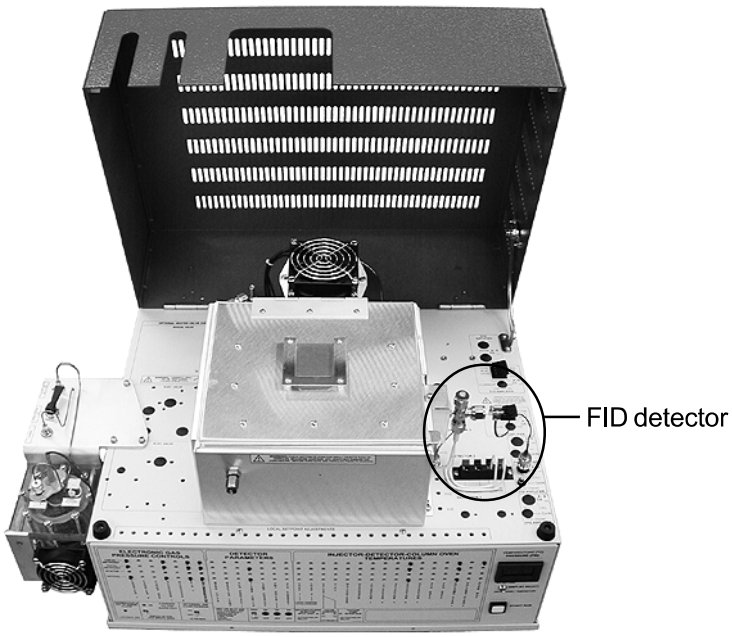
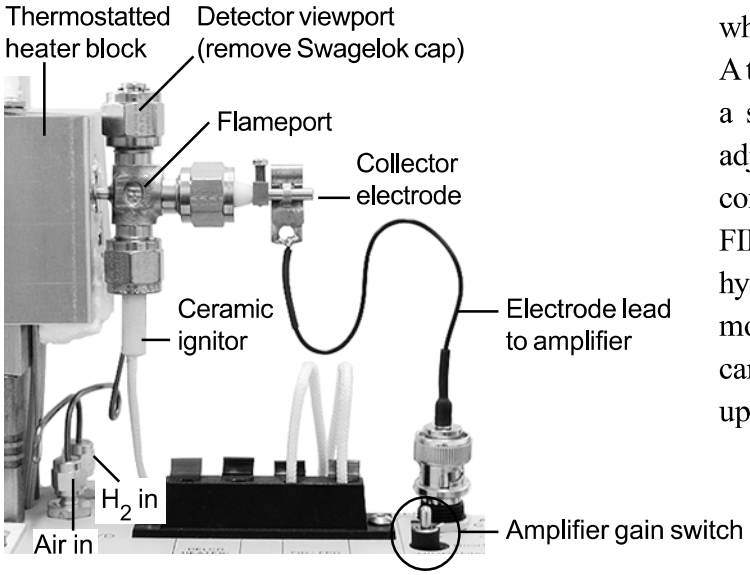
Overview

The Flame Ionization Detector responds to any molecule with a carbon-hydrogen bond, but its response is either poor or nonexistent to compounds such as H_2S, CCl_4, or NH_3. Since the FID is mass sensitive, not concentration sensitive, changes in carrier gas flow rate have little effect on the detector response. It is preferred for general hydrocarbon analysis, with a detection range from 0.1ppm to almost 100%. The FID's response is stable from day to day, and is not susceptible to contamination from dirty samples or column bleed. It is generally robust and easy to operate, but because it uses a hydrogen diffusion flame to ionize compounds for analysis, it destroys the sample in the process.



(SRI Capillary FID GC with built-in Hydrogen Generator)

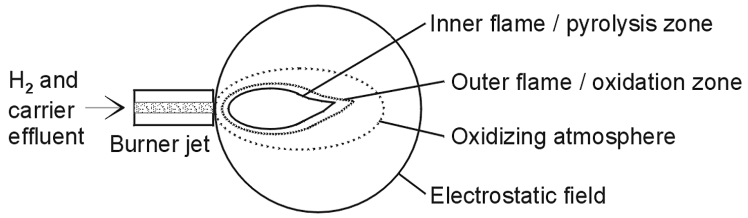
The SRI FID features a unique ceramic ignitor which can run hot continuously, and prevent the flame from extinguishing even with large water injections or pressure surges from column backflush. This ignitor is positioned perpendicular to the stainless steel detector jet and does not penetrate the flame. Opposite this flame is the collector electrode. This positively charged metal tube serves as a collector for the ions released as each sample component elutes from the column(s) and is pyrolyzed in the flame; it doubles as a vent for the FID exhaust gas. The FID is equipped with an electrometer amplifier which has HIGH, HIGH (filtered), and MEDIUM gain settings. On an SRI GC, the hydrogen and air gas flows are controlled using electronic pressure controllers, which are user adjustable via the GC's front panel. A thermostatted aluminum heater block maintains a stable detector temperature which is user adjustable up to 375°C. The optional built-in air compressor may be used to supply the air for the FID, eliminating bulky air cylinders. The built-in hydrogen generator is another option: the standard model can produce 20mL/min for use as both carrier gas and FID combustion gas at pressures up to 25 psi.



Theory of Operation

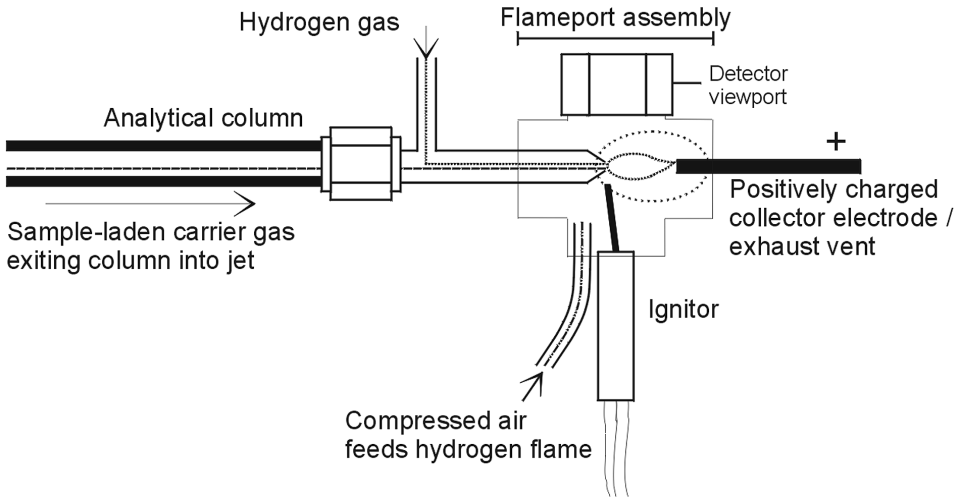
In the SRI FID, the carrier gas effluent from the GC column is mixed with hydrogen, then routed through an unbreakable stainless steel jet. The hydrogen mix supports a diffusion flame at the jet's tip which ionizes the analyte molecules. Positive and negative ions are produced as each sample component is eluted into the flame. A collector electrode attracts the negative ions to the electrometer amplifier, producing an analog signal for the data system input. An electrostatic field is generated by the difference in potential between the positively charged collector electrode and the grounded FID jet. Because of the electrostatic field, the negative ions have to flow in the direction of the collector electrode.

The FID hydrogen diffusion flame



The ratio of air to hydrogen in the combustion mixture should be approximately 10:1. If the carrier flow is higher than normal, the combustion ratio may need to be adjusted. Flow is user adjusted through the Electronic Pressure Controllers (EPC); the rates used to generate test chromatograms at the factory are printed on the right side of the GC in the flow rate chart. The FID temperature must be hot enough so that condensation doesn't occur anywhere in the system; 150°C is sufficient for volatile analytes; for semi-volatiles, use a higher temperature. In addition to using the ignitor to light the flame, it may be left on at an intermediate voltage level to prevent flameout (-750 or 7.5 volts). The ignitor is very durable and will last a long time, even at high temperatures.

FID detector schematic



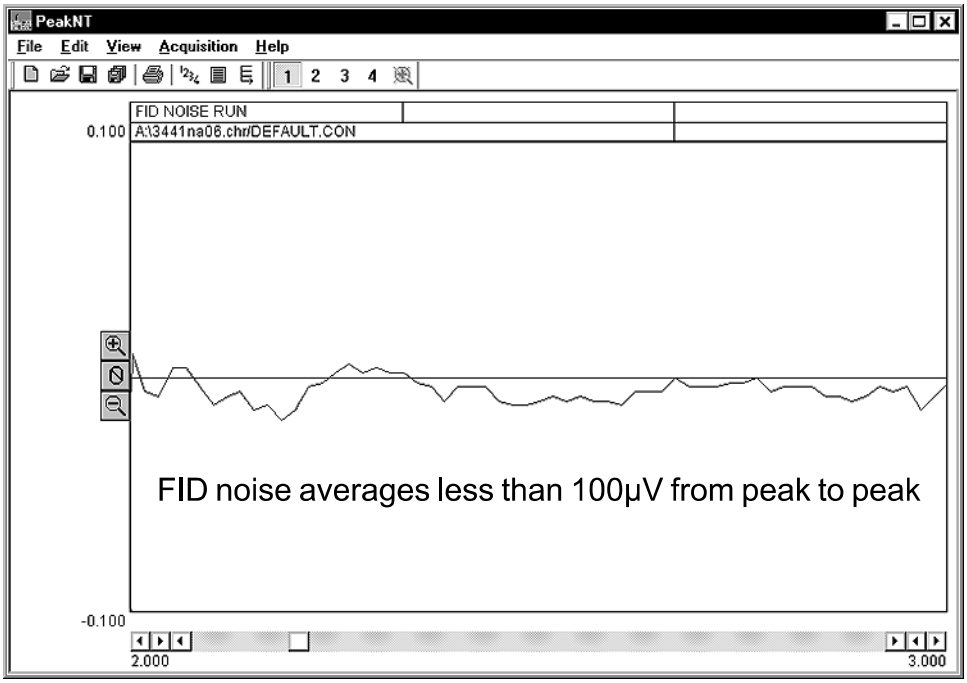
Expected Performance

FID noise run

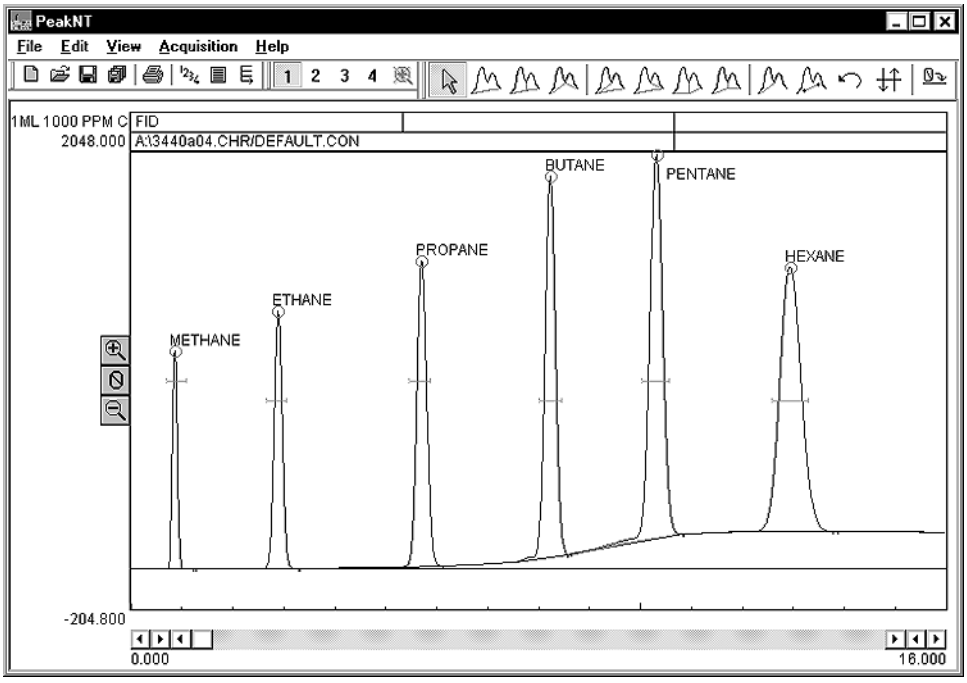
Column: 15m MXT-1
Carrier: Helium @ 10mL/min
FID gain = HIGH
FID temp = 150°C
FID ignitor = -400

Temperature program:

Initial	Hold	Ramp	Final
80°C	15.00	0.00	80°C



C_1-C_6 Hydrocarbon Test Analysis



Sample: 1mL of 1000ppm C_1-C_6
Carrier: Helium @ 10mL/min
FID H_2 at 25psi = 25mL/min
FID air at 6psi = 250mL/min
FID temp = 150°C
FID ignitor = -750
FID gain = HIGH
Valve temp = 90°C

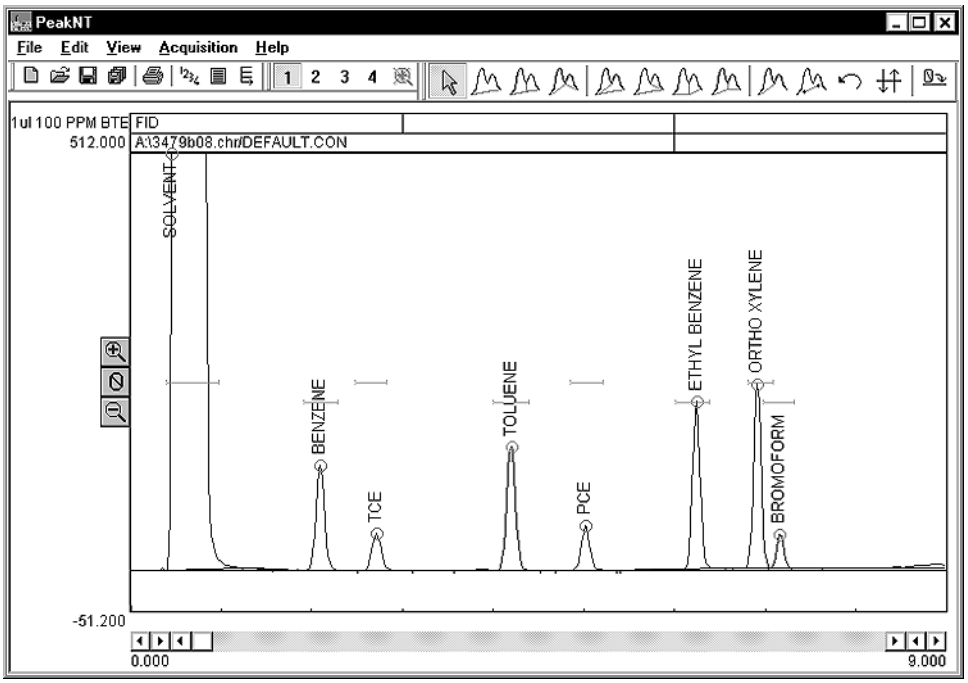
Results:

Component	Retention	Area
Methane	0.850	6979.9260
Ethane	2.866	13623.7580
Propane	5.683	19535.8960
Butane	8.200	26456.5980
Pentane	10.283	33053.9680
Hexane	12.916	39419.0870
	Total	139069.2330

Expected Performance

BTEX Test Analysis

The BTEX chemicals (Benzene, Toluene, Ethylbenzene, and Xylenes) are volatile monoaromatic hydrocarbons found in petroleum products like gasoline. Due to industrial spills and storage tank leakage, they are common environmental pollutants. Groundwater, wastewater, and soil are tested for BTEX chemicals in many everyday situations. The chromatogram below was obtained using an FID-equipped SRI GC.



1µL 100ppm BTEX sample

15m MXT-VOL capillary column

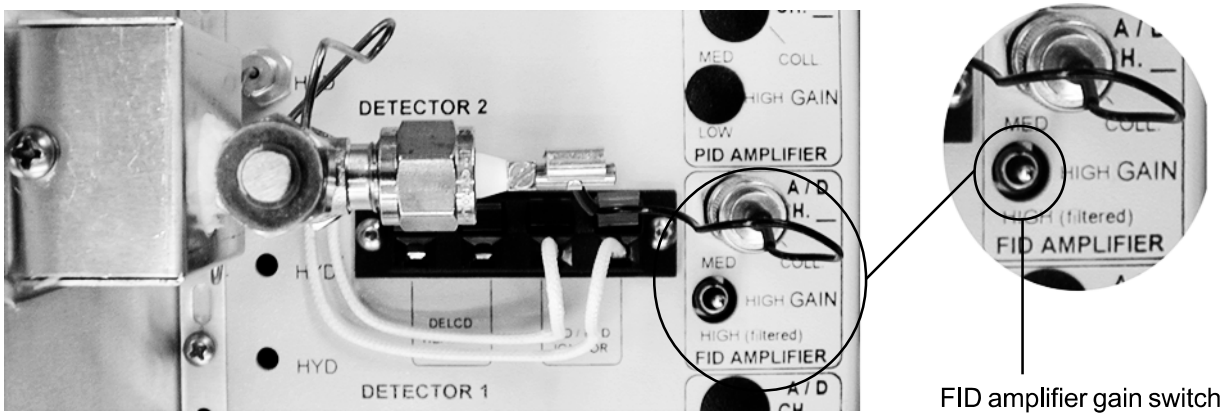
FID gain = HIGH
FID temp = 150°C
FID ignitor = -400

Results:

Component	Retention	Area
Solvent	0.433	95879.7560
Benzene	2.083	837.1000
TCE	2.700	319.2450
Toluene	4.183	1070.1060
PCE	5.000	344.8640
Ethyl Benzene	6.233	1200.3320
Ortho Xylene	6.900	1312.3070
Bromoform	7.150	225.2360
	total	101188.9460

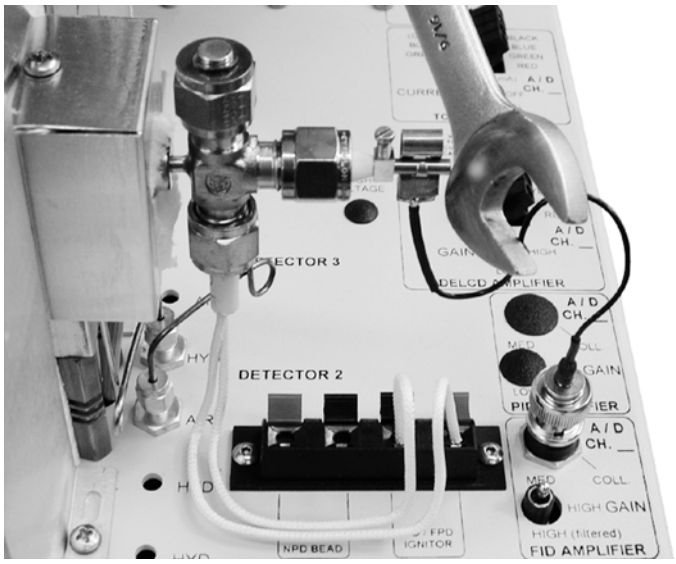
General Operating Procedure

1. Set the FID amplifier gain switch to HIGH for most hydrocarbon applications. If peaks of interest go off the scale (greater than 5000mV), set the gain to MEDIUM. When peaks of interest are 20 seconds wide or more at the base and extra noise immunity is desired, set the gain switch to HIGH (filtered). This setting broadens the peaks slightly.



2. Set the FID hydrogen flow to 25mL/min, and the FID air supply flow to 250mL/min. The approximate pressures required are printed in the gas flow chart on the right-hand side of the GC.

3. Ignite the FID by holding up the ignitor switch for a couple of seconds until you hear a small POP. The ignitor switch is located on the front panel of your SRI GC under the "DETECTOR PARAMETERS" heading (it is labelled vertically: "FLAME IGNITE").



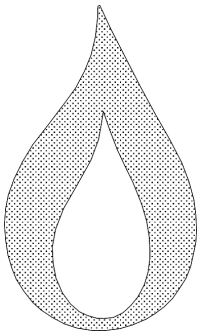
4. Verify that the FID flame is lit by holding the shiny side of a chromed wrench directly in front of the collector outlet/FID exhaust vent. If condensation becomes visible on the wrench surface, the flame is lit.

5. If you wish to keep the ignitor ON to prevent flameout, set the ignitor voltage to -750 by adjusting the trimpot on the "FLAME IGNITE" zone with the supplied screwdriver.

FID Troubleshooting

Whenever you experience problems with your FID, review your operating procedures: check the detector parameters, check to make sure you are on the correct channel of the data system display, check the mixture of hydrogen (25mL/min) and air (250mL/min), check gas pressures and connections, check the oven and detector temperatures, and all the other variables that compose your analysis. Having ruled out operating procedure as the source of the problem, there are two simple diagnostic tests you can perform. Detector problems can be electrical or chemical in nature. Use the Flame ON/OFF test to help determine if the problem is of chemical origin. Use the Wet Finger test to determine if the problem is electrical.

A. Flame ON/OFF Test



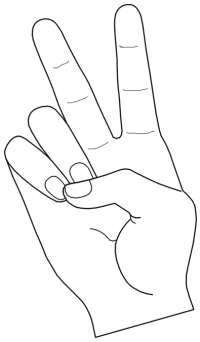
1. Extinguish the flame by turning off the air.

2. Use the wrench test to make sure the flame is OFF. If it is, observe the baseline in the chromatogram window to see whether there is an improvement or no change at all.

3. If baseline noise and high background disappear with the FID flame OFF, the problem is chemical in nature.

4. Isolate the column by capping off the column entrance to the detector with a swagelok-type cap or a nut and septum. Turn the air back on and light the FID flame. If the detector noise is similar to the background that was observed with the flame OFF, the column is suspect.

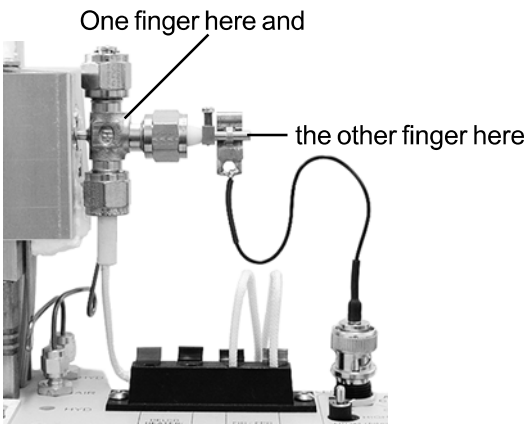
B. Wet Finger Test



1. Make a V sign with the first two fingers of your right hand.

2. Moisten those two fingers (you can achieve sufficient moisture by licking them).

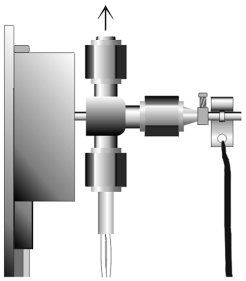
3. Place one finger on the collector electrode, and place the other on bare metal (like the FID detector body or the column oven lid) to ground the collector. Make your contact brief--you need only brush these parts to perform the test. Be careful not to burn yourself; the column oven lid is probably cooler than the FID detector body.



5. Observing the milliVolt reading on the screen. If your contact makes a significant change in the milliVolt reading, then the FID detector electronics are working. The data system signal should jump from zero to the maximum voltage (5,000mV), then come back down when you remove your fingers.

Cleaning the FID

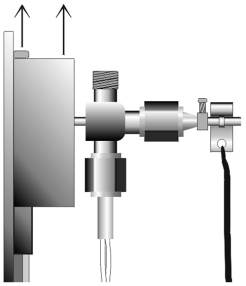
The FID detector rarely requires cleaning or servicing. It may develop a film or coating of combustion desposits in the flameport with extended use. Use the FID detector viewport to check for visible deposits. If you're experiencing problems with your FID detector, try cleaning it, even if you can't see deposits through the viewport.



1. Unscrew the viewport cap nut and examine the flameport interior for coatings or films. If residue is found, the collector electrode and the flameport will need cleaning.

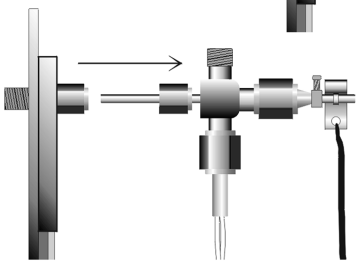
2. Remove flameport assembly from the heater block

   a. Disconnect the FID air supply line at the 1/16" bulkhead fitting.

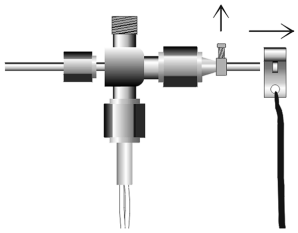


   b. Using a philps head screwdriver, remove the screw on the top of the FID's heater block and pull the aluminum cover up and off.

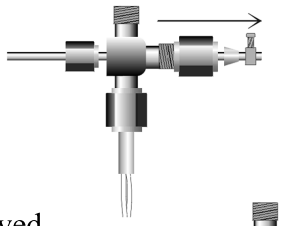
   c. Gently pull off the white insulation to reveal the detector's bulkhead fitting on the column oven wall. Loosen this fitting to disconnect the flameport.



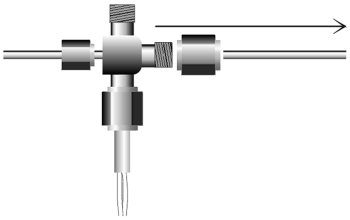
3. Remove the collector electrode



   a. Unclip the electrode lead terminal and slide it off the electrode.



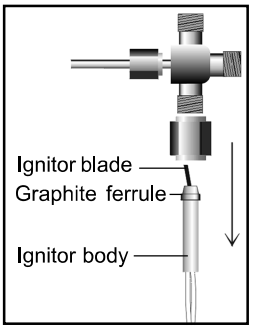
   b. Loosen and remove the nut and ferrule that hold the collector electrode in the flameport body.



   c. Slide the collector electrode out of the nut. Once removed, spin it between your fingers in a piece of sandpaper to clean the stainless steel surface. A wire brush may also be used to scrub the electrode. Once cleaned, set it aside with the ignitor.

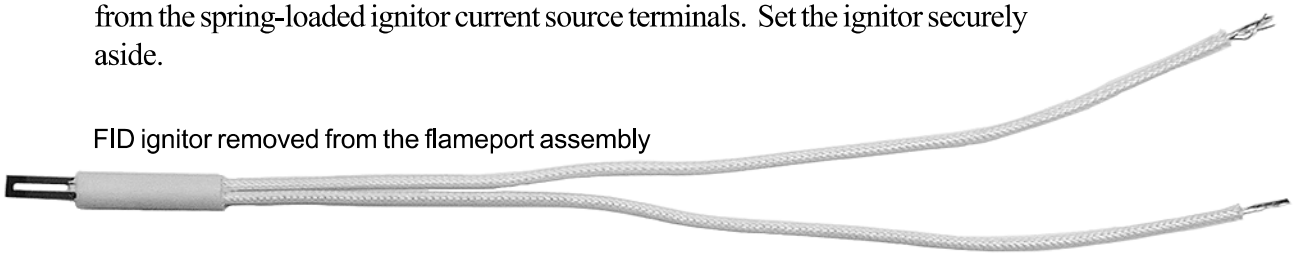
Cleaning the FID continued

4. Remove the FID ignitor element



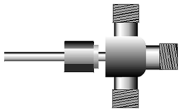
a. The ignitor element is brittle and will break when stressed, so handle the ignitor carefully, mindful of any torque on the blades. While holding the ignitor by the ceramic body with one hand, loosen the 1/4" swagelok-type nut that holds it in place. There is a graphite ferrule inside this nut that secures the ceramic ignitor body when the nut is tightened.

b. Carefully pull the ignitor down out of the flameport. Disconnect the ignitor from the spring-loaded ignitor current source terminals. Set the ignitor securely aside.



FID ignitor removed from the flameport assembly

5. Use a wire brush or a sharp object to remove any residue from the flameport interior, then rinse it with solvent (methanol or methylene chloride), and bake it out in the GC's column oven at 250°C for 10-15 minutes.



Scrape, rinse, and bake out the FID flameport interior

6. Re-assembly

a. Once all the FID parts are cleaned, reverse the disassembly process, starting with the replacement of the ceramic ignitor. Leaving out the cleaning steps, your last step should be reinstalling the flameport assembly onto the heater block. Make sure to position the ignitor so that the blade is slightly below and angled 10-15° toward the jet's tip so that the ignitor will not interfere with the flame or create turbulence.

FID ignitor removed from the flameport; note the slight angle of the blade element

Use the viewport to correctly position the FID ignitor and collector electrode inside the flameport