

## 〈197〉 SPECTROPHOTOMETRIC IDENTIFICATION TESTS

Spectrophotometric tests contribute meaningfully toward the identification of many compendial chemical substances. The test procedures that follow are applicable to substances that absorb IR and/or UV radiation (see *Spectrophotometry and Light-Scattering* (851)).

The IR absorption spectrum of a substance, compared with that obtained concomitantly for the corresponding USP Reference Standard, provides perhaps the most conclusive evidence of the identity of the substance that can be realized from any single test. The UV absorption spectrum, on the other hand, does not exhibit a high degree of specificity. Conformance with both IR absorption and UV absorption test specifications, as called for in a large proportion of compendial monographs, leaves little doubt, if any, regarding the identity of the specimen under examination.

### INFRARED ABSORPTION

Seven methods are indicated for the preparation of previously dried test specimens and Reference Standards for analysis. The reference 〈197K〉 in a monograph signifies that the substance under examination is mixed intimately with potassium bromide. The reference 〈197M〉 in a monograph signifies that the substance under examination is finely ground and dispersed in mineral oil. The reference 〈197F〉 in a monograph signifies that the substance under examination is suspended neat between suitable (for example, sodium chloride or potassium bromide) plates. The reference 〈197S〉 signifies that a solution of designated concentration is prepared in the solvent specified in the individual monograph, and the solution is examined in 0.1-mm cells unless a different cell path length is specified in the individual monograph. The reference 〈197A〉 signifies that the substance under examination is intimately in contact with an internal reflection element for attenuated total reflectance (ATR) analysis. The reference 〈197E〉 signifies that the substance under examination is pressed as a thin sample against a suitable plate for IR microscopic analysis. The reference 〈197D〉 in a monograph signifies that the substance under examination is mixed intimately with an IR-transparent material and transferred to a sample container for diffuse reflection (DR) analysis. The ATR 〈197A〉 and the 〈197E〉 techniques can be used as alternative methods for 〈197K〉, 〈197M〉, 〈197F〉, and 〈197S〉 where testing is performed qualitatively and the Reference Standard spectra are similarly obtained.

Record the spectra of the test specimen and the corresponding USP Reference Standard over the range from about 2.6  $\mu\text{m}$  to 15  $\mu\text{m}$  (3800  $\text{cm}^{-1}$  to 650  $\text{cm}^{-1}$ ) unless otherwise specified in the individual monograph. The IR absorption spectrum of the preparation of the test specimen, previously dried under conditions specified for the corresponding Reference Standard unless otherwise specified, or unless the Reference Standard is to be used without drying, exhibits maxima only at the same wavelengths as that of a similar preparation of the corresponding USP Reference Standard.

Differences that may be observed in the spectra so obtained sometimes are attributed to the presence of polymorphs, which are not always acceptable (see *Procedure* under *Spectrophotometry and Light-Scattering* (851)). Unless otherwise directed in the individual monograph, therefore, continue as follows. If a difference appears in the IR spectra of the analyte and the standard, dissolve equal portions of the test specimen and the Reference Standard in equal volumes of a suitable solvent, evaporate the solution to dryness in similar containers under identical conditions, and repeat the test on the residues.

### ULTRAVIOLET ABSORPTION

The reference 〈197U〉 in a monograph signifies that a test solution and a Standard solution are examined spectrophotometrically, in 1-cm cells, over the spectral range from 200 to 400 nm unless otherwise specified in the individual monograph.

Dissolve a portion of the substance under examination in the designated *Medium* to obtain a test solution having the concentration specified in the monograph for *Solution*. Similarly prepare a Standard solution containing the corresponding USP Reference Standard.

Record and compare the spectra concomitantly obtained for the test solution and the Standard solution. Calculate absorptivities and/or absorbance ratios where these criteria are included in an individual monograph. Unless otherwise specified, absorbances indicated for these calculations are those measured at the maximum absorbance at about the wavelength specified in the individual monograph. Where the absorbance is to be measured at about the specified wavelength other than that of maximum absorbance, the abbreviations (min) and (sh) are used to indicate a minimum and shoulder, respectively, in an absorption spectrum. The requirements are met if the UV absorption spectra of the test solution and the Standard solution exhibit maxima and minima at the same wavelengths and absorptivities and/or absorbance ratios are within specified limits.