

CHEM 4114: Determination of Quinine by Fluorescence Spectroscopy

Objectives: To determine the concentration of quinine (a.k.a. quinidine) in tonic water.

Reference: Skoog, Holler, and Neiman, "Principles of Instrumental Analysis", 5th Edition.

Instrumentation: Perkin Elmer Model LS50B fluorimeter (in Dav. 123A).

Chemicals: conc. H₂SO₄, 1 ppm stock solution of quinine sulfate in 0.1 M H₂SO₄, tonic water sample.

WARNINGS: Follow safe laboratory procedures.

Procedures:

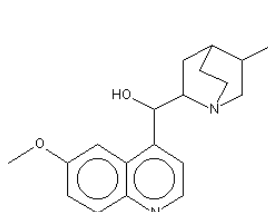
Sample Preparation:

Prepare the following solutions:

2.0 L of 0.1 M H₂SO₄ to dilute all samples.

100.0 mL each of 0.1, 0.05, 0.01, and 0.005 ppm quinine sulfate standards in 0.1 M H₂SO₄.

Obtain the tonic water sample from the TA and dilute by 1:1000 in 0.1 M H₂SO₄. Hint: Unless done very carefully, making a very large dilution is more accurate if done in two or more steps.



Instrument Operation:

Familiarize yourself with the instrument and software before making measurements. Start with the following fluorimeter settings and adjust as necessary to obtain good spectra:

- Resolution (slit width): Excitation: 5 nm, Emission: 5 nm.
- Scan speed: 500 nm/min.

Measurements:

- Do a prescan of the 0.050 ppm standard to find the optimum excitation and emission wavelengths.
- Record excitation and fluorescence spectra of the 0.050 ppm quinine standard.
- Record fluorescence spectra of all solutions and a blank at the optimum excitation wavelength. If the 0.10 ppm standard is off-scale, quantitatively adjust the concentration slightly lower.
- Use the spectra of your blank and standards to estimate the minimum-detectable concentration (defined as having a signal-to-noise ratio of 3). Prepare a new standard of that concentration and record the emission spectrum.

Data Treatment:

1. Plot a working curve of fluorescence signal versus quinine concentration. Verify that the fluorescence signal is linear with concentration through your range of standard concentrations. Calculate the detection limit for quinine based on your spectra the spectra of your blank and standards.
2. Determine the quinine concentration in the tonic water sample. Report your result and the 95-% confidence interval.

Discussion:

How does changing the excitation wavelength affect the wavelength of maximum intensity, λ_{\max} , and the intensity at λ_{\max} in the fluorescence spectrum?