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TECHNOLOGY & OPERATIONS

GAS ON DEMAND

MAKING A CASE FOR IN-HOUSE GENERATION OF CARRIER GAS FOR TOC ANALYSES by Jack Mahan and Peter Froehlich

Total organic carbon (TOC) analyzers are commonly used in applications including the determination of organic matter in water in municipal water supplies and sewage facilities, the monitoring of water used in semiconductor manufacturing and nuclear power plants, and the clean-in-place procedures used in pharmaceutical manufacturing.

TOC analysis includes three discrete steps:

1. Acidification of the sample to remove inorganic carbonaceous material and purgeable organic carbon (e.g., methane)
2. Oxidation of the organic matter in the sample (typically via persulfate in a heated quartz tube) into CO_2
3. Detection of the CO_2 (typically by non-dispersive IR)

"A TOC analyzer can be coupled with a nitrogen analyzer… so that the level of both elements can be determined."

High-purity air or N_2 is used to drive the CO_2 from the oxidation process to the detector, and its purity is a critical issue in the optimization of the sensitivity and operating range of the system. The gas must be free of CO, CO_2 and hydrocarbons (e.g., compressor oils), and is typically supplied to the analyzer at a pressure of 80 to 100 psig and at a flow rate of 400 to 800 mL/min to provide a broad operating range; as an example, organic carbon can be detected over the range from 4 to 25,000 mg/L using the Shimadzu TOC-V_{CSH} Analyzer (Shimadzu Corporation, Tokyo, Japan) shown in Figure 1. In addition to analysis of organic carbon in water, a TOC analyzer can be coupled with a nitrogen analyzer (which converts organic nitrogenous compounds to NO followed by measurement of the chemiluminescence of the NO) so that the level of both elements can be determined.

Figure 1. Shimadzu TOC-V_{CSH} TOC analyzer

Although carrier gas for TOC analysis can be provided by a cylinder obtained from an external source, many laboratories employ an in-house generator to supply the gas. In this article, we will describe how carrier gas

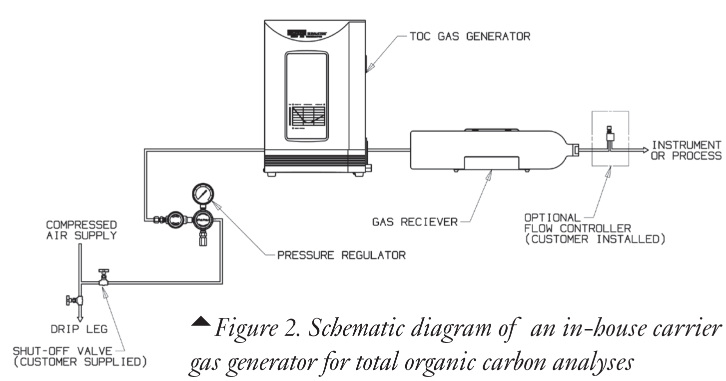

Figure 2. Schematic diagram of an in-house carrier gas generator for total organic carbon analyses

for TOC analysis can be generated in-house from laboratory air and show that this is a safer, more convenient and less expensive approach than the use of a cylinder.

Design of an in-house gas generator for TOC analysis

The initial steps in generating TOC carrier gas involve filtration of the compressed air and oxidization of hydrocarbons. The compressed air is then passed into a pressure swing adsorption (PSA) system to remove water vapor and CO_2, and a final filtration step is employed to remove all particulate matter >0.01 micron. An overall schematic diagram of a typical in-house generator designed to generate high-purity gas for TOC is shown in Figure 2.

The heart of an in-house gas generator for TOC analyses is the PSA system. This system involves pressurizing air and passing it into a chamber that contains molecular sieves that are specially designed to retain the gases that are not desired while allowing the product air to be passed into a storage tank for use with the TOC analyzer. The undesired gases are purged from the molecular sieves on a periodic basis by heating the sieves and reducing the pressure.

A molecular sieve such as activated carbon (charcoal) is used in the PSA system because it has a very large surface area available for adsorption, is extremely porous and can retain a significant amount of the undesired gases before it must be purged. Due to its high degree of micro porosity, 1 g of activated carbon may have a surface area in excess of 500 m_2 (which is equivalent to the area of about two tennis courts), as determined by nitrogen gas adsorption experiments.

The carrier gas supplied by an in-house generator contains extremely low levels of CO_2 and provides superb sensitivity with a TOC analyzer. As an example, the composition of the gas generated by the Parker TOC-1250 TOC gas generator (Figure 3) is presented in Table 1. Although the gas contains approximately 1 percent Ar (Ar is not retained by the molecular sieve), this is not a problem for TOC analysis since Ar is

Gas	Composition
Nitrogen	99.9999%
CO	<1 ppm
CO2	<1 ppm
O2	<1 ppm
H2O	<1 ppm (Dew Point <-100oF [-73oC])
Hydrocarbon (as methane)	<0.1 ppm
Argon	0.9%

Table 1. Composition of nitrogen provided by a typical in-house generator

Figure 3. Parker Balston TOC-1250 TOC gas generator

not detected at the wavelength used to monitor the CO_2 that is generated by the oxidization of the organic compounds. If the compressed air supply contains halogenated hydrocarbons, a scrubber should be installed upstream from the generator, as halogenated hydrocarbons will render the molecular sieves inactive.

The TOC-1250 TOC gas generator shown in Figure 3 can generate gas at flow rates as high as 1,200 mL/min (at an inlet pressure of 150 psig). The total hydrocarbon and CO_2 concentration of the gas from this generator is extremely low and provides an extremely stable baseline for extended periods of time (Figure 4).

Benefits of in-house generation of TOC-grade gas

Safety considerations

In-house generation of TOC-grade gas readily provides the required volume of sufficiently pure gas for superb sensitivity with a TOC analyzer. The gas is available on demand and is present at a lower pressure than the gas from a cylinder, increasing laboratory safety.

A cylinder contains a considerable amount of gas at high pressure; if a leak occurs (e.g., if the valve is compromised), a large quantity of N_2 would be released into the laboratory and would displace the air, leading to the potential of asphyxiation. Since an in-house gas generator typically has a maximum output of 600 to 1,200 mL/min at a pressure of approximately 100 psig, the volume of gas that could escape in the laboratory due to a leak in the system is very small and presents a minimal hazard.

An additional safety concern with the use of cylinder gas involves potential hazards inherent in transporting it from the storage location to the instrument. As an example, if the individual moving the cylinder loses control of it during transport and the valve is damaged, the cylinder can become a guided missile. A typical user, Dr. Michael Lockney, a chemist at Momentive Performance Materials (Sistersville, WV) who uses a TOC analyzer to monitor plant waste water, indicated that they obtained an in-house TOC gas generator to minimize the safety issues and eliminate the concerns involving the handling of gas tanks.

In-house generators include a variety of safety features to minimize the possibility of injury to personnel and facility damage. As an example, if an overpressure or a pressure loss is observed, gas production is immediately terminated and a diagnostic message is generated. If desired, an audible alarm and/or a signal can be sent to an external controller or to the operator. In addition, these systems meet the requirements of a broad range of safety standards, including NFPA and OSHA

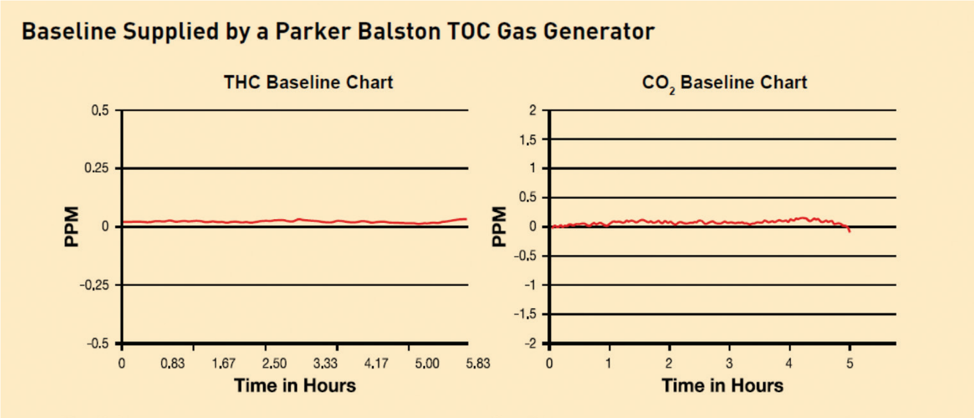

Figure 4. Baselines observed on a TOC analyzer from carrier gas from a Parker Balston generator. Left: total hydrocarbons; right: CO_2

(1910.103), and of regulatory agencies such as the IEC, CSA, UL, cUL and CE.

"The heart of an in-house gas generator for TOC analyses is a pressure swing adsorption (PSA) system."

Convenience

When an in-house gas generator is employed, the TOC-grade gas is readily available on a continuous (24/7) basis or can be generated as required with a short system warm-up. In contrast, when a cylinder is employed, the operator must make certain that the cylinder contains a sufficient amount of gas for the desired operation. In many facilities, replacement cylinders are frequently stored in a remote (outdoor) location for safety reasons, and specially qualified personnel may be required to perform cylinder replacement (replacing a tank is inconvenient in inclement weather). When bottled gas is employed, it is necessary to maintain a supply of spare cylinders and order/return cylinders on a periodic basis.

In contrast, when an in-house generator is employed, very little maintenance is required. Once the system is set up, gas is readily available for an extended period of time with no effort on the part of the analyst. As an example of this point, Dr. Charles Weatherford, the QA supervisor at Metrex Research (Romulus, MI), a division of Sybron Dental Specialties, Inc., reports that their TOC air system has been in use for over eight years with only a minimum of annual maintenance. Similarly, Dr. Lockney indicates that the system at Momentive Performance Materials has been in use for three years with no service issues, and it is not necessary for them to monitor the usage of the generator.

Elimination of contamination

When a cylinder is used to deliver TOC gas, the connection between the source of the gas and the TOC analyzer must be broken when a cylinder is replaced. This can lead to the introduction of contaminants such as water vapor, O_2, CO_2 and other materials that may be present in the laboratory atmosphere into the system. These may have a deleterious effect on the TOC measurement. In contrast, when an in-house generator is employed, a permanent direct connection is made between the generator and the TOC system, thereby practically eliminating the possibility of contamination.

Cost

The overall cost of operation of an in-house generator is considerably lower than the cost of cylinders to provide gas. The only costs for an in-house generator are for electricity and periodic service. The power consumption of the TOC-1250 system is 2 A, so if the generator is used for a 40-hour cycle on a 52-week basis, approximately 500 kWh would be used. At 10c/kWh, the annual operating cost would be approximately $50 and the cost of maintenance and replacement of an in-house generator would be about $500/yr. While the payback period of the generator clearly depends on the amount of gas that is consumed and the local cost of the gas cylinders, the generator can pay for itself in a year in many facilities. When cylinders are used to supply the gas, many other expenses must be considered, including the time cost of ordering the gas and bottle demurrage. These costs are not relevant when an in-house generator is used.

"In-house generators include a variety of safety features to minimize the possibility of injury to personnel and facility damage."

Conclusion

In-house generation of carrier gas for total organic carbon analyses provides the lab with a number of significant benefits compared to the use of cylinders. An in-house generator is safer, as it produces the necessary gas on demand and eliminates the need to handle high-pressure tanks. An in-house generator can provide gas on a 24/7 basis with essentially no maintenance and has an operating cost that is significantly less than the cost of obtaining pressurized cylinders. Since an in-house generator eliminates the requirement for transporting heavy cylinders from the production facility to the point of use, significant environmental benefits are obtained.

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