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APPLICATION NOTE

Analysis of Silicon in Steel to Prevent Sulfidic Corrosion Failures

Using the Niton XL5 Plus XRF Analyzer

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 are 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, silicon analysis of such piping and equipment with portable X-ray fluorescence (XRF) is an ideal choice. The new Thermo Scientific™ Niton™ XL5 Plus allows for fast, accurate, and precise analysis of silicon and other elements in the field.

Niton XL5 Plus XRF Analyzer

The new Niton XL5 Plus is the smallest and lightest high performance XRF metal analyzer in the market. The light weight and small size of the Niton XL5 Plus reduce operator fatigue and enable access to more test points. Compact measurement geometry, a powerful 5W X-ray tube, and the latest silicon drift detector technology

The Niton XL5 Plus in use, analyzing steel piping.

with graphene window, provide the highest performance and best light element sensitivity for the most demanding applications such as low silicon measurement. Niton XL5 Plus delivers fast, accurate elemental analysis in demanding refinery environments. The Niton XL5 Plus provides the refining industry with the following key benefits:

- Unparalleled chemistry and grade identification accuracy for confident results every time
- Outstanding sensitivity for light elements (Mg, Al, Si, P, S)

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Analysis of Silicon in Steel to Prevent Sulfidic Corrosion Using the Niton XL5 Plus XRF Analyzer

- Small size and light weight improves productivity and enables testing in tight spots without operator fatigue
- Flexible user interface enables custom workflow solutions and easy optimization for specific applications such as low silicon measurement.
- Integrated camera and small spot analysis for accurate sample positioning and image capture
- Splashproof, dustproof and rugged housing for harsh environments

Method

Certified reference standards and samples were analyzed after ensuring the surface is clean and clear of any contaminants that could introduce silicon or other elements to our analysis. Data quality objectives dictate the sample preparation requirements and the minimum analysis time used.

Typical metal alloys that are at risk of sulfidation corrosion are carbon and low alloy steels. These alloys will oxidize when exposed to atmospheric conditions. This oxide coating can affect the accuracy of the reading when performing an XRF analysis. As the chromium concentration in the metal gets lower, the oxidation will get worse. It is imperative to remove any corrosion in order to ensure an accurate reading.

In addition to oxidation, there can often be paint or oil or grease on the surface. Paint typically contains metals, such as titanium, zinc or calcium, that can impact the results of analysis. Grease can contain molybdenum and other additives.

In order to get the accurate silicon readings, all surface contamination must be removed in the area to be analyzed. The fastest way to prepare an oxidized surface is to use a right angle die grinder equipped with the proper abrasive media, such as a zirconia/alumina grinding disc. For low silicon analysis, the samples were analyzed for 9, 13, and 33 seconds using both the main filter (3 seconds) and light filter (6, 10, or 30 seconds) after thorough surface preparation.

Results

Figure 1 shows the correlation curve and certified results vs. the Niton XL5 Plus analyzer results. The coefficient of determination (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. As can be seen from the data agreement between laboratory results and the XL5 Plus, the results are very good.

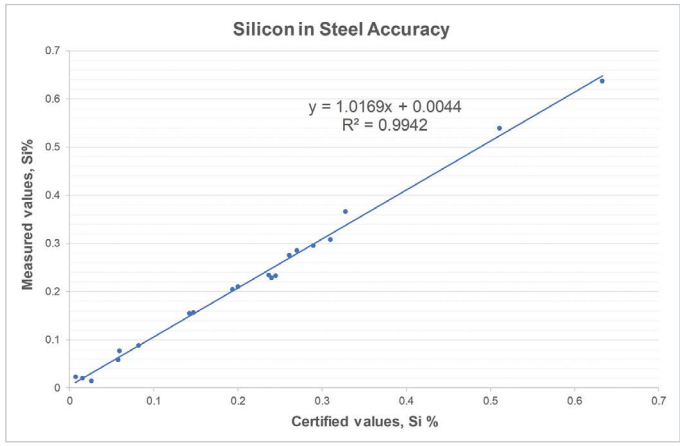

Figure 1: Silicon in steel accuracy using the Niton XL5 Plus analyzer

Figure 2 shows the measurement repeatability data for a low Silicon standard (0.058% Si) and higher Silicon standard (0.147% Si) using 9, 13, and 33 seconds measurement time. The data in Table 1 demonstrates the improvement in precision based on used measurement time.

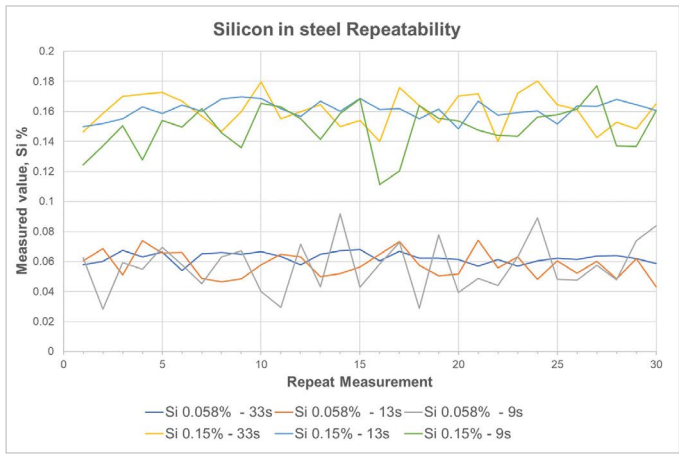

Figure 2: Silicon in steel measurement repeatability using the Niton XL5 Plus analyzer using 9, 13, and 33 seconds measurement time

Table 1: Effect of measurement time on silicon in steel measurement repeatability using the Niton XL5 Plus. All values in wt. %.

Run Number	9 Seconds	13 Seconds	33 Seconds
1	0.037	0.057	0.056
2	0.054	0.044	0.061
3	0.069	0.060	0.057
4	0.064	0.070	0.064
5	0.089	0.062	0.060
6	0.055	0.064	0.064
7	0.046	0.072	0.056
8	0.054	0.061	0.059
9	0.053	0.049	0.067
10	0.079	0.067	0.066
Average	0.060	0.061	0.061
Standard Deviation	0.016	0.009	0.004
Reference Value	0.058	0.058	0.058

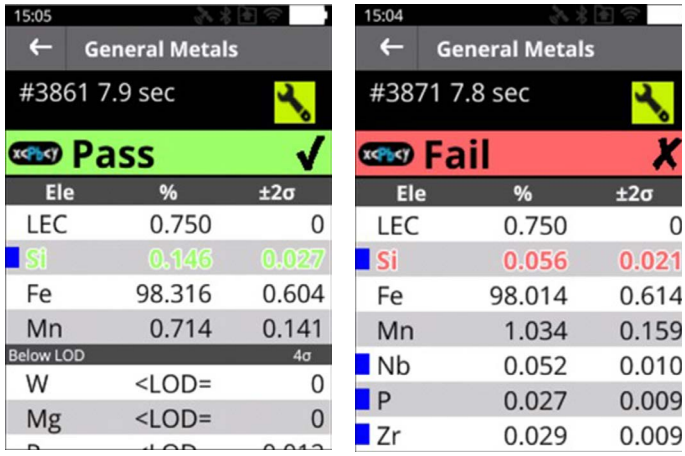

Figure 3: Capture of analytical results above (Pass: pipe not susceptible to sulfidic corrosion) and below (Fail: pipe susceptible to sulfidic corrosion) recommended threshold of 0.1% Si in carbon steel

Conclusion

Results achieved using the Niton XL5 Plus analyzer demonstrates excellent agreement with the laboratory results. The Niton XL5 Plus provides the best light element precision and sensitivity, and enables faster and more reliable low silicon measurement even using short measurement time. Given appropriate sample preparation, the analyzer is able to reliably detect Si levels in steel at levels above and below the threshold of 0.1%, within less than 9 seconds, as shown in Figure 3. Extended measurement time can be used to achieve more precise results when the measured concentration is close to threshold level of 0.1%. As can be seen from Figure 2, Niton XL5 Plus has excellent silicon measurement repeatability, and it can quickly and reliably identify low Si steel which has elevated risk of sulfidation corrosion from higher silicon steel.

Learn more at **thermofisher.com/portableid**

Americas
Boston, USA
+1.978.670.7460
niton@thermofisher.com

Europe, Middle East, Africa
Munich, Germany
+49.89.3681380
niton.eur@thermofisher.com

India
Mumbai, India
+91.226.6803000
ininfo@thermofisher.com

Asia Pacific
New Territories, Hong Kong
+852.2885.4613
niton.asia@thermofisher.com

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