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

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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 CO2 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[2] and Part Two[3], 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 (H20)	GC Columns	Irreversible Degradation (cyano based stationary phase)
	ICP (OES)	Masking some parts of UV spectra
	Infra Red Analyzer	Masking some parts of IR spectra
	Spark discharge, UV Fluorescence may also be affected by water concentration above 5 to 10 ppm	
Oxygen (02)	GC Columns	Irreversible Degradation (cyano based stationary phase)
	ICP (OES)	Masking some parts of UV spectra
	GC-ECD	Noise and background current
	GC-HID	Negative peaks
	GC-TCD	Oxidation of the filament
	Spark discharge, UV Fluorescence or X-Ray Fluorescence may also be affected by oxygen concentration above 5 to 10 ppm	
Heavy Hydrocarbons	GC-FPD	Reduce sensitivity and contamination of windows
	GC-PID	Window contamination
	ICP (MS or OES)	Disturbance of ignition
	ICP (OES)	Contamination of windows
	GC-FID	Reduce sensitivity of the analyzer
	UV Fluorescence or X-Ray Fluorescence may also be affected by hydrocarbon concentration above 5 to 10 ppm	
Halogenated Hydrocarbons	GC-ECD	Reduce sensitivity, damage the ECD Detector
Carbon Dioxide (CO2)	TOC analysis	Reduce sensitivity of the analyzer
	Infra red analyzer	Bind some parts of IR spectra
	ICP (OES)	Bind some parts of UV spectra
	GC	Baseligne drifting at high sensivity

Parts	Standard Equipment; Analysis arround 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	**Alphagaz 1**	**Alphagaz 2**	**Alphagaz 2**
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 belows	Bronze	Bronze or stainless steel	Stainless steel
Gasket	EPDM	EPDN* 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 arround 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	**Alphagaz 1**	**Alphagaz 2**	**Alphagaz 2**
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 O2 in an argon flow (80 cm3/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 C2H2). 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 10-9 atm.cm3/sec can be used for all of your pure gas applications.

Purifiers

For ultra high purity applications (see Part One[2] and Two[3]), 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 arround 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	**Alphagaz 1**	**Alphagaz 2**	**Alphagaz 2**
Material for N2, He, 02, Air H2, CO2, Ar, N20	Copper	Copper or stainless steel	Stainless steel or Electropolished stainless steel
Material for C2H2	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

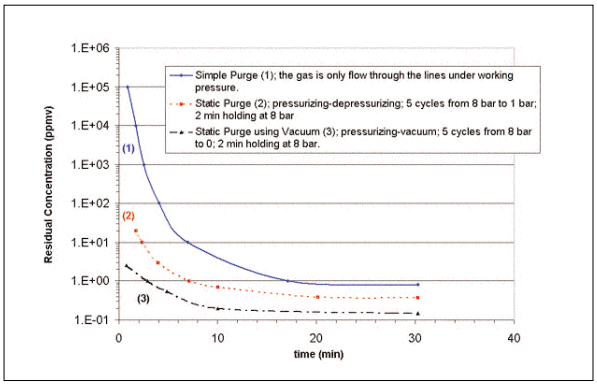

Fig. 1 *Effects of different purging for residual impurity concentrations in piping.*

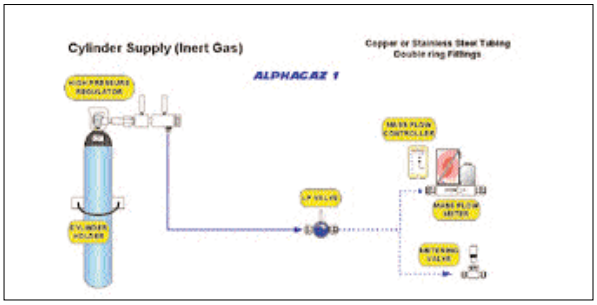

Fig. 2 *Standard equipment using ALPHAGAZ 1 pure gas grade.*

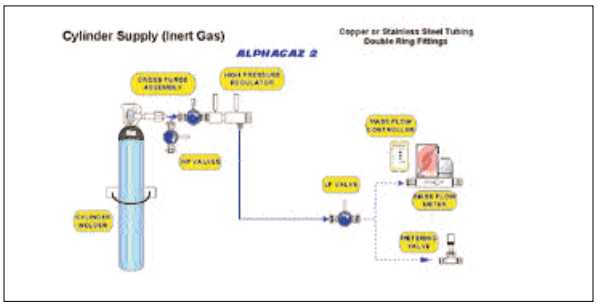

Fig. 3 *high purity equipment using with ALPHAGAZ 2 pure gas grade.*

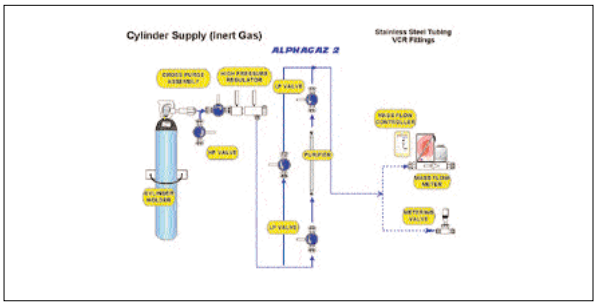

Fig. 4 *Ultra high purity equipment using with ALPHAGAZ 2 pure gas grade.*

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 recommendations, it will be easy to implement cylinder sources of gas in your laboratory. This information can be retrieved from a recent publication[7].

Table 5 Specifications and test results of commercial purifiers.

	Specifications				Results of tests	
Gas	**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

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).

References

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