Analysis of selenium in refinery waste wa-

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App Note

Analysis of selenium in refinery waste water

OndaVia offers a Raman-spectroscopy-based system for rapid, on-site selenium measurement. Selenium is a common byproduct of coal mining, coal-fired power plants, oil refining, metal smelting and some forms of agriculture. OndaVia's simple technology accurately measures and speciates selenium levels in a variety of waters in a few short minutes, even in the presence of other contaminants and common interferences. This speed and convenience is a vast improvement over competing technologies such as ICP-MS.

Raman Spectroscopy

Raman spectroscopy is a powerful tool for chemical analysis—an optical technique that measures the vibrational and rotational modes within a molecular system. The sample is illuminated with monochromatic light (a

ful tool for chemical at measures the vibrain a molecular system. nonochromatic light (a

785-nm diode laser, in our case). The light interacts with Figure 1. OndaVia Analysis System. the molecular bonds, causing some scattered photons to

shift in energy. The resulting scattered light provides structural information that may be used as a "chemical fingerprint". The intensity of the Raman response is weak; however, it is directly proportional to the number of molecules—in other words, it is a direct measure of concentration.

OndaVia Analysis System

At OndaVia, we apply Raman spectroscopy to analytical chemistry. We combine proprietary methods, solvents, software, and algorithms to perform fast, accurate chemical analyses in a range matrices. The OndaVia Analysis System (OV-PP-J003), shown below in Figure 1, consists of a compact Raman spectrometer, proprietary reagents, and consumable, analyte-specific analysis cartridges. The spectrometer measures approximately 12" x 8" by 5". It operates on a regulated 2.5-A, 12-VDC supply using a "power brick" to convert 120-VAC. The system is supplied with our Advanced ORC[™] analysis software and an rugged case for transit and transport.

We currently offer analysis cartridges for an array of amines, anions, and organic compounds. We also offer pre-treatment kits for analysis in complex matrices. For example, our monoethanolamine (MEA) analysis method includes tools for pre-treating refinery sour water for quick, on-site MEA analysis. The OndaVia Analysis System works with all OndaVia analysis cartridges; only one instrument is required to measure any or all analysis cartridges. Furthermore, with OndaVia's Advanced ORC[™] analysis software, the user has access to spectral data for in-house method development or research. The result is a powerful chemical analysis platform.

Selenium analysis

We have developed an analysis kit and associated methods for measuring selenium in a wide variety of sample matrices and at a variety of concentrations, down to single-digit parts-per-billion. Our selenium analysis kit (OV-PP-B005) provides the platform upon which all of our methods are based. Pre-treatment kits are available for coal-mine runoff, refinery waste water, and coal-fired power plant flue-gas desulfurization water.

Our selenium test is highly selective for the selenate (+6) oxidation state of selenium. With a simple oxidation process, the user can convert all selenium in a sample to the fully-oxidized, selenate state to determine the total selenium content. This selectivity enables selenium speciation, if desired, by performing two measurements, one oxidized and one not. The difference between the reported values is the selenite (+4) concentration.

App Note

The pre-treatment kits are designed to reduce interferences to enable ppb-level selenium measurements. The primary interferent is sulfate, which is frequently present at hundreds to thousands of parts per million. A simple solid-phase extraction column selectively reduces sulfate to the single-digit ppm range, where the selenium signal is then readily obtained.

In addition, we offer two measurement ranges: 100-ppb and 1000-ppb. The higher range method has a detection limit of 50-ppb, while the low-range method can determine selenium concentrations to 5-ppb. Lower detection limits are possible by adjusting sample volume; contact OndaVia for additional information.

The method begins by adding the supplied reagent to the sample. This reagent acts as an internal standard, compensating for any selenium loss during the processing steps. A fifteen-minute, unattended oxidation step is performed next, followed up sulfate removal and/or concentration steps. The final step introduces the sample to the instrument, where the concentration is reported to the user on a laptop computer. The full process can be completed in as little as twenty minutes, including the oxidation step.

Analysis of selenium in refinery waste water

We applied this method to refinery process and waste waters obtained from a the San Francisco Bay Area facility, one from the facility's chemical sewer (CS) and one from the induced air flotation unit (IAF). The results are reported in Table 1. We also identified nitrate in both samples, although the customer did not request quantification. Table 1. Extractions across a range of

With our nitrate and sulfate kits, it would be possible to determine these analytes crude oil samples of varying composiwith only a few minutes additional processing time.

Conclusion

OndaVia has developed analysis methods for ppb-level selenium analysis that apply to various waste water streams. Here, we demonstrated the ability to measure selenium in refinery waste water with a simple, easy test that takes less than twenty minutes. This simple approach expands the capabilities of OndaVia's analysis approach into matrices heretofore unreachable with Raman spectroscopy.

Sample	Expected (ppb)	Measured (ppb)
S0016	80	84
S0018	120	129

tions, sources, and properties.