An Executive Summary

Editors' Series: Setting Up a GC Instrument for Analysis

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Gas chromatography instruments must be maintained and set up correctly to provide the best analytical results.

Overview

For a gas chromatography (GC) instrument to provide optimum analytical results, it must be maintained and set up correctly for the required analysis. This article details a step-by-step checklist to ensure a GC instrument is set up correctly. Factors to consider include gas supply, inlet routine maintenance and inspection, column installation, correct column dimensions, and carrier gas types.

Setting Up a GC Instrument

A systematic approach to setting up a GC instrument for analysis involves following the path of carrier gas through the instrument. The path starts at the gas cylinders or generator, moves through the gas filters into the inlet, continues through the electronic pneumatic control system onto the column, and moves into the detector. At each stage, parameters must be set up and maintenance carried out to avoid potential problems (**Figure 1**).

The GC instrument examined in this article was fitted with a flame ionization detector (GC-FID). The carrier gas used can be helium or hydrogen, which are common for capillary columns, or nitrogen, which is typically used for packed columns. Three

Figure 1: The path of carrier gas through a gas chromatography instrument.

- Gas supply - cylinders vs. generators, gas quality, correct use of gas filters
- Inlet - routine inlet maintenance and inspection
- Column - correct installation and conditioning
- Settings - setting the correct column dimensions and carrier gas types

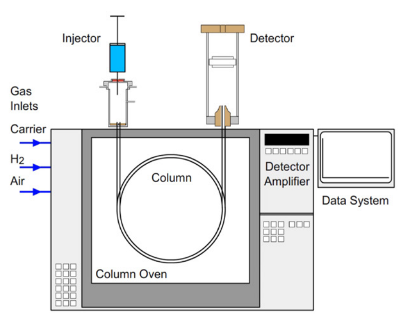


Table I: Makeup gases combination for combustion detectors

For a GC-FID

- Carrier – helium or hydrogen (capillary columns) or nitrogen (packed columns)
- Detector gases – hydrogen (fuel), air (oxidant), helium or nitrogen (makeup)
- Always ensure you have enough gas to run the analysis

Detector	Carrier Gas	Makeup Gas	
		First Choice	Second Choice
FID	Hydrogen Helium Nitrogen	Nitrogen	Helium
NPD	Helium Nitrogen	Nitrogen	Helium
FPD	Hydrogen Helium Nitrogen	Nitrogen	***

Gases for Combustion Detectors. *** *No recommended second choice*

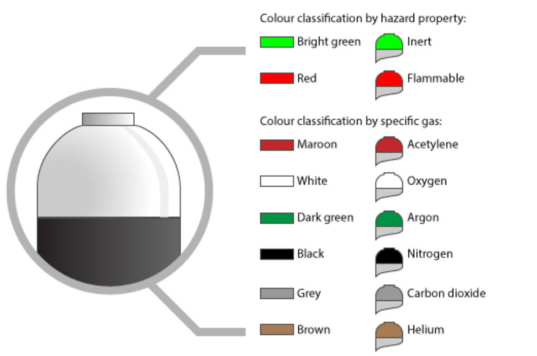
Table II: Makeup gases combination for non-combustion detectors

Detector	Carrier Gas	Makeup Gas	
		First Choice	Second Choice
ECD	Hydrogen Helium	Argon/methane	Nitrogen
	Nitrogen	Nitrogen	Argon/methane
TCD	Hydrogen Helium Nitrogen	**	**

Gases for Non-Combustion Detectors. ** *Must be same as carrier and reference gas*

Figure 2: Controlling gas supply: regulators.

- Cylinder colors can often be matched with colors on the correct regulator



additional gases are required for the FID: a fuel gas (hydrogen), an oxidizer (air), and a makeup gas. The makeup gas choice depends on the carrier gas used. **Table I** shows makeup gas combinations typically used for combustion detectors, while **Table II** shows makeup gas choices for non-combustion detectors.

Gas cylinders. Traditionally, gases have been supplied from high-pressure cylinders, although gas generators have become increasingly common. Gas cylinders are controlled by regulators. In the event of a leak, the regulator increases gas flow, which could result in unsafe conditions. As an example, if the gas was hydrogen and a detector with a flame was in close proximity, an explosion could occur.

Hydrogen generators, in contrast, generate a fixed required amount of gas, which reduces the risk of large amounts of gas being expelled into the lab atmosphere. Both cylinders and generators have associated advantages and disadvantages, particularly related to the cost and safety of each systems.

Safety considerations when using gas cylinders include:

- Move cylinders using approved cylinder trolleys and use safety caps during transport.
- Use tags to indicate the cylinder conditions and to identify each as full, used, or empty.
- At least 200 psi residual gas should be present in a depleted cylinder.
- Chain or strap gas cylinders to the wall when not in use. Store cylinder with the valve close and in a place that does not exceed 51 °C (125 °F).

Gas regulators. The regulator controls the pressure of the gas going to the GC instrument and contains two gauges. The high-pressure gauge indicates the pressure or amount of gas inside the cylinder. It is good practice to mark this gauge at 10% of the starting pressure to provide a warning point for changing the cylinder and avoid it running dry during a run. The low-pressure gauge is adjustable using a regulator knob and indicates the pressure going to the GC system. Most GC instruments operating with an electronic pneumatic control (EPC) unit will operate on a minimum pressure basis. An on/off tap allows gas to move up to the regulator from the cylinder, and there are several designs for on/off taps, some requiring a key.

The connections will also have different threads, depending on the gas they are used for; some may be left- or right-hand threaded. Threads should be inspected for damage that could cause leaks. One should never use PTFE tape on a regulator fitting to stop a leak.

Regulators must be correctly pressure rated for the gas it is supplying by a cylinder or a generator. Regulators and cylinders are often color coded for different gases as seen in **Figure 2**.

Automatic switchover systems ensure a continuous supply of gas to avoid running out of gas partway through an analysis. When switching from one cylinder to the next, pressure balance must be monitored. As with any piece of lab equipment, proper fitting and tightening of fittings and other components is critical. Never over-tighten and always follow the fitting supplier's instructions exactly.

Carrier gases. After connecting and adjusting the air cylinder regulator, the next step is to connect and adjust

Figure 3: Order of gas filter installations.

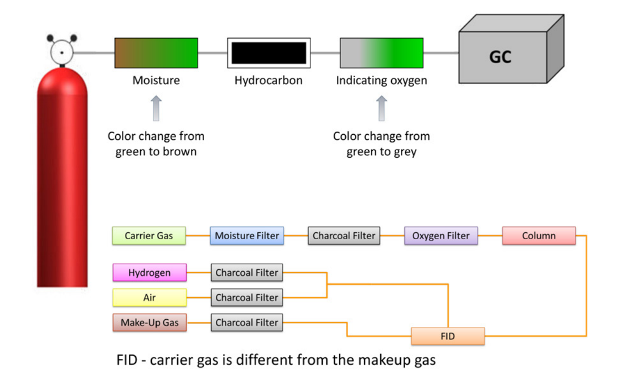


Figure 4: Splitless injection.

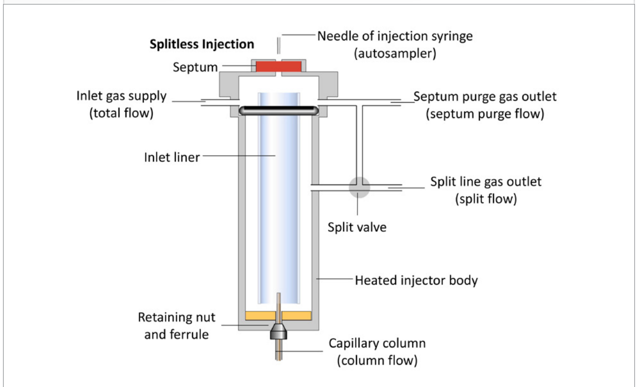
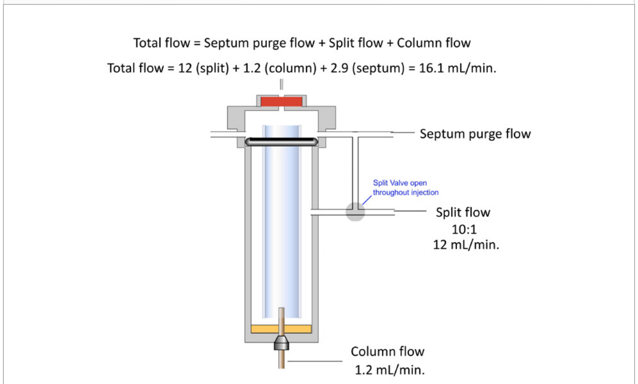


Figure 5: Split injection.



the carrier and makeup gas. These gases should be checked to ensure adequate supply levels are indicated by the high-pressure gauges for each.

Gas filters. The next components to consider in the GC system are the gas filters (traps) that remove contaminants, typically moisture, oxygen, and hydrocarbons from the gas supply. Filters are preferred even when using high-purity gas sources.

Not using the correct filter and in the proper order is among the most common mistakes for filter installation. **Figure 3** shows the order of installation of typical gas filters in a system. Oxygen and moisture traps generally get replaced every six months while hydrocarbon filters are replaced annually.

Depending on the number and location of GC instruments, two trap configurations are generally used. The first is a large-capacity trap on the main line and the second is a set of traps for each GC system. It is best practice to install gas traps as close to the GC instrument as possible. The number of fittings in gas lines should be minimized. The more fittings there are, the greater the risk of ingress of oxygen, moisture or other contaminants.

Of the various flows displayed in a GC instrument, column flow is the most important, as it governs all of the other system flows. Inlet flow can be configured as split injection or splitless injection. In splitless injections, the total amount of gas is a combination of column flow and any septum purge set (**Figure 4**).

The split mode is more complicated as the total flow consists of the column flow, septum purge flow, and the split flow (**Figure 5**). Leaks can occur in the inlet.

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Autosampler. An autosampler should be considered part of the GC flow path and must be correctly set up. Bent needles are a common issue with autosamplers and are typically caused by:

- Using a needle that cannot penetrate the vial septum because it is too narrow and not strong enough.
- Using septum material that is too hard.
- Tightening the septum cap too much.

Plungers can become damaged from long-term use or contamination in the syringe barrel caused by a poor choice of wash solvent. Since most GC systems allow for the use of two wash solvents, it is a good idea to use one that is water miscible and another that is organic. A good autosampler program sequence includes three washes of sample to waste, three pumps of the sample to prime