Linear dynamic range of the Cary 4000, 5000, 6000i: internal diffuse reflectance accessories

Data Sheet

Introduction/Theory

The photometric accuracy and linearity of a spectrophotometer defines its ability to measure an absorbance that can be directly related to a compound of known absorptivity or concentration. Of similar importance is the dynamic range over which the spectrophotometer remains linear. A wide linear dynamic range permits the analysis of highly turbid solutions and a wide range of sample concentrations (optical densities), as well as reducing sample preparation (dilution) requirements.

Diffuse reflectance accessories (DRAs) employ an integrating sphere design. This design greatly improves the efficiency with which light can be collected when analyzing highly scattering samples in either transmission or reflectance modes. For this reason, and because DRAs have their own light detectors, most system performance attributes must be characterized with the DRA installed. This data sheet uses potassium permanganate solution to demonstrate the excellent linear dynamic range of the Agilent Internal DRAs.

Cary 4000, 5000 and 6000i instruments provide a wide linear dynamic range

Agilent Technologies

Materials

- Cary 4000, 5000 or 6000i spectrophotometer with an Internal DRA
- 2 ➡ Attenuator, 1.5 Abs, part number 0110677500
- Quartz cuvettes (10 mm pathlength), part number 6610000800
- Accessory Final Cell Holder, part number 0210187900
- Standard potassium permanganate solutions (0.1, 0.5, 1, 10, 100, 200, 250, 300, 350 and 400 mg/L; freshly prepared from AR Grade $KMnO_4$)

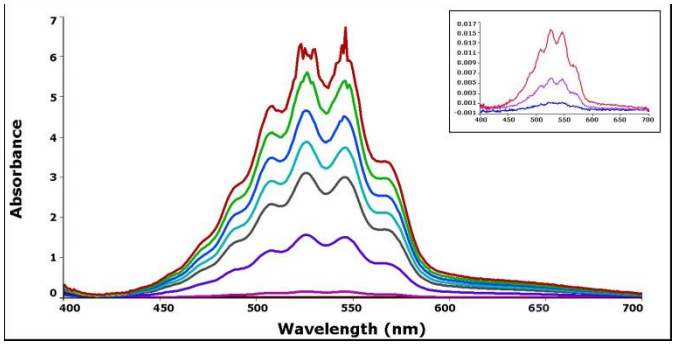
Method

1. Install and align the Internal DRA.
2. Warm up the Cary spectrophotometer for at least 1h prior to use.
3. Recalibrate the DRA by running the Auto-calibrate feature in the Validate application.
4. Install and check the alignment of the cell holder at the transmission port of the DRA beam.
5. Set up the instrument as follows:
   - Wavelength range: 750–400 nm
   - Scan rate: 60.0 nm/min with a data interval of 1.0 nm, signal averaging time 1.0 s
   - SBW: 1.5 nm
   - Zero/Baseline correction: ON
   - Slit height: Reduced
   - All other parameters: default
6. Perform a Zero/Baseline correction on a cuvette filled with water, without reference beam attenuation. Measure the 0.1, 0.5, 1, 10 and 100 mg/L KMnO4 solutions sequentially using the same quartz cuvette.
7. Place a 1.5 Abs attenuation in the reference beam and perform another Zero/Baseline correction. Using the same quartz cuvette as above, measure the 200, 250 and 300 mg/L $KMnO_4$ solution.

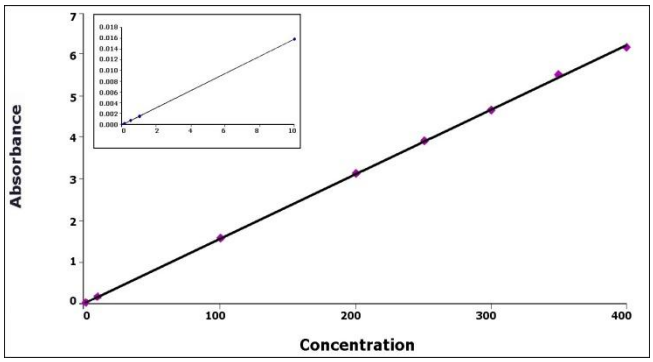
Note: Rinse the cuvette with each new solution before measurement, ensuring that the optical faces are dry and free from fingerprints. Use lint-free tissues if required. You should also ensure that the cuvette is in the same orientation when you replace it for each measurement.

8. Repeat Step 7, this time using 3.0 Abs attenuation in the reference beam (i.e. 1.5 + 1.5 absorbance), and scan the 350 and 400 mg/L $KMnO_4$ solution. Ensure that the two 1.5 Abs attenuators in the reference port are aligned to give approximately 3 Abs attenuation.
9. Construct a calibration curve by plotting the absorbance versus concentration.
10. To confirm the linear dynamic range of your Cary spectrophotometer, perform a linear regression on your data and calculate the coefficient of determination (r^2). This gives an indication of the 'goodness of fit' of your data to a straight line, and hence the dynamic range and linearity of the instrument.

Results



The spectra of standard permanganate solutions can be seen above. The insert shows the spectrum obtained for the lowest concentration standards (0.1 mg/L, 0.5 and 1.0 mg/L), and depicts a spectral profile identical to that of the more concentrated standards.



The plot of absorbance vs concentration (mg/L) above highlights the wide dynamic range and inherent linearity (r^2 = 0.99988) of the Cary spectrophotometers, and confirms that quantitative analysis of permanganate from 0.1 to 400 mg/L is quite feasible at the peak absorption wavelength of 525 nm up to 6 Abs.

The Internal Diffuse Reflectance Accessory

Conclusion

The quantitative analysis of aqueous potassium permanganate demonstrates the excellent photometric accuracy and wide linear dynamic rang of the Agilent DRA systems - an important DRA performance attribute regardless of the application at hand.

www.agilent.com/chem

Published March, 2011
Publication Number 5990-7834EN

Agilent Technologies