

Simple Methods to Measure Powder Samples

<Introduction>

In general for FT/IR, powder samples are measured by 1) KBr Pellet method, 2) Diffuse Reflection method, 3) Solution method, and 4) Microscope Transmission method. Each method has its own advantage and disadvantage and often needs some pretreatments or attachment for sample preparation in order to obtain good spectra. For example:

1. KBr Pellet method: Sample powder needs dilution with KBr and pressing with KBr pellet die and hydraulic press to make a pellet. Sometimes there may need also repeated pretreatments for the best concentration.
2. Diffuse Reflection method: There is no need of pressing but the dilution with KBr is required as KBr Pellet method. A measurement without dilution may cause the peaks reversed at the strong peaks.
3. Solution method: There may need some apparatus to dilute sample with some solvent, and pipettes to pour sample solution into liquid cell.
4. Microscopic Transmission method: There needs IR Microscope and since it adopts MCT detector, Liquid N₂ is required to chill the detector. Hydraulic press may be needed in order to press the powder sample sandwiched between KBr plates.

As mentioned above, usual measurements always need some sample pretreatments. Now the new procedure will be introduced, by which the sample can be placed on the sample compartment without dilution enabling to obtain good spectra.

<Measurement Method>

Existing Diffuse Reflection attachment (DR-81) was used for this measurement with improved optical system and sample cells to delete specular reflection. Sample was rubbed and diffused on the surface of sample cell for measurement. Now Phenacetin, which is generally hard to measure without KBr dilution, was measured.

<Results>

Figure 3 shows zoomed and expanded spectra of Figure 2. Some peaks were found to be reversed or shifted. Figure 4 shows spectra of KBr-diluted sample with existing cell, and of non-diluted sample with improved cell. The spectra show almost no peaks reversed or shifted. As mentioned above, the improved sample cell which has specifically processed surface helps to reduce specular reflection, while existing method is reducing such reflection with KBr-dilution, which proves that it is possible to obtain good quality spectra without KBr-dilution.

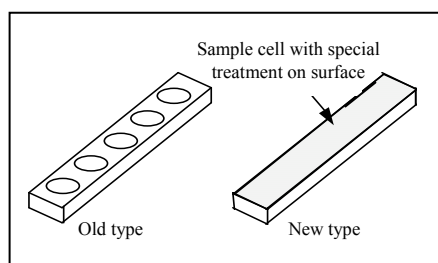


Figure 1. Sample cell for this method

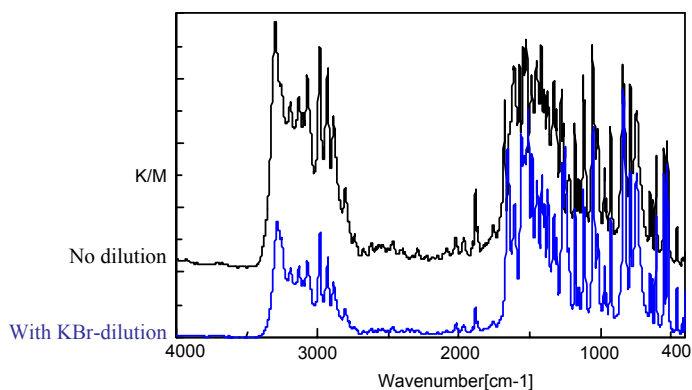


Figure 2. DR-spectra of Phenacetin (in existing sample cell)

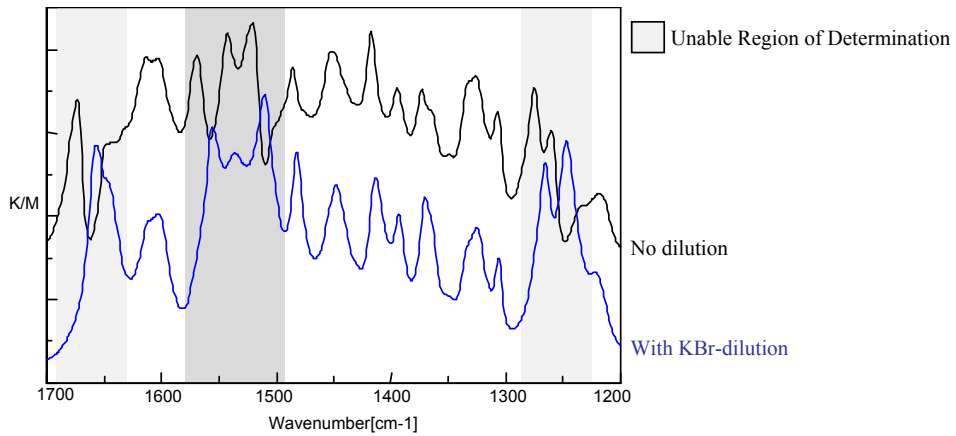


Figure 3. Zoomed and Expanded DR spectra of Phenacetin with existing sample cell

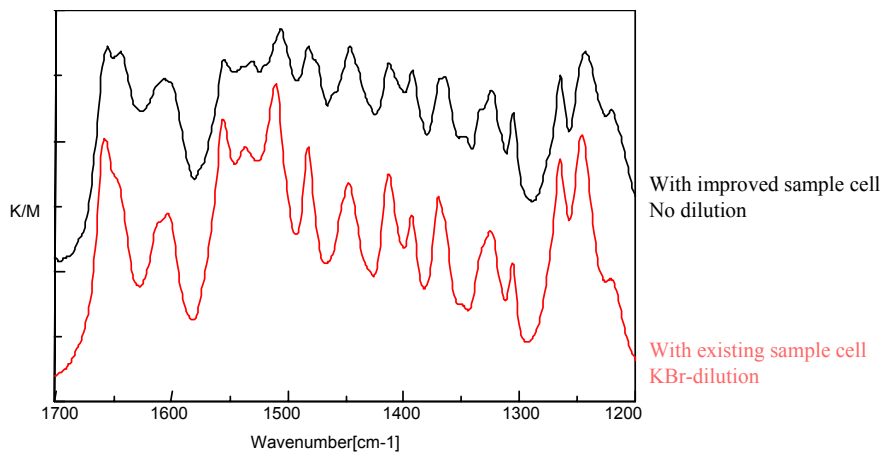


Figure 4. DR spectra of Phenacetin with improved and existing sample cell