

# Choosing the proper gas and gas equipment for the laboratory, Part 1: Relationship between gas purity and detection limits in gas chromatography

Michel Gastiger and Benjamin Jurcik

**M**any analytical techniques require the use of specialty gases (e.g., He, H<sub>2</sub>, N<sub>2</sub>, Ar, O<sub>2</sub>, etc.) and/or their mixtures. The purpose of gases for an analytical procedure depends on the technique, and it is a crucial part of the procedure. The quality of the gas used therefore significantly impacts the potential results. The laboratory manager must understand the effects of gas purity on the quality of the results obtained. Most industrial gas companies offer a range of gas purity levels. Frequently, the effect of the gas quality on the obtainable results is not well understood. An order may be placed without reference to the consequences of the purity on the analytical results.

This paper describes the relationship between the gas purity and the obtainable detection limit for the detectors commonly used in gas chromatography. The required gases and the effect of various impurities are first described, followed by a method of estimating the relationship between carrier gas purity and the obtainable detection limit.

## Specifications

Gas chromatography is an analytical technique that normally uses a large quantity of pure gases. The nature of these gases is shown in *Table 1* for the main detectors in use today.<sup>1</sup>

In gas chromatography, it is well known that water, oxygen, and hydrocarbons are the major critical impurities for chromatographic columns or detector response.<sup>1</sup> *Table 2* shows some disturbances produced by these classic pollutants.

While the consequences of a reduced column lifetime are obvious, the baseline disturbances directly affect the accuracy of the results that are obtainable with the analyzer. In order for analysts to choose the appropriate gas quality in coherence with their analytical needs, a complete product range for analytical chemistry and gas chromatography applications is offered (**Air Liquide Corp.**, Paris, Cedex, France). For each of the gases, a wide range of containers includes cylinders and benchtop generators for on-site production in the case of hydrogen, air, and nitrogen. The simplicity of the range allows the analyst to easily select the appropriate gas purity for the analytical applications (*Table 3*).

*The laboratory manager must understand the effects of gas purity on the quality of the results obtained.*

A general guideline for selecting the correct carrier, fuel, or make-up gases is to use the correct gas purity that does not interfere with analyses or damage equipment. In other words, if one is working in percent levels, there may be no need for the same gas purity used for ppm or subppm analysis.

Table 1

Gases used with commonly used detectors			
Detector*	Carrier gas	Fuel gas	Make-up gas
FID	He, H <sub>2</sub> , N <sub>2</sub>	Air + H <sub>2</sub>	N <sub>2</sub> , He, H <sub>2</sub>
TCD	He, H <sub>2</sub>	None	Same as carrier gas
ECD	N <sub>2</sub> , He, Ar/5% CH <sub>4</sub>	None	N <sub>2</sub> , Ar/5% CH <sub>4</sub>
FPD	N <sub>2</sub> , He	Air + H <sub>2</sub>	Same as carrier gas
HID	He	Air + H <sub>2</sub>	He
NPD	He, H <sub>2</sub> , N <sub>2</sub>	Air + H <sub>2</sub>	He
PID	He, H <sub>2</sub> , N <sub>2</sub>	None	N <sub>2</sub> , He
ELCD	He, H <sub>2</sub>	H <sub>2</sub>	None

\* FID: flame ionization detector, TCD: thermal conductivity (or hotwire) detector, ECD: electron capture detector, FPD: flame photometry detector, HID: helium ionization detector, NPD: nitrogen phosphorus detector, PID: photoionization detector, ELCD: electron conductivity detector.

Table 2

Disturbances caused by classic pollutants	
O <sub>2</sub> or air (in inert gas)	→ Reduce column life
H <sub>2</sub> O	
CO <sub>2</sub>	
CO	→ Baseline disturbances
Hydrocarbons	

Table 3

Gases	Alphagaz 1 and Alphagaz 2 pure gases*	
	Alphagaz 1	Alphagaz 2
Ar, H <sub>2</sub> , He, and N <sub>2</sub>	H <sub>2</sub> O < 3 ppm	H <sub>2</sub> O < 0.5 ppm
	O <sub>2</sub> < 2 ppm	O <sub>2</sub> < 0.1 ppm
	C <sub>n</sub> H <sub>m</sub> < 0.5 ppm	C <sub>n</sub> H <sub>m</sub> < 0.1 ppm
		CO < 0.1 ppm
Air	H <sub>2</sub> O < 3 ppm	CO <sub>2</sub> < 0.1 ppm
	C <sub>n</sub> H <sub>m</sub> < 0.5 ppm	N <sub>2</sub> < 0.1 ppm in Ar and He H <sub>2</sub> < 0.1 ppm in Ar, He, and N <sub>2</sub>

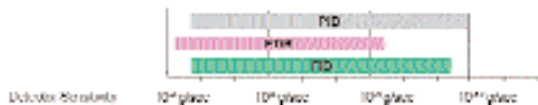
\*Air Liquide Corp.

While the above concepts may appear to be self-evident, the question of how to make an appropriate selection without a series of tests is not evident. Thus, a simple calculation method is given that can be used to make the estimation, and charts are provided for the different detectors.

## Calculation method

### Relationship between gas purity and detector

When a carrier gas containing an impurity arrives at the detector, it produces a signal proportional to the flow rate and to the concentration of this impurity. Also, a GC detector could be represented following its lowest detection limit and its domain of linearity (e.g., FID sensitivity limit: about 10<sup>-12</sup> g of C/sec; domain of linearity: about 10<sup>7</sup>) or its field of use (FTIR) (see *Figure 1*).



**Figure 1** Domain of linearity or field of use for various GC detectors.

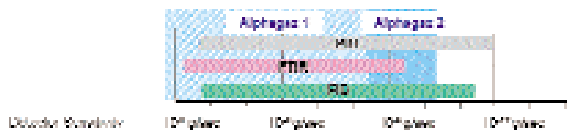
Then, considering that Alphagaz 1 is specified to less than 3 ppm of H<sub>2</sub>O, 2 ppm of O<sub>2</sub>, and 0.5 ppm of C<sub>n</sub>H<sub>m</sub> as critical impurities, and that the mean carrier gas flow rate into the GC is between about 1 and 60 cm<sup>3</sup>/min (packed or capillary columns, with or without makeup), the possible range for the quantity of water arriving to the detector per sec can be calculated to be about 10<sup>-9</sup> g/sec. The same reasoning can provide an estimation for hydrocarbons and O<sub>2</sub> impurities challenging the detector.

Alphagaz 1 is therefore the appropriate carrier gas for analyzing samples when more than 10<sup>-9</sup> g of compound/sec reaches the GC detector. The same calculation for Alphagaz 2 shows that this grade is adapted for less than 10<sup>-10</sup> g of compound/sec (see Figure 2). Following this method, the addition of other detectors is easily done.

### More appropriate scale

While Figure 2 is useful, the units are not immediately evident, since a chemist is more interested in the relationship between the concentration of the analysis and the requisite quality of gas. This problem can be solved by answering the following question: What is the initial sample concentration corresponding to 10<sup>-x</sup> g/sec arriving on the GC detector?

For liquid sample injection (about 1 µL or 1 µg), consider, for instance, that 10<sup>-9</sup> g/sec will be contained in this 1 µg. Also, it is easy to calculate that the sample concentration is near 1 mg/L.



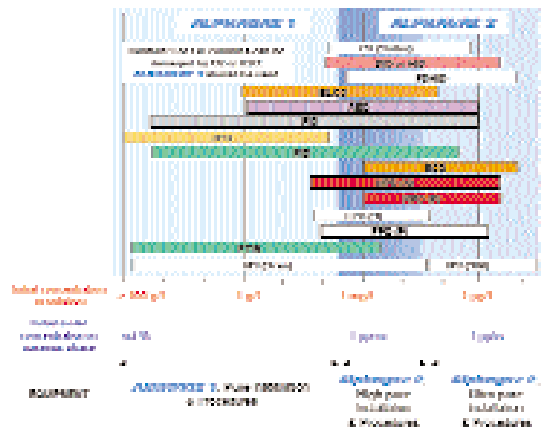
**Figure 2** Relationship between gas purity and domain of linearity or field of use for some GC detectors.

In the case of gas analysis, the injected volume is about 100 µL or 4.5 10<sup>-6</sup> mol of gas. Also, assuming a mean molecular weight of 80 g, 10<sup>-9</sup> g/sec or 1.2 10<sup>-11</sup> mol/sec is present in this volume and the initial concentration will be about 1 ppm.

Using these assumptions, it is easy to transform the initial scale to a working scale. Nevertheless, many hypotheses have been made, and the results must be taken as indications, not as absolute values.

## Conclusions

Figure 3 summarizes the calculations for the frequently used detectors in gas chromatography. It is essential to consider the stability of the column against oxygen and/or water. Some columns (containing cyanogroups, for example) may be damaged by these impurities, irrespective of the sample concentration. In this case, Alphagaz 2 should be selected as the carrier gas.



**Figure 3** Relationship between gas purity, frequently used GC detectors, and level of analysis. FID: flame ionization detector, TCD: thermal conductivity (or hotwire) detector, ECD: electron capture detector, CD: chemiluminescence detector, DID: discharge ionization detector, PDHID: pulsed discharge helium ionization detector, ELCD: electrochemical detector, AED: atomic emission detector, PID: photoionization detector, HID: helium ionization detector, NPD: nitrogen-phosphorus detector, FPD: flame photometric detector, FTIR: infrared detection detector, MSD: mass spectroscopy detector. Scan: full scan operation, SIM: selective ion monitoring.

In the case of analysis of compounds at concentrations below 100 ppb in gaseous phase or 10 µg/L in solution, Alphagaz 2 is recommended, along with appropriate installations and procedures. For example, a cross-purge assembly will be installed between the cylinder and pressure regulator to prevent contamination of the system during cylinder changes. The valves and pressure regulator parts will be all stainless steel, and their leak integrity will be less than 10<sup>-9</sup> atm.cm<sup>3</sup>.sec<sup>-1</sup>. Electropolished tubes will be connected with appropriate fittings or welding. In point of use, gas purifiers will adsorb the last traces of water, oxygen, and hydrocarbons that could interfere with analyses at low concentrations.

## Reference

1. Bulletin 898 A. Bellefonte, PA: Supelco, 1997.

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# Choosing the proper gas and gas equipment in the laboratory, Part 2: Relationship between gas purity and laboratory analyzers

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**G**as purity requirements are directly related to analyzer requirements and the field of use or the working domain. For example, Part 1 of this paper<sup>1</sup> illustrated that high-purity gases such as ALPHAGAZ 2 (AIR LIQUIDE Corp., Paris, France) and dedicated equipment must be used and implemented to analyze low-level concentration samples (e.g., <0.1 ppm) no matter which GC detectors are used. While it is evident that extremely high-purity gases can be used for measurements requiring a higher degree of precision, the reverse is not true. As a result, the authors' selection criteria for gas and material is to use the grades of gas and material necessary to maintain the good working condition of the analyzer and to provide the needed analytical precision.

This paper applies the same logic for the other most common analyzers in the laboratory. For each, the concentration domain to be analyzed will be indicated and the grade of gas recommended will be given. In regard to the grade of gas, dedicated equipment will be chosen.

Analyzers requiring gases have been segmented into the following areas: 1) emission spectroscopy, 2) absorption spectroscopy, 3) mass spectroscopy, 4) thermal analysis, 5) liquid chromatography or supercritical fluid chromatography, 6) surface analysis, 7) gas analysis, and 8) other analysis. Since gas chromatography is the largest analytical technique that uses gas, the relationship between gas purity and chromatography detectors has been described in detail.<sup>1</sup>

For each of the analytical techniques mentioned above, a grade of gas is recommended in relationship to the level of concentration of the sample.

A word of caution is needed: The levels of concentration (in mol/mol or wt/wt) of the sample to be analyzed are indications, not absolute values; there are always specific conditions that may arise requiring the use of a higher gas purity and that cannot be foreseen.

## Pure gases

In order to simplify the choice for the analyst, only two grades for helium, nitrogen, argon, hydrogen, oxygen, acetylene, nitrous oxide, and carbon dioxide (ALPHAGAZ 1 and ALPHAGAZ 2, AIR LIQUIDE Corp.) are proposed. The specifications of both pure gases are given in Table 1. The purity of these gases may be as high as 99.9995% (N<sub>2</sub>) or 99.99999% (H<sub>2</sub>). The large range of flow rate and pressure permits the gas to be fed to one or more analyzers. The typical specifications of

Table 1

Gases	Specifications of ALPHAGAZ 1 and ALPHAGAZ 2	
	ALPHAGAZ 1	ALPHAGAZ 2
Ar, H <sub>2</sub> , He, N <sub>2</sub>	H <sub>2</sub> O < 3 ppm O <sub>2</sub> < 2 ppm C <sub>n</sub> H <sub>m</sub> < 0.5 ppm	H <sub>2</sub> O < 0.5 ppm O <sub>2</sub> , C <sub>n</sub> H <sub>m</sub> , CO, CO <sub>2</sub> < 0.1 ppm N <sub>2</sub> < 0.1 ppm in Ar, He H <sub>2</sub> < 0.1 ppm in Ar, He, N <sub>2</sub>
Air	H <sub>2</sub> O < 3 ppm C <sub>n</sub> H <sub>m</sub> < 0.5 ppm	— H <sub>2</sub> O < 0.5 ppm
O <sub>2</sub>	H <sub>2</sub> O < 3 ppm C <sub>n</sub> H <sub>m</sub> < 0.5 ppm	H <sub>2</sub> , C <sub>n</sub> H <sub>m</sub> , CO, CO <sub>2</sub> < 0.1 ppm N <sub>2</sub> < 4 ppm NO <sub>x</sub> < 15 ppb (typical analysis) H <sub>2</sub> O < 3 ppm
CO <sub>2</sub>	H <sub>2</sub> O < 20 ppm	C <sub>n</sub> H <sub>m</sub> < 2 ppm O <sub>2</sub> < 2 ppm N <sub>2</sub> < 8 ppm
C <sub>2</sub> H <sub>2</sub>	N <sub>2</sub> < 0.4%	—
N <sub>2</sub> O	N <sub>2</sub> < 0.4%	—

Table 2

Gas	Purity	Specifications of ALPHAGAZ FLO generators		
		Maximum output flow rate (L/min)	Maximum output pressure (bar)	Typical analysis
Nitrogen	>99.99%	1–11	Up to 8.6	CO, CO <sub>2</sub> , O <sub>2</sub> < 1 ppm H <sub>2</sub> O < 2 ppm Hydrocarbons < 0.1 ppm
Hydrogen	>99.99999%	0.15–0.55	Up to 4	Not applicable
Air	Not applicable	1–30	Up to 8	Hydrocarbon (as CH <sub>4</sub> ) < 0.1 ppm

Table 3

ALPHAGAZ MIX	Range of ALPHAGAZ MIX	
	Concentrations	Uses
Methane–argon	5 or 10% CH <sub>4</sub> in argon	Electron capture detector, X-ray fluorescence, nuclear counter
Hydrogen–helium	40% H <sub>2</sub> in helium	Flame ionization detector
Hydrogen–argon	0.75–7% H <sub>2</sub> in argon	Spark emission

ALPHAGAZ FLO generators (AIR LIQUIDE Corp.) are given in Table 2. Analyzers that require special mixtures for working (e.g., X-ray fluorescence) are fed with ALPHAGAZ MIX grade (AIR LIQUIDE Corp.) (Table 3).

These specifications do not cover all the possible cases of critical impurities for a given analyzer or analysis; they are designed to ensure the quality of gas necessary for laboratory applications.

### Recommendations

The analyzers presented in this paper are primarily dedicated to the analysis of liquid or solid samples. The impurities that can be contained in gases are only present in gaseous phase and as a result do not interfere with the majority of analyses that will be done. For this reason, use of ALPHAGAZ 1 or ALPHAGAZ FLO is recommended for most analyzers.

Table 4

		Emission spectroscopy Detection level (mol/mol or wt/wt)			
Techniques	Gas %	<1000 ppm	<100 ppm	<10 ppm	<1 ppm
<b>Inductively coupled plasma (ICP)</b>					
Auxiliary plasma flow	Ar	ALPHAGAZ 1			
Plasma flow	Ar	ALPHAGAZ 1			
<b>Optical detector (ICP-OES)*</b>					
Purge of optical part	N <sub>2</sub>	ALPHAGAZ 1			
Purge of optical part	N <sub>2</sub>	ALPHAGAZ FLO			
<b>Mass spectrometer (ICP-MS)</b>					
Auxiliary plasma flow	Ar	N/A	ALPHAGAZ 1		
Plasma flow	Ar	N/A	ALPHAGAZ 1		
<b>Other</b>					
Organic solvent analysis	O <sub>2</sub>	ALPHAGAZ 1			
	H <sub>2</sub>	ALPHAGAZ 1			
<b>Spark emission</b>					
Plasma	Ar	ALPHAGAZ 1**	N/A		
Plasma	Ar + 1-7% H <sub>2</sub>	ALPHAGAZ MIX	N/A		
Purge of optical part	N <sub>2</sub>	ALPHAGAZ 1	N/A		
Purge of optical part	N <sub>2</sub>	ALPHAGAZ FLO	N/A		
<b>GD (glow discharge) OES</b>					
Plasma	Ar	N/A	ALPHAGAZ 2		
Purge of optical part	N <sub>2</sub>	N/A	ALPHAGAZ 1		
Purge of optical part	N <sub>2</sub>	N/A	ALPHAGAZ FLO		
<b>X-ray fluorescence</b>					
Flow counter	Ar-CH <sub>4</sub> (90-10)	ALPHAGAZ MIX			
Flow counter	C <sub>n</sub> H <sub>m</sub> mixtures in He	ALPHAGAZ 1			
Detector cooling	N <sub>2</sub>	Liquid nitrogen			
Liquid analysis	He	ALPHAGAZ 1			
<b>UV fluorescence</b>					
Purge of optical part	N <sub>2</sub>	N/A	ALPHAGAZ 1***		
Purge of optical part	N <sub>2</sub>	N/A	ALPHAGAZ FLO		
Analysis of Hg	Ar	N/A	ALPHAGAZ 1		
Analysis of H <sub>2</sub> S or sulfurs	Air	N/A	ALPHAGAZ FLO		
Analysis of H <sub>2</sub> S or sulfurs	Air	N/A	ALPHAGAZ 1*		
<b>Chemiluminescence or CDL (Chemiluminescence detector)</b>					
Process gas	O <sub>2</sub>	ALPHAGAZ 1			
Process gas	Air	ALPHAGAZ FLO			
Process gas	Air	ALPHAGAZ 1***			
O <sub>3</sub> analysis	Ethylene	N/A	N35		
<b>Flame spectrometry</b>					
Sample introduction	A <sub>2</sub>	ALPHAGAZ 1	N/A		
Flame	O <sub>2</sub>	ALPHAGAZ 1	N/A		
Flame	N <sub>2</sub> O	ALPHAGAZ 1	N/A		
Flame	C <sub>2</sub> H <sub>2</sub>	ALPHAGAZ 1	N/A		

\*OES, optical emission spectroscopy.

\*\*ALPHAGAZ 2 in the case of nitrogen analysis.

\*\*\*Special grade (e.g., POL or VEM) in the case of analysis below 1 ppm.

Table 5

Techniques	Gas	Absorption spectroscopy Detection level (mol/mol or wt/wt)			
		<1000 ppm	<100 ppm	<10 ppm	<1 ppm
<b>Atomic absorption spectroscopy (AAS) with graphite furnace (GFAAS)</b>					
Graphite furnace	Ar	ALPHAGAZ 1			
<b>Atomic absorption with flame (AAS or FAAS)</b>					
Flame C <sub>2</sub> H <sub>2</sub> /Air	Air	ALPHAGAZ 1			
Flame C <sub>2</sub> H <sub>2</sub> /Air	Air	ALPHAGAZ FLO			
Flame	C <sub>2</sub> H <sub>2</sub>	ALPHAGAZ 1			
Flame C <sub>2</sub> H <sub>2</sub> /N <sub>2</sub> O	N <sub>2</sub> O	ALPHAGAZ 1			
Flame C <sub>2</sub> H <sub>2</sub> /N <sub>2</sub> O	C <sub>2</sub> H <sub>2</sub>	ALPHAGAZ 1			
Hydride analysis	N <sub>2</sub>	ALPHAGAZ 1			
Hydride analysis	N <sub>2</sub>	ALPHAGAZ FLO			
<b>Infrared (IR) spectroscopy</b>					
Purge or zero gas	N <sub>2</sub>	ALPHAGAZ 1*			
Purge or zero gas	N <sub>2</sub>	ALPHAGAZ FLO			
<b>FTIR spectroscopy</b>					
Purge or zero gas	N <sub>2</sub>	ALPHAGAZ 1*			
Purge or zero gas	N <sub>2</sub>	ALPHAGAZ FLO			
Cooling of the MCT detector**	N <sub>2</sub>	Liquid nitrogen			
<b>Elemental analysis of C, O, N, H, and S</b>					
N analysis: TCD**	He, Ar	ALPHAGAZ 1	ALPHAGAZ 2		
O analysis: IR analyzer	He, Ar	ALPHAGAZ 1	ALPHAGAZ 2		
C analysis: IR analyzer	O <sub>2</sub>	ALPHAGAZ 1	ALPHAGAZ 2		
S analysis: IR analyzer	O <sub>2</sub>	ALPHAGAZ 1	ALPHAGAZ 2		
H analysis: TCD	Ar, N <sub>2</sub>	ALPHAGAZ 1	ALPHAGAZ 2		
<b>Photoacoustic spectroscopy (PAS)</b>					
N <sub>2</sub> for calibration		ALPHAGAZ 1			
	N/A				
<b>Nuclear magnetic resonance (NMR), electron paramagnetic resonance (EPR) or (ESR)</b>					
	He	Liquid helium			
Cooling	N <sub>2</sub>	Liquid nitrogen			
Cooling	Air	ALPHAGAZ FLO			
Sample spinning					
<b>RAMAN spectroscopy</b>					
	N <sub>2</sub>	ALPHAGAZ 1			
Purge	N <sub>2</sub>	ALPHAGAZ FLO			
Purge	N <sub>2</sub>	Liquid nitrogen			
Cooling of the Ge detector					

\*ALPHAGAZ 2 in the case of CO<sub>2</sub> analysis (below 10 ppm).

\*\*MCT, mercury cadmium telluride; TCD, thermal conductivity detector.

Table 6

Techniques	Mass spectrometry	
	Gas	Grade
<b>MS</b>		
Fast atomic bombardment	Ar, Xe	ALPHAGAZ 1 or standard grades
Chemical ionization	NH <sub>3</sub> , CH <sub>4</sub> , isobutane	ALPHAGAZ 1 or standard grades
Tandem mass spectrometer (MS-MS)	Ar, N <sub>2</sub>	ALPHAGAZ 1 or standard grades
Atmospheric pressure ionization (API)	Ar, N <sub>2</sub>	ALPHAGAZ 1 or standard grades
<b>Coupling LC to MS</b>		
Venturi effect	Air, N <sub>2</sub>	ALPHAGAZ 1
Venturi effect	Air, N <sub>2</sub>	ALPHAGAZ FLO
Curtain gas	N <sub>2</sub> , He	ALPHAGAZ 1
Curtain gas	N <sub>2</sub>	ALPHAGAZ FLO
Nebulization	Air, N <sub>2</sub>	ALPHAGAZ 1
Nebulization	Air, N <sub>2</sub>	ALPHAGAZ FLO
<b>Coupling GC to MS</b>		
Open split coupling	He	ALPHAGAZ 1*

\*ALPHAGAZ 2 for low concentration analysis (below 10 ppm).

Table 7

Thermal analysis	Thermal analysis	
	Gas	Grade
<b>Thermogravimetric analysis (TGA), differential scanning, calorimetry (DSC)</b>		
Inert gases	Ar, N <sub>2</sub> , He	ALPHAGAZ 1
Process gases	O <sub>2</sub> , Air, H <sub>2</sub> , SO <sub>2</sub>	ALPHAGAZ 1 or standard grades
Process or inert gases	Air, N <sub>2</sub> , or H <sub>2</sub>	ALPHAGAZ FLO
Cooling	N <sub>2</sub>	Liquid nitrogen

Table 8

Techniques	Gas	Liquid or supercritical chromatography			
		Detection level (sample in solution)			
		g/L	g/L	g/L	mg/L
		0.1	0.01	10	<1
<b>Liquid chromatography (HPLC or LC)</b>					
Purge of solvent	He	ALPHAGAZ 1			
<b>Supercritical fluid chromatography (SFC)</b>					
Mobile phase	CO <sub>2</sub>	ALPHAGAZ 1 or SFE or SFC grades			
<b>Supercritical fluid extraction (SFE)</b>					
Mobile phase	CO <sub>2</sub>	ALPHAGAZ 1 or SFE or SFC grades			

Table 9

Surface analysis	Surface analysis	
	Gas	Grade
<b>X-ray photoelectron spectroscopy (XPS or electron spectroscopy for chemical analysis [ESCA])</b>		
Sputtering gas	Ar	ALPHAGAZ 1
Cooling of the detector	N <sub>2</sub>	Liquid nitrogen
<b>Auger electron spectroscopy (Auger or AES)*</b>		
Purging	N <sub>2</sub>	ALPHAGAZ 1
Cooling of the detector	N <sub>2</sub>	Liquid nitrogen
<b>Electron microscopy (SEM, TEM)*</b>		
Purging	N <sub>2</sub>	ALPHAGAZ 1
Cooling of EDAX detector	N <sub>2</sub>	Liquid nitrogen
<b>Specific surface (BET)*</b>		
Cooling	N <sub>2</sub>	Liquid nitrogen
Surface measurement	N <sub>2</sub>	ALPHAGAZ 2
Surface measurement	Kr	N48
Zero adjustment	He	ALPHAGAZ 2

\*AES, Auger electron spectroscopy; SEM, scanning electron microscopy; TEM, transmission electron microscopy; BET, Brunnauer, Emmett, and Teller method.

Nevertheless, for the analysis of very dilute samples, ALPHAGAZ 2 should be used to improve the baseline, particularly in cases involving the analysis of O, N, C in steel, or for the analysis of nitrogen in solids by glow discharge spectroscopy. For the analysis of atmospheric pollutants such as NO, NO<sub>2</sub>, or SO<sub>2</sub> by chemiluminescence or UV fluorescence, use of ALPHAGAZ 2 grade is suggested. For more assurance, certain grades such as pollution (POL) or Vehicle Emission Zero (VEM), which include specifications on these pollutants, may also be used. Similarly, two special grades of CO<sub>2</sub> have been developed for supercritical fluid chromatography applications (SFE [supercritical fluid extraction] and SFC [supercritical fluid chromatography] grades) that contain very low levels of heavy hydrocarbons. Except in these particular cases, ALPHAGAZ 1 and ALPHAGAZ FLO are usually the grades recommended.

Tables 4–11 list the most common analyzers using gas and the recommended grade. Only two grades of gas are necessary for supplying the most common laboratory analyzers. Furthermore, ALPHAGAZ FLO provides an alternative solution for supplying the gases, which are well adapted for some analyzers.

Table 10

Techniques	Gas	Other analysis			
		Detection level (sample in solution)			
		g/L	g/L	g/L	mg/L
		0.1	0.01	10	<1
<b>Water analysis (TOC, TOX)*</b>					
Oxidation or flushing	O <sub>2</sub>	ALPHAGAZ 1		ALPHAGAZ 2	
Purging and desorption	N <sub>2</sub> , He, Ar	ALPHAGAZ 1		ALPHAGAZ 2	
Purging and desorption	N <sub>2</sub>	ALPHAGAZ FLO			
<b>Measurement of water in solids (Karl Fischer analysis)</b>					
Water desorption	Air, N <sub>2</sub>	ALPHAGAZ 1		ALPHAGAZ 2	
Water desorption	Air, N <sub>2</sub>	ALPHAGAZ FLO			
<b>Metal analysis in petrochemistry (Wickbold method)</b>					
Flame	O <sub>2</sub>	ALPHAGAZ 1			
Flame	H <sub>2</sub>	ALPHAGAZ 1			
Flame	H <sub>2</sub>	ALPHAGAZ FLO			
Stripping	N <sub>2</sub>	ALPHAGAZ 1			
Stripping	N <sub>2</sub>	ALPHAGAZ FLO			

\*TOC, total organic carbon; TOX, total organic halides.

Table 11

Techniques	Gas	%	Gas analysis			
			Detection level (mol/mol)			
			<1000 ppm	<100 ppm	<10 ppm	<1 ppm
<b>Hygrometry</b>						
Purging or zero gas	N <sub>2</sub>	N/A	ALPHAGAZ 1		ALPHAGAZ 2	
Purging or zero gas	N <sub>2</sub>	N/A	ALPHAGAZ FLO			
<b>Explosivity or toxicity measurements</b>						
Purge or zero gas	Air, N <sub>2</sub>	ALPHAGAZ 1				
Purge or zero gas	Air, N <sub>2</sub>	ALPHAGAZ FLO				
<b>Total hydrocarbon (THC) measurement</b>						
Gas for flame	H <sub>2</sub>	ALPHAGAZ 1		ALPHAGAZ 2		
Gas for flame	H <sub>2</sub>	ALPHAGAZ FLO				
Gas for flame	H <sub>2</sub> /He	ALPHAGAZ MIX				
Gas for flame	Air	ALPHAGAZ 1		ALPHAGAZ 2		
Gas for flame	Air	ALPHAGAZ FLO				
<b>Measurement of O<sub>2</sub> using electrochemical cells coulometric or galvanic or zircon cells</b>						
Purge or zero gas	N <sub>2</sub>	N/A	ALPHAGAZ 1			
Purge or zero gas	N <sub>2</sub>	N/A	ALPHAGAZ FLO			
Zero gas	Air	N/A	ALPHAGAZ 1			
Zero gas	Air	N/A	ALPHAGAZ FLO			
<b>Measurement of O<sub>2</sub> using paramagnetic cells</b>						
Purge or zero gas	N <sub>2</sub>	ALPHAGAZ 1	N/A			
Purge or zero gas	N <sub>2</sub>	ALPHAGAZ FLO	N/A			
Zero gas	Air	ALPHAGAZ 1	N/A			
Zero gas	Air	ALPHAGAZ FLO	N/A			
<b>Measurement of H<sub>2</sub> using electrochemical cells</b>						
Purge or zero gas	N <sub>2</sub>	ALPHAGAZ 1	N/A			
Purge or zero gas	N <sub>2</sub>	ALPHAGAZ FLO	N/A			
<b>Measurement of carbon using electrochemical cells</b>						
Purge or zero gas	N <sub>2</sub>	ALPHAGAZ 1	N/A			
Purge or zero gas	N <sub>2</sub>	ALPHAGAZ FLO	N/A			

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- Gastiger M, Jurcik B. Choosing the proper gas and gas equipment for the laboratory, part 1: relationship between gas purity and detection limits in gas chromatography. *Int Lab* 1999; 29(4A):18.

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# Choosing the proper gas and gas equipment for the laboratory, Part 3: Factors influencing the point-of-use purity

Michel Gastiger and Benjamin Jurcik

**T**his article is the continuation of a series of publications regarding the selection of the correct gas and equipment necessary based on individual laboratory applications.

Gas purity requirements are directly related to analyzer requirements and the field of use or working domain. The requirements for these gases have been detailed in two previous papers.<sup>1,2</sup> In a similar fashion, it is necessary that the gas installation be well adapted to the purity of gas being delivered. This paper discusses how some design features of the gas installation and distribution system affect the purity of the gas at the point of use.

## Impurities and their relation to the gas system

The impurities that are affected by a pure gas system (Ar, H<sub>2</sub>, H<sub>e</sub>, and N<sub>2</sub>) and their sources are described in (Table 1).

Table 1  
Major impurities and their effect on the distribution system

Impurity/ contamination	Sources
Moisture (H <sub>2</sub> O)	Adsorption/desorption from the gas system material (tubes, filters, valves, MFCs, regulators, etc.) Humid air infiltration during cylinder changeout Leaks of humid air (valves, compression fittings, etc.) Pure gas supply system
Air (primarily O <sub>2</sub> or CO <sub>2</sub> )	Air infiltration during cylinder changeout Leaks (valves, compression fittings, etc.) Pure gas supply system
THC (total hydrocarbons)	Component cleaning residue Pure gas supply system

As illustrated in Table 1, however, the gas system (through either normal utilisation, faulty or low-quality installation) and the surface properties of the materials that make up the gas system, can contribute to the delivered point-of-use purity. It is evident that the source gas might be the impurity source for all of the impurities, which is why the selection of the appropriate gas quality is critical for the analytical application. With appropriate component specification and installation techniques, it is possible to obtain a leak-tight distribution system with no added THC, H<sub>2</sub>O, O<sub>2</sub>, or CO<sub>2</sub>.

Moisture, however, is more problematic since typical materials of construction have a large adsorption capacity for moisture and, as a result, there is typically a long purge-out time for moisture. The long purge-out time leads to a slow evolution of the moisture impurity delivered to the point of use.

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*Gas handling installations have been well recognized as playing a key role in the point-of-use purity of carrier gases.*

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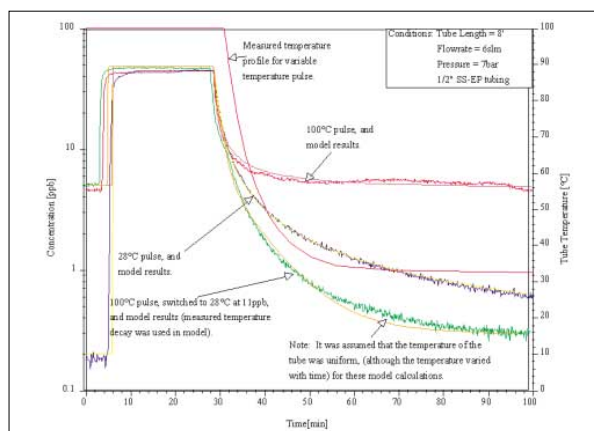
This article uses modeling techniques originally developed to aid the design of ultrahigh-purity (UHP) gas systems for the electronics industry (where state-of-the-art carrier gas specifications are currently less than 1 ppb) in order to compare different types of installations more commonly found in a laboratory.

Gas handling installations have been well recognized as playing a key role in the point-of-use purity of carrier gases. This realization has led to a great deal of work on how to test, compare, and select various components. Moisture is one of the most important impurities to test for because of its slow response characteristics (e.g., dry-down time, time to recover from an upset, etc.). The difficulty in purging moisture arises from the large adsorption capacity of moisture on metallic surfaces.

In order to evaluate the effect of the different components on the overall performance of the UHP distribution system, sophisticated mathematical models have been developed to simulate the transport of moisture in distribution systems.<sup>3,4</sup> These models can be used as a design tool or as an aid to component selection in the distribution system. A simple example of modeling as a design tool is given by McAndrew<sup>5</sup> where the effects of sampling line length, flow rate, and analyzer response time are examined in terms of what types of moisture upsets could be detected. An example of how modeling can be used as an aid to component selection is given by Jurcik,<sup>3</sup> where the effect of different types of components on the dry-down time of a simple distribution system is given. By analyzing the effect of the different components on the performance of the distribution system, the engineer can make a selection of components that gives the desired performance at minimum cost.

## Model description

The modeling of moisture evolution in a gas distribution system is based on a one-dimensional mass balance of the impurity, taking into account the adsorption isotherm, diffusion, and dispersion in the gas phase. In addition, the effect of dead spaces on the delayed purging of systems is taken into account. Local leaks may also be simulated by imposing a given leak rate.



**Figure 1** Comparison of model and experimental results for different temperature pulses.

The different adsorption isotherms of various materials and the characteristics of components have been integrated into a proprietary program: INTAL (Integrated Network Transport Air Liquide) (Air Liquide Corp., France).

An example of the validity of the model is briefly illustrated. Three different experiments were performed and a simulation of the same conditions carried out. The three experiments are described below, and the results of the simulations and measurements are shown in *Figure 1*.

1. A moisture input experiment was performed at room temperature (28 °C). The tube was dried down to ~0.2 ppb, and a 25-min, 42-ppb pulse was introduced into the tube. For all of the experiments with this moisture input the system came to equilibrium at 42 ppb within a few minutes, long before the moisture input was switched off. As a result, the experiments could be analyzed as a step-up followed by a step-down. The moisture concentration at the exit of the tube was monitored by an APIMS (Atmospheric Pressure Ionisation Mass Spectrometer).

2. A moisture input experiment was performed at elevated temperature (100 °C). The tube was dried down to ~4.5 ppb at 100 °C, and a 25-min, 42-ppb pulse was introduced into the tube. The moisture concentration at the exit of the tube was monitored by an APIMS.

3. A moisture input experiment was performed with the baking switched off during the dry-down. The tube was dried down to ~4.5 ppb at 100 °C, and a 25-min, 42-ppb pulse of moisture was introduced into the tube. When the moisture concentration at the outlet of the tube had reached 11 ppb (after 28 min total time) the heating was switched off.

The slow response time of the high-quality 316L SS-EP material can be seen in *Figure 1*. With lower-quality materials (e.g., no electropolishing, etc.), the response time will be slower.

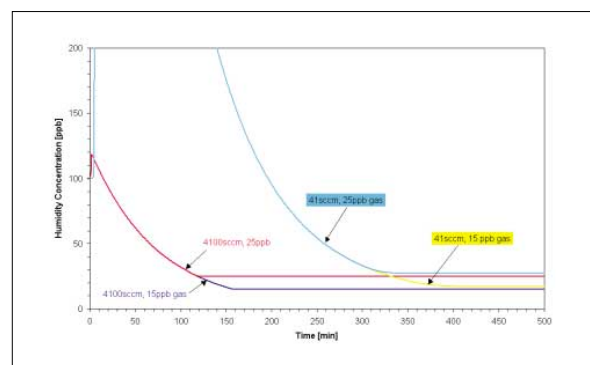
### Example installation

The specific application examined here is the supply of N<sub>2</sub> from high-pressure cylinders for a gas chromatographic (GC) application. The authors estimated the point-of-use moisture concentration as a function of time for two different gas grades and two different types of installations.

The system modeled here is the simplest possible, which supplies a single analyser from a high-pressure cylinder (specifically, a high-pressure cylinder with a regulator, mass flow controller, and tubing). The total length of tubing considered is 5.4 m long. For the simulations, a constant flow rate is considered, starting from the connection to the cylinder valve. It is considered that the entire installation is purged to 100 ppb just before the connection to the cylinder valve. This is, of course, an idealistic initial condition: (With the simple system described, just before connection the line is exposed to air). It was chosen to illustrate the effect (in terms of quantity and duration) the purging of the cylinder valve can have on the impurity content at the point of use. Typical practical leak rates were taken for the two types of installations and do not represent the state of the art, better ultimate results (closer to the source gas) could be attainable.

### Results

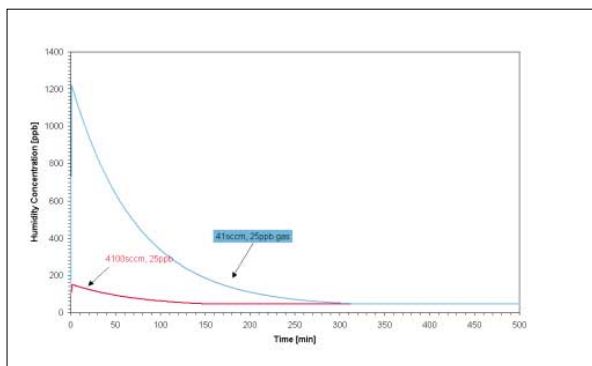
The moisture concentration as a function of time for a high-purity installation is shown in *Figure 2*. Four different cases are simulated: a source gas of 15 and 25 ppb, and a flow rate of 4100 and 41 sccm. The flow rates were chosen to simulate the case in which the purge is performed with the same flow rate as used by the GC, and one that is 10× higher in order to decrease the purge time. The low levels of moisture concentration are selected as representative of levels from a full cylinder (200 bar). At a high pressure such as this, any moisture that is present tends to adsorb on the cylinder walls, and the gas that initially exits the cylinder has a low moisture level. The conditions were chosen, therefore, to mimic the purging of a simple line with the connection of a new cylinder. As can be seen in *Figure 2*, there is a long contamination of moisture in the system from the purging of the cylinder valve. The slight difference between the two ultimate concentrations at the different flow rates is due to the dilution effect as the same leak rate was taken.



**Figure 2** Moisture concentration as a function of time for high-purity installation.

For a standard installation, the results for two different flow rates are shown in *Figure 3*.

In this case, there is a more significant difference between the two flow rates because the final effect of leak rate is much more important in this lower-purity installation. In fact, for this standard installation, in comparison to the high-purity installation, there is not a large difference in duration to reach



**Figure 3** Results of two different flow rates in a standard installation.

the final equilibrium value at the point of use, although there is a significant difference in the magnitude at the point of use. The reason for this phenomenon is that moisture responds much more rapidly at higher concentrations than at lower concentrations. This is illustrated by the fact that the adsorption isotherm of moisture is very steep at low partial pressures (e.g., tens of ppb at a pressure of several bar) of moisture while at higher concentrations there is more surface saturation and therefore the incremental adsorption capacity is much less.

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*When designing and specifying gas installation systems, a supplier who understands the relationship between the material quality, cost, purge time, and the analytical requirements is needed to ensure a high-quality result.*

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### Conclusion

The influence of different installation materials has been demonstrated. Clearly, the purity requirements for a given analyser/analysis need to be taken into

account during the installation. In addition, the time necessary to reach the gas specifications at the point of use is not negligible for moisture due to its strong interaction with the material surfaces. For a typical installation, either several hours are needed to purge the line or a large flow rate is needed. In either case, a cost is associated with this purge (gas or productivity), which should be considered in the installation design. When designing and specifying gas installations systems, a supplier who understands the relationship between the material quality, cost, purge time, and the analytical requirements is needed to ensure a high-quality result. Part 4 of this series will discuss general recommendations for the different types of installations.

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# Guidelines to help to choose the correct gas & gas equipment for the laboratory

## Part 4: Optimal gas and equipment solutions at the inlet of the analyzers

Michel Gastiger and Benjamin Jurcik

### Introduction

In our previous article, we have shown the nature and the source of major contamination encountered in gas distribution systems. Our modeling study illustrates why the purging time, flow rate and procedures are important to respect. Similarly, we have demonstrated that by using low cost materials, the point of use purity of gas contained initially in the cylinder, especially in the case of the moisture, may not be maintained.

In this paper we will propose the correct equipment, piping and installation in order to maintain the quality of gas chosen by the analyst in relation to the application requirements. By using information contained in Parts One and Two, the scientist will be able to choose the correct gas and equipment in order to construct the correct installation needed required for maintaining gas purity at the point of use.

### Effects of Impurity on the analysis.

In TABLE 1, we summarize the major effects of moisture, oxygen, hydrocarbons or CO<sub>2</sub> for an analyzer or an analysis. The reader can find more information and details in the review of J. V. Hinshaw.

In general, these impurities have a detrimental effect for analysis at low concentration level (below ppmv or mg/l). For analysis at higher concentration, these molecules can damage some parts of the analyzer if they are

present at more than some ppm volume.

An illustration of impurity effects is presented by S. Reese where the combined effect of oxygen and high temperature on the separation and resolution of peaks in gas chromatography is demonstrated. Oxygen and high temperature change the composition of the stationary phase and the analyst loses column efficiency, increased column bleed and/or increase column activity toward polar compounds.

R.J. Boyles has pointed out the effect of hydrocarbon outgassing coming from low quality material used in low cost valves or pressure regulators on the gas chromatography efficiency and column life time.

### Recommendations for the choice of the correct equipment.

Using Part One<sup>2</sup> and Part Two<sup>3</sup>, the selection of the correct grade of gas is easy. It is essential to keep this grade through the piping and equipment up to the point of use, the material and the installation must be chosen and assembled correctly. To transport a pure gas from a cylinder (or a bundle, or a liquid storage, or a generator) to an analyzer or an application, it is necessary to use pressure regulator(s), valve(s), fittings and tubing, we will give basic recommendations to help to choose the correct equipment.

Table 1 Some effects of major impurities on analysis.

NOTA: The list above is not exhaustive, but merely illustrates some of the most well known impurity effects.

IMPURITY	Analyzers of Techniques	Effects for concentrations above some ppm or extreme working conditions
Moisture (H <sub>2</sub> O)	GC Columns ICP (OES) Infra Red Analyzer <i>Spark discharge, UV Fluorescence may also be affected by water concentration above 5 to 10 ppm</i>	Irreversible Degradation (cyano based stationary phase) Masking some parts of UV spectra Masking some parts of IR spectra
Oxygen (O <sub>2</sub> )	GC Columns ICP (OES) GC-ECD GC-HID GC-TCD <i>Spark discharge, UV Fluorescence or X-Ray Fluorescence may also be affected by oxygen concentration above 5 to 10 ppm</i>	Irreversible Degradation (cyano based stationary phase) Masking some parts of UV spectra Noise and background current Negative peaks Oxidation of the filament
Heavy Hydrocarbons	GC-FPD GC-PID ICP (MS or OES) ICP (OES) GC-FID <i>UV Fluorescence or X-Ray Fluorescence may also be affected by hydrocarbon concentration above 5 to 10 ppm</i>	Reduce sensitivity and contamination of windows Window contamination Disturbance of ignition Contamination of windows Reduce sensitivity of the analyzer
Halogenated Hydrocarbons	GC-ECD	Reduce sensitivity, damage the ECD Detector
Carbon Dioxide (CO <sub>2</sub> )	TOC analysis Infra red analyzer ICP (OES) GC	Reduce sensitivity of the analyzer Bind some parts of IR spectra Bind some parts of UV spectra Baseligne drifting at high sensivity

Parts	Standard Equipment; Analysis around 10 ppm or 1mg/l	High purity Equipment Analysis around 100 ppb or 10 µg/l	Ultra High Purity Equipment Analysis below 10 ppb or 1 µg/l
Gas Grade	<b>Alphagaz 1</b>	<b>Alphagaz 2</b>	<b>Alphagaz 2</b>
Number of Stages	Single or two stages	Single or two stages	Single or two stages
Body	Brass	Brass or stainless steel	Stainless steel
Diaphragm or bellows	Bronze	Bronze or stainless steel	Stainless steel
Gasket	EPDM	EPDM* or PTFCE	PTFCE or metal/metal
He Leak Rate (mbar.l/sec)	< 10-6	< 10-7	< 10-9

\* Static O-ring

Table 3 General recommendations for the selection of valves.

### High and low pressure VALVES (General recommendations)

Parts	Standard Equipment; Analysis around 10 ppm or 1mg/l	High purity Equipment Analysis around 100 ppb or 10 µg/l	Ultra High Purity Equipment Analysis below 10 ppb or 1 µg/l
Gas Grade	<b>Alphagaz 1</b>	<b>Alphagaz 2</b>	<b>Alphagaz 2</b>
Body	Brass	Stainless steel	Stainless steel
Valve	Polymer coated brass	Polymer coated stainless steel	Polymer coated stainless steel
Seat	Brass	Brass or stainless steel	Stainless steel
Gasket	EPDM*	PTCFE or metal/metal	Metal/metal
He Leak Rate (mbar.l/sec)	< 10-6	< 10-7	< 10-8

\* Dynamic O-ring

## Pressure Regulators and Valves

Table 2 and 3 summarize the general recommendations to be followed. In addition, pressure regulators or valves must be correctly degreased by the manufacturer. Maximum input and output pressure, maximum flow rate must be checked before ordering. Leak rate values give a good global information on the capability of valves and pressure regulators to be used with the selected gas grade.

## Tubing and Fittings

Because of the relatively large diffusion rate of small molecules through the polymeric materials, the use of these materials should be avoided. For example, using the permeability of a polyamide or PTFE, a 1 meter length of 6 mm in diameter pipe introduces about 10 ppm of O<sub>2</sub> in an argon flow (80 cm<sup>3</sup>/min). Classical metal external tube diameters (1/8 or 1/4 in or 6 mm) are compatible with standard pressure and flow rate for laboratory uses. As show in table 4, copper or stainless steel can be used (except copper for C<sub>2</sub>H<sub>2</sub>). Tubes must be degreased and cleaned by the manufacturer.

During the assembly, dirt and metal fragment will be removed. The procedure for the connection of fittings is very well described by the manufacturer and must be correctly applied.

## Mass Flow Meters

This equipment, sold by international manufacturers is well adapted for laboratory applications. Mass flow meter having a leak rate value less than 10<sup>-9</sup> atm.cm<sup>3</sup>/sec can be used for all of your pure gas applications.

## Purifiers

For ultra high purity applications (see Part One<sup>2</sup> and Two<sup>3</sup>), we recommend point of use purifiers. AIR LIQUIDE has checked some of them (Table 5) and is able to propose the correct purifiers required, based upon equipment and analysis.

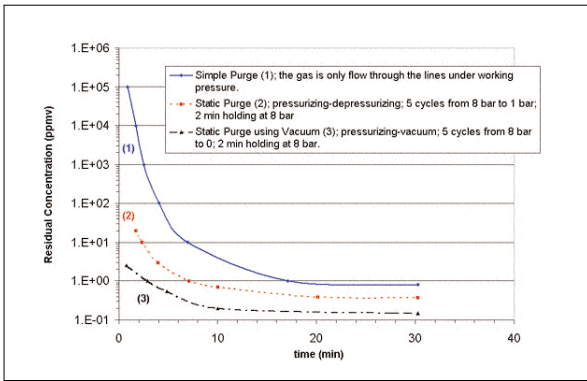
## Purging

ALPHAGAZ 2 gas grade requires a cross purge assembly to avoid air infiltration during the cylinder changeout.

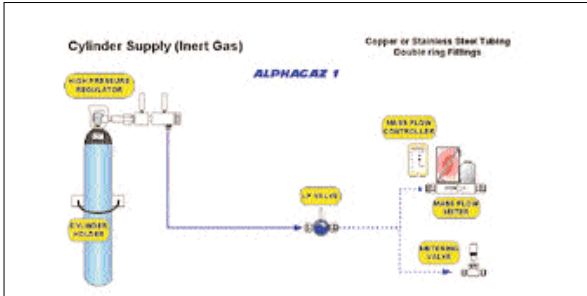
Table 4 General recommendations for choice of tubing and fittings.

### tubing and Fittings (General recommendations)

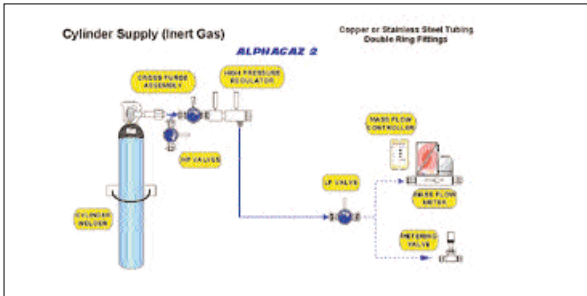
Parts	Standard Equipment; Analysis around 10 ppm or 1mg/l	Analysis around 100 ppb or 10 µg/l	Ultra High Purity Equipment Analysis below 10 ppb or 1 µg/l
Gas Grade	<b>Alphagaz 1</b>	<b>Alphagaz 2</b>	<b>Alphagaz 2</b>
Material for N <sub>2</sub> , He, O <sub>2</sub> , Air H <sub>2</sub> , CO <sub>2</sub> , Ar, N <sub>2</sub> O	Copper	Copper or stainless steel	Stainless steel or Electropolished stainless steel
Material for C <sub>2</sub> H <sub>2</sub>	Stainless steel	Stainless Steel	
Fittings	Brass Double ring type High purity Equipment	Brass or stainless steel double ring Type	Stainless steel VCR™ Type orButt welding



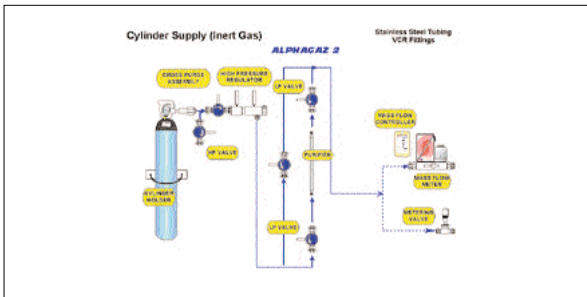
**Fig. 1** Effects of different purging for residual impurity concentrations in piping.



**Fig. 2** Standard equipment using ALPHAGAZ 1 pure gas grade.



**Fig. 3** high purity equipment using with ALPHAGAZ 2 pure gas grade.



**Fig. 4** Ultra high purity equipment using with ALPHAGAZ 2 pure gas grade.

Figure 1 shows the advantage of accurate purge cycles for simple cylinder changeout. For very low concentration analysis levels, purge cycles involving vacuum and pressurization of all parts of piping and all equipment will be performed. Taking into account these recom-

*Table 5 Specifications and test results of commercial purifiers.*

Gas	Specifications			Results of tests		
	Manuf.	Total Cap. (cm3)	Max flow rate (l/min)	Max pressure (bar)	Impurity concent. in input (ppm)	Impurity concent. in output (ppb)
Oxygen	A	100	3.3	200	5 to 10	<5
	B	720	6	83	5 to 50	<5
	C	1000	1	10	5 to 50	<5
Moisture	A	100	303	200	20 to 30	<20
	B	20000	5	8.5	2 to 20	<60
	C	9000	2	10	15 to 100	<400
Hydrocarb. & Halogen. Hydrocarb.	C	n/a	2	10	25(CH4) 25(C2H6) 25(C3H8) 25(nC4H10) 0.19 (C2F2C12)	no absorp. no absorp. <20 <20 <1

mendations, it will be easy to implement cylinder sources of gas in your laboratory. This information can be retrieved from a recent publication<sup>7</sup>.

### Conclusions and proposals of standard installations.

In conclusion, we propose three installation diagrams illustrating the correct assembly of basis equipment to connect a cylinder (or bundle) to an analyzer (Figures 2-4). For uninterrupted feed, AL proposes automatic changeover manifold; it provides a continuous gas supply when the pressure of the first cylinder drops to a preset level, by automatically changing for the other cylinder (or bundle).

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