

Application News

Quantitative Analysis of High-Concentration Samples without Dilution

—Introducing the UV-2700i Plus—

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User Benefits

- ◆ With its double monochromator and low stray light diffraction gratings, the UV-2700i Plus can analyze high-concentration samples at up to 8 Abs without dilution or other sample preparation steps.
- ◆ The spectrum-based quantitative analysis feature enables viewing and considering the entire absorbance spectrum during quantitative analysis.

■ Introduction

UV-Vis spectrophotometers are used for quantitative analysis of analytes in solutions or to assess optical components. When quantitatively analyzing analytes in solutions, UV-Vis spectrophotometers measure absorbance since this varies proportionally with analyte concentrations. However, at absorbances greater than 2 Abs, most UV-Vis spectrophotometers cannot maintain the linear relationship between analyte concentrations and absorbance due to the impact of stray light (light outside the wavelength that is being used for analysis). This can be addressed by diluting the sample, which reduces the concentration of the analyte to a level that can be measured by the instrument. (For solids, the sample is cut into a thin section.)

The UV-2700i Plus UV-Vis spectrophotometer, however, has two diffraction gratings (a double monochromator) that diffract light and uses low stray light diffraction gratings that enable it to measure absorbances of up to 8 Abs.* This wide dynamic range makes it suitable for quantitatively analyzing samples that are not easy to dilute and for assessing the transmission characteristics of low-transmission samples.

This Application News article describes using the UV-2700i Plus to analyze a high-concentration sample and also the spectrum-based quantitative analysis feature available in LabSolutions™ UV-Vis, Shimadzu's control software for UV-Vis spectrophotometers.

* Measurements of up to 8 Abs are in the wavelength range of 400 to 650 nm

■ Overview of Equipment and Quantitative Analysis Features

The UV-2700i Plus (Fig. 1) has Shimadzu's low stray light diffraction gratings (marketed as Lo-Ray-Ligh® gratings), which increase its measurement range up to 8 Abs (0.000001 % transmission). This wide measurement range enables more detailed analysis and eliminates the need for dilution and other complex sample preparation steps.



Fig. 1 UV-2700i Plus

Also, the LabSolutions UV-Vis control software for Shimadzu UV-Vis spectrophotometers has features that many users need. In addition to the standard quantitative analysis feature that creates a calibration curve from measurements taken at predetermined wavelengths, it offers a spectrum-based quantitative analysis, which records complete absorption spectra before creating a calibration curve at any specified wavelength. The standard quantitative analysis feature is suited to routine analysis as it only measures absorbance at a predetermined wavelength (or vicinity), giving shorter analysis times. However, the spectrum-based quantitative analysis feature records the entire absorbance spectrum, allowing users to investigate which absorbance wavelength is optimal for quantitative analysis of a specific analyte and then tailor the analysis conditions based on knowledge of the effects of impurities on absorption. So it is ideal when analyzing samples for the first time or when sample contamination is a concern.

■ Quantitative Analysis of Potassium Permanganate Solution

Standard Quantitative Analysis Feature

Calibration curve linearity at high absorbances was verified by preparing a potassium permanganate (KMnO₄) solution at concentrations in which the highest absorption peak was equivalent to 1 to 8 Abs (65 to 520 mg/L), and samples were measured using the standard quantitative analysis feature. Each measurement was performed using a high-absorbance measurement kit and a 4 Abs neutral density (ND) filter on the reference side.

The standard quantitative analysis feature is accessed by selecting "Quantification" in the LabSolutions UV-Vis launch window (Fig. 2). It has three settings for the measurement method: "Point," "Area," and "Maximum value." Point measures and integrates absorbance at a fixed wavelength, and since it offers a high degree of repeatability, it was selected in this example. (See Table 1 for the measurement conditions.)

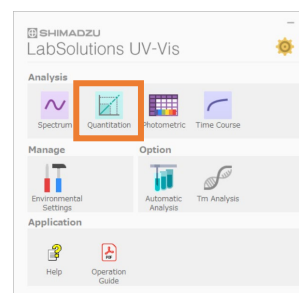


Fig. 2 LabSolutions UV-Vis Launch Window

Table 1 Analysis Conditions for Standard Quantitative Analysis Feature

Equipment:	UV-2700i Plus High absorbance measurement kit*
Measured Wavelength:	525.0 nm
Integration Time:	2.0 sec
Slit Width:	5.0 nm

*Comes as standard with UV-2700i Plus

The calibration curve in Fig. 3 is created from $KMnO_4$ solutions prepared at concentrations equivalent to 1 to 8 Abs. It shows that good linearity ($r^2 = 0.99999$) was achieved even at high absorbances near 8 Abs.

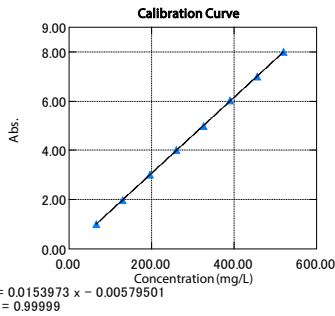


Fig. 3 $KMnO_4$ Solution Calibration Curve Using Standard Quantitative Analysis Feature

Spectrum-Based Quantitative Analysis Feature

Two more solutions with absorption peaks equivalent to 9 and 10 Abs (585 and 650 mg/L) were prepared for analysis by the spectrum-based quantitative analysis feature.

This feature is accessed by selecting "Spectrum" in the LabSolutions UV-Vis launch window (Fig. 2) and then configuring "Evaluation" - "Table settings" - "Type" (Fig. 4).

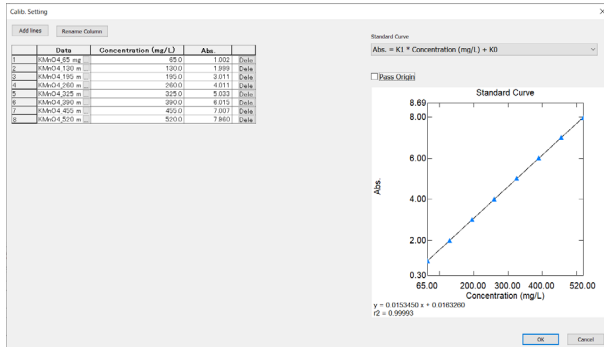


Fig. 4 Configuration Window for Spectrum-Based Quantitative Analysis Feature

The detailed analysis conditions used to measure the absorbance spectra are shown in Table 2, and the absorbance spectra recorded at each $KMnO_4$ solution concentration are shown in Fig. 5.

Table 2 Analysis Conditions for Spectrum Measurement

Equipment:	UV-2700i Plus High absorbance measurement kit
Measured Wavelength Range:	400 to 650 nm
Data Interval:	0.1 nm
Scanning Speed:	Low light scatter (medium speed)
Slit Width:	5.0 nm

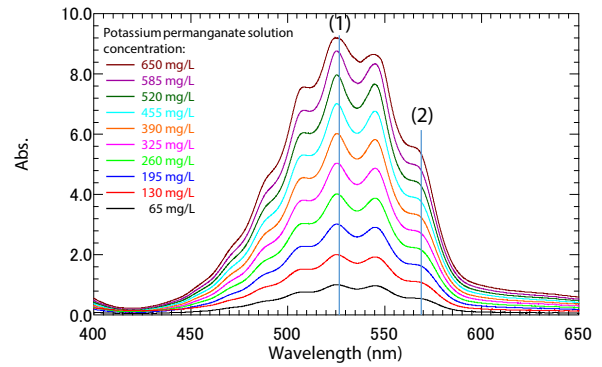


Fig. 5 Absorption Spectra of $KMnO_4$ Solution at Various Concentrations

Peak intensity was not proportional to concentrations above 8 Abs, where absorbance appeared to become saturated (Fig. 5). Next, wavelengths on the absorption spectra were selected, and calibration curves were prepared at these wavelengths using the spectrum-based quantitative analysis feature. In this example, the wavelengths (1) and (2) in Fig. 5 were selected. Wavelength (1), which is at the largest peak in the absorption spectrum (525 nm), was used to prepare the calibration curve (Fig. 3). Two calibration curves were prepared at wavelength (1): one with measurements up to 8 Abs and the other with absorbance above 8 Abs (585 and 650 mg/L). Wavelength (2), which is positioned at a shoulder peak (565 nm), was used to prepare a calibration curve with a maximum sample concentration of 650 mg/L. (See Fig. 6 to 8 for the calibration curves.)

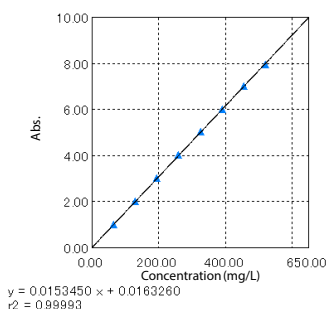


Fig. 6 KMnO_4 Solution Calibration Curve
Using Spectrum-Based Quantitative Analysis Feature
Wavelength Used in Quantitative Analysis: 525 nm,
Range of Quantification: 65 to 520 mg/L

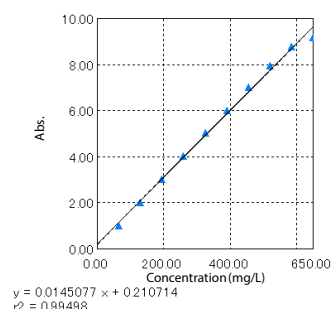


Fig. 7 KMnO_4 Solution Calibration Curve
Using Spectrum-Based Quantitative Analysis Feature
Wavelength Used in Quantitative Analysis: 525 nm,
Range of Quantification: 65 to 650 mg/L

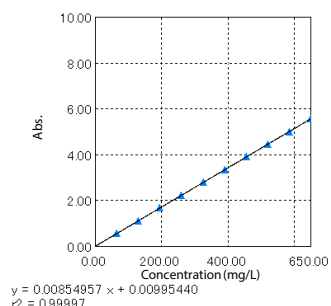


Fig. 8 KMnO_4 Solution Calibration Curve
Using Spectrum-Based Quantitative Analysis Feature
Wavelength Used in Quantitative Analysis: 565nm,
Range of Quantification: 65 to 650 mg/L

Even though Fig. 6 was not produced using the standard quantitative analysis feature, good linearity was achieved ($r^2 = 0.99993$) across an absorption range of up to 8 Abs. Adding higher concentration samples with an absorption of over 8 Abs negatively affected the linearity of the calibration curve ($r^2 = 0.99498$) (Fig. 7). When this negative effect is observed, good linearity can be achieved by selecting a wavelength where the absorption does not exceed 8 Abs at the highest sample concentrations. Selecting a different measured wavelength can reduce the slope of the calibration curve and produce good linearity even at these higher sample concentrations (Fig. 8).

The spectrum-based quantitative analysis feature allows the user to vary the parameters in quantitative analysis, based on knowledge of the full absorbance spectrum, and select a wavelength best suited to quantifying the sample.

Conclusion

This Application News article describes quantitative analysis of a high-concentration sample of potassium permanganate solution. It showed that the double monochromator and the low stray light diffraction gratings of the UV-2700i Plus UV-Vis spectrophotometer enable it to perform quantitative analysis at absorbances of up to 8 Abs. The spectrum-based quantitative analysis feature included with LabSolutions UV-Vis can check for peak saturation across the absorbance spectrum and use this information to select an absorption wavelength that will create a calibration curve with good linearity across the sample concentration range.

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