Method Development for ppm Level Phosphorus Analysis in Challenging Matrix with a Benchtop XRF Spectrometer

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A quality control lab had a need to replace an aging fixed channel wavelength spectrometer for several methods that were used to monitor production raw material and product quality. An energy dispersive system was selected and purchased for this purpose. This study describes the development and validation of a method to determine the concentration of phosphorus (P) at low levels (< 10 ppm, w/w) in an organic liquid containing a high concentration of silicon (~14 wt.%) using a benchtop energy dispersive x-ray fluorescence spectrometer. Matrix-matched standards were prepared and used for method calibration. Initial results with peak deconvolution alone gave an unsatisfactory calibration fit and poor measurement repeatability due to the variability in the deconvolution of the large Si peak that was adjacent to the P KA peak. To avoid this problem, ranges of interest (ROI) were selected for both the P (peak) and P+ (background), this combined with deconvolution significantly improved the calibration fit and repeatability of the measurement especially at low concentrations. This method is applicable for the analysis of LOD (3) to 70 ppm of phosphorus in the organic liquid. Instrument precision, method precision, accuracy and LOD/LOQ were determined.