A new experimental technique called Variance Spectroscopy will be described. It probes spectral variations among small regions of bulk samples resulting from statistical variations in composition. The method is applied to liquid suspensions of unsorted SWCNTs, which contain several distinct structural species emitting photoluminescence at characteristic near-IR wavelengths. Using dilute suspensions, focused excitation, sensitive multichannel detection, and quick data collection, we capture several thousand emission spectra representing independent spatial regions of the sample. The data sets are analyzed to find emission intensity mean and variance at each wavelength. We then combine the mean and variance spectra to obtain information unavailable from conventional methods, including abundances of different emissive species and their relative emission efficiencies. The data are further analyzed for correlations between intensity fluctuations at different wavelengths. This gives novel two-dimensional maps with off-diagonal peaks showing spatially correlated concentration variations for nanotubes of different types. These are from fragile “loose” aggregates of spectrally unperturbed SWCNTs, which may be precursors of familiar nanotube bundles. Results will be presented showing SWCNT loose aggregates under different conditions. We also use the method to study sonication-induced sample changes. Variance spectroscopy should prove a valuable tool for characterizing samples of carbon nanotubes or other nanoparticles with spectral heterogeneity.