ATONARP
AMS-1000

High-Pressure Mass Spectrometers Offer Robust, Real Time, In-Situ Analysis

Application Note

Introduction

Industrial process control is facing one of its greatest challenges. Billions of dollars of pharmaceuticals and advanced industrial processes will be manufactured where fast, accurate, molecular analysis is critical to the quality of the end-product. There's a pressing need for in-situ molecular analysis that delivers real-time results that are accurate, repeatable, and quantitative from a small, rugged, and easy-to-integrate solution. However, existing molecular analysis solutions such as residual gas analysis (RGA), gas chromatography mass spectrometry (GC--MS), and optical solutions like near infrared (NIR) and optical emission spectroscopy (OES) have limitations that make them impractical solutions.

Current solutions for molecular gas analysis applications have significant shortcomings.

- Mass spectrometry and residual gas analysis solutions have required low pressure vacuum on the order of 1 x10^{-6} Torr to operate. Low pressure operation requires sizable and expensive vacuum pumps and ensuring there are no leaks makes installation and maintenance complex and costly.
- Gas chromatography (GC) has historically been used to isolate mixed gas components for quantitative analysis, but this process is slow, has significant consumable running costs, and a high maintenance burden.
- Fourier transform infrared (FTIR) optical spectroscopy techniques can detect the presence of specific molecules, but getting quantitative data is very difficult, rendering them less useful in process control applications.

So how about ruggedized, high pressure mass spectrometry? To be a useful process control tool, it must be process control production-ready. The basic production requirements are: to be fast and accurate, small and easy to install, field-serviceable, robust, deliver stable and repeatable results, support easy software and system integration, and be low maintenance.

Atonarp AMS-1000 is a molecular sensor (quadrupole mass spec) solution that delivers on these requirements with: as many as 100 samples per second operation; sensitivity down to part per million (ppm) concentrations; small (20.4 x 20.0 x 25.6 cm) form factor; integrated vacuum pumps; and a rugged Faraday cup sensor that delivers high stability and infinite lifetime.

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Robust | Reliable | Repeatable

Accurate | Sensitive | Cloud-Ready

AMS-1000 Key Hardware Features

- 1 amu (FWHM) Resolution
- 100 amu Range
- Real-time Data from 10mS/amu
- Quantitative mol% and Pressure Data
- High 5x10^{-3} Torr Operating Pressure
- Integrated Roughing and Turbo Pumps
- <5min Start-up Time Out-of-the-box
- Ultra-small <0.01m^3 Form Factor
- Field-Replaceable Sensor and Filament
- Internet-of-Things (IoT) Ethernet Port
- Minimum Partial Pressure as Low as 1x10^{-9} Torr for N_2
- 1ppm Sensitivity for N_2 at 1x10^{-3} Torr
- <100W Power at 24V DC
- >6 Orders of Magnitude Linear Range

At the heart of AMS-1000 is the miniature molecular sensor. It is about 5 cc and is mounted on a 1.33in. conflat flange. It features an array of nine quadrupole mass filters operating in parallel. A single electron impact ion source supplies ions to the individual quadrupoles to be filtered and collected on individual Faraday cups connected together. Driven at 11MHz radiofrequency (RF), the sensor is able to filter ions on the basis of mass to charge ratio with unit mass resolution from ultra-high vacuum (UHV) to pressures as high as several mTorr. Making the quadrupole small is offset by increasing the control electronics RF. The combination provides real-time, quantitative molecular analysis and virtually eliminates memory effects while offering increased sensitivity by allowing the system to work at higher pressures. Intuitive web browser software, accessible via an Ethernet connection and industry standard data file support (.json), make software integration, process data logging, and setup easy. To ease hardware integration, industrial analog and digital I/O are available, and can be used to control (and get input from) standard industrial sensors, actuators, or PLCs.

The ability to do real-time, quantitative molecular analysis in-situ is an enabling capability for many industrial processes. Examples are given below for the pharmaceutical and industrial manufacturing markets demonstrating how a small, rugged molecular sensor (mass spectrometry) is enabling disruptive process control techniques.

Mass Spectrometry for Pharmaceuticals

In aseptic pharmaceutical manufacturing, a common and critical solution for preservation and transportation of pharmaceutical product is to use lyophilization (freeze-drying) to stabilize and preserve the pharmaceutical until needed at point of care. The success of this process is dominated by two critical activities: accurate detection of the secondary drying endpoint and ensuring the purity of the product by detecting contamination.

Historically, a Pirani gauge was used to detect the second drying endpoint, but the lack of accuracy and dynamic range of the Pirani gauge can cause residual water to remain in the pharmaceutical products, resulting in spoilage. Conservatively long secondary drying times are used to mitigate the limitations of the Pirani gauge solution. However, these result in inefficiencies plus the increased possibility of contamination. Pirani gauge solutions don't detect possible contamination, and historically, post-processing, lot-based sampling would be needed to ensure pharmaceutical product quality.

Using a small, rugged molecular sensor significantly improves the lyophilization process by rapidly and accurately detecting the secondary drying end point. Additionally, with appropriate input filtering, molecular sensor solutions allow detection of minute levels of the silicone oil used for temperature management in the fabric of the lyophilization chamber, which allows mitigation of pharmaceutical product contamination.

AMS-1000 is part of the LyoSentinel™ lyophilization solution.

The Atonarp AMS system features a dual path sampling frontend interface to meet the two objectives. An inlet valve enables automatic switching between two paths with an asymmetric duty cycle. One low duty cycle sampled path is for an unfiltered sample, which is used to detect silicone oil presence. The other, a high duty cycle sampled path, is for a filtered sample for endpoint detection and has a silicon oil filter to minimize contamination of the molecular sensor from silicone oil leaks. Such a scheme protects the molecular sensor from accumulation of oil inside the vacuum chamber, as most of the time is spent on monitoring moisture and other process gases, such as N_2.

Measurements made during the freeze drying process can be stored in the cloud (on public or enterprise networks), where additional analysis can be performed to provide process insights and reporting, plus predictions and recommendations for possible process optimization. The cloud-based software tool chain enables the intelligent Internet of Things (IIoT). By moving the data to the cloud, a wealth of existing machine learning and artificial intelligence tools can efficiently uncover process optimizations. With a molecular sensor, the data being analyzed now is rich, quantitative, real-time molecular data—not simply retrospective temperature, pressure, and machine status information. The wealth and detail of collected data provides a

path of continuous improvement, possibly further maximizing product quality, process efficiency, yield, and profitability.

An Alternative to Gas Chromatography for Natural Gas Management

Raw natural gas must go through many stages or purification and blending before it can be used in residential and industrial applications.

Different types of contaminants need to be removed including:

- Gases such as helium, hydrogen sulfide, carbon dioxide, nitrogen, oxygen, and water vapor
- Liquid condensate and crude oil
- Trace elements such as mercury and radon

After contaminants are removed, gas is sold based on the energy content (not its volume), which is assessed based on the mix of $C_xH_{(2x+2)}$ components on the gas blends. A significant amount of testing is required to meet various standards (GPA 2145, 2172, 2177, 2186, 2261, 2286, TP-17, ASTM D1945, D1946 and D3588, ISO 6974 and 6976, Din51872-5 and UOP53) and contracts.

Real-time, accurate, and quantified molecular sensor solutions based on AMS-1000 and Aston™ from Atonarp are cost-effective alternatives to gas chromatography solutions.

AMS-1000 Front Panel

Evolving Industrial Process Control

Increasingly, industrial process control applications like hot rolling or continuous casting metals, specialty coatings, low pressure and atmospheric plasma etching, and process gas blending/mass flow are optimized with the use of molecular sensor technology. Additionally, air quality environmental compliance using molecular sensors to quantify byproduct and exhaust gases provide real-time, sensitivity, stability, and selectivity.

ASTON Metrology Platform

Atonarp's molecular sensor family extends to ASTON™, which has been optimized for semiconductor manufacturing applications where immunity to corrosive gases and ppb level sensitivity are additional requirements.

For more details on Aston visit: http://www.atonarp.com/aston.

Late in the 19th century, J.J. Thompson created the first mass spectrometer. One hundred and twenty years later, mass spectrometry technology has been refined into a robust, process ready, real-time molecular sensor solution. Rugged, fast, and quantitative molecular sensors can now be part of the process architect's arsenal for applications as diverse as:

- Paper Products
- Food and Beverage
- Stone, Clay and Glass
- Oil and Gas, Petroleum Refining
- Primary Metals
- Rubber and Plastics
- Semiconductors (Etch, Abatement & Deposition)
- Lyophilization (Freeze-Drying)
- Fermentation Monitoring and Control
- Thin -Film Vacuum Coating
- Leak Detection
- Environmental Monitoring
- Safety and Security
- Precision Gas Blending and Mass Flow Control