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

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This 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.[1,2] 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_2, H_e, and N_2) and their sources are described in (*Table 1*).

Table 1

Major impurities and their effect on the distribution system

Impurity/ contamination	Sources
Moisture (H_2O)	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_2 or CO_2)	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_2O, O_2, or CO_2.

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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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.[3,4] 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[5] 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,[3] 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.

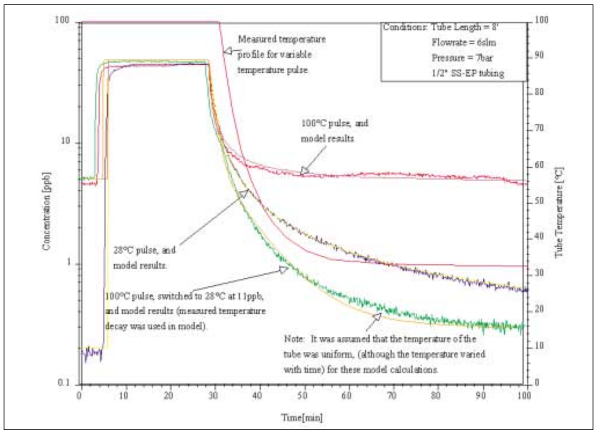

Figure 1 *Comparison of model and experimental results for different temperature pulses.*

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_2 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.

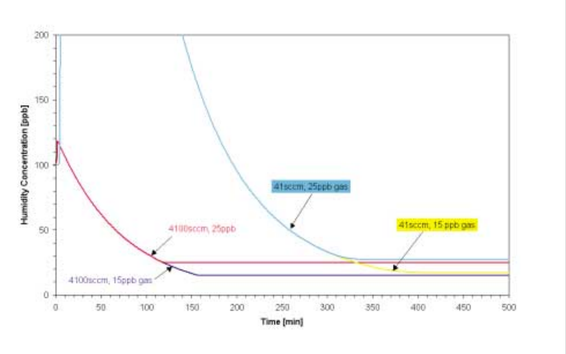

Figure 2 *Moisture concentration as a function of time for high-purity installation.*

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

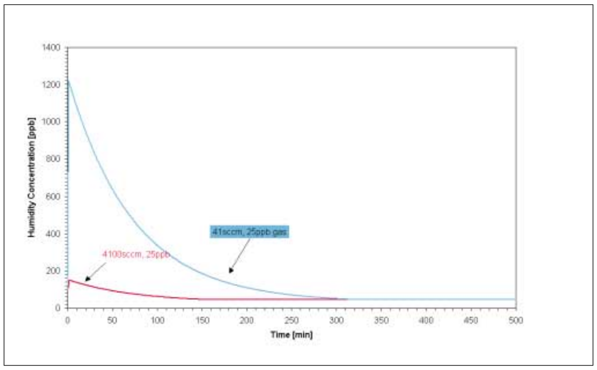

Figure 3 *Results of two different flow rates in a standard installation.*

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.

> *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.*

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.

References

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