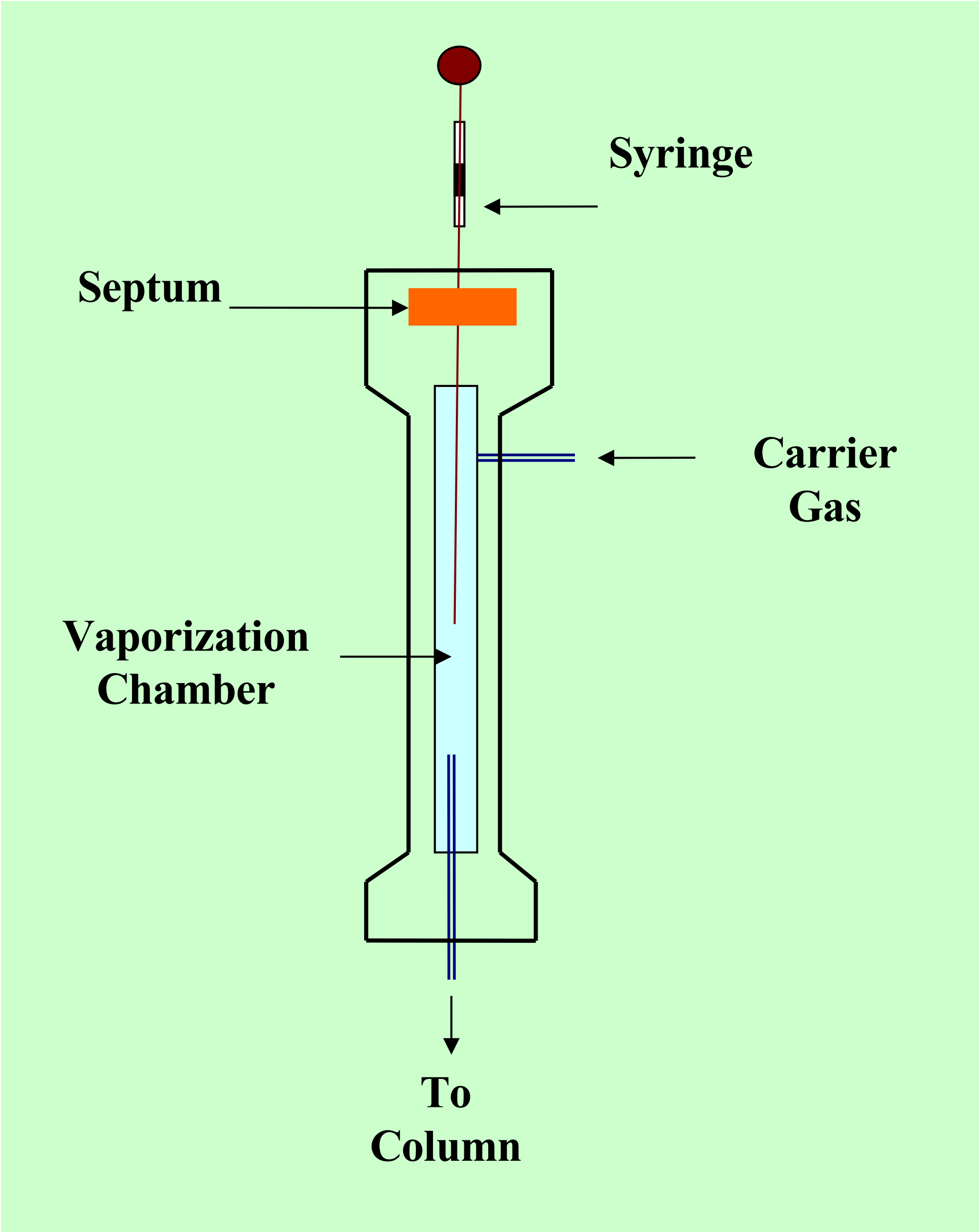
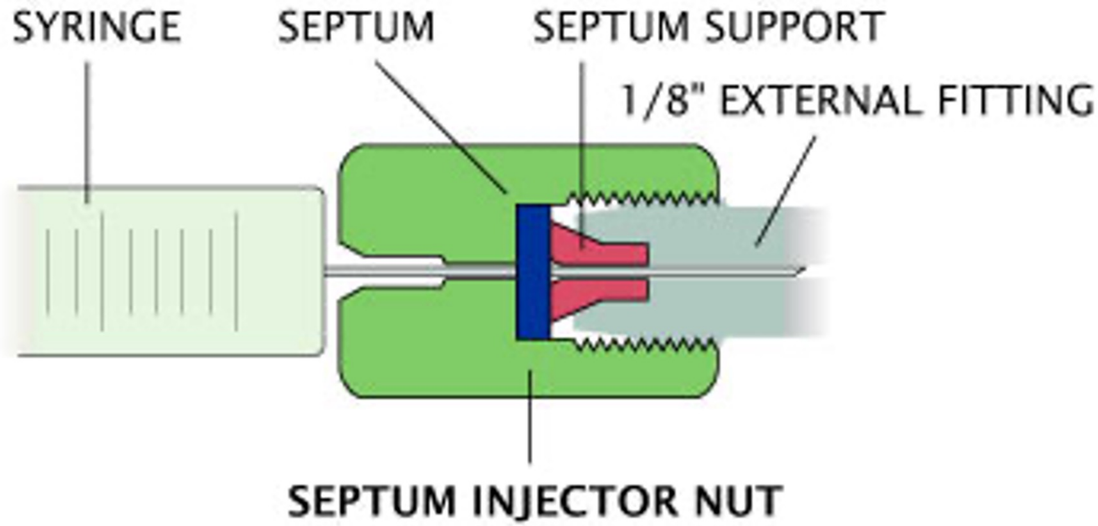
Gas Chromatography

Lecture 2

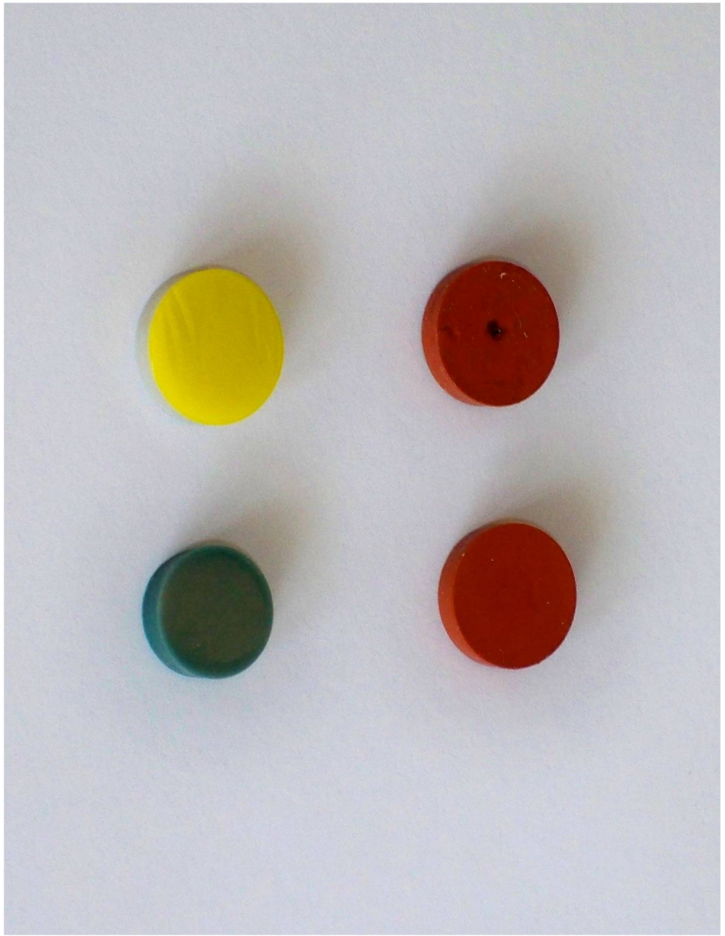
Injectors

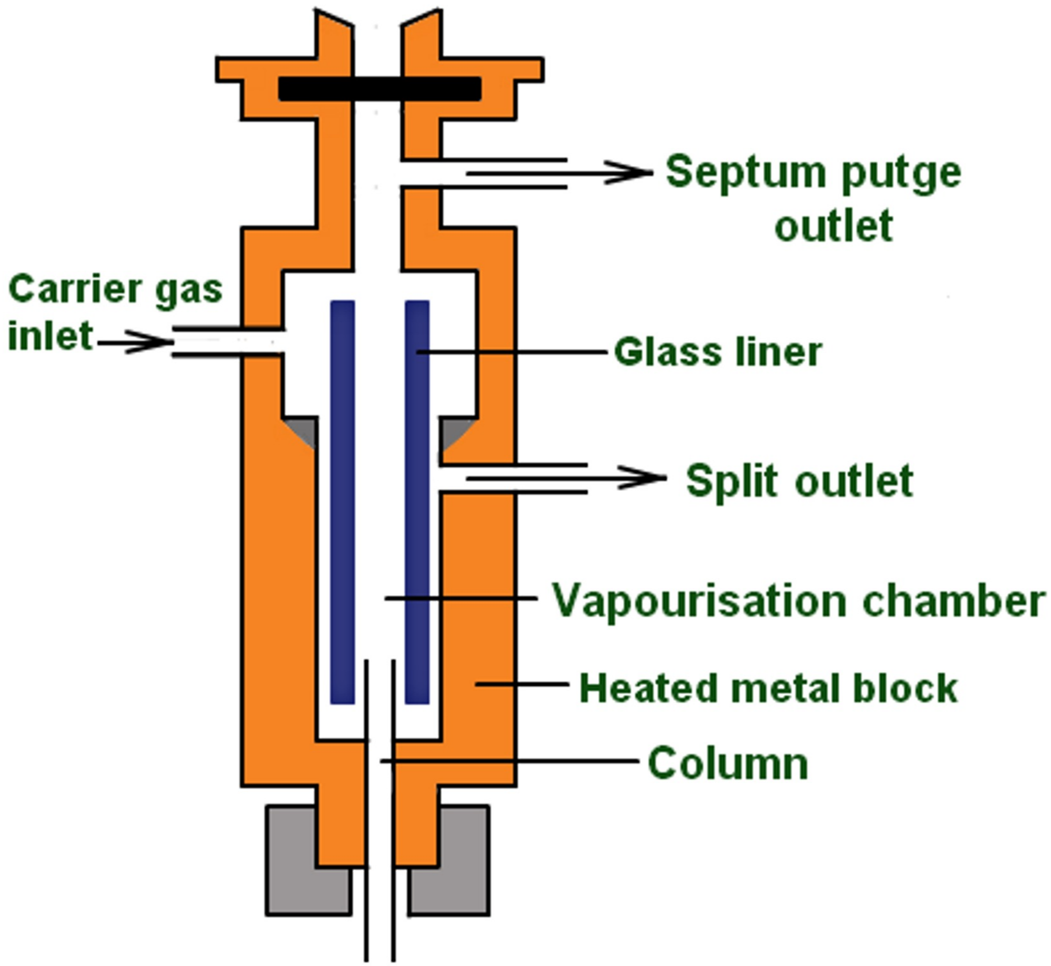
Septum type injectors are the most common. These are composed of a glass tube where vaporization of the sample takes place. The sample is introduced into the injector through a self-sealing silicone rubber septum. The carrier gas flows through the injector carrying vaporized solutes. The temperature of the injector should be adjusted so that flash vaporization of all solutes occurs. If the temperature of the injector is not high enough (at least 50 degrees above highest boiling component), band broadening will take place.


Syringe
Septum
Carrier
Gas
Vaporization
Chamber
To
Column

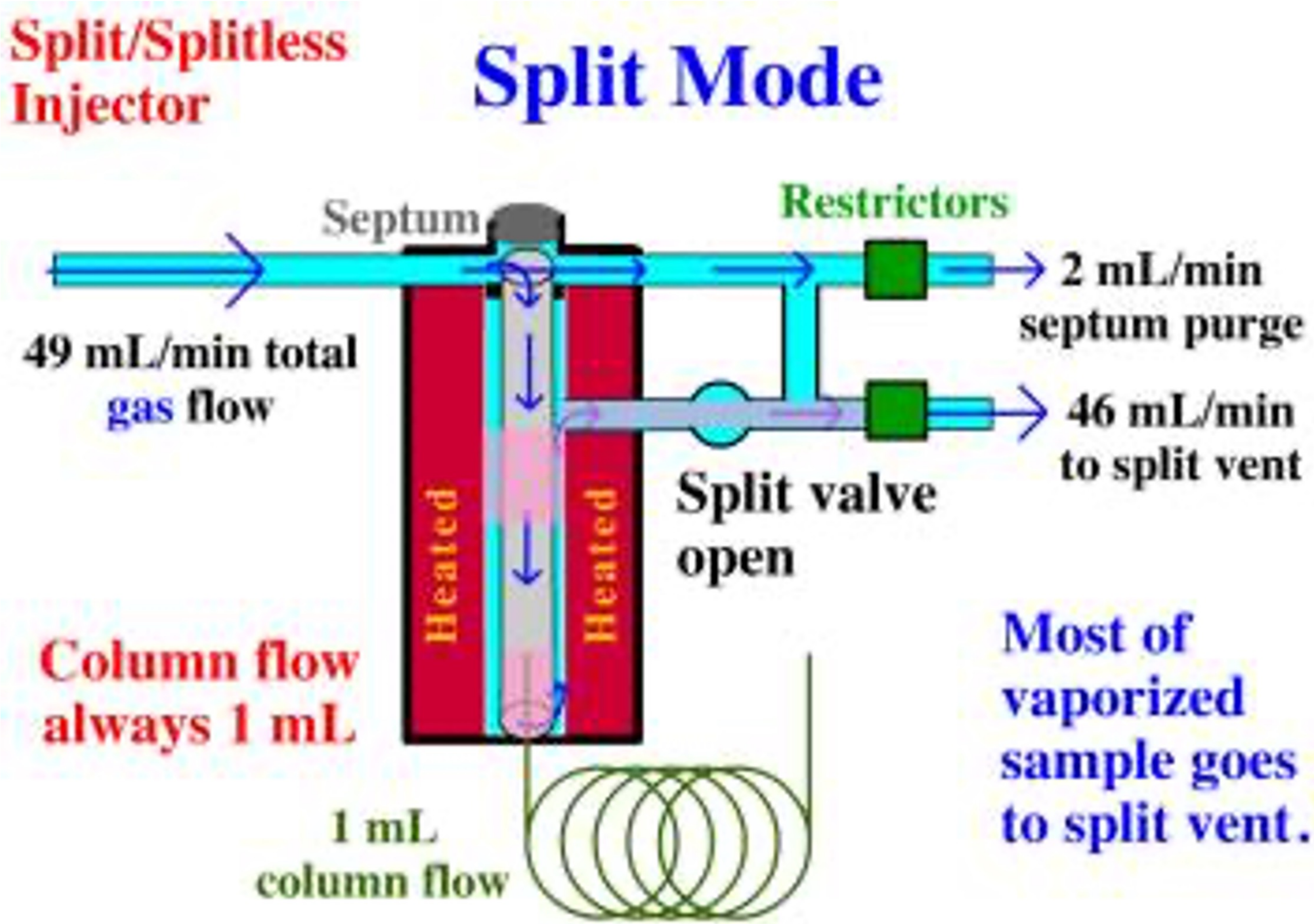

SYRINGE
SEPTUM
SEPTUM SUPPORT
1/8" EXTERNAL FITTING
SEPTUM INJECTOR NUT


MERLIN
S
MICROSEAL

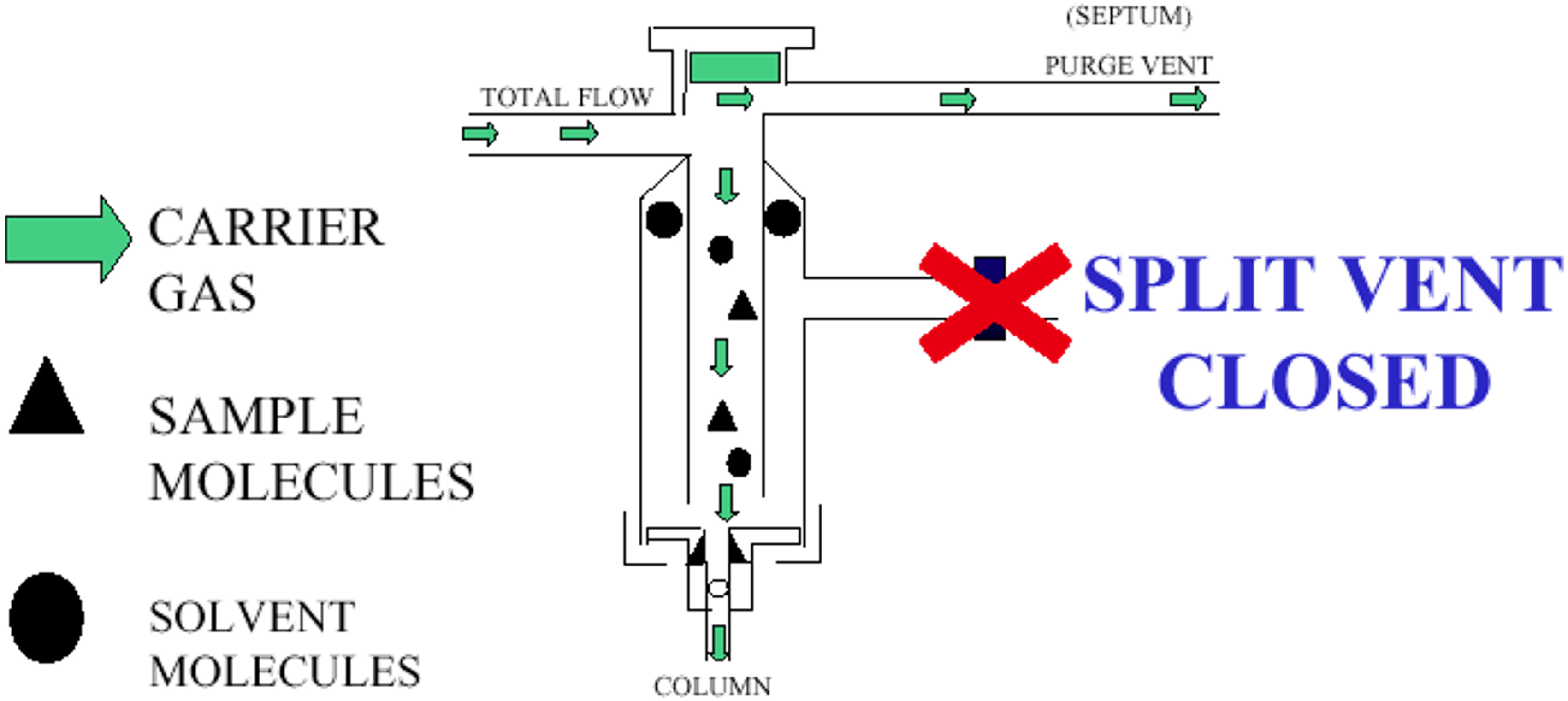


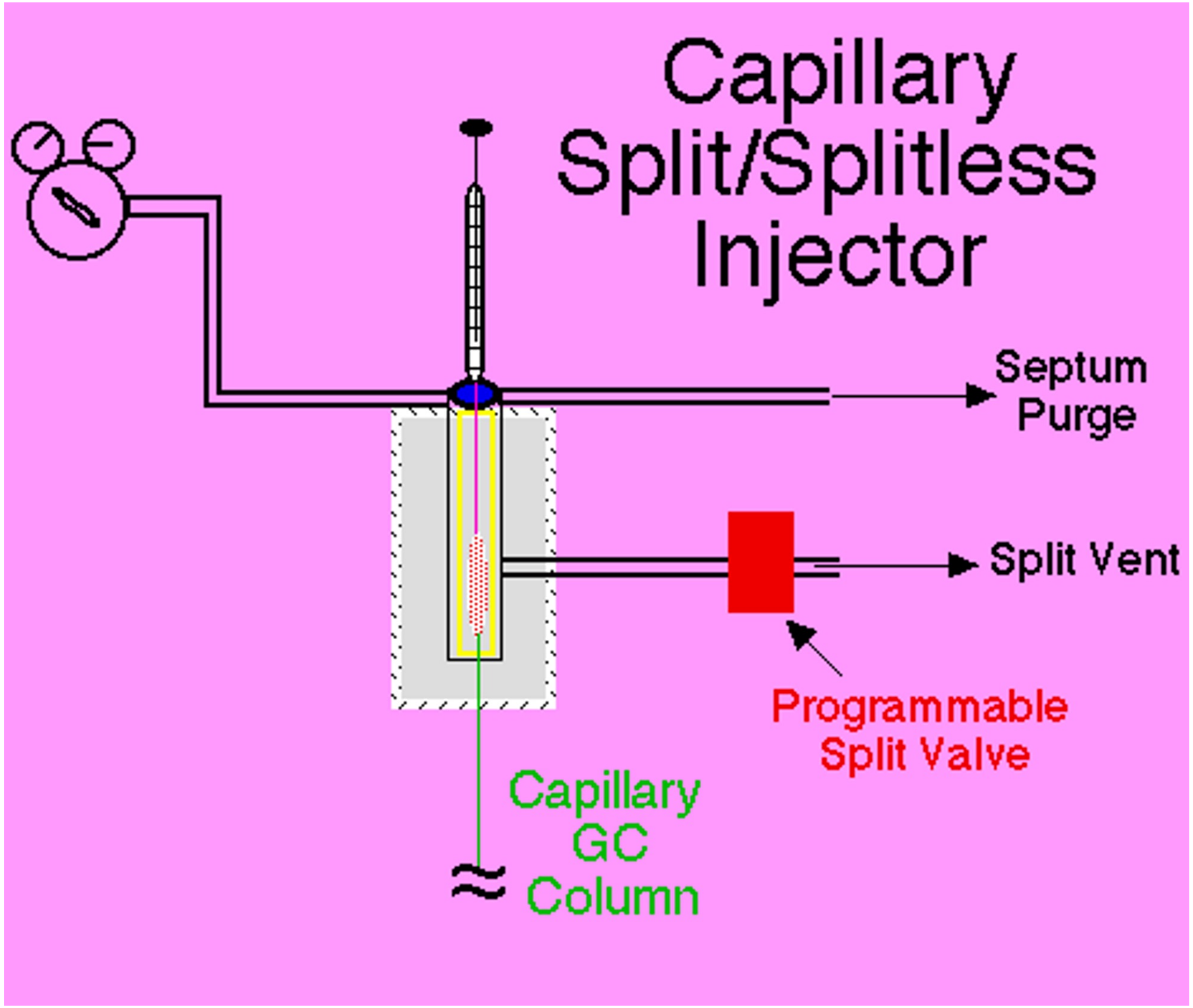


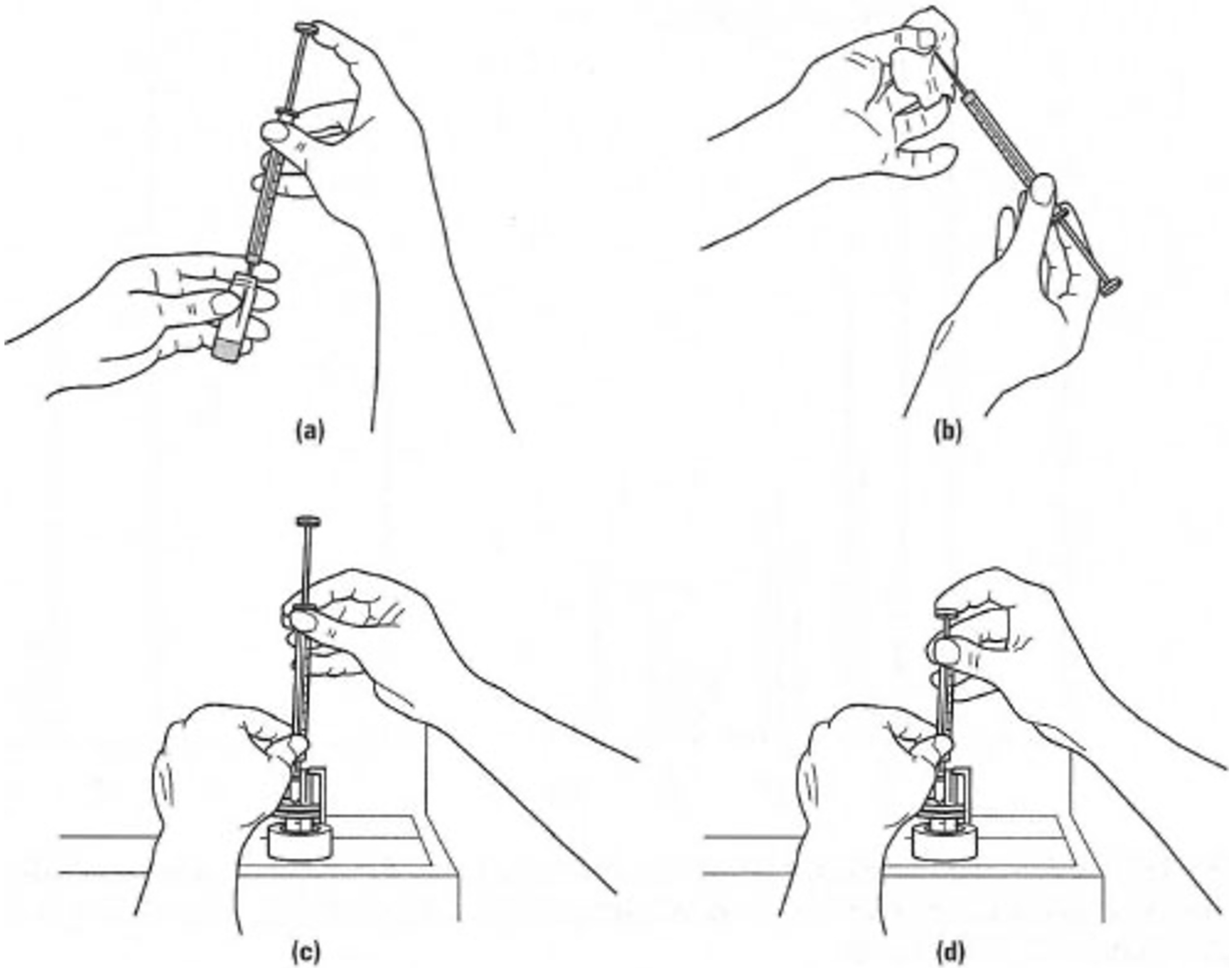
The split / splitless injector


Split/Splitless
Injector
Split Mode
Septum
Restrictors
2 mL/min
septum purge
49 mL/min total
gas flow
46 mL/min
to split vent
Split valve
open
Heated
Heated
Column flow
always 1 mL
Most of
vaporized
sample goes
to split vent.
1 mL
column flow

Splitless Injection




Capillary
Split/Splitless
Injector
Septum
Purge
Split Vent
Programmable
Split Valve
Capillary
GC
Column



(a) (b) (c) (d)

Column Configurations and Ovens

The column in chromatography is undoubtedly the heart of the technique. A column can either be a packed or open tubular. Traditionally, packed columns were most common but fast developments in open tubular techniques and reported advantages in terms of efficiency and speed may make open tubular columns the best choice in the near future. Packed columns are relatively short (~2meters) while open tubular columns may be as long as 30-100 meters

Packed columns are made of stainless steel or glass while open tubular columns are usually made of fused silica. The temperature of the column is adjusted so that it is close to the average boiling point of the sample mixture. However, temperature programming is used very often to achieve better separations. The temperature of the column is assumed to be the same as the oven which houses the column. The oven temperature should be stable and easily changed in order to obtain reproducible results.

Detection Systems

Several detectors are available for use in GC. Each detector has its own characteristics and features as well as drawbacks. Properties of an ideal detector include:

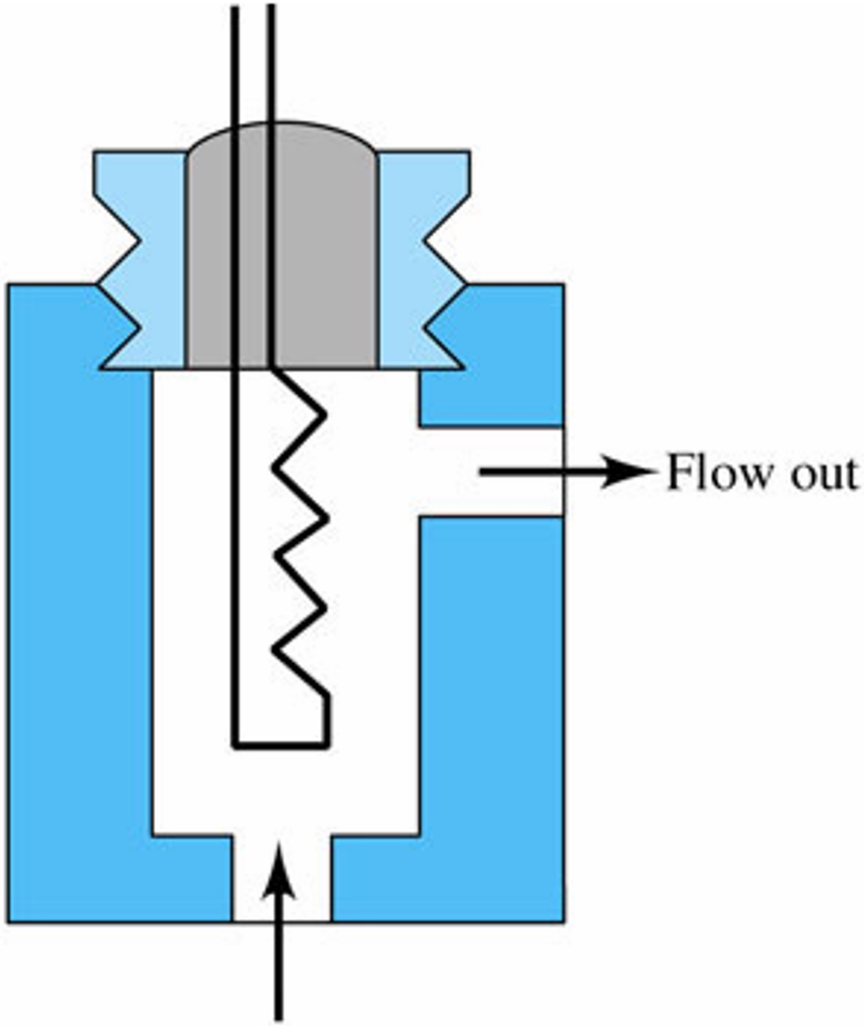
1. High sensitivity
2. Minimum drift
3. Wide dynamic range
4. Operational temperatures up to 400 °C.
5. Fast response time
6. Same response factor for all solutes
7. Good reliability (no fooling)
8. Nondestructive
9. Responds to all solutes (universal)

a. Thermal Conductivity Detector (TCD)

This is a nondestructive detector which is used for the separation and collection of solutes to further perform some other experiments on each purely separated component. The heart of the detector is a heated filament which is cooled by helium carrier gas. Any solute passes across the filament will not cool it as much as helium does because helium has the highest thermal conductivity. This results in an increase in the temperature of the filament which is related to concentration. The detector is simple, nondestructive, and universal but is not very sensitive and is flow rate sensitive.

Table 24-4 Thermal conductivity at 273 K and 1 atm

Gas	Thermal conductivity J/(K • m • s)
H_2	0.170
He	0.141
NH_3	0.021 5
N_2	0.024 3
C_2H_4	0.017 0
O_2	0.024 6
Ar	0.016 2
C_3H_8	0.015 1
CO_2	0.014 4
Cl_2	0.007 6



(a)

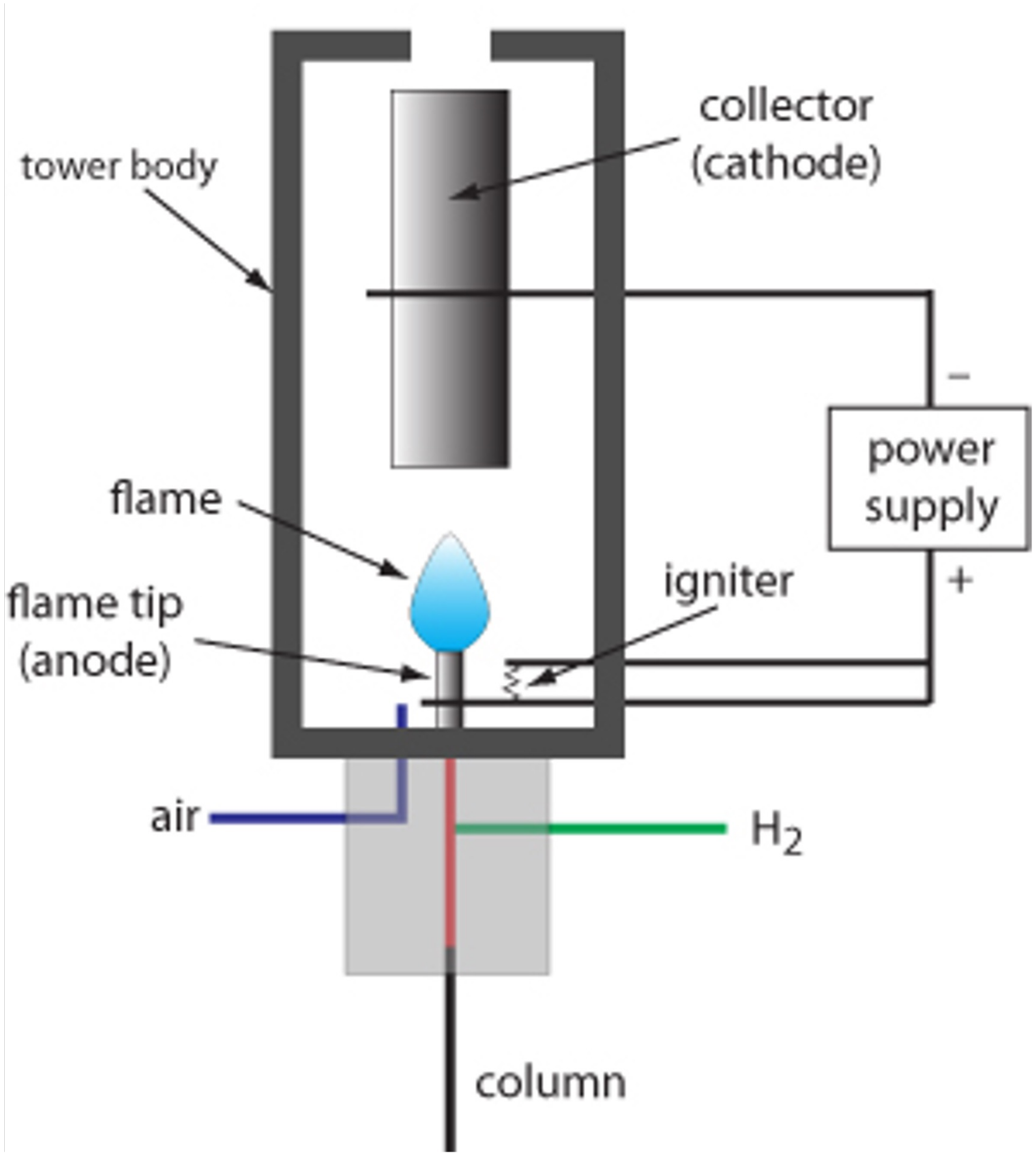
Note that gases should always be flowing through the detector including just before, and few minutes after, the operation of the detector. Otherwise, the filament will melt. Also, keep away any oxygen since oxygen will oxidize the filament and results in its destruction.

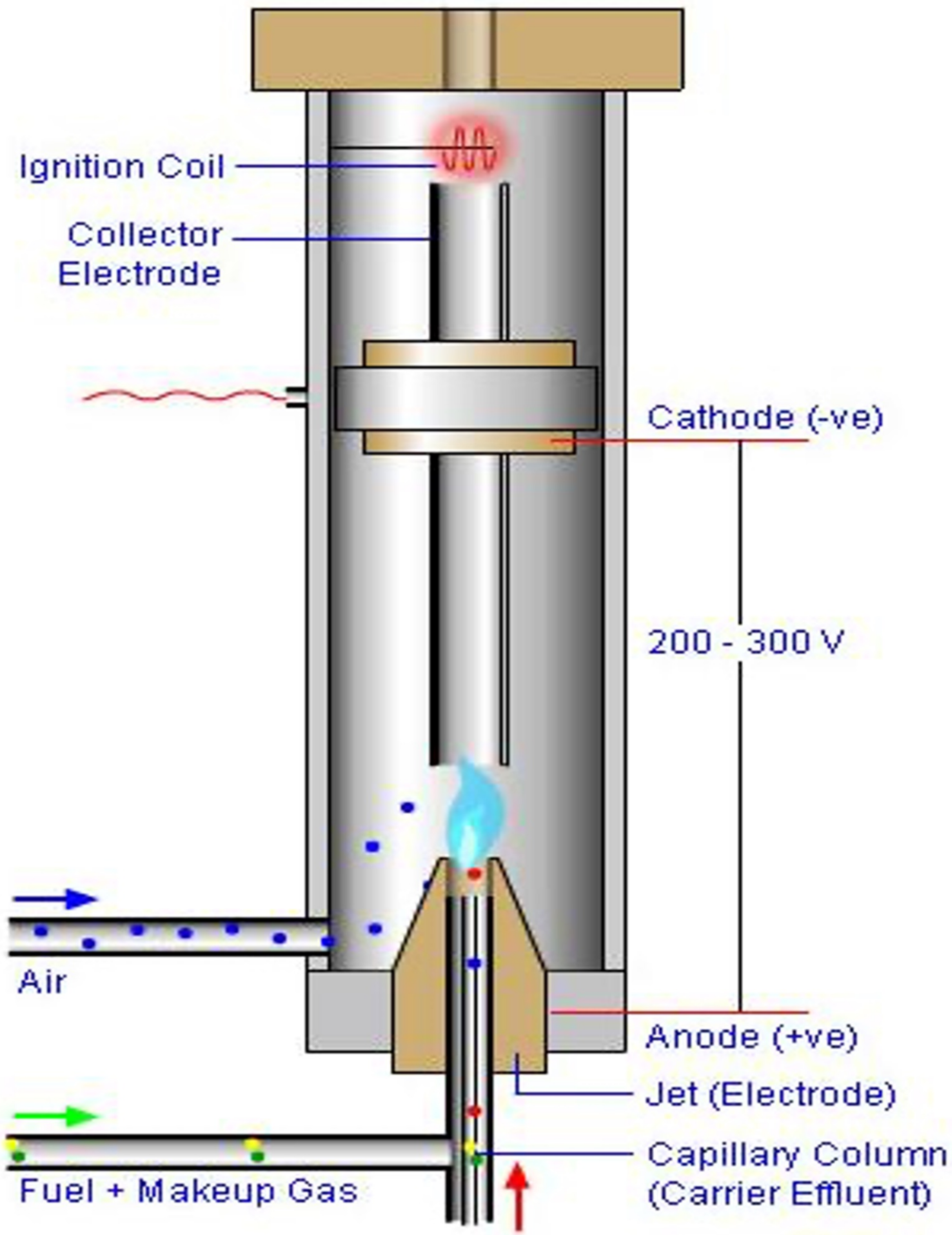
Remember that TCD characteristics include:

1. **Rugged**
2. **Wide dynamic range (10^5)**
3. **Nondestructive**
4. **Insensitive (10^{-8} g/s)**
5. **Flow rate sensitive**

b. Flame Ionization Detector (FID)

This is one of the most sensitive and reliable destructive detectors. Separate two gas cylinders, one for fuel and the other for O_2 or air are used in the ignition of the flame of the FID. The fuel is usually hydrogen gas. The flow rate of air and hydrogen should be carefully adjusted in order to successfully ignite the flame.


tower body
collector
(cathode)
power
supply
flame
flame tip
(anode)
igniter
air
H2
column


Ignition Coil
Collector
Electrode
Cathode (-ve)
200 - 300 V
Air
Anode (+ve)
Jet (Electrode)
Capillary Column
(Carrier Effluent)
Fuel + Makeup Gas

The FID detector is a mass sensitive detector where solutes are ionized in the flame and electrons emitted are attracted by a positive electrode, where a current is obtained.

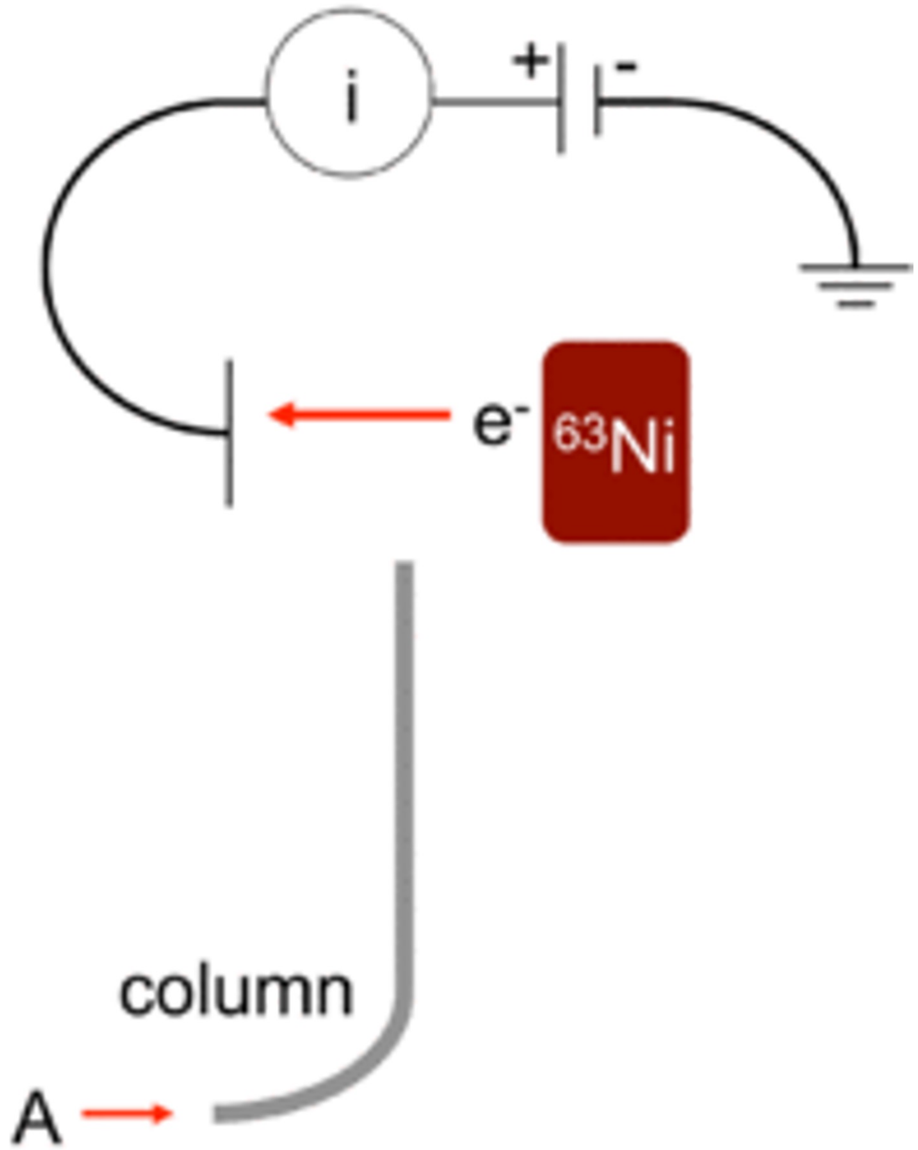
The FID detector is not responsive to air, water, carbon disulfide. This is an extremely important advantage where volatile solutes present in water matrix can be easily analyzed without any pretreatment.

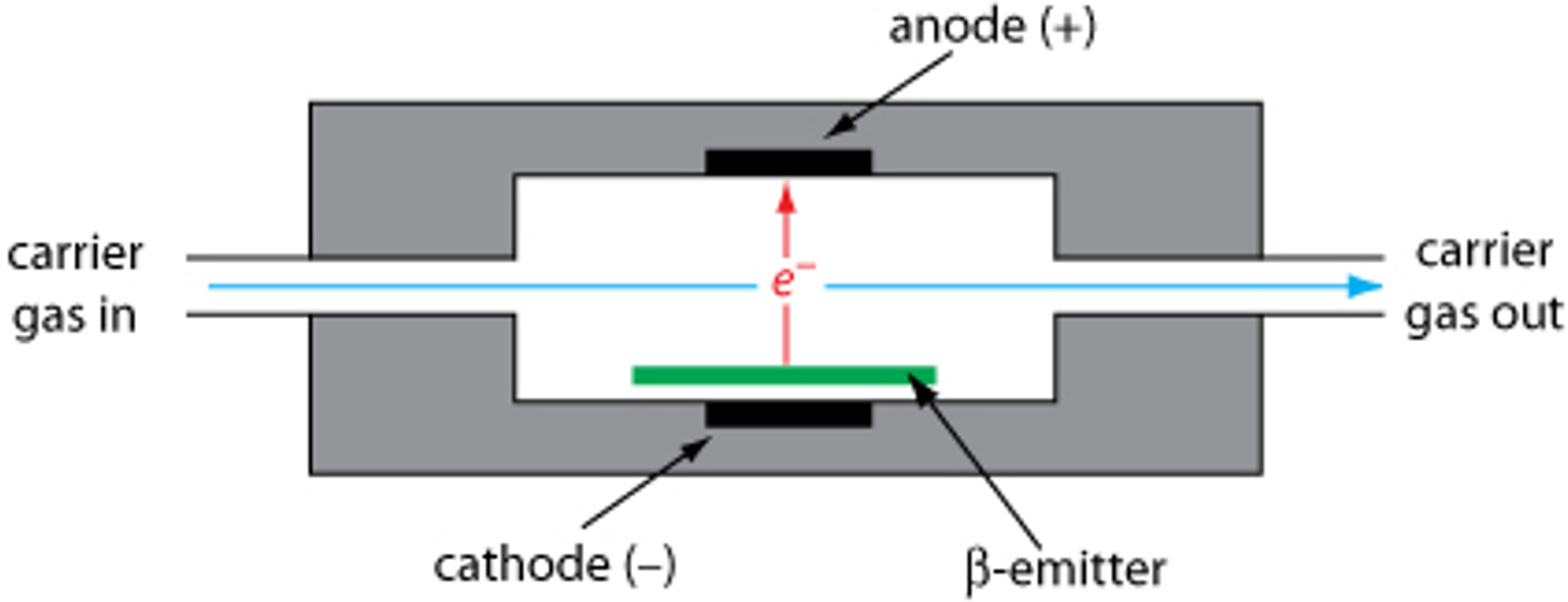
Remember that FID characteristics include:

- Rugged
- Sensitive (10^{-13} g/s)
- Wide dynamic range (10^7)
- Signal depends on number of carbon atoms in organic analytes which is referred to as mass sensitive rather than concentration sensitive
- Weakly sensitive to carbonyl, amine, alcohol, amine groups
- Not sensitive to non-combustibles – H_2O, CO_2, SO_2, NO_x
- Destructive

Electron Capture Detector (ECD)

This detector exhibits high intensity for halogen containing compounds and thus has found wide applications in the detection of pesticides and polychlorinated biphenyls. The mechanism of sensing relies on the fact that electronegative atoms, like halogens, will capture electrons from a β emitter (usually ^{63}Ni). In absence of halogenated compounds, a high current signal will be recorded due to high ionization of the carrier gas, which is N_2, while in presence of halogenated compounds the signal will decrease due to lower nitrogen ionization.


i
+
-
e-
63Ni
column
A


anode (+)
carrier
gas in
e⁻
carrier
gas out
cathode (–)
β-emitter

Remember the following facts about ECD:

1. Electrons from a β-source ionize the carrier gas (nitrogen)
2. Organic molecules containing electronegative atoms capture electrons and decrease current
3. Simple and reliable
4. Sensitive (10^{-15} g/s) to electronegative groups (halogens)
5. Largely non-destructive
6. Insensitive to amines, alcohols and hydrocarbons
7. Limited dynamic range (10^2)
8. Mass sensitive detector