Analysis of Residual Elements in Steel Pipe with the Thermo Scientific Niton XL3t 900 GOLDD Series

Application

Residual element concentrations in carbon steel pipe can be a critical indicator of the expected life and performance of finished components in petrochemical applications. Particular elements of interest include Cr, Cu, and Ni, as well as Mo, Sn, V, Sb, As, and Pb. The manufacturing of carbon steel is becoming more dependant on recycled product. As this happens, residual element concentrations in finished materials increase. Further, residual elements are difficult to remove or lower using simple metallurgical techniques during the melting process. Because residual levels can be strong indicators and affecters of material properties, including corrosion resistance, residual element analysis is increasingly a major concern in both installed and newly purchased materials.

A case study cited in the September 2004 issue of *Hydrocarbon Processing*[1] showed that HF alkylation units can be subject to selective corrosion in a unique manner resulting from elevated levels of residual Cr, Cu, and Ni. In this case study, a failure in the form of a pressure leak caused by internal corrosion was discovered at a weld between a 90° elbow and a straight section of pipe. Chemical testing determined that the sum of Cr, Cu, and Ni was greater than 0.50%. The article's authors suggest a maximum allowable value of 0.20% for the sum of these three elements. This action level was later adopted by a licensee of HF alkylation units.

Method

Using a handheld Thermo Scientific Niton XL3t 900 x-ray fluorescence (XRF) analyzer with geometrically optimized large area drift detector (GOLDD™) technology, we analyzed a sample of A106 Grade B pipe after light surface preparation to remove oxidation. First, a laboratory analysis using an optical emission spectrometer was performed to validate the material test report submitted by the pipe manufacturer. Next, ten individual XRF readings were collected using a measurement time of 45 seconds (15 seconds main filter and 30 seconds light filter). Helium purge of 65 cubic centimeters per minute was used to enhance light element analysis of Si. However, He is not required for silicon analysis or residual element analysis. (See Table 2 for complete detection limits with and without helium.) We then collected the encrypted results on the instrument and downloaded them to the analyzer's Niton Data Transfer (NDT©) data program.

Results

Table 1 shows the results obtained with the Niton® XL3t 900 GOLDD analyzer. The material test report (MTR) chemistry is reported on the top line of the table. An independent laboratory analysis generally verified the MTR results, but calculated a slightly higher sum of the three critical residual elements (Cu, Cr, and Ni) at 0.211%, in good agreement with the XRF results.

XRF Test Results of A106 Grade B Pipe							
	Mn	Si	Ni	Cr	Mo	Cu	RE Sum
Material Test Report	0.650	0.180	0.090	0.070	0.030	0.040	0.200
XRF Average Analysis	0.673	0.198	0.096	0.072	0.033	0.047	0.215
2 sigma Standard Deviation	0.013	0.021	0.008	0.002	0.002	0.005	0.015

Table 1. Comparison of lab results and Niton XRF analyzer results.

The XRF average analysis is the average of ten individual test results using the 45 second test times. The two sigma standard deviation was calculated from the ten individual results.

Limits of detection (LODs) for the Niton XL3t 900 GOLDD are listed in Table 2 below. LODs are calculated as three standard deviations (99.7% confidence interval) for each element. Testing time for determination of detection limits is set for 60 seconds per filter (180 seconds total). Helium was used at a rate of 65 cc per minute for determining the silicon detection limit. Without helium purge, the silicon detection limit is slightly higher.

There are a wide range of accessories for field use with Thermo Scientific Niton XRF analyzers, including heat shields and extension poles for testing hot pipes in use. Additionally, EPI “button” technology provides the optimal method for archiving test results. Factory-installed grade identification libraries contain most commonly used alloy specifications and can be modified “on the fly” to meet application demands and operator needs. Analysis averaging can be performed in real time on the analyzer; NDT report generation also provides an easy-to-use method for transferring test results into common data base programs for data analysis.

Detection Limits for Niton XL3t 900 GOLDD						
	Mn	Si	Ni	Cr	Mo	Cu
Detection Limits – He Purge	0.020	0.024	0.020	0.003	0.002	0.009
Detection Limits without He	0.020	0.075	0.020	0.003	0.002	0.009

Table 2. Limits of detection (LODs) for Niton XL3t 900 with GOLDD technology.

Comments

Refinery inspectors need confidence in residual chemistry analysis. Residual concentrations can be critical to hundredths of a percent, to accurately predict variances in material strength, hardness, and corrosion resistance. HF alkylation is an increasingly central process in the refining industry for the production of petrochemical products. As gasoline requirements have evolved, so, too, has the dependence on this process that offers a very clean burning product.

Breakthrough GOLDD technology, providing light element detection without helium or vacuum purging, achieves lower detection levels and higher sample throughput for true lab-quality performance. We continue to push the performance of handheld XRF instruments to levels never achieved before. For additional information, contact your local sales representative or visit us at www.thermo.com/niton.

Example of residual element concentrations.

The Niton XL3t GOLDD analyzers give you the near instantaneous feedback you need for success.

[1]Tim Munsterman and Anelsy G. Mayorga, “Preferential Corrosion of Welds in HR Service,” *Hydrocarbon Processing* (September 2004): 113-119.

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