

B. GENERAL TESTS

Infrared Spectrophotometry

Infrared Spectrophotometry is designed to identify or determine the sample by measuring absorption of infrared radiation of wavenumbers in a region of 4,000 to 400 cm^{-1} , at various wavenumbers, when it passes through the sample. This method uses the property that the infrared absorption spectrum of a substance is characteristic of its chemical structure. Infrared spectra are shown in charts drawn by plotting the wavenumbers on the abscissa and the transmittances or absorbances on the ordinate.

Unless otherwise specified, when the spectrum of the sample is similar in the intensity of absorption at the same wavenumber to the spectrum of the Reference Standard or the corresponding Reference Spectrum given in section 10 of REGENTS, SOLUTIONS AND OTHER REFERENCE MATERIALS, the sample is identified as concordant with the expected substance. When the spectrum of the sample measured in a solid state is different in the position and relative intensity of the absorption maximum from the Reference Standard spectrum or the Reference Spectrum, treat both the sample and the Reference Standard under the same conditions as directed in the individual monograph, then record the spectra.

When comparing the two spectra, care should be taken to allow for the possibility the two spectra are different in resolving power because different instruments are used to measure sample spectrum and the Reference Spectrum. The greatest variations based on the difference in resolving power between two instruments are likely to occur between 4000 cm^{-1} and 2000 cm^{-1} .

For Fourier-transform infrared spectrophotometers, the precision of wavenumbers is invariable through the total scanning region because the resolving power of them is constant, regardless of wavenumber

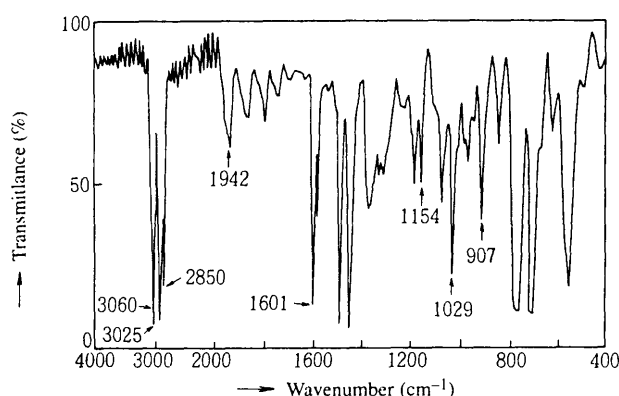
Infrared Reference Spectra ranging between 4000 cm^{-1} and 400 cm^{-1} are given in section 10 in the heading of REAGENT, SOLUTIONS, OTHER REFERENCE MATERIALS for the substances for which identification tests are specified in the individual monographs.

Apparatus and Procedure Use a dispersive infrared spectrophotometer or a Fourier-transform infrared spectrophotometer.

Before using the infrared spectrophotometer, adjust it as specified in the operating manual. The linearity of the absorbance between 20% and 80% of transmittance (%) should be within $\pm 1\%$. The reproducibility of the transmittance should be within $\pm 0.5\%$ in two consecutive measurements. The reproducibility of wavenumber should be within $\pm 5 \text{ cm}^{-1}$ at about 3,000 cm^{-1} and within $\pm 1 \text{ cm}^{-1}$ at about 1,000 cm^{-1} . In addition, adjust the instrument so that a spectrum exhibits

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absorptions at the wavenumbers as indicated in the following figure when measurement is made on a polystyrene film (about 0.03 mm thick).



Preparation of Sample According to an appropriate one of the methods below, prepare the sample so that the transmittance of the most intense absorption bands should be within a range of 20 to 80%. For the optic plate, use sodium chloride, potassium bromide, or thallium iodide bromide.

(1) Potassium Bromide Disk Method Place 1 to 2 mg of a solid sample and 100 to 200 mg of dried potassium bromide for infrared spectrophotometry into an agate mortar, quickly reduce to fine particles protecting from moisture, mix completely, and transfer into a die. Press the surface of the disk at 500 to 1,000 N/cm² under reduced pressure of not more than 0.7 kPa for 5 to 8 minutes, and use this disk for the measurement.

(2) Solution Method Prepare a solution of the solid or liquid sample in the specified solvent, inject the solution into a fixed cell for liquid, and use this cell for the measurement. Place the similar cell containing the same solvent for the compensation beam. The thickness of the fixed cell is generally 0.1 mm or 0.5 mm.

(3) Paste Method Crush finely a solid sample and knead well with liquid paraffin in the mortar. Hold the paste between two optic plates without any air gap, and measure.

(4) Liquid Film Method Hold 1 to 2 drops of liquid sample as a capillary film held between two optic plates, and measure the liquid layer between the plates. If it is necessary to thicken the liquid layer, place rings of aluminum foil or a similar material between the two optic plates so that the liquid sample lies between the plates.

(5) Thin Film Method Dissolve the sample in the specified solvent, and apply it to one optic plate. Evaporate the solvent by drying with hot air, and measure the thin film adhered on the plate. If the sample is a film with a thickness of not more than 0.02 mm, measure the film just as it is.

(6) Gas Sample Measurement Put the sample gas in a gas cell with a light path of 5 to 10 cm in length, previously evacuated, under pressure specified in the

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individual monograph, and measure. A long cell with the light path of not shorter than 1 m is also used if necessary.