

Reactive and Inert Gas Analysis



Advanced Capabilities for Impurity Range from ppt to %

Pure gases and gas mixtures are employed throughout industrial manufacturing processes, as primary reagents to be incorporated into a device, as well as for

- Chemical production
- Chamber cleaning
- Dilution and chemical transport
- Inert atmospheres set up

These gases span a wide range of reactivity, from inert gases (helium, argon, compressed air, sulfur hexafluoride), to air-sensitive organometallics, to highly reactive etching and chamber-cleaning molecules. Industrial gases are manufactured both off-site and on-site (e.g. air separation plants), and are delivered through a variety of containers (pipelines, tube-trailers, cylinders, and specialized canisters). Industrial gases can be packaged as single-phase, ‘compressed gases’, or as two-phase, ‘liquefied gases’. In the case of semiconductor manufacturing, cleanroom air itself can be viewed as a critical purified gas.

Industrial users of gases go to great lengths to maintain gas purity, from the source container to the point of use. Gas analysis is used to verify gas purity at any point in this chain.

Balazs NanoAnalysis tackles challenging gas analysis problems by taking advantage of our unique combination of reactive gas handling knowledge, ultra-pure chemicals, and world-class analytical facilities. From straightforward compressed gas analysis, to air sampling, to customized wet-chemical digestion schemes, Balazs focuses the resources of the world’s largest industrial gas company on **your problems**.

Typical Gases Analyzed	Impurities	Methods
Inert (N ₂ , O ₂ , H ₂ , CDA)	Air components, others	GC-MS, GC-DID
Rare (He, Ne, Ar, Kr, Xe)	Air components, rare gases	GC-MS
Reactive (chlorosilanes, HCl, NH ₃)	Trace metals, air components, others	FTIR, other proprietary methods
Advanced precursors (organosilicons, organometallics)	Assays, impurity surveys	GC-MS
Halocarbons (freons)	Pure-gas and gas mixture re-certifications, air components, others	GC-MS, GC-DID
Cleanroom air	Most volatile contaminants, sulfur compounds	FTIR, trace sulfur monitor, other methods

Bulk Industrial Gases



Fig 1. Balazs gas sampling cylinder

Inert 'bulk' gases comprise N_2 , O_2 , H_2 , Ar, He, CO_2 , and clean dry air (CDA). These gases are consumed in large volumes for purging, inerting, plasma generation, and chemical transport. Critical impurities in bulk gases (see, for example, ITRS Roadmap, 2008 Update, Table YE9) include moisture, O_2 , N_2 , CO, CO_2 , and hydrocarbons. Gas analysis can be done on-line to quantify impurities at pptV to low-ppbV concentrations, or off-line, utilizing 'grab sampling' methods, to achieve low-ppbV reporting limits. Metal impurities in bulk gases exist in the form of non-volatile particles, which are efficiently removed by current particle filtration technology. Balazs focuses on off-line analysis of bulk gases via portable gas cylinders (see Fig. 1), and a broad array of analytical tools, including gas chromatography mass spectrometry (GC-MS) and Fourier-transform infrared spectroscopy (FTIR). This approach allows bulk gases at any location to be rapidly and cost-effectively sampled and analyzed using state-of-the-art laboratory equipment.

Reactive Gases

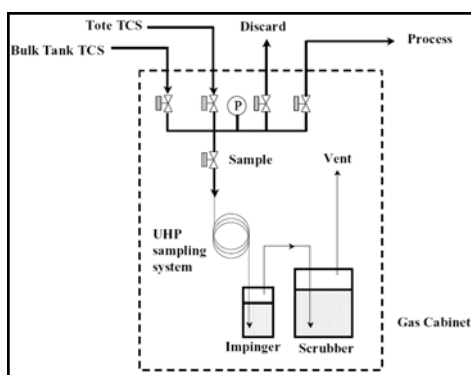


Fig 2. Gas-sampling scheme for an on-site trichlorosilane bulk-tank qualification

Reactive gases include compressed and liquefied gases used for chemical vapor deposition (CVD), such as SiH_4 and NF_3 , chamber-cleaning agents (perfluorinated hydrocarbons, fluorine, ClF_3), highly toxic doping gases (AsH_3 and PH_3), as well as customized, organometallic precursors. Reactive gases are supplied both in pure form and as precision mixtures. Critical impurities in reactive gases include trace metals, moisture, air components, and impurities carried over from the manufacturing process. The diversity of reactive gas properties presents sampling and analysis challenges not encountered in bulk gas analysis. Balazs has engineered a variety of methods to identify and quantify impurities in reactive gases. A principal focus has been quantification of metallic impurities in both the gas and liquid phases of reactive gases. Our sampling methods (see Fig. 2) have been applied for on-site sampling of chlorosilanes, HCl, and carbon monoxide, as well as in the laboratory for NH_3 , HF, organometallics, organoamines, and others. Reporting limits for metal impurities in reactive gases range from 10 to 100 pptW for 30-40 elements. Balazs' laboratory in Dallas, TX routinely devises sampling and analysis schemes for challenging gases.

Clean Dry Air (CDA and XCDA)

Compressed air is used extensively in semiconductor and other industrial settings. CDA is typically dehumidified, filtered, and scrubbed to remove hydrocarbons. It is used primarily to drive pneumatics but also for breathing air in self-contained and airline-supplied systems. A specially-purified, 'XCDA', grade of compressed air is used to purge expensive optics in advanced photolithography systems. CDA impurity analysis is done either on-line, or off-line using the gas sample cylinders described above. XCDA-grade compressed air typically specifies acids, bases, organics, and refractory impurities at low-pptV levels. Consequently, advanced sampling methods are necessary to quantify impurities at the prescribed detection limits.

Stepper Gases

High-purity nitrogen, helium, compressed air, and other gas mixtures are required for purging and cooling the optics in advanced photolithography applications. Such optics are sensitive to minute concentrations of airborne molecular contaminants (AMC) and are expensive to repair or replace. Balazs has been certified by ASML to certify stepper gases meet ASML's stringent specifications. In addition, Balazs offers laboratory-based analytical services for stepper gas delivery modules and purifiers.