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

Michel Gastiger and Benjamin Jurcik

Gas purity requirements are directly related to analyzer requirements and the field of use or the working domain. For example, Part 1 of this paper[1] 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 u se 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.[1]

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_2) or 99.99999% (H_2). The large range of flow rate and pressure permits the gas to be fed to one or more analyzers. The typical specifications of 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*).

Table 1

Specifications of ALPHAGAZ 1 and ALPHAGAZ 2

Gases	ALPHAGAZ 1	ALPHAGAZ 2
Ar, H_2, He, N_2	H_2O < 3 ppm O_2 < 2 ppm C_nH_m < 0.5 ppm	H_2O < 0.5 ppm O_2, C_nH_m, CO, CO_2 < 0.1 ppm N_2 < 0.1 ppm in Ar, He H_2 < 0.1 ppm in Ar, He, N_2
Air	H_2O < 3 ppm C_nH_m < 0.5 ppm	— H_2O < 0.5 ppm
O_2	H_2O < 3 ppm C_nH_m < 0.5 ppm	H_2, C_nH_m, CO, CO_2 < 0.1 ppm N_2 < 4 ppm NO_x < 15 ppb (typical analysis) H_2O < 3 ppm
CO_2	H_2O < 20 ppm	C_nH_m < 2 ppm O_2 < 2 ppm N_2 < 8 ppm
C_2H_2	N_2 < 0.4%	—
N_2O	N_2 < 0.4%	—

Table 2

Specifications of ALPHAGAZ FLO generators

Gas	Purity	Maximum output flow rate (L/min)	Maximum output pressure (bar)	Typical analysis
Nitrogen	>99.99%	1–11	Up to 8.6	CO, CO_2, O_2 < 1ppm H_2O < 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_4) < 0.1 ppm

Table 3

Range of ALPHAGAZ MIX

ALPHAGAZ MIX	Concentrations	Uses
Methane–argon	5 or 10% CH_4 in argon	Electron capture detector, X-ray fluorescence, nuclear counter
Hydrogen–helium	40% H_2 in helium	Flame ionization detector
Hydrogen–argon	0.75–7% H_2 in argon	Spark emission

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

Techniques	Gas	%	Detection level (mol/mol or wt/wt) <1000 ppm	<100 ppm	<10 ppm	<1 ppm
Inductively coupled plasma (ICP)						
Auxiliary plasma flow	Ar		ALPHAGAZ 1			
Plasma flow	Ar		ALPHAGAZ 1			
*Optical detector (ICP-OES)**						
Purge of optical part	N_2		ALPHAGAZ 1			
Purge of optical part	N_2		ALPHAGAZ FLO			
Mass spectrometer (ICP-MS)						
Auxiliary plasma flow	Ar		N/A	ALPHAGAZ 1		
Plasma flow	Ar		N/A	ALPHAGAZ 1		
Other						
Organic solvent analysis	O_2		ALPHAGAZ 1			
	H_2		ALPHAGAZ 1			
Spark emission						
Plasma	Ar		ALPHAGAZ 1**		N/A	
Plasma	Ar + 1–7% H_2		ALPHAGAZ MIX		N/A	
Purge of optical part	N_2		ALPHAGAZ 1		N/A	
Purge of optical part	N_2		ALPHAGAZ FLO		N/A	
GD (glow discharge) OES						
Plasma	Ar		N/A	ALPHAGAZ 2		
Purge of optical part	N_2		N/A	ALPHAGAZ 1		
Purge of optical part	N_2		N/A	ALPHAGAZ FLO		
X-ray fluorescence						
Flow counter	Ar–CH_4 (90–10)		ALPHAGAZ MIX			
Flow counter	C_nH_m mixtures in He		ALPHAGAZ 1			
Detector cooling	N_2		Liquid nitrogen			
Liquid analysis	He		ALPHAGAZ 1			
UV fluorescence						
Purge of optical part	N_2		N/A	ALPHAGAZ 1***		
Purge of optical part	N_2		N/A	ALPHAGAZ FLO		
Analysis of Hg	Ar		N/A	ALPHAGAZ 1		
Analysis of H_2S or sulfurs	Air		N/A	ALPHAGAZ FLO		
Analysis of H_2S or sulfurs	Air		N/A	ALPHAGAZ 1*		
Chemiluminescence or CDL (Chemiluminescence detector)						
Process gas	O_2		ALPHAGAZ 1			
Process gas	Air		ALPHAGAZ FLO			
Process gas	Air		ALPHAGAZ 1***			
O_3 analysis	Ethylene		N/A		N35	
Flame spectrometry						
Sample introduction	A_2		ALPHAGAZ1		N/A	
Flame	O_2		ALPHAGAZ 1		N/A	
Flame	N_2O		ALPHAGAZ 1		N/A	
Flame	C_2H_2		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

Absorption spectroscopy

Techniques	Gas	%	Detection level (mol/mol or wt/wt) <1000 ppm	<100 ppm	<10 ppm	<1 ppm
Atomic absorption spectroscopy (AAS) with graphite furnace (GFAAS)						
Graphite furnace	Ar		ALPHAGAZ 1			
Atomic absorption with flame (AAS or FAAS)						
Flame C_2H_2/Air	Air		ALPHAGAZ 1			
Flame C_2H_2/Air	Air		ALPHAGAZ FLO			
Flame	C_2H_2		ALPHAGAZ 1			
Flame C_2H_2/N_2O	N_2O		ALPHAGAZ 1			
Flame C_2H_2/N_2O	C_2H_2		ALPHAGAZ 1			
Hydride analysis	N_2		ALPHAGAZ 1			
Hydride analysis	N_2		ALPHAGAZ FLO			
Infrared (IR) spectroscopy						
Purge or zero gas	N_2		ALPHAGAZ 1*			
Purge or zero gas	N_2		ALPHAGAZ FLO			
FTIR spectroscopy						
Purge or zero gas	N_2		ALPHAGAZ 1*			
Purge or zero gas	N_2		ALPHAGAZ FLO			
Cooling of the MCT detector**	N_2		Liquid nitrogen			
Elemental analysis of C, O, N, H, and S						
N analysis: TCD**	He, Ar		ALPHAGAZ 1		ALPHAGAZ 2	
O analysis: IR analyzer	He, Ar		ALPHAGAZ 1		ALPHAGAZ 2	
C analysis: IR analyzer	O_2		ALPHAGAZ 1		ALPHAGAZ 2	
S analysis: IR analyzer	O_2		ALPHAGAZ 1		ALPHAGAZ 2	
H analysis: TCD	Ar, N_2		ALPHAGAZ 1		ALPHAGAZ 2	
Photoacoustic spectroscopy (PAS)						
N_2 for calibration			ALPHAGAZ 1			
N/A						
Nuclear magnetic resonance (NMR), electron paramagnetic resonance (EPR) or (ESR)	He		Liquid helium			
Cooling	N_2		Liquid nitrogen			
Cooling	Air		ALPHAGAZ FLO			
Sample spinning						
RAMAN spectroscopy	N_2		ALPHAGAZ 1			
Purge	N_2		ALPHAGAZ FLO			
Purge	N_2		Liquid nitrogen			
Cooling of the Ge detector						

*ALPHAGAZ 2 in the case of CO_2 analysis (below 10 ppm).
**MCT, mercury cadmium telluride; TCD, thermal conductivity detector.

Table 6

Mass spectrometry

Techniques	Gas	Grade
MS		
Fast atomic bombardment	Ar, Xe	ALPHAGAZ 1 or standard grades
Chemical ionization	NH_3, CH_4, isobutane	ALPHAGAZ 1 or standard grades
Tandem mass spectrometer (MS-MS)	Ar, N_2	ALPHAGAZ 1 or standard grades
Atmospheric pressure ionization (API)	Ar, N_2	ALPHAGAZ 1 or standard grades
Coupling LC to MS		
Venturi effect	Air, N_2	ALPHAGAZ 1
Venturi effect	Air, N_2	ALPHAGAZ FLO
Curtain gas	N_2, He	ALPHAGAZ 1
Curtain gas	N_2	ALPHAGAZ FLO
Nebulization	Air, N_2	ALPHAGAZ 1
Nebulization	Air, N_2	ALPHAGAZ FLO
Coupling GC to MS		
Open split coupling	He	ALPHAGAZ 1*

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

Table 7

Thermal analysis

Thermal analysis	Gas	Grade
Thermogravimetric analysis (TGA), differential scanning, calorimetry (DSC)		
Inert gases	Ar, N_2, He	ALPHAGAZ 1
Process gases	O_2, Air, H_2, SO_2. . .	ALPHAGAZ 1 or standard grades
Process or inert gases	Air, N_2, or H_2	ALPHAGAZ FLO
Cooling	N_2	Liquid nitrogen

Table 8

Liquid or supercritical chromatography

		Detection level (sample in solution)				
		0.1	0.01	10	<1	
Techniques	**Gas**	**g/L**	**g/L**	**g/L**	**mg/L**	**mg/L**
Liquid chromatography (HPLC or LC)						
Purge of solvent	He	ALPHAGAZ 1				
Supercritical fluid chromatography (SFC)						
Mobile phase	CO_2	ALPHAGAZ 1 or SFE or SFC grades				
Supercritical fluid extraction (SFE)						
Mobile phase	CO_2	ALPHAGAZ 1 or SFE or SFC grades				

Table 9

Surface analysis

Surface analysis	Gas	Grade
X-ray photoelectron spectroscopy (XPS or electron spectroscopy for chemical analysis [ESCA])		
Sputtering gas	Ar	ALPHAGAZ 1
Cooling of the detector	N_2	Liquid nitrogen
Auger electron spectroscopy (Auger or AES)*		
Purging	N_2	ALPHAGAZ 1
Cooling of the detector	N_2	Liquid nitrogen
Electron microscopy (SEM, TEM)*		
Purging	N_2	ALPHAGAZ 1
Cooling of EDAX detector	N_2	Liquid nitrogen
Specific surface (BET)*		
Cooling	N_2	Liquid nitrogen
Surface measurement	N_2	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_2, or SO_2 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_2 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

Other analysis

		Detection level (sample in solution)				
		0.1	0.01	10	<1	
Techniques	**Gas**	**g/L**	**g/L**	**g/L**	**mg/L**	**mg/L**
Water analysis (TOC, TOX)*						
Oxidation or flushing	O_2	ALPHAGAZ 1				ALPHAGAZ 2
Purging and desorption	N_2, He, Ar	ALPHAGAZ 1				ALPHAGAZ 2
Purging and desorption	N_2	ALPHAGAZ FLO				
Measurement of water in solids (Karl Fischer analysis)						
Water desorption	Air, N_2	ALPHAGAZ 1				ALPHAGAZ 2
Water desorption	Air, N_2	ALPHAGAZ FLO				
Metal analysis in petrochemistry (Wickbold method)						
Flame	O_2	ALPHAGAZ 1				
Flame	H_2	ALPHAGAZ 1				
Flame	H_2	ALPHAGAZ FLO				
Stripping	N_2	ALPHAGAZ 1				
Stripping	N_2	ALPHAGAZ FLO				

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

Table 11

Gas analysis

			Detection level (mol/mol)			
			<1000	<100	<10	<1
Techniques	**Gas**	**%**	**ppm**	**ppm**	**ppm**	**ppm**
Hygrometry						
Purging or zero gas	N_2	N/A	ALPHAGAZ 1		ALPHAGAZ 2	
Purging or zero gas	N_2	N/A	ALPHAGAZ FLO			
Explosivity or toxicity measurements						
Purge or zero gas	Air, N_2		ALPHAGAZ 1			
Purge or zero gas	Air, N_2		ALPHAGAZ FLO			
Total hydrocarbon (THC) measurement						
Gas for flame	H_2		ALPHAGAZ 1	ALPHAGAZ 2		
Gas for flame	H_2		ALPHAGAZ FLO			
Gas for flame	H_2/He		ALPHAGAZ MIX			
Gas for flame	Air		ALPHAGAZ 1	ALPHAGAZ 2		
Gas for flame	Air		ALPHAGAZ FLO			
Measurement of O_2 using electrochemical cells coulometric or galvanic or zircon cells						
Purge or zero gas	N_2	N/A	ALPHAGAZ 1			
Purge or zero gas	N_2	N/A	ALPHAGAZ FLO			
Zero gas	Air	N/A	ALPHAGAZ 1			
Zero gas	Air	N/A	ALPHAGAZ FLO			
Measurement of O_2 using paramagnetic cells						
Purge or zero gas	N_2 ALPHAGAZ 1		N/A			
Purge or zero gas	N_2 ALPHAGAZ FLO		N/A			
Zero gas	Air ALPHAGAZ 1		N/A			
Zero gas	Air ALPHAGAZ FLO		N/A			
Measurement of H_2 using electrochemical cells						
Purge or zero gas	N_2 ALPHAGAZ 1		N/A			
Purge or zero gas	N_2 ALPHAGAZ FLO		N/A			
Measurement of carbon using electrochemical cells						
Purge or zero gas	N_2 ALPHAGAZ 1		N/A			
Purge or zero gas	N_2 ALPHABAZ FLO		N/A			

Reference

1. 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.

Dr. Gastiger is with the Pure Gases and Mixtures Analysis and Laboratory Market, and Dr. Jurcik is Group Manager, Application of Gases for Electronics and Laboratories, ***AIR LIQUIDE Corp.,*** *Claude-Delorme Research Center, BP 126, 78354 Jouy-en-Josas, Cedex, France; tel.: (33) 1 39 07 62 44; fax: (33) 1 39 07 64 66; e-mail: benjamin.jurcik@airliquide.com.*