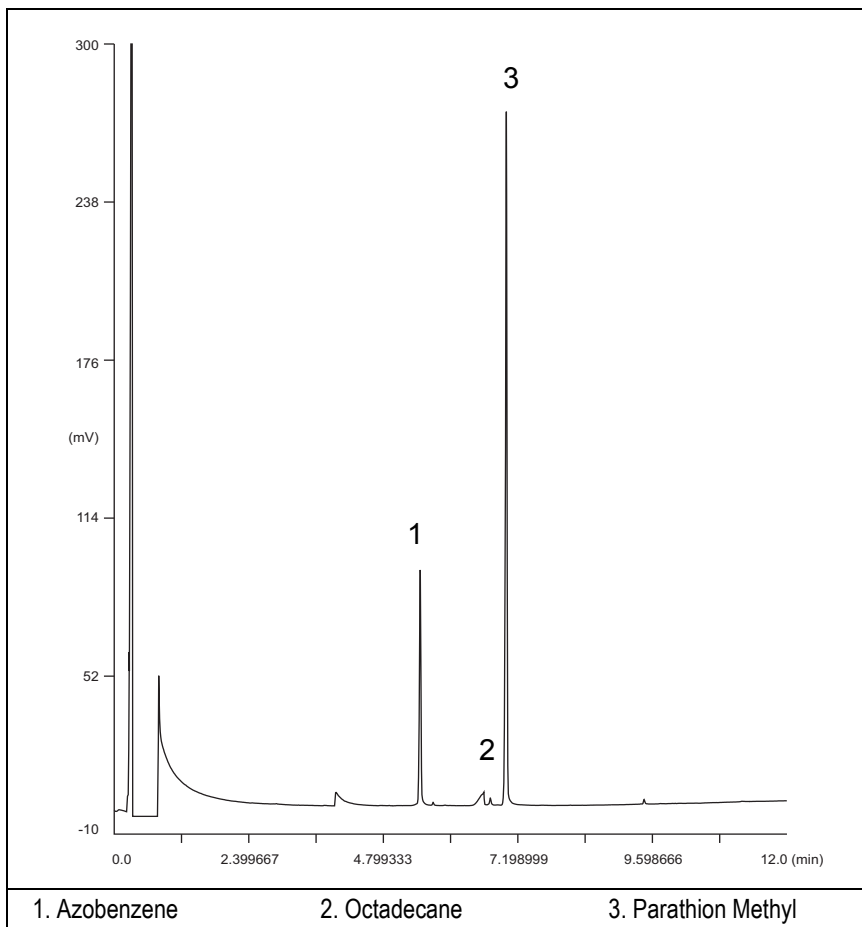


Figure 14-1. NPD-Packed Injection

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

| Acceptance Values | CHROM-CARD | | |
|-------------------|---|------------------------|--------------------------|
| | Baseline Parameters | Analog (1V Full Scale) | Digital (10V Full Scale) |
| | Noise (μV) | < 40 | < 400 |
| | Wander (μV) | < 100 | < 1 000 |
| | Drift ($\mu\text{V/h}$) | < 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 |
| |  | | |

| Computing-integrator (e.g. ChromJet) | |
|---|--|
|  | |

| Acceptance Values | CHROMQUEST | |
|-------------------|---|------------|
| | Baseline Parameters (1V Full Scale) | |
| | Noise (μV) | < 40 |
| | Wander (μV) | < 100 |
| | Drift ($\mu\text{V/h}$) | < 300 |
| | Analytical Results | |
| | Azobenzene Signal-to-noise ratio | > 600 |
| | Parathion Methyl Signal-to-noise ratio | > 1700 |
| | Octadecane Signal-to-noise ratio | Negligible |
| |  | |

| ATLAS | |
|---|------------|
| Baseline Parameters (10V Full Scale) | |
| Noise (μV) | < 400 |
| Wander (μV) | < 1 000 |
| Drift ($\mu\text{V/h}$) | < 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 |
|  | | |

Checkout Using NPD with PPKD Injector

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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 | 290 134 86 |
| | Graphite ferrule for 0.32 mm ID Column | 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: <i>Component</i> <i>Concentration</i> 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 = <i>Refer to Source Ignition</i> 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 “*Performing 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 |
| H ₂ delay time | | Off |
| H ₂ | 2.3 | 2.3 |
| Air | 60 | 60 |
| Mkup N ₂ | 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:

- $H_2 = 2.3 \text{ ml/min}$
 - $Air = 60 \text{ ml/min}$
 - $Mkup N_2 = 15 \text{ ml/min}$
- 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.

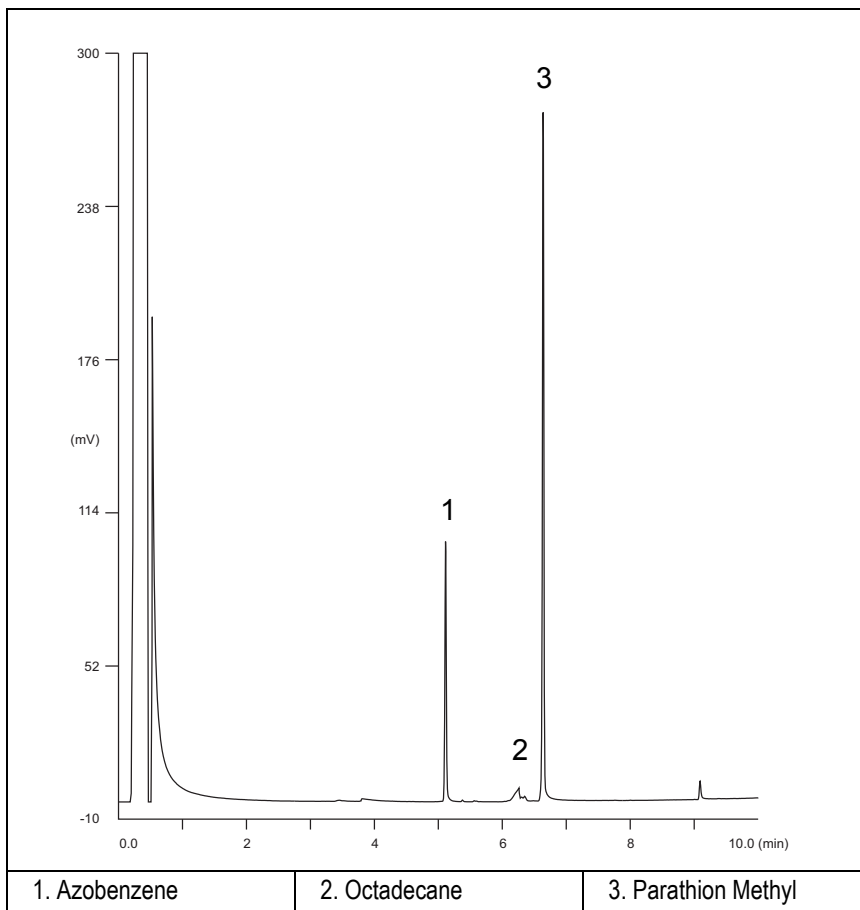


Figure 15-1. NPD-PPKD Injection

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 ($\mu\text{V/h}$) | < 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 ($\mu\text{V/h}$) | < 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 ($\mu\text{V/h}$) | < 3 000 |
| Analytical Results | |
| Azobenzene Signal-to-noise ratio | > 600 |
| Parathion Methyl Signal-to-noise ratio | > 1 700 |
| Octadecane Signal-to-noise ratio | Negligible |
|  | |

| 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 |
|  | |

Checkout Using NPD with PTV Injector

Chapter at a Glance...

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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: <i>Component</i> <i>Concentration</i> 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 = <i>Refer to Source Ignition</i> 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 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 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:
- 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)
 - Increase the Base Temp to 300°C and wait that the NPD cell reaches the correct set temperature.
 - Be sure that the backoff signal is between 0 and 0.5 pA.
 - 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.

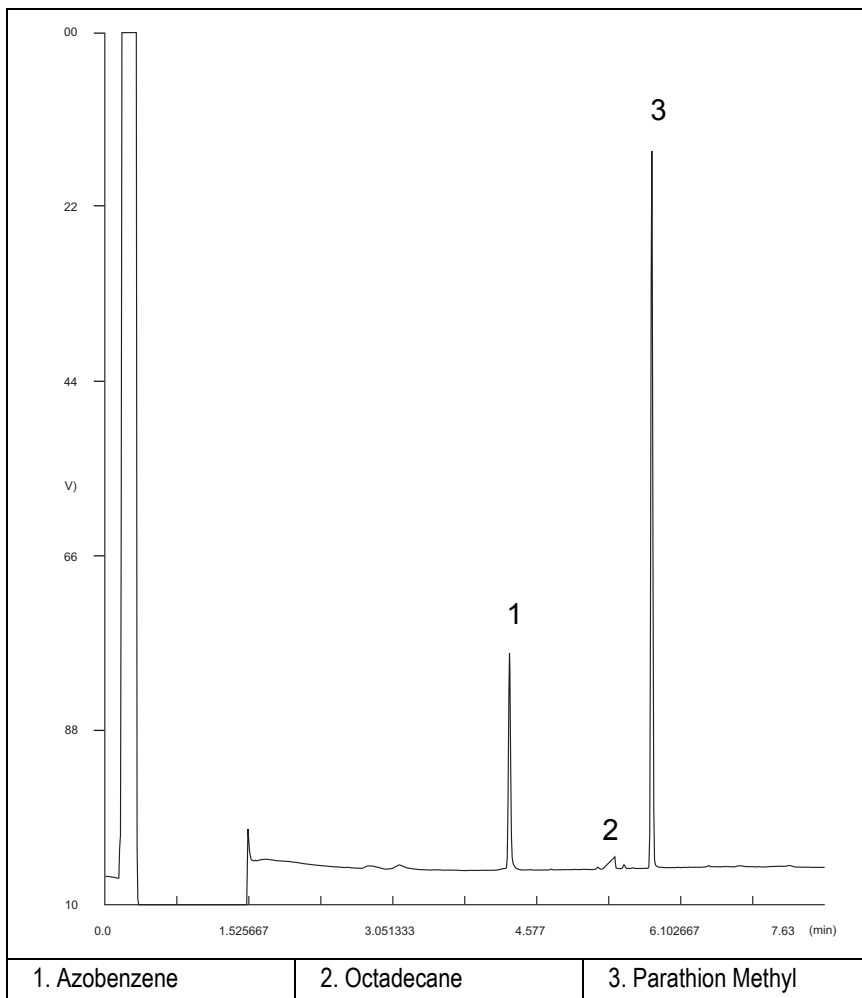


Figure 16-1. NPD-PTV Injection

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 ($\mu\text{V/h}$) | < 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) | |
|---|--|
|  | |

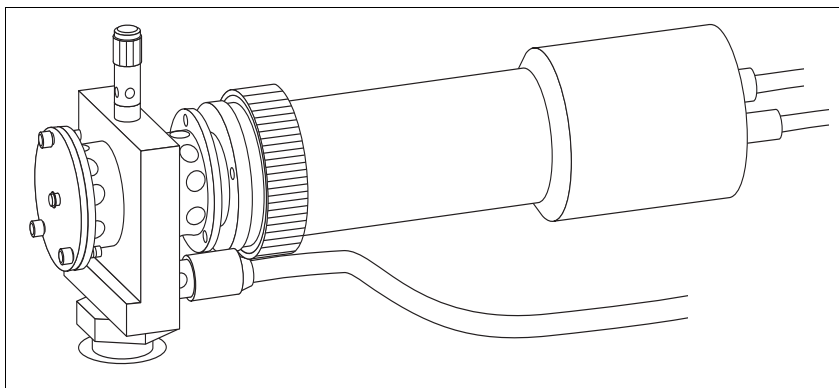
| Acceptance Values | CHROMQUEST | |
|-------------------|---|------------|
| | Baseline Parameters (1V Full Scale) | |
| | Noise (μV) | < 40 |
| | Wander (μV) | < 100 |
| | Drift ($\mu\text{V/h}$) | < 300 |
| | Analytical Results | |
| | Azobenzene Signal-to-noise ratio | > 550 |
| | Parathion Methyl 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 ($\mu\text{V/h}$) | < 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.

Chapter 17, *Checkout Using FPD with S/SL Injector*.

Chapter 18, *Checkout Using FPD with OC Injector*.

Chapter 19, *Checkout Using FPD with PKD Injector*.

Chapter 20, *Checkout Using FPD with PPKD Injector*.

Chapter 21, *Checkout Using FPD with PTV Injector*.

Checkout Using FPD with S/SL Injector

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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: <i>Component</i> <i>Concentration</i> 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 10¹.

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*.

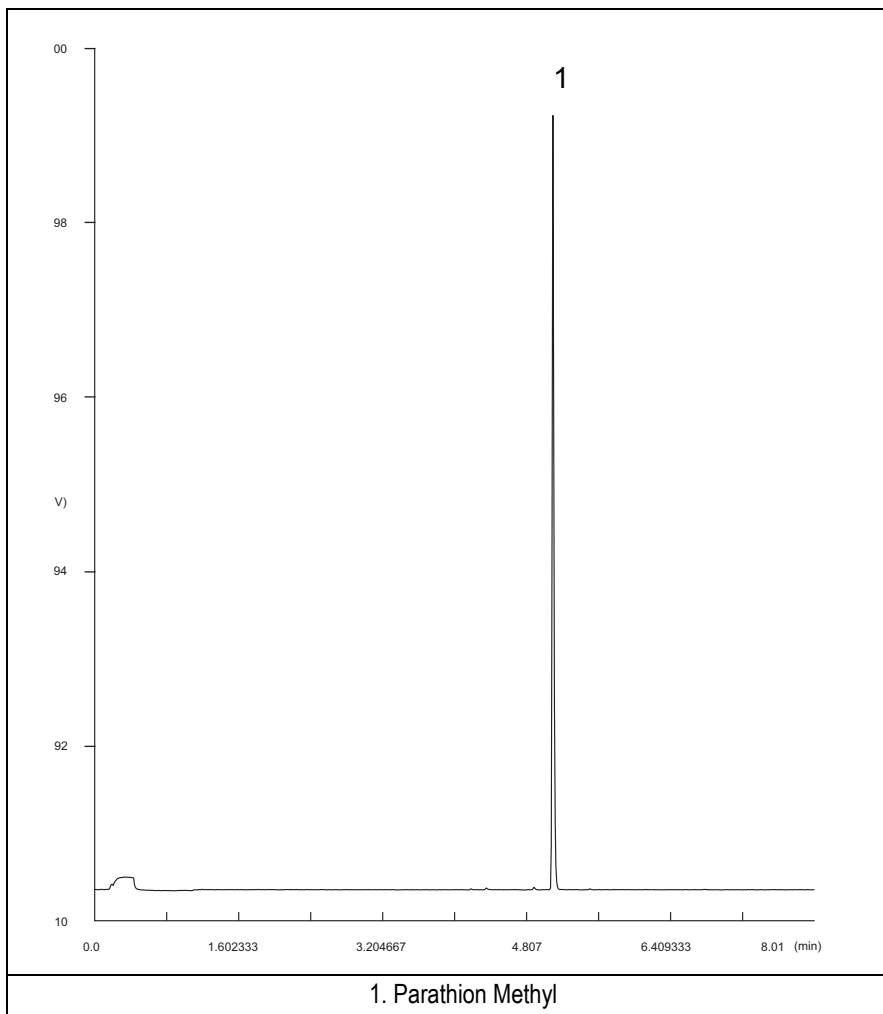


Figure 17-1. FPD-Splitless Injection with 526 nm filter

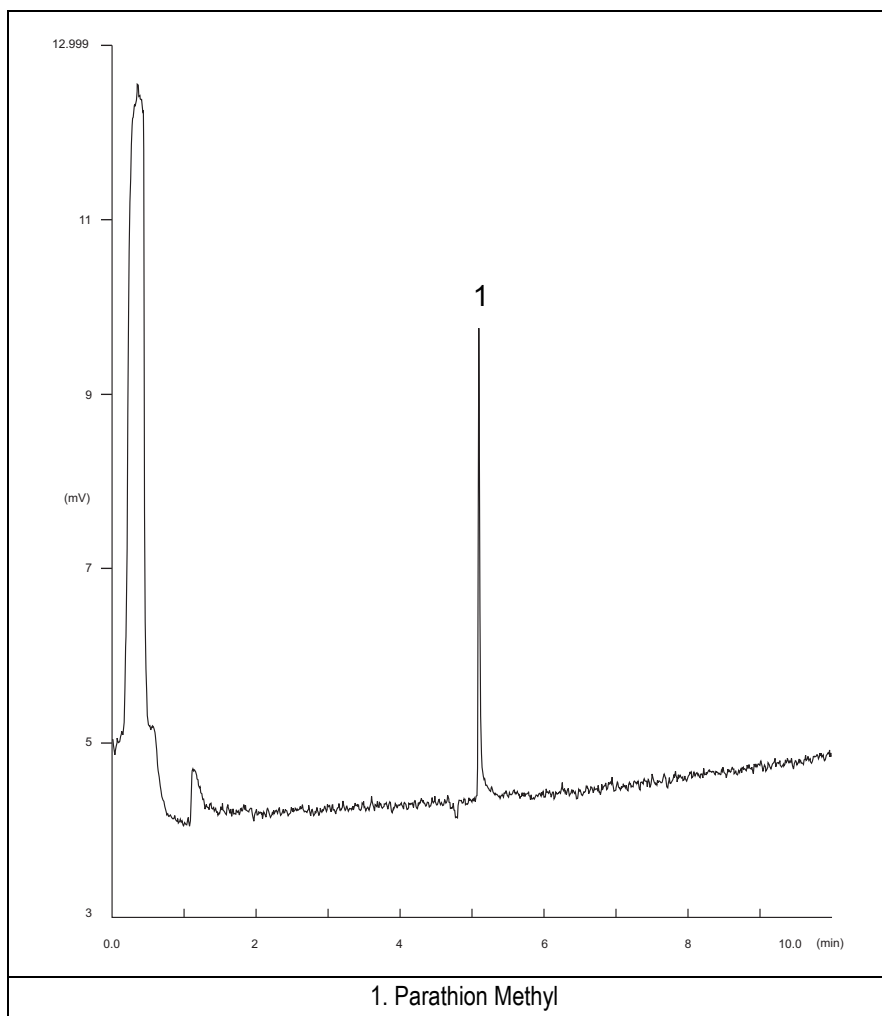


Figure 17-2. FPD-Splitless Injection with 394 nm

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 ($\mu\text{V/h}$) | < 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 ($\mu\text{V/h}$) | < 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 ($\mu\text{V/h}$) | < 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 <i>Peak Height (counts)/noise (counts)</i> . |

Checkout Using FPD with OC Injector

Chapter at a Glance...

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| FPD-OCI Checkout in On-Column Mode | 241 |
|--|-----|

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: <i>Component</i> <i>Concentration</i> 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-fit 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 10¹.

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 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 18.1* or *18.2*.

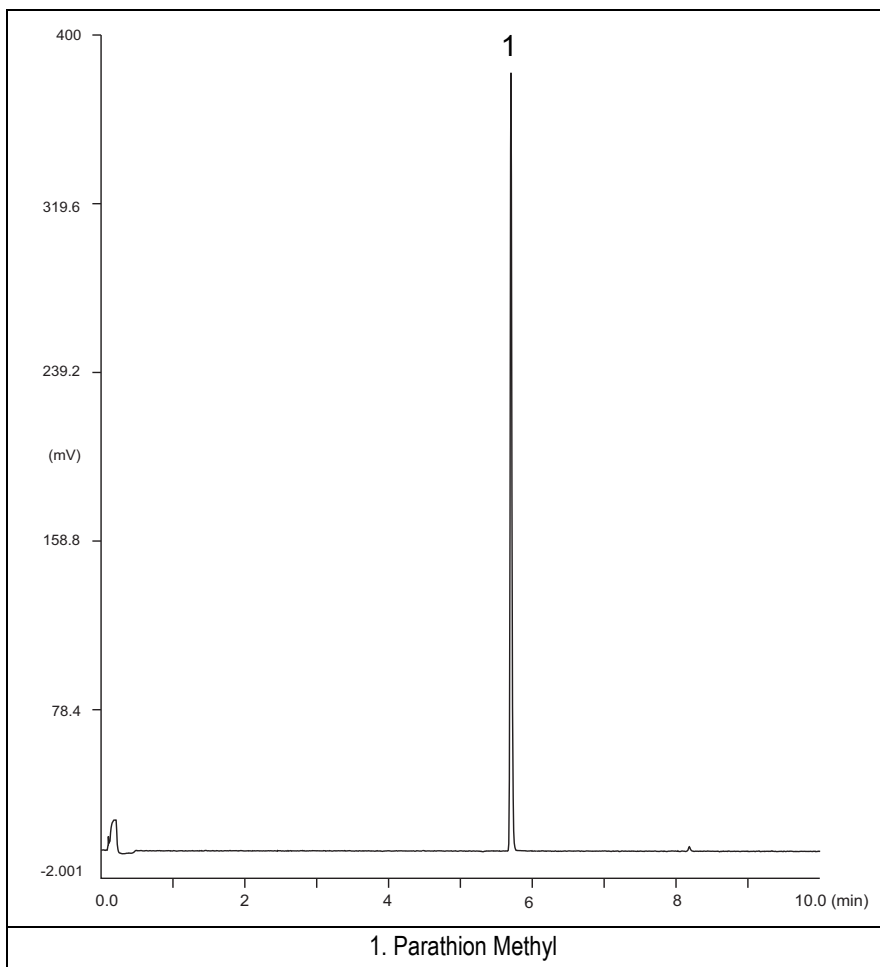


Figure 18-1. FPD-On-Column Injection with 526 nm filter

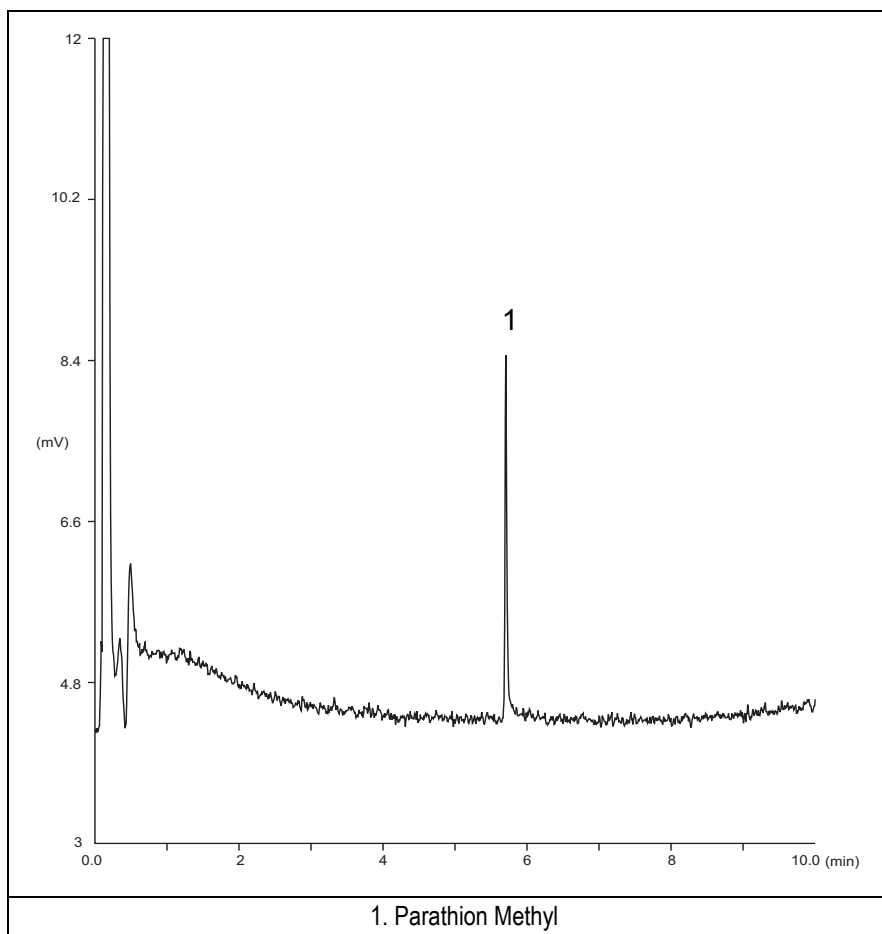


Figure 18-2. FPD-On-Column Injection with 394 nm

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 ($\mu\text{V/h}$) | < 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 ($\mu\text{V/h}$) | < 100 | < 100 |
| | Analytical Results | | |
|  | Parathion Methyl Signal to Noise Ratio | 394 nm (S) Filter | 526 nm (P) Filter |
| | | > 20 | > 1 000 |

| | ATLAS | | |
|---|--|-------------------|-------------------|
| | Baseline Parameters (10V Full Scale) | | |
| | | | 526 nm (P) Filter |
| | Noise (μ V) | < 500 | < 500 |
| | Wander (μ V) | < 1 000 | < 1 000 |
| | Drift (μ V/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 <i>Peak Height (counts)/noise (counts)</i> . |

Checkout Using FPD with PKD Injector

Chapter at a Glance...

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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: <i>Component</i> <i>Concentration</i> 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 10¹.

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 “*Performing 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.** 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. 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 |
| H ₂ | 90 | 90 |
| Air | 115 | 115 |
| Mkup N ₂ | 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*.

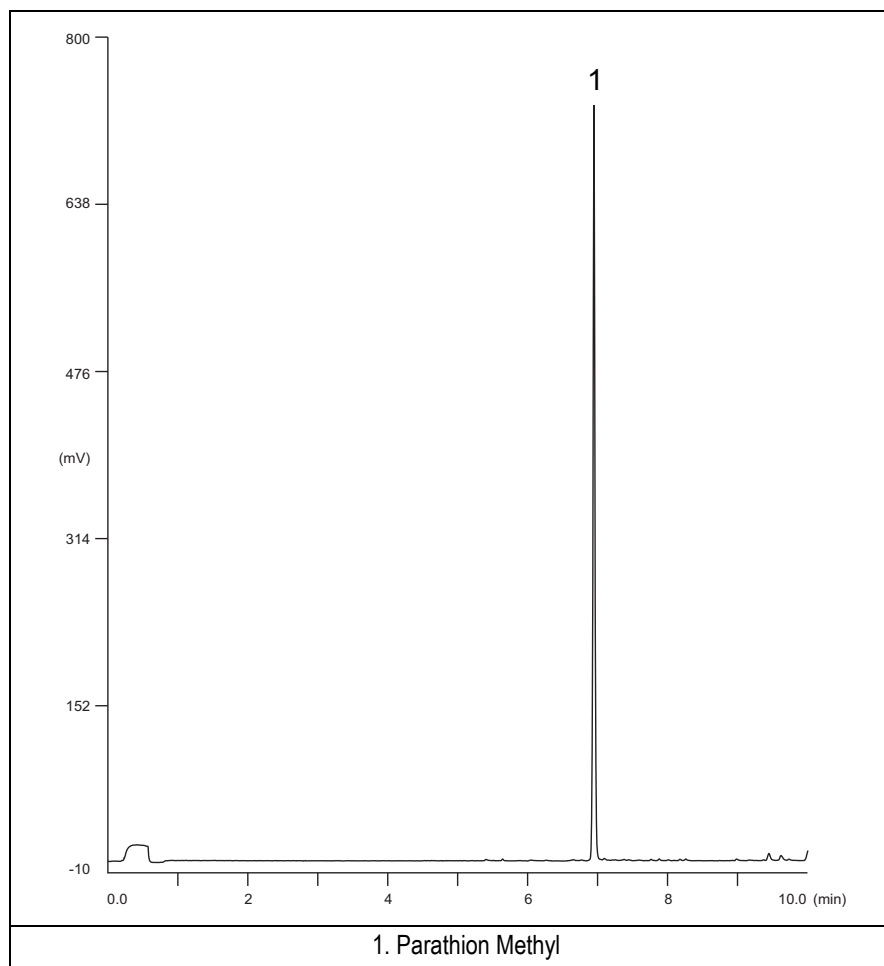


Figure 19-1. FPD-PKD Injection with 526 nm filter

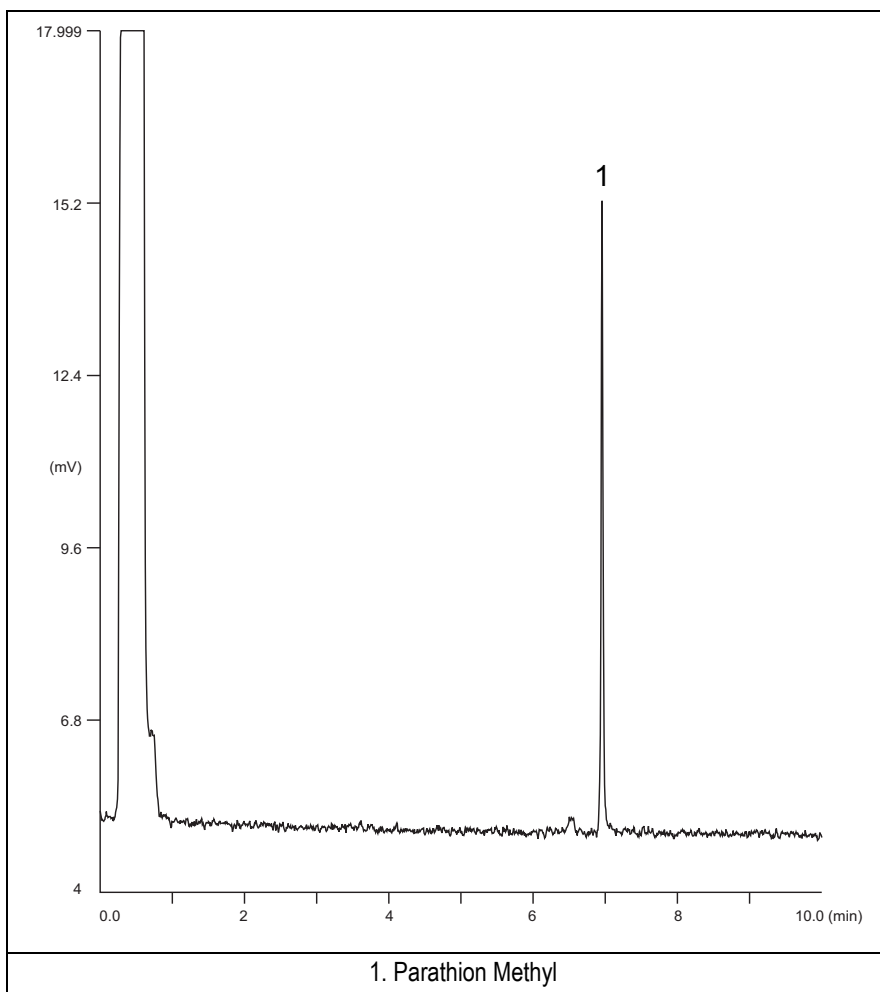


Figure 19-2. FPD-PKD Injection with 394 nm

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 ($\mu\text{V/h}$) | < 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 ($\mu\text{V/h}$) | < 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 ($\mu\text{V/h}$) | < 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 <i>Peak Height (counts)/noise (counts)</i> . |

Checkout Using FPD with PPKD Injector

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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 | 281 071 00 |
| | 394 nm for sulphur | 281 070 00 |
| Test Mixture | Three components in Iso-Octane: | 338 190 06 |
| | <i>Component</i> <i>Concentration</i> | |
| | Azobenzene 1 µg/ml | |
| | Octadecane 1000 µg/ml | |
| | Parathion methyl 1 µg/ml | |
| | | |
| 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 10¹.

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 “*Performing 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.** 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) < |

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.

- 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.

- Verify that High voltage mode is set to **NO**.
- 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*.

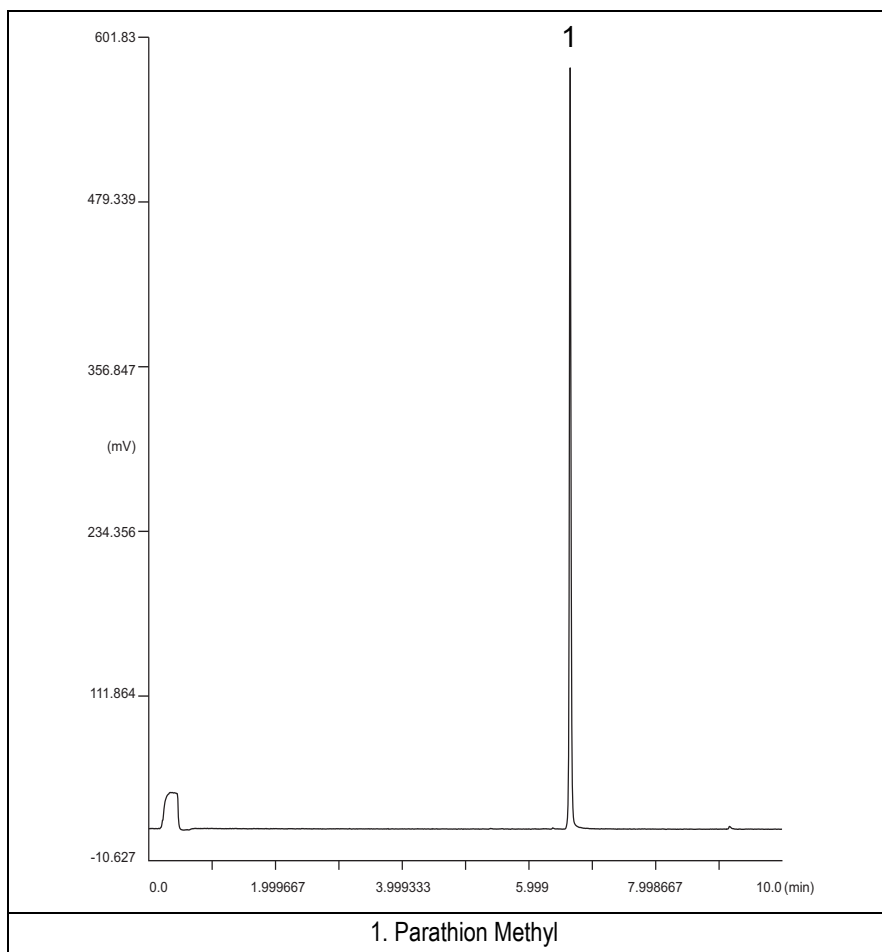


Figure 20-1. FPD-PPKD Injection with 526 nm filter

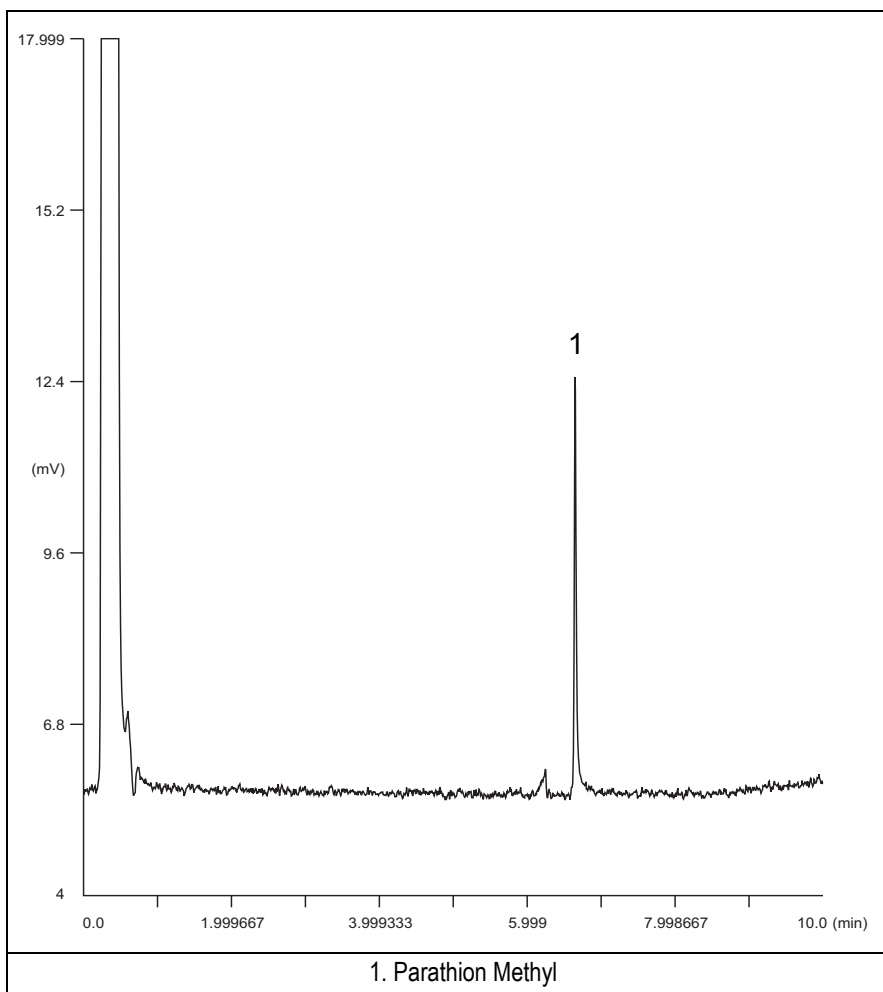


Figure 20-2. FPD-PPKD Injection with 394 nm

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 ($\mu\text{V/h}$) | < 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 ($\mu\text{V/h}$) | < 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 ($\mu\text{V/h}$) | < 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 <i>Peak Height (counts)/noise (counts)</i> . |

Checkout Using FPD with PTV Injector

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| FPD-PTV Checkout in PTV Splitless Mode | 281 |
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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: <i>Component</i> <i>Concentration</i> 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 10¹.

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**. Scroll 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 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*.

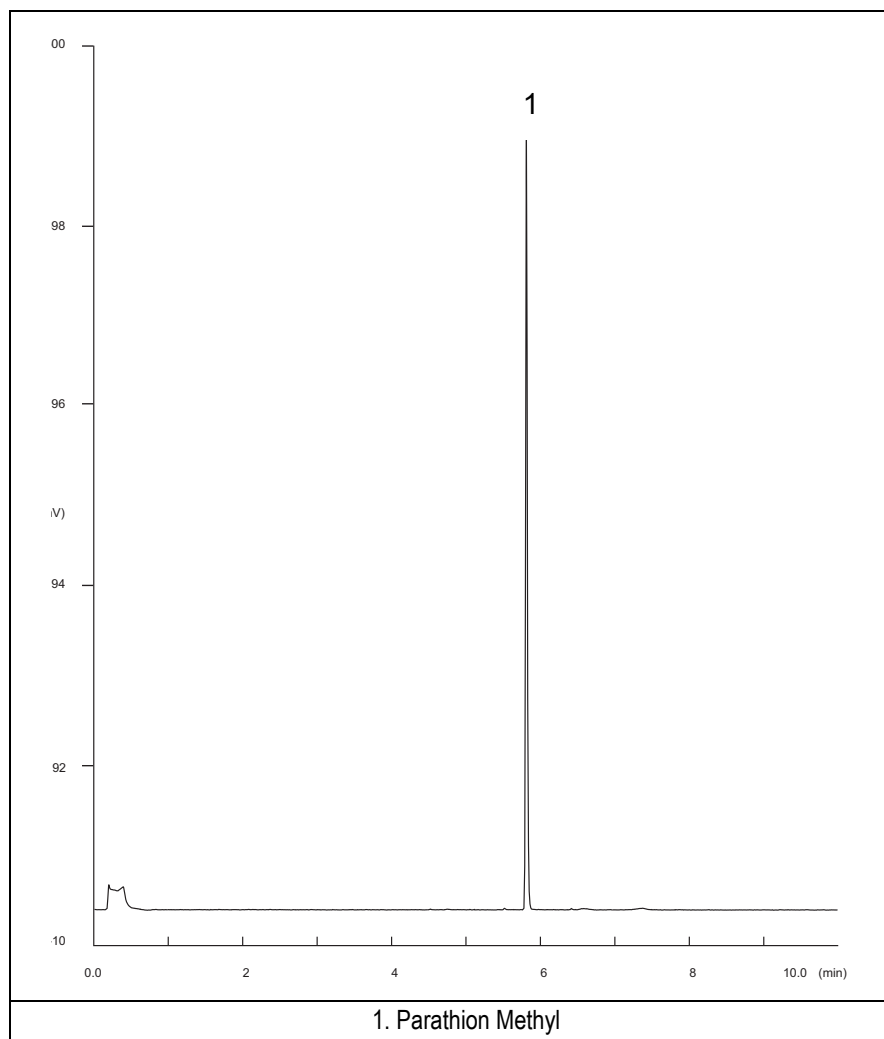


Figure 21-1. FPD-PTV Injection with 526 nm filter

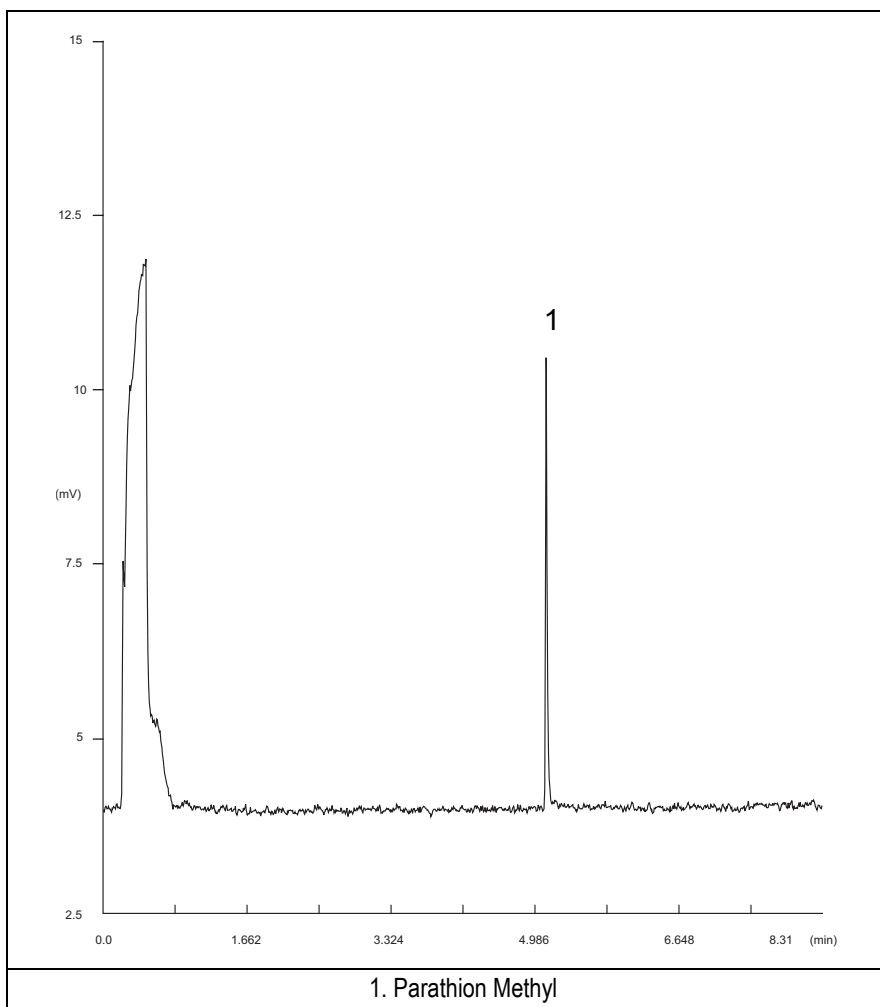


Figure 21-2. FPD-PTV Injection with 394 nm filter

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 ($\mu\text{V/h}$) | < 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 ($\mu\text{V/h}$) | < 100 | < 100 |
| | Analytical Results | | |
|  | Parathion Methyl Signal to Noise Ratio | 394 nm (S) Filter | 526 nm (P) Filter |
| | | > 20 | > 1 000 |

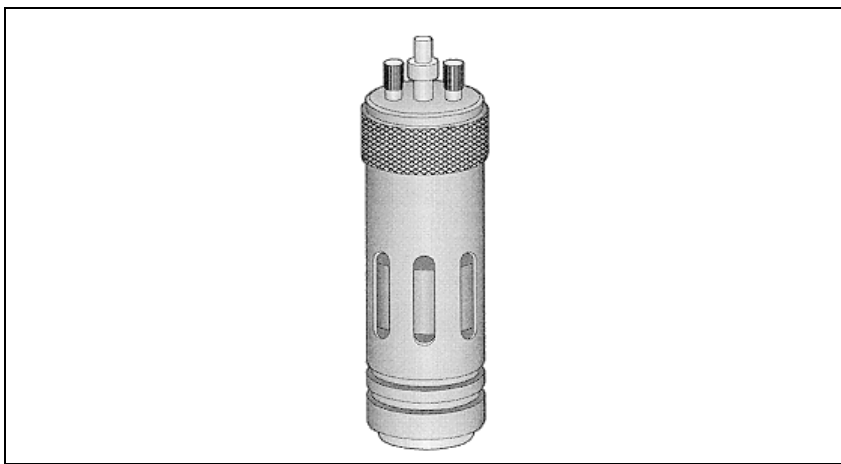
| | ATLAS | | |
|---|--|-------------------|-------------------|
| | Baseline Parameters (10V Full Scale) | | |
| | | | 526 nm (P) Filter |
| | Noise (μV) | < 500 | < 500 |
| | Wander (μV) | < 1 000 | < 1 000 |
| | Drift ($\mu\text{V/h}$) | < 1 000 | < 1 000 |
| | Analytical Results | | |
| | Parathion 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 <i>Peak Height (counts)/noise (counts)</i> . |

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.

Chapter 22, *Checkout Using PID with S/SL Injector*.

Chapter 23, *Checkout Using PID with OC Injector*.

Chapter 24, *Checkout Using PID with PTV Injector*.

Checkout Using PID with S/SL Injector

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| PID-S/SL Checkout in Splitless Mode | 298 |
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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: <i>Component</i> <i>Concentration</i> 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.** 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. 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*.

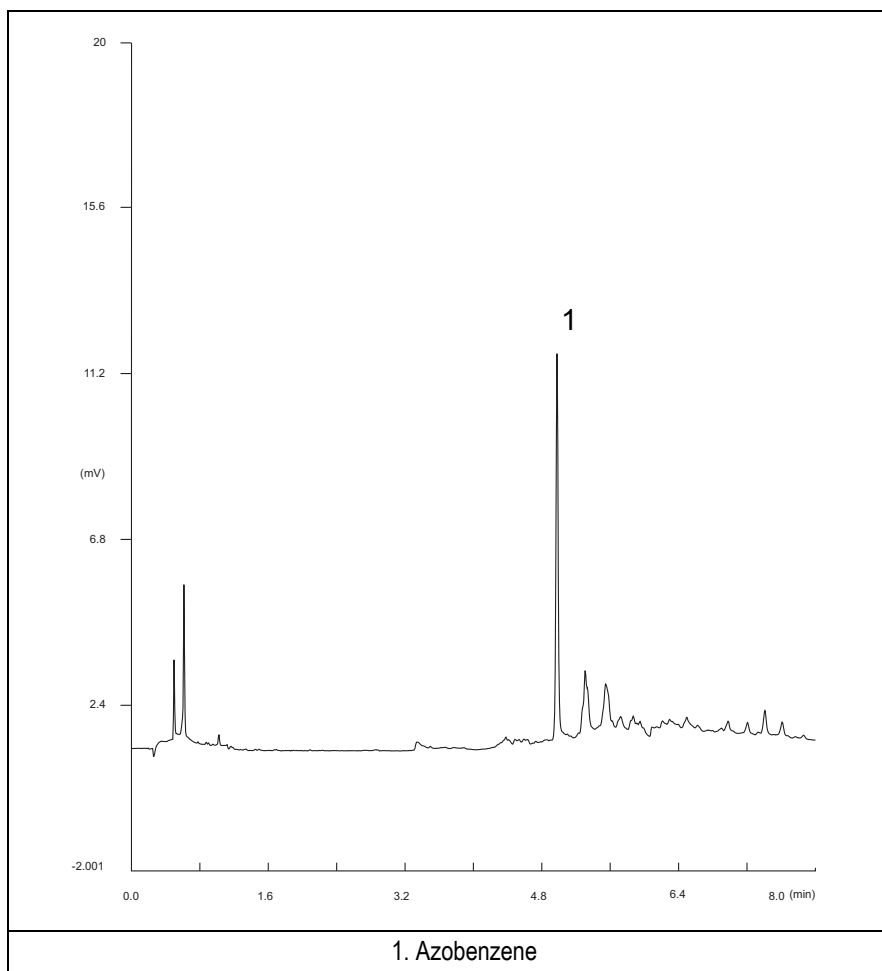


Figure 22-1. PID-Splitless Injection with 8.4 eV UV Lamp

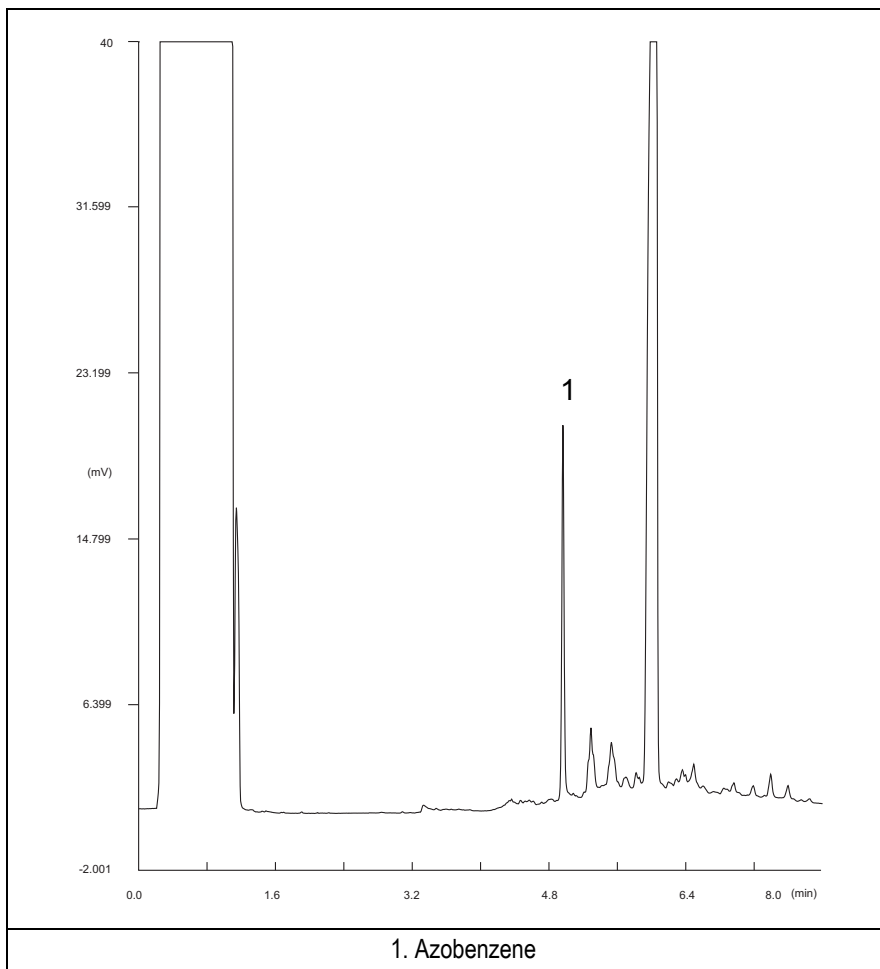
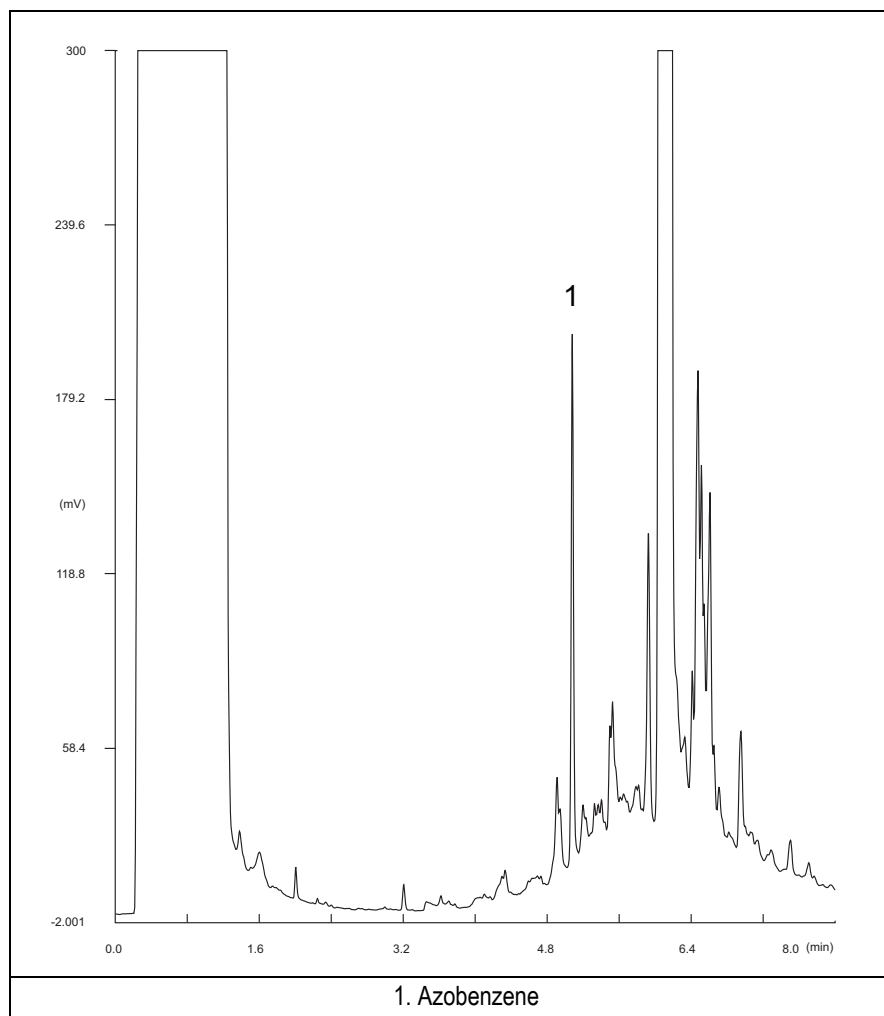


Figure 22-2. PID-Splitless Injection with 9.6 eV UV Lamp

**Figure 22-3.** PID-Splitless Injection with 10.6 eV UV Lamp

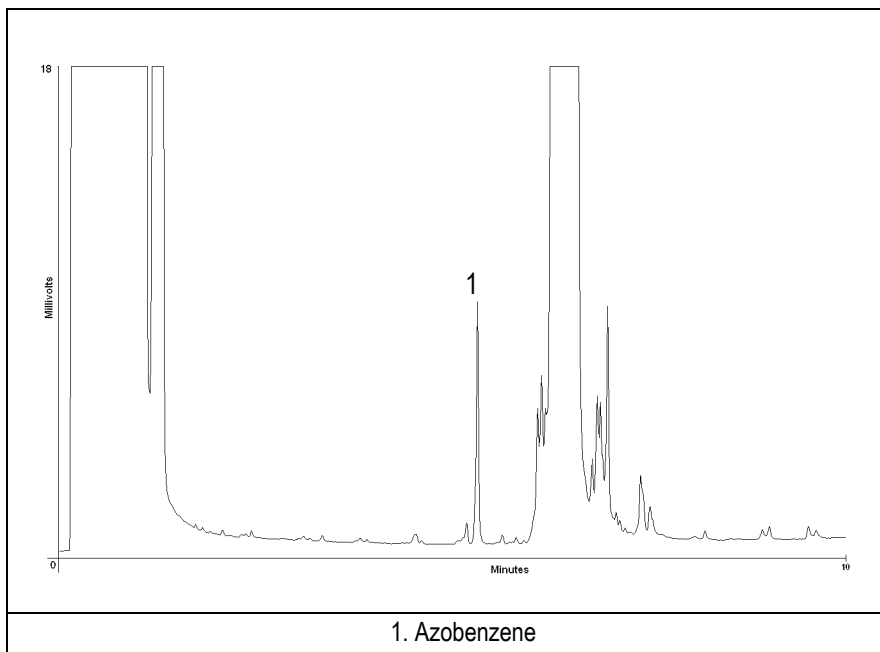


Figure 22-4. PID-Splitless Injection with 11.8 eV UV Lamp

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 ($\mu\text{V/h}$) | < 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 ($\mu\text{V/h}$) | < 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 |
| | | | | | |
| | | | | | |
| | | | | | |



| | 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 ($\mu\text{V/h}$) | < 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 |
|  | | | | | |

| Acceptance Values | 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 |
|  | | | | | |

Checkout Using PID with OC Injector

Chapter at a Glance...

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| Parts Referenced | 308 |
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| Recommended Initial Operations | 310 |

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| PID-OCI Checkout in On-Column Mode..... | 311 |
|---|-----|

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: <i>Component</i> <i>Concentration</i> 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-fit 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.** 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. 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.*

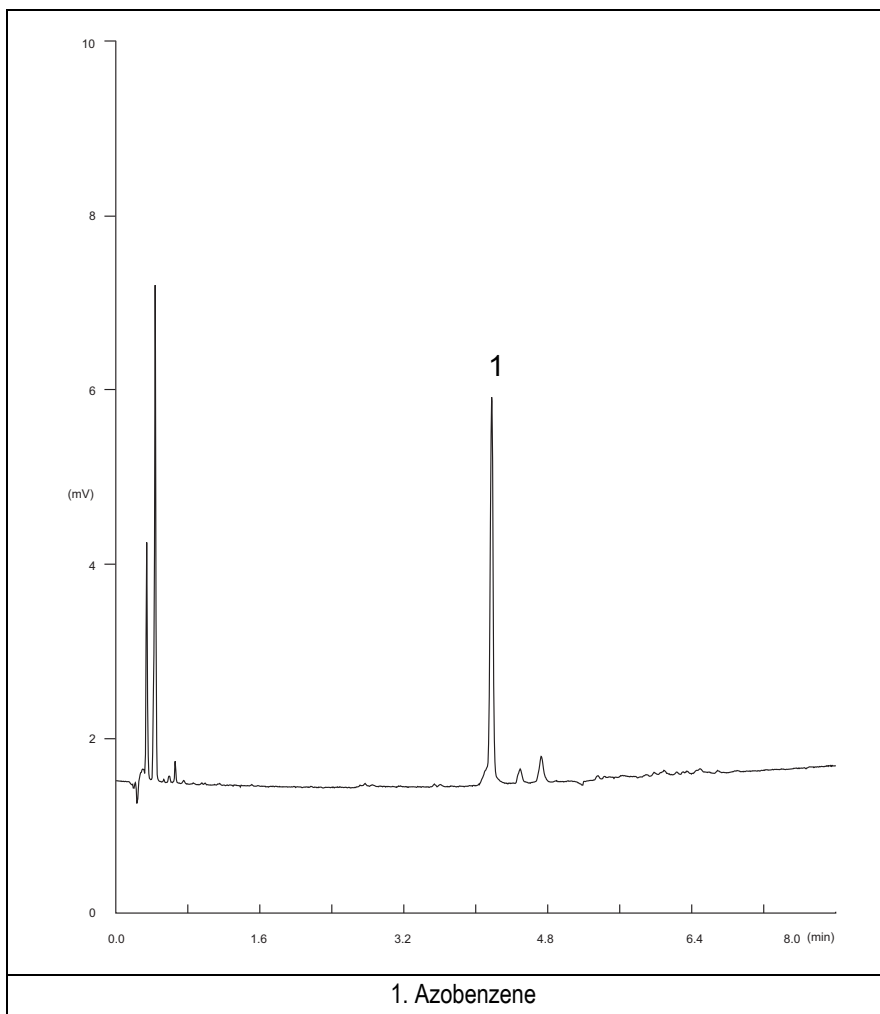


Figure 23-1. PID-On-Column Injection with 8.4 eV UV Lamp

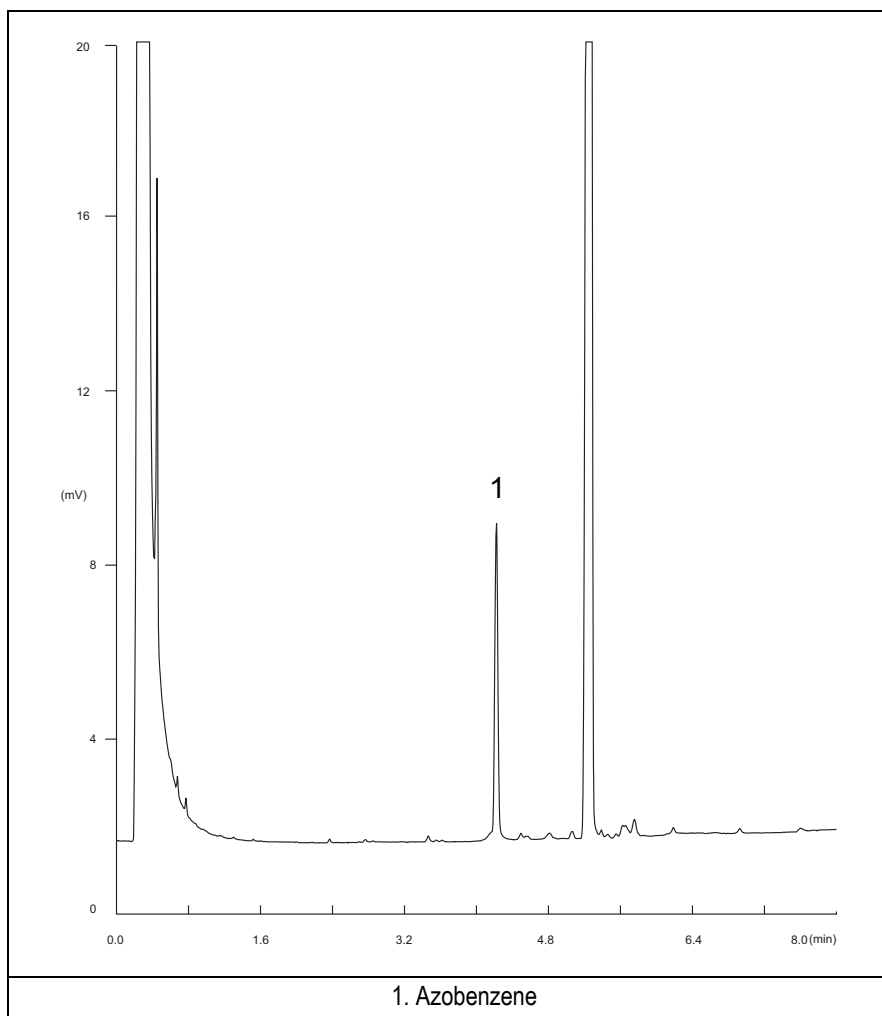


Figure 23-2. PID-On-Column Injection with 9.6 eV UV Lamp

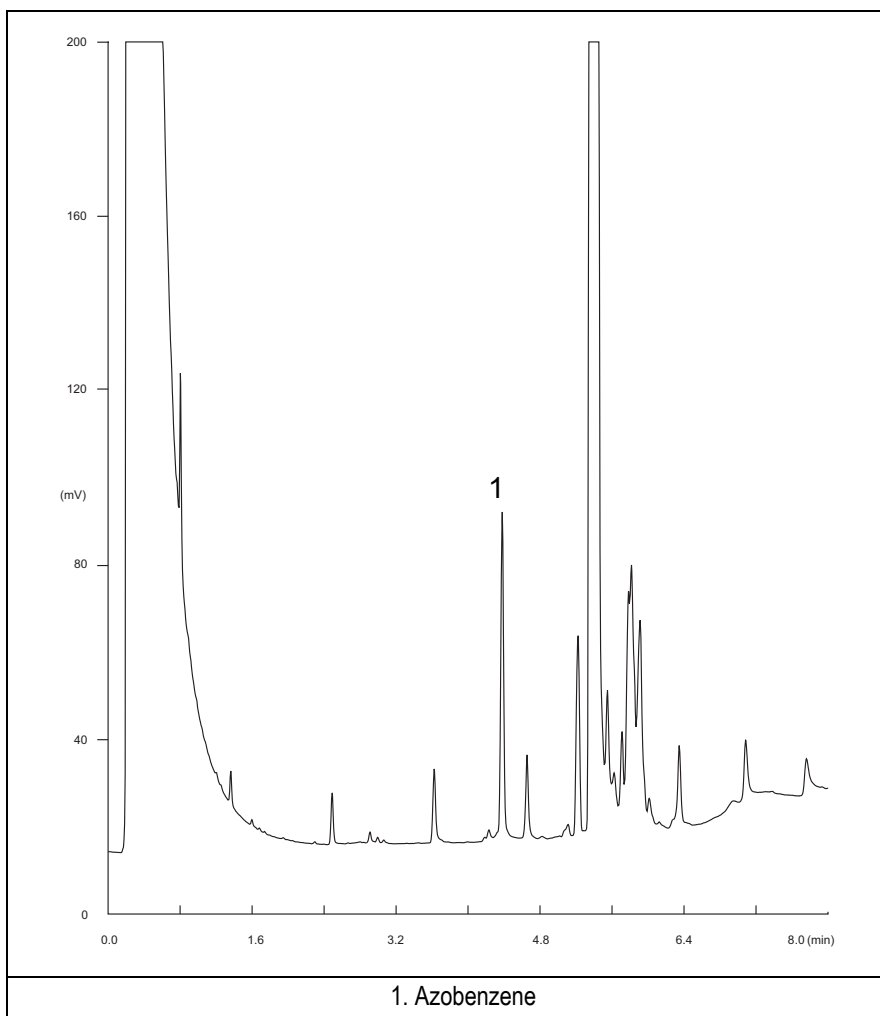


Figure 23-3. PID-On-Column Injection with 10.6 eV UV Lamp

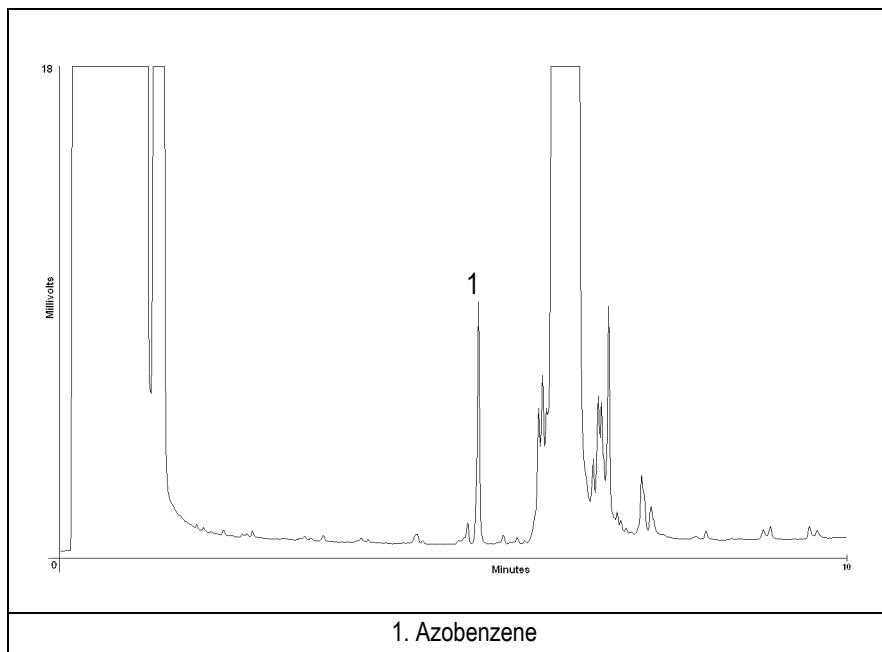


Figure 23-4. PID-On-Column Injection with 11.8 eV UV Lamp

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 |
| | | | | | |
| | | | | | |



| | 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 ($\mu\text{V/h}$) | < 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 |
|  | | | | | |

| Acceptance Values | 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 |
|  | | | | | |

Checkout Using PID with PTV Injector

Chapter at a Glance...

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| Recommended Initial Operations | 325 |

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| PID-PTV Checkout in PTV Splitless Mode | 326 |
|--|-----|

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: <i>Component</i> <i>Concentration</i> 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`. Scroll 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.

**NOTE****Baseline Acquisition and Analysis**

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*.

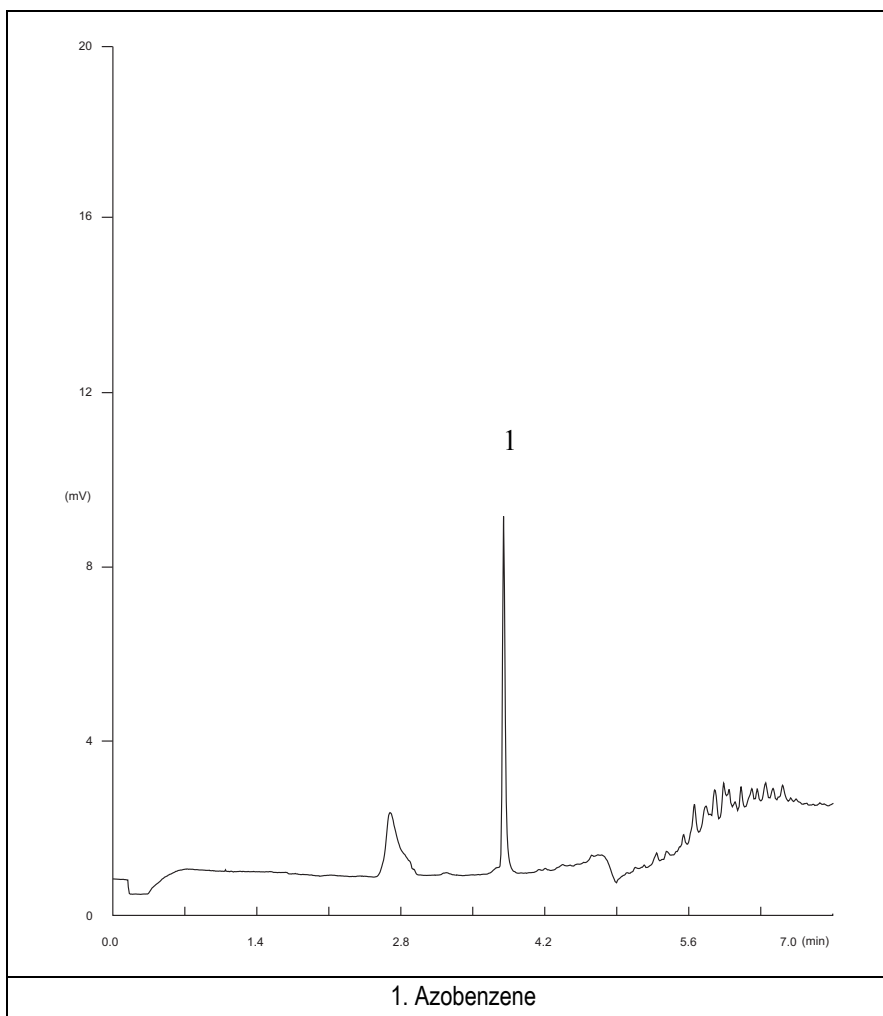


Figure 24-1. PID-PTV Injection with 8.4 eV UV Lamp

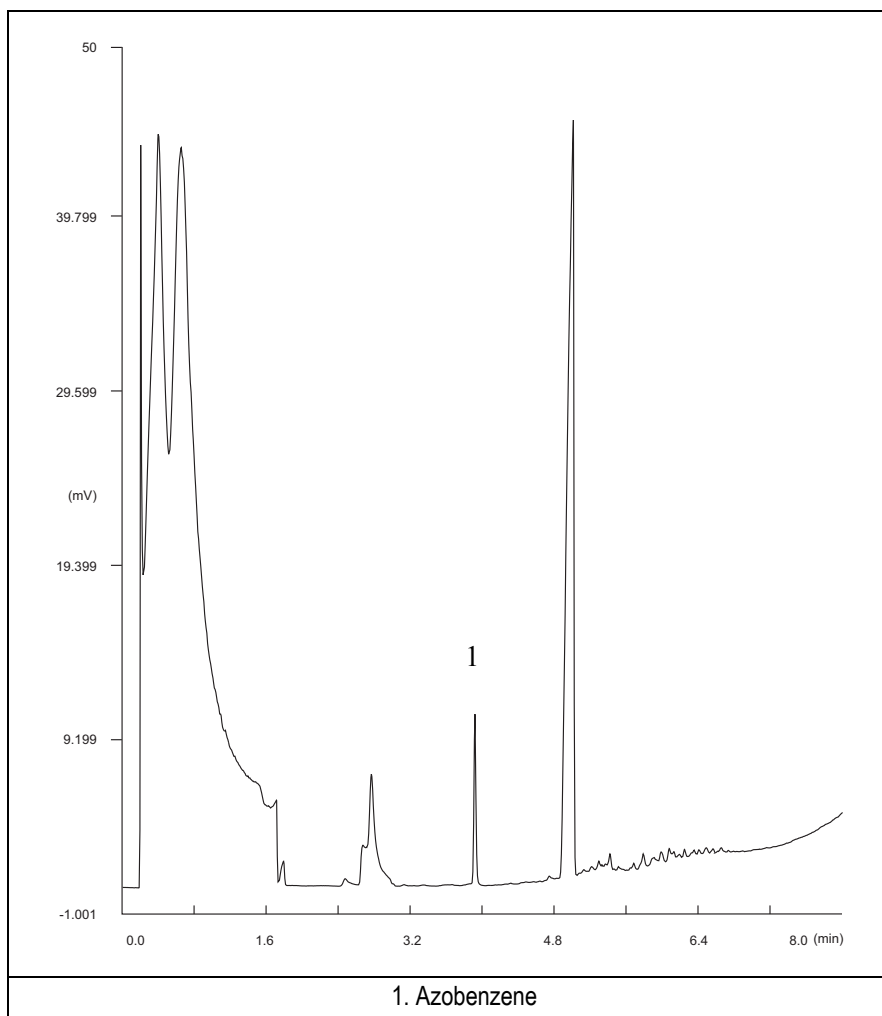


Figure 24-2. PID-PTV Injection with 9.6 eV UV Lamp

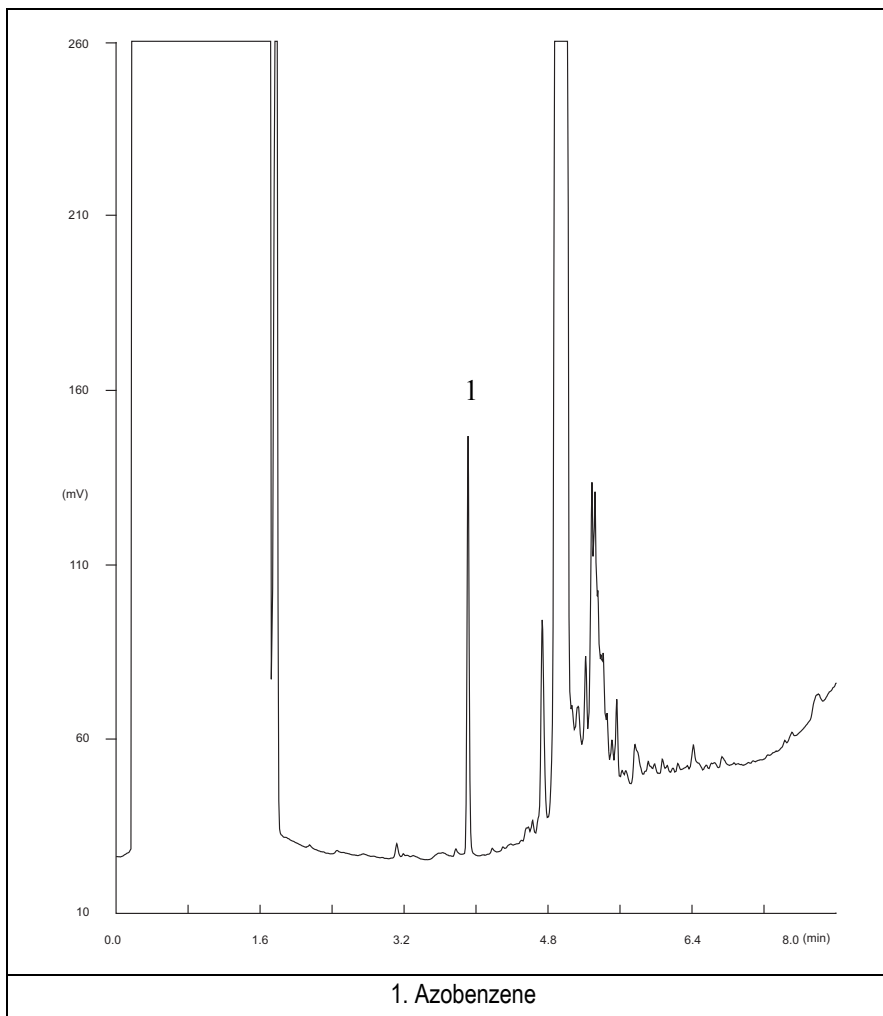


Figure 24-3. PID-PTV Injection with 10.6 eV UV Lamp

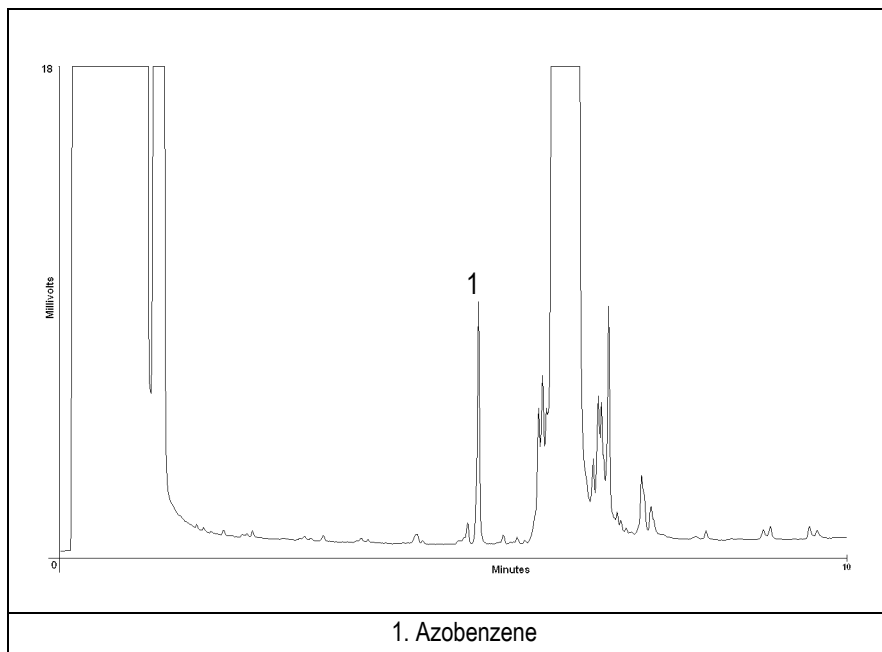


Figure 24-4. PID-PTV Injection with 11.8 eV UV Lamp

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 ($\mu\text{V/h}$) | < 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 ($\mu\text{V/h}$) | < 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 |
| | | | | | |
| | | | | | |

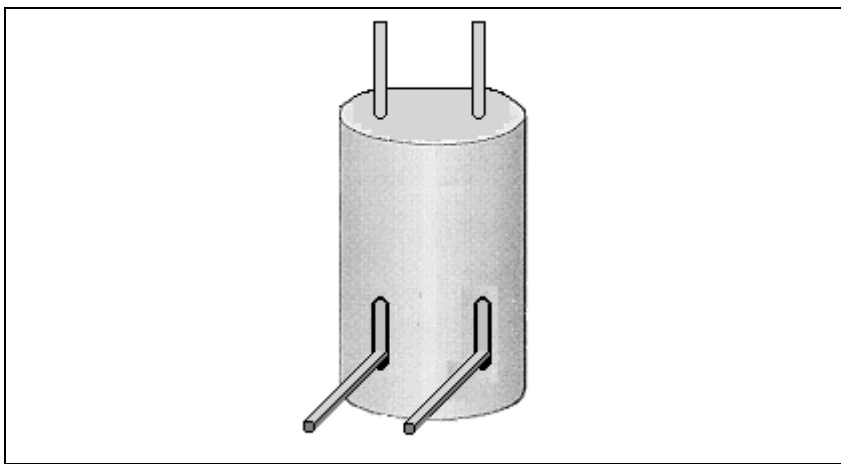


| | ATLAS | | | | |
|---|--|----------|-----------|-------------|----------|
| | Baseline Parameters (10V Full Scale) | | | | |
| | Lamp (eV) | 8.4 | 9.6 | 10.6 | 11.8 |
| | Noise (μV) | < 1 000 | < 500 | < 1 500 | < 1 500 |
| | Wander (μV) | < 1 500 | < 1 000 | < 1 000 | < 1 000 |
| | Drift ($\mu\text{V/h}$) | < 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 |
|  | | | | | |

| Acceptance Values | 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.

Chapter 25, *Checkout Using TCD with S/SL Injector*.

Chapter 26, *Checkout Using TCD with PKD Injector*.

Chapter 27, *Checkout Using TCD with PPKD Injector*.

Checkout Using TCD with S/SL Injector

Chapter at a Glance...

| | |
|--|-----|
| SOP Number: P0326/11/E - 01 September 2009 | 340 |
| Scope | 340 |
| Parts Referenced | 340 |
| Analytical Conditions Required for Splitless Injection | 341 |
| Recommended Initial Operations | 342 |

Operating Procedures

| | |
|---|-----|
| TCD-S/SL Checkout in Splitless Mode | 343 |
|---|-----|

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: <i>Component</i> <i>Concentration</i> 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.** 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. 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*.

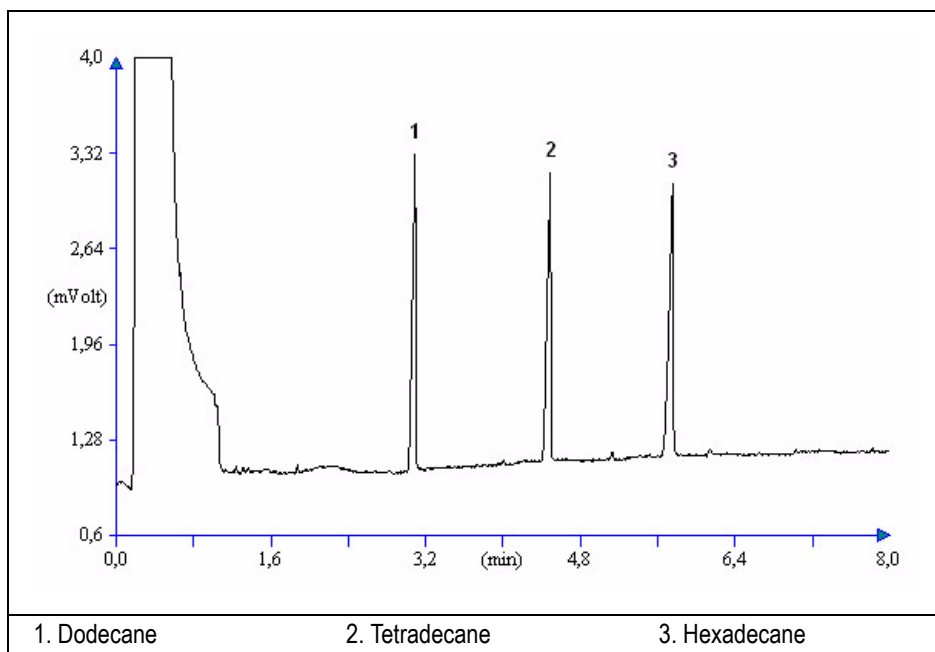





Figure 25-1. TCD-Splitless Injection


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

| Acceptance Values | CHROM-CARD | | |
|---|---------------------------|---|---|
| | Baseline Parameters | Analog (1V Full Scale) | Digital (10V Full Scale) |
| | Noise (μV) | < 30 | < 300 |
| | Wander (μV) | < 140 | < 1 400 |
| | Drift ($\mu\text{V/h}$) | < 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) | |
|---|--|
|  | |

| Acceptance Values | CHROMQUEST | |
|---|---|------------------------------|
| | Baseline Parameters (1V Full Scale) | |
| | Noise (μV) | < 30 |
| | Wander (μV) | < 140 |
| | Drift ($\mu\text{V/h}$) | < 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 ($\mu\text{V/h}$) | < 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. |

Checkout Using TCD with PKD Injector

Chapter at a Glance...

| | |
|--|-----|
| SOP Number: P0328/11/E - 01 September 2009 | 350 |
| Scope | 350 |
| Parts Referenced | 350 |
| Analytical Conditions Required for Packed Injector | 352 |
| Recommended Initial Operations | 353 |

Operating Procedures

| | |
|------------------------|-----|
| TCD-PKD Checkout | 354 |
|------------------------|-----|

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 | 290 134 86 |
| | Graphite ferrule for 0.32 mm ID Column | 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: | 338 190 16 |
| | <i>Component</i> <i>Concentration</i> | |
| | Dodecane 200 µg/ml | |
| | Tetradecane 200 µg/ml | |
| | Hexadecane 200 µg/ml | |
| 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 “*Performing 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*

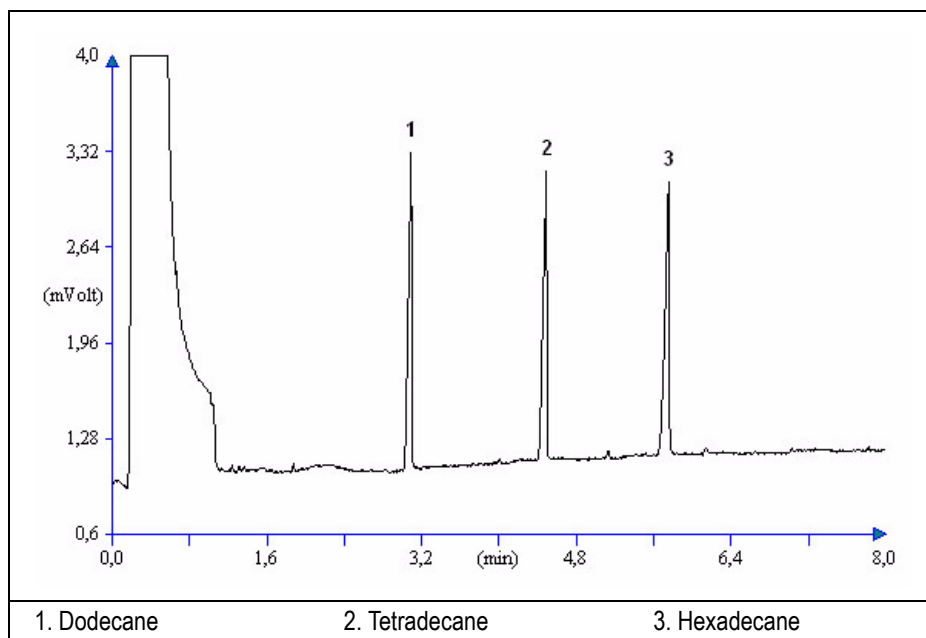





Figure 26-1. TCD-Packed Injection


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 ($\mu\text{V/h}$) | < 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 ($\mu\text{V/h}$) | < 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 ($\mu\text{V/h}$) | < 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. |

Checkout Using TCD with PPKD Injector

Chapter at a Glance...

| | |
|---|-----|
| SOP Number: P0329/11/E - 01 September 2009 | 362 |
| Scope | 362 |
| Parts Referenced..... | 362 |
| Analytical Conditions Required for Purged Packed Injector | 364 |
| Recommended Initial Operations | 365 |

Operating Procedures

| | |
|-------------------------|-----|
| TCD-PPKD Checkout | 366 |
|-------------------------|-----|

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 | 290 134 86 |
| | Graphite ferrule for 0.32 mm ID Column | 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: | 338 190 16 |
| | <i>Component</i> <i>Concentration</i> | |
| | Dodecane 200 µg/ml | |
| | Tetradecane 200 µg/ml | |
| | Hexadecane 200 µg/ml | |
| 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.** 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. 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*

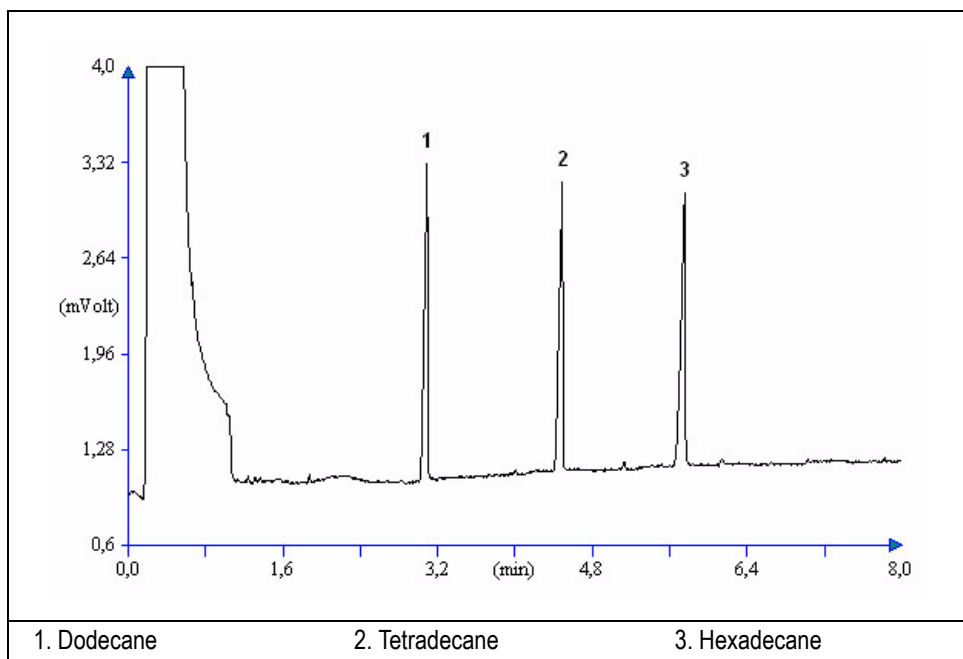





Figure 27-1. TCD-PPKD Injection


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

| Acceptance Values | CHROM-CARD | | |
|---|---------------------------|---|---|
| | Baseline Parameters | Analog (1V Full Scale) | Digital (10V Full Scale) |
| | Noise (μV) | < 30 | < 300 |
| | Wander (μV) | < 140 | < 1 400 |
| | Drift ($\mu\text{V/h}$) | < 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 ($\mu\text{V/h}$) | < 200 |
| | Analytical Results (1V Full Scale) - Area Counts (0.01 μVs) | |
| | Components | > 300 000 for each component |
|  | | |

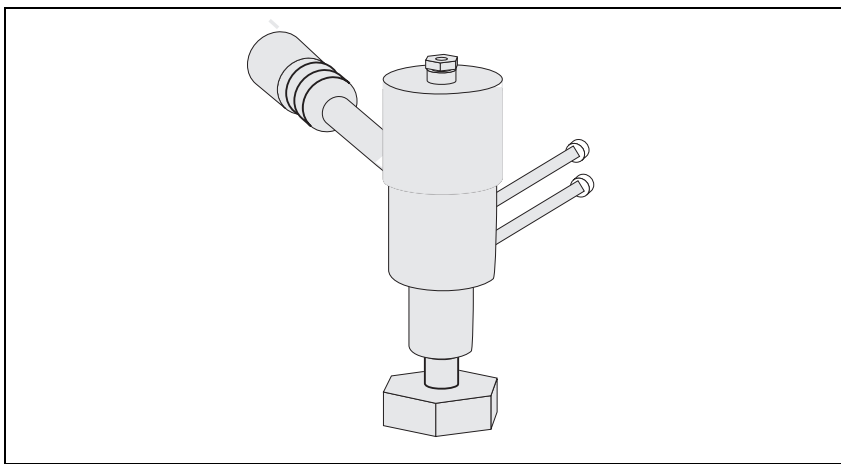
| ATLAS | |
|---|-----------------------------|
| Baseline Parameters (10V Full Scale) | |
| Noise (μV) | < 300 |
| Wander (μV) | < 1 400 |
| Drift ($\mu\text{V/h}$) | < 2 000 |
| Analytical Results (10V Full Scale) - Area Counts (μVs) | |
| Components | > 30 000 for each component |
|  | |

| 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.

Chapter 28, *Checkout Using PDD with S/SL Injector*.

Chapter 29, *Checkout Using PDD with OC Injector*.

Checkout Using PDD with S/SL Injector

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| PDD-S/SL Checkout in Splitless Mode | 379 |
|---|-----|

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: <i>Component Concentration</i> 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.** 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. 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*.

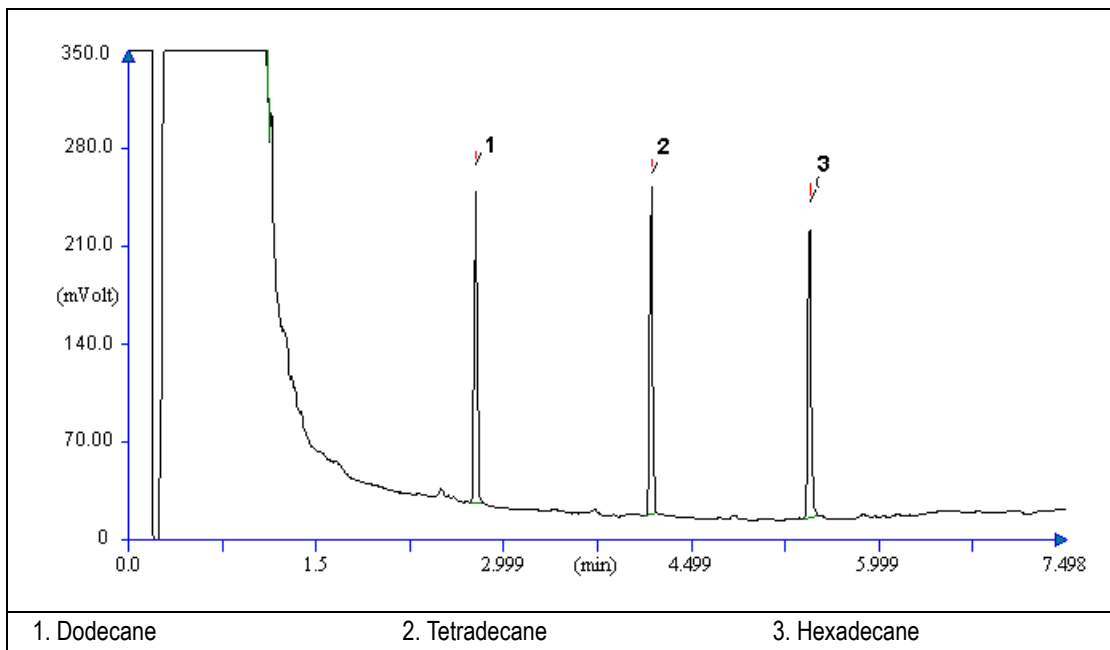





Figure 28-1. PDD-Splitless Injection


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 ($\mu\text{V/h}$) | < 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 ($\mu\text{V/h}$) | < 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 |
|  | | |

| | ATLAS | |
|---|--|--------------------------------|
| | Baseline Parameters (10V Full Scale) | |
| | Noise (μV) | < 500 |
| | Wander (μV) | < 5 000 |
| | Drift ($\mu\text{V/h}$) | < 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 |
|  | | |

| 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 | > 20 000 000 for each component |
| | Area Count Ratio Calculated as C12/C16 | 1 ± 0.1 |
|  | | |

Checkout Using PDD with OC Injector

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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: <i>Component</i> <i>Concentration</i> 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.** 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. 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



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 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 29.1*.

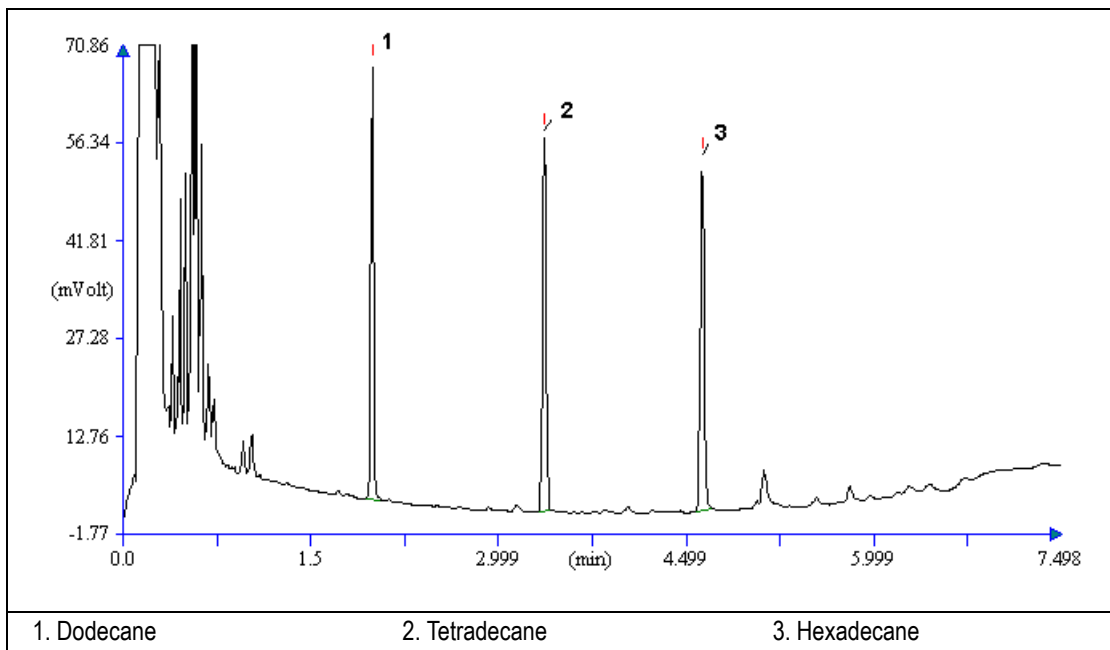





Figure 29-1. PDD-On-Column Injection


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 ($\mu\text{V/h}$) | < 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) | |
|---|--|
|  | |

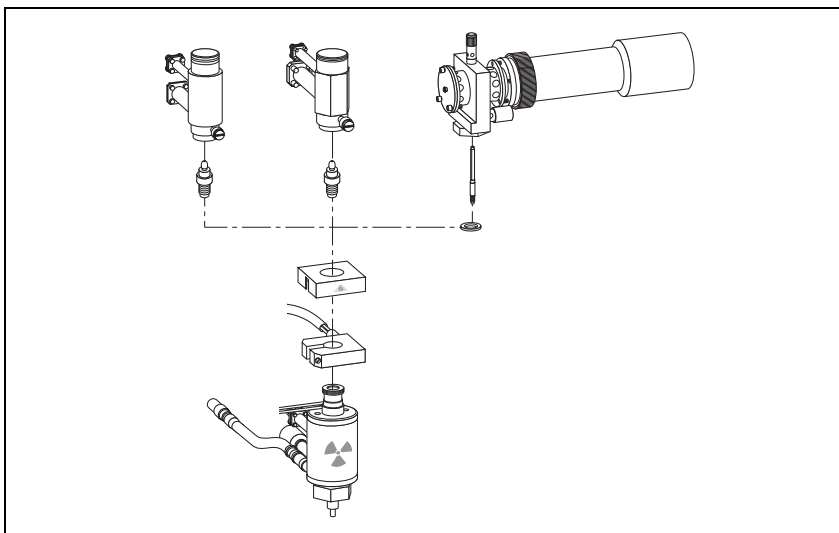
| Acceptance Values | CHROMQUEST | |
|---|---|---------------------------------|
| | Baseline Parameters (1V Full Scale) | |
| | Noise (μV) | < 50 |
| | Wander (μV) | < 500 |
| | Drift ($\mu\text{V/h}$) | < 500 |
| | Analytical Results (1V Full Scale) - Area Counts (0.01 μVs) | |
| | Components | > 10 000 000 for each component |
| | Area Count Ratio Calculated as C12/C16 | 1 ± 0.1 |
|  | | |

| | ATLAS | |
|---|--|--------------------------------|
| | Baseline Parameters (10V Full Scale) | |
| | Noise (μV) | < 500 |
| | Wander (μV) | < 5 000 |
| | Drift ($\mu\text{V/h}$) | < 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.

Chapter 30, [*Checkout Using Tandem FID*](#).

Chapter 31, [*Checkout Using Tandem NPD*](#).

Chapter 32, [*Checkout Using Tandem FPD*](#).

Checkout Using Tandem FID

Chapter at a Glance...

SOP Number: P0383/05/E - 01 September 2009 398

Scope 398

Checkout Overview 399

Important Considerations 400

Operating Procedures

Example of FID Tandem Checkout 401

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.

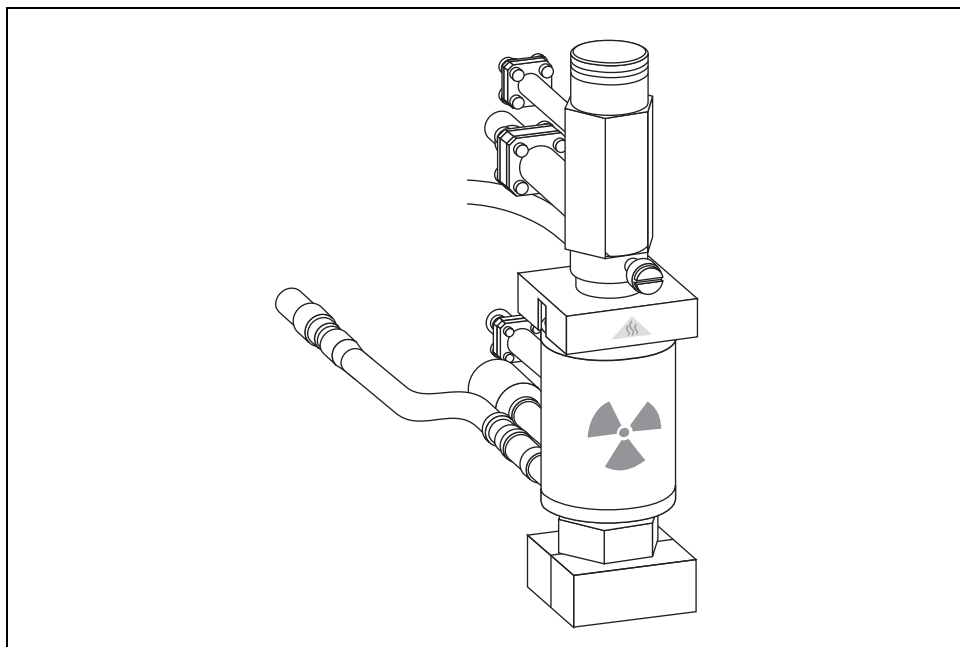


Figure 30-1. FID Tandem

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 | <i>Checkout Using ECD with S/SL Injector</i> on page 95. |
| | <i>Checkout Using ECD with OC Injector</i> on page 107. |
| | <i>Checkout Using ECD with PKD Injector</i> on page 119. |
| | <i>Checkout Using ECD with PPKD Injector</i> on page 131. |
| | <i>Checkout Using ECD with PTV Injector</i> on page 143. |
| FID | <i>Checkout Using FID with S/SL Injector</i> on page 37. |
| | <i>Checkout Using FID with OC Injector</i> on page 47. |
| | <i>Checkout Using FID with PKD Injector</i> on page 57. |
| | <i>Checkout Using FID with PPKD Injector</i> on page 69. |
| | <i>Checkout Using FID with PTV Injector</i> 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.** 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. 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.

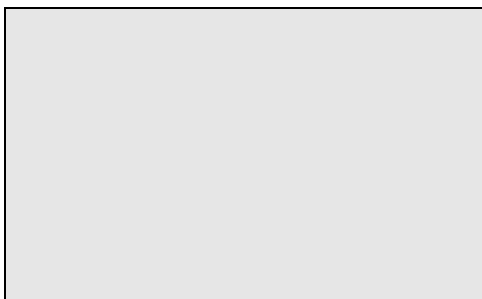
| LEFT INLET (S/SL) ¹ | | |
|--------------------------------|-----------|------|
| Temp | 230 | 230 |
| Pressure | 30.0 | 30.0 |
| Mode: | Splitless | |
| Total flow | (63.0) | |
| Split Flow | 60.0 | 60.0 |
| Splitless time | 0.80 | 0.80 |
| Constant sept purge? | Y< | |

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.

Checkout Using Tandem NPD

Chapter at a Glance...

SOP Number: P0384/06/E - 01 September 2009 406

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Checkout Overview 407

Important Considerations 408

Operating Procedures

Example of NPD Tandem Checkout 409

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.

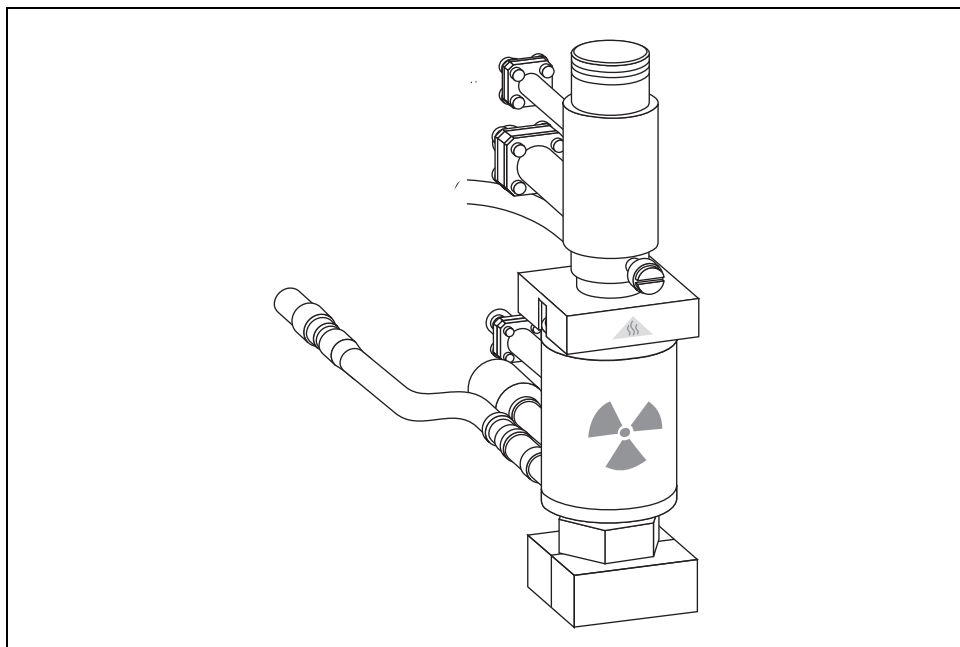


Figure 31-1. NPD Tandem

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 | <i>Checkout Using ECD with S/SL Injector</i> on page 95. |
| | <i>Checkout Using ECD with OC Injector</i> on page 107. |
| | <i>Checkout Using ECD with PKD Injector</i> on page 119. |
| | <i>Checkout Using ECD with PPKD Injector</i> on page 131. |
| | <i>Checkout Using ECD with PTV Injector</i> on page 143. |
| NPD | <i>Checkout Using NPD with S/SL Injector</i> on page 157. |
| | <i>Checkout Using NPD with OC Injector</i> on page 169. |
| | <i>Checkout Using NPD with PKD Injector</i> on page 181. |
| | <i>Checkout Using NPD with PPKD Injector</i> on page 195. |
| | <i>Checkout Using NPD with PTV Injector</i> 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 silcostealed 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.** 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. 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.

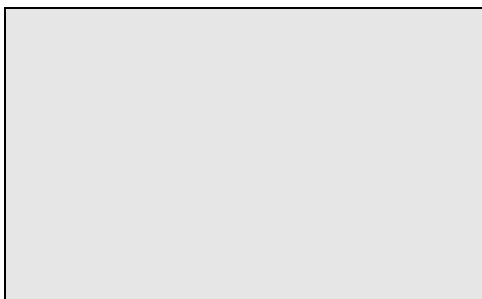
| LEFT INLET (S/SL) ¹ | | |
|--------------------------------|-----------|------|
| Temp | 230 | 230 |
| Pressure | 30.0 | 30.0 |
| Mode: | Splitless | |
| Total flow | (63.0) | |
| Split Flow | 60.0 | 60.0 |
| Splitless time | 0.80 | 0.80 |
| Constant sept purge? | Y< | |

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.

Checkout Using Tandem FPD

Chapter at a Glance...

SOP Number: P0385/06/E - 01 September 2009 414

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Example of FPD Tandem Checkout 417

Example of FPD Tandem Checkout 417

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.

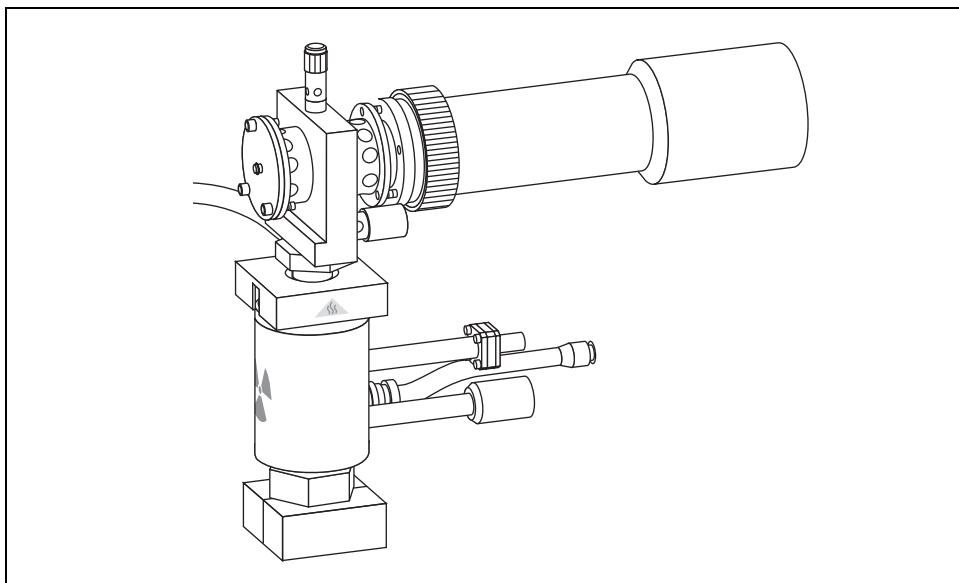


Figure 32-1. FPD Tandem

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 | <i>Checkout Using ECD with S/SL Injector</i> on page 95. |
| | <i>Checkout Using ECD with OC Injector</i> on page 107. |
| | <i>Checkout Using ECD with PKD Injector</i> on page 119. |
| | <i>Checkout Using ECD with PPKD Injector</i> on page 131. |
| | <i>Checkout Using ECD with PTV Injector</i> on page 143. |
| FPD | <i>Checkout Using FPD with S/SL Injector</i> on page 225. |
| | <i>Checkout Using FPD with OC Injector</i> on page 237. |
| | <i>Checkout Using FPD with PKD Injector</i> on page 249. |
| | <i>Checkout Using FPD with PPKD Injector</i> on page 263. |
| | <i>Checkout Using FPD with PTV Injector</i> 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 silcostealed 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.** 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. 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 μ s | | 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 |

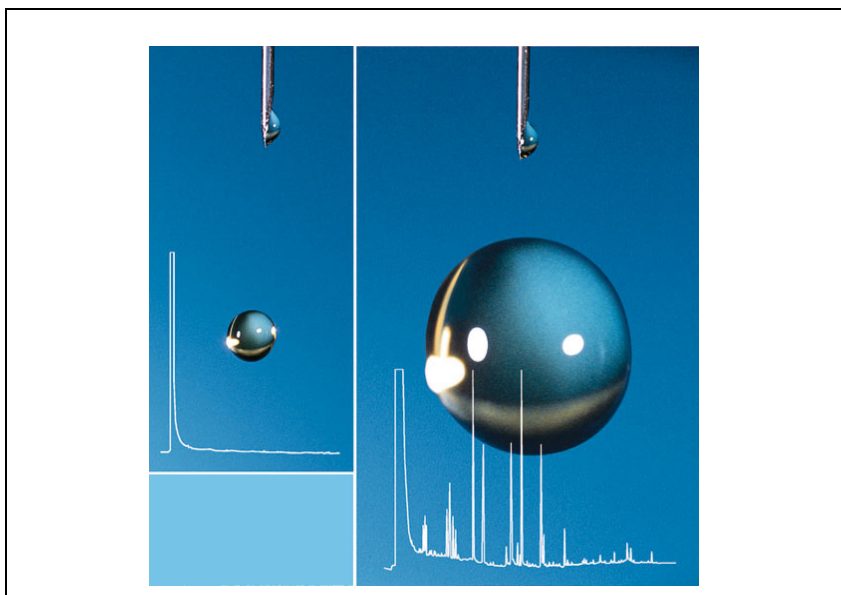
9. Activate your Data System and set the parameters required for the checkout.
10. In the Aux Detector Signal Control Table, scroll to **Auto zero?** and turn it **YES**.
11. Perform a blank analysis injecting pure hexane and press **START** on the GC to begin the checkout run.

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.

Chapter 33, *Checkout Using FID with LVSL Injector*

Chapter 34, *Checkout Using FID with LVOC Injector*

Checkout Using FID with LVSL Injector

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| | |
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| Parts Referenced | 424 |
| Analytical Conditions Required for LVSL Injection..... | 426 |
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Operating Procedures

| | |
|---|-----|
| FID-LVSL Checkout in Splitless Mode | 428 |
|---|-----|

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: | 338 190 32 |
| | <i>Component</i> <i>Concentration</i> | |
| | Dodecane 1 µg/ml | |
| | Tetradecane 1 µg/ml | |
| | Hexadecane 1 µg/ml | |
| 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.** 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. 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.

- 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 | |
| H ₂ | 35 | 35 |
| Air | 350 | 350 |
| Mkup N ₂ | 30 | 30< |

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

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

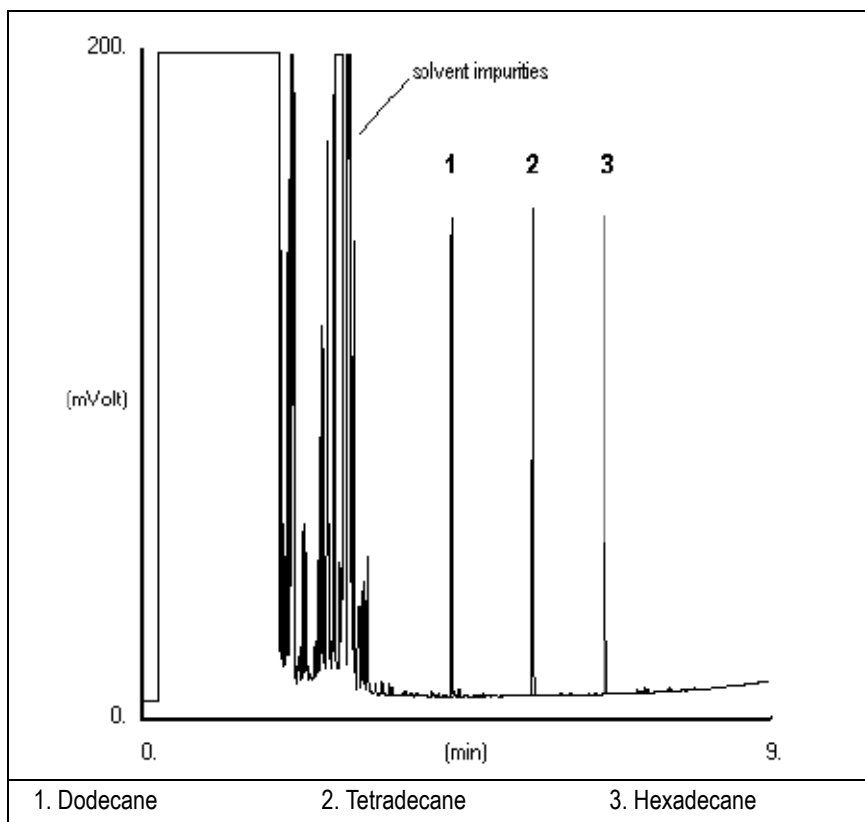





Figure 33-1. FID-LV Splitless Injection


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 |
|  | | |

| ATLAS | |
|---|--------------------------------|
| Baseline Parameters (10V Full Scale) | |
| Noise (μV) | < 300 |
| Wander (μV) | < 500 |
| Drift ($\mu\text{V/h}$) | < 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. |

Checkout Using FID with LVOC Injector

Chapter at a Glance...

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| Recommended Initial Operations | 440 |
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| FID-LVOC Checkout in LVOC Mode | 441 |
|--------------------------------------|-----|

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: | 338 190 32 |
| | <i>Component</i> <i>Concentration</i> | |
| | Dodecane 1 µg/ml | |
| | Tetradecane 1 µg/ml | |
| | Hexadecane 1 µg/ml | |
| 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 = $\geq 0,05$ 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 ($\mu\text{L/s}$) = 10 |
| Sampling depth in vial | Sampling vial depth % = 100 |
| Sample viscosity | Sample pull up speed ($\mu\text{L/s}$) = 5 Delay after bubble elimination (s) = 1 Viscosity delay (s) = 0.3 |
| GC syncro start | Syncro type = Delayed |
| Advanced parameters | Wash solvent depth % = 96 Waste depth % = 20 Needle speed into vial (mm/s) = 100 Solvent filling pull-up speed ($\mu\text{L/s}$) = 10 Bubble elimination pull-up speed ($\mu\text{L/s}$) = 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 syringe 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 ≥ 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*.

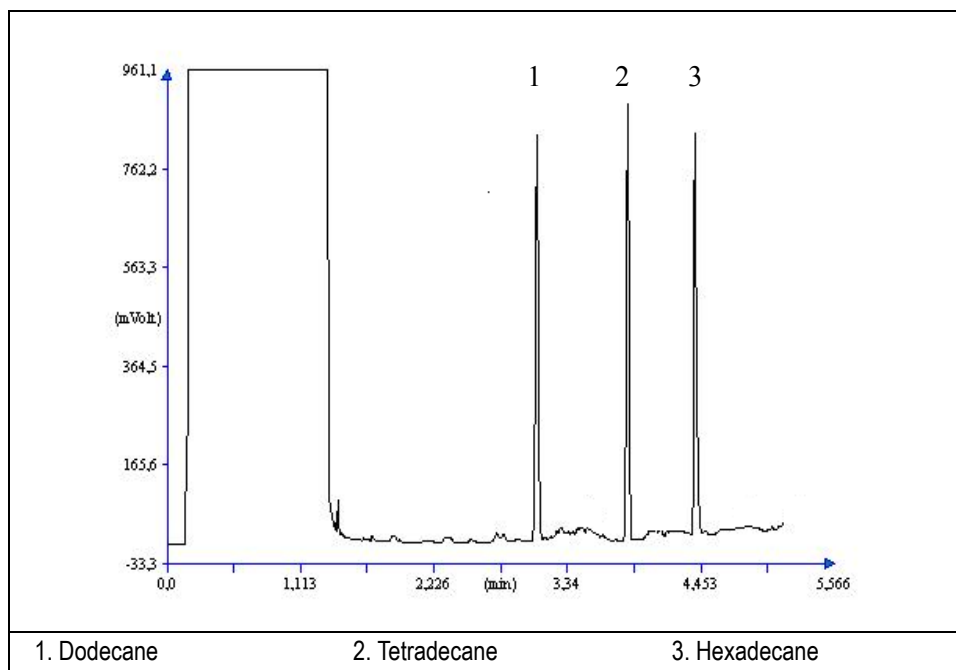





Figure 34-1. FID-LVOC Injection


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

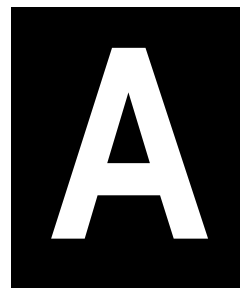
| Acceptance Values | CHROM-CARD | | |
|---|---|---|---|
| | Baseline Parameters | Analog (1V Full Scale) | Digital (10V Full Scale) |
| | Noise (μV) | < 30 | < 300 |
| | Wander (μV) | < 50 | < 500 |
| | Drift ($\mu\text{V/h}$) | < 100 | < 1 000 |
| | Analytical Results | Analog (1V Full Scale) Area 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 ($\mu\text{V/h}$) | < 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 |
|  | | |

| | 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. |



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

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Use http://www.thermo.com/com/cda/resources/resource_detail/1,,12512,00.html address for products information.

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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.)

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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

| | |
|--------|---|
| °C | 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

| | |
|-----|----------------------------|
| °F | 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

| | |
|---------------------|---|
| <i>h</i> | 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 <i>transient</i> |
| 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 <i>surge</i> |
| 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 μ s 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 μ s. |

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:
 $^{\circ}\text{C min}^{-1}$ instead of $^{\circ}\text{C/min}$
 g L^{-1} instead of g/L

A

Acceptance Criteria

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