Using novel laser absorption based analyzers for trace hydrogen sulfide (H\textsubscript{2}S) measurement
Los Gatos Research (LGR)

Measuring trace hydrogen sulfide to determine when to replace spent desulfurizing beds

Measurement made easy

Introduction

Hydrogen-based fuel cells offer a highly attractive source of electrical energy for commercial and military applications. They generate electricity at efficiencies that approach 60%.

Additionally they have zero emissions. Reforming processes for hydrocarbon-based fuels can produce hydrogen for use in fuel cells to generate electric power. But high sulfur content in fuels will poison the reformer catalysts and cell anodes – see Fig. 1, page 2.

To overcome this challenge, certain sulfur-tolerant reformers can convert the fuel sulfur into H\textsubscript{2}S. A desulfurizing bed can then remove the H\textsubscript{2}S prior to the fuel cell.

For more information

Further details of the ABB Analytical products are available for free download from www.abb.com and www.LGRinc.com or by scanning this code:
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The sulfur removal process

The sulfur removal process to absorb H$_2$S has a finite life before it must be replaced. Optimizing and validating the desulfurization process requires a gas-phase analyzer that can to accurately and quickly measure H$_2$S at low concentrations (<1 ppm).

The analysis must be performed in the presence of other reforming gases, such as hydrogen, carbon monoxide, carbon dioxide, nitrogen, various light hydrocarbons, and water vapor.

Conventional sulfur sensors generally don’t work well for this application. Optical sensors based on UV absorption suffer interference from high levels of CO$_2$ in reformed fuels. Solid state sensors based on doped semiconductor films have similar problems with the water vapor and carbon monoxide, both of which are present in high concentrations in reformed fuels. In addition, their non-linear response adversely affects accuracy, limiting their effectiveness to a small dynamic range.

ABB solution

Recent studies conducted by Precision Combustion, Inc. and Los Gatos Research have proven the effectiveness of near-infrared cavity-enhanced optical absorption spectroscopy (off-axis ICOS) to quantify H$_2$S in a reformer stream of hydrocarbon-based fuels. For details, please see F. Dong, C. Junaedi, S. Roychoudhury, and M. Gupta, ‘Rapid, Online Quantification of H$_2$S in JP-8 Fuel Reformate Using Near-Infrared Cavity-Enhanced Laser Absorption Spectroscopy,’ Analytical Chemistry 83 (2011) 4132.

The analyzer, made by LGR, a member of the ABB Group, provides precise H$_2$S mole fraction measurements down to 0.1 ppm or better with a response time of two seconds. The measurement experiences minimal cross-interferences with the other reformate gases present. In fact, the analyzer can, in addition quantify the amounts of carbon dioxide, methane, ethylene, and water vapor present.

Fig. 1: Sulfur must be removed from hydrogen-based fuels to avoid poisoning the fuel cell’s anodes

Fig. 2: Schematic diagram of LGR’s patented Off-Axis Integrated Cavity Output Spectroscopy (OA-ICOS) technique for measurements of trace H$_2$S in complex multi-gas flows
OA-ICOS analyzers from LGR monitor trace gas concentrations by means of a sensitive optical absorption measurement. The technique uses an integrated tunable laser that delivers a detailed scan of an extended spectral region and an optical cavity as a measurement cell to provide an enhanced beam path length for extremely high sensitivity and selectivity.

Off-axis ICOS has many advantages over conventional Cavity Ringdown Spectroscopy (CRDS) techniques. LGR's OA-ICOS analyzers are insensitive to optical alignment, have a much shorter measurement time, and do not require expensive and power-consuming auxiliary components. Most importantly, due to insensitivity to misalignment, these analyzers (including the mirrors) can be serviced by anyone anywhere in minutes.

Reformed fuels usually contain many compounds that can all absorb in the 1.6 µm wavelength region that provides optimum sensitivity for measuring H₂S. Because OA-ICOS instruments produce a fully-resolved and extended spectrum, well-known chemometric fitting techniques can be applied to derive accurate H₂S concentrations.

Chemometric software packages are widely used and available for spectroscopy. Applied to measuring H₂S, the technique has several steps: First the instrument records data spanning the full extent of expected conditions. In this case, for example, it measures multiple gases having known different H₂S trace concentrations along with known concentrations of methane, ethylene, and carbon monoxide concentrations. The analyzer uses the resulting algorithm to convert measured spectra into absolute concentrations of all these gases simultaneously.

The resulting sensitivity and accuracy allows the reliable determination of when a desulfurizing bed has reached its limiting life.

Results
The studies demonstrate the capability of the LGR-patented OA-ICOS spectrometer to record raw data from reformed hydrocarbon-based streams, combine it with standard chemometric analysis methods, and derive trace gas measurements of H₂S continuously and in real time.

The measurements have required accuracy and speed to determine the need to regenerate or replace the desulfurizing bed. In addition, this analyzer may be applied for measurements in other complex flows which require sensitive gas measurements continuously.
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