Determination of High-Refractive Sample using FT/IR in Conjunction with Diffuse Reflection Accessory

The KBr pellet method and the diffuse reflection method are generally used to measure IR spectra of powdered samples. In particulare, the diffuse reflection method is used to measure inorganic materials. However, inorganic materials have a high refractive index, so the preparation of clear KBr pellets is quite difficult. Therefore, these samples are usually diluted using KBr powder or KCI powder in order to decrease their refractive index. For percent reflectance data, peaks usually form in the negative direction. However, peaks in the positive direction, similar to those commonly seen in derivative spectra, can be observed in the wavelength region where the strong absorption band of the sample is located. In addition, if specular reflection occurs in this wavelength region, the peaks in the positive direction are intensified. A custom-made dedicated integrating sphere was designed to gather all of the light occurring due to diffuse reflection while eliminating light occurring due to specular reflection. This method is very effective for measuring inorganic materials that have a high refractive index, such as powdered pigments, and plate samples, such as coatings, paper and synthetic resins.

Figure 1 shows the custom-made integrating sphere for FT/IR. The sphere uses a standard white plate as a reference. The sample is mounted under the integrating sphere. The stage enables the sample to be moved in the verticaldirection. Powdered sample is placed into the sample cell and measurementi is performed while the stage is in direct contact with the bottom of the integrating sphere. Plate samples, such as paper or coated film, are placed directly on the stage and measurement is performed while the stage is in direct contact with the bottom of the integrating sphere. The integrating sphere provides two primary advantages: radiation light occurring due to specular reflection is avoided and the need for sample preparation is eliminated. Thus, the integrating sphere enables various types of solid sample to be measured effortlessly. Figure 2 shows the spectrum for single beam energy obtained using the integrating sphere.

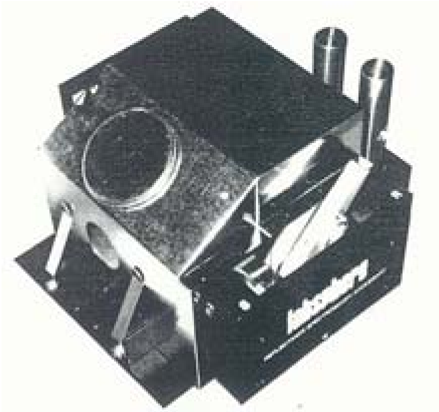

Fig.1 Custom-made dedicated integrating sphere

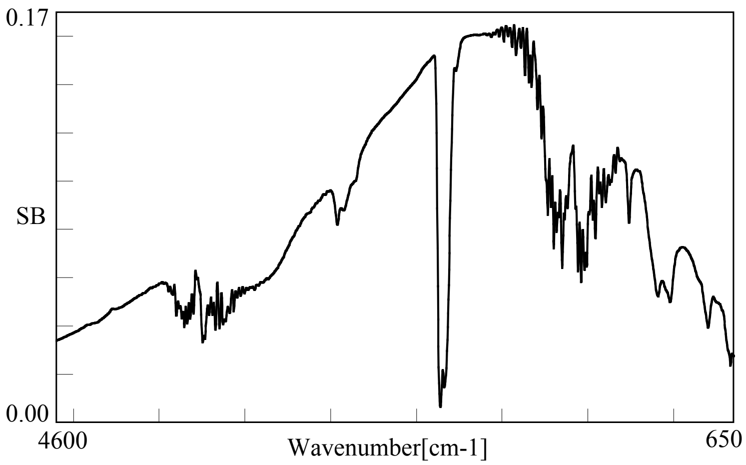

Fig.2 Single beam evergy

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Figure 3 shows the diffuse reflection spectra for Otawa-sand (20 micron) as an example which has high refractive index. Sample was measured without any dilution in order to keep particle size alignment in sample, because keeping particle size alignment is capable of avoiding the influence of specular reflection.
The intensities of specular reflection difference can be observed between the data using difference reflection accessories (DR-81, HDR-500) and using integrating sphere. The peaks in the positive direction can be seen around 1200-1100 cm^{-1} in these spectra, and these observed peaks mean that intensity of specular reflection becomes larger when these inverse direction peaks becomes stronger. The peaks in the positive direction in integrating sphere is much smaller than those of using DR-81, HDR-500, and the intensity of specular reflection seems to decrease much.

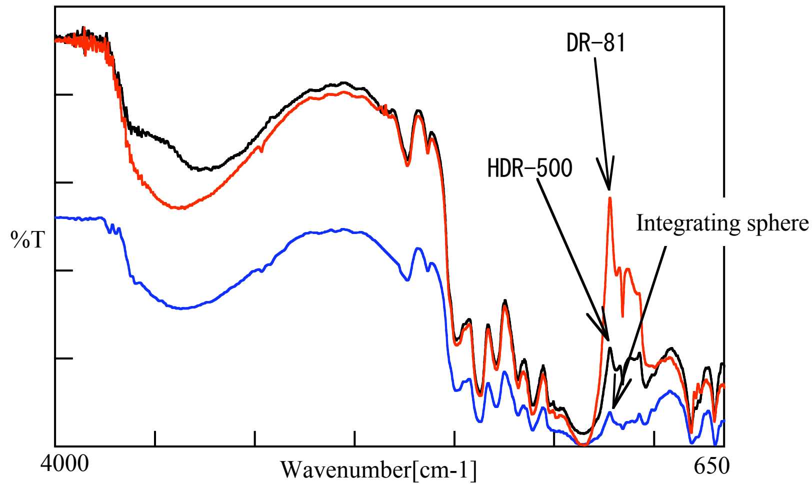

Fig.3 Diffuse spectra of Otawa-sand

Figure 4 shows another example of potassium permanganate ($KmnO_4$) with high refractive index. The peaks in the positive direction using integrating sphere become also much smaller than those of spectra data using diffuse reflection accessories.

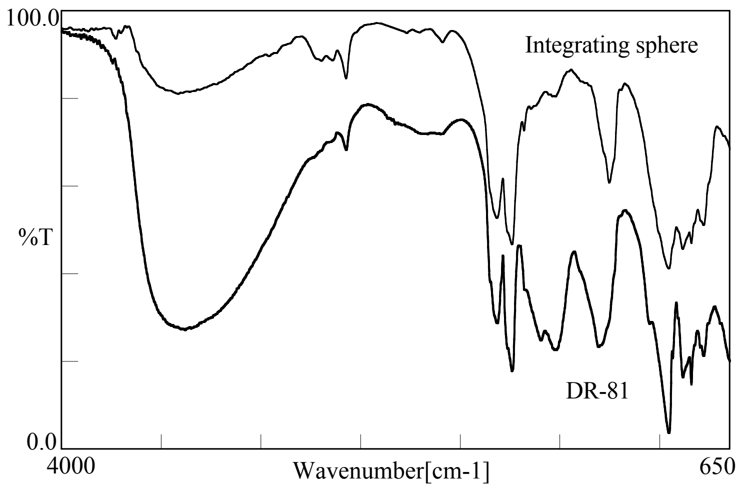

Fig.4 Diffuse spectra of $KmnO_4$