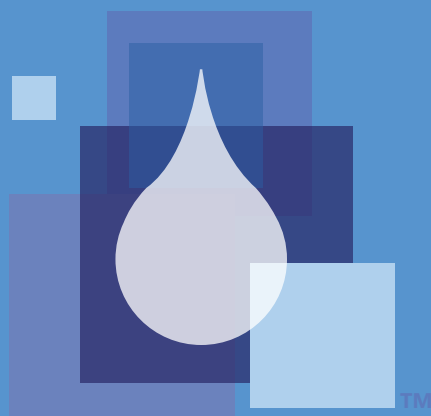


Phosphate and sulfate analysis

OV-AN-0016



OndaVia

Phosphate and sulfate quantitation using Raman spectroscopy

OndaVia develops quantitative analytical methods using Raman spectroscopy. A Raman spectrometer detects the vibrations of chemical bonds. Each set or combination of bonds has a distinct spectrum, providing a chemical ‘fingerprint’ which uniquely identifies a chemical. The intensity of the observed spectrum is linearly related to the concentration in solution. We use this relationship coupled with an internal standard to produce a fast, laboratory-quality test for chemical quantification aqueous solutions. In recent years, due to the miniaturization of lasers, electronics, and microprocessors, Raman spectrometers have decreased in size, and cost, thereby increasing portability and convenience.

This research note details the application of our Raman techniques to create a rapid and accurate phosphate analytical method in the 20-30% by weight range of aqueous phosphate. In this method, the user adds a quantity of sample to a vial or ampoule containing an internal standard. The internal standard solution—or reagent—contains a known quantity of a Raman-active compound. This compound provides a reference against which an unknown concentration of phosphate and/or co-analytes can be measured.

Representative spectra for phosphate (as P_2O_5) are presented in Figure 1. The primary peak from the internal standard is located at 1001-cm^{-1} in the spectrum; all data is scaled to this peak. The signal from the phosphate is illustrated by the peak at 893-cm^{-1} for three concentrations: 20%, 25%, and 30%. There is a clear and strong signal from phosphate, which scales linearly with concentration. There are approximately twenty samples per concentration presented. This data can be used to generate a calibration curve for determining the concentration of phosphate in an unknown sample.

OPAL: benchtop analysis

The OndaVia Portable Analysis Laboratory (OPAL™) is a compact Raman spectrometer and associated test kits for simple, quick chemical analysis. The Raman spectrometer is a portable unit equipped with a 785-nm laser providing approximately 100-mW of optical power at the sample. The spectrometer is packaged in a machined aluminum shell that provides rugged stability as well as a pre-aligned and focused holder for glass ampoules or screw-top vials. The instrument requires a 5V power supply, provided as a line voltage brick with coaxial power connector.

Custom-designed software provides instrument control and handles data acquisition and processing, returning a percent weight concentration in aqueous solutions. Operation of this software and the instrument requires a computer with Windows 10 or later operating system and a free USB port.

Using this system, OndaVia analyzed real-world samples containing phosphate and sulfate at %-level concentrations. These samples are dark in color; the darkness leads to fluorescence, which masks the Raman signal. However, with an appropriate dilution or mixing ratio, it is possible to isolate the Raman spectrum.

Data are presented in Figure 2. The black trace is a real-world sample containing approximately 25% phosphate and 2.8% sulfate. Due to the strong sample color, the sample was diluted 1:1 before analysis. The phosphate signal is strong and clear at 893-cm^{-1} in the sample and in two laboratory standard solutions. The laboratory standard solutions were not diluted and therefore have twice as much phosphate relative to internal standard when compared to the field sample. The signal from the field sample corresponds to approximately 25% phosphate; a new calibration curve is necessary for more precise analysis.

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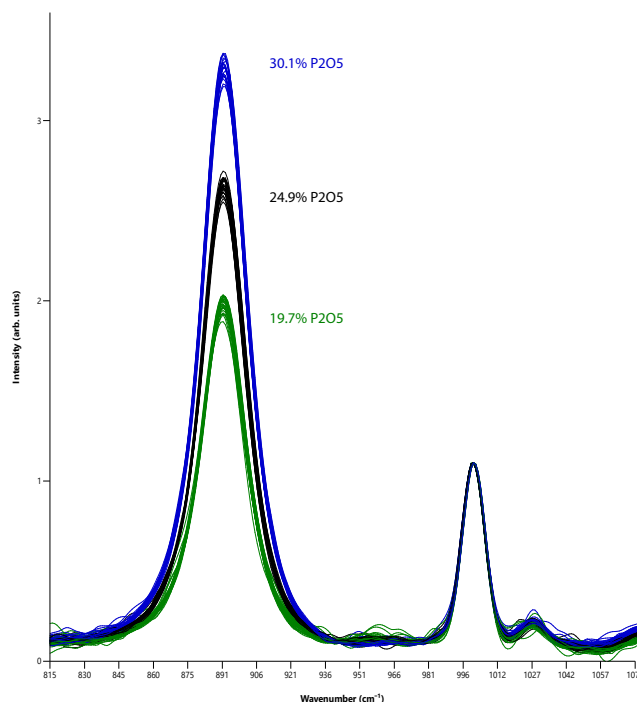


Figure 1. Raman spectrum for P_2O_5 in aqueous solution at 20, 25, and 30% by weight. The spectra are scaled to an internal standard signal using OndaVia's patented and proprietary methods.

Raman spectroscopy is a linear phenomenon. Two different analytes in a sample will generate independent Raman spectra, each corresponding to their respective concentrations. This effect means that we can quantify multiple analytes in a single sample by considering their spectra separately. These phosphate samples also contain sulfate at %-level concentrations. The measured Raman spectrum clearly shows the sulfate signal at 980-cm^{-1} . The corresponding, scaled concentration for sulfate in the field sample is 2.8%.

ORCA: in-line, semi-continuous analysis

The analytical techniques used for benchtop analysis can also be deployed in an automated, inline, semi-continuous analysis system. The OndaVia Raman Controller for Automation (ORCA™) system automates the process of mixing a sample with internal standard and then measuring that sample with a Raman spectrometer.

This system, designed for %-level analysis, contains pumps, valves, control electronics, and Raman spectrometer needed to perform analysis on phosphate solutions. Multiple configurations are available, with and without displays. The fiberglass enclosure is sealed, with access through swage-style compression fittings. Reagent is provided in 4-L jugs designed for approximately 8000 tests before replacement. Clean water must be provided for washing.

Conclusion

Raman spectroscopy is a powerful analytical technique, especially for aqueous solutions where other spectroscopies might fail. OndaVia's proprietary and patented methods make Raman spectroscopy a reliable, simple quantitative technique in many applications where alternatives are not practical.

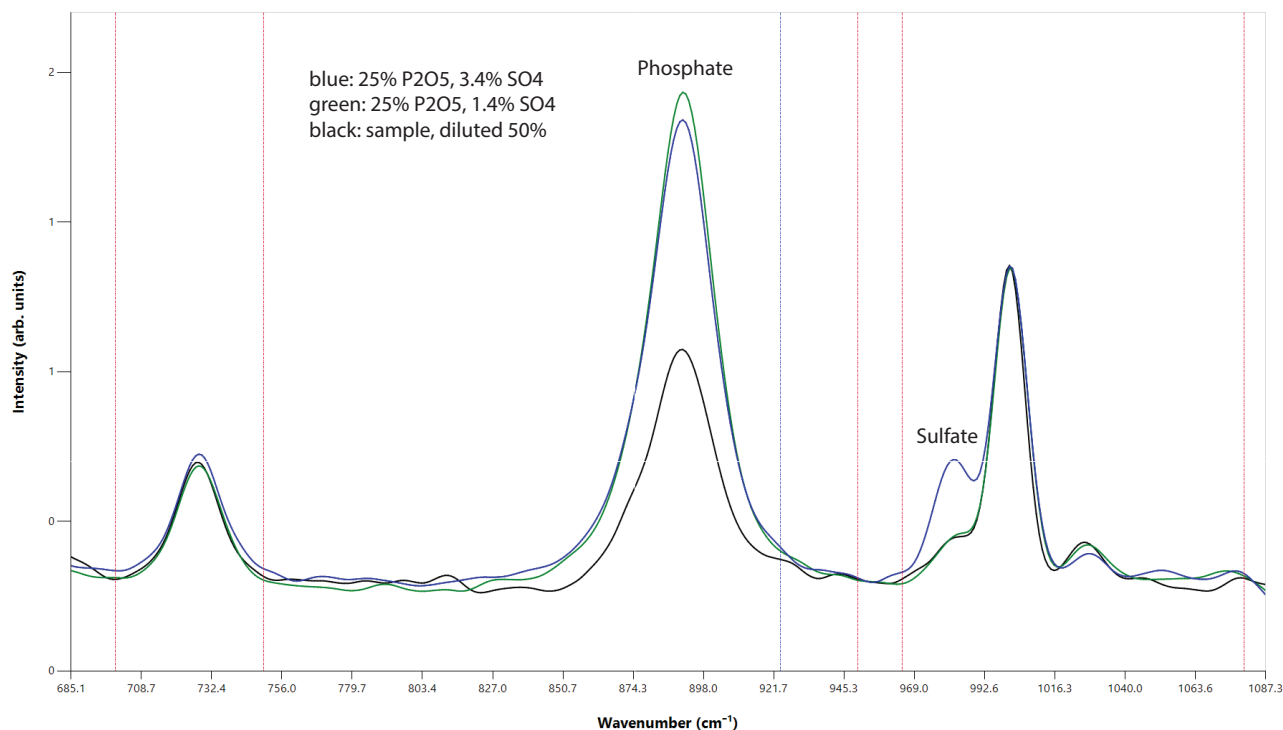


Figure 2. Comparison of real-world field data from a phosphate processing facility against laboratory standard solutions. These samples contain phosphate and sulfate at the %-level, both of which can be quantified simultaneously from a single Raman spectrum.

