

Sample Preparation for the Determination of Residual Concentrations of APQ-Containing Continuous Corrosion Inhibitors in Oilfield Waters using Wilson Analytical Fluorescence Spectrometers

Background:

Fluorescent materials, such as Alkyl Pyridine Quats (APQs), show signals that are directly proportional to concentration only if they fall within the linear range of the calibration curve for the compound being tested. Very high concentrations are often outside of the linear calibration range, and hence cannot be determined correctly without dilution prior to analysis. As our instruments have very good sensitivity for fluorescence measurements, we always dilute oilfield water samples by at least 5-10x with distilled water prior to analysis. This helps to ensure that the samples are well behaved with respect to colour and interferences, and that they fall within the linear calibration range for concentration determinations.

If the corrosion inhibitor residual concentration in the original water sample is completely unknown, then obtaining separate determinations on multiple dilutions is advisable, especially if the corrosion inhibitor level is high. If the same concentration result is obtained for the original sample for all dilutions (after correction for dilution of course), this shows that the sample concentrations measured were all within the linear range of the calibration, and hence that the reported concentration value of the original sample is correct.

If the unknown sample concentration after the initial 10x dilution is known for certain to be within the linear calibration range for the compound being measured, then multiple determinations using separate dilutions are not required.

Procedure:

- (1) Shake the water sample to ensure homogeneity.
- (2) Filter the water sample through a coffee filter to remove any large particles (if required).
- (3) Filter the water sample through a 0.2 μ m syringe filter to remove any fines. If the water still appears cloudy, repeat the 0.2 μ m filtration.
- (4) Dilute the sample 10 X and run the diluted sample on the Wilson Analytical spectrometer. Correct the obtained value for dilution and report the concentration of the original sample.
- (5) If a large concentration value for the original sample is obtained in Step 3, then prepare fresh 20X and 40X dilutions of the original sample. Run the diluted samples on the spectrometer. If all three dilution give values within the linear calibration range (i.e. all three give the same concentration value for the original sample when corrected for dilution), then report the average of the three values as the original sample concentration. If the three dilutions are drastically different, continue diluting and running until all dilutions show the same dilution-corrected concentration for the original sample.