Idaho State Police
Forensic Services

Approval for Quality System Controlled Documents

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#3 Fourier Transform Infrared Spectrometer Analytical Method

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#3
Fourier Transform Infrared Spectrometer Analytical Method

1.0.0 Background

The Fourier Transform Infrared Spectrometer (FTIR) is an analytical instrument that is used to identify compounds based on their infrared absorption properties. The advantages of using FTIR are that it can differentiate stereoisomers and it is fast. A sample can be analyzed in less than five minutes after preparation. The method is limited by the purity of the sample.

2.0.0 Scope

This AM will describe the routine maintenance, performance verification standards, and sample preparation methods necessary to perform quality analysis using a FTIR.

3.0.0 Equipment and Reagents

3.1.0 A FTIR and corresponding analytical software.
3.2.0 IR grade potassium bromide (KBr). Should be kept in a desiccator.
3.3.0 ACS grade solvents.
3.4.0 Hydraulic or other press for making KBr windows.
3.5.0 Any other sample introduction equipment, i.e. Gemini etc.
3.6.0 Reagents
- 3.6.1 Deionized or distilled water.
- 3.6.2 $NaSO_4$.
- 3.6.3 HCl gas, vapor, 10% solution.
- 3.6.4 $NaHCO_3$.
- 3.6.5 Na_2CO_3.
- 3.6.6 Phenylisothiocyanate.

4.0.0 Routine Maintenance

4.1.0 Aside from any necessary cleaning of the outside of the instrument, the sample chamber should be cleaned on a monthly basis. At the same time, the desiccant should be checked and replaced if necessary. Both of these checks will be noted in the maintenance logbook.

4.2.0 Background spectra will be collected before any samples are run. Background spectra should be run once every hour when performing batch analysis.

4.3.0 Monthly performance verification. Using the manufacture's procedures, a performance verification of the instrument is done using polystyrene film. This procedure will be performed monthly and after any maintenance. The "System Validation Report" printout is to be initialed by the analyst and kept in the maintenance logbook. If the verification does not pass and /or there is any other symptom of system failure then consult the manufacturer. Any maintenance is recorded in the logbook.

5.0.0 Standard Library Preparation

In order to confirm the presence of an analyte in a sample, the scan of the sample must match that of a known standard. It is not acceptable to confirm on the basis of a match from a commercially produced library (Georgia State etc.).

5.1.0 Production of valid standard library.

A pure sample of a standard is prepared and analyzed using the same procedures that will be used with an unknown. Once a scan has been produced it can then be stored in an internal library. A match made from this library is acceptable to use for confirmation. Libraries should be made up of all available standards including various salt forms and isomers (d & dl etc.). These standard scans can be produced and entered into the library as they are encountered in casework.

6.0.0 Identification Criteria

If a sample's FTIR spectra matches a spectra of a standard that was prepared the same as the sample, and the second test, if ran, is positive, then the compound is confirmed.

6.1.0 Standard spectra are prepared from authenticated standards and then stored internally for each FTIR instrument, at each laboratory.

6.2.0 FTIR spectra are considered matched if the peaks of the standard are present in the sample, in location, shape, and relative intensities. Any extra major peaks in the sample must be explainable.

7.0.0 Sample Preparation Methods

The following are examples of sample preparation methods for specific substances and or classes of substances.

7.1.0 Cocaine

7.1.1 Pick and Stick. Under a microscope Cocaine HCl appears as flat, mica like crystals. The cocaine can be separated from the cutting agent, added to KBr, and then formed into a pellet. This technique will often yield an IR pure spectra.

7.1.2 Direct. Grind some of the sample with KBr, and form a pellet. This method can be used to determine salt form as long as the sample is relatively pure. Identification

with an appropriate standard is required.

7.1.3 Basic extraction and cleanup. Dissolve sample in water or weak acid. Make basic with $NaHCO_3$. Extract with appropriate non-polar solvent, and dry through $NaSO_4$. Bubble HCl through extract and filter precipitate. Let dry and then mix with KBr, grind, and form a pellet.

7.1.3 Extract with chloroform, or methylene chloride, filter, and then recrystalize.

7.2.0 Heroin

Heroin "Panning" technique from NWAFS newsletter Vol. 12 No. 1. All weights and volumes are approximate.

7.2.1 Dissolve 100mg, preferably more, of sample (Black tar) in 5 mls of 10% HCl. Filter through cotton or glass wool if necessary.

7.2.2 Extract with 5mls of chloroform. Discard the aqueous, acidic, layer.

7.2.3 Back extract with 5 mls of water. Save aqueous layer. Repeat, combining both aqueous layers. Discard chloroform.

7.2.4 Add sodium bicarbonate and extract three times with 5 mls of chloroform. NOTE at this time the solvent can be evaporated onto KBr and analyzed to yield heroin base. May not yield good results due to the polymorphic nature of the base and the base is sticky and it may be difficult to press a good KBr pellet.

7.2.5 Bubble HCl through the chloroform. Dry through a sodium sulfate column. May be analyzed at this stage by drying on KBr.

7.2.6 Using an air stream and heat, recrystalize by doing a solvent exchange with petroleum ether. Evaporate onto KBr.

7.2.7 The spectra of the sample must be compared to a spectra of a standard that was prepared using the same procedure.

7.3.0 Phenethylamines

When purification is necessary the following methods should be used in making KBr pellets. Alternative sample introduction techniques can be used when appropriate.

7.3.1 Amphetamine and Methamphetamine.

Dissolve sample in water or dilute acid. Make basic with Na_2CO_3, or other strong base, and extract with petroleum ether or hexane. Wash extract with water then dry through Na_2SO_4. Bubble HCl gas through solvent and collect the resulting crystals. Wash with additional petroleum ether and let dry. Make KBr pellet. Compare against a known HCl salt standard.

7.3.2 dl-Methamphetamine by PIT derivative.

Dissolve sample in water and make basic with Na_2CO_3. Extract with petroleum ether and dry extract through $NaSO_4$. Add 2 drops of phenylisothiocyanate(PIT) and let stand for 10 minutes. Decant solvent and wash crystals with additional solvent. Dry and make a KBr pellet. Compare with a dl-Methamphetamine standard that was prepared using this procedure.

7.3.3 Direct. Mix and grind sample with KBr. Form into a pellet.

7.3.4 Ephedrine and Pseudoephedrine.

7.3.4.1 Using the same procedure as 7.3.1 will yield the HCl salt.

7.3.4.2 To obtain the base form, dissolve the sample in water, make basic with Na_2CO_3 and extract with petroleum ether or hexane. Evaporate the solvent and make a KBr pellet. Compare against a known base standard.

7.3.4.3 If the sample is a pill(s), crush and add methanol and shake well. Wait approximately one hour and centrifuge. Place supernatant into a clean test tube and allow too evaporate. Take crystals from side of test tube and make a KBr pellet.

8.0.0 History

Revision #	Issue or review date	History	Author or Reviewer
0	4/1/01	Original Issue	D.C. Sincerbeaux
1	8/27/02	Add Section 6 and #	D.C. Sincerbeaux
2	6/30/06	Changed Sec 4.1.0	D.C. Sincerbeaux
3	1/12/07	Added 7.0, 8.0, and 3.3 Dropped 5.2, changed title, Backround, reagent list	D.C. Sincerbeaux
4	7/3/2007	Dropped ACS requirements for dry chemicals	D.C. Sincerbeaux