

### MEASUREMENT OF SULFUR IN REFINERY FLARE GAS STREAMS

## Tunable diode laser absorption spectroscopy (TDLAS) based analysis for compliance with the United States EPA Subpart J(A) regulatory requirement

US EPA regulation NSPS Subpart J(A) has placed an extra investment and operational burden on petroleum refineries to meet the regulatory requirement. AMETEK Process Instruments offers an economical solution to meet the challenge of the measurement of high-range and low-range hydrogen sulfide ( $H_2S$ ) in refinery flare gas streams, with an emphasis on lower capital and operating cost along with improved reliability.

#### The EPA regulation summary

- Routinely combusted flare gases cannot exceed 162 parts per million (ppm)  $H_2S$  on a three-hour rolling average
- Refineries shall continuously measure flare gas lines and record  $H_2S$  on a low-range scale of 0-300 ppm
- Total reduced sulfur (TRS), which is defined as  $H_2S$  + carbonyl sulfide (COS) + carbon disulfide ( $CS_2$ ), shall be continuously measured at a high range with a minimum scale of 0 to 5,000 ppm or 1.3 times the maximum expected concentration of  $H_2S$  (site-specific). In practice the high-range scale tends to be in 0 to 40%  $H_2S$  and higher
- In addition to the  $H_2S$  (low-range) and TRS (high-range) analysis, both flow and British thermal unit (BTU) must be measured. This application bulletin does not address these measurements

### METHODS OF MEASURING THE HIGH-RANGE TRS

The low-range (0 to 300 ppm) is defined as  $H_2S$  only. For the high range the requirement is to measure TRS. The EPA document describes various choices in terms of the high range measurement, to comply with the Subpart J(A) requirement the most practical being:

- 1) Measure just the  $H_2S$  at the high range (not the COS,  $CS_2$ ) and establish a correlation of the ratio of  $CS_2$ , COS to the  $H_2S$  value by grab sample and lab analysis
- 2) Measure total reduced sulfur as  $H_2S$  + COS +  $CS_2$  (using EPA method 15 as a basis)
- 3) Measure total reduced sulfur by oxidizing the sample to sulfur dioxide ( $SO_2$ ) and measure as  $SO_2$  (using EPA ref method 15a as a basis)

The AMETEK solution utilizes the first technique described above; direct measure of  $H_2S$  (only) for the high range utilizing TDLAS. The TDLAS technique is highly specific

to  $H_2S$ , fast-reacting and immune to contamination from high concentrations of  $H_2S$  span gas or high  $H_2S$  event. The reasons for not measuring the COS and  $CS_2$  are the limitation of the spectroscopy to measure COS and  $CS_2$  at the required ranges.

Unlike other technologies, TDLAS is non-contact and resistant to process upsets. It's also virtually maintenance-free. Operational savings over a GC are significant, as shown in Figures 1 and 2. Based on customer input operation savings related to usage of instrument air, carrier gas, calibration gases, electricity, consumable parts and labor can be \$10-20,000 per year. As with any compliance analyzer, reliability and availability is paramount. TDLAS non-contact technology is resilient against common issues that plague other analytical analyzers like contamination of liquids, exposure to process upsets, etc.

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## Limitations with other analytical techniques

- Utilizing a single GC for both the low (H<sub>2</sub>S) and the high-range TRS measurements can be compromised by high-concentration H<sub>2</sub>S daily cylinder gas audits or a high-concentration H<sub>2</sub>S process event
- Utilizing an oxidizing technique, followed by dilution and pulsed fluorescence spectroscopy, has the associated complications of dilution orifices being fouled and introduction of an open flame for the oxidizer
- Both GC and oxidizing (dilution orifice) techniques are subject to damage from the ingress of liquids, which can be a common occurrence due to failure of liquid seals, addition of steam to the flare line, and the presence of entrained liquid hydrocarbon

## Advantages of TDLAS

- TDLAS is a direct, continuous measure and non-contact technique
- The AMETEK 5100HD has a H<sub>2</sub>S reference cell, this ensures line-lock at low concentrations as H<sub>2</sub>S approaches zero. The technique is continuous direct measure – no scrubbing or switching required
- The 5100HD is rated IP65 for installation in a free ventilated building or three-sided shelter, eliminating the need for a temperature-controlled analyzer house and the associated infrastructure in many environments
- An additional safety benefit of installing the analyzer in a free ventilated building or three-sided shelter, is the reduced risk of exposure to H<sub>2</sub>S from the daily (high) concentration H<sub>2</sub>S cylinder gas audits
- Low-maintenance – no carrier gas, no flame, no columns, no dilution, no switching

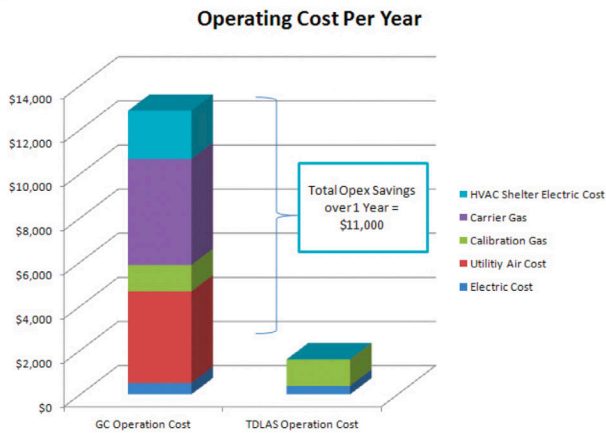


Figure 1. Operation cost of TDLAS versus a process GC

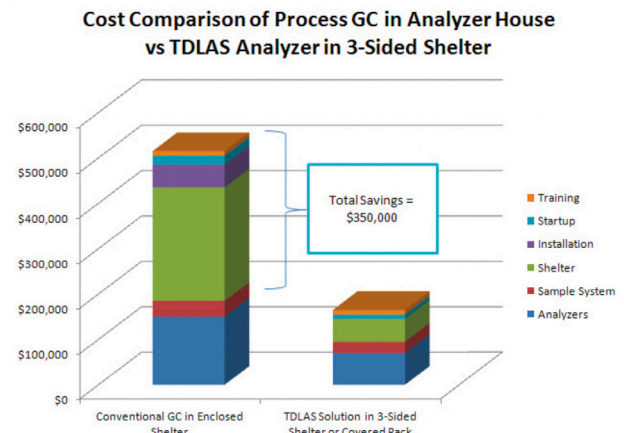


Figure 2. Capital cost of a TDLAS versus a process GC

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