

EMISSIONLESS DIFFUSE REFLECTANCE IR SPECTROSCOPY
AT HIGH TEMPERATURE

Tadashi HATTORI, Miki NIWA, Mamoru TAKAHASHI, and Yuichi MURAKAMI
Department of Synthetic Chemistry, Faculty of Engineering,
Nagoya University, Furo-cho, Chikusa-ku, Nagoya 464

New diffuse reflectance spectrometer in the infrared region was made to eliminate the emission spectrum, and emissionless spectra of powder CaCO_3 were measured in the temperature range from room temperature to 450°C .

An application of infrared technique to the study of catalysis or surface science has been recently widespread in order to investigate surface and bulk structures of powder samples and adsorption phenomena on them. In the trans-*paren*ce ir method, however, much care is necessary in preparing the disk with a good trans-*paren*ce. Powder diffuse reflectance spectroscopy in the infrared region is recommended to avoid the defect.^{1, 2)} Furthermore, measurements of ir spectra at a high temperature are very valuable to know the structure and the behavior of powder samples more precisely. However, such spectra were sometimes disturbed or cancelled out by emission spectra, particularly in the region of low wave number. The emissionless diffuse reflectance ir spectrometer, which was produced by Japan Spectroscopic Co. Ltd. on the basis of new ideas by the authors, allowed us to measure ir spectra at high temperatures.

This instrument is designed to eliminate the emission spectrum, as shown in Fig. 1. Double beams from a light source pass alternatively through sample and reference parts to a half-mirror, and enter the optical system after reflection or passing. Although emissions from both sample and reference parts always enter the optical system, reflected and passed beams go into it only alternatively.

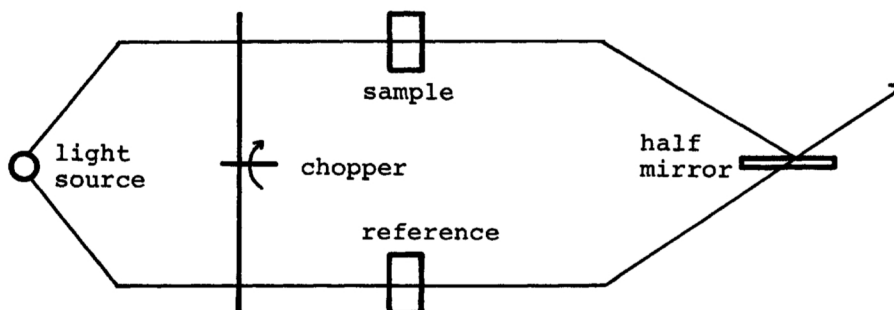
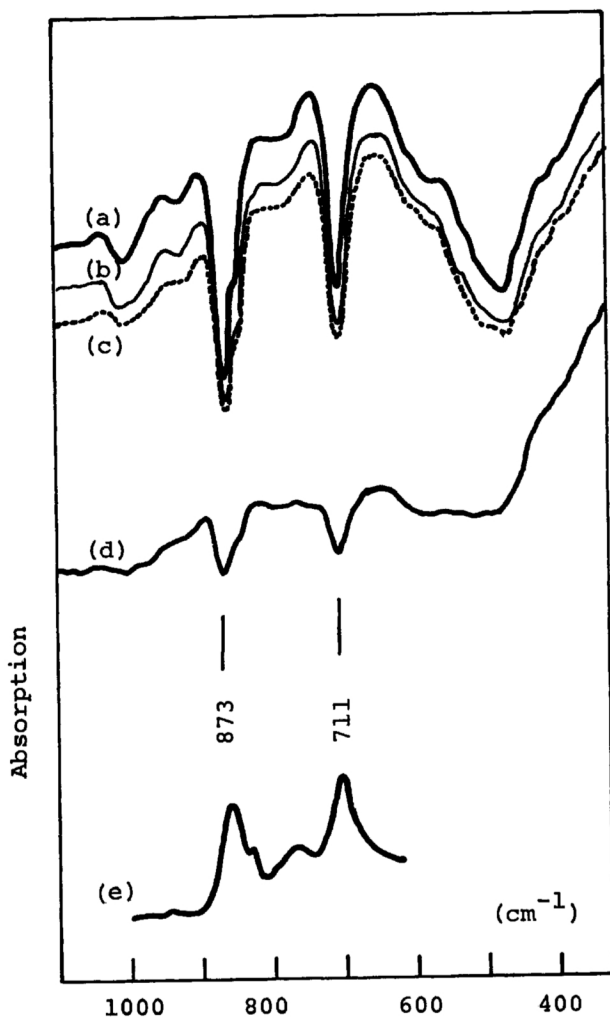


Fig. 1. Basic unit for emissionless diffuse reflectance ir spectrometer.



The emissionless spectrum is then recorded as the difference between these double beams.

Fig. 2 shows diffuse reflectance ir spectra of powder CaCO_3 diluted with KBr (85 wt% of diluent was mixed.) at room temperature (a), 350°C (b), and 450°C (c) and of non-diluted CaCO_3 at 450°C (d) in the region below 1100 cm^{-1} at vacuum (10^{-5} torr). Furthermore, an emission spectrum from CaCO_3 heated at 110°C (e), which was exactly taken as a mirror image of spectra (a)~(d), was shown as an example of the previous diffuse reflectance measurement. The bands at 711 and 873 cm^{-1} were attributed to characteristic absorptions of carbonate ions.^{3, 4} Intensities of the absorption were nearly independent of the measurement temperatures. Sharp absorptions were observed even at high temperatures without disturbance of the emission. By comparison with spectrum (d) of non-diluted CaCO_3 at 450°C , it was clear that the dilution made the absorption distinct.

Fig. 2. Diffuse reflectance ir spectra of powder CaCO_3 .

REFERENCES

- (1) W. W. Wendlandt, "Modern Aspect of Reflectance Spectroscopy," Plenum Press, New York (1968).
- (2) W. W. Wendlandt and H. C. Hecht, "Reflectance Spectroscopy," Interscience Publishers, New York/London/Sydney (1966).
- (3) F. A. Miller, G. L. Carlson, F. F. Bentley, and W. H. Jones, *Spectrochim. Acta*, **16**, 135 (1960).
- (4) M. Mamiya, *Hyomen*, **7**, 45 (1969).

(Received October 5, 1977)