



# Frequently Asked Questions about Chromatographic Gases

A practical primer

# Chromatographic Gases

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# Frequently Asked Questions

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# Frequently Asked Questions about Chromatographic Gases

## What is this document?

It's a cooperative effort between Air Products and Chemicals, Inc. and Agilent Technologies. It is our attempt to bring two complementary viewpoints to bear on one of the major issues in gas chromatography: how to select and use carrier, fuel, and other gases to get the best performance from a gas chromatograph (GC).

This publication is in a question-and-answer format, something like the FAQ (frequently asked questions) files familiar to Internet users. The topics are grouped into Cylinder Safety, General Considerations, Gas Selection, Gas Purity, Gas Distribution, and Gas Cylinders. Air Products and Agilent addresses in the U.S. and Europe are given on the last page.

# Cylinder Safety

## How can I secure and move gas cylinders?

Gas cylinders are heavy and unwieldy. To avoid personal injury or property damage caused by a falling cylinder, these precautions must be followed:

- Always secure the gas cylinder to a stable support using an appropriate clamp so that it cannot be knocked over. Common supports are bench tops, a column or post, or a cylinder rack (if one is available in the laboratory).
- If possible, secure cylinders out of laboratory traffic areas.
- When moving a cylinder, always use a cart designed to carry cylinders (available from your gas supplier). If the cart has a safety chain or strap (it should!), use it. The best carts have four wheels so the cart – instead of you – takes the weight of the cylinder.
- Don't move a cylinder by "rolling" it on its base. It can easily get away from you.
- Keep the cylinder cap tightly in place until you're ready to connect the regulator.
- Never use a mechanic's open-end wrench, screwdriver, or bar to force open a stuck cylinder cap. You could inadvertently rotate the valve hand wheel and release the cylinder contents into the lab while trying to remove the cap. A heavy-duty strap wrench (like an automotive oil filter wrench but sturdier) is useful. If you can't get the cap off, ask your gas supplier to replace the cylinder.

# General Considerations

## Gases contribute to total analytical costs. How can I use them economically?

Reduce flows or stop them altogether when you're not analyzing samples. But there are a few things you should consider before you do so (or cool the heated zones in your GC):

- Although cross-linked stationary phases are quite resilient, they can be degraded by exposure to O<sub>2</sub>, and this degradation is enhanced at high temperature. You might set your GC oven at ambient temperature but the detector end of the column could be exposed to very high temperatures if the detector is hot.
- Be sure to retighten all injector and detector nuts if the inlet and/or detector has been cooled to room temperature. Temperature cycling causes Vespel® ferrules to "creep." Leak-check such fittings regularly.
- Back-pressure regulated split/splitless inlets for capillary columns vent most of the carrier gas that enters them. Forward-pressure regulated inlets can vent a great deal of carrier gas during splitless runs. Only a percent or two of the total carrier gas stream actually enters the column. Some GCs have a gas conservation feature that reduces the flow into the inlet when the instrument is not in use. Use this feature if it is available. Remember to keep the column and detector ovens cool while the carrier gas is only trickling through the column. Air diffusing from minor leaks into a low pressure inlet will

cause the O<sub>2</sub> concentration in the carrier gas to increase and can exacerbate the degradative oxidation of the stationary phase.

- You can turn off detector gases when they are not needed, but you will have to wait while the detector stabilizes and heats to operating temperature when you want to use it again.
- The cost of the gases is a minor part of the total cost of ownership of a GC. You have to decide if the small savings on gases is worth the cost of cold, unequilibrated instruments when you need them.

### Best practice

Always maintain carrier gas flow through the column while it is installed in the GC oven.

## Can I turn off gas flows without disturbing the flow rate settings?

You can stop the flow of gas to your GC with the shut-off valve installed at the outlet of the cylinder pressure regulator. The regulator setting will not be affected. However, this is not a long-term option. A regulator diaphragm that is pressurized without a relieving flow of gas is in a static condition for which it was not designed. It is better to mark the low pressure (outlet) gauge at the desired pressure setting with a laboratory marker and then back out the pressure adjusting knob of the regulator to stop the flow. This procedure will not affect the manual or electronic valve settings in the instrument.

# General Considerations

**Do I have to leak-check the fittings in the gas lines every time I break them?**

**What is the best method for leak-checking?**

Yes, always leak-check all gas line and column fittings you have broken and remade. This is a standard practice that should be followed without exception.

Soap-bubble solutions are an inexpensive means of checking fittings for leaks. However, commercial leak-testing solutions containing ammonia have been implicated in stress corrosion cracking of brass fittings that have been subjected to repeated leak-checking. We recommend that you learn which products contain ammonia and avoid using them on brass fittings in your gas piping systems. Stainless steel fittings are not affected by ammonia.

The best (but more expensive) means of leak-checking chromatography gas lines is a portable helium leak detector which is based on the thermal conductivity of helium. Minute helium leaks at fittings inside and outside the GC can be found rapidly. You can leak-test the air, N<sub>2</sub>, and H<sub>2</sub> lines by pressurizing them with helium.

## Best practice

Always leak-check fittings that have been made or broken, or cooled and re-heated to operating temperatures.

# Gas Selection

**I'm new to chromatography. What kinds of gases are available and what should I be looking for?**

The gases used in gas chromatography are:

1. The mobile phase or carrier gas that moves the sample from the inlet through the column to the detector;
2. The support gases for the detector.

The carrier gas is inert; all it does is carry the analyte zones or bands (you see them as peaks as they exit the column and are recorded) through the inlet and column to the detector.

The detector gases, on the other hand, might participate chemically in the detection process by burning, producing electrons, or initiating reactions on special substrates, or might simply sweep the analyte elution bands through the detector (make-up gas).

The purity requirements depend on the function of the gas, the sensitivity of the analysis, and on the specific detector.

For example,

- Oxygen in the carrier gas will shorten the life of your column by contributing to stationary phase degradation but will have no effect as a makeup gas impurity on the FID signal.
- Parts-per-billion levels of volatile halocarbons in the makeup or detector gas are perfectly invisible to a flame ionization detector (FID) but

dramatically affect the background signal of an electron capture detector (ECD).

- The opposite is true for trace levels of volatile hydrocarbons that affect an FID but not an ECD.
- On the other hand, 1 ppm of total hydrocarbon impurity in the carrier gas of a process GC-FID will be quite insignificant with respect to the amount of analyte on the column and the attenuation of the detector signal.

The general principle is that your GC gases should be free of the impurities that would interfere with your specific analysis or would degrade your chromatographic equipment.

You can select the appropriate gases by comparing the instrument vendor's suggested specifications with the product specifications described in specialty gas suppliers' catalogs or by referring to the gas selector tables in this booklet. Most gas suppliers have special grades of gases for analytical use but there is no consistency among suppliers in naming them or setting the specifications. You have to know what impurities at what levels will interfere with your analysis and select the gas accordingly. [Note: the gas specifications listed in the suppliers' catalogs are not analytical results. They represent the maximum levels permissible within the specific grade of gas. The actual analytical results may be significantly lower than the specifications. On the other hand, all but the 6.0 grades of gas are qualified by statistical quality control. Generally, 10% or fewer of the cylinders in a fill batch will be analyzed to verify gas quality.]

Packed and capillary columns may respond differently to the active impurities in the carrier gas. This difference in response depends on the extent of cross-linking, phase loading, age and condition of the column, and typical temperatures the column is exposed to.

Later in this booklet we develop the theme of gas purity. See that discussion.

## What carrier gas should I use?

That depends on your application, and, in some instances, on your detector. Are you using isothermal packed column GC as a process analyzer? Trace environmental analytical with temperature programmed capillary GC methods? Manufacturing QA analyses where throughput is essential and resolution can be sacrificed? Product R&D support?

### Packed columns

- Helium, nitrogen, and argon can be used as packed column carrier gases. Helium is easily removed by a jet separator in GC-MS. Nitrogen has the advantage of being less expensive than the two noble gases. Argon is useful in analyzing gases for hydrogen and helium content by TCD. Argon/methane mixtures have been used as the carrier gas for isothermal GC with electron capture detection.

### Capillary columns

- Helium is the most commonly used carrier gas because it is inert and non-flammable, and possesses physical properties that permit high resolution, temperature programmed gas chromatography.
- Although hydrogen is flammable, its high diffusivity allows faster linear velocities – and

consequent shorter analyses – with the same separation efficiency that helium provides. Shorter analyses translate into increased throughput, and that means lowered costs per sample.

Reactivity in some situations (catalytic hydrogenation of unsaturated molecules at high inlet temperature) could be a problem when using H<sub>2</sub> as the mobile phase. The potential for chemical reactions in your analytical system must be evaluated. There are also some issues in using hydrogen as the mobile phase in gas chromatography-mass spectrometry (GC-MS): the ion gauges monitoring the ion source pressure are usually calibrated with nitrogen and thus will not accurately report the pressure when H<sub>2</sub> is present. It may be difficult for the low capacity diffusion and turbomolecular pumps to efficiently remove H<sub>2</sub> from the ion source of bench top GC-MS instruments.

- Nitrogen should not be used in temperature programmed capillary GC.

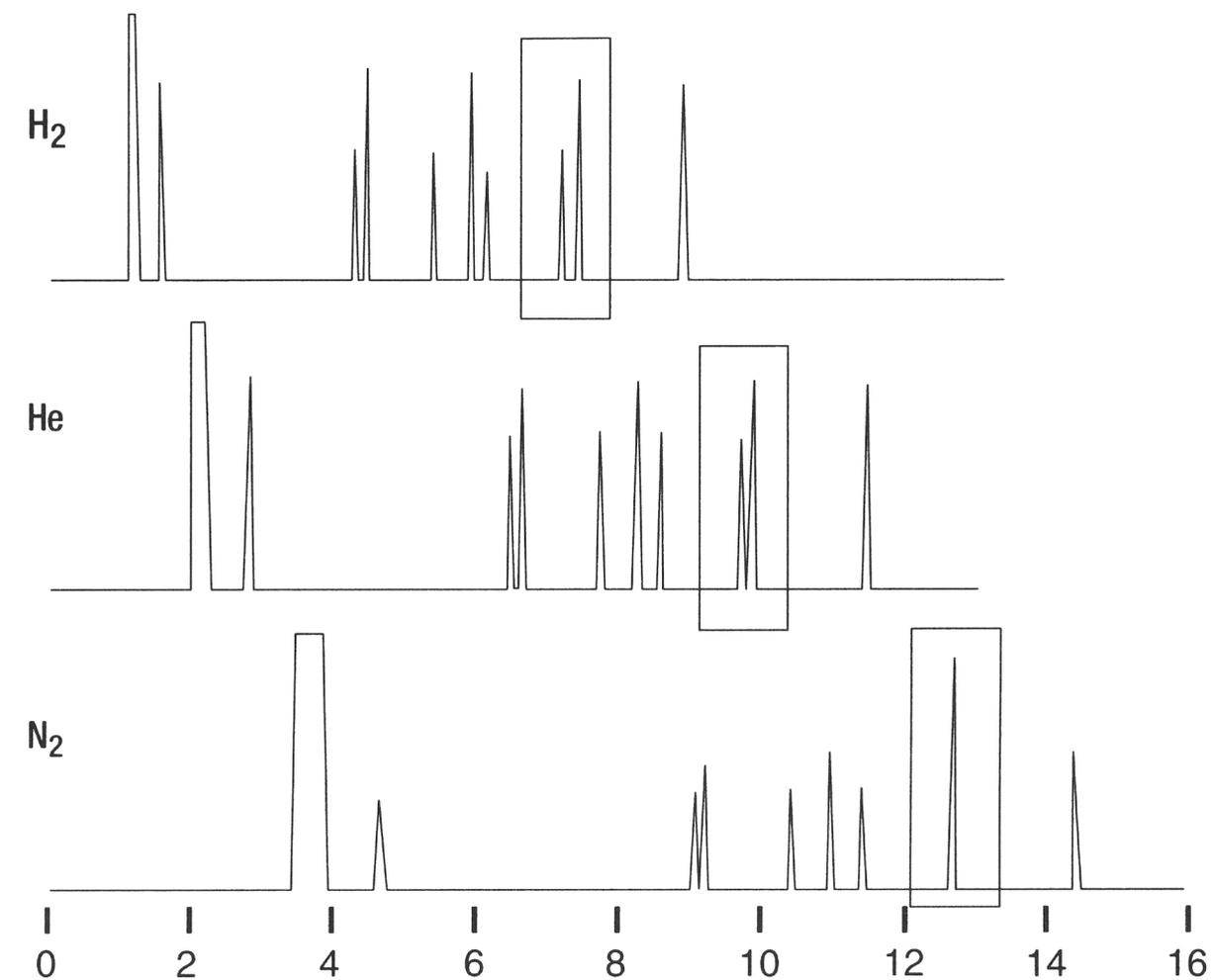
The chromatograms on page 11 illustrate the differences among hydrogen, helium, and nitrogen as carrier gases in temperature programmed capillary GC. The sample is a Supelco isothermal non-polar test mixture. Each carrier gas was set at its optimum linear velocity at the initial temperature of the temperature ramp. Notice the different retention times and resolution. The column and conditions were not changed.

More information on carrier gas selection is given on page 12. You can find it under the figure “Van Deemter plots for capillary columns.”

See your GC manual for instrument-specific information.

# Gas Selection

## Effect of carrier gas on resolution and analysis time

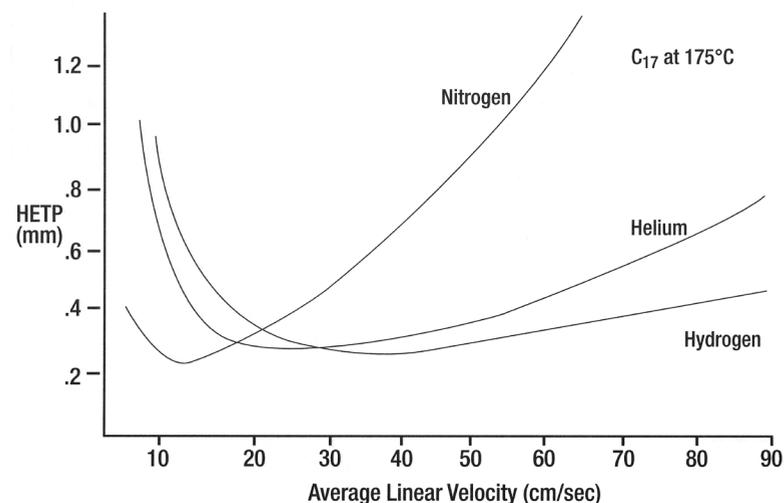


When used as a carrier gas, hydrogen typically provides higher resolution at higher linear velocities than either helium or nitrogen.

Note coelution of test mixture compounds with nitrogen carrier at  $t_R \sim 13$  minutes.

# Gas Selection

## Van Deemter plots for capillary columns



The plot above shows the relationship between “HETP” and the average linear velocity of the carrier gas under isothermal conditions. HETP (or “height equivalent of a theoretical plate”) is one measure of column efficiency. HETP depends on the nature of the carrier gas and its linear velocity (not volume flow rate). The smaller the HETP, the more efficient the separation. You can obtain the highest possible efficiency – best separating power – from your GC when you set the carrier gas linear velocity at the value where HETP is the lowest. For the system characterized above, the optimum occurs at about 12 cm/sec for nitrogen, about 20 cm/sec for helium, and about 36 cm/sec for hydrogen.

Nitrogen is quite different from helium and hydrogen. Although nitrogen’s minimum is lower than either of the other two gases, its

curve is dramatically steeper as the linear velocity increases. Nitrogen is the poorest of the three gases as a carrier gas for temperature-programmed chromatography because slight changes in the linear velocity during a run can lead to significant degradation of efficiency. The efficiency loss (coalescing of the two peaks) illustrated in the previous figure was caused by the decrease in carrier gas linear velocity during a temperature program where the carrier was not flow-controlled.

The minimum HETP with hydrogen, on the other hand, is insensitive to large changes in the linear velocity. The linear velocity can be set at any value between about 30 cm/sec and 60 cm/sec without losing separation efficiency during the run. On balance, hydrogen is the preferred carrier gas for capillary columns.

# Gas Selection – Hydrogen

## I’m worried about using hydrogen as a carrier gas. What is the danger of it exploding?

Yes, hydrogen is a flammable gas. If it is mixed with air in sufficient proportions (greater than 4% concentration in air) and exposed to a heat source, it will explosively ignite. But this is not a significant risk in modern GCs. These are the issues in capillary GC:

- The actual carrier flow rate is low in open tubular columns, and the column effluent is either burned in the detector or vented from it. Such low quantities of hydrogen exiting from a non-destructive GC detector pose no safety issues in a laboratory with typical air exchanges and/or exhaust system.
- The total flow in a back pressure-regulated inlet is divided between the column and either the splitter or the septum purge (depending on the status of the inlet purge valve). Regardless of actual route out of the inlet, the excess carrier gas continually exits through the split vent on the front of the GC. This outlet is the major source of hydrogen flowing out of the GC, and should be captured and vented to the laboratory exhaust system.
- A forward pressure-regulated inlet permits high carrier gas flow only during the time the splitter is open. Again, the effluent from the split vent should be captured and vented to the laboratory exhaust system.

- Under certain circumstances, hydrogen could possibly build up inside a GC oven and reach the 4% lower explosive limit (LEL). These are the conditions:
  - the carrier gas is not electronically pressure controlled,
  - the column breaks close to the injector body nut,
  - the GC oven is held at a high temperature for a long period of time to prevent the oven exhaust doors from opening,
  - sufficient time elapses after the column breaks to build up the H<sub>2</sub> concentration.
- Electronic pressure control of the carrier gas will power down the GC if it detects a leak. Be cautious with a powered-down GC that uses hydrogen as the carrier. Remove all possibilities of a spark or other ignition source by grounding the case before opening the door.
- In the highly unlikely event that an explosion occurs, the spring-loaded doors and exhaust flaps on most modern GC will relieve the pressure surge and protect the instrument.

The following warnings are taken from an Agilent GC manual:

## Using hydrogen

**Warning:** When using hydrogen (H<sub>2</sub>) as the carrier gas or fuel gas, be aware that hydrogen (H<sub>2</sub>) gas can flow into the oven and create an explosion hazard. Therefore, be sure that the supply is off until all connections are made, and ensure that the inlet and detector column fittings are either connected to a column or

# Gas Selection – Hydrogen Safety

capped at all times when hydrogen (H<sub>2</sub>) gas is supplied to the instrument.

**Warning:** Hydrogen (H<sub>2</sub>) is flammable. Leaks, when confined in an enclosed space, may create a fire or explosion hazard. In any application using hydrogen (H<sub>2</sub>), leak test all connections, lines, and valves before operating the instrument. Always turn off the hydrogen (H<sub>2</sub>) supply at its source before working on the instrument.

## Additional Safety Practices when using Hydrogen

The most important safety practice is to know and understand the properties, uses, and safety precautions for hydrogen before using the gas or associated equipment. Ask your gas supplier for copies of the Material Safety Data Sheets for hydrogen and for any related information. Then read the information.

When using hydrogen, these safety practices should be followed:

- Provide adequate ventilation in both work and storage areas.
- Plumb all instrument vents to an exhaust system.
- Be sure that electrical equipment in the laboratory conforms to national and local electrical codes.
- Post “No Smoking” and “No Open Flames” notices in storage and work areas.
- Keep the valve protection cap on the hydrogen

cylinder until the cylinder is put in use.

- Use a regulator recommended for high-pressure hydrogen control.
- Add properly sized safety relief valves and flow limiting devices to protect the GC and purifiers from over pressurization in the event of a regulator failure.

## Hydrogen Generators

If you prefer to eliminate hydrogen cylinders altogether, or if regulations make it inconvenient or impossible to use high-pressure hydrogen cylinders, consider using a hydrogen generator. A hydrogen generator is a safe, reliable, and low-pressure source that produces H<sub>2</sub> only on demand.

# Gas Purity

## What purity gas should I use?

**That depends primarily on the sensitivity of the analyses you perform.** (We deal with this subject further in the section on the “9’s”, and give you some directions in the tables on page 18.) You also have to consider the impact of gas impurities on your columns. Your column vendor can tell you the impurities and their concentrations in the carrier gas that are critical for specific stationary phases. The instrument vendor’s specifications are generic, offering no column-specific information, but they do point you in the right direction.

There are two approaches to ensuring the purity of your instrument gases:

1. Buy low-grade gases and purify them with point-of-use purifiers;
2. Buy high-grade gases and either dispense with the purifier trains or consider them as insurance against the potential rogue cylinder that could slip through the gas supplier’s statistical quality control defenses.

The optimum solution is a grade of gas that is guaranteed on a per cylinder basis to exceed the instrument vendor’s specifications for H<sub>2</sub>O, O<sub>2</sub>, and THC. The problem with per cylinder guarantees is that the analytical costs incurred by the gas supplier to ensure the gas quality of each cylinder are passed along to the customer. These are expensive gases. The BIP® helium, nitrogen, and argon products offered around the world by Air Products (Airgas, Inc., carries this product line in the USA), on the other hand, are guaranteed at <20

ppb H<sub>2</sub>O and <10 ppb O<sub>2</sub> on a per cylinder basis at a reasonable cost.

A purifier train is a complex solution that is also not inexpensive. Purifier trains require up-front capital costs and disciplined preventive maintenance labor to ensure they do not add exactly the same contaminants that they are designed to eliminate back to the gas streams. And ultimately, you are relying on the vendor’s guarantees that the purifiers will meet the specifications because you typically cannot test their performance.

There are advantages and disadvantages of material and manpower costs, consistency of gas quality, and peace-of-mind associated with each approach. You must decide which costs and risks you are willing to live with to obtain the gas purity your instruments and columns need.

Here are some technical issues to keep in mind.

- The lower the guaranteed maximum levels – the specifications – of H<sub>2</sub>O, O<sub>2</sub>, and THC in the gas, the longer your columns will last and the less background noise you will have to deal with (which means greater sensitivity and extended linear dynamic range of the detector).
- Selective detectors are also affected by ultra-trace levels of contaminants that are not normally certified in chromatography gases. For example, the ECD can be contaminated with halocarbons, both volatile solvents and semivolatile lubricant oligomers, in the carrier and detector gases. (See “Contamination of

Electron Capture Detectors with Volatile and Semivolatile Halocarbon Impurities in GC Gas Distribution Systems,” D. L. Vassilaros, LC-GC, Vol. 12, p.94, 1994.) Many specialty gas suppliers offer special certification and gas grades for selective detectors.

- Consider the sensitivity of your instrument and the degree of accuracy required in your analysis. If you are working at percent levels, the purity of your gases is not as important as if you were working at the ppm level. (Unless, of course, you use the same instrument for both purposes.) Consistency of purity, however, is always critically important.

Contact your gas supplier for more information. By specifying the proper grade of gas for your application, you can extend column life and reduce downtime while ensuring the baseline quality you need.

## How many 9’s are enough for my needs?

This is really asking the same question about gas purity needs as the previous question but is framed more quantitatively. Gas suppliers commonly specify the purity of their products in terms of 99.999% (5 nines), 99.9999% (6 nines), and so on. The purer the gas, the more 9’s. These numbers are calculated by subtracting the total impurity specifications (as if they were actual analytical results – which they are not) from 100%. This number is called the “total purity”.

Note: Impurity specifications are always written as “<” (less than). According to arithmetic rules for dealing with inequalities, subtracting a “<” from 100% results in a “>”; thus, the total 9’s should

be read as “>99.999%” purity. The 9’s specify the lowest possible guaranteed purity of the labeled product.

The 9’s are used to define different grades of gas. For example, the highest grade of gas is specified as being >99.9999% or 6 9’s (or 6.0), and a lower grade is defined at >99.9995% or 5 9’s 5 (5.5). In North America you will encounter gas grade names like “research,” “carrier,” “ultrahigh purity,” “high purity,” “zero,” and so on, and grade designations like 6.0, 5.5, 5.0, and so forth in Europe. Each of these grades has a different 9’s specification.

The problem with summarizing gas purity with 9’s is that there is no standard procedure for deriving them. It varies from product-to-product and from vendor-to-vendor. You must look at the specifications used to produce the total purity. We’ll go into more detail in the next section.

**Remember:** Your GC gases must be pure enough so that the impurities – and there are always some – don’t interfere with your analysis or damage your equipment. We recommend that you select your gas according to the key impurity levels, and not on the basis of total purity descriptors.

The specific impurities can selectively affect your detector. Suppose you have a cylinder of helium carrier gas containing mid-ppb levels of methylene chloride. This gas will be perfectly acceptable for a thermal conductivity or flame ionization detector, but an electron capture detector with its high selectivity and sensitivity for halocarbons will be rendered useless by the methylene chloride background at that concentration.

The same considerations hold for your columns. The resistance of your column’s stationary phase to oxidation depends on the type of phase. More polar columns are significantly less stable in the presence of oxygen and heat than are the methyl silicone phases.

So, we can’t really answer your question just by counting 9’s. We suggest that you consult a gas supplier who understands the question. All of the major vendors do.

Fortunately, the knowledgeable gas suppliers have prepared charts (using their terminology, of course) that can help you determine the appropriate gases for your GC applications. The following chart was developed by Air Products-Europe. The BIP<sup>®</sup> grades have exactly the same specifications everywhere they are offered. If you use a different supplier, this chart can still be a useful guide, but be aware that identical or similar names may refer to different purity levels.

# Gas Purity

## Recommended gas grade (Europe) for GC applications

Detector	Carrier and Detector Gases	Minimum Analyte Detection Level*			
		Trace (0-1 ppm)	1-1000 ppm	1000 ppm - 1%	1 - 100%
FID	He	BIP® Plus	BIP®	BIP®	BIP®
	N <sub>2</sub>	BIP® Plus	BIP®	BIP®	BIP®
	H <sub>2</sub>	BIP® Plus	BIP®	BIP®	BIP®
	Air	Zero Plus	Zero Plus	Zero	Zero
TCD	He	BIP® Plus	BIP®	BIP®	BIP®
	H <sub>2</sub>	BIP® Plus	BIP®	BIP®	BIP®
	N <sub>2</sub>	BIP® Plus	BIP®	BIP®	BIP®
	Ar	BIP® Plus	BIP®	BIP®	BIP®
ECD	He	BIP® ECD	BIP® ECD	BIP® ECD	N/A
	N <sub>2</sub>	BIP® ECD	BIP® ECD	BIP® ECD	N/A
	5 or 10% CH <sub>4</sub> in Ar	Experis® mixture	Experis® mixture	Experis® mixture	N/A
GC-MS	He	BIP® Plus	BIP®	BIP®	BIP®
	N <sub>2</sub>	BIP® Plus	BIP®	BIP®	BIP®
	H <sub>2</sub>	BIP® Plus	BIP®	BIP®	N/A
	Ar	BIP® Plus	BIP®	BIP®	BIP®
FPD	He	BIP® Plus	BIP®	BIP®	N/A
	N <sub>2</sub>	BIP® Plus	BIP®	BIP®	N/A
	H <sub>2</sub>	BIP® Plus	BIP®	BIP®	N/A
	Synthetic Air	Zero Plus	Zero Plus	Zero	N/A
PID	He	BIP® Plus	BIP®	BIP®	N/A
	N <sub>2</sub>	BIP® Plus	BIP®	BIP®	N/A
HID or DID	He	BIP® Plus	BIP® Plus	BIP®	N/A
	Purge He	BIP® Plus	BIP®	BIP®	N/A

\*Concentration in ppm of analyte in final sample.

For detailed specifications of the above gases, please refer to the Air Products European Special Gases Catalogue or UHP Data Sheet.

For optimum column performance, we recommend BIP®, BIP® Plus, and BIP® ECD gases which typically contain O<sub>2</sub> <0.01ppm and H<sub>2</sub>O <0.02ppm guaranteed *per cylinder* because of a built-in purifier.

## Revisiting the ‘Nines Mirage’

The fundamental problem with counting 9’s is that a single number just doesn’t tell you enough. You don’t know how that number was derived.

There are several ways to manipulate gas purity labels. One of the easiest is to constrain the number of analyzed (or reported) contaminants to arrive at a target purity. In the table below both gases are claimed to be >99.999% pure, yet Gas B is clearly the better product. The purity specification of Gas A was obtained by selective omission.

## Purity of Gas A and Gas B

Contaminant specifications in molar ppm

	Gas A	Gas B
O <sub>2</sub>	<2	<2
Argon	NA	<4
CO <sub>2</sub>	NA	<2
Total hydrocarbons	<5	<1
Water	<3	<1
Total analyzed impurities	<10	<10
<b>Total purity</b>	<b>&gt;99.999%</b>	<b>&gt;99.999%</b>

NA = Not Analyzed

# Gas Purity

## I use purifiers in my gas lines so I don't have to worry about gas purity, do I?

Maybe, but you could be working under a false sense of security. You don't have to worry about gas purity provided that:

- you are not trying to purify a low purity grade of gas into research grade, and
- your purifiers are sized, specified, and installed correctly, and
- you have indicating purifiers to tell you the status of your purifiers, and
- you discipline yourself to a regular preventive maintenance program on your purifier train.

Otherwise, you have something to think about because you don't really know the condition of your purifier train. If the purifiers are not regularly maintained, they will eventually saturate with the material they were designed to remove, especially if you are using a low grade of gas. Then they become ineffective or a source of contamination when breakthrough begins to occur. This is one of the risks of purifying industrial gases for ultra-high purity applications.

We prefer to think of the purifier train as an insurance policy to protect your instruments from unexpected contaminants or inconsistent contaminant levels in your gases. Like any insurance policy, a premium (preventive maintenance) is due at a regular interval to assure continuing coverage.

- Purifiers cannot be ignored once they are in place – they need maintenance. Your GC manufacturer, columns supplier, and gas supplier have information available about purifier maintenance.
- We suggest a log sheet in the laboratory that records when they were installed and when maintenance (regeneration or replacement) is due.
- A list of part numbers, vendors, and prices should be attached to the log sheet to make it easy to order replacements.

We described some of the issues with purifier trains several pages ago in response to the larger question, "What purity gas should I use?" As we said earlier, you have to decide which approach you want to take to ensure high purity gases reach your instruments. The fact is, unless you use gases that have been qualified *per cylinder* or correctly specified and fully maintained purifiers, or do your own gas qualification analyses, you cannot be confident in the purity of your GC gases from cylinder-to-cylinder.

# Gas Distribution

## What materials should I use for my gas distribution system?

This is easily answered. Use clean metal components throughout.

How is this done? Just follow a few simple rules:

- Be certain that all components are clean before assembling them. It is almost impossible to clean contaminated components after they are assembled.
- Use only regulators with stainless steel diaphragms that are specified for use with high-purity gases. Many manufacturers have both an "industrial" and "analytical grade" line of regulators. You want the top grade. Industrial regulators are not compatible with analytical gases.
- Use copper or stainless steel tubing for all distribution lines. Stainless steel is good for fixed manifolds. Copper is appropriate for the lines to the instruments.
- Make all connections metal-to-metal. SWAGELOCK® or other high quality compression fittings are a good choice. Do not use o-rings, gaskets, or lubricants.
- If you must use pipe thread connections, seal them with virgin-grade Teflon® tape. Never use oil-based sealants that can contaminate your system beyond recovery.

## Gas Distribution – Tubing

I can get refrigeration tubing at the hardware store more cheaply than I can buy tubing from a GC company. Can I use it?

No, definitely not. Or at least, not as you receive it from the hardware store.

Copper tubing is formed by a series of dies. The process must be lubricated, and waxes and oils are used for the job. The tubing is not cleaned to chromatographic standards before sale.

The tubing you purchase from a GC manufacturer or supply house has been cleaned for chromatography applications. It's ready for immediate use.

Can I clean the tubing myself?

Certainly, but it's a messy and awkward job.

One approach is to rinse the tubing with methylene chloride and then dry it under a stream of carrier gas. Another is to use methylene chloride followed with an acetone rinse, and then dry it with carrier gas.

Now for the problems.

- This operation should be carried out in a fume hood, and the size of the tubing could make this awkward.
- Don't use Tygon or similar plastic tubing to connect your solvent reservoir to the metal

tubing. The solvent will extract plasticizer from the plastic and deposit it inside the metal tubing, or dissolve the plastic tubing itself and contaminate the metal tubing.

- Do not use methylene chloride or any other halogenated solvent to clean tubing intended for ECD applications. It is difficult to remove the detectable residues from the metal surface.
- Dispose of the used solvent safely.

It would be very convenient to use colored plastic tubing for my carrier and detector gases. Is that OK?

Please don't; you're just asking for problems. This is because plastics are permeable to gases. Oxygen, water, and other vapors in the laboratory atmosphere are absorbed by the plastic and migrate into the gas stream inside. The advantage of the color-coding is lost by the increased purifier maintenance.

Similarly, oil, grease, and practically anything else that contacts the plastic will work its way through into the gas stream. The result is a noisy baseline that interferes with high-sensitivity analyses, ghost peaks that aren't related to the sample composition, column stationary phase degradation, and other avoidable problems.

If you want tubing flexibility so you can turn a GC to get at the back, coil an extra meter or two of tubing behind the instrument.

## Gas Distribution – Pressure Regulators

I've got some old regulators in a box. Can or should I use them?

Probably not, if this is all you know about them. Regulators can pick up contamination just as columns and tubing can. When you put them into service, they begin to contaminate the gas that flows through them.

Consider these questions:

- What is the rated outlet pressure of the regulator?
- Does the regulator have a stainless steel diaphragm?
- Do you know for sure what gas service it was in before being put in the box?
- Did it come from a gas distribution system that had problems?
- Is it rated for analytical service and, if so, has it been properly maintained?

If your answer to any of these is "I don't know", then we recommend that you buy a new regulator. You will spend money up front and it may be inconvenient, but at least you know what you're getting into, and you have not created problems for yourself.

However, if the regulator has been capped – both the cylinder connection and the outlet fitting – to prevent air ingress, and if it was suitable for chromatographic use in the first place, and if it was tagged with all relevant information, and if it was wrapped in a lint-free cloth for storage, then you can feel confident that the regulator will perform at the required level.

What specifications should I look for on regulators?

There are only a few, but they're all important.

- Select single-stage or two-stage regulation. Single-stage regulators are appropriate where precise pressure control is not needed, or where this regulator follows or will be followed by another stage of regulation. A single-stage regulator will permit the outlet flow rate to increase as the cylinder pressure decreases. Two-stage regulators are essentially two regulators in the same body. The first stage reduces the high cylinder pressure to an intermediate level and the second stage further reduces this to the required outlet pressure. A two-stage regulator prevents the outlet flow rate drifting as a function of the cylinder pressure.
- Use the correct regulator for the required gas service. Avoid adapted regulators because the internal materials of regulators may vary with the gas they are intended for.
- The GC manufacturer will tell you what pressure is needed. Select a regulator such that you will be operating just above the center of the scale on the outlet gauge. This makes it easier to set pressures and allows you to use higher-than-normal pressure when you need it.
- Be sure that the regulator has a stainless steel diaphragm.

Diaphragms of Neoprene or other elastomers outgas volatile organic and inorganic materials than can contaminate the gas stream.

# Gas Distribution – Pressure Regulators

Additionally, exposure of an elastomeric diaphragm to air leaking into the regulator can lead to increased levels of O<sub>2</sub> in the gas stream. Stainless steel diaphragms avoid this problem. It is worth the small extra cost to preserve the purity of the gas you purchased.

- Verify that the regulator is rated for high-purity gas work.

**Important:** Do not use adapters on regulators.

## Best practice

A tee purge (block-and-bleed) valve assembly is the ideal interface between your regulator and the cylinder. Its isolation valve protects the regulator and all downstream plumbing from air contamination when changing cylinders. And the vent valve permits sequential cycle purging of the cylinder valve outlet and tee purge assembly volume to remove atmospheric contaminants prior to opening the isolation valve. See below for details.

## How do I connect a regulator?

1. **Identify the regulator.** Examine the label and the inlet and outlet pressure gauges. Verify that the gauge is intended for the gas to be used and that the input gauge range is adequate for the pressure in the cylinder.
2. **Inspect the regulator.** Examine it for evidence of damage or contamination. If there is any evidence of physical damage (broken gauge glass, dents, damaged threads, and so on) or of foreign material inside the regulator, do not use it.

3. **Inspect the cylinder valve outlet.** Look for evidence of damage to the outlet threads or bull-nosed sealing surface, or of contamination. Remove any foreign material (spider webs, dust, water, etc.) before connecting the regulator.
4. **Do not crack the cylinder valve open to purge the outlet.** This process also aspirates air back into the cylinder. Best practice is to invest in a tee purge (block-and-bleed) valve assembly. See page 24 below.
5. **Connect the regulator to the cylinder.** Be careful to avoid cross-threading the nut which will damage the fitting. Tighten the nut securely. Never use Teflon® tape on this fitting.
6. **Close the regulator.** To close the regulator, turn the adjusting knob to the full counterclockwise position. The regulator must be closed before opening the cylinder valve to avoid the chance of damaging the diaphragm and allowing high cylinder pressure downstream of the regulator.

**Important:** Always open the gas cylinder valve slowly to avoid pressure shocks to the regulator.

## Best practice

See page 27 for instructions on cycle-purging the tee purge assembly after installing the regulator in a new cylinder.

## What delivery pressure should I set on my regulator?

The GC manual may give you values or guidelines for selecting a pressure. Follow these recommendations. Typical regulator pressure for GC gases ranges from 40 to 60 psig (3 to 5 barg).

If the GC manual gives flow (rather than pressure) recommendations, you will have to experiment a bit. You need a pressure that gives you the flows you need, plus about 10 psi (0.7 bar) to allow for occasional extra draw. Because column flow resistance varies with temperature, check your pressure settings at the oven temperatures you will use.

Avoid using unnecessarily high pressures – you might blow out gauges or fittings that are not rated for high pressure.

## What about pressure relief devices?

Some regulators are equipped with safety relief valves designed to protect the regulator in the event of overpressurization of the regulator. These devices will not necessarily protect your GC and gas distribution components downstream of the regulator. Consult your gas supplier for details on external relief valve sizing, captured ports, and flow limiting devices. Over-pressure protection is absolutely required on all Air Products high-pressure gas handling systems in the plants and in the laboratories.

# Gas Distribution – Troubleshooting

My baseline just rose off the scale – could it be contaminated gas?

How do I find out?

What do I do about it?

Yes, the gas could be the source of contamination. But volatile and semi-volatile impurities in GC systems have several possible sources. We can't give you a thorough troubleshooting primer here but we will mention a few ideas.

Consider the timing of the problem. Did the baseline rise begin after a new cylinder of carrier or detector gas was installed? Or did it begin after the installation of a new regulator, valve, or tubing? In either case, there is a strong probability that the baseline problem is associated with the change in the distribution system. Regulators and valves can be lubricated.

If the problem is not associated with a system change, it could be that a purifier has become saturated and is now letting contaminants pass through. When this is the cause, the problem generally comes on gradually.

To isolate and correct the problem:

1. If there are any purifiers, filters, or oxygen scrubbers in the system, bypass them with a length of clean metal tubing.
2. If a cylinder or distribution system component was changed, replace the changed component or cylinder.
3. Open the inlet splitter as far as possible to permit a 100-200 cc/min flow through the carrier gas pneumatics. (Open the detector gas valves or line regulators to obtain 100-200 cc/min flow through the detector.) Purge the lines for 5 to 10 minutes, reset your system to its standard operating conditions, and monitor the baseline. If the contamination was a volatile material, and if this procedure eliminated it, the baseline will drop rapidly.
4. To test the purifiers, reinstall them in the line already proven to be clean one at a time. Watch the baseline patiently after each installation to find the bad one.

We suggest using a BIP® cylinder to assist troubleshooting baseline problems. The built-in purifier guarantees the gas won't contribute to the problem and permits you to find its cause faster.

If you want to continue using purifiers, regenerate them or install new ones after you complete the testing.

# Tee Purge (Block and Bleed) Valve Assembly

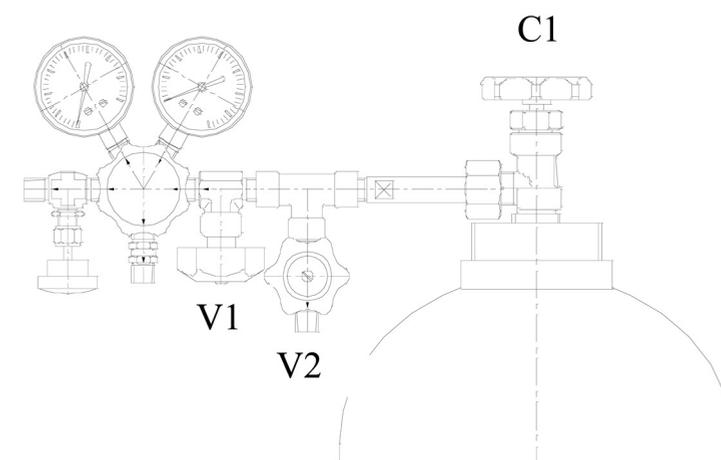
What is a tee purge valve assembly?

Look at the picture below. The tee purge assembly is installed on the upstream side of your regulator between the regulator body and the compressed gas fitting. It consists of two valves, a vent valve and an isolation valve.

Its purpose is to prevent air from entering your gas distribution system and GC after changing a compressed gas cylinder. You can seal off the downstream plumbing with the isolation valve, and cycle purge the assembly after installing the cylinder to eliminate the air that entered.

Procedures for cycle purging a tee purge assembly after changing a cylinder:

1. Close C1 (cylinder valve)
2. Close V1 (isolation valve)
3. Open and then close V2 (vent valve) to depressurize
4. Exchange cylinders
5. Open and then close C1 to pressurize the assembly
6. Open and then close V2 rapidly
7. Repeat steps 5 and 6 up to ten times
8. Open C1 slowly and then V1 slowly.



# Gas Cylinders

## What is the difference between tank, cylinder, and bottle?

'Bottle' often means the small "lecture bottle" size; however, it can refer to a typical laboratory gas cylinder. 'Tank' and 'cylinder' are often interchangeably used in the U.S. although a 'tank' can also refer to a larger, non-portable storage vessel. On the other hand, 'tank' in Europe is not used to refer to a 'cylinder' of compressed gas in the laboratory; it is reserved to describe larger storage vessels.

## When should I change a cylinder?

Before it is empty.

But doesn't this decrease the usable amount of gas I paid for?

Yes, it does, but here are two reasons for not exhausting a cylinder:

- By replacing the cylinder at about 150 psig (10 barg), you reduce the risk of running out of carrier or detector gas in the middle of an important analysis.
- You also protect your purifier train and instrument from contamination. Contaminants which are adsorbed or condensed on the cylinder walls such as moisture and oils desorb as the cylinder pressure decreases, thus increasing their concentration in the gas.

**Note:** BIP® gases produce consistent quality gas from full pressure to the reseating pressure – about 45 psig (3 barg) – of the residual pressure valve built into the cylinder valve.)

## Changing cylinders is difficult and time-consuming. How can I minimize this disruption?

It shouldn't be that serious a disruption if you're using separate cylinders for each GC. Just keep an eye on the cylinder pressure gauge and make the change when the pressure reaches about 150 psig (10 barg) and you are between runs.

If a single cylinder is supplying several GCs, changes will be more frequent and you have to halt the instruments to change an empty cylinder. There is a better approach. Most gas vendors offer an automatic changeover regulator or manifold. This connects two or more gas cylinders, an active cylinder or set of cylinders from which gas is being withdrawn, and reserve cylinders. When the pressure in the active cylinder falls below some preset level, the regulator automatically switches to the reserve cylinder. You can change the empty cylinder at your convenience without interrupting the analyses.

The automatic changeover system must have purge valves that permit you to purge the air from the valves and flexible lines after installing a new cylinder.

You might also choose to use gas generators, thus eliminating some cylinders entirely.

## I read something about an eductor or dip tube. What is it?

An eductor or dip tube extends from the cylinder valve to the bottom of the cylinder. It allows withdrawal of compressed, liquefied gases as the liquid. There are two types of eductor tubes, full-length and gooseneck tubes.

**Full-length eductor tubes.** Full-length tubes are used for liquid withdrawal only. Carbon dioxide cylinders supplying liquid CO<sub>2</sub> for cryogenic cooling of GC ovens or sample focusing units are equipped with these tubes.

**Gooseneck eductor tubes.** Gooseneck tubes can withdraw either gas or liquid. With the cylinder in the normal upright position, gas is withdrawn. In the horizontal position with the valve outlet facing up, liquid is withdrawn.

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