CH424 Instrumental Analysis Lab

Infrared Analysis of Xylene Mixtures

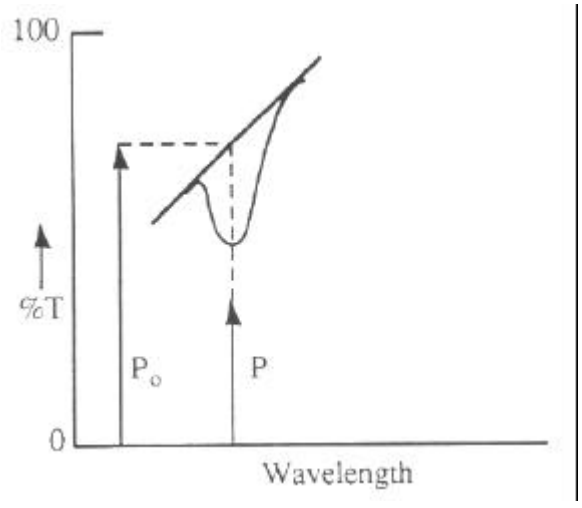
(Reproduced, with minor modification, from CH 424 Instrumental Analysis Course Manual, Spring 1996, written by Jim Peterson)

Introduction

Quantitative infrared analysis is based on Beer's law, but a number of problems can preclude its direct application. The use of an internal standard provides a convenient means of overcoming all of the difficulties. In this experiment the composition of a mixture of m-xylene and p-xylene is determined using o-xylene as the standard. The bands used for the analysis are:

m-xylene	768 (or 691) cm^{-1}
o-xylene	739 cm^{-1}
p-xylene	795 cm^{-1}

The spectrometer is usually set to read percent transmittance. The reading can be treated by drawing a baseline tangent to both shoulders of the band of interest and then determining P_o and P as indicated in the diagram below. Note that the position of zero transmittance must be established accurately for this procedure to work satisfactorily.



The absorbance is calculated for each band and the ratio of the absorbance of the desired constituent to that of the internal standard is plotted versus the percent composition of the solution with respect to the constituent.

Reagent

Using cyclohexane as solvent, prepare five standard solutions with composition: 20% (v/v) o-xylene and 10.0, 15.0, 21.0, 25.0, 30.0% (v/v) m-xylene. Prepare a second set of five standards using p-xylene instead of m-xylene. Prior to recording data, cyclohexane solutions must be dispersed in Nujol (a viscous mixture of liquid alkanes) to reduce evaporation. **Note:** Dispose of xylenes in a container reserved for aromatic compounds.

Procedure

Mix one drop of a standard solution with one drop of Nujol onto an appropriate salt plate and then drop a second salt plate on top. (Note that the sodium chloride plates are easily etched by moisture on your hands. Hold them by the edges and clean them carefully with carbon tetrachloride between samples and when finished.) Record the infrared spectrum of the standard between 900 and 600 cm-1 Continue in this fashion with the other solutions to prepare a calibration curve for both m-xylene and p-xylene.

Obtain from your instructor an unknown consisting of a mixture of m-xylene and p-xylene in cyclohexane. Add o-xylene to 20% (v/v) as an internal standard and then proceed to record its infrared spectrum in the same manner as for the standards. Determine the composition of the unknown mixture by comparing the measured absorbance ratios with the calibration curves. Report the composition of the mixture as it was supplied to you (i.e. before the addition of the internal standard) and in terms of 95% confidence limits.

Questions

i) Why is it not necessary to carefully measure the amount of sample and Nujol which are mixed?

ii) What is the significance of the particular frequency range used in this experiment? Is it specific for xylenes or is it also useful for other substituted benzenes?

iii) Name another spectroscopic technique which can perform this quantitation. What are the advantages/disadvantages of the IR technique with respect to the one you name.

References

H. Veening, *J. Chem. Ed.*, 43, 319-320 (1966).

G. D. Christian in "Analytical Chemistry" (3rd ed.) p. 389, Wiley (1980).