Analysis of Silicon Content in Steel and Sulfidic Corrosion with Thermo Scientific Niton Analyzers

Introduction
Sulfidic corrosion of piping and equipment within the refining industry continues to be a significant cause of leaks and issues that can lead to early replacements, unplanned outages, and incidents potentially resulting in loss of property and injury to workers. Carbon steels with low-silicon (<0.10%) content can corrode at an accelerated rate when exposed to hydrogen-free sulfidation corrosion conditions.

According to the American Petroleum Institute (API) Recommended Practice 939-C (Guidelines for Avoiding Sulfidation Corrosion Failures in Oil Refineries), one-third of high-temperature sulfidic corrosion failures is the result of low silicon content. API RP 939-C is a subcomponent of the larger API RP 578 PMI program – the verification of correct alloy installation in all sulfidation surfaces, both proactive and reactive.

Examples of equipment where hydrogen-free sulfidation occurs include crude/vacuum, fluid catalytic cracker, coker, and visbreaker units. Hydroprocessing and hydrocracking units experience hydrogen-free sulfidation corrosion in their feed and distillation sections.

To help prevent these incidents from occurring, elemental analysis of such piping and equipment with portable x-ray fluorescence (XRF) is an ideal choice. The Thermo Scientific™ Niton™ XL3t GOLDD+ and Niton XL3t Ultra XRF analyzers allows for fast, accurate, and precise analysis of silicon and other elements in the field.

Thermo Scientific Niton XL3t XRF Analyzers
The Niton XL3t GOLDD+ and Niton XL3t Ultra analyzers quickly detect elements from magnesium (Mg) to uranium (U). Both of these instruments are capable of measuring Silicon in low concentrations. The Niton XL3t Ultra, which is equipped with the new large area silicon drift detector, provides highest performance and light element sensitivity for the most demanding applications such as low silicon measurement. These instruments make it fast and straightforward to perform silicon analysis. A quick sample preparation is required because most of the carbon steel piping systems typically have some level of surface corrosion and contamination that will interfere with the analysis. Typically, a small area is treated with a portable grinding tool equipped with an abrasive disk to provide a clean area for analysis. Both of these instruments deliver fast, accurate elemental analysis and alloy-grade identification in demanding conditions.

These analyzers provide the refining industry with the following key benefits:
- Unparalleled accuracy for confidence in results every time
- Light element detection (Mg, Al, Si, P, S) without vacuum or helium (He) purge
- Optional helium purge for enhanced light element performance
- Integrated camera and optional small spot analysis for accurate sample positioning and image capture
- Waterproof, dustproof, and rugged housing for harsh environments
Method
Sixteen certified reference standards and samples were analyzed directly after ensuring the surface was clean and clear of any contaminants that could introduce silicon or other elements to our analysis. Data quality objectives dictate sample preparation requirements and minimum analysis time for these measurements. For this application, the samples were analyzed for 60 seconds using both the main filter (5 seconds) and light filter (55 seconds) after thorough surface preparation.

Results
Figure 1 shows the correlation curve, illustrating certified results vs. the Niton XL3t Ultra analyzer results. The coefficient of determination ($R^2$) for each element is provided in the figures. The $R^2$ value is a measure of how closely the data sets correlate with each other, where a perfect correlation would have an $R^2$ of 1. Figure 2 shows the measurement repeatability data for a single standard with a given value for silicon of 0.10% for Niton XL3t Ultra using a 60 second measurement time. The data in Table 1 demonstrates the improvement in precision between Niton XL3t GOLDD+, Niton XL3t Ultra and Niton XL3t Ultra with optional helium purge.

Conclusion
Results achieved using the Niton XL3t GOLDD+ and Niton XL3t Ultra analyzer demonstrate excellent agreement with the laboratory results. The Niton XL3t Ultra provides the best light element precision and sensitivity for the low silicon measurement. Given appropriate sample preparation and no introduction of contamination, the analyzer is able to reliably detect Si levels in steel at a less than 0.05%.

Helium purge allows for improved detection limits for the both XL3t GOLDD+ and XL3t Ultra in the same amount of time if lower levels of measurement are required. Otherwise, helium purge analysis can be used to achieve the same level of precision as without helium in less time. In this case, the user can reduce total analysis time with helium purge to 25 seconds and achieve the same performance as air path in 60 seconds.

To discuss your particular applications and performance requirements, or to schedule an on-site demonstration to see for yourself how Thermo Scientific portable XRF analyzers can help save you time and money, please contact your local Thermo Scientific portable XRF analyzer representative. You can also email us at niton@thermofisher.com, or visit our website at www.thermoscientific.com/niton.