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### Gases and gas handling in the laboratory environment

Stephen Harrison takes a look at how to safely handle high purity gases in the lab

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*"Today's specialty gases for calibrating and measuring gases and for measuring liquids and solids are on the cutting edge of technology"*

Over the past decade, manufacturing and process plants across the full spectrum of industry have advanced exponentially to meet the growing and evolving demands of the markets they serve. In turn, the design and function of the laboratories which support these plants have had to respond to these changes. Accordingly, the gases and gas technologies used in testing, analysis and measurement, and the associated gas equipment and supply systems have advanced in quantum leaps.

Pure gases today come in various purities from 'industrial' or 'technical' grades to several high purity 'specialty gases' grades. Within the pure specialty grades, purity can vary from the 4.6 grade – being 99.996% pure – to the 7.0 grade at 99.99999% purity. Higher purity means fewer and lower levels of the impurities that cause problems with instrumentation and analytical measurement.

High purity gases keep instruments running at optimum levels. These include helium, hydrogen, argon and nitrogen as 'carrier' gases and 'purge' gases for gas and liquid chromatography and spectrometry. These are required for sample measurement in the gas, liquid or solid phase. High purity gases, such as nitrogen, and gas mixtures are also used for instrument span calibration and to set the zero reading: they are especially required for sample measurement in the gas phase.

Quality is therefore a critical requirement in the supply of pure gas. Pure gas cylinders are provided on the basis of supplier quality assurance, or with a certificate to validate the cylinder contents. Increasingly, quality conscious customers and stringent industrial quality systems are demanding individual certification of analysis for each cylinder supplied.

Gas mixtures also involve a range of quality levels and require different types of certificate. The most basic mixtures — such as welding gas mixtures — are filled using mass production techniques to keep cost and price low, while specialty gas mixtures are filled in more controlled environments. Mixtures for process applications, such as food packaging or laser gas mixtures, are often supplied based on the manufacturers' quality systems. However, calibration gas mixtures always require a certificate of analysis so that the reported values can be used by the customer to precisely calibrate their instrument.

Various certificate types exist in the quest to achieve increasing levels of accuracy, traceability and accreditation. The most sophisticated of these are validated by independent organisations such as NIM (China), NATA (Australia), UKAS (UK) or DAP (Germany) to international standards such as ISO17025 or ISO Guide 34.

The characteristics of gases used to calibrate instruments have also made huge strides. Today's specialty gases for calibrating and measuring gases and for measuring liquids and solids are on

the cutting edge of technology.

In a significant leap forward in the supply of calibration gases, Linde Gases – including their UK business, BOC – last year launched its groundbreaking HiQ 60 calibration gases, a range of gases, including pure gases and gas mixtures, with an extended five year shelf life and flat-line guarantee, allowing for greater reliability in the accuracy of instrument measurement and longer term usability of gas.

“Previously, gas suppliers across the market offered product expiration guarantees generally limited to 36 months, with many products available with only 12 or 24 months of shelf life,” says Steve Harrison, head of specialty gases and specialty equipment. “Gas products with a more limited shelf life can impact measurement accuracy, as gas stability in terms of consistency and quality can change over time. Where consistency or purity of the gas has been compromised, this can result in expensive system re-calibration procedures, additional cylinder changeovers and wasted human resource time.

“The most important requirement of any gas used for calibration purposes is that it can accurately and repeatedly report values of the instrument being measured. HiQ 60 gas products alleviate concerns associated with quality or reliability issues for a period of up to five years and give customers peace of mind.”

Sound analytical protocols are at the heart of work taking place at laboratories

every day and in environmental analysis and testing, a range of sophisticated instruments and next generation gas chromatography and mass spectrometry techniques play a vital role in the identification and qualification of environmental pollutants. Both techniques and equipment require high quality specialty gases for instrument operation and/or calibration, in addition to dedicated high purity gas distribution systems.



*Gases and gas supply systems used in testing, analysis and measurement have advanced in quantum leaps*

Since the reliability of analysis is only as good as the quality of gas being used, distribution systems and equipment for high-purity and specialty gas mixtures must be able to meet increasing demands for high standards of performance and new analysing methods. Impurities occurring in as low concentrations as parts per billion (ppb) can have serious consequences.

Complementing its HiQ solutions, Linde Gases' REDLINE gas supply equipment for high-purity gases and specialty gases is a high-tech, precision designed range of products, modularly designed to slot into central gas supply systems, containing gas panels, valves, points-of-use and cylinder regulators suitable for purities up to 6.0 (99,9999%). Some of the most specialised regulators also have the capability for vacuum dosage and high precision low pressure adjustments. For corrosive mixtures there are models available with the relevant purge assemblies. Additionally, Linde's BASELINE gas equipment range can provide customers with an entry level range of specialty gases cylinder regulators which are appropriate for more flexible systems or short term project work where requirements change from time to time – for example, in universities and R&D labs.

“The flow and pressure required at the work place should be constant; regardless of cylinder or system pressure and regardless of the flow,” Harrison says. “A good regulator should give a constant flow and maintain the pressure that is set on the secondary side where the gas is

delivered to the application. We've achieved this with the REDLINE product range."

To maintain the quality of gas during cylinder changeover or in supply lines to ensure that samples are not contaminated en route to the lab, a system purge is an essential procedure. Purge assemblies allow the introduction of a purge gas into the system after cylinder changeover. Even with standard carrier gases, a system purge is recommended to remove the potential air and moisture contamination that can occur. The most popular purge assemblies available are referred to as Inlet Tee Purge, Outlet Tee Purge, or Cross Purge.

As part of its ongoing commitment to innovation, providing sustainable, safe and environmentally friendly products and solutions, Linde also recently introduced a new range of welded panels as part of its REDLINE gas supply equipment range. The panels, which combine face seal connections with advanced welding techniques, mitigate potential for gas leakage compared with more traditional panels which use standard threaded connections. The welded panels are used to enhance gas supply safety of corrosive or toxic substances such as chlorine, ammonia, hydrogen chloride and hydrogen sulphide.

Over a period of time, standard threaded connection panels are subject to minor vibration which can result in potential hazardous gas leakage. The use of welded connections creates a permanent connection, while the use of face seal fittings ensure a leak-tight service from vacuum to positive pressure at the gas supply and distribution connection.

"Distribution systems for high-purity gases and specialty gases must meet increasing demands for high standards of performance, new analysing methods and production refinements," says Harrison. "Impurities occurring in just a few ppm or ppb can have serious consequences. The demands made on regulators and valves in these environments are extremely high. Components must be capable of dealing with high and low pressures, large and small flows. They must be suitable for high-purity inert gases as well as reactive, flammable, corrosive or toxic gases."

Gas detection systems are critical in a lab setting to protect personnel from the consequences of a gas leak. This can be achieved by installing a fixed detection system in the form of sensors, which are suitable for production laboratories where the types of gas being used are predictable from year to year. The alternative is to utilise detection instruments carried by each person in the lab such as the Linde G-TECTA range, which sound an alert when gas is detected in the laboratory environment. This option is very flexible and therefore suitable for use in R&D laboratories where the gases being handled can change from month to month.

Many of these specialty gases can be hazardous — toxic, flammable and stored at high pressure. Legislation is rapidly tightening to boost the safety of gas handling and usage to entirely new levels. This legislation impacts on product registration, classification and labelling, packaging and transportation, storage, product information and product disposal.

Two of the biggest regulatory developments driving legislative change are REACH and the GHS. REACH is a European Community Regulation on chemicals and their safe use. In the process of being implemented, REACH deals with the Registration, Evaluation, Authorisation and Restriction of Chemical substances. The purpose of this regulation is to ensure a high level of protection for human health and the environment. This includes the promotion of alternative methods for assessment of hazards of chemicals, as well as the free movement of substances on the market of the European Union. REACH makes industry responsible for assessing and managing the risks posed by chemicals and providing appropriate safety information to their users. Substances in volumes over one ton per year that are either manufactured or imported into the EU (even in

preparations/mixtures and articles), now have to be registered.

The Globally Harmonised System of Classification and Labelling of Chemicals (GHS), addresses the classification of chemicals by types of hazard and proposes harmonised hazard communication elements, including labels and safety data sheets. It aims at ensuring that information on physical hazards and toxicity from chemicals be made available to enhance the protection of human health and the environment during the handling, transport and use of these chemicals. The GHS also provides a basis for harmonisation of rules and regulations of chemicals at national, regional and worldwide level, an important factor also for trade facilitation. \*\*

The global implementation of GHS will affect over sixty countries and will directly impact all pure gases in the EU countries from December 1, 2010, with the re-classification of gas mixtures being implemented from 2015.

Yet another critical piece of the gas safety jigsaw is the quality standard ISO 10156 which impacts on how flammable and oxidising gases are defined, labelled and where and how they should be stored.

While most countries have systems for the classification of hazardous chemicals – including gases – to ensure safe transportation, storage, use and disposal, to date various national or regional systems have not always been compatible. This incompatibility has often meant the re-labelling, or the use of multiple labels, on a product, increasing the risks involved in handling gases. For organisations operating at an international level, the need to comply with multiple regulations on hazard classification and labelling has meant that end users may perceive inconsistent label warnings, therefore increasing the risks involved in handling a particular gas, in addition to the added time or barriers companies may face to align their classification and labelling with local requirements

“Regardless of the extent of gas usage in a lab or industrial setting, teaching personnel about the properties and potential risks of different gases will improve safety,” comments Harrison.

“Preventive maintenance is another critical component of safety when it comes to gas handling. We recommend that routine inspections are carried out by our trained professionals to ensure that pressurised gas systems are always in compliance with local regulations and fully optimised to operate at their peak performance. Any deviations from regulations, or early warning signs such as leaks or breakdowns, should be addressed immediately.”

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