



Monitoring waste water for selenium

Coal powers the generation of over one third of the world's electricity, vital to our everyday living. NALCO Water partners with coal-fired power plants globally to develop safe, reliable, and cost-effective solutions to meet environment, health and safety standards with a measurable return on investment.

It is critical to prevent selenium (Se) or any other toxic metals from industrial waste to stream or seep into our natural water sources. Coal power plants require massive amounts of water, especially for cooling towers; therefore, they are typically located near lakes, rivers or seas. Although coal only contains trace amounts of Se, when it is stockpiled, sprayed and exposed to snow and rain, it can become concentrated while leaching into the ground or streaming as runoff. At coal power generation plants Se may concentrate and discharge into waste streams, from scrubber effluent or during routine equipment maintenance and boiler cleaning.

Se is a vital nutrient in appropriate amounts, but dangerous to all forms of life in concentrated amounts. Therefore, most coal mining and power generation operations utilize processes to minimize its discharge. Waste water treatment systems provided by NALCO Water are key components of coal mining and power generation waste management.

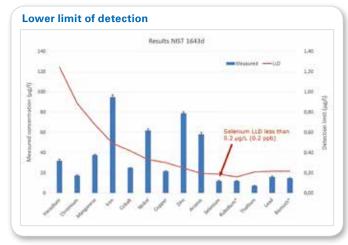
NALCO Water is the world's leading provider of water treatment and process improvements. They offer a number of tools to efficiently analyze evaluate, and treat waster water operations from primary clarification to discharge.

How does Bruker's TXRF help NALCO Water?

TXRF has no need for gases, hazardous chemicals, or labs with hoods and exhausts, it simply needs a power supply. It takes less than 20 minutes from sample preparation to final results for TXRF analysis. Further advantages are:

- Non-destructive measurements, samples can be used again or saved
- Daily calibrations are not required
- No cross contamination from one sample to the other
- Does not require daily or weekly maintenance checks
- Can be operated by a technician.

Bruker's S2 PICOFOX and S4 T-STAR® TXRF spectrometers provide straightforward, quick, and accurate trace measurements of Se at levels well below the EPA daily maximum of 23 µg/L (ppb) from scrubber waste water.



Water sample measurement of NIST 1643d standard by S4 T-STAR® and calculated LLDs show a detection limit for Se of $< 0.2 \,\mu\text{g/L}$.

NALCO Water collects samples to analyze Se levels with the S2 PICOFOX from waste water treatment systems to ensure the waste water is being cleaned properly. Testing is done for biological, chemical, and physical Se removal systems. Ash or settling pond water samples are also collected and measured to monitor Se which can seep into ground water and spill over during adverse weather conditions or when edges collapse.

Preparing samples for Se measurements with TXRF

Sample preparation for water is fast and straightforward. An aliquot of 1 mL of sample is transferred directly to a micro vial. A gallium standard solution (1 g/L) is added directly to the sample in order to get a concentration of 1 mg/L and shaken to ensure the internal standard is evenly dispersed throughout the sample. TXRF disks are pretreated with 5 μ l of Serva® solution, a Si based solution which allows the disk to become hydrophobic and allows for more even drying. 10 μ l of sample are pipetted onto the disk and dried on a hot plate before being placed in the instrument.

NALCO Water uses a 1:1 by volume mixture of the sample and 70% nitric acid to obtain low level readings. For samples with less than 20 ppb Se, 3 µl increments are pipetted in. The procedure of pipetting, drying, pipetting again is repeated six times.



Results before and after waste water treatment measured with S2 PICOFOX, concentrations given in mg/L.

Element	Reference sample		Waste water	
	1 ppm Se	0.015 ppm Se	before Se treatment	after Se treatment
s	425	74.3	569	412
К	2.05	4.42	16.6	12.3
Са	515	39.5	1404	1027
Se	0.93	0.011	0.346	< 0.011
Br	0.015	0.041	204	98.9
Sr	0.787	0.624	5.54	3.28

Results before and after waste water treatment

"NALCO Water, an Ecolab Company, uses Bruker's S2 PICOFOX TXRF to monitor selenium and other elements for confirmation of waste water treatment system efficacy."

TXRF measurements with the S2 PICOFOX show concentration differences of Se, as well as that for other elements, before and after waste water treatment (see table above). After waste water treatment, the element concentrations are significantly lower and almost zero for Se.

For larger sample load requirements, the S4 T-STAR®'s 90 position autosampler trays enables higher throughput than the S2 PICOFOX's 25 disk sample changer. The S4 T-STAR® also provides up to three excitation modes for applications where trace analysis of lighter elements (atomic number # 11-13) and heavier elements (atomic number # 41-53) are required.



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