Standard Operating Procedures

The HP 5890
Gas Chromatograph

HP part number
05890-90390

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Warranty

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Safety Information

This manual contains safety information that should be followed by the user to ensure safe operation.

WARNING

A warning calls attention to a condition or possible situation that could cause injury to the user.

CAUTION

A caution calls attention to a condition or possible situation that could damage or destroy the product or the user's work.

The *Operating Manual* supplied with the Hewlett-Packard 5890 Gas Chromatograph (HP GC) contain **WARNING** and **CAUTION** messages that inform the user about potential hazards that accompany working on and/or operating the instrument. Refer to them for complete safety information.

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Wilmington, DE 19808-1610

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FPD CHECKOUTS

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Overview

The following procedures are protocols and criteria used by Hewlett-Packard in performing final chemical checkout on 5890 instruments as they exit the manufacturing process. The intent here is to provide parallel means for the user to determine if a given 5890 continues to perform according to the original chemical checkout testing expectations.

In performing these protocols, please be aware of the following:

Due to the variety of 5890 configurations and design modifications made over time to various 5890 components, some instructions are stated in general terms. This assumes that in following SOP's, the user knows how to effectively use the given 5890 and will consult its specific operating manual(s) if questions should arise.

Acceptance criteria (area counts, retention times, baseline noise, etc.) in all cases are stated based upon use of an HP 3396 Integrator receiving ANALOG (i.e., non-digital) input from the 5890. This configuration is also used in 5890 manufacturing and was chosen because 5890 analog output is standard across all 5890 instruments and is independent of any given communication card option (HP-IL, HP-IB, RS-232C, none) which may be present.

As single-point measurement methods, meeting acceptance criteria described in these SOP's does not necessarily assure performance capability required for any given analytical method you may wish to use. Performance capability for any given analytical method must be separately demonstrated within the context of that method.

Using This Handbook

This handbook contains Standard Operating Procedures (SOPs) for the HP 5890 Gas Chromatograph. These SOPs include procedures and good laboratory practices (GLPs) which apply to operation and maintenance of the HP 5890 Gas Chromatograph.

The SOPs carry the following information on each page:

- Date of issue
- Page number
- Number of pages in SOP

The SOPs have open fields for you to add information specific to your company. On each page of the SOPs you can add an SOP number. On the first page of the SOPs you can add:

- Company name and official stamp
- SOP number
- Revision number
- Replacement revision number
- Authorizing person
- Reviewing person
- Effective date
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Standard Operating Procedure

Title: Column Adapter / Make-Up Gas Fitting, Installation

Procedure Number: _____

Revision Number: _____

Replaces Revision: _____

Approved by: _____

Reviewed by: _____

Effective Date: _____

Company stamp:

Distribution List

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Scope

Use the following procedure to properly install an appropriate column or make-up gas adapter in various inlet and detector types.

Instrumentation

This SOP applies to all 5890 Gas Chromatographs and standard inlet and detector options.

General Overview

With the exception of Split/Splitless, Split-Only, and Cool On-Column Capillary Column Inlets which are designed for use only with capillary columns, other inlet types, and all detector types, are designed to accommodate a variety of column types through installation of appropriate adapters. The following comments are for both general considerations and those associated with specific inlet and detector types.

Related SOPs

- Liner / Insert, Installation, SOP No. _____
- Column, Installation, SOP No. _____

Comments, General:

With the exception of the Thermal Conductivity Detector, all adapters are 1/4-inch in outer diameter. Those for the Thermal Conductivity Detector are 1/8-inch in outer diameter. All adapters are inserted fully into the inlet or detector in installation.

In all cases, installation of a nut and ferrule(s) to obtain a gas tight seal is best done by first tightening by fingers to insure no cross-threading occurs. If the nut does not turn easily by fingers, it is often evidence of distorted threads caused by over-tightening and/or by extended high temperature exposure in prior installations.

Over-tightening the nut during installation may so distort/damage sealing ferrule(s) that, in fact, a leak-free seal cannot be achieved no matter how tight. Follow vender recommendations for the particular chosen sealing ferrule(s).

Note that tightening recommendations are often DIFFERENT for installation of an adapter with NEW ferrule(s) as opposed to those for reinstalling an adapter with existing ferrule(s) from a previous installation. Generally, one does not tighten preset ferrule(s) to the same degree as one might for new ferrule(s).

A variety of types of ferrules exist to provide a gas-tight seal. Each type has its own particular set of advantages and disadvantages in usage:

a. Stainless steel nut and ferrules are used in applications requiring extended exposure to very high temperatures. In selecting stainless steel, be aware of the following:

   - The ferrules will form a permanent set to the given adapter and, therefore, cannot be later removed. If leakage occurs despite reasonable tightening, a new adapter is the best solution.
   - In cases of over-tightening, a stainless steel nut and ferrules can harden enough to permanently distort threads on the inlet/detector body.

b. Brass nut and ferrules are used in applications requiring more-moderate temperatures, or less-extended exposure to very high temperatures. In selecting brass, be aware of the following:

   - The ferrules will form a permanent set to the given adapter and, therefore, cannot be later removed. If leakage occurs despite reasonable tightening, a new adapter is the best solution.
   - Over long periods of usage, brass can both oxidize and distort in shape leading to increasing difficulty in installation/removal of an adapter. A new adapter is the best solution.

c. Composite material ferrules (Vespel®, graphitized Vespel®, graphite, etc.) are used in applications requiring moderate temperatures, or short-duration exposure to high temperatures. They have an advantage over metal ferrules in that they are easily removed from the adapter when necessary. In selecting such ferrules, be aware of the following:

   - Vespel®-type ferrules may fail immediately if over-tightened (by splitting, being crushed, etc.); soft graphite ferrules may extrude into the fitting if over-tightened. In such failure cases, inspect both the nut and fitting for pieces of ferrule and remove any found.
   - In usage, Vespel®-type ferrules can thermally degrade giving eventual failure by leakage and thereby leading to need for replacement.
   - Vespel®-type ferrules tend to shrink upon first exposure to typical gas chromatographic temperatures. One should plan to recheck tightness after initial usage of a new ferrule or, alternatively, the ferrule may be pre-shrunk before installation by placing it in an oven at 250 °C for at least 4 hours.

In tightening or loosening the nut on the adapter while securing the adapter into the inlet/detector body, use of two wrenches in opposition to each other is strongly recommended:

NOTE: Depending upon the given 5890 instrument and its specific configuration of inlet(s) and detector(s), the particular inlet or detector may have an insulating cup present which prevents access to the base of the inlet or detector body. In these cases, access is achieved in final tightening of nut and ferrule(s) by temporarily removing the cup and allowing it to hang on the connected column.

a. If using metal ferrules (brass or stainless steel) where torque forces are relatively high, one wrench should be placed on the nut portion of the inlet/detector body to prevent its being twisted during tightening or loosening performed with the second wrench on the nut which secures the adapter and ferrules into the inlet/detector body.

b. If using a composite ferrule (Vespel®-type, graphite, etc.) where torque forces are relatively mild, one wrench should be placed on the nut portion of the adapter body to prevent its rotation during tightening or loosening performed by the second wrench on the nut which secures the adapter and ferrule into the inlet/detector body. Rotation of the adapter within a composite ferrule system may damage the ferrule.

NOTE: In installing the capillary column make-up gas adapter in either the Thermal Conductivity or Electron Capture Detector, it is especially important to use a second wrench to prevent rotation of the adapter. Rotation of the make-up gas adapter may break its connected tube.

Comments, Packed and Purged Packed Column Inlets

a. The inlet body without any adapter is designed to accommodate a 1/4-inch glass packed column where the column itself can be inserted fully to serve as a liner.

b. Some adapters provided for these inlets are designed to accommodate a drop-in glass insert. The insert MUST be present in these adapters to provide proper expansion volume at sample injection time.

Comments, Flame Ionization and Nitrogen-Phosphorus Detectors:

a. The detector make-up gas function is internal and, therefore, no separate make-up gas adapter is provided.

b. The detector body without any adapter is designed to accommodate a 1/4-inch glass packed column where the column itself can be inserted fully to serve as a liner.

c. Some adapters provided for these detectors may require a jet exchange to optimize detector performance for the particular column type being installed.

Comments, Thermal Conductivity Detector:

a. The detector make-up gas function is externally provided by a separate make-up gas adapter located inside the oven next to the detector and designed to accommodate capillary columns only. No make-up gas is used in packed column applications.

b. The detector body without any adapter is designed to accommodate a 1/8-inch metal packed column.

Comments, Electron Capture Detector:

a. The detector make-up gas function is externally provided by a separate make-up gas adapter located inside the oven next to the detector and designed to accommodate capillary columns only. No make-up gas is used in packed column applications.

b. The detector body without any adapter is designed to accommodate a 1/4-inch glass packed column where the column itself can be inserted fully to serve as a liner.

Comments, Flame Photometric Detector:

a. The detector make-up gas function is internal and, therefore, no separate make-up gas adapter is provided.

b. The detector body without any adapter is designed to accommodate a 1/4-inch glass packed column where the column itself can be inserted fully to serve as a liner

Standard Operating Procedure

Title: Column, Installation

Procedure Number: _____

Revision Number: _____

Replaces Revision: _____

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Reviewed by: _____

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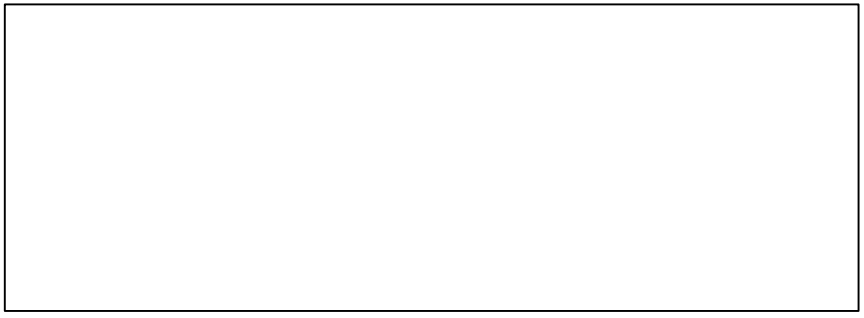
Company stamp:



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Scope

Use the following procedure to properly install a column in various inlet and detector types, and/or to condition or bakeout a column.

Instrumentation

This SOP applies to all 5890A Gas Chromatographs and standard inlet and detector options.

General Overview

Split/Splitless, Split-Only, and Cool On-Column Capillary Column Inlets are designed for use only with capillary columns. Through the use of column adapters, other inlet types, and all detector types, can accommodate installation of a variety of column types. The following are column installation considerations, both general and those associated with specific inlet and detector types.

The following comments assume you have ALREADY installed the correct hardware (column adapters, jets, inserts, etc.) appropriate for the column to be installed into the given inlet and/or detector. Consult your user documentation if in doubt.

Related SOPs

- Column Adapter / Make-Up Gas Fitting, Installation, SOP No. _____

Comments, General

With respect to inlet types, the Split/Splitless, Split-Only, and Cool On-Column Inlets are designed for use only with capillary columns. Packed and Purged Packed Inlets are inherently designed to accommodate a 1/4-inch glass packed column fully inserted into the inlet body. Other column types are accommodated through use of an appropriate adapter inserted fully into the inlet body.

With respect to detector types, the Thermal Conductivity Detector is inherently designed for use with a 1/8-inch metal packed column. Other detector types are inherently designed for use with a 1/4-inch glass packed column fully inserted into the detector body. Other column types are accommodated in all detector types through use of an appropriate adapter inserted fully into the detector body.

In all cases, installation of a nut and ferrule(s) to obtain a gas tight seal is best done by first tightening by fingers to insure no cross-threading occurs. If the nut does not turn easily by fingers, it is often evidence of distorted threads caused by over-tightening and/or by extended high temperature exposure in prior installations.

Over-tightening the nut during installation may distort/damage sealing ferrule(s) to the extent that, a leak-free seal cannot be achieved no matter how tight. In the case of glass or fused silica column types, the column itself can be broken if the fitting is over-tightened. Follow vendor recommendations for the particular chosen sealing ferrule(s).

Note that tightening recommendations are often DIFFERENT for installation of a column with NEW ferrule(s) as opposed to those for reinstalling an adapter with existing ferrule(s) from a previous installation. Generally, one does not tighten preset ferrule(s) to the same degree as one might for new ferrule(s).

A variety of types of ferrules exist to provide a gas-tight seal. Each type has its own particular set of advantages and disadvantages in usage:

a. Stainless steel nut and ferrules are used in metal packed column applications requiring extended exposure to very high temperatures. In selecting stainless steel, be aware of the following:

   - The ferrules will form a permanent set to the given column and, therefore, cannot be later removed. If leakage occurs despite reasonable tightening, a new column or cutting off the bad section of an existing column is the best solution.
   - In cases of over-tightening, a stainless steel nut and ferrules can harden enough to permanently distort threads on the inlet/detector body.

b. Brass nut and ferrules are used in metal packed column applications requiring more-moderate temperatures, or less-extended exposure to very high temperatures. In selecting brass, be aware of the following:

   - The ferrules will form a permanent set to the given column and, therefore, cannot be later removed. If leakage occurs despite reasonable tightening, a new column or cutting off the bad section of an existing column is the best solution.
   - Over long periods of usage, brass can both oxidize and distort in shape leading to increasing difficulty in installation/removal of a column. A new column, or cutting off the bad section of an existing column, is the best solution.

c. Composite material ferrules (Vespel®, graphitized Vespel®, graphite, etc.) that are compatible with virtually any column type are used in applications requiring moderate temperatures or short-duration exposure to high temperatures. They have an advantage over metal ferrules in that they are easily removed from the adapter when necessary. In selecting such ferrules, be aware of the following:

- Vespel®-type ferrules may fail immediately if over-tightened (by splitting, being crushed, etc.); soft graphite ferrules may extrude into the fitting if over-tightened. In such failure cases, inspect both the nut and fitting for pieces of ferrule and remove any found.
- In usage, Vespel®-type ferrules can thermally degrade giving eventual failure by leakage and thereby leading to need for replacement.
- Vespel®-type ferrules tend to shrink upon first exposure to typical gas chromatographic temperatures. One should plan to recheck tightness after initial usage of a new ferrule or, alternatively, the ferrule may be pre-shrunk before installation by placing it in an oven at 250 °C for at least 4 hours.

In tightening or loosening the nut securing the column onto the column adapter or inlet/detector body, use of two wrenches in opposition to each other is strongly recommended:

NOTE: Depending upon the given 5890 instrument and its specific configuration of inlet(s) and detector(s), the particular inlet or detector may have an insulating cup present which prevents access to the base of the inlet or detector body. In these cases, access is achieved in final tightening of nut and ferrule(s) by temporarily removing the cup and allowing it to hang on the connected column.

- If using metal ferrules (brass or stainless steel) where torque forces are relatively high, one wrench should be placed on the nut portion of the column adapter or inlet/detector body to prevent its being rotated during tightening or loosening performed with the second wrench on the nut which secures the column and ferrules onto the column adapter or inlet/detector body.
- If using a composite ferrule (Vespel®-type, graphite, etc.) where torque forces are relatively mild, one wrench should be placed on the nut portion of the column adapter or inlet/detector body to prevent rotation during tightening or loosening performed by the second wrench on the nut which secures the column and ferrule onto the column adapter or inlet/detector body. Rotation within a composite ferrule system may damage the ferrule.

NOTE: If installing a capillary column in either the Thermal Conductivity or Electron Capture Detector, it is especially important to use a second wrench to prevent rotation of the already installed make-up gas adapter. Rotation of the make-up gas adapter may break its connected tube.

Specific Comments, Installing Packed Columns

To minimize both dead volume and mechanical distortion of the column and ferrules in installing a metal column with new metal ferrules, the column should be pushed fully into the receiving fitting, then withdrawn by approximately 1 mm before the nut is tightened to permanently set the ferrules to form the gas tight seal. A reference mark made on the column to aid in maintaining its position during tightening is recommended.

For convenience, the column may be properly positioned and its new ferrules pre-set outside the GC by using anything having the appropriate mating fitting.

Specific Comments, Installing Capillary Columns

To assure a gas tight seal without undue tightening of the nut, the inner diameter of the chosen ferrule should match the outer diameter of the column to be installed as closely as possible.

In some installation situations, it is necessary to make a reference mark on the column. For these cases, suitable marking fluid should be readily available.

In placing the column nut and new ferrule onto a capillary column, ferrule material may enter the column. If this occurs, it is recommended that the column end be broken off at a scribe mark made by a suitable, sharp glass scribing tool. The freshly-cut end should be inspected for burrs and other irregularities and should be at right angles to the column length.

Following are comments related to installation of a column with a NEW ferrule into specific inlet and detector types:

1. Packed Column, Purged Packed Column, Split/Splitless Capillary Column, and Split-Only Capillary Column Inlets:

NOTE: Packed Column and Purged Packed Column Inlets are NOT suitable for use with capillary columns OTHER than those having a nominal 0.53 mm outer diameter (for example, HP Series 530-µ columns).

   a. On a suitable work surface, adjust the column position such that 3 ± 1 mm (a length suitable for most applications) extends beyond the threaded portion of the column nut.

b. Using a suitable marking fluid, mark the column at the opposite end of the column nut as a reference aid during installation.

c. While maintaining the column position in reference to the nut, install the column and nut into the inlet column fitting.

d. Using fingers only, tighten the column nut just enough to feel resistance in moving the column, then, if necessary, correctly postion the column in reference to the nut and previously-made mark on the column.

e. Tighten the column nut further following vendor recommendations to avoid ferrule damage through overtightening.

2. On-Column Capillary Column Inlets:

a. Install the column by gently inserting the column fully into the column fitting until you FIRST feel resistance to further insertion.

b. Tighten the column nut following vendor recommendations to avoid ferrule damage through overtightening.

3. Flame Ionization, Nitrogen-Phosphorus, and Thermal Conductivity Detectors:

a. Install the column by gently inserting the column fully into the column fitting until it stops (approximately 40 mm).

b. Using fingers only, tighten the column nut just enough to feel resistance in moving the column.

c. Withdraw the column by 1 mm from its fully-inserted position. A suitable reference mark made on the column is recommended as an aid in judging the 1-mm withdrawal.

d. Tighten the column nut further following vendor recommendations to avoid ferrule damage through overtightening.

4. Electron Capture Detectors:

NOTE: Older 5890 instruments use an ECD make-up gas adapter which provides a hard stop to column insertion. More recent instruments use a make-up gas adapter which is open-ended. The following process accommodates either situation.

a. On a suitable work surface, adjust the column position such that 74 ± 1 mm extends beyond the threaded portion of the column nut.

b. Using a suitable marking fluid, mark the column at the opposite end of the column nut as a reference aid during installation.

c. While maintaining the column position in reference to the nut, install the column and nut into the inlet column fitting.

d. Using fingers only, tighten the column nut just enough to feel resistance in moving the column, then GENTLY attempt to insert the column further by an additional 1 to 2 mm:

- If further insertion IS possible, withdraw the column and correctly postion it in reference to the nut and previously-made mark on the column.
- If a hard stop is encountered preventing further insertion, withdraw the column by 1 mm from the fully-inserted position. If necessary, an additional reference mark can be made on the column as an aid in judging the 1-mm withdrawal.

e. Tighten the column nut further following vendor recommendations to avoid ferrule damage through overtightening.

5. Flame Photometric Detectors:

a. On a suitable work surface, adjust the column position such that approximately 162 mm extends beyond the threaded portion of the column nut.

b. Using a suitable marking fluid, mark the column at the opposite end of the column nut as a reference aid during installation.

c. While maintaining the column position in reference to the nut, install the column and nut into the inlet column fitting.

d. Using fingers only, tighten the column nut just enough to feel resistance in moving the column, then, if necessary, correctly postion the column in reference to the nut and previously-made mark on the column.

e. Tighten the column nut further following vendor recommendations to avoid ferrule damage through overtightening.

Column Conditioning / Bakeout

New columns often exhibit high levels of stationary phase bleed. Older columns may exhibit bleed problems from slow-moving sample components accumulated in the column over many injections. Either situation typically causes increasing drift and/or wander in the chromatographic baseline as column temperature increases during temperature-programmed analyses.

In either case, a simple procedure may be used to condition the column to improve its bleed behavior when returned to normal service.

1. Install the column at the inlet ONLY. In leaving the detector unconnected, material exiting the column is prevented from contaminating the detector. The detector column connection fitting should be plugged to prevent entry of other foreign material from the oven as well.
2. Provide carrier gas flow through the column. Any flow rate normal for the type of column in its intended application is acceptable.

WARNING: If Hydrogen is the normal carrier gas choice, due to possible explosion hazard within the oven, temporarily switch the inlet to a supply of Helium or Nitrogen.

3. Set the oven to a temperature at or somewhat greater than the highest temperature to which the column is exposed in its analytical applications.

NOTE: Do NOT exceed the vendor-specified isothermal temperature limit for the specific column to be conditioned. Permanent damage to the column and/or its stationary phase may occur.

4. Shortest time of exposure to elevated temperature is obtain through experience, depending upon the type of column, type and thickness of stationary phase, usage in applications, etc. Usually several hours exposure is a sufficient starting point. The process may be repeated if needed.
5. To minimize thermal shock to the column, at the end of the conditioning time, allow the oven and column to cool gradually while maintaining carrier flow. Gradual cooling may be achieved simply by switching the oven off.

Standard Operating Procedure

Title: Electronic Flow Sensor (EFS) Calibration

Procedure Number: _____

Revision Number: _____

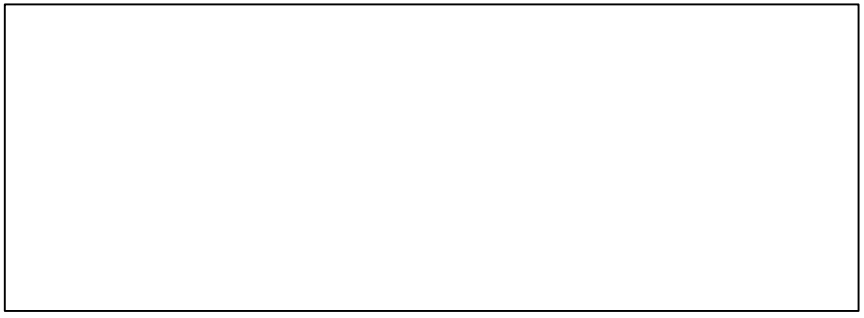
Replaces Revision: _____

Approved by: _____

Reviewed by: _____

Effective Date: _____

Company stamp:



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Scope

Use the following procedure to verify proper Electronic Flow Sensor (EFS) calibration.

Frequency

The procedure should be followed when displayed carrier gas flow rate in either one or both channels does not closely match true carrier flow rate(s) as measured via an independent flow measuring device. The procedure may also be performed any time EFS behavior is suspect, or after EFS or main electronics PC board is replacement.

Instrumentation

This SOP applies to all 5890A Gas Chromatographs.

Parts/Equipment Referenced

1. Digital Flowmeter, 0.5 - 500.0 mL/min, P/N 9301-1004
2. EFS Flow-Measurement Adapter, P/N 05890-80620
3. TCD/NPD/ECD Flow-Measurement Adapter, P/N 5020-8231

Related SOPs

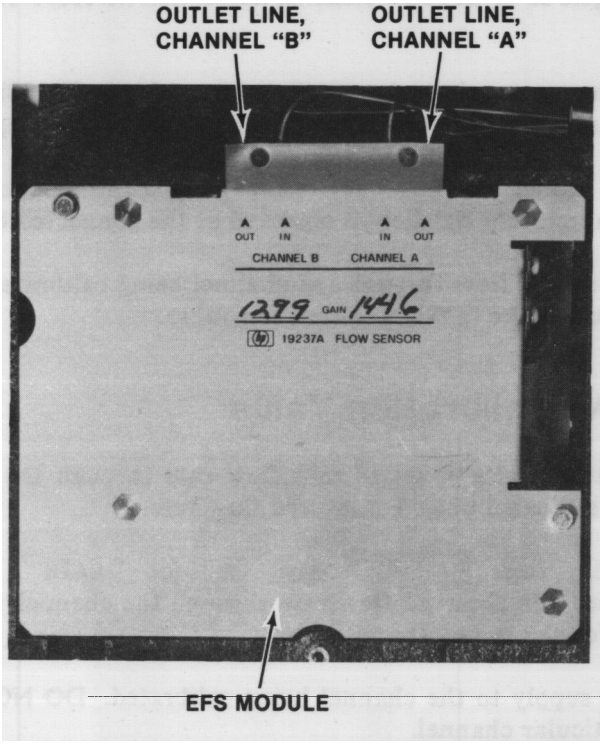
None

Procedure

NOTE: Though it is not necessary to remove columns to perform this procedure, it will be necessary to turn off carrier gas flows. For this reason, it is suggested that the oven and heated zones be cooled and detector(s) be shut down.

NOTE: The EFS is electrically heated. To assure proper calibration, if the 5890 is not already on, it should be switched on and the EFS allowed to thermally equilibrate for at least 1 (one) hour.

1. Remove the 5890 left (as viewed from the front of the instrument) side panel to expose the EFS located toward the instrument rear. Note its four tubes labeled "IN" and "OUT" for flow channels "A" and "B".



2. Noting locations for later reconnection, carefully disconnect the "IN"/"OUT" pair of tubes for the channel to be calibrated.

3. For the channel to be calibrated, ensure the associated gas type is that which is normally used as the carrier gas through the channel. Enter the following key sequence:

   `[FLOW] [A] (or [B] ) [1] (or [2] ; or [3] ; or [4] ) [ENTER]`

   where [1] is pressed if using Helium; [2] is pressed if using Nitrogen; [3] is pressed if using Hydrogen; [4] is pressed if using Argon/Methane.

4. The channel to be calibrated must first be "zeroed" (setting the displayed value to '0' (zero) with no gas flow) and then its calibration Gain value established.

   a. Enter the following key sequence:

      `[CLEAR] [.] [2] [ENTER]`

   b. Noting the 5890 display, "GAIN A" is displayed followed by two numeric values. The "A" indicates the EFS channel to be calibrated. If channel "B" is to be calibrated, press [B].

c. The first numeric value is the current flow rate measured by the indicated channel. It should be at or near '0' (zero) in value and stable with no observed drift or wander. If necessary, allow more time for the value to become stable.

d. The second value is the current calibration Gain value for the given channel.

NOTE: Record the current calibration Gain value for the given channel: if problems are encountered in obtaining a successful new calibration, it may be re-entered to restore current EFS operating behavior.

5. Assuming NO gas is flowing through the disconnected channel, press [ZERO] to display the current "zero" correction value for the given channel.

NOTE: Record the current "zero" correction value for the given channel: if problems are encountered in obtaining a successful new calibration, it may be re-entered to restore current EFS operating behavior.

Asumming a stable flow rate value is displayed, press [ENTER] to "zero" the channel.

NOTE: Record the new "zero" correction value for the given channel: if battery failure should occur, which protects 5890 setpoint storage, the value may be re-entered.

6. To establish the new calibration Gain value for the given channel:

a. Reconnect the "IN" tube to its original location, usually the flow control for the associated inlet.

b. Connect the appropriate gas to the inlet: the gas type used for calibration should be that which is normally used as the carrier gas through the given inlet and EFS channel (matching the gas-type choice made in Step 3).

c. Connect the EFS flow-measurement adapter to the "OUT" tube and connect the independent flowmeter. If needed, the TCD/NPD/ECD flow-measurement adapter may be used to handle differences in tube diameters.

d. Press [FLOW] and, observing flow rate at the independent flowmeter, establish a suitable gas flow through the EFS channel by adjusting the flow control to which the EFS channel "IN" tube was reconnected.

The flow rate established should be in the middle of the range of flow rates normally used. For example, if flow rates through the inlet connected to the given channel are typically in the range between 100 and 200 mL/min, 150 mL/min would be a resonable choice for calibration purposes.

NOTE: Flow through the given channel represents TOTAL inlet flow which includes both column flow plus flow from any inlet vents.

e. Allow sufficient time for the displayed flow rate at both the 5890 display and at the independent flowmeter to stabilize such that no wander or drift is observed.

f. When stability is achieved, observe the flow rate value measured at the independent flowmeter. Enter this value: [independent flowmeter value] [ENTER]

g. The word "CALIBRATING" will appear for a short period of time and, thereafter, the EFS-measured flow rate and new calibration Gain value is displayed indicating successful completion of the calibration process for the given channel. The displayed flow rate value should now match that of the independent flowmeter.

NOTE: Record the new calibration Gain value for the given channel: if battery failure should occur, which protects 5890 setpoint storage, the value may be re-entered.

7. Return to Step 2 and repeat the process if another channel is to be calibrated.

NOTE: If calibration cannot be successfully accomplished, and the independent flow measuring device is known to be working properly and correctly calibrated, consult your 5890 service and user documentation for additional information. If necessary, then contact your local Hewlett-Packard Service representative.

8. This completes the calibration procedure. Press [CLEAR] to exit calibration Gain entry, disconnect and remove the independent flowmeter and flow-measurement adapter(s), and reconnect the EFS "OUT" tube(s).

Standard Operating Procedure

Title: FPD Sulfur / Phosphorus Mode Filter, Installation

Procedure Number: _____

Revision Number: _____

Replaces Revision: _____

Approved by: _____

Reviewed by: _____

Effective Date: _____

Company stamp:

Distribution List

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Scope

Use the following procedure to properly install a selective filter (Phosphorus detection mode or Sulfur detection mode) in a Flame Photometric Detector (FPD).

Instrumentation

This SOP applies to all 5890A Gas Chromatographs with an FPD.

General Overview

The procedure describes how to gain access to the mode filter located in the flame housing assembly to which the photomultiplier tube (PMT) housing is attached.

Related SOPs

None

Procedure

WARNING: If the unit has been previously in operation, the FPD area may be hot enough to cause serious burns. If necessary, switch off the heated zone and allow sufficient cooling time.

WARNING: The FPD flame should be extinguished both to prevent possible fire or explosion hazard and to prevent internal water condensation as the detector cools. Shut off both FPD air and Hydrogen supplies during the procedure.

WARNING: To prevent possible permanent PMT damage during the procedure, switch off the detector electrically.

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[DET] [B] [OFF]
```

For a dual PMT configuration, ALSO enter:

```
[DET] [A] [OFF]
```

1. Remove the detector cover such that the PMT housing is fully exposed (long black tube extending towards the rear of the instrument).
2. The PMT housing (with the PMT itself retained inside) is next removed to expose the filter which is located in the flame housing assembly:
   a. Remove the coil spring securing the PMT housing to its support bracket.

b. CAREFULLY remove the PMT housing from the flame housing assembly by sliding it toward the rear of the instrument. In doing so, note the following:

- Intentionally, to prevent light leakage to the PMT, there is a close fit between the PMT housing and flame housing assembly. Some effort may be required to separate the two sections.
- In separating the PMT housing and flame housing assembly, avoid mechanical stress (bending, rotating, etc.) to the flame housing assembly. Excessive stress may fracture the fused silica liner present in the transfer tube between the column connection inside the oven and the jet located inside the flame housing assembly itself.
- The filter is located at the end of the tube from which the PMT housing is being removed. It is unsecured (with the PMT housing removed) and may fall out as the PMT housing comes free. This is to be avoided as falling onto exposed metal surfaces may create permanent scratches in the filter surface.
- The PMT can be permanently damaged through mechanical shock: it is suggested that the removed PMT housing (and PMT within) be temporarily secured such that it cannot fall from the top of the instrument.
- The PMT has an optical quality window exposed when the PMT housing is removed: it is suggested the newly-open end of the housing be covered to prevent entry of dust or other foreign matter.
- The newly exposed tube portion of the flame housing assembly has an O-ring recessed into a groove near its end which is the light seal for the PMT. Inspect the O-ring thoroughly: it should present a close fit within its groove (no sign of stretch or sag) and no sign of physical deformation, scratches, cuts, etc. If in doubt, it should be replaced as any light leakage will cause baseline and/or noise problems in detector usage.

NOTE: In removing, handling, and/or storing the filter, treat it similar to any item of high optical quality: permanent scratches, discolorations, surface residues, etc. may cause non-ideal behavior in the given detection mode and may indicate need for replacement.

3. Inspect the filter to be installed, handling it only by its edge to prevent fingerprints:

- One side should exhibit color, either deep blue-purple or yellow-green, depending upon the filter selected.
- The opposite side may be "silvered" to present a mirror-like reflective surface.

- Alternatively, if NEITHER side exhibits a mirror-like reflective surface, inspect the edge of the filter for an imprinted arrow (" > ").

4. Install the new filter into the tube portion of the flame housing assembly.
   - The REFLECTIVE, "silvered" side of the filter must be facing inward, TOWARD THE FLAME to properly reflect heat back from the filter itself, and from the PMT.
   - If instead, only an arrow (" > ") was found, the filter must be oriented such that the arrow points toward the PMT (AWAY from the flame).
5. Reinstall the PMT housing, again taking care to avoid mechanical stress on the flame housing assembly, then replace the coil spring to secure the PMT housing and the detector cover.
6. Restore gas flow rates and temperature setpoints. Relight the FPD flame and switch on the detector electrically:

   ```
   [DET] [B] [ON]
   ```

For a dual PMT configuration, ALSO enter:

```
[DET] [A] [ON]
```

Standard Operating Procedure

Title: Liner / Insert, Installation

Procedure Number: _____

Revision Number: _____

Replaces Revision: _____

Approved by: _____

Reviewed by: _____

Effective Date: _____

Company stamp:

Distribution List

- _____
- _____
- _____
- _____
- _____
- _____

Scope

Use the following procedure to properly install a liner (Packed, Purged Packed, Split/Splitless Capillary, and Split-Only Capillary Column Inlets) or insert (Cool On-Column Capillary Column Inlets).

Frequency

Glass insert / liner replacement frequency is highly dependent upon working variables such as sample cleanliness, sample make-up, temperature, etc. Therefore, frequency must be a local decision based upon experience. As a general guideline however, the decision should err on the conservative side, changing the glass insert / liner more frequently than might be truly necessary.

An alignment insert, used in Cool On-Column Capillary Column Inlets, serves to align the column and syringe needle. Therefore, it is replaced only if the present column is to be replaced by a column of DIFFERENT outer diameter.

Instrumentation

This SOP applies to all 5890A Gas Chromatographs and standard inlet options.

General Overview

Section "A" describes glass insert replacement applicable to all Packed and Purged Packed Column Inlets. Section "B" describes glass liner replacement applicable to all Split/Splitless and Split-Only Capillary Column Inlets. A third Section, "C", describes alignment insert replacement applicable to all Cool On-Column Capillary Column Inlets.

In all three cases, exchange is performed at the top of the 5890 (i.e., without need to enter the column oven).

Related SOPs

1. Inlet Septum, Installation, SOP No. _____
2. Column Adapter / Make-Up Gas Fitting, Installation, SOP No. _____

Section A: Packed and Purged Packed Column Inlets

WARNING: **If the unit has been previously in operation, the inlet area may be hot enough to cause serious burns. If necessary, switch off the appropriate heated zone and allow sufficient cooling time.**

WARNING: **If Hydrogen is used as carrier gas through the inlet, possible fire or explosion hazard exists if Hydrogen is allowed to flow during glass insert exchange. It is recommended that Hydrogen carrier flow be switched off during the procedure.**

NOTE: As carrier gas flow through the column ceases during glass insert exchange, it is recommended that column oven temperature be reduced to a conservative value during the procedure.

Procedure

1. Depending upon the inlet type:
   a. For the Packed Column Inlet, remove the septum retainer nut located at the top of the inlet and the septum.
   b. For the Purged Packed Column inlet, remove the combined assembly of septum retainer nut, septum, and top insert weldment. Inspect the O-ring seal between the top insert weldment and inlet body and replace it if necessary.
2. Generally, the old glass insert may now be removed through the now-exposed top of the inlet body. A fibrous, cylindrical object of suitable outer diameter (such as a match stick, large-diameter tooth pick, etc.) can be inserted into the insert from the top, then used to extract it from the inlet body.

   If the old glass insert cannot be extracted through the top of the inlet body, then, from inside the oven, the column adapter containing the insert must be removed from the inlet body. This may require first disconnecting the column to prevent possible damage and/or loss of gas-tight sealing.
3. Install the glass insert simply by dropping it into the column adapter, oriented with its unflared end entering the adapter first.

   Note that if the column adapter was never removed from the inlet body in extracting the old glass insert, the new insert should disappear entirely from view at the top of the inlet body. If its flared end is too large to pass through the top of the inlet body, try a different insert. If necessary, the column adapter must be removed from the inlet body. This may require first disconnecting the column to prevent possible damage and/or loss of gas-tight sealing.

4. Replace items removed to exchange glass inserts. Restore inlet gas flow rate(s) and temperature setpoints.

Section B: Split/Splitless and Split-Only Capillary Column Inlets

WARNING: **If the unit has been previously in operation, the inlet area may be hot enough to cause serious burns. If necessary, switch off the appropriate heated zone and allow sufficient cooling time.**

WARNING: **If Hydrogen is used as carrier gas through the inlet, possible fire or explosion hazard exists if Hydrogen is allowed to flow during glass liner exchange. It is recommended that Hydrogen carrier flow be switched off during the procedure.**

NOTE: For Split/Splitless or Split-Only Capillary Column Inlets, if either the old glass liner to be replaced, or the new glass liner to be installed contains unsecured material (glass wool, for example), the material may become dislodged if carrier gas is allowed to flow during exchange. In this case it is recommended that carrier flow be switched off during the procedure.

NOTE: As carrier gas flow through the column ceases during glass liner exchange, it is recommended that column oven temperature be reduced to a conservative value during the procedure.

Procedure:

1. Remove the combined assembly of septum retainer nut, septum, and split/splitless insert weldment. CAREFULLY bend attached tubing just enough to gain access to the top of the inlet body and to the liner to be replaced.
2. CAREFULLY remove the old liner and its O-ring seal. If fragments of O-ring material adhere to the metal surfaces (often a sign of lengthy service at high temperature), they must be removed.

NOTE: Avoid allowing fragments of either the O-ring seal or liner (if broken) from entering the inlet itself. If necessary, temporarily block the hole into the inlet body with a clean, round object of suitable diameter. Having a vaccuum cleaner available is also recommended in this situation.

3. Install the new liner with an appropriate seal: inner diameter of the new seal should closely match the outer diameter of the replacement liner. The new seal should never be loose on the liner, or have to be forced onto the liner. Leakage may occur if an inappropriately-sized seal is used.

   Also note that some liner styles may require a specific orientation in installation, especially those containing unsecured material (for example, glass wool). Follow vendor recommendations, bearing in mind that gas flow through the liner is always from the top of the inlet (septum location) to its bottom (column location).

4. Carefully replace the inlet top assembly removed to exchange liners, firmly tightening the nut portion of the split/splitless insert weldment. Restore inlet gas flow rate(s) and temperature setpoints.

Section C: Cool On-Column Capillary Column Inlets

WARNING: **If the unit has been previously in operation, the oven and inlet area may be hot enough to cause serious burns. If necessary, switch off the oven (and inlet heated zone if applicable) and allow sufficient cooling time.**

WARNING: **If Hydrogen is used as carrier gas through the inlet, possible fire or explosion hazard exists if Hydrogen is allowed to flow during alignment insert exchange. It is recommended that Hydrogen carrier flow be switched off during the procedure.**

NOTE: The Cool On-Column Capillary Column Inlet has a small coil spring located in the inlet body just below the septum location. The spring is removed in this procedure and can be easily lost. It is an integral part of the inlet assembly, helping to maintain correct column and syringe needle alignment. Proceed with vigilance.

NOTE: As carrier gas flow through the column ceases during alignment insert exchange, it is recommended that column oven temperature be reduced to a conservative value during the procedure.

Procedure:

1. If not already done, remove the column to be replaced by one of DIFFERENT outer diameter.
2. Depending upon the inlet configuration, remove the cooling tower or septum nut base assembly located at the top of the inlet.
3. CAREFULLY remove the small coil spring now visible, extending just above the top of the inlet body. Place the spring in a safe place.
4. Remove the old alignment insert by pushing it out at the top of the inlet body from the inlet column fitting inside the oven. A piece of straight wire of suitable diameter, such as a syringe plunger, can be used. Place the removed insert in a safe place to be used again if needed.

6. Replace the coil spring and cooling tower or septum nut base assembly containing, tighten the nut fully using your fingers only.
7. Install the new diameter column and restore inlet gas flow rate(s) and temperature setpoints.

Standard Operating Procedure

Title: Oven Temperature Setpoint Calibration

Procedure Number: _____

Revision Number: _____

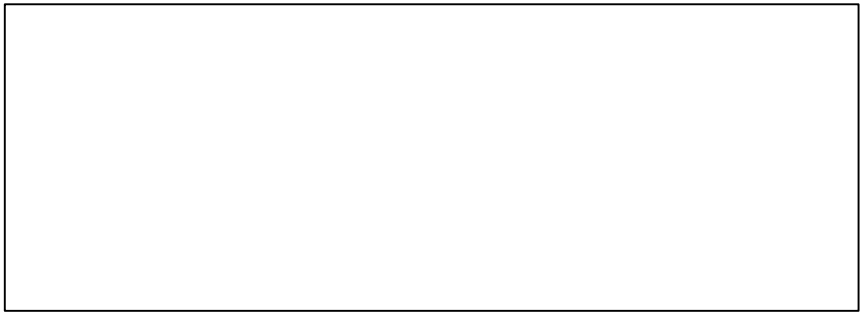
Replaces Revision: _____

Approved by: _____

Reviewed by: _____

Effective Date: _____

Company stamp:



Distribution List

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Scope

Use the following procedure to verify proper oven setpoint calibration.

Frequency

The procedure should be followed when it is critical to have retention time information closely matched between two or more separate 5890's, for example when the same analysis is being performed on multiple 5890's. The procedure may also be performed any time oven behavior is suspect, or after any oven parts replacement, particularly if the temperature sensor or main electronics PC board is replaced.

Instrumentation

This SOP applies to all 5890A Gas Chromatographs.

Parts/Equipment Referenced

1. Wahl 392VXD Heat-Prober Precision Thermometer and Wahl 305D7368-3 Platinum-RTD Temperature Sensor or equivalent temperature measuring device.

Related SOPs

None

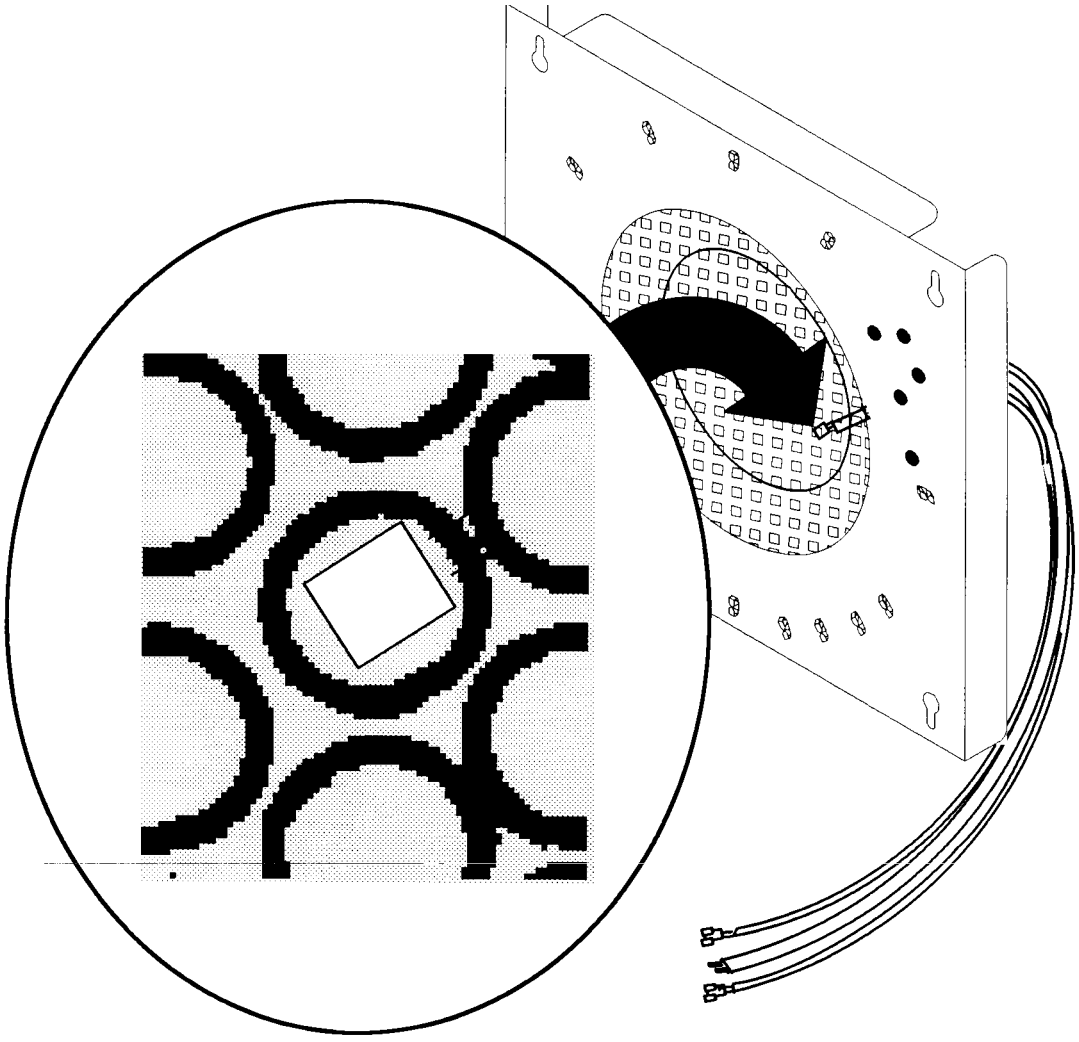
Procedure

WARNING: **If the unit has been previously in operation, areas may be hot enough to cause serious burns. Switch off heated zones and the oven and allow the unit sufficient time to cool.**

NOTE: It is not necessary to remove columns, turn off gas flows, or in any other way disturb instrument setup in performing this procedure.

1. Note the oven temperature sensor location and locate the sensor portion of the precision thermometer as close as possible to it on the column side of the oven heater shroud.

CAUTION: **In placing the independent sensor, be careful to NOT disturb the 5890 oven sensor as it is fragile. Also, its position is critical to proper oven behavior.**



2. Carefully route leads from the sensor portion of the precision thermometer such that there is no interference with oven door closure and/or with good contact between oven door insulation and the oven front.
3. Set oven temperature to a value chosen to be in the center of the range of interest:

   `[OVEN TEMP] [chosen value] [ENTER]`

For example, if the unit is generally used for analyses requiring oven temperatures between 100 and 300 °C, a value of 200 °C should be used.

Allow the unit at least 1 (one) hour to thermally stabilize.

4. Enter the key sequence and note the 5890 display:
   `[CLEAR] [.] [1] [ENTER]`

   a. The first value is the current oven temperature to 0.01 °C. It should be stable with no observed drift or wander.

   b. The second value is the current calibration correction value.

NOTE: Record the current calibration correction value: if problems are encountered in obtaining a successful new calibration, it may be re-entered to restore current oven operating behavior.

5. The new calibration correction value is calculated as:
   Calibration Correction Value = Temperature(PT) - Temperature(5890)

   where 'Temperature(PT)' is that reported by the independent precision thermometer and 'Temperature(5890)' is that reported at the 5890 display.

   For example, if the precision thermometer reports a temperature of '148.73' °C and '150.00' °C is displayed at the 5890, the new calibration correction value is '-1.27' °C.

NOTE: Record the new calibration correction value: if battery failure should occur, which protects 5890 setpoint storage, the value may be re-entered.

6. Enter the new calibration correction (including the sign if negative):
   `[calibration correction value] [ENTER]`

NOTE: The calibration correction is limited to values between ± 10.00 °C (inclusive). If the calibration value is outside this range, and the independent precision thermometer is known to be working properly and is correctly calibrated, consult your 5890 service and user documentation for additional information. If necessary, then contact your local Hewlett-Packard Service representative.

7. This completes the calibration procedure. Press [CLEAR] to exit oven calibration, cool the oven, and remove the precision thermometer sensor and its leads.

Standard Operating Procedure

Title: Inlet Septum, Installation

Procedure Number: _____

Revision Number: _____

Replaces Revision: _____

Approved by: _____

Reviewed by: _____

Effective Date: _____

Company stamp:

Distribution List

- _____
- _____
- _____
- _____
- _____
- _____

Scope

Use the following procedure to properly install a septum into an inlet.

Frequency

Septum replacement frequency is highly dependent upon working variables such as septum temperature, septum material, syringe needle quality, injection methodology and/or technique, etc. Therefore, frequency must be a local decision based upon experience. As a general guideline however, your decision should err on the conservative side, changing the septum more frequently than might be truly necessary.

Instrumentation

This SOP applies to all 5890A Gas Chromatographs and standard inlet options.

General Overview

Septum replacement is described in two separate sections: Section "A" is applicable to all Packed, Purged Packed, Split/Splitless Capillary, and Split-Only Capillary Column Inlets. Section "B" is applicable to all Cool On-Column Capillary Column Inlets.

Related SOPs

1. Liner / Insert, Installation, SOP No. _____

2. Column Adapter / Make-Up Gas Fitting, Installation, SOP No. _____

Section A: Packed Column, Purged Packed Column, Split/Splitless Capillary Column, and Split-Only Capillary Column Inlets

WARNING: If the unit has been previously in operation, the inlet area may be hot enough to cause serious burns. If necessary, switch off the appropriate heated zone and allow sufficient cooling time.

WARNING: If Hydrogen is used as carrier gas through the inlet, possible fire or explosion hazard exists if Hydrogen is allowed to flow during septum exchange. It is recommended that Hydrogen carrier flow be switched off during the procedure.

NOTE: For Split/Splitless or Split-Only Capillary Column Inlets, if a liner is present containing unsecured material (glass wool, for example), the material may become dislodged if carrier gas is allowed to flow during septum exchange. In this case, it is recommended that carrier flow be switched off during the procedure.

NOTE: As carrier gas flow through the column ceases during septum exchange, it is recommended that column oven temperature be reduced to a conservative value during the procedure.

Procedure

1. Remove the septum retainer nut located at the top of the inlet.
2. Remove the old septum. If fragments of septum material adhere to the metal surfaces (often a sign of lengthy service at high temperature), they must be removed.

NOTE: Avoid allowing septum material fragments from entering the inlet itself. If necessary, temporarily block the hole into the inlet with a clean, round, wood toothpick or similar clean, round object of suitable diameter. Having a vaccuum cleaner available is also recommended in this situation.

3. Install the new septum. Note that the procedure differs slightly based upon diameter (9.5 versus 11 mm) of the replacement septum:
   a. For a 9.5-mm diameter septum, simply drop the septum in place, replace the septum retainer nut, and tighten the nut fully using fingers only.
   b. For an 11-mm diameter septum, the septum must be pressed evenly into place, working around its edges.

      Replace the septum retainer nut and tighten it using your fingers only until you first begin to feel resistance. Then tighten further by 1/4- to 1/2-turn.

      Note that fully tightening the nut with an 11-mm diameter septum may compress the septum material to an extent that it is not easily pierced by a syringe needle, possibly leading to a bent needle or to septum "coring" whereby the needle carries small pieces of septum material into the inlet.
4. Restore inlet gas flow rate(s) and temperature setpoints.

Section B: Cool On-Column Capillary Column Inlets

WARNING: If Hydrogen is used as carrier gas through the inlet, possible fire or explosion hazard exists if Hydrogen is allowed to flow during septum exchange. It is recommended that Hydrogen carrier flow be switched off during the procedure.

NOTE: The Cool On-Column Capillary Column Inlet has a small coil spring located in the inlet body just below the septum location. Although the spring is not removed in this procedure, it can be easily lost. It is an integral part of the inlet assembly, helping to maintain correct column and syringe needle alignment. Proceed with vigilance.

NOTE: As carrier gas flow through the column ceases during septum exchange, it is recommended that column oven temperature be reduced to a conservative value during the procedure.

Procedure:

1. Depending upon the inlet configuration, remove the cooling tower or septum nut base assembly located at the top of the inlet.
2. Remove the old septum from the underside of the removed cooling tower or septum base assembly. A pointed tool such as a "thumbtack" may be needed to pry out the old septum. Any remaining fragments of septum material must be removed.
3. Insert the replacement septum into the cooling tower or septum nut base assembly. Note that if a "duck bill" septum is being installed (for manual injection use only), the septum "bill" must be oriented to point into the inlet body (toward the column).
4. Replace the cooling tower or septum nut base assembly containing the new septum and tighten the nut fully using fingers only.
5. Restore inlet gas flow rate(s) and temperature setpoints.

Standard Operating Procedure

Title: FID (Flame Ionization Detector) Checkout Using a Packed or Purged Packed Column Inlet

Procedure Number: _____

Revision Number: _____

Replaces Revision: _____

Approved by: _____

Reviewed by: _____

Effective Date: _____

Company stamp:

Distribution List

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- _____
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Scope

Use the following procedure to verify proper FID operation with either inlet type.

Parts/Equipment Referenced

- GC Instrument Performance Evaluation ("Checkout") Column, P/N 19095S-100
- FID/TCD Performance Evaluation ("Checkout") Sample, P/N 18710-60170
- HP3396A Integrator
- 10-µl syringe, P/N 9301-0810 or equivalent
- 5890A-to-3396A Analog Signal Cable, P/N 35900-60610
- 5890A-to-3396A Remote Start Cable, P/N 03394-60560
- Soap Film Flowmeter (500-ml volume, multi-range), P/N 9301-0981 or equivalent
- FID Flow-Measurement Adapter, P/N 19301-60660
- Chromatographic Grade Purity Gases: Air and Hydrogen to support the FID, Helium as Carrier, and Nitrogen or Helium as Make-Up

Related SOPs

- Inlet Septum, Installation, SOP No. _____
- Liner / Insert, Installation, SOP No. _____
- Column Adapter / Make-Up Gas Fitting, Installation, SOP No. _____
- Column, Installation, SOP No. _____

Procedure

WARNING: If the unit has been previously in operation, areas may be hot enough to cause serious burns. Switch off heated zones and the oven and allow the unit sufficient time to cool.

1. Following SOPs cited above in the Related SOPs section:
   a. At the Packed Column Inlet, install a new septum, a capillary column adapter, new liner, and the Checkout Column.
   b. At the FID, install an 0.011-inch Capillary/Series 530-µ jet, capillary column adapter, and the remaining end of the Checkout Column.

2. Between the 5890 and 3396

   a. Install the analog signal cable from the 'Signal 1' output receptacle on the 5890 and the 3396 Integrator.

   b. Install the remote start cable from the 'Remote' receptacle on the 5890 and the 3396 Integrator.

   c. At the 5890, assign Signal 1 to the FID to be evaluated:

      `[SIG 1] [A] { or [B] }`

   d. Switch on the integrator.

3. To insure carrier flow through the column during oven and zone heat-up, the following flow rates should be established (more precise values will be set later in the procedure)

   a. Insert the flow-measurement adapter fully into the top of the FID, connect the flowmeter, and establish a carrier flow rate (Helium) of 20–25 ml/min.

   b. If using the Purge Packed Inlet, note that some have an adjustable septum purge flow. In such cases, connect the flowmeter to the septum purge vent and adjust flow rate (Helium) to 1–2 ml/min.

4. Set the following oven and heated zone setpoint values:

   ```
   [INJ A TEMP] { or [INJ B TEMP] } [2] [0] [0] [ENTER]
   [DET A TEMP] { or [DET B TEMP] } [2] [5] [0] [ENTER]
   ```

   Oven Program (1 ramp):

NOTE: Depending upon the given 5890, the '[INIT VALUE]' key referenced in the following steps may be labeled '[INIT TEMP]'. In such cases, the '[FINAL VALUE]' key is labeled '[FINAL TEMP]'.

```
[INIT VALUE] [1] [1] [0] [ENTER]
[INIT TIME] [0] [ENTER]
[RATE] [1] [5] [ENTER]
[FINAL VALUE] [1] [5] [0] [ENTER]
[FINAL TIME] [1] [ENTER]
[OVEN TEMP] [ON]
```

   a. Allow the unit at least ½-hour to thermally stabilize.

5. After thermal stabilization, set the following flow rate values:

WARNING: Due to possible explosion hazard within the flowmeter, under no circumstances should both air and hydrogen be allowed to flow at the same time.

   a. As measured at the FID using the flowmeter and FID flow measurement adapter, set:

      ```
      Carrier (Helium) at 20 ± 1 ml/min.
      FID (Hydrogen) at 33 ± 1 ml/min.
      FID (Air) at 400 ± 20 ml/min.
      FID Make-Up (Nitrogen or Helium) at 10 ± 1 ml/min.
      ```

   b. After all flow rates are set, remove the flowmeter and flow-measurement adapter, then turn on all gas flows.

   c. If the Purge Packed Inlet is being used, as measured at the inlet vent using the flowmeter, verify:

   d. Septum Purge flow rate (Helium) at 1 - 2 ml/min.

6. At the 5890, turn on the FID electrically and display its signal output to determine flame-off background offset:

   ```
   [DET] [A] { or [B] } [ON]
   [SIG 1] [SIG 1]
   ```

   A stable value < 2.0 display units is acceptable criteria to continue. If not met, stop here and consult your 5890 service and user documentation for additional information. If necessary, then contact your local Hewlett-Packard Service representative.

7. Ignite the FID flame. Typically, a 'pop' sound is heard as the flame ignites.

   a. Observe the FID signal at the display. Successful flame ignition typically results in a permanent increase in signal output. If no appreciable increase is observed, perform the next check, otherwise continue to Step 8.

   b. Hold a cool, shiny, metal object, such as a chrome-plated wrench, just above the FID chimney. Any observed water condensation signifies the flame is indeed ignited.

   c. If no increase in signal output has been observed even though the flame is ignited, consult your 5890 service and user documentation for additional information. If necessary, then contact your local Hewlett-Packard Service representative.

d. If the flame has not ignited, recheck all flow rates and attempt ignition again. If the flame cannot be lit even though flow rates are correct, consult your 5890 service and user documentation for additional information. If necessary, then contact your local Hewlett-Packard Service representative.

8. With the flame lit, observe the FID signal at the display. This is the flame-on background offset. A stable value between 5 and 15 display units is acceptable criteria to continue to Step 9.

   Typically, the value may be > 15 display units indicating system bakeout is necessary:

   a. Reset oven and heated zone temperatures to the following values:

      ```
      [INJ A TEMP] { or [INJ B TEMP] } [2] [5] [0] [ENTER]
      [DET A TEMP] { or [DET B TEMP] } [4] [0] [0] [ENTER]
      [INIT VALUE] [2] [5] [0] [ENTER]
      ```

   b. Monitor the displayed detector signal: it should rise quickly to some higher value as the 5890 heats to the higher temperatures. Then, over longer time, the signal should decrease to some reasonably constant value.

   c. Restore the original setpoint temperatures specified in Step 4 and allow the unit at least ½-hour to thermally stabilize. Observe the displayed detector signal to determine if it is now within the acceptable range between 5 and 15 display units, verify all gas flow rates, then continue to Step 9.

   d. If the displayed detector signal is still too great to continue, repeat the bakeout process. If repeated bakeout cycles fail to give the acceptable signal level, consult your 5890 service and user documentation for additional information. If necessary, then contact your local Hewlett-Packard Service representative.

9. To check signal noise, wander, and drift, perform the following steps:

   a. At the 5890, enter a Range value of '0' (zero) for Signal 1:

      ```
      [SIG 1] [RANGE ^( )] [0] [ENTER]
      ```

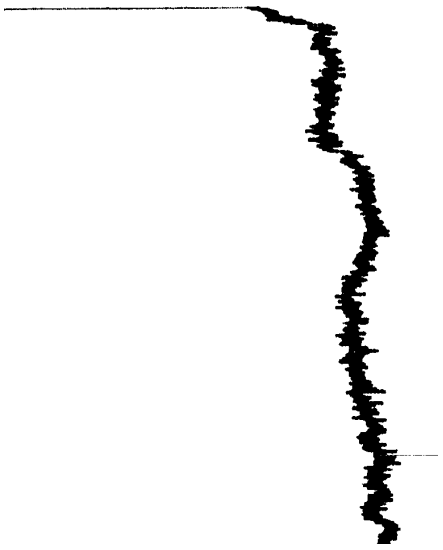
   b. At the 3396 Integrator, enter the following setpoint values:

      ```
      [ATT 2^] [-] [2] [ENTER]
      [ZERO] [5] [0] [ENTER]
      [CHT SP] [1] [ENTER]
      ```

   c. Press [PLOT] to begin plotting the FID signal.

d. At an elapsed time of 1 minute, enter the following key sequence and allow plotting to continue for another 10 minutes:

```
[ATT 2^] [0] [ENTER]
```



e. Using the method shown, measure the plot to check for the following:

- Noise in the first 1-minute portion of the plot should be < 24 mm in width.
- Wander over any given 2-minute period in the 10-minute portion of the plot should be < 14 mm in width.
- Drift over any given 5-minute period in the 10-minute portion of the plot should be < 30 mm in width.

f. If these criteria are not met, repeat the test. If, after repeated testing, the criteria cannot be met, stop here and consult your 5890 service and user documentation for additional information. If necessary, then contact your local Hewlett-Packard Service representative.

10. To perform chemical checkout, do the following steps:

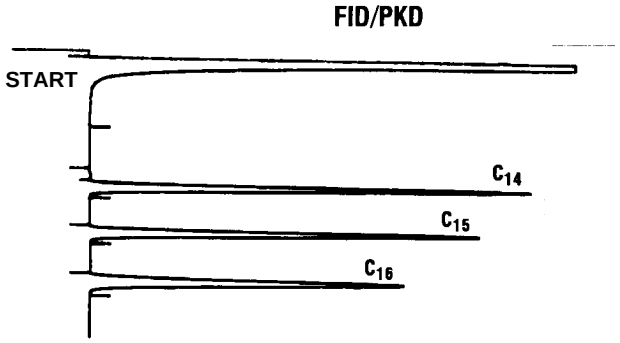
a. At the 5890, enter a Range value of '8' for Signal 1:

```
[SIG 1] [RANGE ^( )] [8] [ENTER]
```

b. At the 3396 Integrator, enter the following setpoint values:

```
[ZERO] [1] [0] [ENTER]
[ATT 2^] [3] [ENTER]
[CHT SP] [1] [ENTER]
[PK WD] [.] [0] [4] [ENTER]
[THRSH] [2] [ENTER]
[AR REJ] [0] [ENTER]
[TIME] [3] [.] [7] [5] [STOP]
```

c. Inject 1 ul of the Checkout Sample and press [START] at the 5890 to begin the checkout run.



11. The resulting chromatogram should appear similar to that shown in Figure 2. The following criteria indicate successful completion of FID chemical checkout:

    - Area counts for components labeled 'C14', 'C15', and 'C16' should each be > 125000.
    - Retention time for the component labeled 'C16' should be between 2.0 and 3.5 minutes.

    a. If these criteria are not met, repeat the test. If, after repeated testing, the criteria cannot be met, consult your 5890 service and user documentation for additional information.

    b. If necessary, then contact your local Hewlett-Packard Service representative.

Standard Operating Procedure

Title: FID (Flame Ionization Detector) Checkout Using a Split-Only or Split/Splitless (in Split Mode) Capillary Column Inlet

Procedure Number: _____

Revision Number: _____

Replaces Revision: _____

Approved by: _____

Reviewed by: _____

Effective Date: _____

Company stamp:

Distribution List

- _____
- _____
- _____
- _____
- _____
- _____

Scope

Use the following procedure to verify proper FID operation with either inlet type.

Parts/Equipment Referenced

- HP-1 Column, P/N 19095Z-121
- Capillary Inlet Evaluation (Split Mode) Sample, P/N 8500-4789
- HP3396A Integrator
- 10-µl syringe, P/N 9301-0810 or equivalent
- 5890A-to-3396A Analog Signal Cable, P/N 35900-60610
- 5890A-to-3396A Remote Start Cable, P/N 03394-60560
- Soap Film Flowmeter (500-ml volume, multi-range), P/N 9301-0981 or equivalent
- FID Flow-Measurement Adapter, P/N 19301-60660
- Chromatographic Grade Purity Gases: Air and Hydrogen to support the FID, Helium as Carrier, and Nitrogen or Helium as Make-Up

Related SOPs

- Inlet Septum, Installation, SOP No. _____
- Liner / Insert, Installation, SOP No. _____
- Column Adapter / Make-Up Gas Fitting, Installation, SOP No. _____
- Column, Installation, SOP No. _____

Procedure

WARNING: If the unit has been previously in operation, areas may be hot enough to cause serious burns. Switch off heated zones and the oven and allow the unit sufficient time to cool.

1. Following SOPs cited above in Related SOPs section.

    a. At the Capillary Column Inlet, install a new septum, a new split liner and O-ring seal, and the HP-1 Column.

    b. At the FID, install an 0.011-inch Capillary/Series 530-µ jet, capillary column adapter, and remaining end of the HP-1 Column.

2. Between the 5890 and 3396:

   a. Install the analog signal cable from the 'Signal 1' output receptacle on the 5890 and the 3396 Integrator.

   b. Install the remote start cable from the 'Remote' receptacle on the 5890 and the 3396 Integrator.

   c. At the 5890, assign Signal 1 to the FID to be evaluated: [SIG 1] [A] { or [B] }

   d. Switch on the integrator.

3. To insure carrier flow through the column during oven and zone heat-up, the following flow rates should be established (more precise values will be set later in the procedure):

   a. Insert the flow-measurement adapter fully into the top of the FID, connect the flowmeter, and establish a carrier flow rate (Helium) of 10 - 20 ml/min.

   b. Connect the flowmeter to the inlet septum purge vent and adjust flow rate (Helium) to 4 - 6 ml/min.

   c. Connect the flowmeter to the inlet split flow vent and adjust flow rate (Helium) to 175 - 225 ml/min.

4. If a Split/Splitless Capillary Inlet is being used, verify the inlet is operating in Split Mode (continuous Purge On condition).

   At the 5890, enter the following three key sequences:

   ```
   [PURGE] [A] { or [B] } [ON]
   [PURGE] [A] { or [B] } [TIME] [OFF] [0] [ENTER]
   [PURGE] [A] { or [B] } [TIME] [ON] [0] [ENTER]
   ```

5. Set the following oven and heated zone setpoint values:

   ```
   [INJ A TEMP] { or [INJ B TEMP] } [2] [0] [0] [ENTER]
   [DET A TEMP] { or [DET B TEMP] } [2] [5] [0] [ENTER]
   ```

   Oven Program (1 ramp):

NOTE: Depending upon the given 5890, the '[INIT VALUE]' key referenced in the following steps may be labeled '[INIT TEMP]'. In such cases, the '[FINAL VALUE]' key is labeled '[FINAL TEMP]'.

```
[INIT VALUE] [1] [0] [0] [ENTER]
[INIT TIME] [0] [ENTER]
[RATE] [2] [0] [ENTER]
[FINAL VALUE] [1] [8] [0] [ENTER]
[FINAL TIME] [1] [ENTER]
[OVEN TEMP] [ON]
```

   d. Allow the unit at least ½-hour to thermally stabilize.

6. After thermal stabilization, set the following flow rate values:

WARNING: Due to possible explosion hazard within the flowmeter, under no circumstances should both air and hydrogen be allowed to flow at the same time.

   a. As measured at the FID using the flowmeter and FID flow measurement adapter, set:

      ```
      Carrier (Helium) at 15 ± 1 ml/min.
      FID (Hydrogen) at 30 ± 1 ml/min.
      FID (Air) at 400 ± 20 ml/min.
      FID Make-Up (Nitrogen or Helium) at 20 ± 1 ml/min.
      ```

   b. After all flow rates are set, remove the flowmeter and flow-measurement adapter, then turn on all detector gas flows.

   c. As measured at the inlet vents using the flowmeter, set: Split flow rate (Helium) at 200 ± 20 ml/min. Septum Purge flow rate (Helium) at 5 ± 1 ml/min.

7. At the 5890, turn on the FID electrically and display its signal output to determine flame-off background offset:

   ```
   [DET] [A] { or [B] } [ON]
   [SIG 1] [SIG 1]
   ```

   A stable value < 2.0 display units is acceptable criteria to continue. If not met, stop here and consult your 5890 service and user documentation for additional information. If necessary, then contact your local Hewlett-Packard Service representative.

8. Ignite the FID flame. Typically, a 'pop' sound is heard as the flame ignites.

   a. Observe the FID signal at the display. Successful flame ignition typically results in a permanent increase in signal output. If no appreciable increase is observed, perform the next check, otherwise continue to Step 9.

   b. Hold a cool, shiny, metal object, such as a chrome-plated wrench, just above the FID chimney. Any observed water condensation signifies the flame is indeed ignited.

      If no increase in signal output has been observed even though the flame is ignited, consult your 5890 service and user documentation for additional information. If necessary, then contact your local Hewlett-Packard Service representative.

c. If the flame has not ignited, recheck all flow rates and attempt ignition again. If the flame cannot be lit even though flow rates are correct, consult your 5890 service and user documentation for additional information. If necessary, then contact your local Hewlett-Packard Service representative.

9. With the flame lit, observe the FID signal at the display. This is the flame-on background offset. A stable value between 5 and 15 display units is acceptable criteria to continue to Step 10.

   Typically, the value may be > 15 display units indicating system bakeout is necessary:

   a. Reset oven and heated zone temperatures to the following values:
      ```
      [INJ A TEMP] { or [INJ B TEMP] } [2] [5] [0] [ENTER]
      [DET A TEMP] { or [DET B TEMP] } [4] [0] [0] [ENTER]
      [INIT VALUE] [2] [5] [0] [ENTER]
      ```

   b. Monitor the displayed detector signal: it should rise quickly to some higher value as the 5890 heats to the higher temperatures. Then, over longer time, the signal should decrease to some reasonably constant value.

   c. Restore the original setpoint temperatures specified in Step 5 and allow the unit at least ½-hour to thermally stabilize. Observe the displayed detector signal to determine if it is now within the acceptable range between 5 and 15 display units, verify all gas flow rates, then continue to Step 10.

   d. If the displayed detector signal is still too great to continue, repeat the bakeout process. If repeated bakeout cycles fail to give the acceptable signal level, consult your 5890 service and user documentation for additional information. If necessary, then contact your local Hewlett-Packard Service representative.

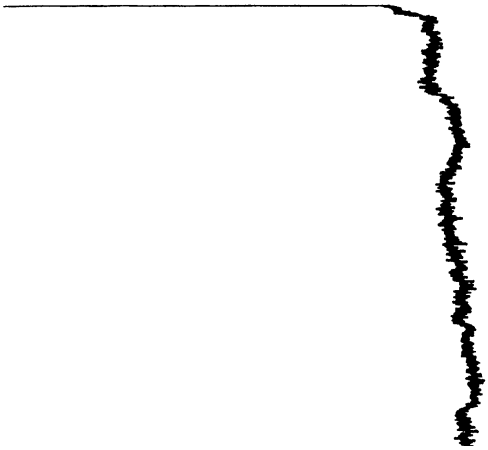
10. To check signal noise, wander, and drift, perform the following steps:

    a. At the 5890, enter a Range value of '0' (zero) for Signal 1:
       ```
       [SIG 1] [RANGE ^( )] [0] [ENTER]
       ```

    b. At the 3396 Integrator, enter the following setpoint values:
       ```
       [ATT 2^] [-] [2] [ENTER]
       [ZERO] [5] [0] [ENTER]
       [CHT SP] [1] [ENTER]
       ```

    c. Press [PLOT] to begin plotting the FID signal.

    d. At an elapsed time of 1 minute, enter the following key sequence and allow plotting to continue for another 10 minutes:
       ```
       [ATT 2^] [0] [ENTER]
       ```



e. Using the method shown, measure the plot to check for the following:

- Noise in the first 1-minute portion of the plot should be < 24 mm in width.
- Wander over any given 2-minute period in the 10-minute portion of the plot should be < 14 mm in width.
- Drift over any given 5-minute period in the 10-minute portion of the plot should be < 30 mm in width.

f. If these criteria are not met, repeat the test. If, after repeated testing, the criteria cannot be met, stop here and consult your 5890 service and user documentation for additional information. If necessary, then contact your local Hewlett-Packard Service representative.

11. To perform chemical checkout, do the following steps:

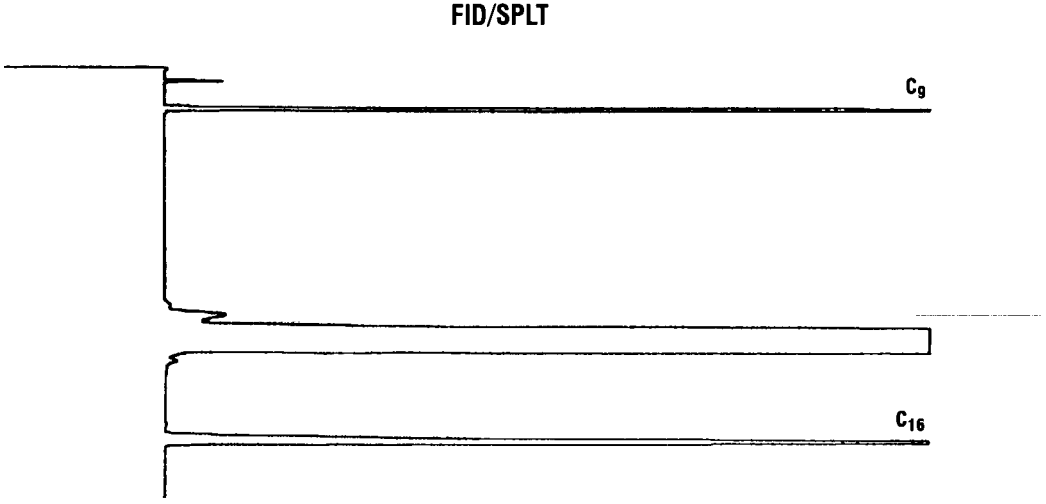
a. At the 5890, enter a Range value of '7' for Signal 1:

```
[SIG 1] [RANGE ^( )] [7] [ENTER]
```

b. At the 3396 Integrator, enter the following setpoint values:

```
[ZERO] [1] [0] [ENTER]
[ATT 2^] [5] [ENTER]
[CHT SP] [1] [.] [5] [ENTER]
[PK WD] [.] [0] [4] [ENTER]
[AR REJ] [5] [0] [0] [0] [ENTER]
[TIME] [5] [STOP]
```

c. Inject 1 µl of the Capillary Inlet Evaluation (Split Mode) Sample and press [START] at the 5890 to begin the checkout run.



d. The resulting chromatogram should appear similar to that shown in the figure above. The following criteria indicate successful completion of FID chemical checkout:

- Area counts for components labeled 'C9' and 'C16' should each be > 500,000.
- The area counts ratio calculated as 'C9'/'C16' should be 1.00 ± 0.05.

e. If these criteria are not met, repeat the test. If, after repeated testing, the criteria cannot be met, consult your 5890 service and user documentation for additional information. If necessary, then contact your local Hewlett-Packard Service representative.

Standard Operating Procedure

Title: FID (Flame Ionization Detector) Checkout Using a Cool On-Column Capillary Column Inlet

Procedure Number: _____

Revision Number: _____

Replaces Revision: _____

Approved by: _____

Reviewed by: _____

Effective Date: _____

Company stamp:

Distribution List

- _____
- _____
- _____
- _____
- _____
- _____

Scope

Use the following procedure to verify proper FID operation with this inlet type.

Parts/Equipment Referenced

1. GC Instrument Performance Evaluation ("Checkout") Column, P/N 19095S-100
2. FID/TCD Performance Evaluation ("Checkout") Sample, P/N 18710-60170
3. HP3396A Integrator
4. 10-ul syringe, P/N 9301-0810 or equivalent
5. 5890A-to-3396A Analog Signal Cable, P/N 35900-60610
6. 5890A-to-3396A Remote Start Cable, P/N 03394-60560
7. Soap Film Flowmeter (500-ml volume, multi-range), P/N 9301-0981 or equivalent
8. FID Flow-Measurement Adapter, P/N 19301-60660
9. Chromatographic Grade Purity Gases: Air and Hydrogen to support the FID, Helium as Carrier, and Nitrogen or Helium as Make-Up

Related SOPs

- Inlet Septum, Installation, SOP No. _____
- Liner / Insert, Installation, SOP No. _____
- Column Adapter / Make-Up Gas Fitting, Installation, SOP No. _____
- Column, Installation, SOP No. _____

Procedure

WARNING: If the unit has been previously in operation, areas may be hot enough to cause serious burns. Switch off heated zones and the oven and allow the unit sufficient time to cool.

1. Following SOPs cited above in the Related SOPs section:
   a. At the Cool On-Column Capillary Column Inlet, install a new septum, a Series 530-µ capillary column alignment guide, and the Checkout Column.

   b. At the FID, install an 0.011-inch Capillary/Series 530-µ jet, capillary column adapter, and remaining end of the Checkout Column.

2. Between the 5890 and 3396:

   a. Install the analog signal cable from the 'Signal 1' output receptacle on the 5890 and the 3396 Integrator.

   b. Install the remote start cable from the 'Remote' receptacle on the 5890 and the 3396 Integrator.

   c. At the 5890, assign Signal 1 to the FID to be evaluated:

      ```
      [SIG 1] [A] { or [B] }
      ```

   d. Switch on the integrator.

3. To insure carrier flow through the column during oven and zone heat-up, the following flow rates should be established (more precise values will be set later in the procedure):

   a. Insert the flow-measurement adapter fully into the top of the FID, connect the flowmeter, and establish a carrier flow rate (Helium) of 10 - 20 ml/min.

   b. If applicable, connect the flowmeter to the inlet septum purge vent and adjust flow rate to (Helium) 4 - 6 ml/min.

4. Set the following oven and heated zone setpoint values:

   If applicable, enable Oven Tracking:

   ```
   [gold] [DET A TEMP] [ON]
   [INJ A TEMP] { or [INJ B TEMP] } [ON]
   [DET A TEMP] { or [DET B TEMP] } [2] [5] [0] [ENTER]
   ```

   Oven Program (1 ramp):

NOTE: Depending upon the given 5890, the '[INIT VALUE]' key referenced in the following steps may be labeled '[INIT TEMP]'. In such cases, the '[FINAL VALUE]' key is labeled '[FINAL TEMP]'.

```
[INIT VALUE] [6] [0] [ENTER]
[INIT TIME] [.] [5] [ENTER]
[RATE] [2] [0] [ENTER]
[FINAL VALUE] [1] [8] [5] [ENTER]
[FINAL TIME] [1] [ENTER]
[OVEN TEMP] [ON]
```

Allow the unit at least ½-hour to thermally stabilize.

5. After thermal stabilization, set the following flow rate values:

WARNING: Due to possible explosion hazard within the flowmeter, under no circumstances should both air and hydrogen be allowed to flow at the same time.

   a. As measured at the FID using the flowmeter and FID flow measurement adapter, set:

      ```
      Carrier (Helium) at 15 ± 1 ml/min.
      FID (Hydrogen) at 30 ± 1 ml/min.
      FID (Air) at 400 ± 20 ml/min.
      FID Make-Up (Nitrogen or Helium) at 20 ± 1 ml/min.
      ```

   b. After all flow rates are set, remove the flowmeter and flow-measurement adapter, then turn on all gas flows.

   c. If applicable, as measured at the inlet septum purge vent using the flowmeter, verify:

   d. Septum Purge flow rate (Helium) at 5 ± 1 ml/min.

6. At the 5890, turn on the FID electrically and display its signal output to determine flame-off background offset:

   ```
   [DET] [A] { or [B] } [ON]
   [SIG 1] [SIG 1]
   ```

   A stable value < 2.0 display units is acceptable criteria to continue. If not met, stop here and consult your 5890 service and user documentation for additional information. If necessary, then contact your local Hewlett-Packard Service representative.

7. Ignite the FID flame. Typically, a 'pop' sound is heard as the flame ignites.

   a. Observe the FID signal at the display. Successful flame ignition typically results in a permanent increase in signal output. If no appreciable increase is observed, perform the next check, otherwise continue to Step 8.

   b. Hold a cool, shiny, metal object, such as a chrome-plated wrench, just above the FID chimney. Any observed water condensation signifies the flame is indeed ignited.

      If no increase in signal output has been observed even though the flame is ignited, consult your 5890 service and user documentation for additional information. If necessary, then contact your local Hewlett-Packard Service representative.

c. If the flame has not ignited, recheck all flow rates and attempt ignition again. If the flame cannot be lit even though flow rates are correct, consult your 5890 service and user documentation for additional information. If necessary, then contact your local Hewlett-Packard Service representative.

8. With the flame lit, observe the FID signal at the display. This is the flame-on background offset. A stable value between 5 and 15 display units is acceptable criteria to continue to Step 9.

   Typically, the value may be > 15 display units indicating system bakeout is necessary:

   a. Reset oven and heated zone temperature to the following values:

      ```
      [DET A TEMP] { or [DET B TEMP] } [4] [0] [0] [ENTER]
      [INIT VALUE] [2] [5] [0] [ENTER]
      ```

   b. Monitor the displayed detector signal: it should rise quickly to some higher value as the 5890 heats to the higher temperatures. Then, over longer time, the signal should decrease to some reasonably constant value.

   c. Restore the original setpoint temperatures specified in Step 4 and allow the unit at least ½-hour to thermally stabilize. Observe the displayed detector signal to determine if it is now within the acceptable range between 5 and 15 display units, verify all gas flow rates, then continue to Step 9.

   d. If the displayed detector signal is still too great to continue, repeat the bakeout process. If repeated bakeout cycles fail to give the acceptable signal level, consult your 5890 service and user documentation for additional information. If necessary, then contact your local Hewlett-Packard Service representative.

9. To check signal noise, wander, and drift, perform the following steps:

   a. At the 5890, enter a Range value of '0' (zero) for Signal 1:

      ```
      [SIG 1] [RANGE ^( )] [0] [ENTER]
      ```

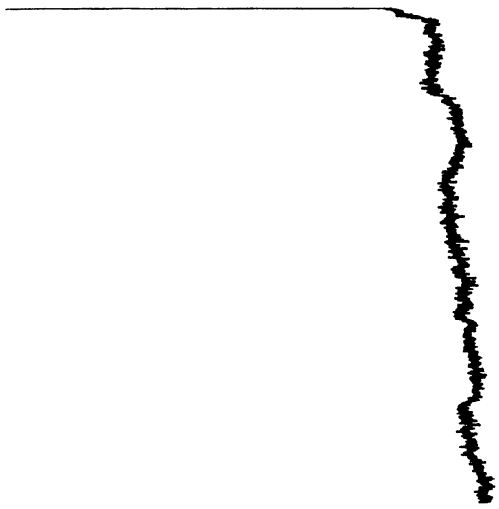
   b. At the 3396 Integrator, enter the following setpoint values:

      ```
      [ATT 2^] [-] [2] [ENTER]
      [ZERO] [5] [0] [ENTER]
      [CHT SP] [1] [ENTER]
      ```

   c. Press [PLOT] to begin plotting the FID signal.

   d. At an elapsed time of 1 minute, enter the following key sequence and allow plotting to continue for another 10 minutes:

      ```
      [ATT 2^] [0] [ENTER]
      ```



e. Using the method shown, measure the plot to check for the following:

- Noise in the first 1-minute portion of the plot should be < 24 mm in width.
- Wander over any given 2-minute period in the 10-minute portion of the plot should be < 14 mm in width.
- Drift over any given 5-minute period in the 10-minute portion of the plot should be < 30 mm in width.

f. If these criteria are not met, repeat the test. If, after repeated testing, the criteria cannot be met, stop here and consult your 5890 service and user documentation for additional information. If necessary, then contact your local Hewlett-Packard Service representative.

10. To perform chemical checkout, do the following steps:

a. At the 5890, enter a Range value of '4' for Signal 1:

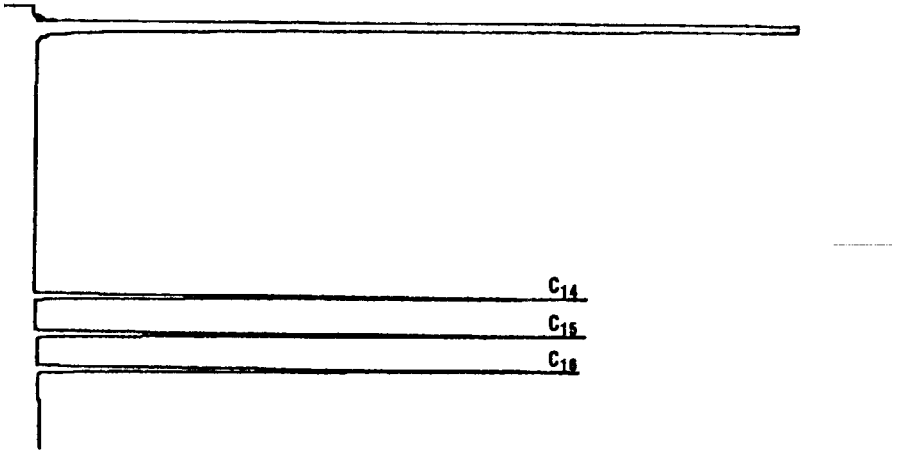
```
[SIG 1] [RANGE ^( )] [4] [ENTER]
```

b. At the 3396 Integrator, enter the following setpoint values:

```
[ZERO] [1] [0] [ENTER]
[ATT 2^] [7] [ENTER]
[CHT SP] [1] [ENTER]
[PK WD] [.] [0] [4] [ENTER]
[THRSH] [5] [ENTER]
[AR REJ] [1] [0] [0] [0] [0] [0] [0] [ENTER]
[TIME] [7] [.] [7] [5] [STOP]
```

c. Inject 1 ul of the Checkout Sample and press [START] at the 5890 to begin the checkout run.

FID/COC



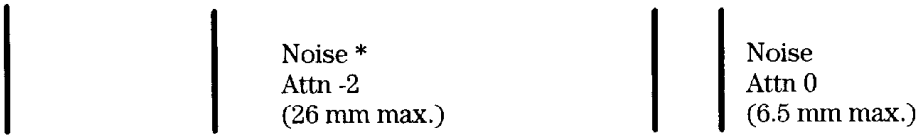
d. The resulting chromatogram should appear similar to that shown in the figure above. The following criteria indicate successful completion of FID chemical checkout:

- Area counts for components labeled 'C14', 'C15', and 'C16' should each be > 2,000,000.
- The area counts ratio calculated as 'C16'/'C14' should be 1.00 ± 0.02.

e. If these criteria are not met, repeat the test. If, after repeated testing, the criteria cannot be met, consult your 5890 service and user documentation for additional information. If necessary, then contact your local Hewlett-Packard Service representative.

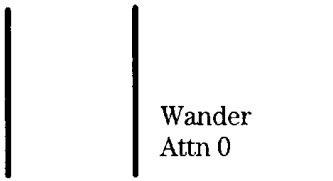
FID Noise Test Overlay
(for use with 3396 Integrator)

Directions: Place each measurement section of overlay at top of plot and then move it down plot making sure plot stays between lines. The fines on the overlay must be kept parallel to edges of the plot paper during measurement.



Integrator Settings

Zero	=	50
Attn 2	=	0
Cht Sp	=	1.0
Pk Wd	=	0.04
Thrsh	=	0
Ar Rej	=	0



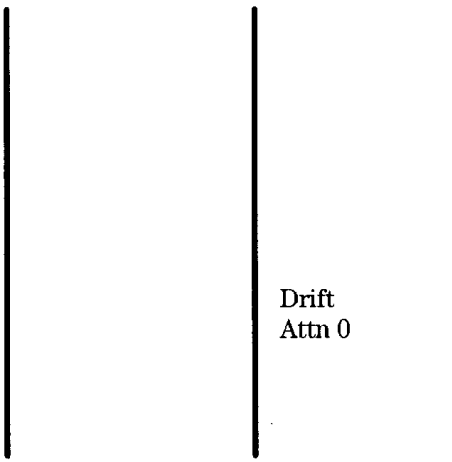
5890 Settings

Range = 0

Offset: Between 5 and 15 pA

Test Time: 10 minutes at Attn 0
1 minute at Attn -2*

No Spikes allowed on plot



* Use only if noise is borderline 6.5 mm wide at Attn 0.

Standard Operating Procedure

Title: TCD (Thermal Conductivity Detector) Checkout Using a Packed or Purged Packed Column Inlet

Procedure Number: _____

Revision Number: _____

Replaces Revision: _____

Approved by: _____

Reviewed by: _____

Effective Date: _____

Company stamp:

Distribution List

- _____
- _____
- _____
- _____
- _____
- _____

Scope

Use the following procedure to verify proper TCD operation with either inlet type.

Parts/Equipment Referenced

- GC Instrument Performance Evaluation ("Checkout") Column, P/N 19095S-100
- FID/TCD Performance Evaluation ("Checkout") Sample, P/N 18710-60170
- HP3396A Integrator
- 10-ul syringe, P/N 9301-0810 or equivalent
- 5890A-to-3396A Analog Signal Cable, P/N 35900-60610
- 5890A-to-3396A Remote Start Cable, P/N 03394-60560
- Soap Film Flowmeter (500-ml volume, multi-range), P/N 9301-0981 or equivalent
- TCD/NPD/ECD Flow-Measurement Adapter, P/N 5020-8231
- Chromatographic Grade Purity Gas: Helium as Carrier, Reference, and Make-Up

Related SOPs

- Inlet Septum, Installation, SOP No. _____
- Liner / Insert, Installation, SOP No. _____
- Column Adapter / Make-Up Gas Fitting, Installation, SOP No. _____
- Column, Installation, SOP No. _____

Procedure

WARNING: If the unit has been previously in operation, areas may be hot enough to cause serious burns. Switch off heated zones and the oven and allow the unit sufficient time to cool.

1. Following SOPs cited above in Related SOPs section:

    a. At the Packed Column Inlet, install a new septum, a capillary column adapter and new liner, and the Checkout Column.

    b. At the TCD, install the make-up gas fitting and remaining end of the Checkout Column.

2. At the 5890, verify that the TCD to be evaluated has its 'Gas Type' set to 'H2/HE' and its 'Sensitivity' set to 'HIGH':

    a. Locate the 'Gas Type' switch at the top of the 5890, adjacent to the TCD to be evaluated. If needed, set the switch to its 'H2/HE' position.

    b. Depending upon the particular 5890, do one of the following to set 'Sensitivity':

    - On some 5890s, TCD 'Sensitivity' is set by a switch adjacent to the 'Gas Type' switch. If a 'Sensitivity' switch is present, set it to the 'HIGH' position.
    - For other 5890s, there is no TCD 'Sensitivity' switch: the function is set through the keyboard. If no switch is present, enter the following key sequence to set the TCD to be evaluated to high sensitivity:

      `[gold] [DET] [A] { or [B] } [ON]`

3. Between the 5890 and 3396:

    a. Install the analog signal cable from the 'Signal 1' receptacle on the 5890 and the 3396 Integrator.

    b. Install the remote start cable from the 'Remote' receptacle on the 5890 and the 3396 Integrator.

    c. At the 5890, assign Signal 1 to the TCD to be evaluated:

    `[SIG 1] [A] { or [B] }`

    d. Switch on the integrator.

4. If present, remove the protective cap found installed on the TCD exhaust vent at the top of the detector, then insert the flow-measurement adapter fully onto the TCD exhaust vent and connect the flowmeter:

    a. Establish the following flow rates:

    ```
    Carrier (Helium) at 20 ± 1 ml/min.
    TCD Reference (Helium) at 30 ± 1 ml/min.
    TCD Make-Up (Helium) at 1 - 2 ml/min.
    ```

    Then remove the flowmeter and flow-measurement adapter.

    b. If using the Purge Packed Inlet, note that some have an adjustable septum purge flow. In such cases, connect the flowmeter to the septum purge vent and adjust flow rate (Helium) to 1 - 2 ml/min.

5. Set the following oven and heated zone setpoint values:

```
[INJ A TEMP] { or [INJ B TEMP] } [2] [5] [0] [ENTER]
[DET A TEMP] { or [DET B TEMP] } [3] [0] [0] [ENTER]
```

Oven Program (1 ramp):

NOTE: Depending upon the given 5890, the '[INIT VALUE]' key referenced in the following steps may be labeled '[INIT TEMP]'. In such cases, the '[FINAL VALUE]' key is labeled '[FINAL TEMP]'.

```
[INIT VALUE] [6] [0] [ENTER]
[INIT TIME] [1] [ENTER]
[RATE] [1] [5] [ENTER]
[FINAL VALUE] [1] [5] [0] [ENTER]
[FINAL TIME] [1] [ENTER]
[OVEN TEMP] [ON]
```

Allow the unit at least ½-hour to thermally stabilize.

6. After thermal stabilization, at the 5890, turn on the TCD electrically and allow about 3 minutes for stabilization.

Display its signal output to determine background offset:

```
[DET] [A] { or [B] } [ON]
[SIG 1] [SIG 1]
```

a. A stable offset at any value between 10 and 30 display units (inclusive) is acceptable. Continue to Step 8.

b. If offset is < 5 display units, stop here and consult your 5890 service and user documentation for additional information. If necessary, then contact your local Hewlett-Packard Service representative.

c. If offset is between 5 and 10 display units, do the following:

- Reset the TCD temperature to 400º C and allow at least ½-hour for thermal stabilization.
- At this elevated temperature, offset of 5 display units or greater is acceptable. Continue to Step 8. Otherwise, stop here and consult your 5890 service and user documentation for additional information. If necessary, then contact your local Hewlett-Packard Service representative.

d. If offset is > 30 display units, there may be chemical contamination contributing to the signal. Continue to Step 7.

7. Perform bakeout of the TCD in the following manner:

   a. Reset the TCD temperature to 400º C and allow at least ½-hour for thermal stabilization.

   b. Monitor the displayed detector signal: for chemical contamination, it should rise quickly to some higher value as the TCD heats to the higher temperature. Then, over longer time, the signal should decrease to some reasonably constant value.

   c. Restore the original setpoint temperature of 300º C and allow at least ½-hour for thermal stabilization.

   d. Observe the displayed detector signal to determine if it is now within the acceptable range between 10 and 30 display units, verify all gas flow rates, then continue to Step 8.

   e. If the displayed detector signal is still too great to continue, repeat the bakeout process. If repeated bakeout cycles fail to give the acceptable signal level, consult your 5890 service and user documentation for additional information. If necessary, then contact your local Hewlett-Packard Service representative.

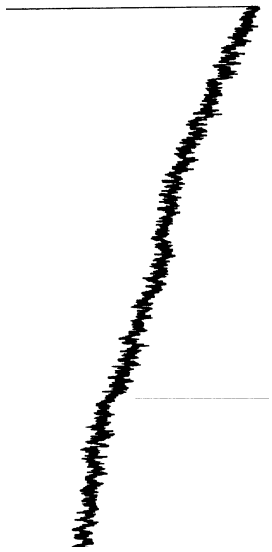
8. To check signal noise, wander, and drift, perform the following steps:

   a. At the 5890, enter a Range value of '0' (zero) for Signal 1:
   `[SIG 1] [RANGE ^( )] [0] [ENTER]`

   b. At the 3396 Integrator, enter the following setpoint values:
   `[ZERO] [5] [0] [ENTER]`
   `[ATT 2^] [0] [ENTER]`
   `[CHT SP] [1] [ENTER]`

   c. Press [PLOT] to begin plotting the TCD signal.

   d. Allow plotting to continue for at least 30 minutes.



e. Using the method shown, measure the plot to check for the following:

- Noise over any given 1-minute portion of the plot should be < 6.5 mm in width (between the inner pair of lines). Up to 4 spikes are allowed in any given portion provided they do not exceed limits represented by the outer pair of lines.
- Wander over any given 5-minute portion of the plot should be < 22 mm in width.
- Drift over the full 30-minute plot should be < 43 mm in width.

f. If these criteria are not met, repeat the test. If, after repeated testing, the criteria cannot be met, stop here and consult your 5890 service and user documentation for additional information. If necessary, then contact your local Hewlett-Packard Service representative.

9. To perform chemical checkout, do the following steps:

a. At the 5890, reset the oven Initial Temperature and allow at least ½-hour for thermal stabilization:

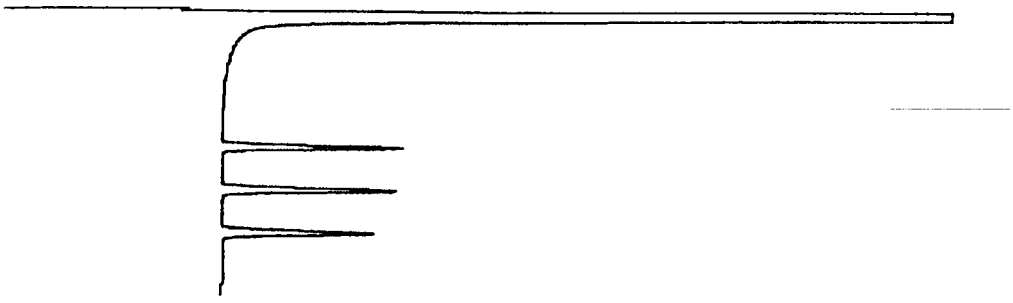
```
[INIT VALUE] [1] [1] [0] [ENTER]
```

b. At the 3396 Integrator, enter the following setpoint values:

```
[ZERO] [1] [0] [ENTER]
[ATT 2^] [7] [ENTER]
[CHT SP] [1] [ENTER]
[PK WD] [.] [0] [4] [ENTER]
[THRSH] [3] [ENTER]
[AR REJ] [0] [ENTER]
[TIME] [5] [STOP]
```

c. Inject 3 µl of the Checkout Sample and press [START] at the 5890 to begin the checkout run.

TCD/PKD



d. The resulting chromatogram should appear similar to that shown in the figure above. The following criteria indicate successful completion of TCD chemical checkout:

- Area counts for components labeled 'C14', 'C15', and 'C16' should each be > 1,000,000.
- The area counts ratio calculated as 'C14'/'C16' should be 1.05 ± 0.05.

e. If these criteria are not met, repeat the test. If, after repeated testing, the criteria cannot be met, consult your 5890 service and user documentation for additional information. If necessary, then contact your local Hewlett-Packard Service representative.

Standard Operating Procedure

Title: TCD (Thermal Conductivity Detector) Checkout Using a Split-Only or Split/Splitless (in Split Mode) Capillary Column Inlet

Procedure Number: _____

Revision Number: _____

Replaces Revision: _____

Approved by: _____

Reviewed by: _____

Effective Date: _____

Company stamp:

Distribution List

- _____
- _____
- _____
- _____
- _____
- _____

Scope

Use the following procedure to verify proper TCD operation with either inlet type.

Parts/Equipment Referenced

- GC Instrument Performance Evaluation ("Checkout") Column, P/N 19095S-100
- Capillary Inlet Evaluation (Split Mode) Sample, P/N 8500-4789
- HP3396A Integrator
- 10-ul syringe, P/N 9301-0810 or equivalent
- 5890A-to-3396A Analog Signal Cable, P/N 35900-60610
- 5890A-to-3396A Remote Start Cable, P/N 03394-60560
- Soap Film Flowmeter (500-ml volume, multi-range), P/N 9301-0981 or equivalent
- TCD/NPD/ECD Flow-Measurement Adapter, P/N 5020-8231
- Chromatographic Grade Purity Gas: Helium as Carrier, Reference, and Make-Up

Related SOPs

- Inlet Septum, Installation, SOP No. _____
- Liner / Insert, Installation, SOP No. _____
- Column Adapter / Make-Up Gas Fitting, Installation, SOP No. _____
- Column, Installation, SOP No. _____

Procedure

WARNING: If the unit has been previously in operation, areas may be hot enough to cause serious burns. Switch off heated zones and the oven and allow the unit sufficient time to cool.

1. Following SOPs cited above in the Related SOPs section:
   a. At the Packed Column Inlet, install a new septum, a capillary column adapter and new liner, and the Checkout Column.
   b. At the TCD, install the make-up gas fitting and remaining end of the Checkout Column.

2. At the 5890, verify that the TCD to be evaluated has its 'Gas Type' set to 'H2/HE' and its 'Sensitivity' set to 'HIGH':

   a. Locate the 'Gas Type' switch at the top of the 5890, adjacent to the TCD to be evaluated. If needed, set the switch to its 'H2/HE' position.

   b. Depending upon the particular 5890, do one of the following to set 'Sensitivity':

      - On some 5890s, TCD 'Sensitivity' is set by a switch adjacent to the 'Gas Type' switch. If a 'Sensitivity' switch is present, set it to the 'HIGH' position.
      - For other 5890s, there is no TCD 'Sensitivity' switch: the function is set through the keyboard. If no switch is present, enter the following key sequence to set the TCD to be evaluated to high sensitivity:

        `[gold] [DET] [A] { or [B] } [ON]`

3. Between the 5890 and 3396:

   a. Install the analog signal cable from the 'Signal 1' output receptacle on the 5890 and the 3396 Integrator.

   b. Install the remote start cable from the 'Remote' receptacle on the 5890 and the 3396 Integrator.

   c. At the 5890, assign Signal 1 to the TCD to be evaluated:

      `[SIG 1] [A] { or [B] }`

   d. Switch on the integrator.

4. If a Split/Splitless Capillary Inlet is being used, verify the inlet is operating in Split Mode (continuous Purge On condition). At the 5890, enter the following three key sequences:

   ```
   [PURGE] [A] { or [B] } [ON]
   [PURGE] [A] { or [B] } [TIME] [OFF] [0] [ENTER]
   [PURGE] [A] { or [B] } [TIME] [ON] [0] [ENTER]
   ```

5. If present, remove the protective cap found installed on the TCD exhaust vent at the top of the detector, then insert the flow-measurement adapter fully onto the TCD exhaust vent and connect the flowmeter:

a. Establish the following flow rates:

```
Carrier (Helium) at 15 ± 1 ml/min.
TCD Reference (Helium) at 37 ± 1 ml/min.
TCD Make-Up (Helium) at 10 ± 1 ml/min.
```

Then remove the flowmeter and flow-measurement adapter.

b. As measured at the inlet vents using the flowmeter, set:

```
Split flow rate (Helium) at 200 ± 10 ml/min.
Septum Purge flow rate (Helium) at 5 ± 1 ml/min.
```

Then remove the flowmeter.

6. Set the following oven and heated zone setpoint values:

```
[INJ A TEMP] { or [INJ B TEMP] } [2] [5] [0] [ENTER]
[DET A TEMP] { or [DET B TEMP] } [3] [0] [0] [ENTER]
```

Oven Program (1 ramp):

NOTE: Depending upon the given 5890, the '[INIT VALUE]' key referenced in the following steps may be labeled '[INIT TEMP]'. In such cases, the '[FINAL VALUE]' key is labeled '[FINAL TEMP]'.

```
[INIT VALUE] [6] [0] [ENTER]
[INIT TIME] [1] [ENTER]
[RATE]  [1] [0] [ENTER]
[FINAL VALUE] [1] [5] [0] [ENTER]
[FINAL TIME] [2] [ENTER]
[OVEN TEMP] [ON]
```

Allow the unit at least ½-hour to thermally stabilize.

7. After thermal stabilization, at the 5890, turn on the TCD electrically and allow about 3 minutes for stabilization. Display its signal output to determine background offset:

```
[DET] [A] { or [B] } [ON]
[SIG 1] [SIG 1]
```

a. A stable offset at any value between 10 and 30 display units (inclusive) is acceptable. Continue to Step 9.

b. If offset is < 5 display units, stop here and consult your 5890 service and user documentation for additional information. If necessary, then contact your local Hewlett-Packard Service representative.

c. If offset is between 5 and 10 display units, do the following:

- Reset the TCD temperature to 400º C and allow at least ½-hour for thermal stabilization.
- At this elevated temperature, offset of 5 display units or greater is acceptable. Continue to Step 9. Otherwise, stop here and consult your 5890 service and user documentation for additional information. If necessary, then contact your local Hewlett-Packard Service representative.

d. If offset is > 30 display units, there may be chemical contamination contributing to the signal. Continue to Step 8.

8. Perform bakeout of the TCD in the following manner:

a. Reset the TCD temperature to 400º C and allow at least ½-hour for thermal stabilization.

b. Monitor the displayed detector signal: for chemical contamination, it should rise quickly to some higher value as the TCD heats to the higher temperature. Then, over longer time, the signal should decrease to some reasonably constant value.

c. Restore the original setpoint temperature of 300º C and allow at least ½-hour for thermal stabilization.

d. Observe the displayed detector signal to determine if it is now within the acceptable range between 10 and 30 display units, verify all gas flow rates, then continue to Step 9.

e. If the displayed detector signal is still too great to continue, repeat the bakeout process. If repeated bakeout cycles fail to give the acceptable signal level, consult your 5890 service and user documentation for additional information. If necessary, then contact your local Hewlett-Packard Service representative.

9. To check signal noise, wander, and drift, perform the following steps:

a. At the 5890, enter a Range value of '0' (zero) for Signal 1:

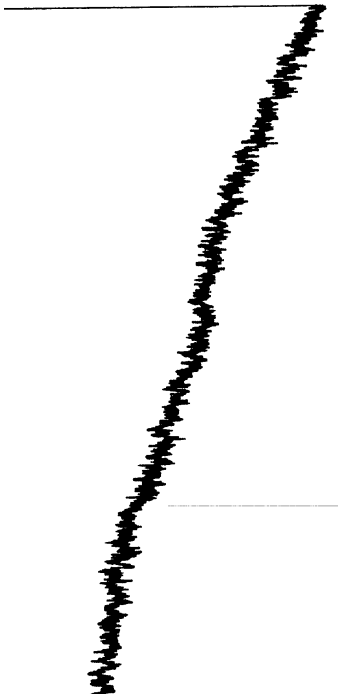
```
[SIG 1] [RANGE ^( )] [0] [ENTER]
```

b. At the 3396 Integrator, enter the following setpoint values:

```
[ZERO] [5] [0] [ENTER]
[ATT 2^] [0] [ENTER]
[CHT SP] [1] [ENTER]
```

c. Press [PLOT] to begin plotting the TCD signal.

d. Allow plotting to continue for at least 30 minutes.



e. Using the method shown, measure the plot to check for the following:

- Noise over any given 1-minute portion of the plot should be < 8 mm in width (between the inner pair of lines). Up to 4 spikes are allowed in any given portion provided are < 22 mm wide.
- Wander over any given 5-minute portion of the plot should be < 22 mm in width.
- Drift over the full 15-minute plot should be < 42 mm in width.

f. If these criteria are not met, repeat the test. If, after repeated testing, the criteria cannot be met, stop here and consult your 5890 service and user documentation for additional information. If necessary, then contact your local Hewlett-Packard Service representative.

10. To perform chemical checkout, do the following steps:

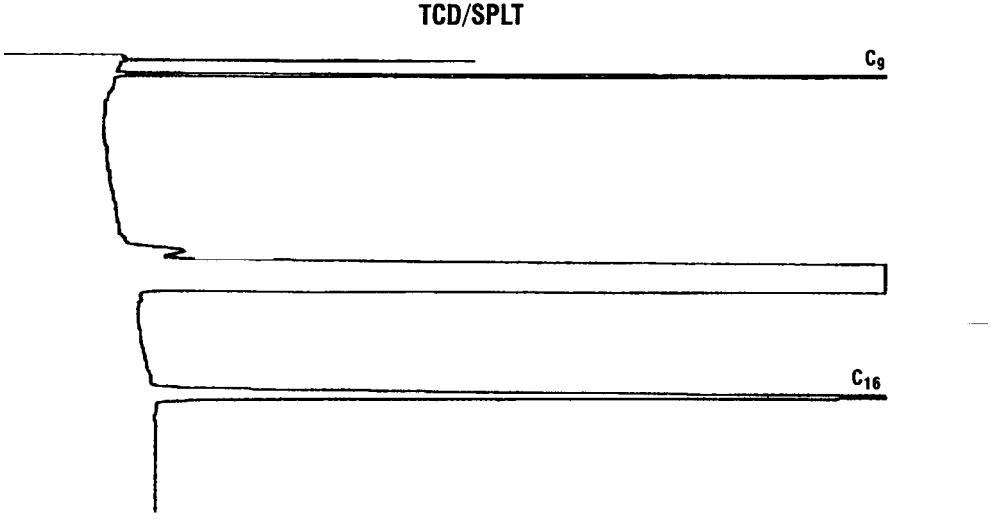
a. At the 5890, reset the oven Initial Temperature and allow at least ½-hour for thermal stabilization:

```
[INIT VALUE] [1] [0] [0] [ENTER]
```

b. At the 3396 Integrator, enter the following setpoint values:

```
[ZERO] [1] [0] [ENTER]
[ATT 2^] [4] [ENTER]
[CHT SP] [1] [ENTER]
[PK WD] [.] [0] [4] [ENTER]
[THRSH] [3] [ENTER]
[AR REJ] [5] [0] [0] [0] [ENTER]
[TIME] [8] [STOP]
```

c. Inject 1 ul of the Checkout Sample and press [START] at the 5890 to begin the checkout run.



d. The resulting chromatogram should appear similar to that shown in the figure above. The following criteria indicate successful completion of TCD chemical checkout:

   - Area counts for the component labeled 'C9' should be > 720,000.
   - The area counts ratio calculated as 'C9'/'C16' should be 1.00 ± 0.05.

e. If these criteria are not met, repeat the test. If, after repeated testing, the criteria cannot be met, consult your 5890 service and user documentation for additional information. If necessary, then contact your local Hewlett-Packard Service representative.

Standard Operating Procedure

Title: TCD (Thermal Conductivity Detector) Checkout Using a Cool On-Column Capillary Column Inlet

Procedure Number: _____

Revision Number: _____

Replaces Revision: _____

Approved by: _____

Reviewed by: _____

Effective Date: _____

Company stamp:

Distribution List

- _____
- _____
- _____
- _____
- _____
- _____

Scope

Use the following procedure to verify proper TCD operation with this inlet type.

Parts/Equipment Referenced

1. GC Instrument Performance Evaluation ("Checkout") Column, P/N 19095S-100
2. FID/TCD Performance Evaluation ("Checkout") Sample, P/N 18710-60170
3. HP3396A Integrator
4. 10-ul syringe, P/N 9301-0810 or equivalent
5. 5890A-to-3396A Analog Signal Cable, P/N 35900-60610
6. 5890A-to-3396A Remote Start Cable, P/N 03394-60560
7. Soap Film Flowmeter (500-ml volume, multi-range), P/N 9301-0981 or equivalent
8. TCD/NPD/ECD Flow-Measurement Adapter, P/N 5020-8231
9. Chromatographic Grade Purity Gas: Helium as Carrier, Reference, and Make-Up

Related SOPs

- Inlet Septum, Installation, SOP No. _____
- Liner / Insert, Installation, SOP No. _____
- Column Adapter / Make-Up Gas Fitting, Installation, SOP No. _____
- Column, Installation, SOP No. _____

Procedure

WARNING: If the unit has been previously in operation, areas may be hot enough to cause serious burns. Switch off heated zones and the oven and allow the unit sufficient time to cool.

1. Following SOPs cited above in the Related SOPs section:
   a. At the Cool On-Column Capillary Column Inlet, install a new septum, a Series 530-u capillary column alignment guide, and the Checkout Column.
   b. At the TCD, install the make-up gas fitting and remaining end of the Checkout Column.

2. At the 5890, verify that the TCD to be evaluated has its 'Gas Type' set to 'H2/HE' and its 'Sensitivity' set to 'HIGH':

   a. Locate the 'Gas Type' switch at the top of the 5890, adjacent to the TCD to be evaluated. If needed, set the switch to its 'H2/HE' position.

   b. Depending upon the particular 5890, do one of the following to set 'Sensitivity':

      - On some 5890s, TCD 'Sensitivity' is set by a switch adjacent to the 'Gas Type' switch. If a 'Sensitivity' switch is present, set it to the 'HIGH' position.
      - For other 5890s, there is no TCD 'Sensitivity' switch: the function is set through the keyboard. If no switch is present, enter the following key sequence to set the TCD to be evaluated to high sensitivity:

        ```
        [gold] [DET] [A] { or [B] } [ON]
        ```

3. Between the 5890 and 3396:

   a. Install the analog signal cable from the 'Signal 1' output receptacle on the 5890 and the 3396 Integrator.

   b. Install the remote start cable from the 'Remote' receptacle on the 5890 and the 3396 Integrator.

   c. At the 5890, assign Signal 1 to the TCD to be evaluated:

      ```
      [SIG 1] [A] { or [B] }
      ```

   d. Switch on the integrator.

4. If present, remove the protective cap found installed on the TCD exhaust vent at the top of the detector, then insert the flow-measurement adapter fully onto the TCD exhaust vent and connect the flowmeter:

   a. Establish the following flow rates:

      ```
      Carrier (Helium) at 20 ± 1 ml/min.
      TCD Reference (Helium) at 37 ± 1 ml/min.
      TCD Make-Up (Helium) at 5 ± 1 ml/min.
      ```

      Then remove the flowmeter and flow-measurement adapter.

   b. If applicable, connect the flowmeter to the inlet septum purge vent and adjust flow rate (Helium) to 4 - 6 ml/min.

5. Set the following oven and heated zone setpoint values:

   If applicable, enable Oven Tracking:

   ```
   [gold] [DET A TEMP] [ON]
   [INJ A TEMP] { or [INJ B TEMP] } [ON]
   [DET A TEMP] { or [DET B TEMP] } [3] [0] [0] [ENTER]
   ```

   Oven Program (1 ramp):

NOTE: Depending upon the given 5890, the '[INIT VALUE]' key referenced in the following steps may be labeled '[INIT TEMP]'. In such cases, the '[FINAL VALUE]' key is labeled '[FINAL TEMP]'.

   ```
   [INIT VALUE] [6] [0] [ENTER]
   [INIT TIME] [.] [5] [ENTER]
   [RATE] [2] [0] [ENTER]
   [FINAL VALUE] [1] [8] [5] [ENTER]
   [FINAL TIME] [1] [ENTER]
   [OVEN TEMP] [ON]
   ```

   Allow the unit at least ½-hour to thermally stabilize.

6. After thermal stabilization, at the 5890, turn on the TCD electrically and allow about 3 minutes for stabilization. Display its signal output to determine background offset:

   ```
   [DET] [A] { or [B] } [ON]
   [SIG 1] [SIG 1]
   ```

   a. A stable offset at any value between 10 and 30 display units (inclusive) is acceptable. Continue to Step 8.

   b. If offset is < 5 display units, stop here and consult your 5890 service and user documentation for additional information. If necessary, then contact your local Hewlett-Packard Service representative.

   c. If offset is between 5 and 10 display units, do the following:

      - Reset the TCD temperature to 400° C and allow at least ½-hour for thermal stabilization.
      - At this elevated temperature, offset of 5 display units or greater is acceptable. Continue to Step 8. Otherwise, stop here and consult your 5890 service and user documentation for additional information. If necessary, then contact your local Hewlett-Packard Service representative.

   d. If offset is > 30 display units, there may be chemical contamination contributing to the signal. Continue to Step 7.

7. Perform bakeout of the TCD in the following manner:

   a. Reset the TCD temperature to 400º C and allow at least ½-hour for thermal stabilization.

   b. Monitor the displayed detector signal: for chemical contamination, it should rise quickly to some higher value as the TCD heats to the higher temperature. Then, over longer time, the signal should decrease to some reasonably constant value.

   c. Restore the original setpoint temperature of 300º C and allow at least ½-hour for thermal stabilization.

   d. Observe the displayed detector signal to determine if it is now within the acceptable range between 10 and 30 display units, verify all gas flow rates, then continue to Step 8.

   e. If the displayed detector signal is still too great to continue, repeat the bakeout process. If repeated bakeout cycles fail to give the acceptable signal level, consult your 5890 service and user documentation for additional information. If necessary, then contact your local Hewlett-Packard Service representative.

8. To check signal noise, wander, and drift, perform the following steps:

   a. At the 5890, enter a Range value of '0' (zero) for Signal 1:

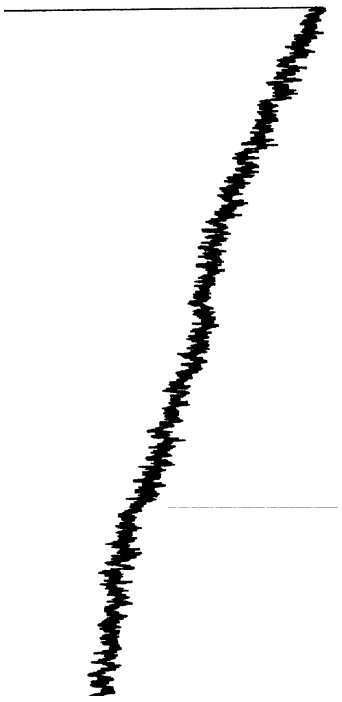
      ```
      [SIG 1] [RANGE ^( )] [0] [ENTER]
      ```

   b. At the 3396 Integrator, enter the following setpoint values:

      ```
      [ZERO] [5] [0] [ENTER]
      [ATT 2^] [0] [ENTER]
      [CHT SP] [1] [ENTER]
      ```

   c. Press [PLOT] to begin plotting the TCD signal.

   d. Allow plotting to continue for at least 30 minutes.



e. Using the method shown, measure the plot to check for the following:

- Noise over any given 1-minute portion of the plot should be < 6.5 mm in width (between the inner pair of lines). Up to 4 spikes are allowed in any given portion provided they do not exceed limits represented by the outer pair of lines.
- Wander over any given 5-minute portion of the plot should be < 22 mm in width.
- Drift over the full 30-minute plot should be < 43 mm in width.

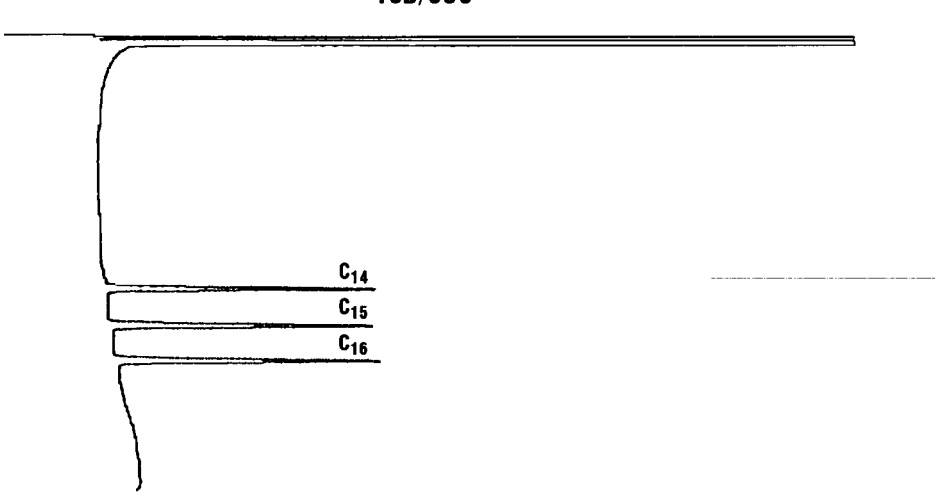
f. If these criteria are not met, repeat the test. If, after repeated testing, the criteria cannot be met, stop here and consult your 5890 service and user documentation for additional information. If necessary, then contact your local Hewlett-Packard Service representative.

9. To perform chemical checkout, do the following steps:

   a. At the 3396 Integrator, enter the following setpoint values:

```
[ZERO] [1] [0] [ENTER]
[ATT 2^] [7] [ENTER]
[CHT SP] [1] [ENTER]
[PK WD] [.] [0] [4] [ENTER]
[THRSH] [5] [ENTER]
[AR REJ] [1] [0] [0] [0] [0] [0] [ENTER]
[TIME] [8] [STOP]
```

   b. Inject 3 ul of the Checkout Sample and press [START] at the 5890 to begin the checkout run.



   c. The resulting chromatogram should appear similar to that shown in the figure above. The following criteria indicate successful completion of TCD chemical checkout:

      - Area counts for components labeled 'C14', 'C15', and 'C16' should each be > 1,000,000.
      - The area counts ratio calculated as 'C14'/'C16' should be 1.05 ± 0.05.

   d. If these criteria are not met, repeat the test. If, after repeated testing, the criteria cannot be met, consult your 5890 service and user documentation for additional information. If necessary, then contact your local Hewlett-Packard Service representative.

TCD NOISE TEST OVERLAY
(Use with HP 3396 ONLY)

Directions: Place each measured section of overlay over last 30 minutcs of the test plot (baseline) and follow directions below for each. Use the dashed centerlines to align the lines with the edges of the test plots.

Noise: Move 1 minute noise measurement down the length of the baseline to assure width of baselines no greater than measured width (inner two lines). You may have up to 4 spikes in any given 1 minute as long as they remain within the two outer lines.

Wander: Move 5 minute wander measurement down the length of the baseline to assure that baseline wander remains within lines for any given 5 minutes. Lines must be parallel to the edge of the plot paper.

Drift: Place drift measurement over the last 30 minutes of baseline. Baseline drift must remain within the lines. Lines must be parallel to the edge of the plot paper.

Integrator Settings:

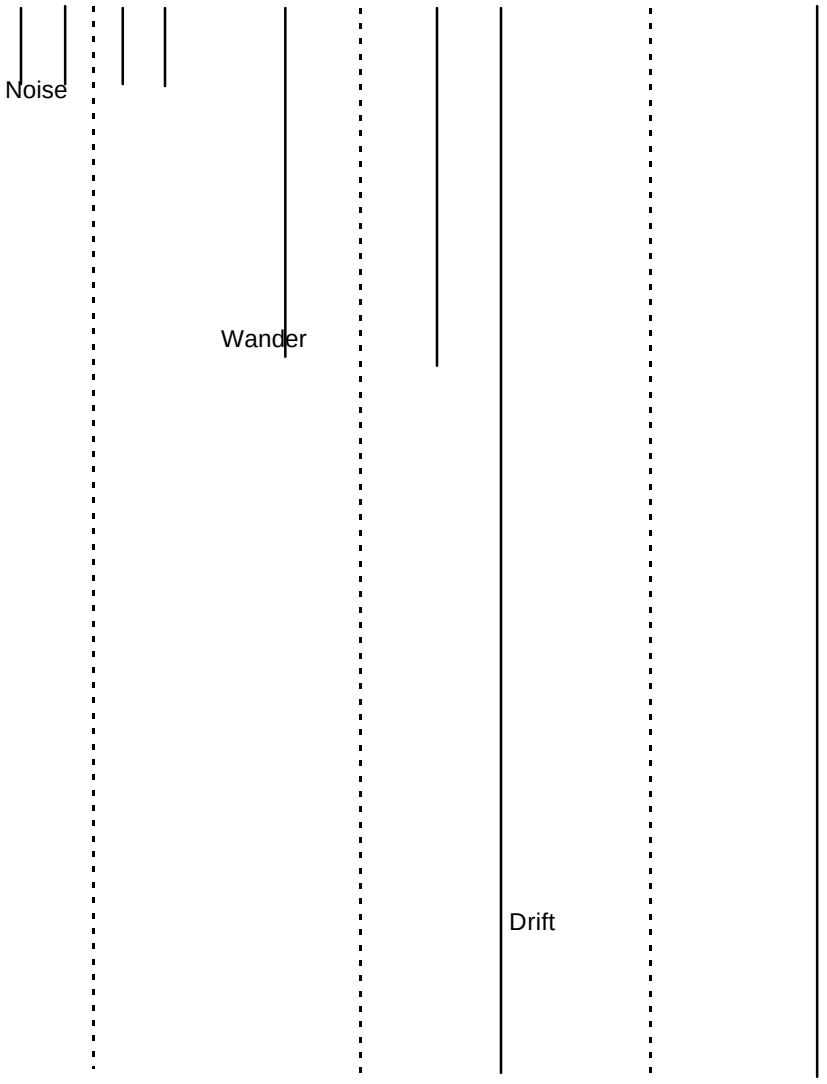
Zero:	50
Attn:	0
Cht Sp :	1
Pk Wd:	0.04
Thrsh:	0
Ar Rej:	0

5890 Settings:

Range: 0

Offset: Below 30 µV

Test Time: 30 Minutes



Standard Operating Procedure

Title: ECD (Electron Capture Detector) Checkout Using a Packed or Purged Packed Column Inlet

Procedure Number: _____

Revision Number: _____

Replaces Revision: _____

Approved by: _____

Reviewed by: _____

Effective Date: _____

Company stamp:

Distribution List

- _____
- _____
- _____
- _____
- _____
- _____

Scope

Use the following procedure to verify proper ECD operation with either inlet type.

Parts/Equipment Referenced

- GC Instrument Performance Evaluation ("Checkout") Column, P/N 19095S-100
- ECD Performance Evaluation ("Checkout") Sample, P/N 18713-60040
- HP3396A Integrator
- 10-µl syringe, P/N 9301-0810 or equivalent
- 5890A-to-3396A Analog Signal Cable, P/N 35900-60610
- 5890A-to-3396A Remote Start Cable, P/N 03394-60560
- Soap Film Flowmeter (500-ml volume, multi-range), P/N 9301-0981 or equivalent
- TCD/NPD/ECD Flow-Measurement Adapter, P/N 5020-8231
- Chromatographic Grade Purity Gas: Nitrogen as Carrier and Make-Up

Related SOPs

- Inlet Septum, Installation, SOP No. _____
- Liner / Insert, Installation, SOP No. _____
- Column Adapter / Make-Up Gas Fitting, Installation, SOP No. _____
- Column, Installation, SOP No. _____

Procedure

WARNING: If the unit has been previously in operation, areas may be hot enough to cause serious burns. Switch off heated zones and the oven and allow the unit sufficient time to cool.

WARNING: Ensure the ECD is properly vented before beginning this procedure

1. Following SOPs cited above in the Related SOPs section:
   a. At the Packed Column Inlet, install a new septum, a capillary column adapter and new liner, and the Checkout Column.

b. At the ECD, install the make-up gas adapter and remaining end of the Checkout Column.

2. Between the 5890 and 3396

   a. Install the analog signal cable from the 'Signal 1' output receptacle on the 5890 and the 3396 Integrator.

   b. Install the remote start cable from the 'Remote' receptacle on the 5890 and the 3396 Integrator.

   c. At the 5890, assign Signal 1 to the ECD to be evaluated:

      ```
      [SIG 1] [A] { or [B] }
      ```

   d. Switch on the integrator.

3. To insure carrier flow through the column during oven and zone heat-up, the following flow rates should be established (more precise values will be set later in the procedure):

   a. Insert the flow-measurement adapter fully onto the ECD vent tube, connect the flowmeter, and establish a carrier flow rate (Nitrogen) of 25 - 35 ml/min.

   b. Also establish a Make-Up gas flow rate (Nitrogen) of 25 - 35 ml/min.

   c. If using the Purge Packed Inlet, note that some have an adjustable septum purge flow. In such cases, connect the flowmeter to the septum purge vent and adjust flow rate (Nitrogen) to 1 - 2 ml/min.

4. Set the following oven and heated zone setpoint values:

   ```
   [INJ A TEMP] { or [INJ B TEMP] } [2] [0] [0] [ENTER]
   [DET A TEMP] { or [DET B TEMP] } [3] [0] [0] [ENTER]
   ```

   Oven Program (isothermal):

NOTE: Depending upon the given 5890, the '[INIT VALUE]' key referenced in the following steps may be labeled '[INIT TEMP]'. In such cases, the '[FINAL VALUE]' key is labeled '[FINAL TEMP]'.

```
[INIT VALUE] [1] [6] [0] [ENTER]
[INIT TIME] [3] [ENTER]
[RATE] [0] [ENTER]
[FINAL VALUE] [0] [ENTER]
[FINAL TIME] [0] [ENTER]
[OVEN TEMP] [ON]
```

Allow the unit at least ½-hour to thermally stabilize.

5. After thermal stabilization, set the following flow rate values:

   a. As measured at the ECD vent tube using the flowmeter and flow-measurement adapter, set:

      ```
      Carrier (Nitrogen) at 30 ± 1 ml/min.
      ECD Make-Up (Nitrogen) at 30 ± 1 ml/min.
      ```

   b. After all flow rates are set, remove the flowmeter and flow-measurement adapter, then turn on all gas flows.

   c. If the Purge Packed Inlet is being used, as measured at the inlet vent using the flowmeter, verify: Septum Purge flow rate (Nitrogen) at 1 - 2 ml/min.

6. At the 5890, turn on the ECD electrically and display its signal output to determine its background offset:

   ```
   [DET] [A] { or [B] } [ON]
   [SIG 1] [SIG 1]
   ```

   A stable value < 30 display units is acceptable criteria to continue to Step 7. Typically, the value may be much higher indicating system bakeout is necessary:

   a. Reset oven and inlet temperatures to the following values:

      ```
      [INJ A TEMP] { or [INJ B TEMP] } [2] [5] [0] [ENTER]
      [INIT VALUE] [2] [5] [0] [ENTER]
      ```

   b. Monitor the displayed detector signal: it should rise quickly to some higher value as the 5890 heats to the higher temperatures. Then, over longer time, the signal should decrease to some reasonably constant value.

   c. Restore the original setpoint temperatures specified in Step 4 and allow the unit at least ½-hour to thermally stabilize. Observe the displayed detector signal to determine if it is now within the acceptable range below 30 display units: If so, verify all gas flow rates, then continue to Step 7.

   d. If the displayed detector signal is still too great to continue, repeat the bakeout process. If repeated bakeout cycles fail to give the acceptable signal level, consult your 5890 service and user documentation for additional information. If necessary, then contact your local Hewlett-Packard Service representative.

7. Note the displayed offset value from Step 6: this value will be used in the next series of steps. To check signal noise, wander, and drift, perform the following:

   a. At the 5890, enter a Range value of '0' (zero) for Signal 1:

      ```
      [SIG 1] [RANGE ^( )] [0] [ENTER]
      ```

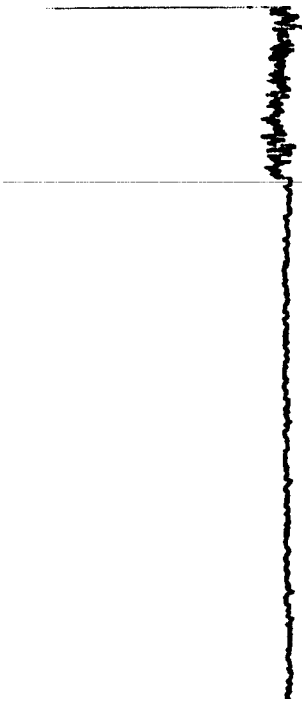
   b. At the 3396 Integrator, enter the following setpoint values:

      ```
      [ATT 2^] [-] [2] [ENTER]
      [ZERO] [5] [0] [ENTER]
      [CHT SP] [1] [ENTER]
      ```

   c. Press [PLOT] to begin plotting the ECD signal.

   d. At an elapsed time of 2 minutes, make the following key stroke entries at the 3396 Integrator and allow plotting to continue for at least 10 minutes longer:

      ```
      [ATT 2^] [0] [ENTER]
      [ZERO] [ENTER]
      ```



e. Using the method shown, measure the plot to check for the following:

- Maximum allowed noise, in mm, in the first 2-minute portion of the plot, is calculated from the previously-noted displayed offset value:

  `Max. noise (in mm) = 0.415 x observed signal offset`

  If observed noise is within acceptable limits, note the actual value for use later in Step 8.

- Wander and/or drift is acceptable if, over the 10-minute portion of the plot, the plot remains on the page.

f. If these criteria are not met, repeat the test. If, after repeated testing, the criteria cannot be met, stop here and consult your 5890 service and user documentation for additional information. If necessary, then contact your local Hewlett-Packard Service representative.

8. To perform chemical checkout, do the following steps:

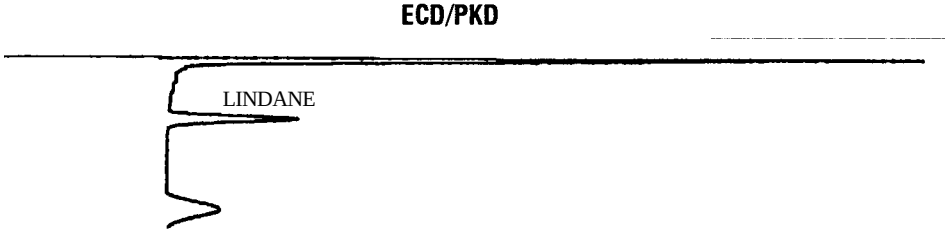
a. At the 5890, enter a Range value of '2' for Signal 1:

```
[SIG 1] [RANGE ^( )] [2] [ENTER]
```

b. At the 3396 Integrator, enter the following setpoint values:

```
[ZERO] [1] [0] [ENTER]
[ATT 2^] [5] [ENTER]
[CHT SP] [1] [ENTER]
[PK WD] [.] [1] [6] [ENTER]
[THRSH] [5] [ENTER]
[AR REJ] [0] [ENTER]
[TIME] [3] [STOP]
```

c. Inject 1 µl of the Checkout Sample and press [START] at the 5890 to begin the checkout run.



d. The resulting chromatogram should appear similar to that shown in the figure above. The following criteria indicate successful completion of ECD chemical checkout:

   - Minimum allowed area counts for the component labeled 'lindane' is calulated from the observed noise as:

     `Min. area counts = 21,300 x observed noise (in mm)`

e. If this criteria is not met, repeat the test. If, after repeated testing, the criteria cannot be met, consult your 5890 service and user documentation for additional information. If necessary, then contact your local Hewlett-Packard Service representative.

NOTE: After completion of the performance checkout procedure, it may be necessary to test for possible radioactive contamination of the checkout column, makeup gas adapter, ECD vent tube, etc. Commonly called a "wipe test", it should be performed in accordance with your local ECD usage requirements.

Standard Operating Procedure

Title: ECD (Electron Capture Detector) Checkout Using a Split-Only or Split/Splitless (in Split Mode) Capillary Column Inlet

Procedure Number: _____

Revision Number: _____

Replaces Revision: _____

Approved by: _____

Reviewed by: _____

Effective Date: _____

Company stamp:

Distribution List

- _____
- _____
- _____
- _____
- _____
- _____

Scope

Use the following procedure to verify proper ECD operation with either inlet type.

Parts/Equipment Referenced

- GC Instrument Performance Evaluation ("Checkout") Column, P/N 19095S-100
- ECD Performance Evaluation ("Checkout") Sample, P/N 18713-60040
- HP3396A Integrator
- 10-ul syringe, P/N 9301-0810 or equivalent
- 5890A-to-3396A Analog Signal Cable, P/N 35900-60610
- 5890A-to-3396A Remote Start Cable, P/N 03394-60560
- Soap Film Flowmeter (500-ml volume, multi-range), P/N 9301-0981 or equivalent
- TCD/NPD/ECD Flow-Measurement Adapter, P/N 5020-8231
- Chromatographic Grade Purity Gas: Nitrogen as Carrier and Make-Up

Related SOPs

- Inlet Septum, Installation, SOP No. _____
- Liner / Insert, Installation, SOP No. _____
- Column Adapter / Make-Up Gas Fitting, Installation, SOP No. _____
- Column, Installation, SOP No. _____

Procedure

WARNING: If the unit has been previously in operation, areas may be hot enough to cause serious burns. Switch off heated zones and the oven and allow the unit sufficient time to cool.

WARNING: Ensure the ECD is properly vented before beginning this procedure.

1. Following SOPs cited above in the Related SOPs section:
   a. At the Capillary Column Inlet, install a new septum, a new split liner and O-ring seal, and the Checkout Column.

b. At the ECD, install the make-up gas adapter and remaining end of the Checkout Column.

2. Between the 5890 and 3396:

   a. Install the analog signal cable from the 'Signal 1' output receptacle on the 5890 and the 3396 Integrator.

   b. Install the remote start cable from the 'Remote' receptacle on the 5890 and the 3396 Integrator.

   c. At the 5890, assign Signal 1 to the ECD to be evaluated:

      ```
      [SIG 1] [A] { or [B] }
      ```

   d. Switch on the integrator.

3. To insure carrier flow through the column during oven and zone heat-up, the following flow rates should be established (more precise values will be set later in the procedure):

   a. Insert the flow-measurement adapter fully onto the ECD vent tube, connect the flowmeter, and establish a carrier flow rate (Nitrogen) of 10 - 20 ml/min.

   b. Also establish a Make-Up gas flow rate (Nitrogen) of 55 - 65 ml/min.

   c. Connect the flowmeter to the inlet septum purge vent and adjust flow rate (Nitrogen) to 4 - 6 ml/min.

   d. Connect the flowmeter to the inlet split flow vent and adjust flow rate (Nitrogen) to 55 - 65 ml/min.

4. If a Split/Splitless Capillary Inlet is being used, verify the inlet is operating in Split Mode (continuous Purge On condition). At the 5890, enter the following three key sequences:

   ```
   [PURGE] [A] { or [B] } [ON]
   [PURGE] [A] { or [B] } [TIME] [OFF] [0] [ENTER]
   [PURGE] [A] { or [B] } [TIME] [ON] [0] [ENTER]
   ```

5. Set the following oven and heated zone setpoint values:

   ```
   [INJ A TEMP] { or [INJ B TEMP] } [2] [0] [0] [ENTER]
   [DET A TEMP] { or [DET B TEMP] } [3] [0] [0] [ENTER]
   ```

   Oven Program (isothermal):

NOTE: Depending upon the given 5890, the '[INIT VALUE]' key referenced in the following steps may be labeled '[INIT TEMP]'. In such cases, the '[FINAL VALUE]' key is labeled '[FINAL TEMP]'.

```
[INIT VALUE] [1] [7] [0] [ENTER]
[INIT TIME] [3] [.] [5] [ENTER]
 [RATE] [0] [ENTER]
[FINAL VALUE] [0] [ENTER]
[FINAL TIME] [0] [ENTER]
[OVEN TEMP] [ON]
```

Allow the unit at least ½-hour to thermally stabilize.

6. After thermal stabilization, set the following flow rate values:

   a. As measured at the ECD vent tube using the flowmeter and flow-measurement adapter, set:

   ```
   Carrier (Nitrogen) at 15 ± 1 ml/min.
   ECD Make-Up (Nitrogen) at 60 ± 2 ml/min.
   ```

   b. After all flow rates are set, remove the flowmeter and flow-measurement adapter, then turn on all gas flows.

   c. As measured at the inlet vents using the flowmeter, set:

   ```
   Split flow rate (Nitrogen) at 60 ± 2 ml/min.
   Septum Purge flow rate (Nitrogen) at 5 ± 1 ml/min.
   ```

7. At the 5890, turn on the ECD electrically and display its signal output to determine its background offset:

   ```
   [DET] [A] { or [B] } [ON]
   [SIG 1] [SIG 1]
   ```

   A stable value < 30 display units is acceptable criteria to continue to Step 8. Typically, the value may be much higher indicating system bakeout is necessary:

   a. Reset oven and inlet temperatures to the following values:

   ```
   [INJ A TEMP] { or [INJ B TEMP] } [2] [5] [0] [ENTER]
   [INIT VALUE] [2] [5] [0] [ENTER]
   ```

   b. Monitor the displayed detector signal: it should rise quickly to some higher value as the 5890 heats to the higher temperatures. Then, over longer time, the signal should decrease to some reasonably constant value.

   c. Restore the original setpoint temperatures specified in Step 4 and allow the unit at least ½-hour to thermally stabilize. Observe the displayed detector signal to determine if it is now within the acceptable range below 30 display units: if so, verify all gas flow rates, then continue to Step 8.

d. If the displayed detector signal is still too great to continue, repeat the bakeout process. If repeated bakeout cycles fail to give the acceptable signal level, consult your 5890 service and user documentation for additional information. If necessary, then contact your local Hewlett-Packard Service representative.

8. Note the displayed offset value from Step 7: the value will be used in the next series of steps. To check signal noise, wander, and drift, perform the following:

   a. At the 5890, enter a Range value of '0' (zero) for Signal 1:

      ```
      [SIG 1] [RANGE ^( )] [0] [ENTER]
      ```

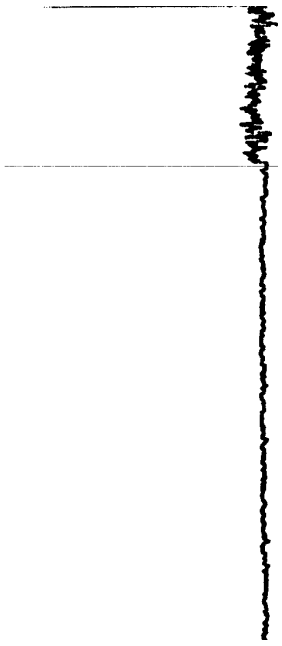
   b. At the 3396 Integrator, enter the following setpoint values:

      ```
      [ATT 2^] [-] [2] [ENTER]
      [ZERO] [5] [0] [ENTER]
      [CHT SP] [1] [ENTER]
      ```

   c. Press [PLOT] to begin plotting the ECD signal.

   d. At an elapsed time of 2 minutes, make the following key stroke entries at the 3396 Integrator and allow plotting to continue for at least 10 minutes longer:

      ```
      [ATT 2^] [0] [ENTER]
      [ZERO] [ENTER]
      ```



e. Using the method shown, measure the plot to check for the following:

- Maximum allowed noise, in mm, in the first 2-minute portion of the plot, is calculated from the previously-noted displayed offset value:

  `Max. noise (in mm) = 0.415 x observed signal offset`

  If observed noise is within acceptable limits, note the actual value for use later in Step 9.

- Wander and/or drift is acceptable if, over the 10-minute portion of the plot, the plot remains on the page.

f. If these criteria are not met, repeat the test. If, after repeated testing, the criteria cannot be met, stop here and consult your 5890 service and user documentation for additional information. If necessary, then contact your local Hewlett-Packard Service representative.

9. To perform chemical checkout, do the following steps:

   a. At the 3396 Integrator, enter the following setpoint values:

```
[ZERO] [1] [0] [ENTER]
[ATT 2^] [5] [ENTER]
[CHT SP] [1] [ENTER]
[PK WD] [.] [0] [4] [ENTER]
[THRSH] [5] [ENTER]
[AR REJ] [0] [ENTER]
[TIME] [3] [.] [5] [STOP]
```

   b. Inject 1 ul of the Checkout Sample and press [START] at the 5890 to begin the checkout run.

   c. The resulting chromatogram should appear similar to that shown in the figure above. The following criteria indicate successful completion of ECD chemical checkout:

      - Minimum allowed area counts for the component labeled 'lindane' is calulated from the observed noise as:

        `Min. area counts = 11,400 x observed noise (in mm)`

   d. If this criteria is not met, repeat the test. If, after repeated testing, the criteria cannot be met, consult your 5890 service and user documentation for additional information. If necessary, then contact your local Hewlett-Packard Service representative.

NOTE: After completion of the performance checkout procedure, it may be necessary to test for possible radioactive contamination of the checkout column, makeup gas adapter, ECD vent tube, etc. Commonly called a "wipe test", it should be performed in accordance with your local ECD usage requirements

Standard Operating Procedure

Title: ECD (Electron Capture Detector) Checkout Using a Cool On-Column Capillary Column Inlet

Procedure Number:_____

Revision Number:_____

Replaces Revision:_____

Approved by:_____

Reviewed by:_____

Effective Date:_____

Company stamp:

Distribution List

- _____
- _____
- _____
- _____
- _____
- _____

Scope

Use the following procedure to verify proper ECD operation with this inlet type.

Parts/Equipment Referenced

- GC Instrument Performance Evaluation ("Checkout") Column, P/N 19095S-100
- ECD Performance Evaluation ("Checkout") Sample, P/N 18713-60040
- HP3396A Integrator
- 10-ul syringe, P/N 9301-0810 or equivalent
- 5890A-to-3396A Analog Signal Cable, P/N 35900-60610
- 5890A-to-3396A Remote Start Cable, P/N 03394-60560
- Soap Film Flowmeter (500-ml volume, multi-range), P/N 9301-0981 or equivalent
- TCD/NPD/ECD Flow-Measurement Adapter, P/N 5020-8231
- Chromatographic Grade Purity Gas: Nitrogen as Carrier and Make-Up

Related SOPs

- Inlet Septum, Installation, SOP No. _____
- Liner / Insert, Installation, SOP No. _____
- Column Adapter / Make-Up Gas Fitting, Installation, SOP No. _____
- Column, Installation, SOP No. _____

Procedure

WARNING: If the unit has been previously in operation, areas may be hot enough to cause serious burns. Switch off heated zones and the oven and allow the unit sufficient time to cool.

WARNING: Ensure the ECD is properly vented before beginning this procedure.

1. Following SOPs cited above in the Related SOPs section:
    a. At the Cool On-Column Capillary Column Inlet, install a new septum, a Series 530-u capillary column alignment guide, and the Checkout Column.

b. At the ECD, install the make-up gas adapter and remaining end of the Checkout Column.

2. Between the 5890 and 3396:

   a. Install the analog signal cable from the 'Signal 1' output receptacle on the 5890 and the 3396 Integrator.

   b. Install the remote start cable from the 'Remote' receptacle on the 5890 and the 3396 Integrator.

   c. At the 5890, assign Signal 1 to the ECD to be evaluated:

      ```
      [SIG 1] [A] { or [B] }
      ```

   d. Switch on the integrator.

3. To insure carrier flow through the column during oven and zone heat-up, the following flow rates should be established (more precise values will be set later in the procedure):

   a. Insert the flow-measurement adapter fully onto the ECD vent tube, connect the flowmeter, and establish a carrier flow rate (Nitrogen) of 10 - 20 ml/min.

   b. Also establish a Make-Up gas flow rate (Nitrogen) of 55 - 65 ml/min.

   c. If applicable, connect the flowmeter to the inlet septum purge vent and adjust flow rate to (Nitrogen) 7 - 9 ml/min.

4. Set the following oven and heated zone setpoint values:

   If applicable, enable Oven Tracking:

   ```
   [gold] [DET A TEMP] [ON]
   [DET A TEMP] { or [DET B TEMP] } [3] [0] [0] [ENTER]
   ```

   Oven Program (isothermal):

NOTE: Depending upon the given 5890, the '[INIT VALUE]' key referenced in the following steps may be labeled '[INIT TEMP]'. In such cases, the '[FINAL VALUE]' key is labeled '[FINAL TEMP]'.

```
[INIT VALUE] [1] [7] [0] [ENTER]
[INIT TIME] [3] [.] [5] [ENTER]
[RATE] [0] [ENTER]
[FINAL VALUE] [0] [ENTER]
[FINAL TIME] [0] [ENTER]
[OVEN TEMP] [ON]
```

d. Allow the unit at least ½-hour to thermally stabilize.

5. After thermal stabilization, set the following flow rate values:

   a. As measured at the ECD vent tube using the flowmeter and flow-measurement adapter, set:

      ```
      Carrier (Nitrogen) at 15 ± 1 ml/min.
      ECD Make-Up (Nitrogen) at 60 ± 2 ml/min.
      ```

   b. After all flow rates are set, remove the flowmeter and flow-measurement adapter, then turn on all gas flows.

   c. If applicable, as measured at the inlet septum purge vent using the flowmeter, verify: Septum Purge flow rate (Nitrogen) at 8 ± 1 ml/min.

6. At the 5890, turn on the ECD electrically and display its signal output to determine its background offset:

   ```
   [DET] [A] { or [B] } [ON]
   [SIG 1] [SIG 1]
   ```

   A stable value < 30 display units is acceptable criteria to continue to Step 7. Typically, the value may be much higher indicating system bakeout is necessary:

   a. Reset oven and inlet temperatures to the following values:

      ```
      [INJ A TEMP] { or [INJ B TEMP] } [2] [5] [0] [ENTER]
      [INIT VALUE] [2] [5] [0] [ENTER]
      ```

   b. Monitor the displayed detector signal: it should rise quickly to some higher value as the 5890 heats to the higher temperatures. Then, over longer time, the signal should decrease to some reasonably constant value.

   c. Restore the original setpoint temperatures specified in Step 4 and allow the unit at least ½-hour to thermally stabilize. Observe the displayed detector signal to determine if it is now within the acceptable range below 30 display units: if so, verify all gas flow rates, then continue to Step 7.

   d. If the displayed detector signal is still too great to continue, repeat the bakeout process. If repeated bakeout cycles fail to give the acceptable signal level, consult your 5890 service and user documentation for additional information. If necessary, then contact your local Hewlett-Packard Service representative.

7. Note the displayed offset value from Step 6: the value will be used in the next series of steps. To check signal noise, wander, and drift, perform the following:

   a. At the 5890, enter a Range value of '0' (zero) for Signal 1:

      ```
      [SIG 1] [RANGE ^( )] [0] [ENTER]
      ```

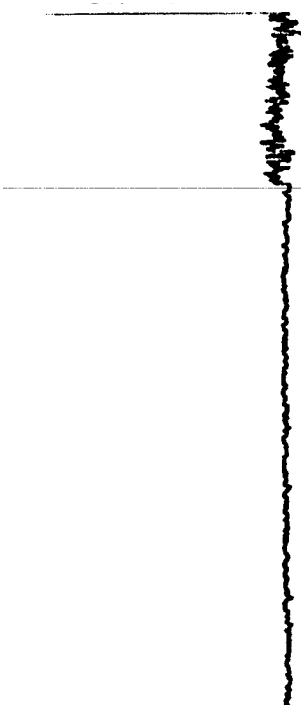
   b. At the 3396 Integrator, enter the following setpoint values:

      ```
      [ATT 2^] [-] [2] [ENTER]
      [ZERO] [5] [0] [ENTER]
      [CHT SP] [1] [ENTER]
      ```

   c. Press [PLOT] to begin plotting the ECD signal.

   d. At an elapsed time of 2 minutes, make the following key stroke entries at the 3396 Integrator and allow plotting to continue for at least 10 minutes longer:

      ```
      [ATT 2^] [0] [ENTER]
      [ZERO] [ENTER]
      ```



e. Using the method shown, measure the plot to check for the following:

- Maximum allowed noise, in mm, in the first 2-minute portion of the plot, is calculated from the previously-noted displayed offset value:

  ```
  Max. noise (in mm) = 0.415 x observed signal offset
  ```

  If observed noise is within acceptable limits, note the actual value for use later in Step 8.

- Wander and/or drift is acceptable if, over the 10-minute portion of the plot, the plot remains on the page.

f. If these criteria are not met, repeat the test. If, after repeated testing, the criteria cannot be met, stop here and consult your 5890 service and user documentation for additional information. If necessary, then contact your local Hewlett-Packard Service representative.

8. To perform chemical checkout, do the following steps:

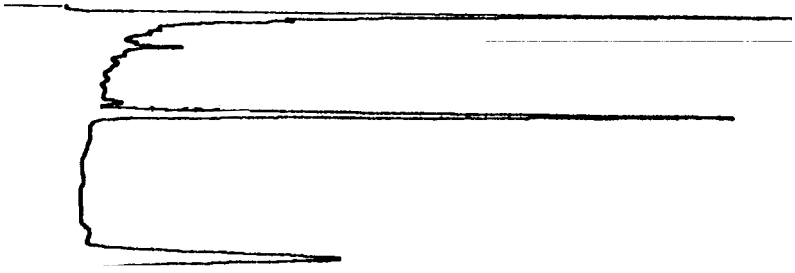
a. At the 5890, enter a Range value of '2' for Signal 1:

```
[SIG 1] [RANGE ^( )] [2] [ENTER]
```

b. At the 3396 Integrator, enter the following setpoint values:

```
[ZERO] [1] [0] [ENTER]
[ATT 2^] [5] [ENTER]
[CHT SP] [1] [ENTER]
[PK WD] [.] [0] [4] [ENTER]
[THRSH] [5] [ENTER]
[AR REJ] [0] [ENTER]
[TIME] [3] [.] [5] [STOP]
```

c. Inject 1 ul of the Checkout Sample and press [START] at the 5890 to begin the checkout run.



d. The resulting chromatogram should appear similar to that shown in the figure above. The following criteria indicate successful completion of ECD chemical checkout:

- Minimum allowed area counts for the component labeled 'lindane' is calulated from the observed noise as:

  `Min. area counts = 14,200 x observed noise (in mm)`

e. If this criteria is not met, repeat the test. If, after repeated testing, the criteria cannot be met, consult your 5890 service and user documentation for additional information. If necessary, then contact your local Hewlett-Packard Service representative.

NOTE: After completion of the performance checkout procedure, it may be necessary to test for possible radioactive contamination of the checkout column, makeup gas adapter, ECD vent tube, etc. Commonly called a "wipe test", it should be performed in accordance with your local ECD usage requirements.

Standard Operating Procedure

Title: FPD (Flame Photometric Detector) Checkout Using a Packed or Purged Packed Column Inlet

Procedure Number: _____

Revision Number: _____

Replaces Revision: _____

Approved by: _____

Reviewed by: _____

Effective Date: _____

Company stamp:

Distribution List

- _____
- _____
- _____
- _____
- _____
- _____

Scope

Use the following procedure to verify proper FPD operation with either inlet type.

Parts/Equipment Referenced

- GC Instrument Performance Evaluation ("Checkout") Column, P/N 19095S-100
- FPD Performance Evaluation ("Checkout") Sample, P/N 19305-60580
- HP3396A Integrator
- 10-ul syringe, P/N 9301-0810 or equivalent
- 5890A-to-3396A Analog Signal Cable, P/N 35900-60610
- 5890A-to-3396A Remote Start Cable, P/N 03394-60560
- Soap Film Flowmeter (500-ml volume, multi-range), P/N 9301-0981 or equivalent
- Chromatographic Grade Purity Gases: Air and Hydrogen to support the FPD, and Nitrogen as Carrier

Related SOPs

- Inlet Septum, Installation, SOP No. _____
- Liner / Insert, Installation, SOP No. _____
- Column Adapter / Make-Up Gas Fitting, Installation, SOP No. _____
- Column, Installation, SOP No. _____
- FPD Sulfur / Phosphorus Mode Filter, Installation, SOP No. _____

Procedure

WARNING: If the unit has been previously in operation, areas may be hot enough to cause serious burns. Switch off heated zones and the oven and allow the unit sufficient time to cool.

1. Following SOPs cited above in the Related SOPs section:
   a. At the Packed Column Inlet, install a new septum, a capillary column adapter and new liner, and the Checkout Column.

b. At the FPD, install the make-up gas adapter and remaining end of the Checkout Column.

c. If not already configured for the desired FPD operating mode, Sulfur or Phosphorus, install the appropriate filter.

2. Between the 5890 and 3396:

a. Install the analog signal cable from the 'Signal 1' output receptacle on the 5890 and the 3396 Integrator.

b. Install the remote start cable from the 'Remote' receptacle on the 5890 and the 3396 Integrator.

c. At the 5890, assign Signal 1 to the FPD to be evaluated:

```
[SIG 1] [A] { or [B] }
```

d. Switch on the integrator.

3. To insure carrier flow through the column during oven and zone heat-up, the following flow rates should be established (more precise values will be set later in the procedure):

a. At the FPD exhaust vent, connect the flowmeter and establish a carrier flow rate (Nitrogen) of 15 - 25 ml/min.

b. If using the Purge Packed Inlet, note that some have an adjustable septum purge flow. In such cases, connect the flowmeter to the septum purge vent and adjust flow rate (Helium) to 1 - 2 ml/min.

4. Set the following oven and heated zone setpoint values:

```
[INJ A TEMP] { or [INJ B TEMP] } [2] [0] [0] [ENTER]
[DET B TEMP] [2] [0] [0] [ENTER]
```

Oven Program (1 ramp):

NOTE: Depending upon the given 5890, the '(INIT VALUE)' key referenced in the following steps may be labeled '(INIT TEMP)'. In such cases, the '(FINAL VALUE)' key is labeled '(FINAL TEMP)'.

```
[INIT VALUE] [1] [1] [0] [ENTER]
[INIT TIME] [0] [ENTER]
[RATE] [1] [0] [ENTER]
[FINAL VALUE] [1] [7] [0] [ENTER]
[FINAL TIME] [3] [ENTER]
[OVEN TEMP] [ON]
```

Allow the unit at least ½-hour to thermally stabilize.

5. After thermal stabilization, set the following flow rate values:

WARNING: Due to possible explosion hazard within the flowmeter, under no circumstances should both air and hydrogen be allowed to flow at the same time.

a. As measured at the FPD exhaust vent using the flowmeter, set:

```
Carrier (Nitrogen) at 20 ± 1 ml/min.
FPD (Hydrogen) at 75 ± 2 ml/min.
FPD (Air) at 100 ± 3 ml/min.
FPD Make-Up at 0 (zero) ml/min. (off)
```

b. After all flow rates are set, remove the flowmeter and then turn on all gas flows.

c. If the Purge Packed Inlet is being used, as measured at the inlet vent using the flowmeter, verify: Septum Purge flow rate (Nitrogen) at 1 - 2 ml/min.

6. At the 5890, turn on the FPD electrically and display its signal output to determine flame-off background offset:

```
[DET] [A] { or [B] } [ON]
[SIG 1] [SIG 1]
```

A stable value of 200 ± 60 display units is acceptable criteria to continue. Note the actual value for later reference. If an acceptable value is not attained, stop here and consult your 5890 service and user documentation for additional information. If necessary, then contact your local Hewlett-Packard Service representative.

7. Ignite the FPD flame. Typically, a 'pop' sound is heard as the flame ignites. If necessary, ignition can be achieved by turning off Hydrogen flow and then depressing and holding in the ignitor button while restoring Hydrogen flow.

a. Observe the FPD signal at the display. Successful flame ignition typically results in a permanent increase in signal output. If no appreciable increase is observed, perform the next check, otherwise continue to Step 8.

b. Hold a cool, shiny, metal object, such as a chrome-plated wrench, at the FPD exhaust vent. Any observed water condensation signifies the flame is indeed ignited.

If no increase in signal output has been observed even though the flame is ignited, consult your 5890 service and user documentation for additional information. If necessary, then contact your local Hewlett-Packard Service representative.

c. If the flame has not ignited, recheck all flow rates and attempt ignition again. If the flame cannot be lit even though flow rates are correct, consult your 5890 service and user documentation for additional information. If necessary, then contact your local Hewlett-Packard Service representative.

8. With the flame lit, observe the FPD signal at the display. This is the flame-on background offset. A stable value not greater than 120 display units above the previously-determined flame-off offset value is acceptable criteria to continue to Step 9.

   Typically, the value may be > 120 display units greater indicating system bakeout is necessary:

   a. Reset oven and heated zone temperatures to the following values:

      ```
      [INJ A TEMP] { or [INJ B TEMP] } [2] [5] [0] [ENTER]
      [DET B TEMP] [2] [5] [0] [ENTER]
      [INIT VALUE] [2] [2] [5] [ENTER]
      ```

   b. Monitor the displayed detector signal: it should rise quickly to some higher value as the 5890 heats to the higher temperatures. Then, over longer time, the signal should decrease to some reasonably constant value. Maintain the unit at these higher temperatures for at least 1 hour.

   c. Restore the original setpoint temperatures specified in Step 4 and allow the unit at least ½-hour to thermally stabilize. Observe the displayed detector signal to determine if it is now stable at a value not greater than 120 display units above the previously-determined flame-off offset value. If so, this is acceptable criteria to continue: verify all gas flow rates, then continue to Step 9.

   d. If the displayed detector signal is still too great to continue, repeat the bakeout process. If repeated bakeout cycles fail to give the acceptable signal level, consult your 5890 service and user documentation for additional information. If necessary, then contact your local Hewlett-Packard Service representative.

9. To check signal noise, wander, and drift, for either Sulfur or Phosphorus mode, perform the following steps:

   a. At the 5890, enter a Range value of '5' for Signal 1:

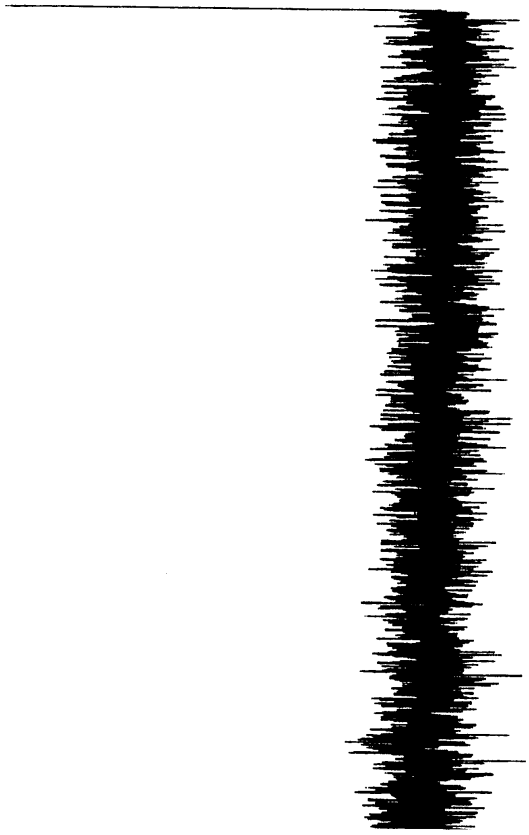
      ```
      [SIG 1] [RANGE ^( )] [5] [ENTER]
      ```

   b. At the 3396 Integrator, enter the following setpoint values:

      ```
      [ATT 2^] [0] [ENTER]
      [ZERO] [5] [0] [ENTER]
      [CHT SP] [1] [ENTER]
      ```

c. Press (PLOT) to begin plotting the FPD signal.

d. Allow plotting to continue for at least 5 minutes.



e. Using the method shown, measure the plot to check for the following:

- Noise, Wander, and Drift over any given 5-minute portion of the plot should be < 32.4 mm in width.
- No more than two noise spikes are to be allowed in any given 1-minute portion of the plot.

f. If these criteria are not met, repeat the test. If, after repeated testing, the criteria cannot be met, stop here and consult your 5890 service and user documentation for additional information. If necessary, then contact your local Hewlett-Packard Service representative.

10. To perform chemical checkout, do the following steps:

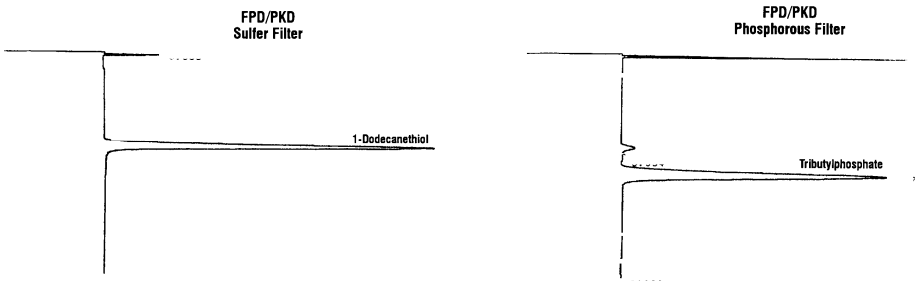
a. At the 3396 Integrator, enter the following setpoint values:

```
[ZERO] [1] [0] [ENTER]
[ATT 2^] [9] [ENTER]
[CHT SP] [1] [ENTER]
[PK WD] [.] [1] [6] [ENTER]
[THRSH] [2] [ENTER]
[AR REJ] [0] [ENTER]
[TIME] [7] [STOP]
```

b. At the 3396 Integrator, enter the following key sequence to disable automatic tangent skimming:

```
[TIME] [0] [INTG()] [4] [ENTER]
```

c. Inject 1 ul of the Checkout Sample and press (START) at the 5890 to begin the checkout run.



d. Depending upon whether the FPD is in Sulfur or Phosphorus mode, the resulting chromatogram and report should appear similar to one of those shown in Figure 2. The following criteria indicate successful completion of FPD chemical checkout:

- In Sulfur mode, area counts for the peak labeled '1-dodecanethiol' should be > 2,400,000; the peak labeled 'tributylphosphate' have negligible area counts.

- In Phosphorus mode, area counts for the peak labeled 'tributylphosphate' should be > 6,900,000; the peak labeled '1-dodecanethiol' have negligible area counts.

e. If these criteria are not met, repeat the test. If, after repeated testing, the criteria cannot be met, consult your 5890 service and user documentation for additional information. If necessary, then contact your local Hewlett-Packard Service representative.

Standard Operating Procedure

Title: FPD (Flame Photometric Detector) Checkout Using a Split-Only or Split/Splitless (in Split Mode) Capillary Column Inlet

Procedure Number: _____

Revision Number: _____

Replaces Revision: _____

Approved by: _____

Reviewed by: _____

Effective Date: _____

Company stamp:

Distribution List

- _____
- _____
- _____
- _____
- _____
- _____

Scope

Use the following procedure to verify proper FPD operation with either inlet type operating in split mode.

Parts/Equipment Referenced

- HP-1 Column, P/N 19095Z-121
- FPD Performance Evaluation ("Checkout") Sample, P/N 19305-60580
- HP3396A Integrator
- 10-ul syringe, P/N 9301-0810 or equivalent
- 5890A-to-3396A Analog Signal Cable, P/N 35900-60610
- 5890A-to-3396A Remote Start Cable, P/N 03394-60560
- Soap Film Flowmeter (500-ml volume, multi-range), P/N 9301-0981 or equivalent
- Chromatographic Grade Purity Gases: Air and Hydrogen to support the FPD, and Nitrogen as Carrier

Related SOPs

- Inlet Septum, Installation, SOP No. _____
- Liner / Insert, Installation, SOP No. _____
- Column Adapter / Make-Up Gas Fitting, Installation, SOP No. _____
- Column, Installation, SOP No. _____

Procedure

WARNING: If the unit has been previously in operation, areas may be hot enough to cause serious burns. Switch off heated zones and the oven and allow the unit sufficient time to cool.

1. Following SOPs cited above in the related SOPs section:
   a. At the Capillary Column Inlet, install a new septum, a new split liner and O-ring seal, and the HP-1 Column.
   b. At the FPD, install the make-up gas adapter and remaining end of the Checkout Column.

c. If not already configured for the desired FPD operating mode, Sulfur or Phosphorus, install the appropriate filter.

2. Between the 5890 and 3396:

   a. Install the analog signal cable from the 'Signal 1' output receptacle on the 5890 and the 3396 Integrator.

   b. Install the remote start cable from the 'Remote' receptacle on the 5890 and the 3396 Integrator.

   c. At the 5890, assign Signal 1 to the FPD to be evaluated:

      ```
      [SIG 1] [A] { or [B] }
      ```

   d. Switch on the integrator.

3. To insure carrier flow through the column during oven and zone heat-up, the following flow rates should be established (more precise values will be set later in the procedure):

   a. At the FPD exhaust vent, connect the flowmeter and establish a carrier flow rate (Nitrogen) of 15 - 25 ml/min.

   b. Connect the flowmeter to the inlet septum purge vent and adjust flow rate (Nitrogen) to 4 - 6 ml/min.

   c. Connect the flowmeter to the inlet split flow vent and adjust the flow rate (Nitrogen) to 175 - 225 ml/min.

4. If a Split/Splitless Capillary Inlet is being used, verify the inlet is operating in Split Mode (continuous Purge On condition). At the 5890, enter the following three key sequences:

   ```
   [PURGE] [A] { or [B] } [ON]
   [PURGE] [A] { or [B] } [TIME] [OFF] [0] [ENTER]
   [PURGE] [A] { or [B] } [TIME] [ON] [0] [ENTER]
   ```

5. Set the following oven and heated zone setpoint values:

   ```
   [INJ A TEMP] { or [INJ B TEMP] } [2] [0] [0] [ENTER]
   [DET B TEMP] [2] [0] [0] [ENTER]
   ```

Oven Program (1 ramp):

NOTE: Depending upon the given 5890, the '[INIT VALUE]' key referenced in the following steps may be labeled '[INIT TEMP]'. In such cases, the '[FINAL VALUE]' key is labeled '[FINAL TEMP]'.

```
[INIT VALUE] [1] [1] [0] [ENTER]
[INIT TIME] [0] [ENTER]
[RATE] [1] [0] [ENTER]
[FINAL VALUE] [1] [7] [0] [ENTER]
[FINAL TIME] [3] [ENTER]
[OVEN TEMP] [ON]
```

Allow the unit at least ½-hour to thermally stabilize.

6. After thermal stabilization, set the following flow rate values:

WARNING: Due to possible explosion hazard within the flowmeter, under no circumstances should both air and hydrogen be allowed to flow at the same time.

a. As measured at the FPD exhaust vent using the flowmeter, set:

```
Carrier (Nitrogen) at 20 ± 1 ml/min.
FPD (Hydrogen) at 75 ± 2 ml/min.
FPD (Air) at 100 ± 3 ml/min.
FPD Make-Up at 0 (zero) ml/min. (off)
```

b. After all flow rates are set, remove the flowmeter and then turn on all gas flows.

c. As measured at the inlet vents using the flowmeter, set:

```
Split flow rate (Nitrogen) at 200 ± 20 ml/min.
Septum Purge flow rate (Nitrogen) at 5 ± 1 ml/min.
```

7. At the 5890, turn on the FPD electrically and display its signal output to determine flame-off background offset:

```
[DET] [A] { or [B] } [ON]
[SIG 1] [SIG 1]
```

A stable value of 200 ± 60 display units is acceptable criteria to continue. Note the actual value for later reference. If an acceptable value is not attained, stop here and consult your 5890 service and user documentation for additional information. If necessary, then contact your local Hewlett-Packard Service representative.

8. Ignite the FPD flame. Typically, a 'pop' sound is heard as the flame ignites. If necessary, ignition can be achieved by turning off Hydrogen flow and then depressing and holding in the ignitor button while restoring Hydrogen flow.

    a. Observe the FPD signal at the display. Successful flame ignition typically results in a permanent increase in signal output. If no appreciable increase is observed, perform the next check, otherwise continue to Step 9.

    b. Hold a cool, shiny, metal object, such as a chrome-plated wrench, at the FPD exhaust vent. Any observed water condensation signifies the flame is indeed ignited.

    If no increase in signal output has been observed even though the flame is ignited, consult your 5890 service and user documentation for additional information. If necessary, then contact your local Hewlett-Packard Service representative.

    c. If the flame has not ignited, recheck all flow rates and attempt ignition again. If the flame cannot be lit even though flow rates are correct, consult your 5890 service and user documentation for additional information. If necessary, then contact your local Hewlett-Packard Service representative.

9. With the flame lit, observe the FPD signal at the display. This is the flame-on background offset. A stable value not greater than 120 display units above the previously-determined flame-off offset value is acceptable criteria to continue to Step 10.

    Typically, the value may be > 120 display units greater indicating system bakeout is necessary:

    a. Reset oven and heated zone temperatures to the following values:

    ```
    [INJ A TEMP] { or [INJ B TEMP] } [2] [5] [0] [ENTER]
    [DET B TEMP] [2] [5] [0] [ENTER]
    [INIT VALUE] [2] [2] [5] [ENTER]
    ```

    b. Monitor the displayed detector signal: it should rise quickly to some higher value as the 5890 heats to the higher temperatures. Then, over longer time, the signal should decrease to some reasonably constant value. Maintain the unit at these higher temperatures for at least 1 hour.

    c. Restore the original setpoint temperatures specified in Step 4 and allow the unit at least ½-hour to thermally stabilize. Observe the displayed detector signal to determine if it is now stable at a value not greater than 120 display units above the previously-determined flame-off offset value. If so, this is acceptable criteria to continue: verify all gas flow rates, then continue to Step 10.

d. If the displayed detector signal is still too great to continue, repeat the bakeout process. If repeated bakeout cycles fail to give the acceptable signal level, consult your 5890 service and user documentation for additional information. If necessary, then contact your local Hewlett-Packard Service representative.

10. To check signal noise, wander, and drift, for either Sulfur or Phosphorus mode, perform the following steps:

    a. At the 5890, enter a Range value of '5' for Signal 1:

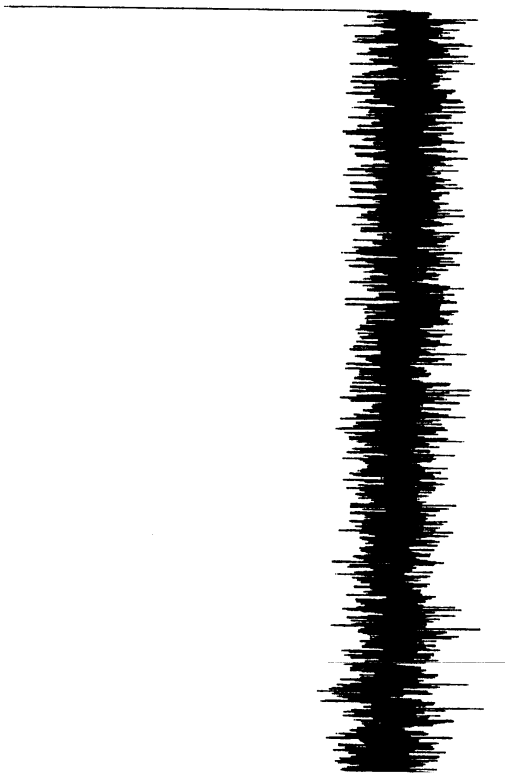
    ```
    [SIG 1] [RANGE ^( )] [5] [ENTER]
    ```

    b. At the 3396 Integrator, enter the following setpoint values:

    ```
    [ATT 2^] [0] [ENTER]
    [ZERO] [5] [0] [ENTER]
    [CHT SP] [1] [ENTER]
    ```

    c. Press [PLOT] to begin plotting the FPD signal.

d. Allow plotting to continue for at least 5 minutes.



f. If these criteria are not met, repeat the test. If, after repeated testing, the criteria cannot be met, stop here and consult your 5890 service and user documentation for additional information. If necessary, then contact your local Hewlett-Packard Service representative.

11. To perform chemical checkout, do the following steps:

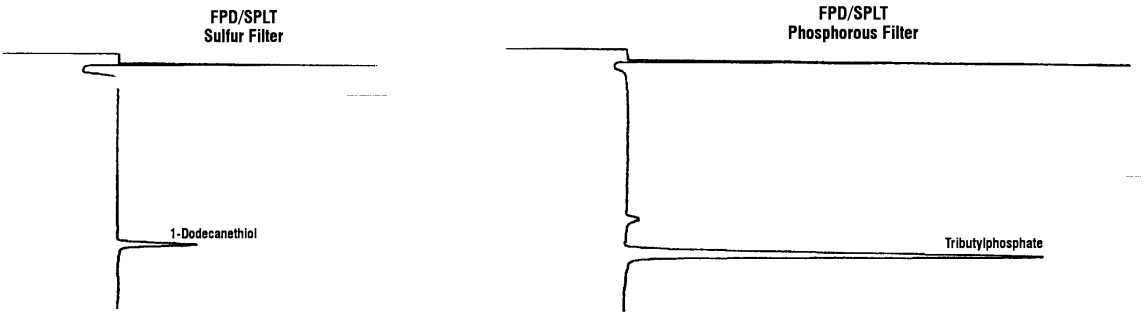
    a. At the 3396 Integrator, enter the following setpoint values:

```
[ZERO] [1] [0] [ENTER]
[ATT 2^] [5] [ENTER]
[CHT SP] [1] [ENTER]
[PK WD] [.] [1] [6] [ENTER]
[THRSH] [2] [ENTER]
[AR REJ] [0] [ENTER]
[TIME] [7] [STOP]
```

b. At the 3396 Integrator, enter the following key sequence to disable automatic tangent skimming:

```
[TIME] [0] [INTG()] [4] [ENTER]
```

c. Inject 2 ul of the Checkout Sample and press [START] at the 5890 to begin the checkout run.



d. Depending upon whether the FPD is in Sulfur or Phosphorus mode, the resulting chromatogram should appear similar to one of those shown in the figure above. The following criteria indicate successful completion of FPD chemical checkout:

- In Sulfur mode, area counts for the peak labeled '1-dodecanethiol' should be > 46,500; the peak labeled 'tributylphosphate' have negligible area counts.
- In Phosphorus mode, area counts for the peak labeled 'tributylphosphate' should be > 1,265,000; the peak labeled '1-dodecanethiol' have negligible area counts.

e. If these criteria are not met, repeat the test. If, after repeated testing, the criteria cannot be met, consult your 5890 service and user documentation for additional information. If necessary, then contact your local Hewlett-Packard Service representative.

Standard Operating Procedure

Title: FPD (Flame Photometric Detector) Checkout Using a Cool On-Column Capillary Column Inlet

Procedure Number: _____

Revision Number: _____

Replaces Revision: _____

Approved by: _____

Reviewed by: _____

Effective Date: _____

Company stamp:

Distribution List

- _____
- _____
- _____
- _____
- _____
- _____

Scope

Use the following procedure to verify proper FPD operation with this inlet type.

Parts/Equipment Referenced

- GC Instrument Performance Evaluation ("Checkout") Column, P/N 19095S-100
- FPD Performance Evaluation ("Checkout") Sample, P/N 19305-60580
- HP3396A Integrator
- 10-ul syringe, P/N 9301-0810 or equivalent
- 5890A-to-3396A Analog Signal Cable, P/N 35900-60610
- 5890A-to-3396A Remote Start Cable, P/N 03394-60560
- Soap Film Flowmeter (500-ml volume, multi-range), P/N 9301-0981 or equivalent
- Chromatographic Grade Purity Gases: Air and Hydrogen to support the FPD, and Nitrogen as Carrier

Related SOPs

- Inlet Septum, Installation, SOP No. _____
- Liner / Insert, Installation, SOP No. _____
- Column Adapter / Make-Up Gas Fitting, Installation, SOP No. _____
- Column, Installation, SOP No. _____

Procedure

WARNING: If the unit has been previously in operation, areas may be hot enough to cause serious burns. Switch off heated zones and the oven and allow the unit sufficient time to cool.

1. Following SOPs cited above in the Related SOPs section:
   a. At the Cool On-Column Capillary Column Inlet, install a new septum, a Series 530-u capillary column alignment guide, and the Checkout Column.
   b. At the FPD, install the make-up gas adapter and remaining end of the Checkout Column.
   c. If not already configured for the desired FPD operating mode, Sulfur or Phosphorus, install the appropriate filter.

2. Between the 5890 and 3396:

   a. Install the analog signal cable from the 'Signal 1' output receptacle on the 5890 and the 3396 Integrator.

   b. Install the remote start cable from the 'Remote' receptacle on the 5890 and the 3396 Integrator.

   c. At the 5890, assign Signal 1 to the FPD to be evaluated:

      ```
      [SIG 1] [A] { or [B] }
      ```

   d. Switch on the integrator.

3. To insure carrier flow through the column during oven and zone heat-up, the following flow rates should be established (more precise values will be set later in the procedure):

   a. At the FPD exhaust vent, connect the flowmeter and establish a carrier flow rate (Nitrogen) of 15 - 25 ml/min.

   b. If applicable, connect the flowmeter to the inlet septum purge vent and adjust flow rate (Nitrogen) to 4 - 6 ml/min.

4. Set the following oven and heated zone setpoint values:

   a. If applicable, enable Oven tracking:

      ```
      [gold] [DET A TEMP] [ON]
      [INJ A TEMP] { or [INJ B TEMP] } [ON]
      ```

   b. `[DET B TEMP] [2] [0] [0] [ENTER]`

   c. Oven Program (2 ramps):

NOTE: Depending upon the given 5890, the '[INIT VALUE]' key referenced in the following steps may be labeled '[INIT TEMP]'. In such cases, the '[FINAL VALUE]' key is labeled '[FINAL TEMP]'.

```
[INIT VALUE] [9] [0] [ENTER]
[INIT TIME] [1] [ENTER]
[RATE] [2] [0] [ENTER]
[FINAL VALUE] [1] [1] [0] [ENTER]
[FINAL TIME] [0] [ENTER]
[OVEN TEMP] [ON]
[RATE] [A] [1] [0] [ENTER]
[FINAL VALUE] [A] [1] [7] [0] [ENTER]
[FINAL TIME] [A] [3] [ENTER]
```

d. Allow the unit at least ½-hour to thermally stabilize.

5. After thermal stabilization, set the following flow rate values:

WARNING: Due to possible explosion hazard within the flowmeter, under no circumstances should both air and hydrogen be allowed to flow at the same time.

a. As measured at the FPD exhaust vent using the flowmeter, set:

```
Carrier (Nitrogen) at 20 ± 1 ml/min.
FPD (Hydrogen) at 75 ± 2 ml/min.
FPD (Air) at 100 ± 3 ml/min.
FPD Make-Up at 0 (zero) ml/min. (off)
```

b. After all flow rates are set, remove the flowmeter and then turn on all gas flows.

c. If applicable, as measured at the inlet vent using the flowmeter, verify:

```
Septum Purge flow rate (Nitrogen) at 5 ± 1 ml/min.
```

6. At the 5890, turn on the FPD electrically and display its signal output to determine flame-off background offset:

```
[DET] [A] { or [B] } [ON]
[SIG 1] [SIG 1]
```

A stable value of 200 ± 60 display units is acceptable criteria to continue. Note the actual value for later reference. If an acceptable value is not attained, stop here and consult your 5890 service and user documentation for additional information. If necessary, then contact your local Hewlett-Packard Service representative.

7. Ignite the FPD flame. Typically, a 'pop' sound is heard as the flame ignites. If necessary, ignition can be achieved by turning off Hydrogen flow and then depressing and holding in the ignitor button while restoring Hydrogen flow.

a. Observe the FPD signal at the display. Successful flame ignition typically results in a permanent increase in signal output. If no appreciable increase is observed, perform the next check, otherwise continue to Step 8.

b. Hold a cool, shiny, metal object, such as a chrome-plated wrench, at the FPD exhaust vent. Any observed water condensation signifies the flame is indeed ignited.

If no increase in signal output has been observed even though the flame is ignited, consult your 5890 service and user documentation for additional information. If necessary, then contact your local Hewlett-Packard Service representative.

c. If the flame has not ignited, recheck all flow rates and attempt ignition again. If the flame cannot be lit even though flow rates are correct, consult your 5890 service and user documentation for additional information. If necessary, then contact your local Hewlett-Packard Service representative.

8. With the flame lit, observe the FPD signal at the display. This is the flame-on background offset. A stable value not greater than 120 display units above the previously-determined flame-off offset value is acceptable criteria to continue to Step 9.

   Typically, the value may be > 120 display units greater indicating system bakeout is necessary:

   a. Reset oven and heated zone temperatures to the following values:

      ```
      [DET B TEMP] [2] [5] [0] [ENTER]
      [INIT VALUE] [2] [2] [5] [ENTER]
      ```

   b. Monitor the displayed detector signal: it should rise quickly to some higher value as the 5890 heats to the higher temperatures. Then, over longer time, the signal should decrease to some reasonably constant value. Maintain the unit at these higher temperatures for at least 1 hour.

   c. Restore the original setpoint temperatures specified in Step 4 and allow the unit at least ½-hour to thermally stabilize. Observe the displayed detector signal to determine if it is now stable at a value not greater than 120 display units above the previously-determined flame-off offset value. If so, this is acceptable criteria to continue: verify all gas flow rates, then continue to Step 9.

   d. If the displayed detector signal is still too great to continue, repeat the bakeout process. If repeated bakeout cycles fail to give the acceptable signal level, consult your 5890 service and user documentation for additional information. If necessary, then contact your local Hewlett-Packard Service representative.

9. To check signal noise, wander, and drift, for either Sulfur or Phosphorus mode, perform the following steps:

   a. At the 5890, enter a Range value of '5' for Signal 1:

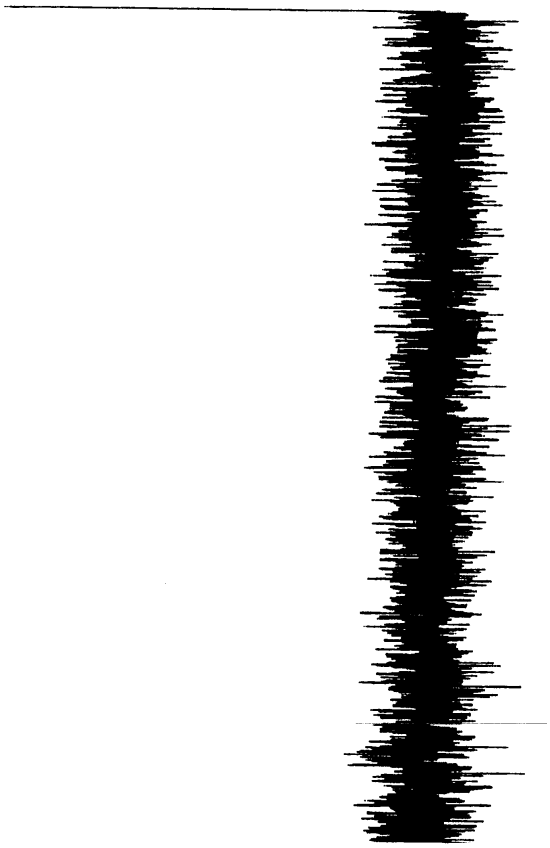
      ```
      [SIG 1] [RANGE ^( )] [5] [ENTER]
      ```

b. At the 3396 Integrator, enter the following setpoint values:

```
[ATT 2^] [0] [ENTER]
[ZERO] [5] [0] [ENTER]
[CHT SP] [1] [ENTER]
```

c. Press [PLOT] to begin plotting the FPD signal.

d. Allow plotting to continue for at least 5 minutes.



e. Using the method shown, measure the plot to check for the following:

- Noise, Wander, and Drift over any given 5-minute portion of the plot should be < 32.4 mm in width.

- No more than two noise spikes are to be allowed in any given 1-minute portion of the plot.

f. If these criteria are not met, repeat the test. If, after repeated testing, the criteria cannot be met, stop here and consult your 5890 service and user documentation for additional information. If necessary, then contact your local Hewlett-Packard Service representative.

10. To perform chemical checkout, do the following steps:

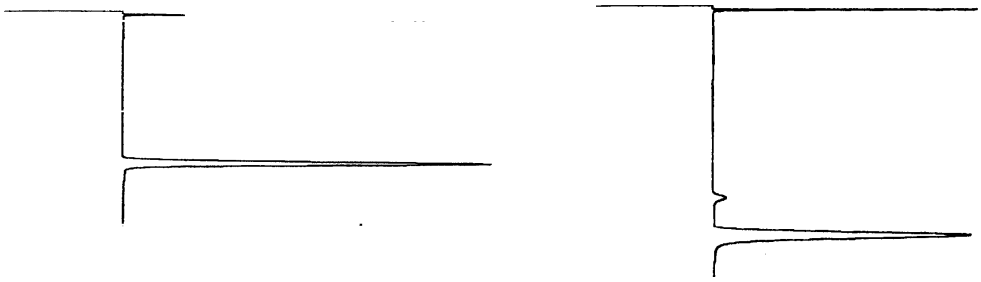
a. At the 3396 Integrator, enter the following setpoint values:

```
[ZERO] [1] [0] [ENTER]
[ATT 2^] [9] [ENTER]
[CHT SP] [1] [ENTER]
[PK WD] [.] [1] [6] [ENTER]
[THRSH] [2] [ENTER]
[AR REJ] [0] [ENTER]
[TIME] [7] [STOP]
```

b. At the 3396 Integrator, enter the following key sequence to disable automatic tangent skimming:

```
[TIME] [0] [INTG()] [4] [ENTER]
```

c. Inject 1 ul of the Checkout Sample and press [START] at the 5890 to begin the checkout run.



d. Depending upon whether the FPD is in Sulfur or Phosphorus mode, the resulting chromatogram and report should appear similar to one of those shown in Figure 2. The following criteria indicate successful completion of FPD chemical checkout:

- If in Sulfur mode, area counts for the peak labeled '1-dodecanethiol' should be > 2,400,000; the peak labeled 'tributylphosphate' have negligible area counts.
- If in Phosphorus mode, area counts for the peak labeled 'tributylphosphate' should be > 6,900,000; the peak labeled '1-dodecanethiol' have negligible area counts.

e. If these criteria are not met, repeat the test. If, after repeated testing, the criteria cannot be met, consult your 5890 service and user documentation for additional information. If necessary, then contact your local Hewlett-Packard Service representative.

FPD Noise Test Overlay
(Use with HP3396 ONLY)

Directions: Place overlay over noise baseline to make sure baseline width is a maximum of 32.4 mm.

Integrator Settings:

Zero: 50
Attn: 0
Cht Sp: 1.0
Pk Wd: 0.16
Thrsh: 0
Ar Rej: 0

5890 Settings:

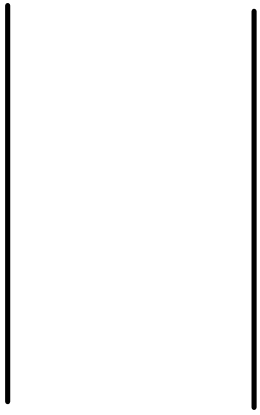
Range: 5

Offset:

No more then
120pA over
"flame off" offset

Test Time:

5 Minutes



Special Instructions:

32.4mm max. peak to peak
2 random spikes per cm maximum.
Sulfur and Phosphorus specs are same.
Noise, Wander, and Drift combined 32.4 mm max.

Standard Operating Procedure

Title: NPD (Nitrogen Phosphorus Detector) Checkout Using a Packed or Purged Packed Column Inlet

Procedure Number:_____

Revision Number:_____

Replaces Revision:_____

Approved by:_____

Reviewed by:_____

Effective Date:_____

Company stamp:

Distribution List

- _____
- _____
- _____
- _____
- _____
- _____

Scope

Use the following procedure to verify proper NPD operation with either inlet type.

Parts/Equipment Referenced

- GC Instrument Performance Evaluation ("Checkout") Column, P/N 19095S-100
- NPD Performance Evaluation ("Checkout") Sample, P/N 18789-60060
- HP3396A Integrator
- 10-ul syringe, P/N 9301-0810 or equivalent
- 5890A-to-3396A Analog Signal Cable, P/N 35900-60610
- 5890A-to-3396A Remote Start Cable, P/N 03394-60560
- Soap Film Flowmeter (500-ml volume, multi-range), P/N 9301-0981 or equivalent
- TCD/NPD/ECD Flow-Measurement Adapter, P/N 5020-8231
- Chromatographic Grade Purity Gases: Air and Hydrogen to support the NPD, Helium as Carrier and as Make-Up

Related SOPs

- Inlet Septum, Installation, SOP No. _____
- Liner / Insert, Installation, SOP No. _____
- Column Adapter / Make-Up Gas Fitting, Installation, SOP No. _____
- Column, Installation, SOP No. _____

Procedure

WARNING: If the unit has been previously in operation, areas may be hot enough to cause serious burns. Switch off heated zones and the oven and allow the unit sufficient time to cool.

1. Following SOPs cited above in the Related SOPs section:

    a. At the Packed Column Inlet, install a new septum, a capillary column adapter and new liner, and the Checkout Column.

    b. At the NPD, install an 0.011-inch Capillary/Series 530-µ jet, capillary column adapter, and remaining end of the Checkout Column.

c. If needed, install also a new NPD Collector.

2. Between the 5890 and 3396:

   a. Install the analog signal cable from the 'Signal 1' output receptacle on the 5890 and the 3396 Integrator.

   b. Install the remote start cable from the 'Remote' receptacle on the 5890 and the 3396 Integrator.

   c. At the 5890, assign Signal 1 to the NPD to be evaluated:

      ```
      [SIG 1] [A] { or [B] }
      ```

   d. Switch on the integrator.

3. To insure carrier flow through the column during oven and zone heat-up, the following flow rates should be established (more precise values will be set later in the procedure):

   a. If present, remove the protective cap found installed on the NPD exhaust vent at the top of the detector, then insert the flow-measurement adapter fully onto the NPD exhaust vent and connect the flowmeter. Establish a carrier flow rate (Helium) of 15 - 25 ml/min.

   b. If using the Purge Packed Inlet, note that some have an adjustable septum purge flow. In such cases, connect the flowmeter to the septum purge vent and adjust flow rate (Helium) to 1 - 2 ml/min.

4. Set the following oven and heated zone setpoint values:

   ```
   [INJ A TEMP] { or [INJ B TEMP] } [1] [7] [0] [ENTER]
   [DET A TEMP] { or [DET B TEMP] } [2] [2] [0] [ENTER]
   ```

   Oven Program (isothermal):

NOTE: Depending upon the given 5890, the '[INIT VALUE]' key referenced in the following steps may be labeled '[INIT TEMP]'. In such cases, the '[FINAL VALUE]' key is labeled '[FINAL TEMP]'.

```
[INIT VALUE] [1] [7] [0] [ENTER]
[INIT TIME] [3] [ENTER]
[RATE] [0] [ENTER]
[FINAL VALUE] [0] [ENTER]
[FINAL TIME] [0] [ENTER]
[OVEN TEMP] [ON]
```

Allow the unit at least ½-hour to thermally stabilize.

5. After thermal stabilization, set the following flow rate values:

WARNING: **Due to possible explosion hazard within the flowmeter, under no circumstances should both air and hydrogen be allowed to flow at the same time.**

a. As measured at the NPD using the flowmeter and TCD/NPD flow measurement adapter, set:

```
Carrier (Helium) at 20 ± 1 ml/min.
NPD (Hydrogen) at 4 ± 1 ml/min.
NPD (Air) at 90 ± 10 ml/min.
NPD Make-Up (Helium) at 10 ± 1 ml/min.
```

b. After all flow rates are set, remove the flowmeter and flow-measurement adapter, then turn on all gas flows.

c. If the Purge Packed Inlet is being used, as measured at the inlet vent using the flowmeter, verify: Septum Purge flow rate (Helium) at 1 - 2 ml/min.

6. At the 5890, turn on the NPD electrically and display its signal output to determine its background offset:

CAUTION: **To prevent possible damage to the Collector, verify the NPD Collector power control is set to '000' (zero, fully counterclockwise) BEFORE turning on the NPD.**

```
[DET] [A] { or [B] } [ON]
[SIG 1] [SIG 1]
```

A stable value < 2.0 display units is acceptable criteria to continue. If not met, stop here and consult your 5890 service and user documentation for additional information. If necessary, then contact your local Hewlett-Packard Service representative.

7. In powering up the NPD Collector, pay close attention to the displayed detector offset as power is increased:

a. Observing the NPD signal, increase power at the NPD Collector power control to a setting of '300' and wait at least 3 minutes.

- If the observed increase is not more than 1 (one) display unit, continue to Step 7. b.
- If the observed increase is > 1 display unit, reduce power at the NPD Collector power control to a setting of '000' and wait until the displayed offset decreases to < 2.0 display units. Then repeat this first step.

- If repeated cycles fail, stop here and consult your 5890 service and user documentation for additional information. If necessary, then contact your local Hewlett-Packard Service representative.

b. Increase power at the NPD Collector power control to a setting of '450' and wait at least 2 minutes.

c. While monitoring the signal display, cautiously increase the power setting in small steps until a sudden increase in signal is observed, signifying successful Collector ignition.

If no sudden and dramatic increase in signal output has been observed by the time a power setting of '800' is achieved, gas flow rates may be incorrect, a new Collector may be required, or service is required. Reduce the power setting to '000' (zero), recheck gas flow rates and replace the Collector. If necessary, consult your 5890 service and user documentation for additional information. If still necessary, then contact your local Hewlett-Packard Service representative.

8. Adjust the NPD Collector power control as needed to achieve a stable offset at any value between 20 and 30 display units. Offset may be considered stable if it is observed to be within ± 0.5 display units in any given 5-second time period.

If, after an extended time period, no stable offset can be achieved, gas flow rates may be incorrect, a new Collector may be required, or service is required. Reduce the power setting to '000' (zero), recheck gas flow rates and replace the Collector. If necessary, consult your 5890 service and user documentation for additional information. If still necessary, then contact your local Hewlett-Packard Service representative.

9. To check signal noise, wander, and drift, perform the following steps:

a. At the 5890, enter a Range value of '0' (zero) for Signal 1:

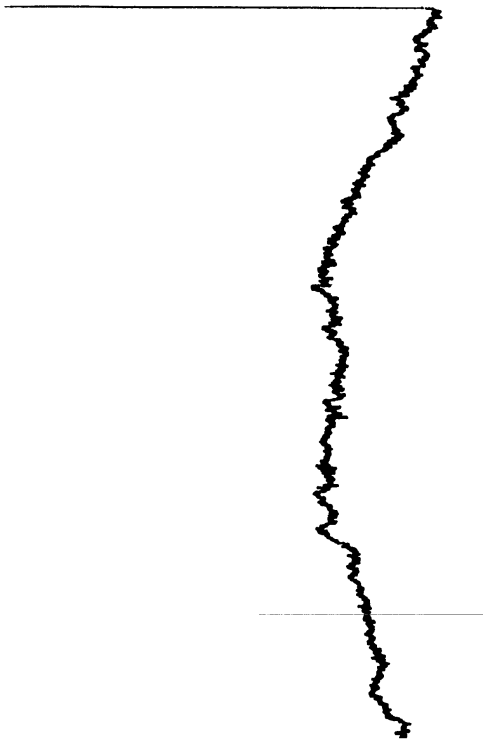
```
[SIG 1] [RANGE ^( )] [0] [ENTER]
```

b. At the 3396 Integrator, enter the following setpoint values:

```
[ATT 2^] [2] [ENTER]
[ZERO] [5] [0] [ENTER]
[CHT SP] [1] [ENTER]
```

c. Press [PLOT] to begin plotting the NPD signal.

d. Allow plotting to continue for at least 5 minutes.



e. Using the method shown, measure the plot to check for the following:

- Noise for any given 5-minute portion in the plot should be < 1 mm in width.
- Wander and drift for any given 5-minute portion in the plot should be remain within the confines of the page.

f. If these criteria are not met, repeat the test. If, after repeated testing, the criteria cannot be met, stop here and consult your 5890 service and user documentation for additional information. If necessary, then contact your local Hewlett-Packard Service representative.

10. To perform chemical checkout, do the following steps:

a. At the 3396 Integrator, enter the following setpoint values:

```
[ZERO] [2] [0] [ENTER]
[ATT 2^] [8] [ENTER]
[CHT SP] [1] [ENTER]
```

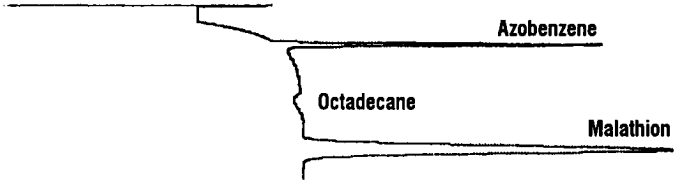
```
[PK WD] [.] [0] [4] [ENTER]
[THRSH] [6] [ENTER]
[AR REJ] [0] [ENTER]
[TIME] [3] [STOP]
```

b. At the 3396 Integrator, enter the following key sequences:

```
[TIME] [0] [INTG()] [9] [ENTER]
[TIME] [0] [.] [5] [INTG()] [-] [9] [ENTER]
[TIME] [1] [.] [4] [INTG()] [0] [ENTER]
```

c. Inject 3 µl of the Checkout Sample and press [START] at the 5890 to begin the checkout run.

NPD/PKD



d. The resulting chromatogram should appear similar to that shown in the figure above. The following criteria indicate successful completion of NPD chemical checkout:

- Area counts for the component labeled 'azobenzene' should be > 1,000,000.
- Area counts for the component labeled 'malathion' should be > 1,500,000.
- Area counts for the component labeled 'octadecane' should be < 0.25 of the area counts found for 'azobenzene' and < 0.12 of the area counts found for 'malathion'. A negative area count value for 'octadecane' is acceptable.

e. If these criteria are not met, repeat the test. Note that unacceptable area count values may be the result of incorrect gas flow rates, a new Collector may be required, or service is required. Reduce the power setting to '000' (zero), recheck gas flow rates and replace the Collector. If necessary, consult your 5890 service and user documentation for additional information. If still necessary, then contact your local Hewlett-Packard Service representative.

Standard Operating Procedure

Title: NPD (Nitrogen Phosphorus Detector) Checkout Using a Split-Only or Split/Splitless (in Split Mode) Capillary Column Inlet

Procedure Number: _____

Revision Number: _____

Replaces Revision: _____

Approved by: _____

Reviewed by: _____

Effective Date: _____

Company stamp:

Distribution List

- _____
- _____
- _____
- _____
- _____
- _____

Scope

Use the following procedure to verify proper NPD operation with either inlet type operating in split mode.

Parts/Equipment Referenced

- HP-1 Column, P/N 19095Z-121
- NPD Performance Evaluation ("Checkout") Sample, P/N 18789-60060
- HP3396A Integrator
- 10-ul syringe, P/N 9301-0810 or equivalent
- 5890A-to-3396A Analog Signal Cable, P/N 35900-60610
- 5890A-to-3396A Remote Start Cable, P/N 03394-60560
- Soap Film Flowmeter (500-ml volume, multi-range), P/N 9301-0981 or equivalent
- TCD/NPD/ECD Flow-Measurement Adapter, P/N 5020-8231
- Chromatographic Grade Purity Gases: Air and Hydrogen to support the NPD, Helium as Carrier and as Make-Up

Related SOPs

- Inlet Septum, Installation, SOP No. _____
- Liner / Insert, Installation, SOP No. _____
- Column Adapter / Make-Up Gas Fitting, Installation, SOP No. _____
- Column, Installation, SOP No. _____

Procedure

WARNING: If the unit has been previously in operation, areas may be hot enough to cause serious burns. Switch off heated zones and the oven and allow the unit sufficient time to cool.

1. Following SOPs cited above in the Related SOPs section:
    a. At the Capillary Column Inlet, install a new septum, a new split liner and O-ring seal, and the HP-1 Column.

b. At the NPD, install an 0.011-inch Capillary/Series 530-µ jet, capillary column adapter, and remaining end of the Checkout Column.

c. If needed, install also a new NPD Collector.

2. Between the 5890 and 3396:

a. Install the analog signal cable from the 'Signal 1' output receptacle on the 5890 and the 3396 Integrator.

b. Install the remote start cable from the 'Remote' receptacle on the 5890 and the 3396 Integrator.

c. At the 5890, assign Signal 1 to the NPD to be evaluated:

```
[SIG 1] [A] { or [B] }
```

d. Switch on the integrator.

3. To insure carrier flow through the column during oven and zone heat-up, the following flow rates should be established (more precise values will be set later in the procedure):

a. If present, remove the protective cap found installed on the NPD exhaust vent at the top of the detector, then insert the flow-measurement adapter fully onto the NPD exhaust vent and connect the flowmeter. Establish a carrier flow rate (Helium) of 15 - 25 ml/min.

b. Connect the flowmeter to the inlet septum purge vent and adjust flow rate (Helium) to 4 - 6 ml/min.

c. Connect the flowmeter to the inlet split flow vent and adjust flow rate (Helium) to 70 - 90 ml/min.

4. If a Split/Splitless Capillary Inlet is being used, verify the inlet is operating in Split Mode (continuous Purge On condition). At the 5890, enter the following three key sequences:

```
[PURGE] [A] { or [B] } [ON]
[PURGE] [A] { or [B] } [TIME] [OFF] [0] [ENTER]
[PURGE] [A] { or [B] } [TIME] [ON] [0] [ENTER]
```

5. Set the following oven and heated zone setpoint values:

```
[INJ A TEMP] { or [INJ B TEMP] } [2] [0] [0] [ENTER]
[DET A TEMP] { or [DET B TEMP] } [2] [2] [0] [ENTER]
```

Oven Program (isothermal):

NOTE: Depending upon the given 5890, the '[INIT VALUE]' key referenced in the following steps may be labeled '[INIT TEMP]'. In such cases, the '[FINAL VALUE]' key is labeled '[FINAL TEMP]'.

```
[INIT VALUE] [1] [8] [0] [ENTER]
[INIT TIME] [5] [ENTER]
[RATE] [0] [ENTER]
[FINAL VALUE] [0] [ENTER]
[FINAL TIME] [0] [ENTER]
[OVEN TEMP] [ON]
```

Allow the unit at least ½-hour to thermally stabilize.

6. After thermal stabilization, set the following flow rate values:

WARNING: Due to possible explosion hazard within the flowmeter, under no circumstances should both air and hydrogen be allowed to flow at the same time.

a. As measured at the NPD using the flowmeter and TCD/NPD flow measurement adapter, set:

```
Carrier (Helium) at 15 ± 1 ml/min.
NPD (Hydrogen) at 4 ± 1 ml/min.
NPD (Air) at 90 ± 10 ml/min.
NPD Make-Up (Helium) at 10 ± 1 ml/min.
```

b. After all flow rates are set, remove the flowmeter and flow-measurement adapter, then turn on all gas flows.

c. As measured at the inlet vents using the flowmeter, set: Split flow rate (Helium) at 80 ± 5 ml/min. Septum Purge flow rate (Helium) at 5 ± 1 ml/min.

7. At the 5890, turn on the NPD electrically and display its signal output to determine its background offset:

CAUTION: To prevent possible damage to the Collector, verify the NPD Collector power control is set to '000' (zero, fully counterclockwise) BEFORE turning on the NPD.

```
[DET] [A] { or [B] } [ON]
[SIG 1] [SIG 1]
```

A stable value < 2.0 display units is acceptable criteria to continue. If not met, stop here and consult your 5890 service and user documentation for additional information. If necessary, then contact your local Hewlett-Packard Service representative.

8. In powering up the NPD Collector, pay close attention to the displayed detector offset as power is increased:

   a. Observing the NPD signal, increase power at the NPD Collector power control to a setting of '300' and wait at least 3 minutes.

      - If the observed increase is not more than 1 (one) display unit, continue to Step 8.b.
      - If the observed increase is > 1 display unit, reduce power at the NPD Collector power control to a setting of '000' and wait until the displayed offset decreases to < 2.0 display units. Then repeat this first step.

        If repeated cycles fail, stop here and consult your 5890 service and user documentation for additional information. If necessary, then contact your local Hewlett-Packard Service representative.

   b. Increase power at the NPD Collector power control to a setting of '450' and wait at least 2 minutes.

   c. While monitoring the signal display, cautiously increase the power setting in small steps until a sudden increase in signal is observed, signifying successful Collector ignition.

      If no sudden and dramatic increase in signal output has been observed by the time a power setting of '800' is achieved, gas flow rates may be incorrect, a new Collector may be required, or service is required. Reduce the power setting to '000' (zero), recheck gas flow rates and replace the Collector. If necessary, consult your 5890 service and user documentation for additional information. If still necessary, then contact your local Hewlett-Packard Service representative.

9. Adjust the NPD Collector power control as needed to achieve a stable offset at any value between 20 and 30 display units. Offset may be considered stable if it is observed to be within ± 0.5 display units in any given 5-second time period.

   If, after an extended time period, no stable offset can be achieved, gas flow rates may be incorrect, a new Collector may be required, or service is required. Reduce the power setting to '000' (zero), recheck gas flow rates and replace the Collector. If necessary, consult your 5890 service and user documentation for additional information. If still necessary, then contact your local Hewlett-Packard Service representative.

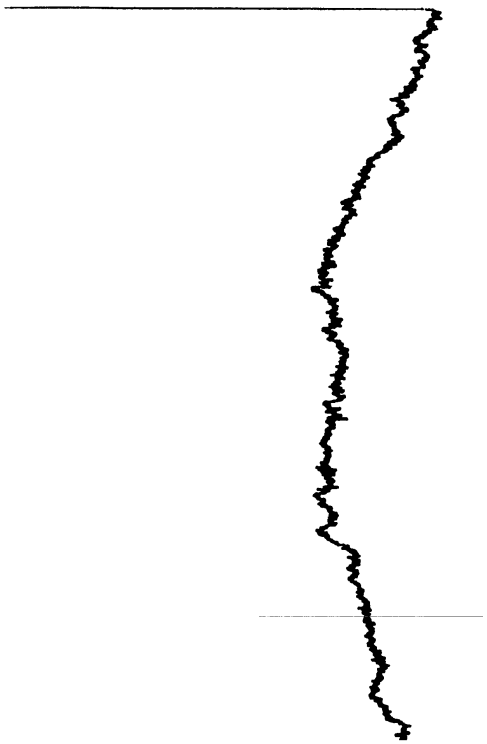
10. To check signal noise, wander, and drift, perform the following steps:
    a. At the 5890, enter a Range value of '0' (zero) for Signal 1:

       ```
       [SIG 1] [RANGE ^( )] [0] [ENTER]
       ```

    b. At the 3396 Integrator, enter the following setpoint values:

       ```
       [ATT 2^] [2] [ENTER]
       [ZERO] [5] [0] [ENTER]
       [CHT SP] [1] [ENTER]
       ```

    c. Press [PLOT] to begin plotting the NPD signal.
    d. Allow plotting to continue for at least 5 minutes.



e. Using the method shown, measure the plot to check for the following:

- Noise for any given 5-minute portion in the plot should be < 1 (one) mm in width.
- Wander and drift for any given 5-minute portion in the plot should be remain within the confines of the page.

f. If these criteria are not met, repeat the test. If, after repeated testing, the criteria cannot be met, stop here and consult your 5890 service and user documentation for additional information. If necessary, then contact your local Hewlett-Packard Service representative.

11. To perform chemical checkout, do the following steps:

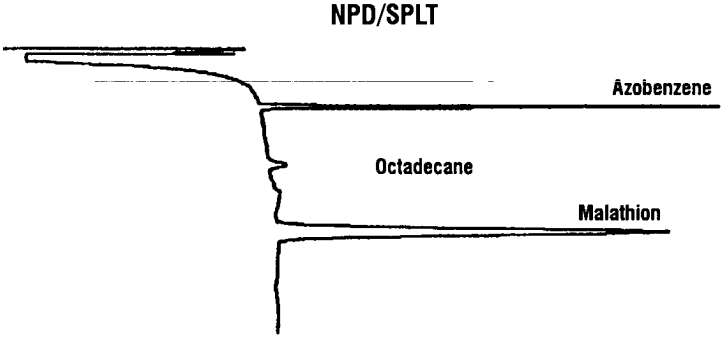
a. At the 3396 Integrator, enter the following setpoint values:

```
[ZERO] [2] [0] [ENTER]
[ATT 2^] [5] [ENTER]
[CHT SP] [1] [ENTER]
[PK WD] [.] [0] [4] [ENTER]
[THRSH] [4] [ENTER]
[AR REJ] [0] [ENTER]
[TIME] [5] [STOP]
```

b. At the 3396 Integrator, enter the following key sequences:

```
[TIME] [0] [INTG()] [9] [ENTER]
[TIME] [.] [7] [INTG()] [-] [9] [ENTER]
[TIME] [2] [.] [5] [INTG()] [0] [ENTER]
```

c. Inject 2 ul of the Checkout Sample and press [START] at the 5890 to begin the checkout run.



d. The resulting chromatogram should appear similar to that shown in the figure above. The following criteria indicate successful completion of NPD chemical checkout:

   - Area counts for the component labeled 'azobenzene' should be > 100,000.
   - Area counts for the component labeled 'malathion' should be > 150,000.
   - Area counts for the component labeled 'octadecane' should be < 0.25 of the area counts found for 'azobenzene' and < 0.12 of the area counts found for 'malathion'. A negative area count value for 'octadecane' is acceptable.

e. If these criteria are not met, repeat the test. Note that unacceptable area count values may be the result of incorrect gas flow rates, a new Collector may be required, or service is required. Reduce the power setting to '000' (zero), recheck gas flow rates and replace the Collector. If necessary, consult your 5890 service and user documentation for additional information. If still necessary, then contact your local Hewlett-Packard Service representative.

Standard Operating Procedure

Title: NPD (Nitrogen Phosphorus Detector) Checkout Using a Cool On-Column Capillary Column Inlet

Procedure Number: _____

Revision Number: _____

Replaces Revision: _____

Approved by: _____

Reviewed by: _____

Effective Date: _____

Company stamp:

Distribution List

- _____
- _____
- _____
- _____
- _____
- _____

Scope

Use the following procedure to verify proper NPD operation with this inlet type.

Parts/Equipment Referenced

- GC Instrument Performance Evaluation ("Checkout") Column, P/N 19095S-100
- NPD Performance Evaluation ("Checkout") Sample, P/N 18789-60060
- HP3396A Integrator
- 10-ul syringe, P/N 9301-0810 or equivalent
- 5890A-to-3396A Analog Signal Cable, P/N 35900-60610
- 5890A-to-3396A Remote Start Cable, P/N 03394-60560
- Soap Film Flowmeter (500-ml volume, multi-range), P/N 9301-0981 or equivalent
- TCD/NPD/ECD Flow-Measurement Adapter, P/N 5020-8231
- Chromatographic Grade Purity Gases: Air and Hydrogen to support the NPD, Helium as Carrier and as Make-Up

Related SOPs

- Inlet Septum, Installation, SOP No. _____
- Liner / Insert, Installation, SOP No. _____
- Column Adapter / Make-Up Gas Fitting, Installation, SOP No. _____
- Column, Installation, SOP No. _____

Procedure

WARNING: If the unit has been previously in operation, areas may be hot enough to cause serious burns. Switch off heated zones and the oven and allow the unit sufficient time to cool.

1. Following SOPs cited above in the Related SOPs section:
   a. At the Cool On-Column Capillary Column Inlet, install a new septum, a Series 530-µ capillary column alignment guide, and the Checkout Column.
   b. At the NPD, install an 0.011-inch Capillary/Series 530-µ jet, capillary column adapter, and remaining end of the Checkout Column.

c. If needed, install also a new NPD Collector.

2. Between the 5890 and 3396:

   a. Install the analog signal cable from the 'Signal 1' output receptacle on the 5890 and the 3396 Integrator.

   b. Install the remote start cable from the 'Remote' receptacle on the 5890 and the 3396 Integrator.

   c. At the 5890, assign Signal 1 to the NPD to be evaluated:

      ```
      [SIG 1] [A] { or [B] }
      ```

   d. Switch on the integrator.

3. To insure carrier flow through the column during oven and zone heat-up, the following flow rates should be established (more precise values will be set later in the procedure):

   a. If present, remove the protective cap found installed on the NPD exhaust vent at the top of the detector, then insert the flow-measurement adapter fully onto the NPD exhaust vent and connect the flowmeter. Establish a carrier flow rate (Helium) of 15 - 25 ml/min.

   b. If applicable, connect the flowmeter to the inlet septum purge vent and adjust flow rate to (Helium) 4 - 6 ml/min.

4. Set the following oven and heated zone setpoint values:

   a. If applicable, enable Oven Tracking:

      ```
      [gold] [DET A TEMP] [ON]
      [INJ A TEMP] { or [INJ B TEMP] } [ON]
      ```

   b. `[DET A TEMP] { or [DET B TEMP] } [2] [2] [0] [ENTER]`

   c. `Oven Program (isothermal):`

NOTE: Depending upon the given 5890, the '[INIT VALUE]' key referenced in the following steps may be labeled '[INIT TEMP]'. In such cases, the '[FINAL VALUE]' key is labeled '[FINAL TEMP]'.

```
[INIT VALUE] [1] [7] [0] [ENTER]
[INIT TIME] [5] [ENTER]
[RATE] [0] [ENTER]
[FINAL VALUE] [0] [ENTER]
[FINAL TIME] [0] [ENTER]
[OVEN TEMP] [ON]
```

d. Allow the unit at least ½-hour to thermally stabilize.

5. After thermal stabilization, set the following flow rate values:

WARNING: Due to possible explosion hazard within the flowmeter, under no circumstances should both air and hydrogen be allowed to flow at the same time.

a. As measured at the NPD using the flowmeter and TCD/NPD flow measurement adapter, set:

```
Carrier (Helium) at 15 ± 1 ml/min.
NPD (Hydrogen) at 4 ± 1 ml/min.
NPD (Air) at 90 ± 10 ml/min.
NPD Make-Up (Helium) at 10 ± 1 ml/min.
```

b. After all flow rates are set, remove the flowmeter and flow-measurement adapter, then turn on all gas flows.

c. If applicable, as measured at the inlet septum purge vent using the flowmeter, verify:

```
Septum Purge flow rate (Helium) at 5 ± 1 ml/min.
```

6. At the 5890, turn on the NPD electrically and display its signal output to determine its background offset:

CAUTION: To prevent possible damage to the Collector, verify the NPD Collector power control is set to '000' (zero, fully counterclockwise) BEFORE turning on the NPD.

```
[DET] [A] { or [B] } [ON]
[SIG 1]  [SIG 1]
```

A stable value < 2.0 display units is acceptable criteria to continue. If not met, stop here and consult your 5890 service and user documentation for additional information. If necessary, then contact your local Hewlett-Packard Service representative.

7. In powering up the NPD Collector, pay close attention to the displayed detector offset as power is increased:

a. Observing the NPD signal, increase power at the NPD Collector power control to a setting of '300' and wait at least 3 minutes.

- If the observed increase is not more than 1 (one) display unit, continue to Step 7. b.
- If the observed increase is > 1 display unit, reduce power at the NPD Collector power control to a setting of '000' and wait until the displayed offset decreases to < 2.0 display units. Then repeat this first step.

If repeated cycles fail, stop here and consult your 5890 service and user documentation for additional information. If necessary, then contact your local Hewlett-Packard Service representative.

b. Increase power at the NPD Collector power control to a setting of '450' and wait at least 2 minutes.

c. While monitoring the signal display, cautiously increase the power setting in small steps until a sudden increase in signal is observed, signifying successful Collector ignition.

If no sudden and dramatic increase in signal output has been observed by the time a power setting of '800' is achieved, gas flow rates may be incorrect, a new Collector may be required, or service is required. Reduce the power setting to '000' (zero), recheck gas flow rates and replace the Collector. If necessary, consult your 5890 service and user documentation for additional information. If still necessary, then contact your local Hewlett-Packard Service representative.

8. Adjust the NPD Collector power control as needed to achieve a stable offset at any value between 20 and 30 display units. Offset may be considered stable if it is observed to be within ± 0.5 display units in any given 5-second time period.

   If, after an extended time period, no stable offset can be achieved, gas flow rates may be incorrect, a new Collector may be required, or service is required. Reduce the power setting to '000' (zero), recheck gas flow rates and replace the Collector. If necessary, consult your 5890 service and user documentation for additional information. If still necessary, then contact your local Hewlett-Packard Service representative.

9. To check signal noise, wander, and drift, perform the following steps:

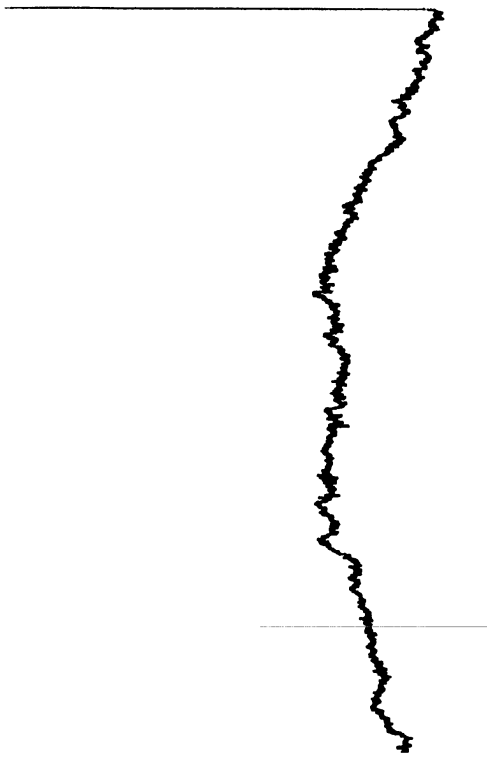
   a. At the 5890, enter a Range value of '0' (zero) for Signal 1:

      ```
      [SIG 1] [RANGE ^( )] [0] [ENTER]
      ```

   b. At the 3396 Integrator, enter the following setpoint values:

      ```
      [ATT 2^] [2] [ENTER]
      [ZERO] [5] [0] [ENTER]
      [CHT SP] [1] [ENTER]
      ```

   c. Press [PLOT] to begin plotting the NPD signal.



e. Using the method shown, measure the plot to check for the following:

- Noise for any given 5-minute portion in the plot should be < 1 mm in width.
- Wander and drift for any given 5-minute portion in the plot should be remain within the confines of the page.

f. If these criteria are not met, repeat the test. If, after repeated testing, the criteria cannot be met, stop here and consult your 5890 service and user documentation for additional information. If necessary, then contact your local Hewlett-Packard Service representative.

10. To perform chemical checkout, do the following steps:

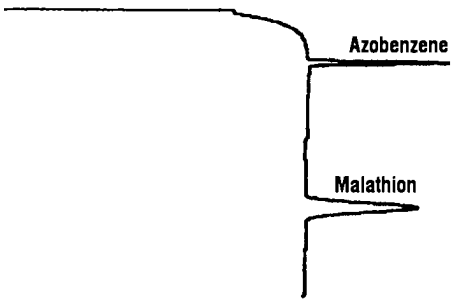
    a. At the 3396 Integrator, enter the following setpoint values:

    ```
    [ZERO] [2] [0] [ENTER]
    [ATT 2^] [8] [ENTER]
    [CHT SP] [1] [ENTER]
    [PK WD] [.] [0] [4] [ENTER]
    [THRSH] [6] [ENTER]
    [AR REJ] [0] [ENTER]
    [TIME] [5] [STOP]
    ```

    b. At the 3396 Integrator, enter the following key sequences:

    ```
    [TIME] [0] [INTG()] [9] [ENTER]
    [TIME] [.] [7] [INTG()] [-] [9] [ENTER]
    [TIME] [2] [.] [5] [INTG()] [0] [ENTER]
    ```

    c. Inject 3 ul of the Checkout Sample and press [START] at the 5890 to begin the checkout run.



    d. The resulting chromatogram should appear similar to that shown in the figure above. The following criteria indicate successful completion of NPD chemical checkout:

    - Area counts for the component labeled 'azobenzene' should be > 1,000,000.
    - Area counts for the component labeled 'malathion' should be > 1,500,000.

- Area counts for the component labeled 'octadecane' should be < 0.25 of the area counts found for 'azobenzene' and < 0.12 of the area counts found for 'malathion'. A negative area count value for 'octadecane' is acceptable.

e. If these criteria are not met, repeat the test. Note that unacceptable area count values may be the result of incorrect gas flow rates, a new Collector may be required, or service is required. Reduce the power setting to '000' (zero), recheck gas flow rates and replace the Collector. If necessary, consult your 5890 service and user documentation for additional information. If still necessary, then contact your local Hewlett-Packard Service representative.

NPD Noise Test Overlay

USE WITH HP3390, 3392, & 3396

Directions: The noise test for the NPD is a decision made on the part of the operator. Below you will find examples and guide lines to make the best decision.

NPD test examples not to scale)

Integrator settings:

Zero: 50
Attn: 2
Cht Sp: 1.0
Pk WD: 0.04
Thrsh: 0
Ar Rej: 0

5890 Settings

Range: 0

Offset:

Between 20-30pA

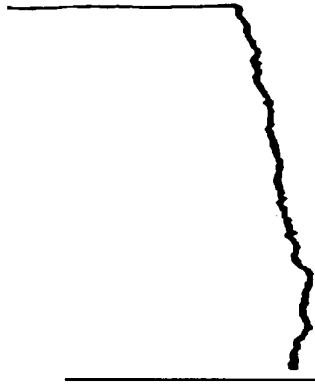
Test Time:

5 minutes

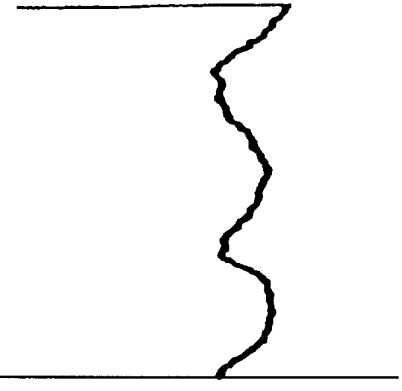
Special Requirements:

2mm max. baseline width (3396 only)
Plot must remain on paper
Slope cannot be too steep
Last 5 minutes of plot is section to be evaluated

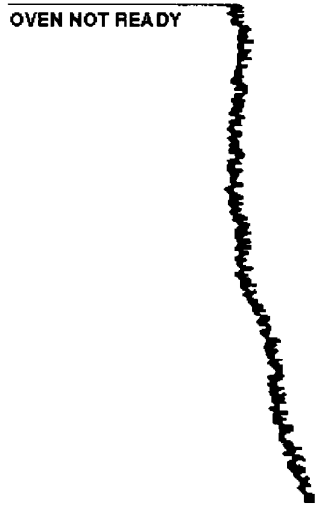
Good Noise
Straight
Still on page
PASSES



Good noise
Not too Steep
Still on page
PASSES



Noise Bad
Straight
Still on page
FAILS



Noise Bad
Slope too steep
Still on page
FAILS