

Identification, ATR Infrared Spectral Analysis

Applicable Products: Carbopol® Polymers and Pemulen™ Polymeric Emulsifiers

Scope: This procedure describes an identification method for Carbopol® polymers and Pemulen™ polymeric emulsifiers by comparing the Attenuated Total Reflective (ATR) infrared spectra of a sample to the spectrum acquired from a known Carbopol polymer or Pemulen polymeric emulsifiers.

Abstract: The IR spectrum of a chemical compound is unique, with the exception of optical isomers. The interaction between electromagnetic radiation and the molecules of a substance results in a number of maxima that serve as a unique fingerprint of the chemical.

Interferences: There are no interferences.

Safety Precautions:

1. Wear safety goggles and gloves and follow good laboratory practices.
2. Polymer dust is irritating to the respiratory passages and inhalation should be avoided.
3. See Material Safety Data Sheet (MSDS) for additional safety and handling information.

Apparatus:

1. Fourier Transform Infrared (FT-IR) spectrometer operating at 4 cm⁻¹ resolution over a range of 4000 - 650 cm⁻¹ and utilizing Happ-Genzel, medium Beer-Norton, or an equivalent apodization function in the Fourier transform process (see Note 1).
2. ATR accessory: a horizontal stage, single bounce 45° incidence-angle unit, equipped with a clamp that can be used to press the analyte material into intimate contact with the ATR crystal. The clamp typically uses a pressure sensitive slip clutch such that a reproducible pressure is applied to every sample (see Note 2).
3. ATR crystal (see Note 3).

Reagents: None.

Procedure:

1. With the ATR accessory installed in the spectrometer but without any sample on the ATR crystal, acquire a background spectrum. The acquisition time/number of scans should be at least four times that used to acquire ordinary transmission spectra (see Note 4).
2. Deposit sufficient neat sample to completely cover the ATR crystal. If the material is particularly coarse, grinding is recommended.
3. Using the sample clamp, press the material into intimate contact with the ATR crystal by tightening the clamp screw until the clutch slips.
4. Acquire a spectrum of the material using the same scan conditions as for the background. Perform a Fourier transform and ratio against the Fourier transformed background spectrum.
5. Use the FT-IR software to identify the key peak positions.
6. If the spectrum is to be plotted, the peak positions can be annotated into the spectrum.

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Interpretations:

The spectrum of a material is compared to the spectrum of a known Carbopol® polymer or Pemulen™ polymeric emulsifier. Infrared trans-mission spectra exhibit main bands at or near 1710, 1454, 1414, 1245, 1172, 1115 and 801 wave numbers (cm⁻¹) with the strongest band near 1710 cm⁻¹. ATR spectra differ from transmission spectra in several respects.

First, ATR spectral intensities are diminished in the higher frequency regions and enhanced in the lower frequency regions, relative to a standard transmission spectrum. The degree to which the higher frequency bands are diminished and the lower frequency bands are enhanced is a function of the specific ATR crystal material used (or more specifically, to its index of refraction).

Secondly, the peak maxima can shift, usually to a slightly lower frequency. While most of the shifts are only 1-2 cm⁻¹, some of the bands can shift over 10 cm⁻¹.

A comparison of the wave numbers noted in the USP from a transmission spectrum of a Carbopol polymer with a germanium (n~4.0) ATR spectrum and one acquired using diamond (n~2.4) is shown in the following table. Since zinc selenide and KRS-5 have essentially the same refractive indices as diamond, spectra generated with these crystals are expected to be essentially identical to the diamond ATR spectrum.

USP*	1710	1454	1414	1245	1172	801
ATR (n~4.0)	1702	1452	1412	1231	1169	797
ATR (n~2.4)	1698	1451	1412	1228	1164	795

*USP lists a band at 1115 cm⁻¹. This band is not referenced in the table because it is very weak.

Therefore, when comparing peak maximum values of an ATR spectrum with a spectrum with standard transmission values, the sample may appear to fail the identification test. For this reason, ATR spectra should always be compared to ATR references only.

In order to meaningfully compare different ATR spectra, it is preferable that all have been acquired using the same ATR crystal material. At a minimum, all must have been acquired using materials of essentially the same index of refraction.

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Notes:

1. Some instrument operating parameters will vary depending upon the particular instrument manufacturer. The choice of operating parameters at any location is less important than assuring that all spectra are acquired in a consistent fashion. Because the ATR process has a low optical throughput, the acquisition time/number of scans should be at least four times that used for standard transmission spectra on the same instrument.
2. Devices of this type are manufactured by Pike Technologies, Harrick Scientific, Spectra-Tech, Smiths Detection, Specac and others.
3. Typically, the ATR crystal material will be germanium, zinc selenide or diamond. If diamond is used, it may be sandwiched to a zinc selenide or KRS-5 (thallous bromide/thallous iodide) backing crystal. The use of a crystal assembly that is entirely KRS-5 should be avoided, due to this material's toxicity, softness and partial solubility in aqueous solutions. The limited spectral range of silicon renders it an unsuitable crystal choice for Carbopol polymer spectra.
4. Due to the limited signal available from single-bounce style ATR accessories, their use is not recommended with a conventional dispersive infrared spectrometer.

References:

- Current edition of the United States Pharmacopeia <197>
- Current edition of the European Pharmacopeia

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