SHIMADZU
Excellence in Science

Application News

DSC-60A Plus Differential Scanning Calorimeter
DTG-60A Simultaneous Thermogravimetry/Differential Thermal Analyzer

Measurement and Automatic Analysis of Resin Using Autosampler and Template Function

Atsushi Kawaguchi

User Benefits

- Use of an autosampler makes it possible to automate the series of processes from measurement to analysis, report preparation, and outputting.
- Automatic analysis using the template function can also support analysis of data measured using multiple temperature programs.
- The autosampler can respond flexibly to addition of samples and urgent samples, even after the start of measurement.
- The autosampler can also be added after purchase of the instrument.

■ Introduction

Thermal analysis instruments such as the DSC differential scanning calorimeter and TG-DTA simultaneous thermogravimetry/differential thermal analyzer are used to evaluate the physical properties of a wide range of specimens, including polymer materials, pharmaceuticals, and inorganic materials. When measuring a large number of samples, for example, in quality control applications, stable analysis is possible and productivity can be improved by using an autosampler.

This article introduces a resin measurement using the DSC-60A Plus thermal analyzer equipped with an autosampler. With an autosampler mounted on the DSC-60A Plus or DTG-60A, automatic measurement of a maximum of 24 samples is possible. By using the template and output functions, it is also possible to automate the entire series of processes from measurement to analysis, report preparation, and outputting. As shown in Fig. 1, the autosampler for these instruments is housed inside the instrument itself, which also contributes to space-saving.

Fig. 1 Appearance of (Left) DSC-60A Plus and (Right) DTG-60A
(The red boxes show the autosamplers.)

■ Analysis Samples

Polypropylene pellets were used in these measurements. The samples were cut to 1 to 2 mm squares, 5 mg was weighed and placed in the aluminum crimp cell for the autosampler, and the samples were measured after setting the lid. A total of 3 cells of the same type were prepared and used in the autosampler measurements. Fig. 2 shows the appearances of a polypropylene sample after cutting and the aluminum crimp cell for the autosampler, and Table 1 shows the measurement conditions.

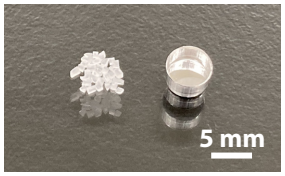


Fig. 2 Appearances of (Left) Polypropylene after Cutting and (Right) Aluminum Crimp Cell of Autosampler

Table 1 Measurement Conditions

Instrument	: DSC-60A Plus
Cell	: Aluminum crimp cell for autosampler
Heating and cooling rate	: 20 ˚C/min
Temperature conditions	: Room temperature → 200 ˚C → 25 ˚C → 200 ˚C
Atmosphere	: Nitrogen 50 ml/min

■ Measurement of Three Polypropylene Samples Using Autosampler

In measurements using an autosampler, it is necessary to input the measurement conditions in a dedicated table on the LabSolutions™ TA software. In addition to general items such as the temperature program and file information, the "Insert/Remove Temperature," "Post Processing," and "Output" conditions can also be set.

In the "Insert/Remove Temperature" function shown in Fig. 3, the "Sample Insert Temperature" and "Sample Remove Temperature" conditions are input. As an example, if the "Sample Insert Temperature" is 25 ˚C ±10 ˚C for 1 minute, the instrument temperature is adjusted to the range of 15 ˚C to 35 ˚C when measurement of the next sample starts, and after 1 minute, the next cell to be measured is inserted from the tray into the sensor. "Sample Remove Temperature" is the same condition as "Sample Insert Temperature" when removing the cell from the device. When the aimed change occurs at a high temperature, data around room temperature are not necessary in some cases. By using these items, the next measurement can be started without waiting for the instrument to return to room temperature, shortening the waiting time between measurements and improving productivity.

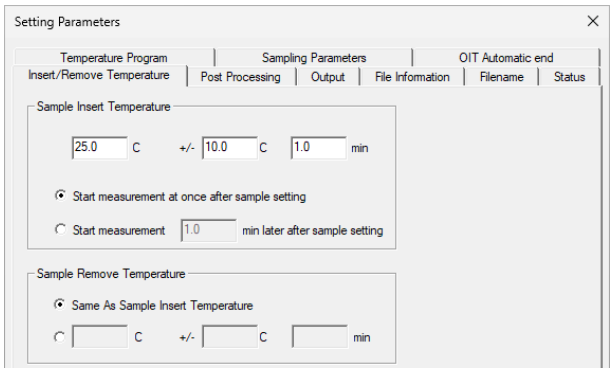


Fig. 3 Insert/Remove Temperature" Setting Screen

The "Post Processing" function, shown in Fig. 4, is used to set the "Signal Shift," "Axis Unit," and "Template" conditions. When "Starting point is zero" is selected for "Signal Shift," an offset is applied to the data after measurement, and the starting point becomes 0. This makes it possible to align the starting points of the data from all measurements. "Axis Unit" allows the user to set the data units on the X-axis and Y-axis. "Template" is a condition related to automatic revision (data processing) and automatic analysis of the data. Data for similar samples which were previously measured and corrected or analyzed is searched by using "Template." When "Perform Data Processing" or "Perform Analysis" is checked, the revisions or analysis in the searched data are all automatically applied to the data when measurement is completed.

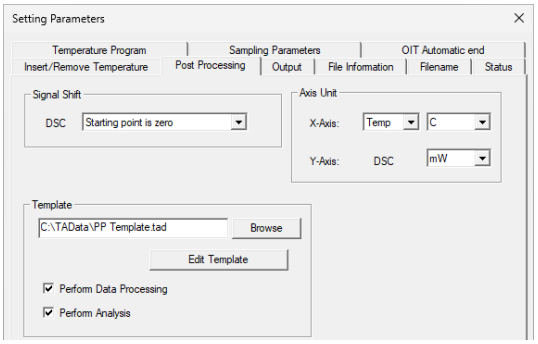


Fig. 4 "Post Processing" Setting Screen

Checking "Print" or "Save ASCII file" of the "Output" function, shown in Fig. 5, makes it possible to automatically print the data after measurements or automatically convert the data to text data. Using this function in combination with the template function makes it possible to carry out all the processes from measurement to analysis, report preparation, and outputting automatically.

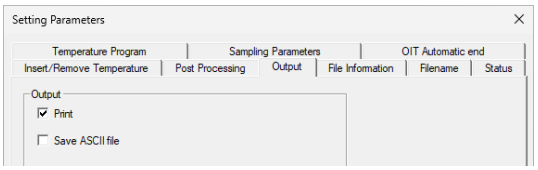


Fig. 5 "Output" Setting Screen

Fig. 6 shows the data for the polypropylene sample after analysis using the template function in this measurement. The same sample as in this measurement was measured by the same program as in Table 1, and the temperature indication is shown on the x-axis. In measurements involving repeated heating and cooling, as in this experiment, if the temperature indication is shown on the x-axis, the obtained DSC curve will normally form a loop with three heating and cooling curves still connected. However, LabSolutions TA excludes the low- and high-temperature ends of the looped DSC curve and divides the display into three DSC curves, which show the heating curve twice and the cooling curve once, making it possible to analyze each of the curves.

In this figure, the first heating data show the peak temperature of the melting peak, the first cooling data show the peak temperature of the crystallization peak, and the second heating data show the peak temperature of the melting peak and the heat of melting in the temperature range from 120 ˚C to 180 ˚C.

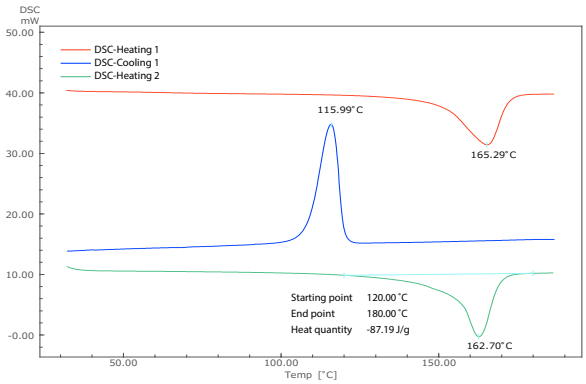


Fig. 6 DSC Curves of Polypropylene after Analysis Using Template Function

Figs. 7 to 9 show the results of the three measurements with the autosampler.

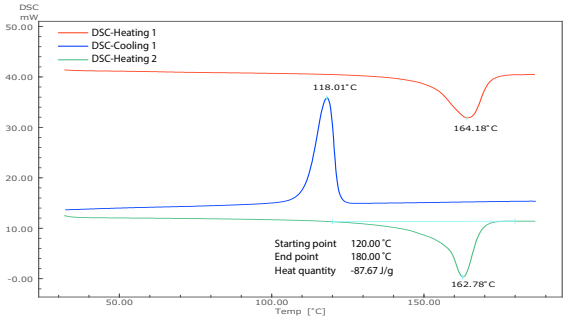


Fig. 7 Results of 1st Measurement
*Only the vertical positions of the curves were adjusted manually.

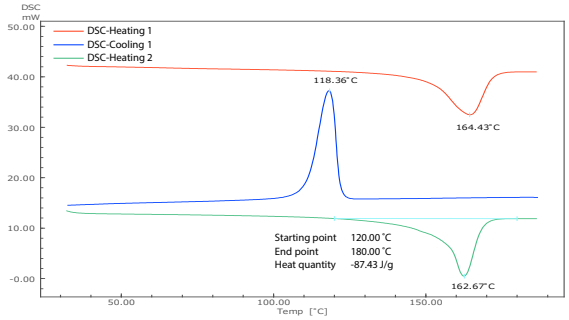


Fig. 8 Results of 2nd Measurement
*Only the vertical positions of the curves were adjusted manually.

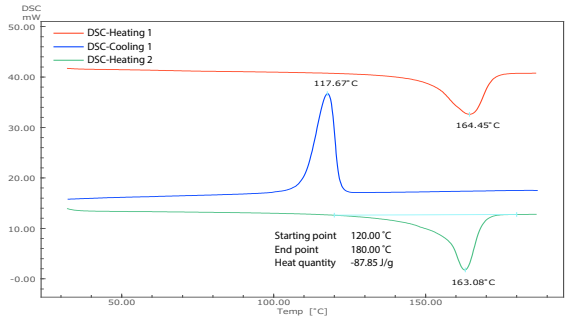


Fig. 9 Results of 3rd Measurement
*Only the vertical positions of the curves were adjusted manually.

As shown in Figs. 7 to 9, by using the template function, it was possible to divide the obtained DSC curves into three curves, in the same manner as in Fig. 6, and analyze the respective curves separately, and to align the appearance of the curves uniformly by automatically changing the colors of the curves, the display of the legend, and other features. A check of the peak temperatures and the heat of melting also shows that good reproducibility was possible in the three measurements. Thus, by measurement with the autosampler and use of the template function, DSC measurements can be conducted with good reproducibility, and the flow of the processes through data analysis can be carried out efficiently.

■ Conclusion

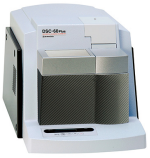
Measurements of three polypropylene samples were carried out using the autosampler of the DSC-60A Plus differential scanning calorimeter. By using the template function of the LabSolutions TA program, it was possible to divide the data obtained by repeated heating and cooling into three curves, and analyze the data automatically. Use of the template function together with other functions such as the output function can improve productivity in DSC and TG-DTA measurements.

01-00990-EN First Edition: Aug. 2025

SHIMADZU
Shimadzu Corporation
www.shimadzu.com/an/

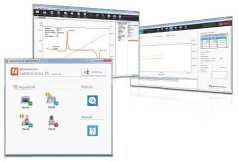
› Please fill out the survey

Related Products Some products may be updated to newer models.



› DSC-60 Plus Series
Differential Scanning Calorimeter

› DTG-60 Series



› LabSolutions TA
Workstation Software for Thermal Analyzer

Related Solutions

› Hydrocarbon Processing Industry (Petrochemical, Ch

› Price Inquiry

› Product Inquiry

› Technical Service / Support Inquiry

› Other Inquiry