

Application News

No. A514

Spectrophotometric Analysis

Introducing Area Imaging with AIM-9000 and AIMsolution

AIMsolution control software for the AIM-9000 infrared microscope comes with an optional mapping program. Measurements performed with the mapping program integrate seamlessly with normal point measurements, and involve simple configuration of sample image capture, measurement size, aperture size, and measurement pitch all in the same window. The mapping program provides detailed analysis of microscopic sample regions by performing area measurements to analyze the in-plane distribution of components, and line measurements useful for depth-based sample analysis.

An example application is described here that uses the AIMsolution mapping program to perform area measurements.

■ Measurement Sample

A transmission method was used to take area mapping measurements of a pharmaceutical powder that contains a number of components. The sample was first inserted into a diamond cell and compressed to a thickness appropriate for transmission measurements. After compression, the diamond cell was installed on the stage, and area measurements were performed.

A visible image of the entire sample is shown in Fig. 1. The visible image shows areas of different colors in the sample, indicating it is probably a mixture of a number of components.

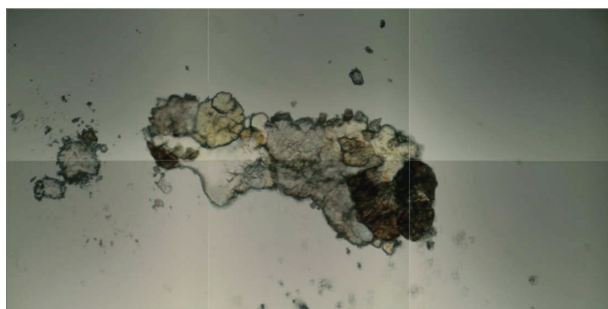


Fig. 1 Visible Image of Sample

Sample measurements were performed in an area of 375 μm (vertical) by 600 μm (horizontal). The measurement area is shown in Fig. 2. A 25 μm by 25 μm aperture size was set at each measurement position. The blue lattice shows the aperture of each measurement position, with the measurement positions being selected to leave no gaps. The yellow box is a background measurement position. Aperture size for background measurements is set automatically to the same aperture size used for sample measurements. Measurements were performed with the parameters shown in Table 1.

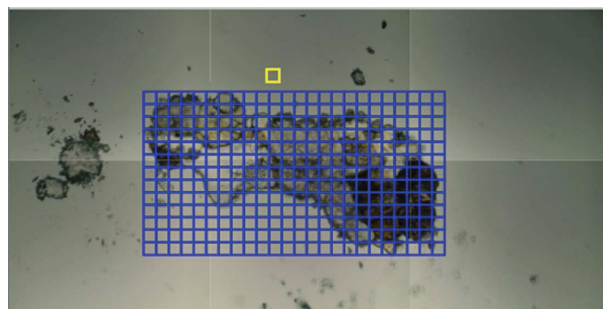


Fig. 2 Image Displayed after Measurement Area Selection

Table 1 FTIR Measurement Conditions

Instrument	: IRTTracer-100 / AIM-9000
Resolution	: 8 cm^{-1}
Accumulation	: 2
Apodization	: Sqr-Triangle
Aperture Size	: 25 μm \times 25 μm
Step	: 25 μm
Detector	: MCT

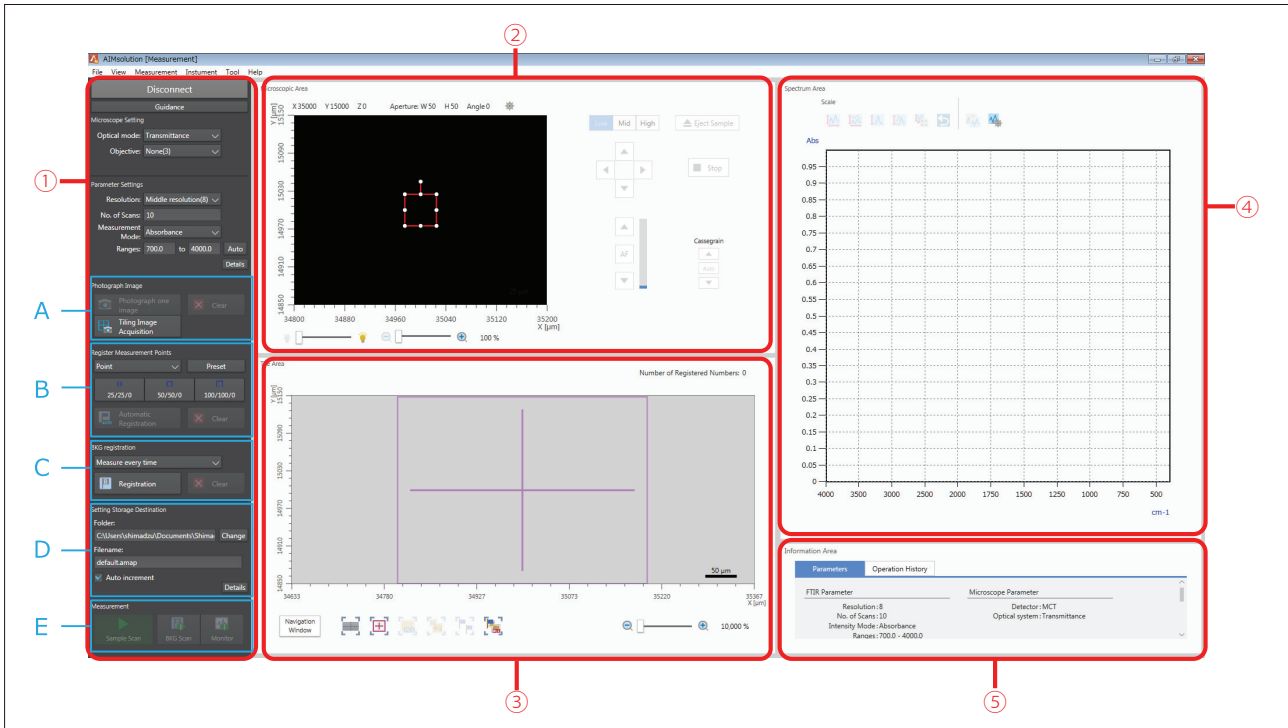


Fig. 3 Measurement Program Configuration Window

■ Measurement Procedure

The AIMsolution measurement program window is shown in Fig. 3. The window is divided into five areas.

- (1) [Measurement Parameter Configuration Area]
Parameters are configured here.
- (2) [Microscope Area]
Displays microscopic images. Stage movement and focal adjustment are also performed with buttons in this area.
- (3) [Tile Area]
Displays images captured by the microscope side-by-side.
(Displaying images side-by-side is called tiling.)
Specifying positions for area measurement is also mainly performed in this area.
- (4) [Spectrum Area]
Displays spectra.
- (5) [Information Area]
Information on configured parameters and logs are displayed in this area.

The microscope can be controlled and all necessary information can be viewed in a single window.

A flowchart of the measurement procedure is shown in Fig. 4.

The AIMsolution mapping program is designed to make configuration of area measurements extremely easy. The measurement procedure is very simple, with configuration performed in order of sections A to E of the measurement parameter configuration area.

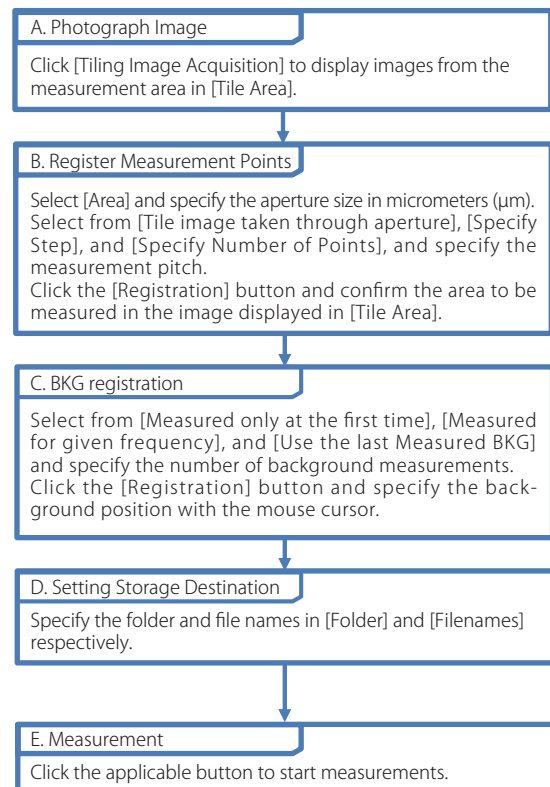


Fig. 4 Flowchart of Sample Measurement

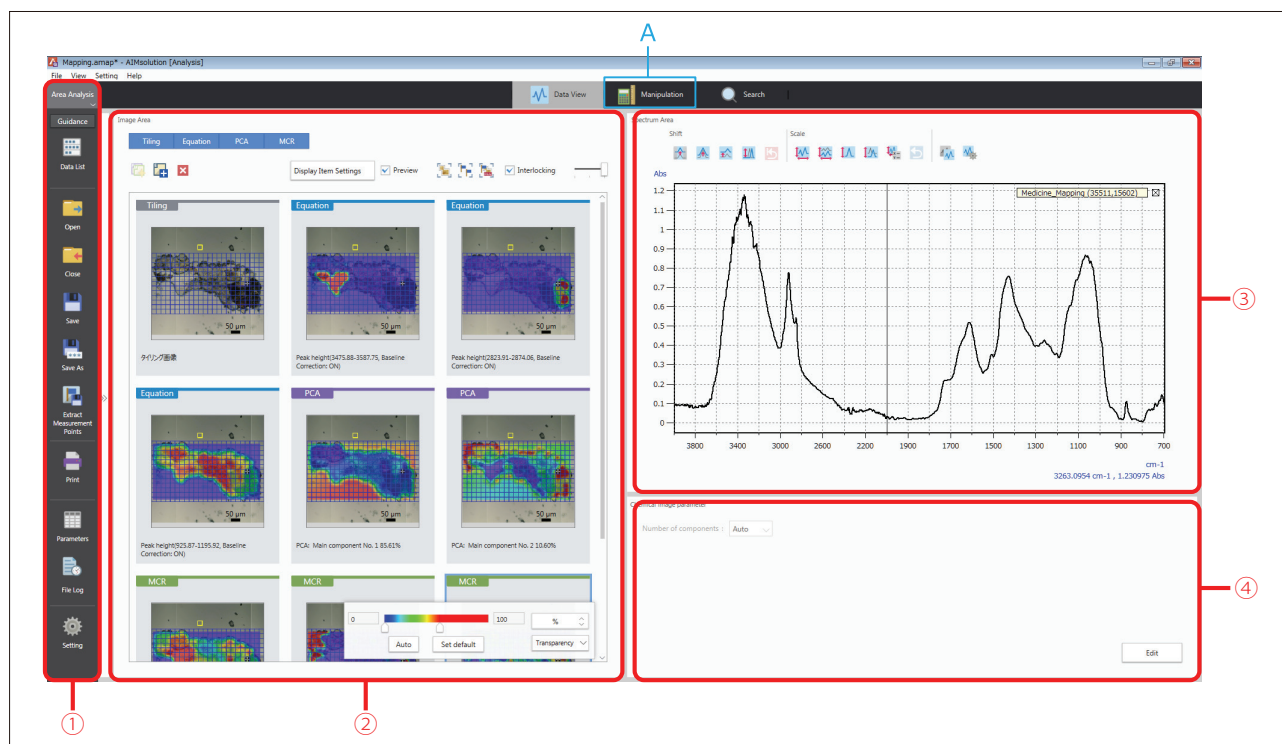


Fig. 5 Area Data Analysis Program Window

■ Area Data Analysis (Data Analysis of Area Measurement Results)

Analysis of the area measurement results is performed by the Area Data Analysis part of the analysis program. A view of the Area Data Analysis screen is shown in Fig. 5.

The data analysis screen is divided into 4 areas.

- (1) [Toolbar]
Contains tool icons.
- (2) [Image Area]
Displays tiling images and the results of data analysis (results of calculations).
The results of data analysis are called chemical imaging.
- (3) [Spectrum Area]
Displays spectra, including measurement point spectra, multivariate analysis loading spectra, and component spectra.
- (4) [Chemical Imaging Parameters]
Displays parameters used during data analysis (computation), and allows them to be edited.
Wavelength range can be specified for peak height and peak area, and the number of factors for multivariate analysis.

To perform preprocessing of measurement data, change windows by clicking the [Manipulation] button in Fig. 5 (A). Preprocessing included in the software are baseline correction, atmospheric correction, advanced ATR Correction, Kramers-Kronig analysis, and difference (compute difference spectra).

In the default settings, the results of area measurement data analysis are displayed as chemical imaging, with large numbers represented in red and small numbers in blue.

Computing formula factors that can be used to create chemical imaging are as follows: Intensity at specified position, relative intensity at specified position, peak height, relative peak height, peak area, relative peak area, and degree of coincidence.

Other than computing formulae, chemical imaging can also be created using multivariate analysis. Multivariate analyses available to the user are principal component analysis (PCA) and multivariate curve resolution (MCR). PCA and MCR data analyses can be implemented with a single click, without the need to configure complicated parameters.

An example of chemical imaging is shown in Fig. 6. The image in Fig. 6 is created using OH group peak height in the vicinity of 3500 cm^{-1} . The image shows that the left portion of the sample contains a large amount of material with OH groups. Chemical imaging allows distributions to be visually displayed in a way that is easy to understand.

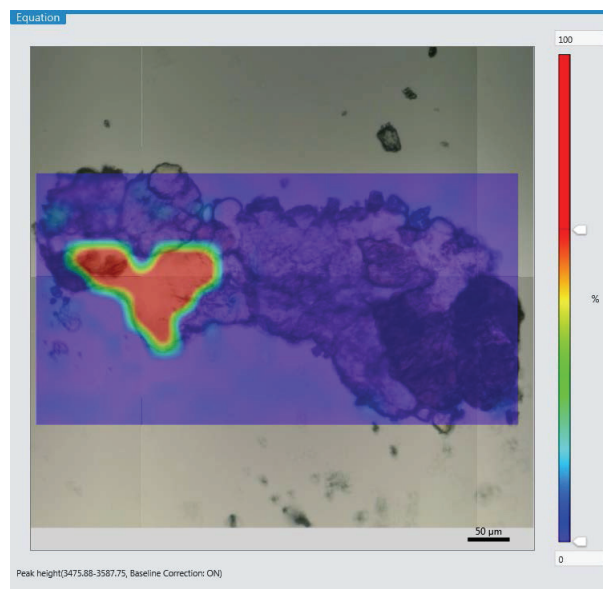


Fig. 6 An Example of Chemical Imaging

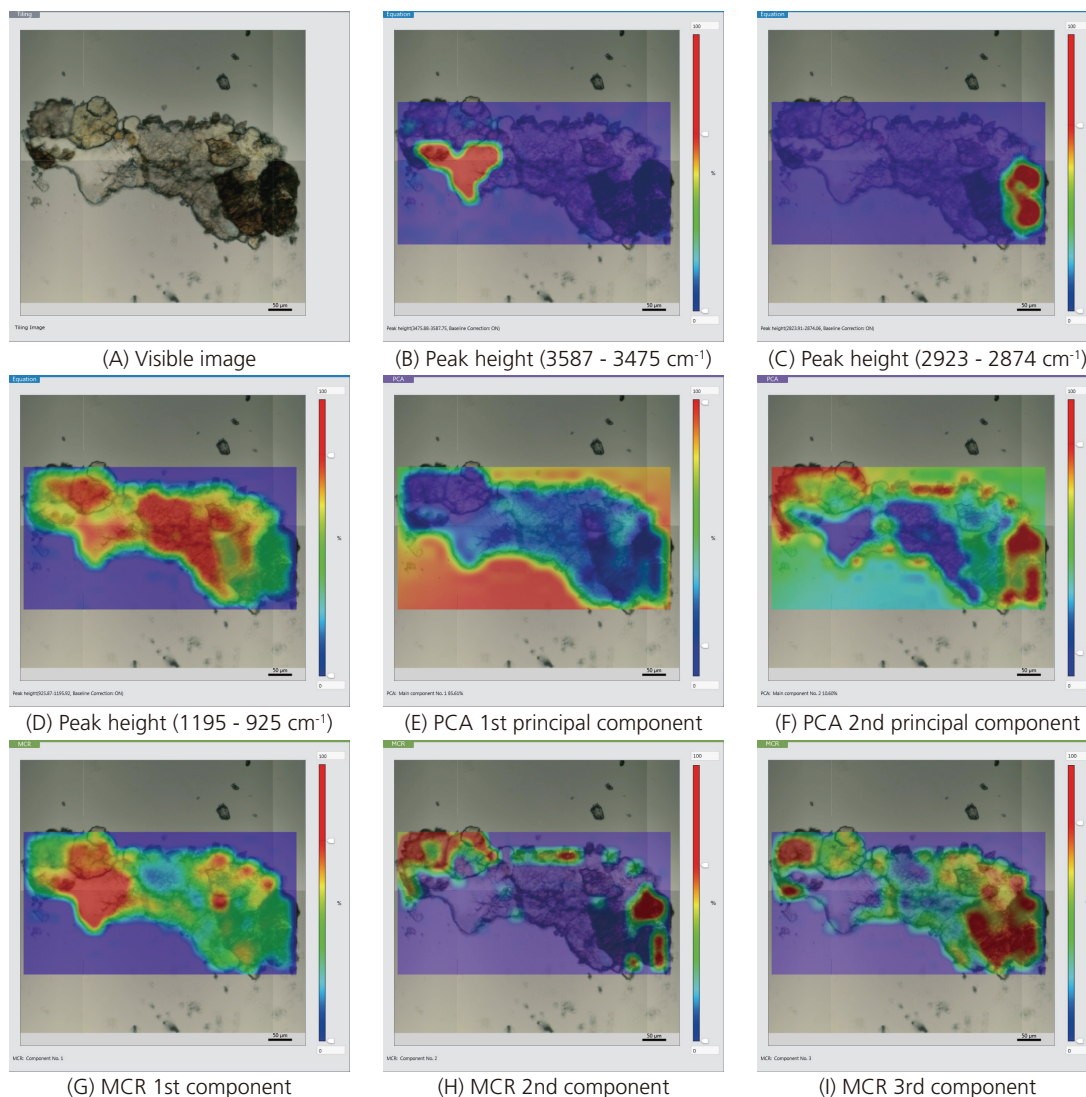


Fig. 7 Chemical Imaging of Sample

Results of Data Analysis

Chemical imaging obtained from the measured sample using several techniques is shown in Fig. 7. From the top left: (A) visible image, (B) peak height at 3587-3475 cm^{-1} , (C) peak height at 2923-2874 cm^{-1} , (D) peak height at 1195-925 cm^{-1} , (E) PCA 1st principal component, (F) PCA 2nd principal component, (G) MCR 1st component, (H) MCR 2nd component, and (I) MCR 3rd component.

The chemical imaging in Fig. 7 displays OH group

absorption in (B), CH group absorption in (C), CO group absorption in (D), and difference from the mean of all sample data in (E). While (D) and (G) show different concentration levels in the sample, some components seem to be present throughout the sample. Based on (C), (F), (H), and (I), it is highly likely that a component present on the right end of the sample is different from those in the other portions.

Conclusion

Performing area measurements with AIMSolution allows for a diverse array of analytical results as images, including the results of multivariate analyses such as PCA and MCR in addition to peak height and peak area.

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