Scanning Of Forensic Samples On Ftir - Sample Preparation

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Abstract

A variety of methods are used for sample preparation to study the infrared spectrum of solids. One of them is preparation of KBr disc and its scanning under transmission mode. An attempt has been made to calibrate the method for preparation of KBr disc with the solid samples. All possible variables, which affect the quality of KBr disc vis-a-vis their effect on quality of IR spectra have been studied with special references to one of the drug i.e. Atropine sulphate. The variables have been suitably controlled to produce the best quality of IR spectra for the sample under study. The advantages of having prepared a quality disc have been discussed for FTIR in the paper.

INTRODUCTION

For studying solid samples on FTIR formation of KBr disc, is one of the most accepted methods of routine. Drugs, Fibers, Poisons, Polymers and Explosive of forensic interest are mostly analyzed in their solid form using FTIR for the purpose of their identification and characterization [1-5]. The method is not only nondestructive but is useful when samples are available in very small quantity. Scanning of the samples using FTIR is preferred in transmission mode.

KBr is an important sample matrix for FTIR scanning. The KBr used should be of IR grade and must be pure. Before preparing the disc, KBr should be vacuum dried as per U.S. pharmacopoeia. To avoid water uptake, the KBr is stored in a sealed bottle, which is placed in a drying cabinet heated to 40 oC. Beside the quality of KBr, the number of sample molecules in the beam and path length are two important factors to be monitored in preparing disc for the sample. Materials that are not self-supporting need to be dispersed in potassium bromide (KBr) to study their spectra.

The other factors, which affect the IR spectra, are error in measuring which may be sometimes due to errors in preparation of the disc such as it may have poor distribution of sample, larger amount of sample with KBr or holes in the disc these may be minute, which could not be noticed while its preparation. The greatest obstacle for the analysis of powdered solid is making the particle size small, uniform and reproducible. Reproducibility in particle size can be improved by grinding the same sample for same time period.

The concentration of the sample and sample size are critical when preparing the KBr disc. Organic sample may be mixed with KBr powder at a sample concentration of 0.5%, while inorganic sample can be prepared up to same concentration provided that path length/thickness are correctly adjusted. Scratches and dents in the cylindrical anvil harpers the performance and can cause the anvil to get stuck inside the collar. The die set should never be compressed when empty. Considering various aspect of disc preparation the method has been standardized to study the FTIR spectra.

MATERIALS

35 mm mortar and pestle, Spatula, Auto press comprising of SS Die (13mm), pellet holder, plunger, envil and EXCALIBER BIO-RAD FTIR Instrument.

Potassium bromide (KBr) IR grade and Sample (Atropine sulphate) procured from M/s Pike Technologies and M/s Loba Chemie respectively.

Recommended Method:

To prepare a good quality disc, a standard method should be followed. The following points should be carefully followed in preparing the disc:

Place about 500 mg of KBr into a mortar and grind it until there is no evidence of crystalinity. Transfer this KBr powder into the drying box at a temperature of 40 oC.

Place 10 mg of solid sample into the mortar and again grind

it until a fine powder is form.

Weigh 1-2 mg of solid fine powder of sample (as per requirement of the die) and 200-300 mg of dry fine powder of KBr. Transfer these weighed quantities into a mortar and mix well with the help of a spatula. Do not grind during the mixing procedure, since reduction in particle size is not required and will lead to absorption of moisture by KBr.

Assemble bottom and top portion of KBr press assembly and place one of the 13 mm die with the polished surface up inside the press.

Transfer the KBr sample mixture to KBr press assembly. Make sure that 100% of the sample is transferred to press assembly.

Place second die inside the KBr press assembly with polished side down so that KBr sample mixture is now sandwiched between the polished surfaces of the each die.

Transfer KBr press assembly to press.

Connect vacuum line to evacuate air from the KBr press assembly with a vacuum pump.

Keep vacuum on, and slowly compress die in KBr press assembly until a pressure of 200 kg/cm2 is achieved on gauge. Make sure that pressure release valve is closed.

After 60 s, slowly open the pressure release valve to release the pressure and also disconnect the vacuum line.

Remove bottom portion of KBr assembly and turn upside down and place back on press.

Put the plunger and turn large knob on top of press and slowly press out bottom die and KBr disc to remove the die from the press. Check that the disc is translucent and the sample is homogenously distributed in the disc.

Before generating the IR spectrum, check the disc for the following faults:

• If the disc breaks on removal from the die, this indicates that the disc is too thin caused by too little powder too much pressure for long time. The remedy for this fault is to increase the sample load or by applying the correct pressure.

• The disc may not be translucent due to following reasons:

Too much sample/KBr powder

Poorly dispersed sample

Uneven distribution of powder in die

Moisture in the disc

Poor pressure or for too short time

All these faults can be remedied by regrinding the sample/KBr and by taking appropriate amounts of sample/KBr or by applying the correct pressure.

The disc turns brown due to sample being an oxidizing agent and checks the spectrum for halide degradation. The disc may have hole in it due to sticking of material that may be remedied by proper cleaning of die before sample preparation.

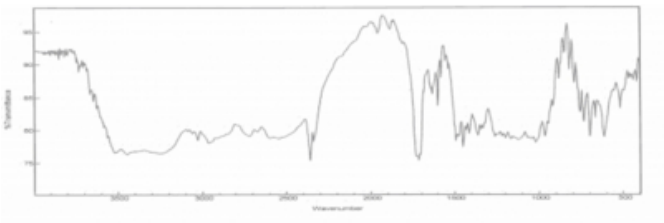
After that immediately place the KBr disc into KBr sample holder and place into sample compartment to generate the spectrum because moisture in air will cloud the disc.

Record the spectra and check for the following faults:

• If the transmittance does not reach to 10%, it means the sample is poorly dispersed or having hole in the disc (Figure 1).

Figure 1

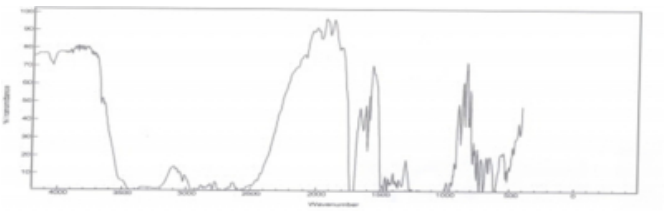
Figure 1: Too weak spectrum



• If too much sample is mixed with KBr, the spectrum will not be clear and transmittance may exceed 10 % (Figure 2).

Figure 2

Figure 2: Too strong spectrum



• Sometimes slopping baseline is obtained which may be due again poorly dispersed sample or the sample is too hard like a polymer which changes the refractive index and bands appear like first derivation and this effect is known is Christiansen effect.

If the standard method is used for preparing the disc as stated above will be transparent, and spectra will be correct (Figure 3) and can be compared with the standard spectrum (Figure 4).

Figure 3

Figure 3: Correct spectrum

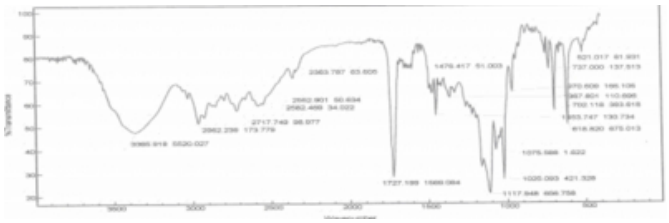
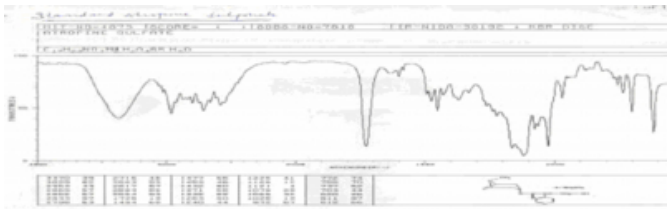


Figure 4

Figure 4: Standard spectrum



CONCLUSION

A standard procedure is to be followed for preparing KBr disc of the sample to be scanned by FTIR. Any deviations in the preparation of sample on account of quality, quantity of KBr, pressure used to make the disc and handling of die, press assembly etc. leads to poor quality of sample preparation affecting thereby the quality of FTIR spectra. The repetition of the quality of spectra especially from forensic angle can be obtained by following strictly the method recommended in this paper for the preparation of sample.

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