520 Gases

Gas requirements

Gases for packed columns

The carrier gas you use depends upon the type of detector and the performance requirements. Table 520-1 lists gas recommendations for packed column use. In general, makeup gases are not required with packed columns.

Table 520-1 Gas Recommendations for Packed Columns

Detector	Carrier Gas	Comments	Detector anode purge or reference gas
Electron Capture	Nitrogen	Maximum sensitivity	Nitrogen
	Argon/ Methane	Maximum dynamic range	Argon/Methane
Flame Ionization	Nitrogen	Maximum sensitivity	Hydrogen and air for detector
	Helium	Acceptable alternative	
Flame Photometric	Hydrogen		Hydrogen and air for detector
	Helium		
	Nitrogen		
	Argon		
Nitrogen-Phosphorus	Helium	Optimum performance	Hydrogen and air for detector
	Nitrogen	Acceptable alternative	
Thermal Conductivity	Helium	General use	Reference must be same as carrier
	Hydrogen	Maximum sensitivity (Note A)	
	Nitrogen	Hydrogen detection (Note B)	
	Argon	Maximum hydrogen sensitivity (Note B)	

Note A: Slightly greater sensitivity than helium. Incompatible with some compounds.
Note B: For analysis of hydrogen or helium. Greatly reduces sensitivity for other compounds.

Gases for capillary columns

When used with capillary columns, GC detectors require a separate makeup gas for optimum sensitivity. For each detector and carrier gas, there is a preferred choice for makeup gas. Table 520-2 lists gas recommendations for capillary columns.

Table 520-2 Gas Recommendations for Capillary Columns

Detector	Carrier gas	Preferred makeup gas	Second choice	Detector anode purge or reference gas
Electron Capture	Hydrogen	Argon/Methane	Nitrogen	Anode purge must be same as makeup
	Helium	Argon/Methane	Nitrogen	
	Nitrogen	Nitrogen	Argon/Methane	
	Argon/ Methane	Argon/Methane	Nitrogen	
Flame Ionization	Hydrogen	Nitrogen	Helium	Hydrogen and air for detector
	Helium	Nitrogen	Helium	
	Nitrogen	Nitrogen	Helium	
Flame Photometric	Hydrogen	Nitrogen		Hydrogen and air for detector
	Helium	Nitrogen		
	Nitrogen	Nitrogen		
	Argon	Nitrogen		
Nitrogen-Phosphorus	Helium	Nitrogen	Helium**	Hydrogen and air for detector
	Nitrogen	Nitrogen	Helium**	
Thermal Conductivity	Hydrogen*	Must be same as carrier and reference gas	Must be same as carrier and reference gas	Reference must be same as carrier and makeup
	Helium			
	Nitrogen			

* When using hydrogen with a thermal conductivity detector, vent the detector exhaust to a fume hood or a dedicated exhaust to avoid buildup of hydrogen gas.

**Helium is not recommended as a makeup gas at flow rate> 5 mL/min Flow rates above 5 mL/min shorten detector life.

Gas purity

Some gas suppliers furnish “instrument” or “chromatographic” purity grades of gas that are specifically intended for chromatographic use. We recommend these grades for use with the GC.

Generally, all gas supplies used should be in the 99.995% to 99.9995% purity range. Only very low levels (≤ 0.5 ppm) of oxygen and total hydrocarbons should be present. Oil-pumped air supplies are not recommended because they may contain large amounts of hydrocarbons.

The addition of high-quality moisture and hydrocarbon traps immediately after the main tank pressure regulator is highly recommended. Refer to the next section, The gas plumbing, for more information on using traps.

Table 520-3 Gas Purity Recommendations

Carrier gases and capillary makeup gases	
Helium	99.9995%
Nitrogen	99.9995%
Hydrogen	99.9995%
Argon/Methane	99.9995%
Detector support gases	
Hydrogen	99.9995%
Air (dry)	Zero-grade or better

The gas plumbing

WARNING All compressed gas cylinders should be securely fastened to an immovable structure or permanent wall. Compressed gases should be stored and handled in accordance with the relevant safety codes.

Gas cylinders should not be located in the path of heated oven exhaust.

To avoid possible eye injury, wear eye protection when using compressed gas.

Follow the general plumbing diagram when preparing gas supply plumbing.

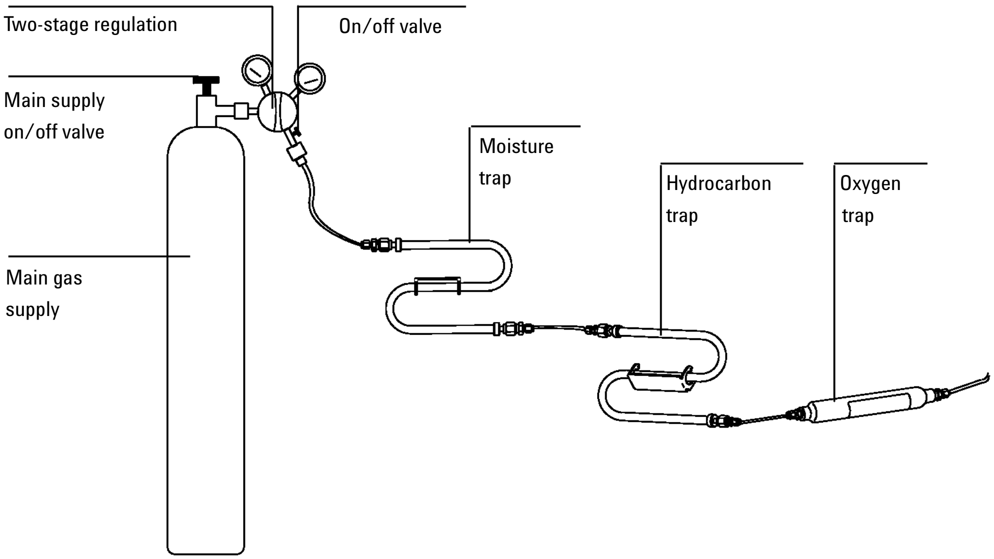

Figure 520-1 General plumbing diagram

- Two-stage regulators are strongly recommended to eliminate pressure surges. High-quality, stainless-steel diaphragm-type regulators are especially recommended.

- On/off valves mounted on the outlet fitting of the two-stage regulator are not essential but are very useful. Be sure the valves have stainless-steel, packless diaphragms.
- FID, FPD, and NPD detectors require a dedicated air supply. Operation may be affected by pressure pulses in air lines shared with other devices.
- Flow- and pressure-controlling devices require at least 10 psi (138 kPa) pressure differential across them to operate properly. Source pressures and capacities must be high enough to ensure this.
- Auxiliary pressure regulators should be located close to the GC inlet fittings. This insures that the supply pressure is measured at the instrument rather than at the source; pressure at the source may be different if the gas supply lines are long or narrow.

Supply tubing for carrier and detector gases

Caution Do not use methylene chloride or other halogenated solvent to clean tubing that will be used with an electron capture detector. They will cause elevated baselines and detector noise until they are completely flushed out of the system.

Gases should be supplied to the instrument only through preconditioned copper tubing (part no. 5180-4196). Do not use ordinary copper tubing—it contains oils and contaminants.

Caution Do not use plastic tubing to supply detector and inlet gases to the GC. It is permeable to oxygen and other contaminants that can damage columns and detectors, and can melt if near hot exhaust or components.

The tubing diameter depends upon the distance between the supply gas and the GC and the total flow rate for the particular gas. One-eighth-inch tubing is adequate when the supply line is less than 15 feet (4.6 m) long.

Use larger diameter tubing (1/4-inch) for distances greater then 15 feet (4.6 m) or when multiple instruments are connected to the same source. You should also use larger diameter tubing if high demand is anticipated (for example, air for an FID).

Be generous when cutting tubing for local supply lines—a coil of flexible tubing between the supply and the instrument lets you move the GC without moving the gas supply. Take this extra length into account when choosing the tubing diameter.

Two-stage pressure regulators

To eliminate pressure surges, use a two-stage regulator with each gas tank. Stainless steel, diaphragm-type regulators are recommended.

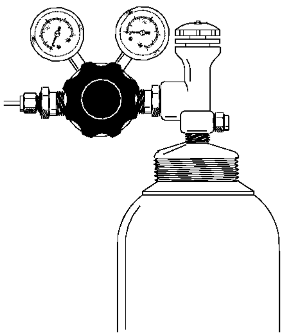

Figure 520-2 Two-stage pressure regulator

The type of regulator you use depends upon gas type and supplier. The *Chemical Analysis Consumables and Accessories* catalog contains information to help you identify the correct regulator, as determined by the Compressed Gas Association (CGA). Agilent Technologies offers pressure-regulator kits that contain all the materials needed to install regulators properly.

Pressure regulator-gas supply tubing connections

The pipe-thread connection between the pressure regulator outlet and the fitting to which you connect the gas tubing must be sealed with Teflon tape. **Instrument grade** Teflon tape (part no. 0460-1266), from which volatiles have been removed, is recommended for all fittings. Do not use **pipe dope** to seal the threads; it contains volatile materials that will contaminate the tubing.

Traps

Using chromatographic-grade gases insures that the gas in your system is pure. However, for optimum sensitivity, it is highly recommended that you install high-quality traps to remove traces of water or other contaminants. After installing a trap, check the gas supply lines for leaks.

Table 520-4 Recommended Traps

Description	Part No.
Preconditioned moisture trap: metal casing, s-shaped trap for carrier gas cleanup. Contains Molecular Sieve 5A, 45/60 mesh, and 1/8-inch fittings.	5060-9084
Hydrocarbon trap: metal casing, s-shaped trap filled with 40/60 mesh activated charcoal, and 1/8-inch fittings	5060-9096
Oxygen trap (for carrier and ECD gases): metal casing, and 1/8-inch brass fittings. Oxygen trap cannot be reconditioned.	3150-0414

Moisture in carrier gas damages columns. We recommend a type 5A Molecular Sieve trap after the source regulator and before any other traps.

A hydrocarbon trap removes organics from gases. It should be placed after a molecular sieve trap and before an oxygen trap, if they are present.

An oxygen trap removes 99% of the oxygen from a gas plus traces of water. It should be last in a series of traps. Because trace amounts of oxygen can damage columns and degrade ECD performance, use an oxygen trap with carrier and ECD gases. Do not use it with FID, FPD, or NPD fuel gases.

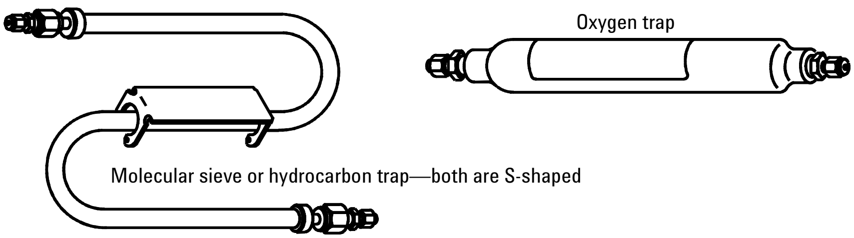

Figure 520-3 Traps

Cryogenic cooling requirements

Cryogenic cooling allows you to cool the oven below ambient temperature. A solenoid valve introduces liquid coolant, either carbon dioxide (CO_2) or nitrogen (N_2), to cool the oven to the desired temperature.

CO_2 and N_2 require different hardware. You must replace the entire valve assembly if you want to change coolants. The liquid CO_2 valve kit is part no. G1565-65510 and the liquid N_2 kit is part no. G1566-65517.

Choosing a coolant

When selecting a coolant, consider these points:

- The lowest temperature you need to reach
- How frequently you will use cryogenic cooling
- The availability and price of coolant
- The size of the tanks in relation to the size of the laboratory
- Liquid N_2 cools reliably to –80°C
- Liquid CO_2 cools reliably to –40°C

CO_2 is the choice for *infrequent* cryogenic cooling because it does not evaporate and is less expensive than N_2. However, a tank of CO_2 contains much less coolant than a tank of N_2 and more CO_2 is used for the same amount of cooling.

Although liquid N_2 evaporates from the tank regardless of frequency of use, N_2 tanks contain more coolant than do CO_2 tanks and therefore may be better for frequent use.

Using carbon dioxide

WARNING Pressurized liquid CO_2 is a hazardous material. If CO_2 escapes its container, it exits at high pressure and low temperatures that can be dangerous to personnel. CO_2 in high concentrations is toxic to humans. Consult your local supplier for recommended safety precautions and delivery system design.

Caution Liquid CO_2 should not be used as a coolant for temperatures below –40°C because the expanding liquid may form solid CO_2—dry ice—in the GC oven. If dry ice builds up in the oven, it can seriously damage the GC.

Liquid CO_2 is available in high-pressure tanks containing 50 pounds of liquid. The CO_2 should be free of particulate material, oil, and other contaminants. These contaminants could clog the expansion orifice or affect the proper operation of the GC.

Additional requirements for the liquid CO_2 system include:

- The tank must have an internal dip tube or eductor tube to deliver liquid CO_2 instead of gas (see Figure 520-4).
- The liquid CO_2 must be provided to the GC at a pressure of 700 to 1,000 psi at a temperature of 25°C.
- Use 1/8-inch diameter heavy-wall stainless steel tubing for supply tubing. The tubing should be between 5 to 50 feet long.
- Coil and fasten the ends of the tubing to prevent it from “whipping” if it breaks.
- Do not install a pressure regulator on the CO_2 tank, as vaporization and cooling would occur in the regulator instead of the oven.
- Do not use a padded tank (one to which another gas is added to increase the pressure).

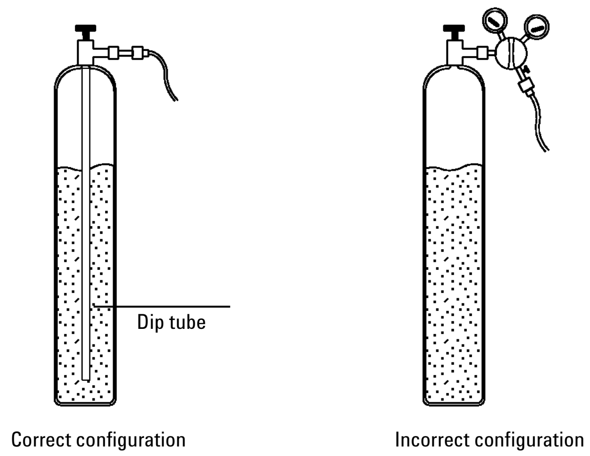

Figure 520-4 Correct and incorrect liquid CO_2 tank configuration

WARNING Do not use copper tubing or thin-wall stainless steel tubing with liquid CO_2. Both harden at stress points and may explode.

Using liquid nitrogen

WARNING Liquid nitrogen is a hazard because of the extremely low temperatures and high pressures that may occur in improperly designed supply systems.

Liquid nitrogen can present an asphyxiant hazard if vaporizing nitrogen displaces oxygen in the air. Consult local suppliers for safety precautions and design information.

Liquid nitrogen is supplied in insulated Dewar tanks. The correct type for cooling purposes is a *low-pressure* Dewar equipped with a dip tube—to deliver liquid rather than gas—and a safety relief valve to prevent pressure build-up. The relief valve is set by the supplier at 20 to 25 psi.

WARNING If liquid nitrogen is trapped between a closed tank valve and the cryo valve on the GC, tremendous pressure will develop and may cause an explosion. For this reason, keep the delivery valve on the tank open so that the entire system is protected by the pressure relief valve.

To move or replace a tank, close the delivery valve and carefully disconnect the line at either end to let residual nitrogen escape.

Additional requirements for the liquid N_2 system include:

- Nitrogen must be provided to the GC as a liquid at 20 to 30 psi.
- The supply tubing for liquid N_2 must be *insulated*. Foam tubing used for refrigeration and air-conditioning lines is suitable for insulation. Since pressures are low, *insulated* copper tubing is adequate.
- The liquid nitrogen tank should be close (only 5 to 10 feet) to the GC to insure that liquid, not gas, is supplied to the inlet.

Supplying valve actuator air

Some valves use pressurized air for actuation (others are electrically or manually driven). Actuator air must be free of oil, moisture, and particulates. It can be supplied from a dried regulated cylinder, although “house” air supplies or air from a compressor are acceptable.

Most valves require 20 to 40 psi of pressure to operate. High-pressure valves may require 65 to 70 psi.

Valves require a dedicated air supply. Do not share valve air supplies with detectors.

See "Valve Control" in the Agilent 6890 GC Operating Manual/CD-ROM for more valve requirements.