

Analysis of Colored Components on the Surface of Tablets

What are detected as foreign matters on the surface of tablets may be discolored surface as well as resin particles or waste threads attached on the surface. In the earlier edition of Shimadzu Application News (No. 339), we introduced an example of analysis of foreign

matters adhering to the surface by the microscopic ATR method. Here we introduce an example of analysis of colored components on the surface of tablets.

■ Colored Components on Tablets

Discoloration of tablet surface may be caused not only by the color change of the tablet itself, but also by contamination of colored components. The photograph of a tablet with partially yellow discoloration that was used in this analysis is shown in Fig.1. The discolored area looks like permeated into the tablet. Furthermore, the irregularity of the surface of the tablet makes measurement difficult by the use of ATR method, which requires close contact. For this reason, as a preprocessing step, we scraped off the discolored portion and analyzed it by two techniques, i.e., single reflection ATR and transmission microscopy.



Fig.1 Photograph of Contaminants on Tablet

■ Measurement by Single Reflection ATR Method

The discolored and nondiscolored areas of the tablet were scraped away and analyzed using the single reflection ATR accessory (MIRacle). The analytical conditions were listed in Table 1. The spectra of the discolored and nondiscolored (normal part) sample were shown in Fig.2 (red line = discolored, blue line = nondiscolored). The intensity of the spectrum of the discolored sample is considerably low compared to that of nondiscolored due to the small quantity of discolored sample available for analysis. In Fig.2, the spectra's intensities are adjusted in the way such that the intensities of both spectra become almost the same. The result shows the spectrum of the discolored sample overlaps that of nondiscolored. In this case, we need to calculate the subtracting spectrum of the two.

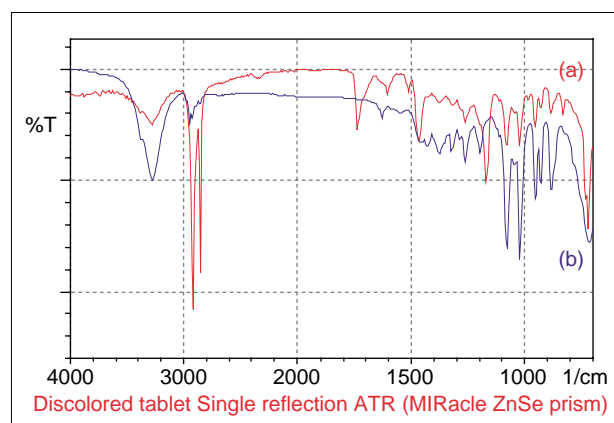


Fig.2 Spectra of Contaminant (a) (discolored) and Normal (b) (nondiscolored) Parts on Tablet by Single Reflection ATR Method

Table 1 Analytical Conditions

Resolution	: 4cm ⁻¹
Accumulation	: 60
Detector	: DLATGS

The subtracting spectrum between the two spectra is shown in Fig.3. And search result of Fig.3 is revealed in Fig.4. The result indicates that the discoloring component on the tablet is carnauba wax. The carnauba wax is a natural wax widely used in lipstick, foundation and as an additive in foods.

It is normally quite difficult to scrape away only discolored section as this case. In addition, spectra of discolored and nondiscolored are overlapped, due to the fact that the single reflection ATR method gives an averaged spectrum on a prism. However, as we have shown, we can obtain the spectrum of discolored part by the subtracting spectrum of nondiscolored spectrum.

■ Measurement by Transmission Microscopy

The same foreign matter was taken from the tablet and spread thinly on a diamond cell. The sample was then measured using the transmission microscopic method. Table 2 lists the analytical conditions. Fig.5 shows an enlarged photograph of the discolored scraping after it has been spread thinly on the diamond cell. Fig.6 shows the spectrum of the 30 by 30 micrometers size sample. In contrast with the spectrum obtained using the single reflection ATR method (Fig.2), we can see that the spectrum of the nondiscolored part does not overlap with that of the discolored one. The spectrum of nondiscolored part often overlaps the spectrum of the discolored one in microscope measurement. In this measurement, however, the spectrum of discolored part is obtained without the subtracting manipulation.



Fig.5 Micrograph of Scraped Contaminants

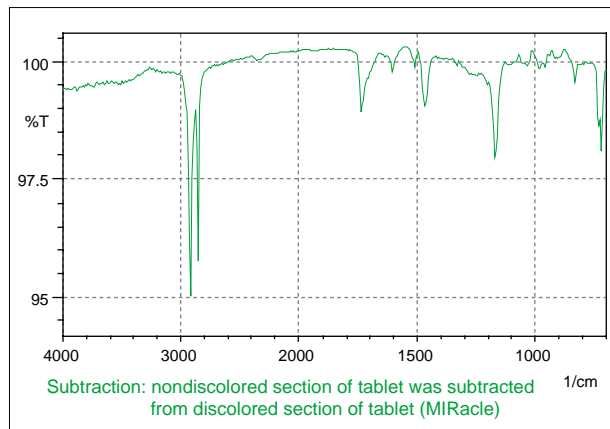


Fig.3 Subtraction Spectrum

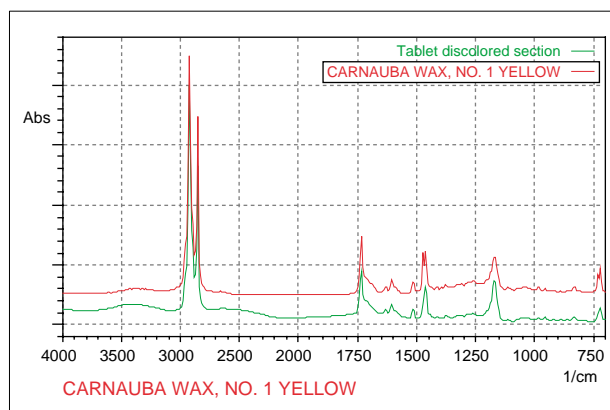


Fig.4 Search Results of Fig.3

Table 2 Analytical Conditions

Resolution	: 8cm ⁻¹
Accumulation	: 60
Detector	: MCT

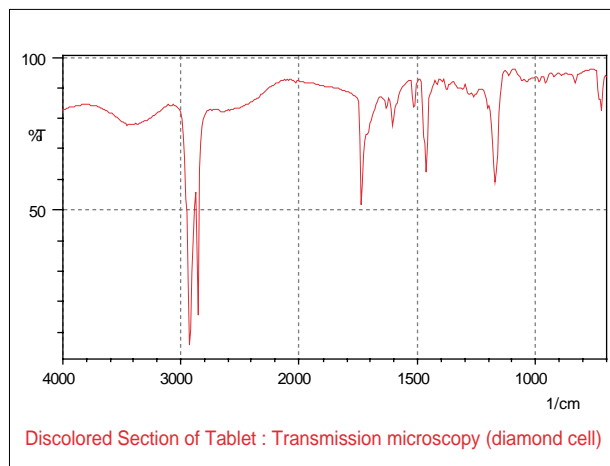


Fig.6 Spectrum of Contaminants on Tablet by Transmission Microscopy Method