Eliminating the Fear Factor

Flame Ionization Detector

Agilent Technologies

Agilent CrossLab
From Insight to Outcome

Agilent

The Flagship GCs from Agilent

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8860

8890

Intuvo

Agilent 8860/8890 Series GC Features – Detectors

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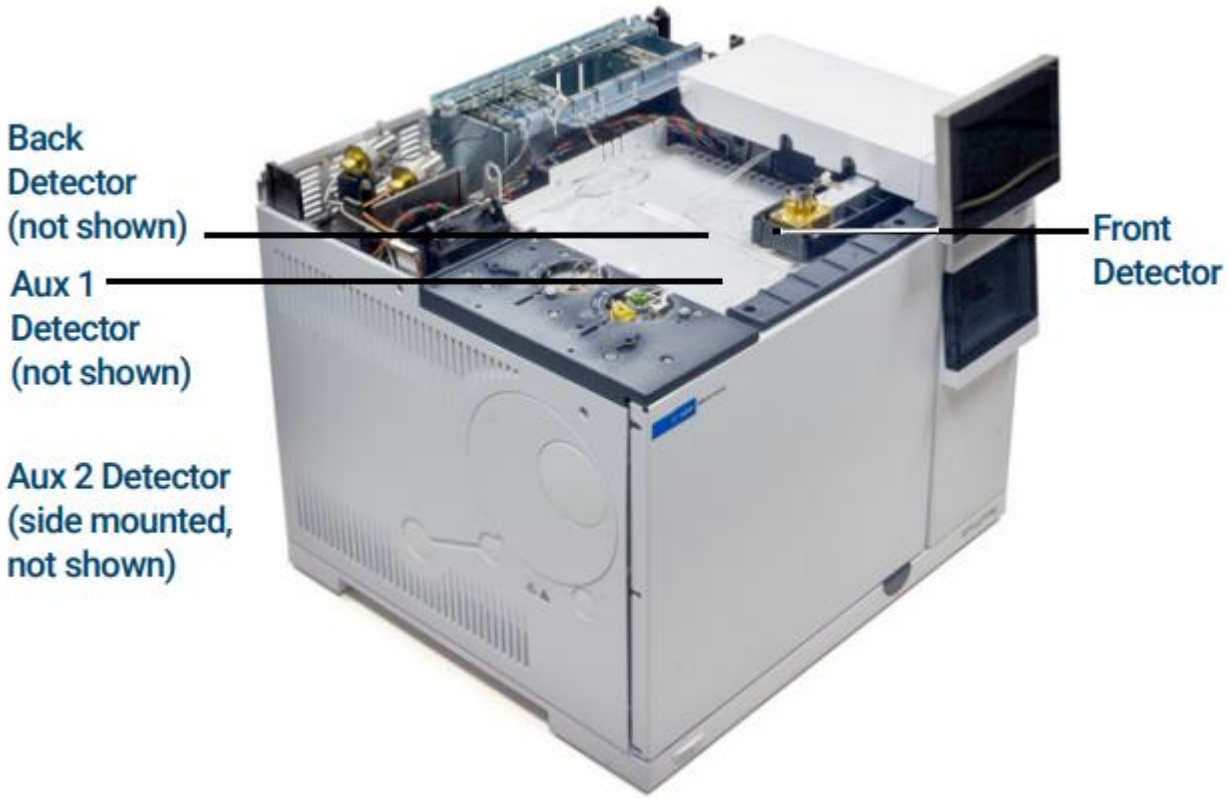

- AUX 1 Detector (TOP) can be TCD or FPD+
- AUX 2 Detector (SIDE) can be TCD, FID, or ECD

YES! This means you can have up to 4 detectors on your 8890 GC or 3 detectors on your 8860 GC!

Agilent Intuvo GC Features – Detectors

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D1 Position

- FID
- TCD
- uECD
- FPD+
- NPD
- XCD

D2 Position

- FID
- TCD
- uECD

Common GC Detectors

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- Flame Ionization Detector (FID)*
- Thermal Conductivity Detector (TCD)*
- Nitrogen-Phosphorous Detector (NPD)*
- Electron Capture Detector (ECD)
- Flame Photometric Detector (FPD)
- Chemiluminescence Detectors (XCD)
- Mass Selective Detector (MSD)

Today's topic is the FID!

GC Detectors

Let's focus on the FID

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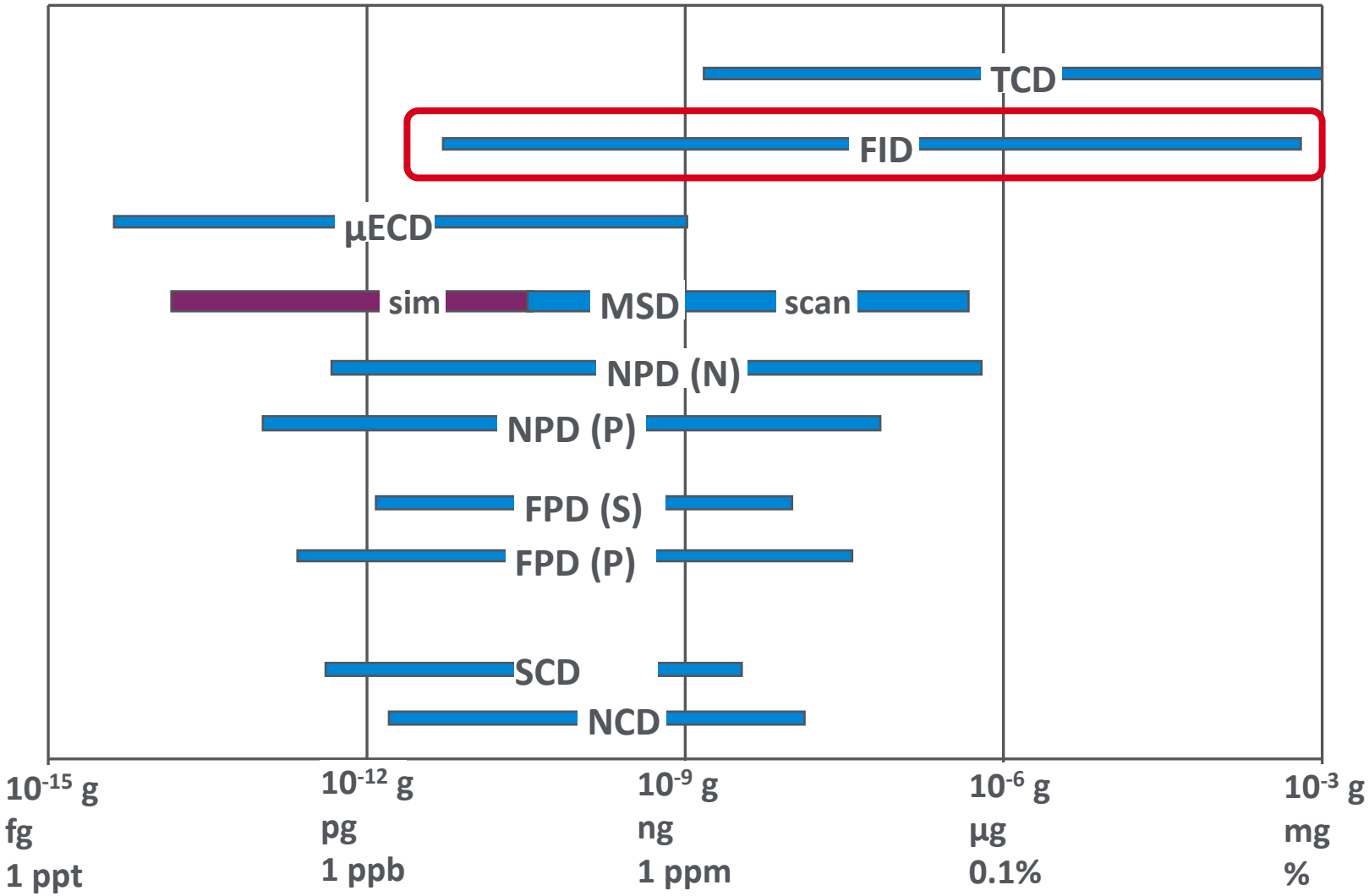
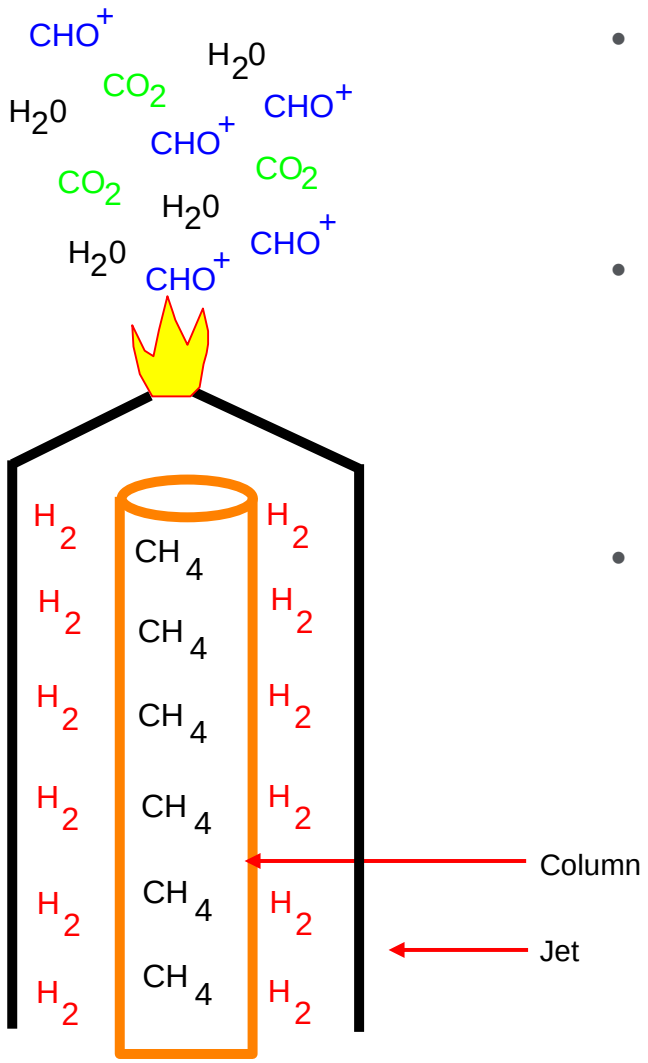


Linear Dynamic Range
Response increases *proportionally* with increased quantity.

Flame Ionization Mechanism



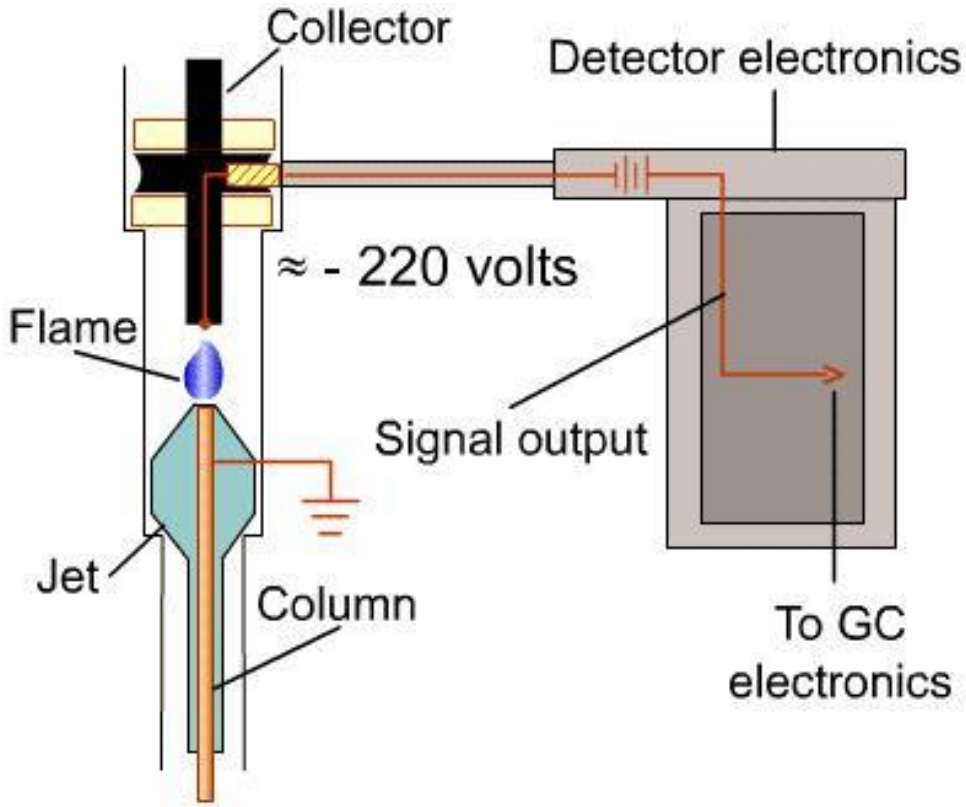
- The FID is a DESTRUCTIVE, mass sensing detector
- Cations generated in the flame are counted and produce the detector signal
- Analytes that have the greatest number of low oxidation state carbons produce the LARGEST signal

SELECTIVITY

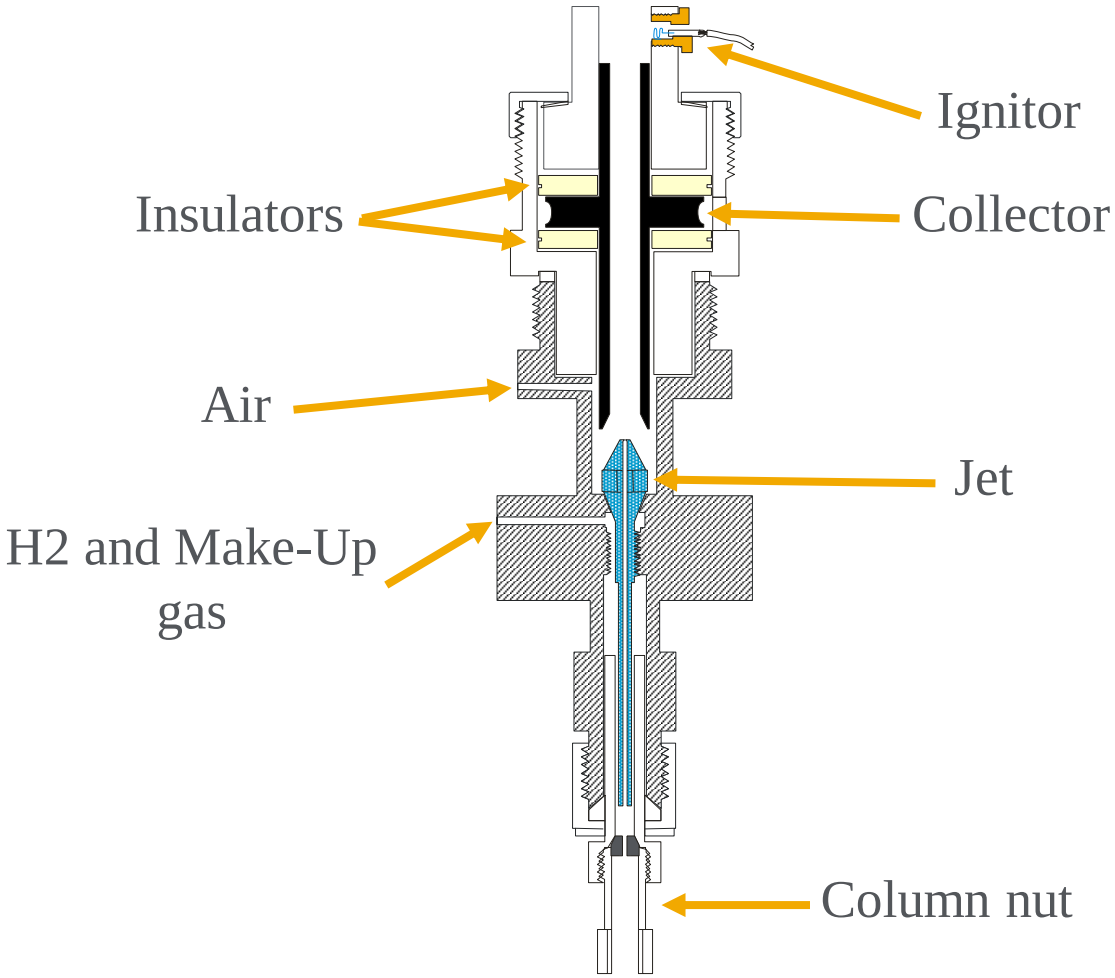
- The FID will respond to ORGANIC compounds (C-H Bonds)
- There is little or NO response for the following:
  - Inert Gases
  - H2O
  - CO2
  - CO
  - N2
  - O2
  - CS2
  - Heavily halogenated compounds

Agilent FID Theory of Operation

- H2 / Air = Flame
- Sample is burned producing positively charged ions
- Positive Ions are attracted to the negative voltage on the collector
- Collector current is converted to FID output in the Electrometer

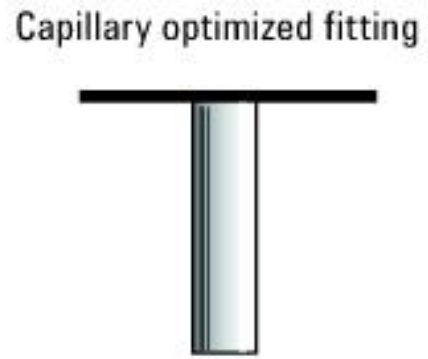


FID Detector Cutaway

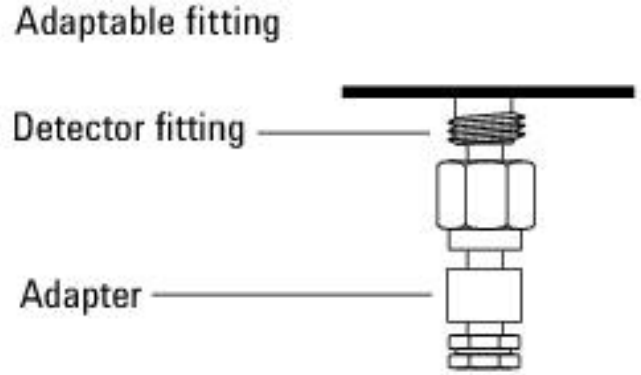


FID Detector Bases

- 6890/7890 GC –
  - Capillary optimized FID base
    OR
    Packed FID base with capillary adapter included
- Intuvo GC –
  - Capillary optimized FID base only -no packed column capabilities on Intuvo
- 8860/8890 GC –
  - Capillary optimized FID base only
    A packed column adapter is included



Capillary columns only



Adapters are available for capillary columns or 1/8” Packed Columns

¼” Packed columns can be installed without an adapter

Agilent 6890/7890/8860/8890/Intuvo FID Jets

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Adaptable FID Jets – 6890/7890		
Jet Type	Part#	Jet Tip ID
Capillary	**19244-80560**	**0.29 mm 0.011 in.**
Packed	**18710-20119**	**0.47 mm 0.018 in.**
Packed Wide Bore	**18789-80070**	**0.79 mm 0.030 in.**
High Temp	**G1531-80620**	**0.47 mm 0.018 in.**

Capillary-Optimized FID Jets – 6890/7890		
Jet Type	Part#	Jet Tip ID
Capillary	**G1531-80560**	**0.29 mm 0.011 in.**
High Temp	**G1531-80620**	**0.47 mm 0.018 in.**

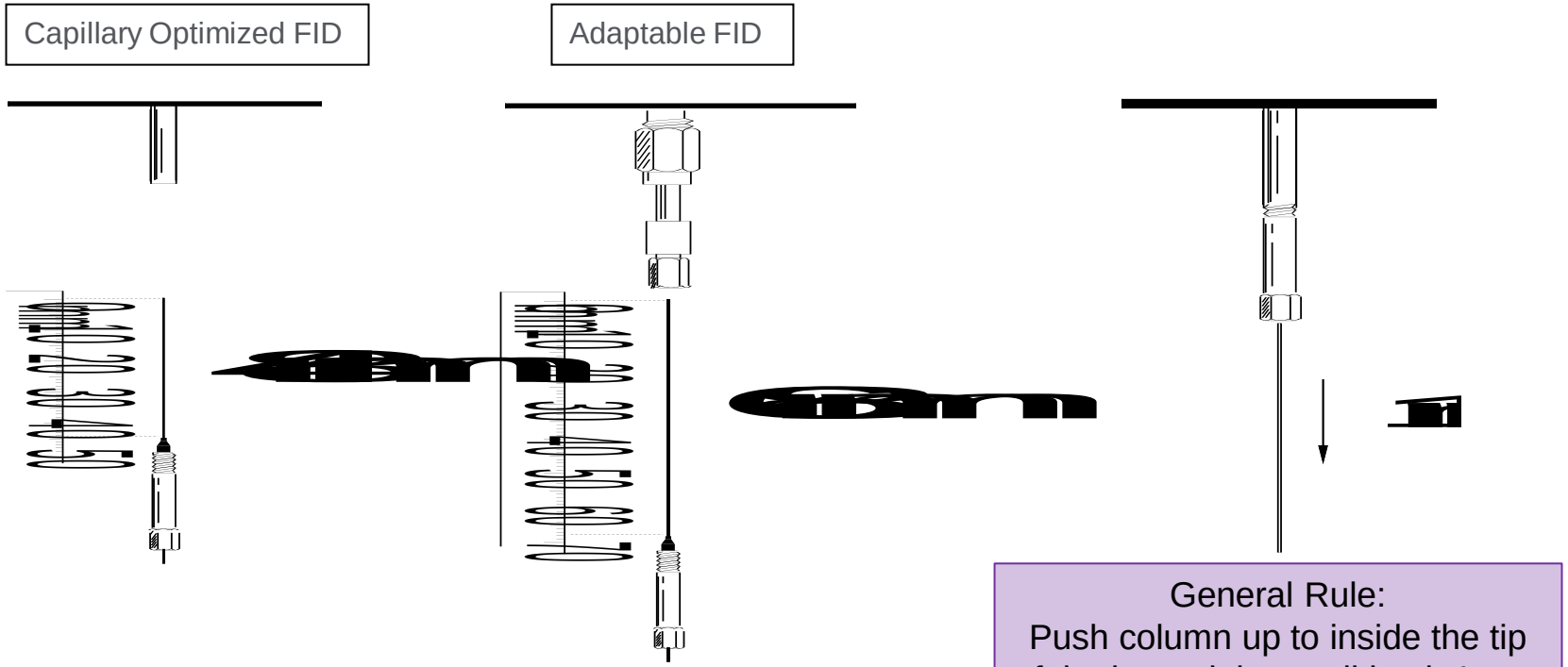
Intuvo/8860/8890 FID Jet		
Jet Type	Part#	Jet Tip ID
Jet	**G4591-20320**	**0.011 in.**

Agilent's NEW FID Jet Design!

Backwards Compatible back to 6890 GC

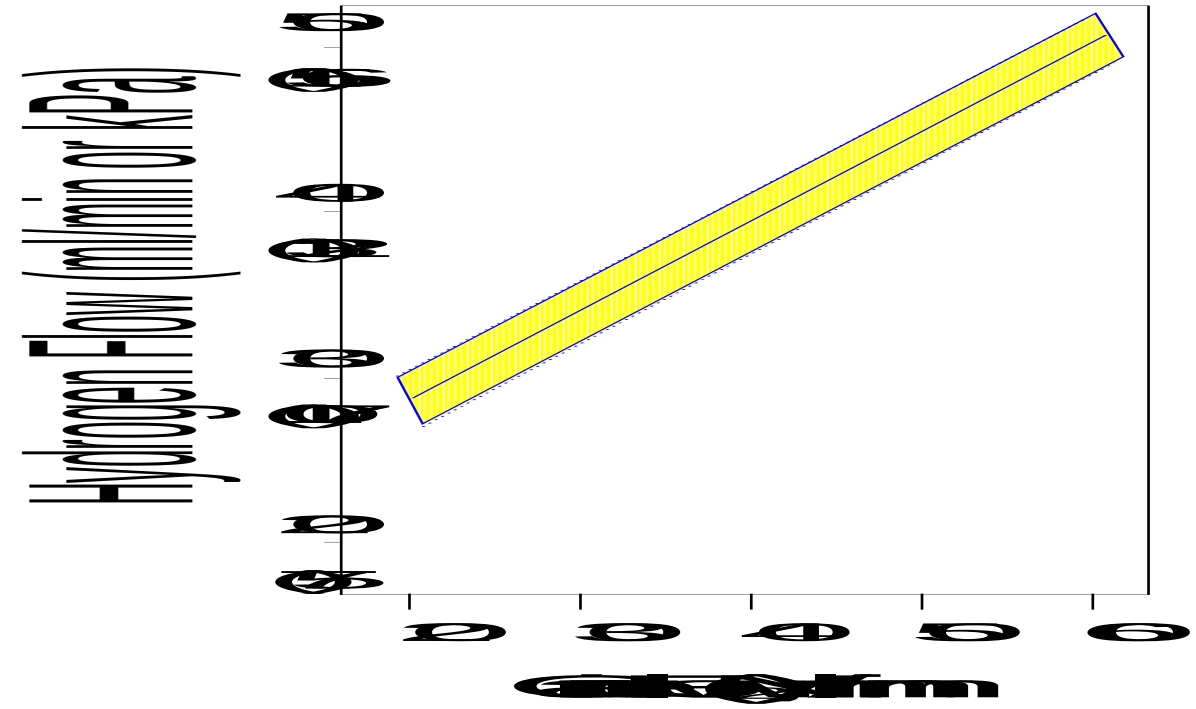
FID Setup - Column Installation - FID

Column Installation – From Troubleshooting and Maintenance Manuals



General Rule:
Push column up to inside the tip of the jet and then pull back 1 mm

FID – Optimizing Gas Flows for Sensitivity



Gas Flow Guidelines:

Hydrogen 30-35 ml/minute

Carrier + Makeup 30-35 ml/minute

Air – 400 ml/minute

Nitrogen is the recommended makeup gas

H2-to-Air ratio should be between 8-12%

Total Inert Gas should have ~1:1 ratio to H2

FID Makeup Gas Modes

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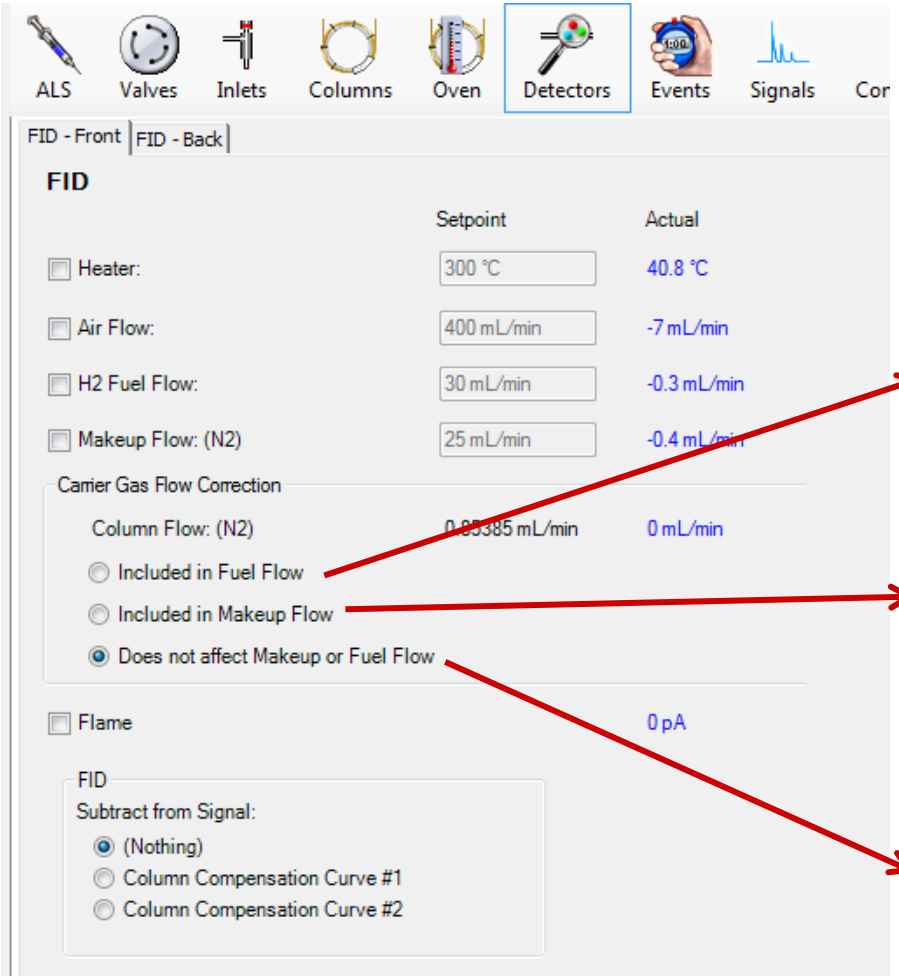
Method Editor Menu in Software



AKA: Column + Fuel = Constant

Select if using **Hydrogen Carrier**

The detector sees Constant Hydrogen Flow if carrier flow changes.

AKA: Column + Makeup = Constant

Best for Column – "**Constant Pressure**" Mode

Makeup is ramped during the run to compensate for dropping carrier flow – Detector sees constant Column + Makeup flow

AKA: Constant Makeup & Fuel Flow

Best for "**Constant Flow**" mode on the Column (He or N2)

Makeup Gas and Hydrogen Control are independent of the Carrier and remain constant throughout the analysis

FID – Tips and Tricks

- It is best to operate the FID at 300°C or hotter – But always 20°C hotter than the highest GC Oven Temp

- Optimizing Flows 1:1 ratio of Carrier + Makeup to H2 flow
  - Nitrogen is the recommended makeup gas

Gas Flow Guidelines:

Hydrogen 30-35 ml/minute

Carrier + Makeup 30-35 ml/minute (≈1:1 ratio to H2)

Air – 400 ml/minute

- Make sure you check the base of the FID for any chunks of graphite when changing the column or jet to prevent those chunks from getting into the jet and causing a partial blockage

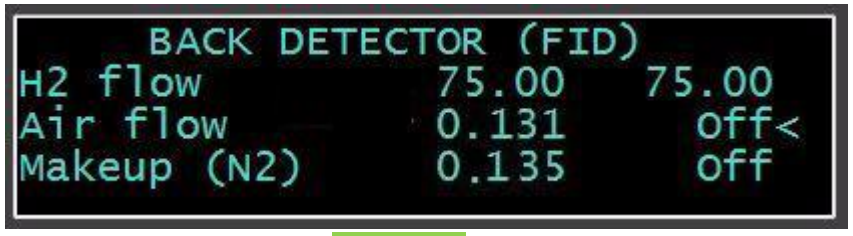
- Check your Lit Offset. Default is 2pA and sometimes with very clean gases the baseline will drop below 2pA causing your GC to think the flame is not it.

- Use the right **jet** for your application
  - High oven temps, bleeding columns, or clogging jets often – try the High Temp FID Jet

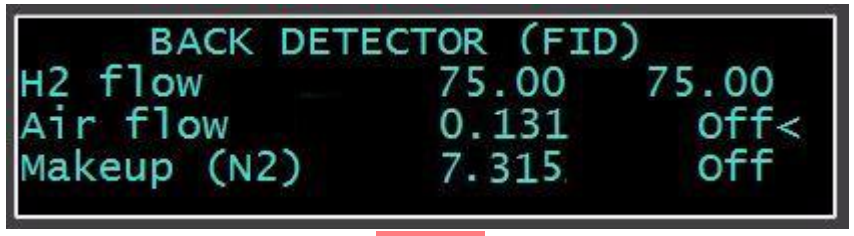
Wider opening to help prevent clogging

FID Troubleshooting

Flame Won't Light - Jet Diagnostic Test – 7890 Series GC



Pass



Fail

- **Set H2 Flow to 75 ml/minute**
- Turn the Makeup Gas "OFF"
- **Monitor Makeup Gas "Actual Flow"**
  - **Low flow reading/minimal change – Jet is clear!**
  - **High Flow reading/big change - indicates Jet Plugging**…

FID Jet Restriction Test – How does it Work?

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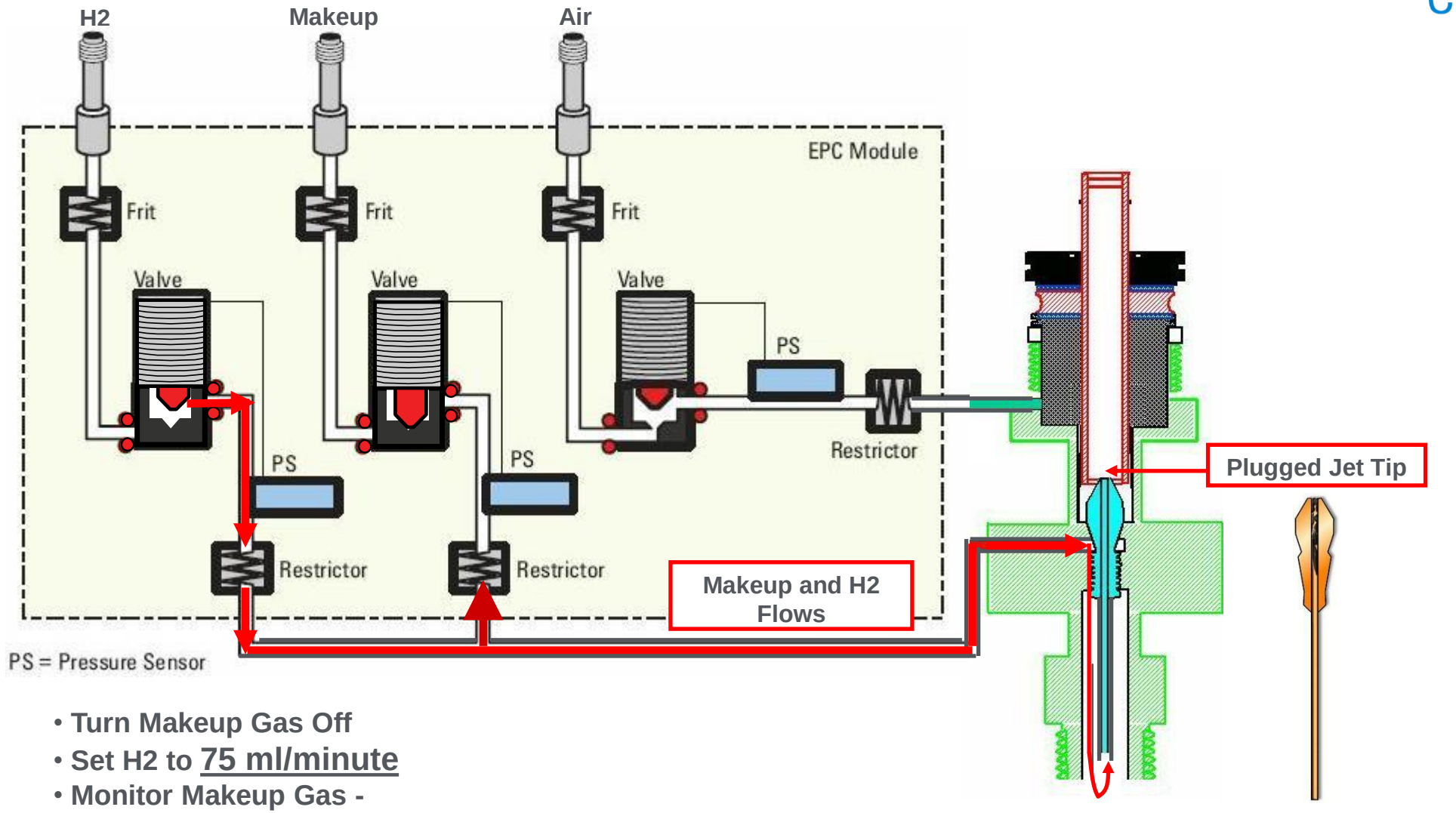

- **Turn Makeup Gas Off**
- **Set H2 to <u>75 ml/minute</u>**
- **Monitor Makeup Gas -**

An increase in the Makeup Gas flow reading indicates pressure backing up from the jet

Jet Diagnostic – Built into 8860/8890/Intuvo GC

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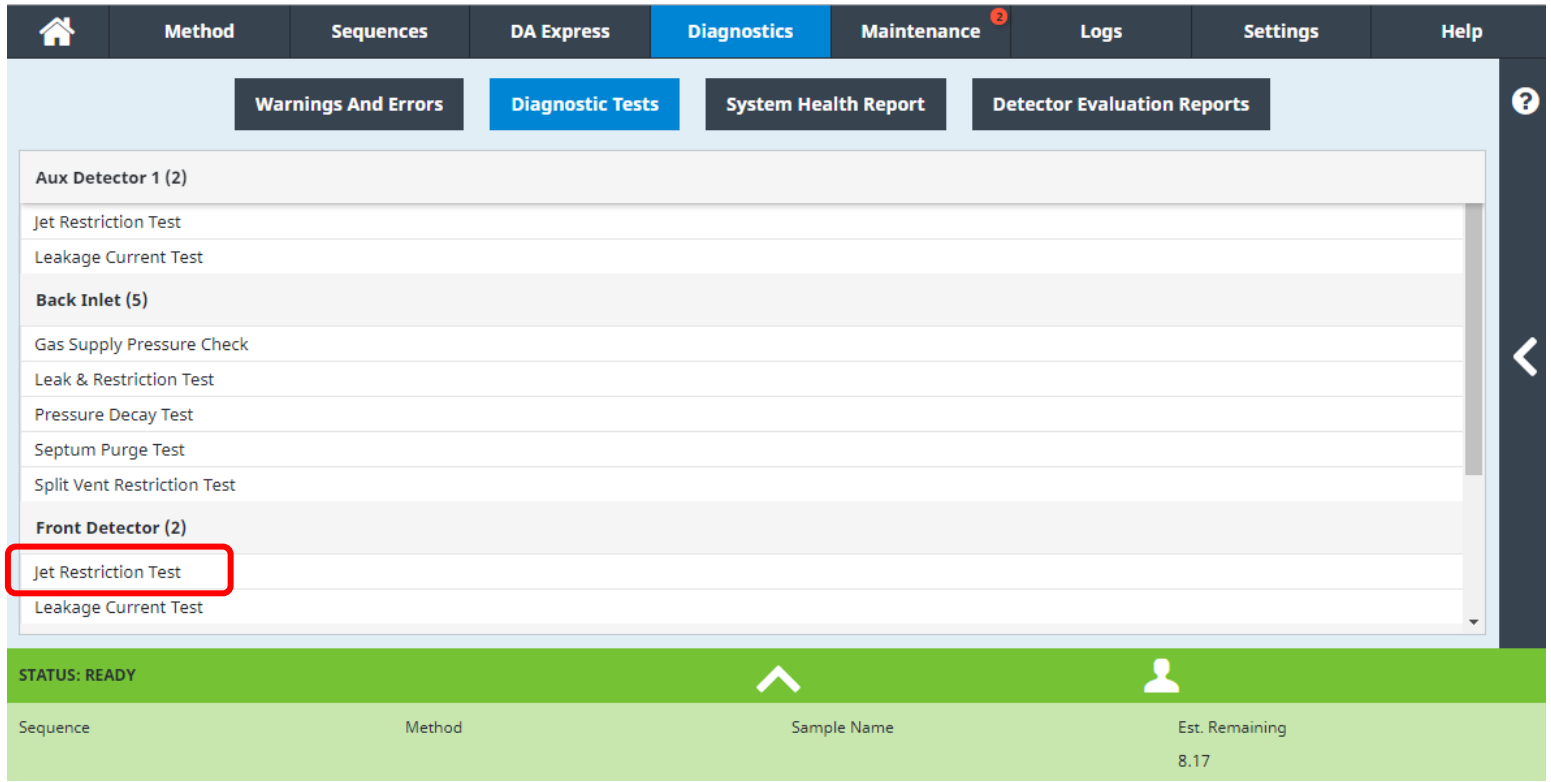
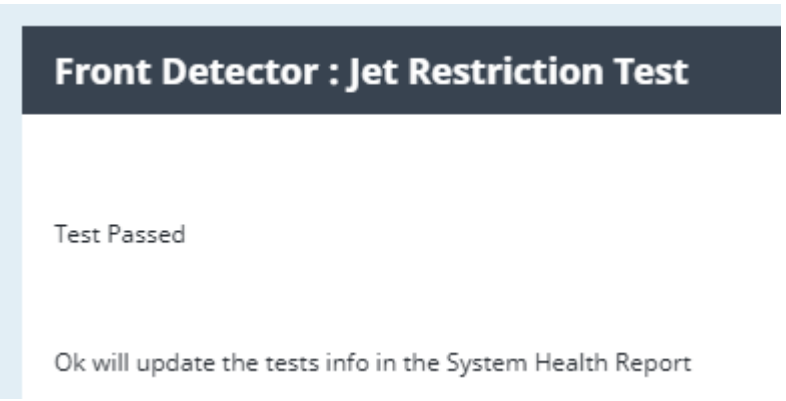
Now the GC has the ability to run the Jet Restriction test from the front panel and generate a pass or fail.

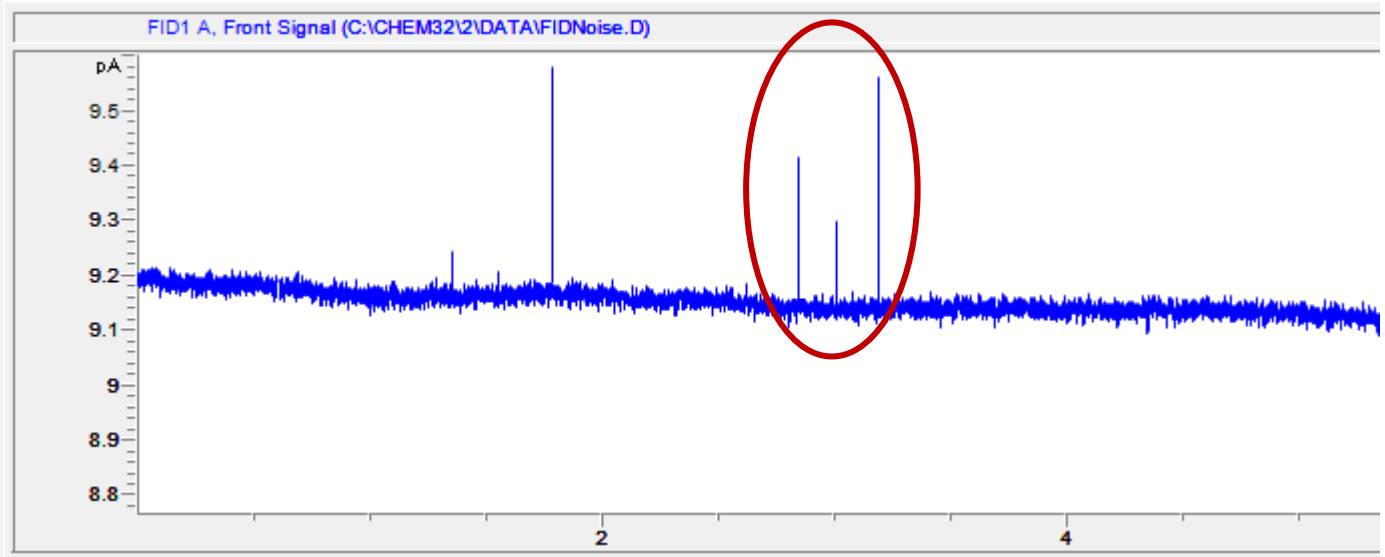




A quick, easy, un-intrusive test that can be run from the GC or the browser interface.

FID Troubleshooting

Baseline Spiking



Possible Causes:

- Contamination of the jet, collector, and/or the base by particulates, stationary phase, graphite
  - Clean and Sonicate or Replace the Jet
  - Thoroughly clean the collector and chimney assembly
  - Thoroughly clean the base of the FID – especially if ferrule chunks are visible

- Electronic Noise – much less likely
  - Vibration
  - Electrical – Grounding problems, Power line disturbances, Power Line conditioners
  - Strong RF Interference

FID Troubleshooting
Baseline Drift and Wander

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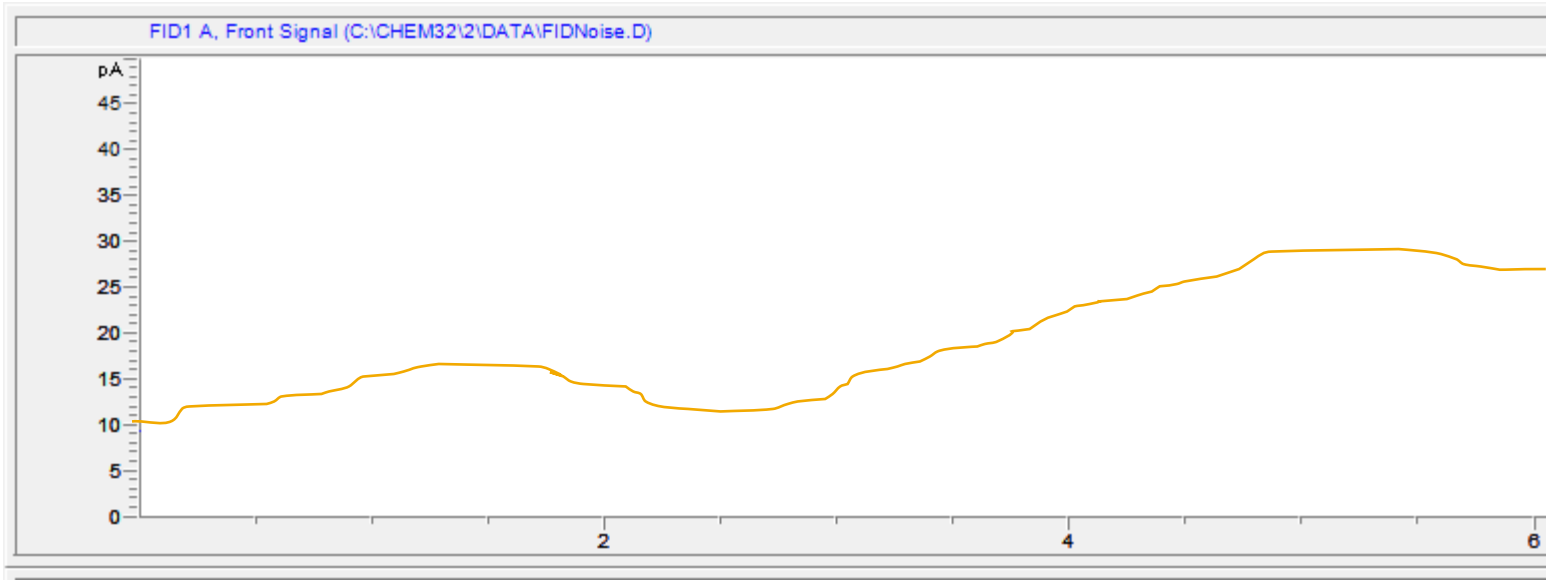

Possible Causes:

- Contaminated/Saturated Gas Supply Traps
- Buildup of high MW sample contamination in the GC Column
- Column Bleed
- Contamination in the Base of the Detector
- Gas Leaks
- Column Fitting Leaks

New!
Smart
Gas Clean
Sensor

Baseline Evaluation – Intelligent GCs

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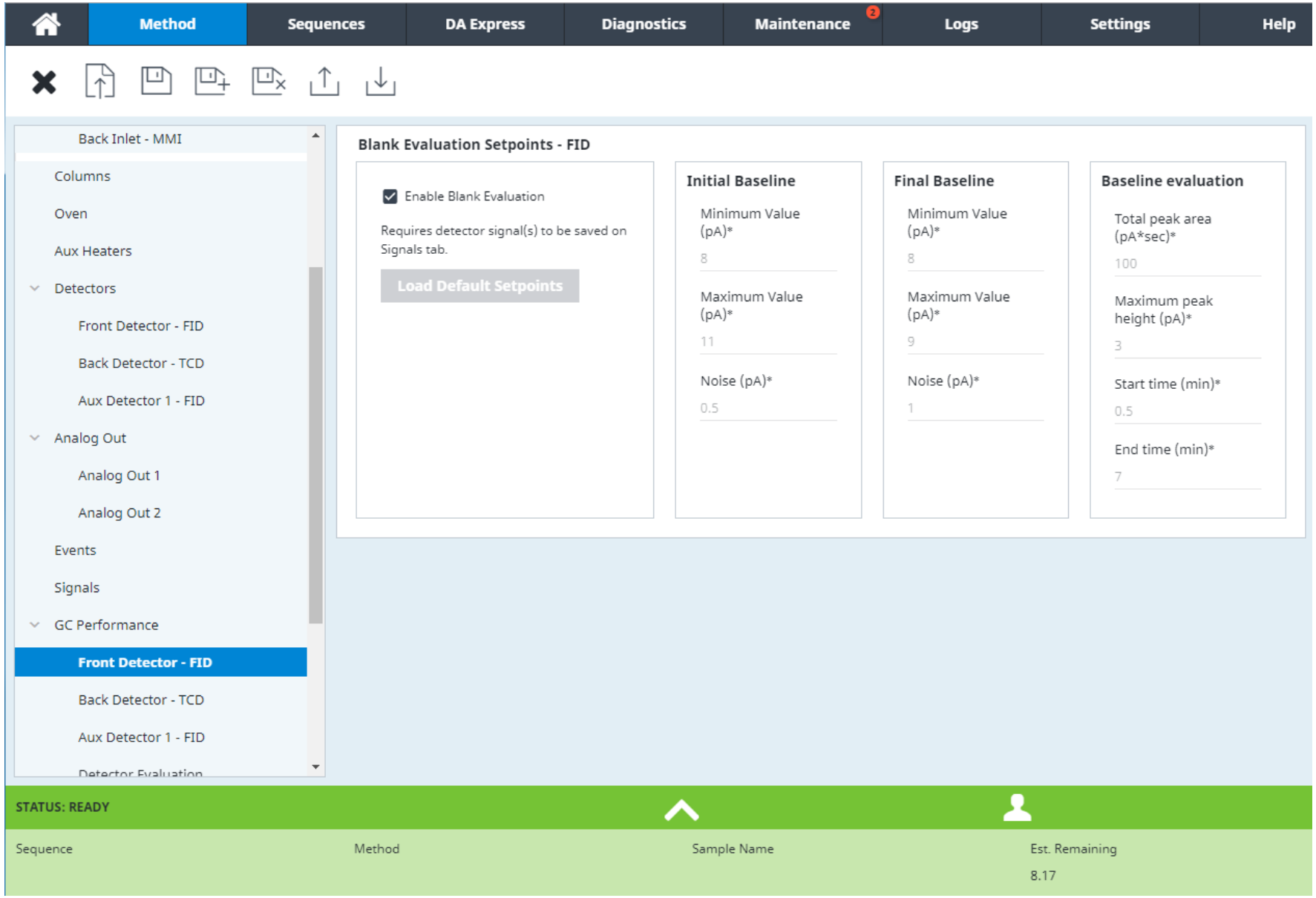

- Blank Evaluation allows you to set up expected signal levels at the start and end of the run and confirms they are as expected.
- When you run a blank, your smart GC monitors the blank and returns either a log entry if the blank is not blank, or stops the sequence.

FID Troubleshooting
Measuring Flows

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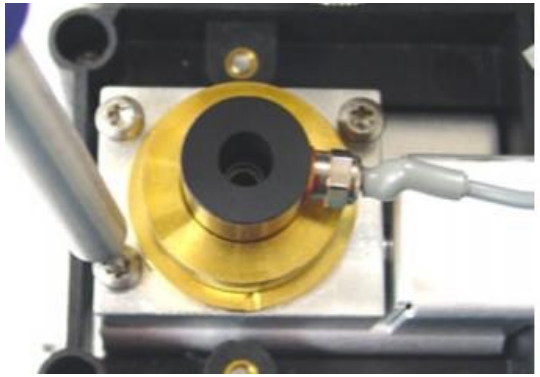
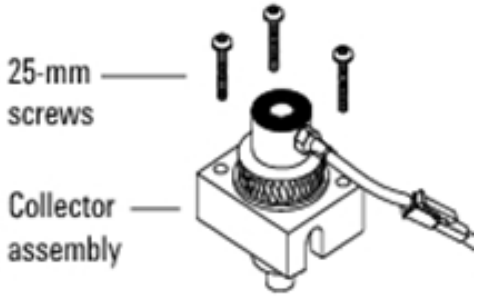
Gather your flowmeter and adapter tube first!

- Set oven temp to Ambient (35C)
- Turn off column flow
- Shut off all detector gases
- Turn off FID flame
- Cool detector
- Connect adapter to detector by inserting into FID as far as possible
- Turn on one detector flow at a time allowing the gas to equilibrate in the flowmeter tubing before taking a measurement

A separate adapter (19301-60660) is supplied for the FID. Insert the adapter into the detector exhaust vent as far as possible. You will feel resistance as the adapter O-ring is forced into the detector exhaust vent. Twist and push the adapter during insertion to ensure a good seal.

FID Maintenance

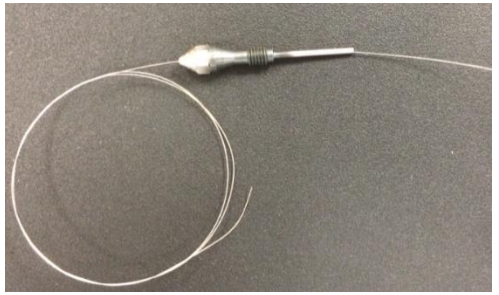


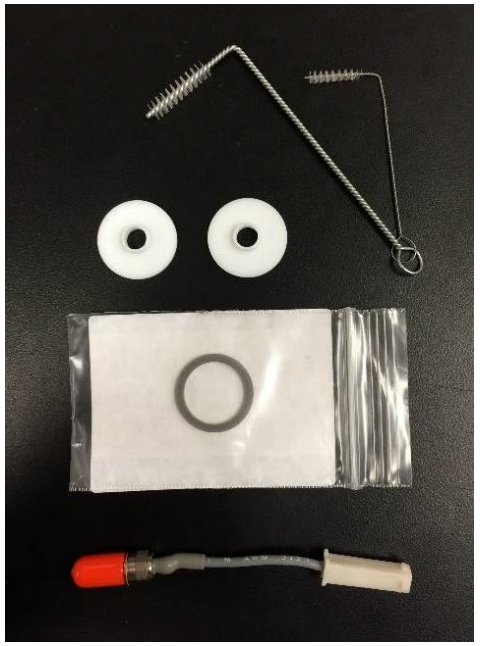


Rebuilding Kit: G1531-67001
Entire Collector Assembly for Extremely corrosive applications

Cleaning Kit: G1531-67000
Includes Small Parts:

- Teflon Collector Insulators
- Silicone Rubber Base Gasket
- Ignitor
- Cleaning brushes for Collector
- Cleaning Wires for Jet

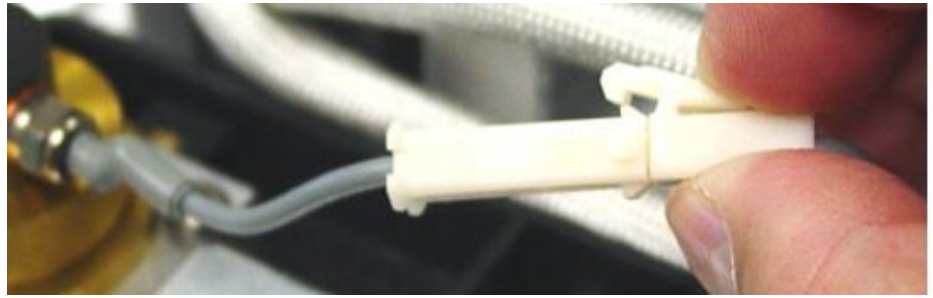




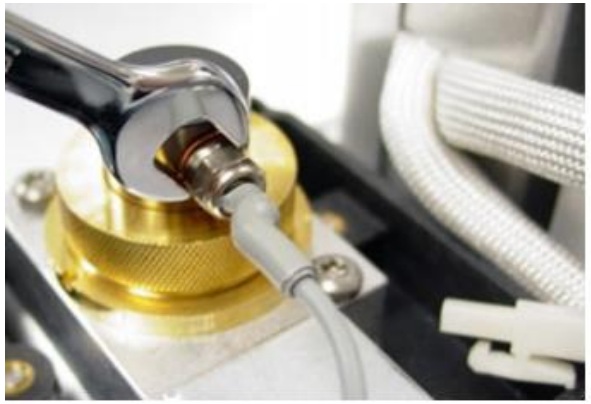
Note: The FID Jet must always be ordered separately

Now let's step through a complete FID Maintenance/Rebuild Procedure together!

FID Maintenance Procedures
Ignitor Replacement



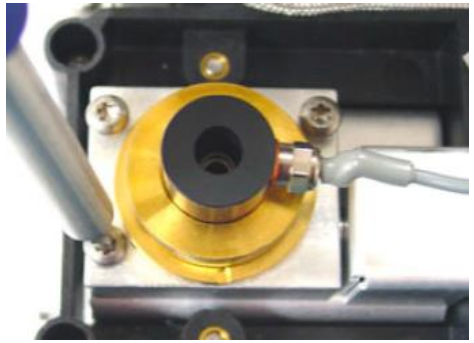
Disconnect the Ignitor



Remove with 5/16” wrench

Install the new Ignitor with
Copper Washer –
Tighten with 5/16” wrench

FID Maintenance Procedures
Collector Removal



Loosen 3-T20 Screws

A common mistake people make is NOT removing the three screws **FIRST.** This makes getting to the jet quite difficult and you can damage the interconnect assembly and spring. Make sure you remove the screws first.

Pull the Collector Assembly straight up

FID Maintenance Procedures

Collector Disassembly and Cleaning

Unscrew the knurled nut

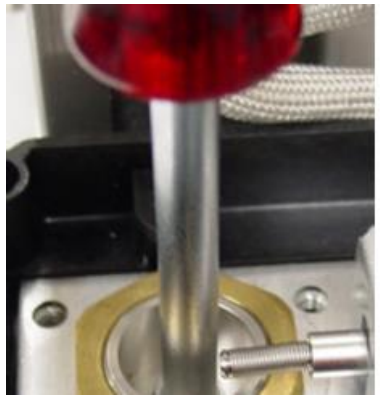
Remove the castle

Remove the collector and Upper/lower PTFE Insulators

Remove/replace the silicone rubber gasket

FID Maintenance Procedures

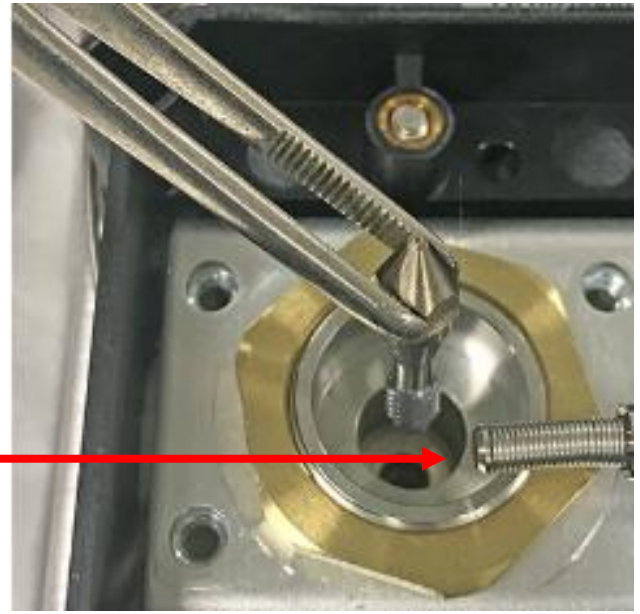
Jet Removal, Collector Removed



Using a ¼” Nut Driver, Loosen the Jet

Remove the Jet

Be careful not to damage the Interconnect Spring



FID Maintenance Procedures

Check for Graphite Chunks in the Detector Base

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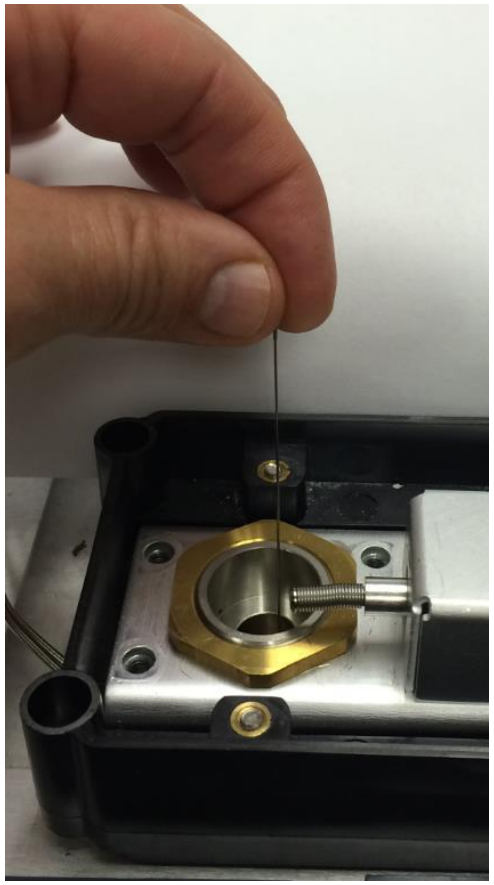
Indicator ….**Was the Column hard to remove or to re-install?**

Shine a flashlight up
From the oven to check
For impacted graphite



Clean out the Detector Base – Top down
A syringe plunger works well

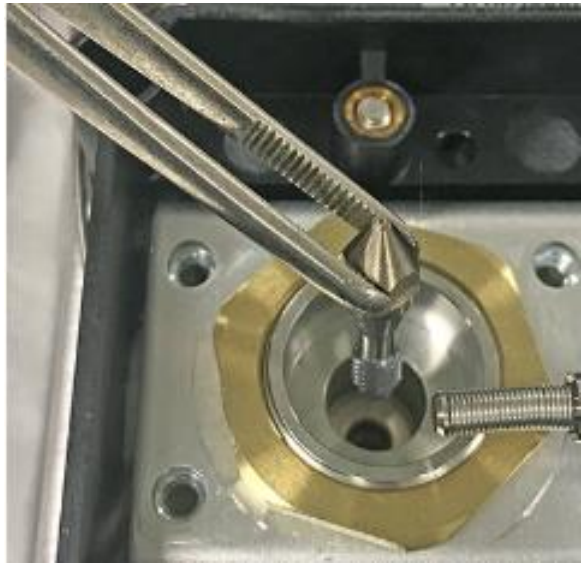
Look for graphite chunks
falling into the oven

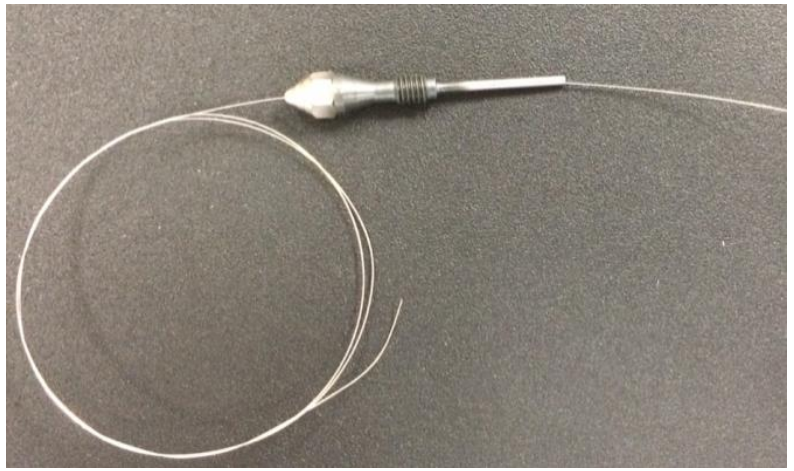
FID Maintenance Procedures

Jet Cleaning or Replacement

Jet sealing surface – if this is Compromised, replace the jet

Agilent recommends replacing the Jet





If the jet is plugged you can clean with a .010” cleaning wire

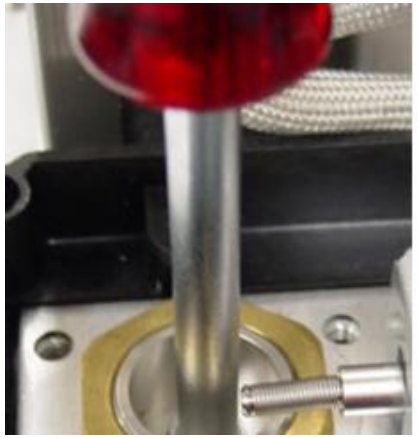
Jet Cleaning Wire (0.010”) 9301-0985

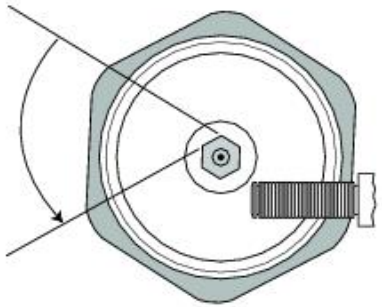
FID Maintenance Procedures

Jet Installation

FID Jet

Interconnect Spring



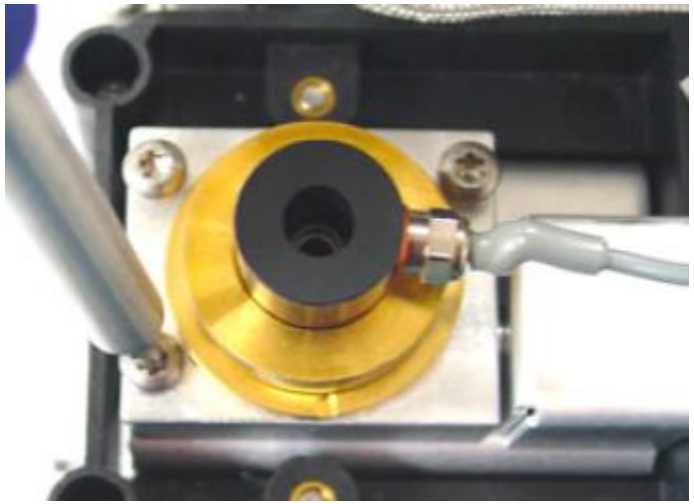


Using a ¼” Nut Driver,
Tighten the FID Jet 1/6th of
A turn past Hand Tight
(For a New Jet only)

FID Maintenance Procedures

Re-install the Collector

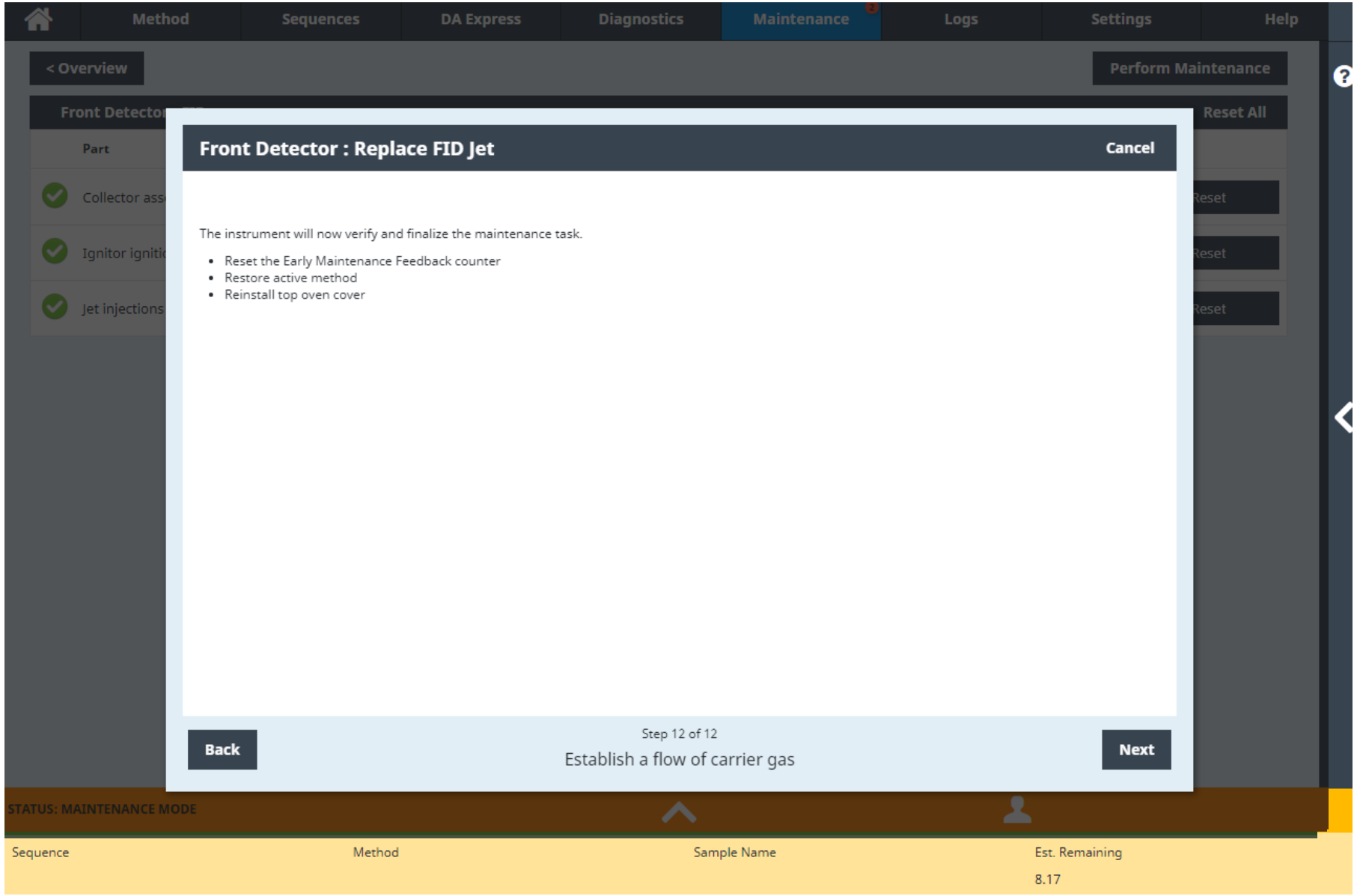
Position the cleaned or replacement collector directly over the detector base and push straight down.
The interconnect spring will “pop” into place!



Tighten 3-T20 Screws evenly and firmly

Re-connect Ignitor electrical cable

Intelligent GC Features – Self Guided Maintenance



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These on-board procedures take you through common maintenance operations, guiding you step-by-step, cooling the instrument zones that are necessary, and even updating logs and EMF counters when finished!

These are accessed by way of the “perform maintenance” link in the maintenance tab.

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For more information regarding Agilent's Gas Chromatography, please visit the website at:
https://www.agilent.com/en/products/gas-chromatography

Videos for Simple Maintenance and Theory can be found on the Agilent YouTube page:
https://www.youtube.com/user/agilent/

Checkout Agilent Communities where you can get answers and share insights:
https://community.agilent.com/welcome

A Sample of Customer Education Courses – GC

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e-Learning

Agilent University Course Code	Course Name
GC-MULTI-2100e	Advanced Operation of the Multi Mode Inlet (MMI)
GC-0GEN-1012s	GC Inlets Theory and Operation
GC-0GEN-1013s	GC Detectors Theory and Operation
SI-7696-1100s	Agilent 7696A Sample Prep WorkBench Operation
GC-MULTI-1240zs	Making Productivity Happen: an Agilent GC eLearning Series
SI-7693-1100s	Agilent 7693A ALS Basic and Advanced Operation and Maintenance
SI-7697-2100fs	Running Start for Agilent 7697A Headspace Sampler

Classroom

Agilent University Course Code	Course Name
GC-0GEN-2000V2	Practical Gas Chromatography – 4 Day (R1915A)
GC-7890-2100cV4	Agilent 7890A/B GC and OpenLAB ChemStation Operation – 5 Day (R1778A)
GC-7890-2200cV3	Agilent 7890A/B GC Maintenance & Troubleshooting – 4 Day (R1914A)
GC-9000-2101cV3	Agilent 7890/9000 GC with OpenLAB 2.3 Essential and Advanced Operation – 4 Days
GC-9000-2103c	Agilent Intuvo 9000 GC with OpenLAB CDS ChemStation Operation – 4 Days
GC-8890-2100c	Agilent 8890 GC Operation with OpenLAB CDS ChemStation Edition – 4 Days

Enroll at Agilent University: http://www.agilent.com/crosslab/university/