

**Drug Chemistry Section
Drug Chemistry Procedure Manual
Effective Date: March 17, 2003**

**Modification of F-1
Prepared by: L. Farren
Approved by: D Koontz
Supersedes: September 1, 1996**

Name of Procedure:

Perkin-Elmer Infrared Spectrophotometry

Suggested Uses:

Collection of quantitative or qualitative data for the identification of controlled and noncontrolled substances.

Apparatus Used to Perform Procedure:

Perkin-Elmer models: 1600, Paragon 1000, PE RXI, Spectrum BX, or Spectrum One
Potassium bromide (infrared grade)
Wig-L-Bug grinding mill
Stainless steel vial and ball
Hydraulic press
Vacuum pump and tubing
Power conditioner
Plotter/pens or printer/ink cartridge
Paper
Spatula
Agate mortar and pestle
Potassium bromide salt plates and holder
Pellet holder
KBr pellet die (13mm)
Polystyrene film standard
Polystyrene film standard spectra (attachment)
Oven
Thermometer

Calibration:

1. Scan background.
2. Place polystyrene film into sample area of instrument.
3. Scan polystyrene film.
4. Print spectrum scan of polystyrene film standard.
5. Record and mark wave numbers 2849 cm^{-1} , 1942 cm^{-1} , and 906 cm^{-1} ($\pm 3\text{ cm}^{-1}$).

Note: The IR Coordinator will evaluate the monthly polystyrene scan to verify the calibration of the instrument, then file and maintain the scan as set forth in the Drug Chemistry Policy and Procedure Manual.

Application of Procedure For Solid Samples:

1. Place approximately 1 milligram of sample and approximately 100 milligrams of potassium bromide in the capsule.
2. Grind mixture in Wig-L-Bug and transfer to pellet die assembly.
3. Press pellet die assembly (vacuum optional) in pellet press and remove pellet.
4. Place pellet in pellet holder.
5. Check background and obtain new background if needed. (Scan empty sample area to acquire data into background region.)
6. Place sample pellet holder in sample area of instrument and scan to acquire data.
7. Data can now be processed in any number of ways including: flattened, abexed and rescaled.
8. Print completed scan and compare to know reference standard.

Application of Procedure For Liquid Samples:

1. Place approximately 100 milligrams of potassium bromide in the capsule.
2. Grind in Wig-L-Bug and transfer to pellet die assembly.
3. Press pellet die assembly (vacuum optional) in pellet press and remove pellet.
4. Place pellet in pellet holder.
5. Smear a thin layer of the liquid sample on one side of the pellet.
6. Check background and obtain new background if needed. (Scan empty sample area to acquire data into background region.)
7. Place sample pellet holder in sample area of instrument and scan to acquire data.
8. Data can now be processed in any number of ways including: flattened, abexed and rescaled.
9. Print completed scan and compare to know reference standard.

Application of Procedure For Volatile Liquid Samples:

1. Check background and obtain new background if needed. (Scan empty sample area to acquire data into background region.)
2. Place a sample of the liquid in a shallow, open container.
3. Place this immediately inside the sample area of the instrument and close the cover.
4. Wait for approximately one minute or until the sample area is saturated with the sample vapors.
5. Scan to acquire data.
6. Data can now be processed in any number of ways including: flattened, abexed and rescaled.
7. Print completed scan and compare to know reference standard.

Safety Concerns:

Make sure capsule is firmly seated in Wig-I-Bug before operating apparatus.
Do not exceed 10 tons of pressure in the pellet press.
Do not look directly into the laser beam emitted from the infrared spectrophotometer.

Literature References:

Moffat, A. C. Ed., **Clarke's Isolation and Identification of Drugs**, 2nd. Ed., The Pharmaceutical Press, 1986.

Mills, III, Terry and Roberson, Conrad J., **Instrumental Data for Drug Analysis**, 2nd Ed., Vols. 1-5, CRC Press, Inc., 1993.

Silverstein, R. M. And Brassler, Clayton G., and Terence C. Morrill, **Spectrometric Identification of Organic Compounds**, New York, Wiley, 1991.

Keller, Roger, **The Sigma Library of FT-IR Spectra**, Edition 1, Vol. 1 and 2, Sigma Chemical Company, Inc., 1986.

Pouchert, Charles J., **The Aldrich Library of Infrared Spectra**, Aldrich Chemical Company, 1981.