

Application Note:

On-Line Monitoring of Caustic & Carbonate with a ClearView® db Photometer

Purpose: To simultaneously measure caustic (NaOH) and sodium carbonate in aqueous process streams, particularly at low (<2%) caustic concentrations, using a ClearView db NIR photometer and fiber optic probe.

Background: The dominance of water in the NIR hampers the measurement of caustic. Nevertheless, there are several successful reports of using full-spectrum FT-NIRs and discrete wavelength photometers for moderate caustic concentrations. Watson and Baughman (Spectroscopy, 2(1) 44-48 (1984)) summarized a typical approach using the spectral region from 1700 to 2300 nm and narrow optical cells of <1 mm. Their calibration (Figure 1) is a caustic calibration used when other species are present, such as carbonate and Na₂S. Our lower range of interest is circled on their calibration.

Approach: A We used the same procedure as described in our article (Spectroscopy, 15 (2) 40-46 (2000)). Samples were thermostated in a sample cell at 35 °C and analyzed with our spectrophotometer. A partial least squares (PLS) analysis of the spectral calibration set was performed. This analysis revealed that several analyzing wavelengths were required in addition to a reference wavelength for both measurements. A ClearView db photometer was configured with those wavelengths. The samples were re-run in the ClearView db and their absorbances were recorded. Multiple linear regression (MLR) coefficients were then calculated.

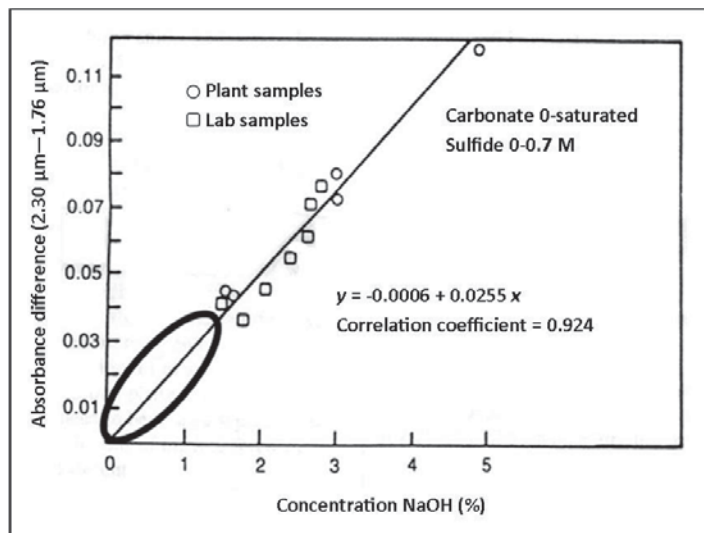


Figure 1: Plot of absorbance difference at 2.30 and 1.76μm versus percent of equivalent NaOH. Samples contained NaOH, Na₂CO₃, and Na₂S, %NaOH calculated assuming Na₂S was hydrolyzed.

ClearView® db Enclosure Options



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Results: The resulting MLR calibrations are shown in Figure 1 and 2. Months of on-line data have demonstrated the linearity of the caustic calibration with a precision of better than ± 0.1 . We have extended this approach to another customer's sample set in the 1-10% caustic range with an $R^2=0.9938$ and an SEE of ± 0.47 . It is pointed out that temperature conditioning of the sample in the slip stream to better than 2°C is important, primarily for caustic.

The ClearView db has the capability to correct the calibration for wider temperature fluctuations using the measured sample temperature from an RTD sensor. Nevertheless, this places demands on the sampling system containing the fiber optic probe. Due to the likely need of cleaning the sapphire windows in the probe with HCl, we recommend a probe with a Hastelloy® B body for concentrations over 20%, titanium for concentrations 60-80%, and Kalrez® 6375 wetted o-ring seals.

Conclusion: Our multiple wavelength ClearView db photometer has the sensitivity and stability to measure low levels of caustic in the presence of carbonate. This has been successfully demonstrated online.

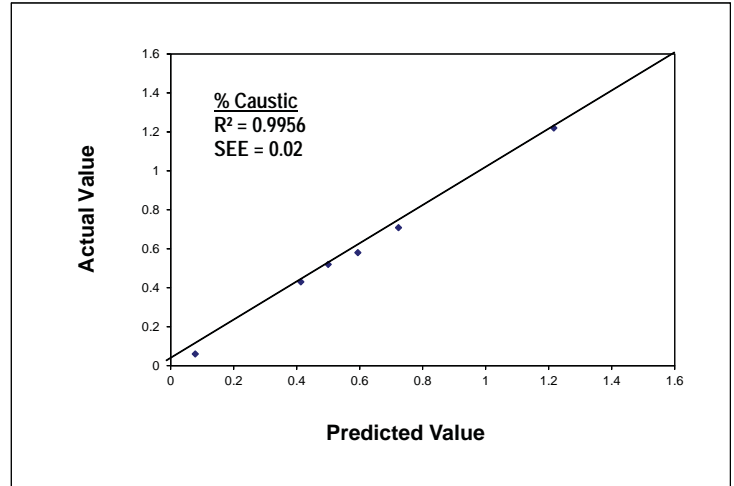


Figure 2

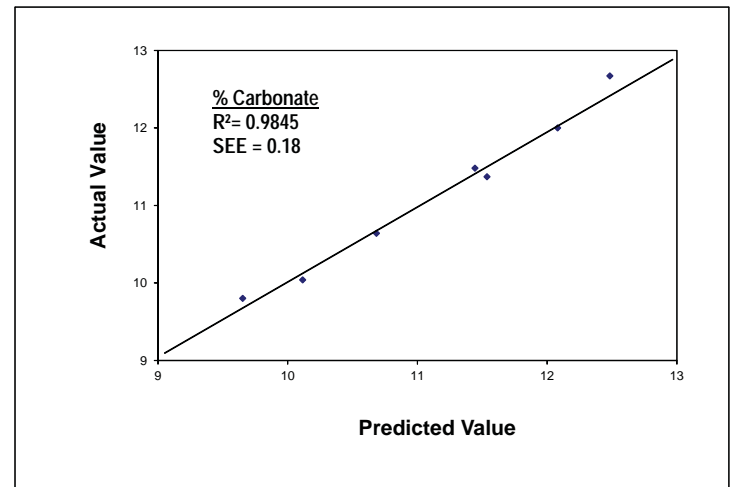


Figure 3